

Supporting Information

Tin-Free Giese Reaction and the Related Radical Carbonylation Using Alkyl Iodides and Cyanoborohydrides

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General information. ^1H NMR spectra were recorded with a JEOL JMN-ECP500 (500 MHz) spectrometer in CDCl_3 . Chemical shifts are reported in parts per million (δ) downfield from internal TMS at 0.00. ^{13}C NMR spectra were recorded with a JEOL JMN-ECP500 (125 MHz) spectrometer and referenced to the solvent peak at 77.00 ppm. Infrared spectra were obtained on a JASCO FT/IR-4100 spectrometer; absorptions are reported in reciprocal centimeters. GC analyses were performed with a Shimadzu GC-18A gas chromatography equipped with column DB-1. GC-MS analyses were performed with a Shimadzu GCMS-QP5050 mass spectrometer. High resolution mass spectra were recorded with a JEOL MS700 spectrometer. The products were purified by flash chromatography on silica gel (Nacalai Tesque Inc. Silica Gel 60, 230-400 mesh) and, if necessary, were further purified by recycling preparative HPLC (Japan Analytical Industry Co. Ltd., LC-908 or LC-918) equipped with GPC columns (JAIGEL-1H + JAIGEL-2H columns) using CHCl_3 as eluent. Photolysis were carried out using a Pyrex round bottomed flask and using a 500 W Xenon short arc lamp (Ushio Co. Ltd., lamp house: SX-U1500XQ, Xenon short arc lamp: UXL-500SX, power supply: BA-X500). EtOH, MeOH, C_6H_6 , THF, acetone, Et_2O were dried and purified by standard distillation techniques. Alkyl iodides **1g**, **1i**, and **1k** were prepared from the corresponding alcohol.¹ **1f** and **1j** were prepared from the corresponding bromides with sodium iodide in dry acetone.² **1h** was prepared from 4-bromobenzyl alcohol and 1,5-diiodopentane by Williamson method using sodium hydride. **2g** was prepared via 1-undecen-3-ol, which was obtained by Grignard reaction of n-octylmagnesium bromide with acrolein, followed by Jones oxidation. Alkenes **2a**, **2b**, **2c**, **2d**, **2e**, and **2f** were distilled prior to use. Other reagents were commercially available and used without further purification.

References

- [1] (a) Olah, G. A.; Narang, S. C.; Gupta, B. G. B.; Malhotra, R. *J. Org. Chem.* **1979**, *44*, 1247; (b) Garcia-Fandiño, R.; Codesido, E. M.; Sobarzo-Sánchez, E.; Castedo, L.; Granja, J. R. *Org. Lett.* **2004**, *6*, 193.
- [2] Daub, G. H.; Castle, R. N.; *J. Org. Chem.* **1954**, *19*, 1571.

Typical Procedure A: Ethyl Undecanoate (**3a**).

A magnetic stirring bar, 1-iodooctane (**1a**) (239.5 mg, 1.0 mmol), ethyl acrylate (**2a**) (150.0 mg, 1.5 mmol), NaBH_3CN (311.7 mg, 5.0 mmol) and methanol (2.0 mL) were placed in a Pyrex 10 mL round-bottomed flask and the mixture was irradiated by Xenon arc lamp (500

W) with stirring for 3 h under argon. Saturated ammonium chloride aqueous solution (1 mL) was added to the reaction mixture. The mixture was poured into water (20 mL) and extracted with Et₂O (3 x 20 mL). The organic layer was washed with brine, and dried over Na₂SO₄, then filtered and concentrated in vacuo. The residue was separated by flash chromatography on silica gel (gradient from hexane/Et₂O = 20/1 to hexane/Et₂O = 10/1). The major fraction contained **3a** (160.7 mg, 75%). The minor fraction contained 2-nonylglutarate (**4a**) (28.8 mg, 9%).

Typical Procedure B: Methyl 4-Oxododecanoate (3i).

A magnetic stirring bar, AIBN (8.7 mg, 0.053 mmol), *n*-Bu₄NBH₃CN (721.5 mg, 2.56 mmol), **1a** (120.1 mg, 0.5 mmol), **2d** (170.1 mg, 1.98 mmol), and methanol (30 mL) were placed in a 100-mL stainless steel autoclave. The autoclave was closed, purged three times with carbon monoxide, pressurized with 91 atm of CO, and then heated at 80 °C for 19 h. Excess CO was discharged at room temperature. Saturated ammonium chloride aqueous solution (10 mL) was added to the reaction mixture. The mixture was poured into water (50 mL) and extracted with Et₂O (3 x 50 mL). The organic layer was washed with brine, and dried over Na₂SO₄, then filtered and concentrated in vacuo. The residue was purified by flash chromatography on silica gel (hexane/AcOEt = 30/1) to give **3i** (70.3 mg, 62%).

Typical Procedure C: 3-Tridecanone (3m).

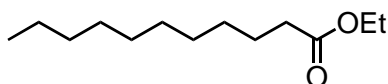
A magnetic stirring bar, *n*-Bu₄NBH₃CN (311.7 mg, 1.5 mmol), **1a** (118.6 mg, 0.49 mmol), **2f** (127.1 mg, 1.5 mmol), and methanol (1.0 mL) were placed in a Pyrex 10 mL round-bottomed flask and the mixture was irradiated by Xenon arc lamp (500 W) with stirring for 6 h under argon. The solvent was removed under reduced pressure. The residue was purified by flash

chromatography on silica gel (hexane/Et₂O = 20/1) to give **3m** (64.9 mg, 67%).

Deuterium Labeling Experiment.

A magnetic stirring bar, **1a** (121.6 mg, 0.51 mmol), **2a** (105.2 mg, 1.1 mmol), NaBD₃CN (162.3 mg, 4.8 mmol, 96 atom% D) and methanol (1.0 mL) were placed in a pyrex 10 mL round-bottomed flask and irradiated by Xenon arc lamp (500 W) with stirring for 3 h under argon. The reaction mixture was poured into water (20 mL) and extracted with Et₂O (20 mL x 3). The organic layer was washed with brine, and dried over Na₂SO₄, then filtered and concentrated in vacuo. The residue was purified by flash chromatography on silica gel (hexane/ Et₂O = 20/1) to give **3 a-d** (46.6 mg). Deuterium incorporation was determined by ¹H NMR.

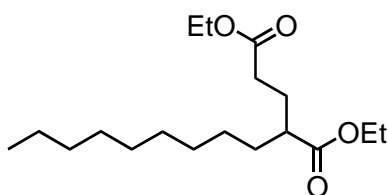
Ethyl Undecanoate (3a).



Colorless oil, (*R*_f = 0.38, hexane/ Et₂O = 10/1); ¹H NMR (CDCl₃, 500 MHz) δ 0.88 (t, *J* = 6.9 Hz, 3H), 1.16-1.38 (m, 17H), 1.55-1.68 (m, 2H), 2.29 (t, *J* = 7.6 Hz, 2H), 4.12 (q, *J* = 7.2 Hz, 2H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.05, 14.21, 22.64, 24.96, 29.12, 29.24, 29.27, 29.43, 29.52, 31.86, 34.36, 60.08, 173.85; IR (neat) 1739 cm⁻¹; MS (EI) *m/z* (rel intensity) 214 (M⁺, 9), 169 (24), 101 (69), 88 (100), 73 (51), 70(49), 61 (37), 60 (35), 57 (33), 55 (48).

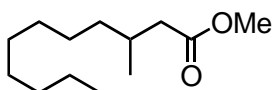
This compound is previously known: Sim, T. C.; Joung, M.; Yoon, N. M. *J. Org. Chem.* **1997**, *62*, 2357.

Diethyl 2-Nonylglutarate (4a).



Colorless oil. ($R_f = 0.23$, hexane/Et₂O = 10/1); ¹H NMR (CDCl₃, 500 MHz) δ 0.88 (t, $J = 6.9$ Hz, 3H), 1.16-1.38 (m, 20H), 1.40-1.49 (m, 1H), 1.57-1.66 (m, 1H), 1.78-1.94 (m, 2H), 2.22-2.41 (m, 3H), 3.98-4.20 (m, 4H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.07, 14.19, 14.28, 22.64, 27.18, 29.26, 29.41, 29.46, 29.49, 31.85, 32.06, 32.30, 44.82, 60.19, 60.34, 173.06, 175.69 (Two signals accidentally superposed each other.); IR (neat) 1736 cm⁻¹; MS (EI) m/z (rel intensity) 269 (M⁺-OEt, 48), 198 (42), 188 (59), 152 (47), 142 (100), 114 (83), 101 (37), 98 (32), 97 (37), 88 (47), 83 (53), 81 (36), 55 (90); HRMS calcd for C₁₈H₃₄O₄ (M⁺) 314.2457, found 314.2452.

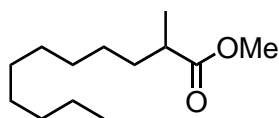
Methyl 3-Methylundecanoate (3b).



Yellow oil, ($R_f = 0.2$, hexane/ Et₂O = 20/1); ¹H NMR (CDCl₃, 500 MHz) δ 0.88 (t, $J = 6.9$ Hz, 3H), 0.93 (d, $J = 6.4$ Hz, 3H), 1.13-1.35 (m, 14H), 1.88-1.99 (m, 1H), 2.10 (dd, $J = 14.7, 7.8$ Hz, 1H), 2.30 (dd, $J = 14.7, 6.0$ Hz, 1H), 3.66 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.07, 19.72, 22.66, 26.89, 29.28, 29.56, 29.73, 30.33, 31.88, 36.72, 41.66, 51.29, 173.78; IR (neat) 1742 cm⁻¹; MS (EI) m/z (rel intensity) 214 (M⁺, 6), 183 (14), 157 (11), 101 (47), 74 (100), 69 (28), 59 (26).

This compound is previously known: Sim, T. C.; Joung, M.; Yoon, N. M. *J. Org. Chem.* **1997**, *62*, 2357.

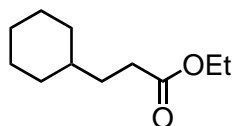
Methyl 2-Methylundecanoate (3c).



Colorless oil, ($R_f = 0.40$, hexane/ Et₂O = 20/1); ¹H NMR (CDCl₃, 500 MHz) δ 0.88 (t, $J = 7.1$ Hz, 3H), 1.14 (d, $J = 6.9$ Hz, 3H), 1.20-1.33 (m, 14H), 1.34-1.45 (m, 1H), 1.59-1.70 (m, 1H), 2.43 (sext, $J = 7.0, 7.0$ Hz, 1H), 3.67 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.07, 17.03, 22.66, 27.23, 29.28, 29.47, 29.49, 29.53, 31.88, 33.82, 39.44, 51.39, 177.37; IR (neat) 1741 cm⁻¹; MS (EI) m/z (rel intensity) 214 (M^+ , 5), 157 (17), 143 (13), 101 (57), 89 (13), 88 (100), 71 (12), 69 (23), 59 (23), 57 (48), 55 (40).

This compound is previously known: Sim, T. C.; Joung, M.; Yoon, N. M. *J. Org. Chem.* **1997**, 62, 2357.

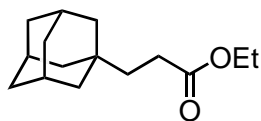
Ethyl 3-Cyclohexylpropionate (3d).



Colorless oil, ($R_f = 0.32$, hexane/ Et₂O = 10/1); ¹H NMR (CDCl₃, 500 MHz) δ 0.81-0.91 (m, 2H), 1.05-1.29 (m, 7H), 1.48-1.55 (m, 2H), 1.60-1.74 (m, 5H), 2.30 (t, $J = 8.0$ Hz, 2H), 4.12 (q, $J = 7.0$ Hz, 2H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.00, 26.03, 26.34, 31.69, 32.17, 32.78, 37.05, 59.84, 173.76; IR (neat) 1735 cm⁻¹; MS (EI) m/z (rel intensity) 184 (M^+ , 1), 139 (14), 121 (20), 101 (100), 88 (81), 73 (42), 55 (95).

This compound is previously known: Shukla, P.; Hsu, Y-C.; Cheng, C-H. *J. Org. Chem.* **2006**, 71, 655.

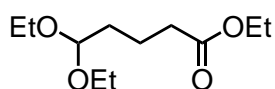
Ethyl 3-(1-Adamantyl)propionate (3e).



Colorless oil, (R_f = 0.30, hexane/ Et₂O = 10/1); ¹H NMR (CDCl₃, 500 MHz) δ 1.26 (t, J = 7.1 Hz, 3H), 1.37-1.49 (m, 7H), 1.58-1.65 (m, 3H), 1.66-1.74 (m, 3H), 1.95 (m, 3H), 2.22-2.28 (m, 2H) 4.12 (q, J = 7.2 Hz, 2H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.14, 28.08, 28.37, 31.80, 36.97, 38.86, 41.93, 60.08, 174.55; IR (neat) 1738 cm⁻¹; MS (EI) m/z (rel intensity) 236 (M⁺, 34), 191 (33), 135 (100), 107 (39), 93 (51), 91 (40), 79 (54), 67 (31).

This compound is previously known: Yamazaki, O.; Togo, H.; Matubayashi, S.; Yokoyama, M. *Tetrahedron* **1999**, 55, 3735.

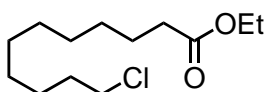
Ethyl 5,5-Diethoxypentanoate (3f).



Colorless oil, (R_f = 0.48, hexane/AcOEt = 5/1); ¹H NMR (CDCl₃, 500 MHz) δ 1.20 (t, J = 7.1 Hz, 6H), 1.25 (t, J = 7.1 Hz, 3H), 1.61-1.74 (m, 4H), 2.33 (t, J = 7.3 Hz, 2H), 3.45-3.53 (m, 2H), 3.61-3.68 (m, 2H), 4.13 (q, J = 7.2 Hz, 2H), 4.49 (t, J = 5.5 Hz, 1H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.06, 15.14, 20.09, 32.80, 33.81, 60.03, 60.84, 102.37, 173.25; IR (neat) 1737 cm⁻¹; MS (EI) m/z (rel intensity) 173 (M⁺-OEt, 6), 127 (11), 103 (11), 99 (16), 97 (32), 85 (100), 75 (11), 73 (13), 70 (22), 57 (46).

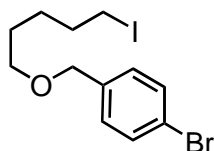
This compound is previously known. See the reference; Paolobelli, A. B.; Ruzziconi, R. *J. Org. Chem.* **1996**, 61, 6434.

Ethyl 11-Chloroundecanoate (3g).



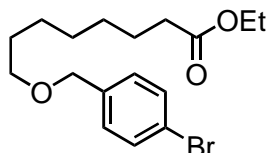
Colorless oil, ($R_f = 0.20$, hexane/ Et₂O = 10/1); ¹H NMR (CDCl₃, 500 MHz) δ 1.20-1.34 (m, 10 H), 1.25 (t, $J = 7.1$ Hz, 3H), 1.36-1.46 (m, 2H), 1.56-1.66 (m, 2H), 1.76 (quint, $J = 7.1$ Hz, 2H), 2.28 (t, $J = 7.6$ Hz, 2H), 3.52 (t, $J = 6.7$ Hz, 2H), 4.12 (q, $J = 7.0$ Hz, 2H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.15, 24.86, 26.76, 28.75, 29.00, 29.10, 29.21, 29.27, 32.54, 34.24, 44.98, 60.00, 173.67; IR (neat) 1737 cm⁻¹; MS (EI) m/z (rel intensity) 248 (M⁺, 1), 205 (12), 203 (21), 115 (14), 101 (64), 88 (100), 83 (22), 73 (41), 70 (47), 60 (45), 57 (27), 55 (68); HRMS calcd for C₁₃H₂₅³⁵ClO₂ (M⁺) 248.1543, found 248.1546.

5-(4-Bromobenzyloxy)pentyl iodide (1h).



Pale yellow oil. ($R_f = 0.48$, hexane/ Et₂O = 10/1); ¹H NMR (CDCl₃, 500 MHz) δ 1.45-1.53 (m, 2H), 1.59-1.68 (m, 2H), 1.85 (quint, $J = 7.2$ Hz, 2H), 3.19 (t, $J = 6.9$ Hz, 2H), 3.46 (t, $J = 6.4$ Hz, 2H), 4.44 (s, 2H), 7.18-7.24 (m, 2H), 7.44-7.50 (m, 2H); ¹³C NMR (CDCl₃, 125 MHz) δ 6.82, 27.20, 28.62, 33.24, 70.12, 72.12, 121.32, 129.18, 131.42, 137.52; IR (neat) 2931, 1641 cm⁻¹; MS (EI) m/z (rel intensity) 382 (M⁺, 1), 171 (69), 169 (100), 91 (14), 90 (24), 89 (24), 63 (11); HRMS calcd for C₁₂H₁₆⁷⁹BrIO (M⁺) 381.9429, found 381.9426.

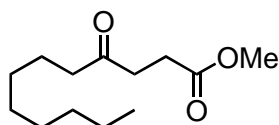
Ethyl 8-[(4-bromophenyl)methoxy]octanoate (3h).



Colorless oil, ($R_f = 0.30$, hexane/ Et₂O = 5/1); ¹H NMR (CDCl₃, 500 MHz) δ 1.25 (t, $J = 7.1$ Hz, 3H), 1.28-1.40 (m, 6H), 1.55-1.66 (m, 4H), 2.28 (t, $J = 7.6$ Hz, 2H), 3.44 (t, $J = 6.6$ Hz, 2H), 4.12 (q, $J = 7.2$, 2H), 4.43 (s, 2H), 7.16-7.24 (m, 2H), 7.42-7.48 (m, 2H); ¹³C NMR

(CDCl₃, 125 MHz) δ 14.14, 24.76, 25.87, 28.92, 28.95, 29.53, 34.18, 59.99, 70.4, 71.92, 121.13, 129.09, 131.28, 137.61, 173.62; IR (neat) 1735 cm⁻¹; MS (EI) m/z (rel intensity) 356 (M⁺, 2), 277 (18), 207 (15), 185 (13), 171 (100), 169 (74), 125 (42), 101 (58), 97 (34), 90 (31), 88 (38), 55 (50); HRMS calcd for C₁₇H₂₅⁷⁹BrO₃ (M⁺) 356.0987, found 356.0991.

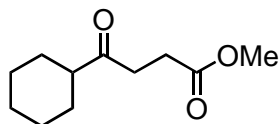
Methyl 4-Oxododecanoate (3i).



Yellow oil; (R_f = 0.25, hexane/ AcOEt = 8/1); ¹H NMR (CDCl₃, 500 MHz) δ 0.88 (t, J = 6.9 Hz, 3H), 1.19-1.33 (m, 10H), 1.53-1.63 (m, 2H), 2.44 (t, J = 7.6 Hz, 2H), 2.59 (t, J = 6.7 Hz, 2H), 2.72 (t, J = 6.7, 2H), 3.68 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.01, 22.57, 23.76, 27.66, 29.06, 29.14, 29.29, 31.75, 36.95, 42.75, 51.67, 173.24, 209.04.

This compound is previously known: Kishimoto, Y.; Ikariya, T. *J. Org. Chem.* **2000**, 65, 7656.

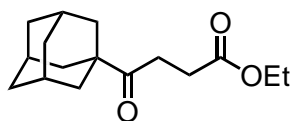
Methyl 4-Oxo-4-cyclohexylbutanoate (3j).



Colorless oil; (R_f = 0.25, hexane/ AcOEt = 5/1); ¹H NMR (CDCl₃, 500 MHz) δ 1.10-1.42 (m, 5H), 1.60-1.70 (m, 1H), 1.72-1.81 (m, 2H), 1.82-1.92 (m, 2H), 2.34-2.42 (m, 1H), 2.58 (t, J = 6.6 Hz, 2H), 2.76 (t, J = 6.4 Hz, 2H), 3.67 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 25.53, 25.75, 27.62, 28.38, 34.90, 50.62, 51.62, 173.29, 211.88.

This compound is previously known: Ryu, I.; Kusano, K.; Yamazaki, H. *J. Org. Chem.* **1991**, 56, 5003.

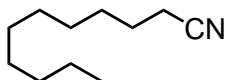
Ethyl 4-Oxo-4-(1-adamantyl)butanoate (3k).



Colorless oil; (R_f = 0.38, hexane/ AcOEt = 5/1); ^1H NMR (CDCl_3 , 500 MHz) δ 1.25 (t, J = 7.1 Hz, 3H), 1.64-1.72 (m, 3H), 1.72-1.78 (m, 3H), 1.82-1.86 (m, 6H), 2.01-2.08 (m, 3H), 2.55 (t, J = 6.7 Hz, 2H), 2.77 (t, J = 6.3 Hz, 2H), 4.12 (q, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 125 MHz) δ 14.12, 27.88, 28.50, 30.98, 36.48, 38.22, 46.07, 60.40, 172.99, 213.66.

This compound is previously known: Cai, Y.; Roberts, B. P.; Tocher, D. A.; Barnett, S. A. *Org. Biomol. Chem.* **2004**, 2, 2517.

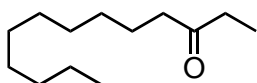
Undecanenitrile (3l).



Colorless oil, (R_f = 0.18, hexane/ Et_2O = 20/1); ^1H NMR (CDCl_3 , 500 MHz) δ 0.88 (t, J = 6.9 Hz, 3H), 1.21-1.37 (m, 12H), 1.39-1.49 (m, 2H), 1.66 (quint, J = 7.5 Hz, 2H), 2.33 (t, J = 7.3 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 13.98, 17.00, 22.55, 25.28, 28.56, 28.66, 29.14, 29.20, 29.35, 31.75, 119.72; IR (neat) 2926, 2247 cm^{-1} ; MS (EI) m/z (rel intensity) 152 ($\text{M}^+ - \text{CH}_3$, 4), 138 (21), 124 (53), 110 (78), 96 (94), 82 (100), 69 (82), 57 (89), 55 (97).

This compound is previously known: Blay, G.; Cardona, L.; Garcia, B.; Lahoz, L.; Pedro, J. R. *Tetrahedron* **1996**, 52, 8611.

3-Tridecanone (3m).

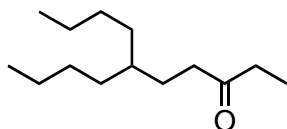


White solid, Mp < 30°C; (R_f = 0.30, hexane/ Et_2O = 5/1); ^1H NMR (CDCl_3 , 500 MHz) δ 0.88 (t,

$J = 6.9$ Hz, 3H), 1.05 (t, $J = 7.3$ Hz, 3H), 1.20-1.34 (m, 14H), 1.52-1.61 (m, 2H), 2.39 (t, $J = 7.3$ Hz, 2H), 2.42 (q, $J = 7.3$, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 7.83, 14.08, 22.66, 23.96, 29.27, 29.29, 29.40, 29.46, 29.55, 31.88, 35.83, 42.44, 211.96; IR (neat) 1718 cm^{-1} ; MS (EI) m/z (rel intensity) 198 (M^+ , 2), 169 (47), 95 (17), 85 (45), 72 (100), 57 (97), 55 (27).

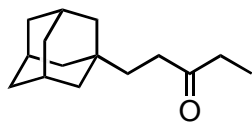
This compound is previously known: Zhang, D.; Ready, J. M. *Org. Lett.* **2005**, 7, 5681.

6-Butyl-3-decanone (3n).



