Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Ullmann *N*-Arylation/2-Amidation Cascade by Self-Relay Copper Catalysis: One-pot Synthesis of Indolo[1,2-*a*]quinazolinones

Supporting Information

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EXPERIMENTAL

1. General Methods.

Melting points were recorded with a Yamato MP21 and are uncorrected. High-resolution MS spectra were recorded with a Micromass AutoSpec 3100 and a JEOL JMS-T100LP mass spectrometers. IR spectra were measured with a Shimadzu IRAffinity-1 spectrometer. The NMR experiments were performed with a JEOL JNM-ECA500 (500 MHz) spectrometer, and chemical shifts are expressed in ppm (δ) with TMS as an internal reference. Column chromatography, Flash column chromatography and Medium Pressure Liquid Chromatography (MPLC) were performed on silica gel (Silica Gel 60N, Kanto Chemical Co., Ltd.). Methyl 5-methoxyindole-3-carboxylate (**1b**), Methyl 5-chloroindole-3-carboxylate (**1c**), Methyl 5-bromoindole-3-carboxylate (**1c**), and Methyl 5-nitroindole-3-carboxylate (**1d**) were prepared according to the reported procedures[1-2].

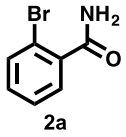
2. Synthesis of 2-Halobenzamides 2a-j:

General procedure A for Preparation of Substrate 2a-j:

DMF (0.1 mL) was added to a mixture of 2-halobenzoic acids (50 mmol) in thionyl chloride (20 mL) at room temperature and reflux. After 3 h, thionyl chloride was removed in vacuo and to give the crude acid chloride. To a solution of the crude acid chloride in THF (80 mL), amine (100 mmol) was added dropwise at 0 °C and the mixture was stirred at room temperature. After 16 h, the mixture was added to 10% HCl solution at 0 °C, extracted with AcOEt (300 mL), washed with brine, and dried over $MgSO_4$. The solvent was removed, and the residue was purified by recrystallization (EtOH) to give 2-halobenzamides **2a-l**.

2-Bromobenzamide (2a).

Following the general procedure A from 2-bromobenzoic acid, **2a** (8.62 g, 86%) was obtained as white solids.

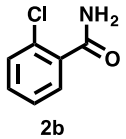


2a

8.62 g, 86%. White solids. Mp: 165-167 °C (EtOH). IR ($CHCl_3$): 3304, 1668 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 6.14 (br s, 2H), 7.29 (td, *J* = 1.7, 8.0 Hz, 1H), 7.37 (td, *J* = 1.2, 8.0 Hz, 1H), 7.60 (dd, *J* = 1.2, 7.7 Hz, 1H), 7.63 (dd, *J* = 1.8, 7.5 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 119.3, 127.7, 130.0, 131.8, 133.7, 136.7, 169.4. HR-ESI-MS *m/z*: Calcd for C_7H_7BrNO [$(M+H)^+$]: 199.9711, 201.9691. Found 199.9715, 201.9690.

2-Chlorobenzamide (2b).

Following the general procedure A from 2-chlorobenzoic acid, **2b** (6.09 g, 78%) was obtained as white solids.

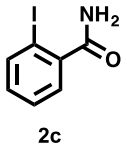


2b

6.09 g, 78%. White solids. Mp: 139-141 °C (EtOH). IR ($CHCl_3$): 3400, 1665 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 7.31-7.35 (m, 1H), 7.37-7.41 (m, 2H), 7.44 (d, *J* = 8.1 Hz, 1H), 7.55 (br s, 1H), 7.83 (br s, 1H). ^{13}C-NMR (DMSO-d_6) δ: 127.5, 129.2, 130.1, 131.1, 137.7, 168.7. HR-ESI-MS *m/z*: Calcd for C_7H_7ClNO [$(M+H)^+$]: 156.0216, 158.0187. Found 156.0216, 158.0192.

2-Iodobenzamide (2c).

Following the general procedure A from 2-iodobenzoic acid, **2c** (8.92 g, 72%) was obtained as white solids.



2c

8.92 g, 72%. White solids. Mp: 185-187 °C (EtOH). IR ($CHCl_3$): 3304, 1670 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 7.11 (td, J = 1.2, 7.5 Hz, 1H), 7.30 (d, J = 7.5 Hz, 1H), 7.39 (t, J = 7.5 Hz, 1H), 7.48 (br s, 1H), 7.79 (br s, 1H), 7.83 (d, J = 8.1 Hz, 1H). ^{13}C-NMR(DMSO-d_6) δ: 93.7, 128.3, 128.5, 131.1, 139.7, 143.7, 171.2. HR-ESI-MS *m/z*: Calcd for C_7H_6INNaO [(M+Na)$^+$]: 269.9392. Found 269.9395.

2-Bromo-*N*-methylbenzamide (2d).

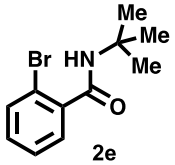
Following the general procedure A from 2-bromobenzoic acid, **2d** (7.59 g, 71%) was obtained as white solids.

2d

7.59 g, 71%. White solids. Mp: 132-134 °C (EtOH). IR ($CHCl_3$): 3324, 1716, 1663 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.00 (d, J = 5.2 Hz, 3H), 6.04 (br s, 1H), 7.25 (td, J = 1.8, 7.5 Hz, 1H), 7.33 (d, J = 8.0 Hz, 1H), 7.50 (dd, J = 1.1, 8.0 Hz, 1H), 7.56 (d, J = 8.0 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 26.8, 119.3, 127.6, 129.7, 131.3, 133.4, 137.9, 168.4. HR-ESI-MS *m/z*: Calcd for $C_8H_8BrNNaO$ [(M+Na)$^+$]: 235.9687, 237.9666. Found 235.9682, 237.9670.

2-Bromo-*N*-(*tert*-buthyl)benzamide (2e).

Following the general procedure A from 2-bromobenzoic acid, **2e** (6.04 g, 47%) was obtained as white solids.

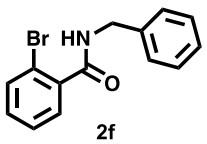


2e

6.04 g, 47%. White solids. Mp: 97-100 °C (EtOH). IR ($CHCl_3$): 3321, 1667 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 1.32 (s, 9H), 7.26-7.29 (m, 2H), 7.36 (t, J = 6.9 Hz, 1H), 7.57 (d, J = 8.0 Hz, 1H), 7.97 (br s, 1H). ^{13}C-NMR (DMSO-d_6) δ: 28.9, 51.4, 119.5, 127.9, 129.1, 130.8, 132.9, 140.7, 167.4 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{11}H_{14}BrNNaO$ [(M+Na)$^+$]: 278.0156, 280.0136. Found 278.0161, 280.0139.

***N*-Benzyl-2-bromobenzamide (2f).**

Following the general procedure A from 2-bromobenzoic acid, **2f** (11.94 g, 82%) was obtained as white solids.

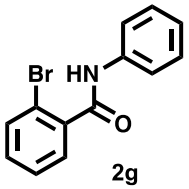


2f

11.94 g, 82%. White solids. Mp: 115-117 °C (EtOH). IR ($CHCl_3$): 3319, 3305, 1663 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.65 (d, J = 5.7 Hz, 2H), 6.28 (br s, 1H), 7.24-7.31 (m, *2*H), 7.33-7.39 (m, 5H), 7.55 (d, J = 7.5 Hz, 1H), 7.57 (d, J = 8.0 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 44.3, 119.4, 127.7, 127.8, 128.1, 128.9, 129.7, 131.4, 133.5, 137.7, 137.8, 167.6 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{14}H_{12}BrNNaO$ [(M+Na)$^+$]: 312.0000, 313.9979. Found 312.0000, 313.9982.

2-Bromo-*N*-phenylbenzamide (2g).

Following the general procedure A from 2-bromobenzoic acid, **2g** (8.00 g, 58%) was obtained as white solids.

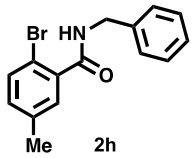


2g

8.00 g, 58%. White solids. Mp: 125-126 °C (EtOH). IR ($CHCl_3$): 3304, 1678, 1600 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 7.16 (t, J = 7.5 Hz, 1H), 7.28 (t, J = 8.0 Hz, 1H), 7.35 (t, J = 8.0 Hz, 3H), 7.56-7.63 (m,

4H), 7.91 (br s, 1H). ^{13}C-NMR ($CDCl_3$) δ: 119.4, 120.2, 124.9, 127.8, 129.2, 129.8, 131.7, 133.6, 137.7, 137.9, 165.8 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{13}H_{10}BrNNaO$ [$(M+Na)^+$]: 297.9843, 299.9823. Found 297.9847, 299.9824.

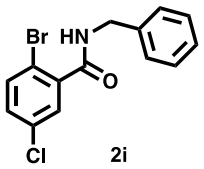
***N*-Benzyl-2-bromo-5-methylbenzamide (2h).**



Following the general procedure A from 2-bromo-5-methylbenzoic acid, **2h** (10.37 g, 68%) was obtained as white solids.

10.37 g, 68%. White solids. Mp: 130-132 °C (EtOH). IR ($CHCl_3$): 3323, 1663 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 2.26 (s, 3H), 4.40 (d, *J* = 5.8 Hz, 2H), 7.14 (dd, *J* = 2.3, 8.6 Hz, 1H), 7.21-7.24 (m, 2H), 7.29-7.35 (m, 4H), 7.48 (d, *J* = 8.0 Hz, 1H), 8.89 (t, *J* = 5.7 Hz, 1H). ^{13}C-NMR (DMSO-d_6) δ: 20.8, 42.9, 116.1, 127.3, 127.8, 128.8, 129.8, 132.0, 133.0, 137.7, 139.4, 139.7, 167.9 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{15}H_{15}BrNNO$ [$(M+H)^+$]: 304.0337, 306.0317. Found 304.0336, 306.0323.

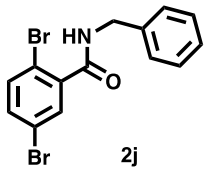
***N*-Benzyl-2-bromo-5-chlorobenzamide (2i).**



Following the general procedure A from 2-bromo-5-chlorobenzoic acid, **2i** (11.69 g, 72%) was obtained as white solids.

11.69 g, 72%. White solids. Mp: 164-165 °C (EtOH). IR ($CHCl_3$): 3306, 1667 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 4.41 (d, *J* = 5.7 Hz, 1H), 7.23 (t, *J* = 6.9 Hz, 1H), 7.29-7.35 (m, 4H), 7.41 (dd, *J* = 2.3, 8.0 Hz, 1H), 7.48 (d, *J* = 2.3 Hz, 1H), 7.65 (d, *J* = 8.6 Hz, 1H), 9.02 (t, *J* = 5.7 Hz, 1H). ^{13}C-NMR (DMSO-d_6) δ: 43.1, 118.0, 127.4, 127.9, 128.9, 129.0, 131.2, 132.9, 135.0, 139.3, 141.1, 166.4 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{14}H_{11}BrClNNaO$ [$(M+Na)^+$]: 345.9610, 347.9590. Found 345.9607, 347.9587.

***N*-Benzyl-2,5-dibromobenzamide (2j).**



Following the general procedure A from 2,5-dibromobenzoic acid, **2j** (11.33 g, 61%) was obtained as white solids.

11.33 g, 61%. White solids. Mp: 176-178 °C (EtOH). IR ($CHCl_3$): 3433, 1668 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 4.40 (d, *J* = 5.8 Hz, 1H), 7.22 (t, *J* = 6.3 Hz, 1H), 7.29-7.34 (m, 4H), 7.53 (dd, *J* = 2.9, 8.6 Hz, 1H), 7.58 (d, *J* = 4.0 Hz, 1H), 7.59 (d, *J* = 2.3 Hz, 1H), 9.01 (t, *J* = 5.8 Hz, 1H). ^{13}C-NMR (DMSO-d_6) δ: 51.5, 87.9, 113.1, 115.6, 117.7, 121.2, 122.7, 124.3, 125.2, 127.0, 130.0, 131.1, 135.3, 138.7, 142.3, 158.0, 166.2 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{14}H_{11}Br_2NNaO$ [$(M+Na)^+$]: 389.9105, 391.9085, 393.9064. Found 389.9111, 391.9076, 393.9064.

3. Synthesis of 3a, 4a, 5:

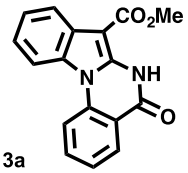
General procedure B for Table 1:

Base (2 mmol) was added to a mixture of indole (1 mmol), **2a** (300 mg, 1.5 mmol), Cu-catalyst (0.1 mmol) in solvent (10 mL) at room temperature and reflux for 16 h. After adding Et_2O (20 mL) to the mixtures at room temperature, insoluble materials were removed by filtration through silicagel pad with suction. The filtrate was concentrated in vaccuo, and the residue was purified by silica gel column chromatography with hexane/AcOEt (5/1) to give **3a** as white solids.

General procedure C for Scheme 2:

Cesium carbonate (652 mg, 2 mmol) was added to a mixture of indole (1 mmol), 2-halobenzamide (1.5 mmol), CuBr (14.4 mg, 0.1 mmol) in DMSO (10 mL) at room temperature and reflux under air. After adding Et_2O (20 mL) to the mixtures at room temperature, insoluble materials were removed by filtration through silicagel pad with suction. The filtrate was concentrated in vaccuo, and the residue was purified by silica gel column chromatography with hexane/AcOEt (5/1) to give **3**.

Methyl 5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3a).

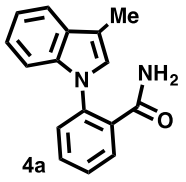


3a

Following the general procedure C, **3a** (214 mg, 73%) was obtained as white solids.

214 mg, 73%. White solids. Mp: 204-207 °C (EtOH). IR ($CHCl_3$): 3404, 1705, 1612, 1597 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.01 (s, 3H), 7.34 (td, *J* = 1.8, 7.7 Hz, 1H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.87 (td, *J* = 1.2, 7.6 Hz, 1H), 8.10 (d, *J* = 8.6 Hz, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 8.35 (d, *J* = 8.6 Hz, 1H), 8.43 (dd, *J* = 1.7, 8.0 Hz, 1H), 10.57 (br s, 1H). ^{13}C-NMR ($CDCl_3$) δ: 51.5, 87.9, 113.1, 115.6, 117.7, 121.2, 122.7, 124.3, 125.2, 127.0, 130.0, 131.1, 135.3, 138.7, 142.3, 158.0, 166.2. HR-ESI-MS *m/z*: Calcd for $C_{17}H_{12}N_2NaO_3$ [$(M+Na)^+$]: 315.0746. Found 315.0748.

2-(3-Methyl-1*H*-indol-1-yl)benzamide (4a).

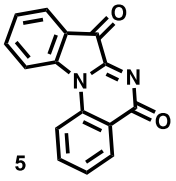


4a

Following the general procedure C, **4a** (159 mg, 64%) was obtained as colorless oil.

159 mg, 64%. Colorless oil. IR ($CHCl_3$): 3400, 1672, 1603, 1584 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 2.38 (s, 3H), 5.30 (br s, 1H), 5.91 (br s, 1H), 7.00 (s, 1H), 7.16-7.19 (m, 3H), 7.36 (d, *J* = 8.1 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.57 (t, *J* = 7.5 Hz, 1H), 7.61-7.63 (m, 1H), 8.01 (d, *J* = 7.5 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 9.8, 110.3, 114.0, 119.4, 120.4, 123.1, 126.2, 128.2, 128.4, 129.5, 131.2, 131.9, 132.2, 136.9, 137.3, 168.4. HR-ESI-MS *m/z*: Calcd for $C_{16}H_{14}N_2NaO$ [$(M+Na)^+$]: 273.1004. Found 273.1002.

Indolo[1,2-*a*]quinazoline-5,7-dione (5).



5

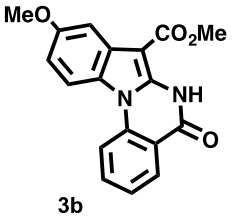
Following the general procedure C, **5** (55 mg, 22%) was obtained as white solids.

55 mg, 22%. White solids. Mp: 256-258 °C (EtOH). IR ($CHCl_3$): 3442, 3236, 1728, 1694, 1599 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 7.45 (t, *J* = 7.5 Hz, 1H), 7.71 (dt, *J* = 4.0, 4.6 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.92 (d, *J* = 4.6 Hz, 2H), 8.29 (d, *J* = 7.5 Hz, 1H), 8.45 (d, *J* = 8.0 Hz, 1H). ^{13}C-NMR (DMSO-d_6) δ: 117.6, 122.8, 123.9, 125.2, 127.5, 127.5, 130.3, 130.4, 135.6, 138.3, 145.5, 146.6,

147.1, 158.2, 182.9. HR-ESI-MS *m/z*: Calcd for $C_{15}H_9N_2O_2$ $[(M+H)^+]$: 249.0664. Found 249.0663.

4. Synthesis of 3a-o, 4b:

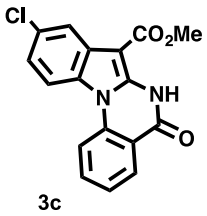
Methyl 9-methoxy-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3b).



3b

Following the general procedure C, **3b** (168 mg, 52%) was obtained as white solids.
168 mg, 52%. White solids. Mp: 196-198 °C (EtOH). IR ($CHCl_3$): 3368, 3300, 1681, 1602 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.89 (s, 3H), 3.98 (s, 3H), 6.86 (d, *J* = 9.2 Hz, 1H), 7.43 (t, *J* = 8.2 Hz, 1H), 7.52 (s, 1H), 7.82 (t, *J* = 7.5 Hz, 1H), 7.88 (dd, *J* = 3.5, 9.2 Hz, 1H), 8.18 (dd, *J* = 3.5, 8.1 Hz, 1H), 8.37 (d, *J* = 8.0 Hz, 1H), 10.41 (br s, 1H). ^{13}C-NMR ($CDCl_3$) δ: 51.4, 55.7, 87.7, 104.0, 110.8, 113.8, 115.2, 117.4, 124.9, 125.5, 128.3, 129.9, 135.2, 138.4, 142.2, 156.9, 157.8, 165.9. HR-ESI-MS *m/z*: Calcd for $C_{18}H_{15}N_2O_4$ [$(M+H)^+$]: 323.1032. Found 323.1030.

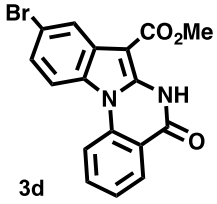
Methyl 9-chloro-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3c).



3c

Following the general procedure C, **3c** (259 mg, 79%) was obtained as white solids.
259 mg, 79%. White solids. Mp: 230-233 °C (EtOH). IR ($CHCl_3$): 3300, 1684, 1607 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.00 (s, 3H), 7.24-7.26 (m, 1H), 7.49 (t, *J* = 8.0 Hz, 1H), 7.86 (t, *J* = 8.6 Hz, 1H), 7.95 (d, *J* = 8.6 Hz, 1H), 8.01 (s, 1H), 8.20 (d, *J* = 8.6 Hz, 1H), 8.41 (d, *J* = 7.5 Hz, 1H), 10.49 (br s, 1H). ^{13}C-NMR ($CDCl_3$) δ: 51.7, 87.5, 113.9, 115.4, 117.7, 120.8, 122.7, 125.5, 128.3, 129.3, 130.1, 135.4, 138.2, 142.7, 157.7, 165.6 (one sp^2 signal was not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{17}H_{11}ClN_2O_3$ [$(M+H)^+$]: 327.0536, 329.0507. Found 327.0535, 329.0512.

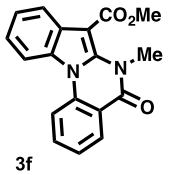
Methyl 9-bromo-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3d).



3d

Following the general procedure C, **3d** (304 mg, 82%) was obtained as white solids.
304 mg, 82%. White solids. Mp: 239-242 °C (EtOH). IR ($CHCl_3$): 3300, 1674, 1608 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.00 (s, 3H), 7.41 (dd, *J* = 1.7, 8.6 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.87 (t, *J* = 8.1 Hz, 1H), 7.92 (d, *J* = 9.2 Hz, 1H), 8.20 (s, 1H), 8.22 (d, *J* = 8.6 Hz, 1H), 8.42 (d, *J* = 7.4 Hz, 1H), 10.49 (br s, 1H). ^{13}C-NMR ($CDCl_3$) δ: 51.7, 87.4, 114.3, 115.4, 117.7, 117.9, 123.8, 125.4, 125.5, 128.7, 129.7, 130.1, 135.4, 138.2, 142.7, 157.7, 165.6 (one sp^2 signal was not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{17}H_{11}BrN_2NaO_3$ [$(M+Na)^+$]: 392.9851, 394.9830. Found 392.9849, 394.9833.

Methyl 6-methyl-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3f).

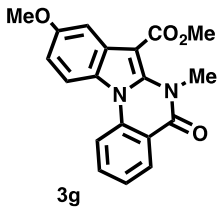


3f

Following the general procedure C, **3f** (260 mg, 85%) was obtained as white solids.
260 mg, 85%. White solids. Mp: 192-193 °C (EtOH). IR ($CHCl_3$): 1697, 1678, 1604 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.77 (s, 3H), 4.01 (s, 3H), 7.35-7.40 (m, 2H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.83 (dt, *J* = 1.8, 7.5 Hz, 1H), 8.10-8.14 (m, 2H), 8.37 (d, *J* = 8.6 Hz, 1H), 8.43 (dd, *J* = 1.2, 7.5 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 36.0, 51.7, 92.8, 113.0, 115.3, 116.9, 121.1, 122.9, 123.8, 124.8, 128.3, 130.0, 130.5, 134.8, 138.0, 141.4, 159.9, 164.7. HR-ESI-MS *m/z*: Calcd for $C_{18}H_{14}N_2NaO_3$ [$(M+Na)^+$]: 329.0902. Found 329.0902.

Methyl 9-methoxy-6-methyl-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3g).

Following the general procedure C, **3g** (239 mg, 71%) was obtained as white solids.

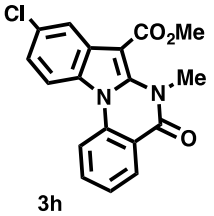


3g

239 mg, 71%. White solids. Mp: 206-208 °C (EtOH). IR ($CHCl_3$): 1684, 1603 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.74 (s, 3H), 3.92 (s, 3H), 3.99 (s, 3H), 6.94 (dd, *J* = 2.9, 9.2 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.59 (d, *J* = 2.3 Hz, 1H), 7.79 (dt, *J* = 1.2, 6.9 Hz, 1H), 7.98 (d, *J* = 9.2 Hz, 1H), 8.26 (d, *J* = 8.6 Hz, 1H), 8.41 (dd, *J* = 1.2, 8.0 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 36.3, 51.7, 55.8, 92.6, 103.9, 111.4, 113.8, 115.0, 116.7, 124.6, 125.2, 129.6, 129.9, 134.8, 137.9, 141.8, 156.5, 159.9, 164.7. HR-ESI-MS *m/z*: Calcd for $C_{19}H_{17}N_2O_4$ [(M+H)$^+$]: 337.1188. Found 337.1181.

