

Asymmetric aldol reaction via memory of chirality

Hidetoshi Watanabe, Tomoyuki Yoshimura, Shimpei Kawakami, Takahiro Sasamori, Norihiro Tokitoh, and Takeo Kawabata*

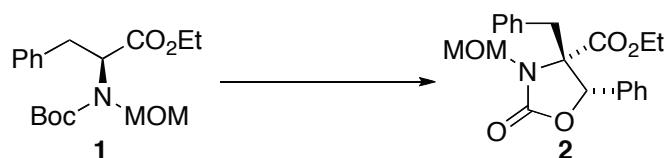
Institute for Chemical Researches, Kyoto University

Electronic Supplementary Information

General Procedure	2
General Procedure for Table 1	2
Synthesis of compound 3	5
Synthesis of compound 4	5
Synthesis of compound 5	6
Synthesis of compound 6	7
Synthesis of compound 7	8
Synthesis of compound 8	9
Conversion of 2 into 10	10
ORTEP representation of 10	11
Synthesis of compound 11	11
Aldol reaction between 11 and benzaldehyde	12
Aldol reaction between 13 and benzaldehyde	13
Spectral data	15

General Procedure. ^1H NMR were measured in CDCl_3 solution and referenced from TMS (0.00 ppm) using JEOL ECX-400 or JEOL AL-400 (400 MHz) spectrophotometers, unless otherwise noted. ^{13}C NMR were measured in CDCl_3 solution and referenced to CDCl_3 (77.5 ppm) using JEOL ECX-400 or JEOL AL-400 (100 MHz) spectrophotometers, unless otherwise noted. Chemical shifts are reported in ppm. When peak multiplicities are reported, the following abbreviations are used: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broadened. IR spectra were recorded on JASCO FT/IR-4200 spectrometer. Mass spectra were obtained on JEOL JMS-700. Elemental analyses were performed with CHN J-science-lab. Microcoder JM10. Optical rotations were determined on HORIBA SEPA-200. Melting points were measured with Yanagimoto Micro Melting Point Apparatus PM-500 and are uncorrected. Enantiomeric excess was determined by HPLC analysis using Shimadzu LC-10AS liquid chromatograph with Chiralpak AD-H or Chiralcel OD-H as chiral stationary phase. Flash column chromatography was performed on Silica Gel (SilicaFlash[®] 60F₂₄₅) and compounds were visualized with UV light and *p*-anisaldehyde stain, phosphomolybdic acid stain or ninhydrin stain. Preparative thin layer chromatography was performed on precoated plates (0.5 mm, silica gel Merck Kieselgel 60F₂₄₅) and visualized with UV light. All reactions were conducted in oven-dried glassware under argon atmosphere. Dehydrated solvents were purchased from Kanto Kagaku or Wako Chemicals and pre-treated with activated MS4Å for 1 day or longer.

General Procedure for Table 1



Procedure I

KHMDS (1.1 eq.) was added to a solution of **1** (1.0 eq.) in toluene/THF at -78°C . After being stirred for 30 min, benzaldehyde (3.0 eq) was added dropwise at same temperature. The resulting mixture was stirred for 2.5 hr and poured into sat. NH_4Cl aq. and extracted with AcOEt . The extracts were washed with sat. NaHCO_3 aq. and brine, and dried over Na_2SO_4 , filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/ Et_2O =1/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=4/1) to obtain **2**.

Procedure II

A solution of **1** (1.0 eq.) was added to a solution of benzaldehyde (3.0 eq.) and KHMDS (1.1 eq.) in toluene/THF at the temperature shown in Table 1. After being stirred for 10 min, the mixture was poured into sat. NH_4Cl aq. and extracted with AcOEt . The extracts were washed with sat. NaHCO_3 aq. and brine, and dried over Na_2SO_4 , filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/ Et_2O =1/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=4/1) to obtain **2**.

Procedure III

Benzaldehyde (0.13 mL, 1.25 mmol) was added to a mixture of **1** (84 mg, 0.25 mmol) in toluene (1.5 mL) at rt and the mixture was cooled to the temperature shown in Table 1. KHMDS (0.37-0.41 M in toluene, 1.83-2.02 mL, 0.75 mmol) was added to this solution and the resulting mixture was stirred for the time shown in Table 1 at same temperature. The reaction mixture was poured into sat. NH_4Cl aq. and extracted with AcOEt . The

extracts were washed with sat. NaHCO_3 aq. and brine, and dried over Na_2SO_4 , filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/ Et_2O =1/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=4/1) to obtain **2**.

2:(4S, 5S)-Ethyl 4-benzyl-3-(methoxymethyl)-2-oxo-5-phenyloxazolidine-4-carboxylate

Colorless oil; $[\alpha]_D^{20} +2.8$ (*c* 1.0, CHCl_3 , 87% ee); ^1H NMR (400 MHz, CDCl_3): δ 7.45–7.26 (m, 10H), 5.38 (s, 1H), 4.83 (ABq, $\Delta\nu_{AB}=0.32$ Hz, $J=11.6$ Hz, 2H), 3.71 (q, $J=7.2$ Hz, 2H), 3.52 (ABq, $\Delta\nu_{AB}=0.031$ Hz, $J=15.6$ Hz, 2H), 3.47 (s, 3H), 0.94 (t, $J=7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.7, 158.5, 134.4, 134.3, 131.4, 129.3, 129.2, 128.6, 128.0, 126.3, 78.9, 76.3, 73.1, 62.4, 57.6, 37.4, 13.8; IR (neat) cm^{-1} : 2935, 1774, 1739, 1496, 1455, 1374, 1233, 1078, 1028, 744, 701; MS (EI) m/z (rel intensity) 369 (M^+ , 5), 338 (10), 296 (40), 278 (100), 220 (20), 202 (40), 158 (20), 130 (20), 91 (60); HRMS (EI) m/z Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_5$ (M^+): 369.1576 Found 369.1581; Anal. Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_5$: C, 68.28; H, 6.28; N, 3.79 Found: C, 68.02; H, 6.36; N, 3.77; HPLC conditions: Daicel Chiralcel OD-H, hexane:2-propanol=95:5, flow 0.5 mL/min, $t_R=32$ (4*R*,5*R*), 37 (4*S*,5*S*) min.

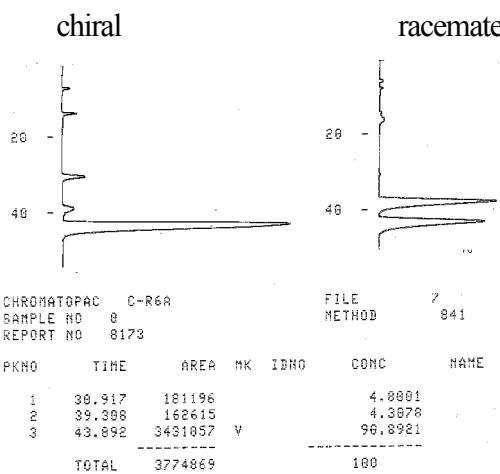
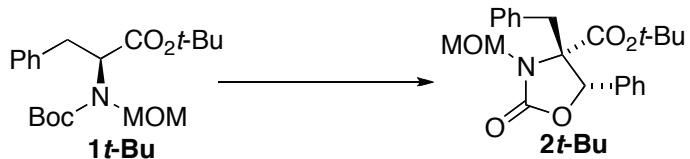


Table 1, entry 13



To a solution of **1t-Bu** (91 mg, 0.25 mmol) and benzaldehyde (0.13 mL, 1.25 mmol) in $t\text{-BuOMe}$ (1.5 mL) was added dropwise KHMDS (0.37 M in PhMe, 2.02 mL, 0.75 mmol) at -50°C . After being stirred for 12 hr, the reaction mixture was poured into sat. NH_4Cl aq. and the resulting mixture was extracted with AcOEt . The extracts were washed with sat. NaHCO_3 aq. and brine, and dried over Na_2SO_4 , filtered and concentrated. The residue was chromatographed on silica gel (hexane/ Et_2O =1/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=4/1) to give **2t-Bu** (31 mg, 31%, 89% ee).

2t-Bu:tert-Butyl 4-benzyl-3-(methoxymethyl)-2-oxo-5-phenyloxazolidine-4-carboxylate

Colorless oil; $[\alpha]_D^{20} +1.9$ (*c* 1.0, CHCl_3 , 86% ee); ^1H NMR (400 MHz, CDCl_3): δ 7.43–7.30 (m, 10H), 5.38 (s, 1H), 4.82 (ABq, $\Delta\nu_{AB}=0.27$ Hz, $J=11.6$ Hz, 2H), 3.50 (s, 2H), 3.48 (s, 3H) 1.07 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3): δ 168.5, 158.7, 135.0, 134.7, 131.5, 129.3, 129.1, 128.8, 128.0, 126.4, 83.8, 78.6, 76.4, 73.0, 57.8, 38.1, 27.6. IR (neat) cm^{-1} : 2930, 1771, 1732, 1454, 1370, 1251, 1081, 1040, 745, 701. MS (EI) m/z (rel intensity) 397 (M^+ , 1), 306 (60), 296 (100), 250 (60), 220 (30), 174 (25), 130 (20), 91 (40), 57 (10); HRMS (EI) m/z Calcd for $\text{C}_{23}\text{H}_{27}\text{NO}_5$ (M^+): 397.1889 Found 397.1875; Anal. Calcd for $\text{C}_{23}\text{H}_{27}\text{NO}_5$: C, 69.50; H, 6.85; N, 3.52%. Found: C, 69.29; H, 7.02; N, 3.49%; HPLC conditions: Daicel Chiralcel OJ-H, hexane: 2-propanol=85:15, flow 0.6 mL/min, $t_R=14$ (major), 22 (minor) min.

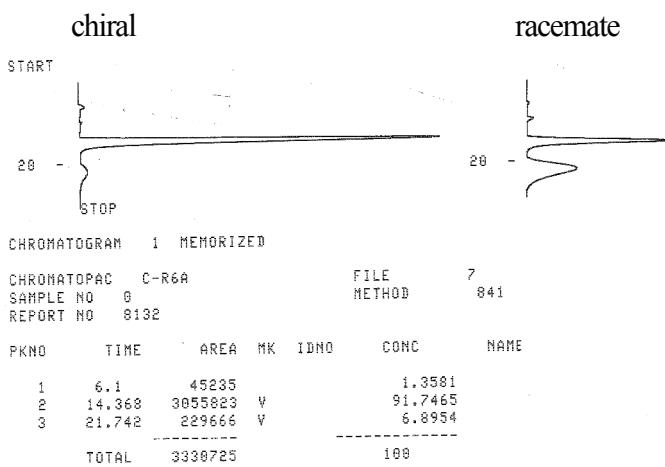
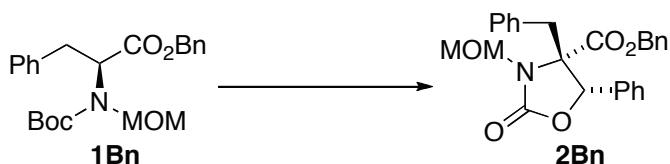


Table 1, entry 14



To a solution of **1Bn** (100 mg, 0.25 mmol) and benzaldehyde (0.13 mL, 1.25 mmol) in *t*-BuOMe (1.50 mL) was added dropwise KHMDS (0.30 M in PhMe, 2.50 mL, 0.75 mmol) at -50 °C. After being stirred for 12 hr, the reaction mixture was poured into sat. NH₄Cl aq. and the resulting mixture was extracted with AcOEt. The extracts were washed with sat. NaHCO₃ aq. and brine, and dried over Na₂SO₄, filtered and concentrated. The residue was chromatographed on silica gel (hexane/Et₂O=1/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=4/1) to give **2Bn** (106 mg, 99%, 82% ee).

2Bn:Benzyl 4-benzyl-3-(methoxymethyl)-2-oxo-5-phenyloxazolidine-4-carboxylate

Colorless oil; [α]_D²⁰ +22 (*c* 1.0, CHCl₃, 91% ee); ¹H NMR (400 MHz, CDCl₃): δ 7.42-7.24 (m, 13H), 7.12-7.09 (m, 2H), 5.39 (s, 1H), 4.80 (ABq, Δv_{AB}=0.33 Hz *J*=11.6 Hz, 2H), 4.63 (ABq, Δv_{AB}=0.035 Hz *J*=12.4 Hz, 2H) 3.52 (ABq, Δv_{AB}=0.024 Hz *J*=15.6 Hz, 2H) 3.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 169.7, 158.5, 134.6, 134.2, 134.2, 131.5, 129.4, 129.3, 128.9, 128.8, 128.8, 128.1, 126.4, 79.1, 76.4, 73.4, 68.3, 57.7, 37.4; IR (neat) cm⁻¹: 3064, 2934, 1770, 1742, 1455, 1374, 1078, 910, 698; MS (EI) *m/z* (rel intensity) 431 (M⁺, 1), 400 (5), 340 (100), 296 (60), 220 (50), 130 (20); HRMS (EI) *m/z* Calcd for C₂₆H₂₅NO₅ (M⁺): 431.1733 Found 431.1725 Anal. Calcd for C₂₆H₂₅NO₅: C, 72.37; H, 5.84; N, 3.25%. Found: C, 72.13; H, 5.90; N, 3.06%; HPLC conditions: Daicel Chiralcel OD-H, hexane: 2-propanol=90:10, flow 0.6 mL/min, *t*_R=27 (major), 33 (minor) min.

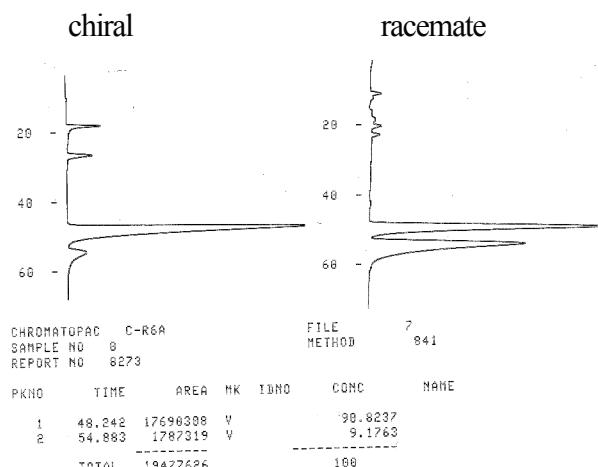
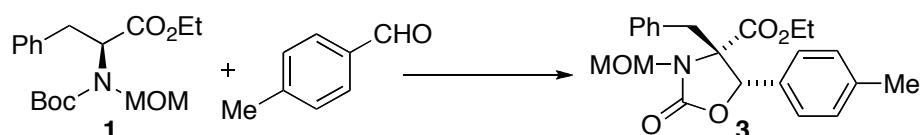


Table 2, entry 2



p-Tolualdehyde (0.15 mL, 1.25 mmol) was added to a solution of **1** (84 mg, 0.25 mmol) in *t*-BuOMe (1.5 mL) and the mixture was cooled to -60 °C and KHMDS (0.40 M in toluene, 1.88 mL, 0.75 mmol) was added slowly. After being stirred for 12 hr at same temperature, the reaction mixture was poured into sat. NH₄Cl aq. and extracted with AcOEt and the extracts were washed with sat. NaHCO₃ aq. and brine, and dried over Na₂SO₄, filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/AcOEt=7/1) to give an oil, which was further purified by PTLC (hexane/AcOEt=3/1) to give **3** (65 mg, 67%, 88% ee).

3:Ethyl 4-benzyl-3-(methoxymethyl)-2-oxo-5-*p*-tolyloxazolidine-4-carboxylate

Colorless oil; [α]_D²⁰ -9.9 (*c* 1.1, CHCl₃, 86% ee); ¹H NMR (400 MHz, CDCl₃): δ 7.44 -7.31 (m, 5H), 7.15 (s, 4H), 5.34 (s, 1H), 4.82 (ABq, Δv_{AB}=0.32 Hz *J*=11.5 Hz, 2H), 3.75 (q, *J*=7.3 Hz, 2H), 3.49 (ABq, Δv_{AB}=0.034 Hz *J*=15.6 Hz, 2H), 3.46 (s, 3H), 2.33 (s, 3H), 0.98 (t, *J*=7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 169.9, 158.8, 139.3, 134.5, 131.6, 131.4, 129.4, 129.3, 128.1, 126.4, 79.2, 76.5, 73.3, 62.5, 57.7, 37.5, 21.6, 14.0; IR (neat) cm⁻¹: 2930, 1773, 1740, 1373, 1232, 1039, 704; MS (EI) *m/z* (rel intensity) 383 (M⁺, 5), 351 (10), 310 (20), 292 (100), 234 (20), 216 (90), 144 (30), 91 (50); HRMS (EI) *m/z* Calcd for C₂₂H₂₅NO₅ (M⁺): 383.1733 Found 383.1739 Anal. Calcd for C₂₂H₂₅NO₅: C, 68.91; H, 6.57; N, 3.65 Found C, 68.70; H, 6.53; N, 3.56; HPLC conditions: Daicel Chiralcel OD-H, hexane:2-propanol=93:7, flow 0.8 mL/min, *t*_R=13 (minor), 16 (major) min.

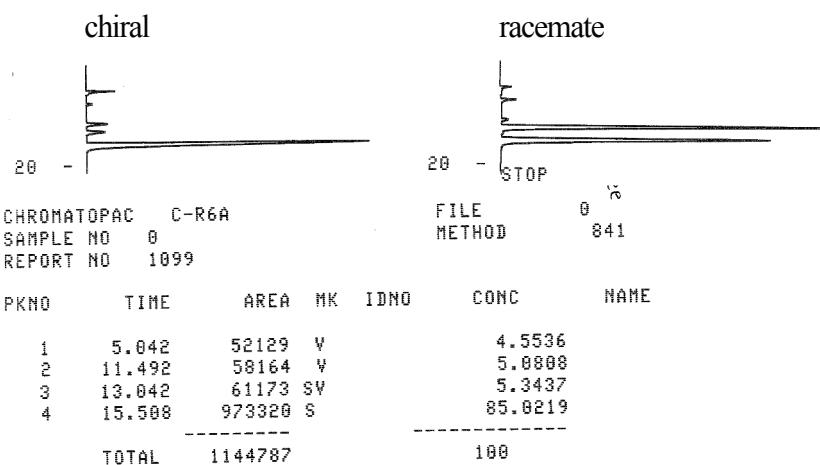
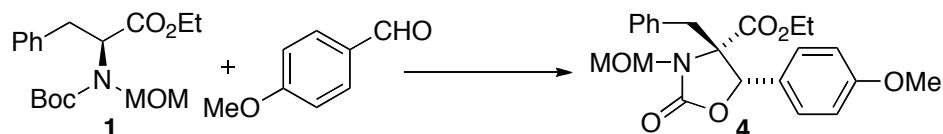


Table 2, entry 3



p-Anisaldehyde (0.15 mL, 1.25 mmol) was added to a solution of **1** (84 mg, 0.25 mmol) in *t*-BuOMe (1.5 mL) and the mixture was cooled to -60 °C and KHMDS (0.38 M in toluene, 1.97 mL, 0.75 mmol) was added slowly. After being stirred for 6 hr at same temperature, the reaction mixture was poured into sat. NH₄Cl aq. and extracted with AcOEt and the extracts were washed with sat. NaHCO₃ aq. and brine, and dried over Na₂SO₄,

filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/AcOEt=7/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=4/1) to obtain **4** (66 mg, 66%, 88% ee).

4:Ethyl 4-benzyl-3-(methoxymethyl)-5-(4-methoxyphenyl)-2-oxooxazolidine-4-carboxylate

Colorless oil; $[\alpha]_D^{20} -14$ (*c* 1.1, CHCl₃, 87% ee); ¹H NMR (400 MHz, CDCl₃): δ 7.44-7.30 (m, 5H), 7.18 (d, *J*=8.8 Hz, 2H), 6.86 (d, *J*=8.8 Hz, 2H), 5.33 (s, 1H), 4.82 (ABq, $\Delta\nu_{AB}=0.32$ Hz *J*=11.2 Hz, 2H), 3.78 (s, 3H), 3.77 (q, *J*=7.2 Hz, 2H), 3.48 (ABq, $\Delta\nu_{AB}=0.031$ Hz *J*=15.6 Hz, 2H), 3.46 (s, 3H), 1.01 (t, *J*=7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 169.9, 160.4, 158.6, 134.4, 131.5, 129.2, 128.0, 127.8, 126.2, 114.0, 79.1, 76.4, 73.3, 62.5, 57.6, 55.6, 37.5, 14.0; IR (neat) cm⁻¹: 2935, 1769, 1741, 1516, 1254, 1032, 704; MS (EI) *m/z* (rel intensity) 399 (M⁺, 40), 308 (100), 264 (90), 232 (90), 191 (30), 151 (30), 135 (20); HRMS (EI) *m/z* Calcd for C₂₂H₂₅NO₆ (M⁺): 399.1682 Found 399.1681; Anal. Calcd for C₂₂H₂₅NO₆: C, 66.15; H, 6.31; N, 3.51 Found C, 65.85; H, 6.38; N, 3.28; HPLC conditions: Daicel Chiralpak AD-H, hexane:2-propanol=85:15, flow 1.0 mL/min, *t_R*=33 (minor), 40 (major) min.

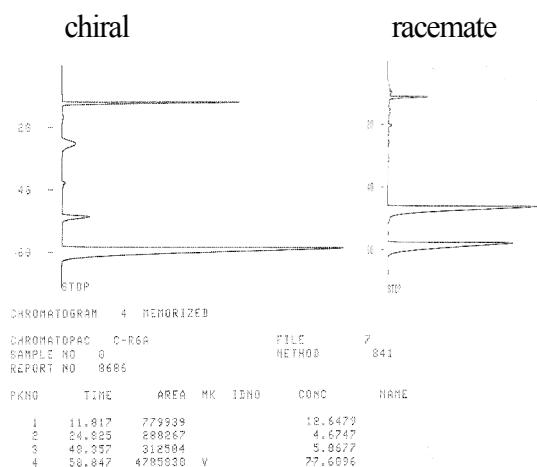
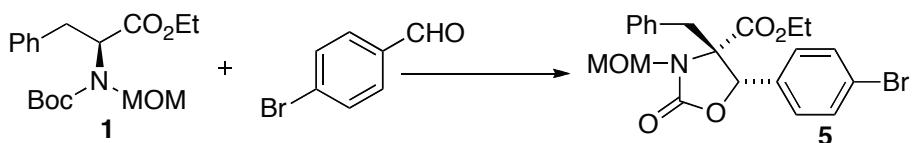


Table 2, entry 4



A mixture of **1** (84 mg, 0.25 mmol) and *p*-bromobenzaldehyde (231 mg, 1.25 mmol) was dissolved in toluene (4.5 mL) and the resulting mixture was cooled to -50 °C. KHMDS (0.37 M in *t*-BuOMe, 2.03 mL, 0.75 mmol) was added dropwise slowly to this solution. After being stirred for 6 hr at same temperature, the reaction mixture was poured into sat. NH₄Cl aq. and extracted with AcOEt. The extracts were washed with sat. NaHCO₃ aq. and brine, and dried over Na₂SO₄, filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/1,4-dioxane=5/1) to give an oil, which was further purified by PTLC (hexane/AcOEt=3/1) to obtain **5** (72 mg, 64%, 78% ee).

5:Ethyl 4-benzyl-5-(4-bromophenyl)-3-(methoxymethyl)-2-oxooxazolidine-4-carboxylate

Colorless oil; $[\alpha]_D^{20} -23$ (*c* 1.1, CHCl₃, 78% ee); ¹H NMR (400 MHz, CDCl₃): δ 7.47 (d, *J*=8.2 Hz, 2H), 7.42-7.33 (m, 5H), 7.12 (d, *J*=8.2 Hz, 2H), 5.32 (s, 1H), 4.83 (ABq, $\Delta\nu_{AB}=0.30$ Hz *J*=11.4 Hz, 2H), 3.77 (q, *J*=6.8 Hz, 2H), 3.49 (ABq, $\Delta\nu_{AB}=0.024$ Hz *J*=15.6 Hz, 2H), 3.42 (s, 3H), 0.99 (t, *J*=6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 169.7, 158.3, 134.2, 133.6, 131.9, 131.5, 129.4, 128.3, 128.1, 123.5, 78.4, 76.4, 72.9, 62.7, 57.8, 37.5, 14.0; IR (neat) cm⁻¹: 2935, 1773, 1739, 1373, 1232 1081, 1040, 704; MS (EI) *m/z* (rel intensity) 449

(M⁺, ⁸¹Br, 3), 447 (M⁺, ⁷⁹Br, 3), 376 (15), 374 (20), 358 (95), 356 (100), 282 (20), 280 (20), 91 (80); HRMS (EI) m/z Calcd for C₂₁H₂₂BrNO₅ (M⁺): 447.0681 Found 447.0682; Anal. Calcd for C₂₁H₂₂BrNO₅: C, 56.26; H, 4.95; N, 3.12 Found C, 56.51; H, 5.17; N, 3.06; HPLC conditions: Daicel Chiralcel OD-H, hexane: 2-propanol=90:10, flow 0.7 mL/min, t_R=18 (minor), 22 (major) min.

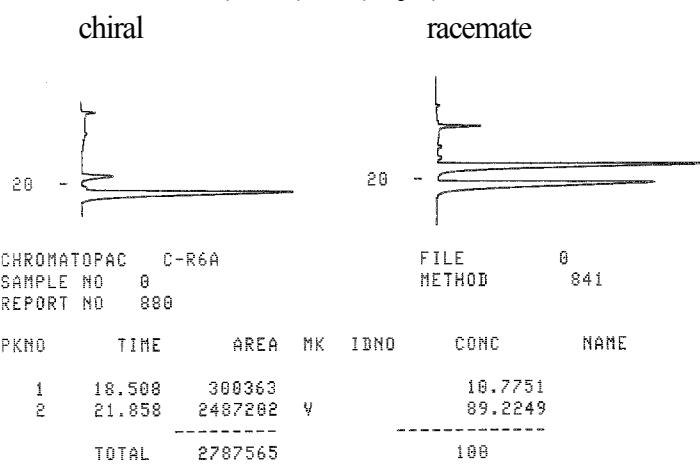
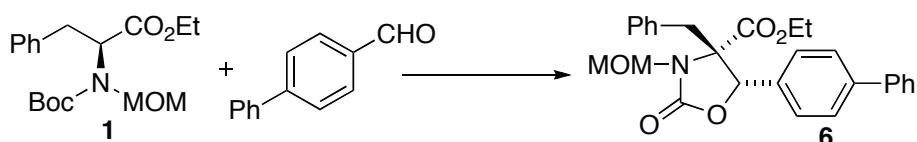


Table 2, entry 5



A mixture of **1** (84 mg, 0.25 mmol) and *p*-phenylbenzaldehyde (228 mg, 1.25 mmol) was dissolved in toluene (4.5 mL) and the resulting mixture was cooled to -50 °C. KHMDS (0.37 M in *t*-BuOMe, 2.03 mL, 0.75 mmol) was added slowly. After being stirred for 12 hr at same temperature, the reaction mixture was poured into sat. NH₄Cl aq. and extracted with AcOEt. The extracts were washed with sat. NaHCO₃ aq. and brine, and dried over Na₂SO₄, filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/AcOEt=6/1) to give an oil, which was further purified by PTLC (hexane/AcOEt=4/1) to obtain **6** (106 mg, 95%, 80% ee).

6:Ethyl 4-benzyl-5-(biphenyl-4-yl)-3-(methoxymethyl)-2-oxooxazolidine-4-carboxylate

Colorless oil; [α]_D²⁰ -58 (c 1.0, CHCl₃, 85% ee); ¹H NMR (400 MHz, CDCl₃): δ 7.59-7.55 (m, 4H), 7.47-7.32 (m, 10H), 5.43 (s, 1H), 4.85 (ABq, Δv_{AB}=0.31 Hz J=11.4 Hz, 2H), 3.75 (q, J=7.3 Hz, 2H), 3.54 (ABq, Δv_{AB}=0.043 Hz J=15.1 Hz, 2H), 3.48 (s, 3H), 0.95 (t, J=7.3 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 169.9, 158.6, 142.3, 140.6, 134.4, 133.5, 131.6, 129.4, 129.3, 128.2, 128.1, 127.5, 127.4, 126.9, 79.0, 76.5, 73.3, 62.6, 57.8, 37.6, 14.0; IR (neat) cm⁻¹: 2933, 1772, 1739, 1373, 1233, 1082, 1038, 764, 700; MS (EI) m/z (rel intensity) 445 (M⁺, 10), 354 (100), 310 (20), 278 (95), 165 (30), 91 (40); HRMS (EI) m/z Calcd for C₂₇H₂₇NO₅ (M⁺): 445.1889 Found 445.1882; Anal. Calcd for C₂₇H₂₇NO₅: C, 72.79; H, 6.11; N, 3.14 Found C, 72.59; H, 6.30; N, 3.06; HPLC conditions: Daicel Chiralpak AD-H, hexane:2-propanol=80:20, flow 1.0 mL/min, t_R=20 (minor), 47 (major) min.

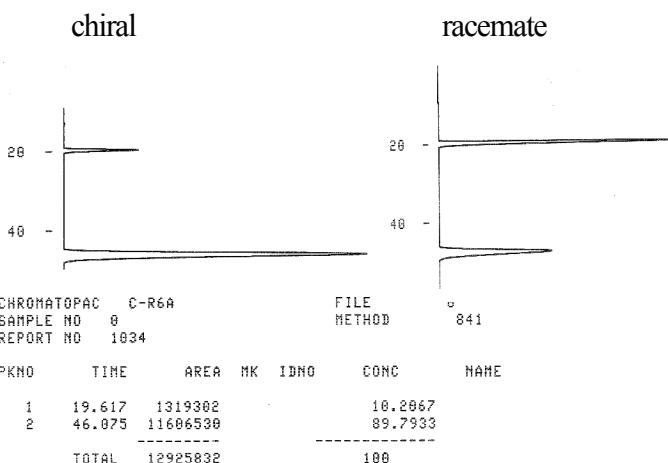
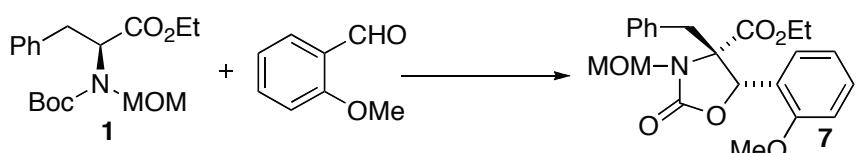


Table 2, entry 6



o-Anisaldehyde (0.15 mL, 1.25 mmol) was added to a solution of **1** (84 mg, 0.25 mmol) in *t*-BuOMe (1.5 mL) and the mixture was cooled to -50 °C and KHMDS (0.44 M in toluene, 1.70 mL, 0.75 mmol) was added slowly. After being stirred for 12 hr at same temperature, the reaction mixture was poured into sat. NH₄Cl aq. and extracted with AcOEt. The extracts were washed with sat. NaHCO₃ aq. and brine, and dried over Na₂SO₄, filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/AcOEt=7/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=7/1) to obtain **7** (67 mg, 67%, 89% ee).

7:Ethyl 4-benzyl-3-(methoxymethyl)-5-(2-methoxyphenyl)-2-oxooxazolidine-4-carboxylate

Colorless oil; $[\alpha]_D^{20} +9.0$ (*c* 1.1, CHCl₃, 89% ee); ¹H NMR (400 MHz, CDCl₃): δ 7.45 (d, *J*=7.2 Hz, 2H), 7.39-7.26 (m, 5H), 6.95 (t, *J*=8.2 Hz, 1H), 6.87 (d, *J*=8.2 Hz, 1H), 5.86 (s, 1H), 4.81 (ABq, $\Delta\nu_{AB}$ =0.31 Hz *J*=11.6 Hz, 2H), 3.91 (s, 3H), 3.61 (q of ABq, $\Delta\nu_{AB}$ =0.095 Hz *J_{AB}*=10.8 Hz, *J_{AX}*=7.2 Hz, 2H), 3.55 (ABq, $\Delta\nu_{AB}$ =0.27 Hz *J*=15.4 Hz, 2H), 3.42 (s, 3H), 0.92 (t, *J*=7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 170.0, 158.7, 156.7, 135.1, 131.6, 130.3, 129.0, 127.8, 127.8, 124.2, 120.9, 110.5, 76.3, 75.3, 73.0, 62.3, 57.7, 55.6, 37.6, 13.9; IR (neat) cm⁻¹: 2937, 1768, 1738, 1495, 1378, 1250, 1036, 756, 704; MS (EI) *m/z* (rel intensity) 399 (M⁺, 10), 368 (10), 308 (100), 276 (10), 264 (10), 232 (40), 151 (20), 135 (10); HRMS (EI) *m/z* Calcd for C₂₂H₂₅NO₆ (M⁺): 399.1682 Found 399.1675; Anal. Calcd for C₂₂H₂₅NO₆: C, 66.15; H, 6.31; N, 3.51 Found C, 66.00; H, 6.31; N, 3.39; HPLC conditions: Daicel Chiralcel OD-H, hexane:2-propanol=95: 5, flow 0.8 mL/min, *t_R*=20 (minor), 23 (major) min.

