Supporting Information for

Ylide Mediated Carbonyl Homologations for the Preparation of Isatin Derivatives

Christina T. Lollar,[a] Katherine M. Krenek,[a] Kevin J. Bruemmer,[a] and Alexander R. Lippert*[a,b]

[a]Department of Chemistry and [b]Center for Drug Discovery, Design, and Delivery (CD4), Southern Methodist University, Dallas, TX 75275-0314, USA

General Methods: All reactions were carried out in dried glassware under an atmosphere of N_2. *N,N*-Diisopropylethylamine was distilled from KOH before use. All other reagents were used without further purification. *N*-Phenyl anthranilic acid was prepared according to a literature procedure.[1] All other reagents were purchased from Sigma-Aldrich (St. Louis, MO), Alfa Aesar (Ward Hill, MA), or Matrix Scientific (Columbia, SC). Column chromatography was performed on Silica gel P60 (SiliCycle). ^{1}H NMR and ^{13}C NMR were measured on a JEOL 500 MHz spectrometer in the Department of Chemistry at Southern Methodist University. Chemical shift units are reported using parts per million (ppm) with the peaks from residual proton signals serving as an internal reference. Coupling constant units are in Hertz (Hz) and splitting patterns are abbreviated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dd, doublet of doublets, td, triplet of doublets; br, broad. High-resolution mass spectroscopy was performed on a Shimadzu IT-TOF (ESI source) at the Shimadzu Center for Advanced Analytical Chemistry at the University of Texas, Arlington. Melting points were measured using a Mel-Temp II (Laboratory Devices, USA) equipped with a Fluke 51 K/J Thermometer. Chemical abbreviations are used as follows: CH_2Cl_2, dichloromethane; EtOAc, ethyl Acetate; THF, tetrahydrofuran; DMF, dimethylformamide; H_2O, water; HBTU, *O*-benzotriazole-*N,N,N',N'*-tetramethyl-uronium-hexafluoro-phosphate; DIPEA, *N,N*-diisopropylethylamine; KOH, potassium hydroxide; K_2CO_3, potassium carbonate; N_2, nitrogen.

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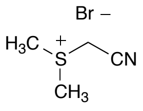
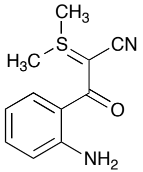
[1] C. Pal, M. K. Kundu, U. Bandyopadhyay, S. Adhikari, *Bioorg. Med. Chem. Lett.* 2011, **21**, 3563.



1

Sulfonium Salt (1):

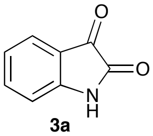
Bromoacetonitrile (7.5 mL, 100 mmol, 1.0 equiv.) was added to dimethyl sulfide (7.2 mL, 100 mmol, 1.0 equiv.) and stirred in a round bottom flask overnight. The solids were carefully broken apart with a spatula and minimal exposure to the atmosphere. Residual reagent was removed *in vacuo* to yield the sulfonium salt **1** as a white solid (17 g, 93%). ^{1}H NMR (500 MHz, CD_3OD) δ 4.85 (s, 2H), δ 3.09 (s, 6H); ^{13}C NMR (125 MHz, CD_3OD) δ 112.04, δ 29.48, δ 25.89.



2a

3-(2-aminophenyl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2a):

2-aminobenzoic acid (135.0 mg, 0.9915 mmol, 1.0 equiv) was dissolved in 5 mL CH_2Cl_2. HBTU (458.2 mg, 1.208 mmol, 1.2 equiv), sulfonium salt **1** (546.7 mg, 3.003 mmol, 3.0 equiv), and DIPEA (0.52 mL, 3.0 mmol, 3.0 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1 M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2a** was used in the next step without further purification. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65–7.63 (d, 1H, *J* = 8 Hz), δ 7.11–7.08 (t, 1H, *J* = 8 Hz), δ 6.61–6.58 (m, 2H), δ 5.23–5.18 (br s, 2H, NH_2 peak), δ 2.73 (s, 6H); HRMS calcd for $C_{11}H_{12}N_2OS$ [M+H]+ 221.0743, found 221.0740.

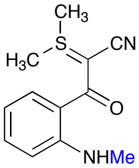


3a

1*H*-indole-2,3-dione (3a):

The sulfur ylide **2a** was dissolved in 2:1 THF:H_2O (2 mL:1 mL) before Oxone (461.4 mg, 0.7501 mmol, 1.5 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL H_2O, extracted three times with 10 mL CH_2Cl_2, washed with 10 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. Purification of the crude product was accomplished by silica gel column chromatography (2:1

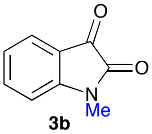
Hexane:EtOAc) to yield 1*H*-indole-2,3-dione **3a** (59.5 mg, 81%). The two-step sequence was reproduced on the same scale to yield **3a** in 88% yield. mp 198–199 ºC (lit.[2] 200–202 ºC); ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.99 (s, 1H), δ 7.63–7.61 (d, 1H, *J* = 7.45 Hz), δ 7.58–7.57 (t, 1H, *J* = 7.45 Hz), δ 7.14–7.11 (t, 1H, *J* = 8 Hz), δ 6.92–6.91 (d, 1H, *J* = 8 Hz); ^{13}C NMR (125 MHz, d6 acetone) δ 183.98, δ 159.98, δ 150.77, δ 138.42, δ 124.65, δ 123.11, δ 118.27, δ 112.35; HRMS calcd for $C_8H_5NO_2$ [M+Na]+ 170.0213, found 170.0211.



2b

2-(dimethyl-l^4-sulfanylidene)-3-[2-(methylamino)phenyl]–3-oxopropanenitrile (2b):

2-(methylamino)benzoic acid (151.17 mg, 0.9380 mmol, 0.94 equiv) was dissolved in 10 mL CH_2Cl_2. HBTU (455.1 mg, 0.9999 mmol, 1.1 equiv), sulfonium salt **1** (546.2 mg, 3.000 mmol, 3.2 equiv), and DIPEA (0.52 mL, 3.0 mmol, 3.2 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M $K_2{\cdot}CO_3$, extracted four times with 10 mL CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2b** was used in the next step without further purification. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (br s, 1H), δ 7.27 (m, 2H), δ 6.61–6.58 (m, 2H), δ 2.80 (s, 3H), δ 2.78 (s, 6H); HRMS calcd for $C_{12}H_{14}N_2OS$ [M+H]+ 235.0900, found 235.0894.



3b

1-methyl-1*H*-indole-2,3-dione (3b):

The sulfur ylide **2b** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (615.1 mg, 0.9999 mmol, 1.1 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL H_2O, extracted three times with 10 mL CH_2Cl_2, washed with 10 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide was used in the next step without further purification. Purification of the crude product was accomplished by silica gel column chromatography (3:1 Hexane:EtOAc) to yield 1-methyl-1*H*-indole-2,3-dione **3b** (97.3 mg, 65%). The two-step sequence

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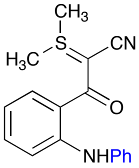
[2] C. S. Marvel, G. S. Hiers, *Org. Synth., Coll. Vol.* 1925, **1**, 327.

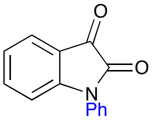
was reproduced on the same scale to yield **3b** in 55% yield. mp 126–129 ºC (lit.[3] 131–133 ºC); ^{1}H NMR (500 MHz, $(CD_3)_2CO$) δ 7.65 (td, 1H, *J* = 8, 1.2 Hz), δ 7.49 (d, 1H, *J* = 7.45), δ 7.12 (td, 1H, *J* = 7.5, 1.2 Hz), δ 7.08 (d, 1H, *J* = 8 Hz), 3.19 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 183.44, δ 158.34, δ 151.55, δ 138.49, δ 125.40, δ 123.94, δ 117.55, δ 110.01, δ 26.33; HRMS calcd for $C_9H_7NO_2$ [M+Na]+ 184.0369, found 184.0376.



2c

2-(dimethyl-l^4-sulfanylidene)-3-oxo-3-[2-(phenylamino)phenyl]propanenitrile (2c):

N-phenylanthranlic acid (47.0 mg, 0.220 mmol, 1.0 equiv) was dissolved in 3 mL CH_2Cl_2. HBTU (100.0 mg, 0.264 mmol, 1.2 equiv), sulfonium salt **1** (120 mg, 0.661 mmol, 3.0 equiv), and DIPEA (0.115 mL, 0.662 mmol, 3.0 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, washed with 20 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2c** was used in the next step without further purification. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.89 (br s, 1H), δ 7.77–7.76 (d, 1H, *J* = 7.5 Hz), δ 7.33–7.32 (d, 1H, *J* = 8 Hz), δ 7.26–7.20 (m, 5H), δ 6.94–6.91 (t, 1H, *J* = 7.5 Hz), δ 6.80–6.77 (t, 1H, *J* = 7.5 Hz), δ 2.79 (s, 6H); HRMS calcd for $C_{17}H_{16}N_2OS$ [M+Cl]- 331.0677, found 331.0678.



3c

1-phenyl-1*H*-indole-2,3-dione (3c):

The sulfur ylide **2c** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (199.3 mg, 0.3519 mmol, 1.6 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a separatory funnel containing 20 mL EtOAc and 20 mL H_2O, extracted three times with 10 mL EtOAc, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. Purification of the crude product was accomplished by silica column chromatography (6:1 Hexane:EtOAc) to yield 1-phenyl-1*H*-indole-2,3-dione **3c** (49 mg, 65%). The two-step sequence was reproduced on the same

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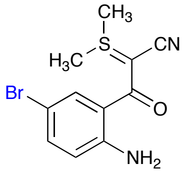
[3] P. G. Gassman, B. W. Cue, Jr., T.-Y. Luh, *J. Org. Chem.* 1977, **42**, 1344.

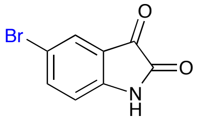
scale to yield **3c** in 87% yield. mp 134–136 ºC (lit.[4] 138–139 ºC); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (dd, 1H, *J* = 7.5, 1.2 Hz), δ 7.57–7.52 (m, 3H), δ 7.47–7.41 (m, 3H), δ 7.17 (t, 1H, *J* = 7.4), δ 6.89 (d, 1H, *J* = 8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 183.01, δ 157.42, δ 151.76, δ 138.44, δ 132.96, δ 130.06, δ 128.94, δ 126.09, δ 125.72, δ 124.41, δ 117.59, δ 111.39; HRMS calcd for $C_{14}H_9NO_2$ [M+H]+ 224.0706, found 224.0701.



2d

3-(2-amino-5-bromophenyl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2d):

2-amino-5-bromobenzoic acid (137.14 mg, 0.6067 mmol, 0.61 equiv) was dissolved in 10 mL CH_2Cl_2. HBTU (455.16 mg, 1.2 mmol, 1.2 equiv), sulfonium salt **1** (546.24 mg, 3.0 mmol, 3.0 equiv), and DIPEA (0.52 mL, 3.0 mmol, 4.9 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2d** was used in the next step without further purification. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76–7.75 (d, 1H, *J* = 2.3), δ 7.21–7.19 (dd, 1H, *J* = 2.3, 8.6), δ 6.53–6.52 (d, 1H, *J* = 8.6), δ 5.27 (br s, 2H), δ 2.76 (s, 6H); HRMS calcd for $C_{11}H_{11}N_2OSBr$ [M+H]+ 298.9848, found 298.9851.



3d

5-bromo-1*H*-indole-2,3-dione (3d):

The sulfur ylide **2d** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (922.7 mg, 1.5 mmol, 1.5 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a separatory funnel containing 20 mL CH_2Cl_2 and 20 mL H_2O, extracted three times with 20 mL CH_2Cl_2, washed with 20 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. Purification of the crude

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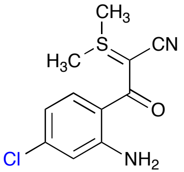
[4] Dombrowski, James E. (44 Mayer Road, Portland, Maine, 04102, US), Mattingly, Phillip Gregory (204 Seafarer Drive, Grayslake, Illinois, 60030, US) 1994, "N-arylation of isatins" ABBOTT LABORATORIES (One Abbott Park Road, Abbott Park, Illinois, 60064-3500, US) EP0369344
http://www.freepatentsonline.com/EP0369344B1.html

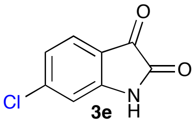
product was accomplished by silica gel column chromatography (2:1 Hexane:EtOAc) to yield 5-bromo-1*H*-indole-2,3-dione **3d** (79.7 mg, 54%). The two-step sequence was reproduced on the same scale to yield the **3d** in 84% yield. mp 250–254 ºC (lit.[5] 255–256 ºC); ^{1}H NMR (500 MHz, DMSO-d6) δ 11.10 (s, 1H), δ 7.70–7.68 (dd, 1H, *J* = 2, 8.5 Hz), δ 7.61 (s, 1H), δ 6.84-6.82 (d, 1H, *J* = 8 Hz); ^{13}C NMR (125 MHz, DMSO-D6) δ 183.71, δ 159.51, δ 150.08, δ 140.55, δ 127.42, δ 120.09, δ 114.82, δ 114.79; HRMS calcd for $C_8H_4NO_2Br$ [M+H]+ 225.9498, found 225.9488.



2e

3-(2-amino-4-chlorophenyl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2e):

4-chloroanthranilic acid (172.5 mg, 1.01 mmol, 1.0 equiv) was dissolved in 10 mL CH_2Cl_2. HBTU (454.4 mg, 1.20 mmol, 1.2 equiv), sulfonium salt **1** (545.5 mg, 3.00 mmol, 3.0 equiv), and DIPEA (0.52 mL, 3.0 mmol, 3.0 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was then poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, washed with 20 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2e** was used in the next step without further purification. ^{1}H NMR (500 MHz, $(CD_3)_2CO$) δ 7.62–7.60 (d, 1H, *J* = 8.5 Hz), δ 6.61–6.61 (d, 1H, *J* = 2 Hz), δ 6.58–6.56 (dd, 1H, *J* = 2, 8.5 Hz), δ 2.78 (s, 6H); HRMS calcd for $C_{11}H_{11}N_2OSCl$ [M+H]+ 255.0353, found 255.0349.



3e

6-chloro-1*H*-indole-2,3-dione (3e):

The sulfur ylide **2e** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (926.3 mg, 1.51 mmol, 1.5 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a separatory funnel containing 20 mL CH_2Cl_2 and 20 mL H_2O, extracted three times with 20 mL CH_2Cl_2. The organic layer was washed with 20 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude material was purified by silica gel column chromatography (2:1

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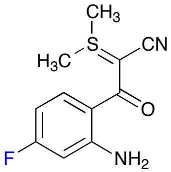
[5] B. Campaigne, W. Archer, *J. Am. Chem. Soc.* 1952, **74**, 5801.

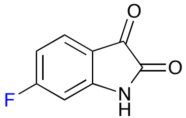
hexanes:EtOAc) to yield 7-methyl-1*H*-indole-2,3-dione **3e** (115.1 mg, 63%). The two-step sequence was reproduced on the same scale to yield **3e** in 57% yield. mp 258–261 ºC (lit.[6] 263 ºC); ^{1}H NMR (500 MHz, $(CD_3)_2CO$) δ 10.13 (s, 1H), δ 7.55–7.53 (d, 1H, *J* = 8 Hz), δ 7.14–7.12 (dd, 1H, *J* = 1.7, 8 Hz), δ 7.05–7.05 (d, 1H, *J* = 1.7 Hz); ^{13}C NMR (125 MHz, $(CD_3)_2CO$) δ 182.57, δ 158.95, δ 151.88, δ 143.35, δ 126.12, δ 123.18, δ 116.99, δ 112.60; HRMS calcd for $C_8H_4NO_2Cl$ [M+H]+ 182.0003, found 182.0003.