Methyl 9-chloro-6-methyl-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3h).

Following the general procedure C, **3h** (299 mg, 88%) was obtained as white solids.

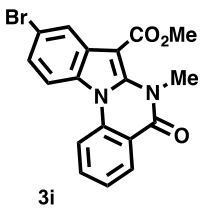


3h

299 mg, 88%. White solids. Mp: 196-198 °C (EtOH). IR ($CHCl_3$): 1699, 1681, 1602 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.75 (s, 3H), 4.01 (s, 3H), 7.29 (dd, *J* = 1.2, 8.6 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 1H), 7.83 (dt, *J* = 1.2, 7.4 Hz, 1H), 8.02 (d, *J* = 9.2 Hz, 1H), 8.07 (d, *J* = 1.7 Hz, 1H), 8.26 (d, *J* = 8.6 Hz, 1H), 8.43 (d, *J* = 8.0 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 36.1, 51.9, 92.4, 113.9, 115.2, 117.0, 120.8, 122.9, 125.2, 128.8, 129.6, 130.1, 135.0, 137.6, 142.3, 159.8, 164.2 (one sp^2 signal was not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{18}H_{13}ClN_2NaO_3$ [(M+Na)$^+$]: 363.0512, 365.0483. Found 363.0512, 365.0484.

Methyl 9-bromo-6-methyl-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3i).

Following the general procedure C, **3i** (351 mg, 91%) was obtained as white solids.

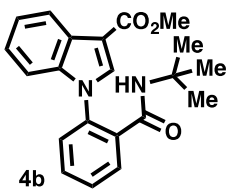


3i

351 mg, 91%. White solids. Mp: 210-212 °C (EtOH). IR ($CHCl_3$): 1701, 1682 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.75 (s, 3H), 4.01 (s, 3H), 7.44 (dd, *J* = 1.7, 8.6 Hz, 1H), 7.46 (t, *J* = 8.0 Hz, 1H), 7.83 (dt, *J* = 1.7, 7.7 Hz, 1H), 7.97 (d, *J* = 8.6 Hz, 1H), 8.22 (d, *J* = 1.7 Hz, 1H), 8.25 (d, *J* = 8.0 Hz, 1H), 8.43 (dd, *J* = 1.2, 8.1 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 36.1, 51.9, 92.3, 114.2, 115.2, 117.0, 117.4, 123.8, 125.2, 125.7, 129.2, 130.0, 130.2, 135.0, 137.6, 142.1, 159.8, 164.2. HR-ESI-MS *m/z*: Calcd for $C_{18}H_{13}BrN_2NaO_3$ [(M+Na)$^+$]: 407.0007, 408.9987. Found 407.0005, 408.9990.

Methyl 1-(2-(*tert*-butylcarbamoyl)phenyl)-1*H*-indole-3-carboxylate (4b).

Following the general procedure C, **4b** (190 mg, 54%) was obtained as colorless viscous oil.

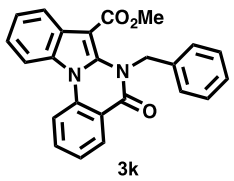


4b

1.90 mg, 54%. Colorless viscous oil. IR ($CHCl_3$): 3428, 3323, 1705, 1664 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 0.84 (s, 9H), 3.92 (s, 3H), 4.88 (br s, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.26 (td, *J* = 1.2, 8.6 Hz, 1H), 7.32 (td, *J* = 1.2, 8.1 Hz, 1H), 7.41 (dd, *J* = 1.7, 8.0 Hz, 1H), 7.55-7.62 (m, 2H), 7.88 (dd, *J* = 2.3, 7.5 Hz, 1H), 7.93 (s, 1H), 8.25 (d, *J* = 8.0 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 27.9, 51.3, 51.6, 109.7, 110.8, 122.0, 123.0, 124.2, 126.4, 127.9, 129.6, 130.7, 131.4, 134.5, 134.6, 135.5, 137.9, 164.9, 165.2 (two sp^3 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{21}H_{22}N_2NaO_3$ [(M+Na)$^+$]: 373.1528. Found 373.1529.

Methyl 6-benzyl-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3k).

Following the general procedure C, **3k** (355 mg, 93%) was obtained as white solids.

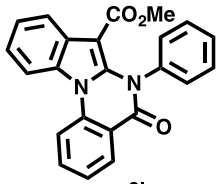


3k

355 mg, 93%. White solids. Mp: 162-164 °C (EtOH). IR ($CHCl_3$): 1693, 1682, 1603 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.85 (s, 3H), 5.94 (s, 2H), 7.11 (d, *J* = 6.9 Hz, 2H), 7.15-7.21 (m, 3H), 7.30-7.33 (m, 2H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.82 (dt, *J* = 1.2, 7.5 Hz, 1H), 7.90-7.92 (m, 1H), 8.09-8.11 (m, 1H), 8.35 (d, *J* = 8.6 Hz, 1H), 8.46 (dd, *J* = 1.7, 8.0 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 47.4, 51.9, 94.2, 113.0, 115.5, 116.9, 121.2, 122.8, 123.6, 124.8, 127.1, 127.4, 128.2, 128.5, 130.4, 134.9, 136.0, 138.1, 138.8. HR-ESI-MS *m/z*: Calcd for $C_{24}H_{18}N_2NaO_3$ [(M+Na) $^+$]: 405.1215. Found 405.1215.

Methyl 5-oxo-6-phenyl-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3l).

Following the general procedure C, **3l** (317 mg, 86%) was obtained as white solids.

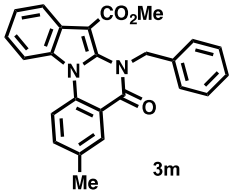


3l

317 mg, 86%. White solids. Mp: 212-215 °C (EtOH). IR ($CHCl_3$): 1686, 1603 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.22 (s, 3H), 7.34-7.38 (m, 2H), 7.41-7.44 (m, 2H), 7.47-7.53 (m, 4H), 7.83 (dt, *J* = 1.2, 7.5 Hz, 1H), 7.93-7.94 (m, 1H), 8.13-8.14 (m, 1H), 8.37 (d, *J* = 8.6 Hz, 1H), 8.42 (dd, *J* = 1.2, 8.0 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 51.5, 93.8, 113.0, 115.4, 117.1, 120.5, 123.0, 123.8, 124.7, 127.8, 128.4, 128.5, 129.2, 130.4, 130.5, 135.2, 138.5, 138.6, 139.0, 159.1, 163.9 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{23}H_{16}N_2NaO_3$ [(M+Na) $^+$]: 391.1059. Found 391.1060.

Methyl 6-benzyl-3-methyl-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3m).

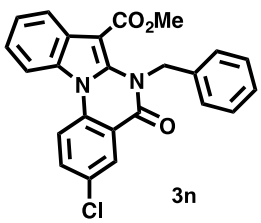
Following the general procedure C, **3m** (319 mg, 80%) was obtained as white solids.



3m

319 mg, 80%. White solids. Mp: 158-160 °C (EtOH). IR ($CHCl_3$): 1692, 1674 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 2.46 (s, 3H), 3.86 (s, 3H), 5.94 (s, 2H), 7.10 (d, *J* = 6.9 Hz, 2H), 7.13-7.21 (m, 3H), 7.27-7.29 (m, 2H), 7.55 (d, *J* = 8.6 Hz, 1H), 7.88-7.90 (m, 1H), 8.01 (d, *J* = 4.0 Hz, 1H), 8.15 (d, *J* = 7.5 Hz, 1H), 8.22 (s, 1H). ^{13}C-NMR ($CDCl_3$) δ: 20.9, 47.4, 51.9, 93.8, 112.9, 115.4, 116.6, 121.1, 122.7, 123.5, 127.1, 127.4, 128.1, 128.5, 130.1, 130.2, 134.8, 135.8, 136.1, 138.7, 160.1, 165.2 (three sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{25}H_{20}N_2NaO_3$ [(M+Na) $^+$]: 419.1372. Found 419.1372.

Methyl 6-benzyl-3-chloro-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3n).



3n

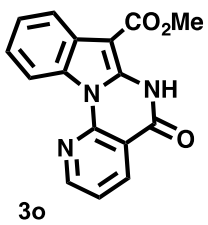
Following the general procedure C, **3n** (300 mg, 72%) was obtained as white solids.

300 mg, 72%. White solids. Mp: 204-206 °C (EtOH). IR ($CHCl_3$): 1697, 1678 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.85 (s, 3H), 5.91 (s, 2H), 7.09 (d, *J* = 7.4 Hz, 2H), 7.14-7.21 (m, 3H),

7.28-7.32 (m, 2H), 7.72 (dd, *J* = 2.3, 8.6 Hz, 1H), 7.88-7.89 (m, 1H), 7.98 (d, *J* = 3.5 Hz, 1H), 8.24 (dd, *J* = 4.0, 9.2 Hz, 1H), 8.39 (d, *J* = 2.0 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 47.5, 52.0, 94.6, 112.7, 116.9, 118.2, 121.3, 123.1, 123.9, 127.1, 127.5, 128.1, 128.6, 129.8, 130.1, 130.4, 134.9, 135.7, 136.5, 138.2, 158.9, 164.9 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{24}H_{17}ClN_2NaO_3$ [$(M+Na)^+$]: 439.0825, 441.0796. Found 439.0825, 441.0798.

Methyl 5-oxo-5,6-dihydropyrido[3',2':5,6]pyrimido[1,2-*a*]indole-7-carboxylate (3o).

Following the general procedure C, **3o** (155 mg, 53%) was obtained as white solids.



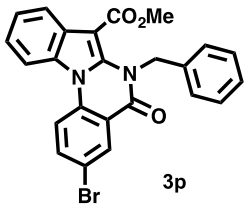
3o

155 mg, 53%. White solids. Mp: 222-224 °C (EtOH). IR ($CHCl_3$): 3304, 1689, 1628 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.99 (s, 3H), 7.29 (dt, *J* = 1.2, 8.6 Hz, 1H), 7.34 (dt, *J* = 1.1, 7.5 Hz, 1H), 7.41 (dd, *J* = 4.6, 7.5 Hz, 1H), 7.96 (d, *J* = 7.5 Hz, 1H), 8.60 (dd, *J* = 2.3, 8.0 Hz, 1H), 8.81 (dd, *J* = 2.3, 5.2 Hz, 1H), 8.92 (d, *J* = 8.0 Hz, 1H), 10.38 (br s, 1H). ^{13}C-NMR ($CDCl_3$) δ: 51.6, 89.1, 112.8, 116.3, 120.3, 120.8, 123.2, 124.8, 126.0, 130.8, 138.2, 141.2, 149.9, 153.9, 158.1, 166.1. HR-ESI-MS *m/z*: Calcd for $C_{16}H_{11}N_3NaO_3$ [$(M+Na)^+$]: 316.0698. Found 316.0700.

5. Synthesis of 3p:

Methyl 6-benzyl-3-bromo-5-oxo-5,6-dihydroindolo[1,2-*a*]quinazoline-7-carboxylate (3p).

Cesium carbonate (652 mg, 2 mmol) was added to a mixture of methyl indole-3-carboxylate (175 mg, 1 mmol), 2,5-dibromobenzamide (554 mg, 1.5 mmol), CuBr (14.4 mg, 0.1 mmol) in DMSO (10 mL) at room temperature and reflux for 48 h under air. After adding Et_2O (20 mL) to the mixtures at room temperature, insoluble materials were removed by filtration through silicagel pad with suction. The filtrate was concentrated in vaccuo, and the residue was purified by silica gel column chromatography with hexane/AcOEt (5/1) to give **3p (**315 mg, 68%) as white solids.



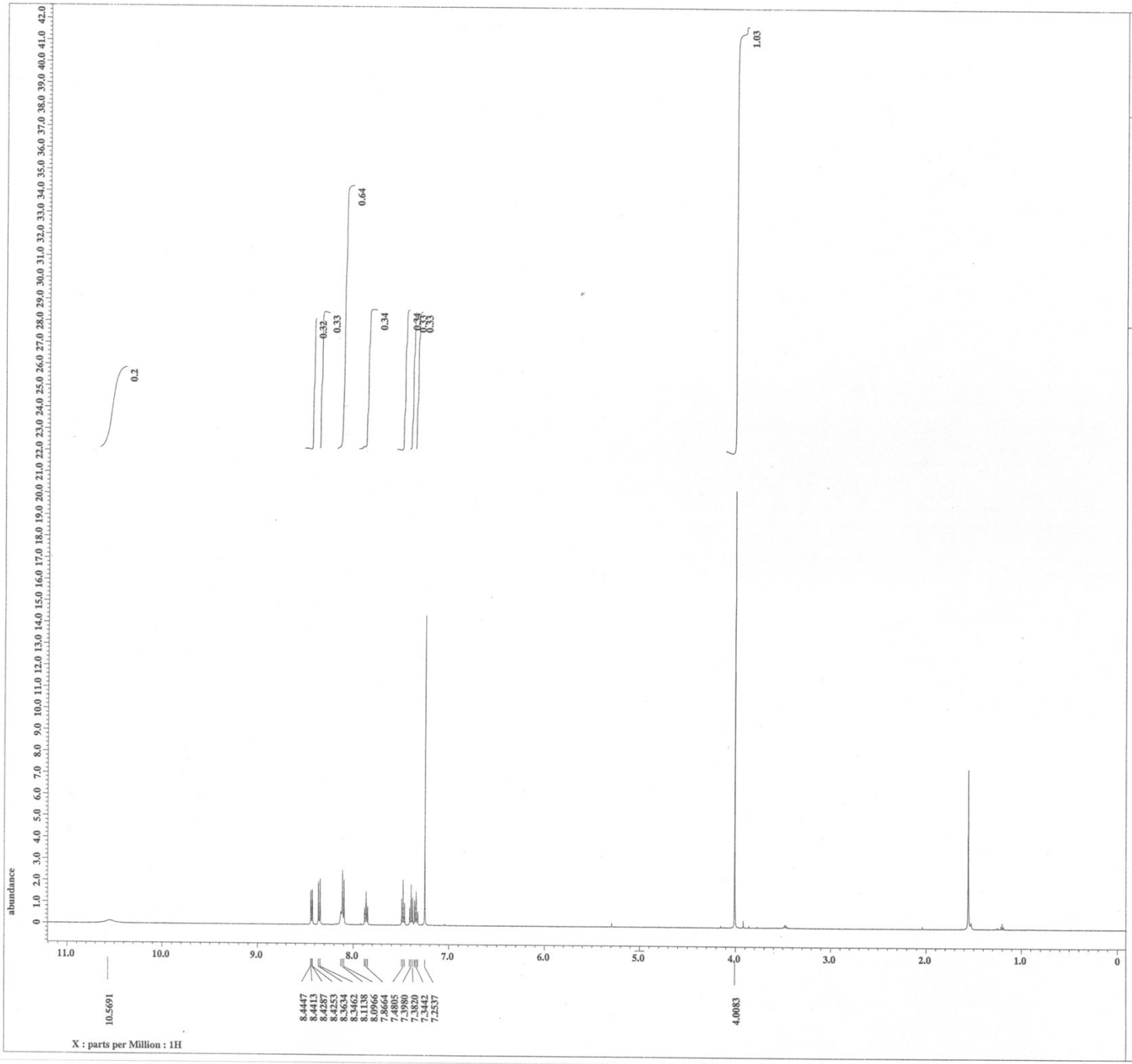
3p

315 mg, 68%. White solids. Mp: 215-217 °C (EtOH). IR ($CHCl_3$): 1697, 1676 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.85 (s, 3H), 5.91 (s, 2H), 7.09 (d, *J* = 6.9 Hz, 2H), 7.14-7.21 (m, 3H), 7.29-7.34 (m, 2H), 7.88-7.91 (m, 2H), 8.00-8.02 (m, 1H), 8.22 (d, *J* = 8.6 Hz, 1H), 8.57 (d, *J* = 2.3 Hz, 1H). ^{13}C-NMR ($CDCl_3$) δ: 47.5, 52.0, 94.7, 112.8, 117.2, 117.8, 118.4, 121.4, 123.2, 123.9, 127.1, 127.5, 128.2, 128.6, 130.2, 132.9, 135.7, 136.9, 137.7, 138.3, 158.9, 164.9 (two sp^2 signals were not observed because of overlapping). HR-ESI-MS *m/z*: Calcd for $C_{24}H_{17}BrN_2NaO_3$ [$(M+Na)^+$]: 483.0320, 485.0300. Found 483.0297, 485.0305.

6. References

(1) Linton, E. C.; Kozlowski, M. C. *J. Am. Chem. Soc.* **2008**, *130*, 16162.

(2) Abe, T.; Yamada, K. *Org. Lett*. **2016**, *18*, 6504.



JEOL

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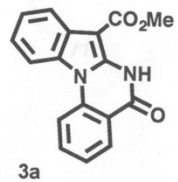
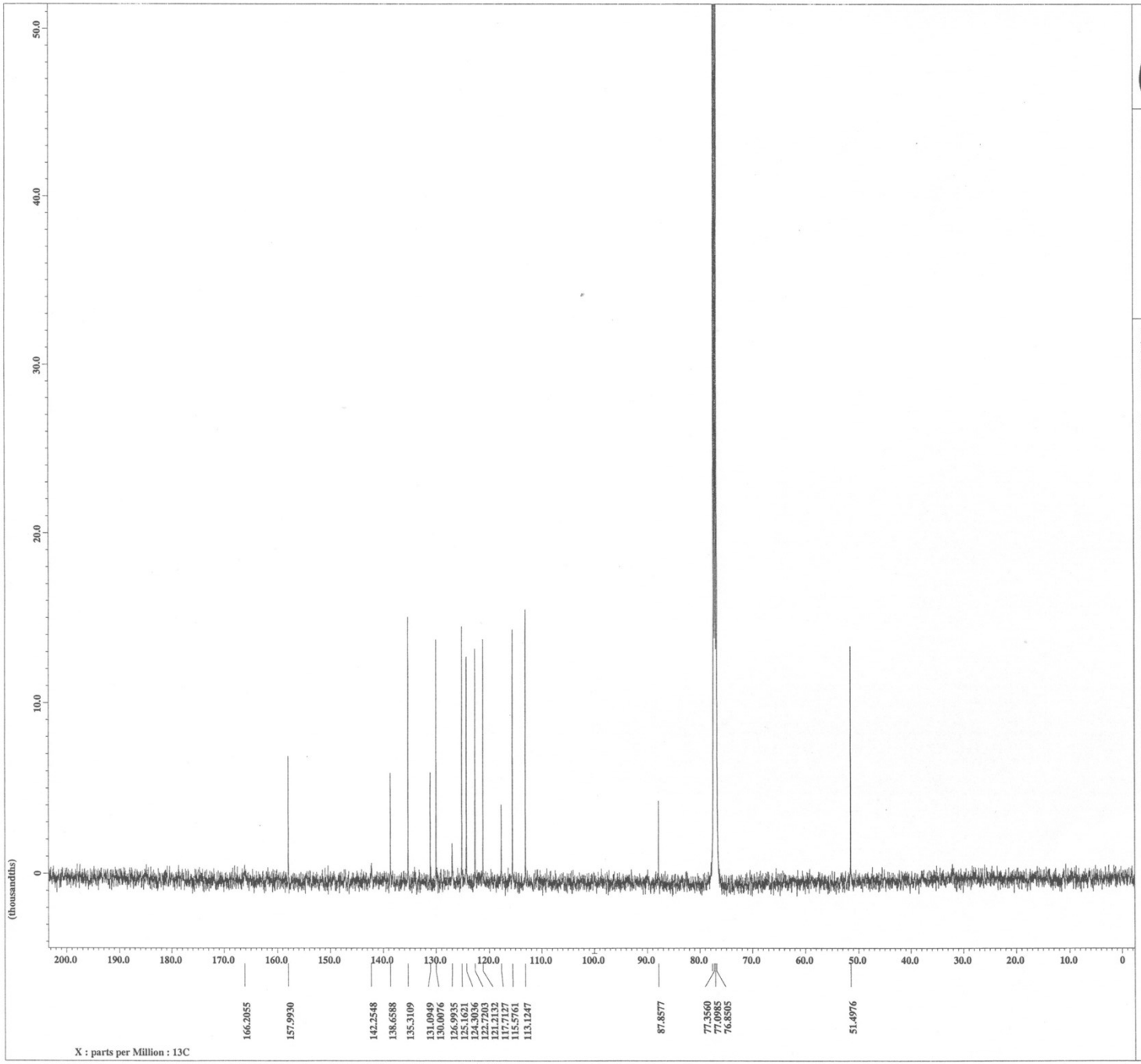
Repetition_time	= 6.74587904[s]
Temp_get	= 22.4[dC]