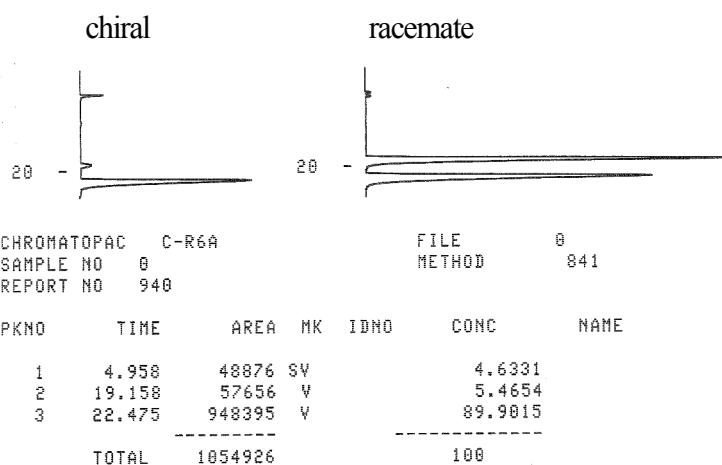
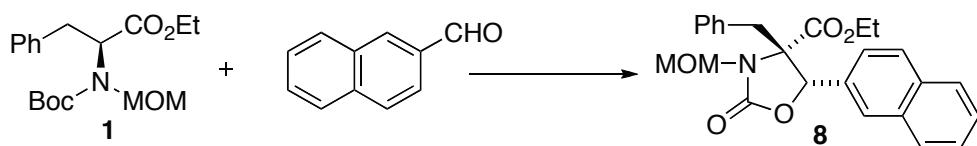


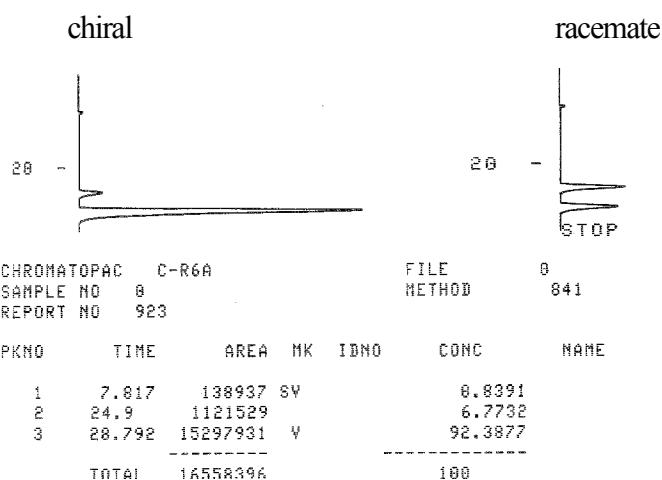
Table 2, entry 7



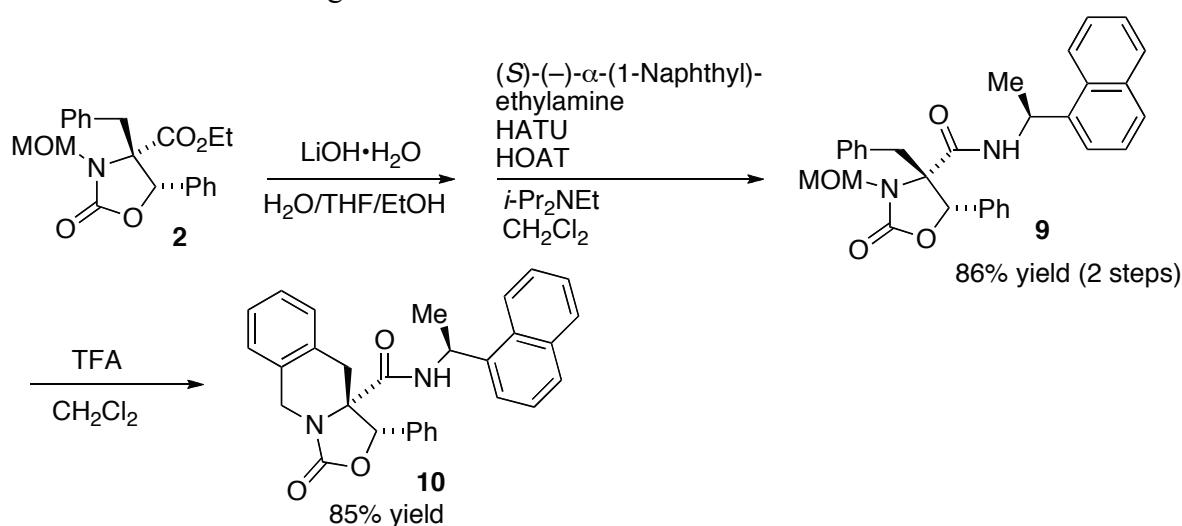
A mixture of **1** (84 mg, 0.25 mmol) and 2-naphthaldehyde (195 mg, 1.25 mmol) was dissolved in toluene (4.5 mL) and the resulting mixture was cooled to -50 °C. KHMS (0.37 M in *t*-BuOMe, 2.03 mL, 0.75 mmol) was added slowly to this solution. After being stirred for 6 hr at same temperature, the reaction mixture was poured into sat. NH₄Cl aq. and extracted with AcOEt. The extracts were washed with sat. NaHCO₃ aq. and brine, and dried over Na₂SO₄, filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/AcOEt=3/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=5/1) to obtain **8** (46 mg, 44%, 86% ee).

8: Ethyl 4-benzyl-3-(methoxymethyl)-5-(naphthalen-2-yl)-2-oxooxazolidine-4-carboxylate

Colorless oil; [α]_D²⁰ -59 (*c* 1.0, CHCl₃, 86% ee); ¹H NMR (400 MHz, CDCl₃): δ 7.82-7.78 (m, 3H), 7.72 (s, 1H), 7.52-7.47 (m, 4H), 7.44-7.32 (m, 4H), 5.56 (s, 1H), 4.86 (ABq, Δv_{AB}=0.32 Hz *J*=11.6 Hz, 2H), 3.60 (q, *J*=7.2 Hz, 2H), 3.57 (ABq, Δv_{AB}=0.087 Hz *J*=15.6 Hz, 2H), 3.48 (s, 3H), 0.79 (t, *J*=7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 169.9, 158.7, 134.5, 133.7, 133.1, 131.8, 131.6, 129.4, 128.5, 128.5, 128.2, 128.1, 127.1, 127.1, 126.1, 123.6, 79.3, 76.5, 73.3, 62.6, 57.8, 37.8, 13.8; IR (neat) cm⁻¹: 2934, 1771, 1739, 1376, 1227, 1083, 1040, 704; MS (EI) *m/z* (rel intensity) 419 (M⁺, 30), 284 (20), 270 (20), 252 (60), 91 (60); HRMS (EI) *m/z* Calcd for C₂₅H₂₅NO₅ (M⁺): 419.1733 Found 419.1749; Anal. Calcd for C₂₅H₂₅NO₅: C, 71.58; H, 6.01; N, 3.34 Found C, 71.48; H, 6.12; N, 3.24; HPLC conditions: Daicel Chiralcel OD-H, hexane:2-propanol=90:10, flow 0.5 mL/min, *t*_R=25 (minor), 29 (major) min.



Determination of Absolute configuration



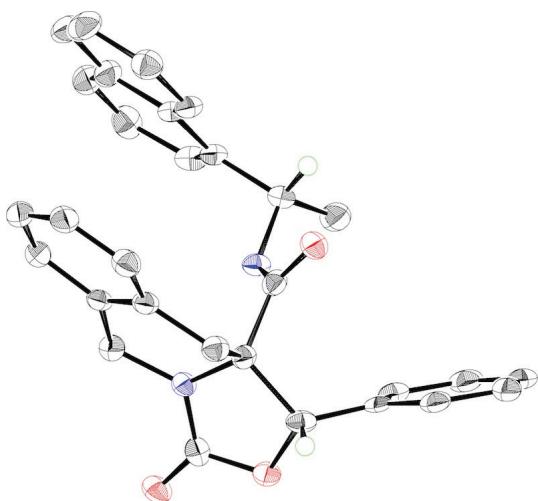
$\text{LiOH}\cdot\text{H}_2\text{O}$ (82 mg, 1.95 mmol) was added to a solution of **2** (239 mg, 0.65 mmol, 85% ee) in $\text{H}_2\text{O}/\text{EtOH}/\text{THF}$ (18 mL, 1/1/1, v/v/v) at 0 °C. The resulting mixture was warmed to 50 °C and stirred for 3 hr. After being cooled to rt, the mixture was diluted with AcOEt and 1 M HCl was added. The aqueous layer was extracted with AcOEt and the extracts were washed with brine and dried over Na_2SO_4 , filtered, and concentrated. The residue was dissolved in CH_2Cl_2 (6.5 mL) and cooled to 0 °C. To this solution were added *O*-(7-azabenzotriazol-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate (HATU, 373 mg, 0.98 mmol), 1-hydroxy-7-benzotriazole (HOAT, 133 mg, 0.98 mmol), (*S*)(-)- α -(1-naphthyl)ethylamine (0.21 mL, 1.30 mmol), and *i*-Pr₂NEt (0.34 mL, 1.95 mL), successively. After being stirred at rt for 48 hr, the mixture was diluted with AcOEt and washed with aqueous 10% citric acid solution, sat. NaHCO_3 aq., and brine and dried over Na_2SO_4 , filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/AcOEt=4/1) to give **9** (277 mg, 89%). Trifluoroacetic acid (6 mL) was added to a solution of **9** (200 mg, 0.40 mmol) in CH_2Cl_2 (2.0 mL) at 0 °C. The resulting mixture was refluxed for 2 hr. After removal of volatiles, the residue was purified through flash silica gel column chromatography (hexane/AcOEt=3/1) to give **10** (157 mg, 85%) as colorless crystals.

10:(1*S*,10*aS*)-*N*-(*(S*)-1-(naphthalen-1-yl)ethyl)-3-oxo-1-phenyl-3,5,10,10*a*-tetrahydro-1*H*-oxazolo[3,4-*b*]-isoquinoline-10*a*-carboxamide**

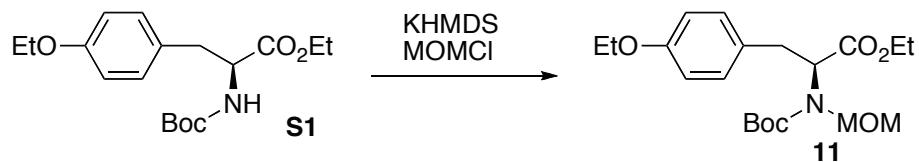
Colorless crystal; mp. 210–213 °C (recrystallized from 1,4-dioxane/petroleum ether) $[\alpha]_D^{20} +15.0$ (*c* 0.39, CHCl_3 , >99% ee); ¹H NMR (400 MHz, CDCl_3): δ 7.78 (d, *J*=8.3 Hz, 1H), 7.73 (d, *J*=8.3 Hz, 1H), 7.46–7.43 (m,

3H), 7.39-7.31 (m, 4H), 7.25-7.21 (m, 2H), 7.18-7.14 (m, 3H), 6.91-6.87 (m, 1H), 6.62 (d, $J=7.8$ Hz, 1H), 5.84 (br d, $J=7.8$ Hz, 1H), 5.54 (s, 1H), 5.22 (dq, $J=7.3$ Hz, 6.8 Hz, 1H), 4.64 (d, $J=17.0$ Hz, 1H), 4.06 (d, $J=14.7$ Hz, 1H), 3.59 (d, $J=17.0$ Hz, 1H), 3.08 (d, $J=14.7$ Hz, 1H), 0.80 (d, $J=6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 165.7, 158.7, 136.8, 135.3, 134.1, 131.3, 131.2, 130.1, 130.0, 129.7, 129.2, 129.1, 127.7, 127.6, 127.1, 126.7, 126.6, 126.1, 125.3, 123.1, 123.0, 84.1, 68.4, 45.09, 44.0, 18.2; IR (neat) cm^{-1} : 2925, 1754, 1668, 1401, 1220, 1004, 776; MS (EI) m/z (rel intensity) 431 (M^+ , 20), 264 (80), 263 (40), 220 (100), 218 (30), 155 (20); HRMS (EI) m/z Calcd for $\text{C}_{30}\text{H}_{26}\text{N}_2\text{O}_3$ (M^+): 462.1943 Found 462.1941.

Crystal data for **10**+[1/2 1,4-dioxane], found $\text{C}_{32}\text{H}_{30}\text{N}_2\text{O}_4$, FW=506.58, T=103 (2) K, Wavelength 0.71069 Å. Crystal system triclinic, Space group *P1* (#1), Unit cell dimensions $a=9.1521$ (3) Å $\alpha=97.7195$ (13)°. $b=14.3285$ (4) Å $\beta=97.2737$ (19)°. $c=20.2138$ (6) Å $\gamma=89.468$ (2)°. Volume=2605.51 (14) Å³, Z=4, Density (calculated)=1.291 Mg/m³, Absorption coefficient=0.085 mm⁻¹, F (000)=1072, Crystal size=0.35 x 0.10 x 0.05 mm³, Reflections collected 23140, Independent reflections 17122 [R (int)=0.0499], Completeness to theta=25.50° 99.4 %, Final R indices [$I>2\sigma(I)$] $R_1=0.0551$, $wR_2=0.1210$ R indices (all data) $R_1=0.0762$, $wR_2=0.1297$. CCDC 867310 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.ac.uk/data_request/cif.



Preparation of precursor **11**



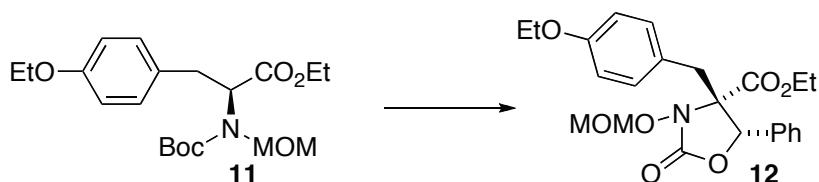
KHMDS (0.32 M in THF, 8.31 mL, 2.66 mmol) was added dropwise slowly to a solution of known **S1** (1.0 g, 2.96 mmol) at -78 °C. The resulting mixture was stirred for 30 min and MOMCl (0.90 mL, 11.8 mmol) was added slowly. After being stirred for 17 hr, the reaction mixture was poured into sat. NH_4Cl aq. and extracted with AcOEt. The extracts were washed with sat. NaHCO_3 aq. and brine, and dried over Na_2SO_4 , filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/1,4-dioxane=20/1) to give **11** (870 mg, 86%, >99% ee).

A 2/3 mixture of rotamers of **11**: (S)-Ethyl 2-(tert-butoxycarbonyl(methoxymethyl)amino)-3-(4-ethoxyphenyl)propanoate

Colorless oil; $[\alpha]_D^{20} -127$ (*c* 0.96, CHCl_3 , >99% ee); ^1H NMR (400 MHz, CDCl_3): δ 7.09, 7.06 (two d, $J=8.7$

Hz and $J=8.7$ Hz, ratio=2:3, 2H), 6.80 (d, $J=8.7$ Hz, 2H), 4.73, 4.60 (two d, $J=11.0$ Hz and $J=11.0$ Hz, ratio=3:2, 2H), 4.27-4.12 (m, 2.4 H), 4.07 (dd, $J=11.0$ Hz, 5.0 Hz, 3/5 H), 4.02-3.97 (m, 12/5 H), 3.81 (d, $J=11.0$ Hz, 3/5H), 3.32, 3.28 (two d, $J=5.0$ Hz and $J=5.0$ Hz, ratio=2:3, 1H), 3.25-3.06 (m, 1H), 3.23, 3.17 (two s, ratio=3:2, 3H), 1.49, 1.48 (two s, ratio=3:2, 9H), 1.40 (t, $J=7.3$ Hz, 3H), 1.30, 1.25 (two t, $J=7.3$ Hz and $J=7.3$ Hz, ratio=3:2, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 171.5, 158.0 and 157.9 (rotamer), 155.3 and 154.7 (rotamer), 130.6, 130.4 and 130.2 (rotamer), 114.9 and 114.7 (rotamer), 81.5 and 81.1 (rotamer), 79.8, 63.7, 61.1 and 61.4 (rotamer), 61.2 and 60.9 (rotamer), 56.2 and 55.8 (rotamer), 35.7 and 34.8 (rotamer), 28.6, 15.2, 14.5; IR (neat) cm^{-1} : 2930, 1740, 1705, 1512, 1243, 1175, 1091, 913, 863; MS (EI) m/z (rel intensity) 381 (M^+ , 10), 276 (10), 250 (20), 220 (100), 135 (90), 107 (50), 57 (50); HRMS (EI) m/z Calcd for $\text{C}_{20}\text{H}_{31}\text{NO}_6$ (M^+): 381.2151 Found 381.2160 Anal. Calcd for $\text{C}_{20}\text{H}_{31}\text{NO}_6$: C, 62.97; H, 8.91; N, 3.67 Found C, 62.88; H, 8.22; N, 3.85; HPLC conditions: Daicel Chiralcel OD-H, hexane:2-propanol=98:2, flow 0.4 mL/min, $t_{\text{R}}=18$ (*R*), 20 (*S*) min.

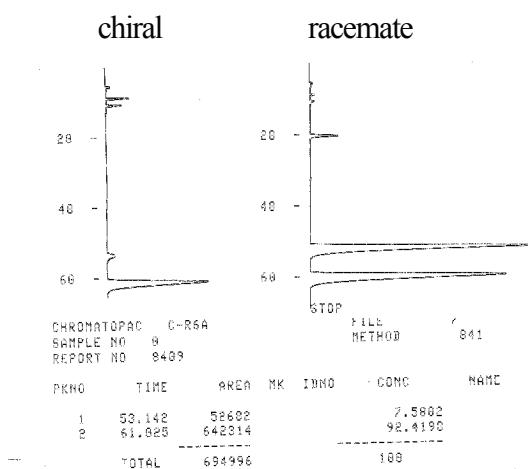
Procedure for aldol reaction between **11** and benzaldehyde



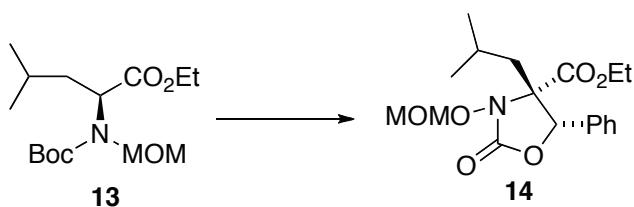
Benzaldehyde (0.13 mL, 1.25 mmol) was added to a solution of **11** (95 mg, 0.25 mmol) in *t*-BuOMe (1.5 mL) and the mixture was cooled to -40 °C and KHMDS (0.39 M in toluene, 1.92 mL, 0.75 mmol) was added dropwise slowly. After being stirred for 6 hr at same temperature, the reaction mixture was poured into sat. NH_4Cl aq. and extracted with AcOEt. The extracts were washed with sat. NaHCO_3 aq. and brine, and dried over Na_2SO_4 , filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/Et₂O=1/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=4/1) to obtain **12** (98 mg, 95%, 85% ee).

12:Ethyl 4-(4-ethoxybenzyl)-3-(methoxymethyl)-2-oxo-5-phenyloxazolidine-4-carboxylate

Colorless oil; $[\alpha]_D^{20} -6.6$ (*c* 1.2, CHCl_3 , 91% ee); ^1H NMR (400 MHz, CDCl_3): δ 7.37-7.30 (m, 5H), 7.09 (ABq, $\Delta\nu_{\text{AB}}=0.37$ Hz, $J=8.4$ Hz, 4H), 5.39 (s, 1H), 4.82 (ABq, $\Delta\nu_{\text{AB}}=0.32$ Hz, $J=11.4$ Hz, 2H), 4.04 (q, $J=7.2$ Hz, 2H), 3.70 (q, $J=7.6$ Hz, 2H), 3.46 (s, 3H), 3.44 (ABq, $\Delta\nu_{\text{AB}}=0.043$ Hz, $J=15.6$ Hz, 2H), 1.42 (t, $J=7.2$ Hz, 3H), 0.93 (t, $J=7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.8, 158.8, 158.6, 134.6, 132.5, 129.3, 128.7, 126.4, 125.9, 115.1, 79.0, 76.3, 73.3, 63.8, 62.4, 57.6, 36.7, 15.2, 13.9; IR (neat) cm^{-1} : 2934, 1770, 1739, 1513, 1248, 913, 733; MS (EI) m/z (rel intensity) 413 (M^+ , 20), 382 (5), 278 (10), 202 (5), 135 (100), 107 (60); HRMS (EI) m/z Calcd for $\text{C}_{23}\text{H}_{27}\text{NO}_6$ (M^+): 413.1838 Found 413.1844; Anal. Calcd for $\text{C}_{23}\text{H}_{27}\text{NO}_6$: C, 66.81; H, 6.58; N, 3.39 Found C, 66.80; H, 6.68; N, 3.34; HPLC conditions: Daicel Chiralpak AD-H, hexane:2-propanol=90:10, flow 0.6 mL/min, $t_{\text{R}}=53$ (minor), 59 (major) min.



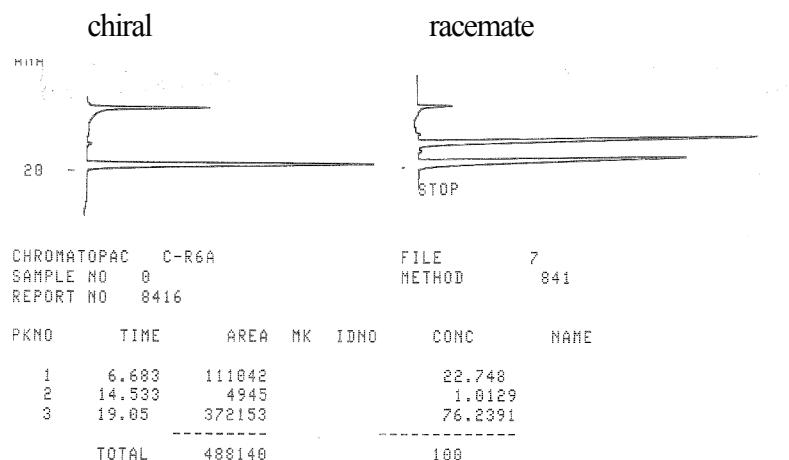
Procedure for aldol reaction between **13** and benzaldehyde



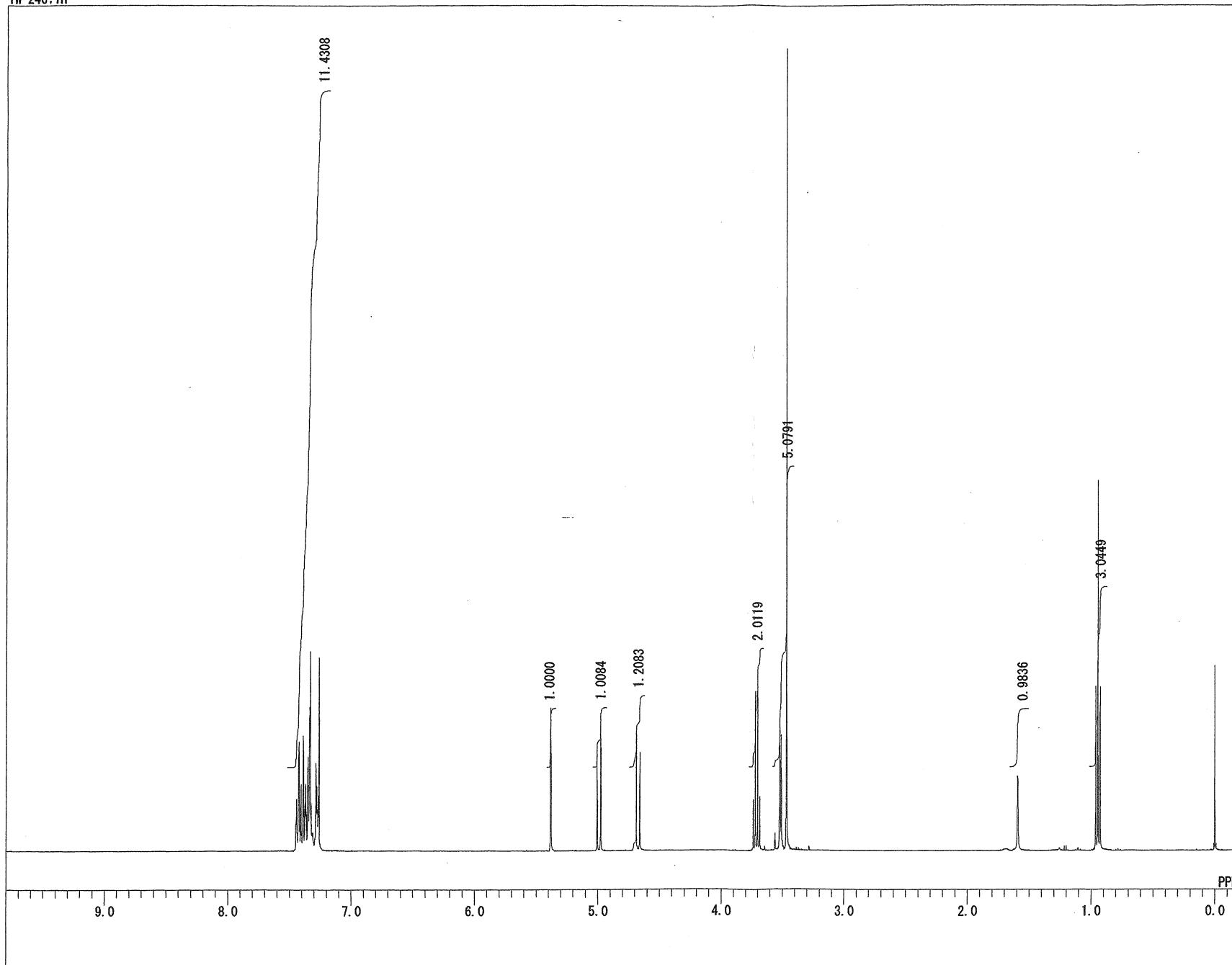
Benzaldehyde (0.13 mL, 1.25 mmol) was added to a solution of **13** (76 mg, 0.25 mmol) in *t*-BuOMe (1.5 mL) and the mixture was cooled to -40 °C and KHMDS (0.44 M in toluene, 1.70 mL, 0.75 mmol) was added dropwise slowly. After being stirred for 12 hr at same temperature, the reaction mixture was poured into sat. NH₄Cl aq. and extracted with AcOEt. The extracts were washed with sat. NaHCO₃ aq. and brine, and dried over Na₂SO₄, filtered, and concentrated. The residue was purified through flash silica gel column chromatography (hexane/AcOEt=6/1) to give an oil, which was further purified by PTLC (hexane/1,4-dioxane=4/1) to obtain **14** (70 mg, 83%, 94% ee).

14:Ethyl 4-isobutyl-3-(methoxymethyl)-2-oxo-5-phenyloxazolidine-4-carboxylate

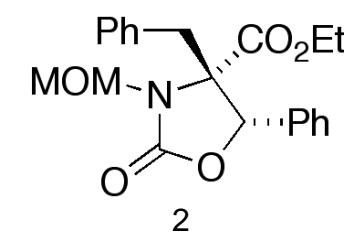
Colorless oil; [α]_D²⁰ +19 (*c* 1.2, CHCl₃, 98% ee); ¹H NMR (400 MHz, CDCl₃): δ 7.38-7.30 (m, 5H), 5.58 (s, 1H), 4.74 (ABq, Δv_{AB}=0.29 Hz *J*=11.8 Hz, 2H), 3.61 (q of ABq, Δv_{AB}=0.080 Hz *J*_{AB}=10.8 Hz, *J*_{AX}=7.2 Hz, 2H), 3.42 (s, 3H), 2.08 (d of ABq, Δv_{AB}=0.18 Hz *J*=15.2 Hz, *J*_{AX}=6.0 Hz, 2H), 2.05 (br sept, *J*= 6.0 Hz, 1H), 1.20 (d, *J*=6.0 Hz, 3H), 1.04 (d, *J*=6.0 Hz, 3H), 0.87 (t, *J*=7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 170.0, 158.8, 134.5, 129.5, 128.8, 126.4, 80.2, 76.0, 73.2, 62.2, 57.4, 40.3, 25.9, 24.5, 23.6, 13.9; IR (neat) cm⁻¹: 2960, 2940, 1772, 1740, 1455, 1374, 1229, 1082, 1029, 751, 697; MS (EI) *m/z* (rel intensity) 335 (M⁺, <1), 304 (1), 278 (1), 262 (100), 232 (10), 218 (10), 186 (15); HRMS (EI) *m/z* Calcd for C₁₈H₂₅NO₅ (M⁺): 335.1733 Found 335.1736; Anal. Calcd for C₁₈H₂₅NO₅: C, 64.46; H, 7.51; N, 4.18 Found C, 64.46; H, 7.64; N, 4.27; HPLC conditions: Daicel Chiralcel OD-H, hexane:2-propanol=90:10, flow 0.5 mL/min, *t*_R=14 (minor), 19 (major) min.

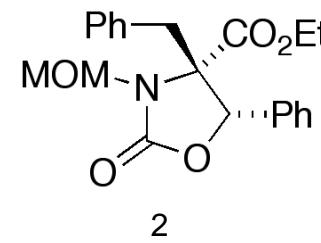
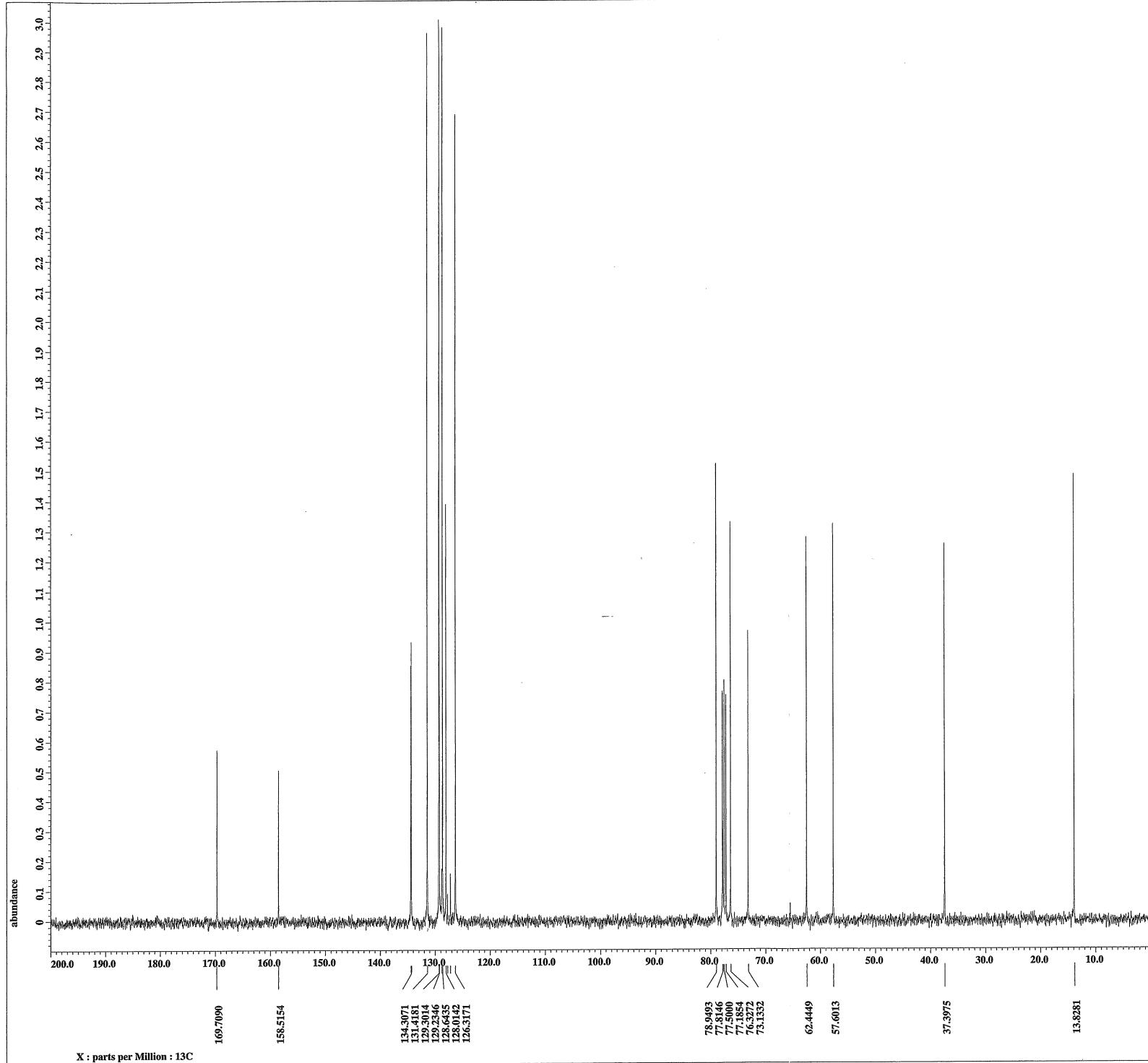