2f

3-(2-amino-4-fluorophenyl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2f):

4–fluoroanthranlic acid (77.4 mg, 0.499 mmol, 1.0 equiv) was dissolved in 5 mL CH_2Cl_2. HBTU (226.7 mg, 0.5977 mmol, 1.2 equiv), sulfonium salt **1** (272.1 mg, 1.494 mmol, 3.0 equiv), and DIPEA (0.26 mL, 1.50 mmol, 3.0 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2f** was used in the next step without further purification. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (br t, 1H, *J* = 6.9 Hz), δ 6.20–6.17 (dd, 1H, *J* = 2.3, 11.5 Hz), δ 6.11 (td, 1H, *J* = 2.3, 11.5 Hz), δ 5.50 (br s, 2H), δ 2.60 (s, 6H); HRMS calcd for $C_{11}H_{11}N_2OFS$ [M+H]+ 239.0649, found 239.0652.



3f

6-fluoro-1*H*-indole-2,3-dione (3f):

The sulfur ylide **2f** was dissolved in 2:1 THF:H_2O (6.4 mL: 3.2 mL) before Oxone (463.3 mg, 0.7531 mmol, 1.5 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was poured into a separatory funnel containing 20 mL CH_2Cl_2 and 20 mL H_2O, extracted three times with 20 mL CH_2Cl_2, washed with 20 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. Purification of the crude product was accomplished by silica gel column chromatography (3:1 Hexane:EtOAc) to yield 6-fluoro-1*H*-indole-2,3-dione **3f** (61.8 mg, 80%). The two-

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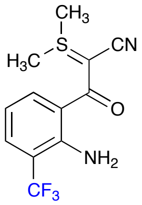
[6] P. W. Sadler, R. L. Warren, *J. Am. Chem. Soc.* 1956, **78**, 1251.

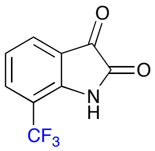
step sequence was reproduced on the same scale to yield **3f** in 83% yield. mp 197–199 ºC (lit.[7] 197 ºC); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (br s, 1H), δ 7.66 (q, 1H), δ 6.81 (t, 1H, J = 1.7 Hz), δ 6.64 (d, 1H, J = 8.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 180.54, δ 170.16, δ 168.20 (d, J = 39.4 Hz), δ 159.05 (d, J = 13.1 Hz), δ 151.31 (d, J = 11.9 Hz), δ 128.70 (d, J = 11.9 Hz), δ 111.36 (d, J = 23.8 Hz), 100.83 (d, J = 27.4 Hz); HRMS calcd for $C_8H_4NO_2F$ [M-H]- 164.0153, found 164.0159.



2g

3-[2-amino-3-(trifluoromethyl)phenyl]-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2g):

6-trifluoromethylanthranlic acid (204.3 mg, 0.9959 mmol, 1.0 equiv) was dissolved in 10 mL CH_2Cl_2. HBTU (453.4 mg, 1.195 mmol, 1.2 equiv), sulfonium salt **1** (543.4 mg, 2.984 mmol, 3.0 equiv), and DIPEA (0.52 mL, 3.0 mmol, 3.0 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2g** was used in the next step without further purification. ^{1}H NMR (500 MHz, $(CD_3)_2CO$) δ 7.77–7.75 (d, 1H, J = 7.5 Hz), δ 7.41–7.39 (d, 1H, J = 7.5 Hz), δ 6.65–6.62 (t, 1H, J = 7.5 Hz), δ 5.70 (br s, 2H), δ 2.84 (s, 6H); HRMS calcd for $C_{12}H_{11}N_2OF_3S$ [M+H]+ 289.0617, found 289.0614.



3g

7-(trifluoromethyl)-1*H*-indole-2,3-dione (3g):

The sulfur ylide **2g** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (930.6 mg, 1.513 mmol, 1.5 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a separatory funnel containing 20 mL CH_2Cl_2 and 20 mL H_2O, extracted three times with 10 mL CH_2Cl_2, 20 mL brine, and then dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. Purification of the crude product was accomplished by silica gel column chromatography (4:1

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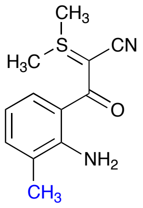
[7] P. W. Sadler, *J. Org. Chem.* 1957, **22**, 283.

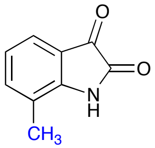
Hexane:EtOAc) to yield 7-(trifluoromethyl)-1*H*-indole-2,3-dione **3g** (210.4 mg, 98%). The two-step sequence was reproduced on the same scale to yield **3g** in 84% yield. mp 187–190 ºC (lit.[8] 189–190 ºC); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25 (br s, 1H), δ 7.81 (d, 1H, *J* = 7.45 Hz), δ 7.77 (d, 1H, *J* = 8 Hz), δ 7.28–7.25 (m, 1H); ^{13}C NMR (125 MHz, $(CD_3)_2CO$) δ 182.32, δ 158.80, δ 147.48, δ 133.89 (q, *J* = 3.6), δ 128.33, δ 123.37 (q, *J* = 270.7 Hz), δ 123.12, δ 119.92, δ 113.46 (q, *J* = 32.2 Hz); HRMS calcd for $C_9H_4NO_2F_3$ [M-H]- 214.0121, found 214.0123.



2h

3-(2-aminophenyl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2h):

3-methylanthranilic acid (151.17 mg, 1.01 mmol, 1.0 equiv) was dissolved in 10 mL CH_2Cl_2. HBTU (457.8 mg, 1.207 mmol, 1.2 equiv), sulfonium salt **1** (561.2 mg, 3.082 mmol, 3.1 equiv), and DIPEA (0.52 mL, 3.0 mmol, 3.0 equiv) were added, in that respective order. The product was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2h** was used in the next step without further purification. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63–7.61 (d, 1H, *J* = 7.5 Hz), δ 7.10–7.09 (d, 1H, *J* = 7.5 Hz), δ 6.64–6.61 (t, 1H, *J* = 8.2 Hz), δ 2.84 (s, 6H); HRMS calcd for $C_{12}H_{14}N_2OS$ [M+H]+ 235.0900, found 235.0902.



3h

7-methyl-1*H*-indole-2,3-dione (3h):

The sulfur ylide **2h** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (924.1 mg, 1.502 mmol, 1.5 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a separatory funnel containing 20 mL CH_2Cl_2 and 20 mL H_2O, extracted three times with 20 mL CH_2Cl_2. The organic layer was washed with 20 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude material was purified by silica gel column chromatography (3:1 hexanes:EtOAc) to yield 7-methyl-1*H*-indole-2,3-dione **3h** (125.8 mg, 84%). The two-step sequence was reproduced on the same scale to yield **3h** in 92% yield.

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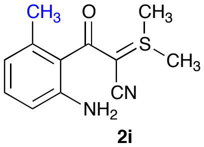
[8] P. M. Maginnity, C. A. Gaulin, *J. Am .Chem. Soc.* 1951, **73**, 3579.

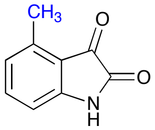
mp 260–263 ºC (lit.[3] mp 267–269 ºC); ^{1}H NMR (500 MHz, $(CD_3)_2CO$) δ 7.99 (br s, 1H), δ 7.38 (d, 1H, *J* = 7.5 Hz), δ 7.04 (t, 1H, *J* = 7.5 Hz), δ 2.26 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 184.27, δ 159.48, δ 149.27, δ 139.52, δ 122.91, δ 122.00, δ 121.80, δ 118.02, 14.85; HRMS calcd for $C_9H_7NO_2$ [M+H]+ 162.0550, found 162.0547.



2i

3-(2-amino-6-methylphenyl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2i):

3-(2-amino-6-methylphenyl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (154.9 mg, 0.9612 mmol, 1.0 equiv) was dissolved in 10 mL CH_2Cl_2. HBTU (478.2 mg, 1.2 mmol, 1.2 equiv), sulfonium salt **1** (576.1 mg, 3.0 mmol, 3.0 equiv), and DIPEA (0.52 mL, 3.0 mmol, 3.1 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a mixture of 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2i** was used in the next step without further purification. HRMS calcd for $C_{12}H_{14}N_2OS$ [M+H]+ 235.0900, found 235.0898.

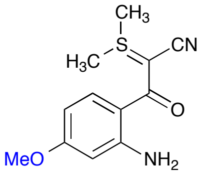


3i

4-methyl-1*H*-indole-2,3-dione (3i):

The crude sulfur ylide **2i** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (970.2 mg, 1.577 mmol, 1.6 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL H_2O, extracted three times with 10 mL of CH_2Cl_2, washed with 20 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. Purification of the crude product was accomplished by silica gel column chromatography (2:1 Hexane:EtOAc) to yield 4-methyl-1*H*-indole-2,3-dione **3i** (72.6 mg, 45%). The two-step sequence was reproduced on the same scale to yield **3i** in 53% yield. mp 188–191 ºC (lit.[7] 189 ºC); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.51 (s, 1H), δ 7.40–7.38 (d, 1H, *J* = 8.1), δ 6.89–6.87 (d, 1H, *J* = 8.1), δ 6.73–6.72 (d, 1H, *J* = 8.1), δ 2.56 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 183.58, δ 159.51, δ 149.22, δ 141.78, δ 138.07, δ 126.31, δ 116.46, δ 109.73, δ 18.23; HRMS calcd for $C_9H_7NO_2$

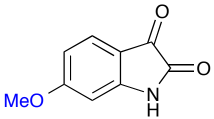
[M+H]+ 162.0550, found 162.0552.



2j

3-(2-amino-4-methoxyphenyl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2j):

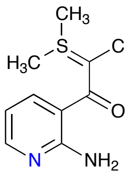
4-methoxyanthranilic acid (167.5 mg, 1.002 mmol, 1.0 equiv) was dissolved in 10 mL CH_2Cl_2. HBTU (452.8 mg, 1.194 mmol, 1.2 equiv), sulfonium salt **1** (452.6 mg, 2.486 mmol, 2.5 equiv), and DIPEA (0.52 mL, 3.0 mmol, 3.0 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL of CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2j** was used in the next step without further purification. HRMS calcd for $C_{12}H_{14}N_2O_2S$ [M+H]+ 251.0849, found 251.0849.



3j

6-methoxy-1*H*-indole-2,3-dione (3j):

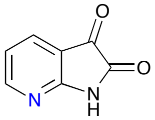
The sulfur ylide **2j** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (920.3 mg, 1.496 mmol, 1.5 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a mixture of 20 mL CH_2Cl_2 and 20 mL H_2O, extracted three times with 10 mL CH_2Cl_2. The organic layer was washed with 20 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. Purification of the crude product was accomplished by silica gel column chromatography (1:1 hexanes:EtOAc) to yield 6-methoxy-1*H*-indole-2,3-dione **3j** (129.5 mg, 73%). The two-step sequence was reproduced on the same scale to yield **3j** in 67% yield. mp 228–230 ºC (lit.[7] 230 ºC); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (br s, 1H), δ 7.58 (d, 1H, J = 8.1 Hz), δ 6.57 (dd, H, J = 2.5, 8.5 Hz), δ 6.40 (d, 1H, J = 2.3 Hz), δ 3.91 (s, 3H); ^{13}C NMR (125 MHz, $(CD_3)_2CO$) δ 181.85, δ 169.10, δ 161.00, δ 154.10, δ 127.85, δ 112.55, δ 109.70, δ 98.60, δ 56.46; HRMS calcd for $C_9H_7NO_3$ [M+Na]+ 200.0318, found 200.0312.



2k

3-(2-aminopyridin-3-yl)-2-(dimethyl-l^4-sulfanylidene)-3-oxopropanenitrile (2k):

2-aminopyridine-3-carboxylic acid (139.8 mg, 1.066 mmol, 1.0 equiv) was dissolved in 10 mL CH_2Cl_2. HBTU (456.4 mg, 1.20 mmol, 1.1 equiv), sulfonium salt **1** (464.4 mg, 2.551 mmol, 2.4 equiv), and DIPEA (0.52 mL, 3.0 mmol, 2.8 equiv) were added, in that respective order. The solution was stirred vigorously for 75 minutes. The reaction mixture was poured into a separatory funnel containing 10 mL CH_2Cl_2 and 10 mL 1M K_2CO_3, extracted four times with 10 mL CH_2Cl_2, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. The crude sulfur ylide **2k** was used in the next step without further purification. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99–7.98 (dd, 1H, *J* = 2, 5 Hz), δ 7.96–7.94 (d, 1H, *J* = 8 Hz), δ 6.52–6.51 (t, 1H, *J* = 4.6 Hz), δ 6.07 (br s, 2H), δ 2.75 (s, 6H); HRMS calcd for $C_{10}H_{11}N_3OS$ [M+H]+ 222.0696, found 222.0691.



3k

1H-pyrrolo[2,3-b]pyridine-2,3-dione (3k):

The sulfur ylide **2k** was dissolved in 2:1 THF:H_2O (12.8 mL:6.4 mL) before Oxone (933.8 mg, 1.518 mmol, 1.4 equiv) was added. The stirring reaction was allowed to proceed for 10 minutes. The reaction mixture was then poured into a mixture of 10 mL CH_2Cl_2 and 10 mL H_2O, extracted three times with 10 mL CH_2Cl_2, washed with 10 mL brine, dried over sodium sulfate, filtered, washed with extra CH_2Cl_2, and concentrated. Purification of the crude product was done by a silica gel column chromatography (1:3 Hexane:EtOAc) to yield 1*H*-pyrrolo[2,3-b]pyridine-2,3-dione **3k** (82.2 mg, 55%). The two-step sequence was reproduced on the same scale to yield **3k** in 53% yield. mp 168–172 ºC (lit.[9] 168–170 ºC); ^{1}H NMR (500 MHz, CD_3OD) δ 8.63 (dd, 1H, *J* = 1.7, 4.6 Hz), δ 8.36 (dd, 1H, *J* = 1.8, 8.0 Hz), δ 7.29 (dd, 1H, *J* = 4.6, 8.0 Hz); ^{13}C NMR (125 MHz, $(CD_3)_2CO$) δ 185.58, δ 164.93, δ 160.01, δ 156.65, δ 133.34, δ 120.16, δ 113.69; HRMS calcd for $C_7H_4N_2O_2$ [M–H]– 147.0200, found 147.0201.