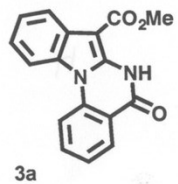
3a



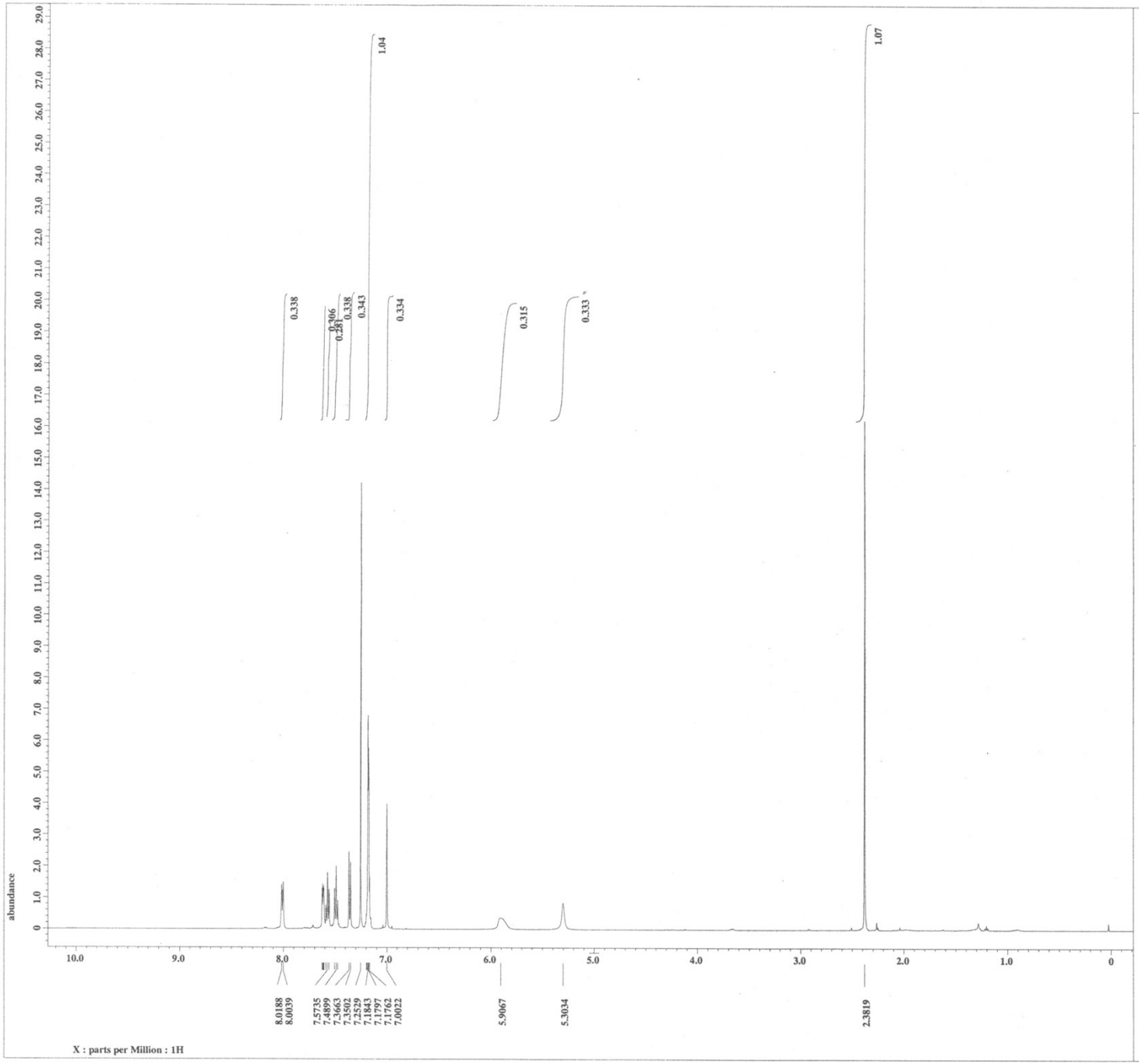
JEOL

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

Derived from: TA160516-2.jdf
```

Filename	= TA160516-5.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#570649
Solvent	= CHLOROFORM-D
Creation_time	= 12-MAY-2016 02:28:10
Revision_time	= 17-MAY-2016 08:47:09
Current_time	= 17-MAY-2016 08:48:48
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 0.83361792[s]
X_domain	= 13C
X_freq	= 125.76529768[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.19959034[Hz]
X_sweep	= 39.3081761[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 21425
Total_scans	= 21425
X_90_width	= 11.3[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 5.5[dB]
X_pulse	= 3.76666667[us]
Irr_atn_dec	= 21.318[dB]
Irr_atn_noe	= 21.318[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	= 2.83361792[s]
Temp_get	= 22.9[dC]

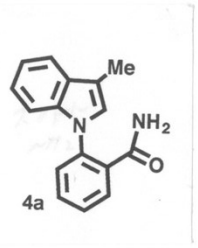


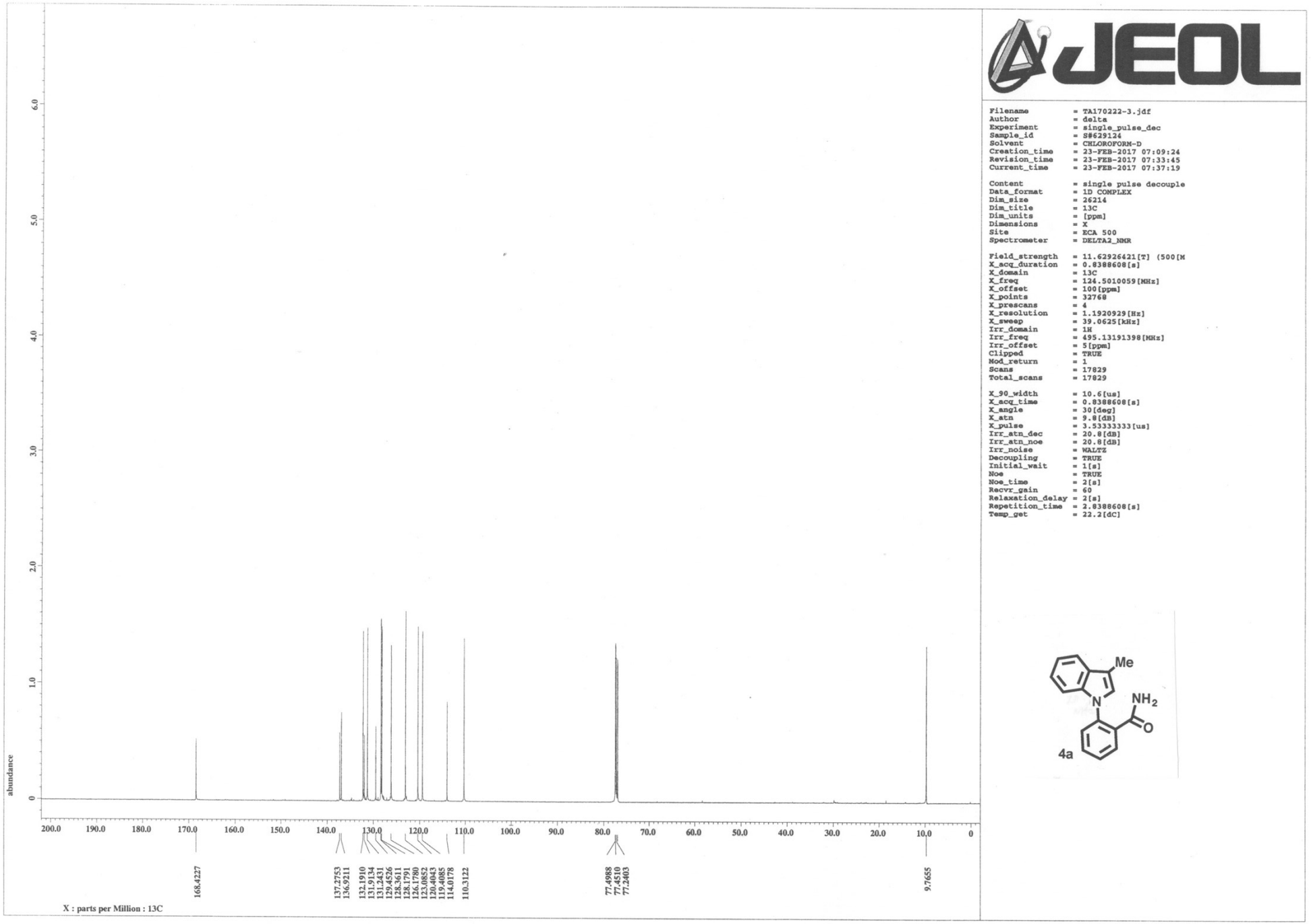
3a



JEOL

Filename	= TA170221-15.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#640168
Solvent	= CHLOROFORM-D
Creation_time	= 21-FEB-2017 17:25:17
Revision_time	= 21-FEB-2017 17:54:39
Current_time	= 21-FEB-2017 17:54:42
Content	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.62926421[T] (500[M
X_acq_duration	= 1.76422912[s]
X_domain	= 1H
X_freq	= 495.13191398[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.5668198[Hz]
X_sweep	= 9.28677563[kHz]
Irr_domain	= 1H
Irr_freq	= 495.13191398[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 495.13191398[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 12.7[us]
X_acq_time	= 1.76422912[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.35[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 32
Relaxation_delay	= 5[s]
Repetition_time	= 6.76422912[s]
Temp_get	= 20.2[dC]



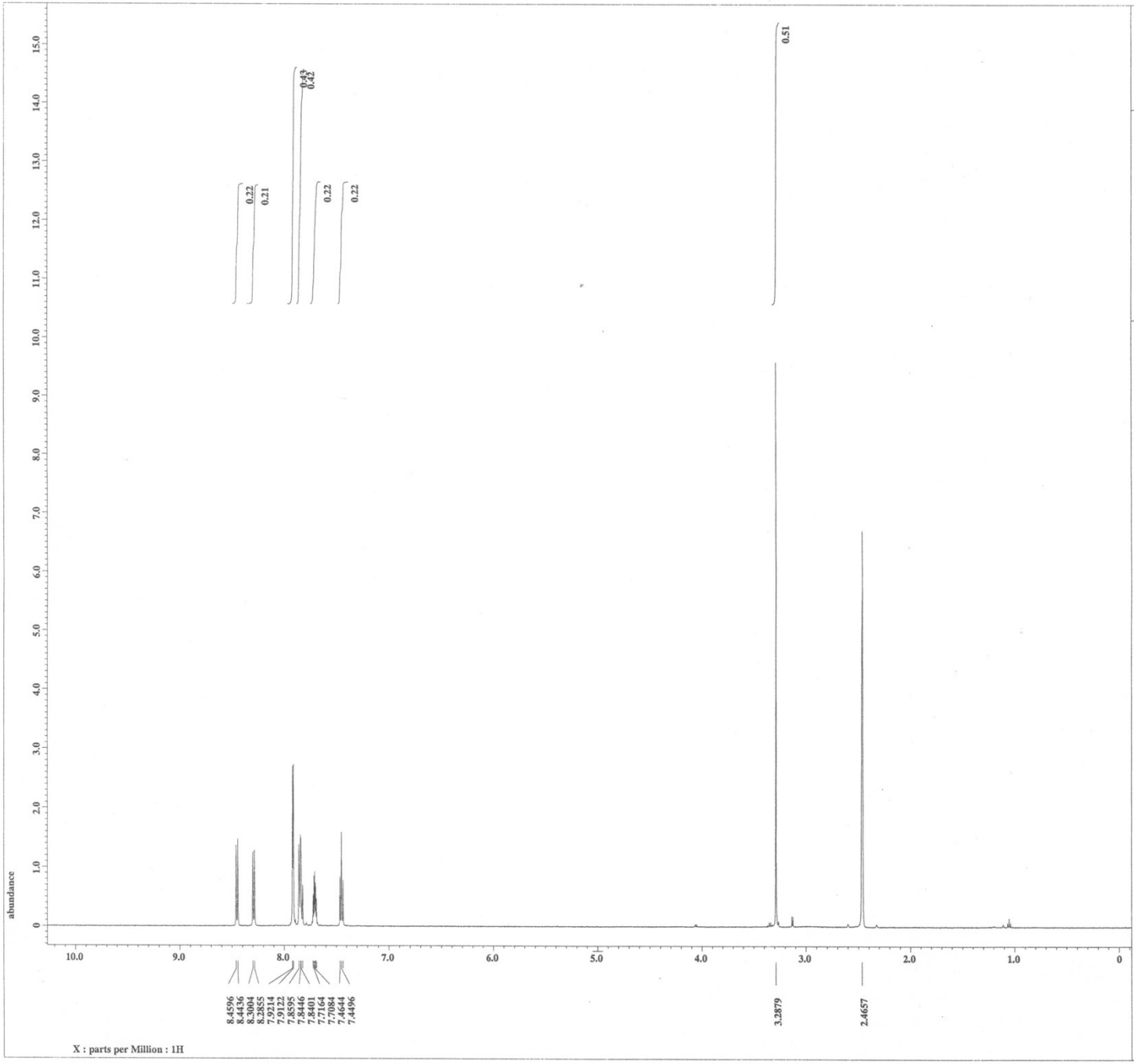


Filename = TA170222-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#629124
Solvent = CHLOROFORM-D
Creation_time = 23-FEB-2017 07:09:24
Revision_time = 23-FEB-2017 07:33:45
Current_time = 23-FEB-2017 07:37:19

Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 17829
Total_scans = 17829

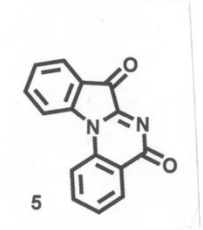
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.8[dB]
X_pulse = 3.53333333[us]
Irr_atn_dec = 20.8[dB]
Irr_atn_noe = 20.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 22.2[dC]

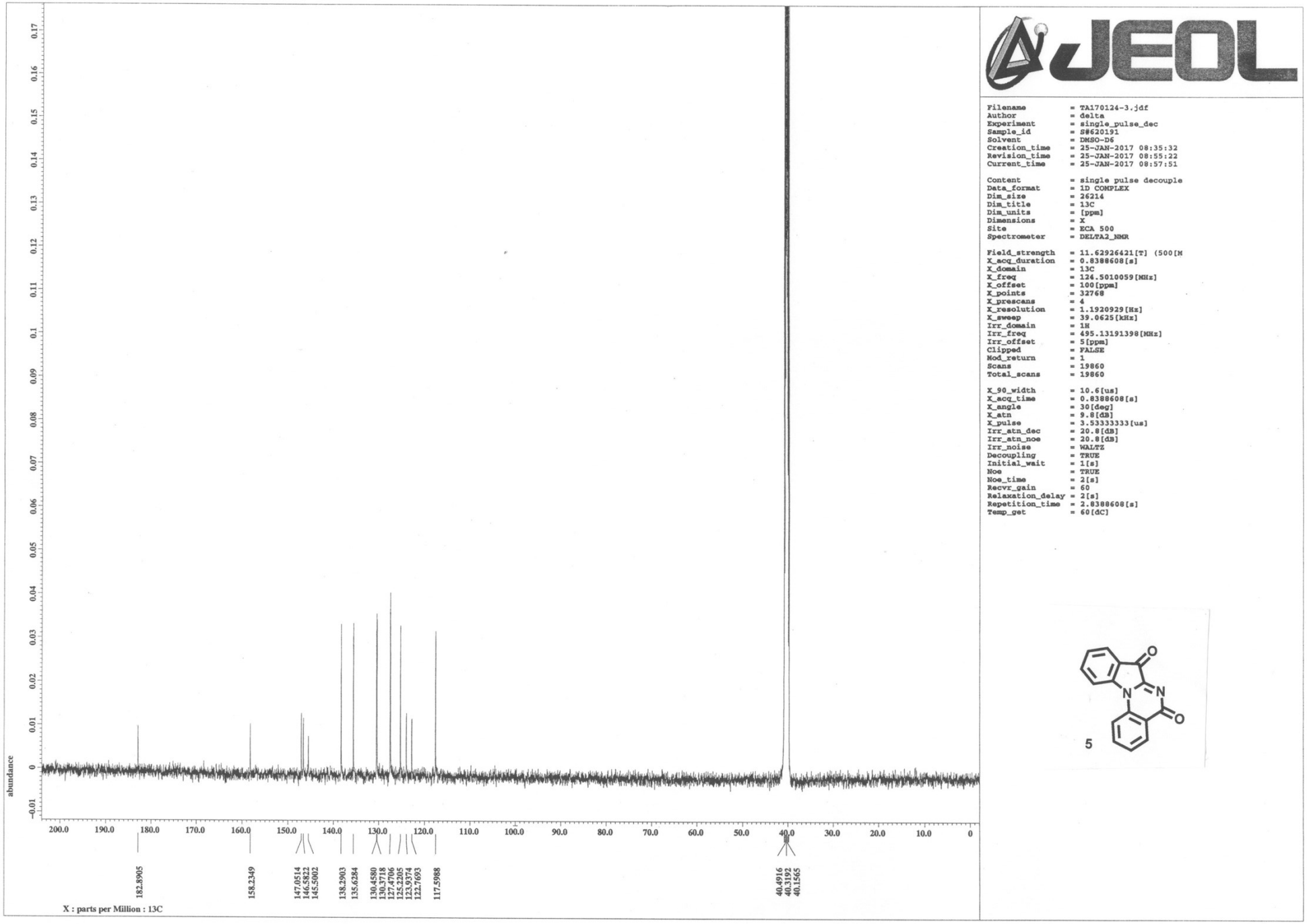


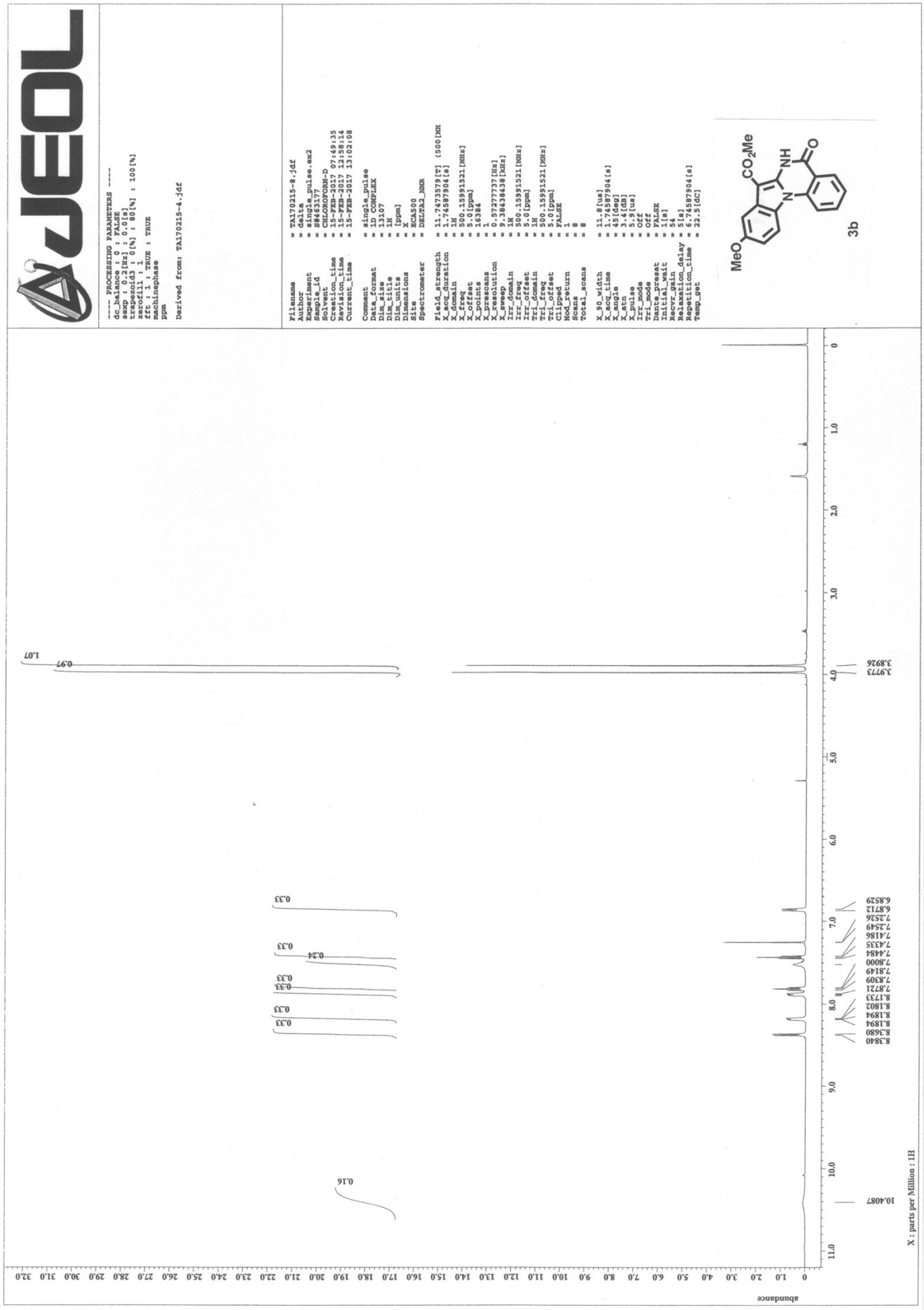
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

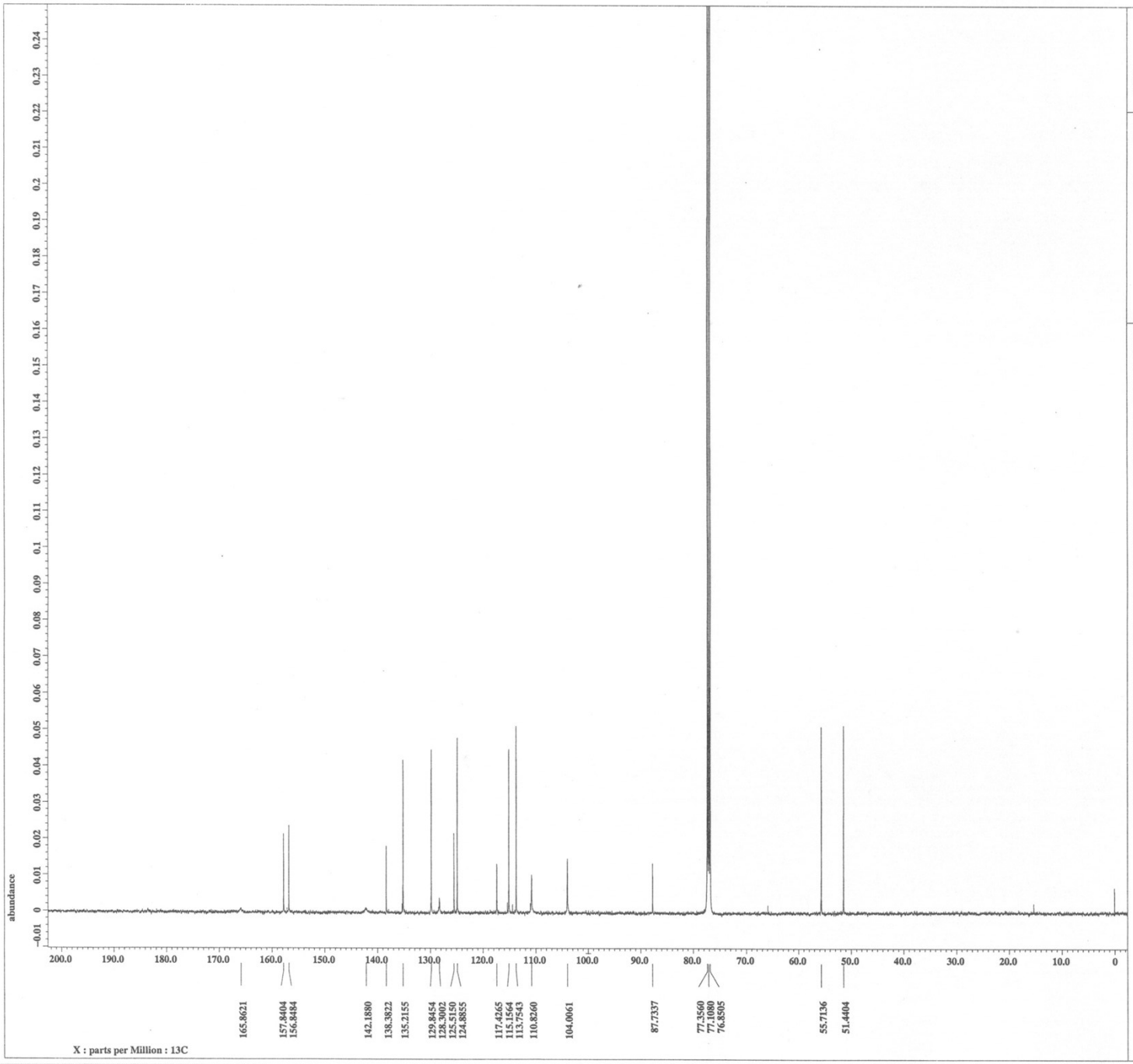
Derived from: TA170123-4.jdf

Filename	= TA170123-7.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#475309
Solvent	= DMSO-D6
Creation_time	= 23-JAN-2017 08:10:35
Revision_time	= 23-JAN-2017 13:17:20
Current_time	= 23-JAN-2017 13:20:18
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 11.8[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.9[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 60
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 22.9[dC]




abundance
X : parts per Million : 13C
182.8905
158.2349
147.0514
146.5822
145.5002
138.2903
135.6284
130.4580
130.3718
127.4706
125.2205
123.9374
122.7693
117.5988
40.4916
40.3192
40.1565
Filename = TA170124-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#620191
Solvent = DMSO-D6
Creation_time = 25-JAN-2017 08:35:32
Revision_time = 25-JAN-2017 08:55:22
Current_time = 25-JAN-2017 08:57:51
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 19860
Total_scans = 19860
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.8[dB]
X_pulse = 3.53333333[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 = 2.8388608[s]
Temp_get = 60[dC]
5

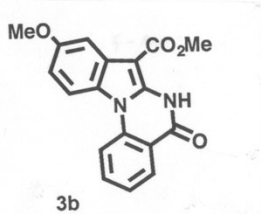

JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zf1 : 1
zf2 : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TA170215-4.jdf
Filename = TA170215-8.jdf
Author = data
Experiment = single_pulse.ex2
Sample_id = S#463177
Solvent = CHLOROFORM-D
Creation_time = 15-FEB-2017 07:49:35
Revision_time = 15-FEB-2017 12:58:14
Current_time = 15-FEB-2017 13:02:08
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = JEOL500
Spectrometer = DELTA2_NMR
Field_strength = 11.74735579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 11.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 5.9[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 54
Relaxation_delay = 5[s]
Repetition_time = 7.46587904[s]
Temp_get = 22.5[dC]
CO2Me
NH
O
N
MeO
3b
1.07
0.97
0.33
0.24
0.33
0.33
0.33
0.33
0.16
3.8926
3.9773
6.8529
6.8712
7.2526
7.2549
7.4186
7.4335
7.4484
7.8000
7.8149
7.8309
7.8721
8.1733
8.1802
8.1894
8.1894
8.3680
8.3840
10.4087
abundance
X : parts per Million : 1H



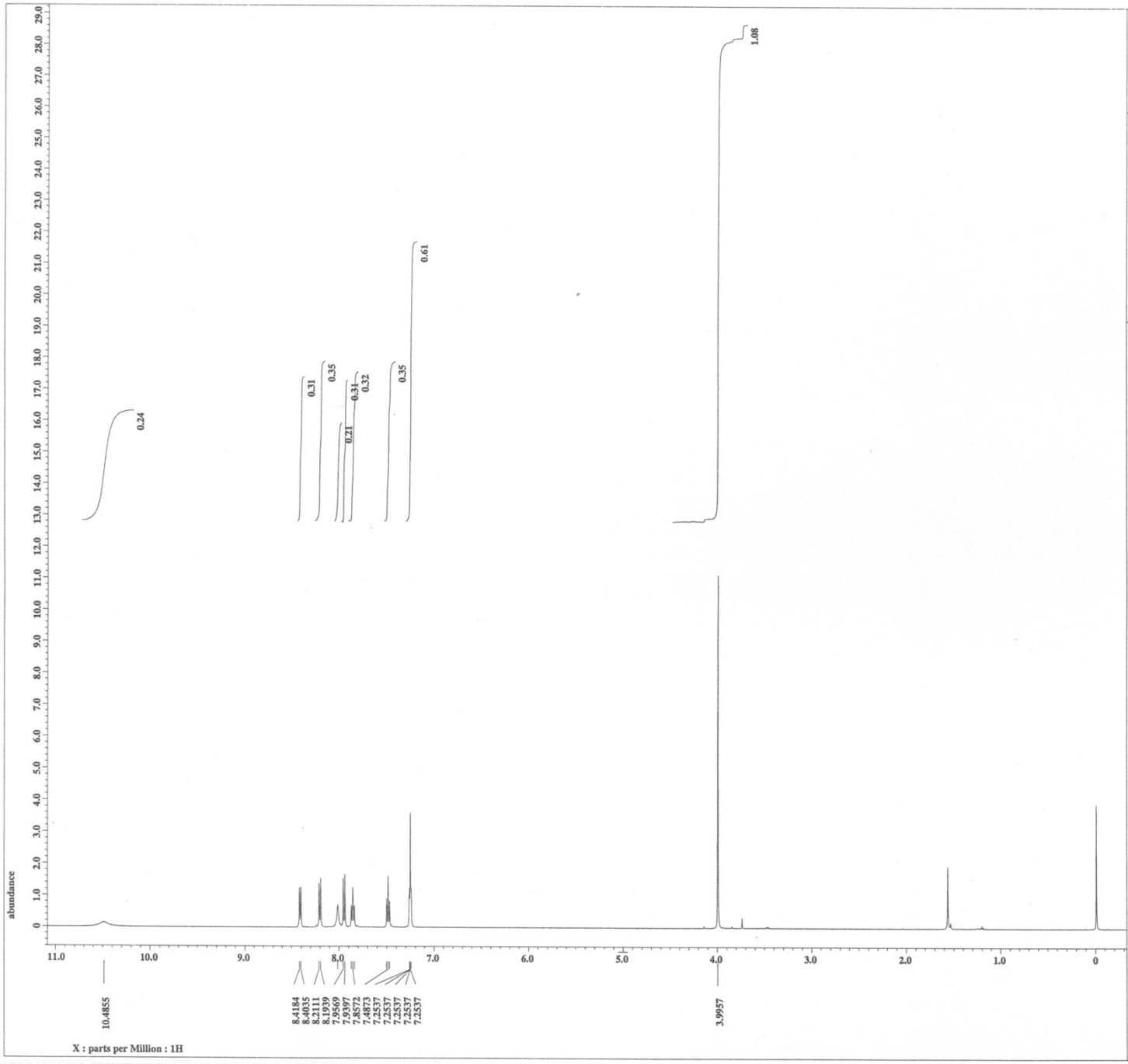
JEOL

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

Derived from: TA170215-13.jdf
```

```
Filename          = TA170215-15.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#616675
Solvent           = CHLOROFORM-D
Creation_time     = 16-FEB-2017 02:39:46
Revision_time     = 16-FEB-2017 07:51:14
Current_time      = 16-FEB-2017 07:53:42

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 18534
Total_scans       = 18534

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 52
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 23.3[dC]
```



