

Asymmetric α -Arylation of Amino Acid Derivatives by Clayden Rearrangement of Ester Enolates via Memory of Chirality

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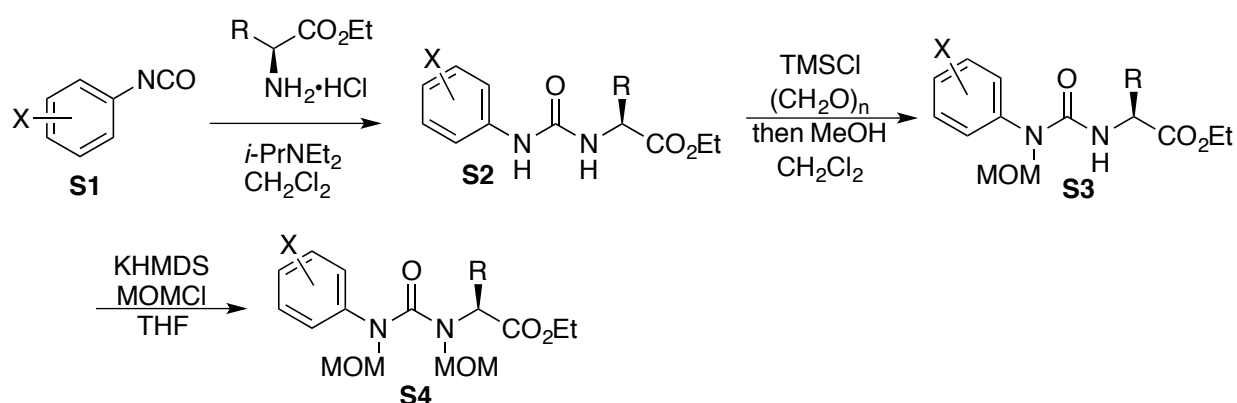
Supporting Information

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General. ^1H NMR were measured in CDCl_3 or $\text{DMSO-}d_6$ and referenced from TMS (0.00 ppm) or DMSO (2.50 ppm) using JEOL ECX-400 (400 MHz) spectrophotometer, unless otherwise noted. ^{13}C NMR were measured in CDCl_3 , acetone- d_6 , or $\text{DMSO-}d_6$ and referenced to CDCl_3 (77.0 ppm), $(\text{CD}_3)_2\text{CO}$ (29.84 ppm), or $(\text{CD}_3)_2\text{SO}$ (39.52 ppm) using JEOL ECX-400 (100 MHz) spectrophotometer, 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; quint, quintet; sept, septet; 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. Flash column chromatography was performed on Silica Gel (SiliaFlash[®] F60 (SILICYCLE) or silica gel 60N (spherical, neutral, KANTO)). Thin layer chromatography (TLC) was performed on precoated plates (0.25 mm, silica gel Merck Kieselgel 60F₂₄₅), and compounds were visualized with UV light followed by *p*-anisaldehyde stain, phosphomolybdic acid stain or, ninhydrin stain. Preparative thin layer chromatography (PTLC) was performed on precoated plates (0.5 mm, silica gel, Merck Kieselgel 60F₂₄₅) and visualized with UV light. Anhydrous THF was purchased from Kanto Kagaku and pre-treated with activated MS4Å for 1 day or longer. Anhydrous toluene purchased from Wako Chemical was distilled over CaH_2 and kept with MS4Å. Anhydrous DMF, which was purchased from Wako Chemical and pre-treated with MS4Å for 1 day, was distilled over P_2O_5 and kept with MS4Å. Hexamethyldisilazane (HMDS) and diisopropylamine (*i*-Pr₂NH) were distilled over CaH_2 and kept with MS4Å. *n*-BuLi (ca. 1.5 M in hexane) was purchased from Kanto Kagaku and titrated with diphenylacetic acid.ⁱ⁾ LDA was freshly prepared from *i*-Pr₂NH (1 eq.) with *n*-BuLi (1 eq.) in THF or toluene at 0 °C for 30 min. LiHMDS was freshly prepared from HMDS (1 eq.) with *n*-BuLi (1 eq.) in THF (when reaction was performed in THF or DMF) or toluene (when reaction was performed in toluene) at 0 °C for 30min. NaHMDS (ca. 2.0 M in THF) was purchased from TCI and titrated with standard solution of *sec*-butanol by using 2,2'-bipyridine as an indicator.ⁱⁱ⁾ KHMDS (ca 0.5 M) was prepared from HMDS (5.2 mL, 25 mmol) with KH (1.2 g) in THF (44.8 mL) or toluene (44.8 mL) under reflux for 1 hr and kept in vial containers and titrated with standard solution of *sec*-butanol by using 2,2'-bipyridine as an indicator.ⁱⁱ⁾

General procedure 1 for synthesis of precursor of aryl migration reaction



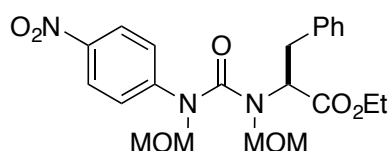
Aryl isocyanate **S1** (1.0 eq.) was added to a solution of amino acid ethyl ester hydrochloride (1.2 eq.) and *i*-Pr₂NEt (1.2 eq.) in CH_2Cl_2 (0.2 M) at rt. After being stirred for 30 min at same temperature, the mixture was concentrated. The residue was purified through silica gel column chromatography to give urea **S2**. Freshly distilled TMSCl (6.0 eq.) was added to a mixture of urea **S2** (1.0 eq.) and paraformaldehyde (6.0 eq.) in CH_2Cl_2 at 0 °C. After being stirred at rt for overnight at rt, the resulting mixture was cooled to 0 °C and MeOH (one and half times volume as large as mole number of urea **S2**) was added. The mixture was stirred for additional 30 min at 0 °C, then poured into sat. NaHCO_3 aq.

i) Kofron, W. G.; Backawski, L. M. *J. Org. Chem.* **1976**, *41*, 1879.

ii) Ireland, R. E.; Meissner, R. S. *J. Org. Chem.* **1991**, *56*, 4566.

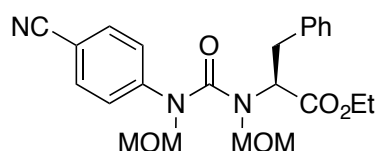
at 0 °C. The aqueous layer was extracted with AcOEt and the combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated. The residue was purified through flash silica gel column chromatography to give mono-methoxymethylated urea **S3**. To a solution of mono-methoxymethylated urea **S3** (1.0 eq.) in THF was added dropwise KHMDS (THF solution, 0.98 eq.) at -78 °C. After being stirred for 15 min at same temperature, MOMCl (3.0-6.0 eq.) was added. The resulting mixture was stirred at -78 °C for overnight and poured into sat. NH₄Cl aq. at rt. The aqueous layer was extracted with AcOEt and the extracts were washed with brine, dried over MgSO₄, filtered, and concentrated. The residue was purified through flash silica gel column chromatography to give **S4**.

Ethyl *N*-(*N*'-methoxymethyl-*N*'-*p*-nitrophenylcarbamoyl)-*L*-phenylalaninate (**2**)



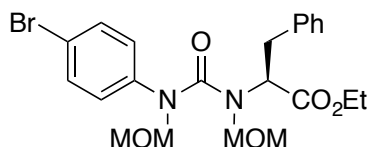
According to general procedure 1, **2** (420 mg, 29% (overall yield)) was obtained from *p*-nitrophenyl isocyanate and *L*-phenylalanine ethyl ester hydrochloride as a yellow oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/2-propanol = 9:1, flow = 1.0 mL/min, λ = 254 nm, t_R = 15 (*S*), 21 (*R*) min; $[\alpha]_D^{22}$ -86 (*c* 0.14, CHCl₃); IR (neat) cm⁻¹: 2935, 1738, 1675, 1514, 1438, 1338, 1281, 1193, 1085; ¹H NMR (400 MHz, CDCl₃): δ 1.28 (t, *J* = 7.3 Hz, 3H), 2.96 (s, 3H), 3.31 (dd, *J* = 9.9, 14.4 Hz, 1H), 3.36 (s, 3H), 3.48 (dd, *J* = 5.7, 14.4 Hz, 1H), 4.17 (d, *J* = 10.1 Hz, 1H), 4.22 (q, *J* = 7.3 Hz, 2H), 4.37 (d, *J* = 10.1 Hz, 1H), 4.67 (dd, *J* = 6.0, 10.1 Hz, 1H), 4.97 (d, *J* = 10.8 Hz, 1H), 5.02 (d, *J* = 10.8 Hz, 1H), 7.00 (dd, *J* = 2.3, 7.1 Hz, 2H), 7.24-7.36 (m, 5H), 8.01 (dd, *J* = 2.3, 7.1 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 14.09 (q), 35.27 (t), 55.85 (q), 56.03 (q), 60.51 (d), 61.54 (t), 80.34 (t), 82.06 (t), 121.15 (d), 124.84 (d), 126.91 (d), 128.64 (d), 129.26 (d), 137.63 (s), 143.48 (s), 158.81 (s), 170.60 (s); MS (FAB) *m/z*: 468 [(M+Na)⁺], 45 (base peak); HRMS (FAB) *m/z* calcd for C₂₂H₂₇N₃NaO₇ [(M+Na)⁺]: 468.1747 found 468.1749.

Ethyl *N*-(*N*'-*p*-cyanophenyl-*N*'-methoxymethylcarbamoyl)-*N*-methoxymethyl-*L*-phenylalaninate (**5**)



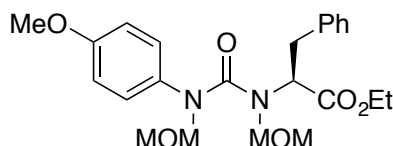
According to the general procedure 1, **5** (671 mg, 44% (overall yield)) was obtained from *p*-cyanophenyl isocyanate and *L*-phenylalanine ethyl ester hydrochloride as a colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/2-propanol = 9:1, flow = 1.0 mL/min, λ = 254 nm, t_R = 19 (*R*), 28 (*S*) min; $[\alpha]_D^{19}$ -77 (*c* 3.2, CHCl₃); IR (neat) cm⁻¹: 2986, 2936, 2225, 1739, 1672, 1604, 1509, 1423, 1356, 1279, 1195, 1085, 755; ¹H NMR (400 MHz, CDCl₃): δ 1.27 (t, *J* = 7.3 Hz, 3H), 2.93 (s, 3H), 3.29 (dd, *J* = 9.6, 14.7 Hz, 1H), 3.34 (s, 3H), 3.46 (dd, *J* = 6.0, 14.7 Hz, 1H), 4.15 (d, *J* = 10.3 Hz, 1H), 4.21 (q, *J* = 7.3 Hz, 2H), 4.34 (d, *J* = 10.3 Hz, 1H), 4.65 (dd, *J* = 6.0, 9.6 Hz, 1H), 4.96 (d, *J* = 10.6 Hz, 1H), 5.01 (d, *J* = 10.6 Hz, 1H), 6.98 (d, *J* = 8.7 Hz, 2H), 7.23-7.35 (m, 5H), 7.42 (d, *J* = 8.7 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 14.02 (q), 35.23 (t), 55.71 (q), 55.92 (q), 60.33 (d), 61.41 (t), 80.20 (t), 82.03 (t), 107.23 (s), 118.66 (s), 122.11 (d), 126.76 (d), 128.53 (d), 129.20 (d), 133.05 (d), 137.66 (s), 147.75 (s), 158.82 (s), 170.65 (s); MS (FAB) *m/z*: 448 [(M+Na)⁺], 45 (base peak); HRMS (FAB) *m/z* calcd for C₂₃H₂₇N₃NaO₅ [(M+Na)⁺]: 448.1848 found 448.1838; Anal. calcd for C₂₃H₂₇N₃O₅: C, 64.93; H, 6.40; N, 9.88 found C, 64.97; H, 6.47; N, 9.71.

Ethyl *N*-(*N*'-*p*-bromophenyl-*N*'-methoxymethylcarbamoyl)-*N*-methoxymethyl-*L*-phenylalaninate (**7**)



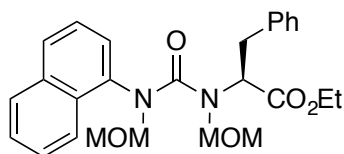
According to the general procedure 1, **7** (852 mg, 52% (overall yield)) was obtained from *p*-bromophenyl isocyanate and L-phenylalanine ethyl ester hydrochloride as a colorless oil; $[\alpha]_D^{22}$ -50 (*c* 1.0, CHCl₃); IR (neat) cm⁻¹: 2984, 2935, 2821, 1738, 1666, 1589, 1491, 1423, 1280, 1073; ¹H NMR (400 MHz, CDCl₃): δ 1.23 (t, *J* = 7.1 Hz, 3H), 2.95 (s, 3H), 3.22 (dd, *J* = 9.0, 14.4 Hz, 1H), 3.34 (s, 3H), 3.40 (dd, *J* = 6.4, 14.4 Hz, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 4.21 (d, *J* = 10.3 Hz, 1H), 4.37 (d, *J* = 10.3 Hz, 1H), 4.57 (dd, *J* = 6.4, 9.0 Hz, 1H), 4.96 (s, 2H), 6.66 (d, *J* = 9.2 Hz, 1H), 7.21-7.33 (m, 7H); ¹³C NMR (100 MHz, CDCl₃): δ 13.86 (q), 35.41 (q), 55.47 (q), 55.76 (q), 60.17 (d), 61.01 (t), 79.61 (t), 82.27 (t), 118.23 (s), 125.31 (d), 126.44 (d), 128.26 (d), 129.09 (d), 132.02 (d), 137.75 (s), 142.75 (s), 159.26 (s), 170.82 (s); MS (EI) *m/z*: 480 [(M+2)⁺, base peak], 478 (M⁺); HRMS (EI) *m/z* calcd for C₂₂H₂₇BrN₂O₅ (M⁺): 478.1103 found 478.1101; calcd for C₂₂H₂₇BrN₂O₅ [(M+2)⁺]: 480.1086 found 480.1086.

Ethyl *N*-methoxymethyl-*N*-(*N'*-methoxymethyl-*N'*-*p*-methoxyphenylcarbamoyl)-L-phenylalaninate (**9**)



According to the general procedure 1, **9** (292 mg, 23% (overall yield)) was obtained from *p*-methoxyphenyl isocyanate and L-phenylalanine ethyl ester hydrochloride as a colorless oil; HPLC conditions: Daicel Chiralpak AD-H, hexane/2-propanol = 9:1, flow = 1.0 mL/min, λ = 254 nm, *t_R* = 19.5 (*S*), 21.3 (*R*) min; $[\alpha]_D^{22}$ -42 (*c* 1.5, CHCl₃); IR (neat) cm⁻¹: 2933, 1737, 1658, 1510, 1246, 1080, 1030; ¹H NMR (400 MHz, CDCl₃): δ 1.22 (t, *J* = 7.1 Hz, 3H), 2.93 (s, 3H), 3.17 (dd, *J* = 8.2, 14.2 Hz, 1H), 3.36 (s, 3H), 3.37 (dd, *J* = 6.9, 14.2 Hz, 1H), 3.77 (s, 3H), 4.13 (q, *J* = 7.1 Hz, 2H), 4.24 (d, *J* = 10.3 Hz, 1H), 4.41 (d, *J* = 10.3 Hz, 1H), 4.53 (dd, *J* = 6.9, 8.2 Hz, 1H), 4.92 (d, *J* = 10.1 Hz, 1H), 4.98 (d, *J* = 10.1 Hz, 1H), 6.75 (d, *J* = 8.7 Hz, 2H), 6.96 (d, *J* = 8.7 Hz, 2H), 7.20-7.30 (m, 5H); ¹³C NMR (100 MHz, CDCl₃): δ 14.02 (q), 35.77 (t), 55.40 (q), 55.59 (q), 56.00 (q), 60.32 (d), 61.07 (t), 79.45 (t), 82.82 (t), 114.45 (d), 126.18 (d), 126.46 (d), 128.33 (d), 129.32 (d), 136.64 (s), 138.20 (s), 157.42 (s), 160.07 (s), 171.31 (s); MS (FAB) *m/z*: 430 (M⁺), 45 (base peak); HRMS (FAB) *m/z* calcd for C₂₃H₃₀N₂O₆ (M⁺): 430.2104 found 430.2104; Anal. calcd for C₂₃H₃₀N₂O₆: C, 64.24; H, 7.00; N, 6.45 found C, 64.21; H, 7.02; N, 6.51.

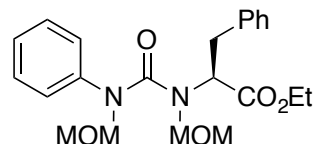
Ethyl *N*-methoxymethyl-*N*-[*N'*-methoxymethyl-*N'*-(naphthalen-1-yl)carbamoyl]-L-phenylalaninate (**11**)



According to the general procedure 1, **11** (463 mg, 56% (overall yield)) was obtained from 1-naphthyl isocyanate and L-phenylalanine ethyl ester hydrochloride as a colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/2-propanol = 9:1, flow = 1.0 mL/min, λ = 254 nm, *t_R* = 13 (*R*), 25 (*S*) min; $[\alpha]_D^{19}$ -46 (*c* 0.50, CHCl₃); IR (neat) cm⁻¹: 3060, 3028, 2983, 2932, 2820, 1739, 1660, 1454; ¹H NMR (400 MHz, CDCl₃): δ 1.18 (t, *J* = 7.1 Hz, 3H), 2.63 (s, 3H), 3.05 (dd, *J* = 8.3, 14.2 Hz, 1H), 3.31 (dd, *J* = 6.9, 14.2 Hz, 1H), 3.39 (s, 3H), 4.09 (q, *J* = 7.1 Hz, 2H), 4.15 (d, *J* = 10.6 Hz, 1H), 4.31 (d, *J* = 10.6 Hz, 1H), 4.50 (br t, *J* = 7.8 Hz, 1H), 5.01 (br s, 2H), 7.08-7.11 (m, 3H), 7.18-7.34 (m, 4H), 7.50-7.58 (m, 2H), 7.75 (d, *J* = 8.2 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 8.03 (d, *J* = 8.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 14.00 (q), 35.78 (t), 55.09 (q), 57.02 (q), 60.51 (d), 61.01 (t), 79.02 (t), 82.23 (t), 122.61 (d), 125.67 (d),

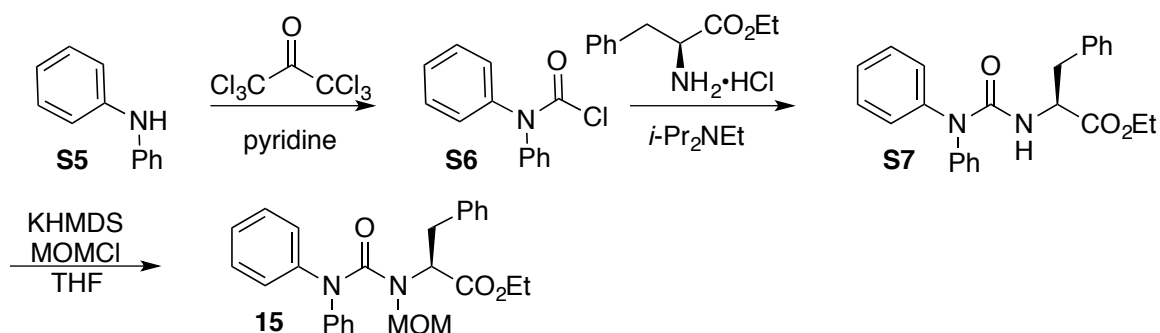
125.89 (d), 126.32 (d), 126.36 (d), 127.04 (d), 127.60 (d), 128.24 (d), 128.67 (d), 129.25 (d), 129.38 (s), 134.77 (s), 138.17 (s), 138.86 (s), 161.14 (s), 171.26 (s); MS (EI) m/z : 450 (M^+), 169 (base peak); HRMS (EI) m/z calcd for $C_{26}H_{30}N_2O_5$ (M^+): 450.2155 found 450.2154; Anal. calcd for $C_{26}H_{30}N_2O_5$: C, 69.31; H, 6.71; N, 6.22 found C, 69.03; H, 6.71; N, 6.17.

Ethyl *N*-methoxymethyl-*N*-(*N'*-methoxymethyl-*N'*-phenylcarbamoyl)-*L*-phenylalaninate (**13**)



According to the general procedure 1, **13** (1.44g, 57% (overall yield) was obtained from phenyl isocyanate and *L*-phenylalanine ethyl ester hydrochloride as a colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/2-propanol = 95:5, flow = 1.0 mL/min, λ = 254 nm, t_R = 12 (*R*), 17 (*S*) min; $[\alpha]_D^{22}$ -42 (c 1.2, $CHCl_3$); IR (neat) cm^{-1} : 2983, 2934, 1738, 1662, 1496, 1273, 1194, 1087; 1H NMR (400 MHz, $CDCl_3$): δ 1.22 (t, J = 7.1 Hz, 3H), 2.92 (s, 3H), 3.37 (s, 3H), 4.14 (q, J = 7.1 Hz, 2H), 4.23 (d, J = 10.1 Hz, 1H), 4.40 (d, J = 10.1 Hz, 1H), 4.98 (d, J = 10.3 Hz, 1H), 5.04 (d, J = 10.3 Hz, 1H), 7.03 (d, J = 7.3 Hz, 2H), 7.12 (t, J = 7.3 Hz, 1H), 7.19-7.31 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.03 (q), 35.69 (t), 55.67 (q), 56.00 (q), 60.27 (d), 61.12 (t), 79.57 (t), 82.71 (t), 124.18 (d), 125.45 (d), 126.51 (d), 128.37 (d), 129.33 (d), 138.13 (s), 143.92 (s), 159.88 (s), 171.18 (s); MS (EI) m/z : 400 (M^+ , base peak); HRMS (EI) m/z calcd for $C_{22}H_{28}N_2O_5$ (M^+): 400.1998 found 400.1999; Anal. calcd for $C_{22}H_{28}N_2O_5$: C, 65.98; H, 7.05; N, 7.00 found C, 65.68; H, 7.13; N, 6.86.

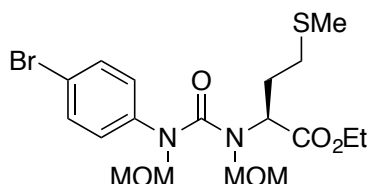
Synthesis of *N*-methoxymethyl-*N*-(*N'*,*N'*-diphenylcarbamoyl)-*L*-phenylalaninate (**15**)



To a solution of triphosgene (1.0 eq.) in CH_2Cl_2 (0.1 M) were added pyridine (6.0 eq.) and aniline **S5** (3.0 eq.) at 0 °C, successively. After being stirred for overnight at rt, the reaction was quenched by addition of sat. NH_4Cl aq. and the organic layer was washed with 1 M HCl aq., and dried over $MgSO_4$, filtered, and concentrated to give **S6**, which was dissolved in CH_2Cl_2 (0.2 M). Phenylalanine ethyl ester hydrochloride (1.2 eq.) and *i*- Pr_2NEt (2.4 eq.) were added to the mixture at 0 °C, successively. The resulting mixture was stirred for overnight at rt and diluted with water. The aqueous layer was extracted with AcOEt. The extracts were washed with brine, dried over $MgSO_4$, filtered, and concentrated. The residue was chromatographed on silica gel to give urea **S7**. To a solution of **S7** (1.0 eq.) was added dropwise KHMDS (THF solution, 0.98 eq.) at -78 °C. After being stirred for 15 min at -78 °C, MOMCl (6.0 eq.) was added dropwise. The resulting mixture was stirred for overnight at same temperature and poured into sat. NH_4Cl aq. The aqueous layer was extracted with AcOEt and the extracts were washed with brine, and dried over $MgSO_4$, filtered, and concentrated. The residual oil was purified through silica gel column chromatography to give **15** (1.33 g, 42% (overall yield)) as a colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/2-propanol = 99:1, flow = 1.0 mL/min, λ = 254 nm, t_R = 65 (*S*), 75 (*R*) min; $[\alpha]_D^{22}$ -32 (c 0.45, $CHCl_3$); IR (neat) cm^{-1} : 3060, 2983, 1737, 1669, 1590, 1493, 1291, 1087; 1H NMR (400 MHz, $CDCl_3$): δ 1.25 (t, J = 7.1 Hz, 3H), 3.10 (s, 3H), 3.19 (dd, J = 8.2, 14.2 Hz, 1H), 3.42 (dd, J

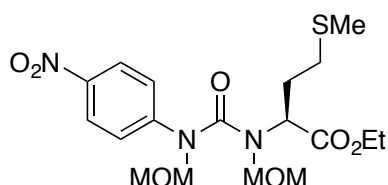
= 6.9, 14.2 Hz, 1H), 4.15 (q, $J = 7.1$ Hz, 2H), 4.42 (d, $J = 11.0$ Hz, 1H), 4.59 (dd, $J = 11.0$ Hz, 1H), 4.67 (dd, $J = 6.9, 8.2$ Hz, 1H), 6.94-6.97 (m, 4H), 7.12 (t, $J = 7.3$ Hz, 2H), 7.19-7.33 (m, 9H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.02 (q), 35.60 (t), 56.03 (q), 60.26 (d), 61.14 (t), 79.30 (t), 125.02 (d), 125.32 (d), 126.46 (d), 128.34 (d), 129.15 (d), 129.27 (d), 137.97 (s), 144.59 (s), 160.07 (s), 170.86 (s); MS (FAB) m/z : 432 (M^+), 196 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{26}\text{H}_{28}\text{N}_2\text{O}_4$ (M^+): 432.2049 found 432.2050.

Ethyl *N*-(*N'*-*p*-bromophenyl-*N'*-methoxymethylcarbamoyl)-*N*-methoxymethyl-L-methioninate (**17**)



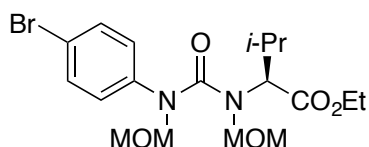
According to the general procedure 1, **17** (209 mg, 17% (overall yield)) was obtained from *p*-bromophenyl isocyanate and L-methionine ethyl ester hydrochloride as a colorless oil; HPLC conditions: Daicel Chiralpak, hexane/2-propanol = 9:1, flow = 1.0 mL/min, $\lambda = 254$ nm, $t_R = 12$ (*R*), 14 (*S*) min; $[\alpha]_D^{22} -17$ (c 0.44, CHCl_3); IR (neat) cm^{-1} : 2926, 1738, 1665, 1490, 1421, 1362, 1261, 1191, 1076; ^1H NMR (400 MHz, CDCl_3): δ 1.26 (t, $J = 6.7$ Hz, 3H), 1.99-2.09 (m, 4H), 2.26-2.36 (m, 1H), 2.51-2.64 (m, 2H), 3.04 (s, 3H), 3.38 (s, 3H), 4.08-4.19 (m, 2H), 4.39-4.48 (m, 3H), 4.97-5.03 (m, 2H), 7.16 (d, $J = 8.7$ Hz, 2H), 7.45 (d, $J = 8.7$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.12 (q), 15.10 (q), 29.74 (t), 31.07 (t), 55.41 (q), 56.12 (q), 58.71 (s), 61.28 (t), 79.77 (t), 82.56 (t), 119.04 (s), 126.21 (d), 132.36 (d), 142.83 (s), 160.04 (s), 171.39 (s); MS (FAB) m/z : 487 [$(\text{M}+2+\text{Na})^+$], 485 [$(\text{M}+\text{Na})^+$], 154 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{18}\text{H}_{27}\text{BrN}_2\text{NaO}_5\text{S}$ [$(\text{M}+\text{Na})^+$]: 485.0722 found 485.0725; calcd for $\text{C}_{18}\text{H}_{27}\text{BrN}_2\text{NaO}_5\text{S}$ [$(\text{M}+2+\text{Na})^+$]: 487.0703 found 487.0709.

Ethyl *N*-methoxymethyl-*N'*-(*N'*-methoxymethyl-*N'*-*p*-nitrophenylcarbamoyl)-L-methioninate (**19**)



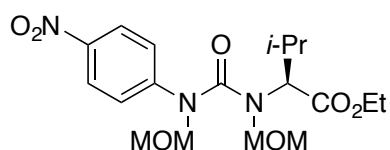
According to the procedure 1, **19** (70 mg, 8% (overall yield)) was obtained from *p*-nitrophenyl isocyanate and L-methionine ethyl ester hydrochloride as a yellow oil; HPLC conditions: Daicel Chiralpak AD-H, hexane/2-propanol = 9:1, flow = 1.0 mL/min, $\lambda = 254$ nm, $t_R = 27$ (*R*), 31 (*S*) min; $[\alpha]_D^{22} -57$ (c 0.66, CHCl_3); IR (neat) cm^{-1} : 2934, 1738, 1673, 1597, 1515, 1426, 1338, 1280, 1193, 1075; ^1H NMR (400 MHz, CDCl_3): δ 1.29 (t, $J = 7.3$ Hz, 3H), 2.05-2.15 (m, 4H), 2.37-2.46 (m, 1H), 2.59-2.71 (m, 2H), 3.05 (s, 3H), 3.41 (s, 3H), 4.17-4.23 (m, 2H), 4.44-4.52 (m, 3H), 5.05 (d, $J = 10.6$ Hz, 1H), 5.11 (d, $J = 10.5$ Hz, 1H), 7.40 (d, $J = 9.2$ Hz, 2H), 8.20 (d, $J = 9.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.12 (q), 15.06 (q), 29.40 (t), 31.09 (t), 55.47 (q), 56.10 (q), 58.87 (d), 61.52 (t), 80.24 (t), 82.04 (t), 121.84 (d), 124.87 (d), 143.87 (s), 149.60 (s), 159.45 (s), 170.97 (s); MS (FAB) m/z 452 [$(\text{M}+\text{Na})^+$], 73 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{18}\text{H}_{27}\text{N}_3\text{NaO}_7\text{S}$ [$(\text{M}+\text{Na})^+$]: 452.1467 found 452.1470.

Ethyl *N*-(*N'*-*p*-bromophenyl-*N'*-methoxymethylcarbamoyl)-*N*-methoxymethyl-L-valinate (**21**)



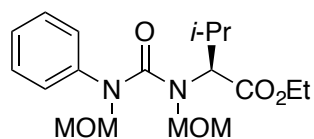
According to the general procedure 1, **21** (260 mg, 55% (overall yield)) was obtained from *p*-bromophenyl isocyanate and L-valine ethyl ester hydrochloride as a colorless oil; HPLC conditions: Daicel Chiralpak AD-H, hexane/2-propanol = 95:5, flow = 1.0 mL/min, λ = 254 nm, t_R = 11.6 (*R*), 12.9 (*S*) min; $[\alpha]_D^{20}$ +12 (*c* 1.1, CHCl₃); IR (neat) cm⁻¹: 2919, 1736, 1668, 1489, 1383, 1277, 1194, 1078; ¹H NMR (400 MHz, CDCl₃): δ 0.92 (d, *J* = 6.8 Hz, 3H), 0.97 (d, *J* = 6.8 Hz, 3H), 1.25 (t, *J* = 6.9 Hz, 3H), 2.16-2.27 (m, 1H), 3.11 (s, 3H), 3.39 (s, 3H), 4.00-4.19 (m, 3H), 4.42 (d, *J* = 10.1 Hz, 1H), 4.55 (d, *J* = 10.1 Hz, 1H), 4.98 (d, *J* = 10.5 Hz, 1H), 5.04 (d, *J* = 10.5 Hz, 1H), 7.17 (d, *J* = 8.7 Hz, 2H), 7.44 (d, *J* = 8.7 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 14.10 (q), 19.35 (q), 19.99 (q), 28.70 (d), 55.56 (q), 56.12 (q), 60.76 (t), 64.78 (d), 78.19 (d), 82.54 (d), 118.93 (s), 126.24 (d), 132.29 (d), 143.04 (s), 161.25 (s), 171.34 (s); MS (FAB) *m/z*: 432 [(M+2)⁺], 430 (M⁺), 154 (base peak); HRMS (FAB) *m/z* calcd for C₁₈H₂₇BrN₂O₅ (M⁺): 430.1103 found 430.1102; calcd for C₁₈H₂₇BrN₂O₅ [(M+2)⁺]: 432.1085 found 432.1087; Anal. calcd for C₁₈H₂₇BrN₂O₅: C, 50.12; H, 6.31; N, 6.49 found C, 49.94; H, 6.28; N, 6.46.

Ethyl *N*-methoxymethyl-*N*-(*N'*-methoxymethyl-*N'*-*p*-nitrophenylcarbamoyl)-L-valinate (**23**)



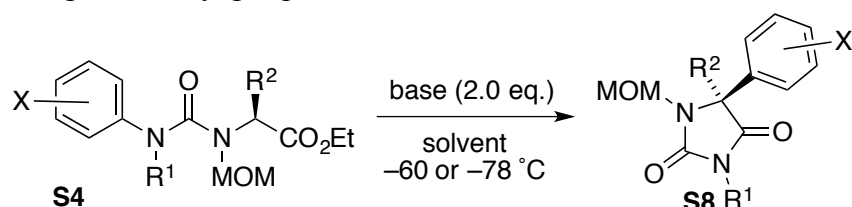
According to the general procedure 1, **23** (120 mg, 43% (overall yield)) was obtained from *p*-nitrophenyl isocyanate and L-valine ethyl ester hydrochloride as a colorless oil; HPLC conditions: Daicel Chiralpak AD-H, hexane/2-propanol = 93:7, flow = 1.0 mL/min, λ = 254 nm, t_R = 16.3 (*R*), 18.1 (*S*) min; $[\alpha]_D^{20}$ +13 (*c* 0.39, CHCl₃); IR (neat) cm⁻¹: 3424, 2921, 1737, 1677, 1596, 1515, 1339, 1279, 1194, 1078; ¹H NMR (400 MHz, CDCl₃): δ 0.98 (d, *J* = 6.9 Hz, 3H), 1.02 (d, *J* = 6.9 Hz, 3H), 1.22 (t, *J* = 6.9 Hz, 3H), 2.23-2.35 (m, 1H), 3.09 (s, 3H), 3.41 (s, 3H), 4.07-4.23 (m, 3H), 4.44 (d, *J* = 10.6 Hz, 1H), 4.60 (d, *J* = 10.6 Hz, 1H), 5.05 (d, *J* = 10.5 Hz, 1H), 5.13 (d, *J* = 10.5 Hz, 2H), 7.39 (d, *J* = 9.2 Hz, 2H), 8.12 (d, *J* = 9.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 14.12 (q), 19.49 (q), 19.91 (q), 28.50 (d), 55.58 (q), 56.16 (q), 60.99 (t), 64.84 (d), 78.52 (t), 82.07 (t), 121.99 (d), 124.85 (d), 143.87 (s), 129.81 (s), 160.68 (s), 170.99 (s); MS (FAB) *m/z*: 420 [(M+Na)⁺, base peak], HRMS (FAB) *m/z* calcd for C₁₈H₂₇N₃NaO₇ [(M+Na)⁺]: 420.1747 found 420.1746; Anal. calcd for C₁₈H₂₇N₃O₇: C, 54.40; H, 6.85; N, 10.57 found C, 54.23; H, 6.85; N, 10.48.

Ethyl *N*-methoxymethyl-*N*-(*N'*-methoxymethyl-*N'*-phenylcarbamoyl)-L-valinate (**25**)



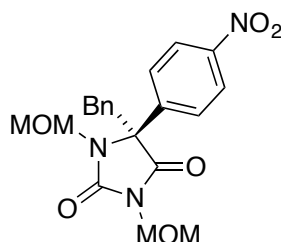
According to the general procedure 1, **25** (503 mg, 55% (overall yield)) was obtained from phenyl isocyanate and L-valine ethyl ester hydrochloride as a colorless oil; $[\alpha]_D^{18}$ +18 (*c* 0.40, CHCl₃); HPLC condition: Daicel Chiralcel OD-H; hexane/2-propanol = 95:5, flow = 0.8 mL/min, λ = 254 nm, t_R = 7.7 (*R*), 8.6 (*S*) min; IR (neat) cm⁻¹: 2970, 2936, 1737, 1667, 1273, 1195, 1079; ¹H NMR (400 MHz, CDCl₃): δ 0.92 (d, *J* = 6.4 Hz, 3H), 0.95 (d, *J* = 6.4 Hz, 3H), 1.25 (t, *J* = 7.3 Hz, 3H), 2.16-2.25 (m, 1H), 3.09 (s, 3H), 3.41 (s, 3H), 4.01-4.19 (m, 3H), 4.41 (d, *J* = 10.6 Hz, 1H), 4.49 (d, *J* = 10.6 Hz, 1H), 5.00 (d, *J* = 10.6 Hz, 1H), 5.09 (d, *J* = 10.6 Hz, 1H), 7.16-7.35 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 14.02 (q), 19.36 (q), 19.83 (q), 28.49 (d), 55.43 (q), 56.03 (q), 60.54 (t), 64.56 (d), 78.01 (t), 82.68 (t), 124.75 (d), 125.68 (d), 129.21 (d), 143.85 (s), 161.40 (s), 171.31 (s); MS (FAB) *m/z*: 375 [(M+Na)⁺], 352 (M⁺); HRMS (FAB) *m/z* calcd for C₁₈H₂₈N₂O₅ (M⁺): 352.1998 found 352.1998; Anal. calcd for C₁₈H₂₈N₂O₅: C, 61.34; H, 8.01; N, 7.95 found C, 61.30; H, 7.99; N, 8.08.

General Procedure 2 for migration of aryl group



To a solution of base (2.0 eq.) in THF or toluene was added dropwise **S4** (1.0 eq.) in anhydrous solvent (THF, DMF, or toluene; total 0.1 M) at $-60\text{ }^{\circ}\text{C}$ (DMF/THF), $-78\text{ }^{\circ}\text{C}$ (THF and toluene), or $0\text{ }^{\circ}\text{C}$ (toluene). After being stirred until complete consumption of **S4** (checked by TLC) at same temperature, the mixture was poured into sat. NH_4Cl aq., immediately and extracted with AcOEt. The extracts were washed with brine, dried over MgSO_4 , filtered, and concentrated. The residue was purified through silica gel column chromatography to give **S8**.

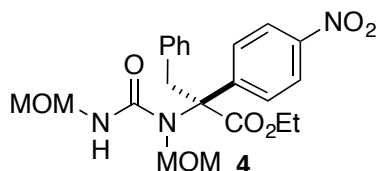
5-Benzyl-1,3-bis(methoxymethyl)-5-*p*-nitrophenylimidazolidin-2,4-dione (**3**) (Table 1, entry 1)



According to the general procedure 2, urea **2** (125 mg, 0.28 mmol) was treated with KHMDS (1.11 mL, 0.50 M in THF, 0.56 mmol) in DMF (1.71 mL, total ratio of DMF/THF = 1.5/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min. The crude product was purified through silica gel column chromatography (AcOEt/hexane = 30/70) to give **3** (105 mg, 93% yield, 83% ee) as a colorless oil; $[\alpha]_D^{20} -2.3$ (*c* 0.53, CHCl_3 , 97% ee); HPLC conditions: Daicel Chiralcel OD-H, hexane/ethanol = 7:3, flow = 0.5 mL/min, $\lambda = 230\text{ nm}$, $t_R = 27$ (minor), 30 (major) min; IR (KBr) cm^{-1} : 1726, 1523, 1351; ^1H NMR (400 MHz, CDCl_3): δ 2.98 (s, 3H), 3.33 (s, 3H), 3.54 (d, $J = 13.7\text{ Hz}$, 1H), 3.85 (d, $J = 13.7\text{ Hz}$, 1H), 4.66 (d, $J = 10.5\text{ Hz}$, 1H), 4.67 (d, $J = 11.0\text{ Hz}$, 1H), 4.76 (d, $J = 10.5\text{ Hz}$, 1H), 4.90 (d, $J = 11.0\text{ Hz}$, 1H), 7.24-7.34 (m, 5H), 7.69 (d, $J = 9.2\text{ Hz}$, 2H), 8.30 (d, $J = 9.2\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 39.29 (t), 57.27 (q), 70.07 (t), 71.38 (s), 73.11 (t), 124.12 (d), 127.95 (d), 128.68 (d), 130.35 (d), 132.56 (s), 143.38 (s), 148.11 (s), 156.19 (s), 172.52 (s); MS (EI) m/z : 399 (M^+), 308 (base peak); HPMS (EI) m/z calcd for $\text{C}_{20}\text{H}_{21}\text{N}_3\text{O}_6$ (M^+): 399.1430 found 399.1430.