C:\WINNMR98\DATA\tw246h.als
TW-246;1H

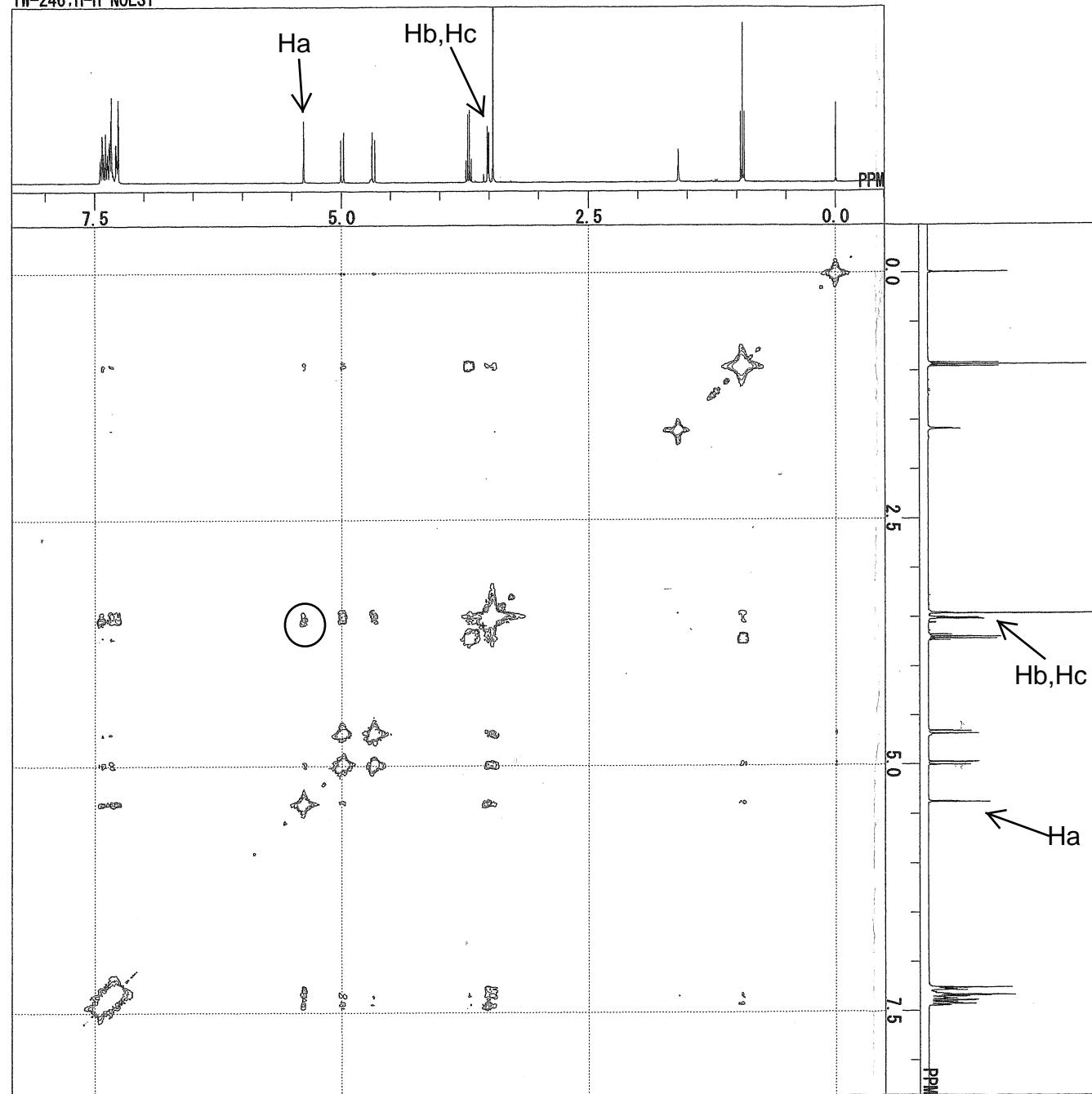


DFILE C:\WINNMR98\DATA\tw246h.als
COMNT TW-246;1H
DATIM Wed Jul 27 09:28:46 2005
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 kHz
OBFIN 10277.0 Hz
POINT 32768
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCl₃
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 19

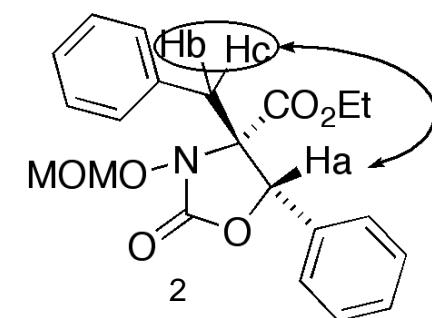




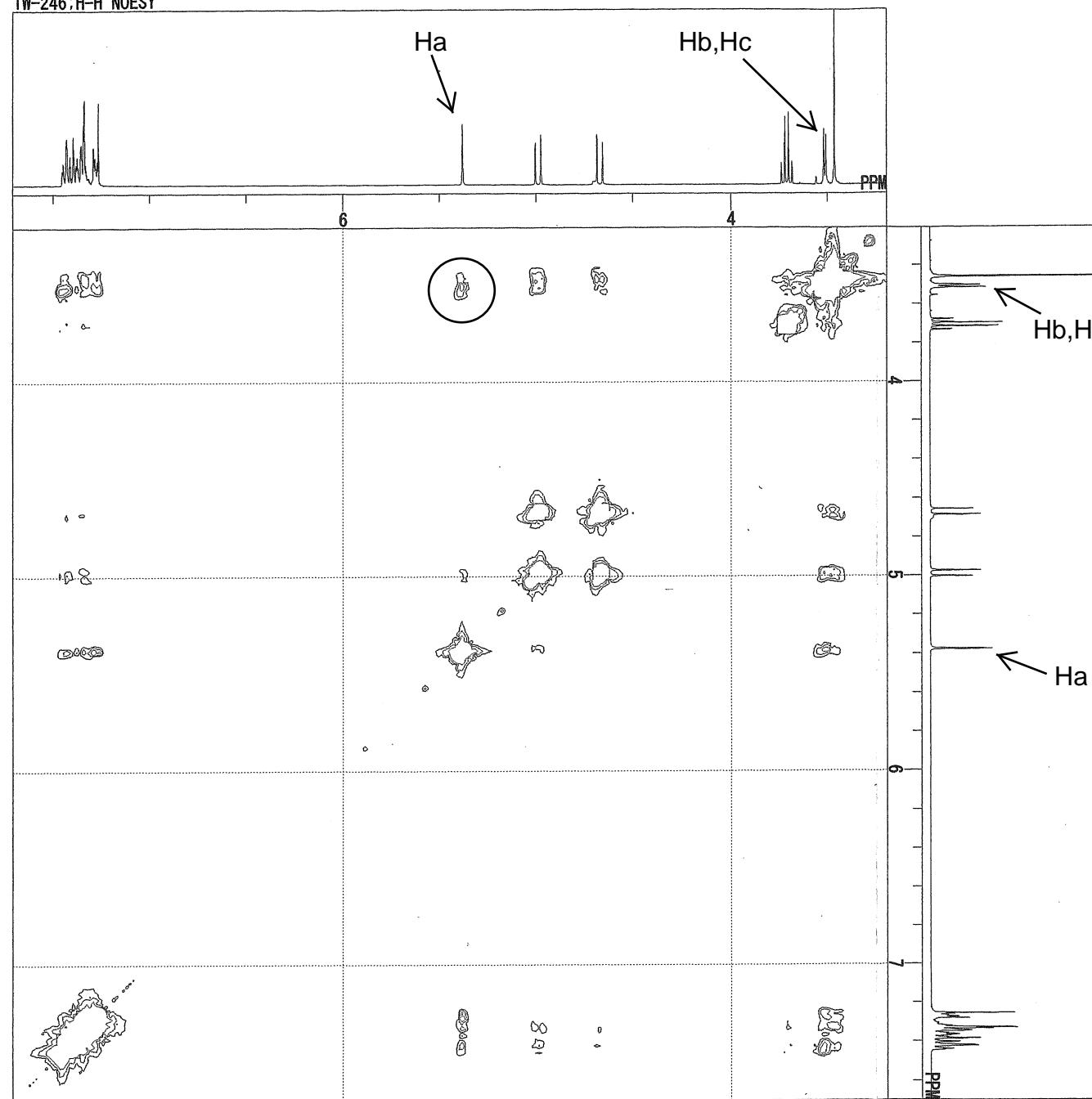
C:\WINNMR98\DATA\tw246noesy. ALS
TW-246;H-H NOESY



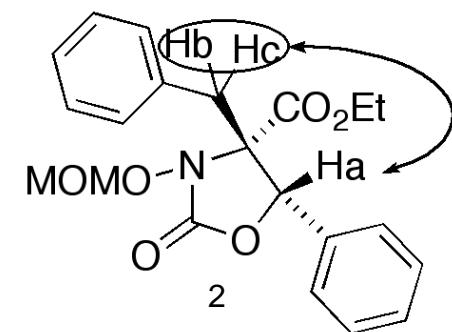
DFILE C:\WINNMR98\DATA\tw246noesy. ALS
COMNT TW-246;H-H NOESY
DATIM Wed Jul 27 13:29:18 2005
EXMOD VNOENH
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134.00 kHz
OBFIN 77.4 Hz
POINT 512
FREQU 3500.1 Hz
CLPNT 512
TODAT 256
CLFRQ 3496.5 Hz
SCANS 16
ACQTM 0.146 sec
PD 1.500 sec
PW1 12.4 us
PW2 12.4 us
PW3 10.0 us
P11 0.286 ms
P12 0.286 ms
P13 1000.00 ms
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCl₃
EXREF 0.00 ppm
CLEXR 0.00 ppm
RGAIN 18
OBATN 511
LOOP1 1



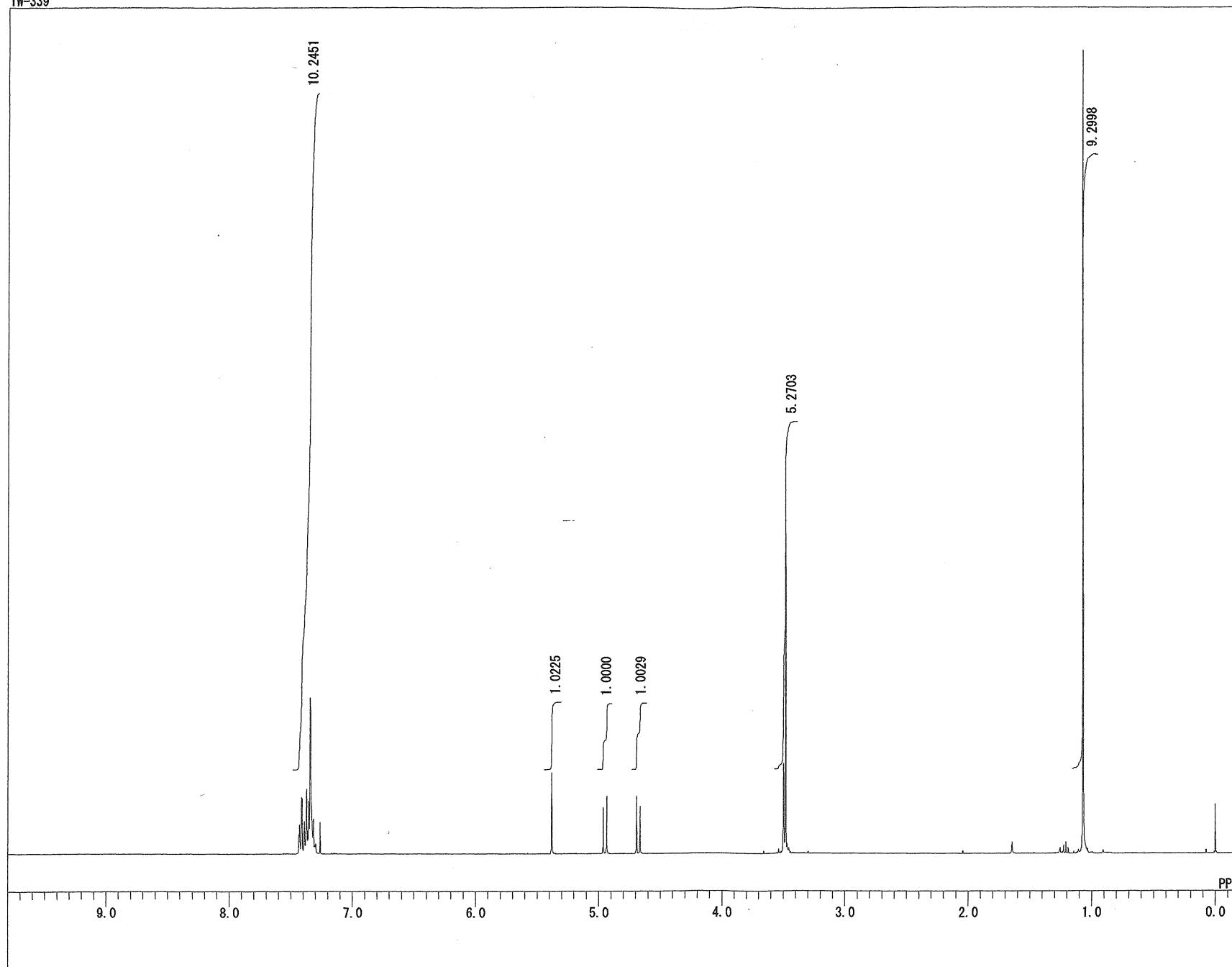
C:\WINNMR98\DATA\tw246noesy. ALS
TW-246;H-H NOESY



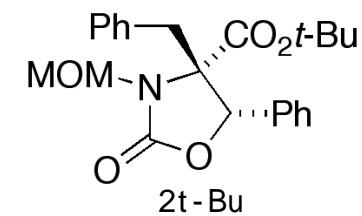
DFILE C:\WINNMR98\DATA\tw246noesy. ALS
COMNT TW-246;H-H NOESY
DATIM Wed Jul 27 13:29:18 2005
EXMOD VNOENH
OBNUC 1H
OBFRO 395.75 MHz
OBSET 134.00 kHz
OBFIN 77.4 Hz
POINT 512
FREQU 3500.1 Hz
CLPNT 512
TODAT 256
CLFRQ 3496.5 Hz
SCANS 16
ACQTM 0.146 sec
PD 1.500 sec
PW1 12.4 us
PW2 12.4 us
PW3 10.0 us
P11 0.286 ms
P12 0.286 ms
P13 1000.00 ms
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCl₃
EXREF 0.00 ppm
CLEXR 0.00 ppm
RGAIN 18
OBATN 511
LOOP1 1

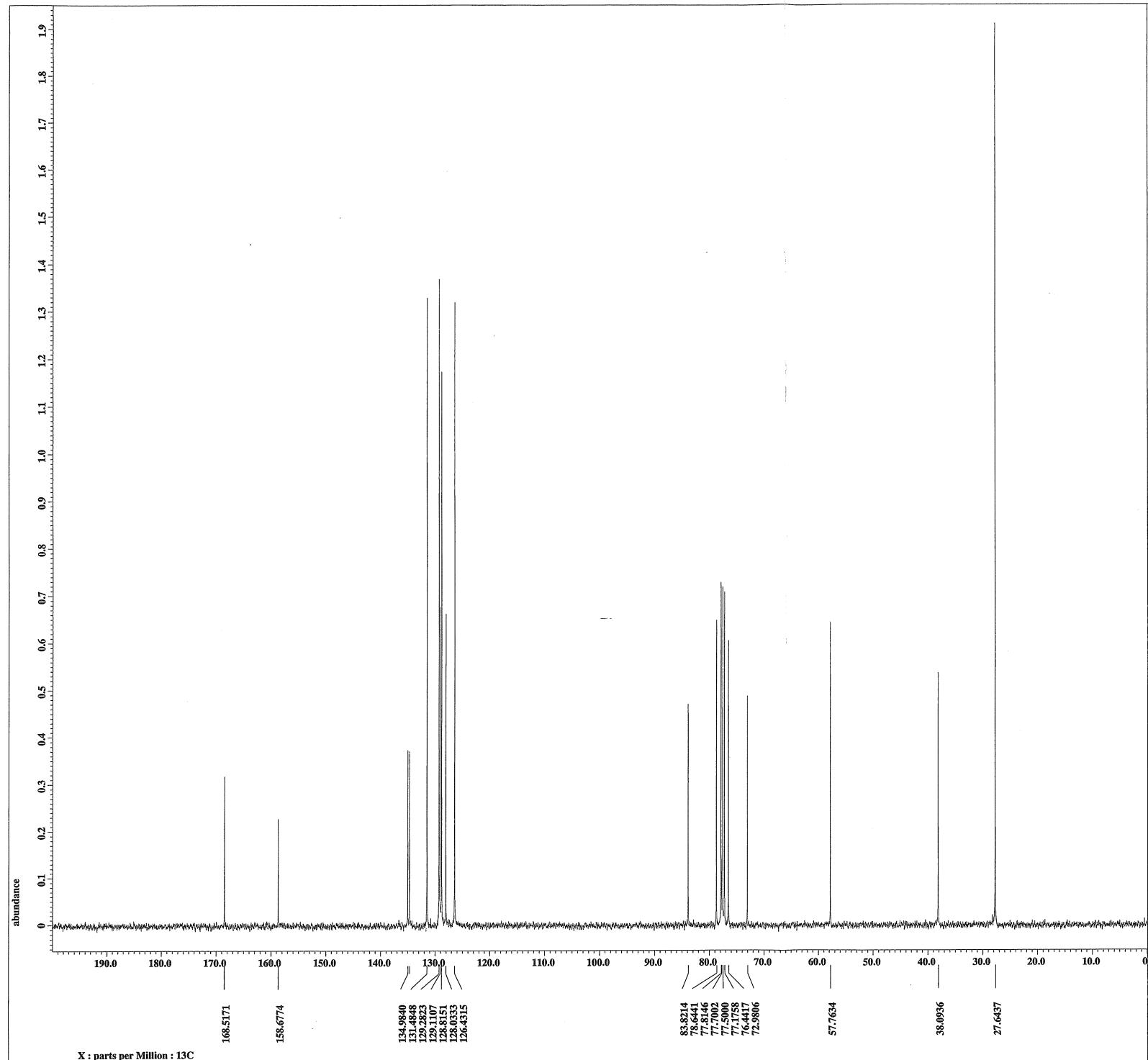


C:\WINNMR98\DATA\tw339.als
TW-339



DFILE C:\WINNMR98\DATA\tw339.als
COMNT TW-339
DATIM Thu Feb 23 10:21:39 2006
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 kHz
OBFIN 10277.0 Hz
POINT 32768
FREQM 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
IRNUC 1H
CTEMP 19.9 °C
SLVNT CDCl₃
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 15





```

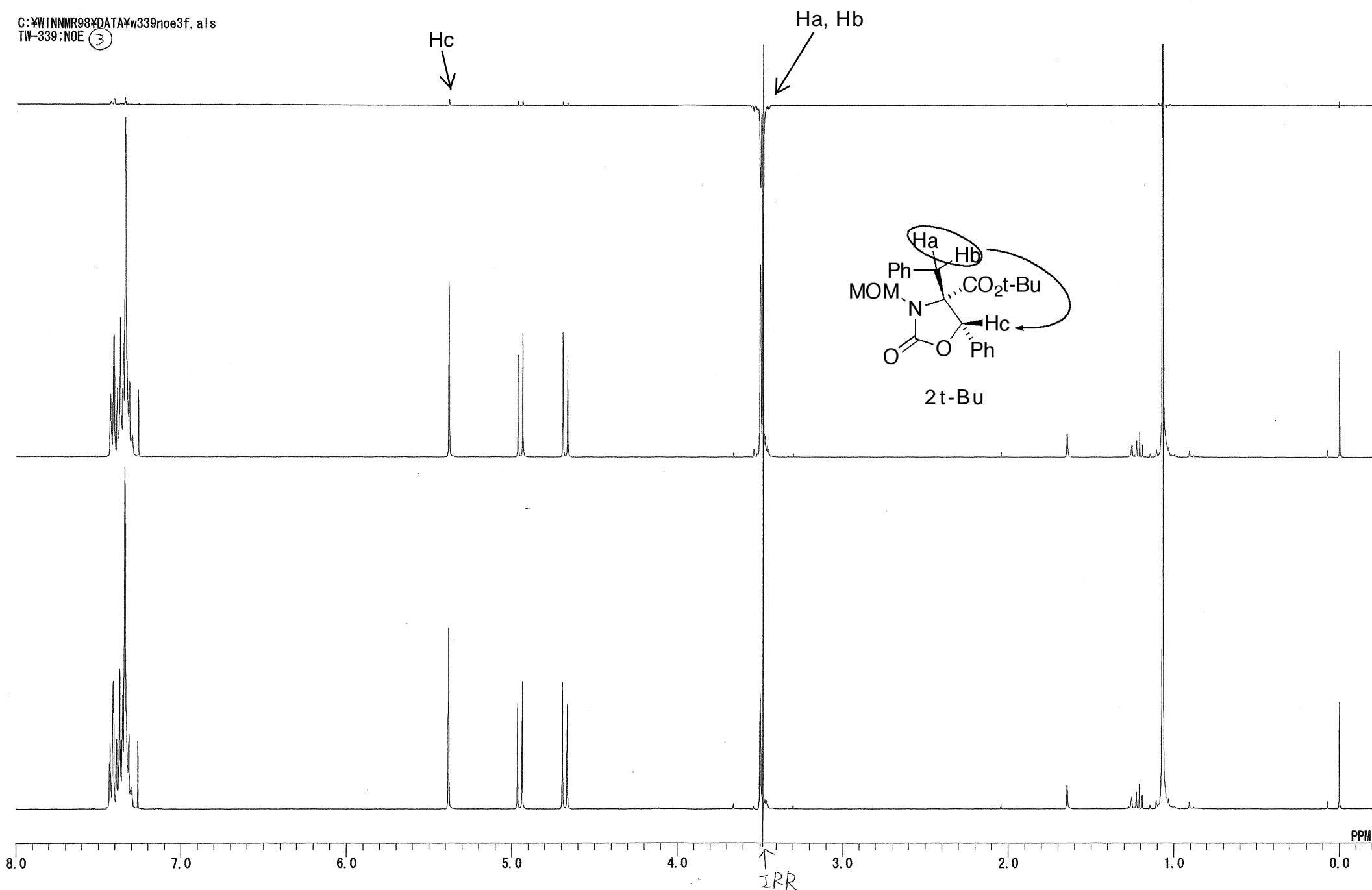
----- PROCESSING PARAMETERS -----
dc_balance = 0 : FALSE
segs : 2.0[Hz] : 0.0[el]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill1 : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TW-786 13C-1.jdf

Filename      = TW-786 13C-4.jdf
Author        = delta
Experiment   = single_pulse_dec
SampleId     = S#786
Solvent      = CHLOROFORM-D
Creation_time = 27-MAR-2007 19:05:26
Revision_time = 28-MAR-2007 00:25:02
Current_time  = 28-MAR-2007 00:25:40
Content       = single pulse decouple
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site          = ECK 400P
Spectrometer  = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_center      = 130
X_freq         = 100.52530333[MHz]
X_offset       = 100[ppm]
X_points      = 32768
X_prescans   = 4
X_resolution  = 0.95846665[Hz]
X_sweep        = 31.40703518[kHz]
Irr_domain    = 1H
Irr_freq       = 399.78219838[MHz]
Irr_offset     = 5[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans          = 236
Total_scans   = 236
X_90_width    = 10.2[us]
X_acq_time    = 1.04333312[s]
X_angle        = 30[deg]
X_atn         = 3.8[dB]
X_pulse        = 3.4[us]
Irr_atn_dec   = 19.8[dB]
Irr_atn_noe   = 19.8[dB]
Irr_noise      = WALZ
Decoupling     = TRUE
Initial_wait   = 1[s]
Noe           = TRUE
Noe_time       = 2[s]
Recvrvr_gain   = 56
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get       = 17.7[dc]

```

C(=O)OC(C(=O)OC(C)C)N()C(=O)OC(C)C

C:\WINNMR98\DATA\w339noe3f.als
TW-339;NOE (3)