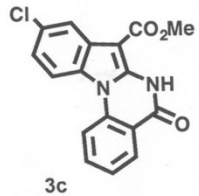
3b



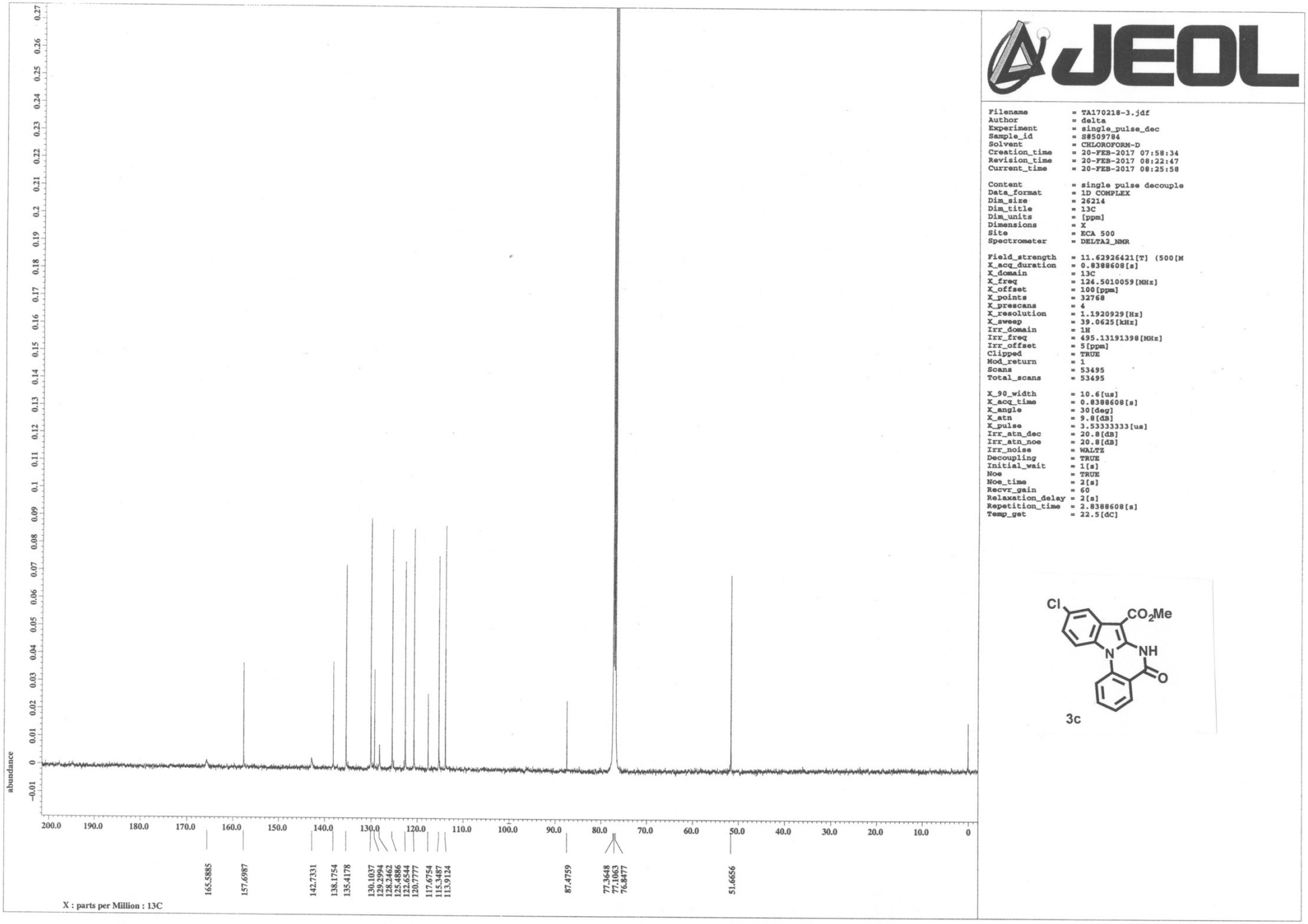
JEOL

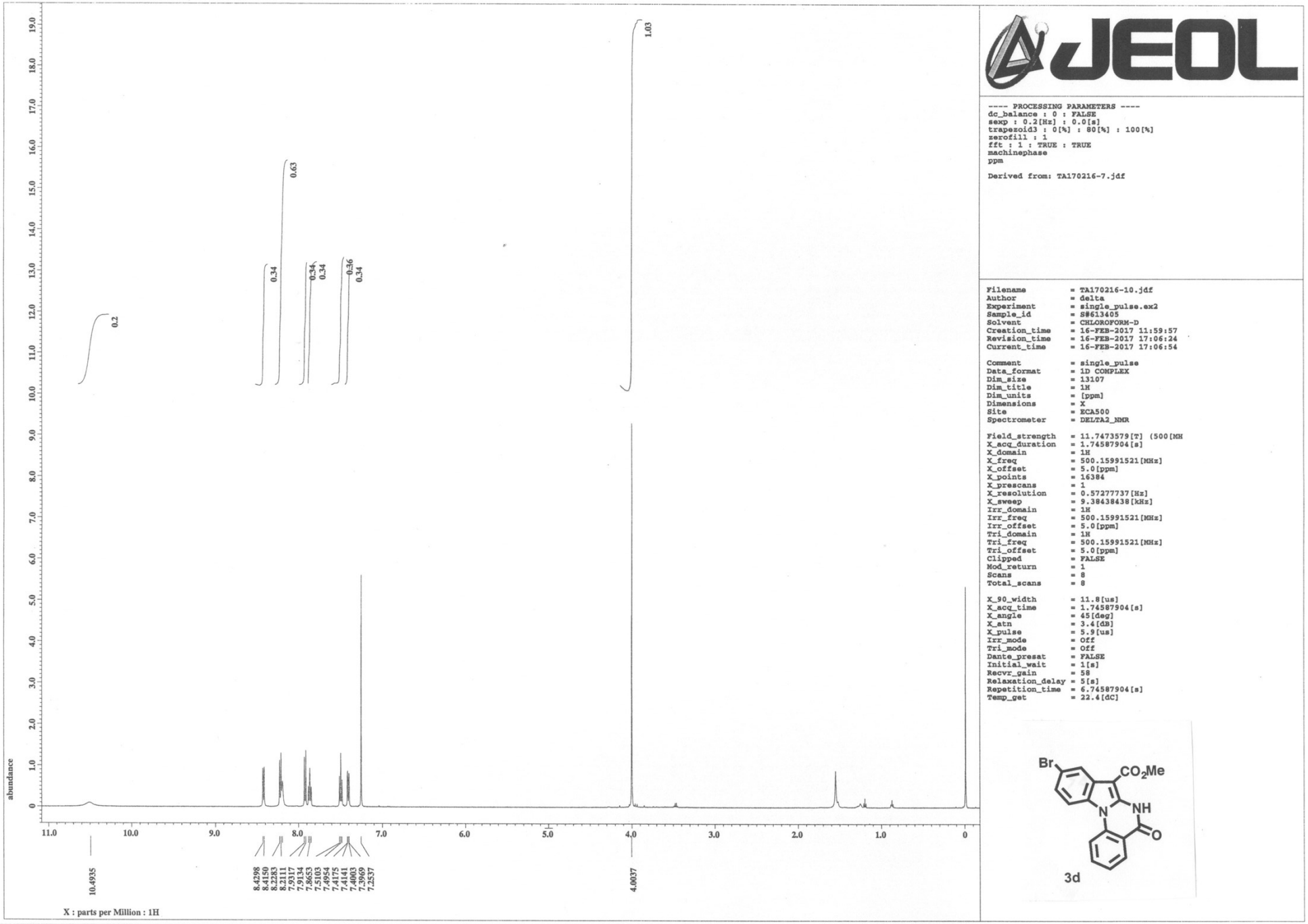
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA170217-4.jdf
```

```
Filename          = TA170217-8.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#352289
Solvent           = CHLOROFORM-D
Creation_time     = 17-FEB-2017 04:44:44
Revision_time     = 17-FEB-2017 09:52:08
Current_time      = 17-FEB-2017 09:53:58

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22[dC]
```



3c


abundance
X : parts per Million : 13C
165.5885
157.6987
142.7331
138.1754
135.4178
130.1037
129.2994
128.2462
125.4886
122.6544
120.7777
117.6754
115.3487
113.9124
87.4759
77.3648
77.1063
76.8477
51.6656
Filename = TA170218-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#509784
Solvent = CHLOROFORM-D
Creation_time = 20-FEB-2017 07:58:34
Revision_time = 20-FEB-2017 08:22:47
Current_time = 20-FEB-2017 08:25:58
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 53495
Total_scans = 53495
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.8[dB]
X_pulse = 3.53333333[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 = 2.8388608[s]
Temp_get = 22.5[dC]
Cl
CO2Me
NH
N
O
3c

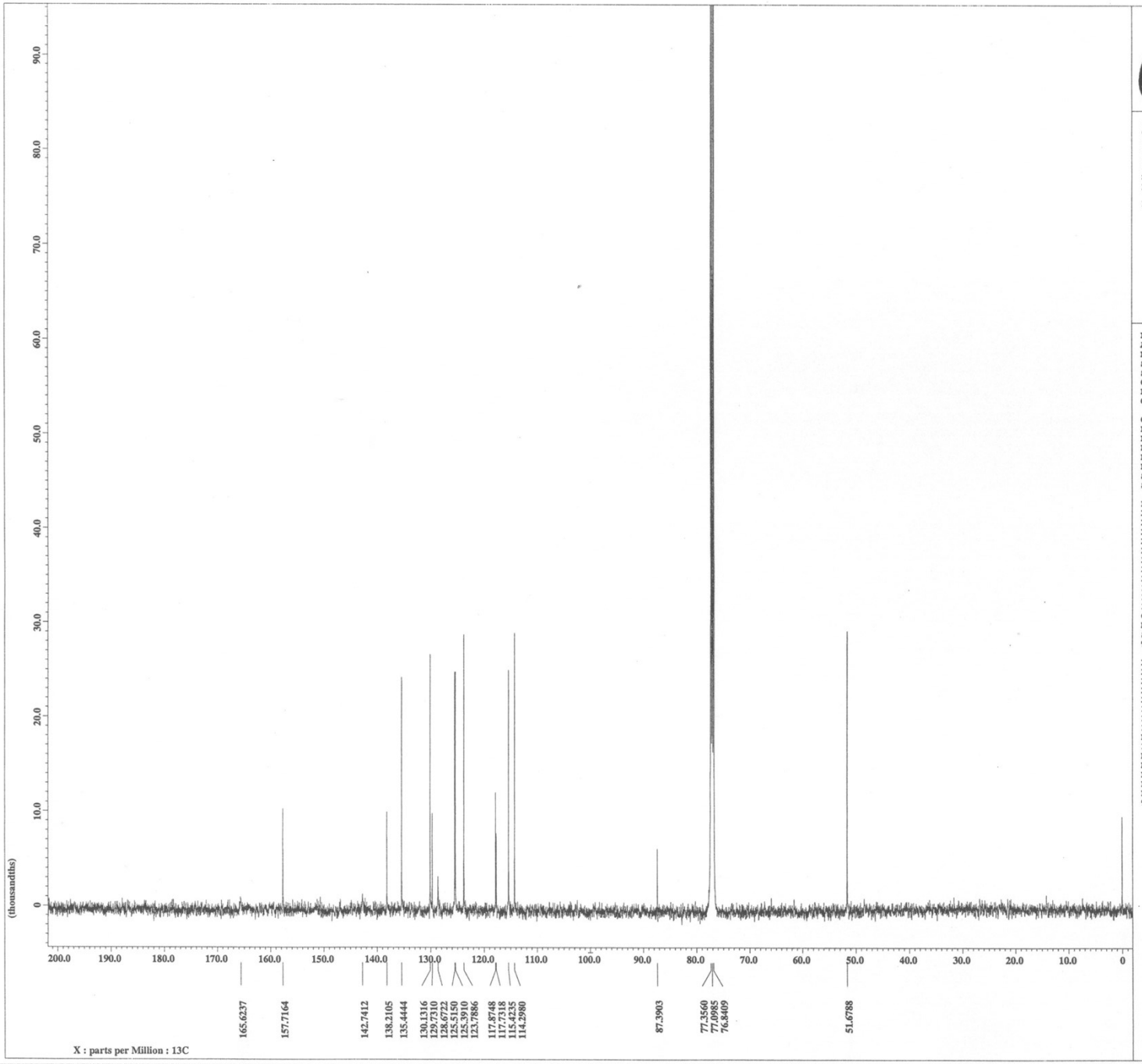


JEOL

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

Derived from: TA170216-7.jdf
```

Filename	= TA170216-10.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#613405
Solvent	= CHLOROFORM-D
Creation_time	= 16-FEB-2017 11:59:57
Revision_time	= 16-FEB-2017 17:06:24
Current_time	= 16-FEB-2017 17:06:54
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 11.8[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.9[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 58
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 22.4[dC]

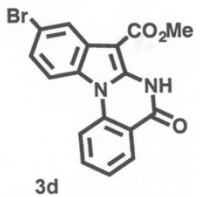
3d



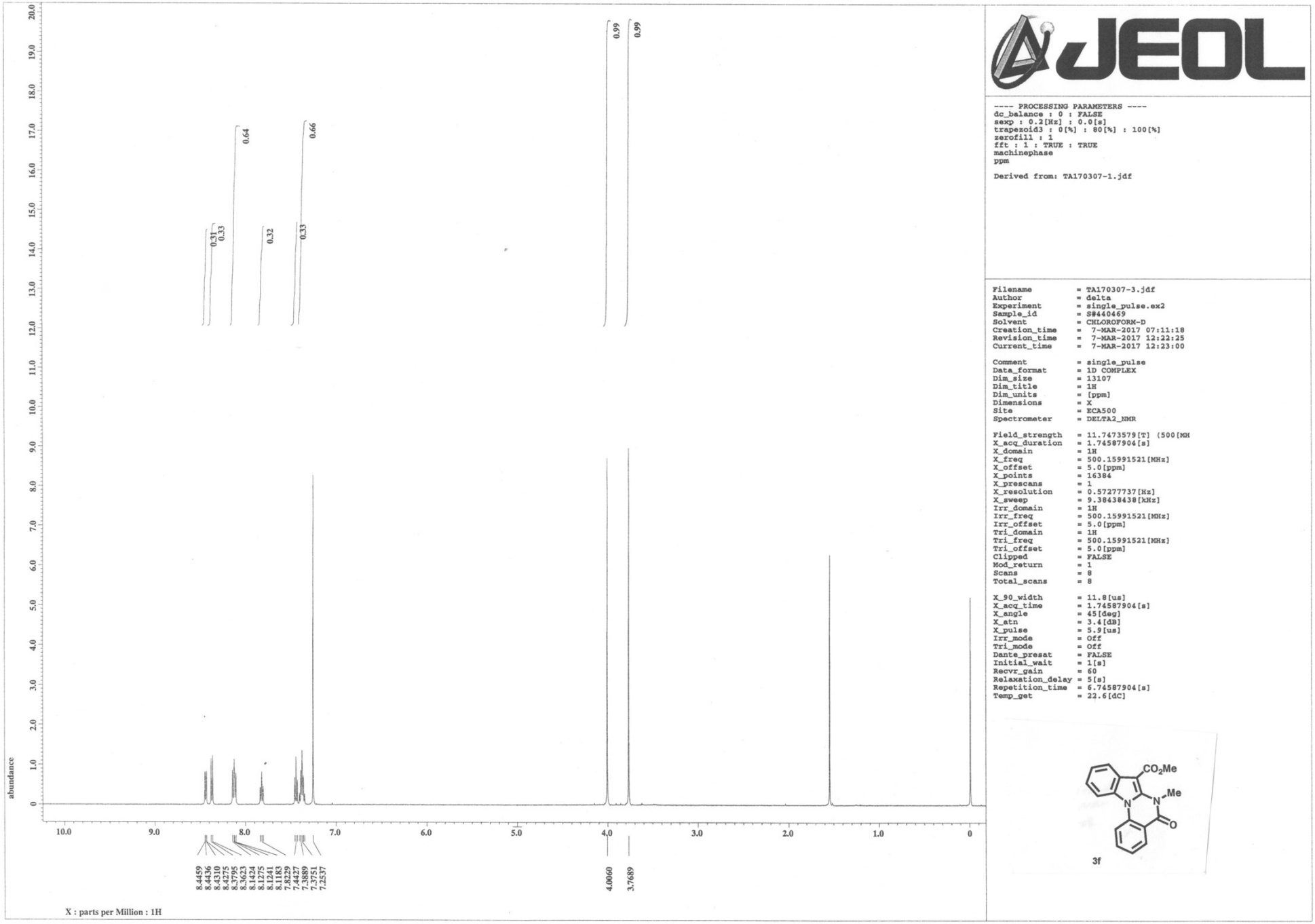
JEOL

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

Derived from: TA170216-8.jdf
```

```
Filename          = TA170216-12.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#614611
Solvent           = CHLOROFORM-D
Creation_time     = 17-FEB-2017 01:59:50
Revision_time     = 17-FEB-2017 07:41:48
Current_time      = 17-FEB-2017 07:43:55