Colorless oil, ($R_f = 0.43$, hexane/ $\text{Et}_2\text{O} = 10/1$); ^1H NMR (CDCl_3 , 500 MHz) δ 0.89 (t, $J = 6.9$ Hz, 6H), 1.05 (t, $J = 7.3$ Hz, 3H), 1.16-1.40 (m, 14H), 1.49-1.56 (m, 2H), 2.34-2.39 (m, 2H), 2.40 (q, $J = 7.3$ Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 7.87, 14.09, 23.07, 27.59, 28.81, 33.09, 35.80, 37.00, 39.81, 212.18; IR (neat) 1717 cm^{-1} ; MS (EI) m/z (rel intensity) 183 ($\text{M}^+ - \text{Et}$, 32), 165 (14), 140 (33), 109 (26), 85 (89), 72 (64), 57 (100); HRMS calcd for $\text{C}_{14}\text{H}_{28}\text{O}$ (M^+) 212.2140, found 212.2142.

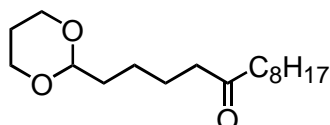
5-Adamantyl-3-pentanone (3o).



Pale yellow oil, ($R_f = 0.18$, hexane/ $\text{Et}_2\text{O} = 15/1$); ^1H NMR (CDCl_3 , 500 MHz) δ 1.04 (t, $J = 7.4$ Hz, 3H), 1.30-1.37 (m, 2H), 1.41-1.48 (m, 6H), 1.57-1.64 (m, 3H), 1.66-1.74 (m, 3H), 1.90-1.97 (m, 3H), 2.31-2.37 (m, 2H), 2.42 (q, $J = 7.3$ Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 7.95, 28.61, 31.80, 35.81, 36.03, 37.08, 37.88, 42.17, 212.55; IR (neat) 1715 cm^{-1} ; MS (EI) m/z (rel intensity) 220 (M^+ , 3), 202 (28), 191 (67), 173 (37), 135 (100), 107 (21), 93 (39), 91

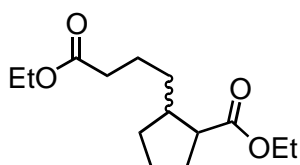
(30), 79 (48), 67 (25), 57 (41); HRMS calcd for C₁₅H₂₄O₁ (M⁺) 220.1827, found 220.1830.

1-(1,3-Dioxan-2-yl)-5-tridecanone (3p).



Colorless solid, Mp = 30–31 °C; (R_f = 0.35, hexane/AcOEt = 5/2); ¹H NMR (CDCl₃, 500 MHz) δ 0.88 (t, *J* = 7.1 Hz, 3H), 1.20-1.43 (m, 13H), 1.51-1.63 (m, 6H), 2.00-2.12 (m, 1H), 2.34-2.42 (m, 4H), 3.71-3.79 (m, 2H), 4.05-4.12 (m, 2H), 4.51 (t, *J* = 5.3 Hz, 1H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.02, 22.57, 23.52, 23.56, 23.82, 25.78, 29.07, 29.20, 29.31, 31.75, 34.86, 42.55, 42.77, 66.81, 102.01, 211.27; IR (neat) 1705 cm⁻¹; MS (EI) *m/z* (rel intensity) 284 (M⁺, 1), 128 (49), 110 (12), 87 (100), 57 (21); HRMS calcd for C₁₈H₃₂O₃ (M⁺) 284.2351, found 284.2350.

Ethyl 2-(Ethoxycarbonyl)cyclopentanebutanoate (3q).



Obtained as a *cis/trans*-isomer mixture in a 67/33 ratio, as determined by GC analysis of the crude reaction mixture. The *cis*- and *trans*-isomers of **3q** were separated using a preparative HPLC. *Cis*-isomer: Colorless oil, (R_f = 0.30, hexane/AcOEt = 10/1); ¹H NMR (CDCl₃, 500 MHz) δ 1.19-1.28 (m, 7H), 1.33-1.49 (m, 2H), 1.50-1.74 (m, 3H), 1.75-1.98 (m, 4H), 2.02-2.12 (m, 1H), 2.21-2.34 (m, 2H), 2.78-2.85 (m, 1H), 4.06-4.17 (m, 4H); ¹³C NMR (CDCl₃, 125 MHz) δ 14.21, 14.31, 23.77, 24.00, 28.35, 30.61, 30.97, 34.45, 43.44, 47.51, 59.84, 60.17, 173.57, 175.40; IR (neat) 1733 cm⁻¹; MS (EI) *m/z* (rel intensity) 211 (M⁺-OEt,

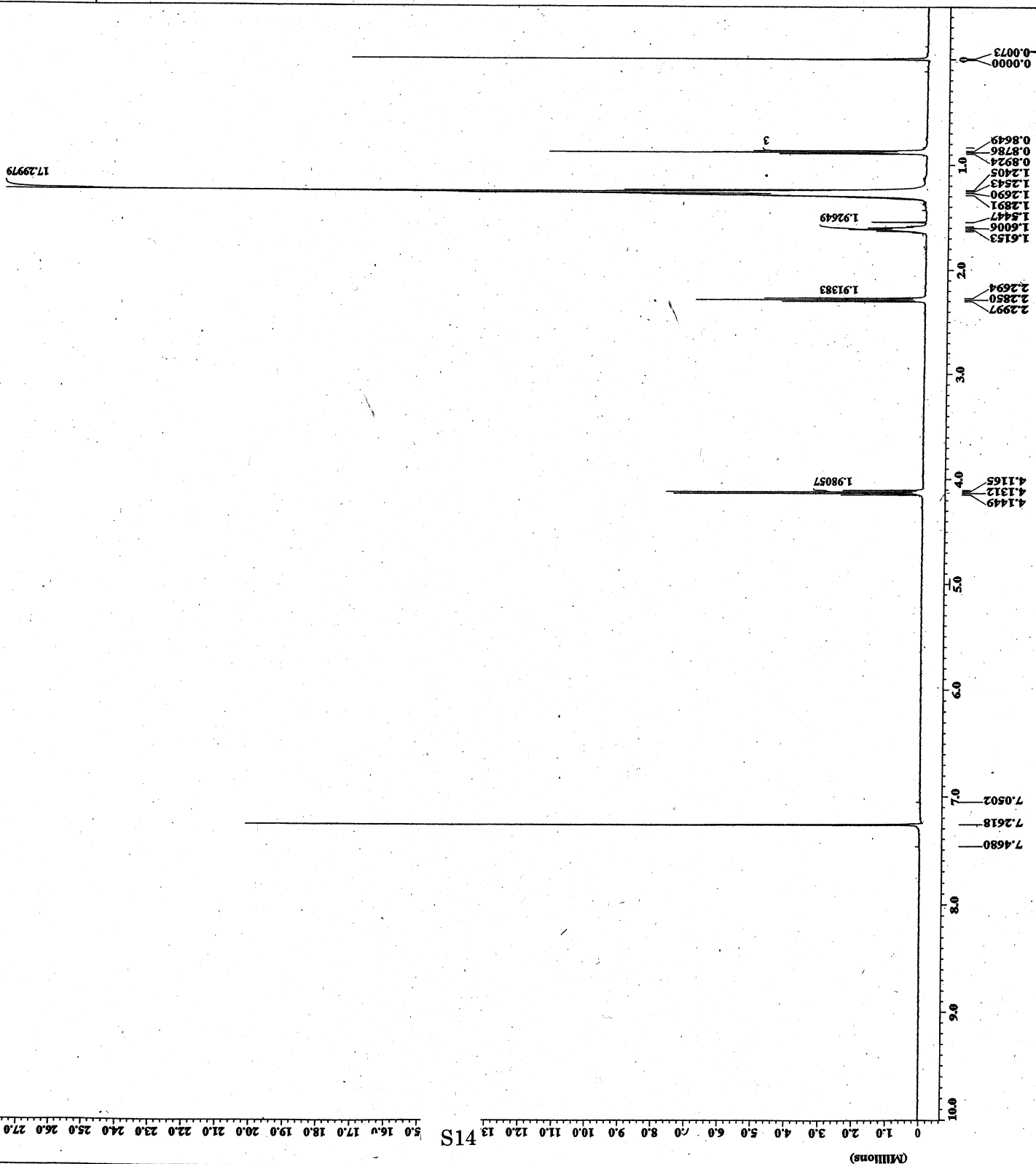
50), 183 (30), 169 (69), 136 (55), 119 (42), 114 (53), 95 (100), 73 (41), 67 (67), 55 (51); HRMS calcd for $C_{14}H_{24}O_4$ (M^+) 256.1675, found 256.1683. *Trans*-isomer: Colorless oil, (R_f = 0.30, hexane/AcOEt = 10/1); 1H NMR ($CDCl_3$, 500 MHz) δ 1.16-1.35 (m, 8H), 1.47-1.74 (m, 5H), 1.78-1.97 (m, 3H), 2.05-2.15 (m, 1H), 2.23-2.35 (m, 3H), 4.09-4.18 (m, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 14.23, 14.29, 23.64, 24.74, 30.33, 32.48, 34.45, 34.77, 44.01, 50.39, 60.18, 60.20, 173.64, 176.58; IR (neat) 1732 cm^{-1} ; MS (EI) m/z (rel intensity) 211 (M^+ -OEt, 57), 182 (58), 169 (61), 136 (100), 95 (97), 67 (72), 55 (48); HRMS calcd for $C_{14}H_{24}O_4$ (M^+) 256.1675, found 256.1669.

The configuration of **3q** was assigned by comparison with 1H NMR spectra of methyl 2-ethylcyclopentanecarboxylate, see: Canonne, P.; Plamondon, J. *Can. J. Chem.* **1989**, 67, 555.

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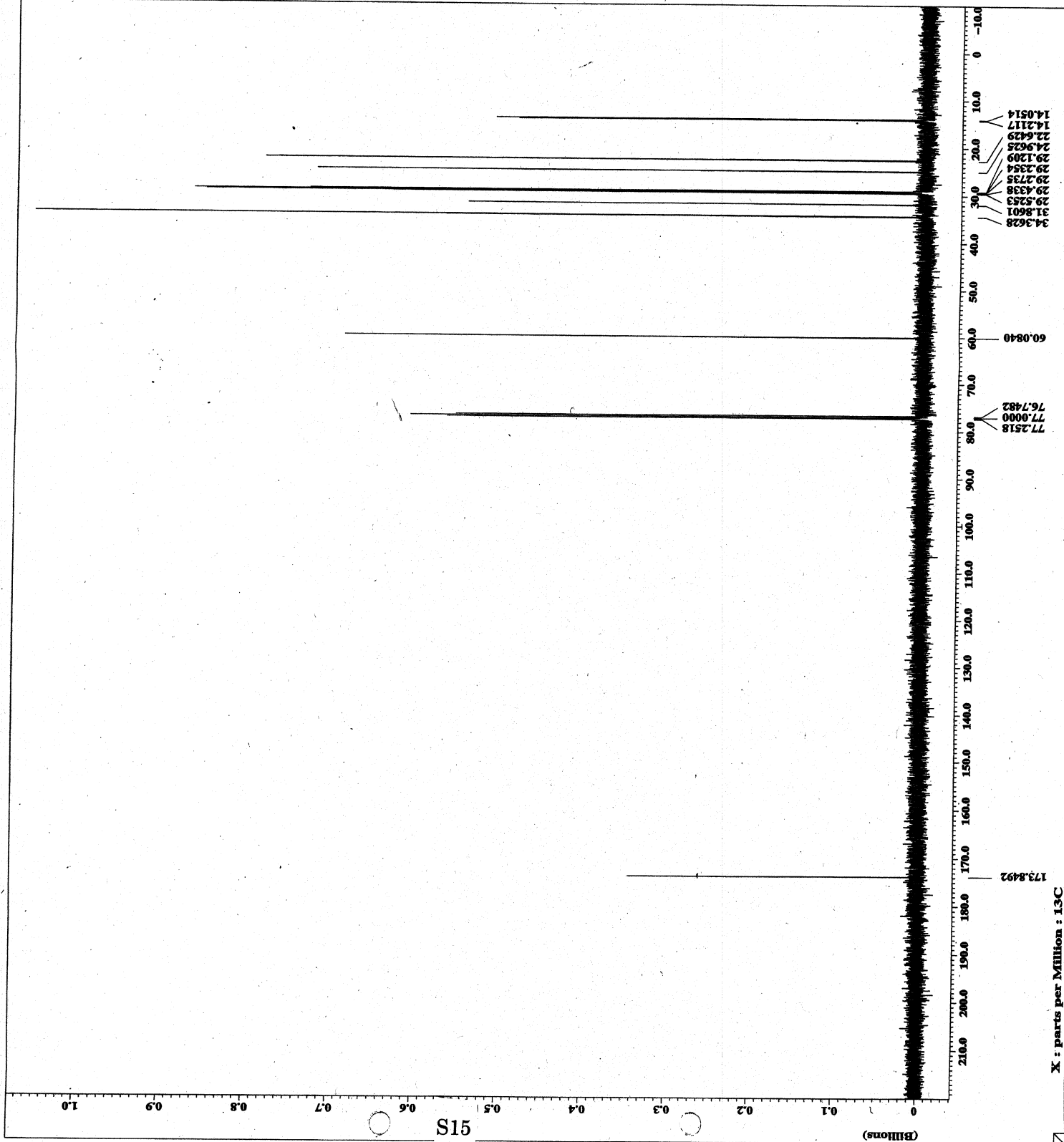
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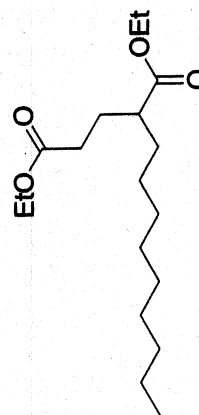
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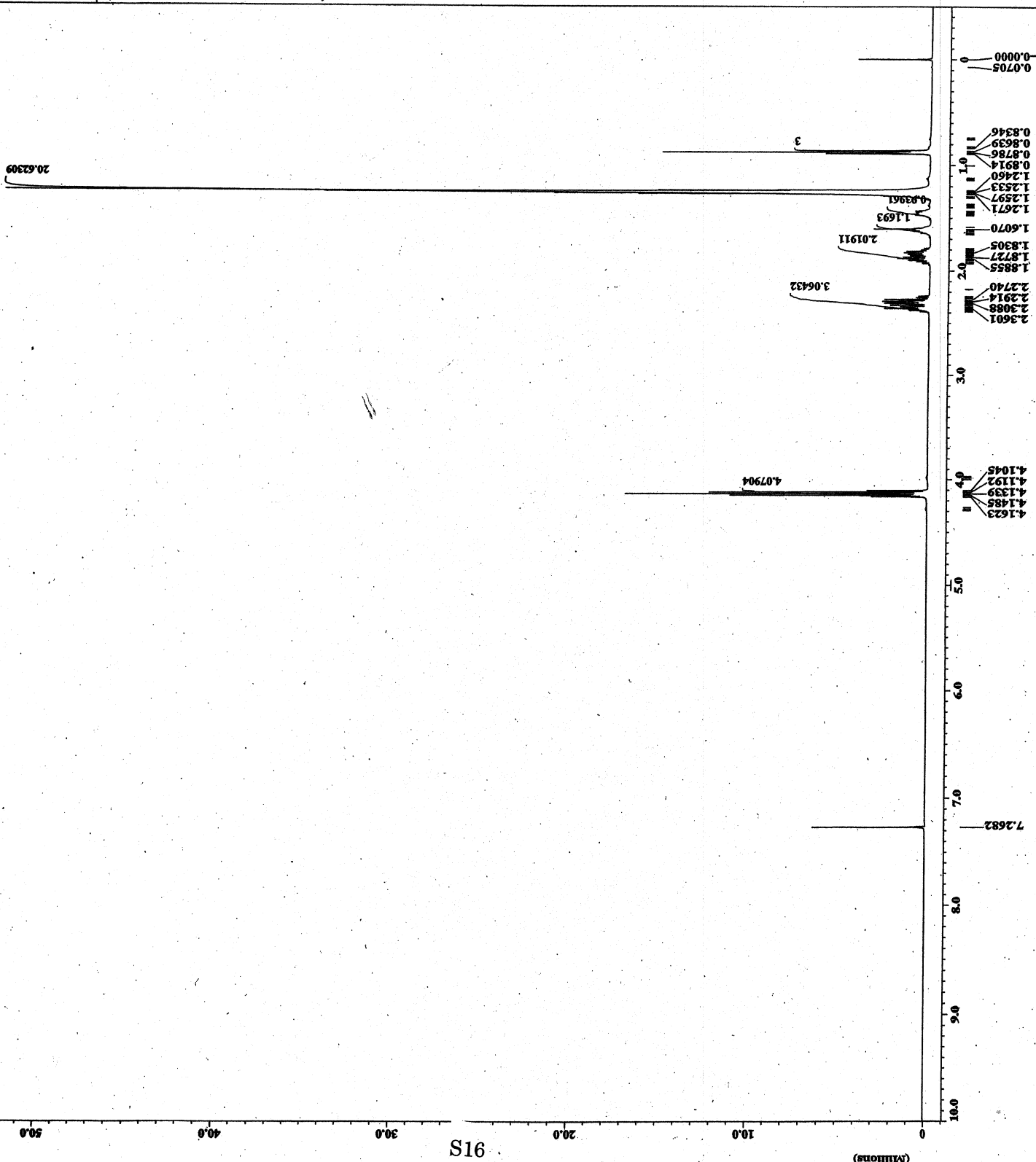
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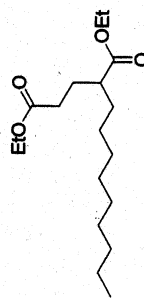
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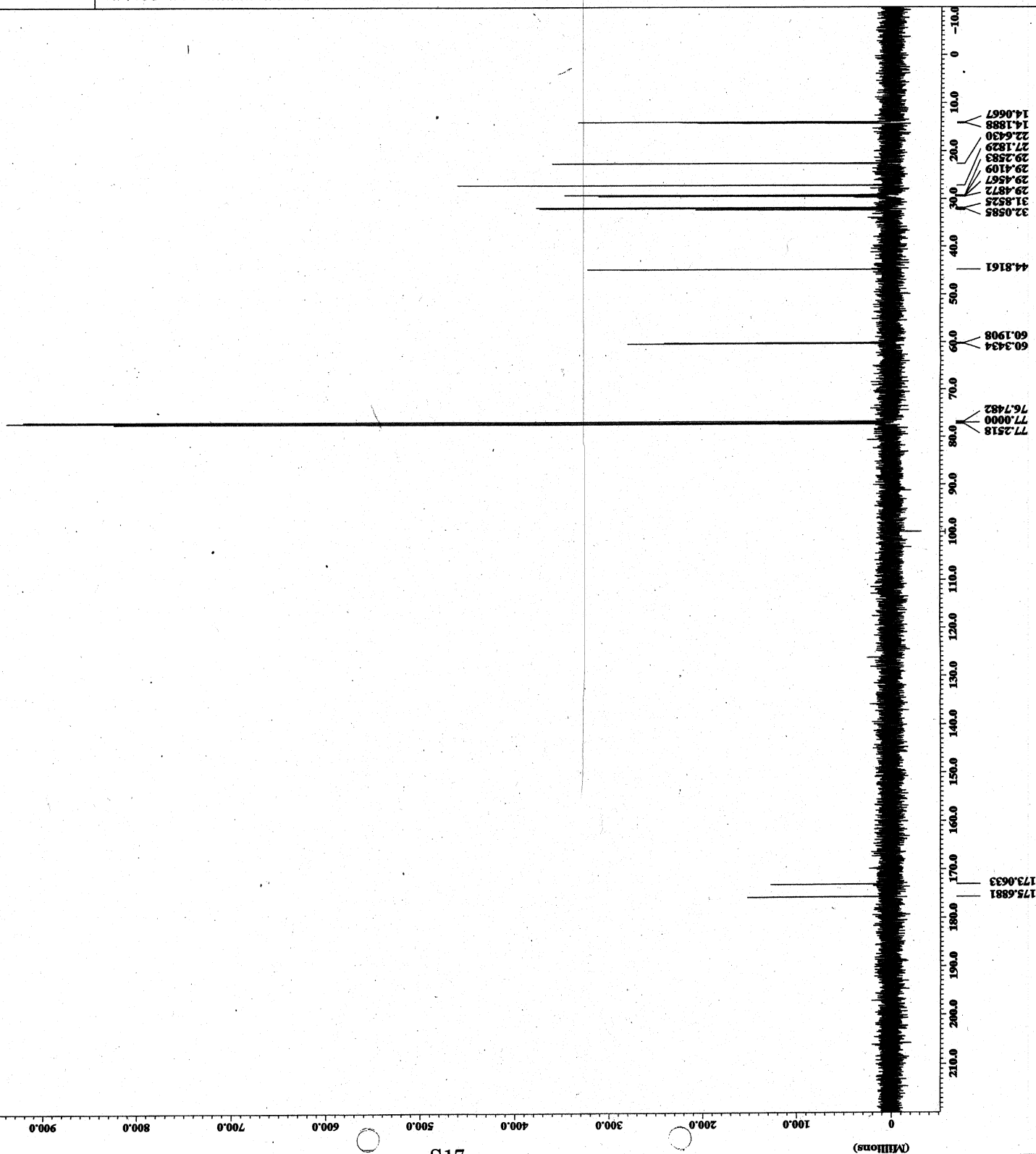
4a



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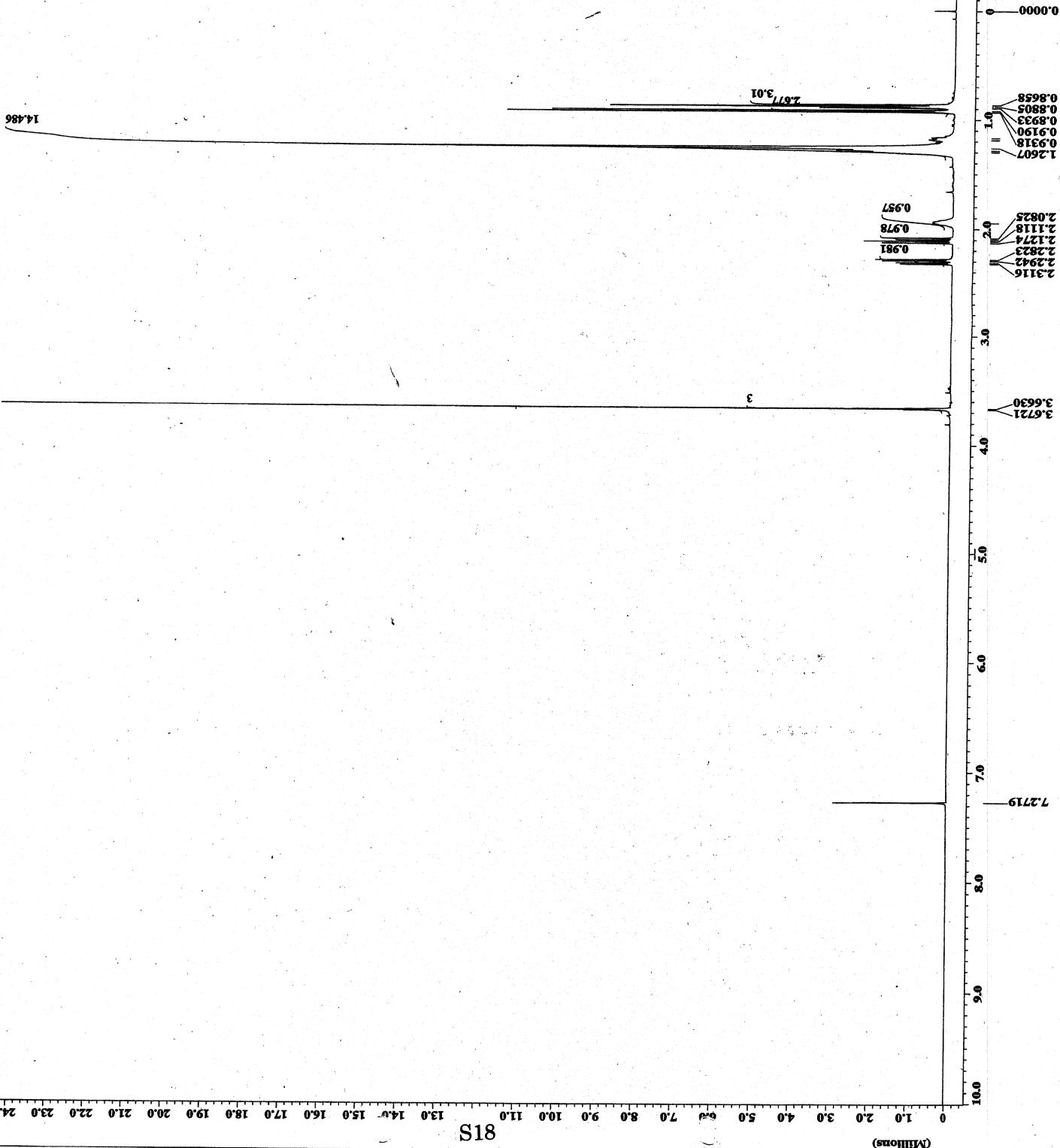
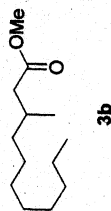