(Table 1, entry 2)

According to the general procedure 2, treatment of **2** (99.2 mg, 0.22 mmol) with NaHMDS (0.23 mL, 1.89 M in THF, 0.44 mmol) in DMF (1.95 mL, total ratio of DMF/THF = 8/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min gave **3** (55.5 mg, 64%, 93% ee) and **4** (23.7 mg, 24%), respectively.



4: yellow oil; IR (neat) cm^{-1} : 3392, 3924, 1737, 1681, 1521, 1350, 1068; ^1H NMR (400 MHz, CDCl_3): δ 1.17 (t, $J = 7.3\text{ Hz}$, 3H), 3.08 (s, 3H), 3.35 (s, 3H), 3.61 (d, $J = 10.1\text{ Hz}$, 1H), 3.74 (d, $J = 10.1\text{ Hz}$, 1H), 3.85 (d, $J = 14.2\text{ Hz}$, 1H), 4.07 (d, $J = 14.2\text{ Hz}$, 1H), 4.15 (q, $J = 7.3\text{ Hz}$, 2H), 4.70 (dd, $J = 6.9, 10.1\text{ Hz}$, 1H), 4.74 (dd, $J = 6.9, 10.1\text{ Hz}$, 1H), 6.47 (br t, $J = 6.9\text{ Hz}$, 1H), 7.03-7.05 (m, 2H), 7.17-7.25 (m, 3H), 7.90 (d, $J = 9.2\text{ Hz}$, 2H), 8.19 (d, $J = 9.2\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, acetone- d_6): δ 14.15 (q), 40.48 (t), 54.64 (q), 55.32 (q), 62.02 (t), 73.19 (t), 73.41 (s), 79.78 (t), 123.36 (d),

127.90 (d), 128.91 (d), 130.36 (d), 131.82 (d), 136.88 (s), 147.38 (s), 148.11 (s), 159.93 (s), 171.27 (s); MS (EI) m/z : 445 (M^+), 267 (base peak); HRMS (EI) m/z calcd for $C_{22}H_{27}N_3O_7$ (M^+): 445.1849 found 445.1839. Partial cyclization of **4** was observed during measurement of its ^{13}C NMR spectrum so the specific rotation of pure **4** could not be determined.

(Table 1, entry 3)

According to the general procedure 2, aryl migration of **2** (134 mg, 0.30 mmol) with LiHMDS (1.0 mL, 0.6 M in THF/hexane, 0.60 mmol) in DMF (2.0 mL, total ratio of DMF/THF = 4.25/1) at -60 °C for 30 min gave **3** (70.6 mg, 59%, 99% ee) and **4** (20.1 mg, 15%), respectively.

(Table 1, entry 4)

According to the general procedure 2, urea **2** (134 mg, 0.30 mmol) was treated with LiHMDS (1.0 mL, 0.6 M in THF/hexane, 0.60 mmol) in DMF (1.98 mL, total ratio of DMF/THF = 4/1) at -60 °C for 30 min. After being warmed to 15 °C over 12 hr, the resulting mixture was quenched by addition of sat. NH_4Cl . Usual work-up followed by purification of crude product gave **3** (81.4 mg, 68%, 97% ee).

(Table 1, entry 5)

According to the general procedure 2, treatment of **2** (120 mg, 0.27 mmol) with KHMDS (1.06 mL, 0.51 M in THF, 0.54 mmol) in THF (1.63 mL) at -78 °C for 30 min gave **3** (86 mg, 80%, 7% ee) and **4** (13.5 mg, 8%), respectively.

(Table 1, entry 6)

According to the general procedure 2, treatment of **2** (107 mg, 0.24 mmol) with NaHMDS (0.26 mL, 1.89 M in THF, 0.48 mmol) in THF (2.16 mL) at -78 °C for 30 min gave **3** (60 mg, 62%, 73% ee) and **4** (22 mg, 20%), respectively.

(Table 1, entry 7)

According to the general procedure 2, treatment of **2** (99 mg, 0.22 mmol) with LiHMDS (0.70 mL, 0.63 M in THF/hexane, 0.44 mmol) in THF (1.5 mL) at -78 °C for 30 min gave **3** (13.6 mg, 18%, -23% ee) and recovered **2** (57.1 mg, 58%), respectively.

(Table 1, entry 8)

According to the general procedure 2, treatment of **2** (111 mg, 0.25 mmol) with KHMDS (1.16 mL, 0.43 M in toluene, 0.50 mmol) in toluene (1.33 mL) at 0 °C for 30 min gave **3** (56.2 mg, 55%, -25% ee) and **4** (49.3 mg, 42%), respectively.

(Table 1, entry 9)

According to the general procedure 2, treatment of **2** (120 mg, 0.27 mmol) with NaHMDS (0.28 mL, 1.90 M in THF) in toluene (2.41 mL) at 0 °C for 30 min gave **3** (55.1 mg, 51%, -83% ee) and **4** (34.7 mg, 29%), respectively.

(Table 1, entry 10)

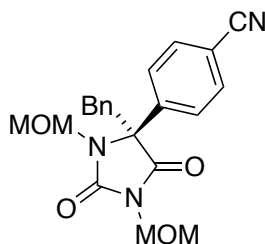
According to the general procedure 2, treatment of **2** (91.3 mg, 0.21 mmol) with NaHMDS (0.22 mL, 1.89 M in THF, 0.41 mmol) in toluene (1.83 mL) at -78 °C for 3.5 hr gave **3** (51 mg, 62%, -61% ee) and **4** (23.8 mg, 26%), respectively.

(Table 1, entry 11)

According to the general procedure 2, treatment of **2** (105 mg, 0.24 mmol) with LiHMDS (0.86 mL, 0.55 M in

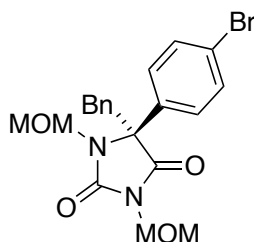
toluene/hexane, 0.47 mmol) in toluene (1.50 mL) at 0 °C for 1 hr gave **3** (35 mg, 37%, -74% ee) and **4** (11.7 mg, 11%), respectively. Small amount of **2** (1.9 mg, 2%) was also recovered.

5-Benzyl-1,3-bis(methoxymethyl)-5-*p*-cyanophenylimidazolidin-2,4-dione (**6**) (Table 2, entry 2)



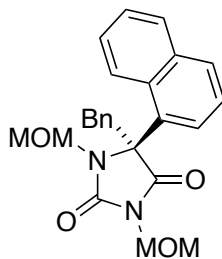
According to the general procedure 2, treatment of **5** (128 mg, 0.30 mmol) with LiHMDS (0.91 mL, 0.66 M in THF/hexane, 0.60 mmol) in DMF (2.09 mL, total ratio of DMF/THF = 5/1) at -60 °C for 30 min gave **6** (102.5 mg, 90%, 98% ee) as a colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/ethanol = 3:2, flow = 0.5 mL/min, $\lambda = 230$ nm, $t_R = 24$ (minor), 27 (major) min; $[\alpha]_D^{22} +2.7$ (*c* 0.64, CHCl₃, 98% ee); IR (KBr) cm⁻¹: 2320, 1780, 1721; ¹H NMR (400 MHz, CDCl₃): δ 2.96 (s, 3H), 3.32 (s, 3H), 3.51 (d, *J* = 13.7 Hz, 1H), 3.82 (d, *J* = 13.7 Hz, 1H), 4.63 (d, *J* = 11.0 Hz, 1H), 4.65 (d, *J* = 10.6 Hz, 1H), 4.75 (d, *J* = 10.6 Hz, 1H), 4.88 (d, *J* = 11.0 Hz, 1H), 7.24-7.30 (m, 5H), 7.60 (d, *J* = 8.3 Hz, 2H), 7.75 (d, *J* = 8.3 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 39.00 (t), 57.13 (q), 57.19 (q), 69.95 (t), 71.39 (s), 73.01 (t), 113.09 (s), 117.92 (s), 127.56 (d), 127.85 (d), 128.56 (d), 130.32 (d), 132.60 (s), 132.71 (d), 141.47 (s), 156.17 (s), 172.56 (s); MS (EI) *m/z*: 379 (M⁺), 288 (base peak); HRMS (EI) *m/z* calcd for C₂₁H₂₁N₃O₄ (M⁺): 379.1532 found 379.1530; Anal. calcd for C₂₁H₂₁N₃O₄: C, 66.48; H, 5.58; N, 11.08 found C, 66.12; H, 5.58; N, 10.98.

5-Benzyl-1,3-bis(methoxymethyl)-5-*p*-bromophenylimidazolidin-2,4-dione (**8**) (Table 2, entry 3)



According to the general procedure 2, treatment of **7** (142 mg, 0.30 mmol) with LiHMDS (0.91 mL, 0.66 M in THF/hexane, 0.60 mmol) in DMF (2.09 mL, total ratio of DMF/THF = 5/1) at -60 °C for 30 min gave **8** (123.9 mg, 97%, 95% ee) as a colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/ethanol = 3:2, flow = 0.5 mL/min, $\lambda = 230$ nm, $t_R = 16$ (minor), 18 (major) min; $[\alpha]_D^{22} -3.6$ (*c* 0.61, CHCl₃, 95% ee); IR (KBr) cm⁻¹: 2935, 1781, 1725, 1449, 1369, 1099; ¹H NMR (400 MHz, CDCl₃): δ 2.95 (s, 3H), 3.32 (s, 3H), 3.50 (d, *J* = 13.7 Hz, 1H), 3.77 (d, *J* = 13.7 Hz, 1H), 4.52 (d, *J* = 11.0 Hz, 1H), 4.65 (d, *J* = 11.0 Hz, 1H), 4.74 (d, *J* = 11.0 Hz, 1H), 4.90 (d, *J* = 11.0 Hz, 1H), 7.24-7.30 (m, 7H), 7.57 (d, *J* = 8.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 38.92 (t), 56.98 (q), 57.15 (q), 69.79 (t), 71.27 (s), 72.95 (t), 123.40 (s), 127.58 (d), 128.27 (d), 128.37 (d), 130.48 (d), 132.21 (d), 133.07 (s), 135.31 (s), 156.29 (s), 173.08 (s); MS (FAB) *m/z*: 457 [(M+2+Na)⁺], 455 [(M+Na)⁺], 154 (base peak); HRMS (FAB) *m/z* calcd for C₂₀H₂₁BrN₂NaO₄ [(M+Na)⁺]: 455.0582 found 455.0584; calcd for C₂₀H₂₁BrN₂NaO₄ [(M+2+Na)⁺]: 457.0565 found 457.0565; Anal. calcd for C₂₀H₂₁BrN₂O₄: C, 55.44; H, 4.89; N, 6.47 found C, 55.10; H, 4.99; N, 6.23.

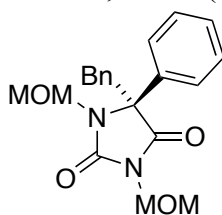
5-Benzyl-1,3-bis(methoxymethyl)-5-(naphthalen-1-yl)imidazolidin-2,4-dione (**12**) (Table 2, entry 5)



According to the general procedure 2, treatment of **11** (122 mg, 0.27 mmol) with LiHMDS (0.82 mL, 0.66 M in THF/hexane, 0.54 mmol) in DMF (1.89 mL, total ratio of DMF/THF = 5/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min gave **12** (51.5 mg, 47%, 84% ee) and recovered **11** (9.6 mg, 8%), respectively.

12: Colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/2-propanol = 85:15, flow = 1.0 mL/min, $\lambda = 230\text{ nm}$, $t_{\text{R}} = 22$ (minor), 45 (major) min; $[\alpha]_{\text{D}}^{22} -14$ ($c\ 1.2$, CHCl_3 , 98% ee); IR (KBr) cm^{-1} : 1776, 1721, 1368, 1101; ^1H NMR (400 MHz, CDCl_3): δ 3.21 (s, 3H), 3.22 (s, 3H), 3.75 (d, $J = 12.4\text{ Hz}$, 1H), 3.97 (d, $J = 12.4\text{ Hz}$, 1H), 4.18 (d, $J = 11.0\text{ Hz}$, 1H), 4.48 (d, $J = 11.0\text{ Hz}$, 1H), 4.51 (d, $J = 11.0\text{ Hz}$, 1H), 4.86 (d, $J = 11.0\text{ Hz}$, 1H), 7.30-7.37 (m, 5H), 7.42-7.46 (m, 2H), 7.48-7.52 (m, 1H), 7.58 (t, $J = 7.8\text{ Hz}$, 1H), 7.91-7.98 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 42.15 (t), 57.18 (q), 57.77 (q), 69.86 (t), 70.40 (s), 72.88 (t), 122.17 (d), 124.92 (d), 125.93 (d), 127.24 (d), 127.73 (d), 127.75 (d), 128.28 (d), 129.85 (d), 129.90 (s), 130.88 (d), 131.16 (d), 133.06 (s), 134.42 (s), 155.90 (s), 174.02 (s); MS (EI^+) m/z : 404 (M^+), 313 (base peak); HRMS (EI) m/z calcd for $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_4$ (M^+): 404.1736 found 404.1737.

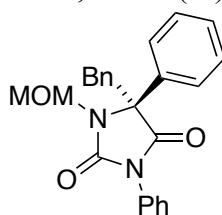
5-Benzyl-1,3-bis(methoxymethyl)-5-phenylimidazolidin-2,4-dione (**14**) (Table 2, entry 6)



According to the general procedure 2, treatment of **13** (74.2 mg, 0.19 mmol) with KHMDS (0.73 mL, 0.51 M in THF, 0.37 mmol) in DMF (1.1 mL, total ratio of DMF/THF = 1.5/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min gave **14** (47.7 mg, 73%, 55% ee) and recovered **13** (27.4 mg, 37%).

14: colorless oil; $[\alpha]_{\text{D}}^{20} -5.6$ ($c\ 0.33$, CHCl_3 , 27% ee); IR (KBr) cm^{-1} : 2933, 1778, 1720, 1448, 1375, 1246, 1180, 1089, 1038; ^1H NMR (400 MHz, CDCl_3): δ 2.96 (s, 3H), 3.31 (s, 3H), 3.55 (d, $J = 13.5\text{ Hz}$, 1H), 3.82 (d, $J = 13.5\text{ Hz}$, 1H), 4.47 (d, $J = 11.2\text{ Hz}$, 1H), 4.67 (d, $J = 10.5\text{ Hz}$, 1H), 4.75 (d, $J = 10.5\text{ Hz}$, 1H), 4.92 (d, $J = 11.2\text{ Hz}$, 1H), 7.25-7.34 (m, 5H), 7.38-7.46 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3): δ 38.92 (t), 56.99 (t), 57.23 (q), 69.81 (t), 71.78 (s), 73.11 (t), 126.54 (d), 127.50 (d), 128.35 (d), 129.22 (d), 130.73 (d), 133.55 (s), 136.22 (s), 156.60 (s), 173.65 (s); MS (EI) m/z : 354 (M^+), 263 (base peak); HRMS (EI) m/z calcd for $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_4$ (M^+): 354.1580 found 354.1581. Enantiomeric excess (ee) was determined by HPLC with chiral stationary after removal of bis(methoxymethyl) group. HPLC conditions: Daicel Chiralpak AD-H, hexane/ethanol = 1:4, flow = 1.0 mL/min, $t_{\text{R}} = 11$ (minor), 20 (major) min.

5-Benzyl-1-methoxymethyl-3,5-diphenylimidazolidin-2,4-dione (**16**) (Table 2, entry 7)

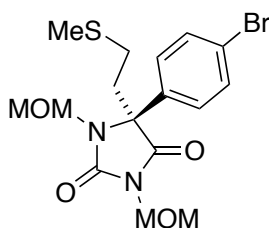


According to the general procedure 2, treatment of **15** (109 mg, 0.25 mmol) with KHMDS (1.0 mL, 0.50 M in THF,

0.5 mmol) in DMF/THF (1.52 mL, total ratio of DMF/THF = 1/1) at $-78\text{ }^{\circ}\text{C}$ for 30 min gave **16** (50.3 mg, 46%, 73% ee) and recovered **15** (44.1 mg, 40%), respectively.

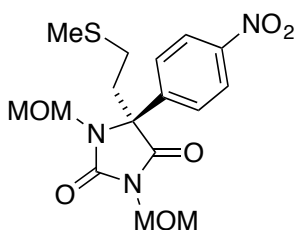
16: colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/2-propanol = 95:5, flow = 1.0 mL/min, $\lambda = 230\text{ nm}$, $t_{\text{R}} = 19$ (minor), 23 (major) min; $[\alpha]_{\text{D}}^{22} +15$ (c 1.4, CHCl_3 , 73% ee); IR (KBr) cm^{-1} : 1775, 1719, 1499, 1421, 1397; ^1H NMR (400 MHz, CDCl_3): δ 3.35 (s, 3H), 3.65 (d, $J = 13.3\text{ Hz}$, 1H), 3.84 (d, $J = 13.3\text{ Hz}$, 1H), 4.53 (d, $J = 11.2\text{ Hz}$, 1H), 4.97 (d, $J = 11.2\text{ Hz}$, 1H), 6.87-6.90 (m, 2H), 7.30-7.39 (m, 8H), 7.43-7.53 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3): δ 39.26 (t), 57.26 (q), 71.57 (s), 73.23 (t), 126.35 (d), 126.76 (d), 127.56 (d), 128.38 (d), 128.92 (d), 129.19 (d), 130.71 (d), 131.08 (s), 133.68 (s), 136.28 (s), 155.91 (s), 172.62 (s); MS (EI) m/z : 386 (M^+), 295 (base peak); HRMS (EI) m/z calcd for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_3$ (M^+): 386.1630 found 386.1624.

5-*p*-Bromophenyl-1,3-bis(methoxymethyl)-5-[2'-(methylthio)ethyl]imidazolidin-2,4-dione (**18**) (Table 3, entry 1)



According to the general procedure 2, treatment of **17** (91.1 mg, 0.20 mmol) with LiHMDS (0.61 mL, 0.65 M in THF/hexane, 0.39 mmol) in DMF (1.36 mL, total ratio of DMF/THF = 5/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min gave **18** (70.8 mg, 86%, 96% ee) as a yellow oil; HPLC conditions: Daicel Chiralpak IA; hexane/2-propanol = 83:17, flow = 1.0 mL/min, $\lambda = 254\text{ nm}$, $t_{\text{R}} = 18$ (minor), 34 (major) min; $[\alpha]_{\text{D}}^{22} +35$ (c 0.91, CHCl_3 , 96% ee); IR (neat) cm^{-1} : 2934, 1781, 1725, 1490, 1443, 1373, 1179, 1087, 1011; ^1H NMR (400 MHz, CDCl_3): δ 2.13 (s, 3H), 2.36-2.48 (m, 2H), 2.59-2.68 (m, 1H), 2.75-2.85 (m, 1H), 3.39 (s, 3H), 3.41 (s, 3H), 4.28 (d, $J = 11.0\text{ Hz}$, 1H), 4.96 (s, 2H), 5.04 (d, $J = 11.0\text{ Hz}$, 1H), 7.17 (d, $J = 8.7\text{ Hz}$, 2H), 7.54 (d, $J = 8.7\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 15.30 (q), 27.60 (t), 33.93 (t), 57.30 (t), 57.70 (t), 69.89 (s), 70.22 (t), 72.50 (t), 123.57 (s), 127.70 (d), 132.50 (d), 135.00 (s), 156.31 (s), 172.97 (s); MS (FAB) m/z : 441 [$(\text{M}+2+\text{Na})^+$], 439 [$(\text{M}+\text{Na})^+$], 154 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{16}\text{H}_{21}\text{BrN}_2\text{NaO}_4\text{S}$ [$(\text{M}+\text{Na})^+$]: 439.0303 found 439.0303; calcd for $\text{C}_{16}\text{H}_{21}\text{BrN}_2\text{NaO}_4\text{S}$ [$(\text{M}+2+\text{Na})^+$]: 441.0284 found 441.0283.

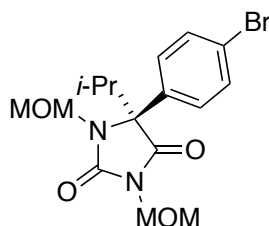
1,3-bis(methoxymethyl)-5-[2'-(methylthio)ethyl]-5-*p*-nitrophenylimidazolidin-2,4-dione (**20**) (Table 3, entry 2)



According to the general procedure 2, treatment of **19** (67.1 mg, 0.16 mmol) with LiHMDS (0.50 mL, 0.66 M in THF/hexane, 0.33 mmol) in DMF (1.16 mL, total ratio of DMF/THF = 5/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min gave **20** (42.7 mg, 71%, 96% ee) as a yellow oil; HPLC conditions: Daicel Chiralcel OD-H; hexane/2-propanol = 9:1, flow = 1.0 mL/min, $\lambda = 270\text{ nm}$, $t_{\text{R}} = 9$ (major), 11 (minor) min; $[\alpha]_{\text{D}}^{21} +32$ (c 1.6, CHCl_3 , 96% ee) IR (neat) cm^{-1} : 2936, 1783, 1725, 1524, 1444, 1350, 1180, 1090; ^1H NMR (400 MHz, CDCl_3): δ 2.14 (s, 3H), 2.39-2.53 (m, 2H), 2.59-2.66 (m, 1H), 2.85-2.92 (m, 1H), 3.40 (s, 3H), 3.42 (s, 3H), 4.37 (d, $J = 11.5\text{ Hz}$, 1H), 4.97 (s, 2H), 5.07 (d, $J = 11.5\text{ Hz}$, 1H), 7.53 (d, $J = 9.2\text{ Hz}$, 2H), 8.27 (d, $J = 8.7\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, DCDCl_3): δ 15.37 (q), 27.57 (t), 34.20 (t), 57.41 (q), 57.80 (q), 69.96 (s), 70.39 (t), 72.62 (t), 124.36 (d), 127.29 (d), 143.01 (s), 148.16 (s), 156.15 (s), 172.23 (s); MS (FAB) m/z : 406 [$(\text{M}+\text{Na})^+$], 154 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{16}\text{H}_{21}\text{N}_3\text{NaO}_6\text{S}$ [$(\text{M}+\text{Na})^+$]: 406.1049 found

406.1050.

5-*p*-Bromophenyl-5-isopropyl-1,3-bis(methoxymethyl)imidazolidin-2,4-dione (**22**) (Table 3, entry 3)



According to the general procedure 2, treatment of **21** (56.9 mg, 0.13 mmol) with LiHMDS (0.47 mL, 0.57 M in THF/hexane, 0.26 mmol) in DMF (1.1 mL, total ratio of DMF/THF = 5/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min gave a 1:2.7 mixture of **22** (98% ee) and α -arylated compound in 91% combined yield.

22: as a colorless oil; HPLC conditions: Daicel Chiralpak AD-H; hexane/2-propanol = 9:1; flow = 1.0 mL/min; λ = 254 nm, t_{R} = 13 (*R*), 17 (*S*) min; $[\alpha]_{\text{D}}^{20}$ +48 (*c* 1.1, CHCl_3 , 98% ee); IR (neat) cm^{-1} : 2920, 2850, 1779, 1723, 1444, 1377, 1245, 1183, 1087, 1094, 1009; ^1H NMR (400 MHz, CDCl_3): 1.05 (d, J = 6.9 Hz, 3H), 1.07 (d, J = 6.9 Hz, 3H), 2.98 (sept, J = 6.9 Hz, 1H), 3.36 (s, 3H), 3.45 (s, 3H), 4.35 (d, J = 11.0 Hz, 1H), 4.93 (d, J = 10.6 Hz, 1H), 4.95 (d, J = 10.6 Hz, 1H), 5.13 (d, J = 11.0 Hz, 1H), 7.14 (d, J = 8.3 Hz, 2H), 7.54 (d, J = 8.3 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 15.49 (q), 16.39 (q), 32.34 (d), 57.03 (q), 57.53 (q), 70.05 (t), 72.76 (t), 74.58 (s), 122.95 (s), 128.42 (d), 132.35 (d), 134.42 (s), 156.90 (s), 173.48 (s); MS (FAB) m/z : 409 $[(\text{M}+2+\text{Na})^+]$, 407 $[(\text{M}+\text{Na})^+]$, 353 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{16}\text{H}_{21}\text{BrN}_2\text{NaO}_4$ $[(\text{M}+\text{Na})^+]$: 407.0582 found 407.0579; calcd for $\text{C}_{16}\text{H}_{21}\text{BrN}_2\text{NaO}_4$ $[(\text{M}+2+\text{Na})^+]$: 409.0564 found 409.0565; Anal. calcd for $\text{C}_{16}\text{H}_{21}\text{BrN}_2\text{O}_4$: C, 49.88; H, 5.49; N, 7.27 found C, 49.88; H, 5.59; N, 7.26.

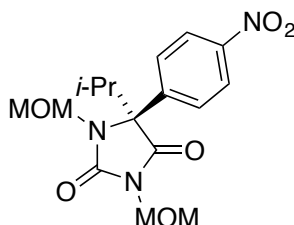
(Table 3, entry 4)

According to the general procedure 2, urea **21** (41.7 mg, 0.097 mmol) was treated with LiHMDS (0.33 mL, 0.58 M in THF/hexane, 0.19 mmol) in DMF (0.81 mL, total ratio of DMF/THF = 5/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min. After being warmed to $20\text{ }^{\circ}\text{C}$ over 12 hr, the resulting mixture was quenched by addition of sat. NH_4Cl . Usual work-up followed by purification of crude product gave **22** (30.0 mg, 80%, 98% ee).

(Table 3, entry 5)

According to the general procedure 2, treatment of **21** (40 mg, 0.093 mmol) with KHMDS (0.42 mL, 0.44 M in THF) in DMF (0.51 mL, total ratio of DMF/THF = 1.2/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min gave **22** (36.3 mg, quant, 56% ee).

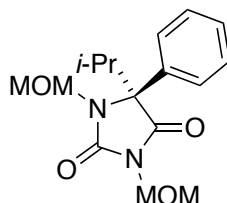
5-Isopropyl-1,3-bis(methoxymethyl)-5-*p*-nitrophenylimidazolidin-2,4-dione (**24**) (Table 3, entry 6)



According to the general procedure 2, urea **23** (65 mg, 0.16 mmol) was treated with LiHMDS (0.55 mL, 0.60 M in THF/hexane, 0.33 mmol) in DMF (1.33 mL, total ratio of DMF/THF = 5/1) at $-60\text{ }^{\circ}\text{C}$ for 30 min. After being warmed to $20\text{ }^{\circ}\text{C}$ over 12 hr, the resulting mixture was quenched by addition of sat. NH_4Cl . Usual work-up followed by purification of crude product gave **24** (56.2 mg, quant, 99% ee) as a colorless oil; HPLC conditions: Daicel Chiralpak AD-H; hexane/2-propanol = 9:1; flow = 1.0 mL/min; λ = 254 nm; t_{R} = 26 (*R*), 29 (*S*) min; $[\alpha]_{\text{D}}^{20}$ +58 (*c* 0.23, CHCl_3);

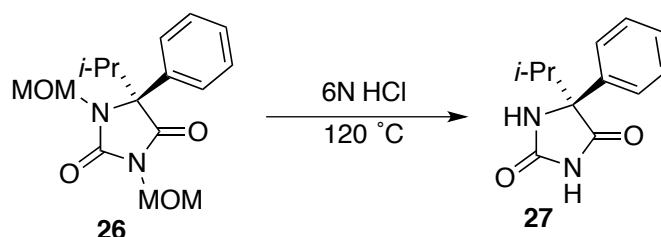
IR (neat) cm^{-1} : 3394, 2919, 2850, 1780, 1723, 1598, 1524, 1445, 1349, 1181, 1092; ^1H NMR (400 MHz, CDCl_3): δ 1.06 (d, $J=6.9$ Hz, 3H), 1.10 (d, $J=6.9$ Hz, 3H), 3.06 (sept, $J=6.9$ Hz, 1H), 3.37 (s, 3H), 3.45 (s, 3H), 4.42 (d, $J=11.4$ Hz, 1H), 4.93 (d, $J=10.6$ Hz, 1H), 4.96 (d, $J=10.6$ Hz, 1H), 5.19 (d, $J=11.4$ Hz, 1H), 7.49 (d, $J=8.7$ Hz, 2H), 8.27 (d, $J=8.7$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 15.51 (q), 16.31 (q), 32.99 (d), 57.13 (q), 57.66 (q), 70.24 (t), 72.86 (t), 124.25 (d), 127.90 (d), 142.63 (s), 147.83 (s), 156.69 (s), 172.77 (s); MS (FAB) m/z : 374 $[(\text{M}+\text{Na})^+]$, 320 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{16}\text{H}_{21}\text{N}_3\text{NaO}_6$: 374.1328 found 374.1327.

5-Isopropyl-1,3-bis(methoxymethyl)-5-phenylimidazolidin-2,4-dione (**26**) (Table 3, entry 7)



According to the general procedure 2, treatment of **25** (97 mg, 0.28 mmol) with KHMDS (1.08 mL, 0.51 M in THF, 0.55 mmol) in DMF (1.72 mL, total ratio of DMF/THF = 1.6/1) at -60 °C for 3 hr gave **26** (72 mg, 86%, 61% ee) as a colorless oil; HPLC conditions: Daicel Chiralpak ID; hexane/2-propanol = 9:1; flow = 1.0 mL/min; $\lambda = 254$ nm; $t_R = 14$ (minor), 17 (major) min; $[\alpha]_D^{22} +28$ (c 0.50, CHCl_3 , 52% ee); IR (neat) cm^{-1} : 2938, 1779, 1723, 1448, 1371, 1183, 1090; ^1H NMR (400 MHz, CDCl_3): δ 1.07 (d, $J=6.9$ Hz, 3H), 1.09 (d, $J=6.9$ Hz, 3H), 3.05 (sept, $J=6.9$ Hz, 1H), 3.36 (s, 3H), 3.45 (s, 3H), 4.36 (d, $J=11.4$ Hz, 1H), 4.93 (d, $J=10.5$ Hz, 1H), 4.96 (d, $J=10.5$ Hz, 1H), 5.15 (d, $J=11.4$ Hz, 1H), 7.23-7.26 (m, 2H), 7.33-7.43 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 15.52 (q), 16.46 (q), 32.12 (d), 56.94 (q), 57.42 (q), 69.95 (t), 72.80 (t), 74.98 (s), 126.65 (d), 128.61 (d), 129.16 (d), 135.21 (s), 157.07 (s), 173.90 (s); MS (FAB) m/z : 329 $[(\text{M}+\text{Na})^+]$, 275 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{16}\text{H}_{22}\text{N}_2\text{NaO}_4$: 329.1477 found 329.1482. The enantiomeric excess was determined after its conversion into **27**.

Conversion of **26** into **27** to determine enantiomeric excess and the absolute configuration of **26**

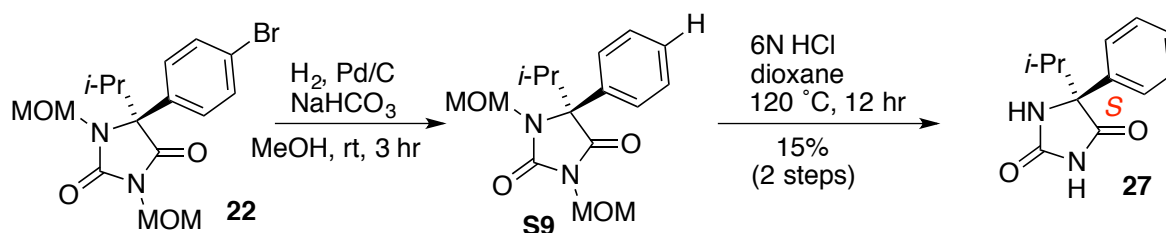


A mixture of **27** (29 mg, 94 μmol) in 6N HCl (1.0 mL) was stirred at 120 °C for 14.5 hr and extracted with AcOEt. The extracts were washed with brine, dried over MgSO_4 , filtered, and concentrated. The residue was purified by PTLC (hexane/AcOEt = 1:1) to give hydantoin (7.7 mg, 38%); HPLC conditions: Daicel Chiralpak AD-H, hexane/ethanol = 1:1, flow = 0.5 mL/min, $\lambda = 230$ nm, $t_R = 11$ (*R*), 17 (*S*) min, $[\alpha]_D^{18} +79$ (c 0.24, EtOH, 61% ee) [lit.ⁱⁱⁱ] $[\alpha]_{589} -150$ (c 1, EtOH) for (*R*)-enantiomer]; ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 0.62 (d, $J=6.9$ Hz, 3H), 0.88 (d, $J=6.4$ Hz, 3H), 2.42-2.49 (m, 1H), 7.29-7.33 (m, 1H), 7.39 (t, $J=7.8$ Hz, 2H), 7.51-7.53 (m, 2H), 8.72 (br s, 1H), 10.74 (br s, 1H); ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$): δ 16.30 (q), 16.67 (q), 35.12 (d), 71.43 (s), 125.50 (d), 127.75 (d), 128.42 (d), 138.67 (s), 157.05 (s), 176.38 (s); MS (FAB) m/z : 219 $[(\text{M}+\text{H})^+]$, 93 (base peak); HRMS (FAB) m/z calcd for $\text{C}_{12}\text{H}_{15}\text{N}_2\text{O}_2$ $[(\text{M}+\text{H})^+]$: 219.1134 found 219.1136. The ^1H NMR and ^{13}C NMR spectra were identified with reported ones.^{iv)}

iii) Coquerel, G.; Petit, S. *J. Cryst. Growth* **1993**, *130*, 173-180.

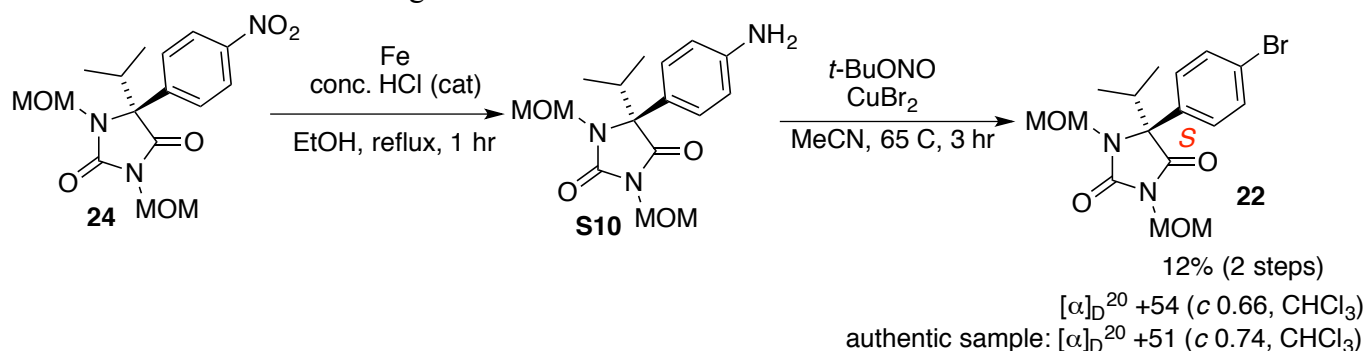
iv) Kleinpeter, E.; Heydenreich, M.; Kalder, L.; Koch, A.; Henning, D.; Kempter, G.; Benassi, R.; Taddei, F. *J. Mol. Struct.* **1997**, *403*, 111-122.

Determination of the absolute configuration of **22**



A mixture of **22** (70 mg, 0.18 mmol), Pd/C (9.7 mg, 10% on carbon, 0.91 μmol), and NaHCO_3 (99.4 mg, 1.18 mmol) in MeOH (2.0 mL) was stirred under H_2 atmosphere at rt for 3 hr.^{v)} The mixture was filtered off and the filtrate was concentrated. The residue was dissolved in AcOEt and washed with water. The aqueous layer was extracted with AcOEt and the combined organic layers were washed with brine, and dried over MgSO_4 , filtered, and concentrated to give **S9**, which was dissolved in dioxane (0.5 mL) and 6N HCl (1.5 mL) was added. The resulting mixture was heated at 120 °C for 12 hr. After being cooled to rt, the mixture was extracted with AcOEt. The extracts were washed with brine, and dried over MgSO_4 , filtered, and concentrated. The residue was purified by PTLC (AcOEt/hexane = 1/1) to give **27** (6.2 mg, 15% (2 steps)) as a colorless solid. The absolute configuration was determined to be *S* by HPLC with chiral stationary. The specific optical rotation of **22** in this scheme was determined to be +51 (c 0.79, CHCl_3).

Determination of the absolute configuration of **24**



To a solution of **24** (156.3 mg, 0.45 mmol) in EtOH (3 mL) were added Fe (248.5 mg, 4.45 mmol), water (0.75 mL), and conc. HCl (5 drops), successively. After being stirred for 1 hr at 95 °C, the mixture was filtered through a pad of Celite. The filtrate was extracted with AcOEt and the extracts were washed with water and brine, and dried over MgSO_4 , filtered, and concentrated to give **S10**, which was dissolved in MeCN (1.5 mL). This solution was added to a stirring solution of *t*-BuONO (0.089 mL, 0.75 mmol) and CuBr_2 (124.2 mg, 0.56 mmol) in MeCN (1.0 mL) at 65 °C.^{vi)} After being stirred for 3 hr at same temperature, the reaction was quenched by addition of 1N HCl (1.0 mL) at rt and the mixture was extracted with AcOEt. The extracts were washed with water and brine, and dried over MgSO_4 , filtered, and concentrated. The residual oil was chromatographed on silica gel (AcOEt/hexane = 3/7) to give an oil, which was further purified by PTLC (AcOEt/ CH_2Cl_2 = 5/95) to obtain **22** (19.9 mg, 12% (2 steps)) as a colorless oil. The ^1H NMR spectrum of **22** was identified with that of **22** which was prepared from **8**. The absolute configuration of **22** was determined to be *S* by comparison of the sign of specific optical rotation with the authentic sample (mentioned above) ($[\alpha]_{\text{D}}^{20} +54$ (c 0.66, CHCl_3), authentic sample: $[\alpha]_{\text{D}}^{20} +51$ (c 0.79, CHCl_3)).

Determination of half-life of racemization

The barrier to racemization of lithium enolate **A** generated from **9** in DMF-THF (2:1) at -78 °C was determined

v) Ramanathan, A.; Jimenez, L. S. *Synthesis* **2010**, 217-220.

vi) Mori, K.; Ehara, K.; Kurihara, K.; Akiyama, T. *J. Am. Chem. Soc.* **2011**, *133*, 6166-6169.

through the periodic quenching of the enolate with methyl iodide. Experiments were performed at the strictly controlled temperature. The decrease in ee ($\ln ee^0/ee^t$) as a function of time for base-treatment of **9** was plotted (Figure 1). The barrier was determined to be 15.2 kcal/mol at -78°C .