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 17762
Total_scans       = 17762

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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   = 2.83361792[s]
Temp_get          = 23.2[dC]
```



3d

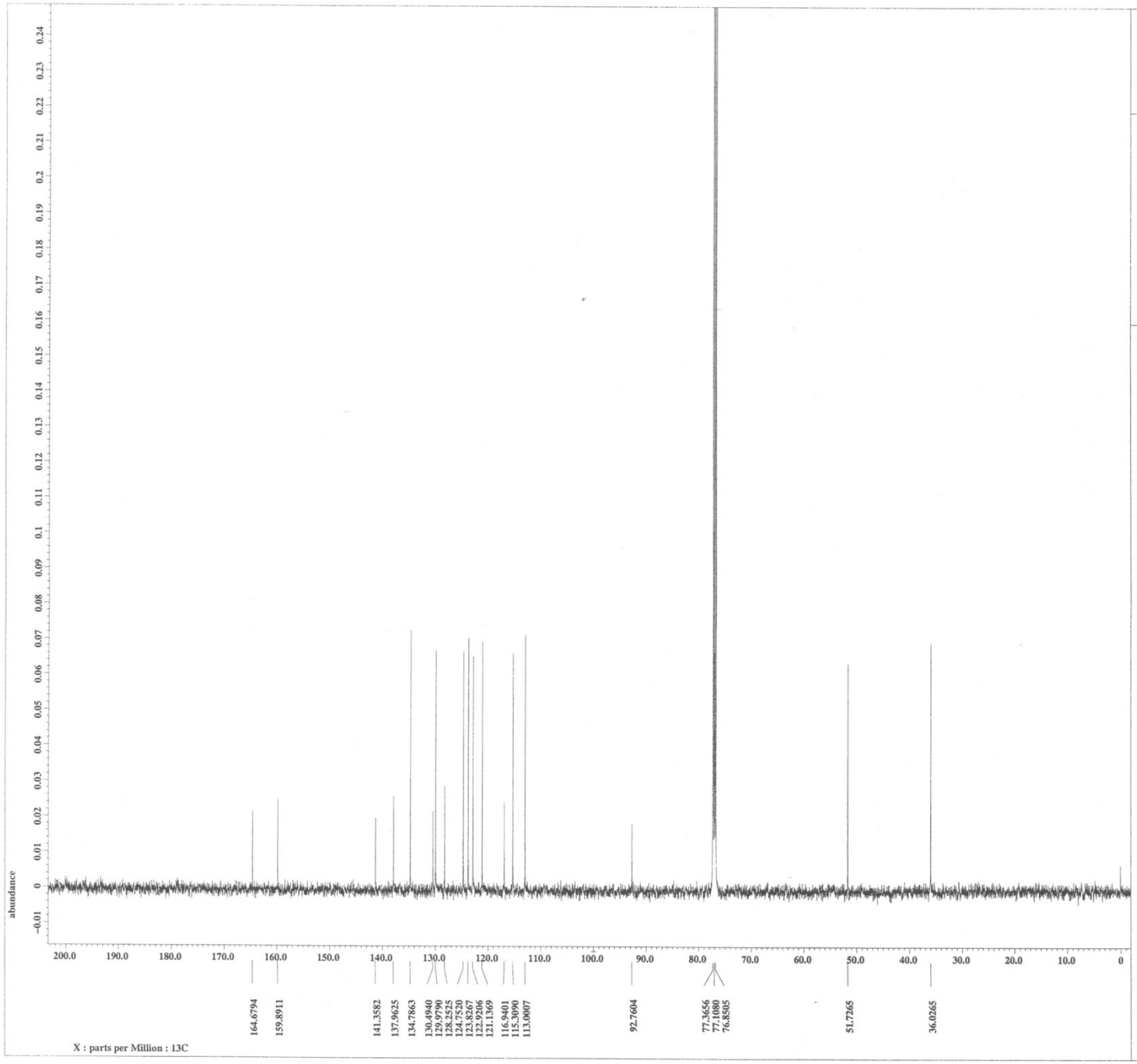


JEOL

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

Derived from: TA170307-1.jdf
```

```
Filename          = TA170307-3.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#440469
Solvent           = CHLOROFORM-D
Creation_time     =  7-MAR-2017 07:11:18
Revision_time     =  7-MAR-2017 12:22:25
Current_time      =  7-MAR-2017 12:23:00

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.6[dC]
```

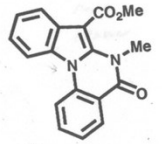

3f



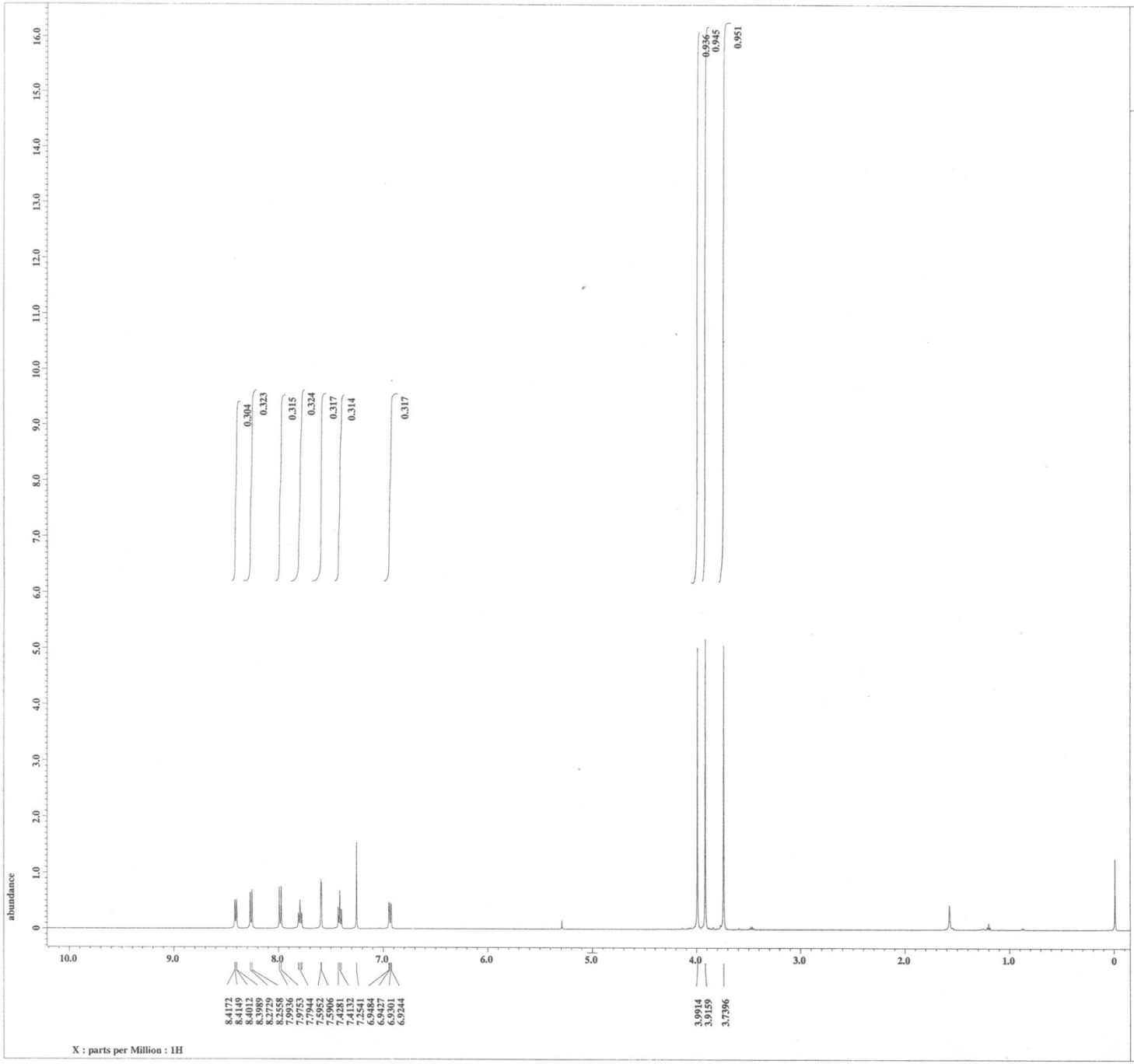
JEOL

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

Derived from: TA170307-4.jdf
```

```
Filename          = TA170307-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#546316
Solvent           = CHLOROFORM-D
Creation_time     =  7-MAR-2017 11:36:25
Revision_time     =  7-MAR-2017 16:41:08
Current_time      =  7-MAR-2017 16:43:09

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1902
Total_scans       = 1902

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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   = 2.83361792[s]
Temp_get          = 23.1[dC]
```

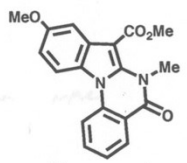


3f

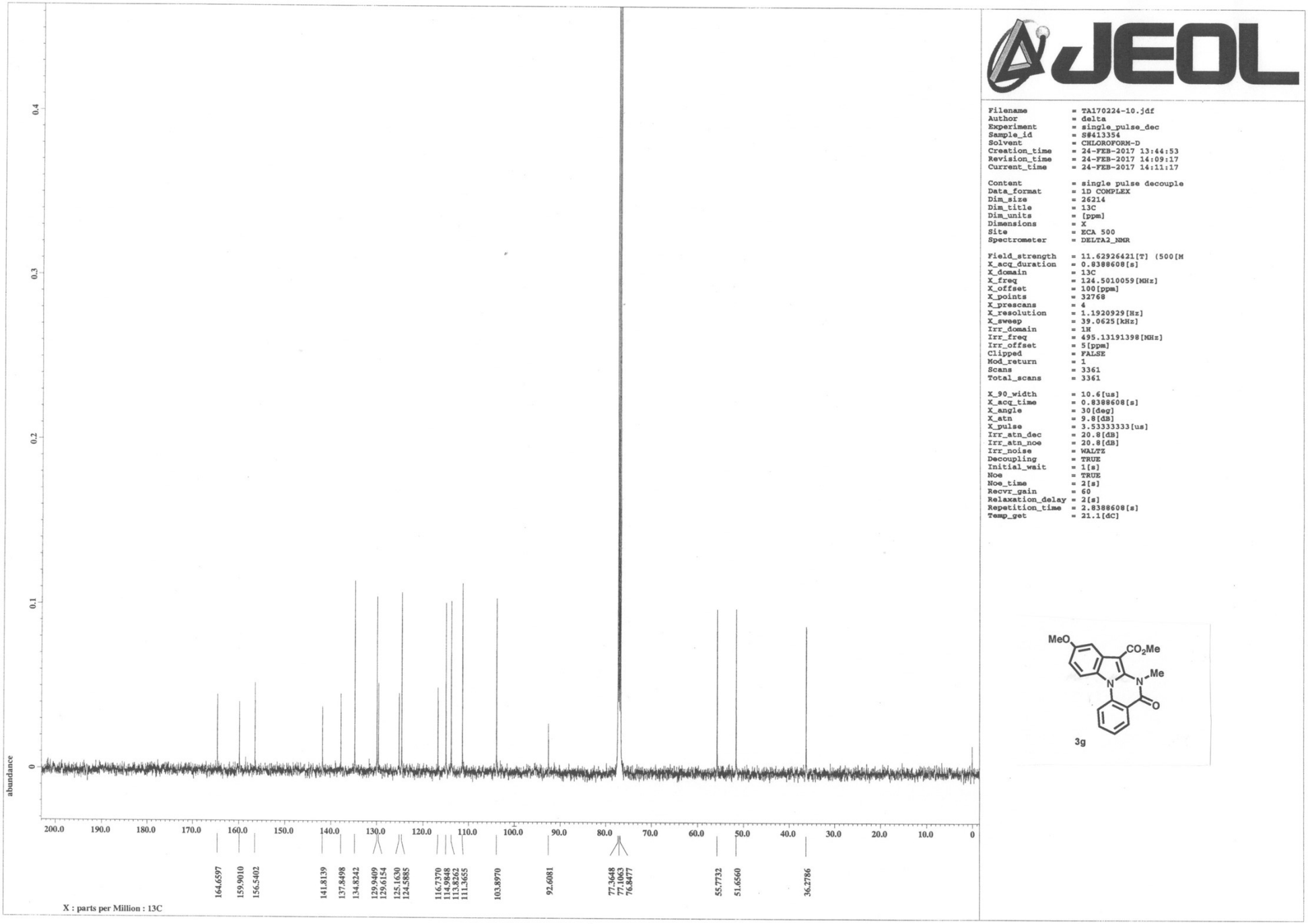


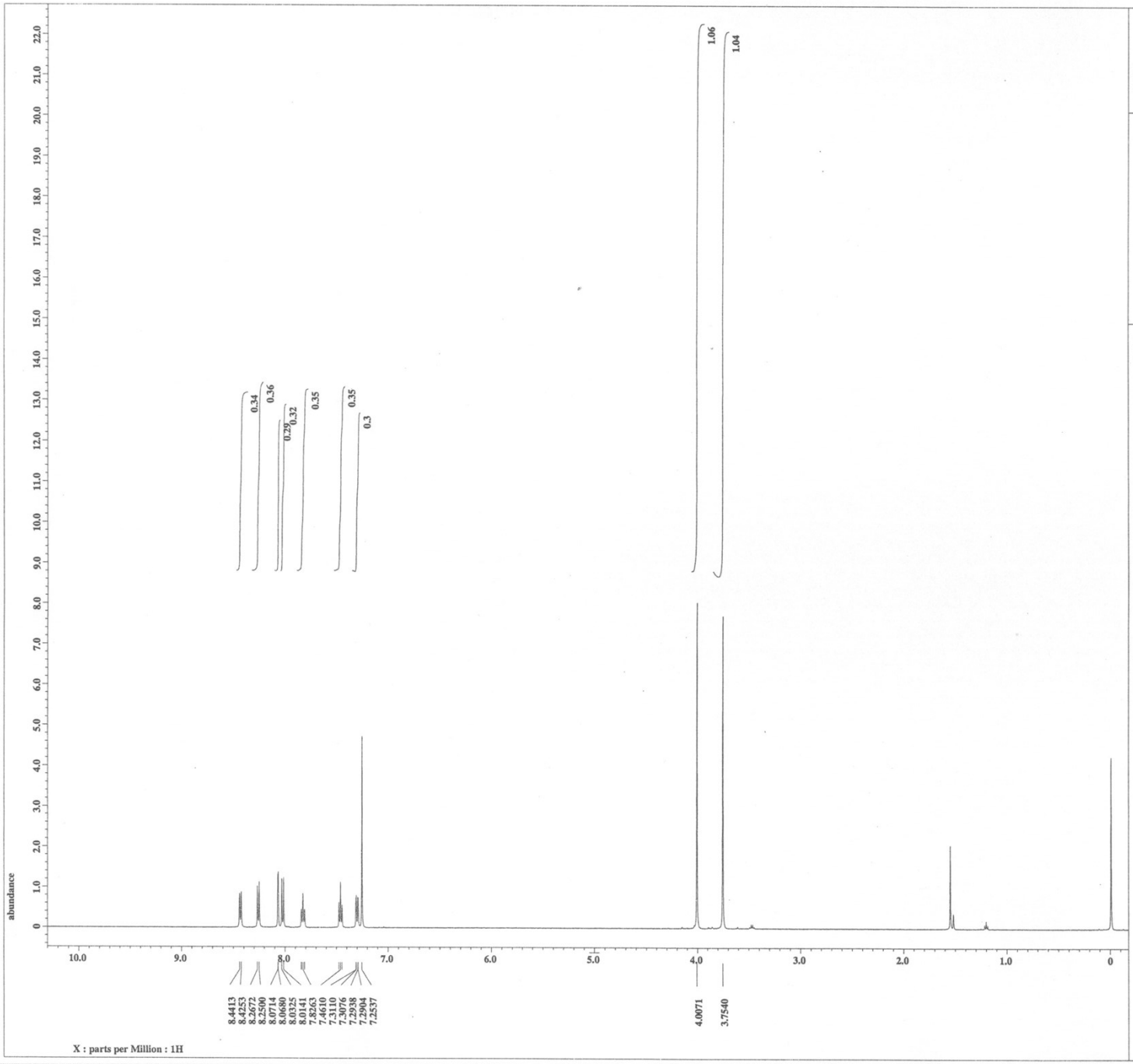
JEOL

Filename	= TA170224-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#368411
Solvent	= CHLOROFORM-D
Creation_time	= 24-FEB-2017 09:52:05
Revision_time	= 24-FEB-2017 10:21:30
Current_time	= 24-FEB-2017 10:24:02
Content	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.62926421[T] (500[M
X_acq_duration	= 1.76422912[s]
X_domain	= 1H
X_freq	= 495.13191398[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.5668198[Hz]
X_sweep	= 9.28677563[kHz]
Irr_domain	= 1H
Irr_freq	= 495.13191398[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 495.13191398[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 12.7[us]
X_acq_time	= 1.76422912[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.35[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 44
Relaxation_delay	= 5[s]
Repetition_time	= 6.76422912[s]
Temp_get	= 20.6[dC]



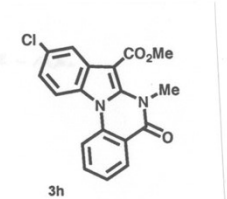
3g


JEOL
Filename = TA170224-10.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#413354
Solvent = CHLOROFORM-D
Creation_time = 24-FEB-2017 13:44:53
Revision_time = 24-FEB-2017 14:09:17
Current_time = 24-FEB-2017 14:11:17
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 3361
Total_scans = 3361
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.8[dB]
X_pulse = 3.53333333[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 = 2.8388608[s]
Temp_get = 21.1[dC]
abundance
0.4
0.3
0.2
0.1
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
164.6597
159.9010
156.5402
141.8139
137.8498
134.8242
129.9409
129.6154
125.1630
124.5885
116.7370
114.9848
113.8262
111.3655
103.8970
92.6081
77.3648
77.1063
76.8477
55.7732
51.6560
36.2786
X : parts per Million : 13C
MeO
CO2Me
N
N
Me
O
3g

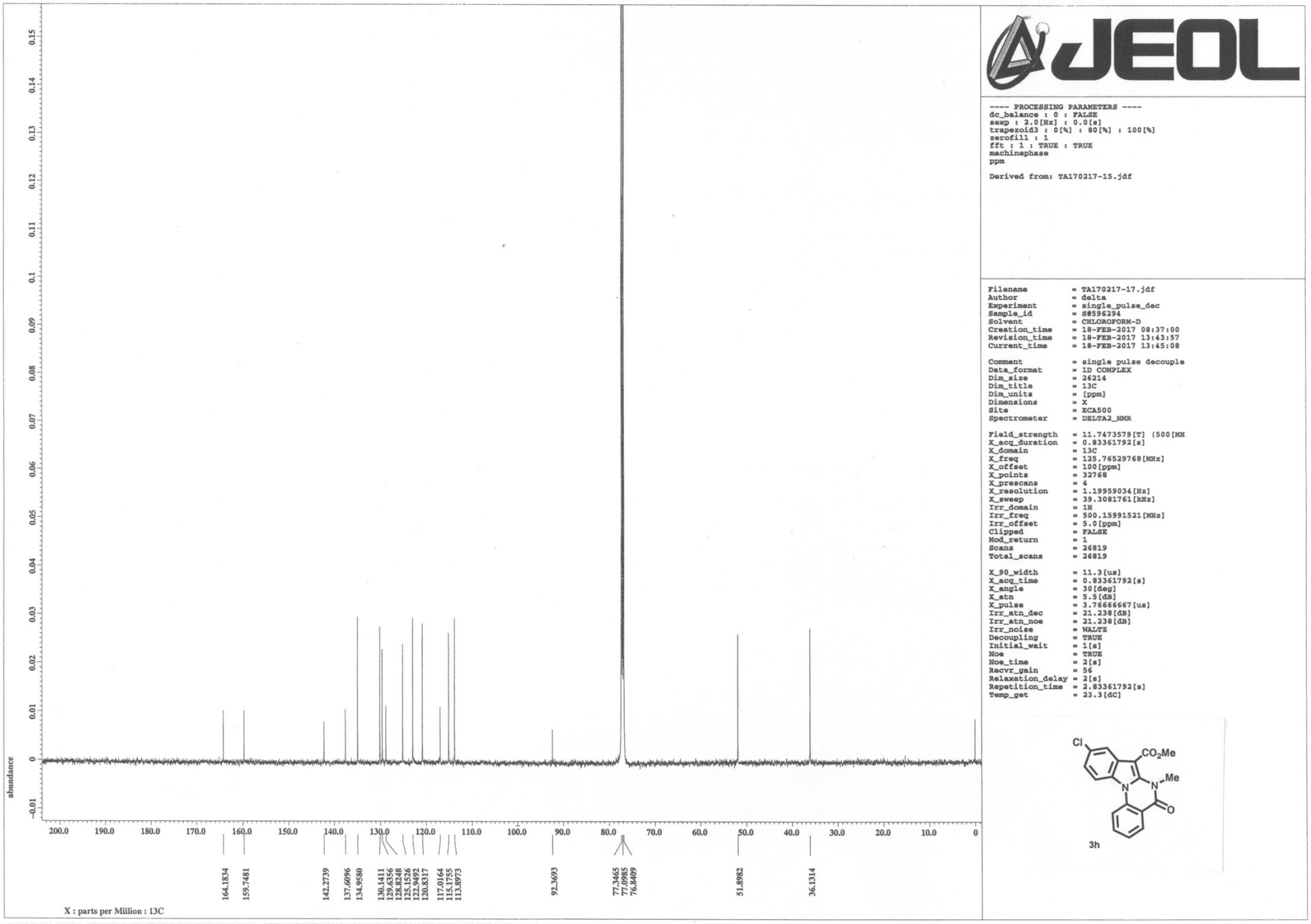


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA170217-7.jdf
```

```
Filename          = TA170217-10.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#488670
Solvent           = CHLOROFORM-D
Creation_time     = 17-FEB-2017 08:32:01
Revision_time     = 17-FEB-2017 13:38:40
Current_time      = 17-FEB-2017 13:39:21

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.4[dC]
```