4a

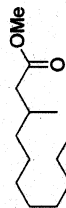


X : parts per Million : 13C

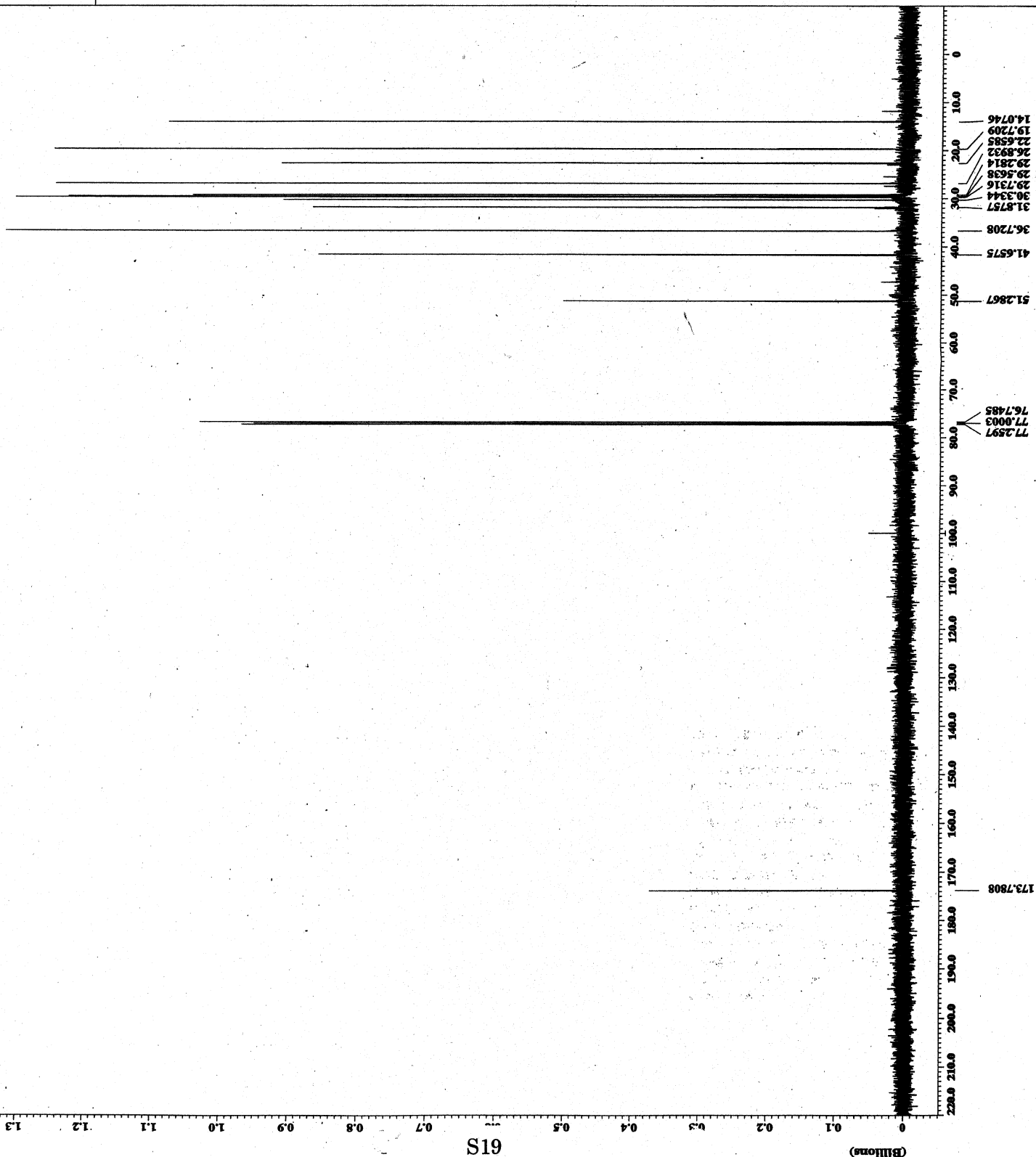
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 Dim 2 : 13C
 Dim Units : [ppm]
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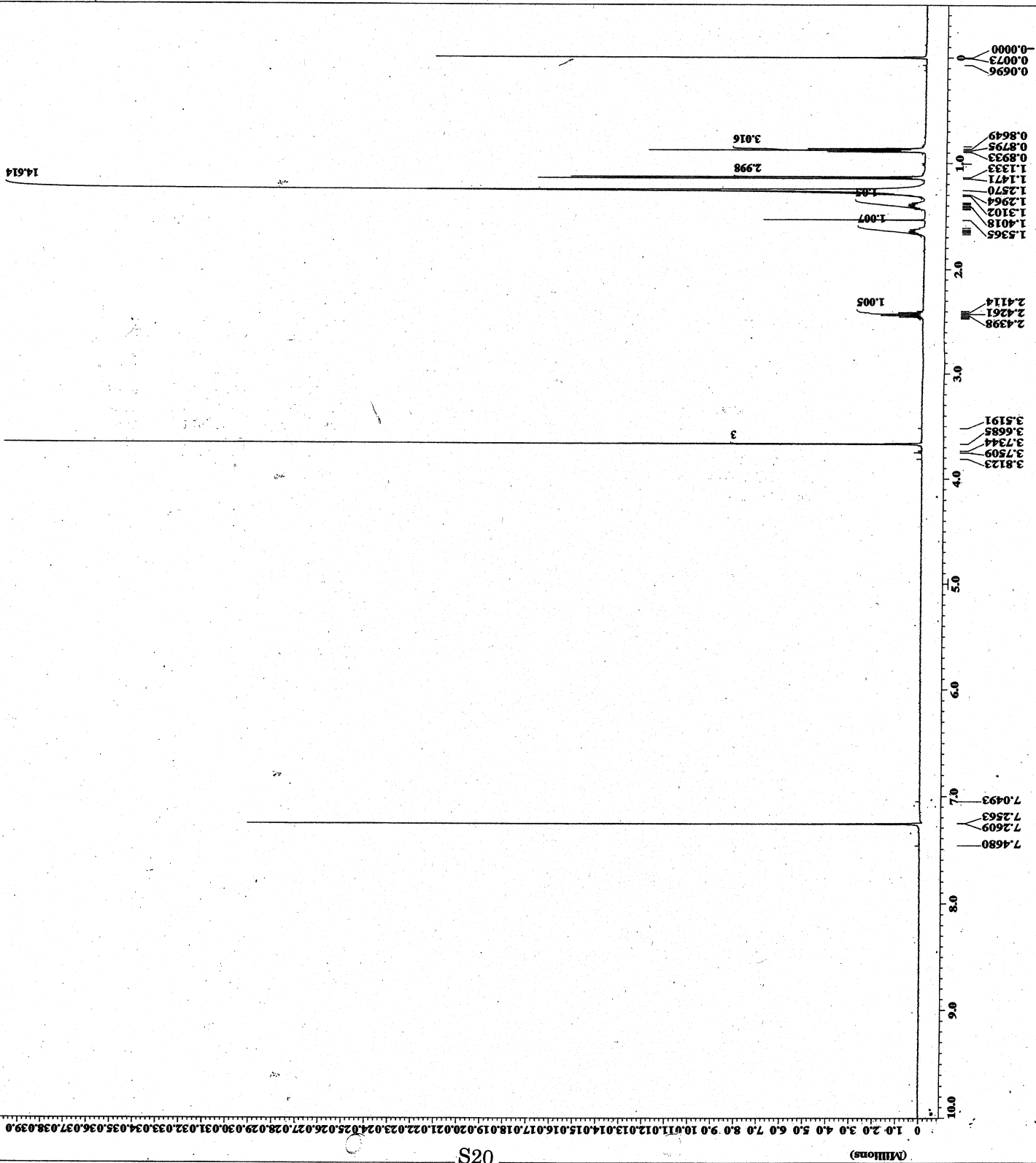
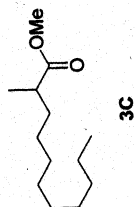
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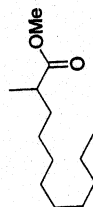
3b



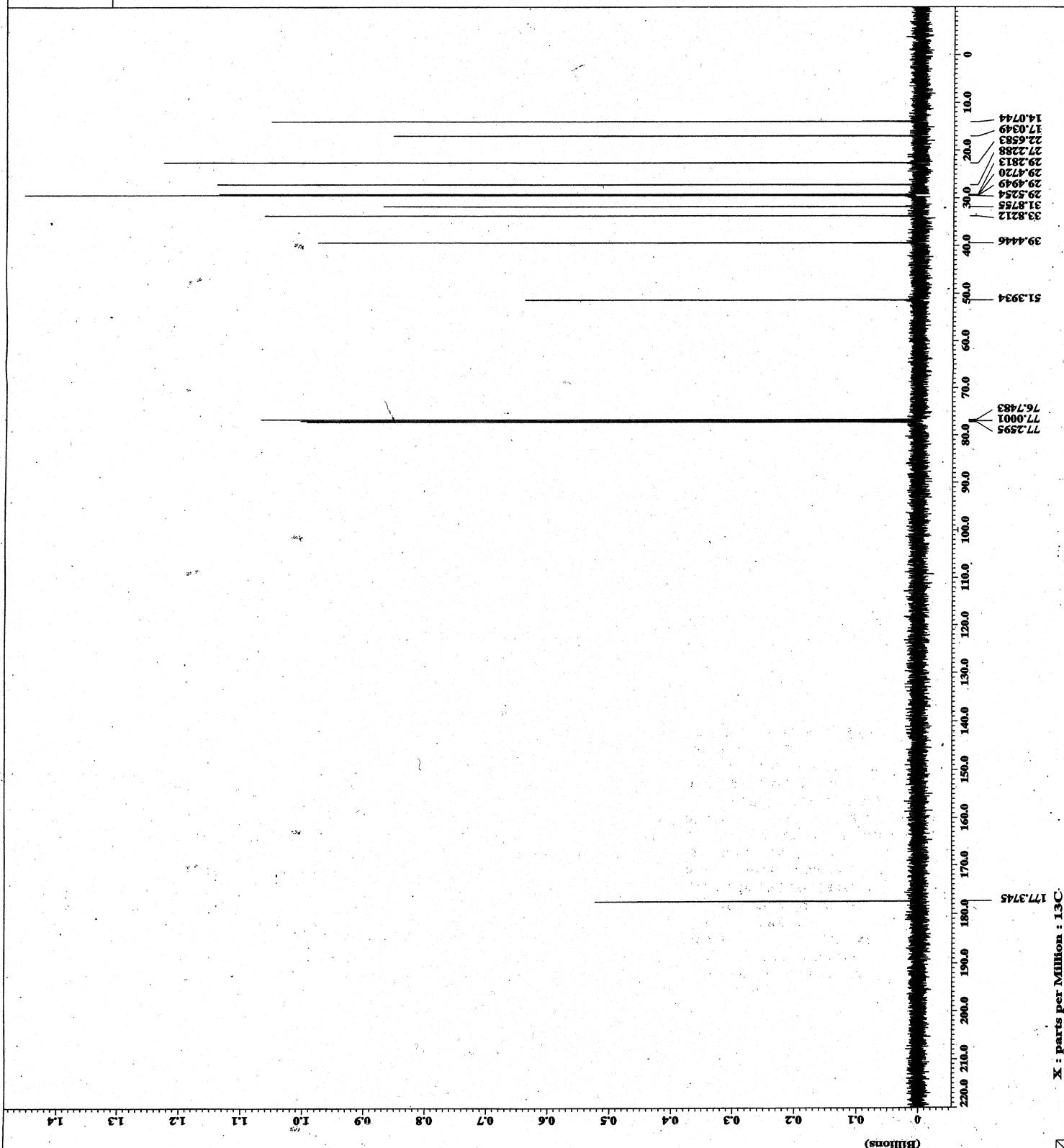
X : parts per Million : 13C



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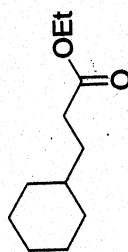
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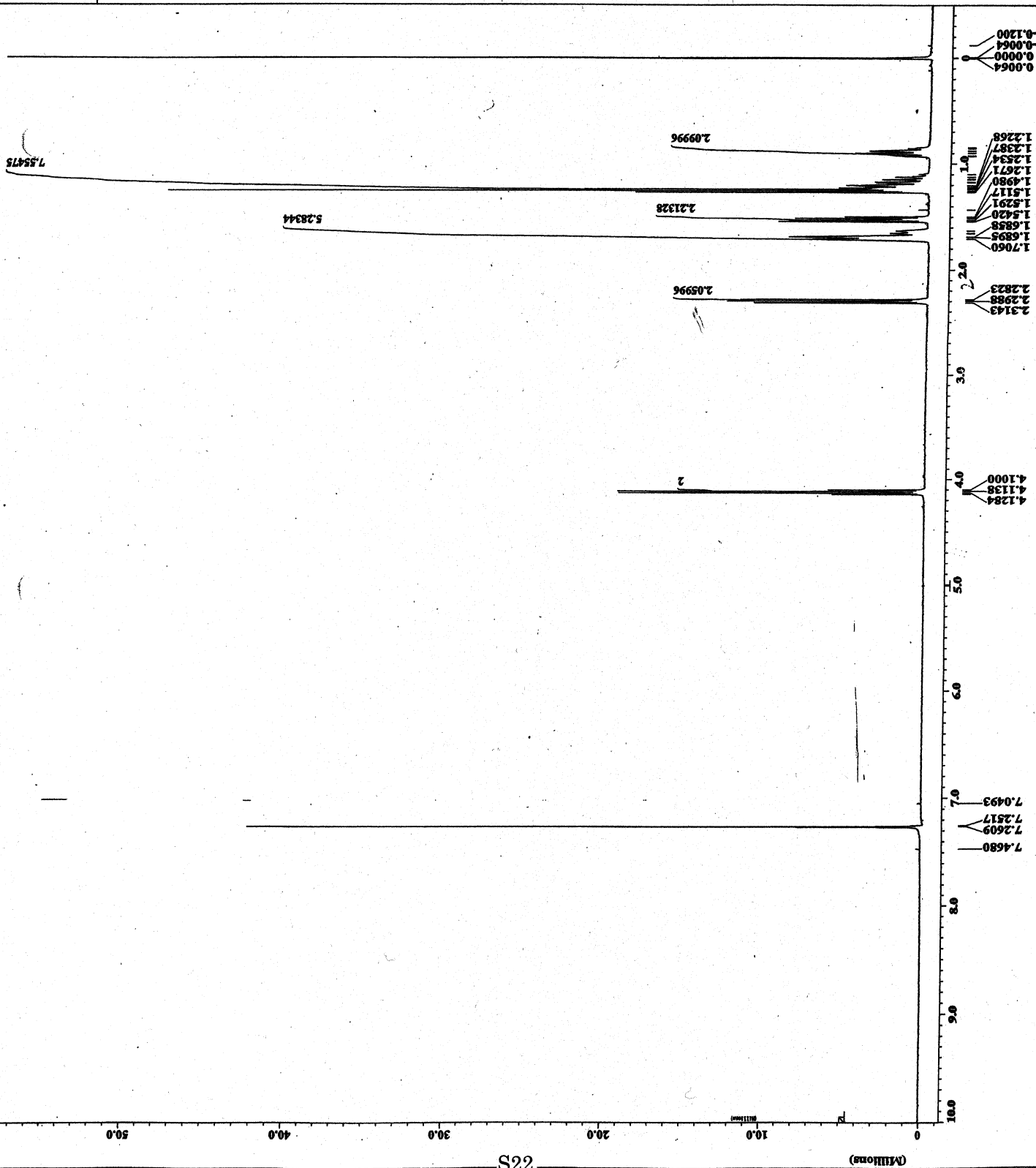
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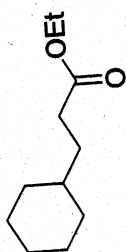
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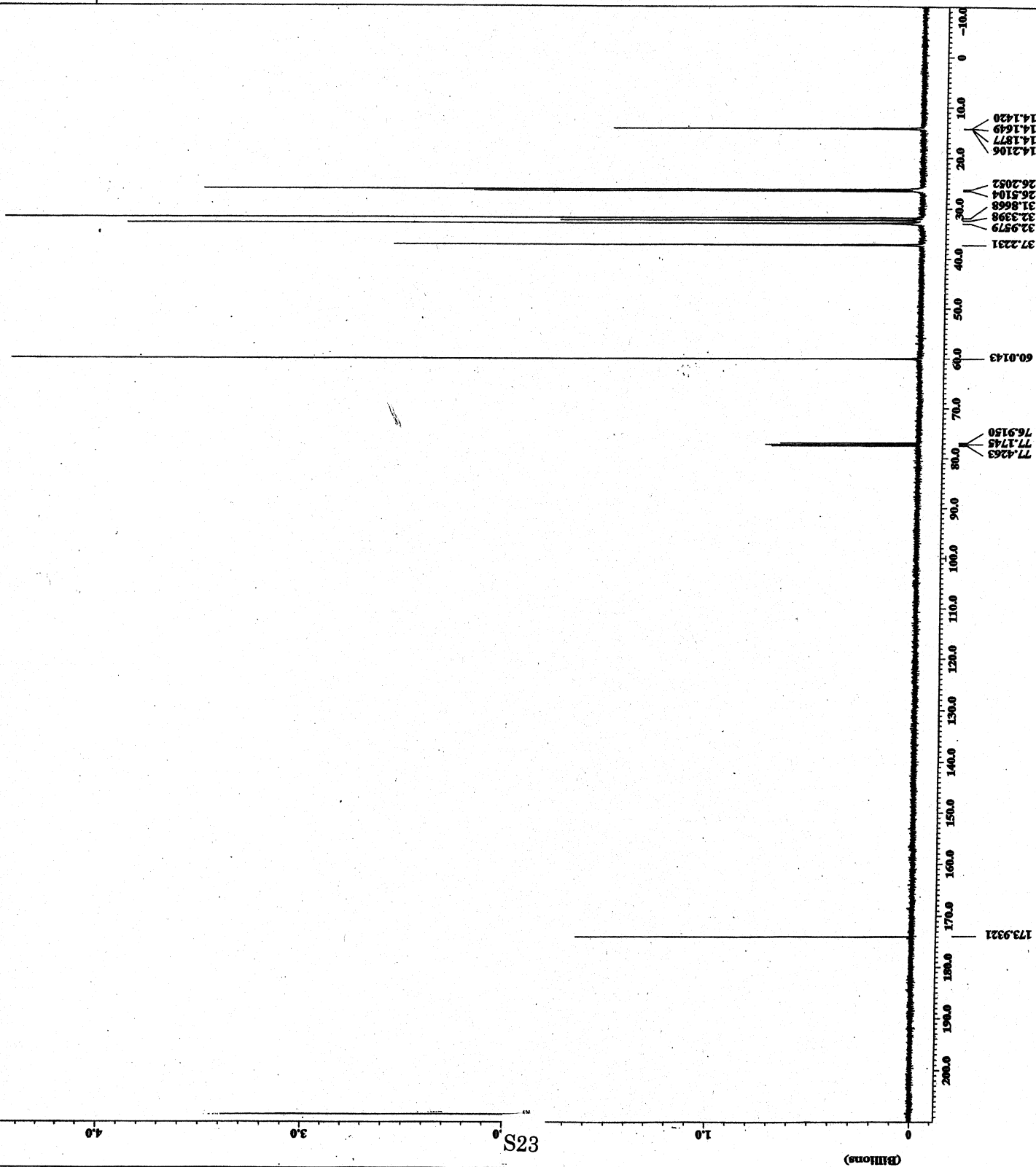
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X : parts per Million : 1H

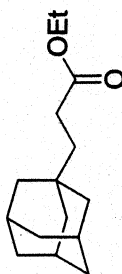
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3d