Table: Ee value at the indicated time

time (min)	ee^t	$\ln (ee^0/ee^t)$
0	26	–
120	17	0.42
240	13	0.69
360	10	0.96
480	7	1.31
600	6	1.47

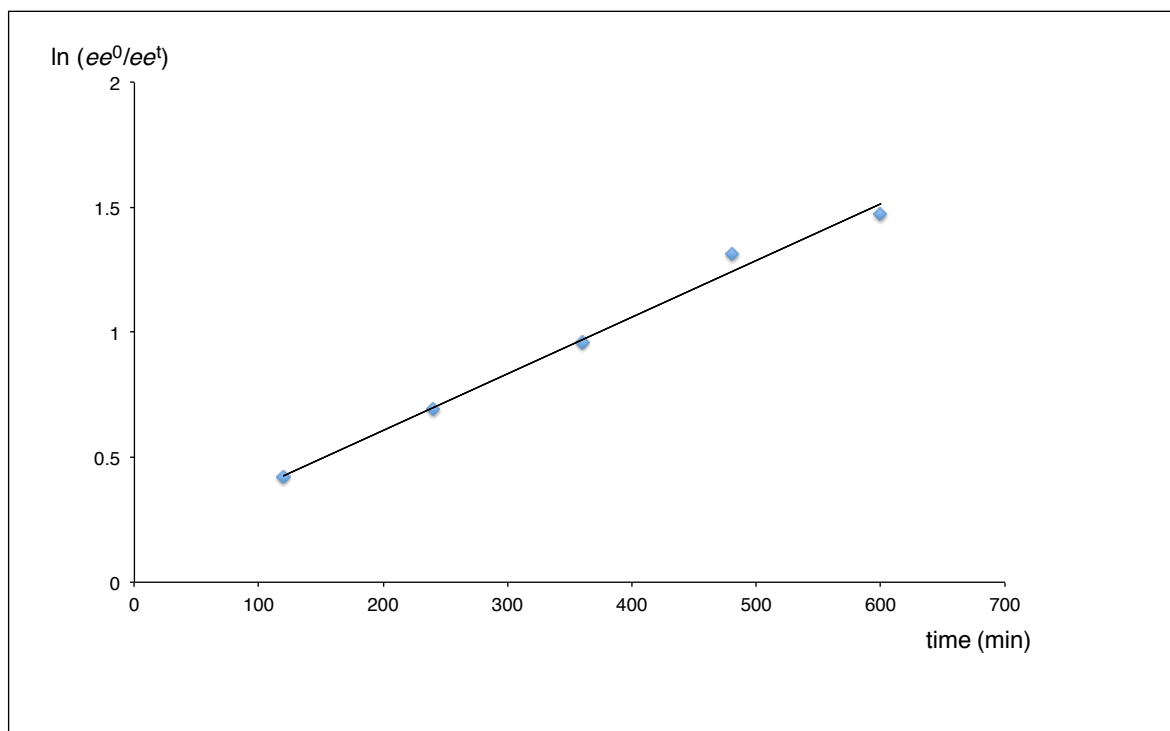
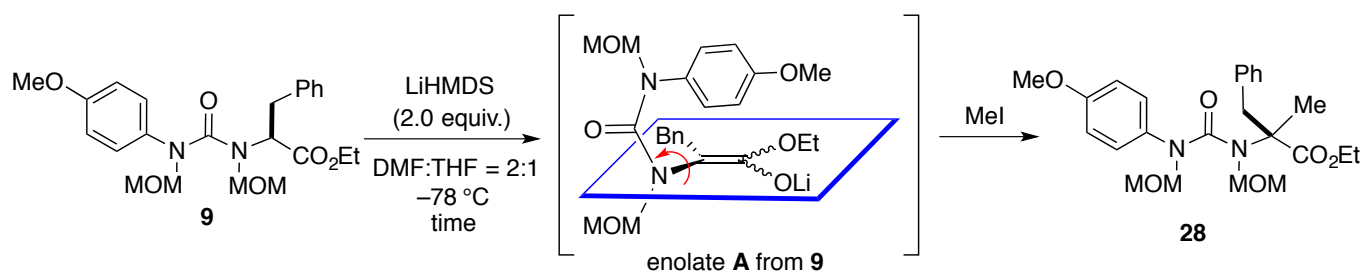
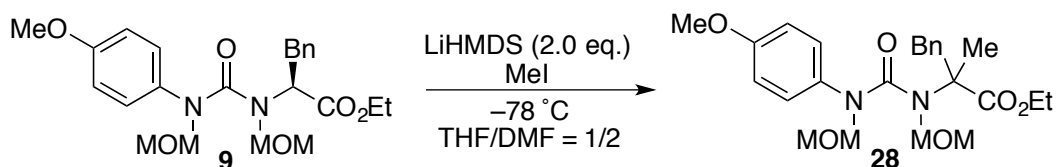


Figure 1. Plot of $\ln (ee^0/ee^t)$ versus time (t) for base-treatment of **9**. ee^0 = the ee value of **28** obtained by the reaction of the enolate immediately after its generation from **9** and LiHMDS with methyl iodide. ee^t = the ee value of **28** obtained by the treatment of **9** with KHMDS for the time indicated followed by addition of methyl iodide. Experiments were performed at the strictly controlled temperature. The barrier to racemization was determined to be 15.2 kcal/mol at -78°C from the slope, $2k = 2.27 \times 10^{-3} \text{ min}^{-1}$ ($r^2 = 0.99$).

Procedure for α -Methylation of **9**



A 0.64 M solution of LiHMDS in THF was prepared as followed:

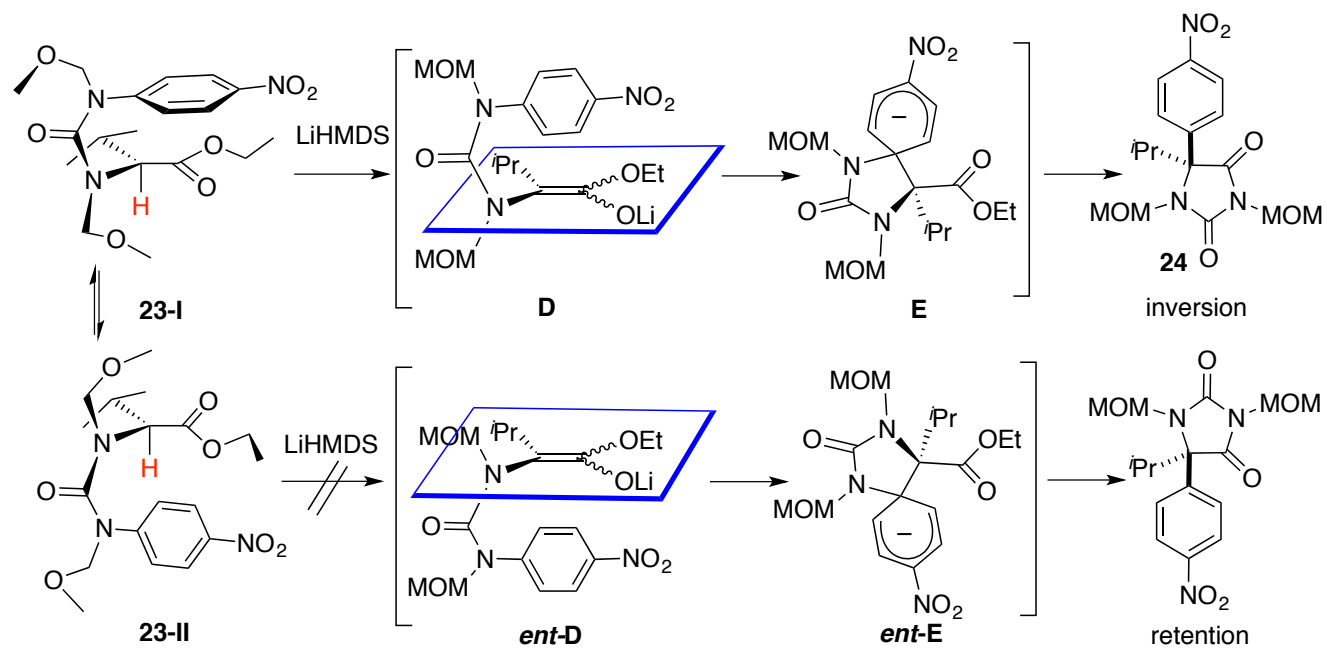
n-BuLi (4.14 mL, 1.55 M in *n*-hexane, 6.4 mmol) was added dropwise to a solution of HMDS (1.33 mL, 6.4 mmol) in THF (4.53 mL) at 0 °C and the resulting mixture was stirred for 30 min at 0 °C. This LiHMDS solution was employed for determination of half-life of racemization.

Methylation of urea **9** was conducted as followed:

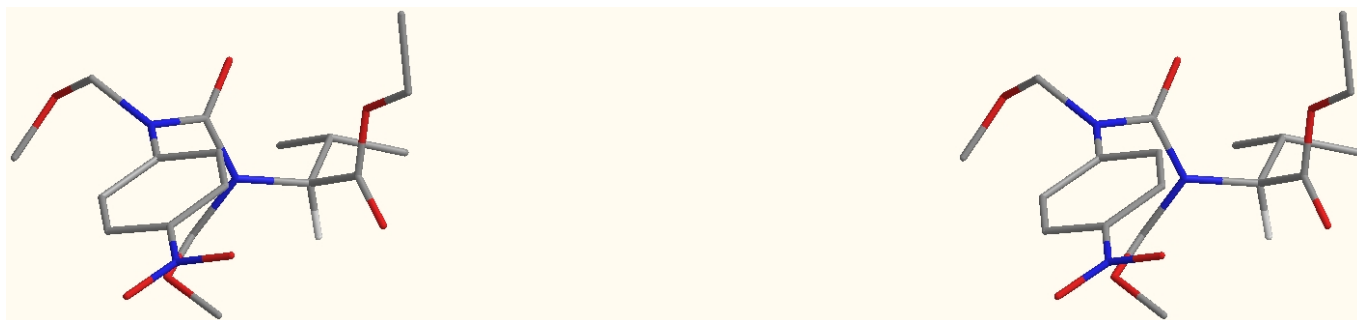
To a solution of LiHMDS (0.64 M in THF, 0.47 mL, 0.30 mmol) in DMF (0.44 mL) was added dropwise a solution of urea **9** (64.6 mg, 0.15 mmol) in DMF/THF (0.60 mL, 2/1, v/v) over 2 min at -78 °C. After being stirred for 1.5 min (time = 0 min), for 2 hr (time = 120 min), for 4 hr (time = 240 min), for 6 hr (time = 360 min), for 8 hr (time = 480 min), or for 10 hr (time = 600 min), MeI (0.19 mL, 3.0 mmol) was added dropwise over 1 min at -78 °C. After being stirred for additional 1.5 min, 5N AcOH/THF (0.2 mL) was added. The resulting mixture was poured into sat. NH₄Cl aq., and then extracted with AcOEt. The extracts were washed with sat. NaHCO₃ aq., water, and brine, and dried over MgSO₄, filtered, and concentrated. The residual oil was purified by PTLC (AcOEt/hexane = 30:70) to give a small amount of **28** (<10% yield) as a colorless oil; HPLC conditions: Daicel Chiralcel OD-H, hexane/2-propanol = 95/5, flow = 0.5 mL/min, λ = 254 nm, t_R = 20 (minor), 23 (major) min; IR (neat) cm⁻¹: 2986, 2935, 1731, 1652, 1512, 1247, 1086, 910, 733; ¹H NMR (400 MHz, CDCl₃): δ 1.17 (t, J = 7.3 Hz, 3H), 1.59 (s, 3H), 2.91 (s, 3H), 3.10 (d, J = 12.8 Hz, 1H), 3.35 (d, J = 12.8 Hz, 1H), 3.41 (s, 3H), 3.81 (s, 3H), 4.02 (d, J = 11.4 Hz, 1H), 4.06-4.18 (m, 2H), 4.38 (d, J = 11.4 Hz, 1H), 4.93 (d, J = 10.1 Hz, 1H), 5.10 (d, J = 10.1 Hz, 1H), 6.88 (d, J = 9.2 Hz, 2H), 7.13-7.16 (m, 2H), 7.21-7.28 (m, 3H), 7.37 (d, J = 9.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 13.87 (q), 21.57 (q), 42.58 (t), 54.42 (q), 55.34 (q), 55.94 (q), 60.88 (t), 65.67 (s), 77.94 (t), 82.69 (t), 114.29 (d), 126.03 (d), 126.72 (d), 127.91 (d), 130.52 (d), 136.15 (s), 137.18 (s), 157.26 (s), 160.95 (s), 173.52 (s); MS (FAB) m/z : 467 [(M+Na)⁺, base peak]; HRMS (FAB) m/z calcd for C₂₄H₃₂N₂NaO₆: 476.2158 found 467.2157.

Conformational analysis of compound **23**

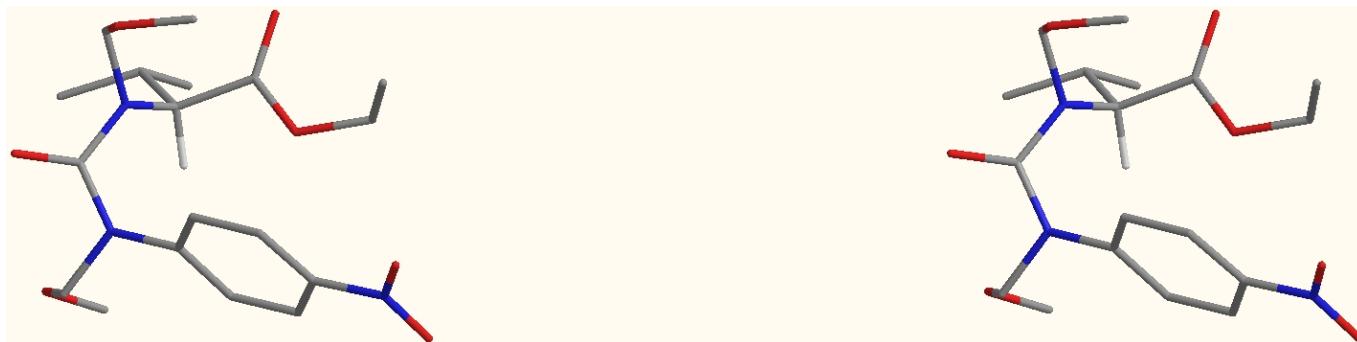
The stable conformers **23-I** and **23-II** were generated by molecular modeling search of **23** (MCMM, 10,000 steps) with OPLS 2005 force field in vacuum using MacroModel (V. 9.0). The **23-II** was found to be the most stable conformer among all possible conformers. The C(α)-H bond in **23-II** is synperiplanar with respect to the N(C=O)N(MOM)Ar bond. On the other hand, the **23-I** was found to be the most stable conformers, in which C(α)-H bond is antiperiplanar with respect to the N(C=O)N(MOM)Ar bond. The difference in potential energies between **23-I** and **23-II** is estimated to be 1.2 kcal/mol (**23-II** is more stable than **23-I**).

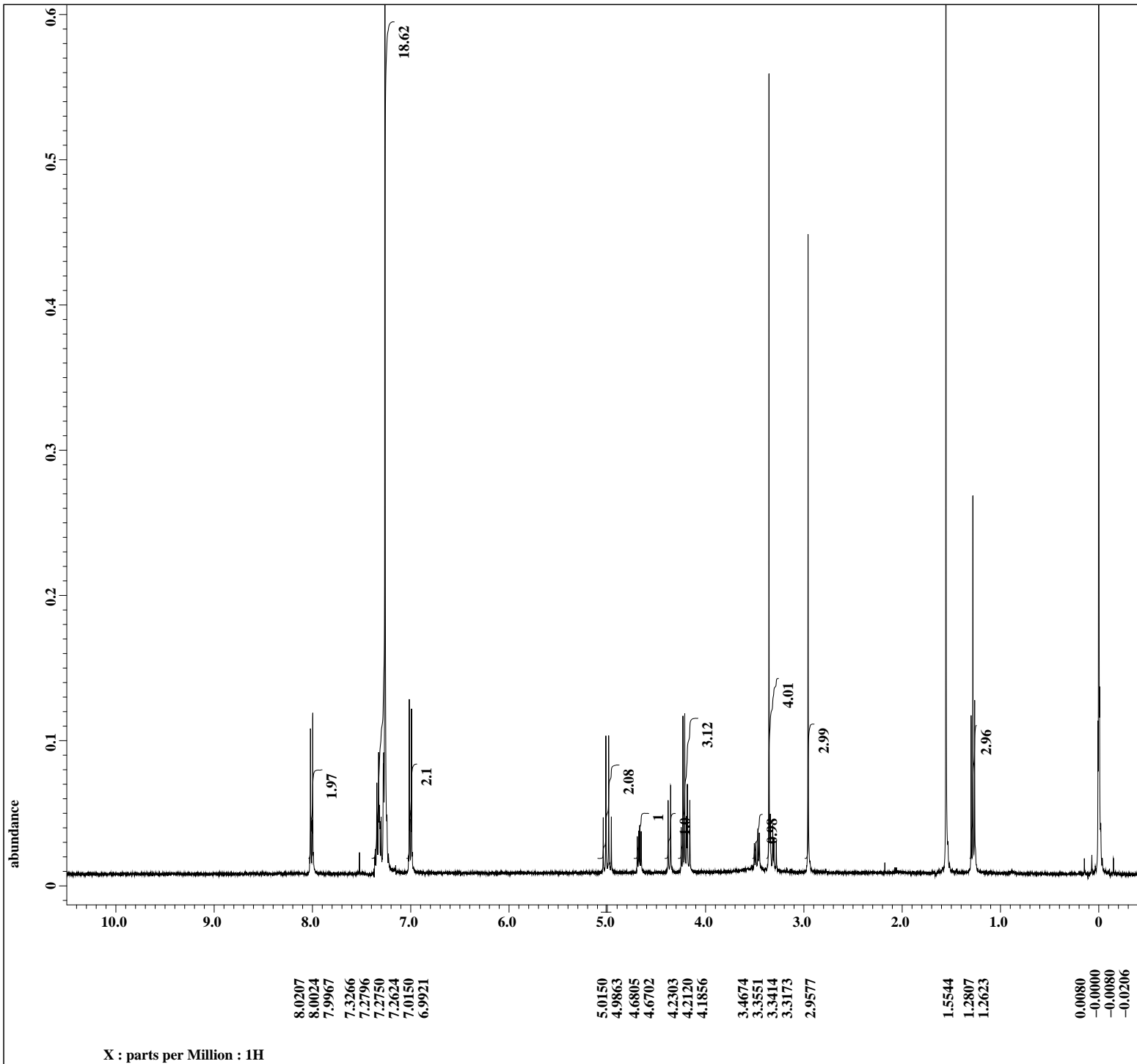


(Stereoview of conformer 23-I)



(Stereoview of conformer 23-II)



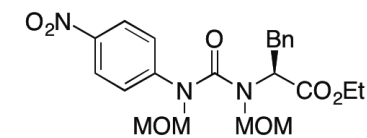


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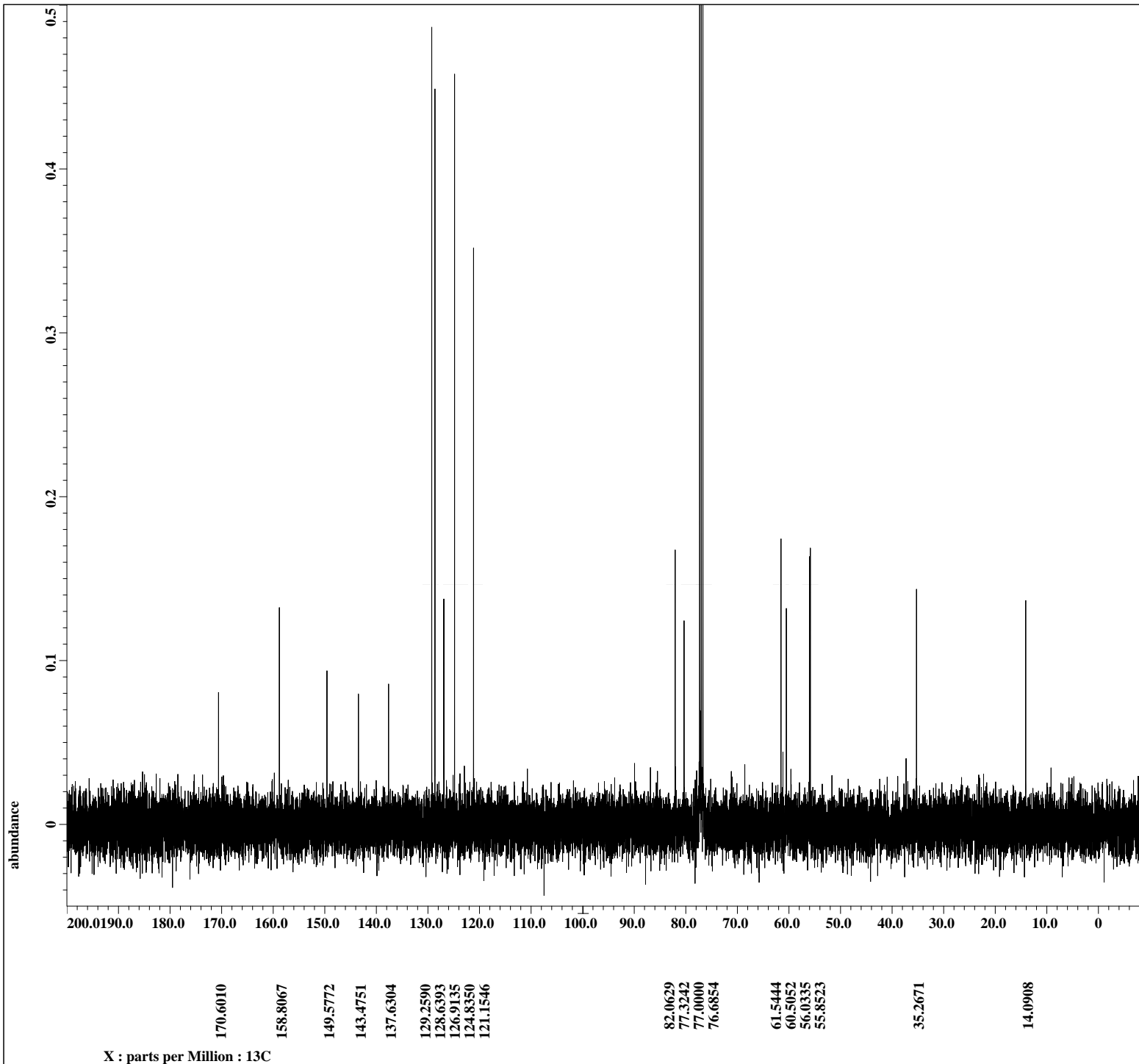
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2

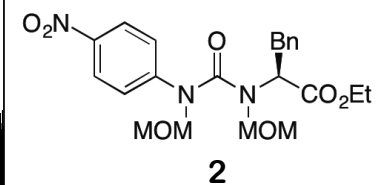


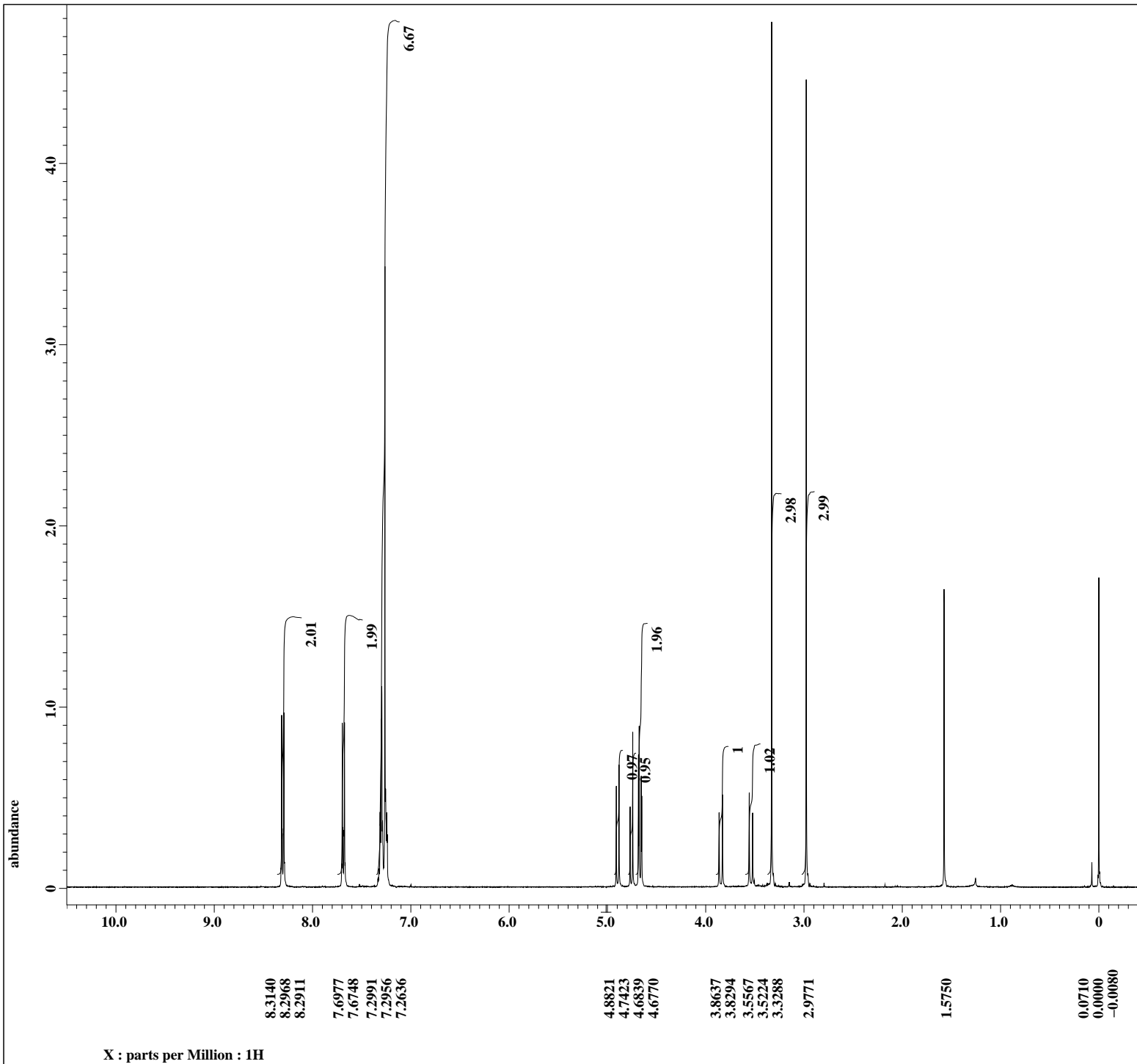
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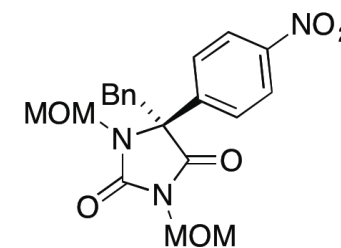


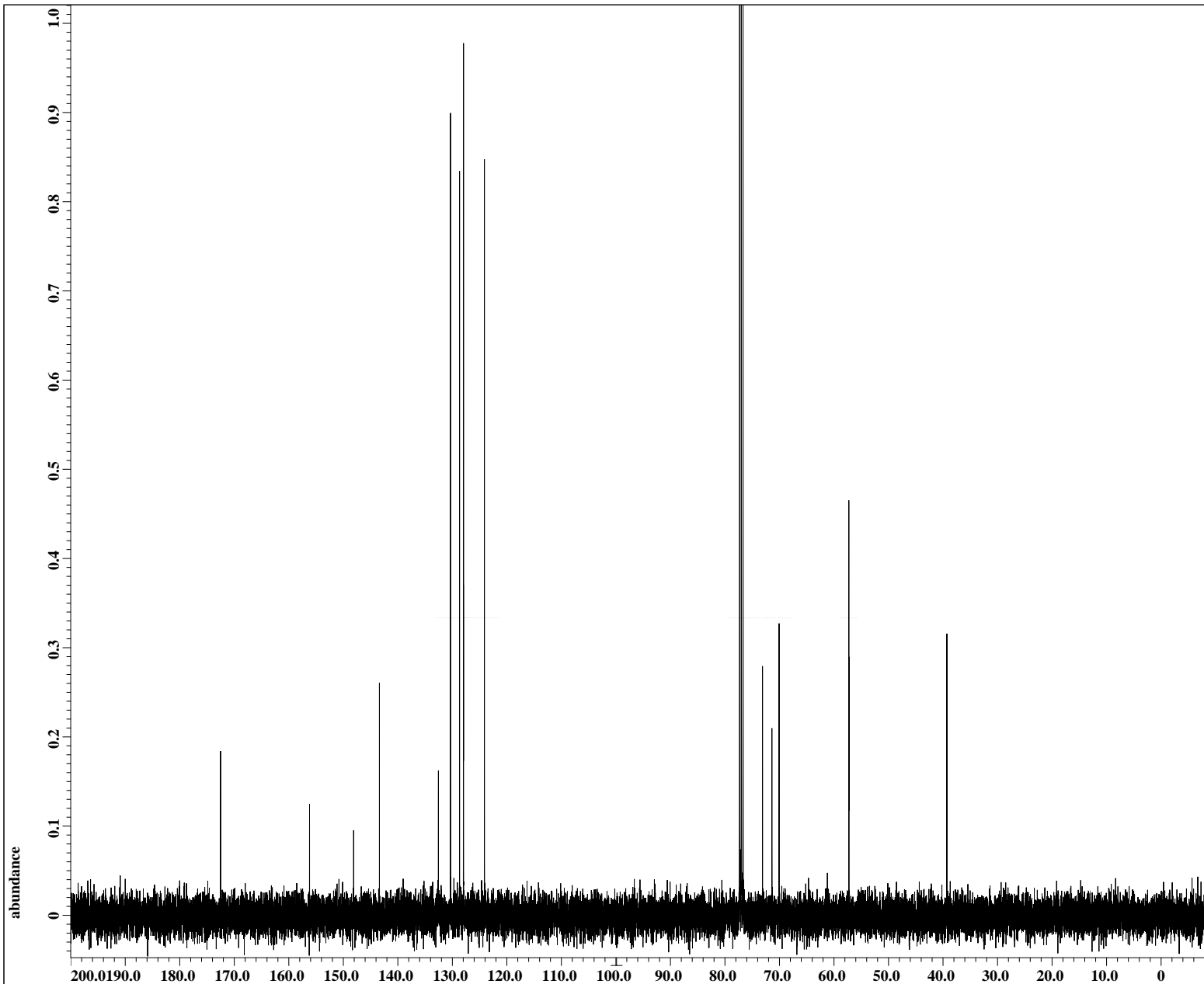
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 Dante_presat = FALSE
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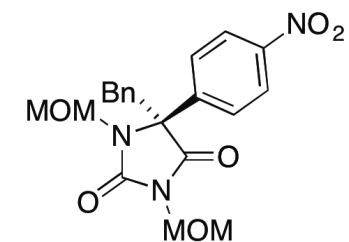
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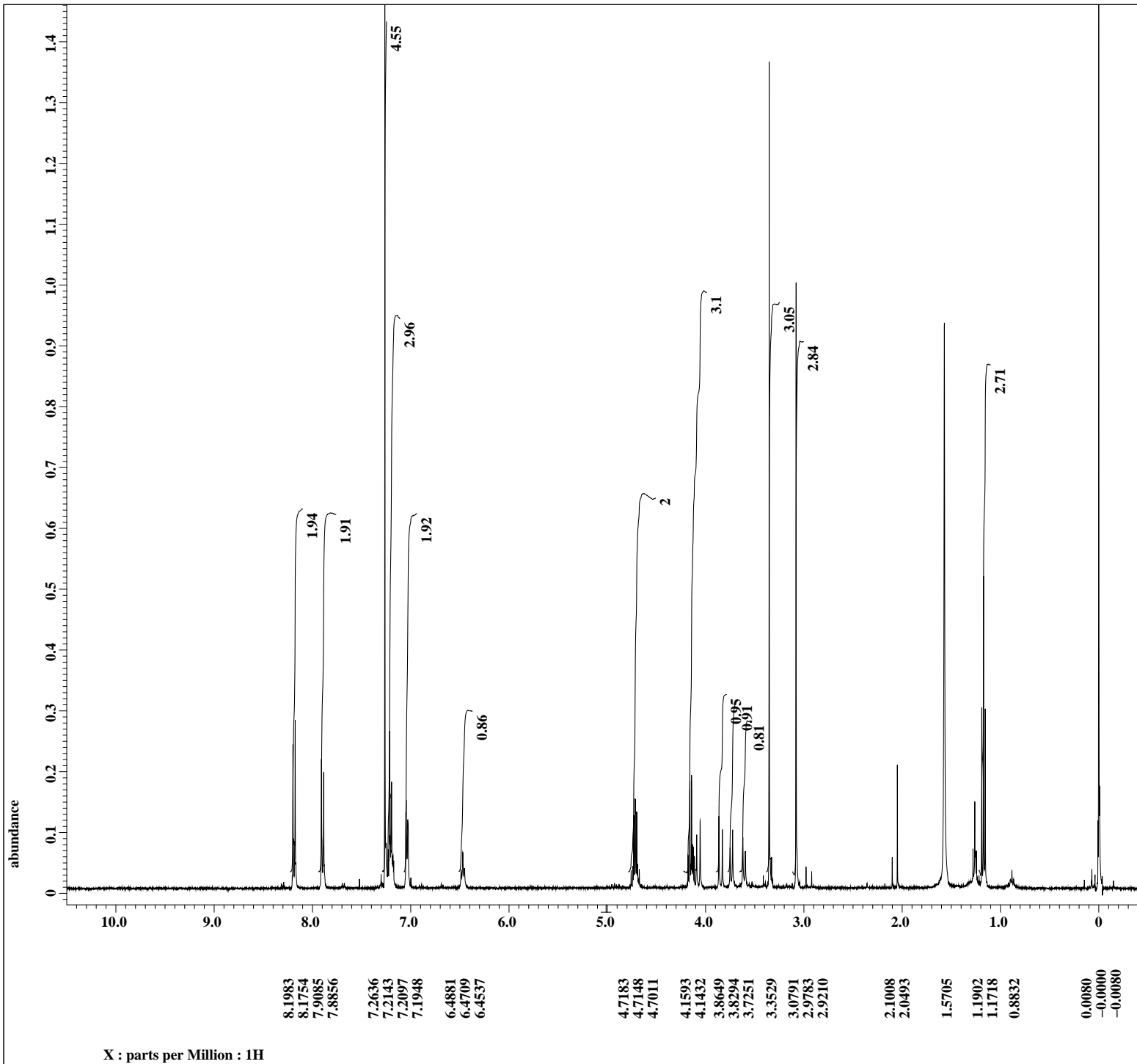
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Initial_wait   = 1[s]
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Noe_time       = 2[s]
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```



3

X : parts per Million : 13C

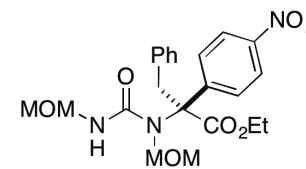


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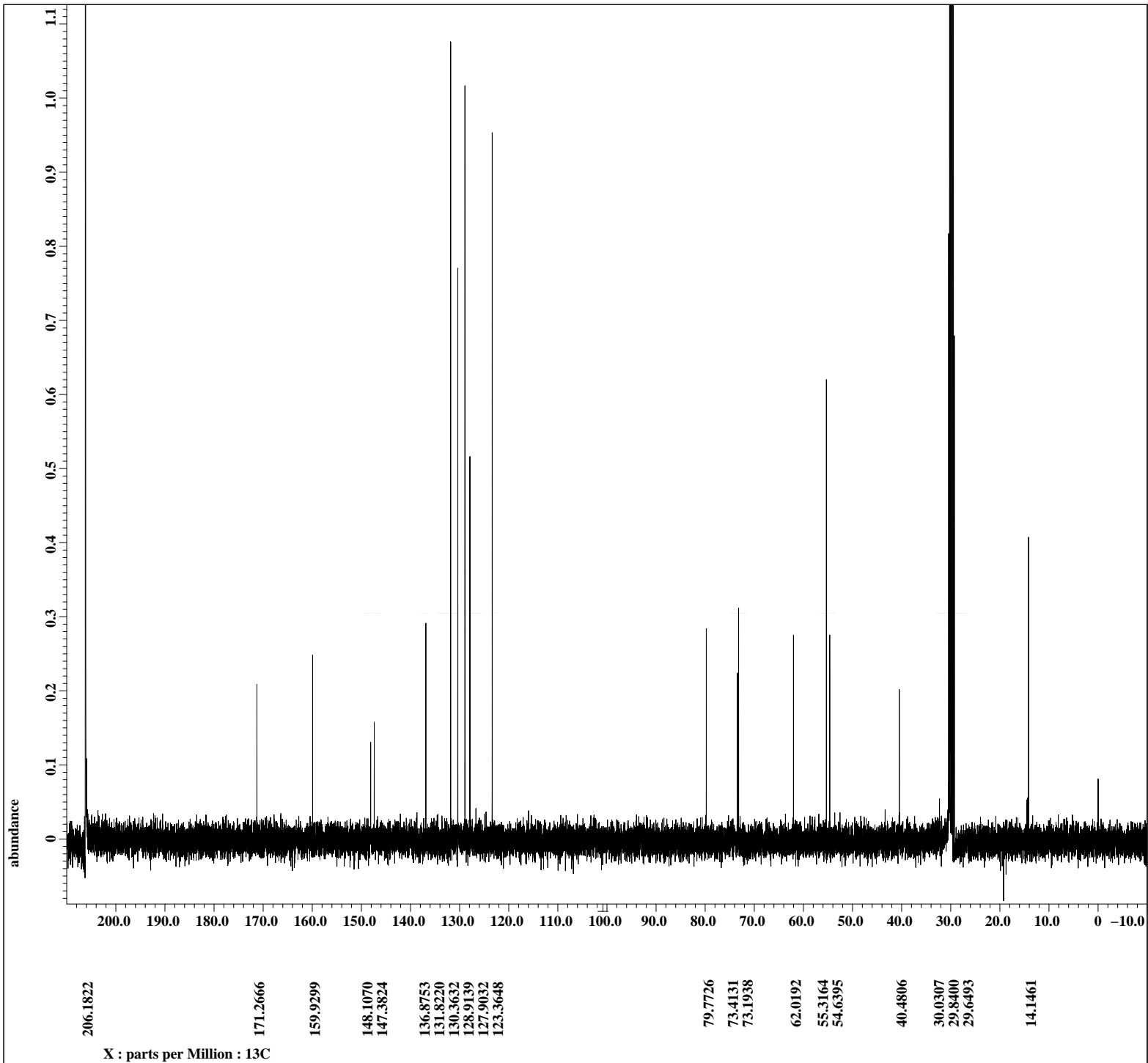
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4

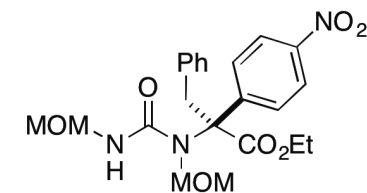


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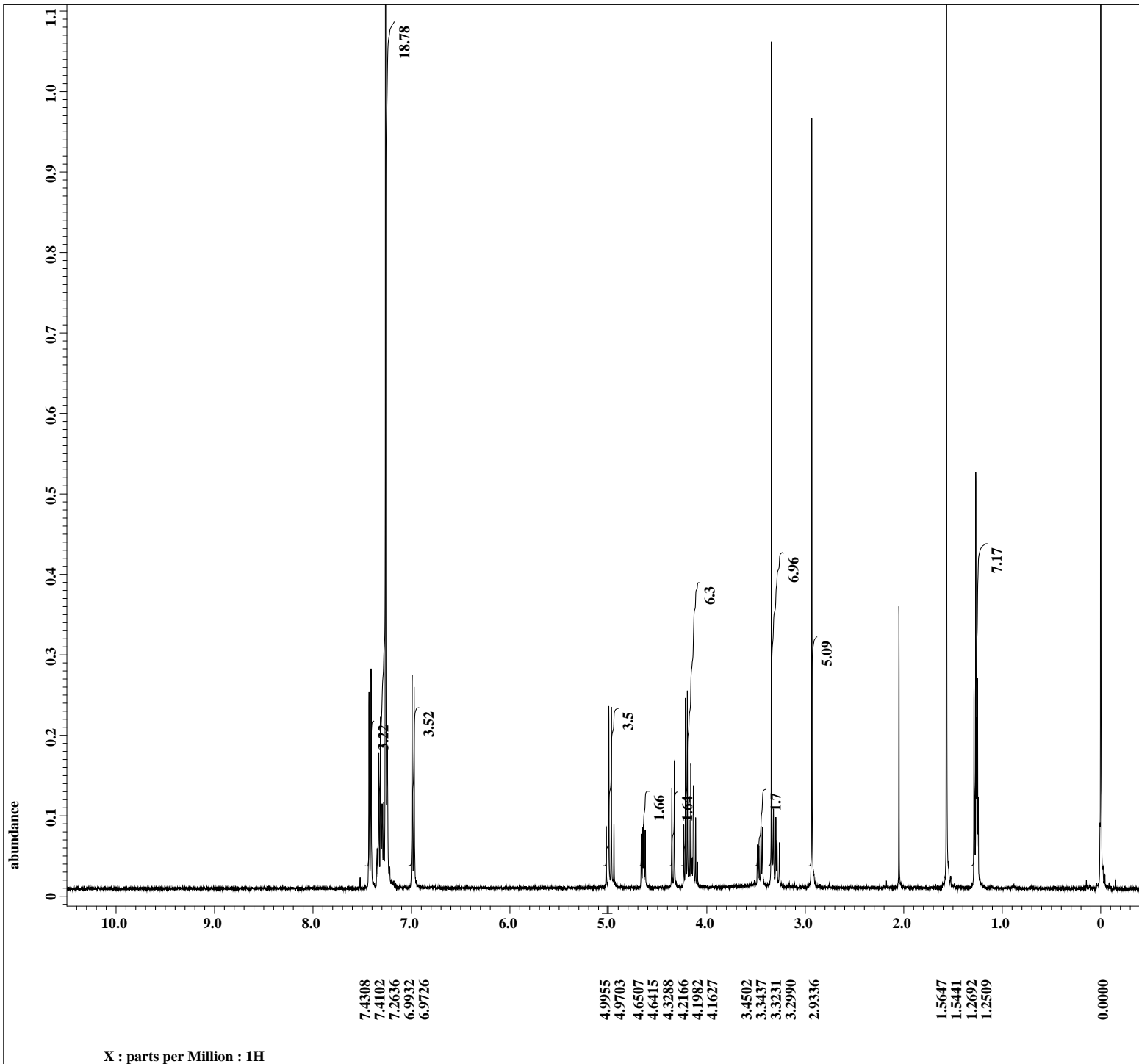
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4