3h

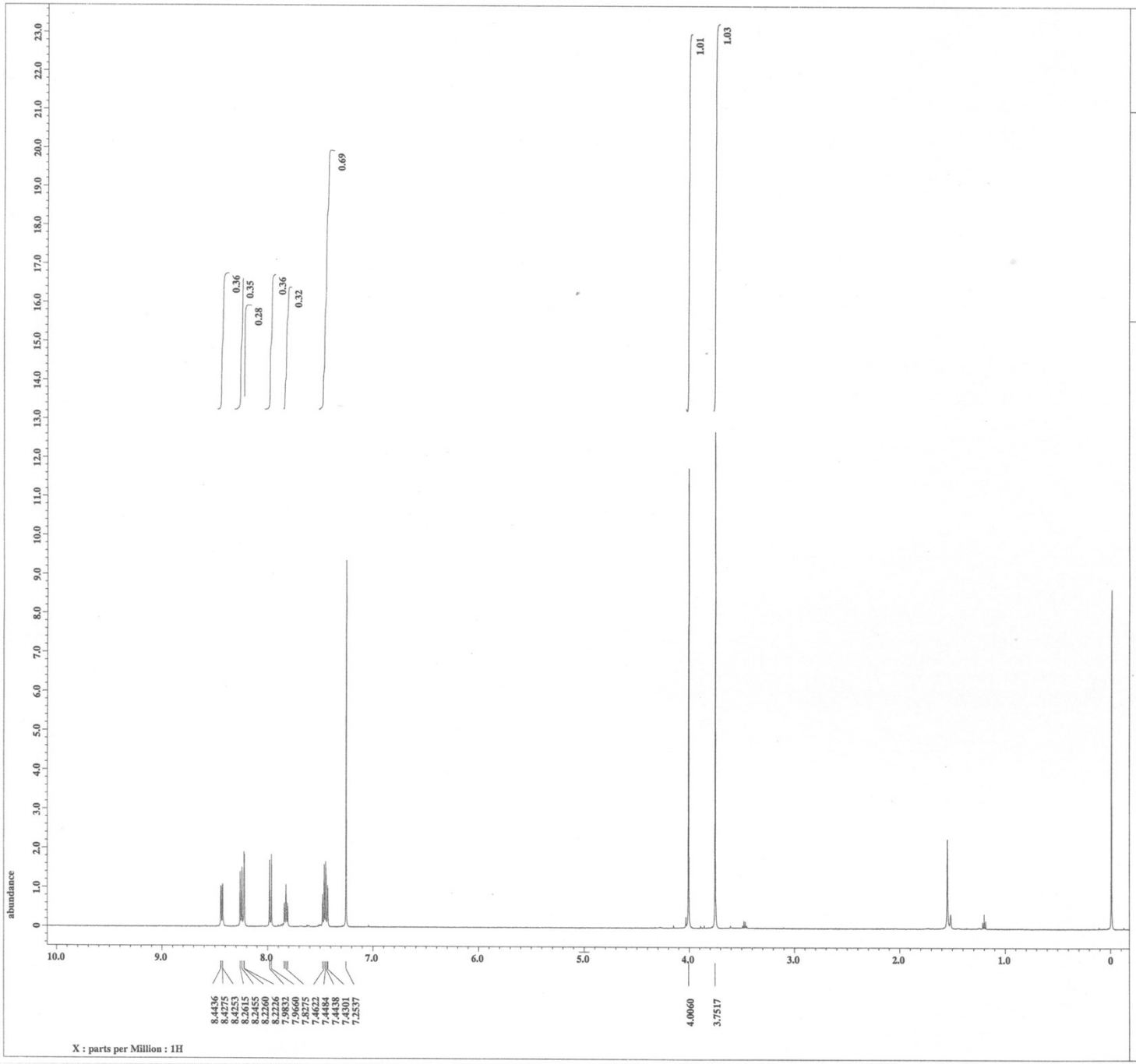


JEOL

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

Derived from: TA170217-15.jdf
```

```
Filename          = TA170217-17.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#596294
Solvent           = CHLOROFORM-D
Creation_time     = 18-FEB-2017 08:37:00
Revision_time     = 18-FEB-2017 13:43:57
Current_time      = 18-FEB-2017 13:45:08

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 26819
Total_scans       = 26819

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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   = 2.83361792[s]
Temp_get          = 23.3[dC]
```

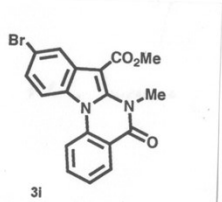

3h



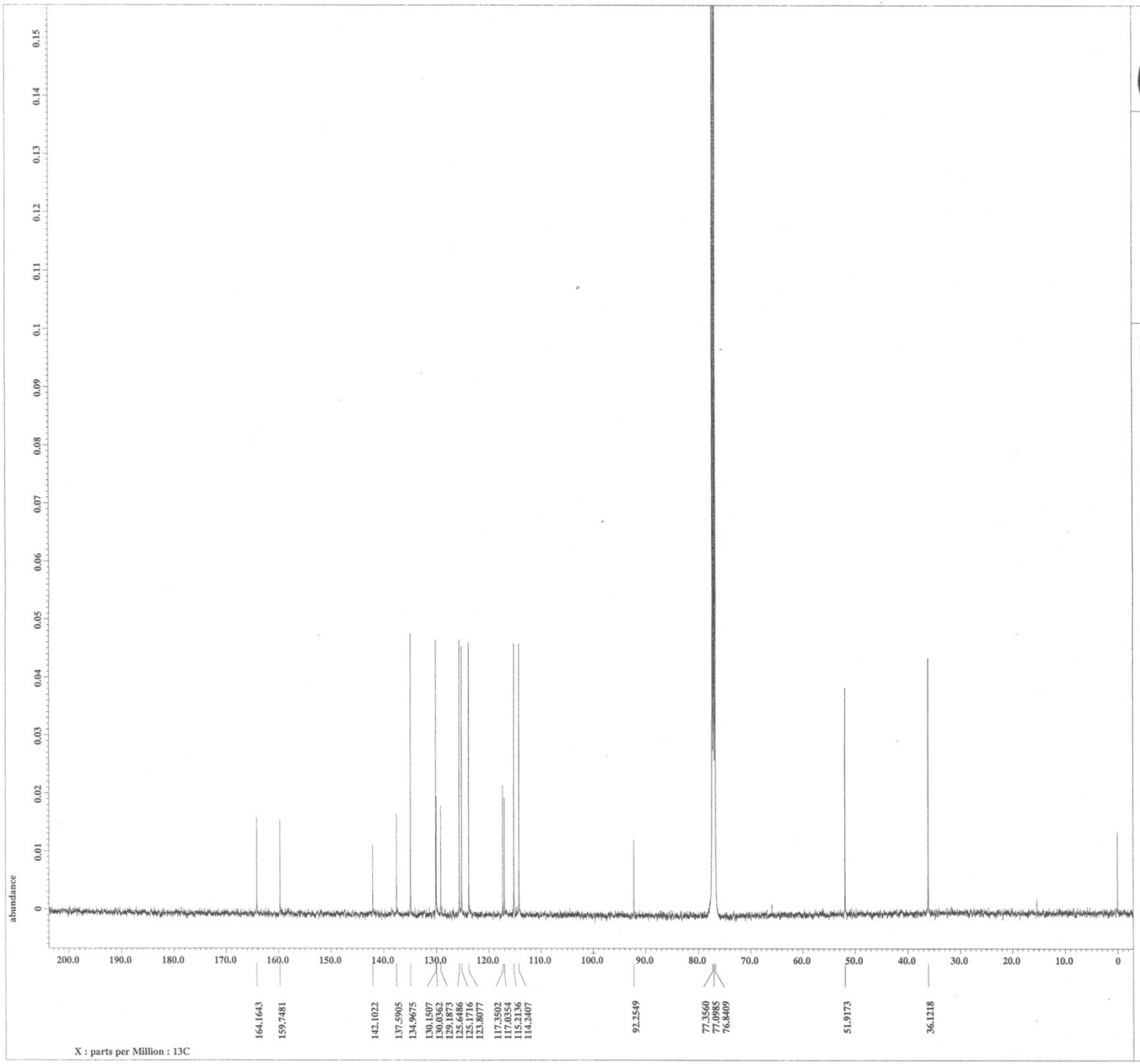
JEOL

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

Derived from: TA170217-1.jdf
```

```
Filename          = TA170217-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#303478
Solvent           = CHLOROFORM-D
Creation_time     = 17-FEB-2017 03:23:23
Revision_time     = 17-FEB-2017 08:30:25
Current_time      = 17-FEB-2017 08:30:47

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.4[dC]
```



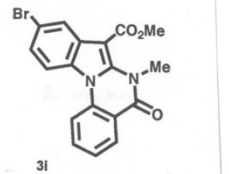
3i



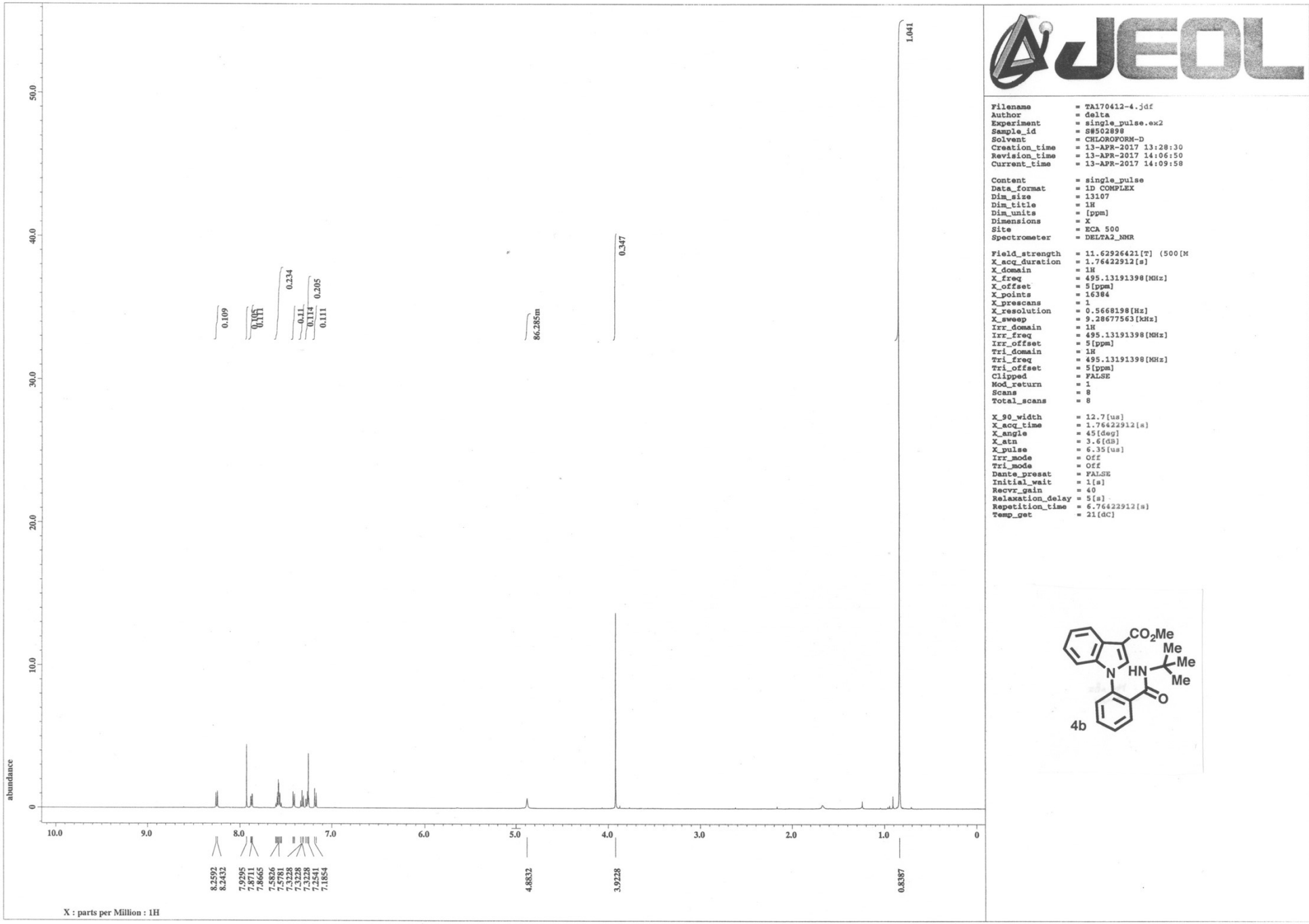
JEOL

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

Derived from: TA170217-16.jdf
```

```
Filename            = TA170217-18.jdf
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = S#497690
Solvent             = CHLOROFORM-D
Creation_time       = 20-FEB-2017 02:48:41
Revision_time       = 20-FEB-2017 07:52:58
Current_time        = 20-FEB-2017 07:55:26

Comment             = single pulse decouple
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA500
Spectrometer        = DELTA2_NMR

Field_strength      = 11.7473579[T]  (500[MH
X_acq_duration      = 0.83361792[s]
X_domain            = 13C
X_freq              = 125.76529768[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 1.19959034[Hz]
X_sweep             = 39.3081761[kHz]
Irr_domain          = 1H
Irr_freq            = 500.15991521[MHz]
Irr_offset          = 5.0[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 53418
Total_scans         = 53418

X_90_width          = 11.3[us]
X_acq_time          = 0.83361792[s]
X_angle             = 30[deg]
X_atn               = 5.5[dB]
X_pulse             = 3.76666667[us]
Irr_atn_dec         = 21.238[dB]
Irr_atn_noe         = 21.238[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     = 2.83361792[s]
Temp_get            = 23.4[dC]
```



3l



Filename = TA170412-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#502898
Solvent = CHLOROFORM-D
Creation_time = 13-APR-2017 13:28:30
Revision_time = 13-APR-2017 14:06:50
Current_time = 13-APR-2017 14:09:58

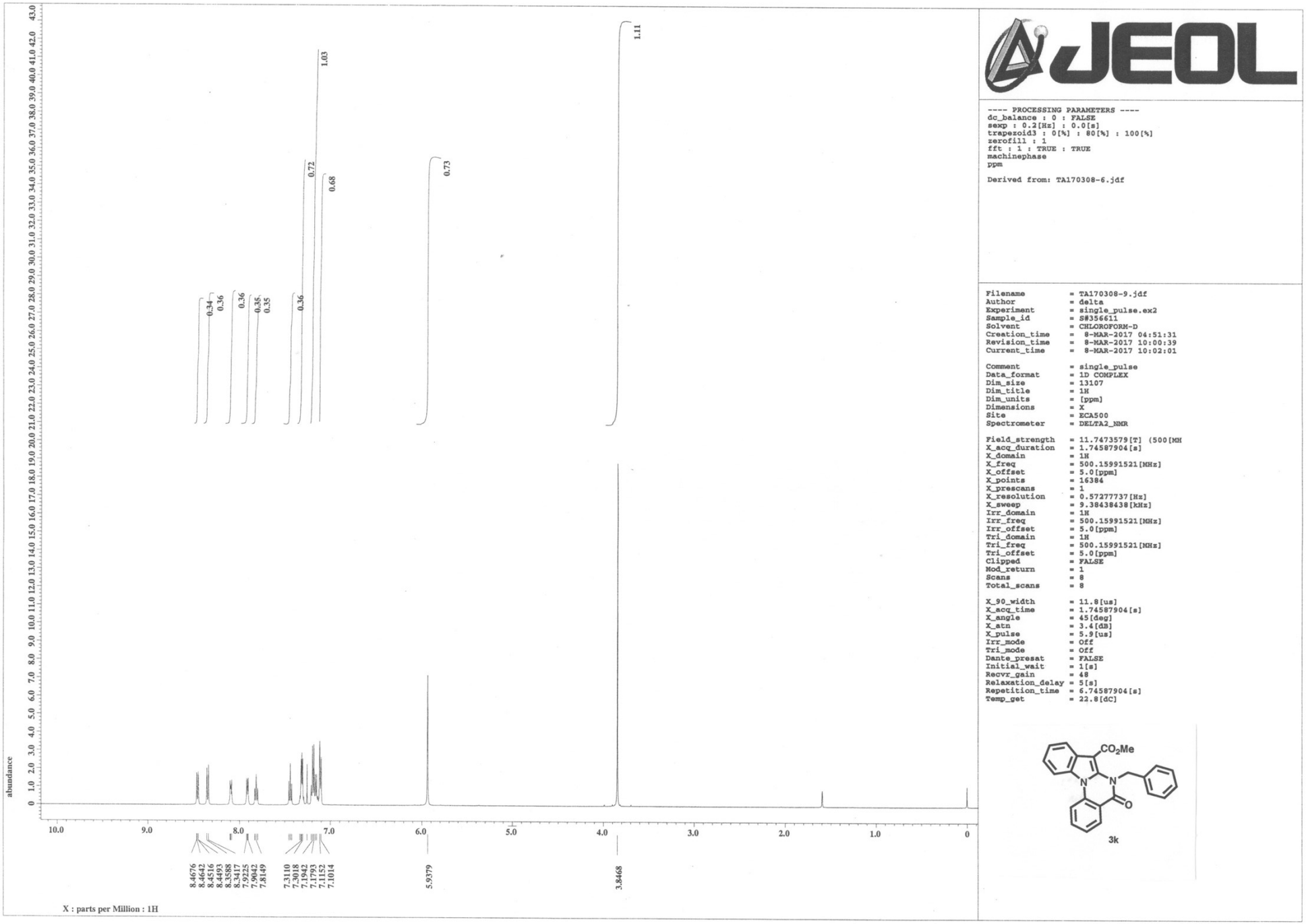
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 12.7[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.6[dB]
X_pulse = 6.35[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 21[dC]

4b

JEOL
Filename = TA170412-9.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SH543106
Solvent = CHLOROFORM-D
Creation_time = 14-APR-2017 08:19:57
Revision_time = 14-APR-2017 08:52:31
Current_time = 14-APR-2017 08:54:43
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 125.50100599[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
X_window = EM
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 22519
Total_scans = 22519
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.0[dB]
X_pulse = 3.53333333[us]
Irr_atn_dec = 20.0[dB]
Irr_atn_noe = 20.0[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Recy_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 21.5[dC]
CO2Me
Me
Me
HN
Me
O
N
4b
27.9771
51.5602
51.3304
77.3744
77.1158
76.8573
110.8197
109.7090
137.8881
134.5848
134.4986
131.4346
130.6973
129.5675
127.9398
126.3790
124.1672
122.9703
121.9841
165.1864
164.9374
X : parts per Million : 13C
abundance

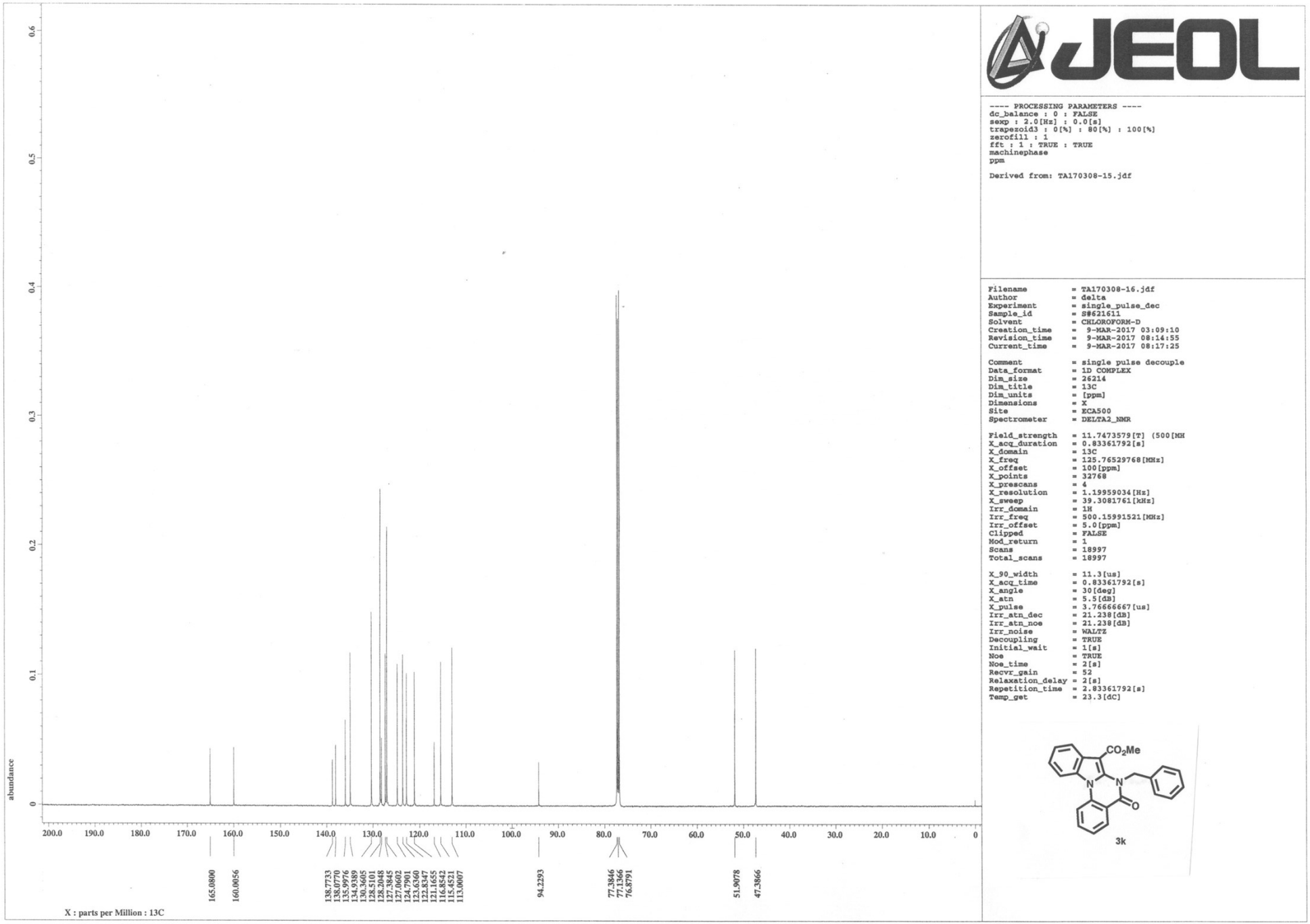


JEOL

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

Derived from: TA170308-6.jdf
```

```
Filename          = TA170308-9.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#356611
Solvent           = CHLOROFORM-D
Creation_time     =  8-MAR-2017 04:51:31
Revision_time     =  8-MAR-2017 10:00:39
Current_time      =  8-MAR-2017 10:02:01

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.8[dC]
```