PPM

8.0

7.0

6.0

5.0

4.0

3.0

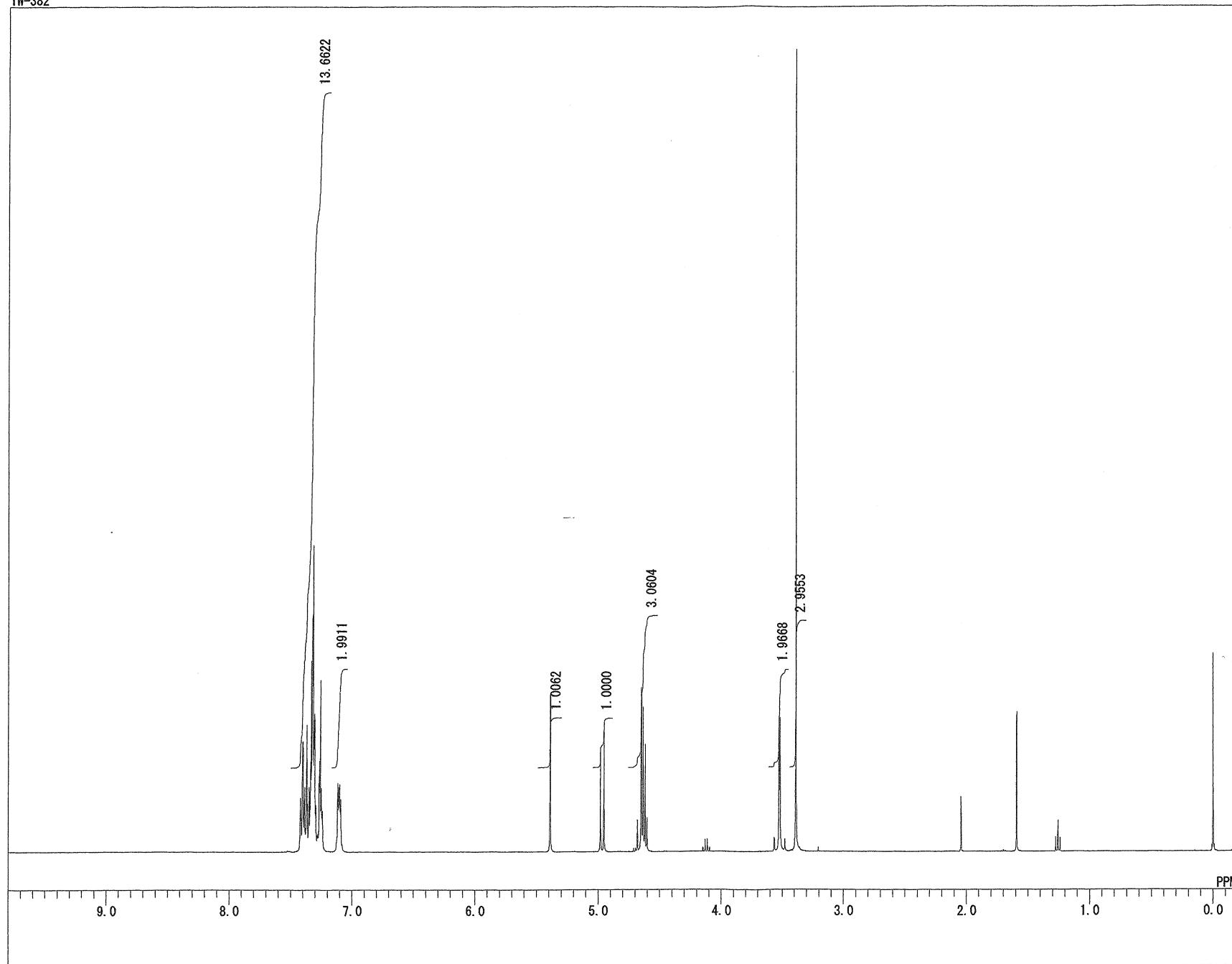
2.0

1.0

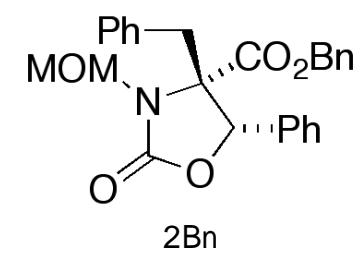
0.0

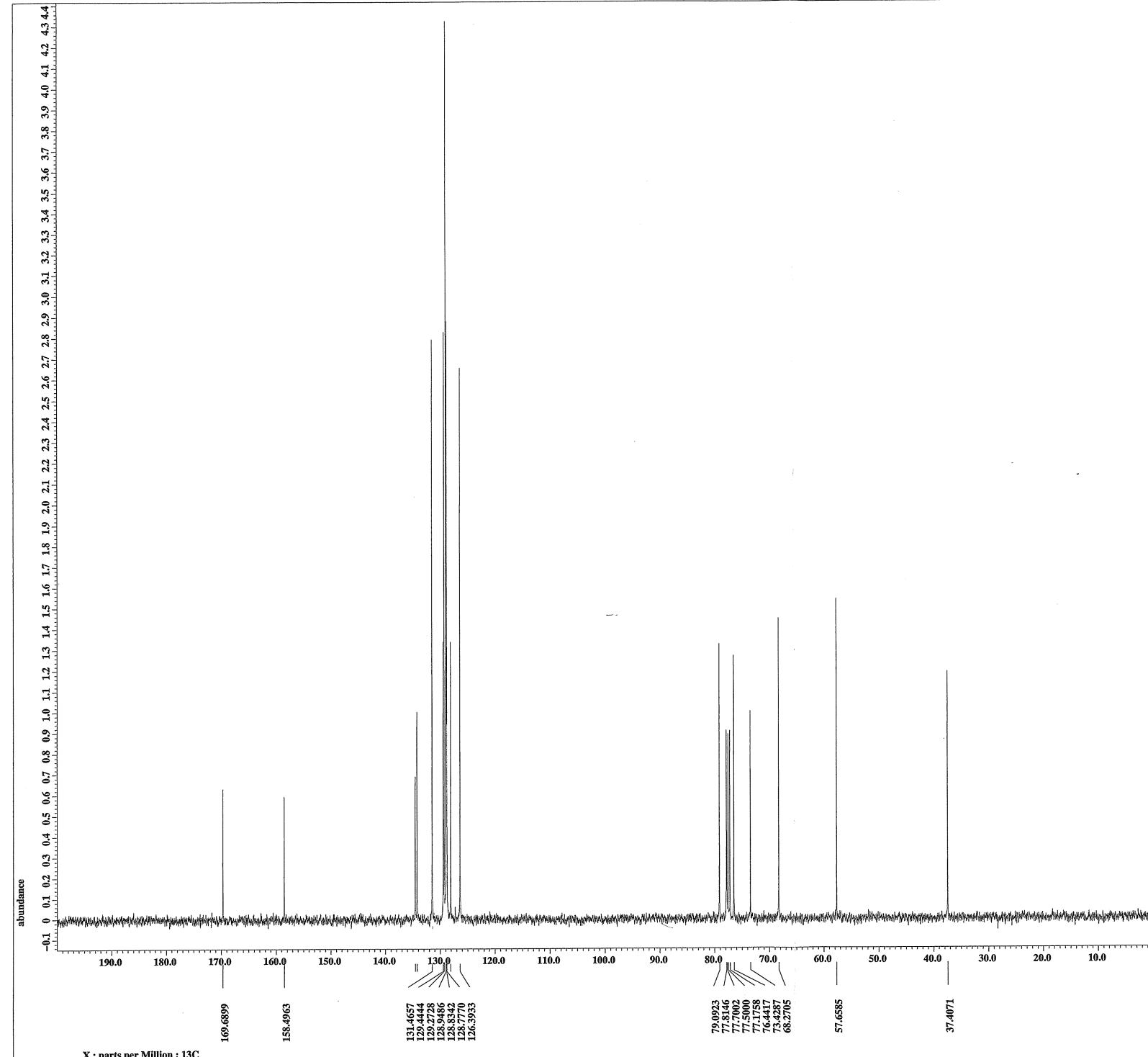
IRR

C:\WINNMR98\DATA\tw382.als
TW-382



DFILE C:\WINNMR98\DATA\tw382.als
COMNT TW-382
DATIM Thu Feb 23 14:07:31 2006
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 KHz
OBFIN 10277.0 Hz
POINT 32768
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 18





```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sep : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: 13C No4-1.jdf

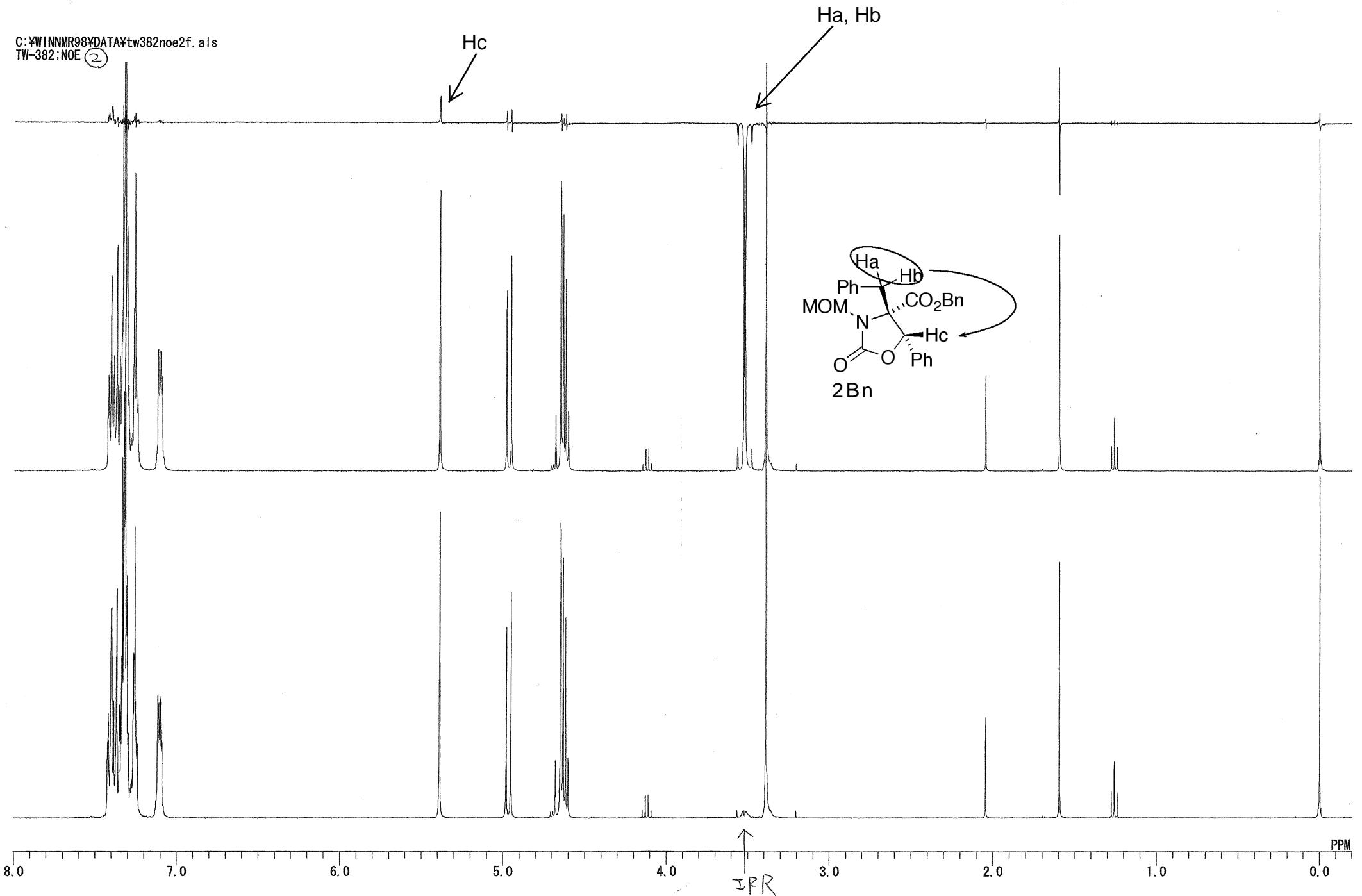
Filename = 13C No4-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#653972
Solvent = CHLOROFORM-D
Creation_time = 22-MAR-2007 12:58:10
Revision_time = 22-MAR-2007 18:18:30
Current_time = 22-MAR-2007 18:20:39

Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400F
Spectrometer = DELTA2_NMR

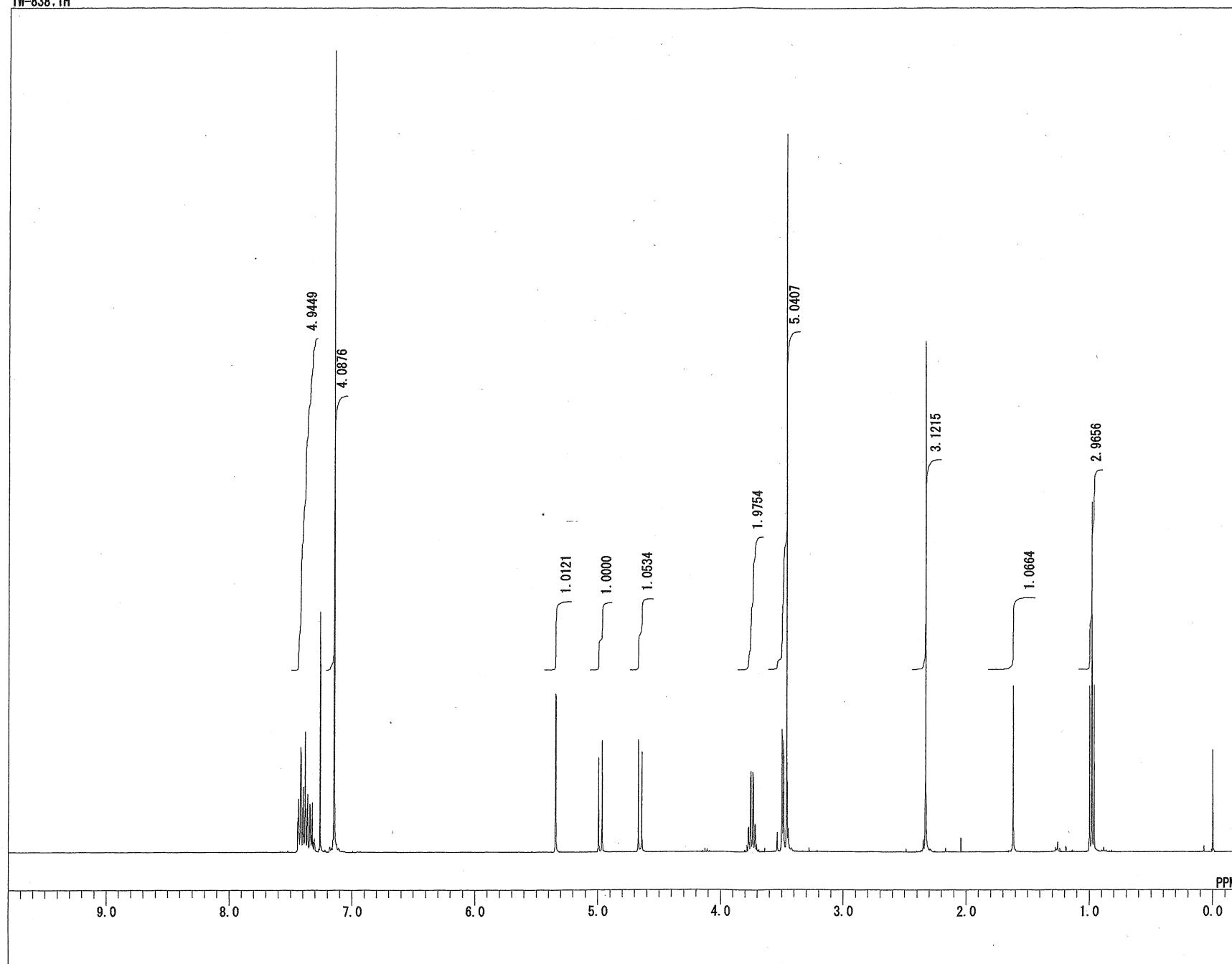
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 52
Total_scans = 52

X_90_width = 10.2[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.8[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 19.8[dB]
Irr_atn_noe = 19.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get = 18.2[dc]

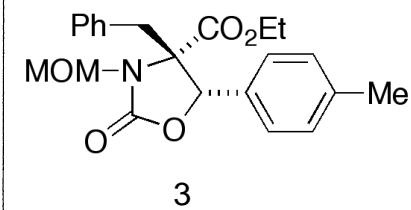
CC(=O)N1[C@@H](C(=O)OC(=O)C2=C1C=C(C=C2)OC(=O)OC(=O)C3=C2C=C(C=C3)OC(=O)OC(=O)C4=C3C=C(C=C4)OC(=O)OC(=O)C5=C4C=C(C=C5)OC(=O)OC(=O)C6=C5C=C(C=C6)OC(=O)OC(=O)C7=C6C=C(C=C7)OC(=O)OC(=O)C8=C7C=C(C=C8)OC(=O)OC(=O)C9=C8C=C(C=C9)OC(=O)OC(=O)C10=C9C=C(C=C10)OC(=O)OC(=O)C11=C10C=C(C=C11)OC(=O)OC(=O)C12=C11C=C(C=C12)OC(=O)OC(=O)C13=C12C=C(C=C13)OC(=O)OC(=O)C14=C13C=C(C=C14)OC(=O)OC(=O)C15=C14C=C(C=C15)OC(=O)OC(=O)C16=C15C=C(C=C16)OC(=O)OC(=O)C17=C16C=C(C=C17)OC(=O)OC(=O)C18=C17C=C(C=C18)OC(=O)OC(=O)C19=C18C=C(C=C19)OC(=O)OC(=O)C20=C19C=C(C=C20)OC(=O)OC(=O)C21=C20C=C(C=C21)OC(=O)OC(=O)C22=C21C=C(C=C22)OC(=O)OC(=O)C23=C22C=C(C=C23)OC(=O)OC(=O)C24=C23C=C(C=C24)OC(=O)OC(=O)C25=C24C=C(C=C25)OC(=O)OC(=O)C26=C25C=C(C=C26)OC(=O)OC(=O)C27=C26C=C(C=C27)OC(=O)OC(=O)C28=C27C=C(C=C28)OC(=O)OC(=O)C29=C28C=C(C=C29)OC(=O)OC(=O)C30=C29C=C(C=C30)OC(=O)OC(=O)C31=C30C=C(C=C31)OC(=O)OC(=O)C32=C31C=C(C=C32)OC(=O)OC(=O)C33=C32C=C(C=C33)OC(=O)OC(=O)C34=C33C=C(C=C34)OC(=O)OC(=O)C35=C34C=C(C=C35)OC(=O)OC(=O)C36=C35C=C(C=C36)OC(=O)OC(=O)C37=C36C=C(C=C37)OC(=O)OC(=O)C38=C37C=C(C=C38)OC(=O)OC(=O)C39=C38C=C(C=C39)OC(=O)OC(=O)C40=C39C=C(C=C40)OC(=O)OC(=O)C41=C40C=C(C=C41)OC(=O)OC(=O)C42=C41C=C(C=C42)OC(=O)OC(=O)C43=C42C=C(C=C43)OC(=O)OC(=O)C44=C43C=C(C=C44)OC(=O)OC(=O)C45=C44C=C(C=C45)OC(=O)OC(=O)C46=C45C=C(C=C46)OC(=O)OC(=O)C47=C46C=C(C=C47)OC(=O)OC(=O)C48=C47C=C(C=C48)OC(=O)OC(=O)C49=C48C=C(C=C49)OC(=O)OC(=O)C50=C49C=C(C=C50)OC(=O)OC(=O)C51=C50C=C(C=C51)OC(=O)OC(=O)C52=C51C=C(C=C52)OC(=O)OC(=O)C53=C52C=C(C=C53)OC(=O)OC(=O)C54=C53C=C(C=C54)OC(=O)OC(=O)C55=C54C=C(C=C55)OC(=O)OC(=O)C56=C55C=C(C=C56)OC(=O)OC(=O)C57=C56C=C(C=C57)OC(=O)OC(=O)C58=C57C=C(C=C58)OC(=O)OC(=O)C59=C58C=C(C=C59)OC(=O)OC(=O)C60=C59C=C(C=C60)OC(=O)OC(=O)C61=C60C=C(C=C61)OC(=O)OC(=O)C62=C61C=C(C=C62)OC(=O)OC(=O)C63=C62C=C(C=C63)OC(=O)OC(=O)C64=C63C=C(C=C64)OC(=O)OC(=O)C65=C64C=C(C=C65)OC(=O)OC(=O)C66=C65C=C(C=C66)OC(=O)OC(=O)C67=C66C=C(C=C67)OC(=O)OC(=O)C68=C67C=C(C=C68)OC(=O)OC(=O)C69=C68C=C(C=C69)OC(=O)OC(=O)C70=C69C=C(C=C70)OC(=O)OC(=O)C71=C70C=C(C=C71)OC(=O)OC(=O)C72=C71C=C(C=C72)OC(=O)OC(=O)C73=C72C=C(C=C73)OC(=O)OC(=O)C74=C73C=C(C=C74)OC(=O)OC(=O)C75=C74C=C(C=C75)OC(=O)OC(=O)C76=C75C=C(C=C76)OC(=O)OC(=O)C77=C76C=C(C=C77)OC(=O)OC(=O)C78=C77C=C(C=C78)OC(=O)OC(=O)C79=C78C=C(C=C79)OC(=O)OC(=O)C80=C79C=C(C=C80)OC(=O)OC(=O)C81=C80C=C(C=C81)OC(=O)OC(=O)C82=C81C=C(C=C82)OC(=O)OC(=O)C83=C82C=C(C=C83)OC(=O)OC(=O)C84=C83C=C(C=C84)OC(=O)OC(=O)C85=C84C=C(C=C85)OC(=O)OC(=O)C86=C85C=C(C=C86)OC(=O)OC(=O)C87=C86C=C(C=C87)OC(=O)OC(=O)C88=C87C=C(C=C88)OC(=O)OC(=O)C89=C88C=C(C=C89)OC(=O)OC(=O)C90=C89C=C(C=C90)OC(=O)OC(=O)C91=C90C=C(C=C91)OC(=O)OC(=O)C92=C91C=C(C=C92)OC(=O)OC(=O)C93=C92C=C(C=C93)OC(=O)OC(=O)C94=C93C=C(C=C94)OC(=O)OC(=O)C95=C94C=C(C=C95)OC(=O)OC(=O)C96=C95C=C(C=C96)OC(=O)OC(=O)C97=C96C=C(C=C97)OC(=O)OC(=O)C98=C97C=C(C=C98)OC(=O)OC(=O)C99=C98C=C(C=C99)OC(=O)OC(=O)C100=C99C=C(C=C100)OC(=O)OC(=O)C101=C100C=C(C=C101)OC(=O)OC(=O)C102=C101C=C(C=C102)OC(=O)OC(=O)C103=C102C=C(C=C103)OC(=O)OC(=O)C104=C103C=C(C=C104)OC(=O)OC(=O)C105=C104C=C(C=C105)OC(=O)OC(=O)C106=C105C=C(C=C106)OC(=O)OC(=O)C107=C106C=C(C=C107)OC(=O)OC(=O)C108=C107C=C(C=C108)OC(=O)OC(=O)C109=C108C=C(C=C109)OC(=O)OC(=O)C110=C109C=C(C=C110)OC(=O)OC(=O)C111=C110C=C(C=C111)OC(=O)OC(=O)C112=C111C=C(C=C112)OC(=O)OC(=O)C113=C112C=C(C=C113)OC(=O)OC(=O)C114=C113C=C(C=C114)OC(=O)OC(=O)C115=C114C=C(C=C115)OC(=O)OC(=O)C116=C115C=C(C=C116)OC(=O)OC(=O)C117=C116C=C(C=C117)OC(=O)OC(=O)C118=C117C=C(C=C118)OC(=O)OC(=O)C119=C118C=C(C=C119)OC(=O)OC(=O)C120=C119C=C(C=C120)OC(=O)OC(=O)C121=C120C=C(C=C121)OC(=O)OC(=O)C122=C121C=C(C=C122)OC(=O)OC(=O)C123=C122C=C(C=C123)OC(=O)OC(=O)C124=C123C=C(C=C124)OC(=O)OC(=O)C125=C124C=C(C=C125)OC(=O)OC(=O)C126=C125C=C(C=C126)OC(=O)OC(=O)C127=C126C=C(C=C127)OC(=O)OC(=O)C128=C127C=C(C=C128)OC(=O)OC(=O)C129=C128C=C(C=C129)OC(=O)OC(=O)C130=C129C=C(C=C130)OC(=O)OC(=O)C131=C130C=C(C=C131)OC(=O)OC(=O)C132=C131C=C(C=C132)OC(=O)OC(=O)C133=C132C=C(C=C133)OC(=O)OC(=O)C134=C133C=C(C=C134)OC(=O)OC(=O)C135=C134C=C(C=C135)OC(=O)OC(=O)C136=C135C=C(C=C136)OC(=O)OC(=O)C137=C136C=C(C=C137)OC(=O)OC(=O)C138=C137C=C(C=C138)OC(=O)OC(=O)C139=C138C=C(C=C139)OC(=O)OC(=O)C140=C139C=C(C=C140)OC(=O)OC(=O)C141=C140C=C(C=C141)OC(=O)OC(=O)C142=C141C=C(C=C142)OC(=O)OC(=O)C143=C142C=C(C=C143)OC(=O)OC(=O)C144=C143C=C(C=C144)OC(=O)OC(=O)C145=C144C=C(C=C145)OC(=O)OC(=O)C146=C145C=C(C=C146)OC(=O)OC(=O)C147=C146C=C(C=C147)OC(=O)OC(=O)C148=C147C=C(C=C148)OC(=O)OC(=O)C149=C148C=C(C=C149)OC(=O)OC(=O)C150=C149C=C(C=C150)OC(=O)OC(=O)C151=C150C=C(C=C151)OC(=O)OC(=O)C152=C151C=C(C=C152)OC(=O)OC(=O)C153=C152C=C(C=C153)OC(=O)OC(=O)C154=C153C=C(C=C154)OC(=O)OC(=O)C155=C154C=C(C=C155)OC(=O)OC(=O)C156=C155C=C(C=C156)OC(=O)OC(=O)C157=C156C=C(C=C157)OC(=O)OC(=O)C158=C157C=C(C=C158)OC(=O)OC(=O)C159=C158C=C(C=C159)OC(=O)OC(=O)C160=C159C=C(C=C160)OC(=O)OC(=O)C161=C160C=C(C=C161)OC(=O)OC(=O)C162=C161C=C(C=C162)OC(=O)OC(=O)C163=C162C=C(C=C163)OC(=O)OC(=O)C164=C163C=C(C=C164)OC(=O)OC(=O)C165=C164C=C(C=C165)OC(=O)OC(=O)C166=C165C=C(C=C166)OC(=O)OC(=O)C167=C166C=C(C=C167)OC(=O)OC(=O)C168=C167C=C(C=C168)OC(=O)OC(=O)C169=C168C=C(C=C169)OC(=O)OC(=O)C170=C169C=C(C=C170)OC(=O)OC(=O)C171=C170C=C(C=C171)OC(=O)OC(=O)C172=C171C=C(C=C172)OC(=O)OC(=O)C173=C172C=C(C=C173)OC(=O)OC(=O)C174=C173C=C(C=C174)OC(=O)OC(=O)C175=C174C=C(C=C175)OC(=O)OC(=O)C176=C175C=C(C=C176)OC(=O)OC(=O)C177=C176C=C(C=C177)OC(=O)OC(=O)C178=C177C=C(C=C178)OC(=O)OC(=O)C179=C178C=C(C=C179)OC(=O)OC(=O)C180=C179C=C(C=C180)OC(=O)OC(=O)C181=C180C=C(C=C181)OC(=O)OC(=O)C182=C181C=C(C=C182)OC(=O)OC(=O)C183=C182C=C(C=C183)OC(=O)OC(=O)C184=C183C=C(C=C184)OC(=O)OC(=O)C185=C184C=C(C=C185)OC(=O)OC(=O)C186=C185C=C(C=C186)OC(=O)OC(=O)C187=C186C=C(C=C187)OC(=O)OC(=O)C188=C187C=C(C=C188)OC(=O)OC(=O)C189=C188C=C(C=C189)OC(=O)OC(=O)C190=C189C=C(C=C190)OC(=O)OC(=O)C191=C190C=C(C=C191)OC(=O)OC(=O)C192=C191C=C(C=C192)OC(=O)OC(=O)C193=C192C=C(C=C193)OC(=O)OC(=O)C194=C193C=C(C=C194)OC(=O)OC(=O)C195=C194C=C(C=C195)OC(=O)OC(=O)C196=C195C=C(C=C196)OC(=O)OC(=O)C197=C196C=C(C=C197)OC(=O)OC(=O)C198=C197C=C(C=C198)OC(=O)OC(=O)C199=C198C=C(C=C199)OC(=O)OC(=O)C200=C199C=C(C=C200)OC(=O)OC(=O)C201=C200C=C(C=C201)OC(=O)OC(=O)C202=C201C=C(C=C202)OC(=O)OC(=O)C203=C202C=C(C=C203)OC(=O)OC(=O)C204=C203C=C(C=C204)OC(=O)OC(=O)C205=C204C=C(C=C205)OC(=O)OC(=O)C206=C205C=C(C=C206)OC(=O)OC(=O)C207=C206C=C(C=C207)OC(=O)OC(=O)C208=C207C=C(C=C208)OC(=O)OC(=O)C209=C208C=C(C=C209)OC(=O)OC(=O)C210=C209C=C(C=C210)OC(=O)OC(=O)C211=C210C=C(C=C211)OC(=O)OC(=O)C212=C211C=C(C=C212)OC(=O)OC(=O)C213=C212C=C(C=C213)OC(=O)OC(=O)C214=C213C=C(C=C214)OC(=O)OC(=O)C215=C214C=C(C=C215)OC(=O)OC(=O)C216=C215C=C(C=C216)OC(=O)OC(=O)C217=C216C=C(C=C217)OC(=O)OC(=O)C218=C217C=C(C=C218)OC(=O)OC(=O)C219=C218C=C(C=C219)OC(=O)OC(=O)C220=C219C=C(C=C220)OC(=O)OC(=O)C221=C220C=C(C=C221)OC(=O)OC(=O)C222=C221C=C(C=C222)OC(=O)OC(=O)C223=C222C=C(C=C223)OC(=O)OC(=O)C224=C223C=C(C=C224)OC(=O)OC(=O)C225=C224C=C(C=C225)OC(=O)OC(=O)C226=C225C=C(C=C226)OC(=O)OC(=O)C227=C226C=C(C=C227)OC(=O)OC(=O)C228=C227C=C(C=C228)OC(=O)OC(=O)C229=C228C=C(C=C229)OC(=O)OC(=O)C230=C229C=C(C=C230)OC(=O)OC(=O)C231=C230C=C(C=C231)OC(=O)OC(=O)C232=C231C=C(C=C232)OC(=O)OC(=O)C233=C232C=C(C=C233)OC(=O)OC(=O)C234=C233C=C(C=C234)OC(=O)OC(=O)C235=C234C=C(C=C235)OC(=O)OC(=O)C236=C235C=C(C=C236)OC(=O)OC(=O)C237=C236C=C(C=C237)OC(=O)OC(=O)C238=C237C=C(C=C238)OC(=O)OC(=O)C239=C238C=C(C=C239)OC(=O)OC(=O)C240=C239C=C(C=C240)OC(=O)OC(=O)C241=C240C=C(C=C241)OC(=O)OC(=O)C242=C241C=C(C=C242)OC(=O)OC(=O)C243=C242C=C(C=C243)OC(=O)OC(=O)C244=C243C=C(C=C244)OC(=O)OC(=O)C245=C244C=C(C=C245)OC(=O)OC(=O)C246=C245C=C(C=C246)OC(=O)OC(=O)C247=C246C=C(C=C247)OC(=O)OC(=O)C248=C247C=C(C=C248)OC(=O)OC(=O)C249=C248C=C(C=C249)OC(=O)OC(=O)C250=C249C=C(C=C250)OC(=O)OC(=O)C251=C250C=C(C=C251)OC(=O)OC(=O)C252=C251C=C(C=C252)OC(=O)OC(=O)C253=C252C=C(C=C253)OC(=O)OC(=O)C254=C253C=C(C=C254)OC(=O)OC(=O)C255=C254C=C(C=C255)OC(=O)OC(=O)C256=C255C=C(C=C256)OC(=O)OC(=O)C257=C256C=C(C=C257)OC(=O)OC(=O)C258=C257C=C(C=C258)OC(=O)OC(=O)C259=C258C=C(C=C259)OC(=O)OC(=O)C260=C259C=C(C=C260)OC(=O)OC(=O)C261=C260C=C(C=C261)OC(=O)OC(=O)C262=C261C=C(C=C262)OC(=O)OC(=O)C263=C262C=C(C=C263)OC(=O)OC(=O)C264=C263C=C(C=C264)OC(=O)OC(=O)C265=C264C=C(C=C265)OC(=O)OC(=O)C266=C265C=C(C=C266)OC(=O)OC(=O)C267=C266C=C(C=C267)OC(=O)OC(=O)C268=C267C=C(C=C268)OC(=O)OC(=O)C269=C268C=C(C=C269)OC(=O)OC(=O)C270=C269C=C(C=C270)OC(=O)OC(=O)C271=C270C=C(C=C271)OC(=O)OC(=O)C272=C271C=C(C=C272)OC(=O)OC(=O)C273=C272C=C(C=C273)OC(=O)OC(=O)C274=C273C=C(C=C274)OC(=O)OC(=O)C275=C274C=C(C=C275)OC(=O)OC(=O)C276=C275C=C(C=C276)OC(=O)OC(=O)C277=C276C=C(C=C277)OC(=O)OC(=O)C278=C277C=C(C=C278)OC(=O)OC(=O)C279=C278C=C(C=C279)OC(=O)OC(=O)C280=C279C=C(C=C280)OC(=O)OC(=O)C281=C280C=C(C=C281)OC(=O)OC(=O)C282=C281C=C(C=C282)OC(=O)OC(=O)C283=C282C=C(C=C283)OC(=O)OC(=O)C284=C283C=C(C=C284)OC(=O)OC(=O)C285=C284C=C(C=C285)OC(=O)OC(=O)C286=C285C=C(C=C286)OC(=O)OC(=O)C287=C286C=C(C=C287)OC(=O)OC(=O)C288=C287C=C(C=C288)OC(=O)OC(=O)C289=C288C=C(C=C289)OC(=O)OC(=O)C290=C289C=C(C=C290)OC(=O)OC(=O)C291=C290C=C(C=C291)OC(=O)OC(=O)C292=C291C=C(C=C292)OC(=O)OC(=O)C293=C292C=C(C=C293)OC(=O)OC(=O)C294=C293C=C(C=C294)OC(=O)OC(=O)C295=C294C=C(C=C295)OC(=O)OC(=O)C296=C295C=C(C=C296)OC(=O)OC(=O)C297=C296C=C(C=C297)OC(=O)OC(=O)C298=C297C=C(C=C298)OC(=O)OC(=O)C299=C298C=C(C=C299)OC(=O)OC(=O)C300=C299C=C(C=C300)OC(=O)OC(=O)C301=C300C=C(C=C301)OC(=O)OC(=O)C302=C301C=C(C=C302)OC(=O)OC(=O)C303=C302C=C(C=C303)OC(=O)OC(=O)C304=C303C=C(C=C304)OC(=O)OC(=O)C305=C304C=C(C=C305)OC(=O)OC(=O)C306=C305C=C(C=C306)OC(=O)OC(=O)C307=C306C=C(C=C307)OC(=O)OC(=O)C308=C307C=C(C=C308)OC(=O)OC(=O)C309=C308C=C(C=C309)OC(=O)OC(=O)C310=C309C=C(C=C310)OC(=O)OC(=O)C311=C310C=C(C=C311)OC(=O)OC(=O)C312=C311C=C(C=C312)OC(=O)OC(=O)C313=C312C=C(C=C313)OC(=O)OC(=O)C314=C313C=C(C=C314)OC(=O)OC(=O)C315=C314C=C(C=C315)OC(=O)OC(=O)C316=C315C=C(C=C316)OC(=O)OC(=O)C317=C316C=C(C=C317)OC(=O)OC(=O)C318=C317C=C(C=C318)OC(=O)OC(=O)C319=C318C=C(C=C319)OC(=O)OC(=O)C320=C319C=C(C=C320)OC(=O)OC(=O)C321=C320C=C(C=C321)OC(=O)OC(=O)C322=C321C=C(C=C322)OC(=O)OC(=O)C323=C322C=C(C=C323)OC(=O)OC(=O)C324=C323C=C(C=C324)OC(=O)OC(=O)C325=C324C=C(C=C325)OC(=O)OC(=O)C326=C325C=C(C=C326)OC(=O)OC(=O)C327=C326C=C(C=C327)OC(=O)OC(=O)C328=C327C=C(C=C328)OC(=O)OC(=O)C329=C328C=C(C=C329)OC(=O)OC(=O)C330=C329C=C(C=C330)OC(=O)OC(=O)C331=C330C=C(C=C331)OC(=O)OC(=O)C332=C331C=C(C=C332)OC(=O)OC(=O)C333=C332C=C(C=C333)OC(=O)OC(=O)C334=C333C=C(C=C334)OC(=O)OC(=O)C335=C334C=C(C=C335)OC(=O)OC(=O)C336=C335C=C(C=C336)OC(=O)OC(=O)C337=C336C=C(C=C337)OC(=O)OC(=O)C338=C337C=C(C=C338)OC(=O)OC(=O)C339=C338C=C(C=C339)OC(=O)OC(=O)C340=C339C=C(C=C340)OC(=O)OC(=O)C341=C340C=C(C=C341)OC(=O)OC(=O)C342=C341C=C(C=C342)OC(=O)OC(=O)C343=C342C=C(C=C343)OC(=O)OC(=O)C344=C343C=C(C=C344)OC(=O)OC(=O)C345=C344C=C(C=C345)OC(=O)OC(=O)C346=C345C=C(C=C346)OC(=O)OC(=O)C347=C346C=C(C=C347)OC(=O)OC(=O)C348=C347C=C(C=C348)OC(=O)OC(=O)C349=C348C=C(C=C349)OC(=O)OC(=O)C350=C349C=C(C=C350)OC(=O)OC(=O)C351=C350C=C(C=C351)OC(=O)OC(=O)C352=C351C=C(C=C352)OC(=O)OC(=O)C353=C352C=C(C=C353)OC(=O)OC(=O)C354=C353C=C(C=C354)OC(=O)OC(=O)C355=C354C=C(C=C355)OC(=O)OC(=O)C356=C355C=C(C=C356)OC(=O)OC(=O)C357=C356C=C(C=C357)OC(=O)OC(=O)C358=C357C=C(C=C358)OC(=O)OC(=O)C359=C358C=C(C=C359)OC(=O)OC(=O)C360=C359C=C(C=C360)OC(=O)OC(=O)C361=C360C=C(C=C361)OC(=O)OC(=O)C362=C361C=C(C=C362)OC(=O)OC(=O)C363=C362C=C(C=C363)OC(=O)OC(=O)C364=C363C=C(C=C364)OC(=O)OC(=O)C365=C364C=C(C=C365)OC(=O)OC(=O)C366=C365C=C(C=C366)OC(=O)OC(=O)C367=C366C=C(C=C367)OC(=O)OC(=O)C368=C367C=C(C=C368)OC(=O)OC(=O)C369=C368C=C(C=C369)OC(=O)OC(=O)C370=C369C=C(C=C370)OC(=O)OC(=O)C371=C370C=C(C=C371)OC(=O)OC(=O)C372=C371C=C(C=C372)OC(=O)OC(=O)C373=C372C=C(C=C373)OC(=O)OC(=O)C374=C373C=C(C=C374)OC(=O)OC(=O)C375=C374C=C(C=C375)OC(=O)OC(=O)C376=C375C=C(C=C376)OC(=O)OC(=O)C377=C376C=C(C=C377)OC(=O)OC(=O)C378=C377C=C(C=C378)OC(=O)OC(=O)C379=C378C=C(C=C379)OC(=O)OC(=O)C380=C379C=C(C=C380)OC(=O)OC(=O)C381=C380C=C(C=C381)OC(=O)OC(=O)C382=C381C=C(C=C382)OC(=O)OC(=O)C383=C382C=C(C=C383)OC(=O)OC(=O)C384=C383C=C(C=C384)OC(=O)OC(=O)C385=C384C=C(C=C385)OC(=O)OC(=O)C386=C385C=C(C=C386)OC(=O)OC(=O)C387=C386C=C(C=C387)OC(=O)OC(=O)C388=C387C=C(C=C388)OC(=O)OC(=O)C389=C388C=C(C=C389)OC(=O)OC(=O)C390=C389C=C(C=C390)OC(=O)OC(=O)C391=C390C=C(C=C391)OC(=O)OC(=O)C392=C391C=C(C=C392)OC(=O)OC(=O)C393=C392C=C(C=C393)OC(=O)OC(=O)C394=C393C=C(C=C394)OC(=O)OC(=O)C395=C394C=C(C=C395)OC(=O)OC(=O)C396=C395C=C(C=C396)OC(=O)OC(=O)C397=C396C=C(C
```

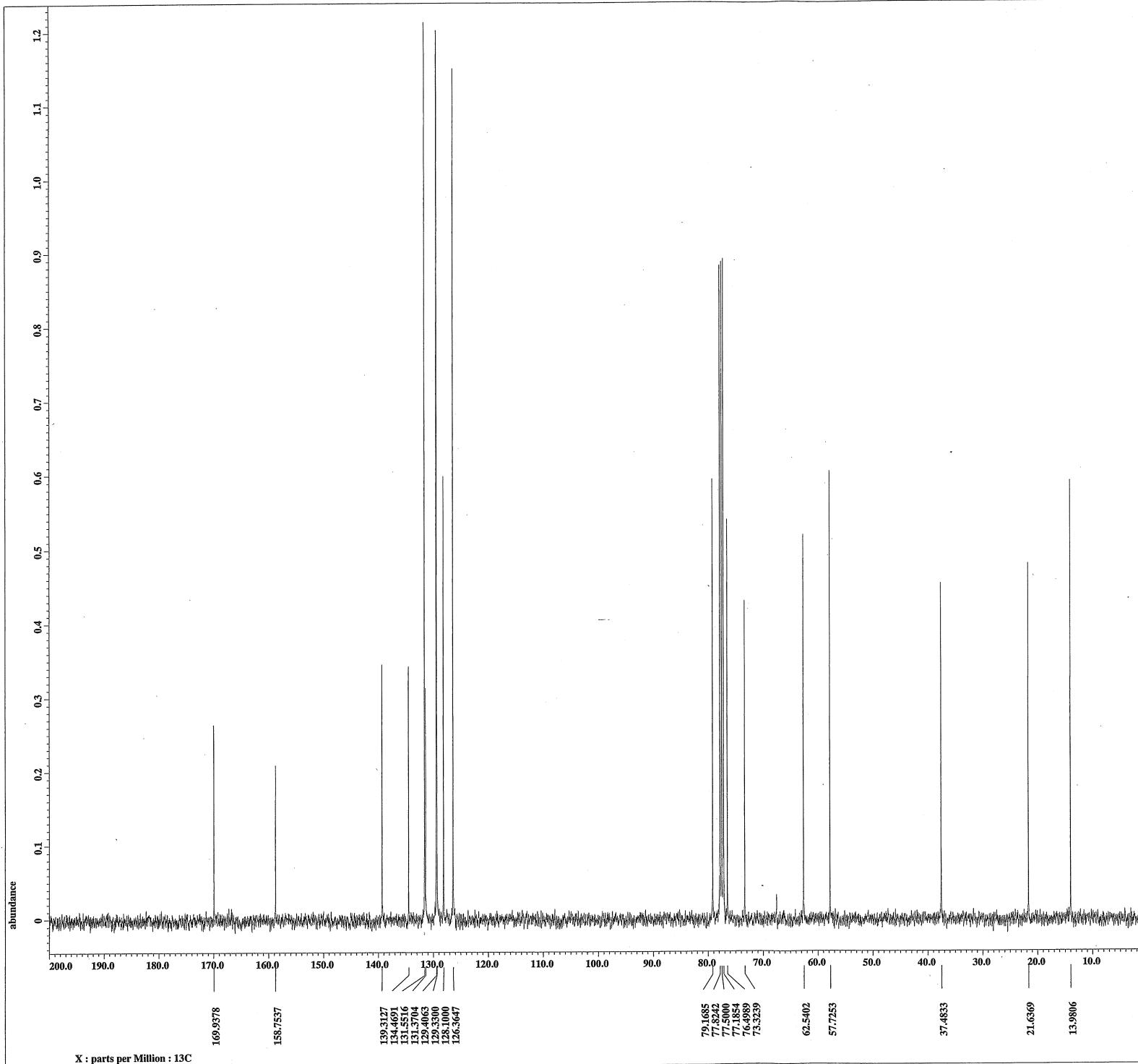


C:\WINNNMR98\DATA\tw838hf.als
TW-838:1H



DFILE C:\WINNNMR98\DATA\tw838hf.als
COMNT TW-838:1H
DATIM Thu Jun 28 09:22:18 2007
OBNUC 1H
EXMOD NON
OBFRO 395.75 MHz
OBSET 124.00 KHz
ORFIN 10277.0 Hz
POINT 32768
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 17