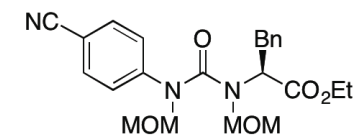


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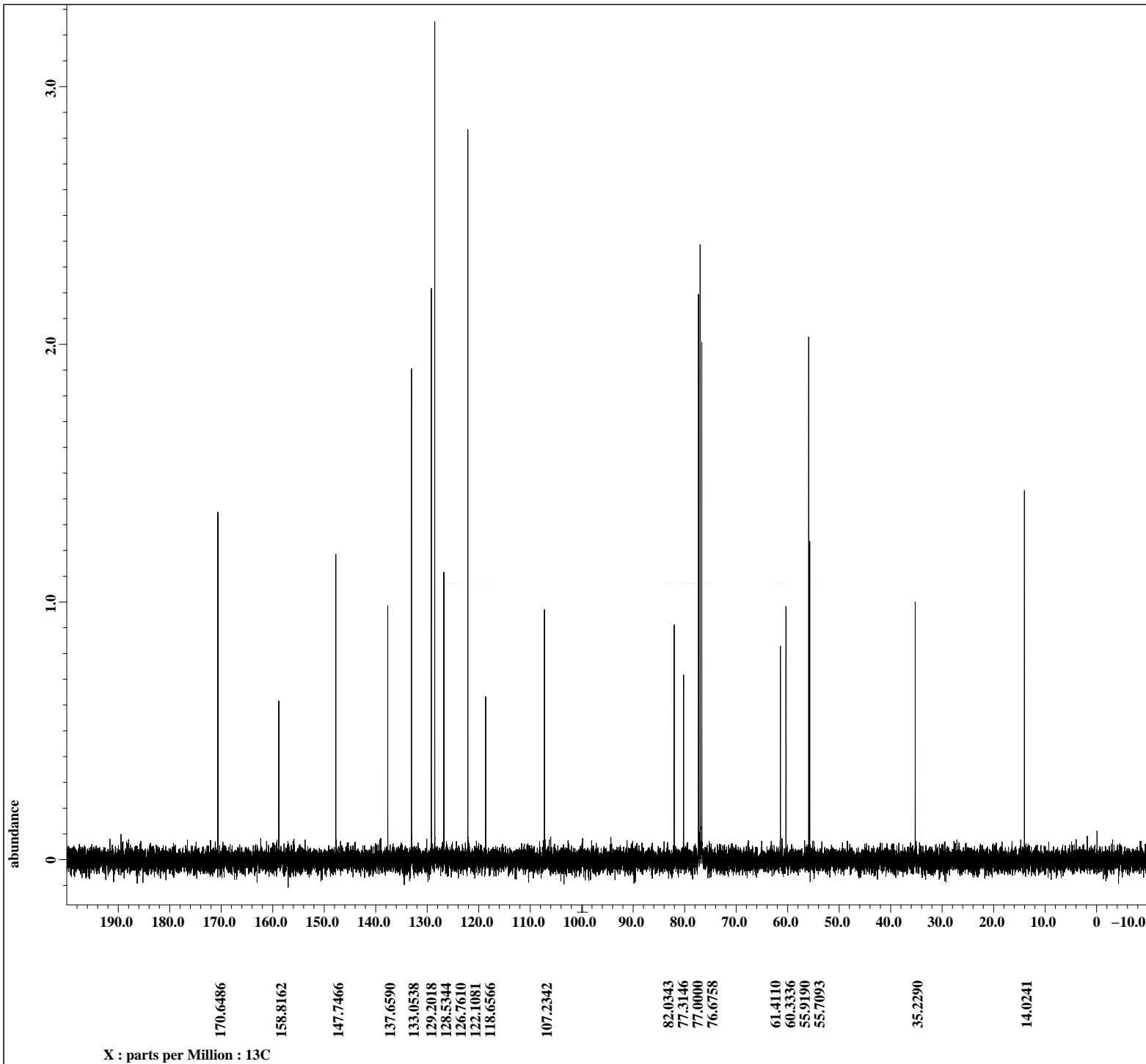
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5

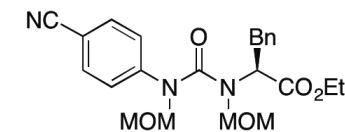


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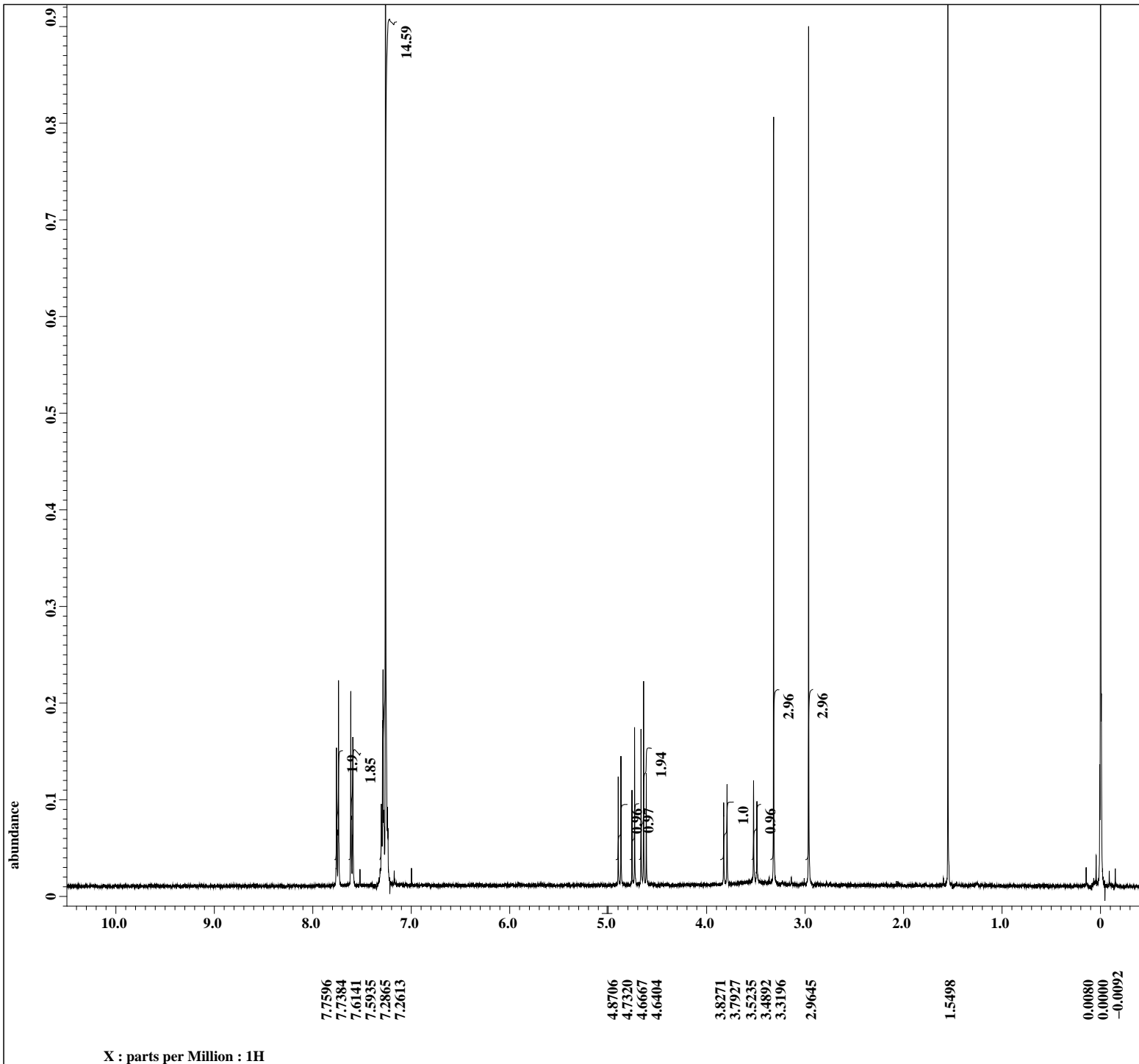
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5

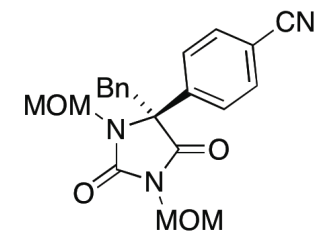


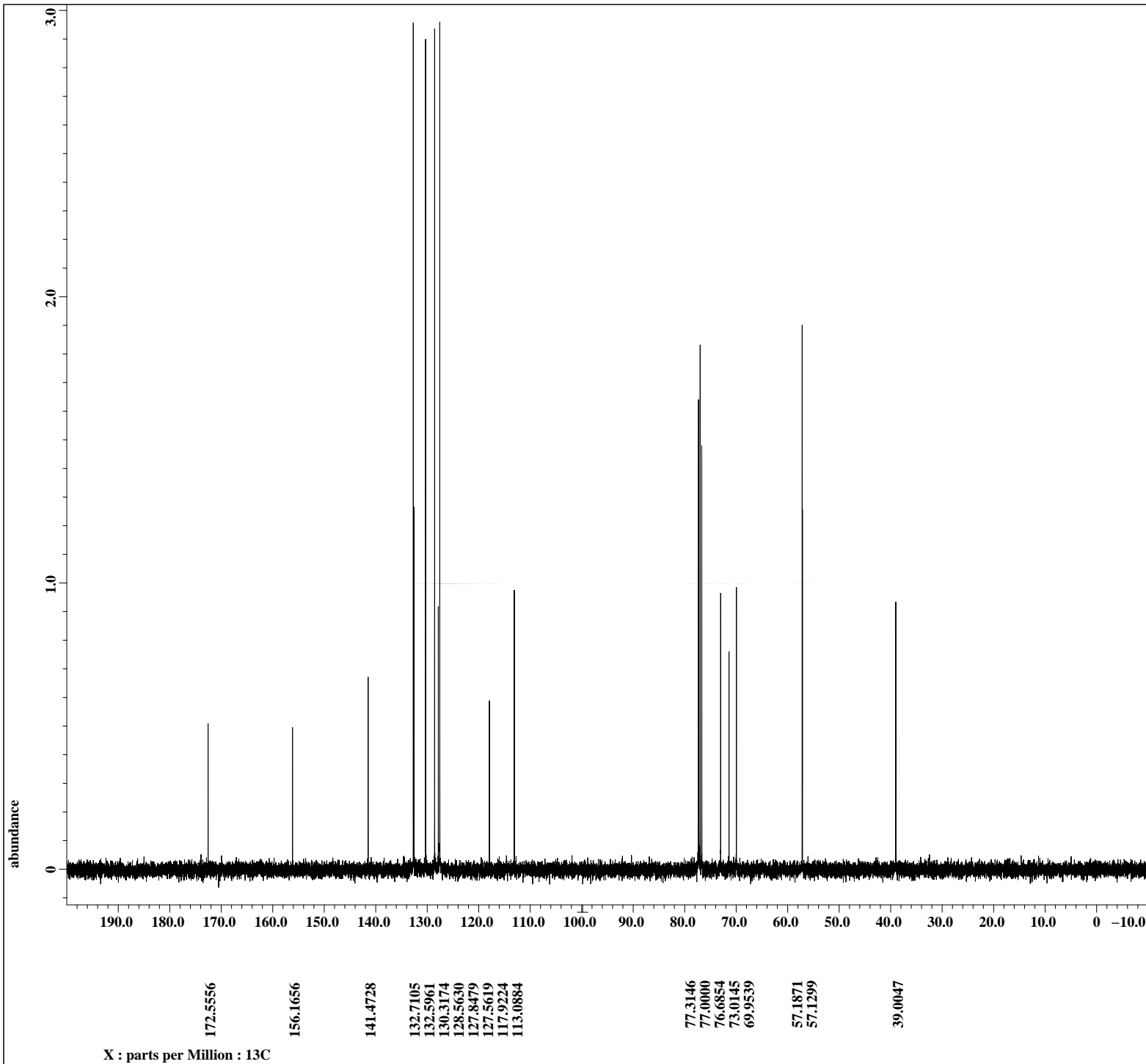
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 Experiment = single_pulse.ex2
 Sample_id = S#23271
 Solvent = CHLOROFORM-D
 Creation_time = 30-DEC-2011 17:31:53
 Revision_time = 16-APR-2013 21:49:41
 Current_time = 16-APR-2013 21:50:39

Comment = 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_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 42
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.7[degC]



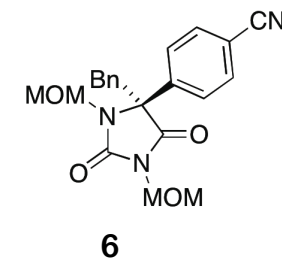


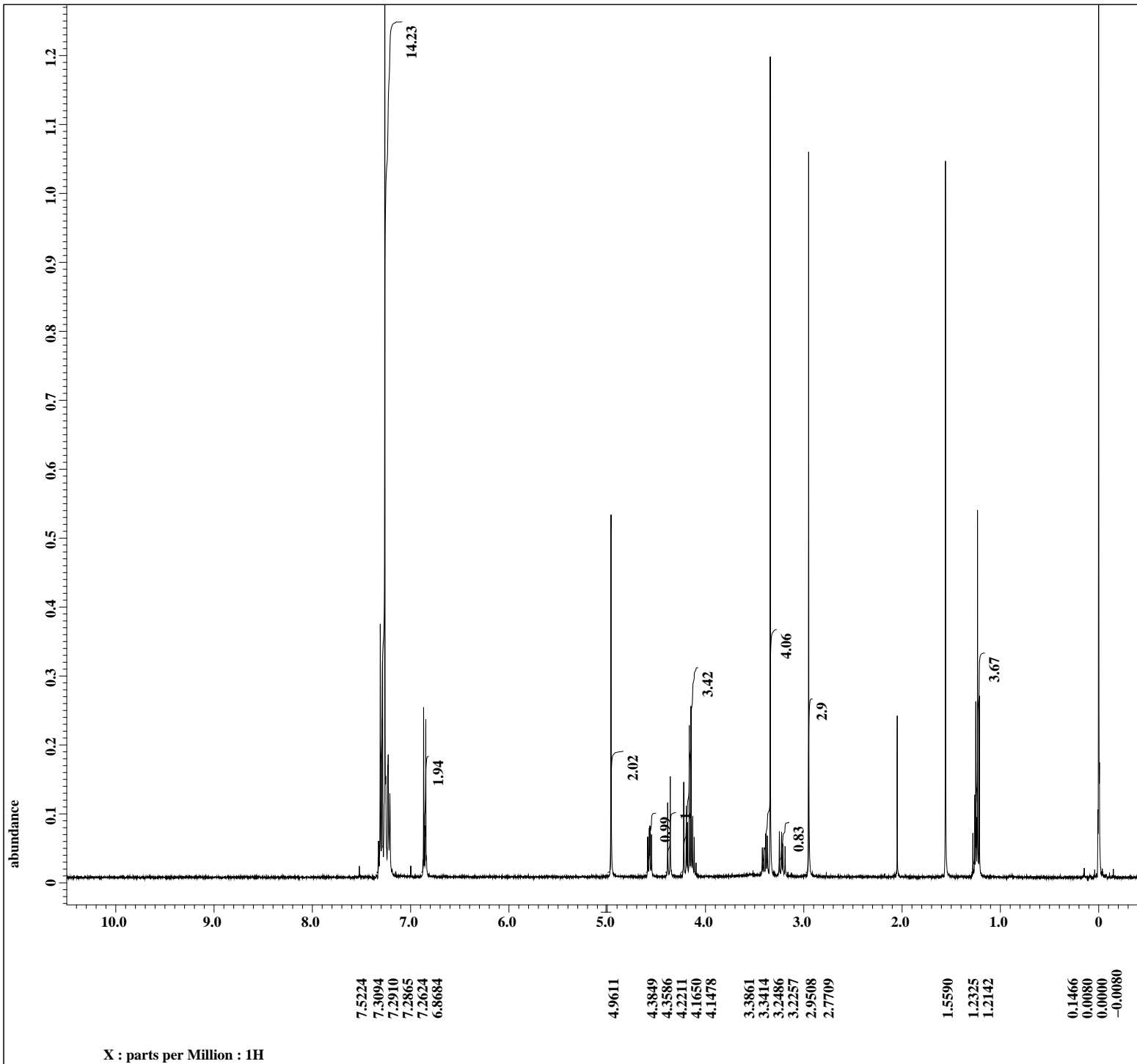
Filename = KT-1731-13C-3-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#33768
 Solvent = CHLOROFORM-D
 Creation_time = 30-DEC-2011 17:56:41
 Revision_time = 16-APR-2013 22:29:53
 Current_time = 16-APR-2013 22:30:38

Comment = 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 = 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 = 130
 Total_scans = 130

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 = 21.5[dB]
 Irr_atn_noe = 21.5[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 = 18.7[dC]





```

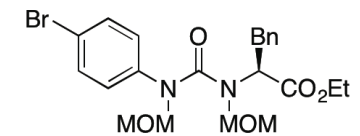
Filename      = KT-1742-product-1-4.j
Author       = delta
Experiment    = single_pulse.ex2
Sample_id    = S#739055
Solvent      = CHLOROFORM-D
Creation_time = 5-JAN-2012 13:24:35
Revision_time = 15-APR-2013 22:33:48
Current_time  = 15-APR-2013 22:34:24

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

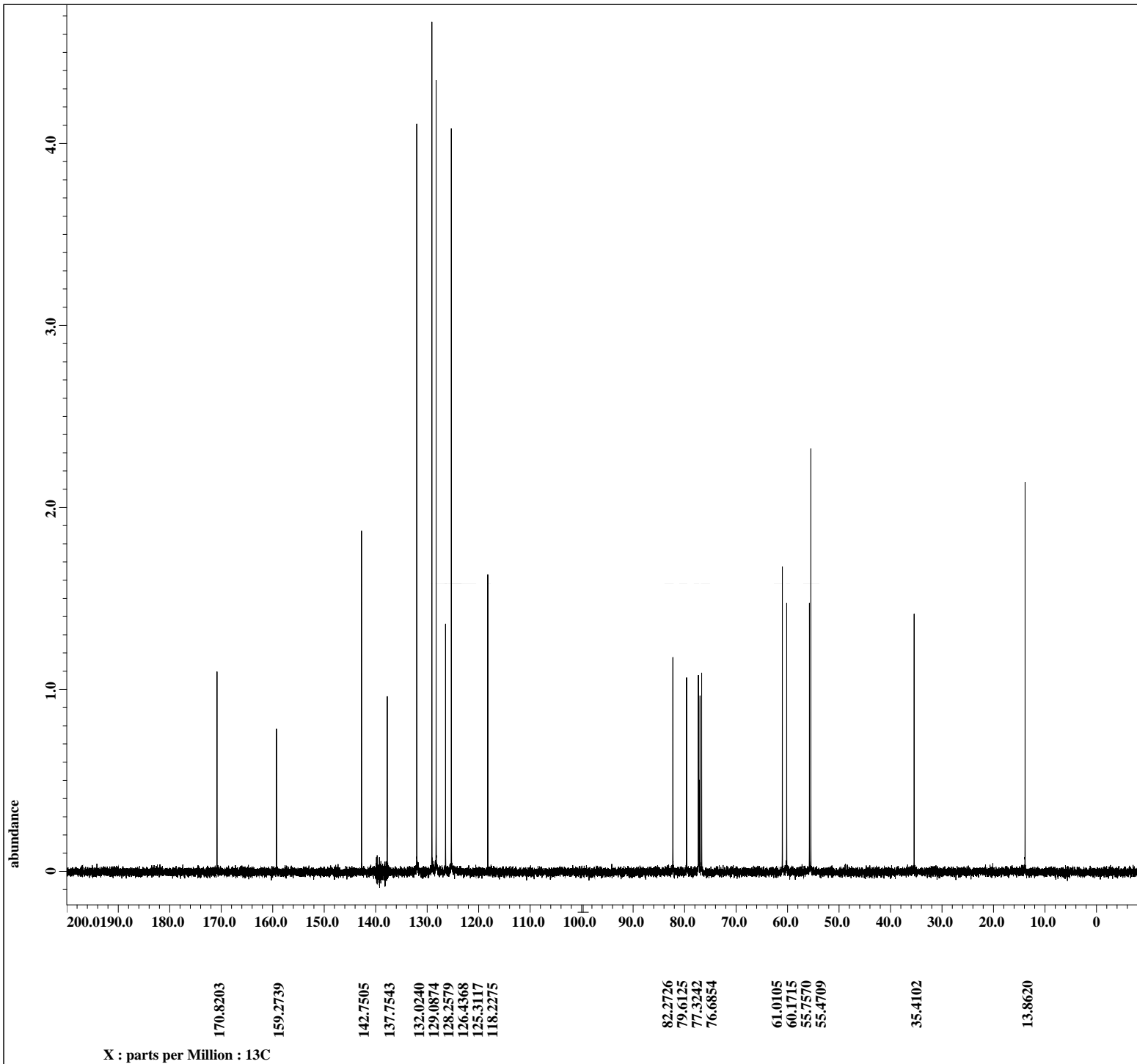
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 2.18365952[s]
X_domain       = 1H
X_freq         = 399.78219838[MHz]
X_offset       = 5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.45794685[Hz]
X_sweep        = 7.5030012[kHz]
Irr_domain     = 1H
Irr_freq       = 399.78219838[MHz]
Irr_offset     = 5[ppm]
Tri_domain     = 1H
Tri_freq       = 399.78219838[MHz]
Tri_offset     = 5[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 8
Total_scans    = 8

X_90_width    = 10[us]
X_acq_time     = 2.18365952[s]
X_angle        = 45[deg]
X_atn          = 0.3[dB]
X_pulse        = 5[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       = 18.5[dc]

```



7

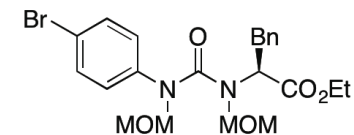


Filename = KT-1742-13C-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#748960
 Solvent = CHLOROFORM-D
 Creation_time = 17-JAN-2012 13:44:32
 Revision_time = 15-APR-2013 22:34:55
 Current_time = 15-APR-2013 22:35:10

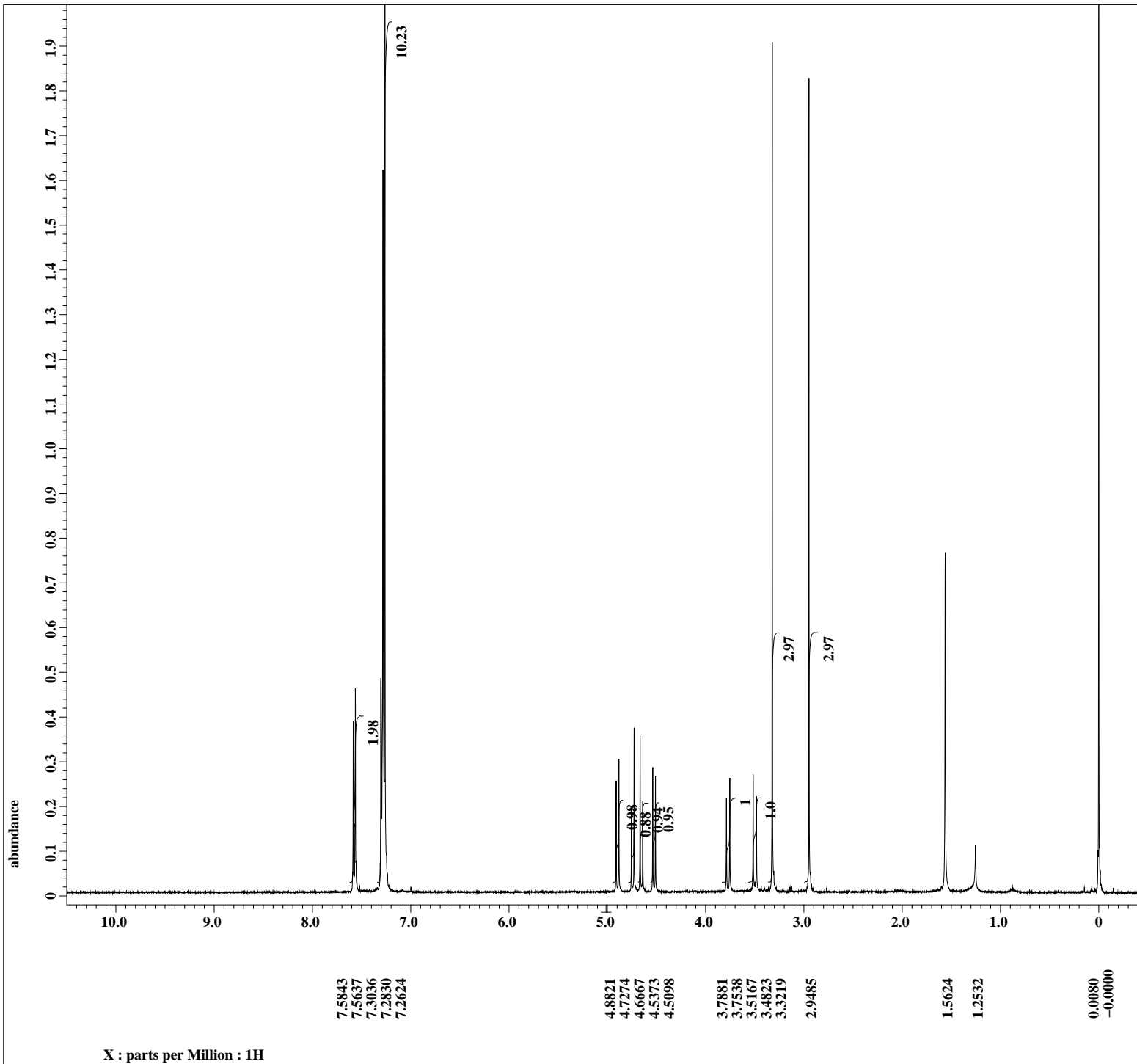
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 66
 Total_scans = 66

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 = 21.5[dB]
 Irr_atn_noe = 21.5[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 54
 Relaxation_delay = 2[s]
 Repetition_time = 3.04333312[s]
 Temp_get = 19.1[dC]



7

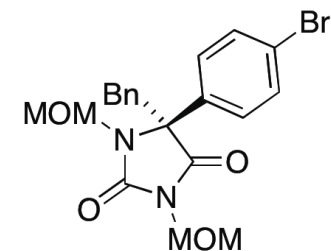


Filename = KT-1745-product-1-2-4
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#31923
 Solvent = CHLOROFORM-D
 Creation_time = 8-JAN-2012 17:46:09
 Revision_time = 17-APR-2013 09:53:00
 Current_time = 17-APR-2013 09:53:37

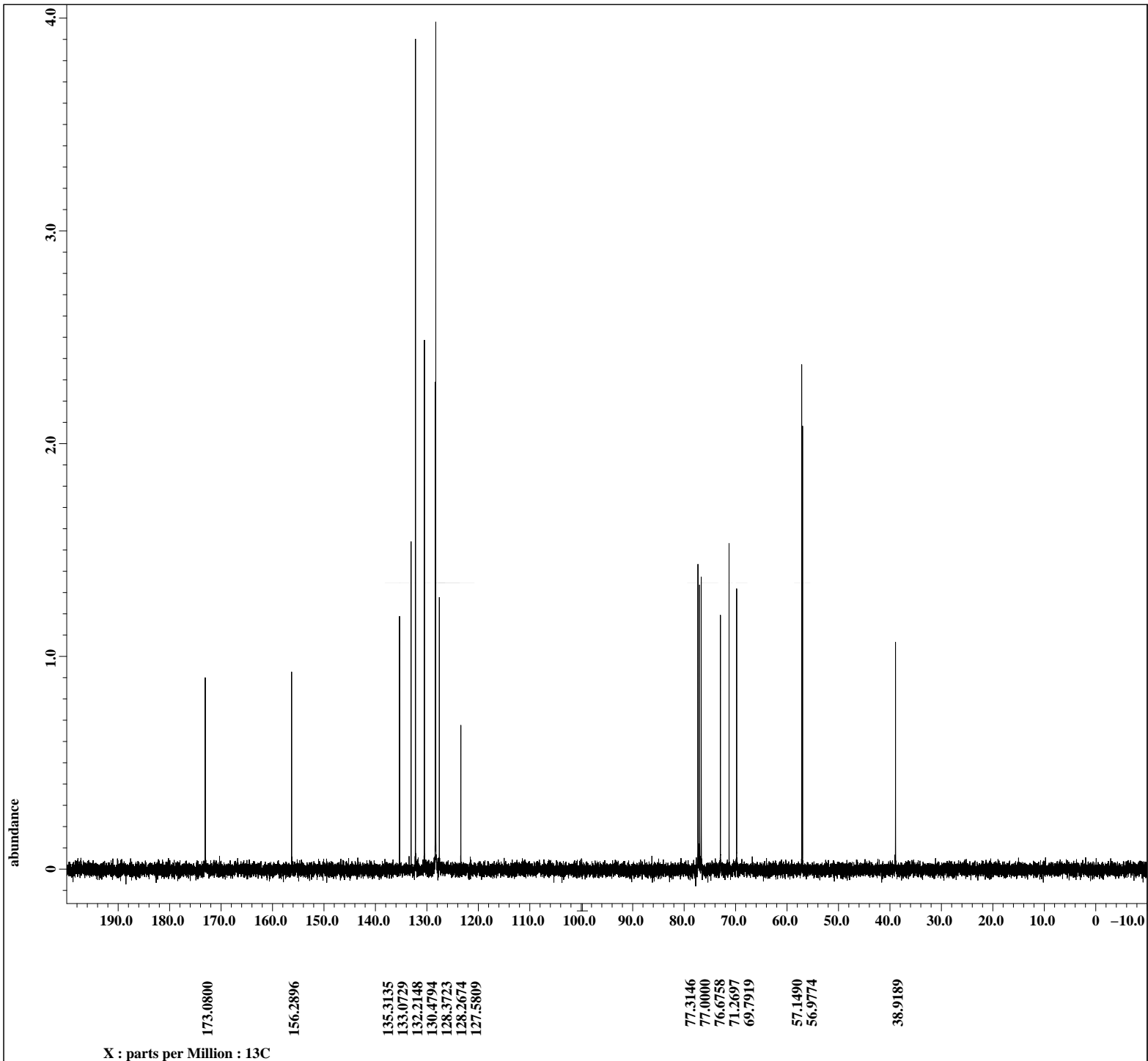
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[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 = 19.2[dC]



8

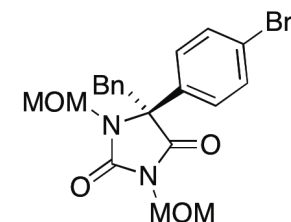


Filename = KT-1745-13C-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#39373
 Solvent = CHLOROFORM-D
 Creation_time = 8-JAN-2012 18:03:00
 Revision_time = 17-APR-2013 09:54:12
 Current_time = 17-APR-2013 09:55:02

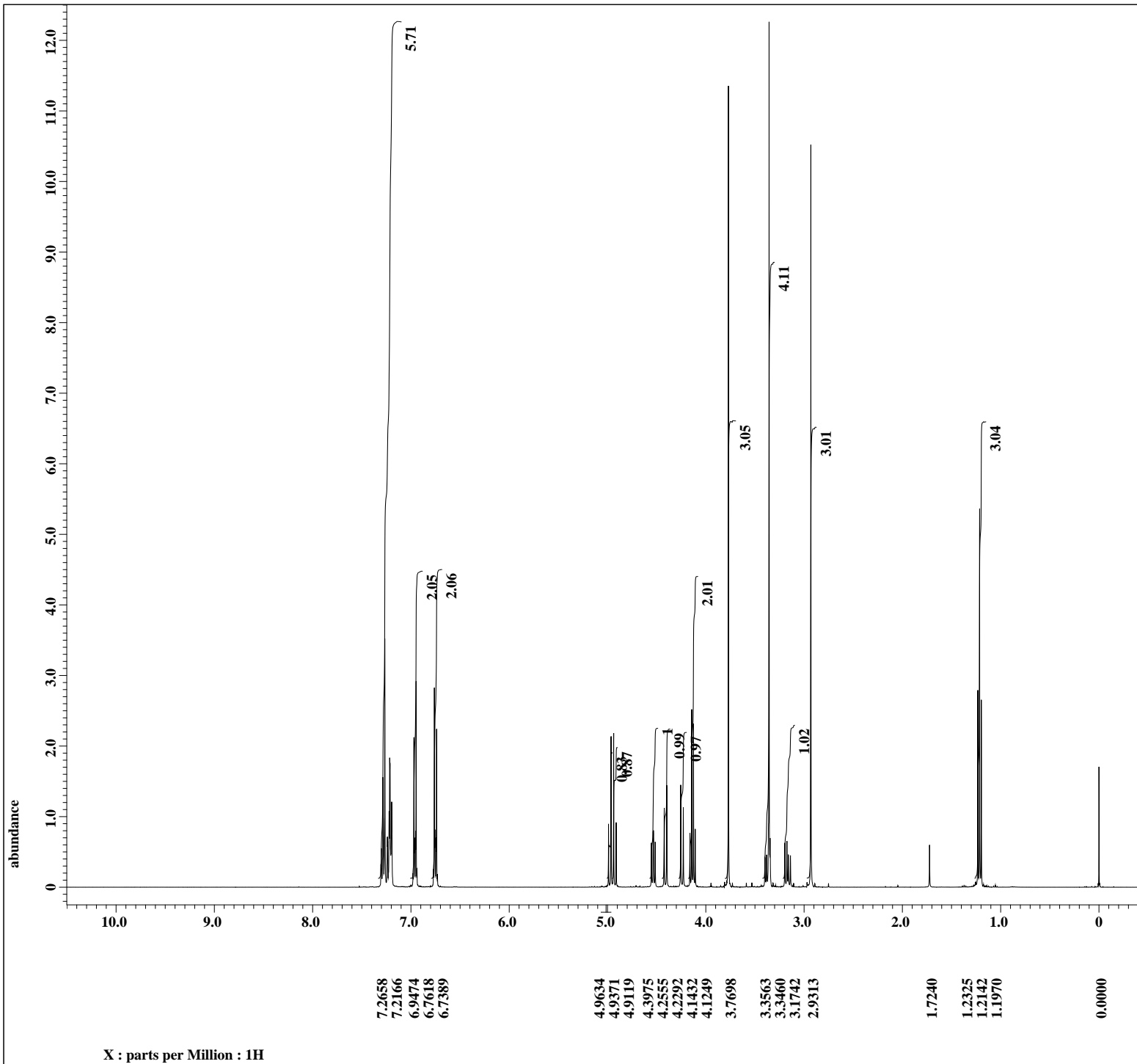
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 70
 Total_scans = 70

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 = 21.5[dB]
 Irr_atn_noe = 21.5[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 = 19.4[dC]



8

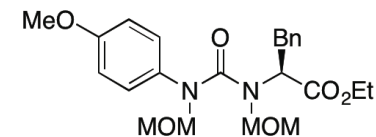


Filename = TY-12-090 H-2-3.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#661184
 Solvent = CHLOROFORM-D
 Creation_time = 6-APR-2013 10:56:09
 Revision_time = 15-APR-2013 22:38:00
 Current_time = 15-APR-2013 22:38:24

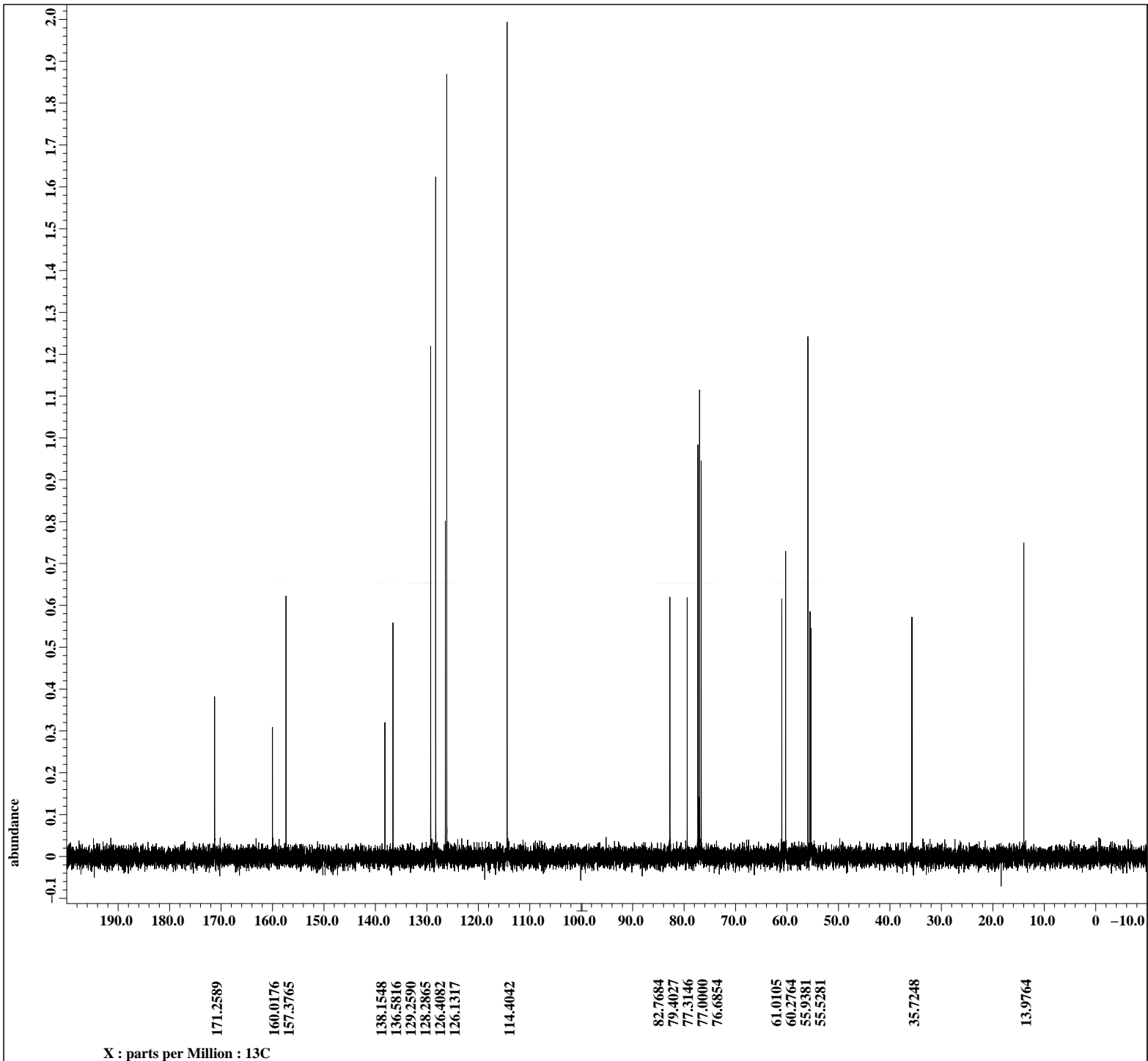
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10.75[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5.375[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 34
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.5[dC]