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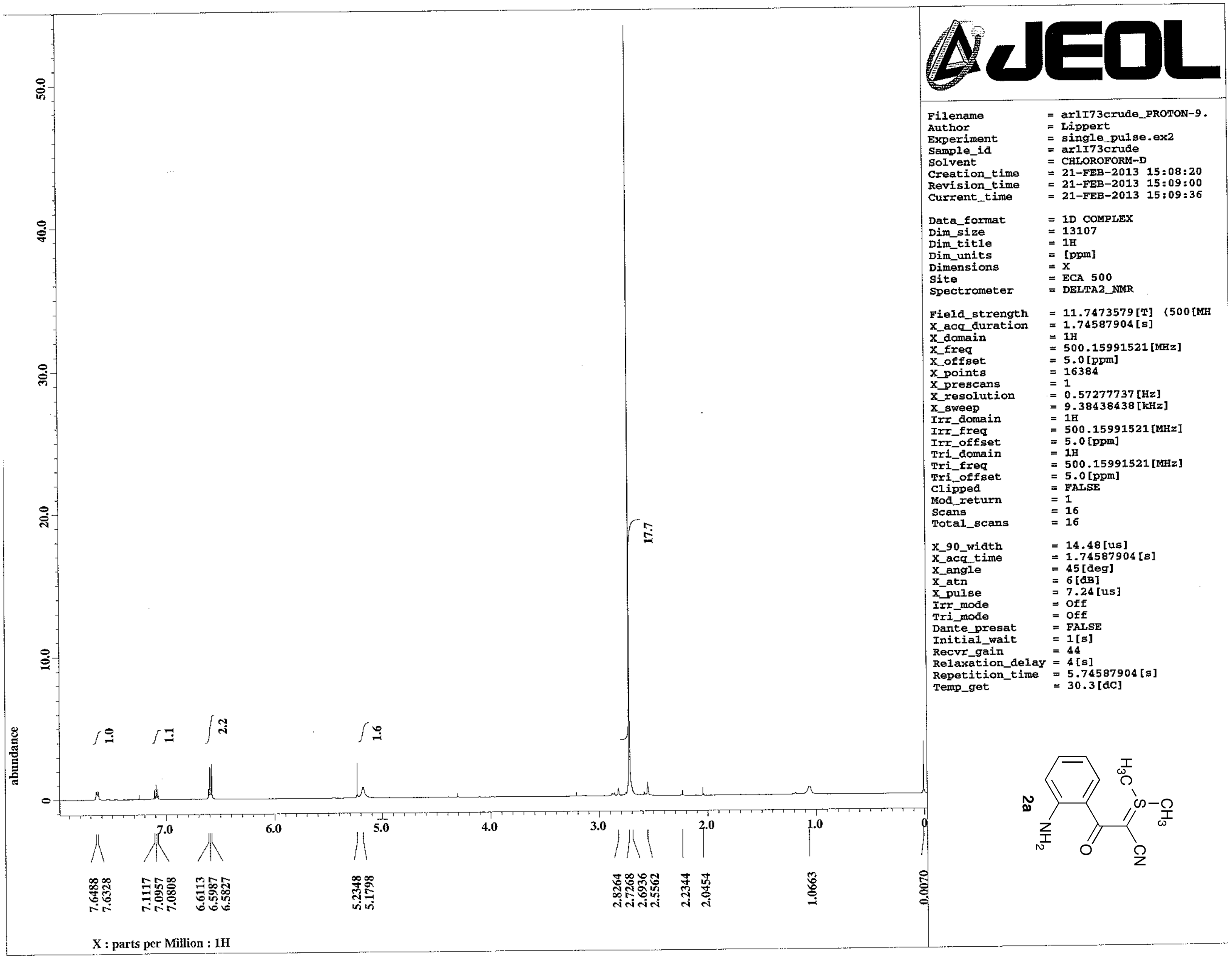
[9] R. Sriram, C. N. S. S. P. Kumar, N. Raghunandan, V. Ramesh, M. Sarangapani, V. J. Rao, *Synth. Commun.* 1993, **42**, 3419.



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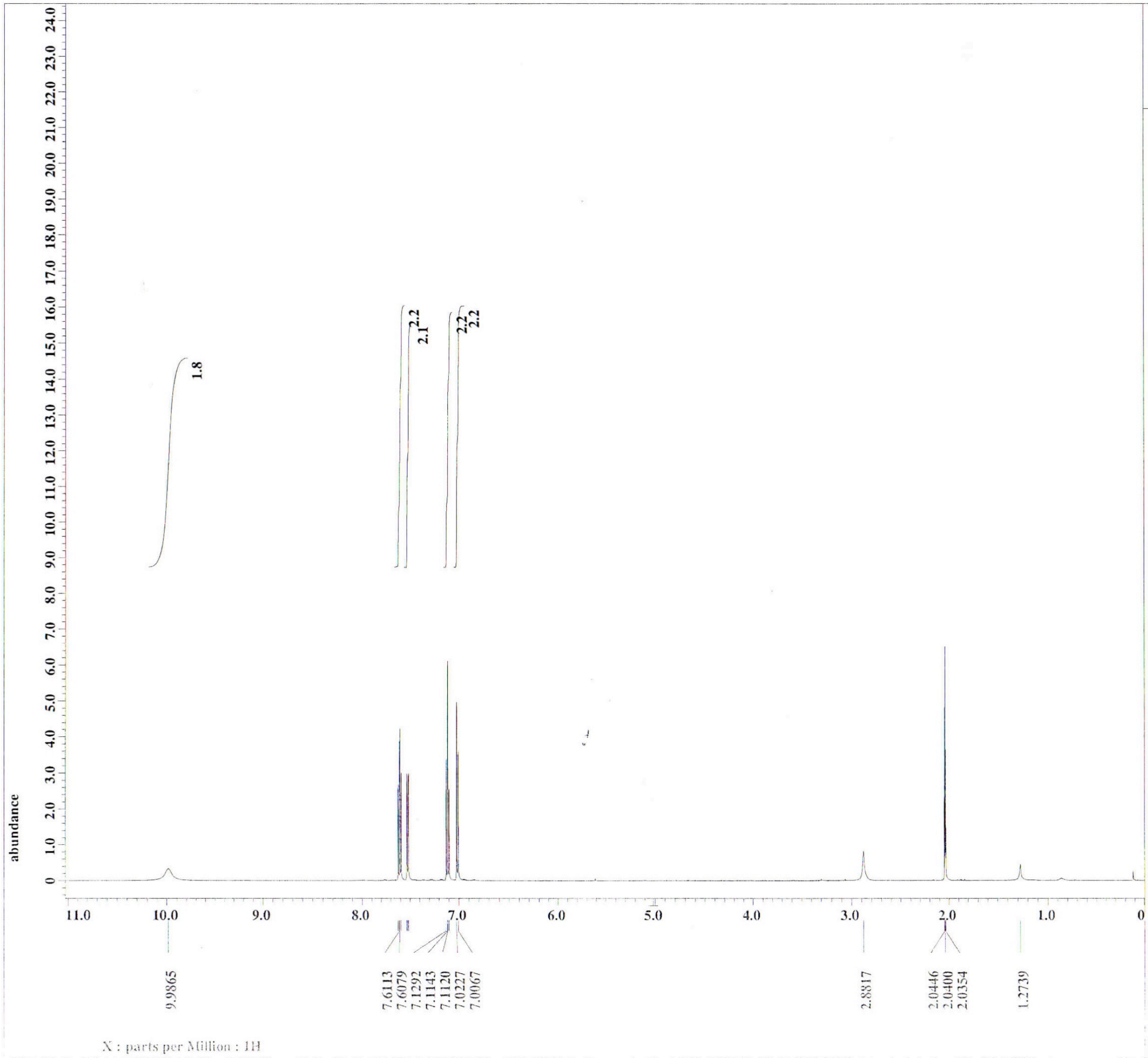

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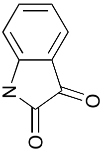
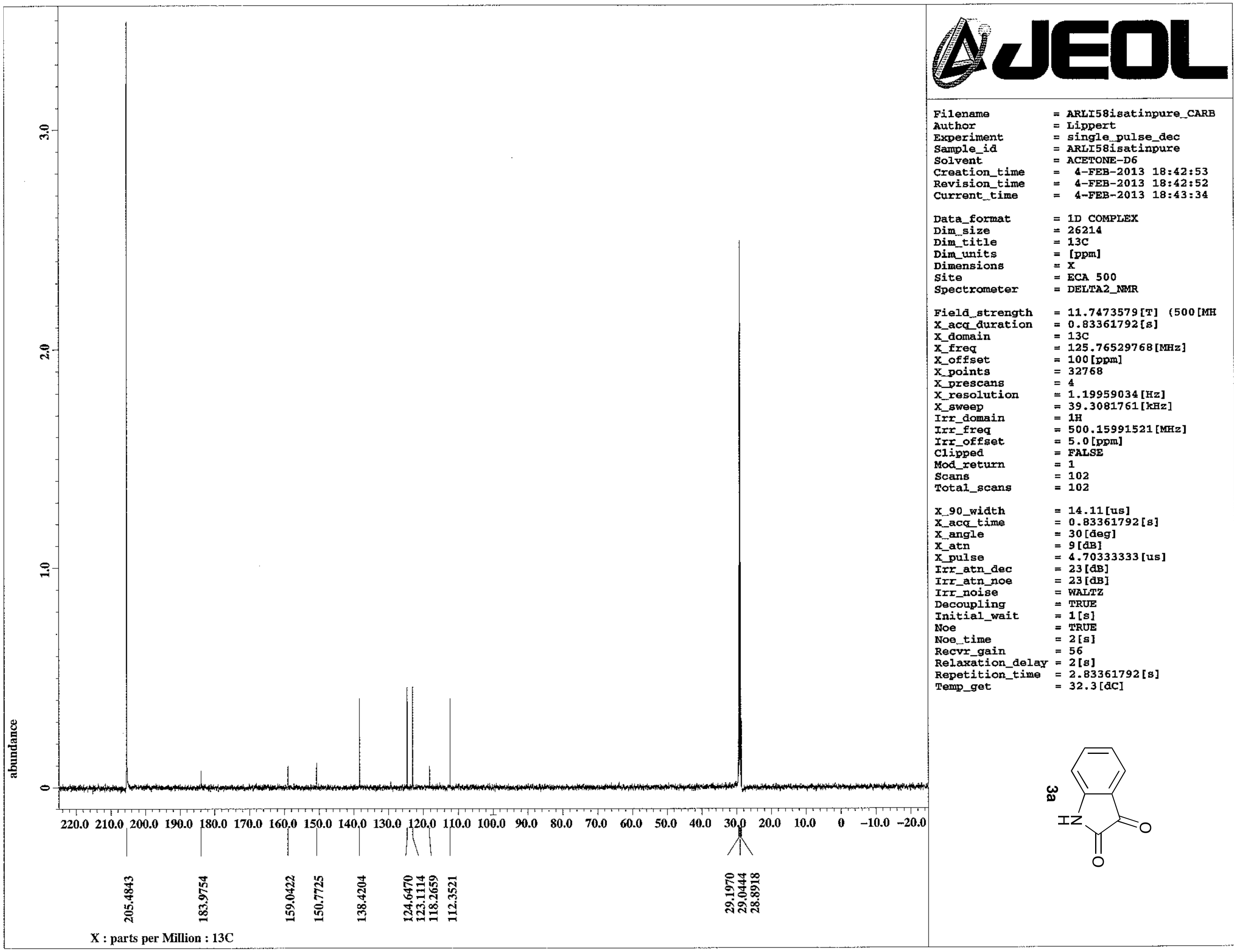


3a



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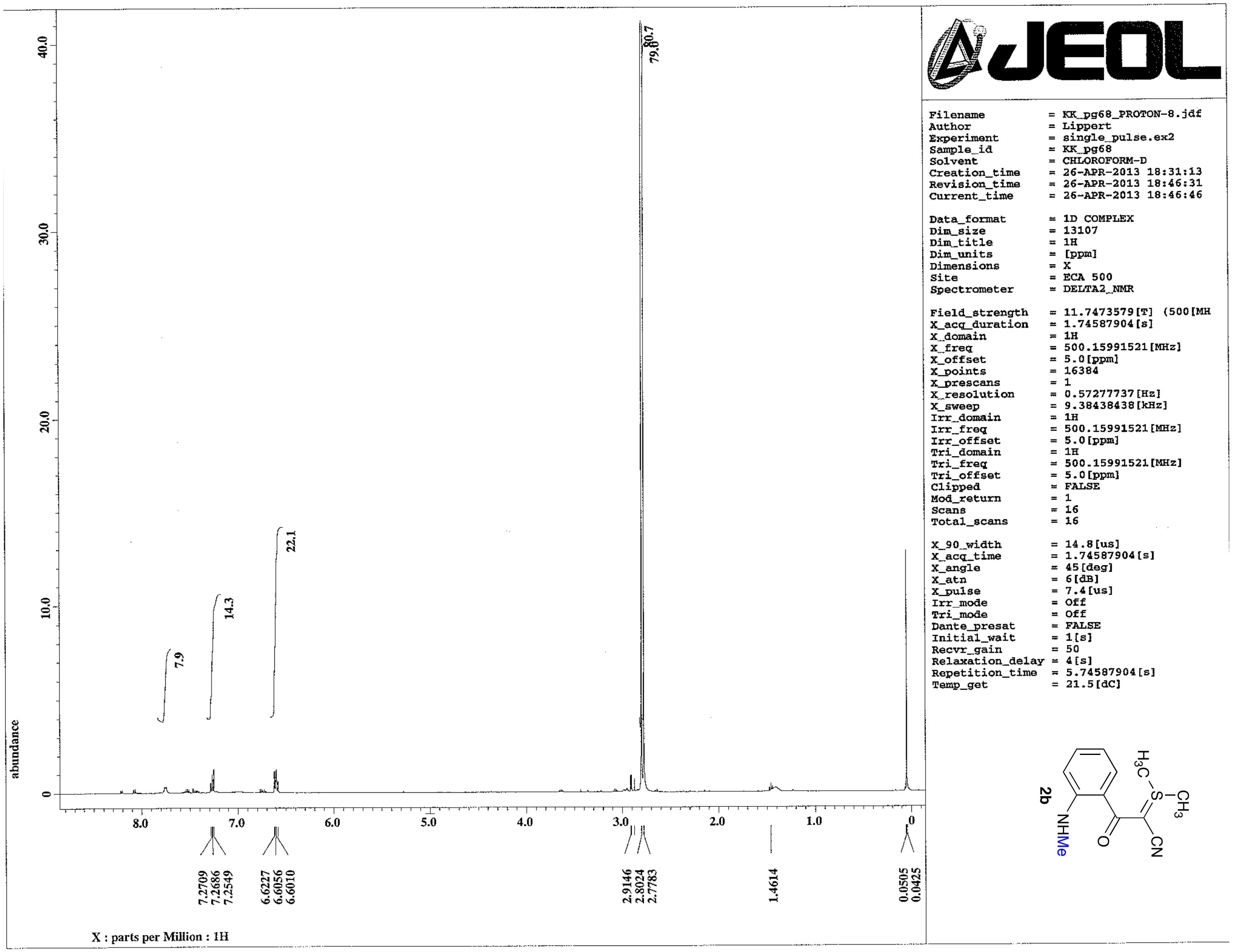