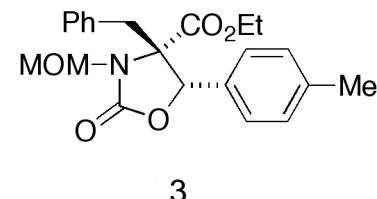
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sep : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TW-830 pure 13C-1.jdf

Filename = TW-830 pure 13C-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S1_13704
Solvent = CHLOROFORM-D
Creation_time = 23-MAY-2007 06:36:00
Revision_time = 23-MAY-2007 11:56:47
Current_time = 23-MAY-2007 11:57:13

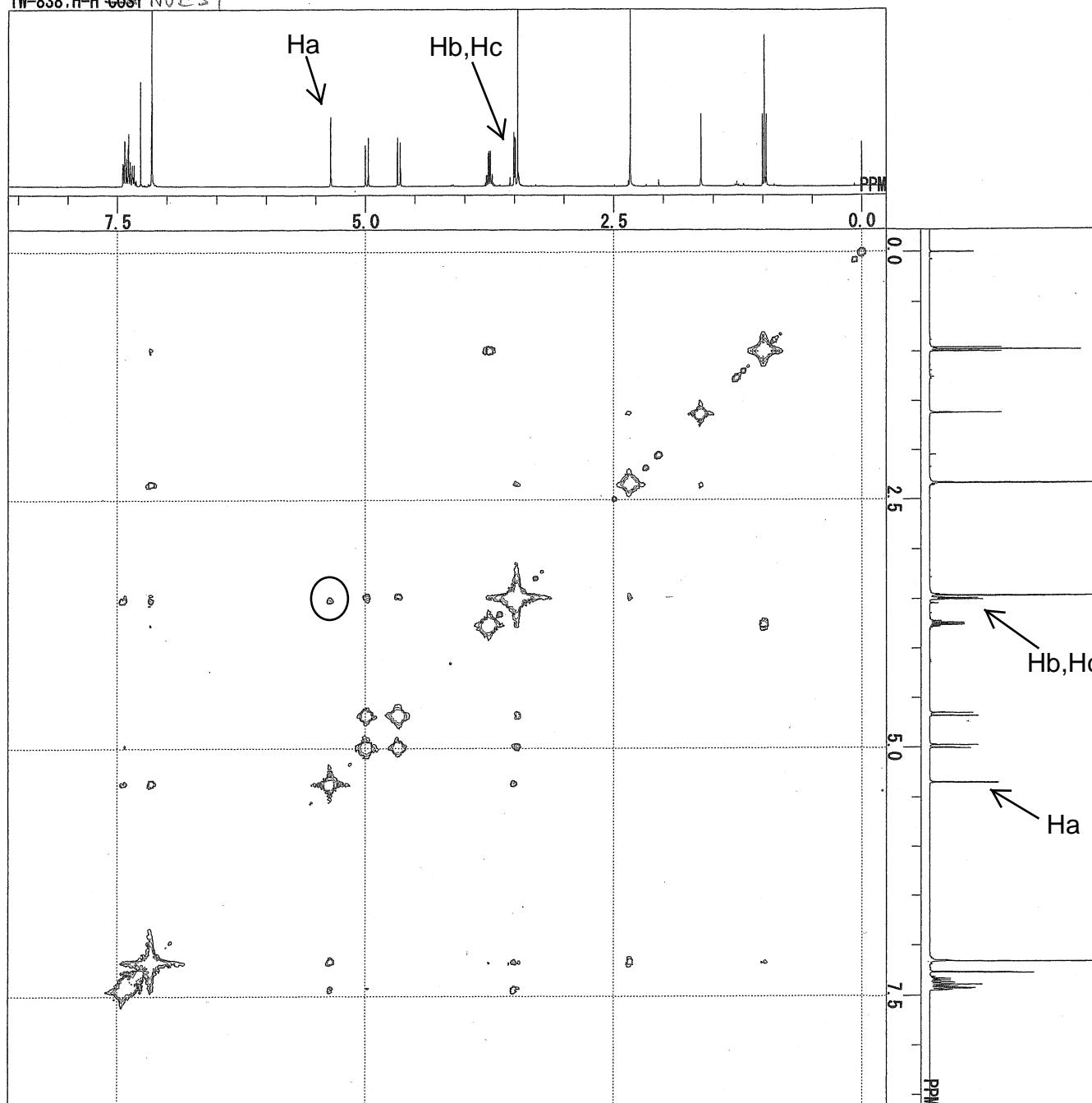
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.389766[M] (400[MHz])
X_ssw_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100 [ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 267
Total_scans = 267

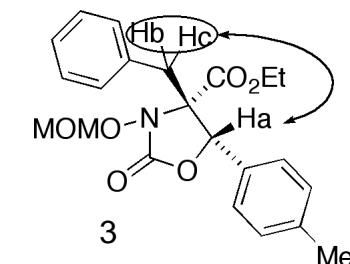
X_90_width = 10.2[us]
X_acc_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.8[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 19.8[dB]
Irr_atn_noe = 19.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recv_r_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get = 18.2[°C]



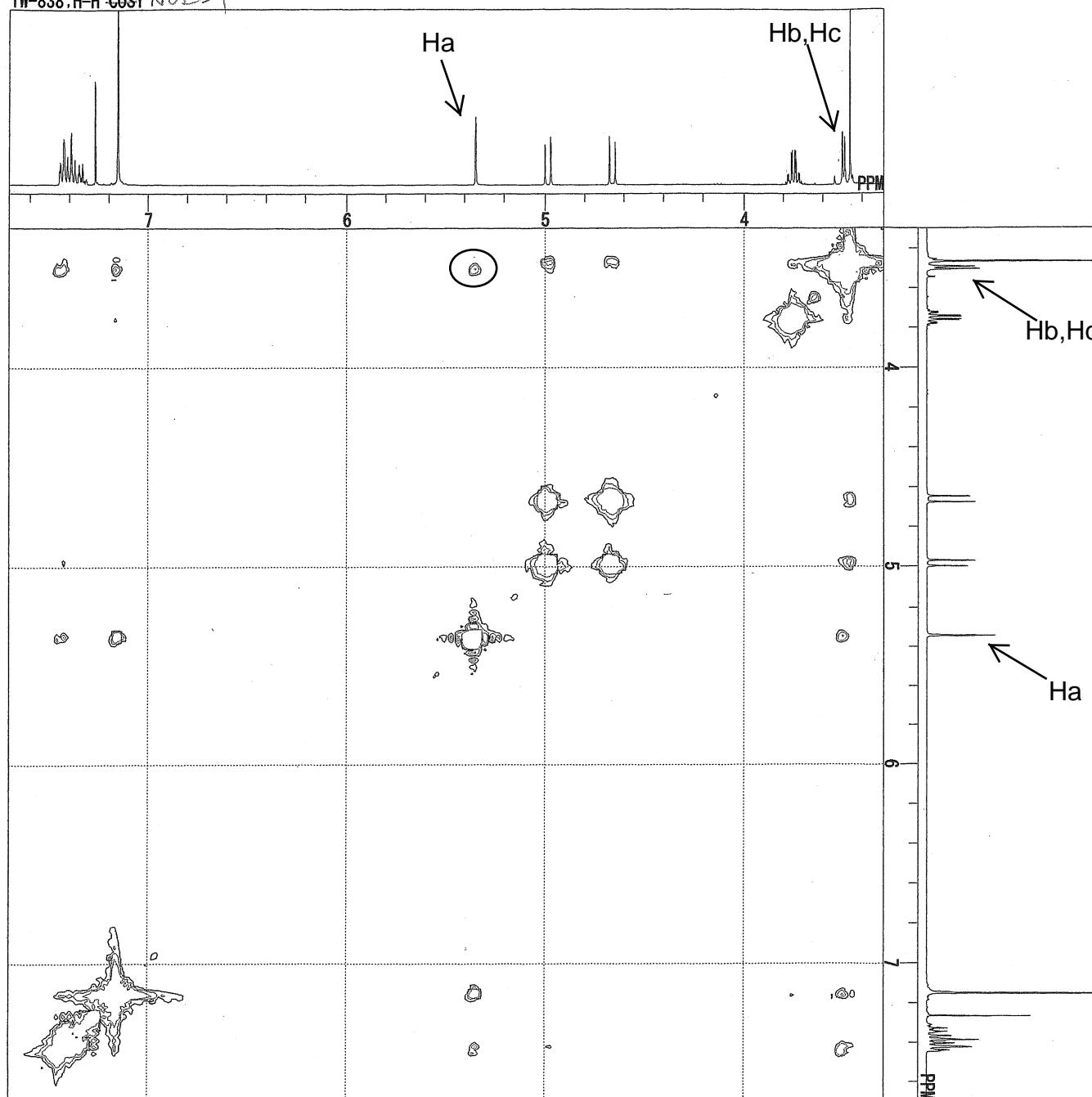
C:\WINNMR98\DATA\tw838noesy.ALS
TW-838:H-H NOESY



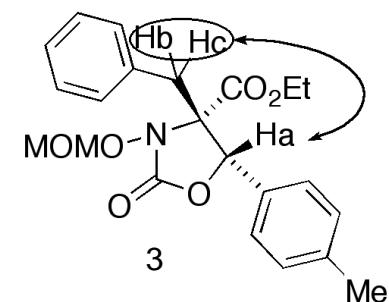
DFILE C:\WINNMR98\DATA\tw838noesy.ALS
COMNT TW-838:H-H NOESY
DATIM Thu Jun 28 13:05:26 2007
EXMOD VNOENH
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134.10 kHz
OBFIN 77.0 Hz
POINT 512
FREQU 3500.1 Hz
CLPNT 512
TODAT 256
CLFRQ 3496.5 Hz
SCANS 16
ACQTM 0.146 sec
PD 1.500 sec
PW1 12.6 us
PW2 12.6 us
PW3 10.0 us
P11 0.286 ms
P12 0.286 ms
P13 1000.00 ms
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCl₃
EXREF 0.00 ppm
CLEXR 0.00 ppm
RGAIN 16
OBATN 511
LOOP1 1



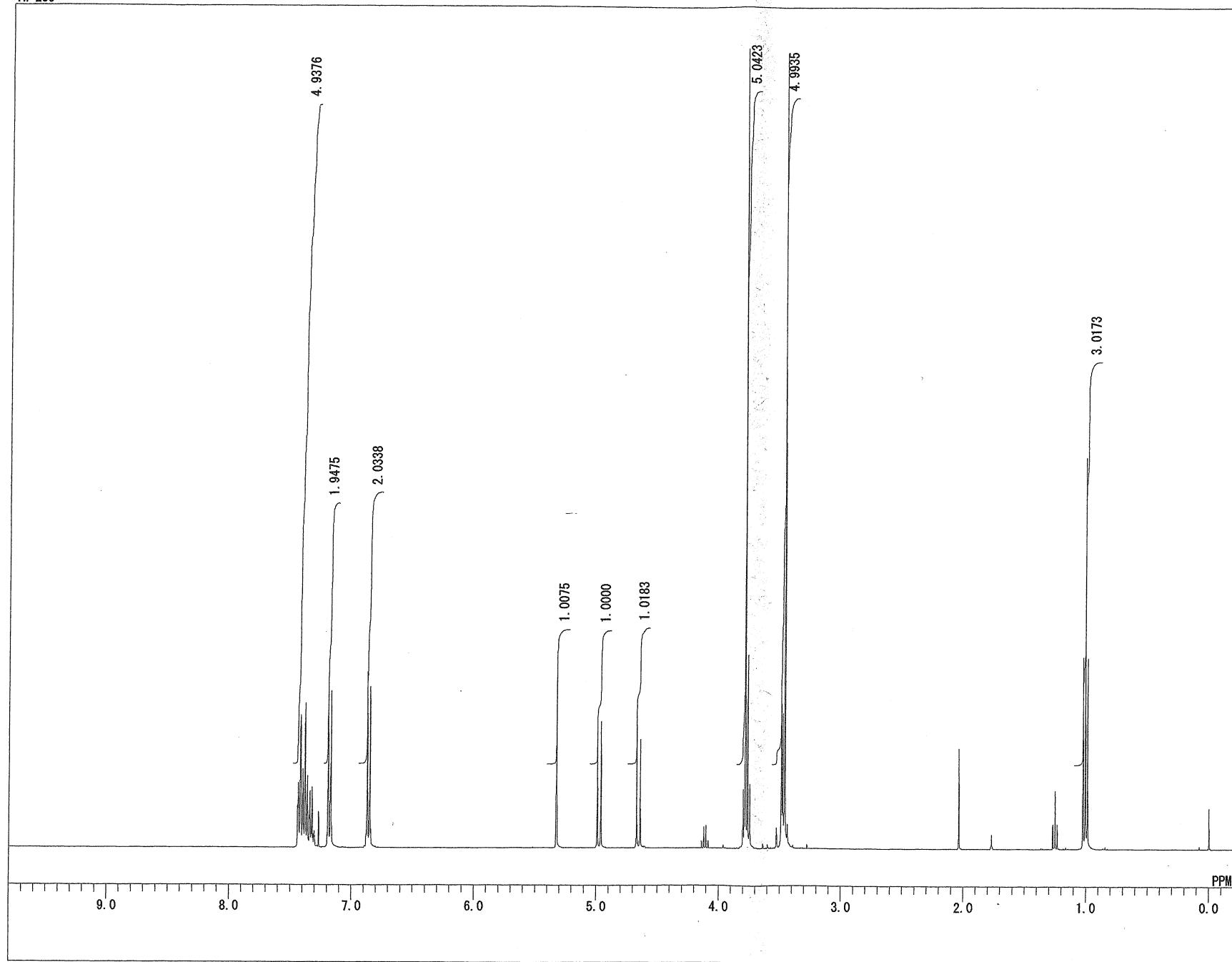
C:\WINNMR98\DATA\tw838noesy.ALS
TW-838;H-H-COSY NOESY



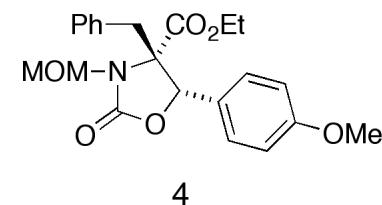
DFILE C:\WINNMR98\DATA\tw838noesy.ALS
COMNT TW-838;H-H-COSY NOESY
DATIM Thu Jun 28 13:05:26 2007
EXMOD VNOENH
OBNUC 1H
OBFREQ 395.75 MHz
OBSET 134.10 KHz
OBFIN 77.0 Hz
POINT 512
FREQU 3500.1 Hz
CLPNT 512
TODAT 256
CLFRQ 3496.5 Hz
SCANS 16
ACQTM 0.146 sec
PD 1.500 sec
PW1 12.6 us
PW2 12.6 us
PW3 10.0 us
P11 0.286 ms
P12 0.286 ms
P13 1000.00 ms
1H 21.6 c
CDCL3 0.00 ppm
SLVNT 0.00 ppm
EXREF CLEXR
RGAIN 16
OBATN 511
LOOP1 1

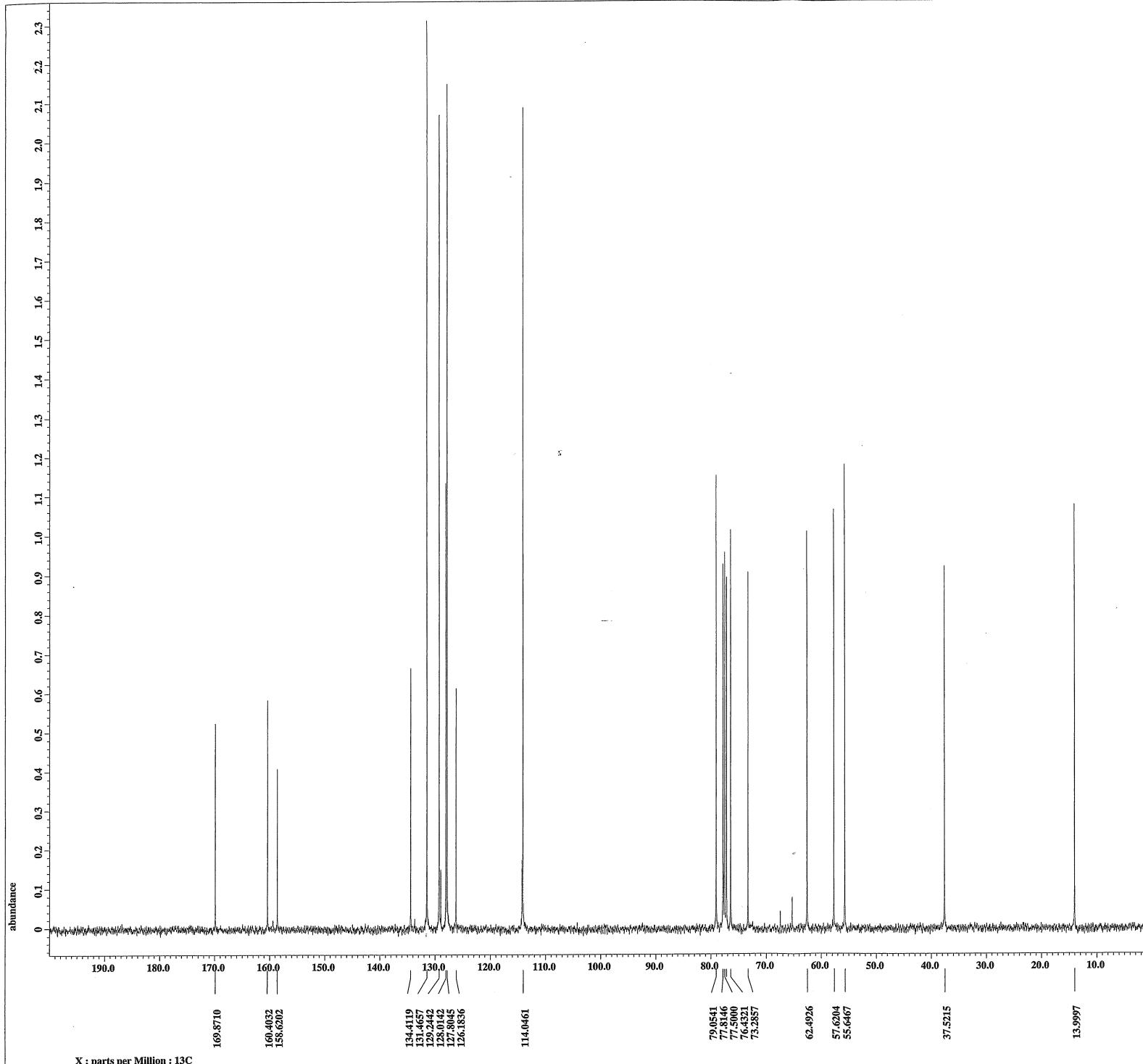


C:\WINNMR98\DATA\tw283.als
TW-283



DFILE C:\WINNMR98\DATA\tw283.als
COMT TW-283
DATM Tue Mar 14 14:31:18 2006
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 kHz
OBFIN 10277.0 Hz
POINT 32768
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCl₃
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 13





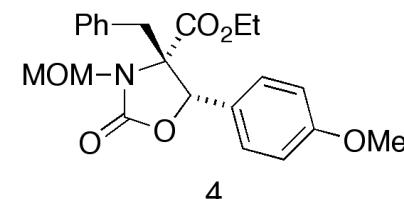
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sepx : 2.0[Hz] : 0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from:  $^{13}\text{C}$  No9, p-OMe-1.jdf
```

Filename = ^{13}C No9, p-OMe-3.jdf
Auther = delta
Experiment = single_pulse_dec
Serie_id = S#01764
Solvent = CHLOROFORM-D
Creation_time = 11-APR-2007 11:33:56
Revision_time = 11-APR-2007 16:53:22
Current_time = 11-APR-2007 16:53:38

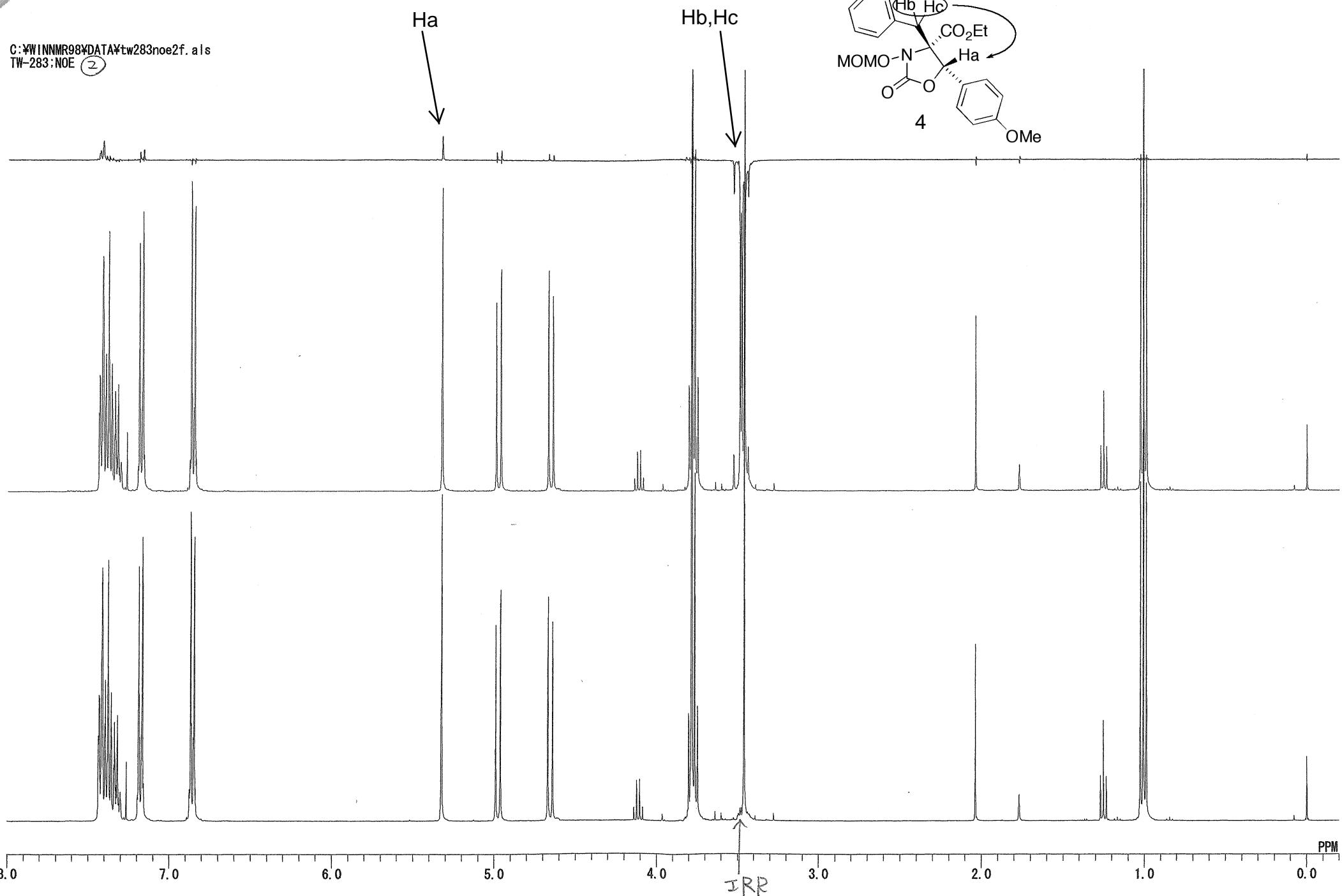
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = ^{13}C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR

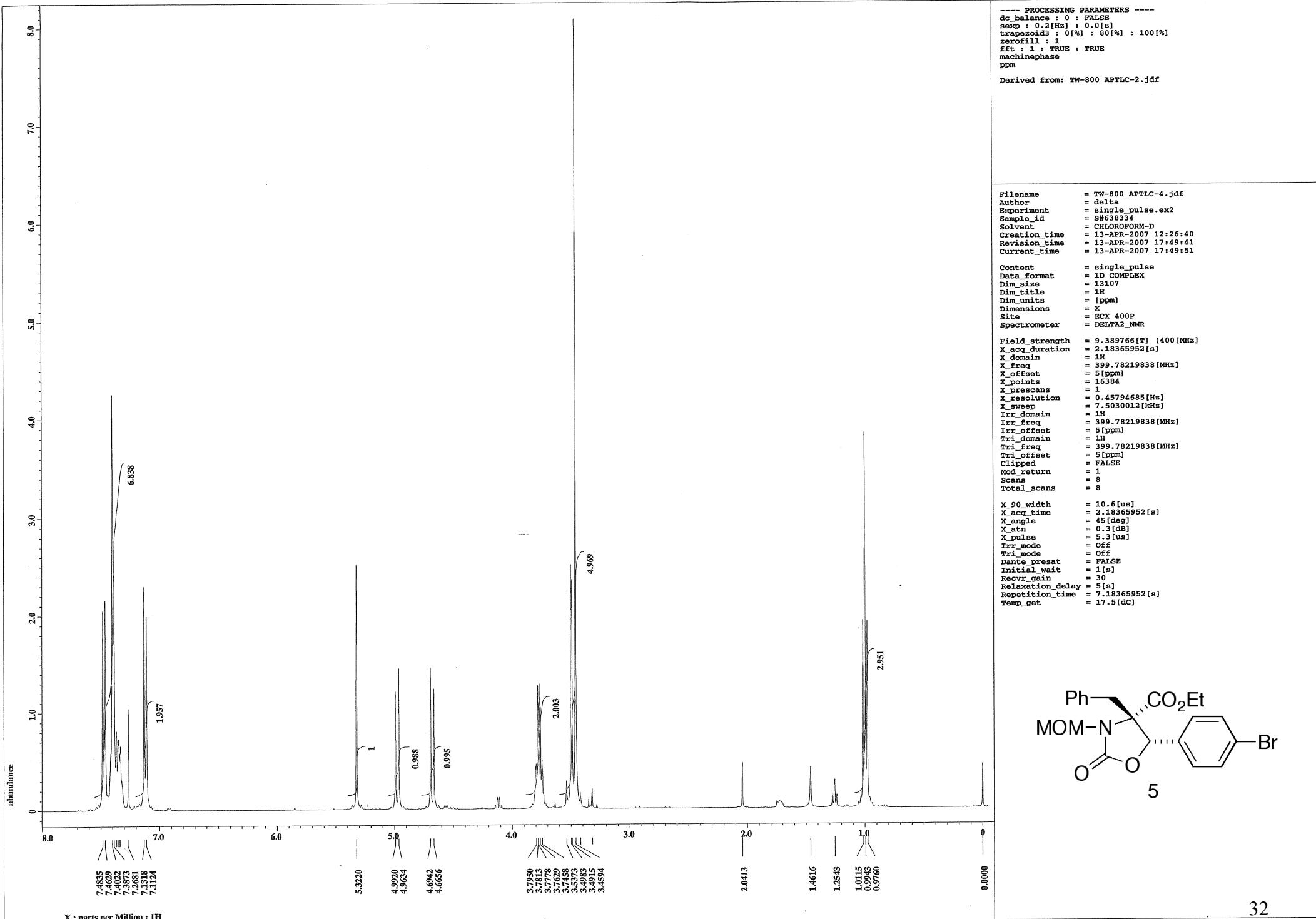
Field_strength = 9.389766[T] (400[MHz])
x_acq_duration = 1.04333312[s]
X_domain = ^{13}C
X_freq = 100.52530333[MHz]
X_offset = 100 [ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 180
Total_scans = 180

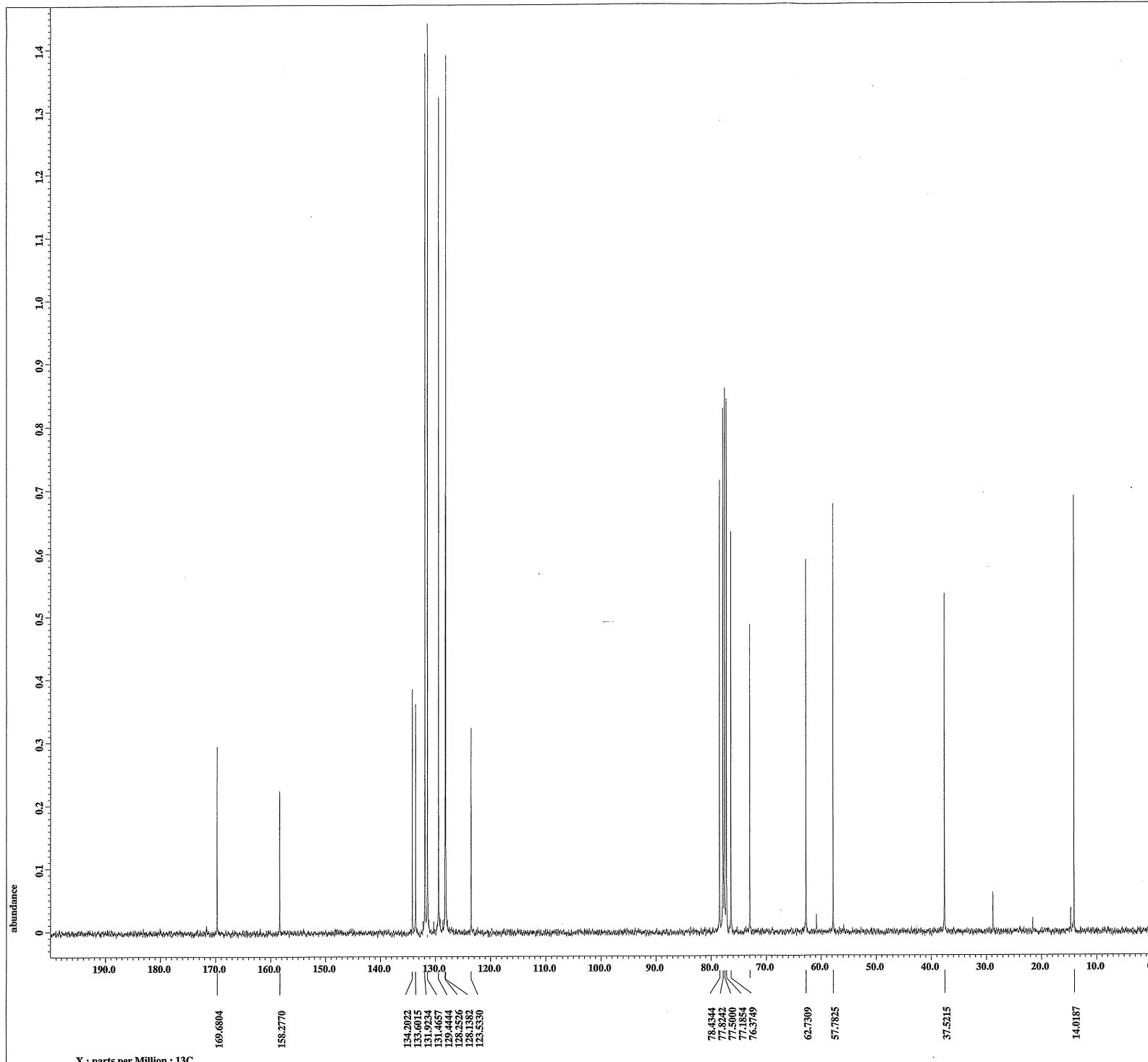
X_90_width = 10.2[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.8[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 19.8[dB]
Irr_atn_noe = 19.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recv_gain = 58
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get = 19[dc]



C:\WINNMR98\DATA\tw283noe2f.als
TW-283:NOE (2)







```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sep : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TW-800 APTLC 13C-1.jdf

Filename = TW-800 APTLC 13C-4.jd
Author =
Experiment = single_pulse_dec
Sample_id = S#642831
Solvent = CHLOROFORM-D
Creation_time = 13-APR-2007 13:26:07
Revision_time = 13-APR-2007 20:17:04
Current_time = 13-APR-2007 20:17:22

Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTAS_NMR

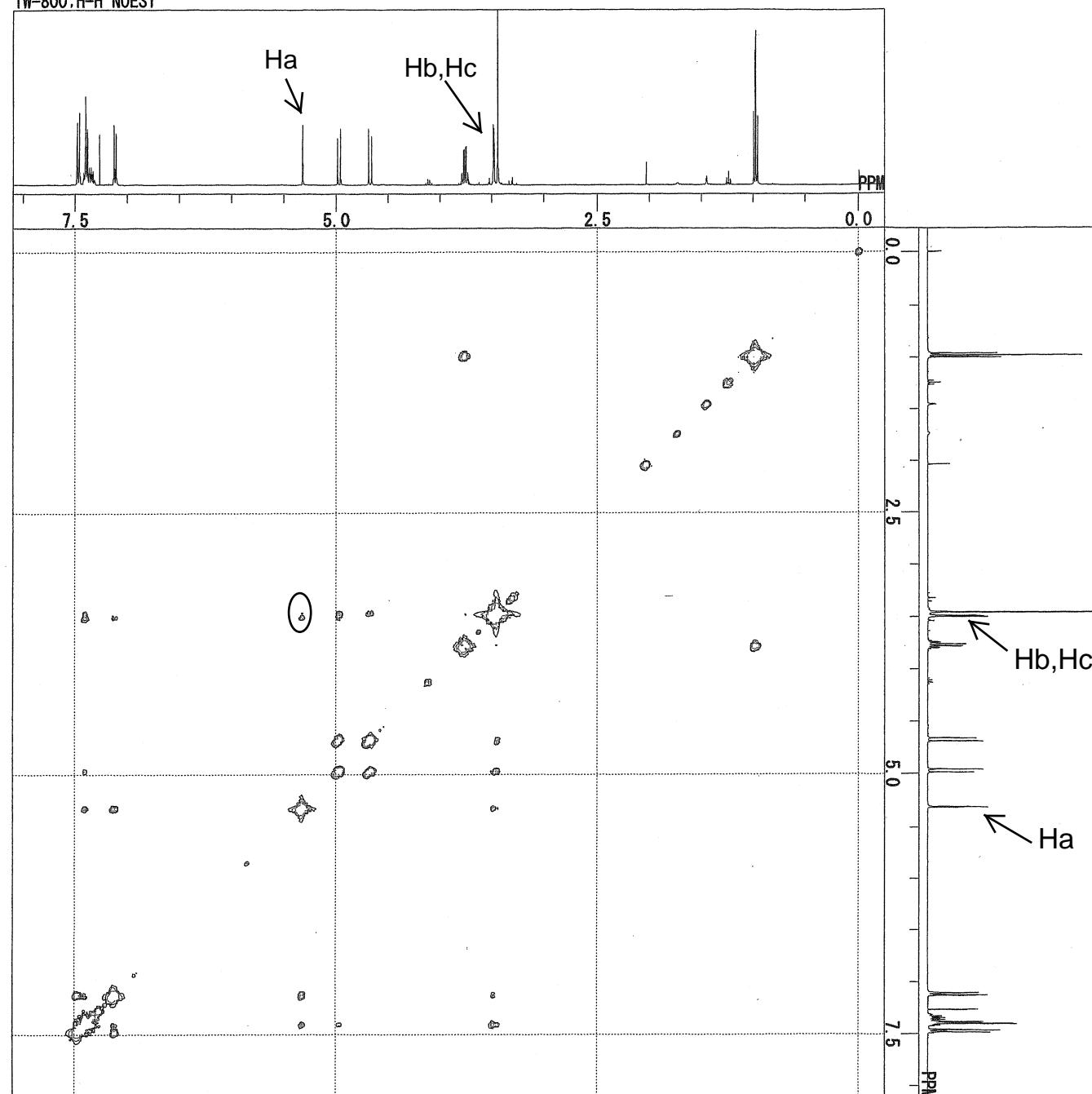
Field_strength = 9.389766["] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 1000
Total_scans = 1000