3k

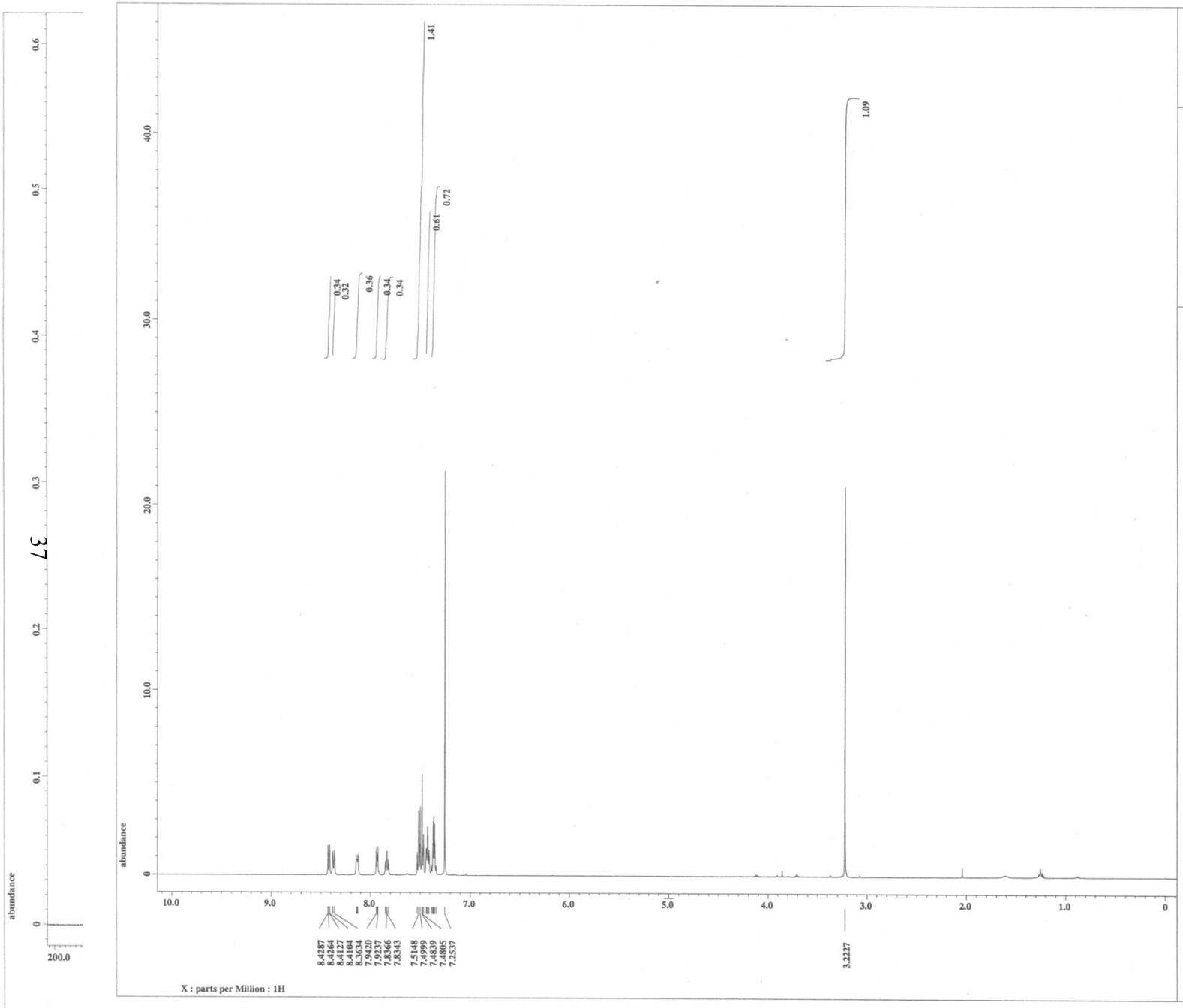


JEOL

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

Derived from: TA170308-15.jdf
```

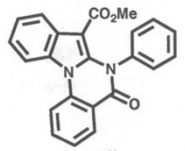
```
Filename          = TA170308-16.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#621611
Solvent           = CHLOROFORM-D
Creation_time     =  9-MAR-2017 03:09:10
Revision_time     =  9-MAR-2017 08:14:55
Current_time      =  9-MAR-2017 08:17:25

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 18997
Total_scans       = 18997

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 52
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 23.3[dC]
```


3k

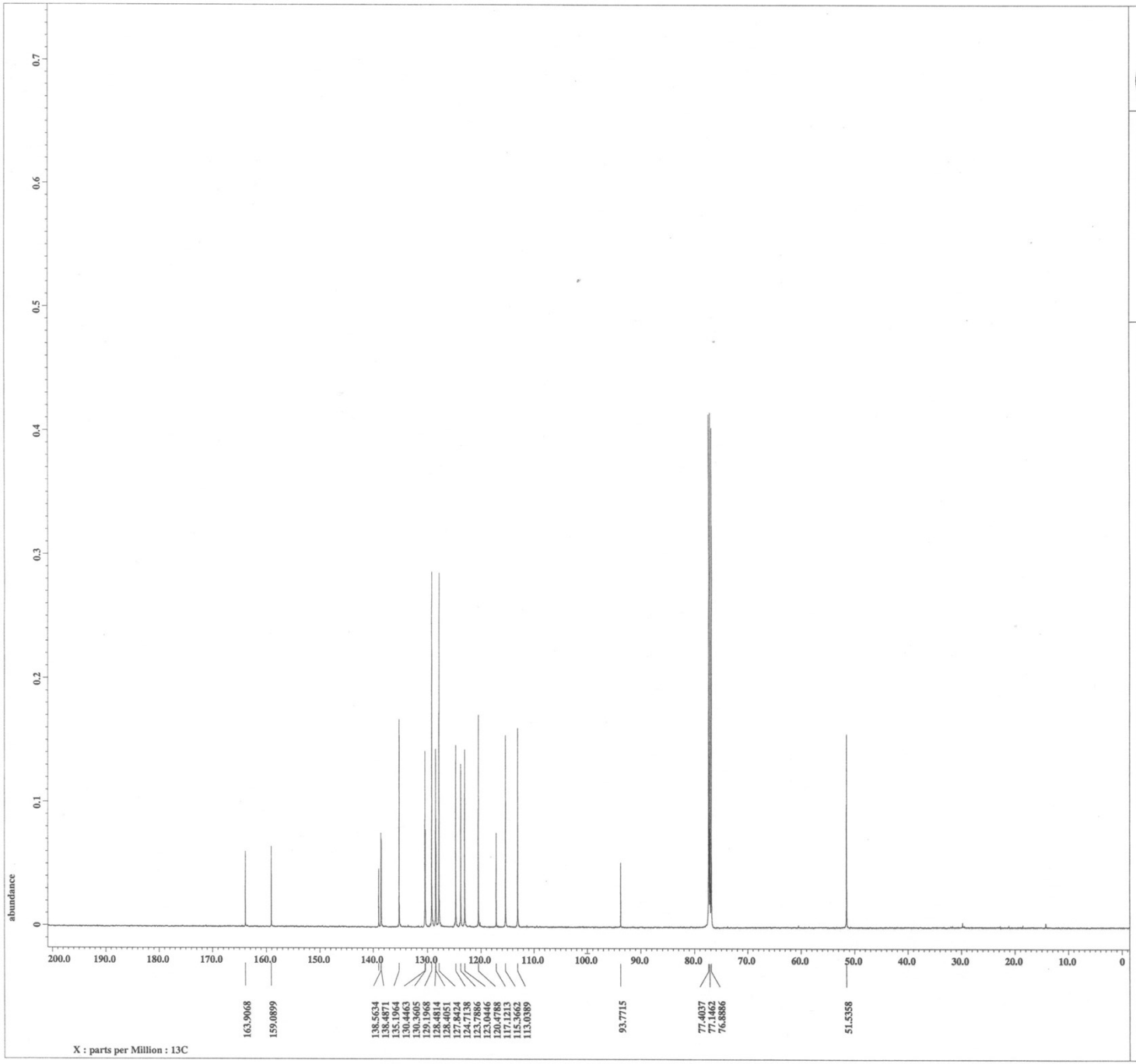


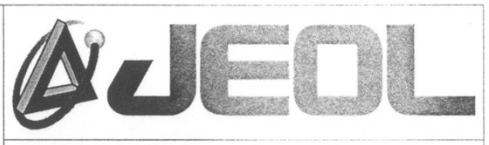
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA170405-1.jdf
```

Filename	= TA170405-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#339406
Solvent	= CHLOROFORM-D
Creation_time	= 5-APR-2017 04:21:23
Revision_time	= 5-APR-2017 09:30:37
Current_time	= 5-APR-2017 09:32:58
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 11.8[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.9[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 50
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 22[dC]



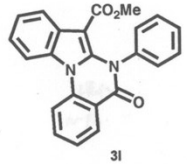
3l



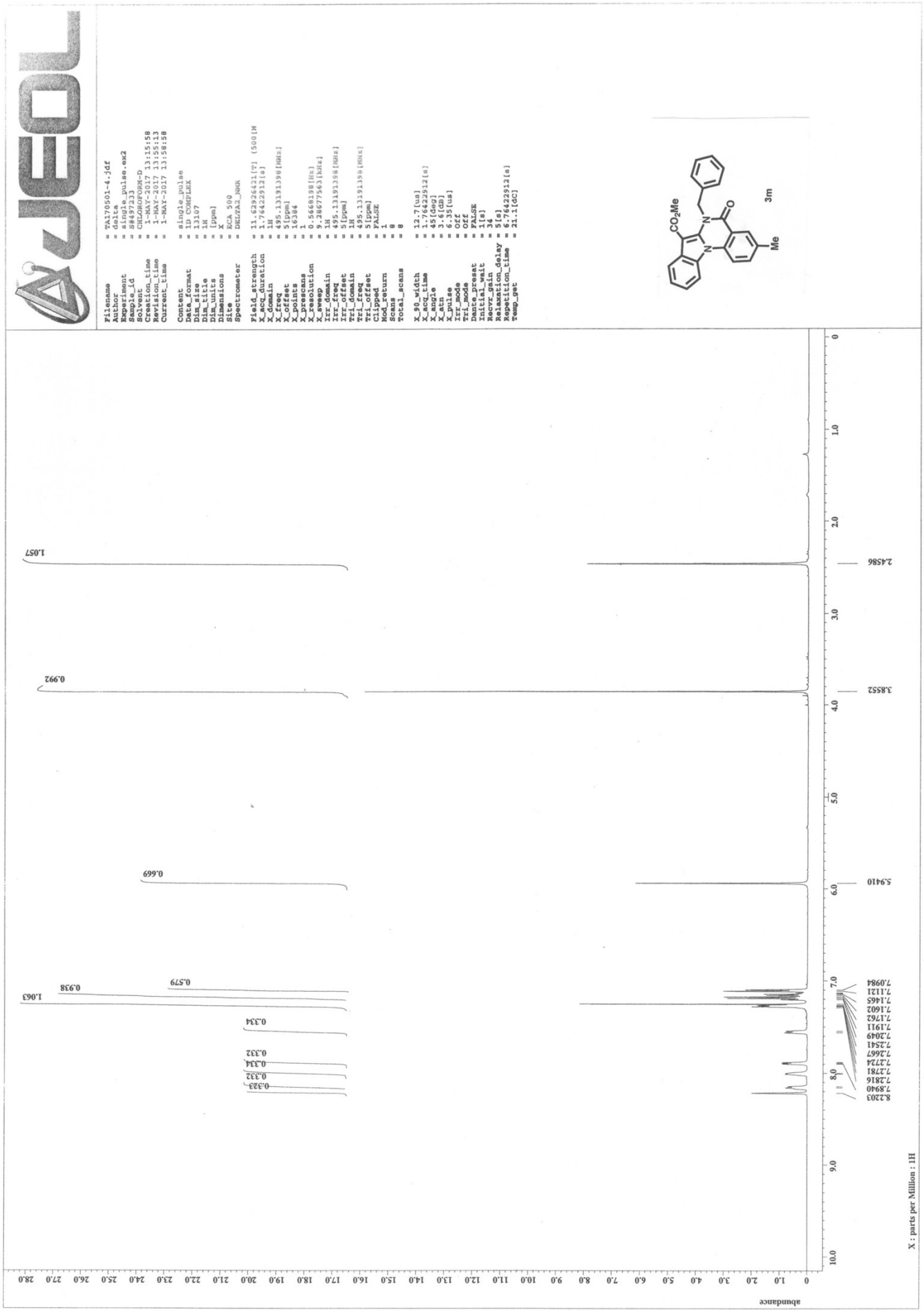


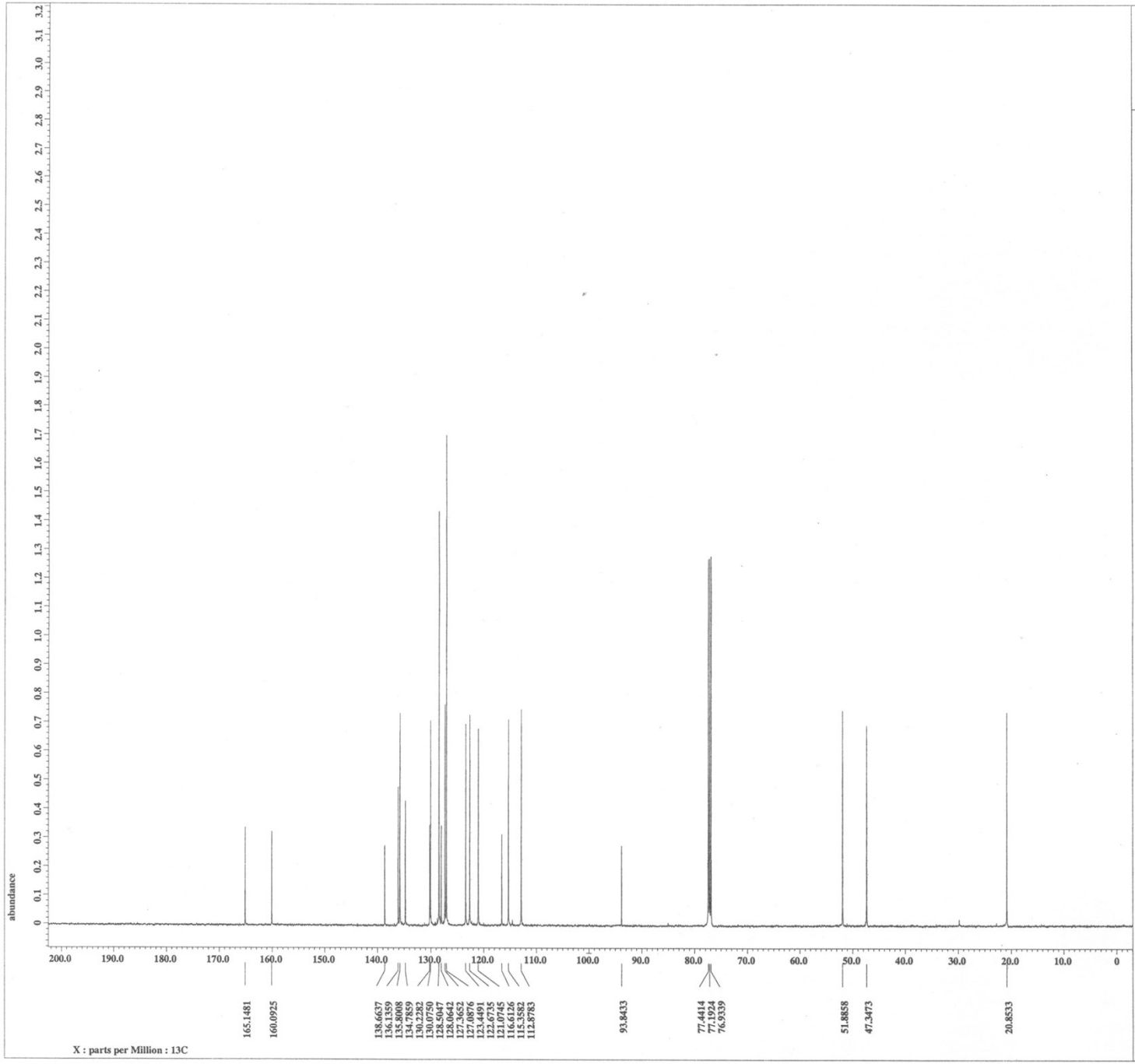
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA170405-11.jdf
```

```
Filename          = TA170405-13.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#540084
Solvent           = CHLOROFORM-D
Creation_time     =  6-APR-2017 02:32:56
Revision_time     =  6-APR-2017 07:38:46
Current_time      =  6-APR-2017 07:41:40

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 21128
Total_scans       = 21128

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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   = 2.83361792[s]
Temp_get          = 22.8[dC]
```



3l


JEOL
Filename = TA170501-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = MAR-231
Solvent = CHLOROFORM-D
Creation_time = 1-MAY-2017 13:15:58
Revision_time = 1-MAY-2017 13:55:13
Current_time = 1-MAY-2017 13:58:58
Content = single pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = JEOL 500
Spectrometer = DELTA2_NMR
Field_strength = 11.629264211[T] (500[M
X_acq_duration = 1.764229121[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.566919[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Xfer_return = 1
Scans = 8
Total_scans = 8
X_90_width = 12.7[us]
X_acq_time = 1.764229121[s]
X_angle = 45[deg]
X_atn = 36[dB]
X_pulse = 6.35[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recycle_time = 5[s]
Relaxation_delay = 5[s]
Repetition_time = 6.764229121[s]
Temp_get = 21.1[dC]
CO2Me
Me
3m
abundance
X : parts per Million : 1H
1.057
0.992
0.669
0.579
0.938
1.063
0.334
0.332
0.334
0.332
0.323
2.4586
3.8552
5.9410
7.0984
7.1121
7.1465
7.1602
7.1762
7.1911
7.2049
7.2541
7.2667
7.2724
7.2781
7.2816
7.8940
8.2203

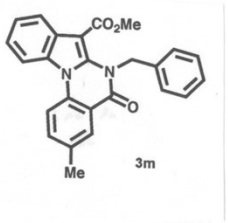


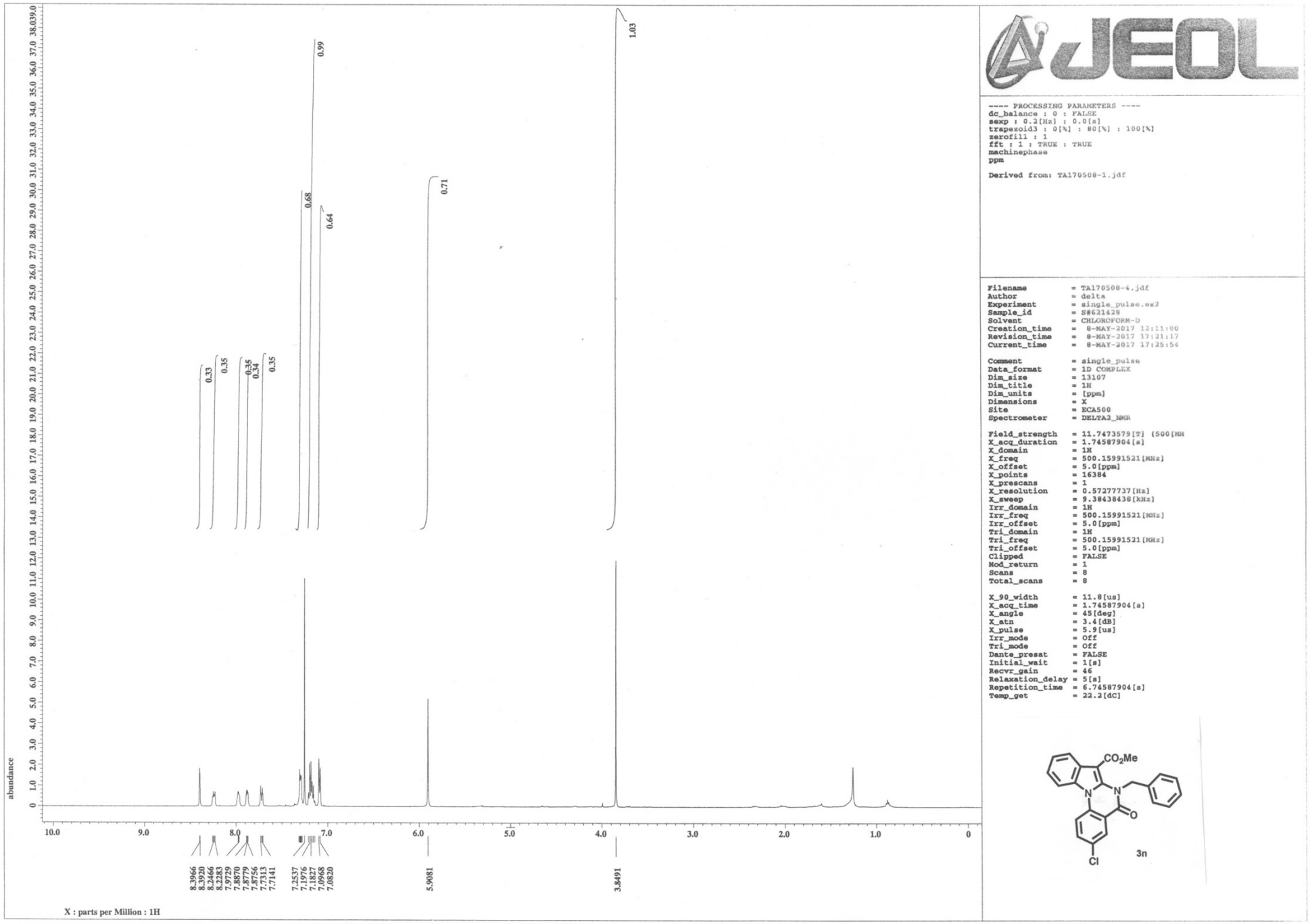
Filename = TA170501-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 88550408
Solvent = CHLOROFORM-D
Creation_time = 1-MAY-2017 17:15:51
Revision_time = 1-MAY-2017 17:52:21
Current_time = 1-MAY-2017 17:54:23

Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 126.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 3215
Total_scans = 3215

X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.8[dB]
X_pulse = 3.53333333[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 = 2.8388608[s]
Temp_get = 22.1[dC]



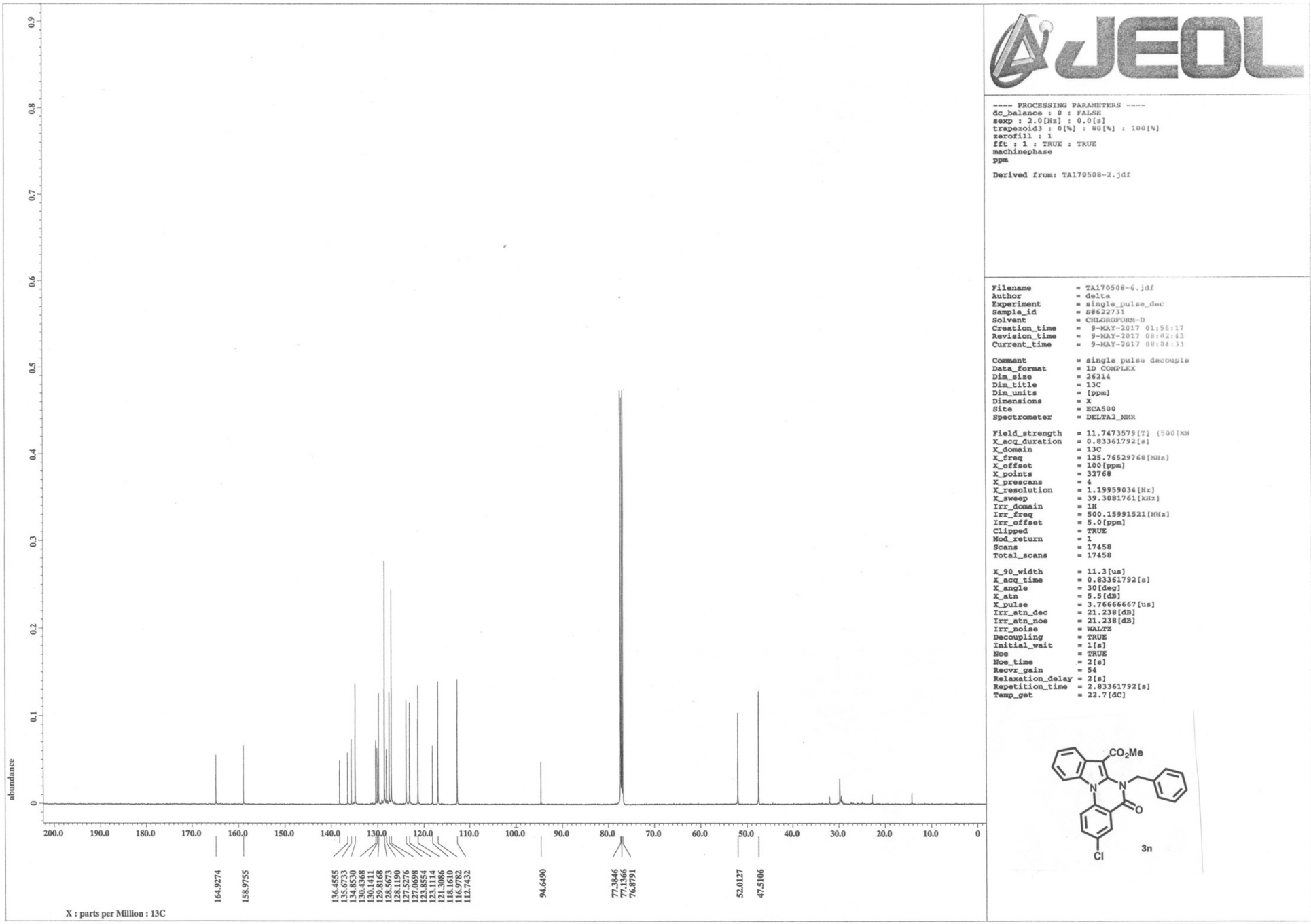


JEOL

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

Derived from: TA170508-1.jdf
```