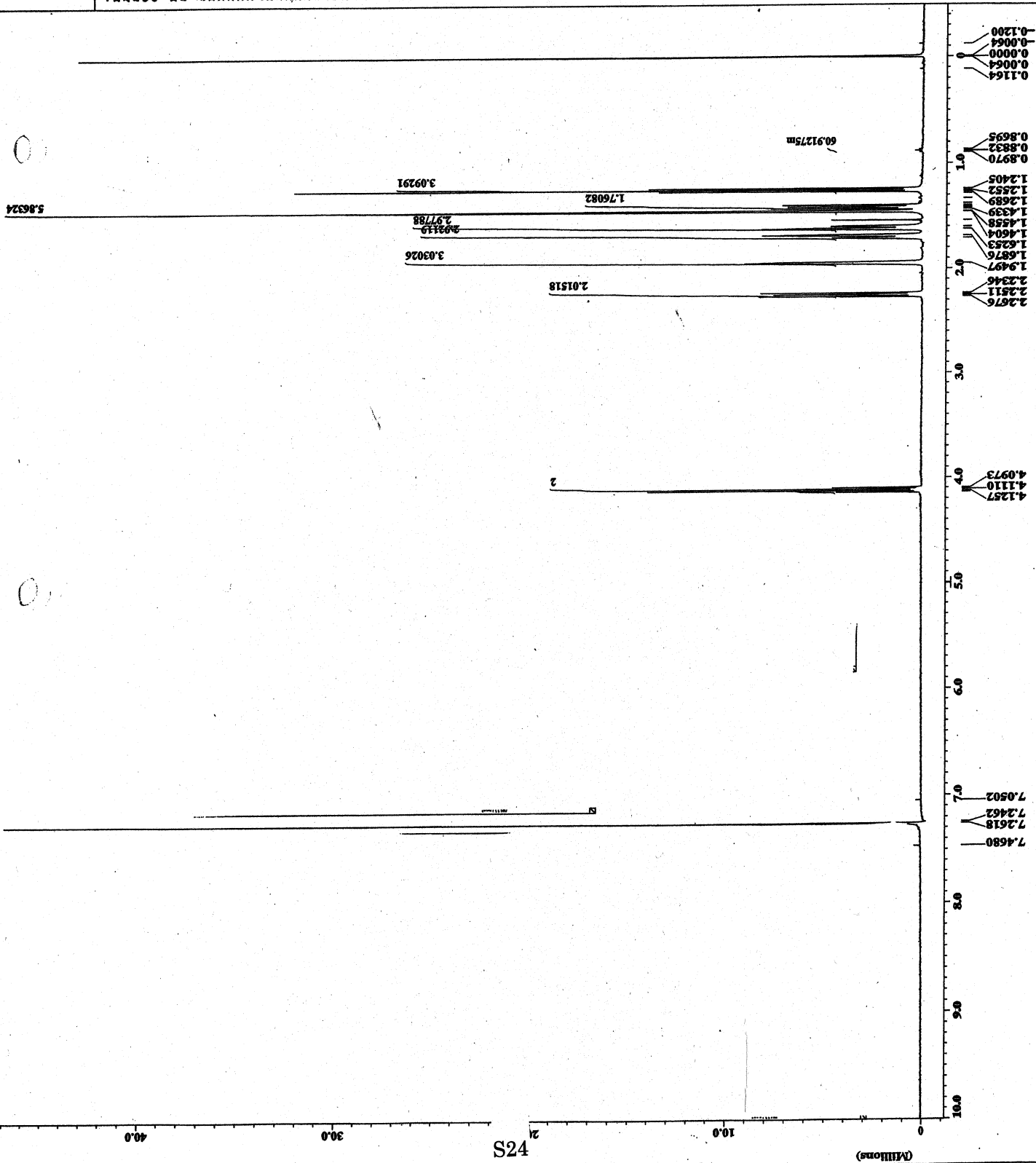


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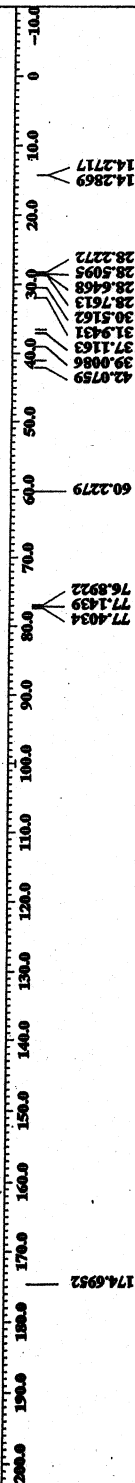
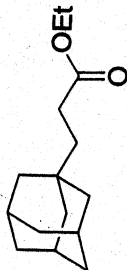
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3e



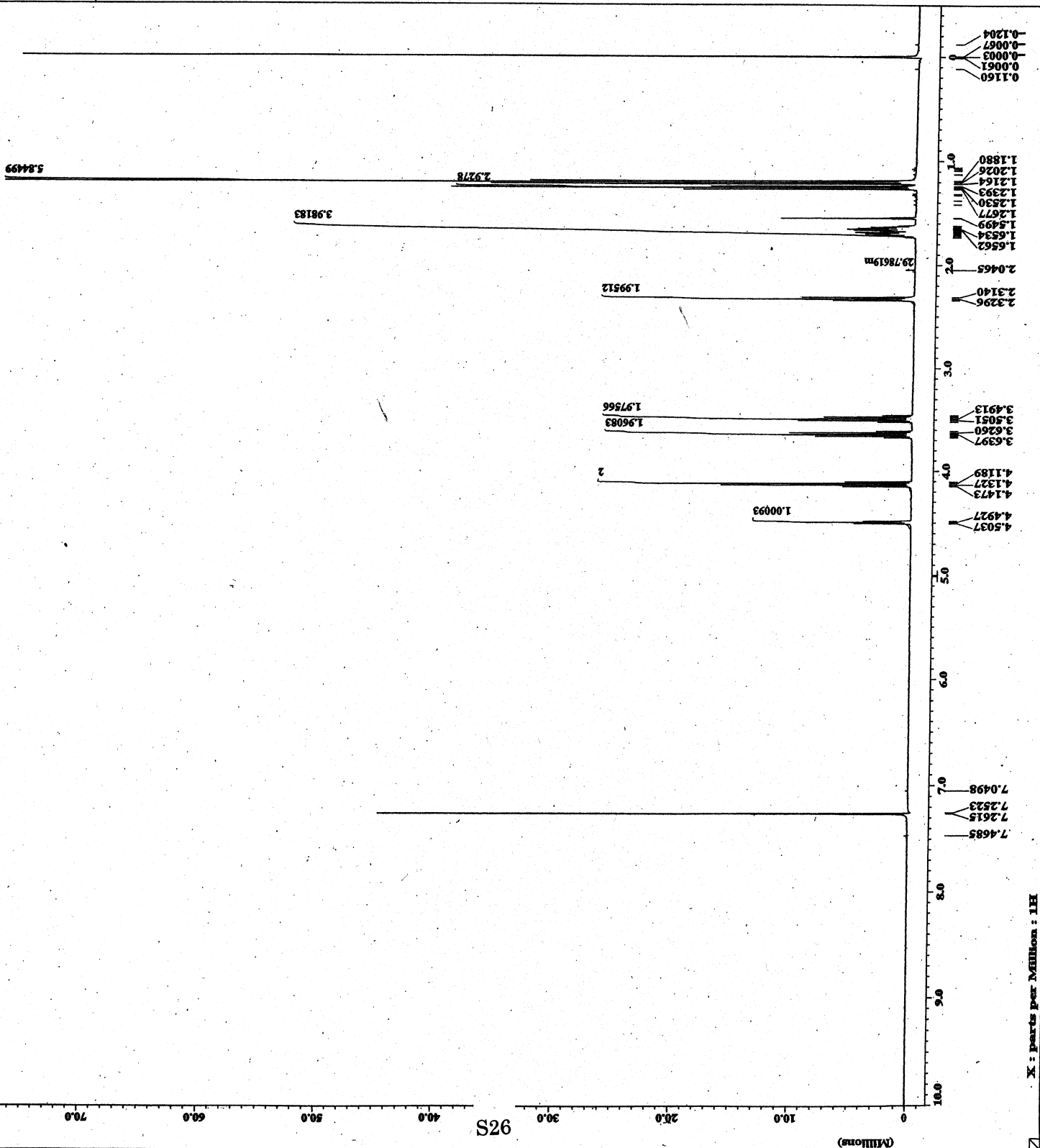
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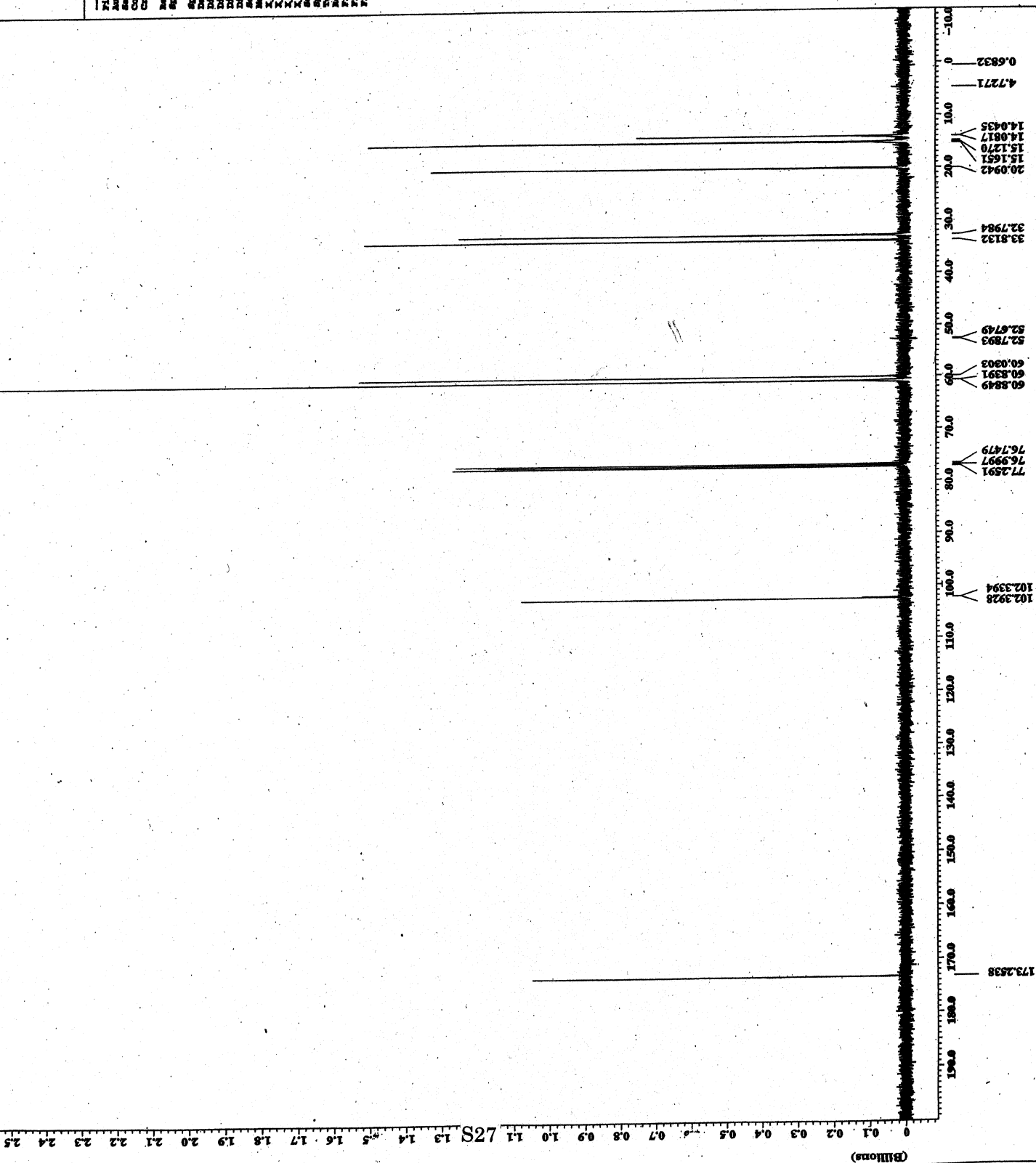
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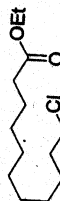
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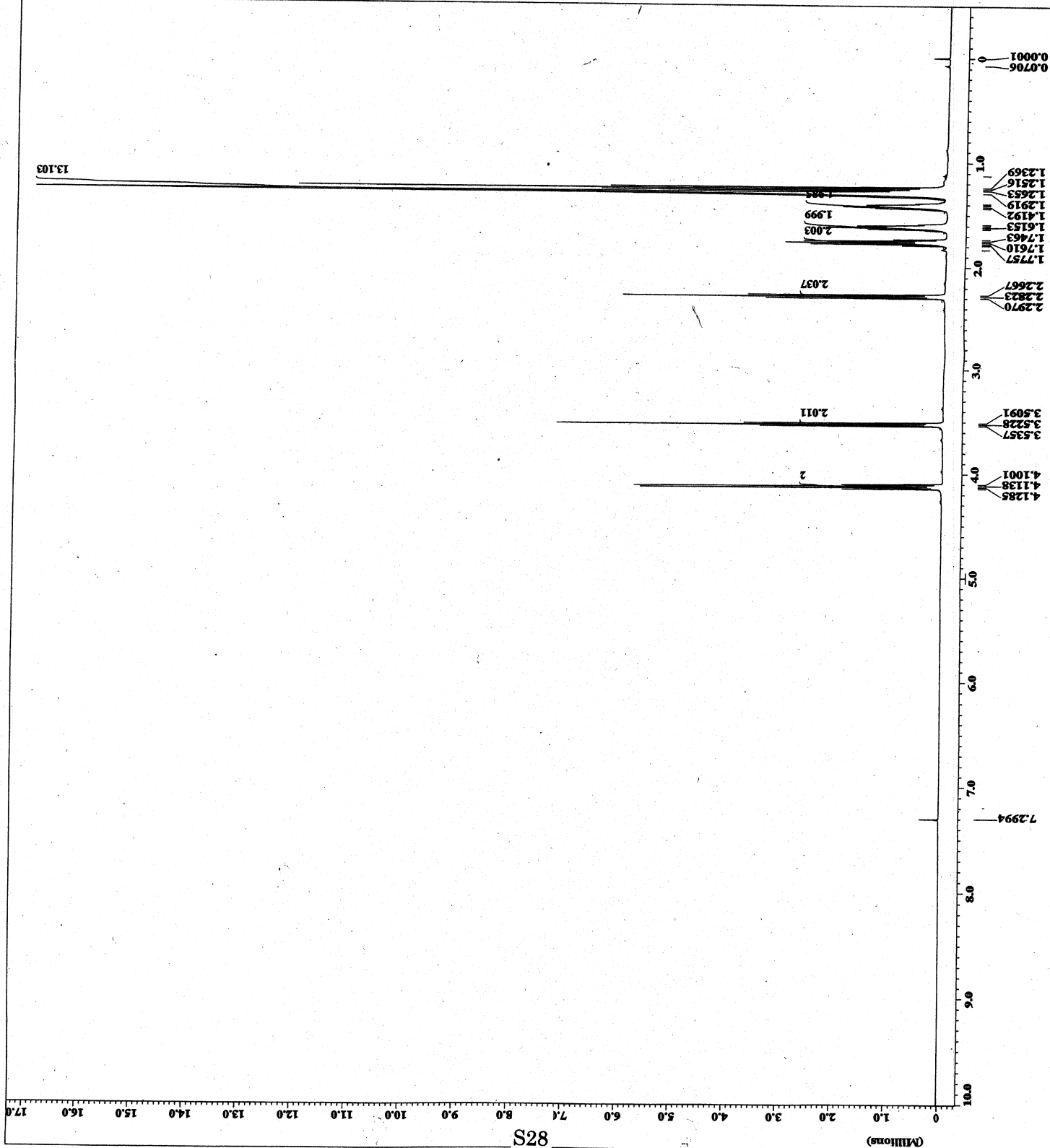
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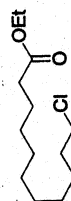
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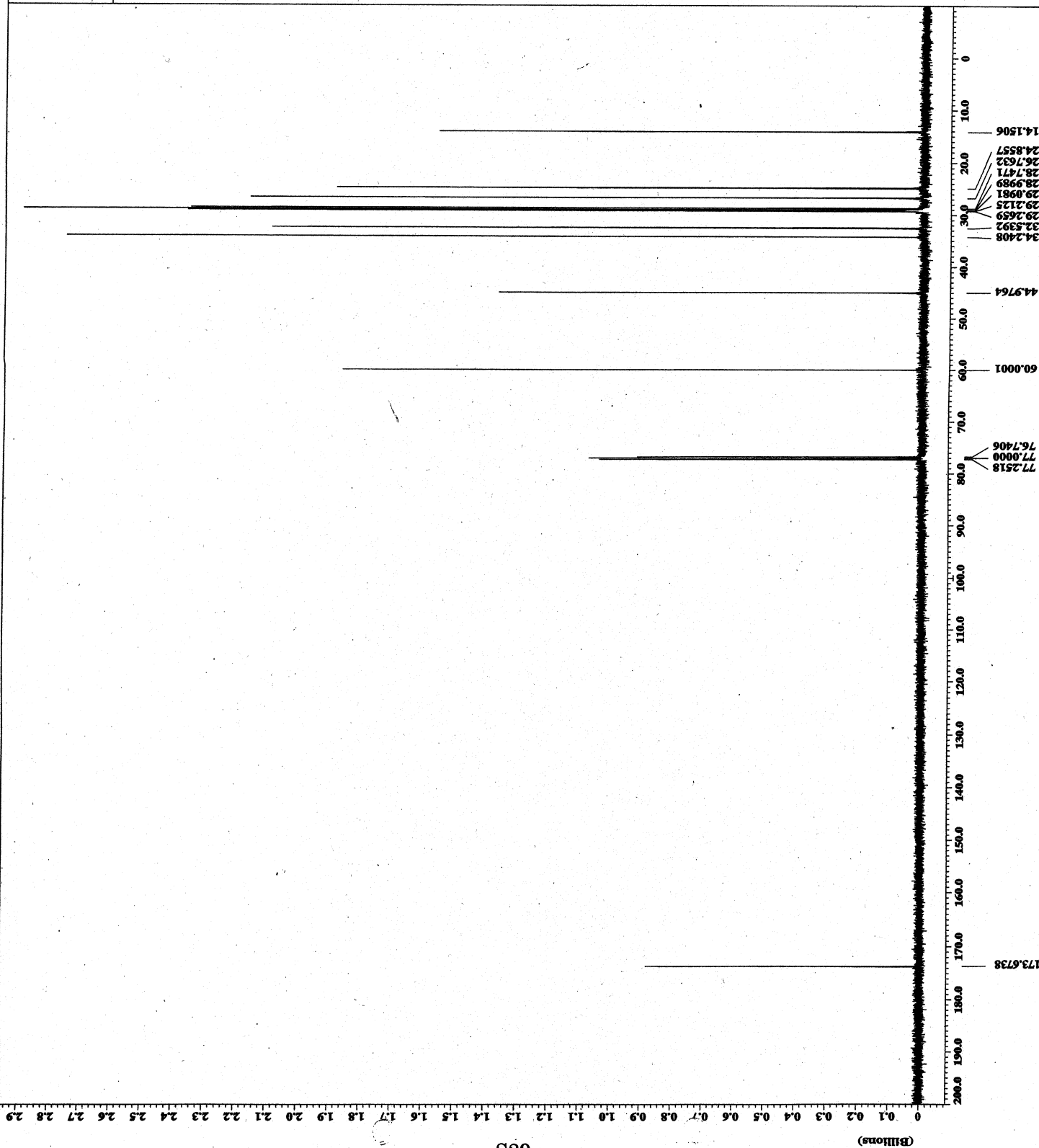
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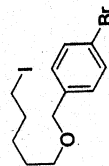


3g

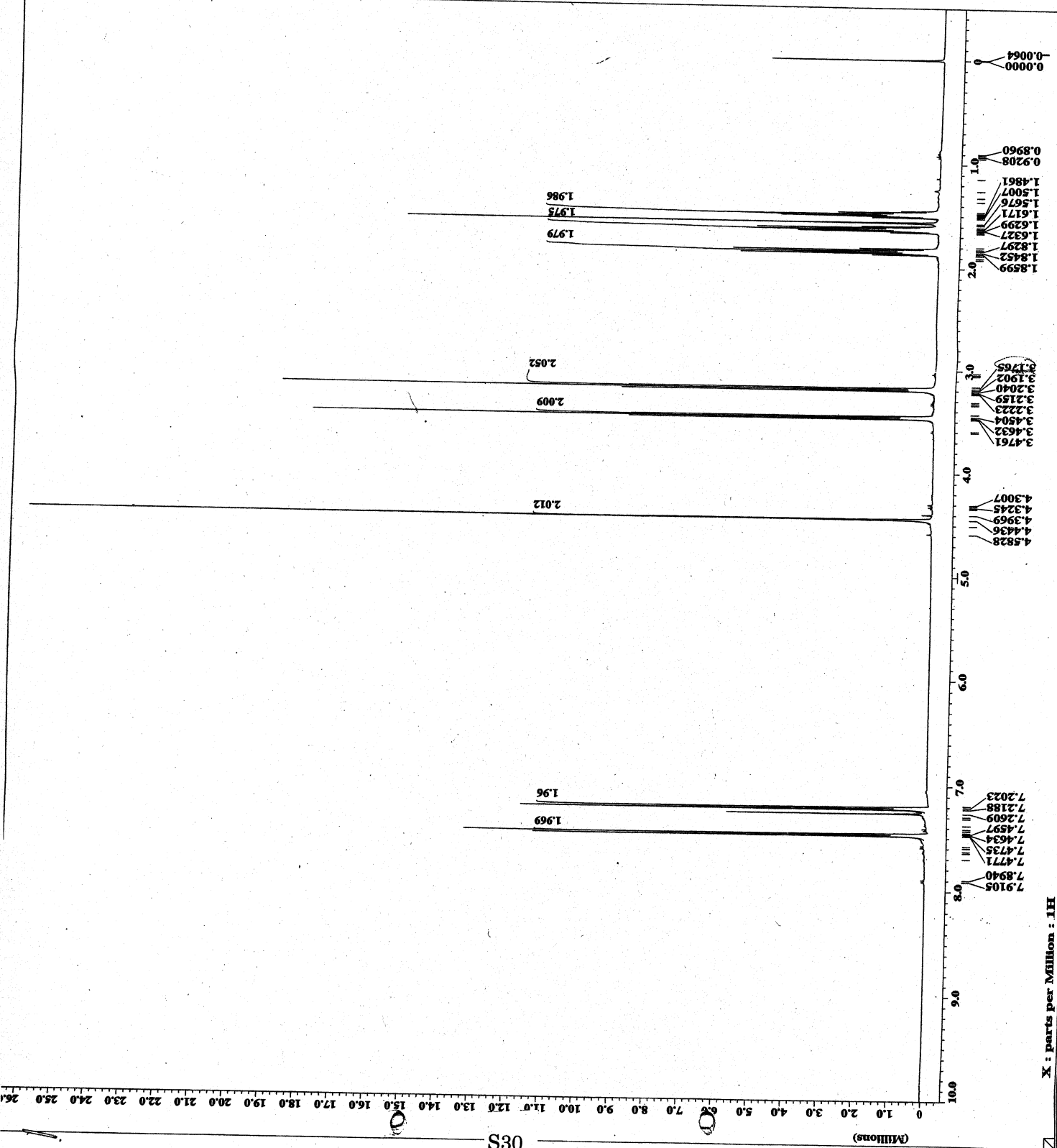


X : parts per Million : 13C

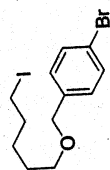
ACQUISITION PARAMETERS
File Name = 1d_spectrum.44
Date = 2006-06-05 06:27:14
Sample ID = 1d_spectrum.44
Constant = 3.0000000000000000
Creation Date = 3-JUN-2006 21:03:49
Revision Date = 5-JUN-2006 06:27:14
Spec file = MCF500
Spec Type = DELTA_MMR
Data Format = ID COMPLEX
Dimensions = 1
Dim 1 Size = 1024
Dim 2 Size = 1024
Dim 3 Size = 1024
Dim 4 Size = 1024
Dim 5 Size = 1024
Mod Return = 1
X Domain = 1
X Offset = 0
X Scale = 1
X Sweep = 1
Solvent = CHLOROFORM-D
Spin Set = 15 [Hz]
Temp Set = 24 [C]
Field Strength = 11.7471579 [T]
Filter Mode = MUYRANWATZ
Filter Width = 3.7511936 [Hz]



1h



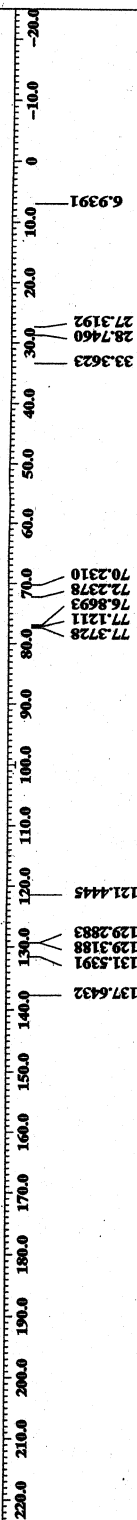
--- ACQUISITION PARAMETERS ---
 File Name : 14_13c_spectrum.156
 Author :
 Sample ID : RST-022
 Content : Single Pulse with Broad
 Creation Date : 3-JUN-2006 23:53:43
 Revision Date : 5-JUN-2006 09:41:37
 Spec Site : MCP500
 Spec Type :
 Data Format :
 Data Name :
 Data Title :
 Data Size : 32768
 Data Units : [ppm]
 Scans : 400
 X offset : 13c
 X offset : 100 [ppm]
 X freq : 125.77787547 [MHz]
 X sweep : 31.44654088 [kHz]
 Solvent : CHLOROFORME-D
 Salt :
 Temp Set : 24.5 [C]
 Recv Gain : 30
 Field strength : 11.7473579 [T]
 Filter Mode : HETCORHANCE
 Filter Width : 15.7266221 [kHz]