X_90_width = 10.2[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.8[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 19.8[dB]
Irr_atn_noe = 19.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recv_gain = 60
Relaxation_delay = 1[s]
Repetition_time = 3.04333312[s]
Temp_get = 19.2[dC]

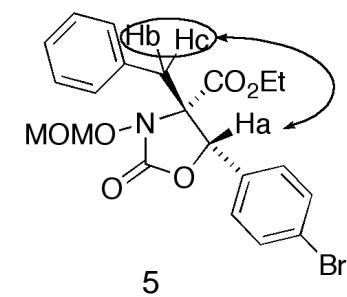
CC(=O)c1ccc(Br)cc1[C@H](COc2ccccc2)[C@@H](N([MOM-])C(=O)OC)c3ccccc3
```

5

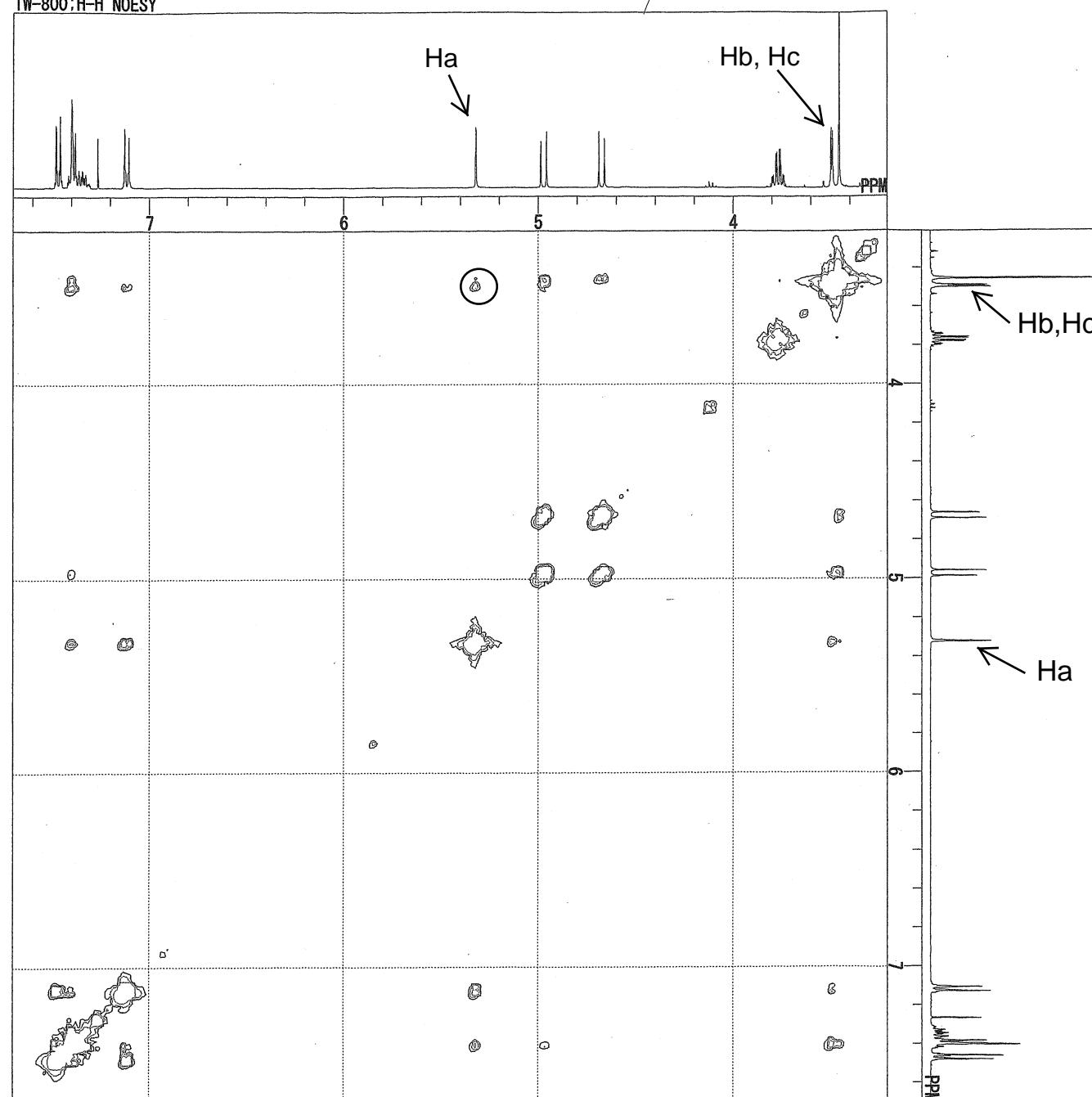
C:\WINNMR98\DATA\tw800noesy.ALS
TW-800;H-H NOESY



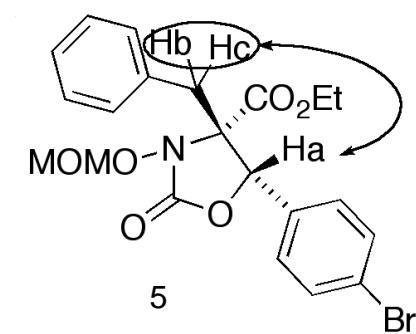
DFILE C:\WINNMR98\DATA\tw800noesy.ALS
COMNT TW-800;H-H NOESY
DATIM Wed Apr 18 15:47:57 2007
EXMOD VNOENH
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134.00 kHz
OBFIN 76.9 Hz
POINT 512
FREQU 3300.3 Hz
CLPNT 512
TODAT 256
CLFRQ 3300.3 Hz
SCANS 16
ACQTM 0.155 sec
PD 1.500 sec
PW1 12.8 us
PW2 12.8 us
PW3 10.0 us
PI1 0.303 ms
PI2 0.303 ms
PI3 1500.00 ms
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCl₃
EXREF 0.00 ppm
CLEXR 0.00 ppm
RGAIN 11
OBATN 511
LOOP1 1



C:\WINNMR98\DATA\tw800noesy.ALS
TW-800:H-H NOESY

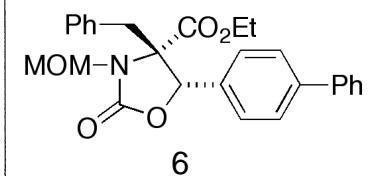
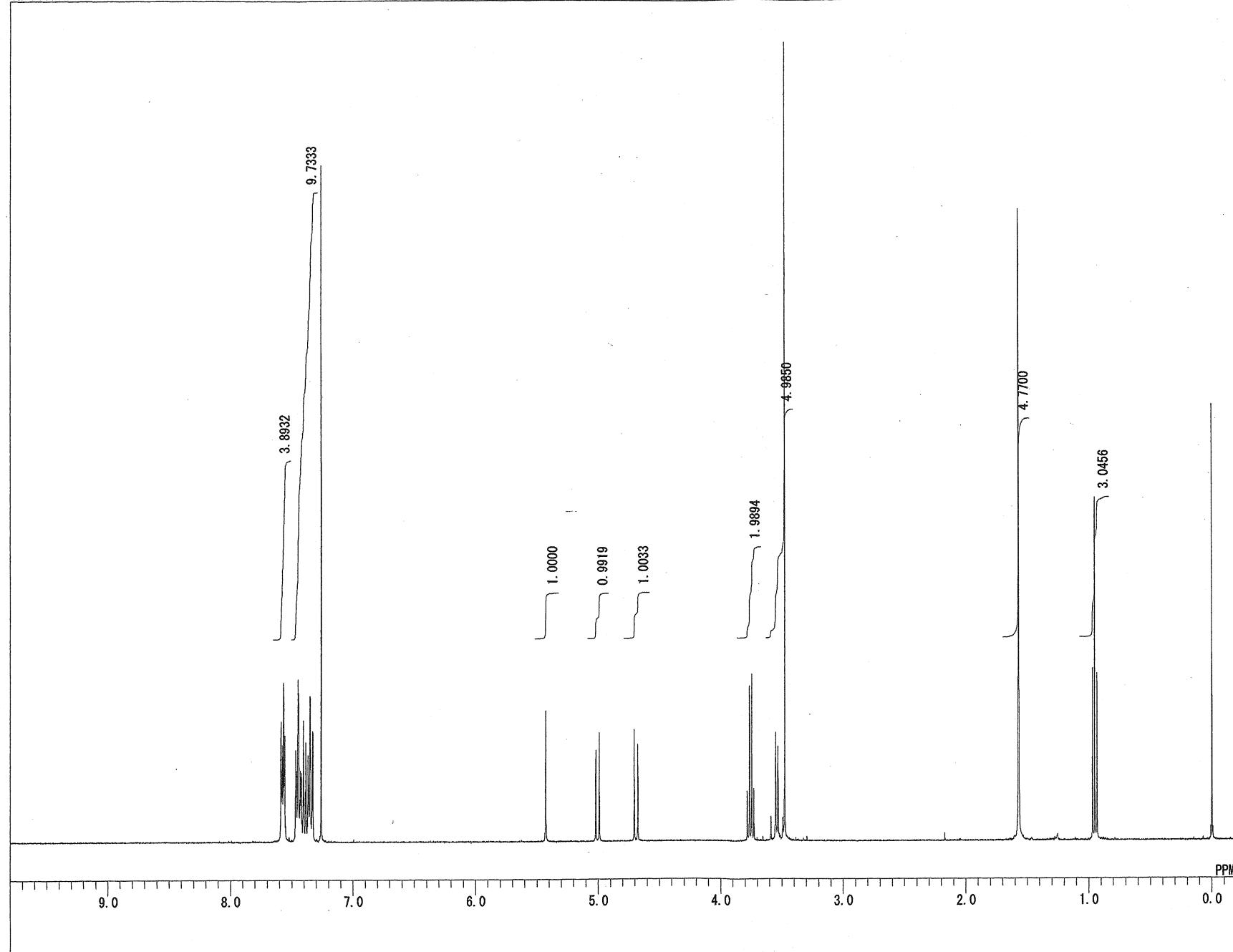


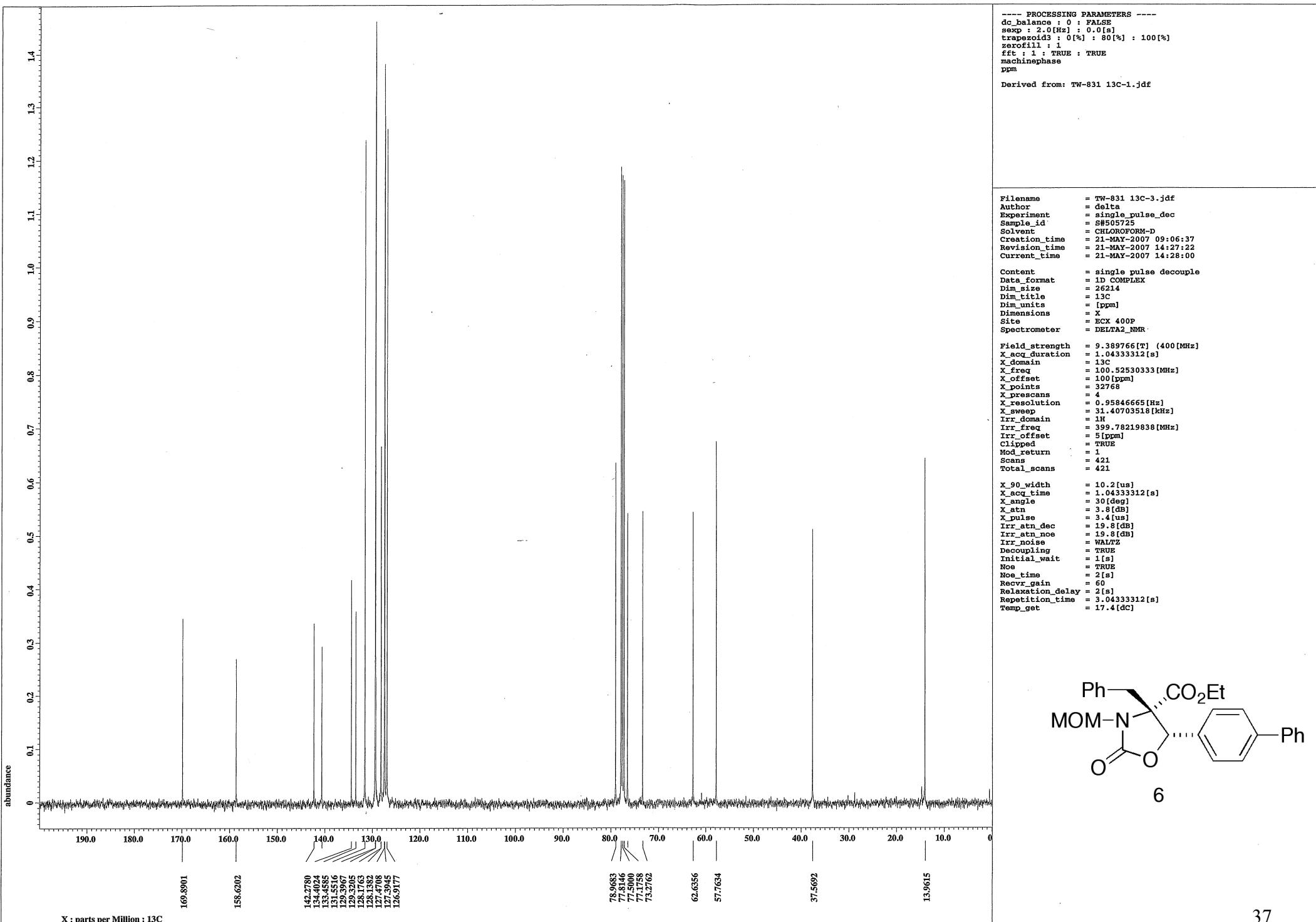
DFILE C:\WINNMR98\DATA\tw800noesy.ALS
COMNT TW-800:H-H NOESY
DATIM Wed Apr 18 15:47:57 2007
EXMOD VNOENH
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134.00 kHz
OBFIN 76.9 Hz
POINT 512
FREQU 3300.3 Hz
CLPNT 512
TODAT 256
CLFRQ 3300.3 Hz
SCANS 16
ACQTM 0.155 sec
PD 1.500 sec
PW1 12.8 us
PW2 12.8 us
PW3 10.0 us
P11 0.303 ms
P12 0.303 ms
P13 1500.00 ms
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCl₃
EXREF 0.00 ppm
CLEXR 0.00 ppm
RGAIN 11
OBATN 511
LOOP1 1



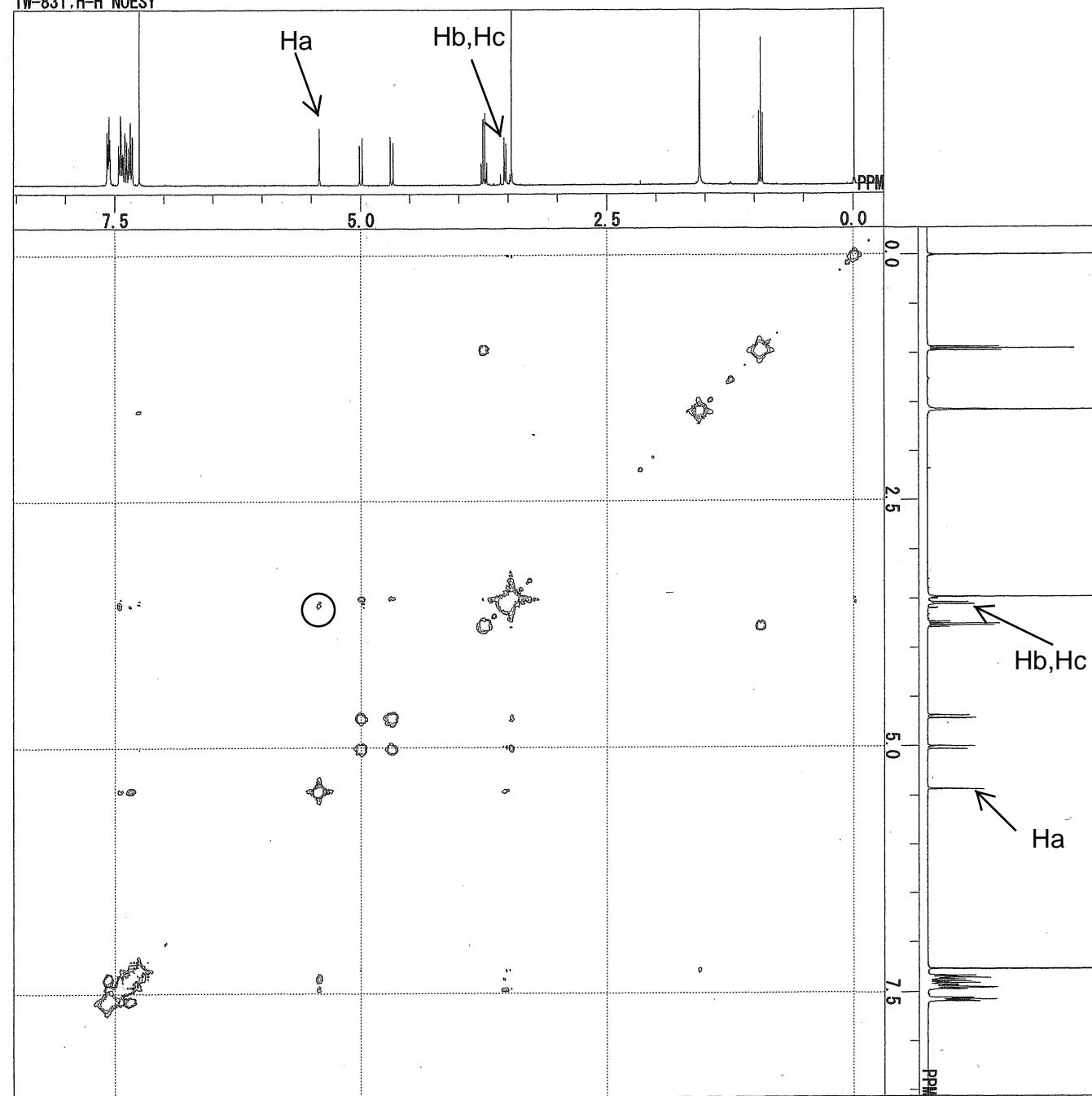
C:\WINNNMR98\DATA\tw831hf.als
TW-831:1H

DFILE C:\WINNNMR98\DATA\tw831hf.als
COMNT TW-831:1H
DATIM Wed Jun 27 12:20:26 2007
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 kHz
OBFIN 10277.0 Hz
POINT 32768
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 22

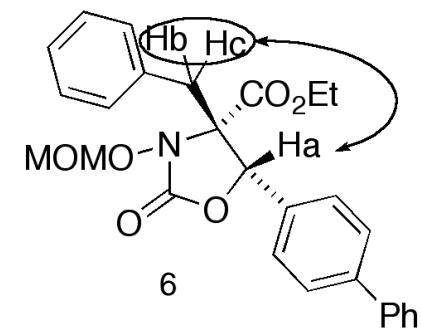




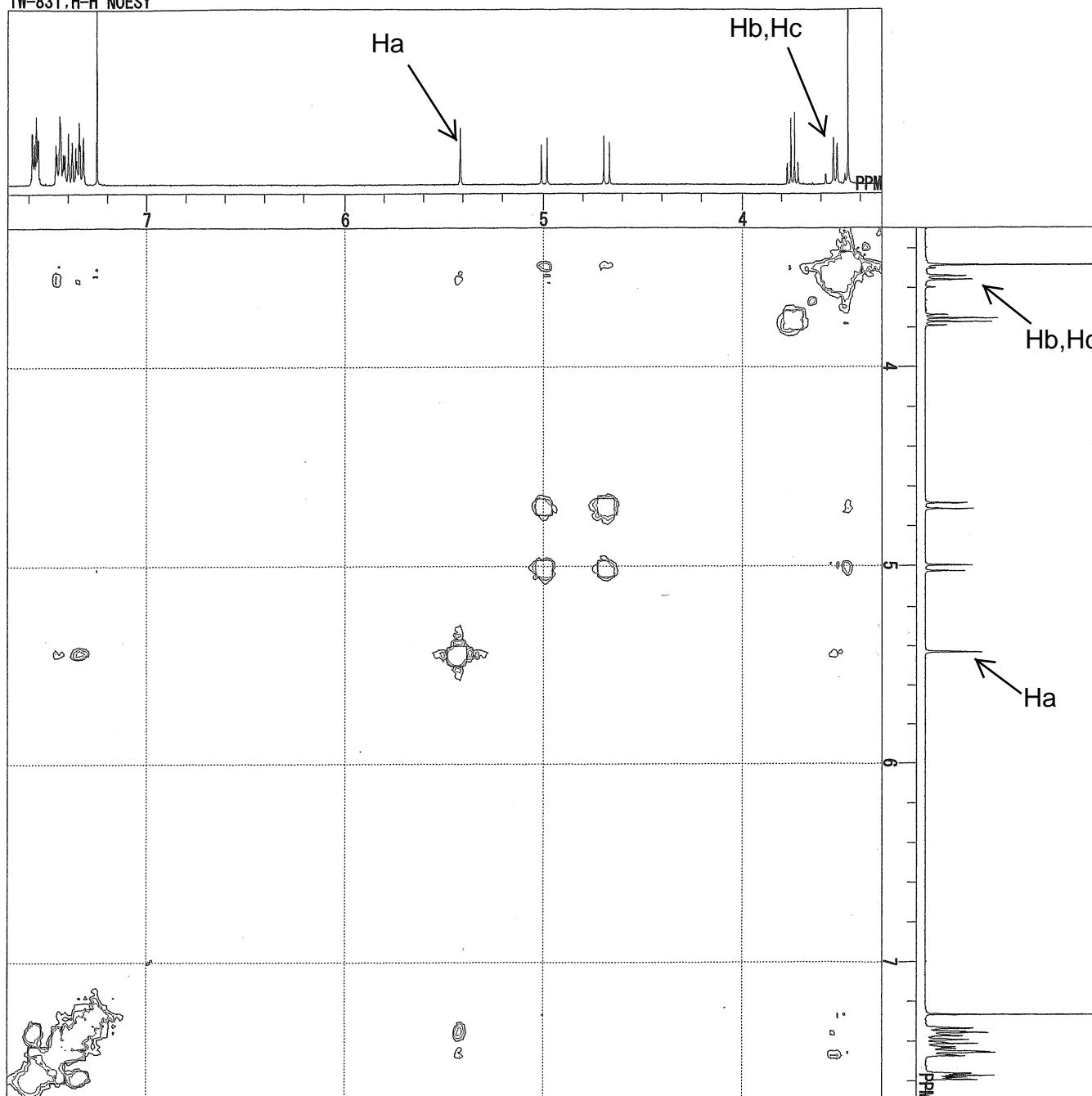
C:\WINNMR98\DATA\tw831noesy.ALS
TW-831:H-H NOESY



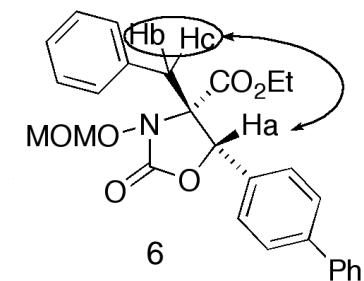
DFILE C:\WINNMR98\DATA\tw831noesy.ALS
COMNT TW-831:H-H NOESY
DATIM Wed Jun 27 14:02:00 2007
EXMOD VNOENH
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134.10 KHz
OBFIN 56.1 Hz
POINT 512
FREQU 3500.1 Hz
CLPNT 512
TODAT 256
CLFRQ 3496.5 Hz
SCANS 8
ACQTM 0.146 sec
PD 1.500 sec
PW1 12.6 us
PW2 12.6 us
PW3 10.0 us
PI1 0.286 ms
PI2 0.286 ms
PI3 1000.00 ms
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCl₃
EXREF 0.00 ppm
CLEXR 0.00 ppm
RGAIN 21
OBATN 511
LOOP1 1



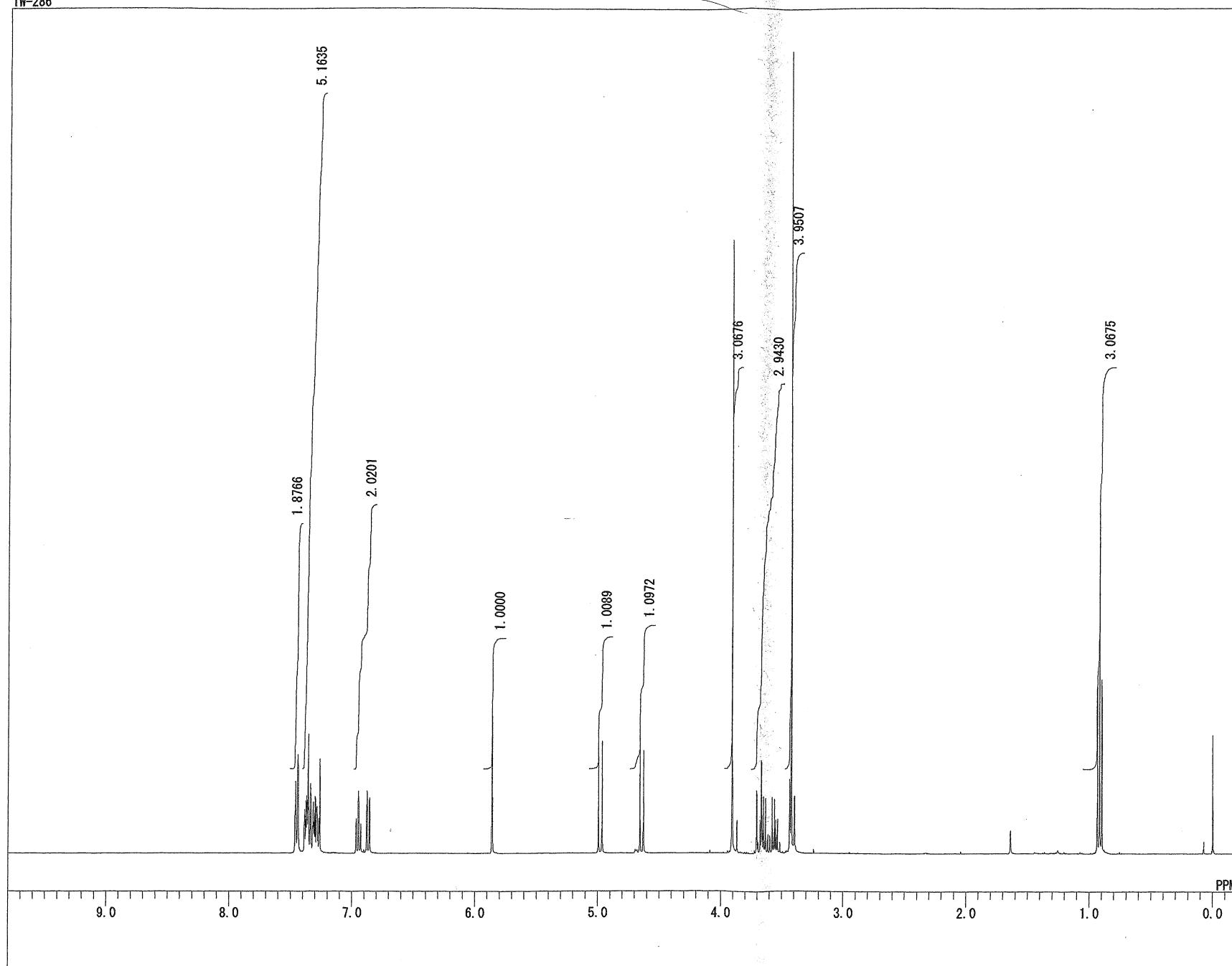
C:\WINNMR98\DATA\tw831noesy.ALS
TW-831:H-H NOESY



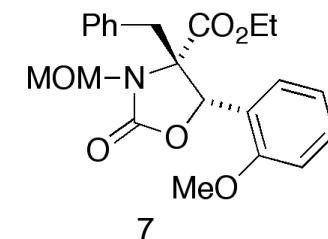
DFILE C:\WINNMR98\DATA\tw831noesy.ALS
COMNT TW-831:H-H NOESY
DATIM Wed Jun 27 14:02:00 2007
EXMOD VNOENH
OBNUC 1H
OBFREQ 395.75 MHz
OBSET 134.10 KHz
OBFIN 56.1 Hz
POINT 512
FREQU 3500.1 Hz
CLPNT 512
TODAT 256
CLFRQ 3496.5 Hz
SCANS 8
ACQTM 0.146 sec
PD 1.500 sec
PW1 12.6 us
PW2 12.6 us
PW3 10.0 us
P11 0.286 ms
P12 0.286 ms
P13 1000.00 ms
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL₃
EXREF 0.00 ppm
CLEXR 0.00 ppm
RGAIN 21
OBATN 511
LOOP1 1

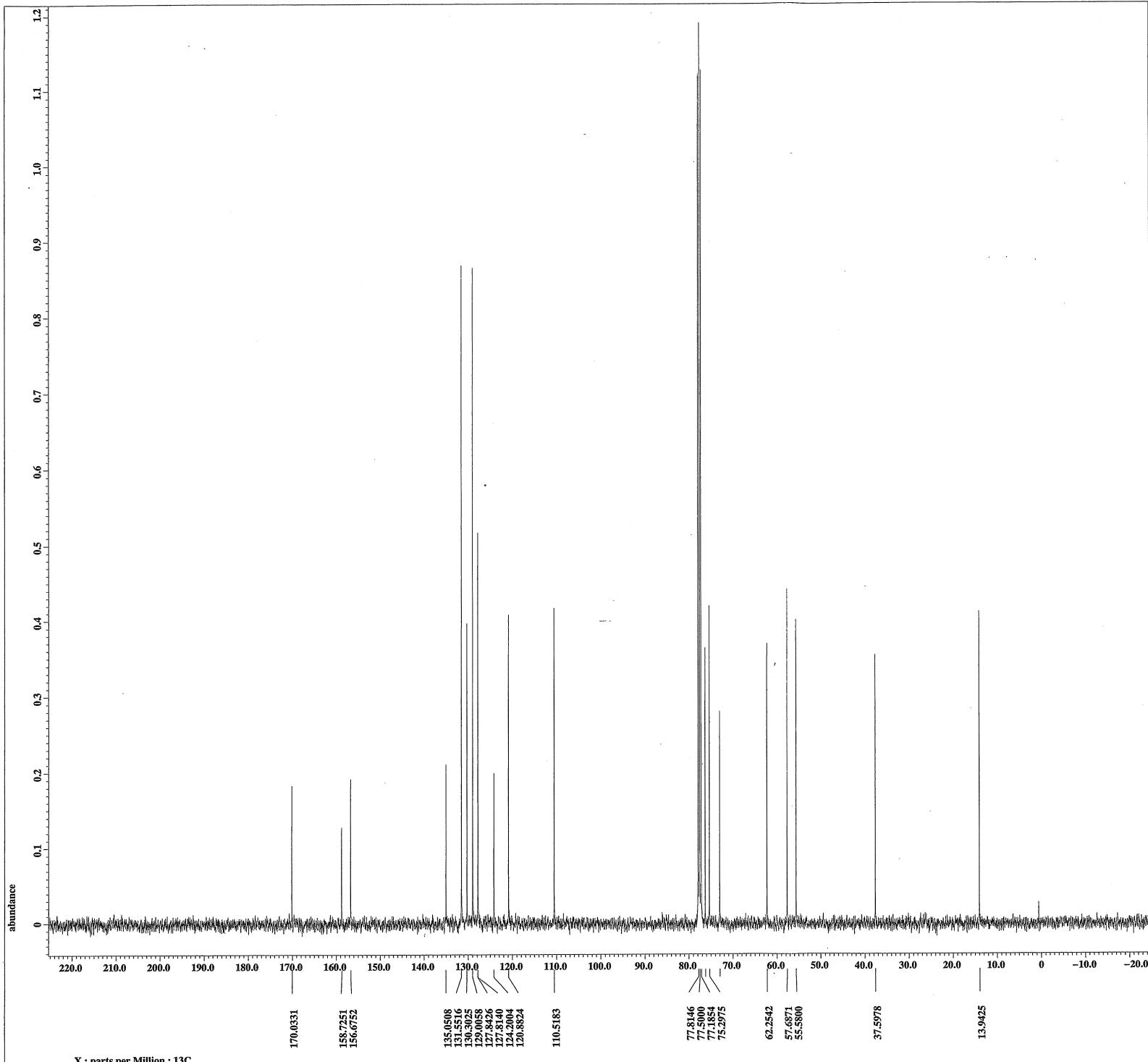


C:\WINNMR98\DATA\tw286.als
TW-286



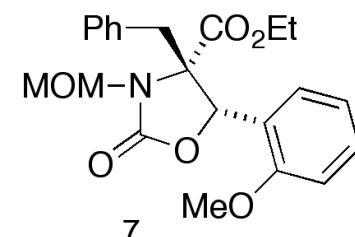
DFILE C:\WINNMR98\DATA\tw286.als
COMNT TW-286
DATIM Wed Mar 15 11:04:36 2006
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 kHz
OBFIN 10277.0 Hz
POINT 32768
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 μ s
IRNUC 1H
CTEMP 19.6 °C
SLVNT CDCl₃
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 17





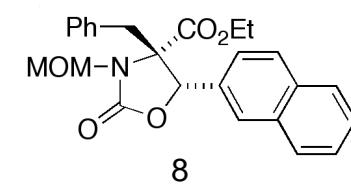
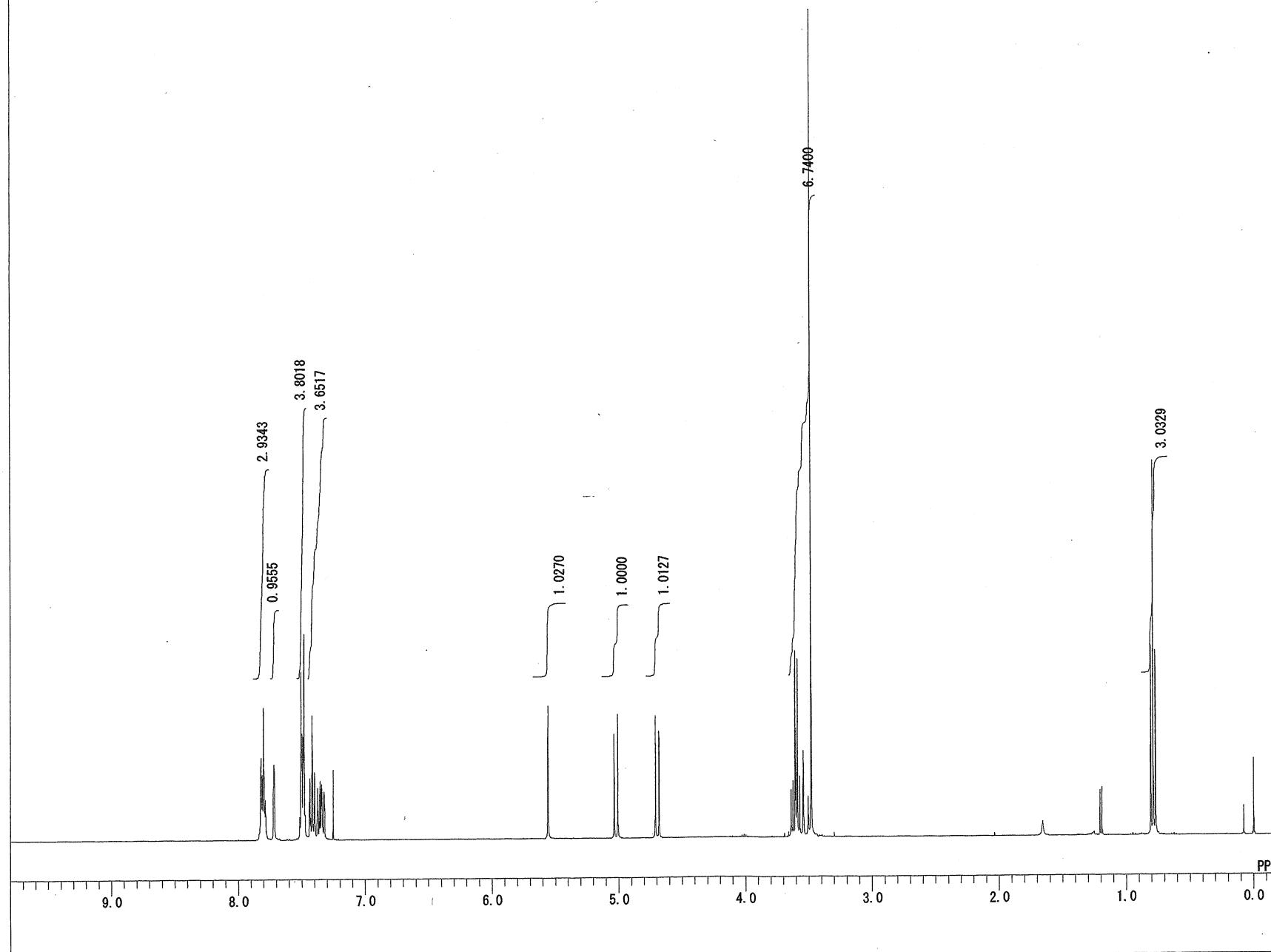
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sep : 2.0[Hz] : 0.0[s]
trapezoids : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from:  $^{13}\text{C}$  No12, o-OMe-1.jdf
```