9

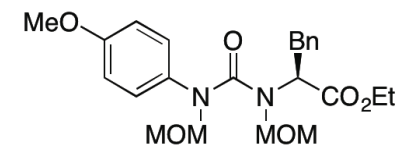


Filename = TY-12-090 13C comp-3.
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = 090
 Solvent = CHLOROFORM-D
 Creation_time = 6-APR-2013 11:03:02
 Revision_time = 15-APR-2013 22:38:56
 Current_time = 15-APR-2013 22:39:06

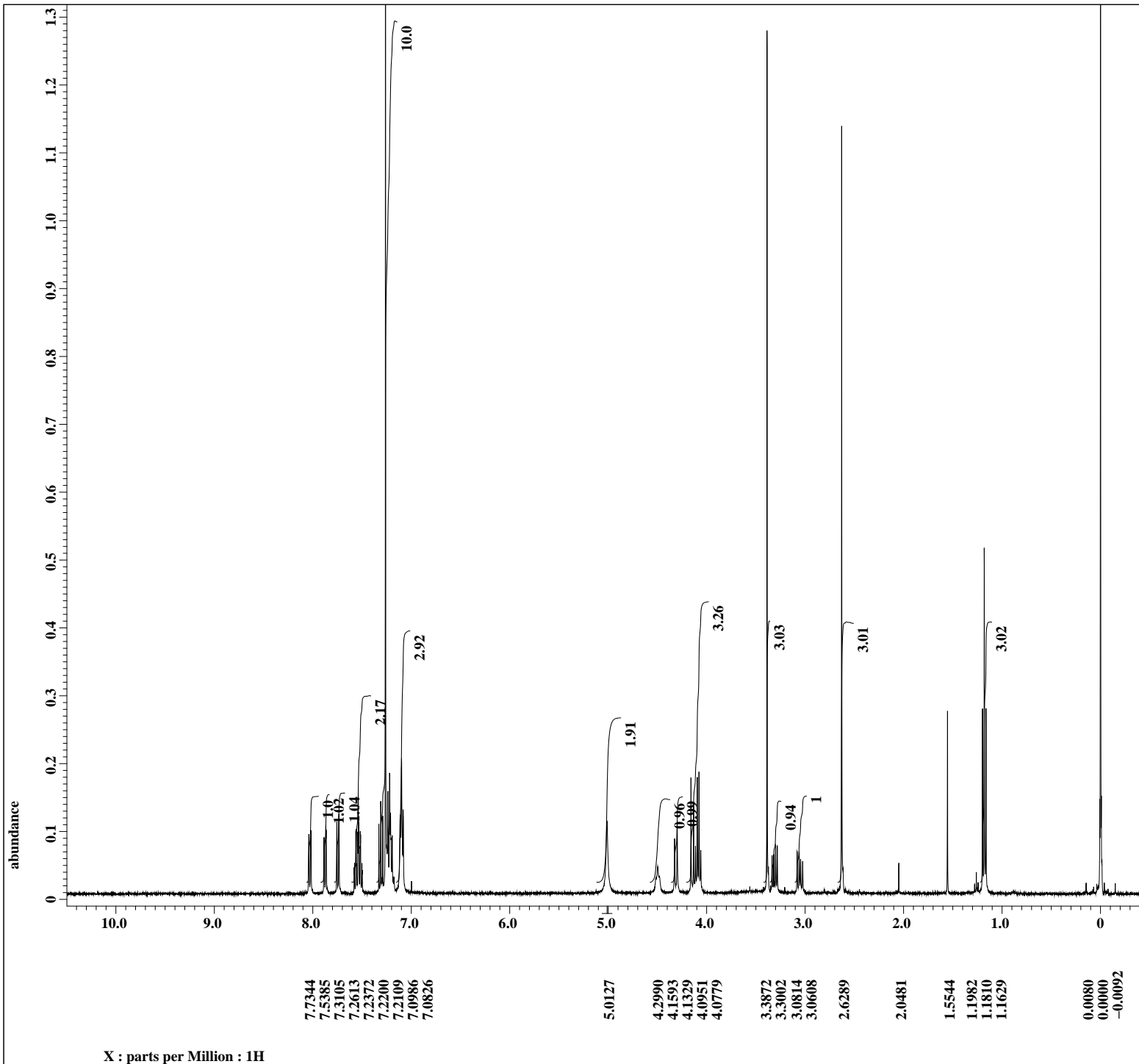
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 64
 Total_scans = 64

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 = 20.8[dB]
 Irr_atn_noe = 20.8[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 54
 Relaxation_delay = 2[s]
 Repetition_time = 3.04333312[s]
 Temp_get = 18.9[dC]



9

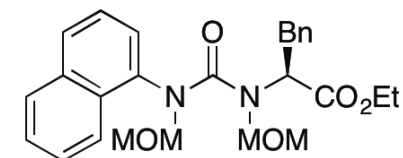


Filename = KT-1729-PTLC-1-up-3.j
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#29838
 Solvent = CHLOROFORM-D
 Creation_time = 25-DEC-2011 17:43:01
 Revision_time = 16-APR-2013 21:10:54
 Current_time = 16-APR-2013 21:11:48

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

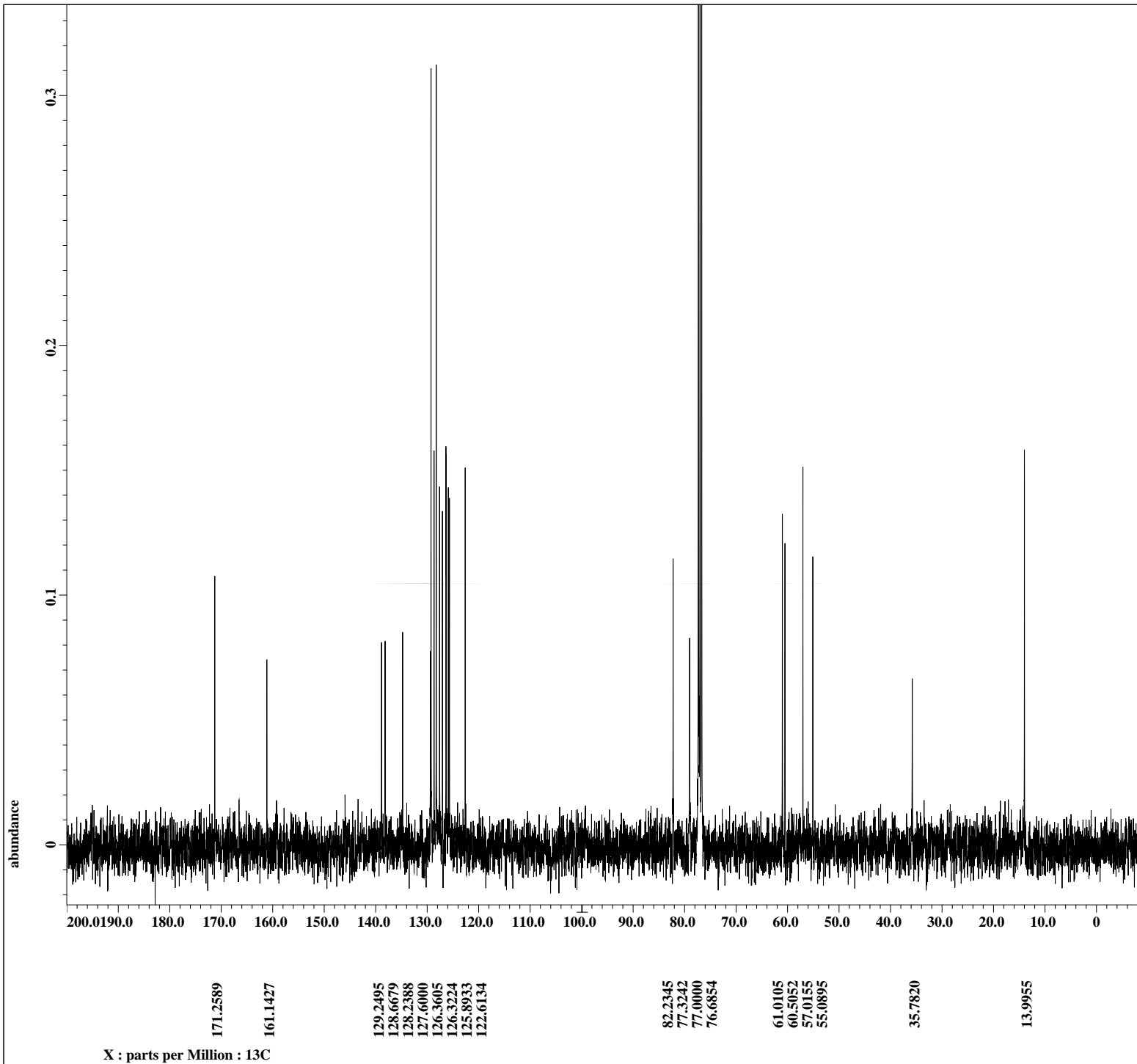
Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 399.78219838[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.45794685[Hz]
 X_sweep = 7.5030012[kHz]
 Irr_domain = 1H
 Irr_freq = 399.78219838[MHz]
 Irr_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[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 = 18.8[degC]



11

X : parts per Million : 1H

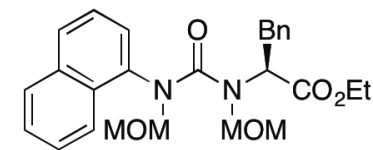


Filename = KT-1729-13C-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#852748
 Solvent = CHLOROFORM-D
 Creation_time = 16-JAN-2012 16:40:56
 Revision_time = 16-APR-2013 21:13:13
 Current_time = 16-APR-2013 21:14:27

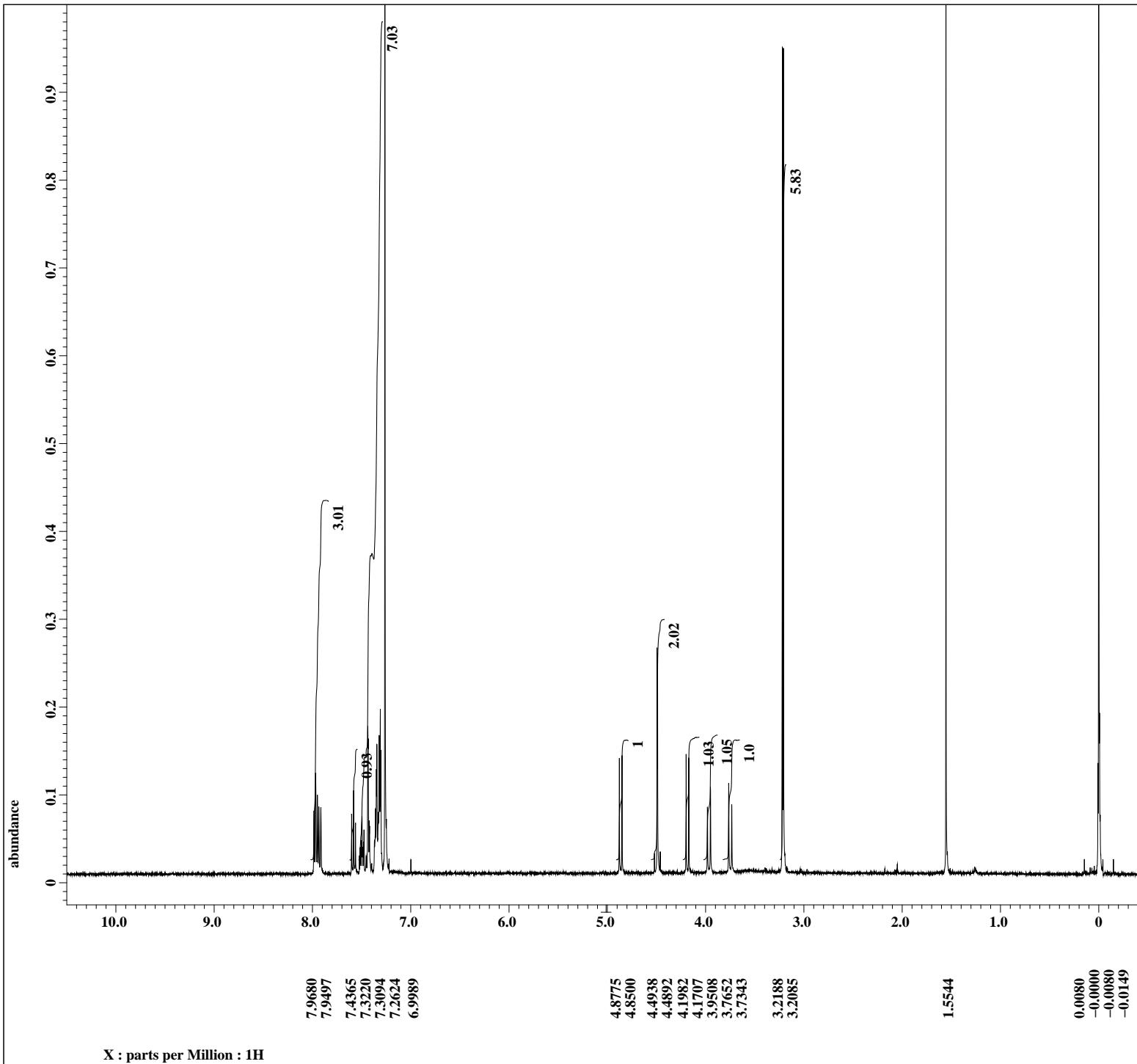
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 130
 Total_scans = 130

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 = 21.5[dB]
 Irr_atn_noe = 21.5[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 = 19.3[dC]



11

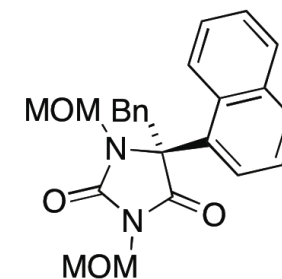


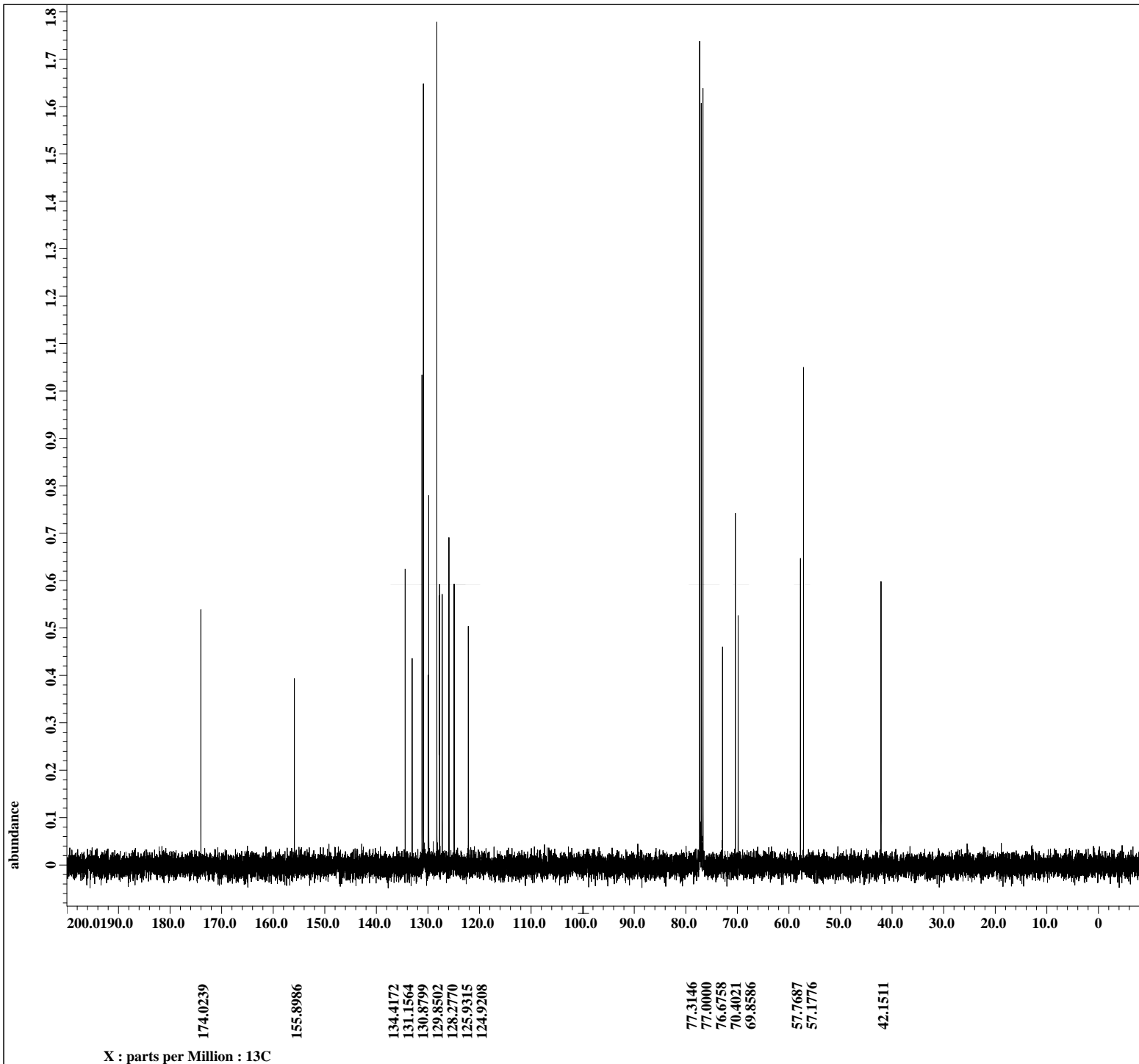
Filename = KT-1733-final-3.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#528016
 Solvent = CHLOROFORM-D
 Creation_time = 4-JAN-2012 07:33:52
 Revision_time = 17-APR-2013 10:10:18
 Current_time = 17-APR-2013 10:10:48

Comment = 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_domain = 1H
 X_freq = 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[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 42
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.1[dC]



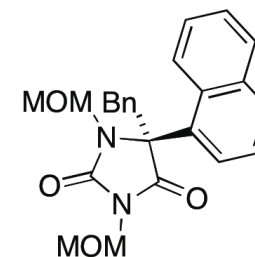


Filename = KT-1733-13C-final-3.j
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#536212
 Solvent = CHLOROFORM-D
 Creation_time = 4-JAN-2012 07:54:30
 Revision_time = 17-APR-2013 10:11:21
 Current_time = 17-APR-2013 10:12:25

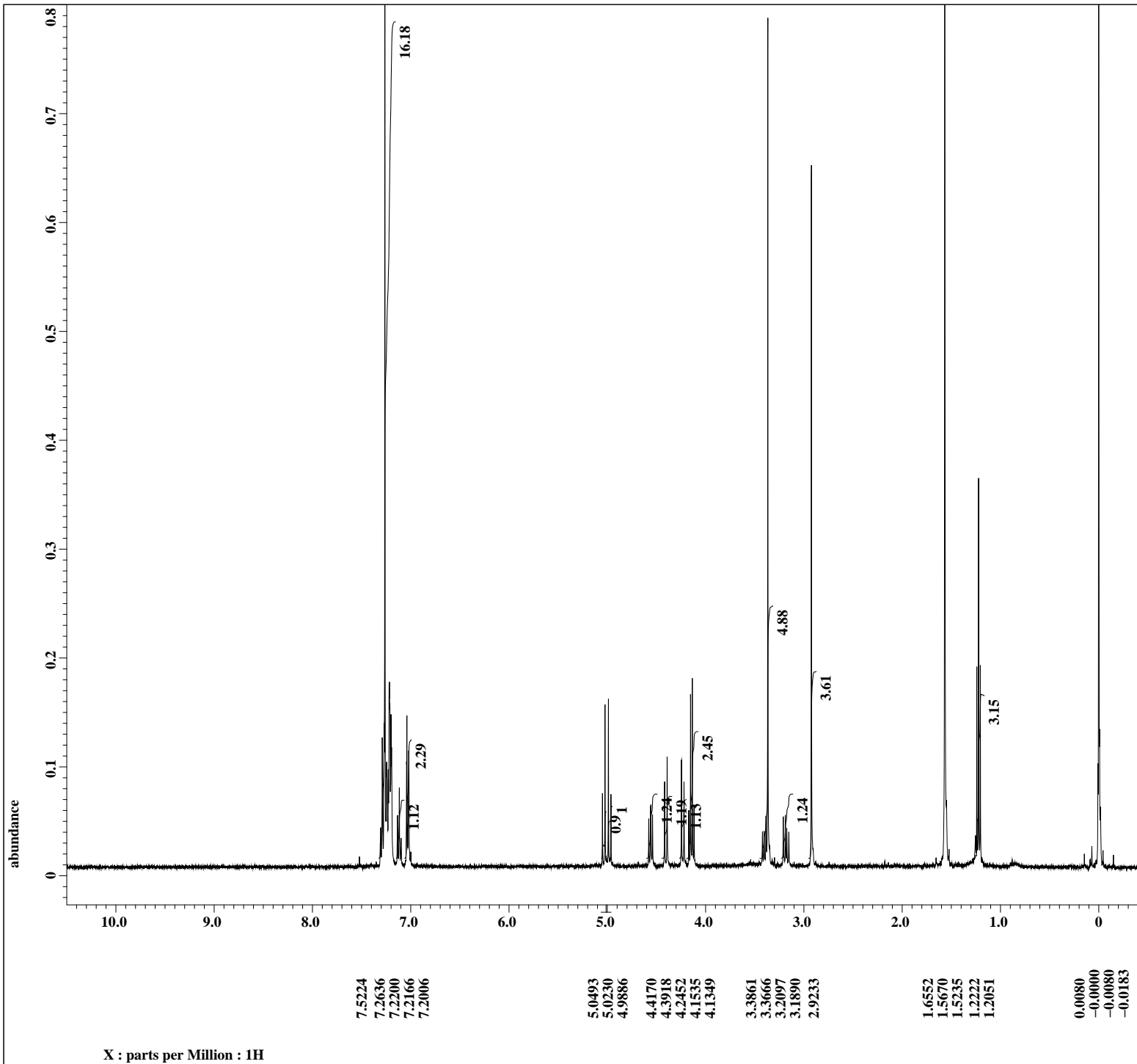
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 142
 Total_scans = 142

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 = 21.5[dB]
 Irr_atn_noe = 21.5[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 = 18.2[dC]



12

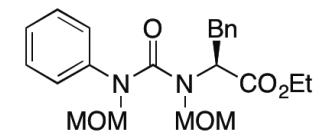


Filename = KT-1615-product-1-3.j
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#515380
 Solvent = CHLOROFORM-D
 Creation_time = 7-OCT-2011 07:25:09
 Revision_time = 15-APR-2013 22:21:09
 Current_time = 15-APR-2013 22:22:00

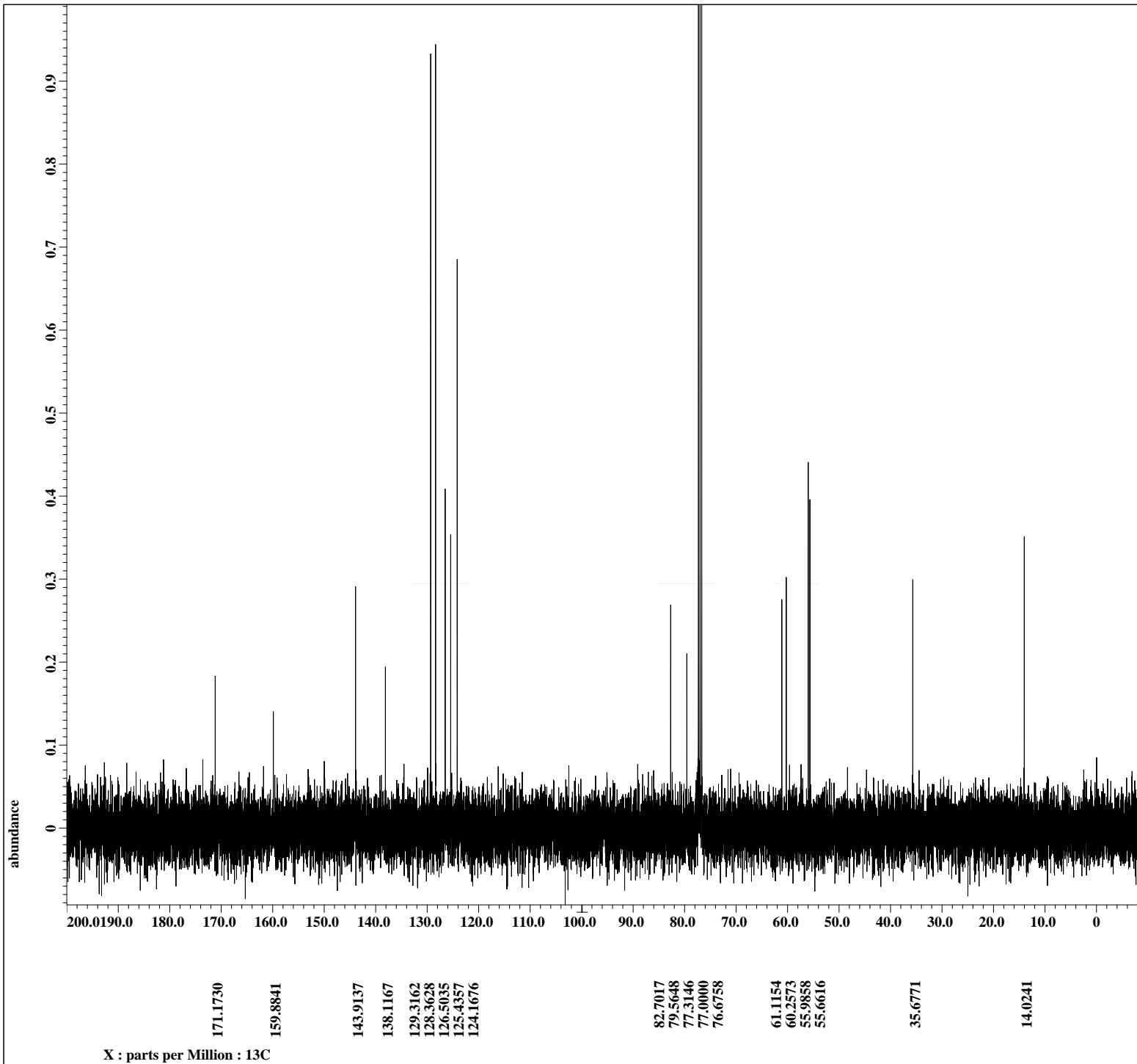
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[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 = 19.1[dC]



13

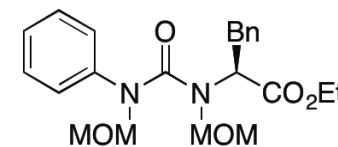


Filename = KT-1615-product-1-13C
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#539465
 Solvent = CHLOROFORM-D
 Creation_time = 7-OCT-2011 08:07:19
 Revision_time = 15-APR-2013 22:23:01
 Current_time = 15-APR-2013 22:23:42

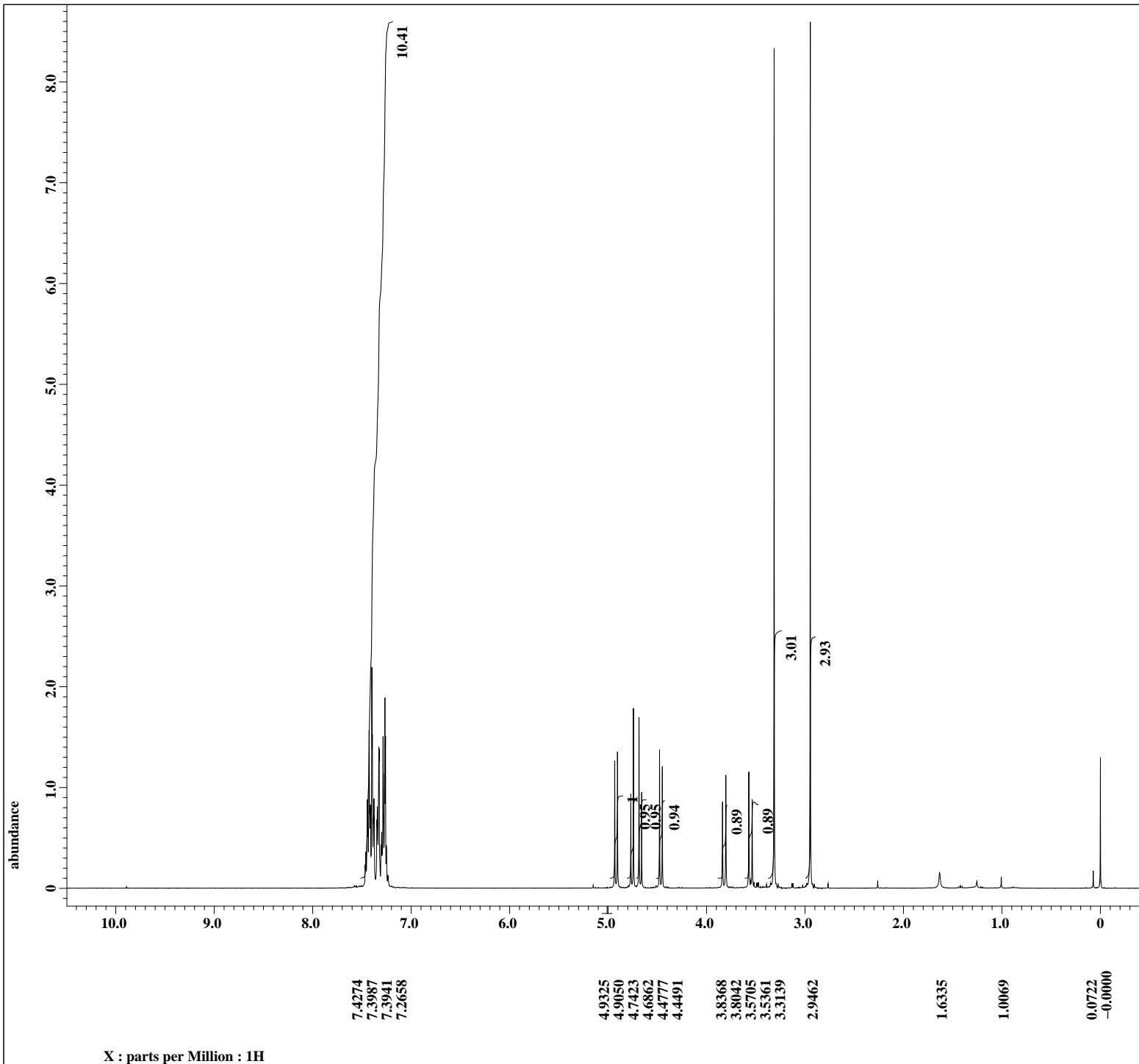
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 64
 Total_scans = 64

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 = 21.5[dB]
 Irr_atn_noe = 21.5[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 = 19.4[dC]



13

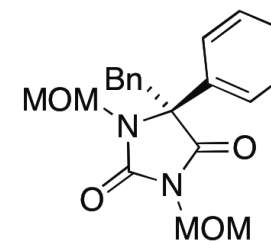


Filename = MOM-MOM-kankatai-3.jd
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#667085
 Solvent = CHLOROFORM-D
 Creation_time = 26-MAR-2013 11:06:24
 Revision_time = 17-APR-2013 14:26:41
 Current_time = 17-APR-2013 14:27:36

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

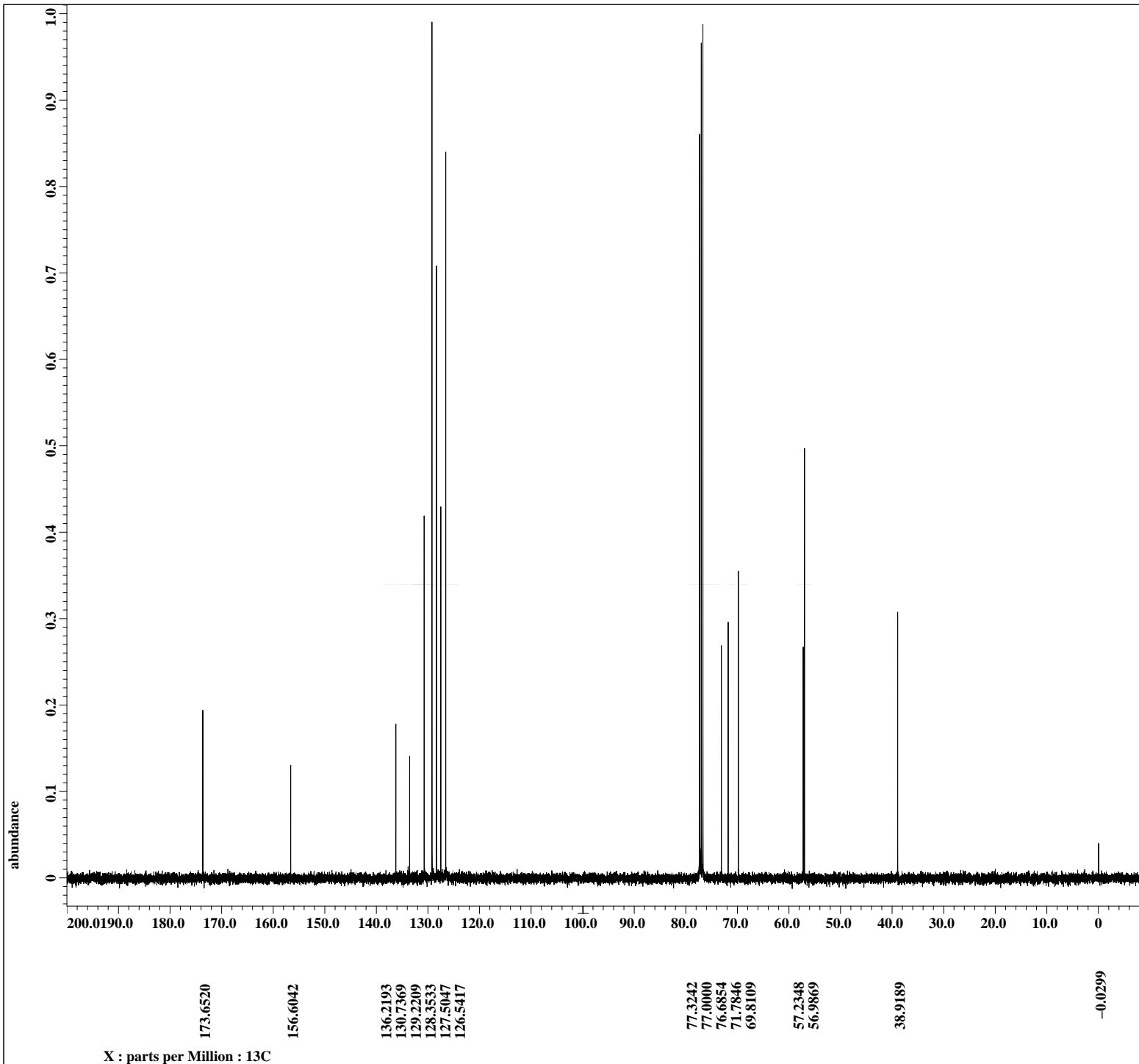
Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 399.78219838[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.45794685[Hz]
 X_sweep = 7.5030012[kHz]
 Irr_domain = 1H
 Irr_freq = 399.78219838[MHz]
 Irr_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

X_90_width = 10.75[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5.375[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 34
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.5[dC]



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X : parts per Million : 1H

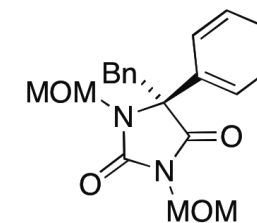


Filename = MOM-MOM-kanka-13C-3.j
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = a
 Solvent = CHLOROFORM-D
 Creation_time = 26-MAR-2013 12:18:07
 Revision_time = 17-APR-2013 14:28:20
 Current_time = 17-APR-2013 14:29:51

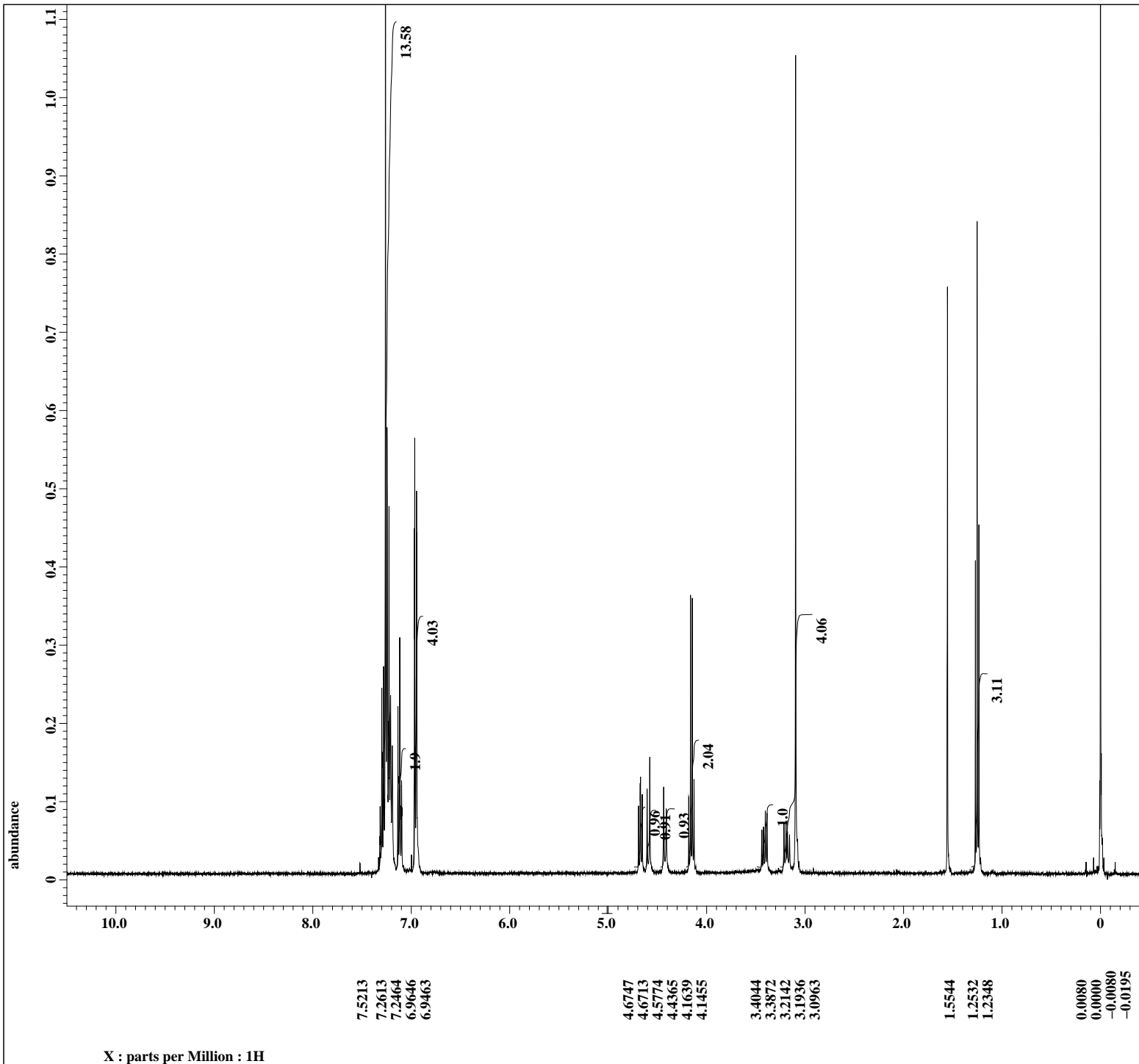
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 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 = 20.8[dB]
 Irr_atn_noe = 20.8[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 54
 Relaxation_delay = 2[s]
 Repetition_time = 3.04333312[s]
 Temp_get = 19.4[dC]



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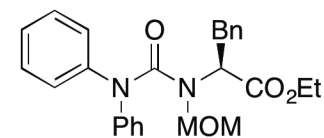


Filename = KT-1644-20120131-3.jd
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#750541
 Solvent = CHLOROFORM-D
 Creation_time = 31-JAN-2012 13:42:55
 Revision_time = 16-APR-2013 21:37:03
 Current_time = 16-APR-2013 21:38:12