1h

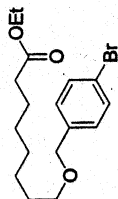
831

(Billions)

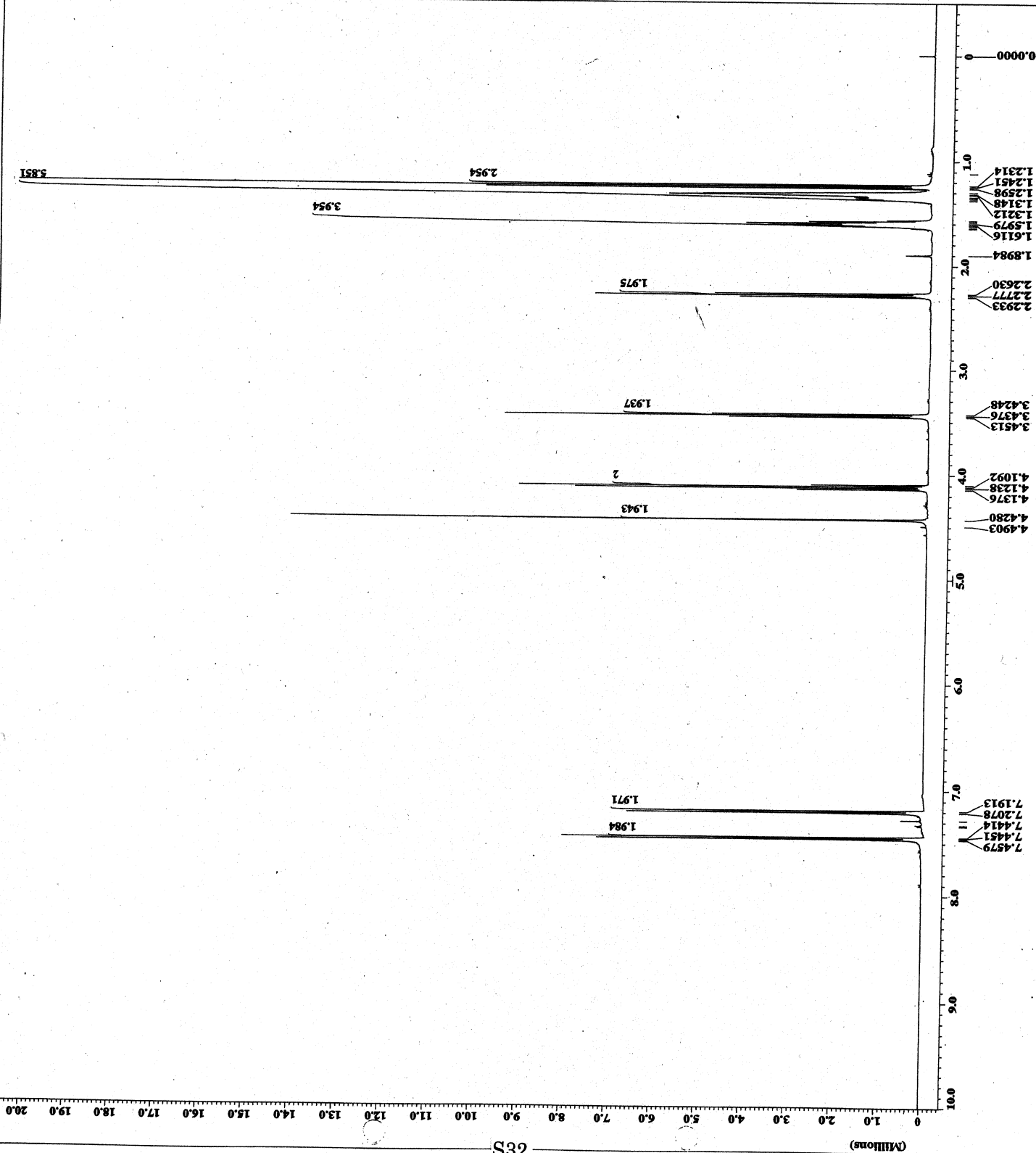


X : parts per Million : 13C

ACQUISITION PARAMETERS
 File Name : 1d_spectrum.108
 Author :
 Sample ID : M0-126-452-3
 Creation Date : 7-JUN-2006 11:07:00
 Revision Date : 8-JUN-2006 21:33:21
 Spec Site : MCP500
 Spec Type : 1D COSY
 Data Format : 1D COSY
 Dimensions : 1H
 Num Title : 1H
 Num Size : 16384
 Num Bits : 16
 Num Channels : 1
 Mod_return : 1H
 X_domain : 5[ppm]
 X_offset : 500.13241602[MHz]
 X_freq : 500.13241602[MHz]
 Solvent : CDCl3
 Spin_get : 17[Hz]
 Temp_get : 22.9[deg]
 Acq_gain : 15
 Filter_strength : 11.747357917
 Filter_width : 3.75119936[Hz]

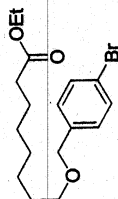


3h

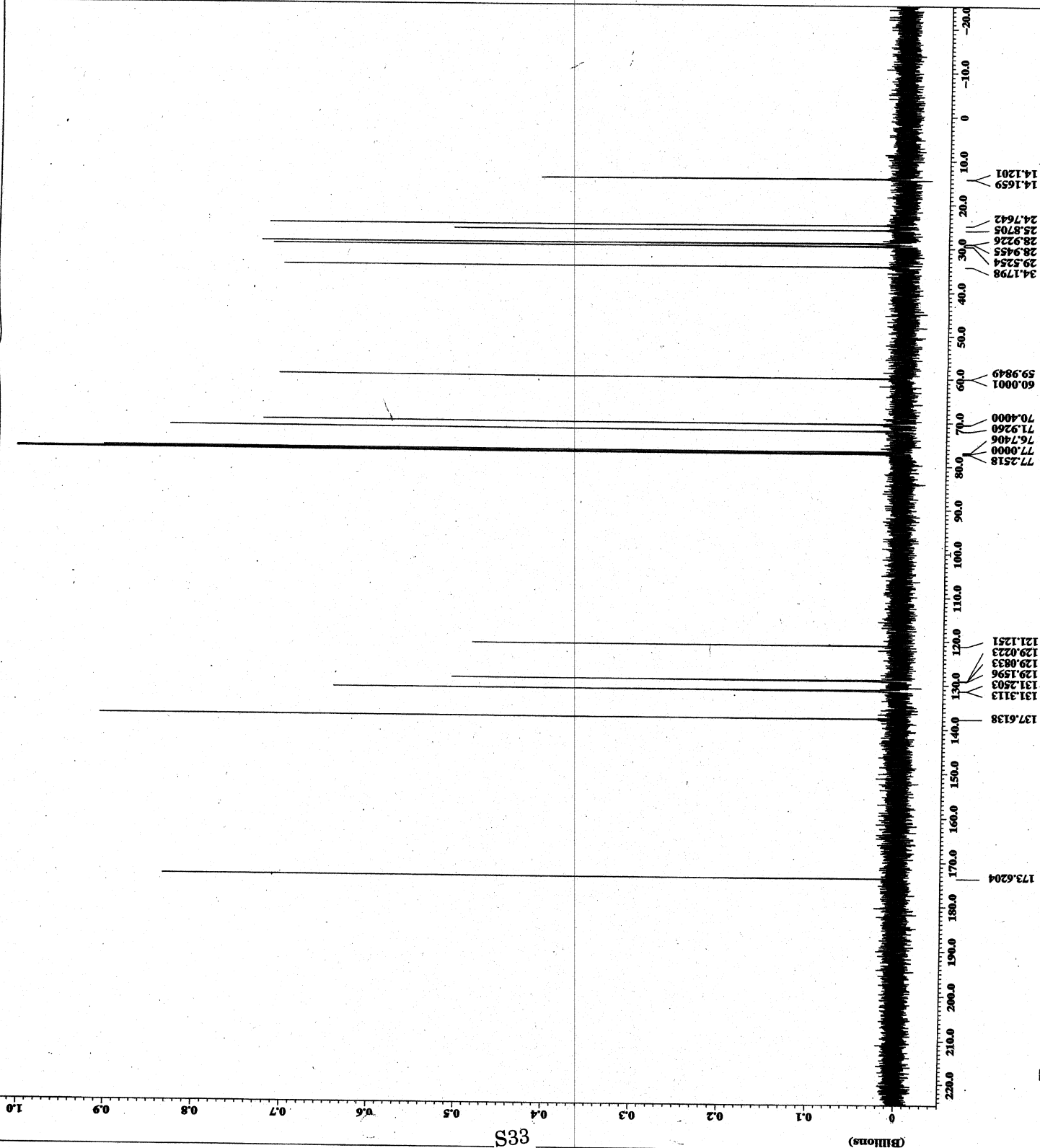


X : parts per Million : 1H

--- ACQUISITION PARAMETERS ---
 File Name = 1d_13c_spectrum.180
 Author =
 Sample ID = HFO-136-E2-3-2
 Comments = Single Pulse with Broad
 Creation Date = 7-JUN-2006 11:25:17
 Revision Date = 8-JUN-2006 21:49:39
 Spec Site = MZ500
 Spec Type = HMQC_1H
 Data Format = 1D Convent
 Dimensions = 13C
 Dim title = 32768
 Dim size = 100
 Dim units = [ppm]
 Mod_return = 13C
 X_domain = 100 [ppm]
 X_offset = 125.77787547 [MHz]
 X_freq = 125.77787547 [MHz]
 X_name = 13C
 Solvent = CDCl3
 Spin_set = 13 [Hz]
 Temp_set = 23.5 [deg]
 Nuc1_gain = 30
 Nuc1_offset = 1.7473579 [Hz]
 Filec_name = 1d_13c_spectrum.180
 Filec_size = 15.72864221 [KHz]

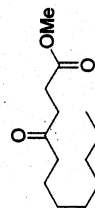


3h

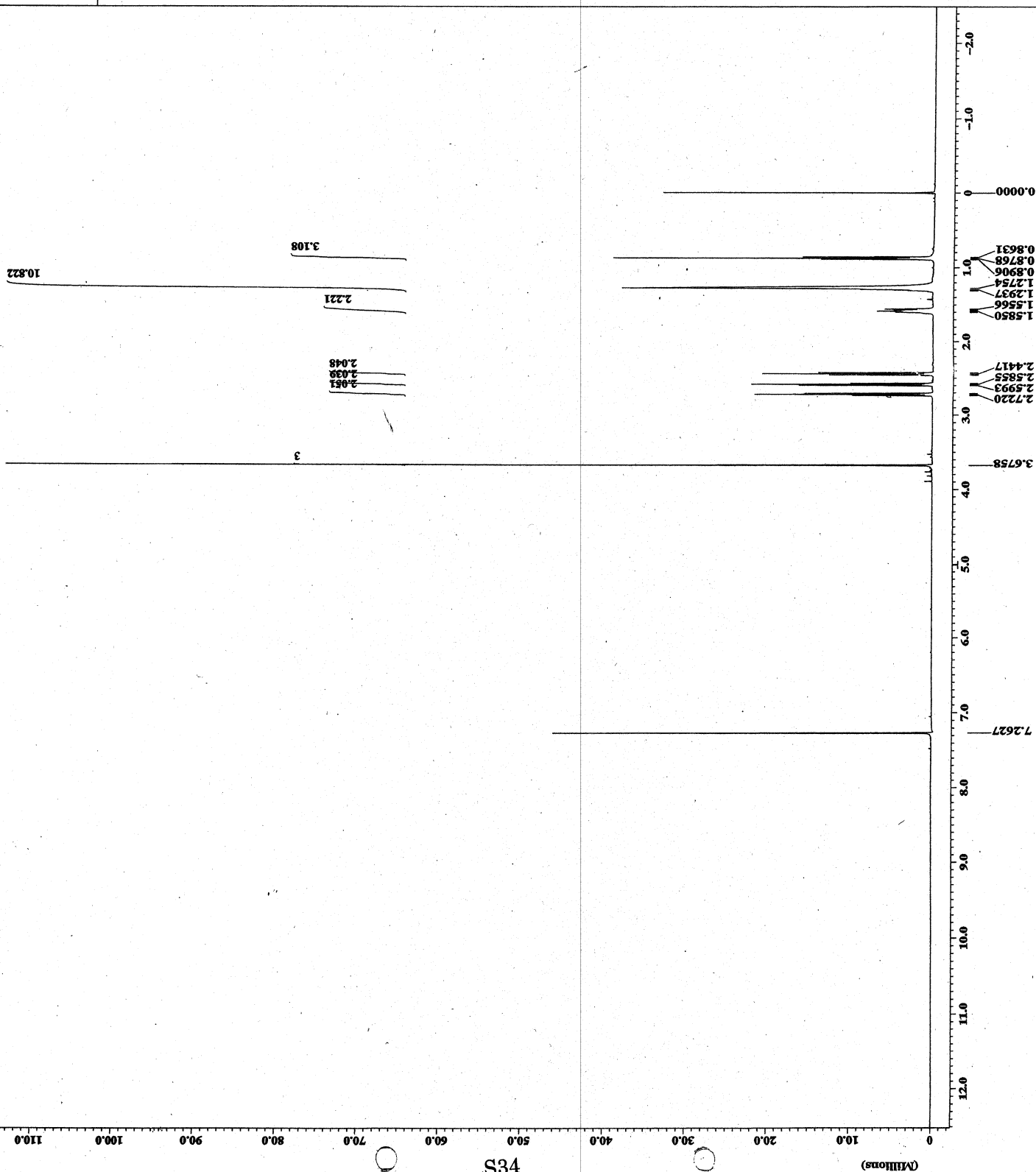


X : parts per Million : 13C

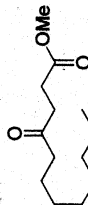
----- ACQUISITION PARAMETERS -----
 File Name = 1d_spectrum.2964
 Author =
 Sample ID =
 Content = Single Pulse Experiment
 Creation Date = 20-OCT-2007 14:33:32
 Revision Date = 22-OCT-2007 09:12:02
 Spec Site = KCP500
 Spec Type = 1H/29A_1H
 Relaxation = 10.000000
 Dimensions = 1H
 Dim Title = 16384
 Dim Size = 16384
 Dim Units = Deg
 Mod Return = 10
 X_domain = 1H
 X_offset = 51ppm
 X_freq = 500.13241602 [MHz]
 X_resol = 7.50750751 [Hz]
 Acq_method = 1D
 Solvent = CDCl3
 Spin_get = 16 [Hz]
 Temp_get = 24.7 [deg]
 Recvr_gain = 25
 Field_strength = 11.7473579 [T]
 Pulse_prog = zgpg30
 Filten_width = 1.75119936 [Hz]



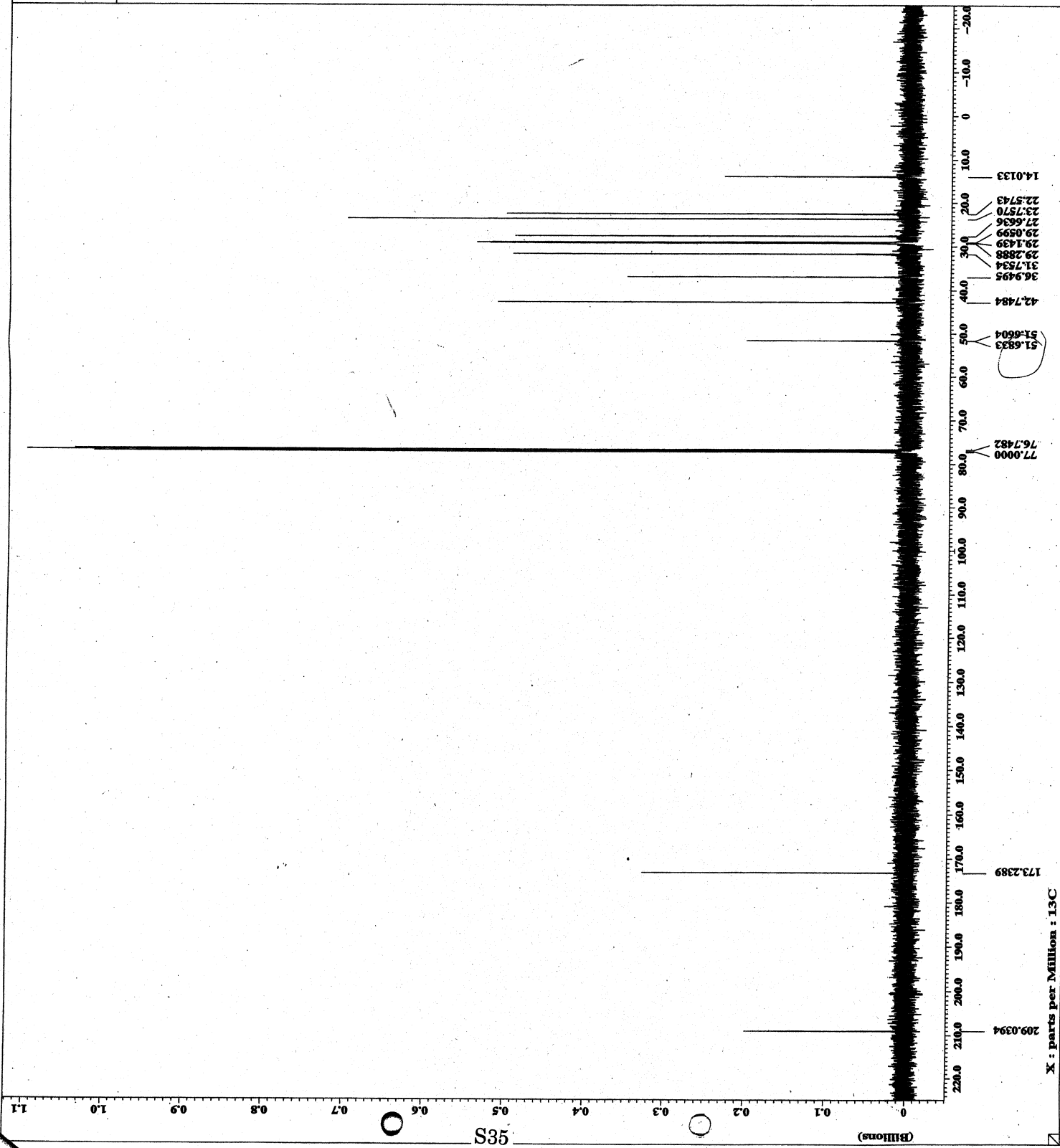
31



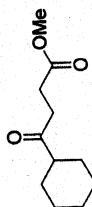
ACQUISITION PARAMETERS
 File Name: 14_13c_spectrum.30
 Author: R
 Sample ID: Single Pulse with Broad
 Creation Date: 20-OCT-2007 14:56:05
 Revision Date: 22-OCT-2007 09:32:41
 Spec File: MCP500
 Spec Type: 1D COSY
 Data Format: 1D COSY
 Dimensions: 13C
 Num Title: 13C
 Num Size: 32768
 Num Units: 13C
 Num: 433
 Mod_return: 1
 X_domain: 13C
 X_offset: 100ppm
 X_freq: 125.77787547[MHz]
 X_solvent: CDCl3
 Solvent: CHLOROFORM-D
 Spin_set: 17Hz
 Temp_set: 26.4[°C]
 Acq_gain: 30
 Filter: 13C Length: 21.7473579[F]
 Filter: 13C Width: 15.72866221[MHz]
 Filter_width: 15.72866221[MHz]



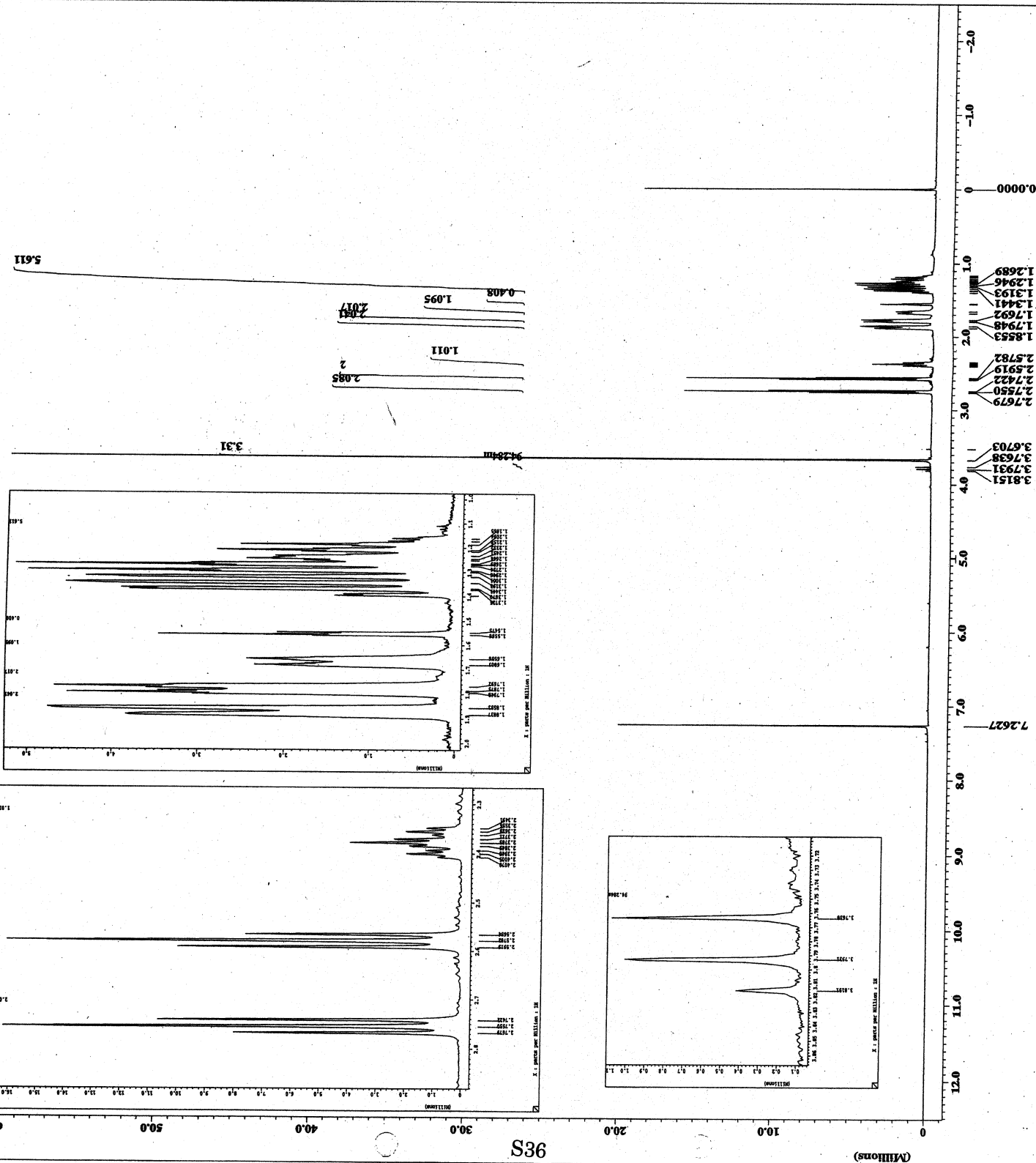
3i



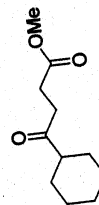
ACQUISITION PARAMETERS
 File Name : 1d_spectrum_3419
 Sample ID : 1150/1515-29
 Context : Single Pulse Experiment
 Creation Date : 31-Oct-2007 10:40:15
 Revision Date : 2-Nov-2007 05:29:15
 Spec Site : MZ500
 Spec Type : 1D NMR
 Data Format : 1D COMPLEX
 Dimensions : 1
 Num Title : 1
 Num Date : 10884
 Num Units : 1
 Scans : 1
 Mod_return : 1
 X_domain : 1
 X_offset : 1
 X_phase : 1
 X_sweep : 1
 Solvent : CHLOROFORM-D
 Spin_set : 16[Hz]
 Temp_set : 24.9[deg]
 Field_strength : 11.7471579[T]
 Filter_mode : MUYSSONWALTZ
 Filter_width : 3.75119936[Hz]