2b

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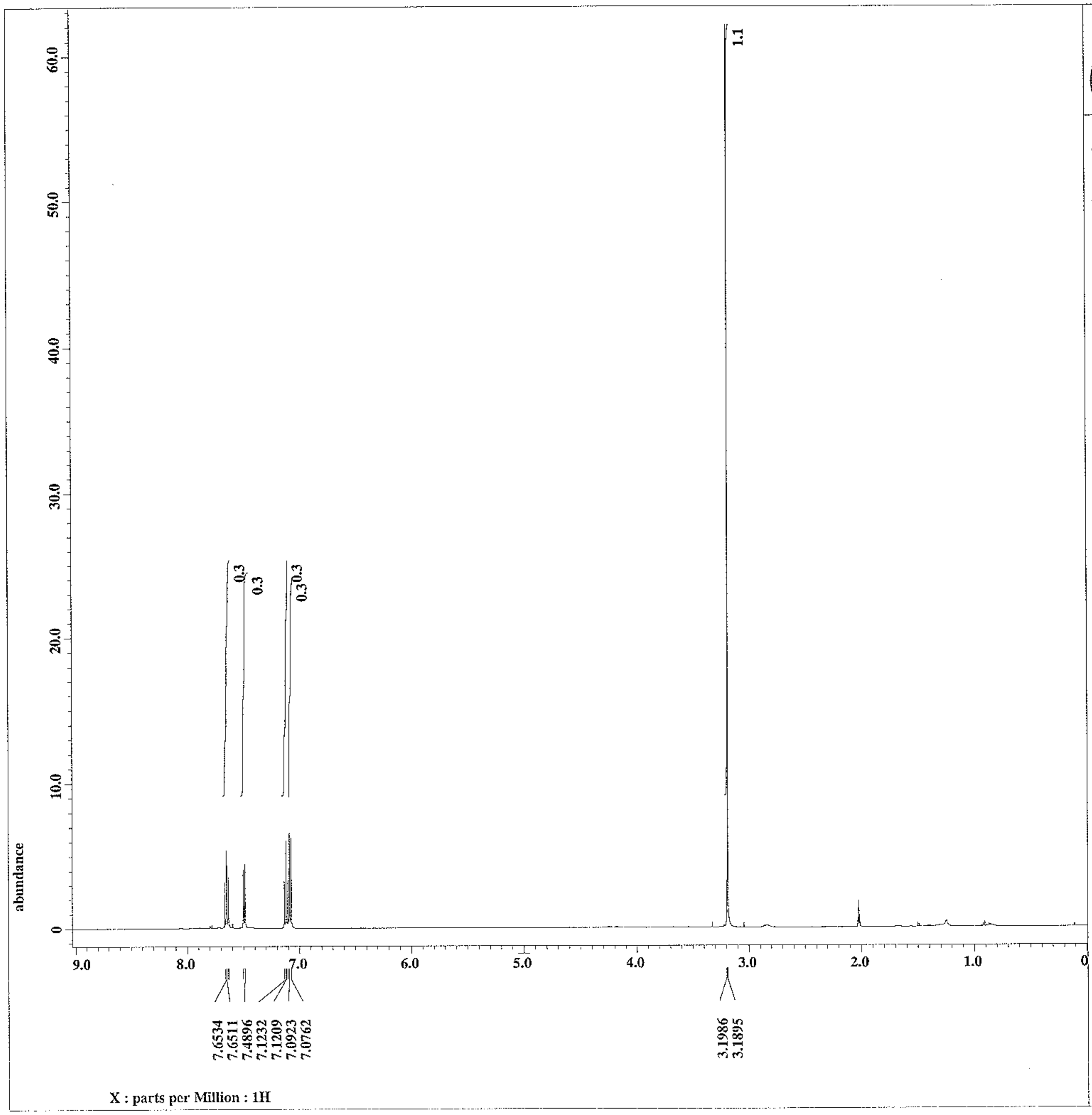

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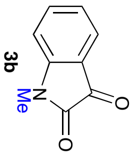
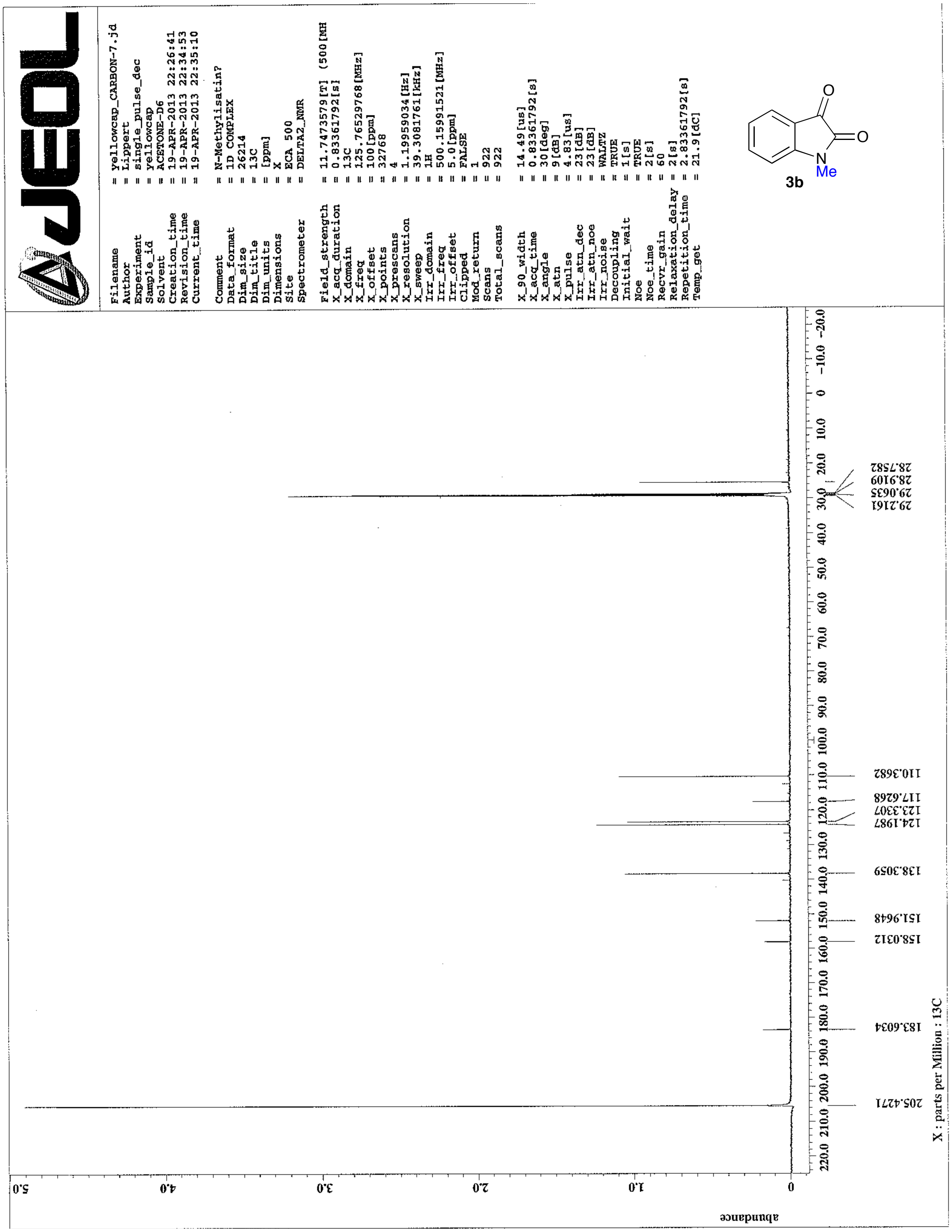
Temp_get            = 21.3[dC]
```



3b

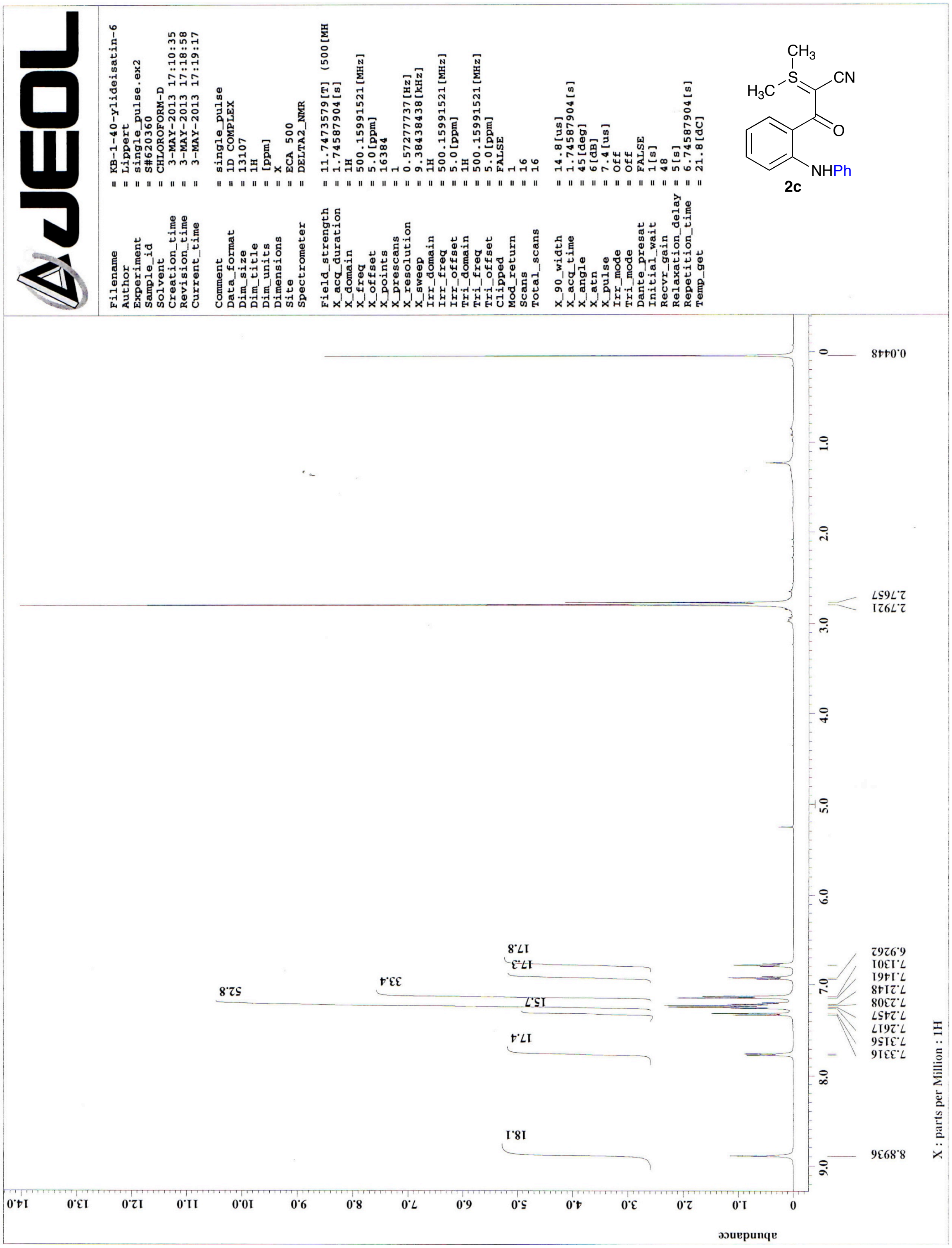


```
Filename          = yellowcap_CARBON-7.jd
Author            = Lippert
Experiment        = single_pulse_dec
Sample_id         = yellowcap
Solvent           = ACETONE-D6
Creation_time     = 19-APR-2013  22:26:41
Revision_time     = 19-APR-2013  22:34:53
Current_time      = 19-APR-2013  22:35:10

Comment           = N-Methylisatin?
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
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             = 922
Total_scans       = 922

X_90_width        = 14.49[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 4.83[us]
Irr_atn_dec       = 23[dB]
Irr_atn_noe       = 23[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          = 21.9[dC]
```



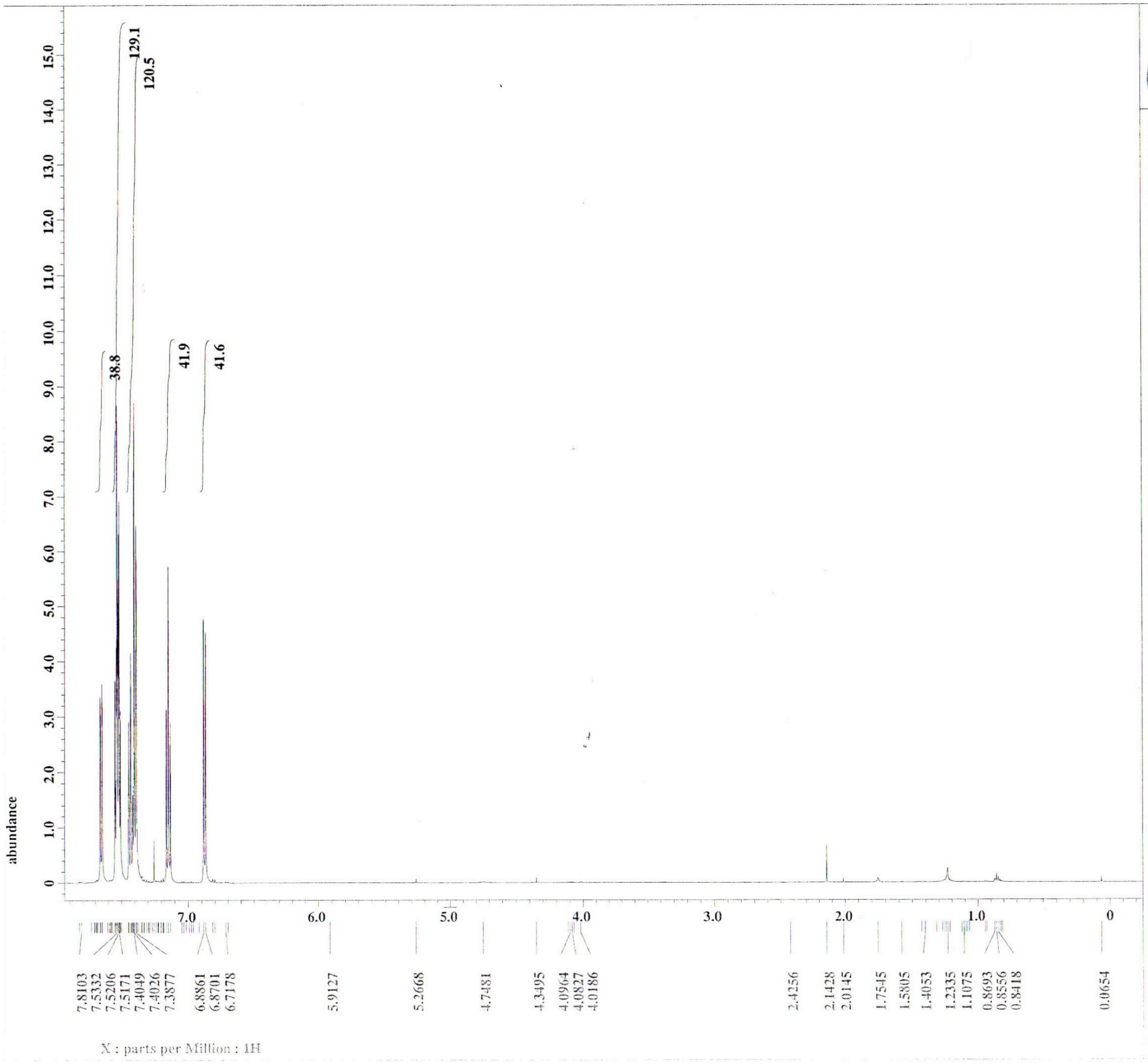
```
Filename             = KB-1-40-ylideisatin-6
Author               = Lippert
Experiment           = single_pulse.ex2
Sample_id            = S#620360
Solvent              = CHLOROFORM-D
Creation_time        =   3-MAY-2013 17:10:35
Revision_time        =   3-MAY-2013 17:18:58
Current_time         =   3-MAY-2013 17:19:17

Comment              = 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       = 11.7473579[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                = 16
Total_scans          = 16

X_90_width           = 14.8[us]
X_acq_time           = 1.74587904[s]
X_angle              = 45[deg]
X_atn                = 6[dB]
X_pulse              = 7.4[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             = 21.8[dC]
```

2c

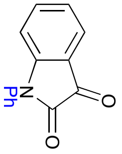


```
Filename            = KB-69-aftercolumn_PRO
Author              = Lippert
Experiment          = single_pulse.ex2
Sample_id           = KB-69-aftercolumn
Solvent             = CHLOROFORM-D
Creation_time       =  6-NOV-2013 20:52:05
Revision_time       =  6-NOV-2013 21:02:45
Current_time        =  6-NOV-2013 21:02:47