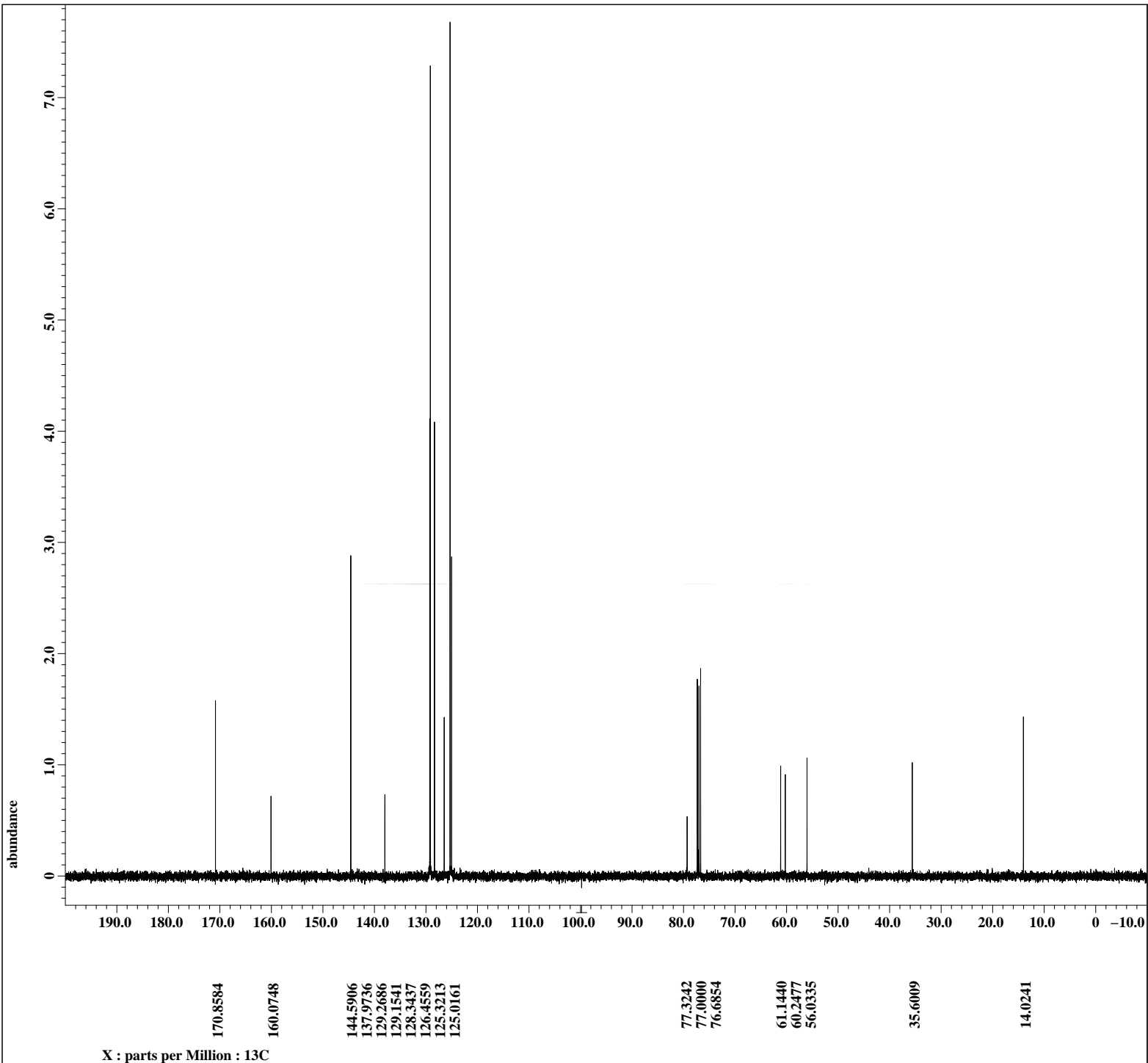
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[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 = 19.2[dC]



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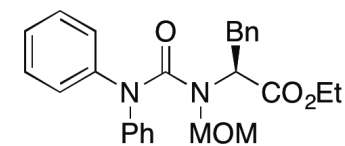


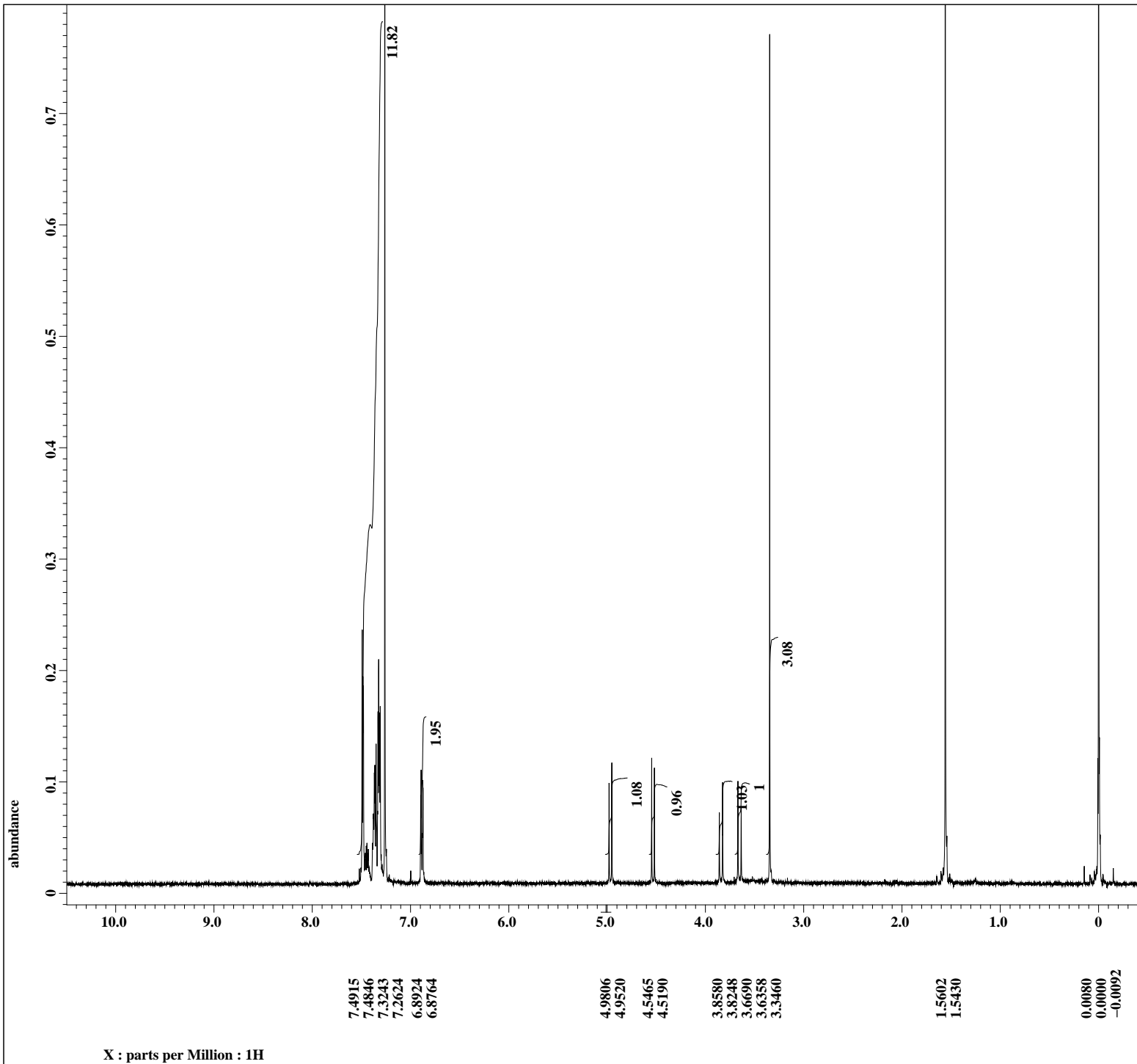
Filename = KT-1644-13C-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#810351
 Solvent = CHLOROFORM-D
 Creation_time = 31-JAN-2012 15:27:04
 Revision_time = 16-APR-2013 21:38:56
 Current_time = 16-APR-2013 21:40:02

Comment = 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 = 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 = 66
 Total_scans = 66

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 = 21.5[dB]
 Irr_atn_noe = 21.5[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 = 19.3[dC]





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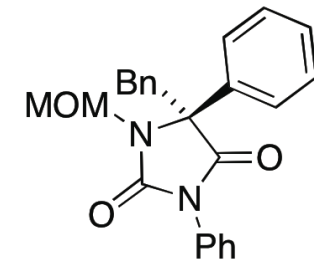
Filename      = KT-1651-again-2-6.jdf
Author       = delta
Experiment   = single_pulse.ex2
Sample_id    = S#555683
Solvent      = CHLOROFORM-D
Creation_time = 2-NOV-2011 08:24:08
Revision_time = 17-APR-2013 11:42:06
Current_time  = 17-APR-2013 11:42:38

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

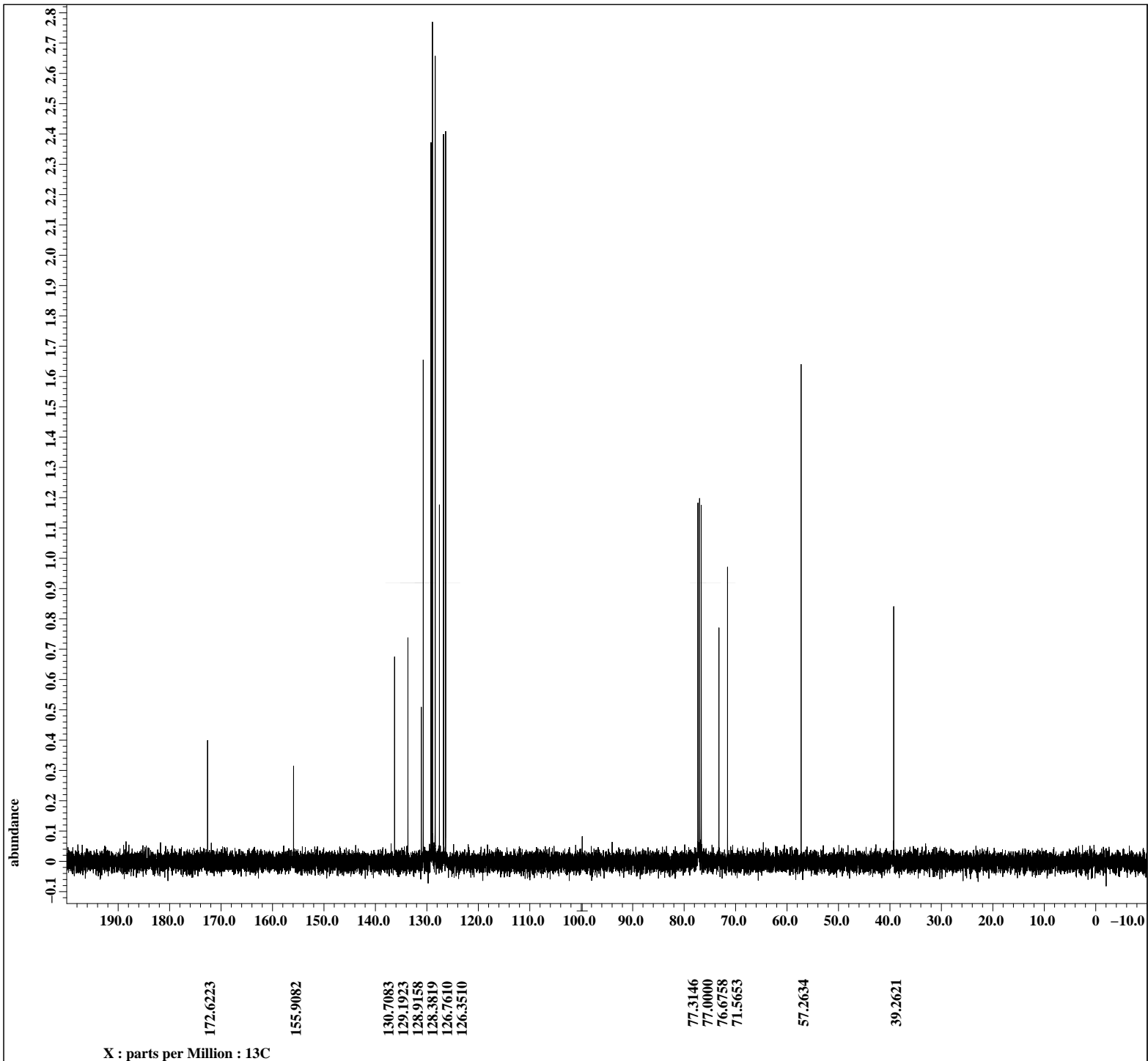
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 2.18365952[s]
X_domain       = 1H
X_freq         = 399.78219838[MHz]
X_offset       = 5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.45794685[Hz]
X_sweep        = 7.5030012[kHz]
Irr_domain     = 1H
Irr_freq       = 399.78219838[MHz]
Irr_offset     = 5[ppm]
Tri_domain     = 1H
Tri_freq       = 399.78219838[MHz]
Tri_offset     = 5[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 8
Total_scans    = 8

X_90_width    = 10[us]
X_acq_time     = 2.18365952[s]
X_angle        = 45[deg]
X_atn          = 0.3[dB]
X_pulse        = 5[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       = 18.6[dC]

```



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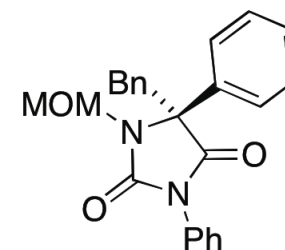
Filename      = KT-1651-13C-3.jdf
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#569470
Solvent      = CHLOROFORM-D
Creation_time = 2-NOV-2011 08:51:24
Revision_time = 17-APR-2013 11:43:08
Current_time  = 17-APR-2013 11:43:53

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

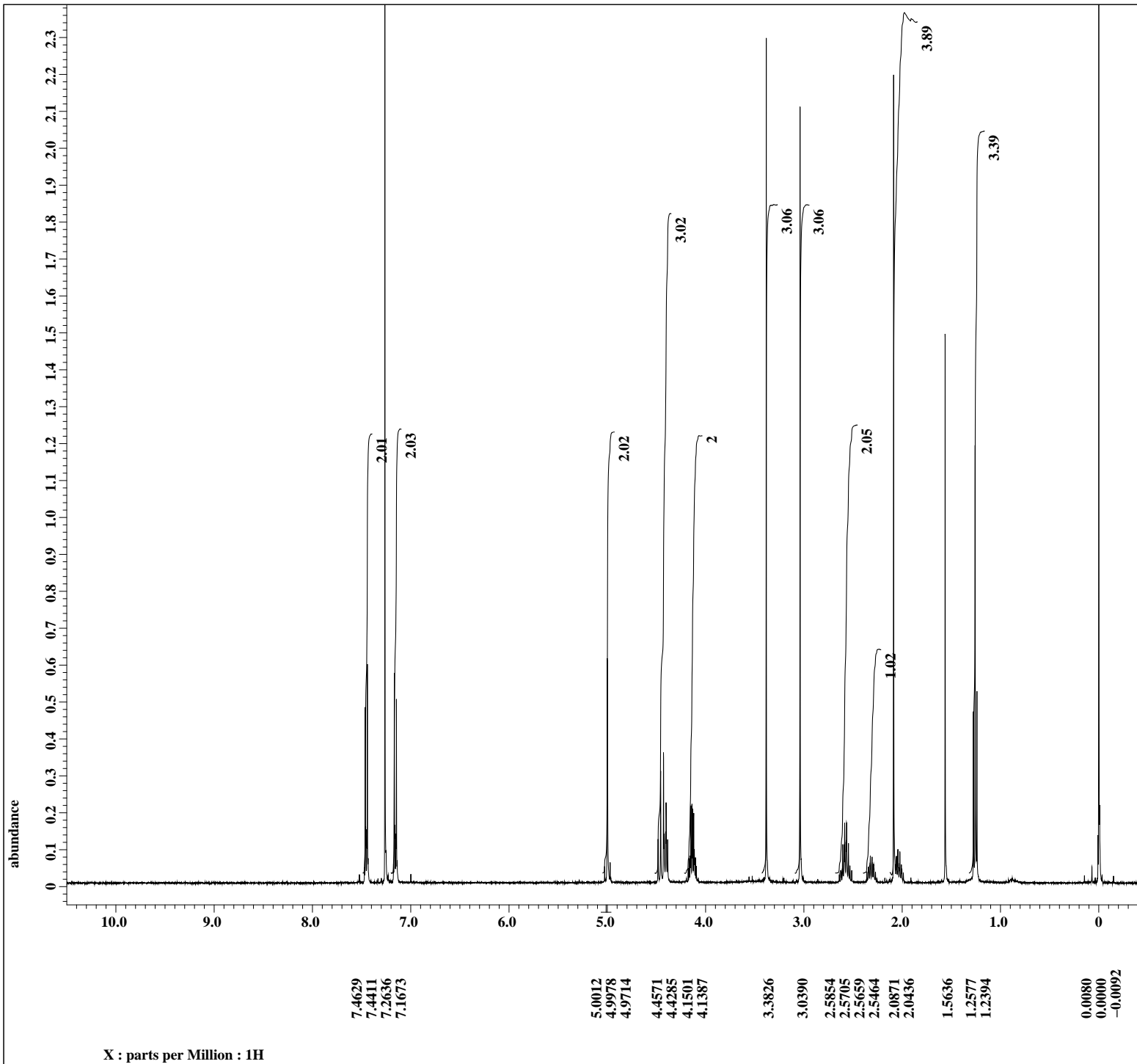
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain       = 13C
X_freq        = 100.52530333[MHz]
X_offset      = 100[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 0.95846665[Hz]
X_sweep       = 31.40703518[kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838[MHz]
Irr_offset    = 5[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 64
Total_scans   = 64

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   = 21.5[dB]
Irr_atn_noe   = 21.5[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      = 19[dC]

```



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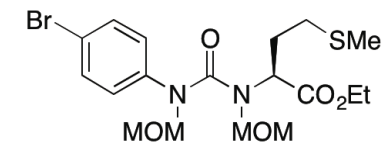


Filename = RH386-column-3.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#338225
 Solvent = CHLOROFORM-D
 Creation_time = 23-MAR-2012 02:07:48
 Revision_time = 17-APR-2013 16:08:37
 Current_time = 17-APR-2013 16:09:18

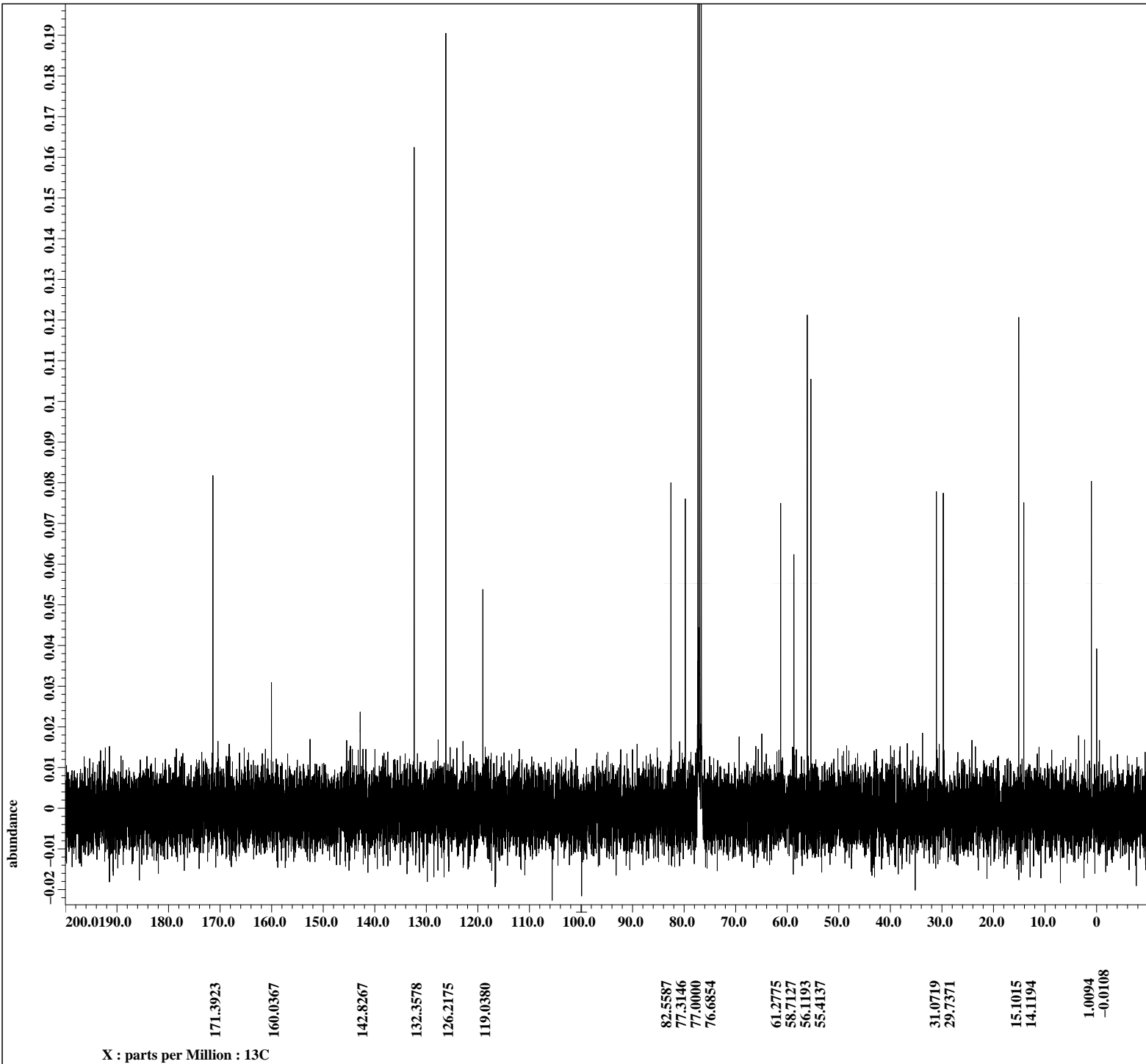
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 42
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.2[degC]



17

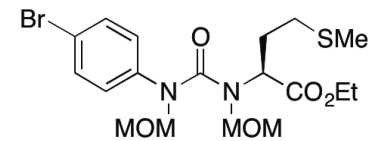


Filename = RH386-13C-5.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#716231
 Solvent = CHLOROFORM-D
 Creation_time = 18-DEC-2012 13:10:33
 Revision_time = 17-APR-2013 16:11:57
 Current_time = 17-APR-2013 16:12:24

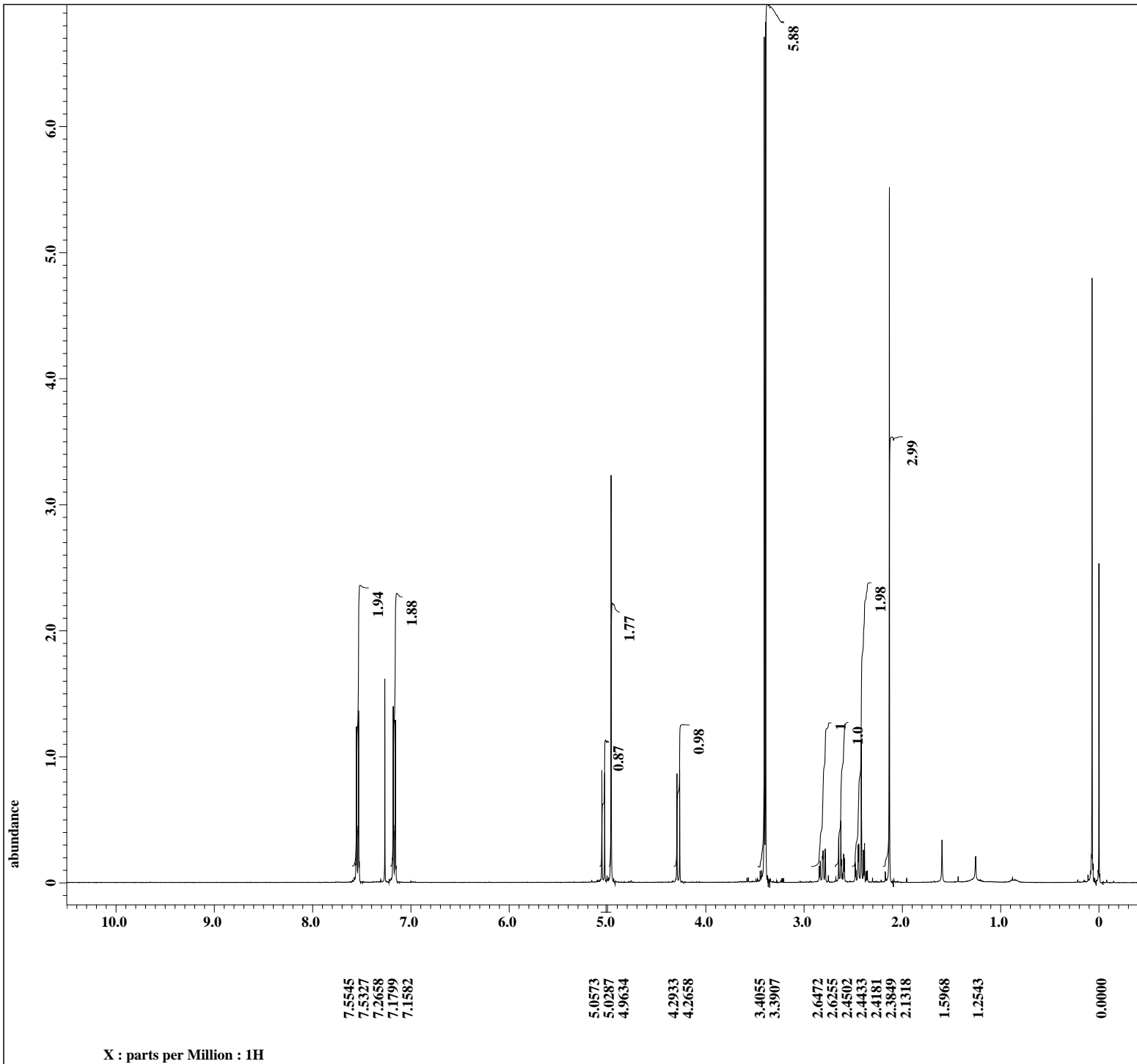
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 600
 Total_scans = 600

X_90_width = 10[us]
 X_acq_time = 1.04333312[s]
 X_angle = 30[deg]
 X_atn = 3.8[dB]
 X_pulse = 3.33333333[us]
 Irr_atn_dec = 20.8[dB]
 Irr_atn_noe = 20.8[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 56
 Relaxation_delay = 2[s]
 Repetition_time = 3.04333312[s]
 Temp_get = 17.9[dC]



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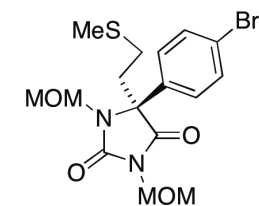


Filename = RH405-20120321-5.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = a
 Solvent = CHLOROFORM-D
 Creation_time = 21-MAR-2013 15:08:05
 Revision_time = 17-APR-2013 11:58:09
 Current_time = 17-APR-2013 11:59:07

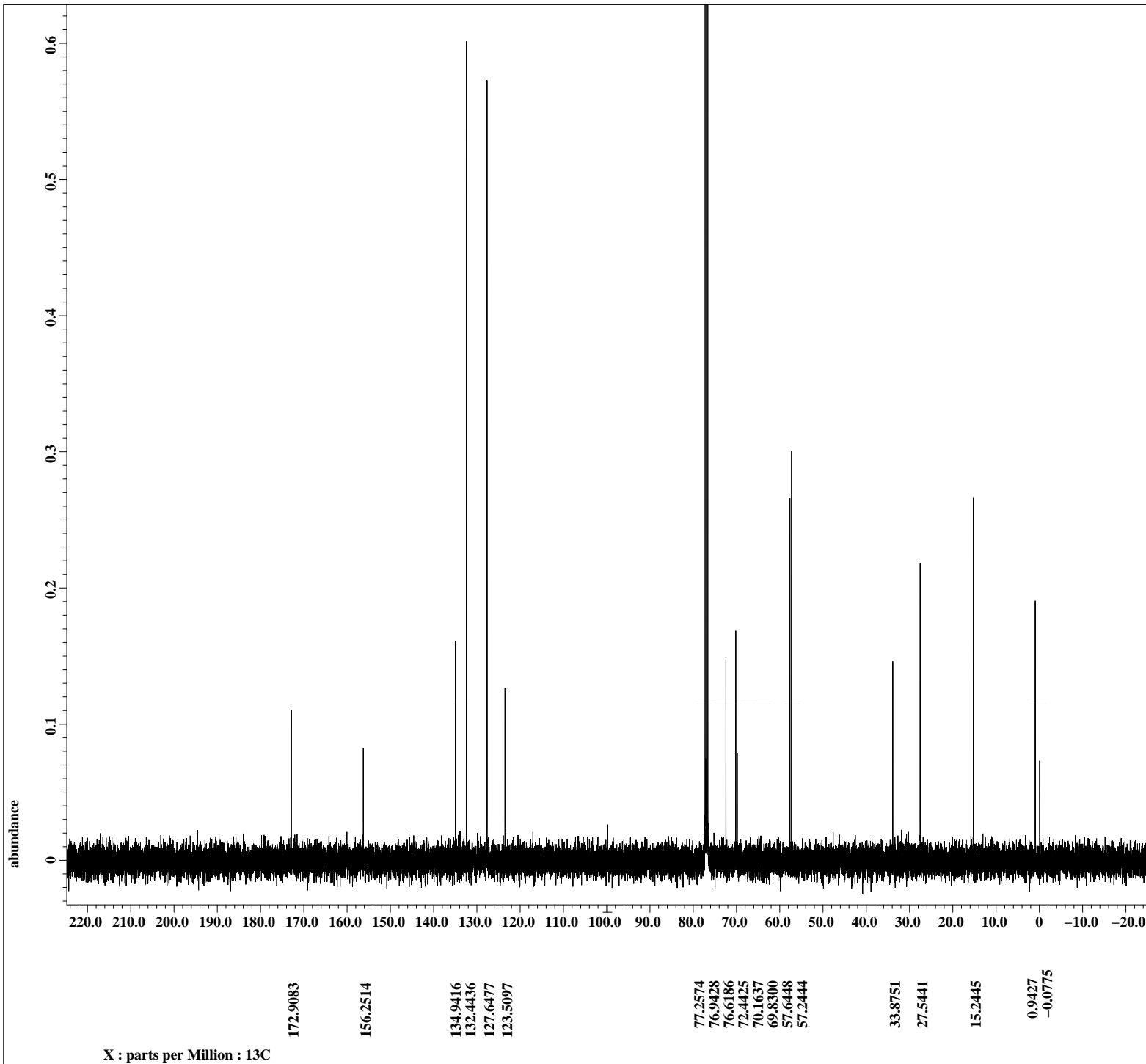
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10.75[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5.375[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 36
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.5[dC]



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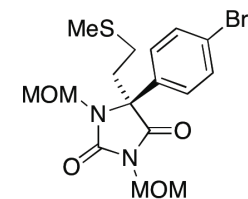
Filename      = RH405-13C-5.jdf
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#585725
Solvent      = CHLOROFORM-D
Creation_time = 18-DEC-2012 09:33:13
Revision_time = 17-APR-2013 12:00:44
Current_time  = 17-APR-2013 12:02:02

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

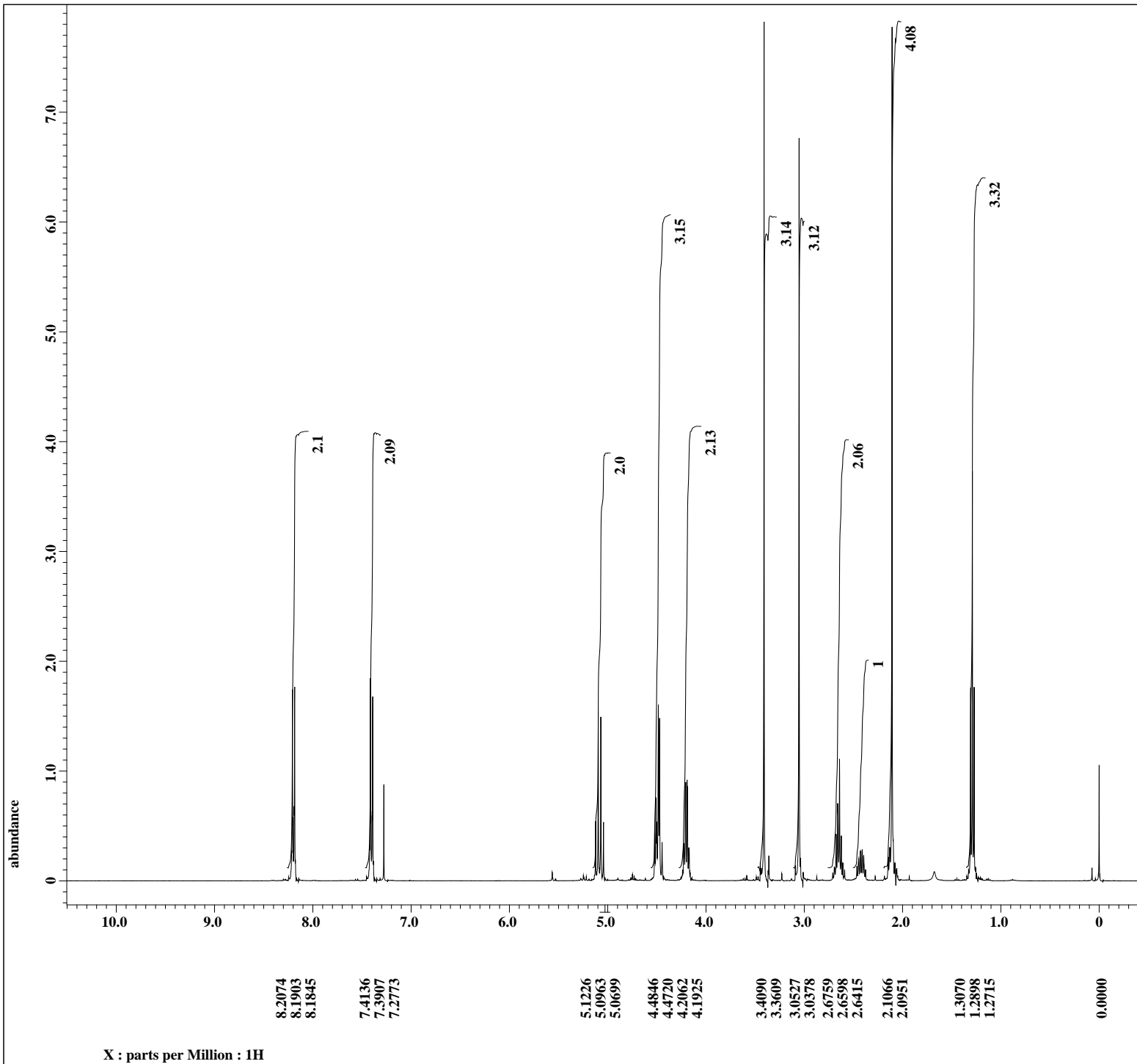
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain       = 13C
X_freq        = 100.52530333[MHz]
X_offset      = 100[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 0.95846665[Hz]
X_sweep       = 31.40703518[kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838[MHz]
Irr_offset    = 5[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 600
Total_scans   = 600