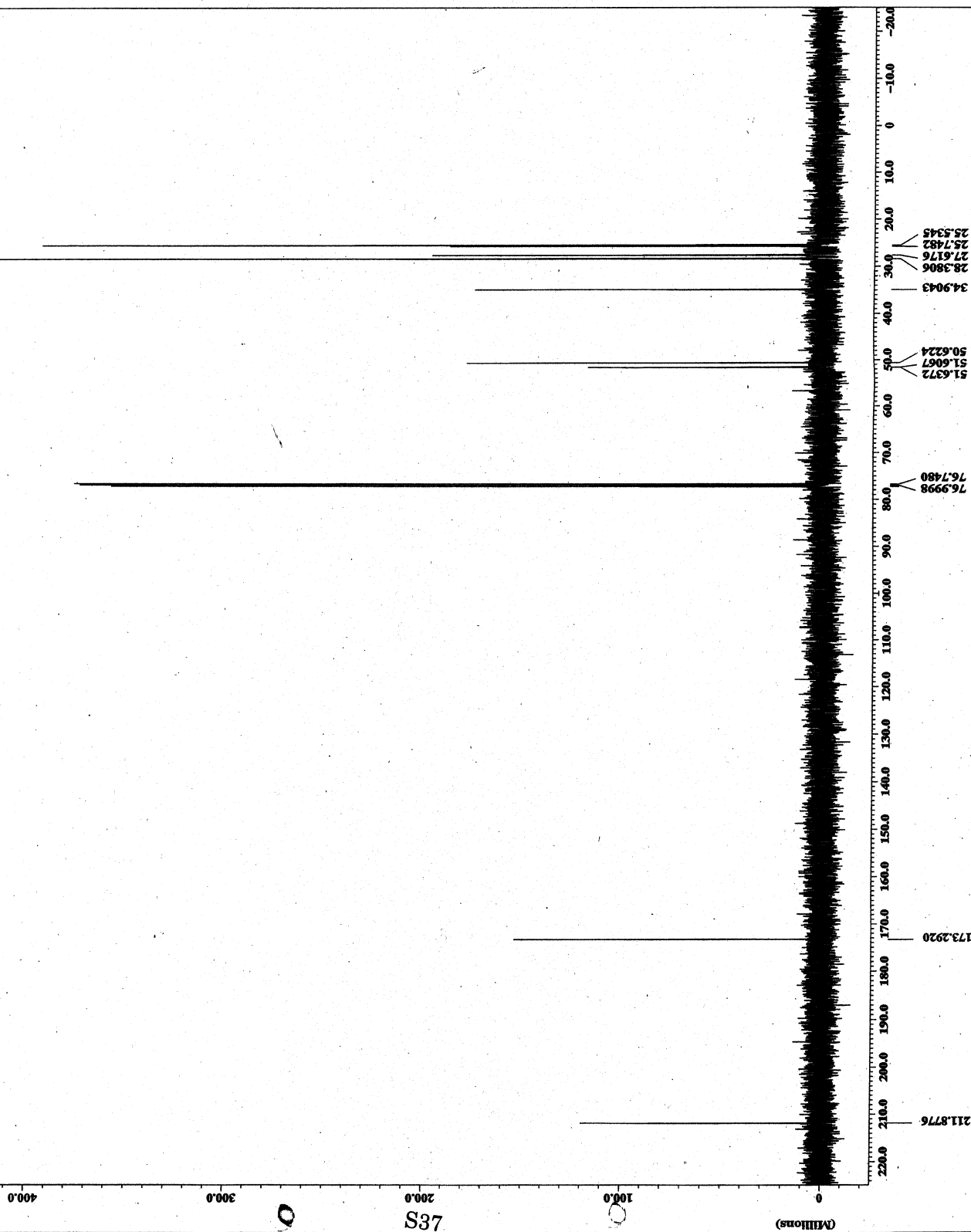
3j



ACQUISITION PARAMETERS
File Name: 10_13c_spectrum.182
Author: c
Sample ID: c
Content: Single Pulse with Broad
Creation Date: 7-NOV-2007 14:52:37
Revision Date: 9-NOV-2007 09:48:59
Spec Site: KCP500
Spec Type: MEXZA_MMR
Data Format: 1D COMPLEX
Data Size: 13C
Data Title: 13C
Data Size: 32768
Data Units: [ppm]
Scan: 153
X1_start: 13C
X1_offset: 100 [ppm]
X_freq: 125.77767547 [MHz]
X_swap: 31.44654088 [kHz]
Solvent: CDCl3
Pulse: zgpg30
Temp_set: 25.5 [degC]
Recov_gain: 30
Field_strength: 11.7473579 [T]
Filter_mode: HETCORH2H
Filter_width: 13.72066221 [kHz]

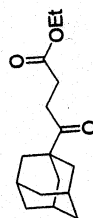


3j

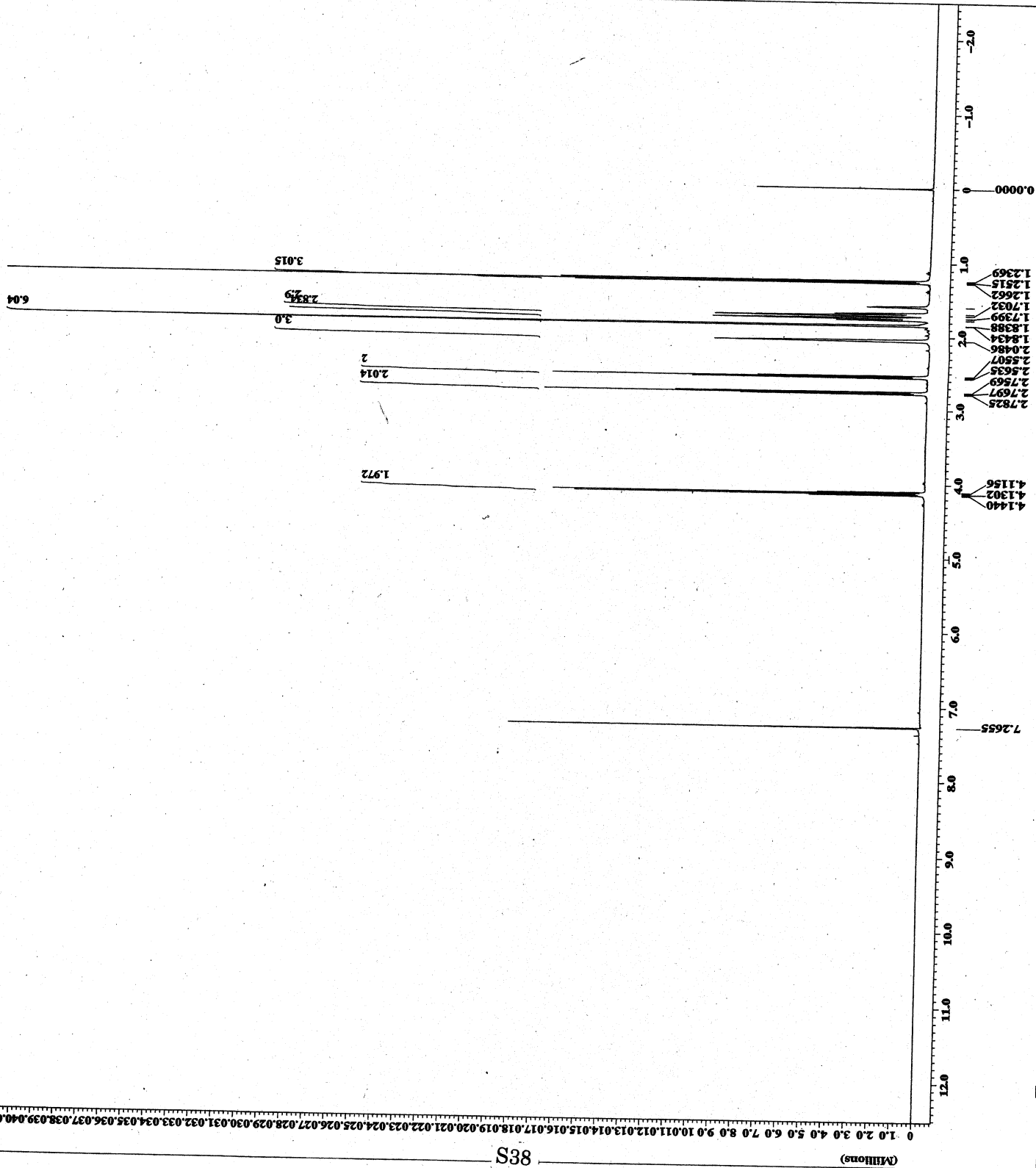


X : parts per Million : 13C

--- ACQUISITION PARAMETERS ---
 File Name = 14_spectrum.190
 Author =
 Sample ID =
 Experiment = Single Pulse Experiment
 Creation Date = 20-MAR-2007 19:10:22
 Revision Date = 22-MAR-2007 13:53:39
 Spec Site = RCP500
 Spec Type = 1H/2D
 Data Format = 1D
 Dimensions = 1H
 Dia Title = 16384
 Dia Size = 16384
 Dia Units = [ppm]
 Name = 1
 Mod_return = 1H
 X_domain = 5 [ppm]
 X_offset = 500.16241602 [MHz]
 X_freq = 7.50750751 [MHz]
 Solvent = CDCl3
 Spin_get = 14 [ms]
 Temp_get = 21.6 [deg]
 Acq_gain = 24
 Recv_strength = 11.7473579 [V]
 Filter_strength = 0.00000000
 Filter_width = 3.75119936 [Hz]

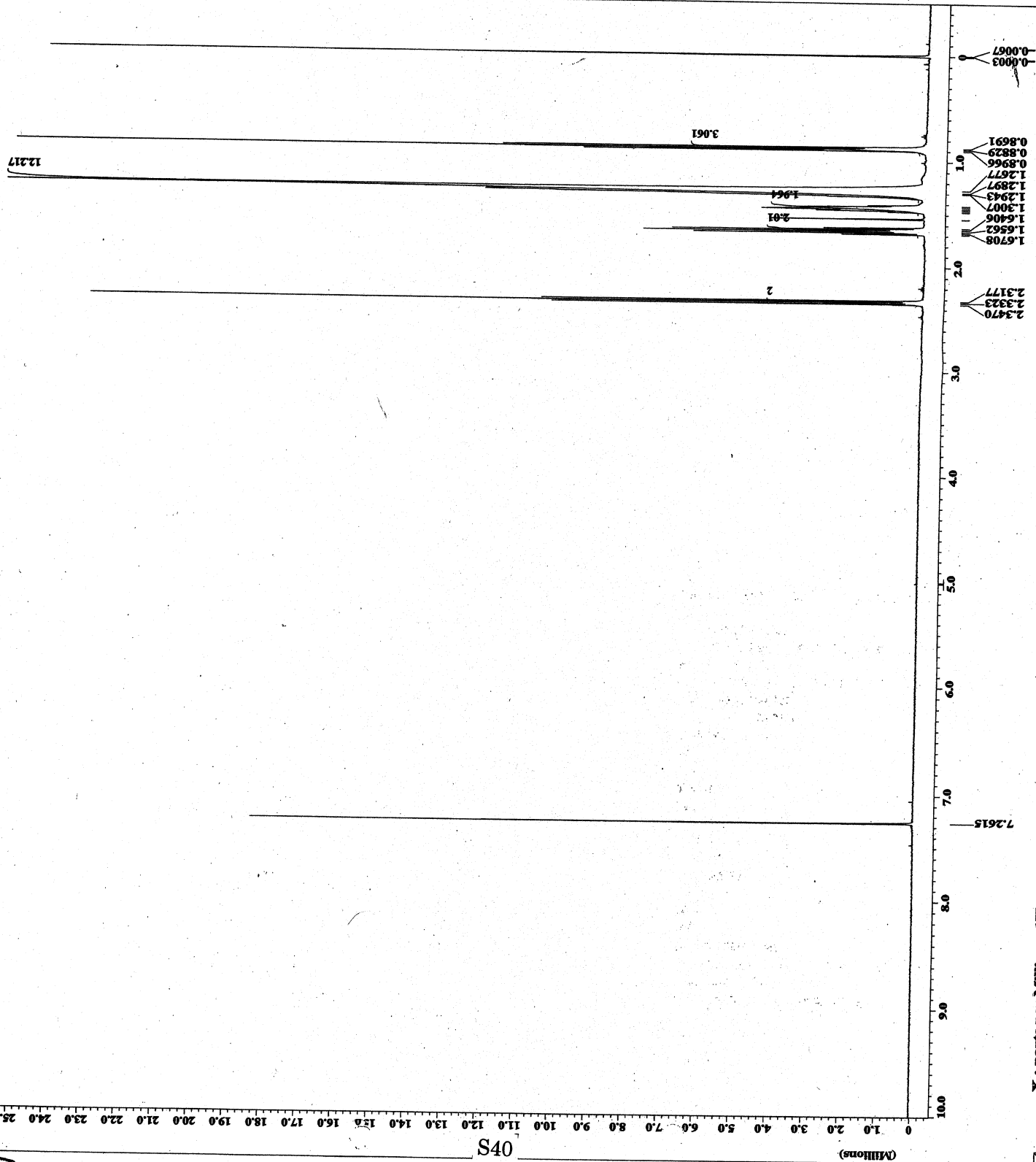


3k



X : parts per Million : 1H

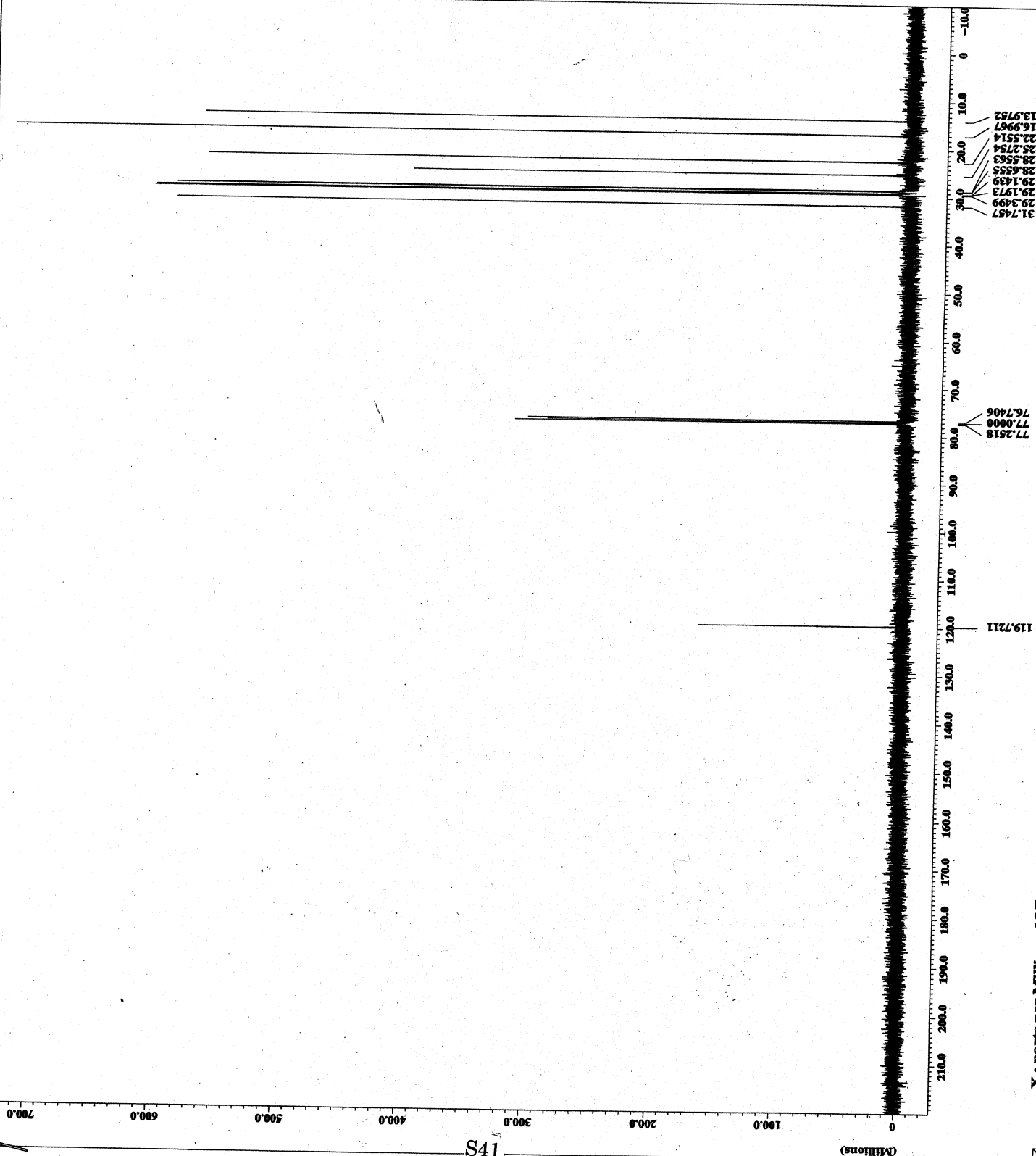
--- ACQUISITION PARAMETERS ---
File Name = 1d_spectrum_499
Author =
Sample ID = R07-205-fs1-7
Content = Single Pulse Experiment
Creation Date = 31-mar-2007 11:04:36
Revision Date = 2-Apr-2007 00:57:21
Spec Site = R07300
Spec Type = MZETA_MMR
Data Format = 1D COMPLEX
Data Points = 1
Data Size = 16384
Data Units = [ppm]
Scans = 8
Acq_return = 1
X_offset = 1
X_freq = 500.16241602 [MHz]
X_sweep = 7.50750751 [kHz]
Solvent = CHLOROFORM-D
SpinJet = 16 [Hz]
Recvr_gain = 24
Field_strength = 11.7473579 [T]
Filter_mode = HETCORHCH
Filter_width = 3.75119936 [kHz]



ACQUISITION PARAMETERS
 File Name = 14_13c_spectrum.105
 Author =
 Sample ID = BPO-205-fid-7
 Content = Single Pulse with Broad
 Creation Date = 31-MAR-2007 11:16:42
 Revision Date = 2-APR-2007 01:07:30
 Spec Site = MCP500
 Spec Type = 13C NMR
 Data Format = 2D COMPLEX
 Dimensions = 13C
 Dim title = 32768
 Dim size = 131
 Dim units = [ppm]
 Mod return = 13C
 X domain = 100 [ppm]
 X offset = 125.77787547 [MHz]
 X freq = 31.44654088 [MHz]
 X temp = 14 [MHz]
 Spin rate = 25.1 [Hz]
 Temp set = 30
 Recvr gain = 11.747379 [V]
 Filtr_d strength = 10 [dB]
 Filtr_type = 10 [dB]
 Filtr_width = 15.7206221 [MHz]



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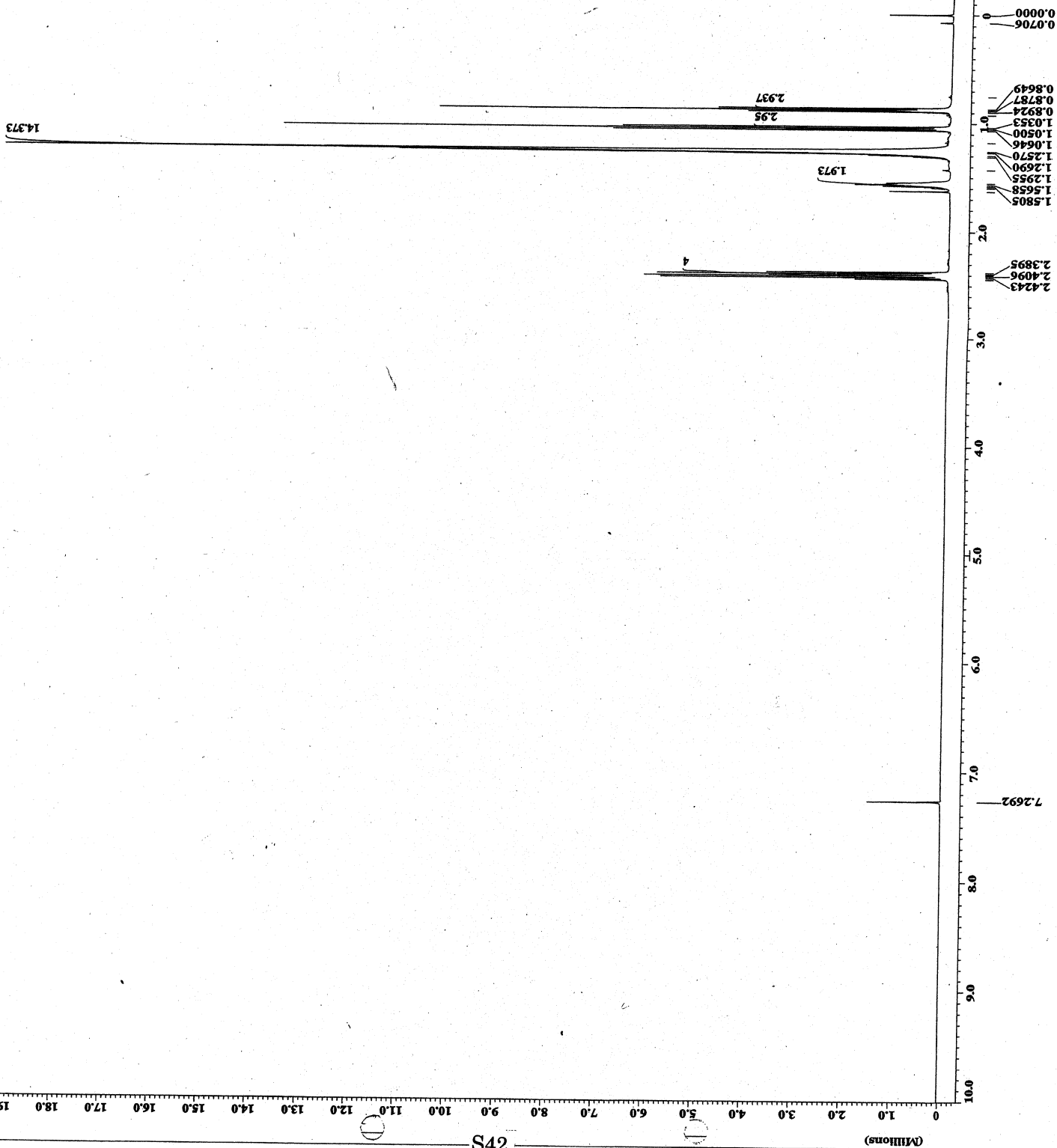