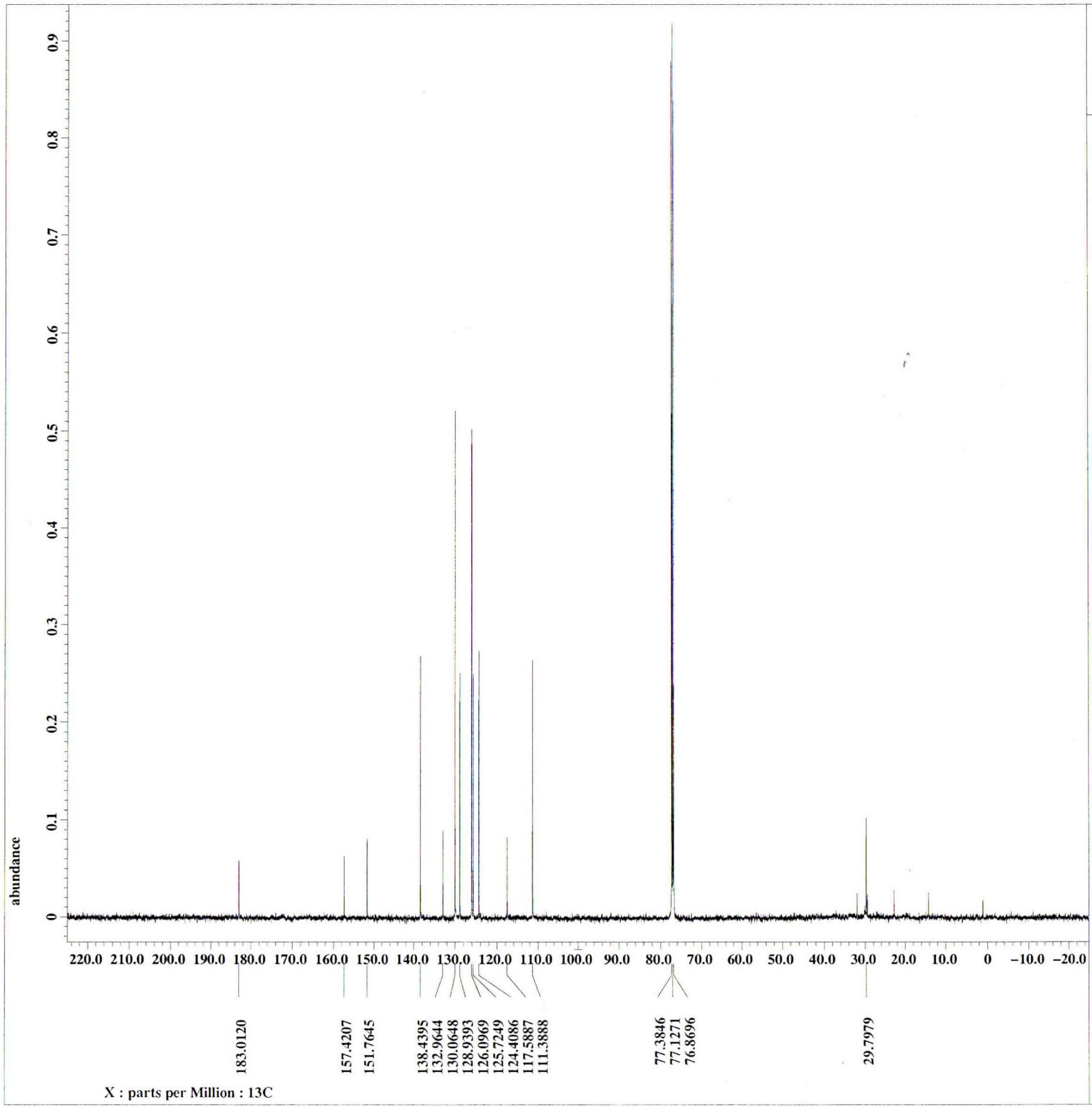
Data_format         = 1D COMPLEX
Dim_size            = 13107
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA 500
Spectrometer        = DELTA2_NMR

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               = 16
Total_scans         = 16

X_90_width          = 14.8[us]
X_acq_time          = 1.74587904[s]
X_angle             = 45[deg]
X_atn               = 6[dB]
X_pulse             = 7.4[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 48
Relaxation_delay    = 4[s]
Repetition_time     = 5.74587904[s]
Temp_get            = 20.6[dC]
```



3c

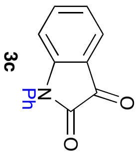


```
Filename           = N-Phenylisatin-13C_CA
Author             = Lippert
Experiment         = single_pulse_dec
Sample_id          = N-Phenylisatin-13C
Solvent            = CHLOROFORM-D
Creation_time      =  6-MAY-2013 19:39:46
Revision_time      =  6-MAY-2013 19:48:43
Current_time       =  6-MAY-2013 19:48:54

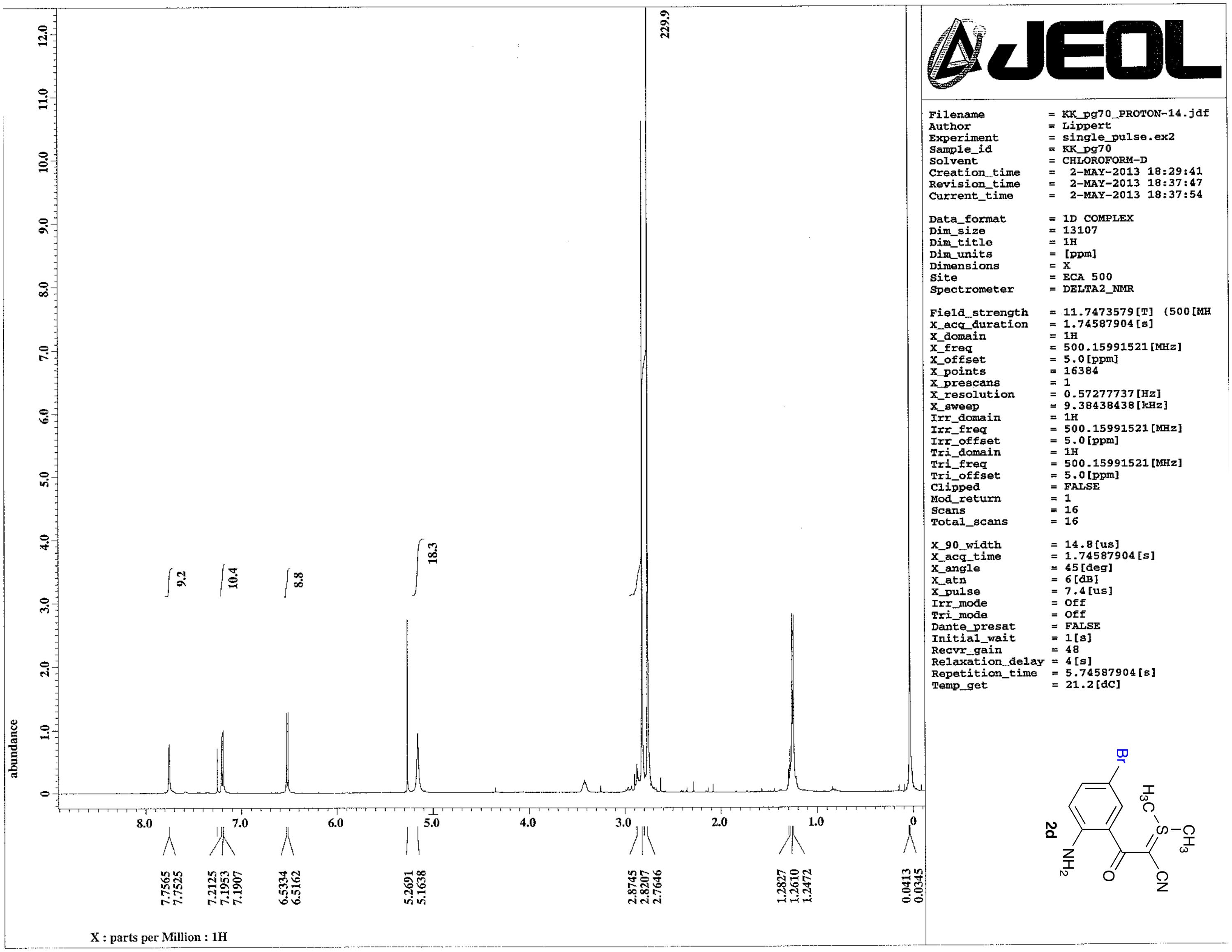
Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
Spectrometer       = DELTA2_NMR

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              = 2048
Total_scans        = 2048

X_90_width         = 14.49[us]
X_acq_time         = 0.83361792[s]
X_angle            = 30[deg]
X_atn              = 9[dB]
X_pulse            = 4.83[us]
Irr_atn_dec        = 23[dB]
Irr_atn_noe        = 23[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 58
Relaxation_delay   = 2[s]
Repetition_time    = 2.83361792[s]
Temp_get           = 21.6[dC]
```



3c



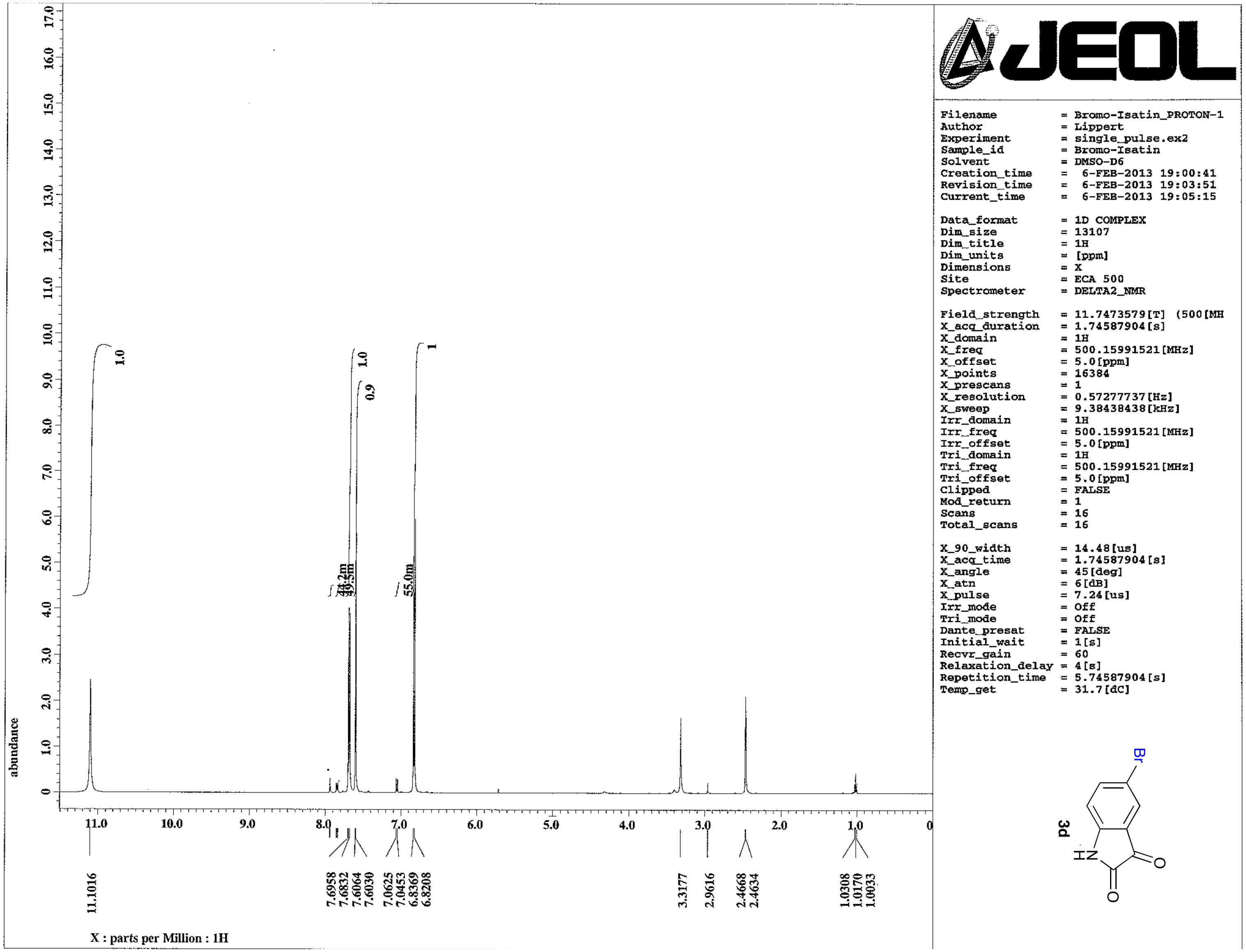
2d

Filename = KK_pg70_PROTON-14.jdf
Author = Lippert
Experiment = single_pulse.ex2
Sample_id = KK_pg70
Solvent = CHLOROFORM-D
Creation_time = 2-MAY-2013 18:29:41
Revision_time = 2-MAY-2013 18:37:47
Current_time = 2-MAY-2013 18:37:54

Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
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 = 16
Total_scans = 16

X_90_width = 14.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 7.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 4[s]
Repetition_time = 5.74587904[s]
Temp_get = 21.2[dC]



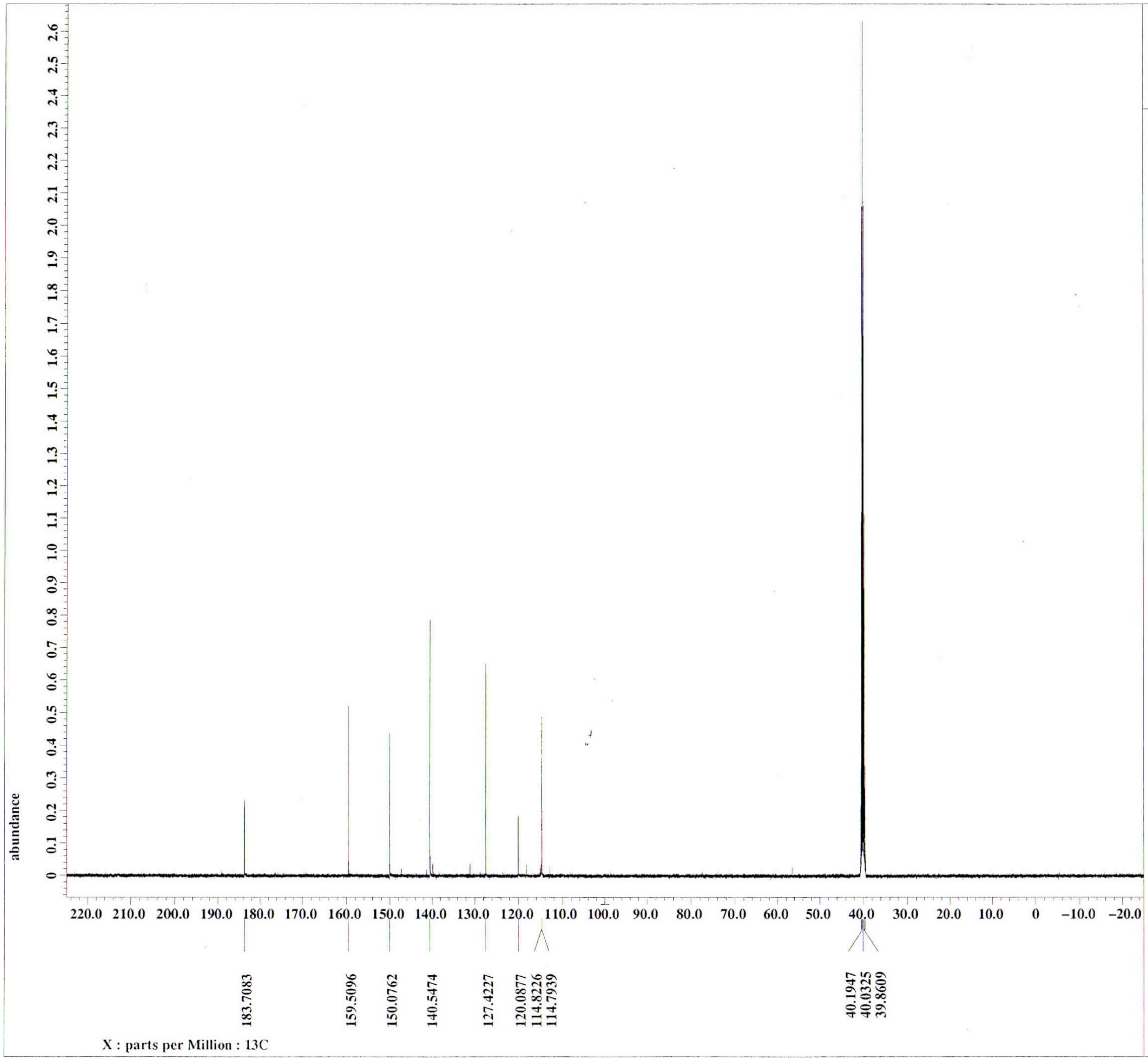
```
Filename           = Bromo-Isatin_PROTON-1
Author             = Lippert
Experiment         = single_pulse.ex2
Sample_id          = Bromo-Isatin
Solvent            = DMSO-D6
Creation_time      =  6-FEB-2013 19:00:41
Revision_time      =  6-FEB-2013 19:03:51
Current_time       =  6-FEB-2013 19:05:15

Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
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              = 16
Total_scans        = 16

X_90_width         = 14.48[us]
X_acq_time         = 1.74587904[s]
X_angle            = 45[deg]
X_atn              = 6[dB]
X_pulse            = 7.24[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 60
Relaxation_delay   = 4[s]
Repetition_time    = 5.74587904[s]
Temp_get           = 31.7[dC]
```

3d



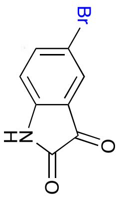
JEOL

```
Filename           = Bromo-Isatin_CARBON-7
Author             = Lippert
Experiment         = single_pulse_dec
Sample_id          = Bromo-Isatin
Solvent            = DMSO-D6
Creation_time      =  6-FEB-2013 20:39:36
Revision_time      = 24-JUN-2013 17:41:41
Current_time       = 24-JUN-2013 17:42:12

Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
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              = 2048
Total_scans        = 2048

X_90_width         = 14.11[us]
X_acq_time         = 0.83361792[s]
X_angle            = 30[deg]
X_atn              = 9[dB]
X_pulse            = 4.70333333[us]
Irr_atn_dec        = 23[dB]
Irr_atn_noe        = 23[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           = 32.4[dC]
```



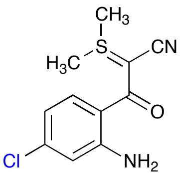
3d

```
Filename          = KK_pg71_PROTON-9.jdf
Author            = Lippert
Experiment        = single_pulse.ex2
Sample_id         = KK_pg71
Solvent           = CHLOROFORM-D
Creation_time     =  2-MAY-2013 18:21:57
Revision_time     =  2-MAY-2013 18:43:19
Current_time      =  2-MAY-2013 18:43:22

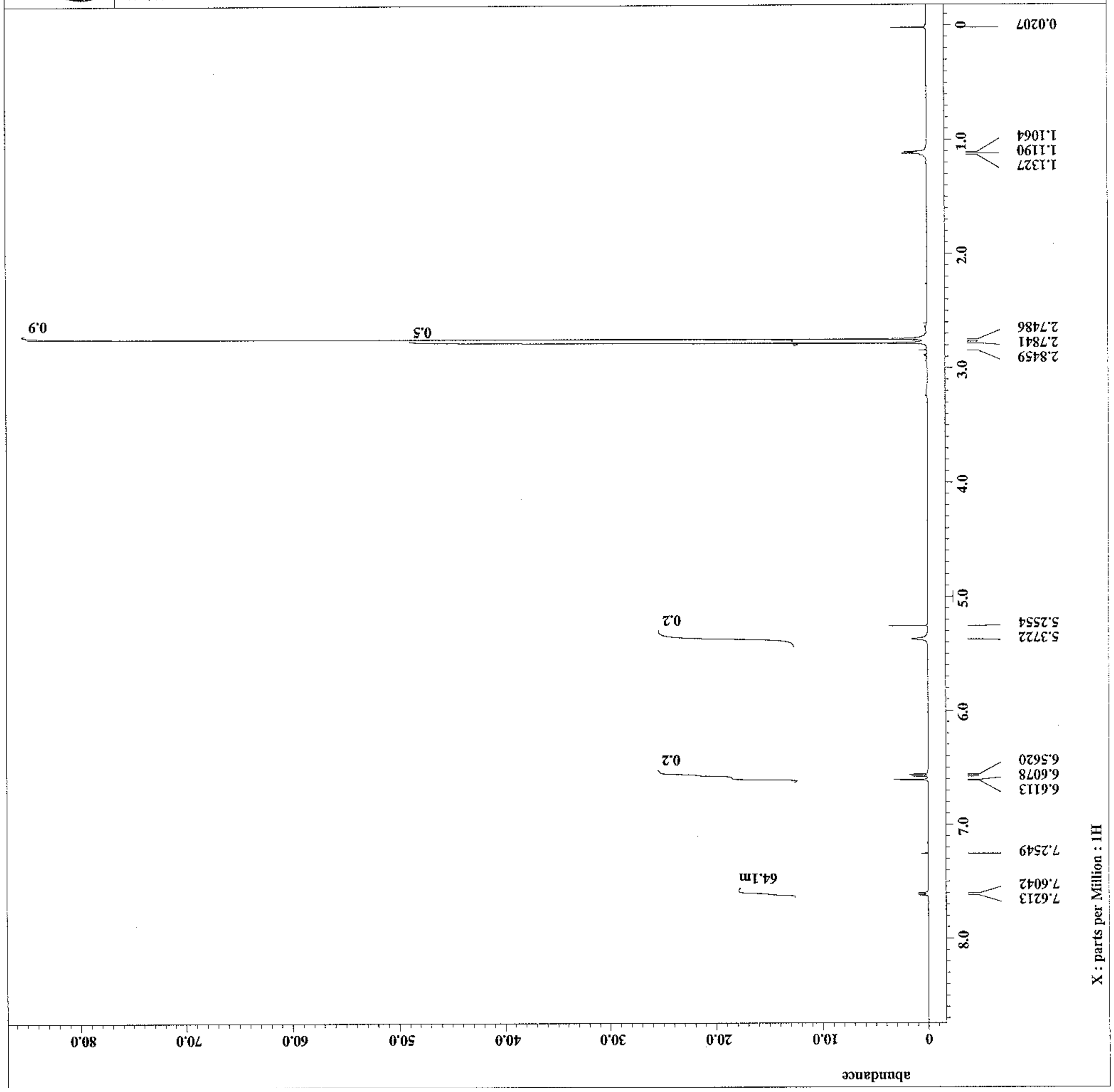
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

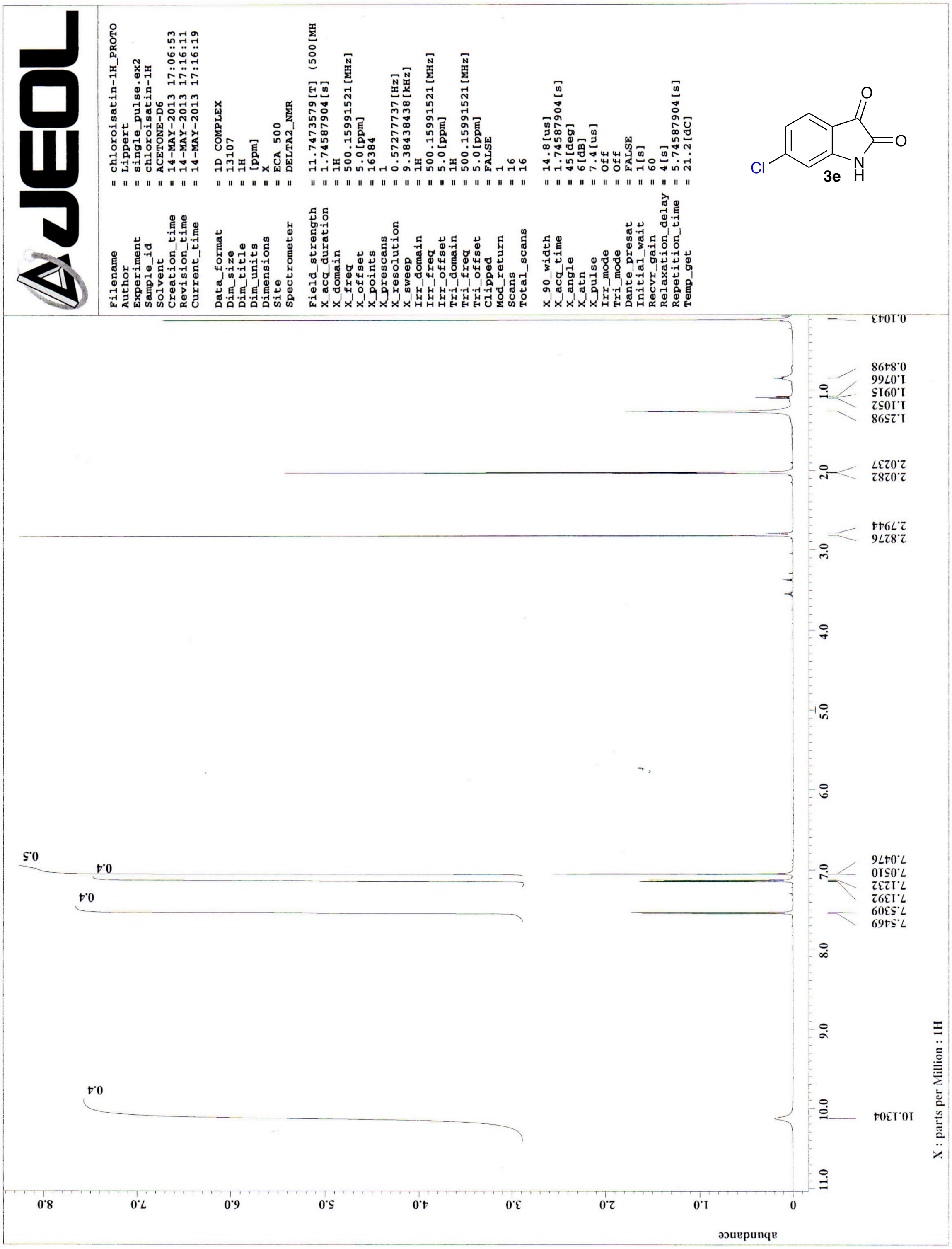
Field_strength    = 11.7473579[T]  (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 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             = 16
Total_scans       = 16

X_90_width        = 14.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 6[dB]
X_pulse           = 7.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 21.1[dC]
```



2e





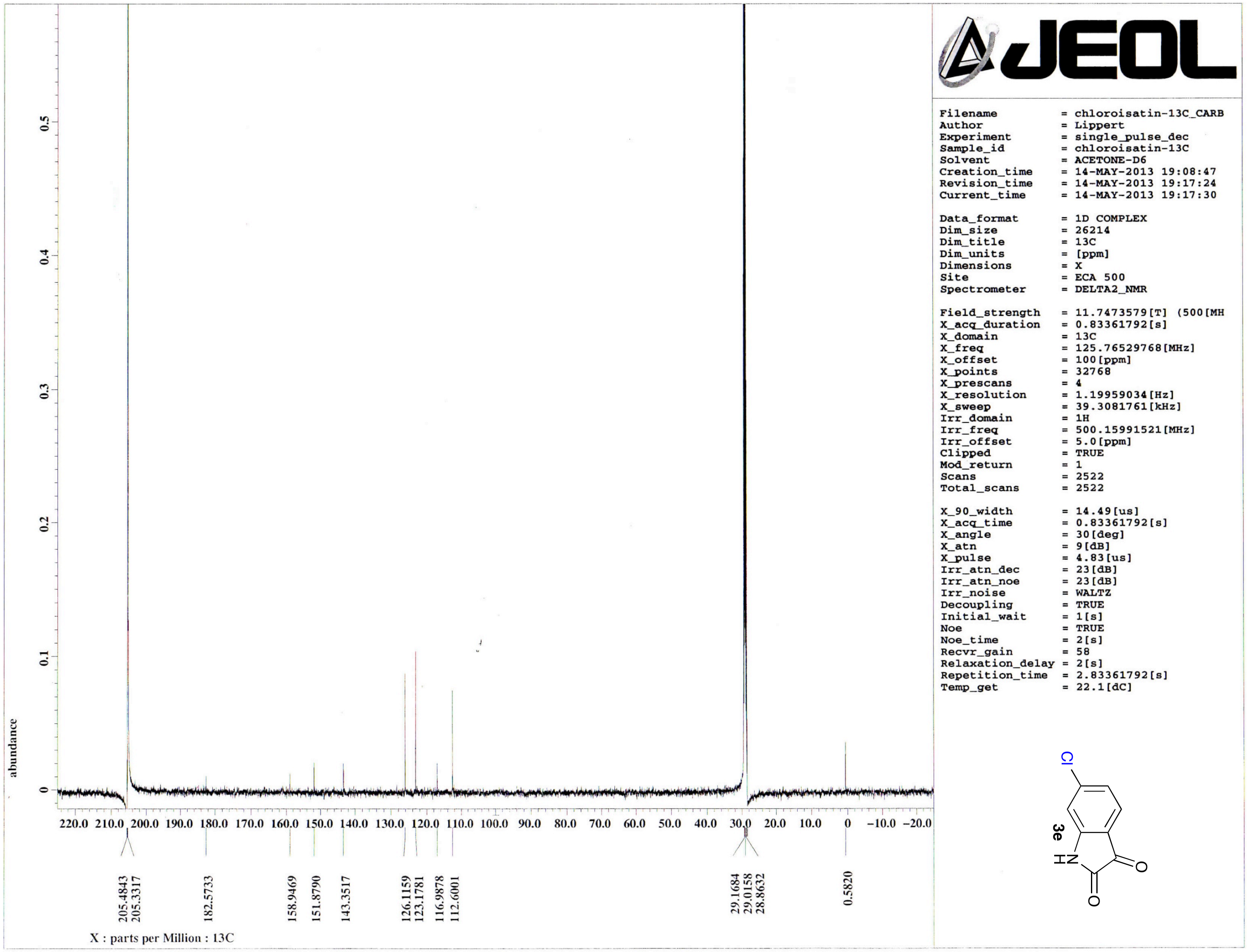
```
Filename            = chloroisatin-1H_PROTO
Author              = Lippert
Experiment          = single_pulse.ex2
Sample_id           = chloroisatin-1H
Solvent             = ACETONE-D6
Creation_time       = 14-MAY-2013 17:06:53
Revision_time       = 14-MAY-2013 17:16:11
Current_time        = 14-MAY-2013 17:16:19

Data_format         = 1D COMPLEX
Dim_size            = 13107
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA 500
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               = 16
Total_scans         = 16