X_90_width   = 10[us]
X_acq_time   = 1.04333312[s]
X_angle      = 30[deg]
X_atn        = 3.8[dB]
X_pulse      = 3.33333333[us]
Irr_atn_dec  = 20.8[dB]
Irr_atn_noe  = 20.8[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]

```



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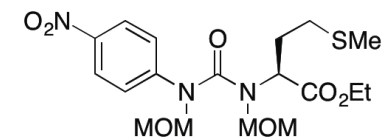
X : parts per Million : 1H

Filename = RH767-6.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = a
 Solvent = CHLOROFORM-D
 Creation_time = 21-MAR-2013 15:31:59
 Revision_time = 17-APR-2013 16:03:55
 Current_time = 17-APR-2013 16:05:44

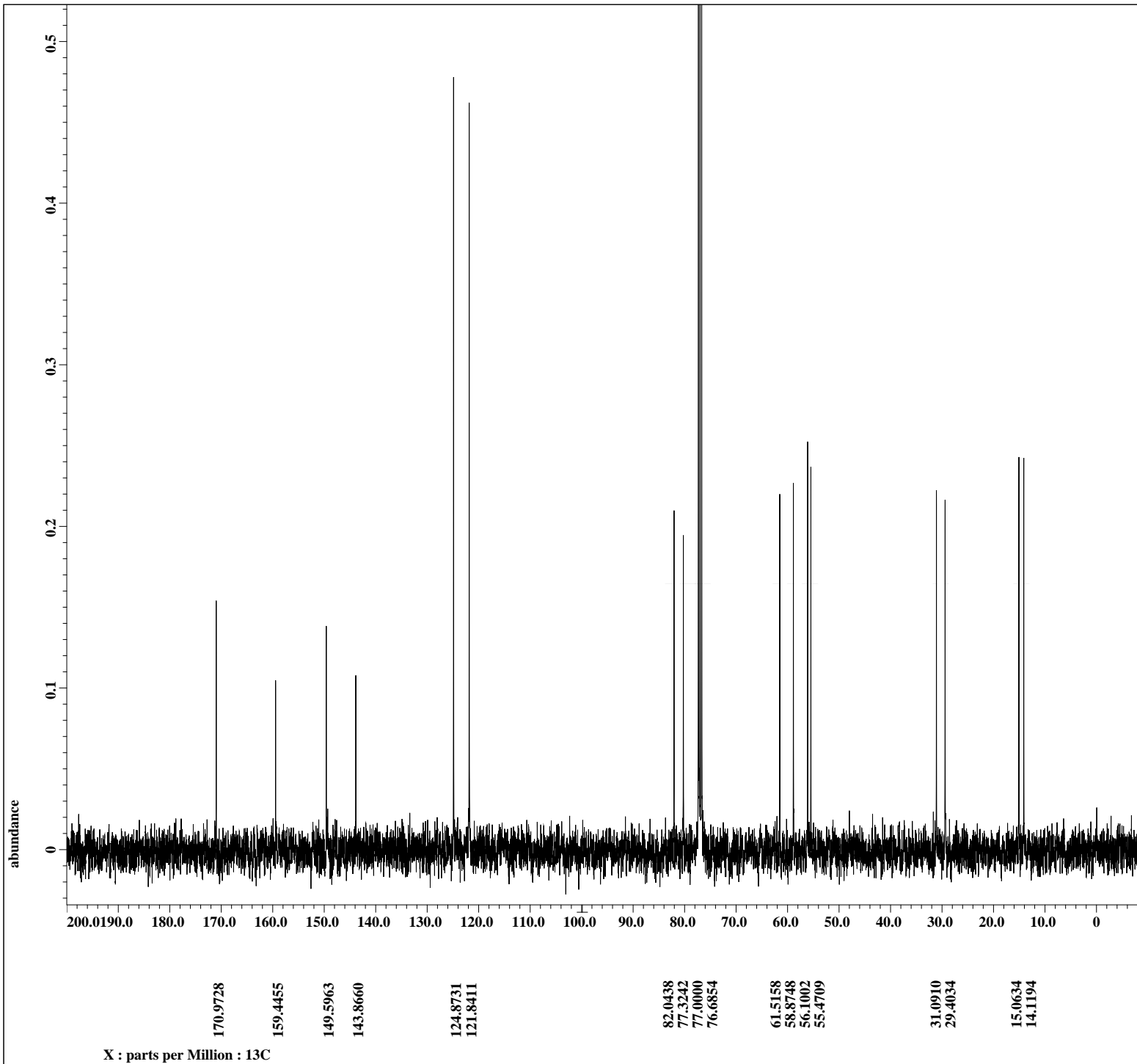
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10.75[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5.375[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 32
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 19.1[dC]



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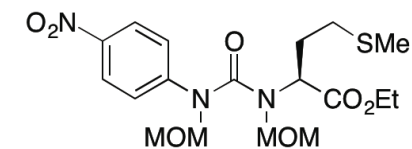


Filename = RH767-13C-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = a
 Solvent = CHLOROFORM-D
 Creation_time = 21-MAR-2013 15:40:42
 Revision_time = 17-APR-2013 16:22:04
 Current_time = 17-APR-2013 16:22:45

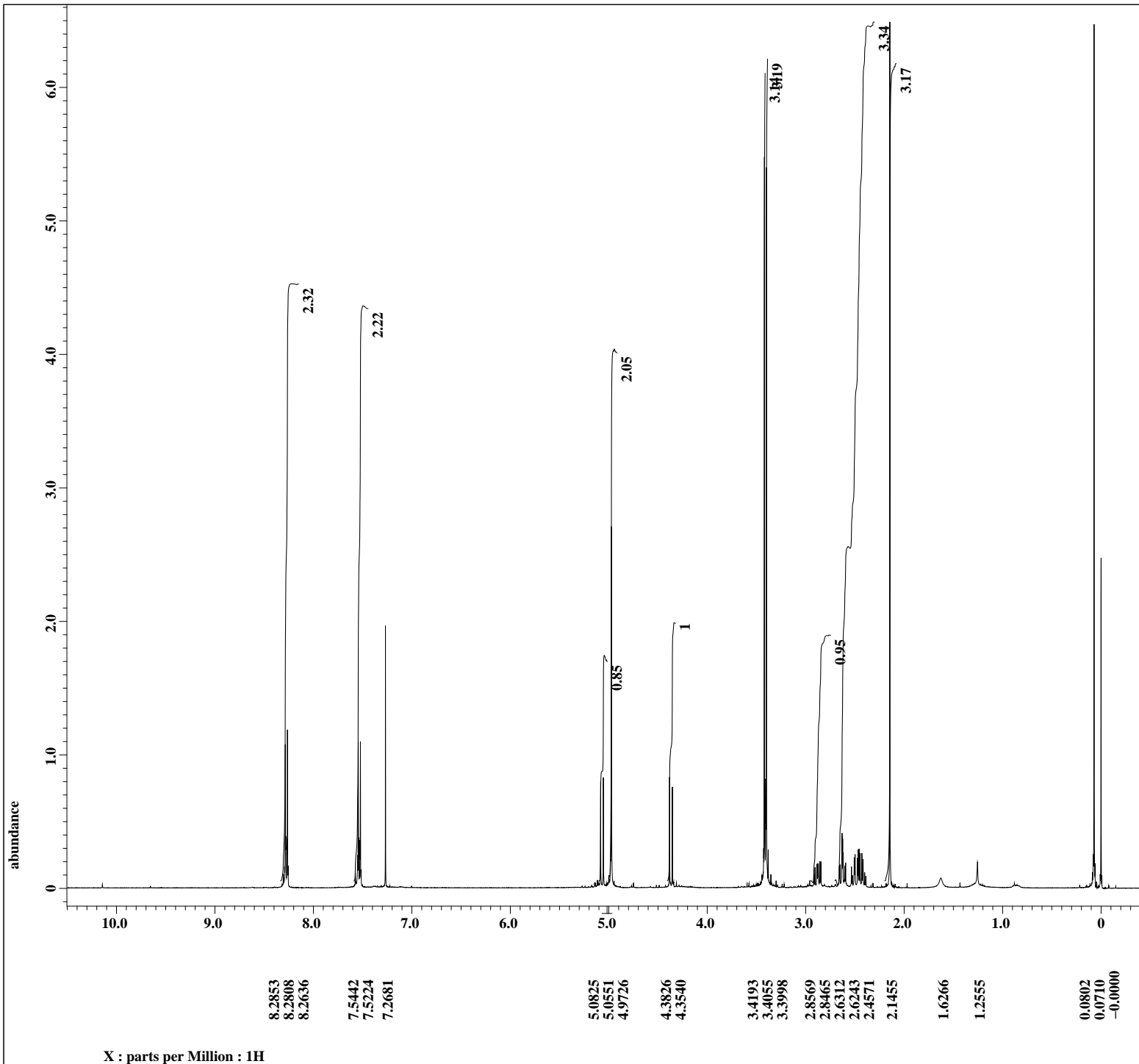
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 70
 Total_scans = 70

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 = 20.8[dB]
 Irr_atn_noe = 20.8[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 = 18.9[dC]



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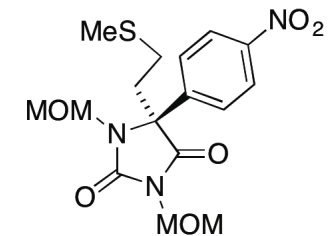
Filename      = RH430-20130321-4.jdf
Author       = delta
Experiment   = single_pulse.ex2
Sample_id    = a
Solvent      = CHLOROFORM-D
Creation_time = 21-MAR-2013 14:52:37
Revision_time = 17-APR-2013 11:47:52
Current_time  = 17-APR-2013 11:49:08

Comment      = 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_domain       = 1H
X_freq         = 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          = 8
Total_scans    = 8

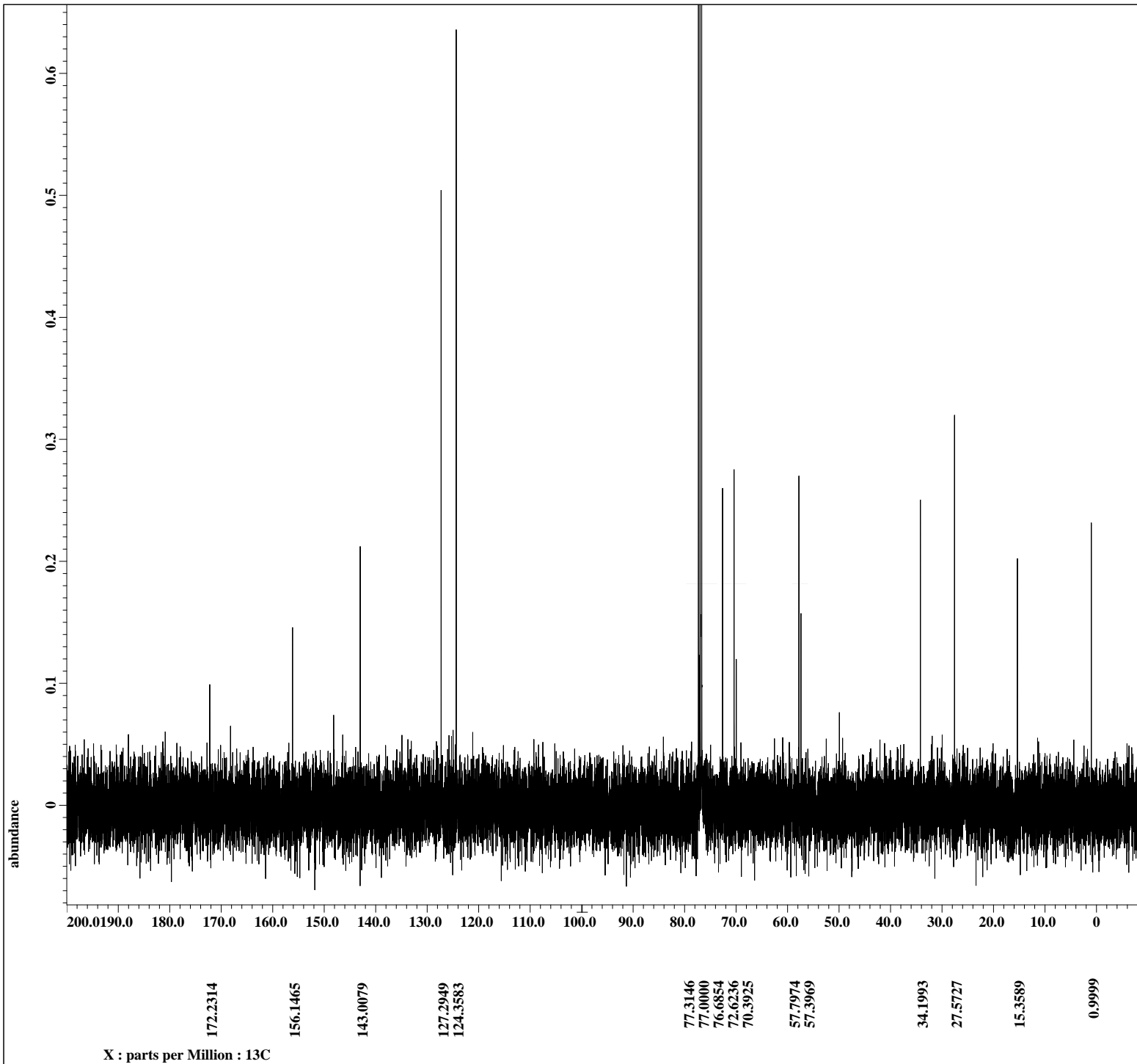
X_90_width    = 10.75[us]
X_acq_time     = 2.18365952[s]
X_angle        = 45[deg]
X_atn          = 0.3[dB]
X_pulse        = 5.375[us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait   = 1[s]
Recvr_gain     = 36
Relaxation_delay = 5[s]
Repetition_time = 7.18365952[s]
Temp_get       = 18.7[dC]

```



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X : parts per Million : 1H

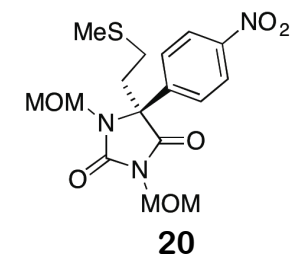


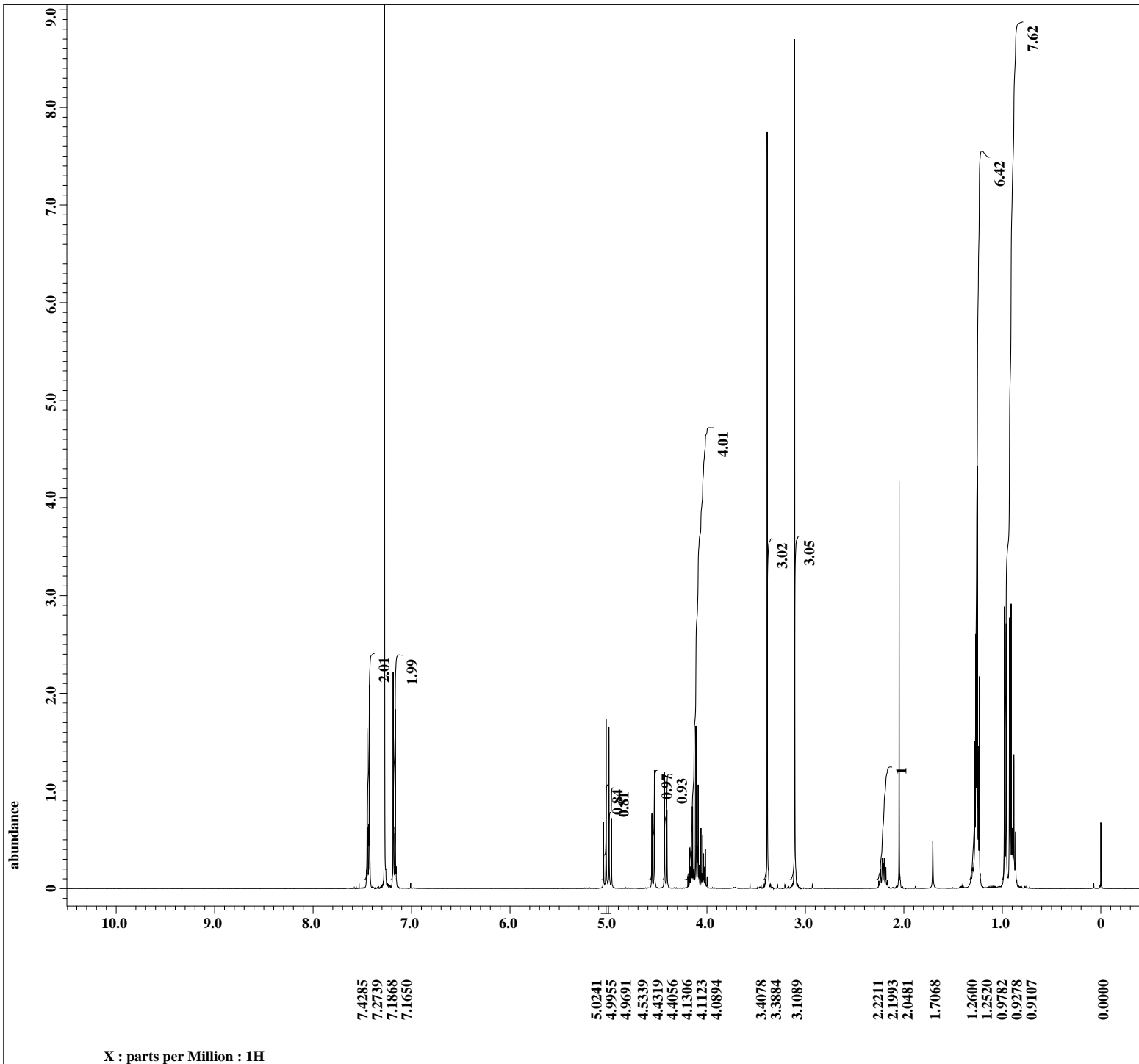
Filename = RH431-13C-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#571413
 Solvent = CHLOROFORM-D
 Creation_time = 18-DEC-2012 08:45:57
 Revision_time = 17-APR-2013 11:50:02
 Current_time = 17-APR-2013 11:51:29

Comment = 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 = 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 = 124
 Total_scans = 124

X_90_width = 10[us]
 X_acq_time = 1.04333312[s]
 X_angle = 30[deg]
 X_atn = 3.8[dB]
 X_pulse = 3.33333333[us]
 Irr_atn_dec = 20.8[dB]
 Irr_atn_noe = 20.8[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 = 17.7[dC]



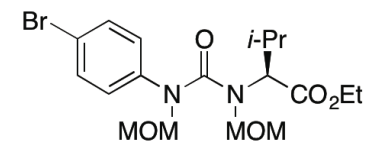


Filename = YP471 Tproj. chiral d
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#576896
 Solvent = CHLOROFORM-D
 Creation_time = 26-MAR-2012 08:45:26
 Revision_time = 16-APR-2013 20:35:35
 Current_time = 16-APR-2013 20:36:52

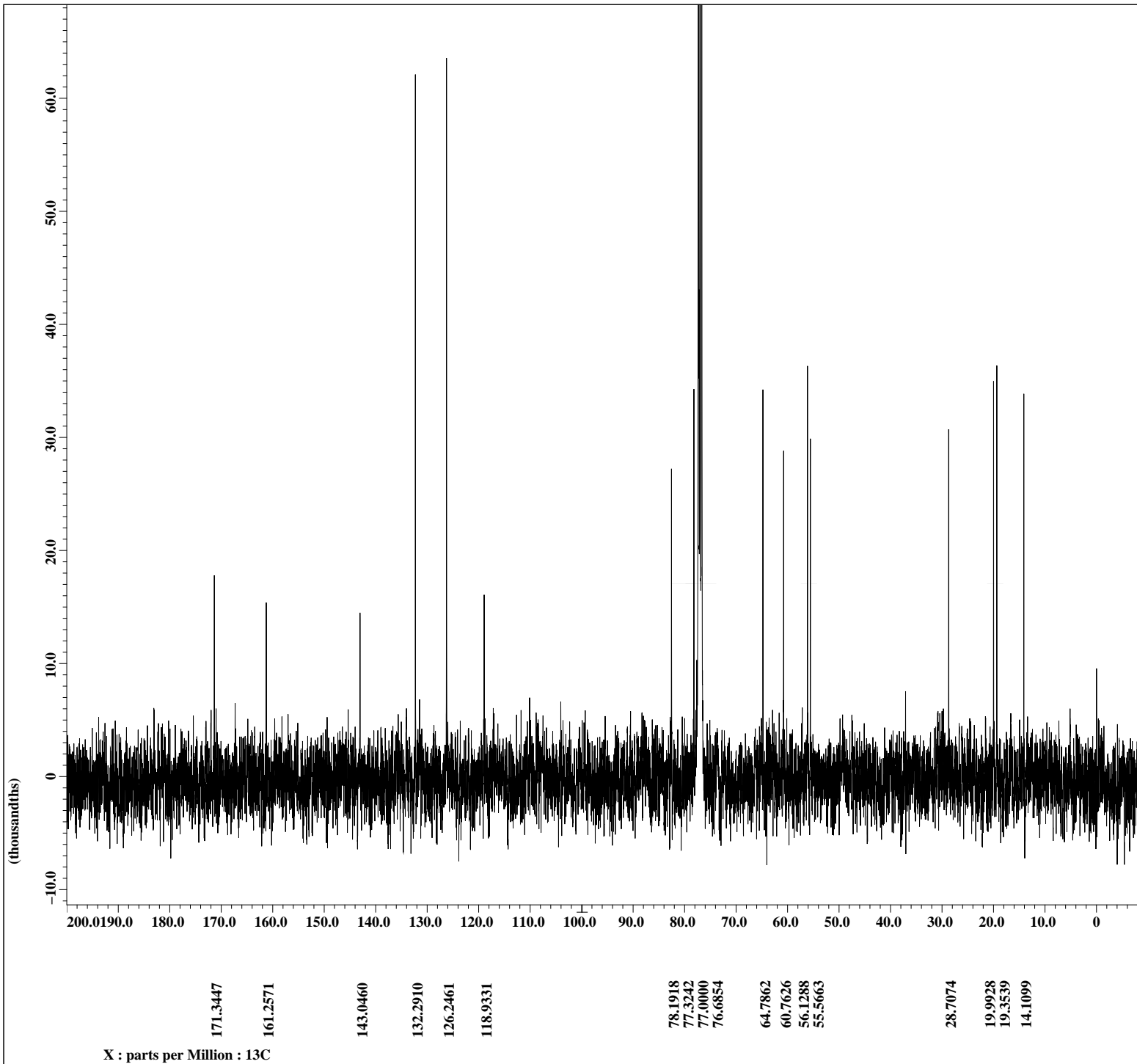
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 32
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.5[dC]



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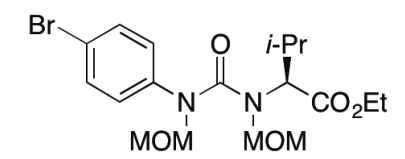
```

Filename      = YP471 Tproj. chiral d
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#794165
Solvent      = CHLOROFORM-D
Creation_time = 26-MAR-2012 15:11:41
Revision_time = 16-APR-2013 20:47:09
Current_time  = 16-APR-2013 20:48:22

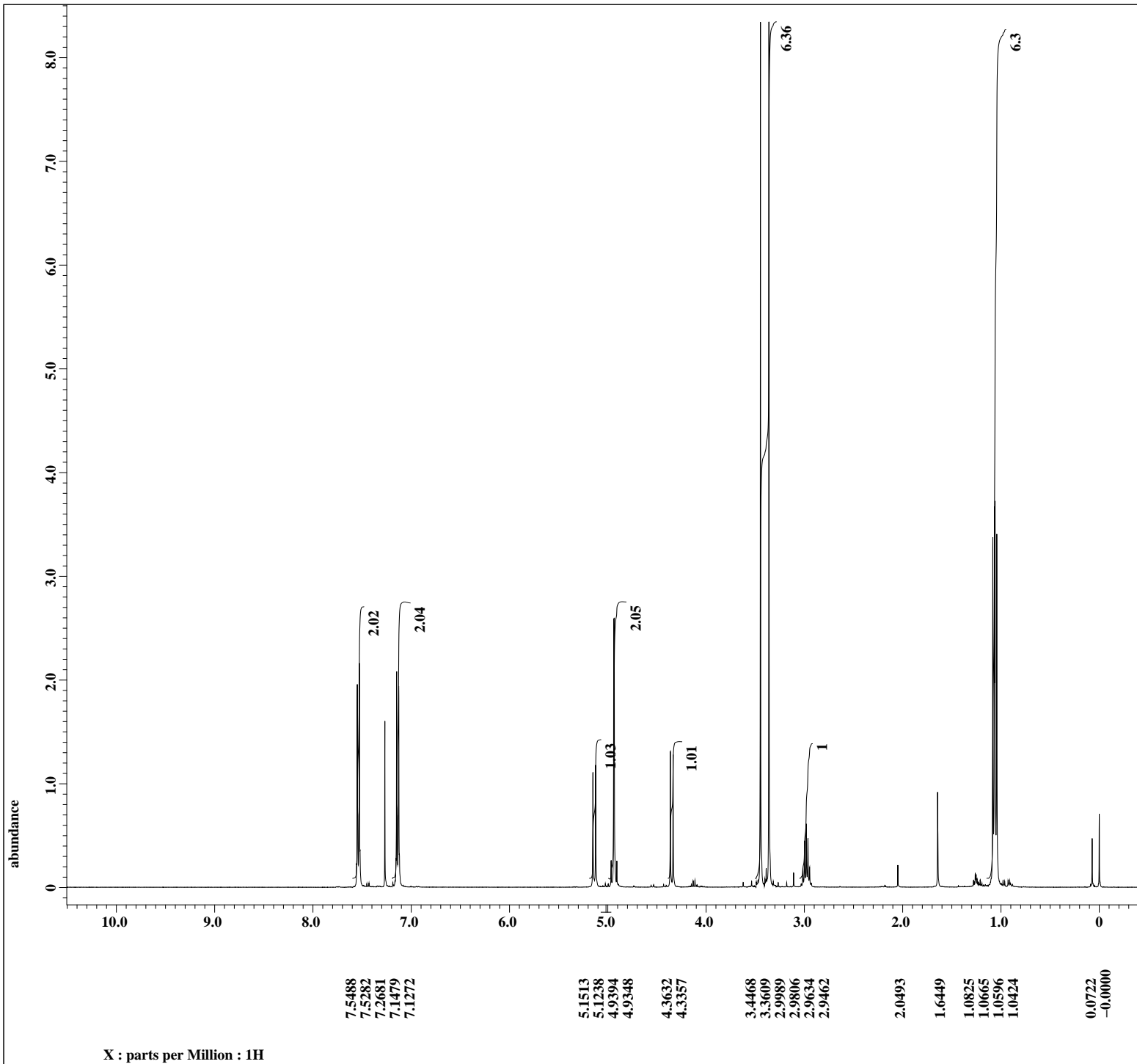
Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 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          = 500
Total_scans    = 500

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    = 21.5[dB]
Irr_atn_noe    = 21.5[dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1[s]
Noe            = TRUE
Noe_time       = 2[s]
Recvr_gain     = 56
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get       = 18.5[dC]
  
```



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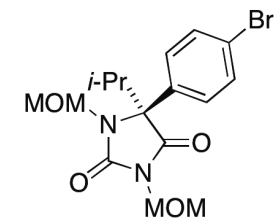


Filename = YP472 T-proj. repurif
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#777053
 Solvent = CHLOROFORM-D
 Creation_time = 5-APR-2012 14:18:38
 Revision_time = 17-APR-2013 12:04:24
 Current_time = 17-APR-2013 12:21:04

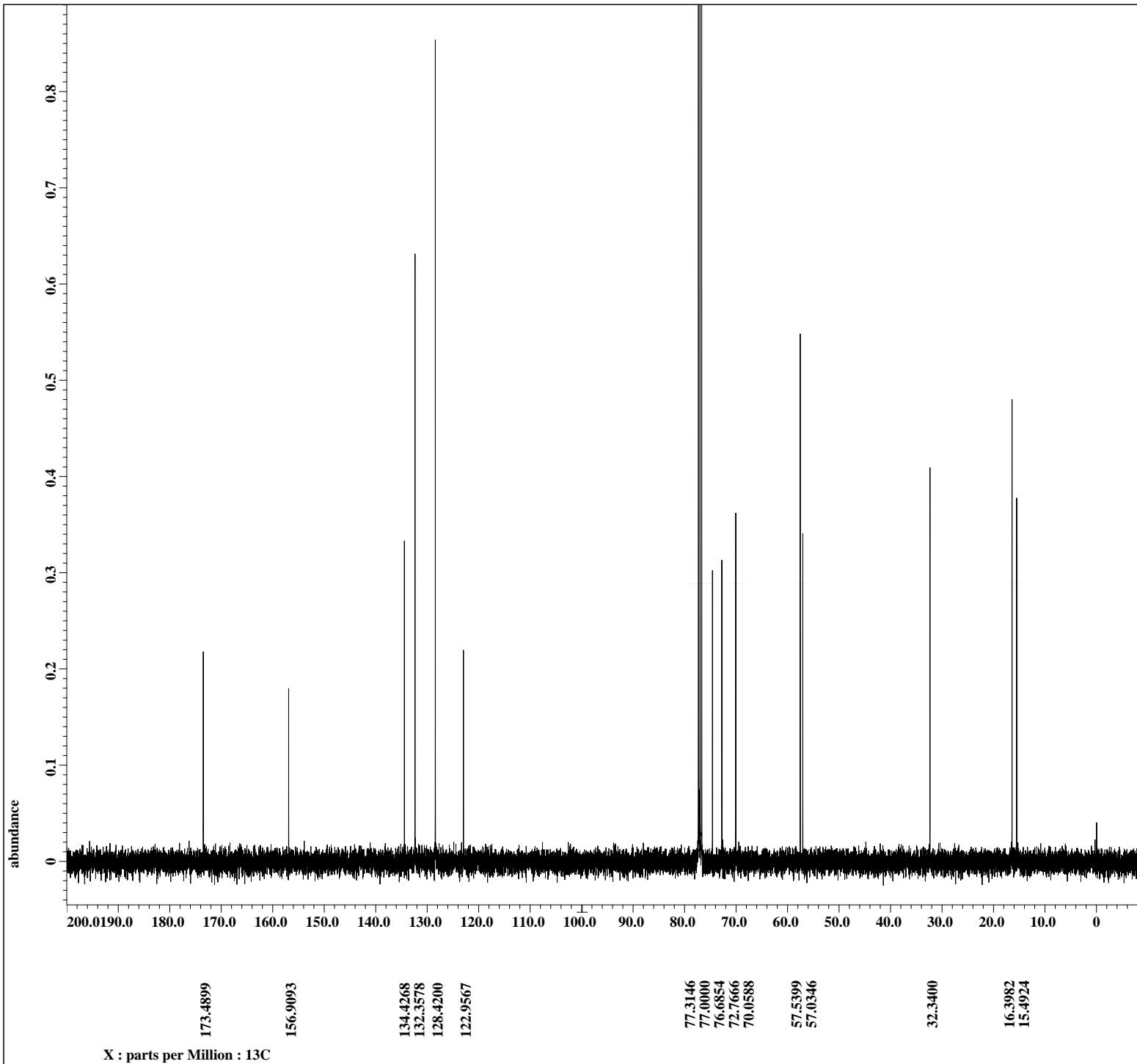
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 36
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.2[dC]



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```

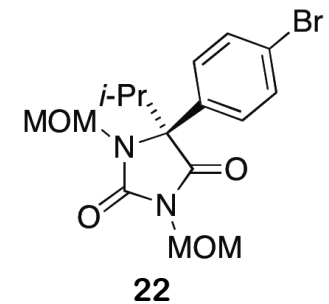
Filename      = YP472 T-proj. repurif
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#778784
Solvent      = CHLOROFORM-D
Creation_time = 5-APR-2012 14:45:40
Revision_time = 17-APR-2013 12:21:45
Current_time  = 17-APR-2013 12:23:18

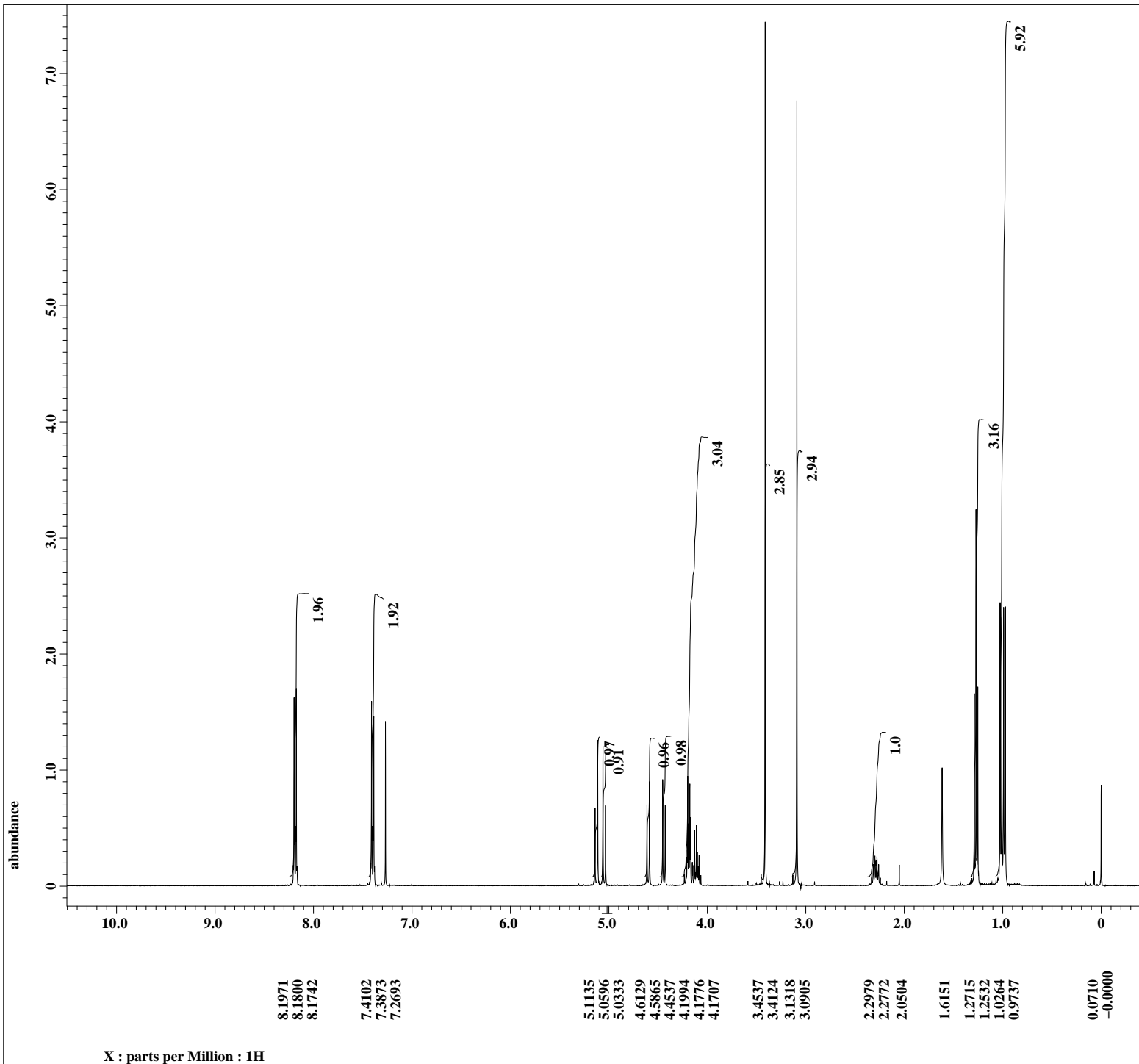
Comment      = single pulse decouple
Data_format  = 1D_COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 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         = 500
Total_scans   = 500