X : parts per Million : 13C

--- ACQUISITION PARAMETERS ---
 File Name : 14_spectrum.4151
 Author :
 Sample ID : RSU-185-414-5
 Content : Single Pulse Experiment
 Creation Date : 28-JUN-2007 17:10:14
 Revision Date : 30-JUN-2007 05:52:56
 Spec Site : MCP500
 Spec Type : NMR
 Spec Name : 1D COMPLEX
 Dimensions : 1H
 Dim Title :
 Dim Size : 16384
 Dim Units : [ppm]
 Num : 4
 Mod :
 X :
 X_domain : 1H
 X_offset : 5 [ppm]
 X_freq : 500.16241602 [MHz]
 X_resol : 7.50750751 [Hz]
 X_acq :
 X_ref :
 Spin :
 Spin_set : 24.8 [Hz]
 Recvz_gain : 21
 Field_strength : 11.7473579 [T]
 Pulse_mode : HETEROMODE
 Filter_width : 3.75119936 [Hz]



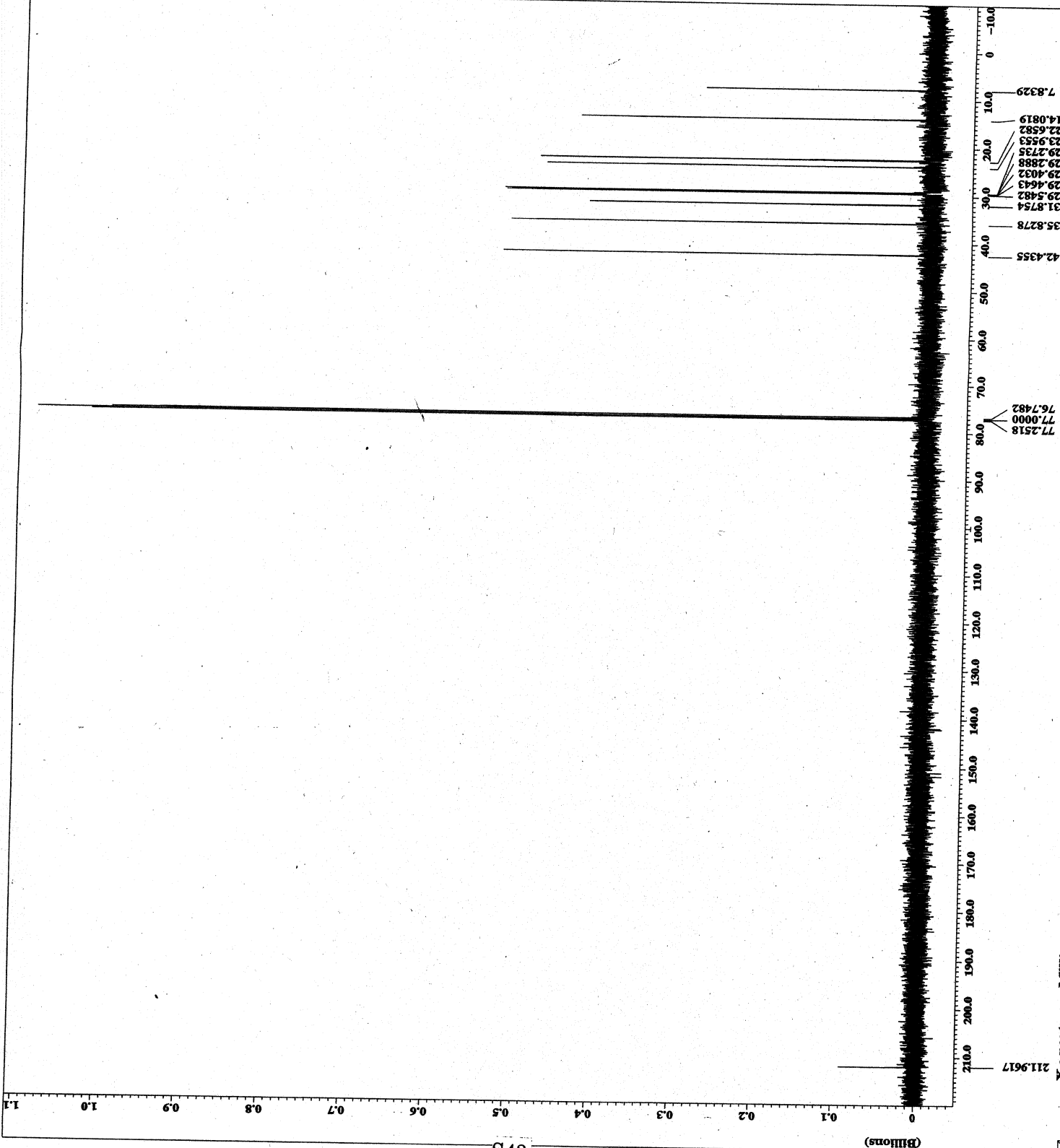
3m



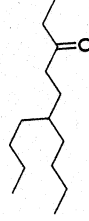
---- ACQUISITION PARAMETERS ----
 File Name = 14_13c_spectrum.197
 Author =
 Sample ID = RSG-185-4rd-5
 Content = Single Pulse with Broad
 Creation Date = 28-JAN-2007 17:25:46
 Revision Date = 30-JAN-2007 06:07:39
 Spec Site = RCP500
 Spec Type = MEXA_MIR
 Data Point = 10
 Dimensions = 13c
 Dim Title =
 Dim Size = 32768
 Dim Units = [ppm]
 Dim Min = 400
 Dim Max = 13c
 X Domain = 13c
 X Offset = 100 [ppm]
 X Freq = 125.77787547 [MHz]
 X Preset = 31.44654088 [MHz]
 Solvent = CHLOROFORM-D
 Spin Set = 13c
 Temp Set = 25.6 [deg]
 Recv Gain = 30
 Field Strength = 11.7473579 [T]
 Filter Mode = HETCORHET
 Filter Width = 15.72064221 [MHz]



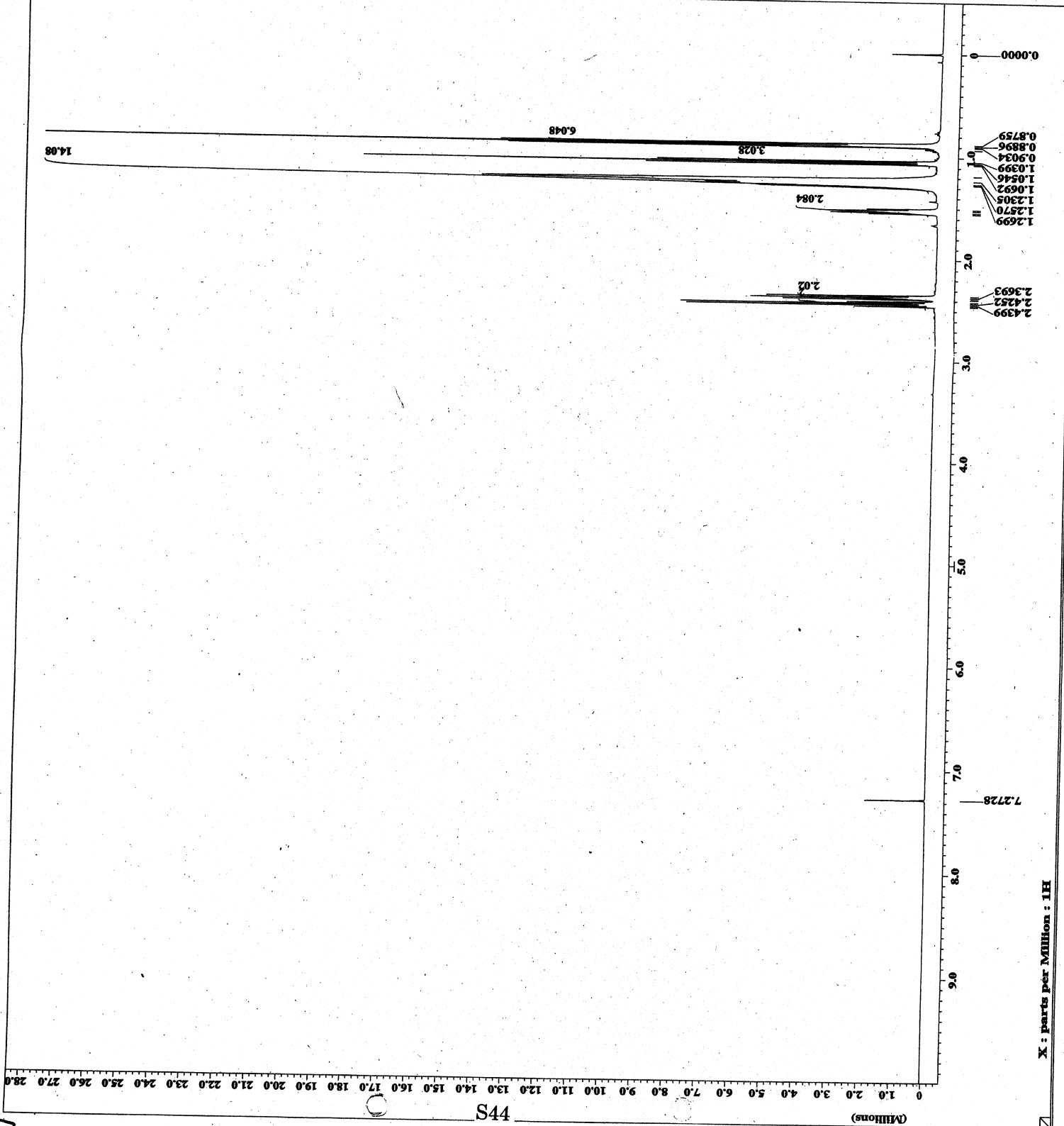
3m



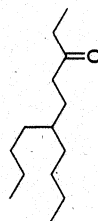
--- ACQUISITION PARAMETERS ---
File Name = 1d_spectrum_6071
Sample ID = RM-193-623
Content = Single Pulse Experiment
Creation Date = 10-MAR-2007 13:34:22
Revision Date = 12-MAR-2007 09:02:57
Spec Site = MCP500
Spec Type = INVEX_MSR
Data Format = 1D COMETEX
Dimensions = X
Dim Size = 18
Dim Units = [ppm]
Scans = 8
Acq_return = 1
X_center = 18
X_offset = 50
X_sweep = 7.50750751 [Hz]
Solvent = CHLOROFORM-D
Spin_get = 17 [Hz]
Spin_set = 24.1 [Hz]
Recvr_gain = 11.7473579 [V]
Field_strength = 11.7473579 [V]
Filter_mode = HETCORHETCOR
Filter_width = 3.75119936 [Hz]



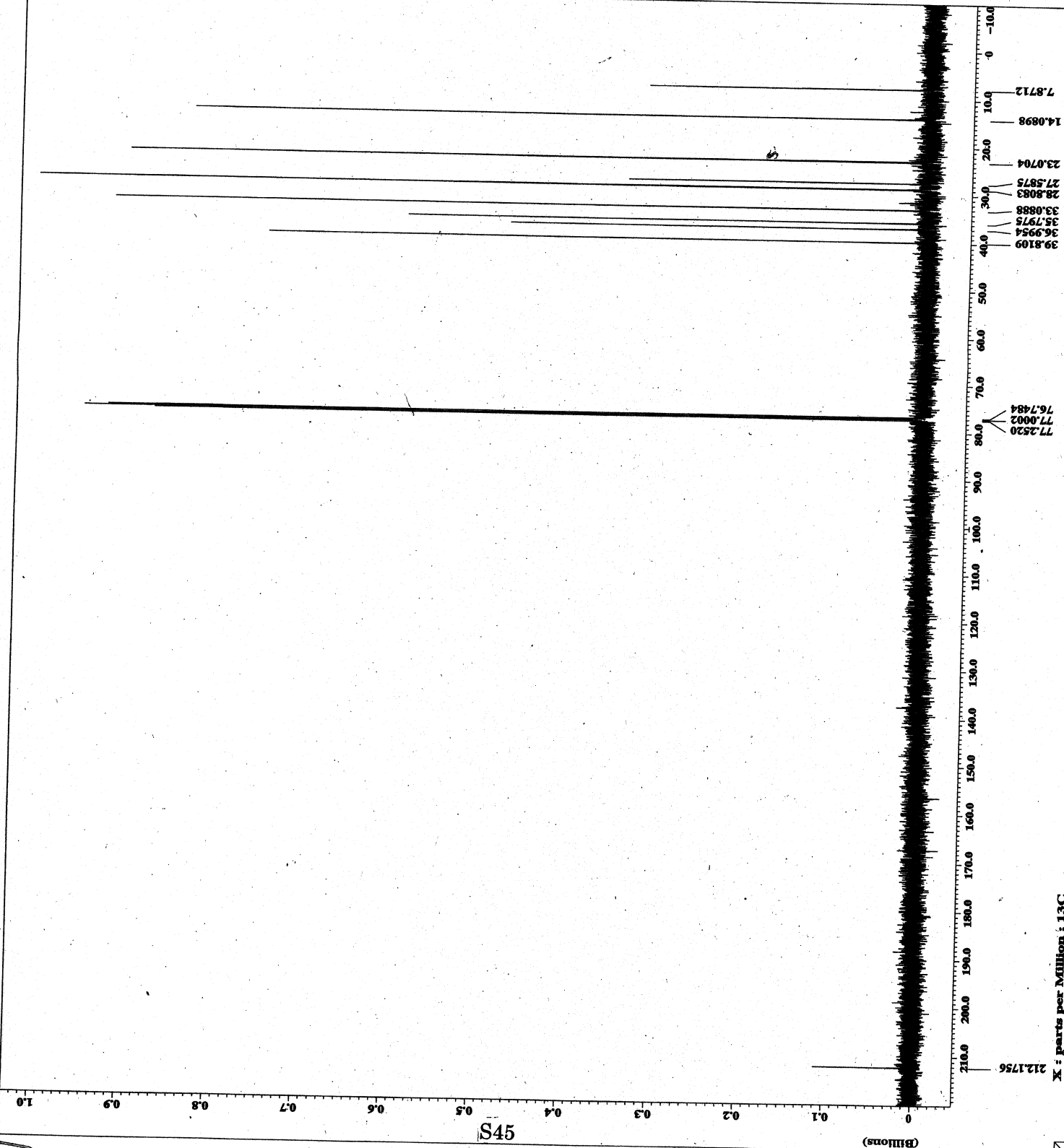
3n



ACQUISITION PARAMETERS
 File Name = 14_13c_spectrum.491
 Author =
 Sample ID = RSP-131-223
 Comment = Single Pulse with Broad
 Creation Date = 10-Mar-2007 13:49:28
 Revision Date = 12-Mar-2007 09:16:52
 Spec Site = RCP500
 Spec Type = 1D COSY
 Data Format = 1D COSY
 Dimensions = 13C
 Dia Title = 13C
 Dia Size = 32768
 Dia Units = 100
 Sound = 1
 Mod_return = 1
 X_domain = 13C
 X_offset = 100 (ppm)
 X_range = 125.77787547 [MHz]
 X_resolution = 15.4654088 [Hz]
 Solvent = CDCl3
 Spin_get = 16 [Hz]
 Temp_get = 25.6 [°C]
 Temp_set = 30
 Field_strength = 11.7473579 [T]
 Pulse_program = zgpg30
 Filter_mode = 1
 Filter_width = 15.72066231 [Hz]

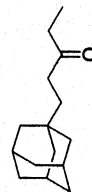


3n

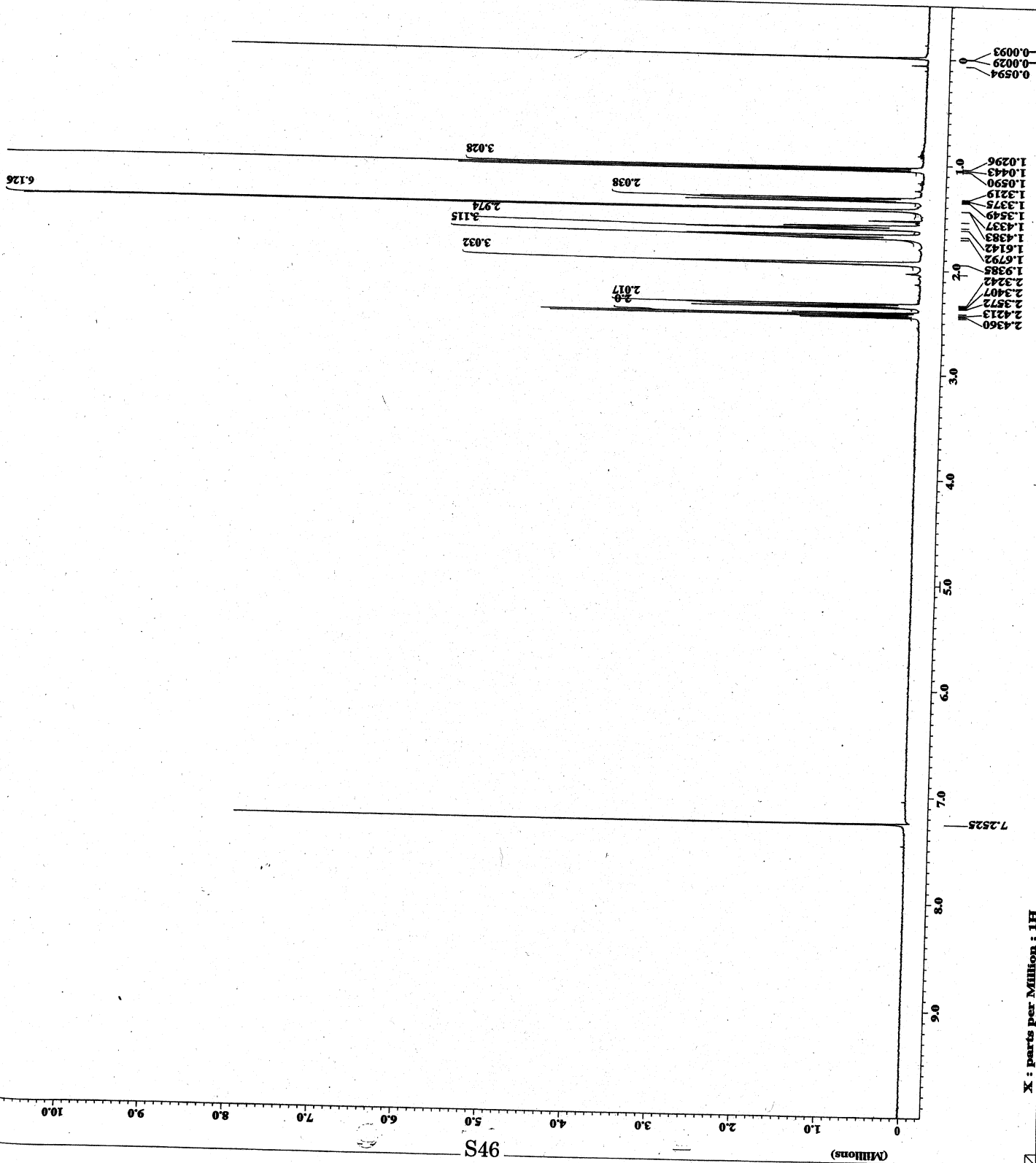


X : parts per Million : 13C

ACQUISITION PARAMETERS
File Name = 1d_spectrum_4722
Sample =
Sample ID = BIP-188-544-7
Content = Sample 188-544-7
Creation Date = 8-22-2007 13:17:30
Revision Date = 10-22-2007 07:12:16
Spec Site = MCF500
Spec Type = NMR
Data Format = 1D COMPLEX
Dimensions = 1
Mix Site = 1884
Mix Units = [ppm]
Scans = 4
Fid_return = 1
X_domain = 1H
X_offset = 0
X_freq = 500.1364160 [MHz]
X_name = 7-20735791 [Hz]
Solvent = CDCl3
Spin_get = 16 [Hz]
Spin_put = 24.5 [Hz]
Recvr_gain = 11.7473579 [V]
Field_strength = 11.7473579 [V]
Filter_mode = NOISEGATE
Filter_width = 3.75119936 [Hz]



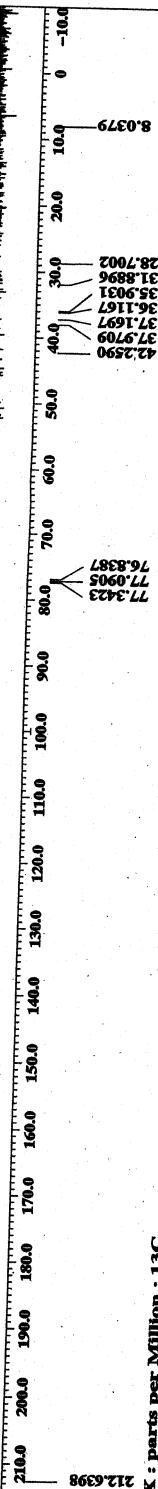
30



ACQUISITION PARAMETERS
File Name = 1d_13c_spectrum.294
Author =
Sample ID = RM-189-474-7
Content = Single Pulse with Broad
Creation Date = 9-28-2007 14:36:57
Revision Date = 11-28-2007 03:30:25
Spec Site = RM300
Spec Type = DELTA_BK
Data Format = ID COMPAK
Dimensions = 1
Dim Size = 13768
Dim Units = [mm]
Scans = 400
Acq_return = 1
C1_offset = 13C [ppm]
X_offset = 125.771447 [ppm]
X_freq = 31.44634088 [MHz]
Solvent = CDCl3/CDCl3-D
Spin_set = 14 [Hz]
Acq_time = 25.2 [min]
Nuc1 = 13C
Field_strength = 11.743759 [T]
Filter_mode = HORTZSCHMIDT
Filter_width = 15.72066221 [kHz]