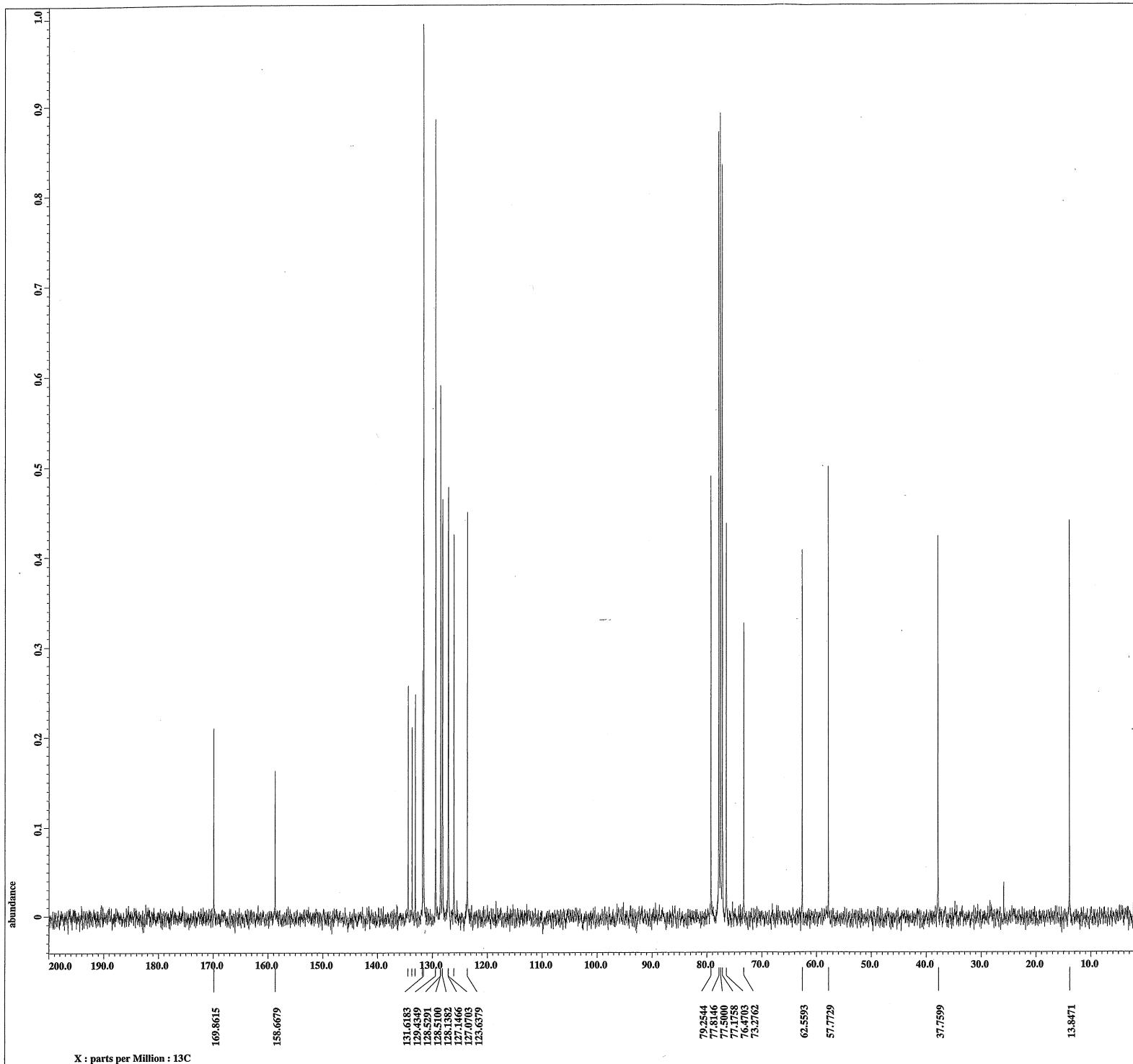
Filename	= ^{13}C No12, o-OMe-4.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S402030
Solvent	= CHLOROFORM-D
Creation_time	= 20-APR-2007 17:17:27
Revision_time	= 20-APR-2007 22:42:01
Current_time	= 20-APR-2007 22:42:33
Content	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= ^{13}C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz])
X_acq_duration	= 1.04333312[s]
X_domain	= ^{13}C
X_freq	= 100.52530333[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95846665[Hz]
X_sweep	= 31.40703518[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[NHz]
Irr_offset	= 5[ppm]
Clipped	= TRUE
Mod_return	= 1
Scans	= 342
Total_scans	= 342
X_90_width	= 10.2[us]
X_acq_time	= 1.04333312[s]
X_angle	= 30[deg]
X_atn	= 3.8[dB]
X_pulse	= 3.4[us]
Irr_atn_dec	= 19.8[dB]
Irr_atn_noe	= 19.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recv_spin	= 5
Relaxation_delay	= 2[s]
Repetition_time	= 3.04333312[s]
Temp_get	= 17.9[dC]



C:\WINNNMR98\DATA\tw810hf.als
TW-810:1H

DFILE C:\WINNNMR98\DATA\tw810hf.als
COMNT TW-810:1H
DATIM Fri May 11 10:39:26 2007
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 kHz
OBFIN 10277.0 Hz
POINT 32768
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 16

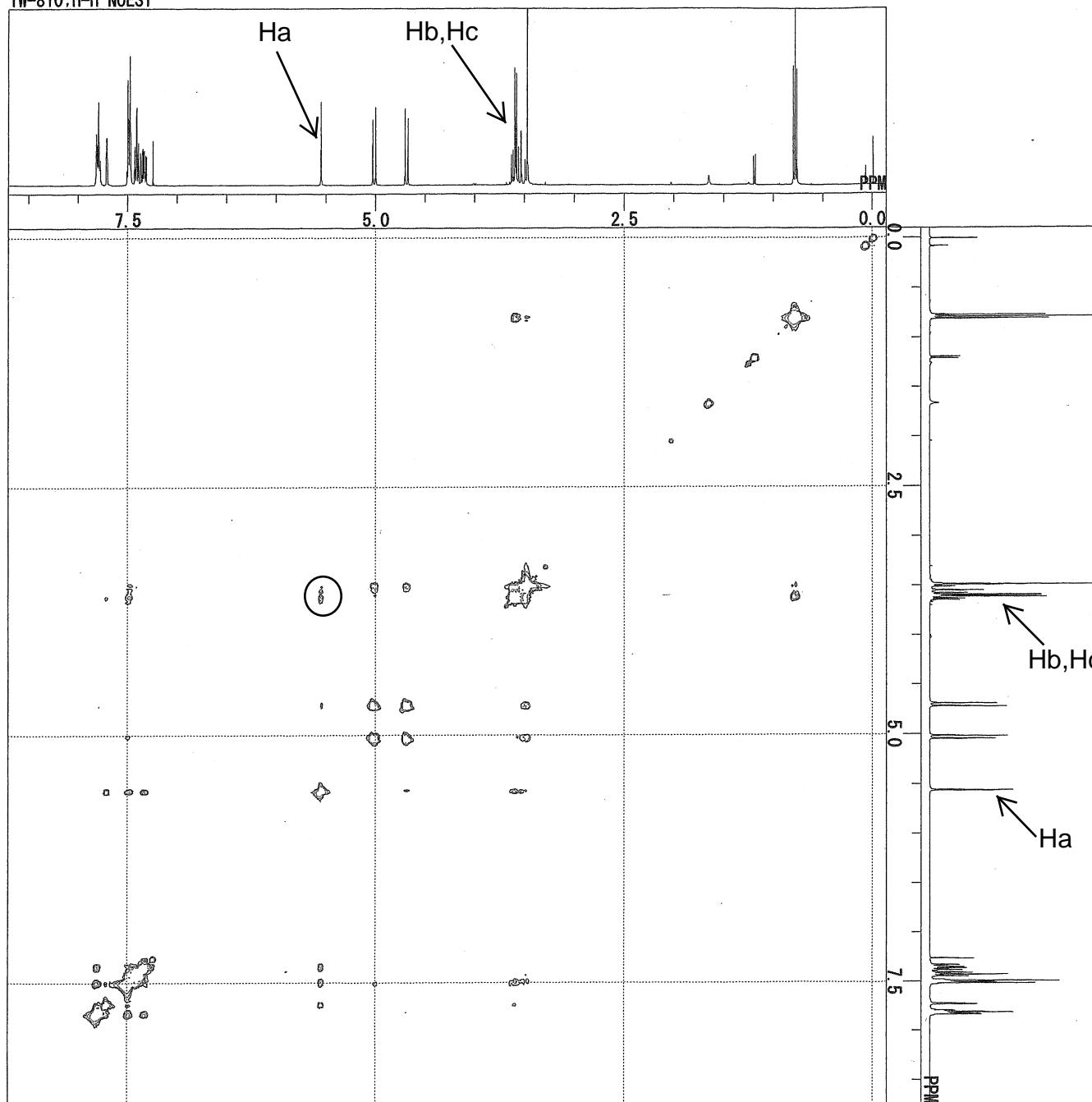




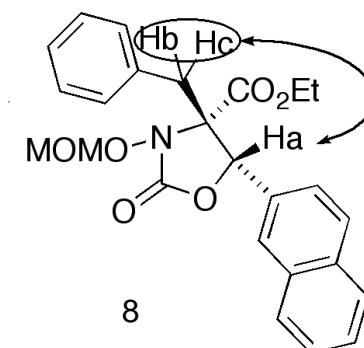
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sewp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill1 : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TW-810 13C-1.jdf
```

Filename	= TW-810 13C-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#49646
Solvent	= CHLOROFORM-D
Creation_time	= 15-MAY-2007 08:26:47
Revision_time	= 15-MAY-2007 13:47:18
Current_time	= 15-MAY-2007 13:47:49
Content	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz])
X_seq_duration	= 1.04333312[s]
X_domain	= 13C
X_free	= 100.52530333[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95846665[Hz]
X_sweep	= 31.40703518[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 152
Total_scans	= 152
X_90_width	= 10.2[us]
X_acq_time	= 1.04333312[s]
X_angle	= 30[deg]
X_atn	= 3.8[db]
X_pulse	= 3.4[us]
Irr_atn_dec	= 19.8[dB]
Irr_atn_noe	= 19.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 58
Relaxation_delay	= 2[s]
Repetition_time	= 3.04333312[s]
Temp_get	= 18[dc]

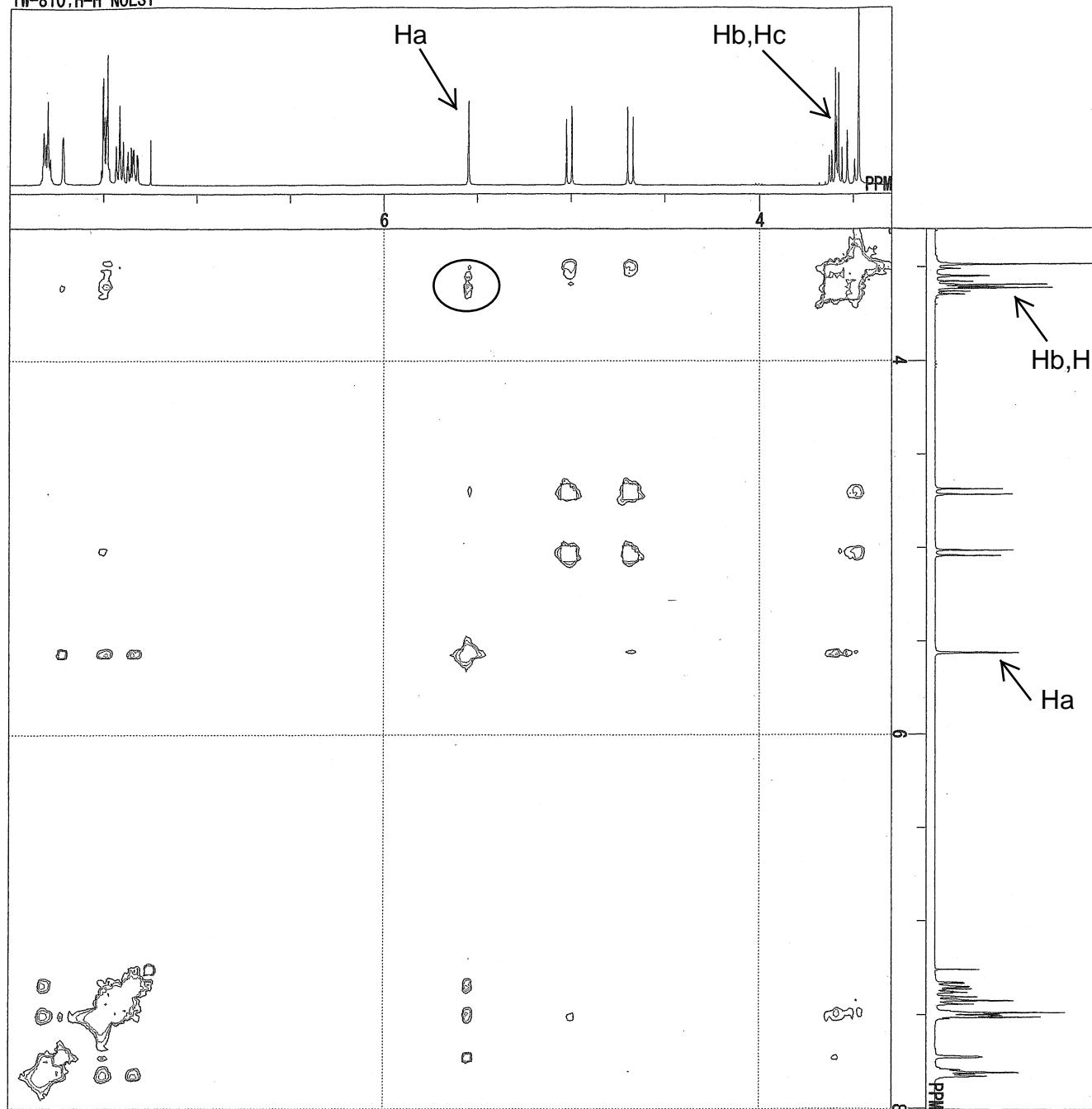
C:\WINNMR98\DATA\tw810noesy.ALS
TW-810:H-H NOESY



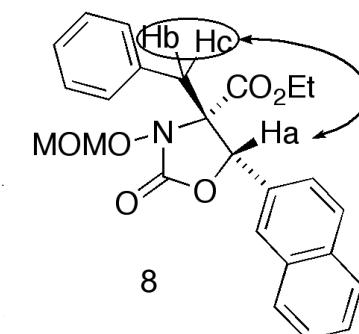
DFILE C:\WINNMR98\DATA\tw810noesy.ALS
COMNT TW-810:H-H NOESY
DATIM Fri May 11 13:20:41 2007
EXMOD VNOENH
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134.20 kHz
OBFIN 27.1 Hz
POINT 512
FREQU 3500.1 Hz
CLPNT 512
TODAT 256
CLFRQ 3496.5 Hz
SCANS 8
ACQTM 0.146 sec
PD 1.500 sec
PW1 12.6 us
PW2 12.6 us
PW3 10.0 us
P11 0.286 ms
P12 0.286 ms
P13 1500.00 ms
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCl₃
EXREF 0.00 ppm
CLEXR 0.00 ppm
RGAIN 15
OBATN 511
LOOP1 1

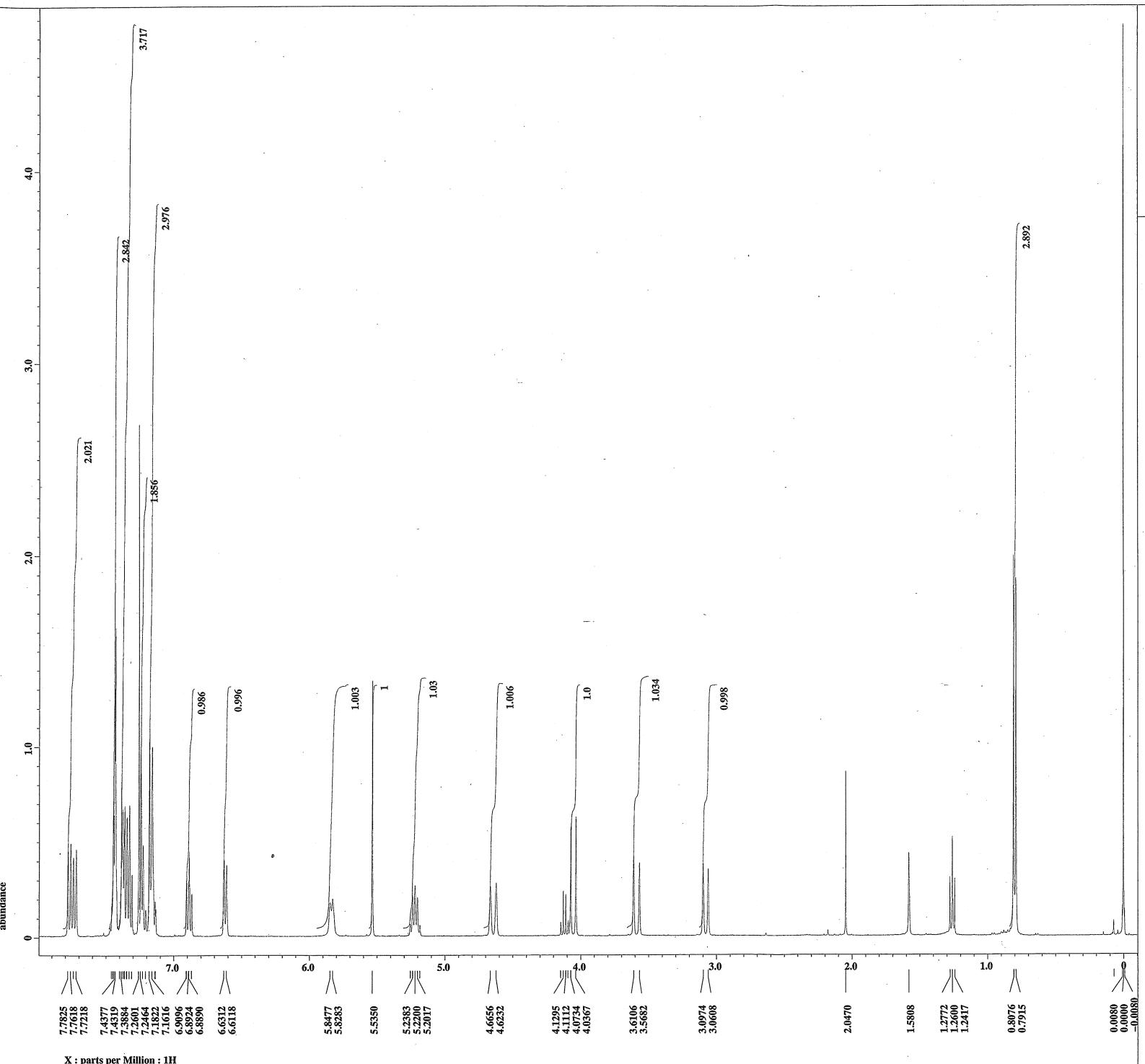


C:\WINNMR98\DATA\tw810noesy.ALS
TW-810;H-H NOESY



DFILE C:\WINNMR98\DATA\tw810noesy.ALS
COMNT TW-810;H-H NOESY
DATIM Fri May 11 13:20:41 2007
EXMOD VNOENH
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134.20 KHz
OBFIN 27.1 Hz
POINT 512
FREQU 3500.1 Hz
CLPNT 512
TODAT 256
CLFRQ 3496.5 Hz
SCANS 8
ACQTM 0.146 sec
PD 1.500 sec
PW1 12.6 us
PW2 12.6 us
PW3 10.0 us
P11 0.286 ms
P12 0.286 ms
P13 1500.00 ms
IRNUC 1H 21.7 c
CTEMP CDCL₃
SLVNT 0.00 ppm
EXREF 0.00 ppm
CLEXR 15
RGAIN 511
OBATN 1
LOOP1 1





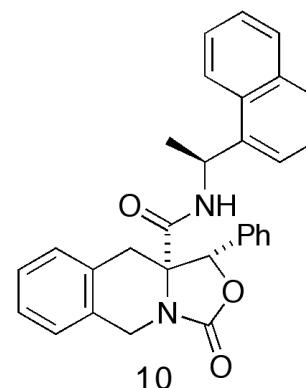
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
secp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TW-1086 for data3-1.jdf
```

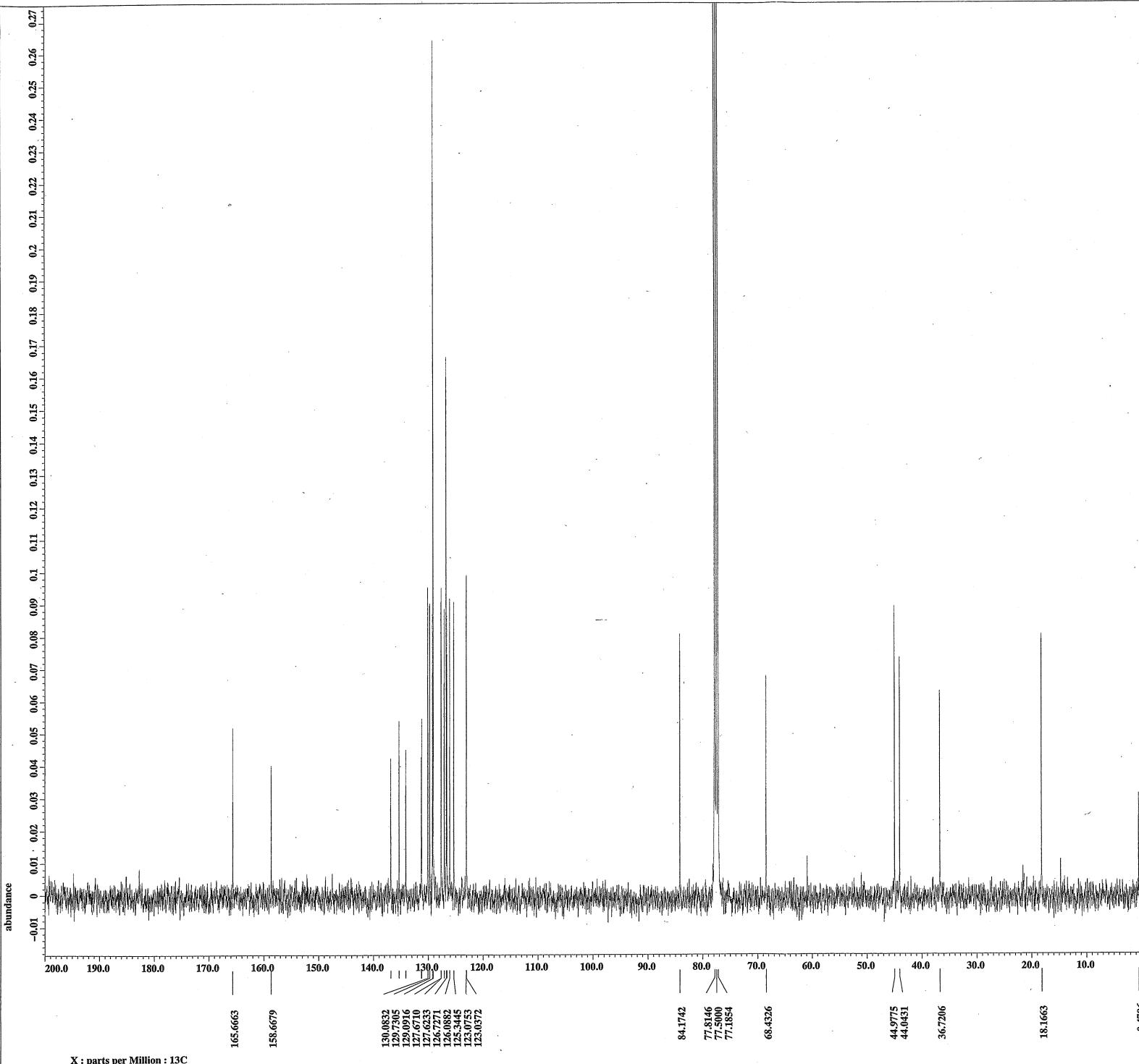
```
Filename      = TW-1086 for data3-4.j
Author        = delta
Experiment   = single_pulse.ex2
Sample_id    = S1086
Solvent       = CHLOROFORM-D
Creation_time = 29-JAN-2009 17:57:44
Revision_time = 30-JAN-2009 00:40:14
Current_time  = 30-JAN-2009 00:44:06

Content       = single_pulse
Data_format   = 1D COMPLEX
Dim_size      = 13107
Dim_title     = 1H
Dim_units     = [ppm]
Dimensions    = X
Site          = ECX 400P
Spectrometer  = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 2.18365952[s]
X_deltan     = 1
X_fraq       = 399.78219838[MHz]
X_offset      = 5[ppm]
X_points      = 16384
X_prescans   = 1
X_resolution  = 0.45794685[Hz]
X_sweep       = 7.5030012[kHz]
Irr_domain   = 1H
Irr_freq      = 399.78219838[MHz]
Irr_offset    = 5[ppm]
Tri_domain   = 1H
Tri_freq      = 399.78219838[MHz]
Tri_offset    = 5[ppm]
Clipped      = FALSE
Mod_return   = 1
Scans         = 16
Total_scans   = 16

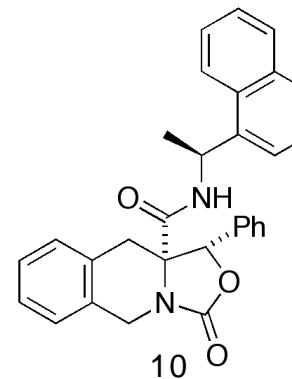
X_90_width   = 10.6[us]
X_acq_time   = 2.18365952[s]
X_angle       = 45[deg]
X_atn        = 0.3[dB]
X_pulse       = 5.3[us]
Irr_mode      = Off
Tri_mode      = Off
Dante_presat = FALSE
Initial_wait  = 1[s]
Recvr_gain   = 40
Relaxation_delay = 5[s]
Repetition_time = 7.18365952[s]
Temp_get      = 18.9[dc]
```