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   = 21.5[dB]
Irr_atn_noe   = 21.5[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      = 18.9[dC]

```



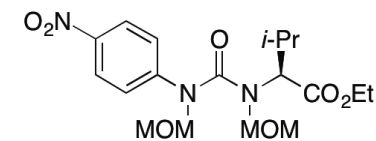


Filename = YP681 T-proj. NO2 L-V
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#646466
 Solvent = CHLOROFORM-D
 Creation_time = 19-MAR-2013 10:32:16
 Revision_time = 16-APR-2013 20:54:22
 Current_time = 16-APR-2013 20:54:46

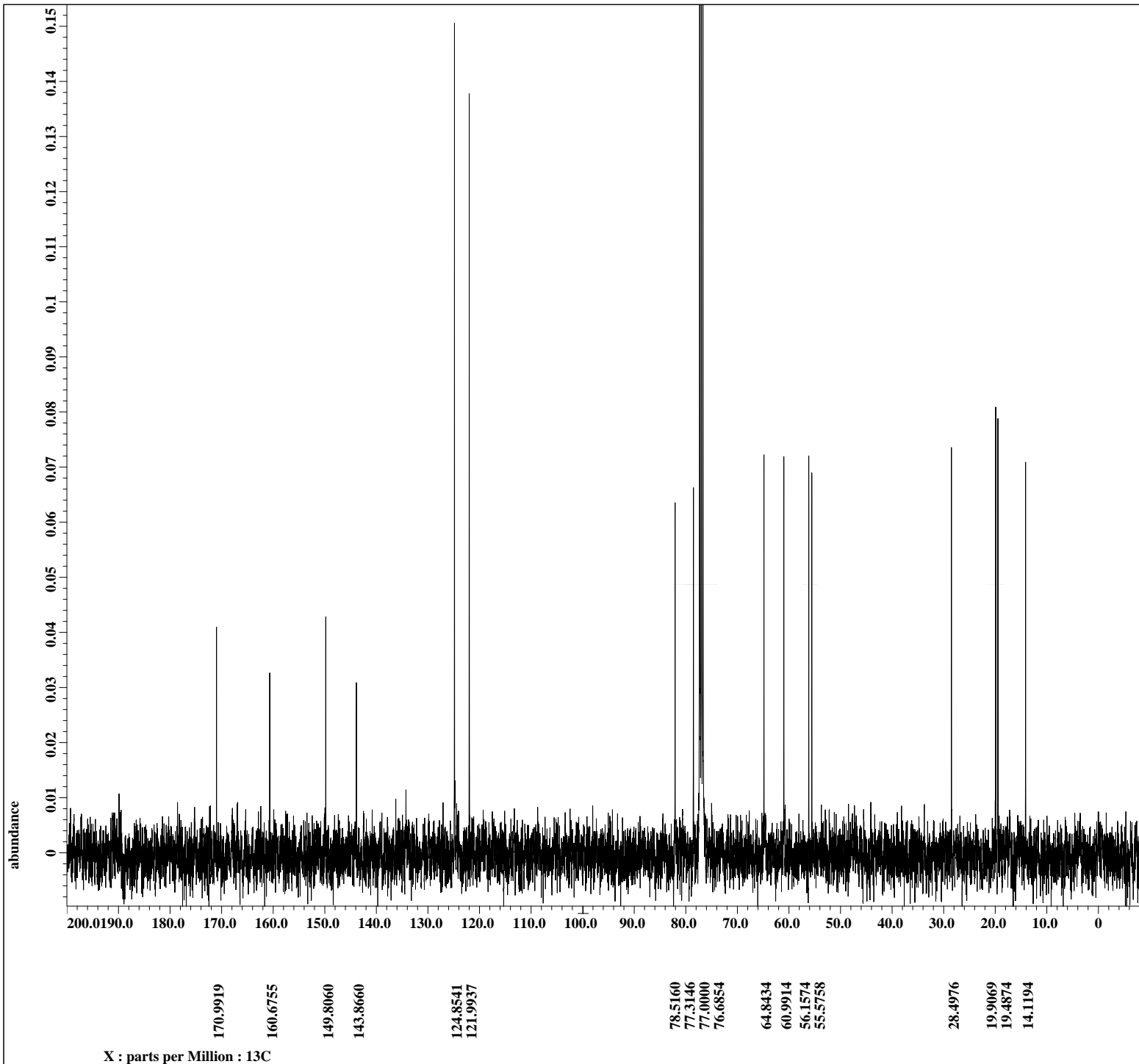
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10.75[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5.375[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 38
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18[dC]



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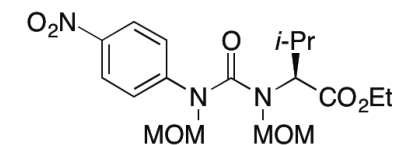


Filename = YP681 T-proj. NO2 L-V
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#647955
 Solvent = CHLOROFORM-D
 Creation_time = 19-MAR-2013 10:45:48
 Revision_time = 16-APR-2013 21:06:34
 Current_time = 16-APR-2013 21:06:54

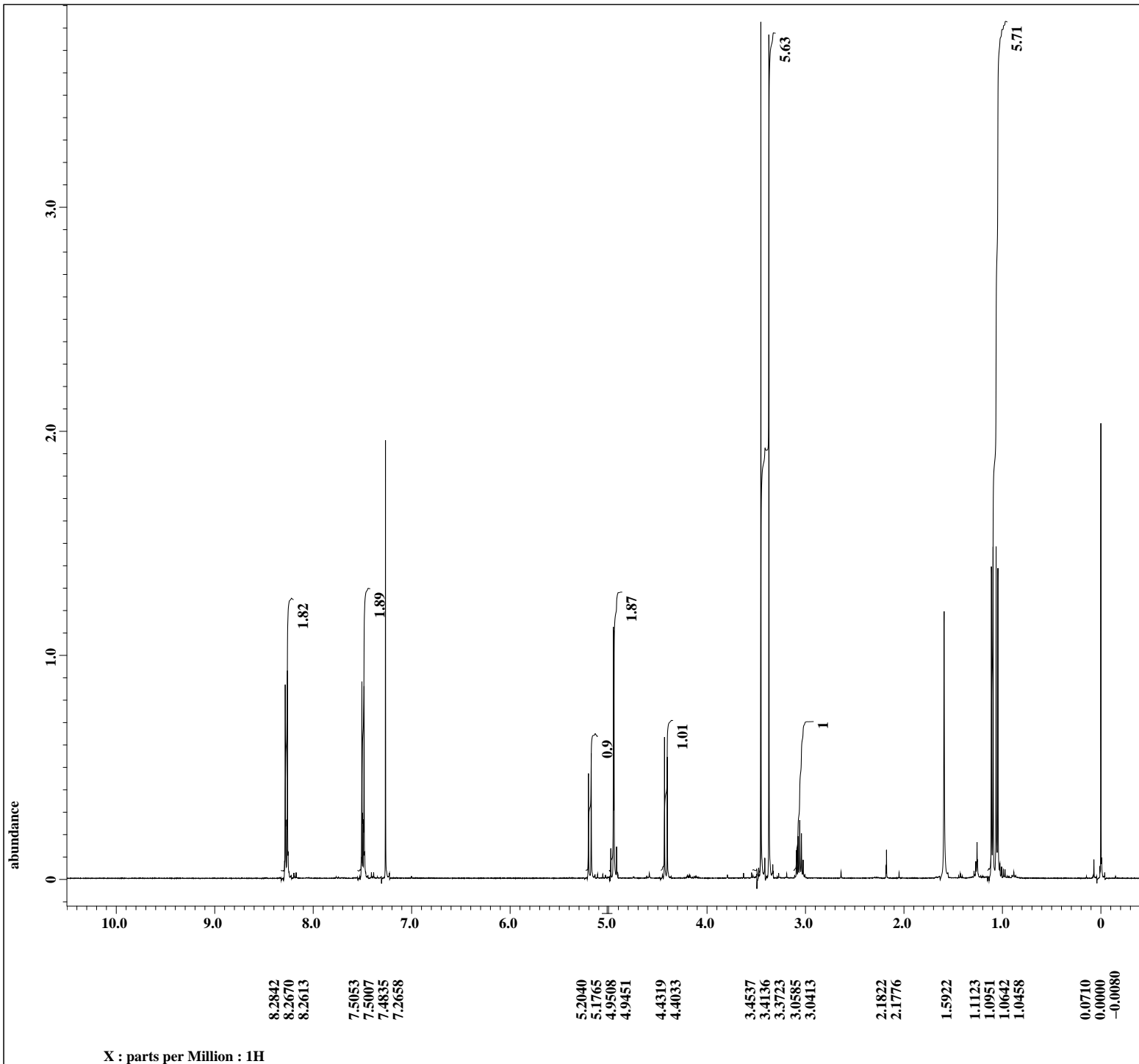
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 238
 Total_scans = 238

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 = 20.8[dB]
 Irr_atn_noe = 20.8[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 56
 Relaxation_delay = 2[s]
 Repetition_time = 3.04333312[s]
 Temp_get = 18.7[dC]



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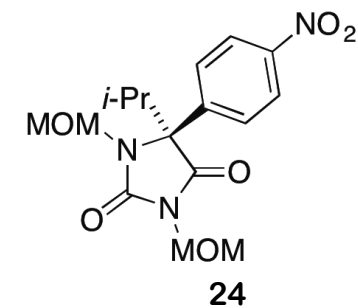


Filename = YP506 T-proj. NO2 LiH
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#371325
 Solvent = CHLOROFORM-D
 Creation_time = 28-JUN-2012 10:20:11
 Revision_time = 17-APR-2013 14:48:51
 Current_time = 17-APR-2013 14:49:57

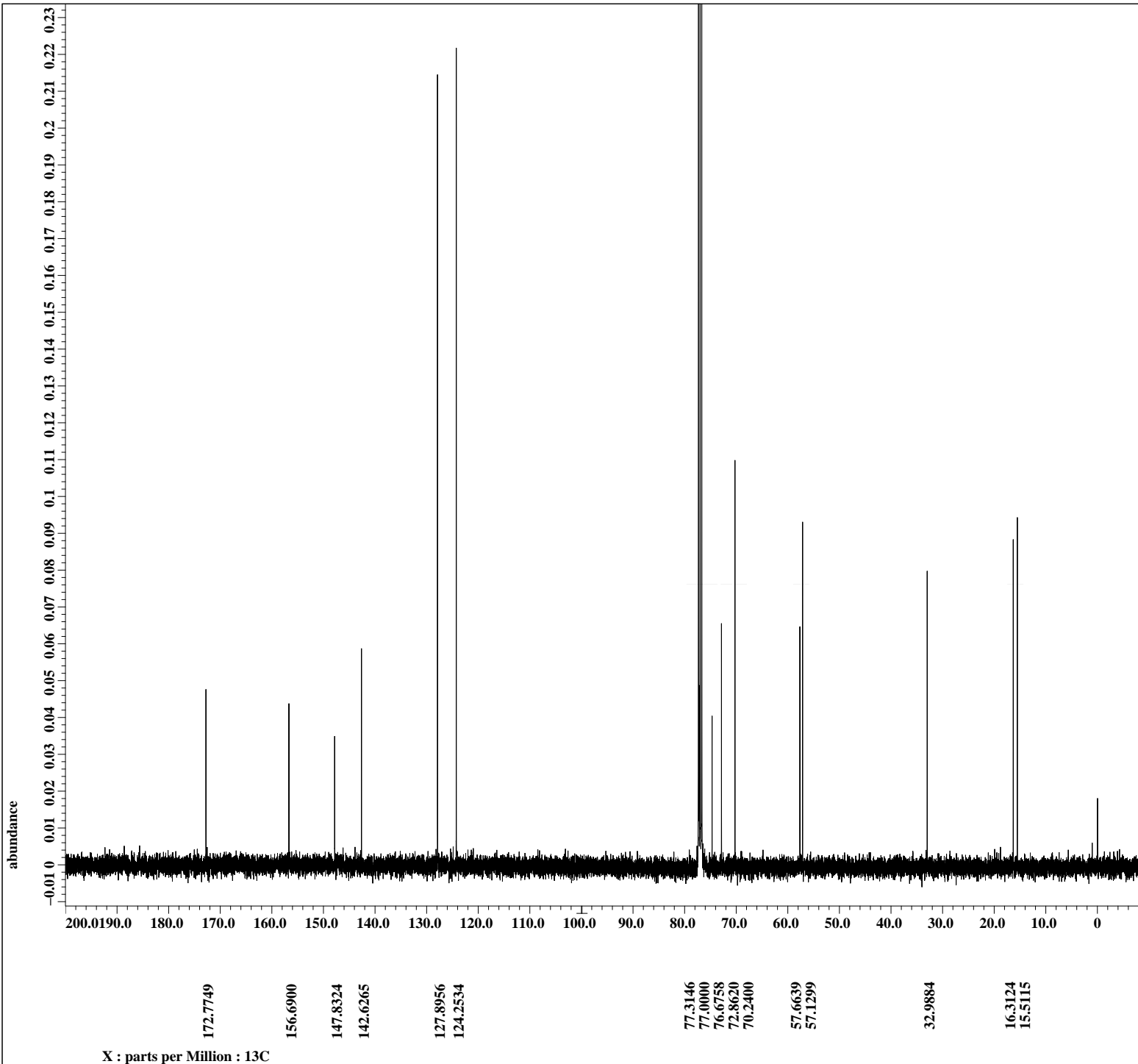
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10.75[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5.375[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 38
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 19.1[degC]



X : parts per Million : 1H

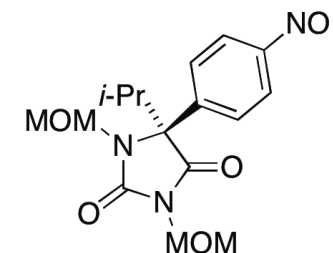


Filename = YP506 T-proj. NO2 LiH
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#79989
 Solvent = CHLOROFORM-D
 Creation_time = 29-JUN-2012 06:16:50
 Revision_time = 17-APR-2013 14:52:43
 Current_time = 17-APR-2013 14:53:13

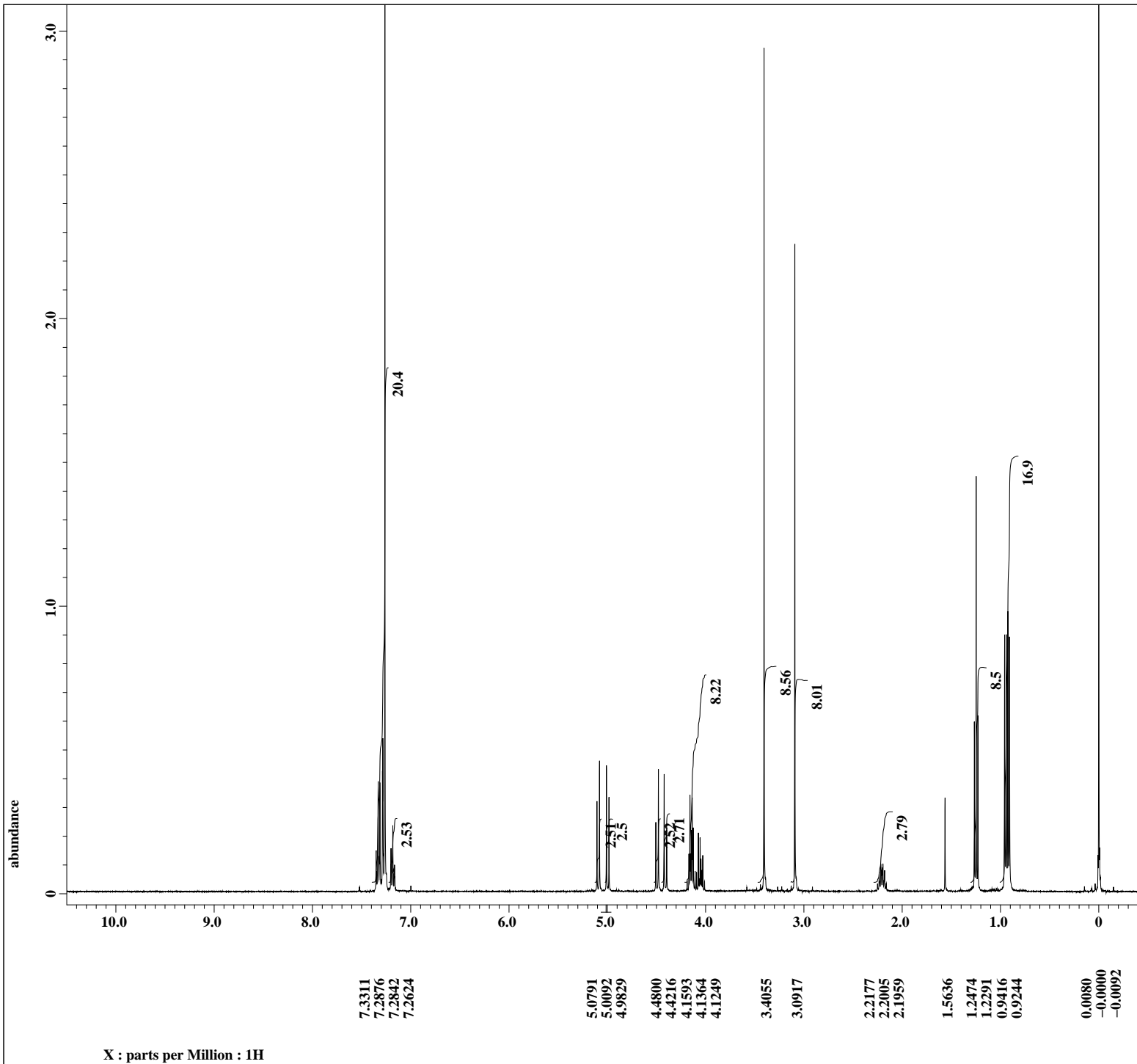
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 4800
 Total_scans = 4800

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 = 20.8[dB]
 Irr_atn_noe = 20.8[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 54
 Relaxation_delay = 2[s]
 Repetition_time = 3.04333312[s]
 Temp_get = 19[dC]



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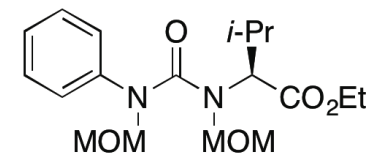


Filename = KT-1763-3.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#837753
 Solvent = CHLOROFORM-D
 Creation_time = 21-FEB-2012 16:07:28
 Revision_time = 17-APR-2013 19:58:39
 Current_time = 17-APR-2013 19:59:24

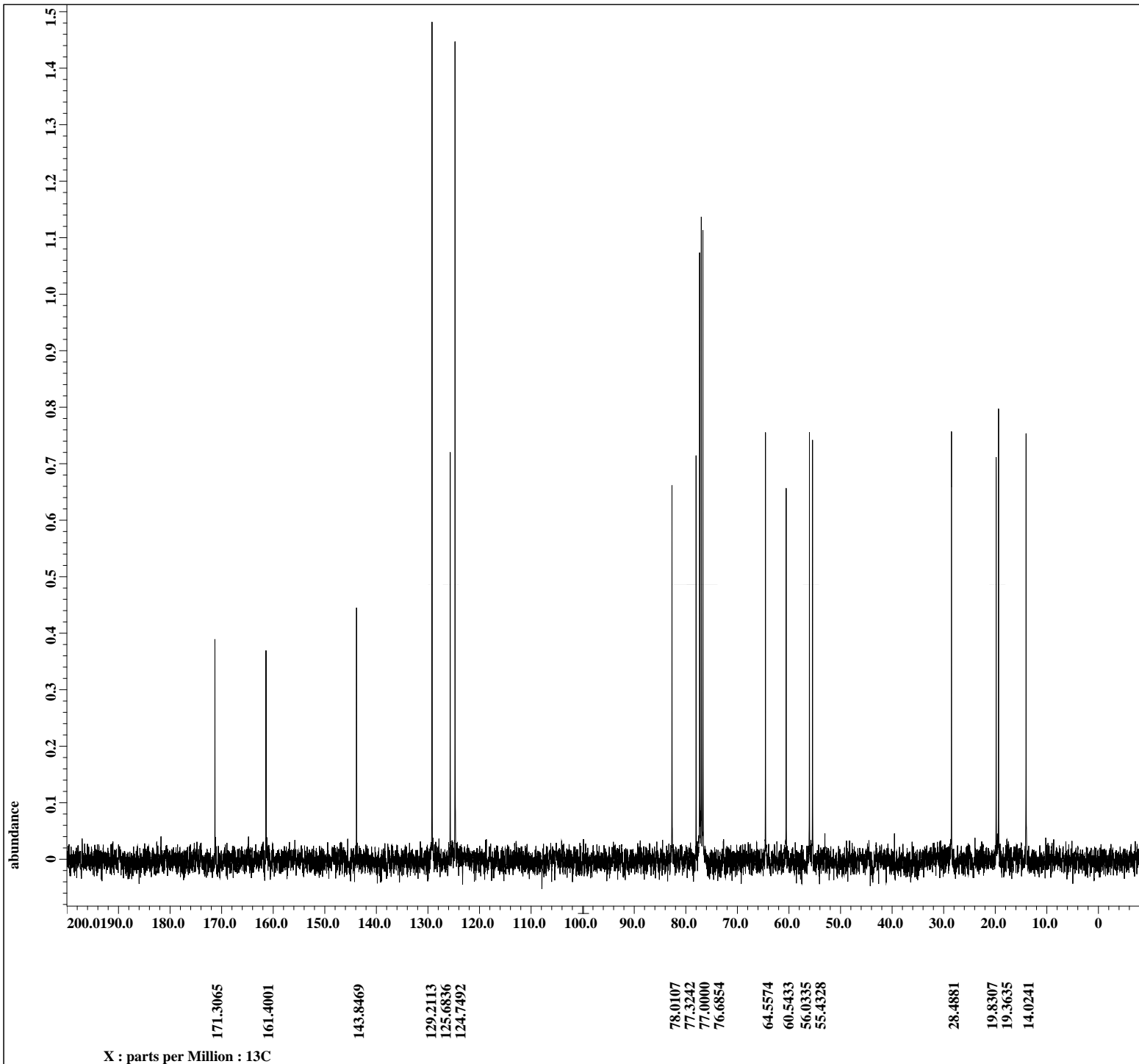
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[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 = 18.6[degC]



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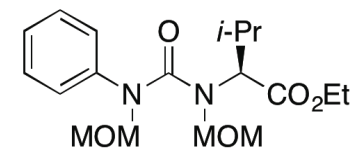


Filename = KT-1763-13C-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#735415
 Solvent = CHLOROFORM-D
 Creation_time = 14-FEB-2012 13:19:37
 Revision_time = 17-APR-2013 20:00:06
 Current_time = 17-APR-2013 20:00:32

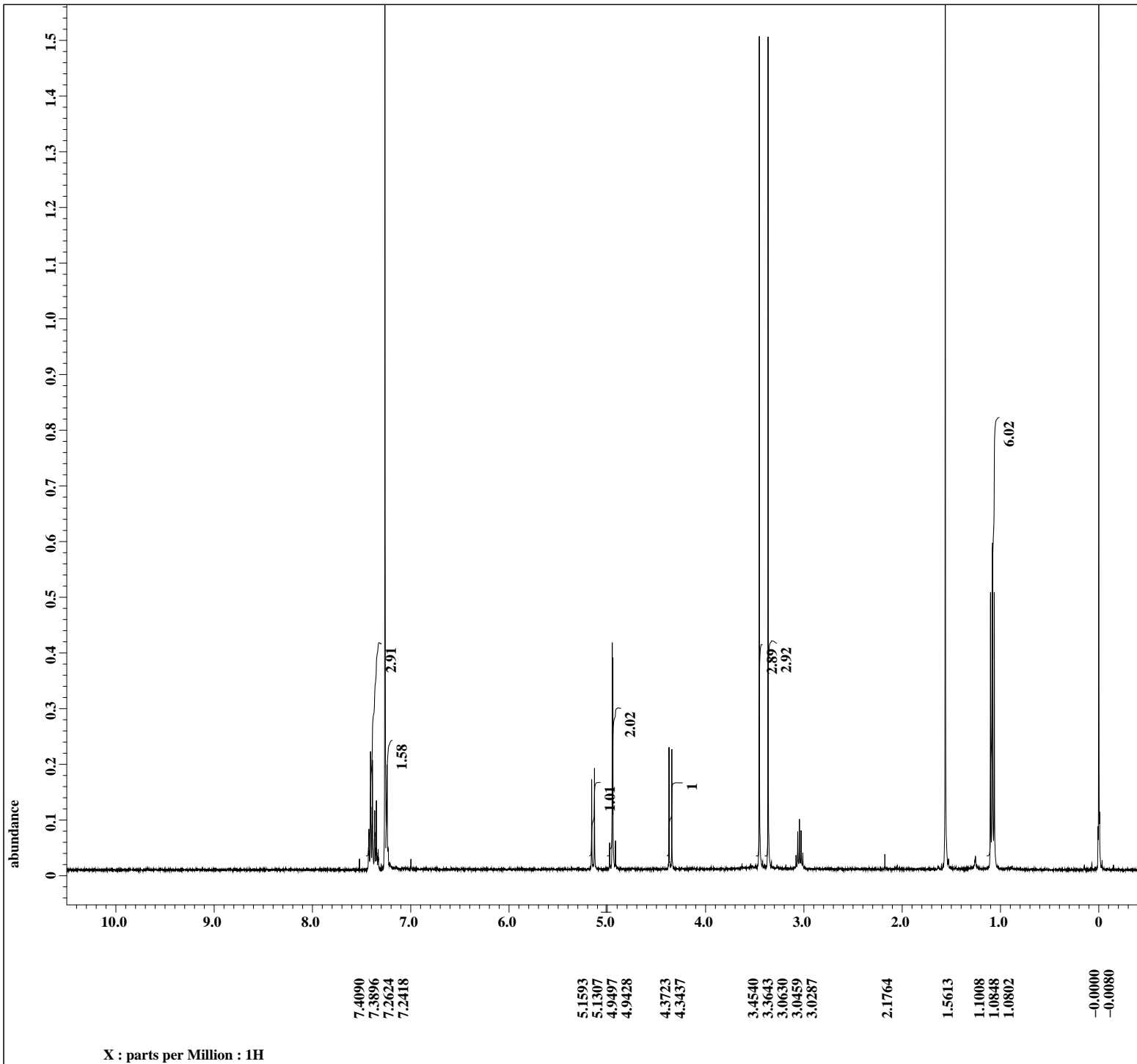
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 34
 Total_scans = 34

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 = 21.5[dB]
 Irr_atn_noe = 21.5[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 = 18.9[dC]



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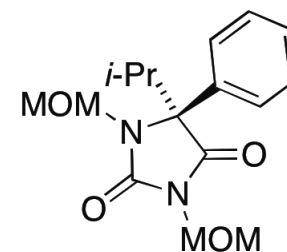
X : parts per Million : 1H

Filename = Kt-1765-product-3.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#347859
 Solvent = CHLOROFORM-D
 Creation_time = 16-FEB-2012 02:30:57
 Revision_time = 17-APR-2013 20:02:57
 Current_time = 17-APR-2013 20:03:30

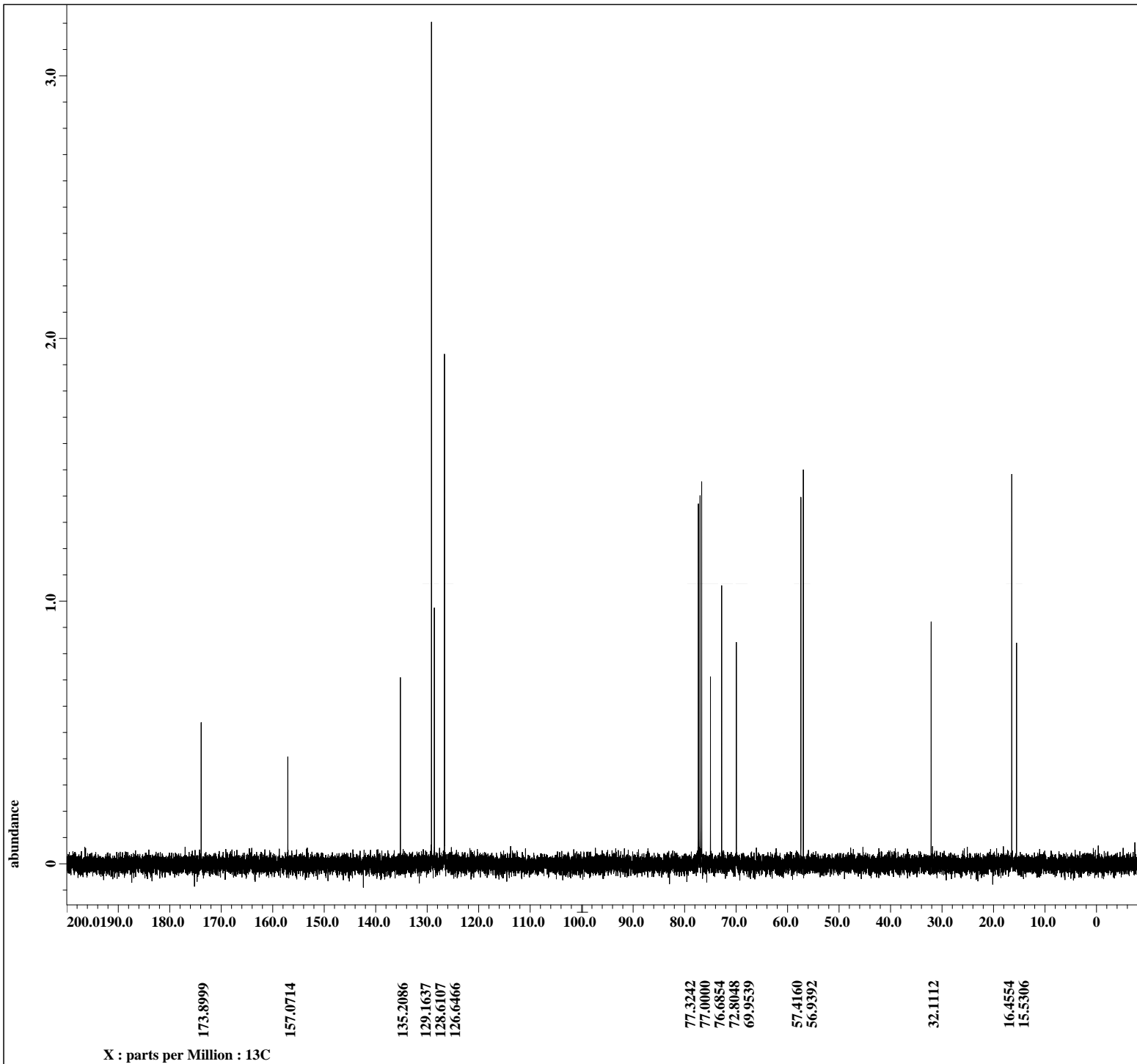
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 42
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 19[dC]



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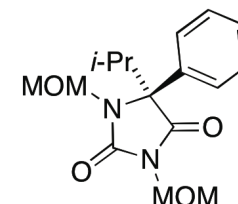


Filename = KT-1765-13C-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#354061
 Solvent = CHLOROFORM-D
 Creation_time = 16-FEB-2012 02:44:04
 Revision_time = 17-APR-2013 20:04:14
 Current_time = 17-APR-2013 20:04:37

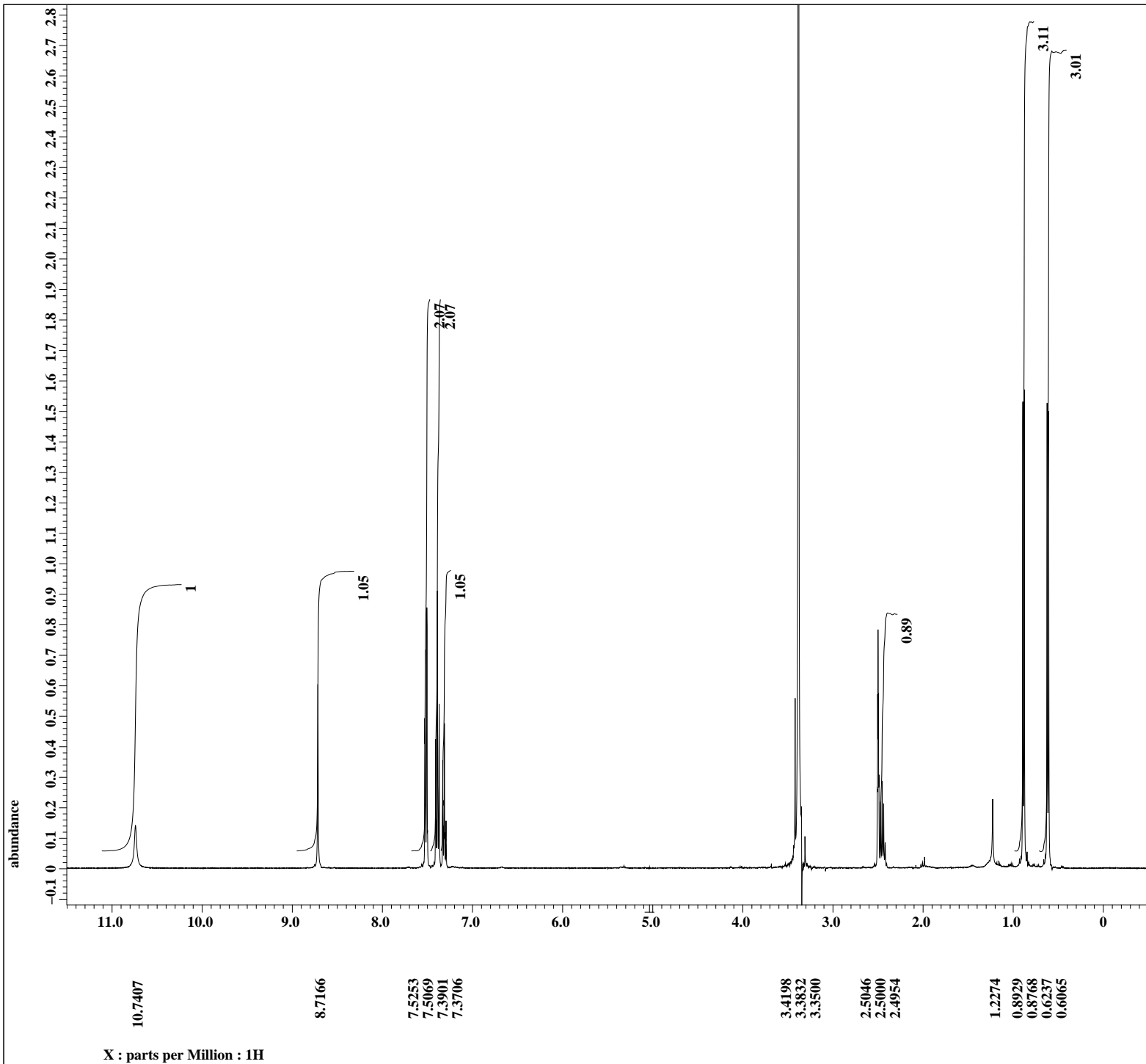
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 42
 Total_scans = 42

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 = 21.5[dB]
 Irr_atn_noe = 21.5[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 56
 Relaxation_delay = 2[s]
 Repetition_time = 3.04333312[s]
 Temp_get = 19.2[dC]



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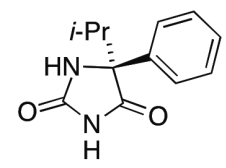
Filename      = TY-12-148-DMSOd6-5.jd
Author       = delta
Experiment    = single_pulse.ex2
Sample_id    = S#714857
Solvent      = DMSO-D6
Creation_time = 23-JUN-2013 12:18:02
Revision_time = 23-JUN-2013 19:58:19
Current_time  = 23-JUN-2013 21:07:23

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

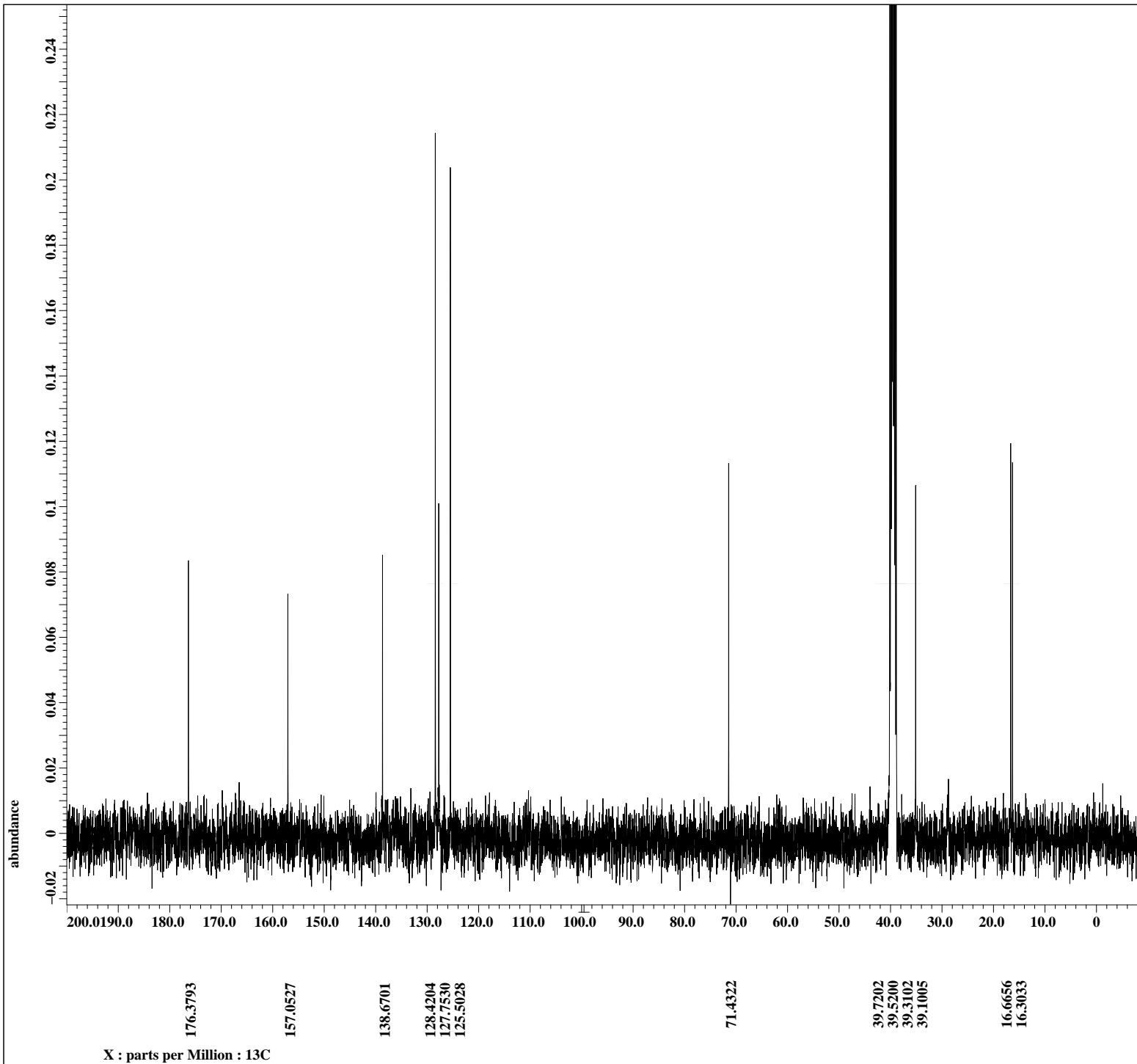
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 2.18365952[s]
X_domain      = 1H
X_freq       = 399.78219838[MHz]
X_offset     = 5[ppm]
X_points     = 16384
X_prescans   = 1
X_resolution = 0.45794685[Hz]
X_sweep     = 7.5030012[kHz]
Irr_domain   = 1H
Irr_freq    = 399.78219838[MHz]
Irr_offset   = 5[ppm]
Tri_domain   = 1H
Tri_freq    = 399.78219838[MHz]
Tri_offset   = 5[ppm]
Clipped     = FALSE
Mod_return  = 1
Scans       = 8
Total_scans = 8

X_90_width  = 10.75[us]
X_acq_time  = 2.18365952[s]
X_angle     = 45[deg]
X_atn      = 0.3[dB]
X_pulse    = 5.375[us]
Irr_mode    = Off
Tri_mode    = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain  = 34
Relaxation_delay = 5[s]
Repetition_time = 7.18365952[s]
Temp_get    = 20.2[dC]

```



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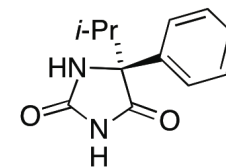


Filename = TY-12-148 comp-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = S#716563
 Solvent = DMSO-D6
 Creation_time = 23-JUN-2013 12:29:52
 Revision_time = 23-JUN-2013 21:08:54
 Current_time = 23-JUN-2013 21:09:11

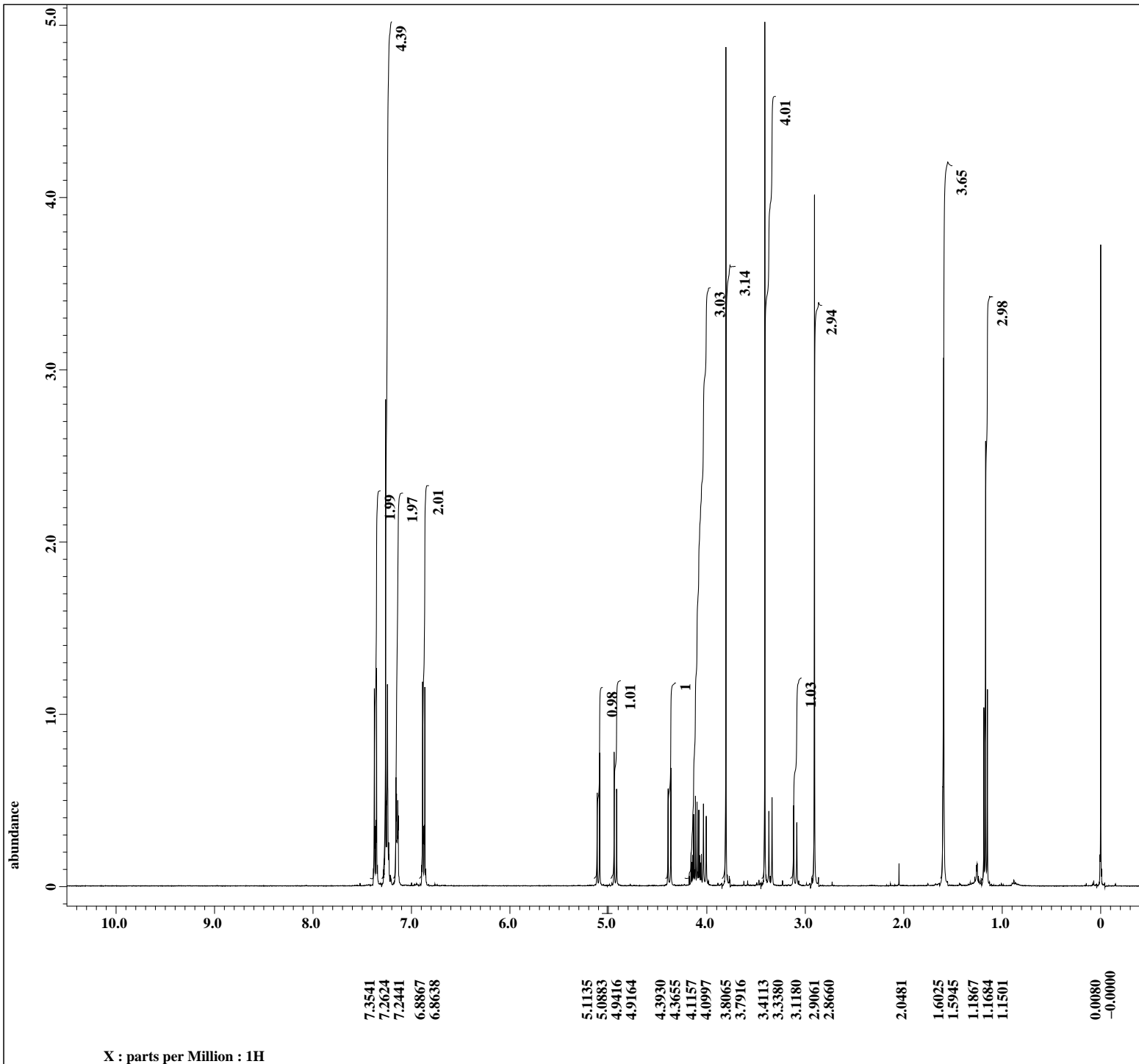
Comment = single pulse decouple
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 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 = 202
 Total_scans = 202

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 = 20.8[dB]
 Irr_atn_noe = 20.8[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 = 20.3[dC]



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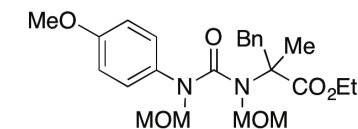
X : parts per Million : 1H

Filename = TY-12-101proton-4.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = S#484816
 Solvent = CHLOROFORM-D
 Creation_time = 13-APR-2013 06:01:58
 Revision_time = 16-APR-2013 18:01:29
 Current_time = 16-APR-2013 18:02:01

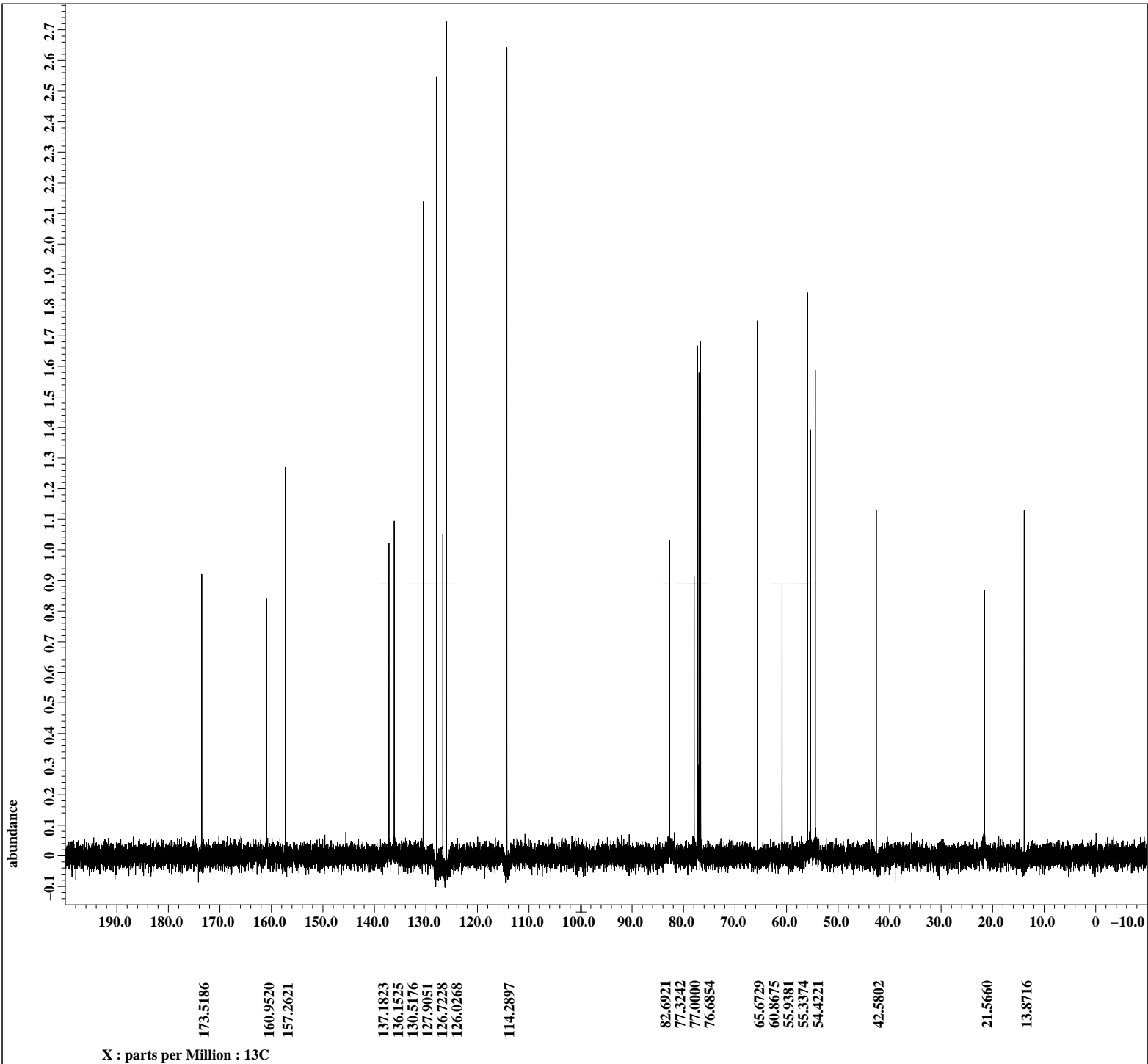
Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 2.18365952[s]
 X_domain = 1H
 X_freq = 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 = 8
 Total_scans = 8

X_90_width = 10.75[us]
 X_acq_time = 2.18365952[s]
 X_angle = 45[deg]
 X_atn = 0.3[dB]
 X_pulse = 5.375[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 38
 Relaxation_delay = 5[s]
 Repetition_time = 7.18365952[s]
 Temp_get = 18.6[dC]



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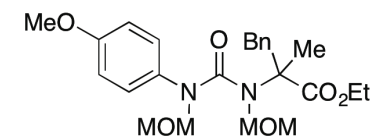


Filename = TY-12-101 13C comp-4.
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = 101
 Solvent = CHLOROFORM-D
 Creation_time = 13-APR-2013 06:15:19
 Revision_time = 16-APR-2013 18:03:32
 Current_time = 16-APR-2013 18:03:50

Comment = single pulse decouple
 Data_format = 1D_REAL
 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 = 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 = 64
 Total_scans = 64

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 = 20.8[dB]
 Irr_atn_noe = 20.8[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 = 18.9[dC]



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