X_90_width          = 14.8[us]
X_acq_time          = 1.74587904[s]
X_angle             = 45[deg]
X_atn               = 6[dB]
X_pulse             = 7.4[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 60
Relaxation_delay    = 4[s]
Repetition_time     = 5.74587904[s]
Temp_get            = 21.2[dC]
```

3e



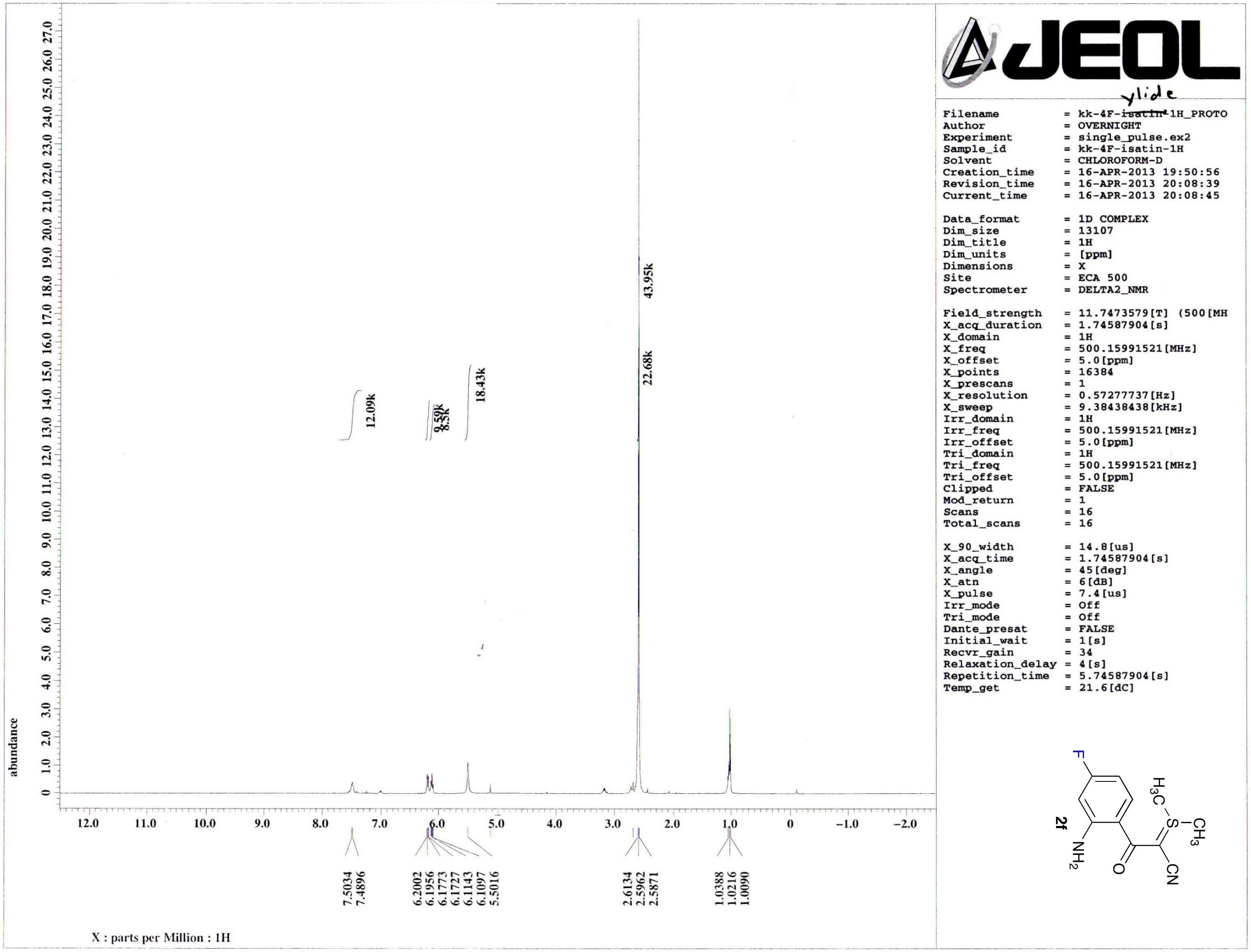
```
Filename          = chloroisatin-13C_CARB
Author            = Lippert
Experiment        = single_pulse_dec
Sample_id         = chloroisatin-13C
Solvent           = ACETONE-D6
Creation_time     = 14-MAY-2013 19:08:47
Revision_time     = 14-MAY-2013 19:17:24
Current_time      = 14-MAY-2013 19:17:30

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
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             = 2522
Total_scans       = 2522

X_90_width        = 14.49[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 4.83[us]
Irr_atn_dec       = 23[dB]
Irr_atn_noe       = 23[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 58
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 22.1[dC]
```

3e

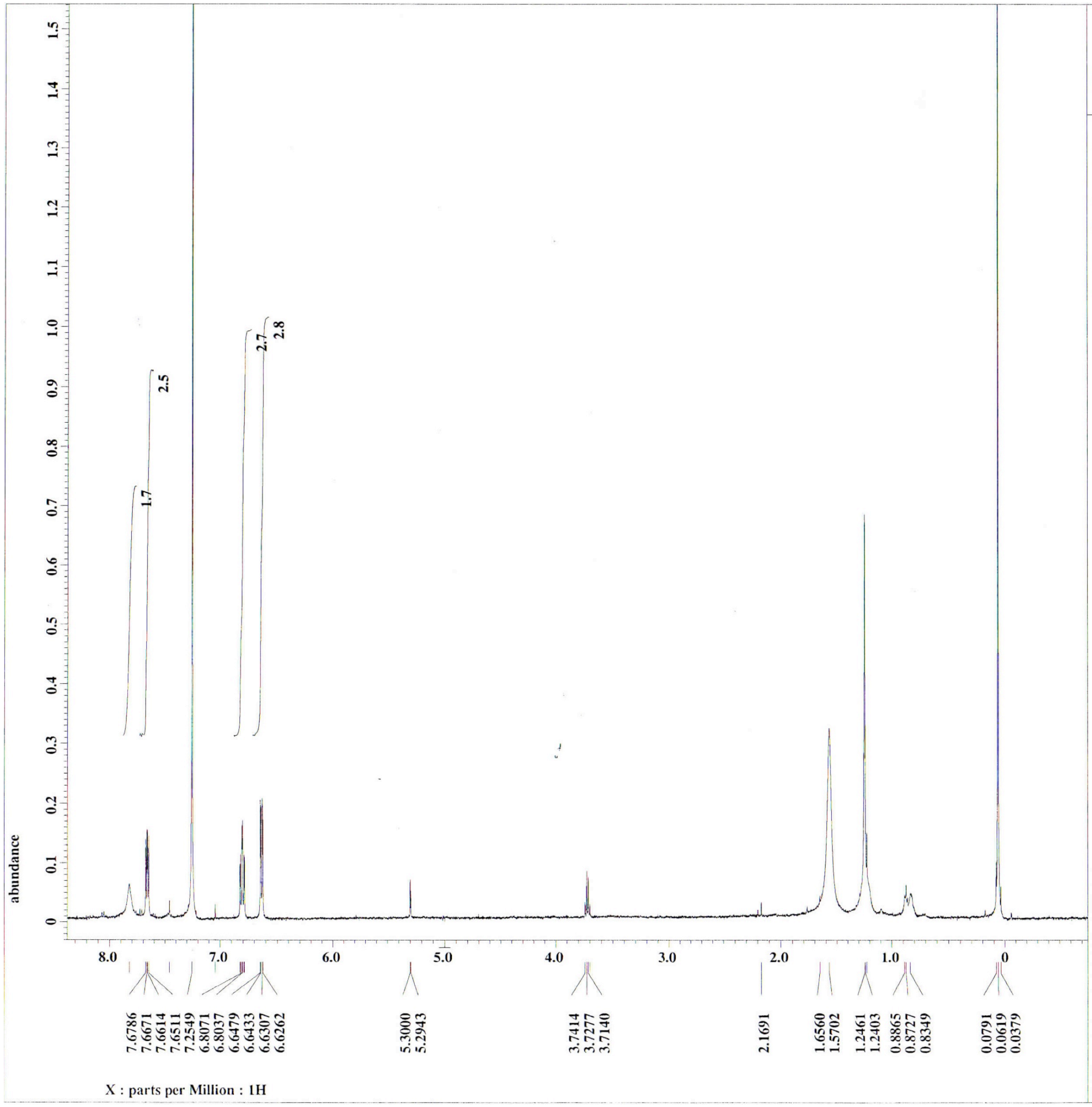


```
Filename          = kk-4F-~~isatin~~ ylide 1H_PROTO
Author            = OVERNIGHT
Experiment        = single_pulse.ex2
Sample_id         = kk-4F-isatin-1H
Solvent           = CHLOROFORM-D
Creation_time     = 16-APR-2013 19:50:56
Revision_time     = 16-APR-2013 20:08:39
Current_time      = 16-APR-2013 20:08:45

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
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             = 16
Total_scans       = 16

X_90_width        = 14.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 6[dB]
X_pulse           = 7.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 21.6[dC]
```

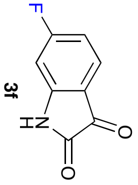


```
Filename          = KK-p45-4Fisatin-4.jdf
Author            = Lippert
Experiment        = single_pulse.ex2
Sample_id         = S#458951
Solvent           = CHLOROFORM-D
Creation_time     =  6-MAY-2013 12:41:38
Revision_time     =  6-MAY-2013 12:50:00
Current_time      =  6-MAY-2013 12:50:57

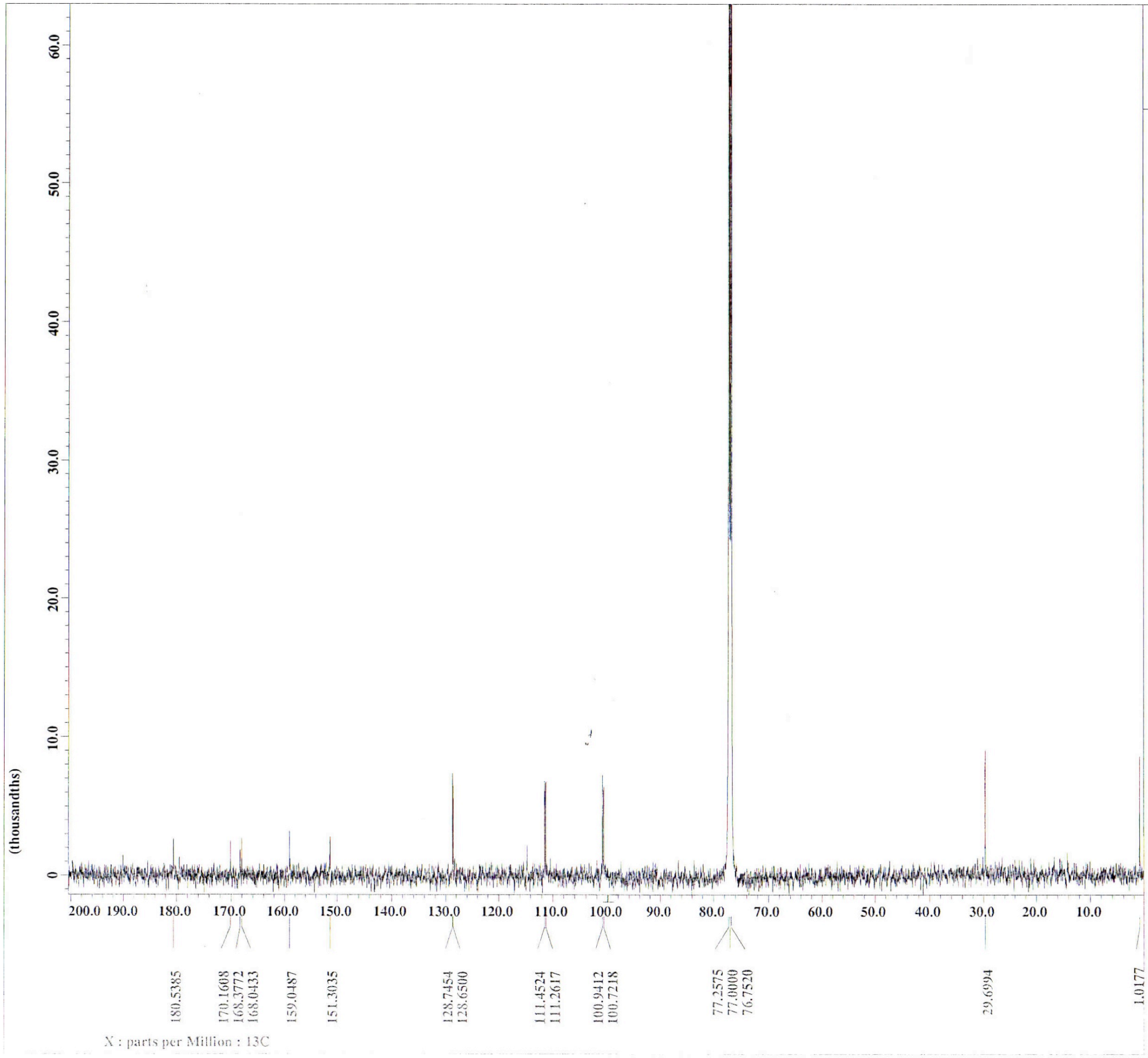
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16
Total_scans       = 16

X_90_width        = 14.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 6[dB]
X_pulse           = 7.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 62
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 21.1[dC]
```



3f



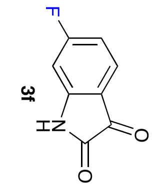
JEOL

```
Filename          = smh-13-c-4-fluoroisat
Author            = Lippert
Experiment        = single_pulse_dec
Sample_id         = smh-13-c-4-fluoroisat
Solvent           = CHLOROFORM-D
Creation_time     = 14-SEP-2013 10:19:11
Revision_time     =  4-NOV-2013 12:26:48
Current_time      =  4-NOV-2013 12:27:17

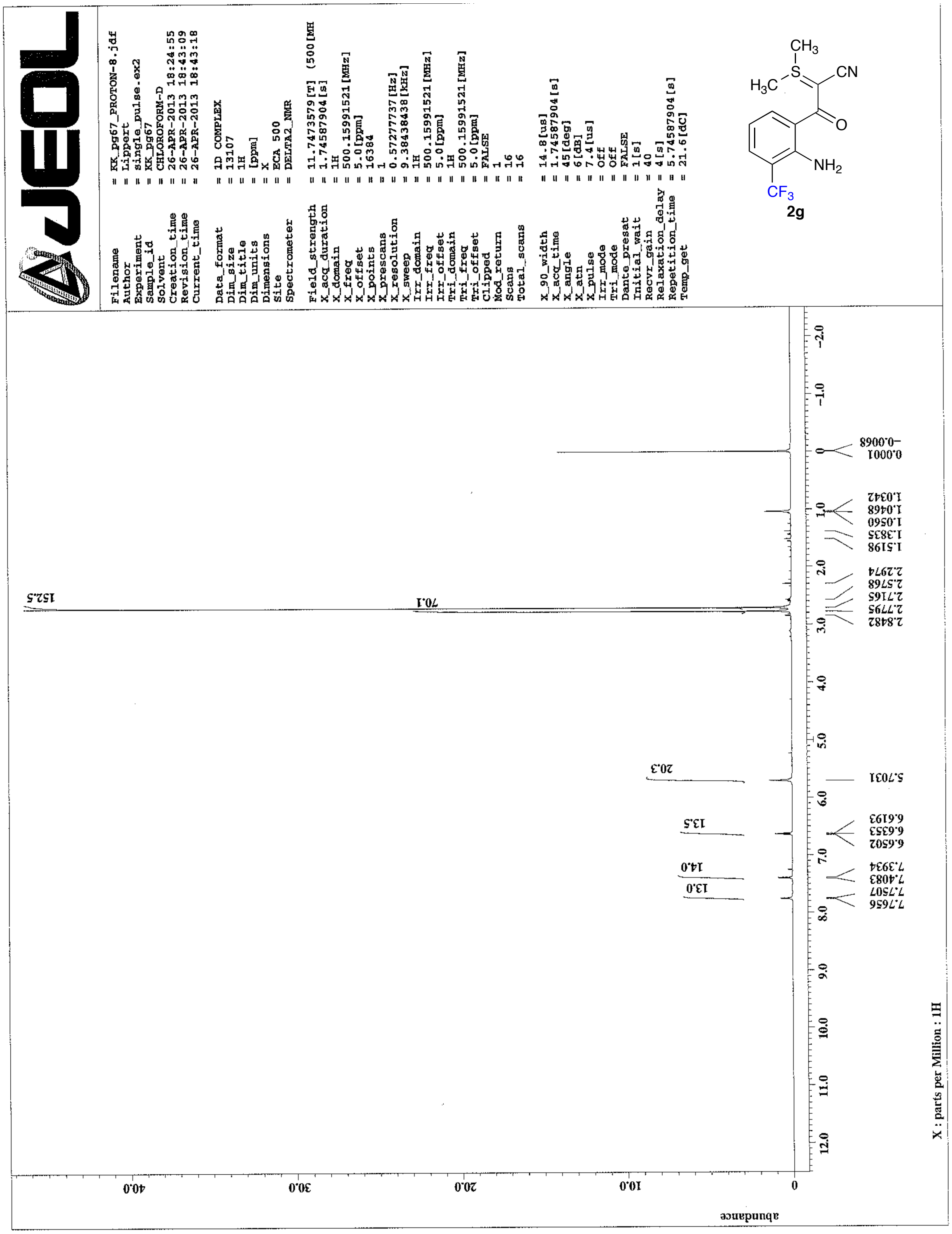
Comment           = ptlc
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