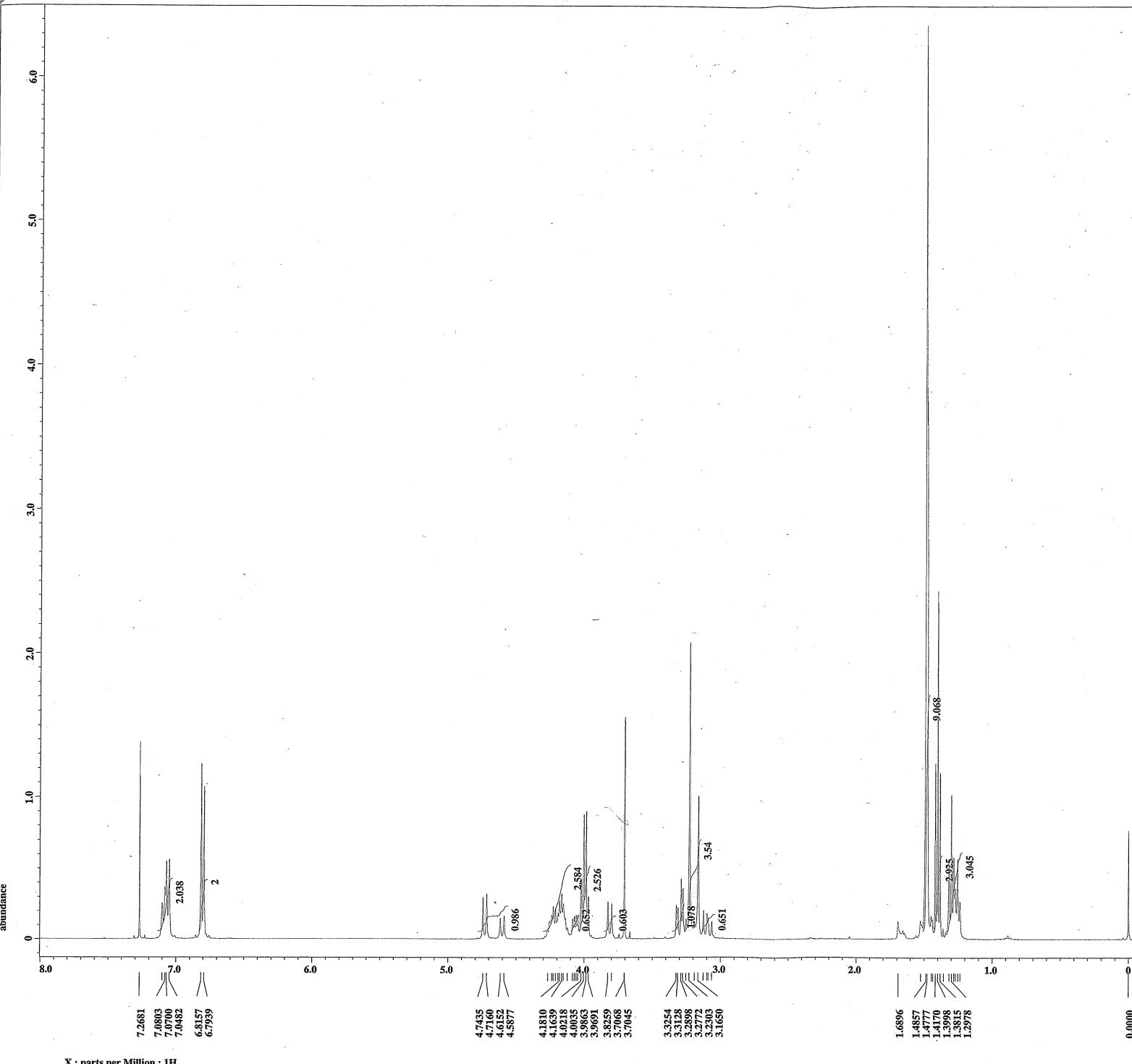




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sepx : 0.0Hz : 0.0[s]
txr_thresholds : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TW-1086 13C-1.jdf
```

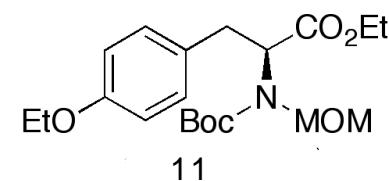
```
Filename = TW-1086 13C-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#3957A
Solvent = CHLOROFORM-D
Creation_time = 29-JAN-2009 19:51:01
Revision_time = 30-JAN-2009 02:21:41
Current_time = 30-JAN-2009 02:22:24
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 3.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 1434
Total_scans = 1434
X_90_width = 10.2[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_attn = 3.8[dB]
X_pulse = 3.4[us]
Irr_attn_dec = 19.8[dB]
Irr_attn_noe = 19.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recv_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get = 19.8[dC]
```

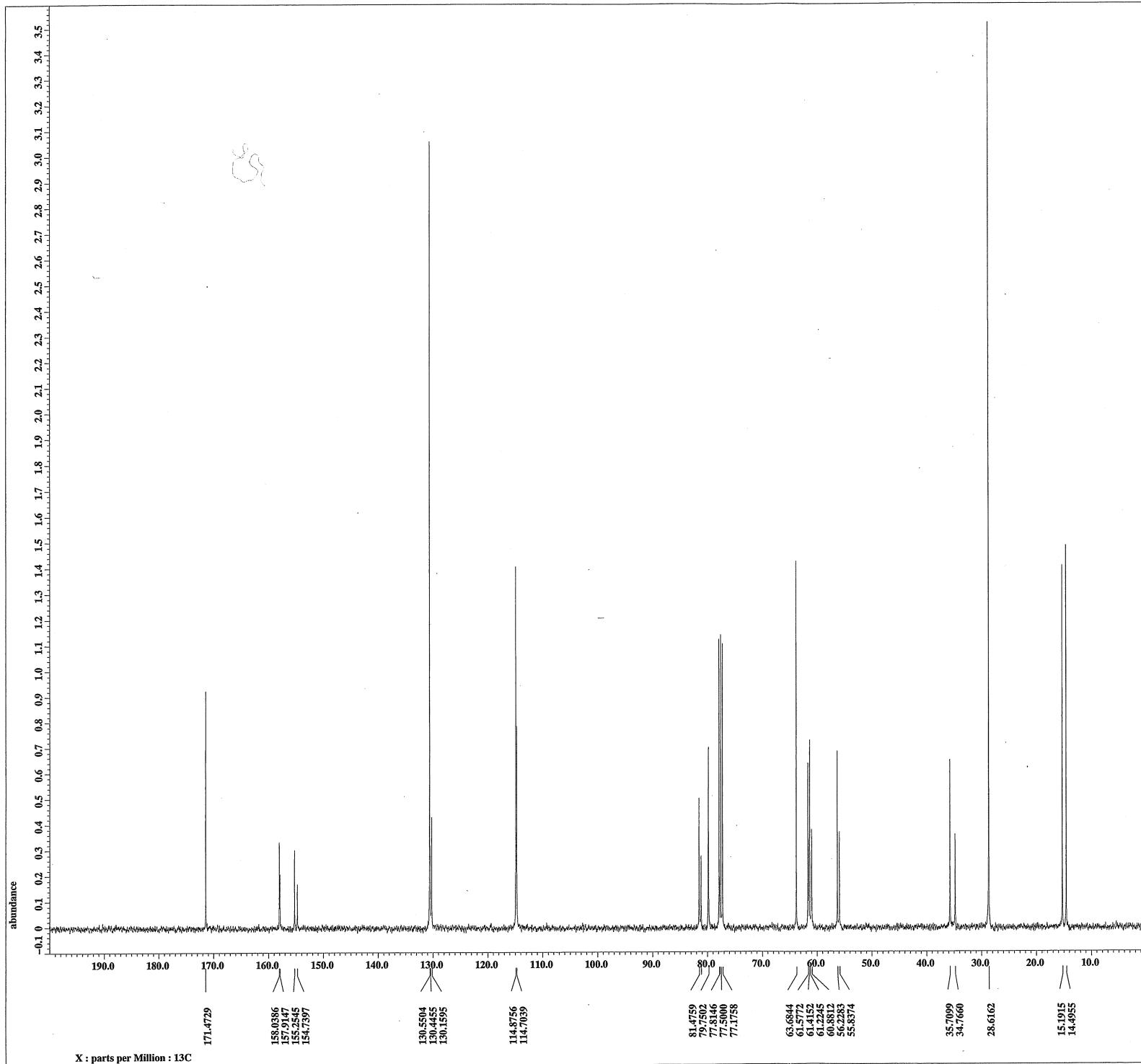




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TW-784-1.jdf
```

Filename	= TW-784-5.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= SH537714
Solvent	= CHLOROFORM-D
Creation_time	= 27-MAR-2007 09:40:34
Revision_time	= 27-JAN-2009 19:50:27
Current_time	= 27-JAN-2009 19:50:38
Content	= single_pulse
Data_format	= 1H_COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= ECX 400P
Site	= DELTA2_NMR
Spectrometer	= 9.389766[T] (400[MHz])
Field_strength	= 2.18365952[s]
X_acq_duration	= 1H
X_domain	= 399.78219838[MHz]
X_freq	= 5[ppm]
X_offset	= 16384
X_prescans	= 1
X_resolution	= 0.45794685[Hz]
X_0	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 10.6[us]
X_acq_time	= 2.18365952[s]
X_angle	= 45[deg]
X_atn	= 0.3[dB]
X_pulse	= 5.3[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 30
Relaxation_delay	= 5[s]
Repetition_time	= 7.18365952[s]
Temp_get	= 17.9[dc]





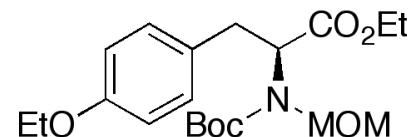
```
---- PROCESSING PARAMETERS ----
dc_balance = FALSE
sepx : 2.0[Hz] : 0.0[s]
trapezoid4d : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from:  $^{13}\text{C}$  No5-1.jdf
```

```
Filename      =  $^{13}\text{C}$  No5-3.jdf
Author        = delta
Experiment   = single_pulse_dec
Sample_id    = S4830207
Solvent       = CDCl3-CD3OD-CDCl3
Creation_time = 20-APR-2007 17:57:28
Revision_time = 20-APR-2007 23:17:45
Current_time  = 20-APR-2007 23:18:36

Content       = single pulse decouple
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     =  $^{13}\text{C}$ 
Dim_units     = [ppm]
Dimensions    = x
Site          = ECX 400P
Spectrometer  = DELTA2_NMR

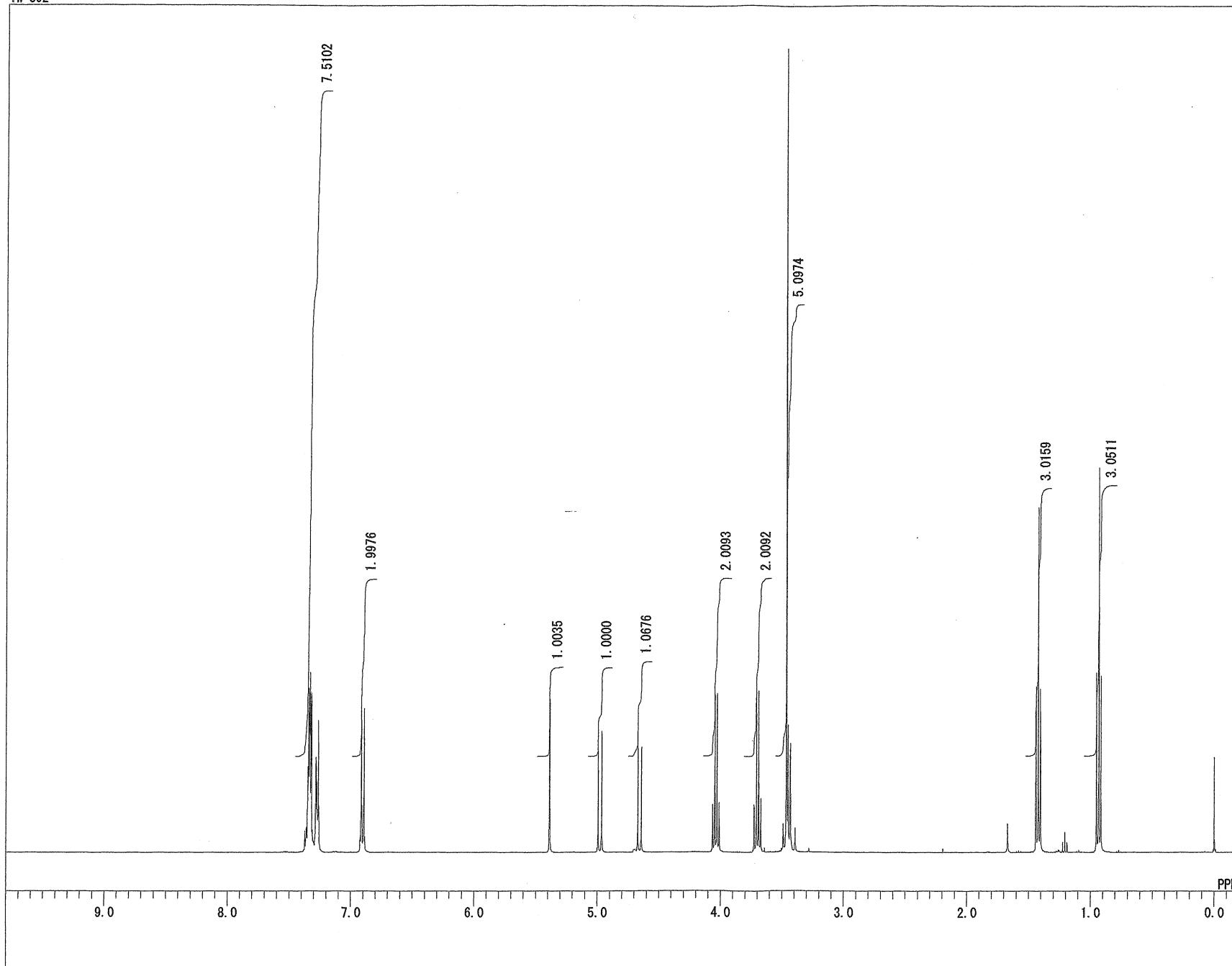
Field_strength = 9.389766[T] (400[MHz])
X_acc_duration = 1.04333312[s]
X_averages    = 13C
X_freq        = 100.52530333[MHz]
X_offset      = 100[ppm]
X_points      = 32768
X_prescans   = 4
X_resolution  = 0.95846665[Hz]
X_sweep       = 31.40703518[kHz]
Irr_domain   = 1H
Irr_freq      = 399.78219838[MHz]
Irr_offset    = 5[ppm]
Clipped       = FALSE
Mod_return   = 1
Scans         = 200
Total_scans   = 200

X_90_width   = 10.2[us]
X_acc_time   = 1.04333312[s]
X_angle       = 30[deg]
X_atn         = 3.8[dB]
X_pulse       = 3.4[us]
Irr_atn_noe  = 19.8[dB]
Irr_atn_dec  = 19.8[dB]
Irr_atn_noe  = 19.8[dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noe          = TRUE
Noe_time      = 2[s]
Recvr_gain   = 60
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get      = 18.3[dC]
```

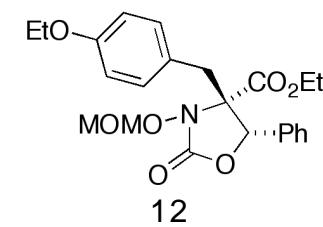


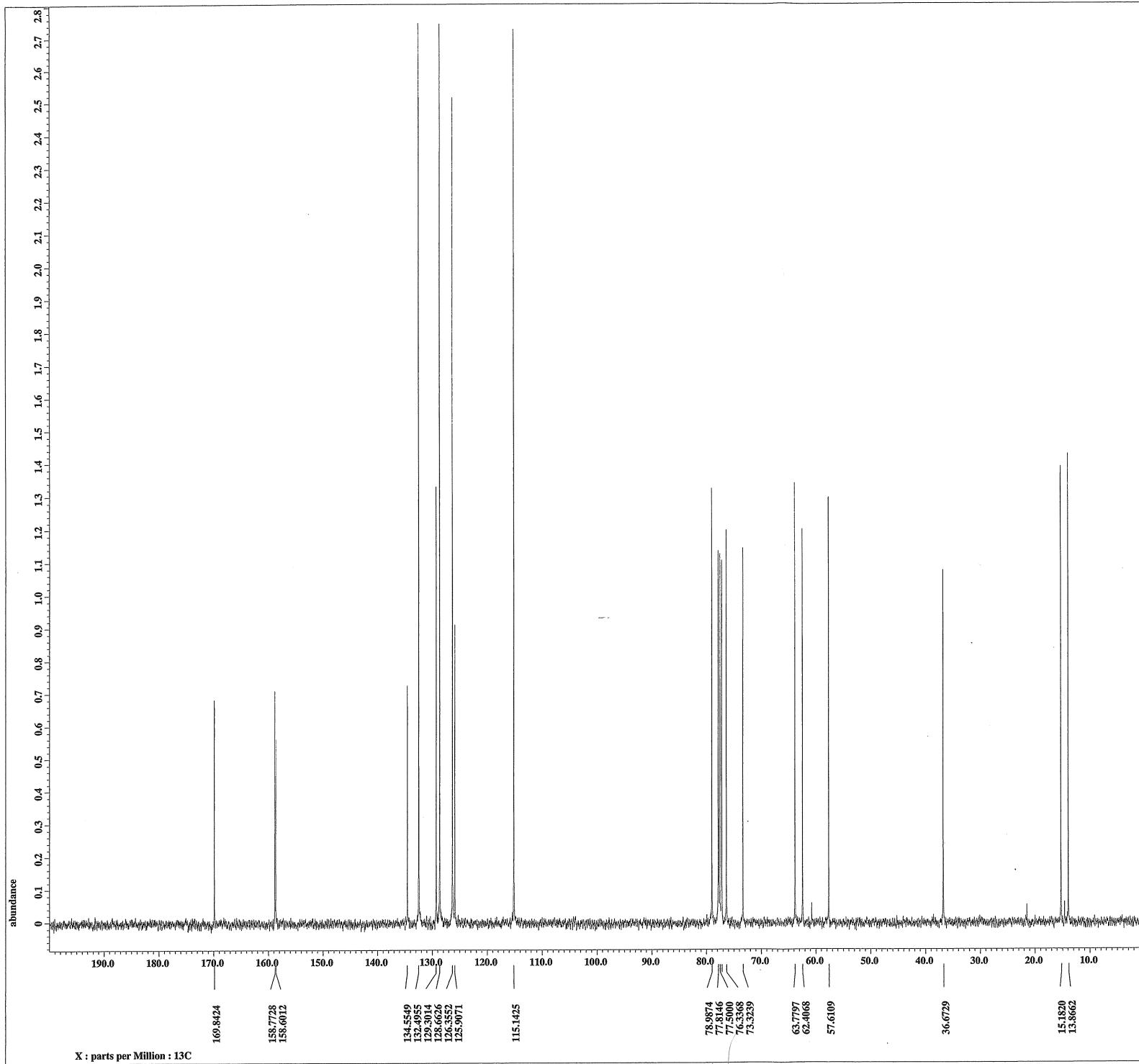
11

C:\WINNMR98\DATA\tw392.als
TW-392



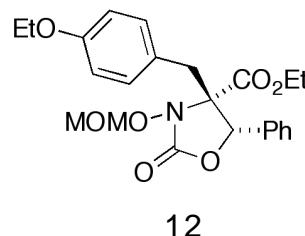
DFILE C:\WINNMR98\DATA\tw392.als
COMNT TW-392
DATIM Thu Feb 23 14:49:47 2006
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 kHz
OBFIN 10277.0 Hz
POINT 32768
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
1H 20.2 c
IRNUC CDCL₃
CTEMP 0.00 ppm
SLVNT 0.00 Hz
EXREF RGAIN 16



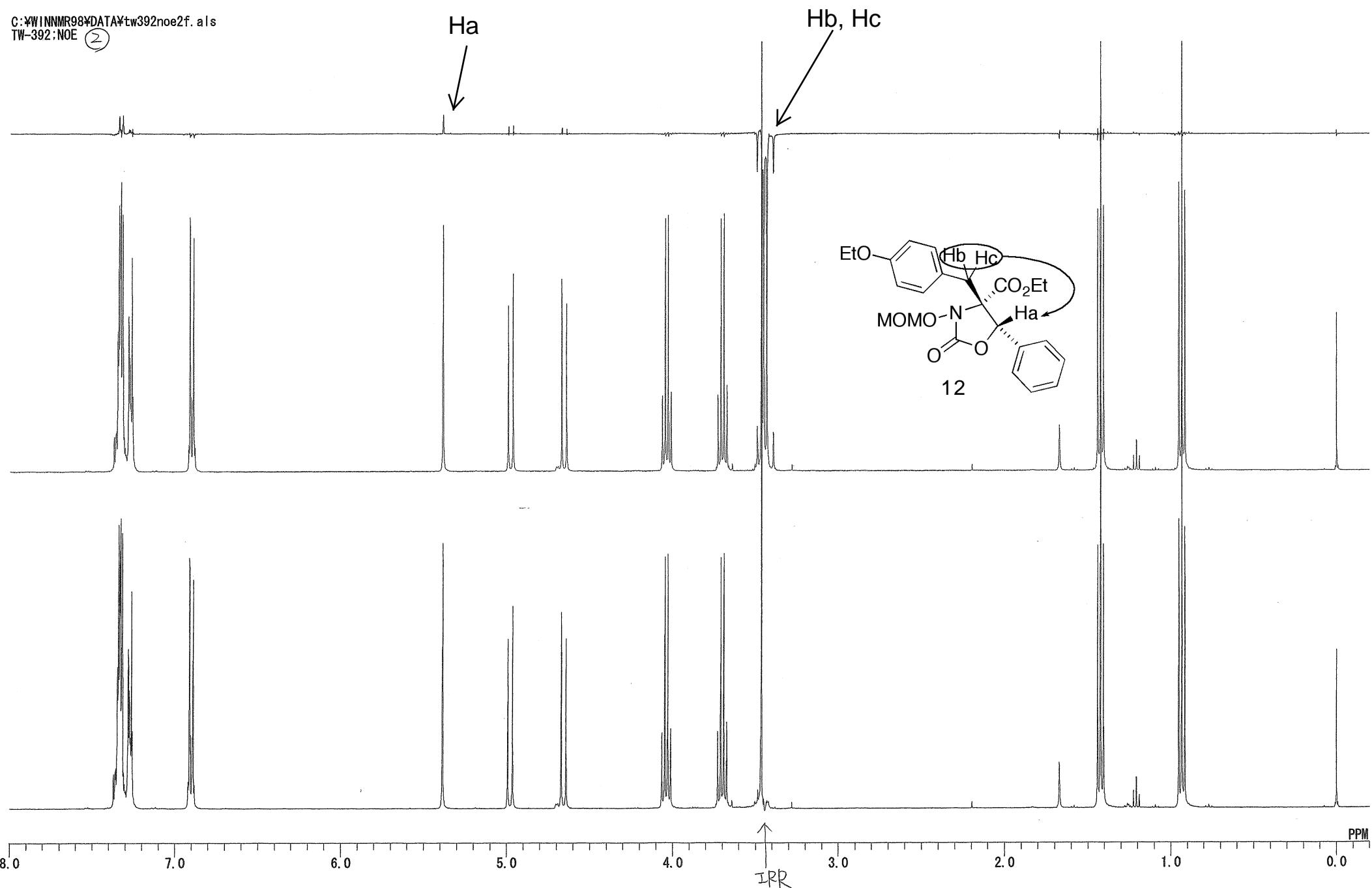


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TW-788 13C-1.jdf
```

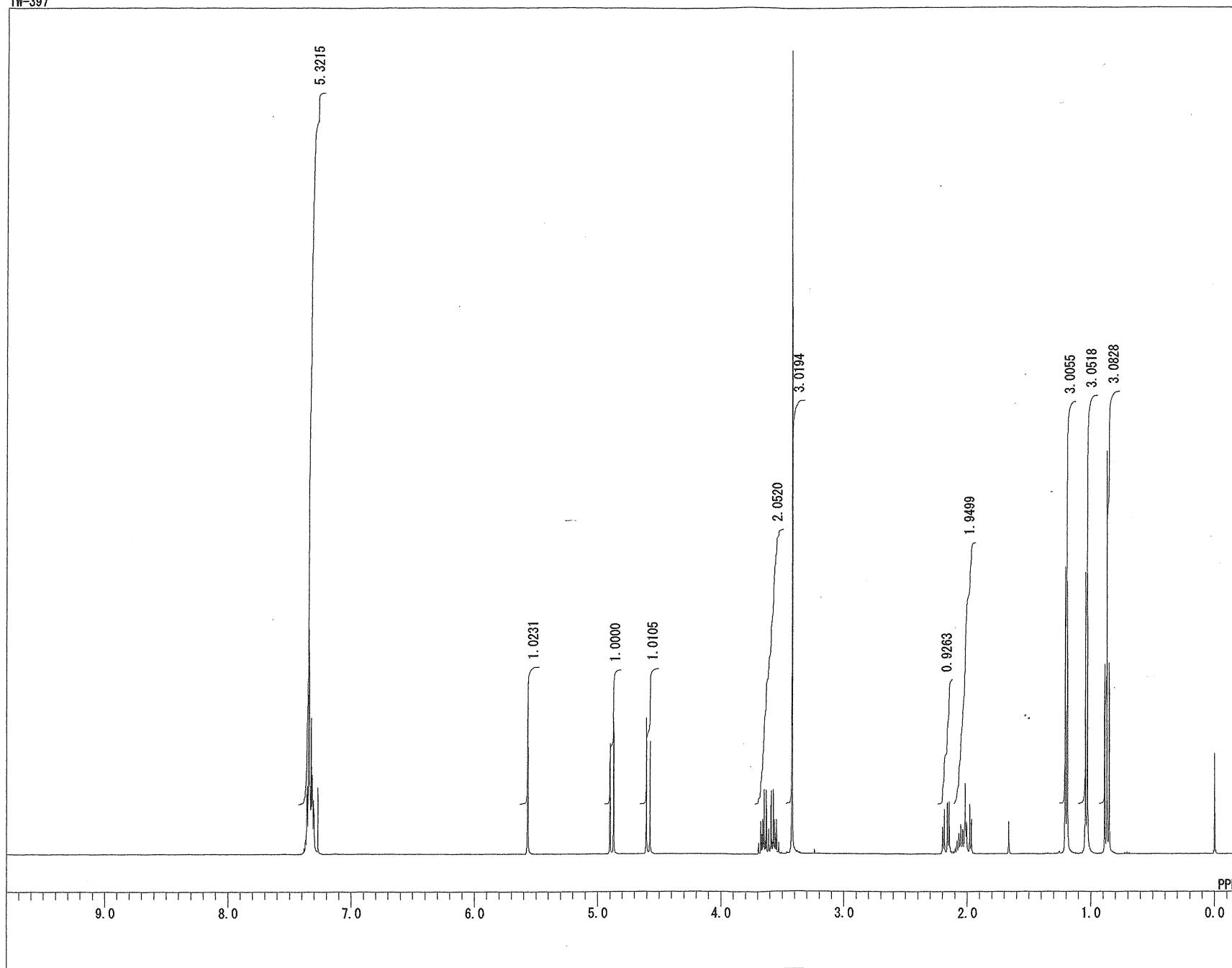
filename = TW-788 13C-3.jdf
author = delbr
Experiment = single_pulse_dec
sample_id = S#76531
Solvent = CHLOROFORM-D
Creation_time = 28-MAR-2007 16:25:02
Revision_time = 28-MAR-2007 21:44:03
Current_time = 28-MAR-2007 21:47:20
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = x
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[M] (400[MHz])
x_acq_duration = 1.04333312[s]
x_domain = 13C
x_freq = 100.52530333[MHz]
x_offset = 100[ppm]
x_points = 32768
x_prescans = 4
x_resolution = 0.95846665[Hz]
x_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 129
Total_scans = 129
x_90_width = 10.2[us]
x_acq_time = 1.04333312[s]
x_angle = 30[deg]
x_atn = 3.8[dB]
x_pulse = 3.4[us]
Irr_atn_dec = 19.8[dB]
Irr_atn_noe = 19.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recv_r_gain = 64
Relaxation_delay = 1[s]
Repetition_time = 3.04333312[s]
Temp_get = 17.9[dc]



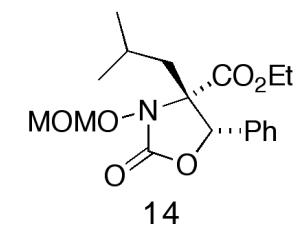
C:\WINNMR98\DATA\tw392noe2f.als
TW-392;NOE (2)

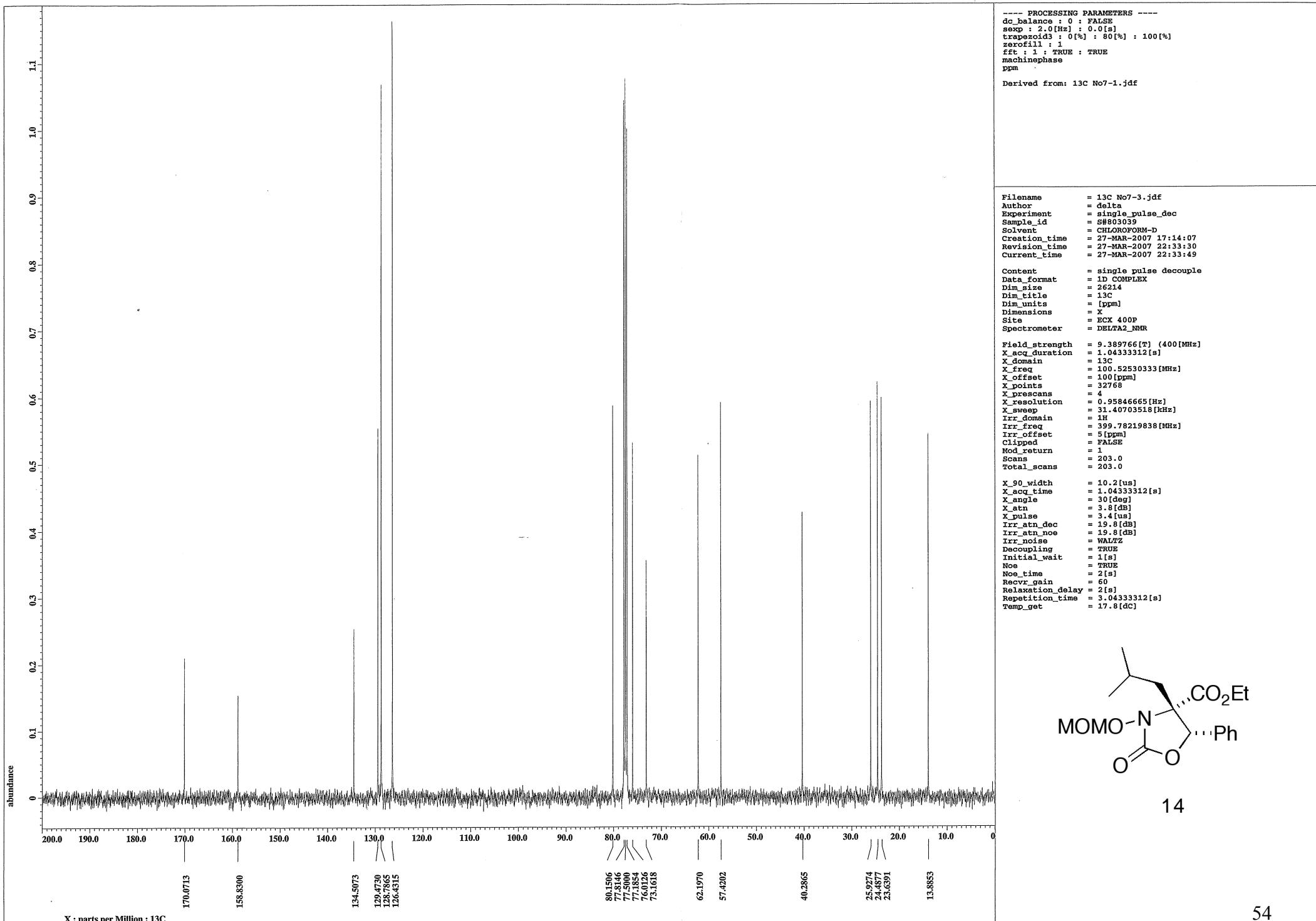


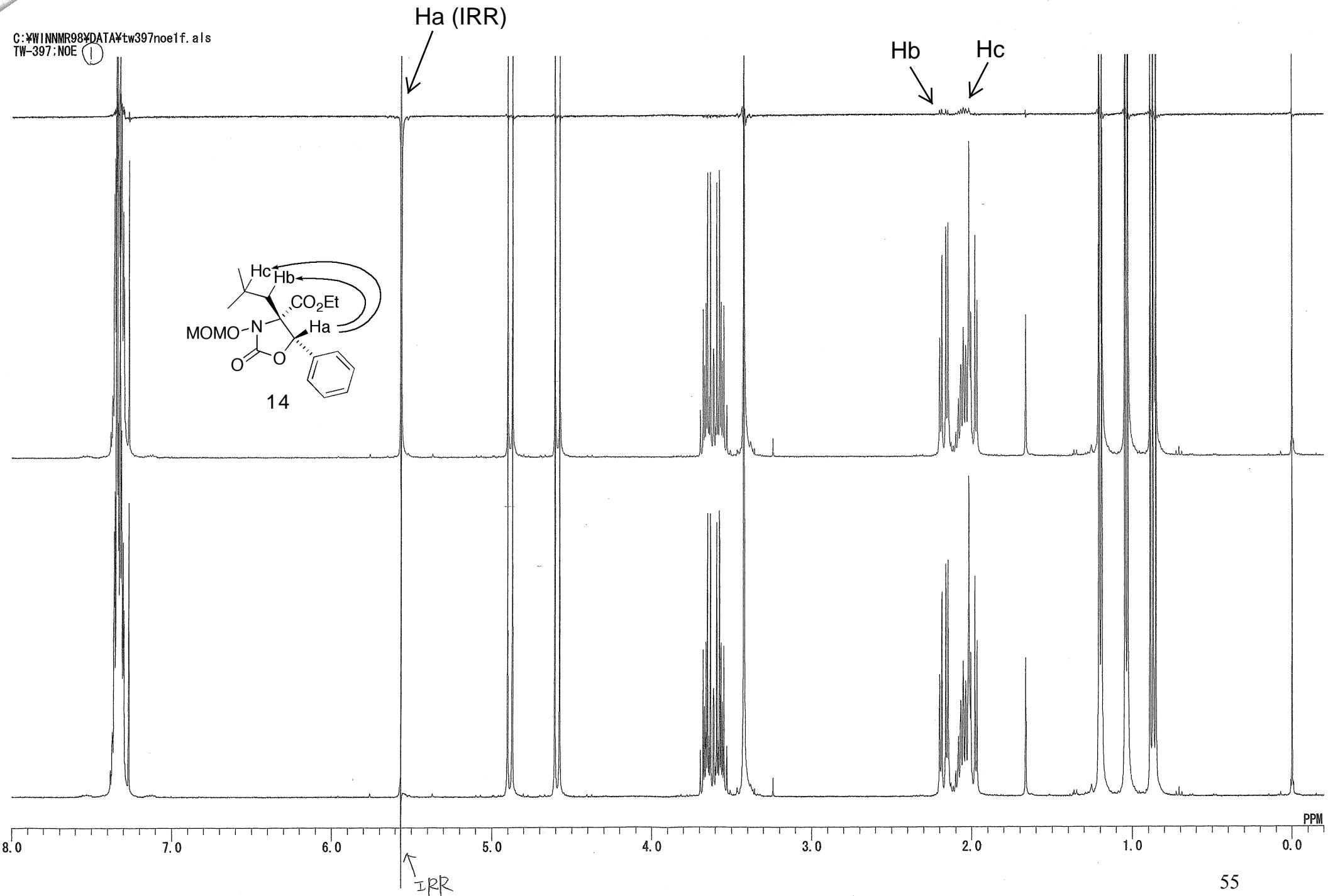
C:\WINNMR98\DATA\tw397.als
TW-397



DFILE C:\WINNMR98\DATA\tw397.als
COMNT TW-397
DATIM Thu Feb 23 16:42:05 2006
OBNUC 1H
EXMOD NON
OBFRQ 395.75 MHz
OBSET 124.00 kHz
OBFIN 10277.0 Hz
POINT 32788
FREQU 7936.5 Hz
SCANS 16
ACQTM 4.129 sec
PD 10.000 sec
PW1 5.0 us
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCl₃
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 16







C:\WINNMR98\DATA\tw397noe2f.als
TW-397:NOE (2)

Ha

Hb (IRR)