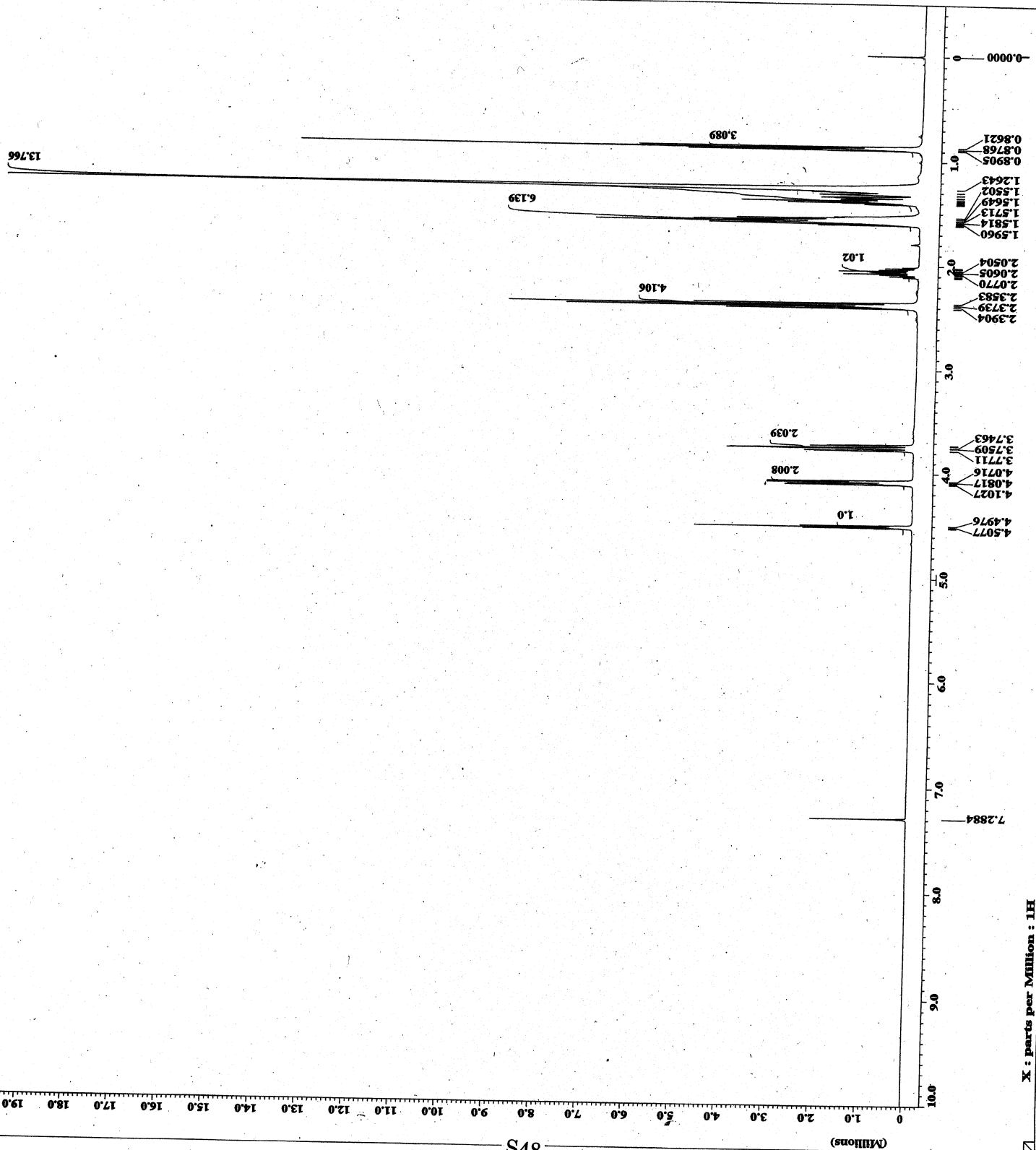
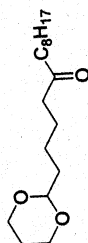


30

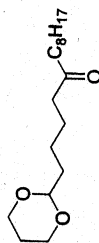


X : parts per Million : 13C

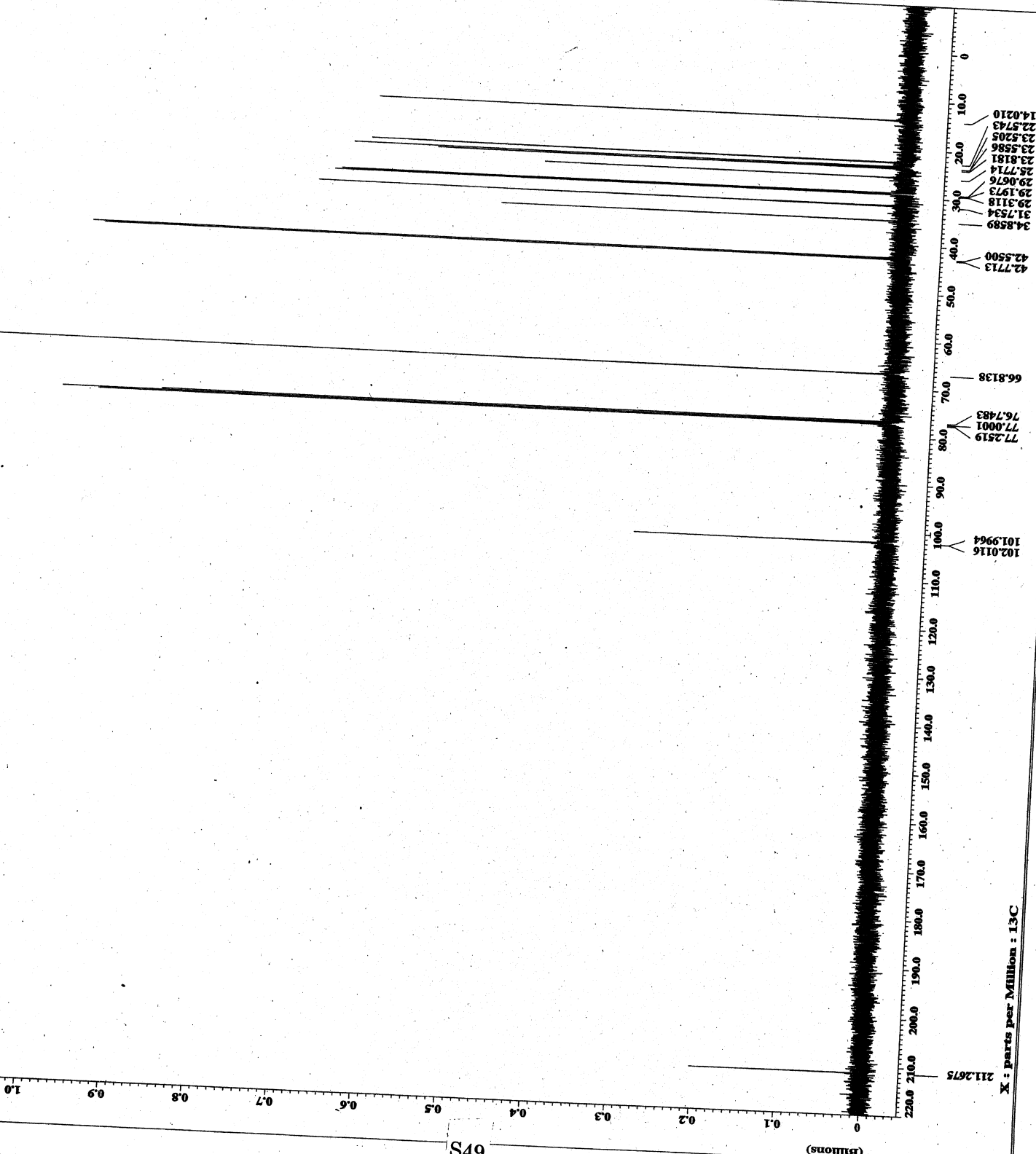
--- ACQUISITION PARAMETERS ---
 File Name = id_spectrum.5994
 Filter ID =
 Sample =
 Content =
 Creation Date = 8-MAR-2007 18:49:30
 Revision Date = 10-MAR-2007 08:14:41
 Spec Site = ECP500
 Spec Type = NMR
 Data Format = 1D COMPAK
 Dimensions =
 Dim Title =
 Dim 1 =
 Dim 2 =
 Dim 3 =
 Dim 4 =
 Dim 5 =
 Mod_return =
 X_domain =
 X_offset =
 X_sweep =
 Solvent =
 Spin_get =
 Temp_get =
 Field_strength =
 Filter_mode =
 Filter_width =



ACQUISITION PARAMETERS
 File Name: 14_13C_spectrum.464
 Author: R50-132-RVAC-Dr1
 Sample ID: Single Pulse with Broad
 Creation Date: 8-Mar-2007 19:05:55
 Revision Date: 10-Mar-2007 09:31:23
 Spec Site: RCT500
 Spec Type: 13C
 Data Format: 13C
 Dimensions: 13C
 Num Title: 13C
 Num Size: 32768
 Num Units: 13C
 Num: 400
 Mod_return: 13C
 X_domain: 100 [ppm]
 X_offset: 125.77787547 [MHz]
 X_freq: 125.77787547 [MHz]
 X_gain: 31.44654088 [dB]
 Solvent: CDCl3
 Spin_set: 15 [Hz]
 Temp_set: 25.3 [degC]
 Field_gen: 30
 Field_Coupling: 11.7473579 [Hz]
 Filter_Mod: 13C
 Filter_Width: 15.72066221 [kHz]



3d

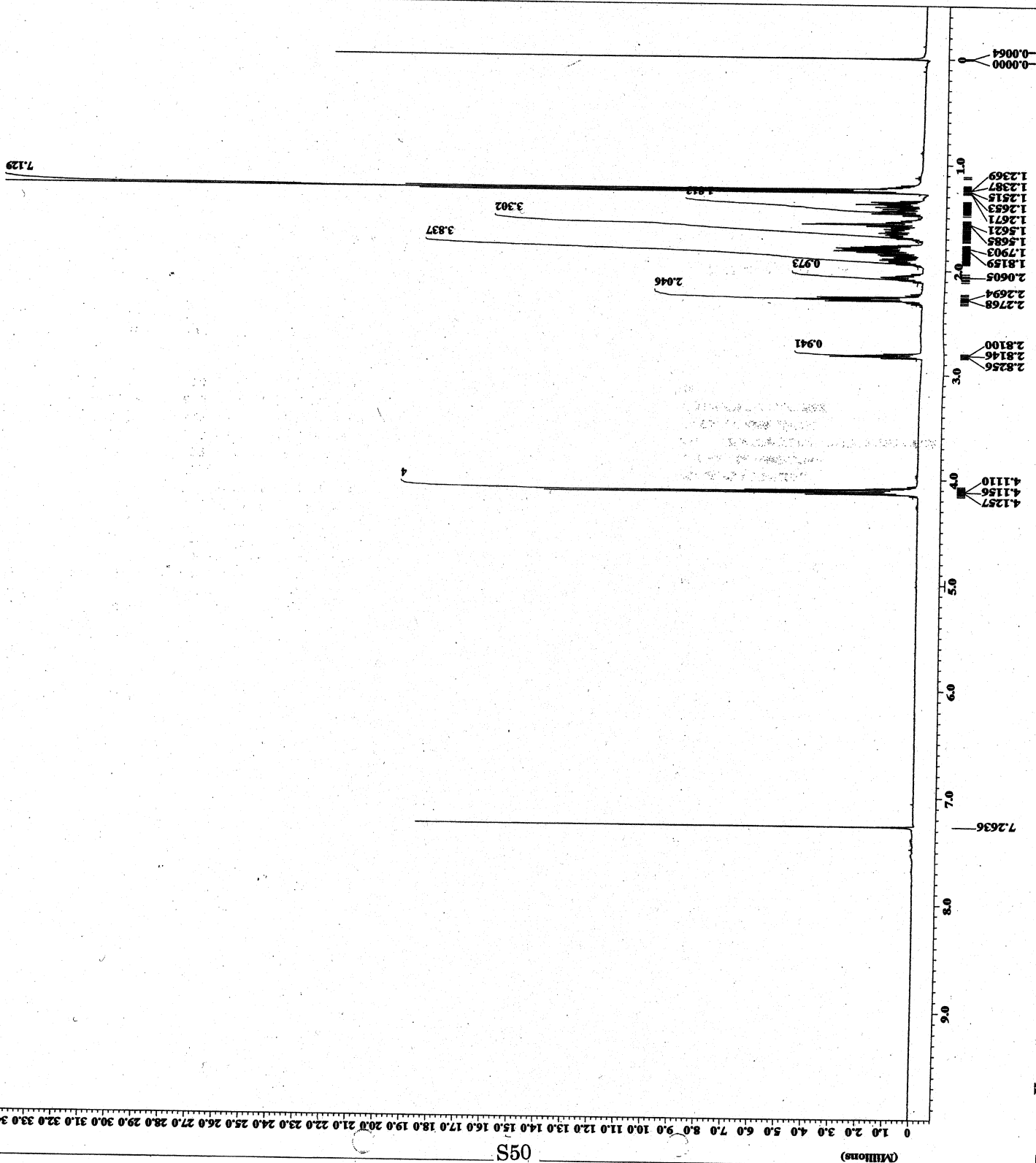
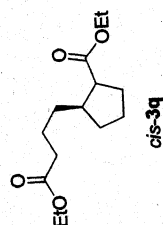


X: parts per Million : 13C

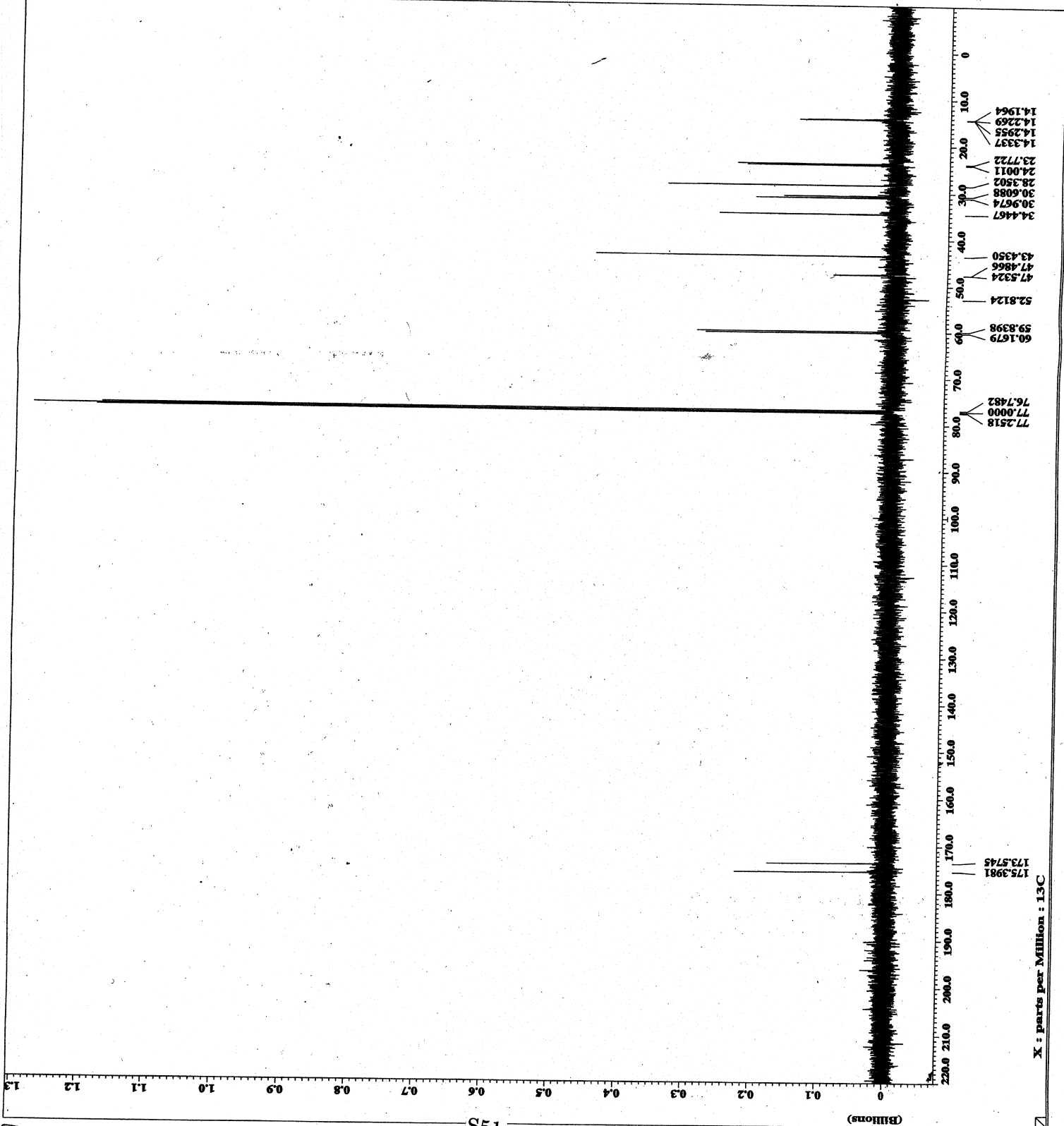
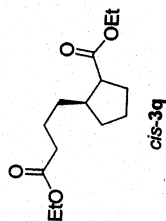
```

File Name      = 18.3
Acquisition Parameters
-
Sample         = RST-103-HVZ-4-254
Sample ID      = Single Pulse Experiment
Creation Date   = 23-Nov-2006 16:00:12
Version Date   = 21-Nov-2007 12:51:10
Spec Site      = MC3500
Data Path       = DATA_RAW
Data Format      = 1D COMPAK
Dimensions      = X
Min title       = 1E
Min title       = 1594
Min title       = 1594
Min title       = 8
Min title       = 8
Min title       = 8
Mod return      = 1
X domain        = 1E
X offset        = 5 [mm]
X sweep         = 7.0E-15241602 [Hz]
Y domain        = 1E
Y offset        = 7.0E-15241602 [Hz]
Y sweep         = 7.0E-15241602 [Hz]
Solvent         = CDCl3/NO2/CDCl3
Spin get        = 15 [Hz]
Pulse get       = 23.2 [deg]
Recf get        = 15
Recf get        = 23.2 [deg]
Filter strength = 15
Filter strength = 23.2 [deg]
Filter mode     = 3
Filter width    = 3.7471579 [Hz]
Filter width    = 3.7471579 [Hz]

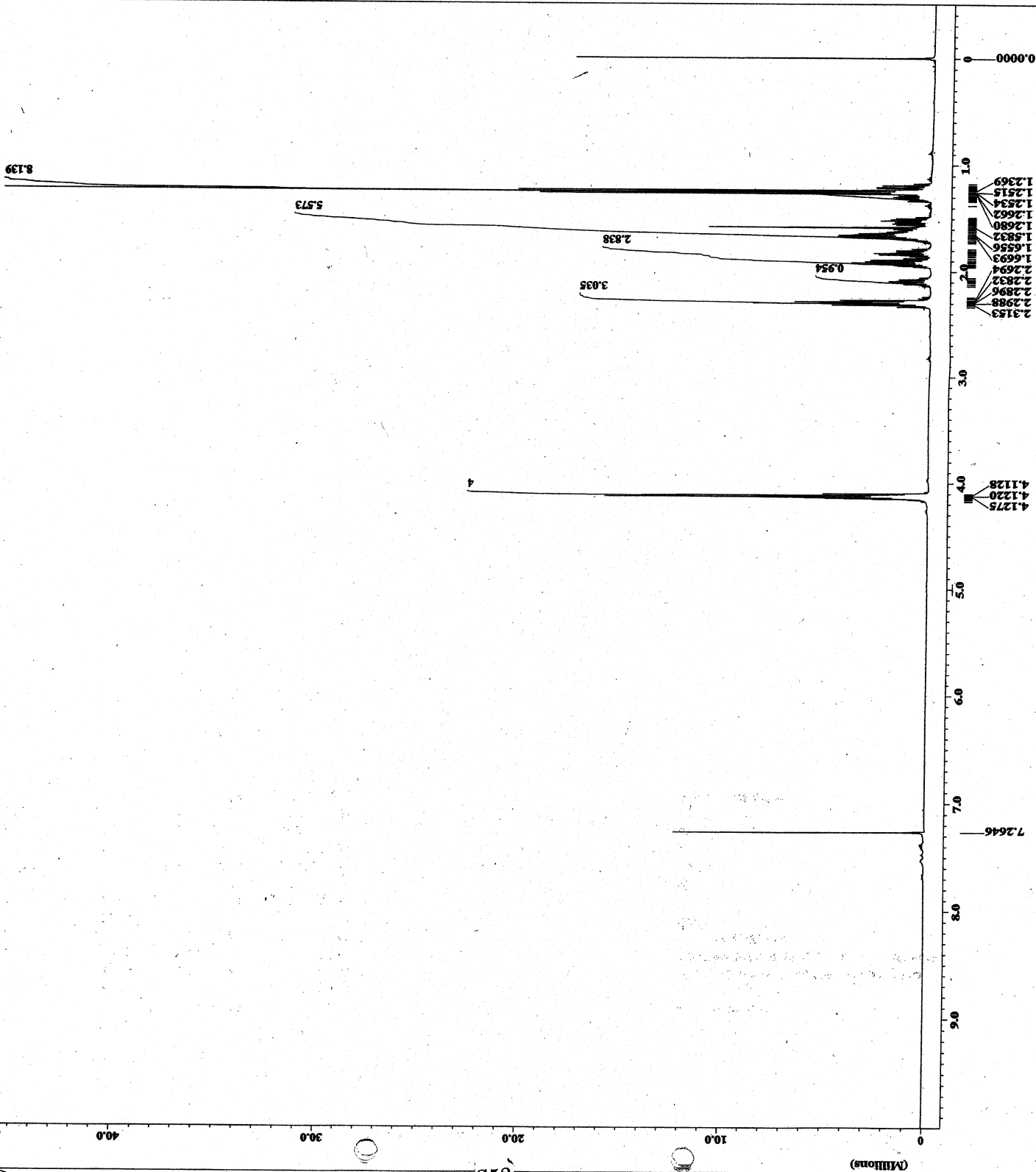
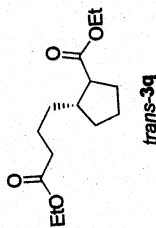
```



--- ACQUISITION PARAMETERS ---
 File Name = 13C.1
 Author =
 Sample ID = MS-101-MPLC-4-fr4
 Compound = Methyl Pile with Broad
 Creation Date = 21-PM-2006 21:24:02
 Revision Date = 28-MR-2007 13:44:58
 Spec Site = MCP500
 Spec Type = 13C NMR
 Data Format = 1D COMPAR
 Dimensions =
 N1a Title = 13C
 N1a Size = 32768
 N1a Units = [ppm]
 N1a Scan = 1
 Mod_return = 13C
 X_domain = 100 [ppm]
 X_offset = 125.77787947 [ppm]
 X_freq = 125.7684084 [ppm]
 X_solvent = CDCl3
 Spin_get = 14 [Hz]
 Temp_get = 23.4 [degC]
 Recvz_gain = 30
 P1d1_strength = 11.7473579 [V]
 P1d1_width = 15.72066221 [mm]
 Filter_width = 15.72066221 [mm]



--- ACQUISITION PARAMETERS ---
 File Name = E.2
 Author =
 Sample ID = RMP-103-2007-4-2-1
 Creation Date = 23-02-2007 17:52:14
 Revision Date = 28-02-2007 12:41:31
 Spec Site = MCP500
 Spec Type = NMR
 Data Format = 1D
 Dimensions = 1D
 NMR Title = 1D
 NMR Size = 16384
 NMR Units = 1
 Mod_Return = 1
 X_domain = 1
 X_offset = 500.16241602 [Hz]
 X_freq = 500.1360751 [Hz]
 Solvent = CDCl3
 Spin_get = 14 [Hz]
 Temp_get = 22.6 [°C]
 Acq_gain = 24
 Filtration = 7473579 [Hz]
 Filtration_width = 3.75119936 [Hz]



--- ACQUISITION PARAMETERS ---
 File Name = C:\S
 Author =
 Sample ID = MSU-103-MSU04-fcl
 Content = Single Pulse with Broad
 Creation Date = 23-FEB-2006 21:04:38
 Revision Date = 28-MAR-2007 12:57:28
 Spec Site = MSU000
 Spec Type = NMR
 Data Format = ID COMPLEX
 Data Name =
 Data Size = 100
 Data Units = [ppm]
 Scans = 1000
 Mod Return = 1
 X Offset = 100
 X Scale = 100
 X Freq = 125.77777777777777 [MHz]
 X Sweep = 31.44554088 [Hz]
 Solvent = CHLOROFORM-D
 Spin Get = 15 [Hz]
 Spin Set = 15 [Hz]
 Recv Gain = 30.1 [dB]
 Field Strength = 11.7473579 [T]
 Filter Mode = NOTREMOVED
 Filter Width = 15.72066221 [kHz]