Filename	= TA170508-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#621428
Solvent	= CHLOROFORM-D
Creation_time	= 8-MAY-2017 12:11:00
Revision_time	= 8-MAY-2017 17:21:17
Current_time	= 8-MAY-2017 17:25:54
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 11.8[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.9[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 46
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 22.2[dC]

3n

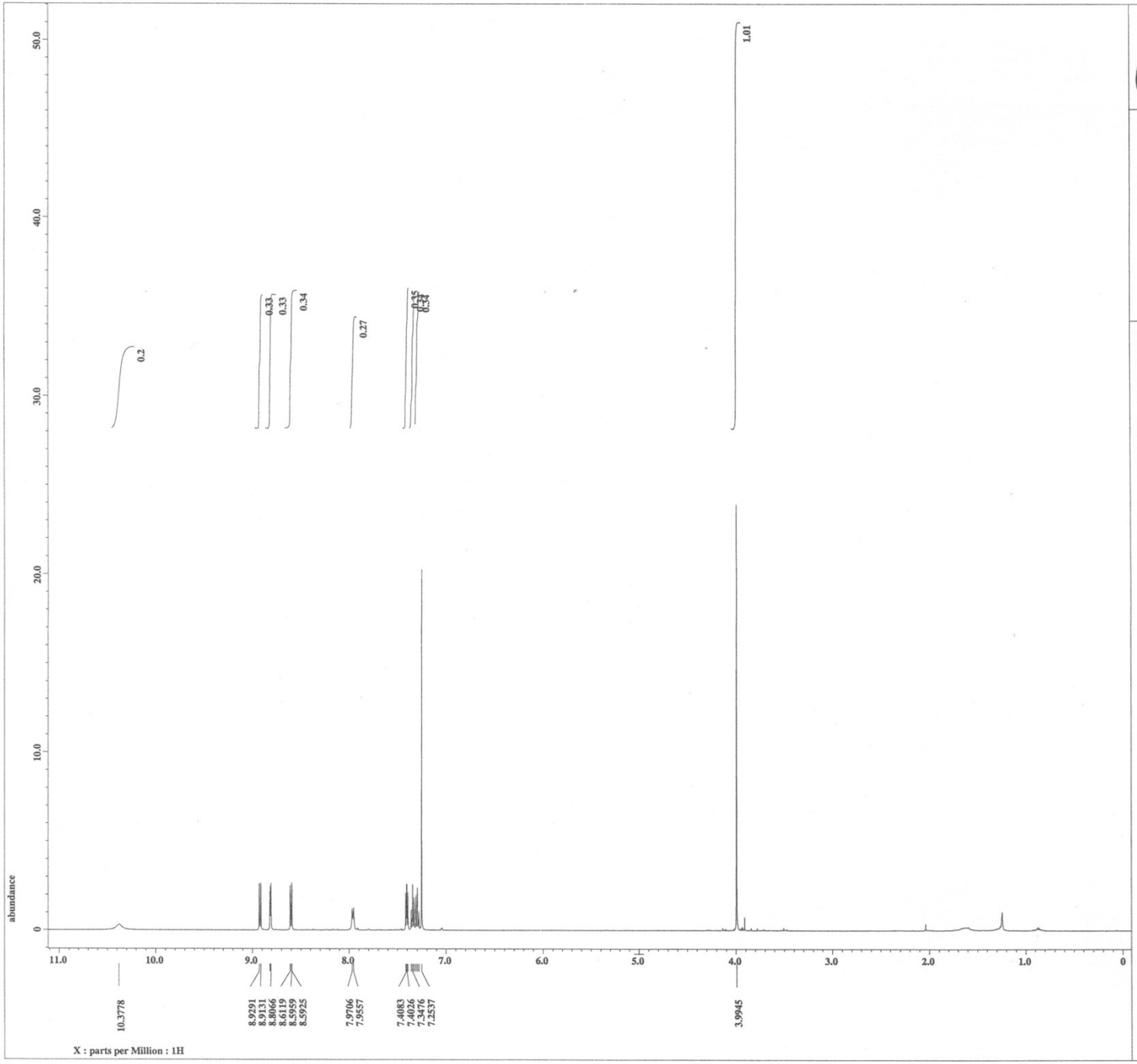


JEOL

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

Derived from: TA170508-2.jdf
```

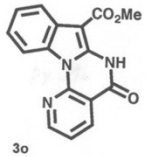
Filename	= TA170508-4.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#622731
Solvent	= CHLOROFORM-D
Creation_time	= 9-MAY-2017 01:56:17
Revision_time	= 9-MAY-2017 08:02:42
Current_time	= 9-MAY-2017 08:04:33
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 0.83361792[s]
X_domain	= 13C
X_freq	= 125.76529768[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.19959034[Hz]
X_sweep	= 39.3081761[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Clipped	= TRUE
Mod_return	= 1
Scans	= 17458
Total_scans	= 17458
X_90_width	= 11.3[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 5.5[dB]
X_pulse	= 3.76666667[us]
Irr_atn_dec	= 21.238[dB]
Irr_atn_noe	= 21.238[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	= 2.83361792[s]
Temp_get	= 22.7[dC]

3n

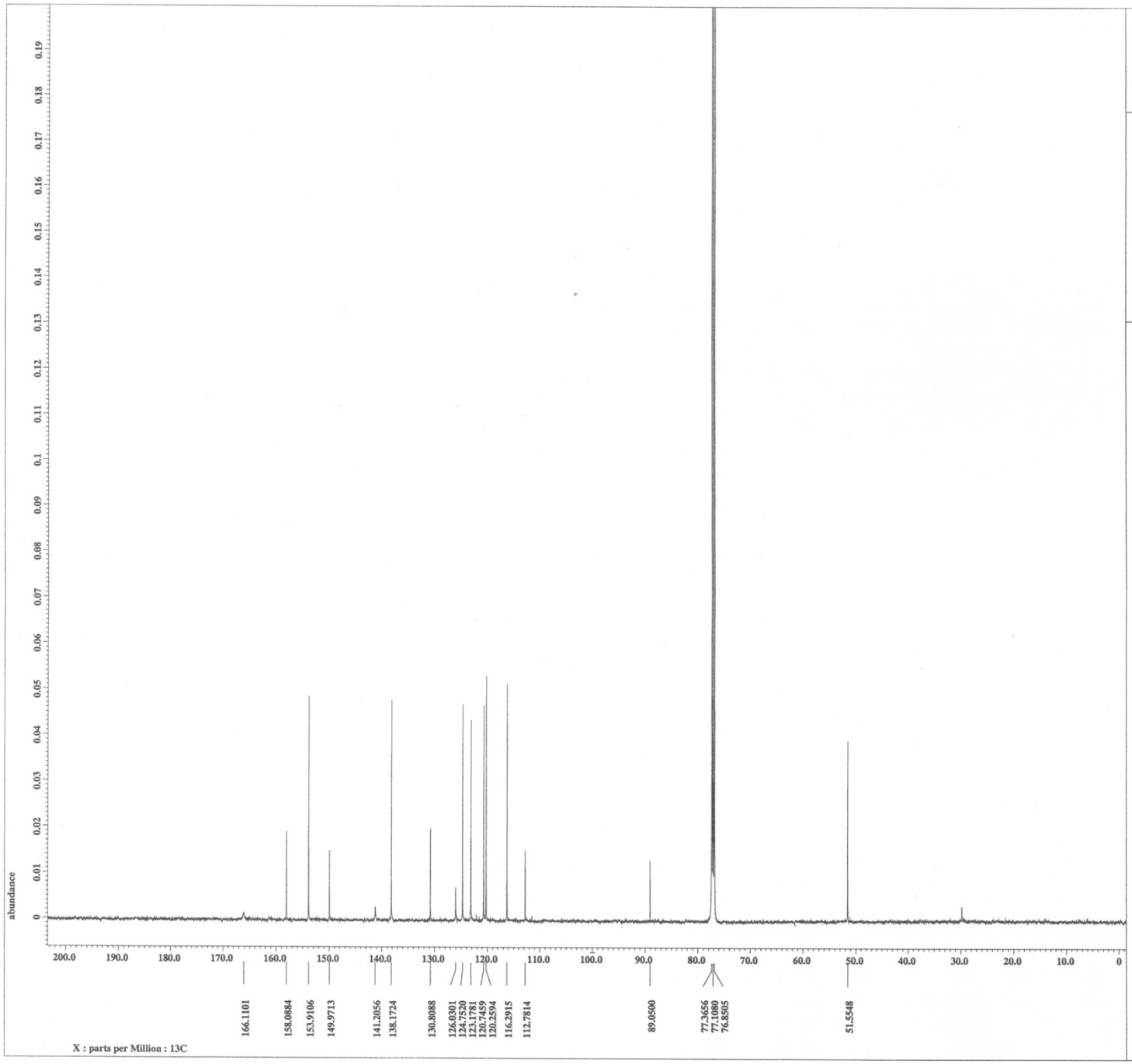


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA170510-3.jdf
```

Filename	= TA170510-7.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#680065
Solvent	= CHLOROFORM-D
Creation_time	= 10-MAY-2017 13:48:12
Revision_time	= 10-MAY-2017 18:58:27
Current_time	= 10-MAY-2017 19:00:52
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 11.8[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.9[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 58
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 22.3[dC]



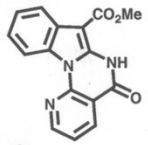
3o



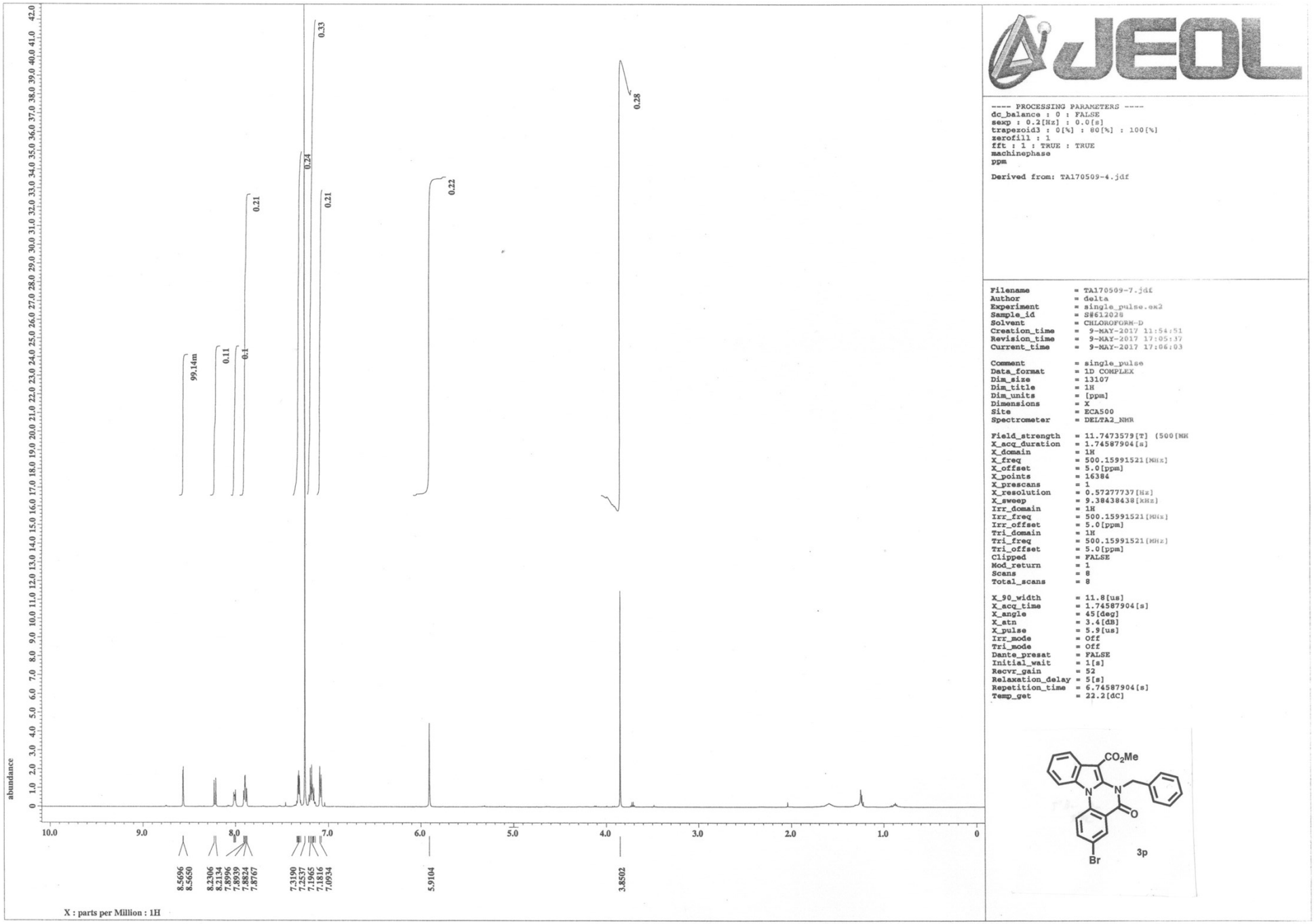
JEOL

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

Derived from: TA170510-4.jdf
```

Filename	= TA170510-6.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#681087
Solvent	= CHLOROFORM-D
Creation_time	= 11-MAY-2017 03:23:24
Revision_time	= 11-MAY-2017 08:35:29
Current_time	= 11-MAY-2017 08:37:33
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 0.83361792[s]
X_domain	= 13C
X_freq	= 125.76529768[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.19959034[Hz]
X_sweep	= 39.3081761[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 17245
Total_scans	= 17245
X_90_width	= 11.3[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 5.5[dB]
X_pulse	= 3.76666667[us]
Irr_atn_dec	= 21.238[dB]
Irr_atn_noe	= 21.238[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 52
Relaxation_delay	= 2[s]
Repetition_time	= 2.83361792[s]
Temp_get	= 22.9[dC]



3o

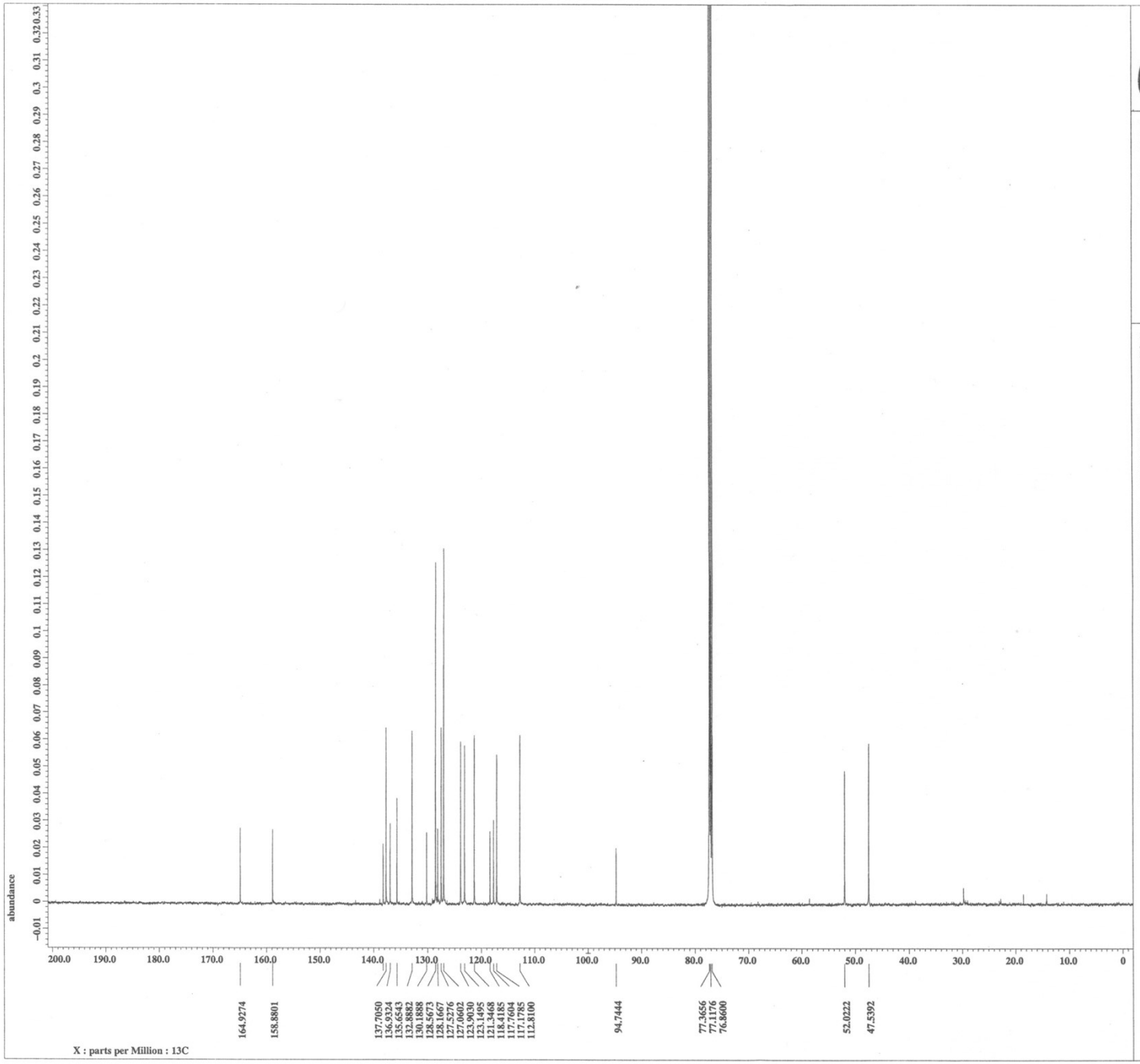


---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA170509-4.jdf

Filename	= TA170509-7.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#612028
Solvent	= CHLOROFORM-D
Creation_time	= 9-MAY-2017 11:54:51
Revision_time	= 9-MAY-2017 17:05:37
Current_time	= 9-MAY-2017 17:06:03
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 11.8[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.9[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 52
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 22.2[dC]

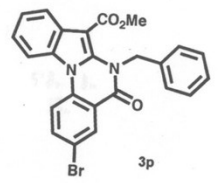
3p



JEOL

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

Derived from: TA170509-5.jdf
```

Filename	= TA170509-7.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#613388
Solvent	= CHLOROFORM-D
Creation_time	= 10-MAY-2017 02:23:12
Revision_time	= 10-MAY-2017 07:31:00
Current_time	= 10-MAY-2017 07:34:48
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 0.83361792[s]
X_domain	= 13C
X_freq	= 125.76529768[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.19959034[Hz]
X_sweep	= 39.3081761[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 18359
Total_scans	= 18359
X_90_width	= 11.3[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 5.5[dB]
X_pulse	= 3.76666667[us]
Irr_atn_dec	= 21.238[dB]
Irr_atn_noe	= 21.238[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	= 2.83361792[s]
Temp_get	= 22.6[dC]



3p