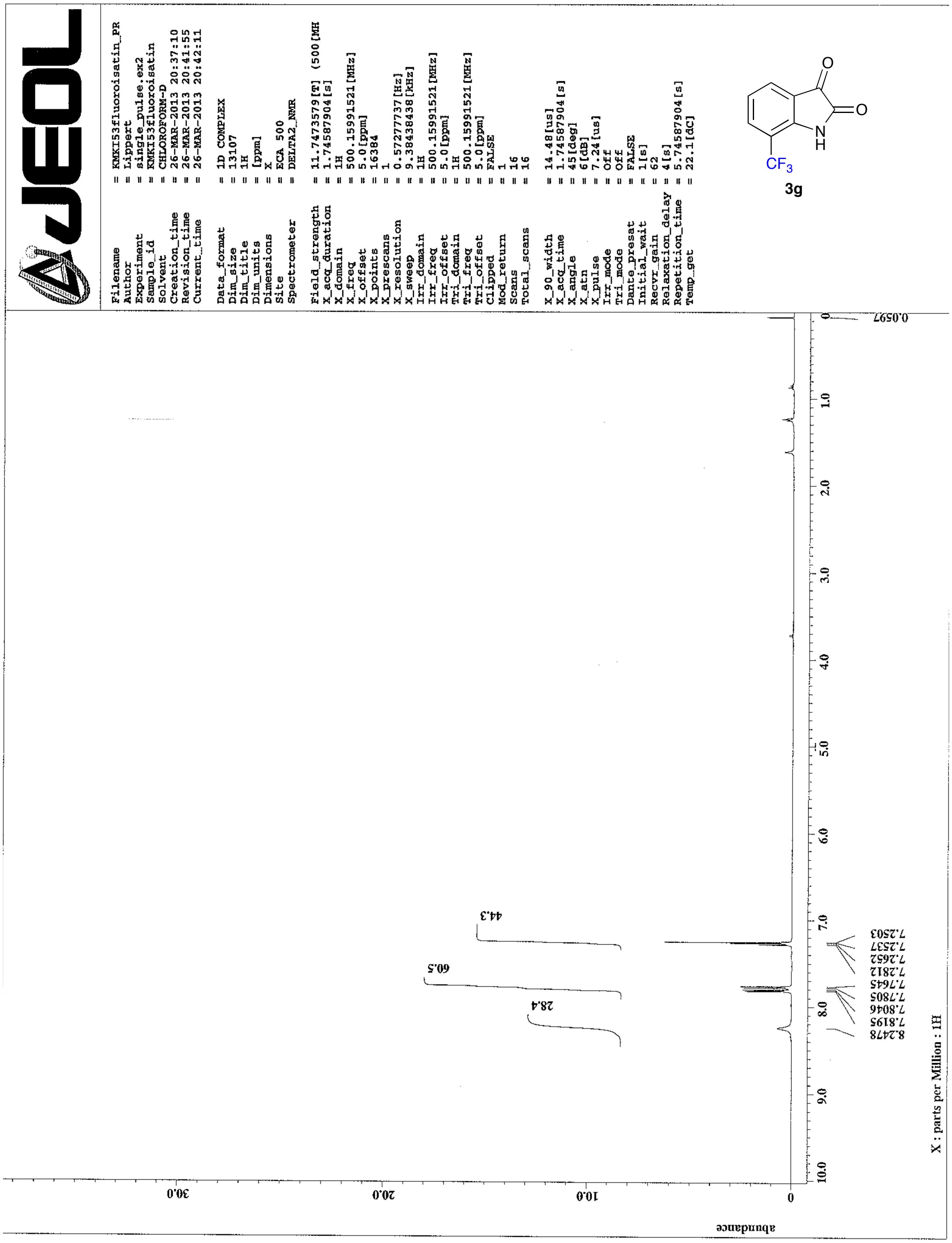
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 19207
Total_scans       = 19207

X_90_width        = 14.49[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 4.83[us]
Irr_atn_dec       = 23[dB]
Irr_atn_noe       = 23[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          = 21.1[dC]
```



3f


JEOL
Filename = KK_pg67_PROTON-8.jdf
Author = Lippert
Experiment = single_pulse.ex2
Sample_id = KK_pg67
Solvent = CHLOROFORM-D
Creation_time = 26-APR-2013 18:24:55
Revision_time = 26-APR-2013 18:43:09
Current_time = 26-APR-2013 18:43:18
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
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 = 16
Total_scans = 16
X_90_width = 14.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 7.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 4[s]
Repetition_time = 5.74587904[s]
Temp_get = 21.6[dC]
CH3
H3C
S
CN
O
NH2
CF3
2g
152.5
70.1
20.3
13.5
14.0
13.0
-0.0068
0.0001
1.0342
1.0468
1.0560
1.3835
1.5198
2.2974
2.5768
2.7165
2.7795
2.8482
5.7031
6.6193
6.6353
6.6502
7.3934
7.4083
7.7507
7.7656
-2.0
-1.0
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
X : parts per Million : 1H
40.0
30.0
20.0
10.0
0
abundance



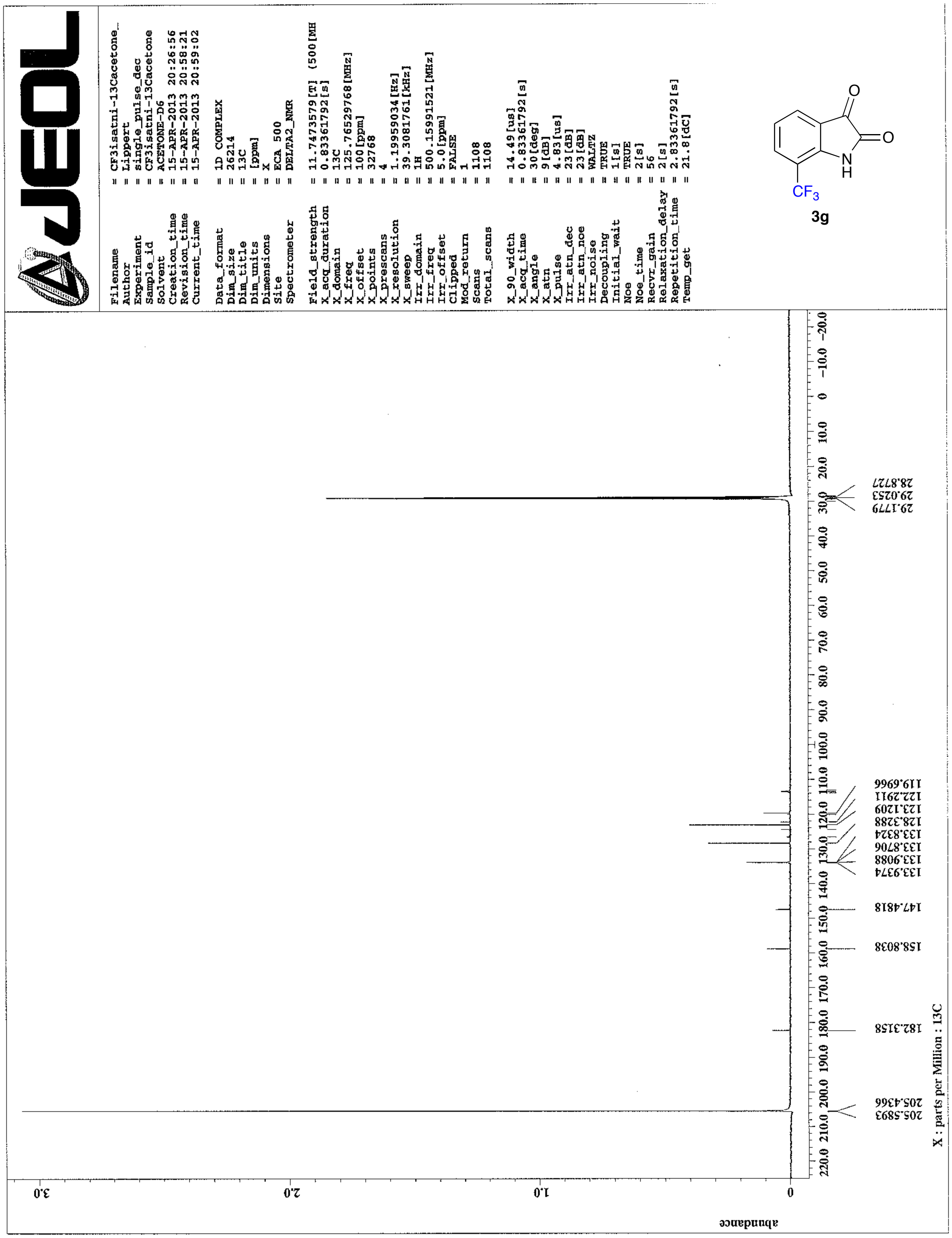
3g

```
Filename           = KMKI53fluoroisatin_PR
Author             = Lippert
Experiment         = single_pulse.ex2
Sample_id          = KMKI53fluoroisatin
Solvent            = CHLOROFORM-D
Creation_time      = 26-MAR-2013 20:37:10
Revision_time      = 26-MAR-2013 20:41:55
Current_time       = 26-MAR-2013 20:42:11

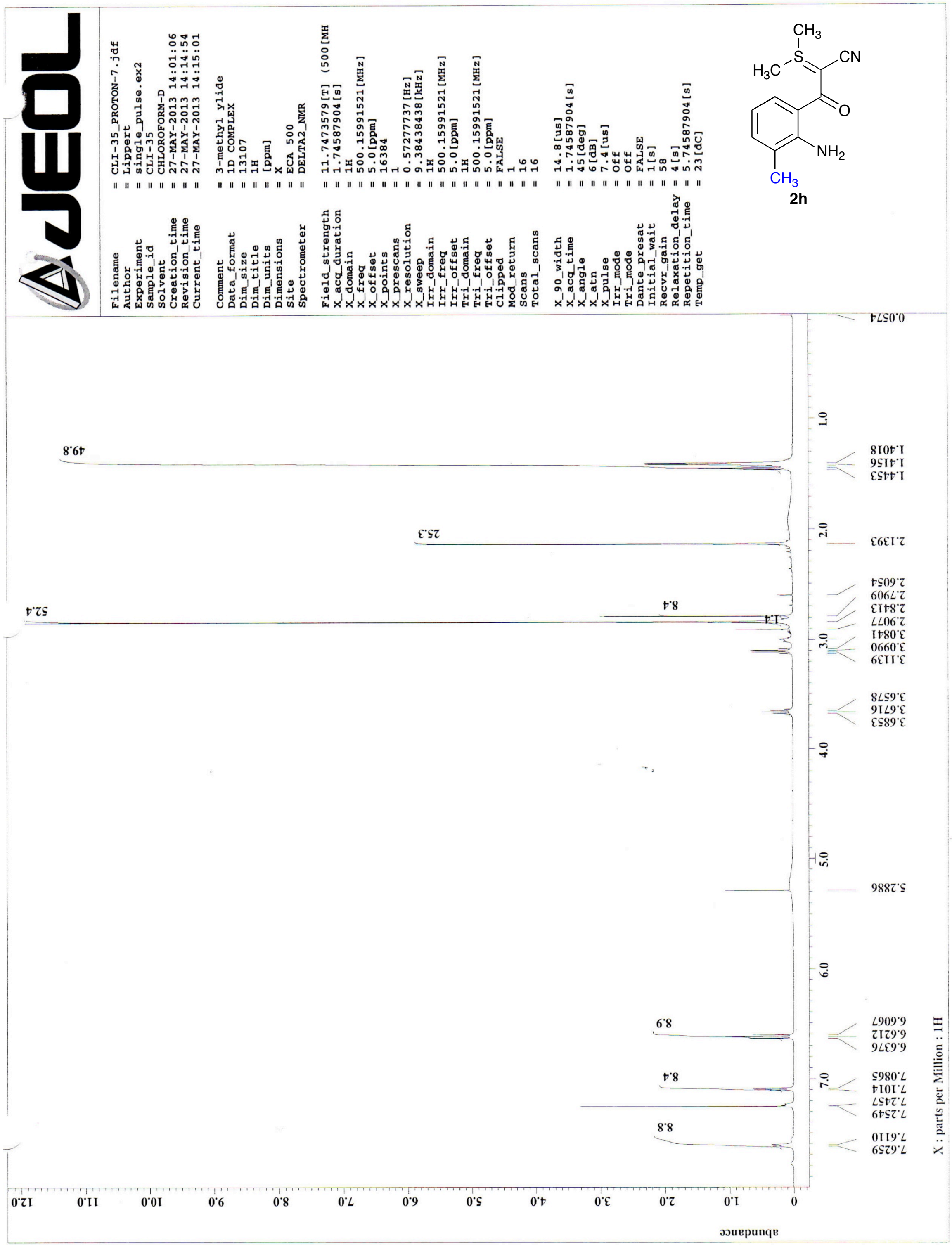
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
Spectrometer       = DELTA2_NMR

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              = 16
Total_scans        = 16

X_90_width         = 14.48[us]
X_acq_time         = 1.74587904[s]
X_angle            = 45[deg]
X_atn              = 6[dB]
X_pulse            = 7.24[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 62
Relaxation_delay   = 4[s]
Repetition_time    = 5.74587904[s]
Temp_get           = 22.1[dC]
```


Filename = CF3isatni-13Cacetone_
Author = Lippert
Experiment = single_pulse_dec
Sample_id = CF3isatni-13Cacetone
Solvent = ACETONE-D6
Creation_time = 15-APR-2013 20:26:56
Revision_time = 15-APR-2013 20:58:21
Current_time = 15-APR-2013 20:59:02
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
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 = 1108
Total_scans = 1108
X_90_width = 14.49[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 4.83[us]
Irr_atn_dec = 23[dB]
Irr_atn_noe = 23[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 = 21.8[dC]
O
O
N
H
CF3
28.8727
29.0253
29.1779
119.6966
122.2911
123.1209
128.3288
133.8324
133.8706
133.9088
133.9374
147.4818
158.8038
182.3158
205.4366
205.5893
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
X : parts per Million : 13C
3.0
2.0
1.0
0
abundance

3g



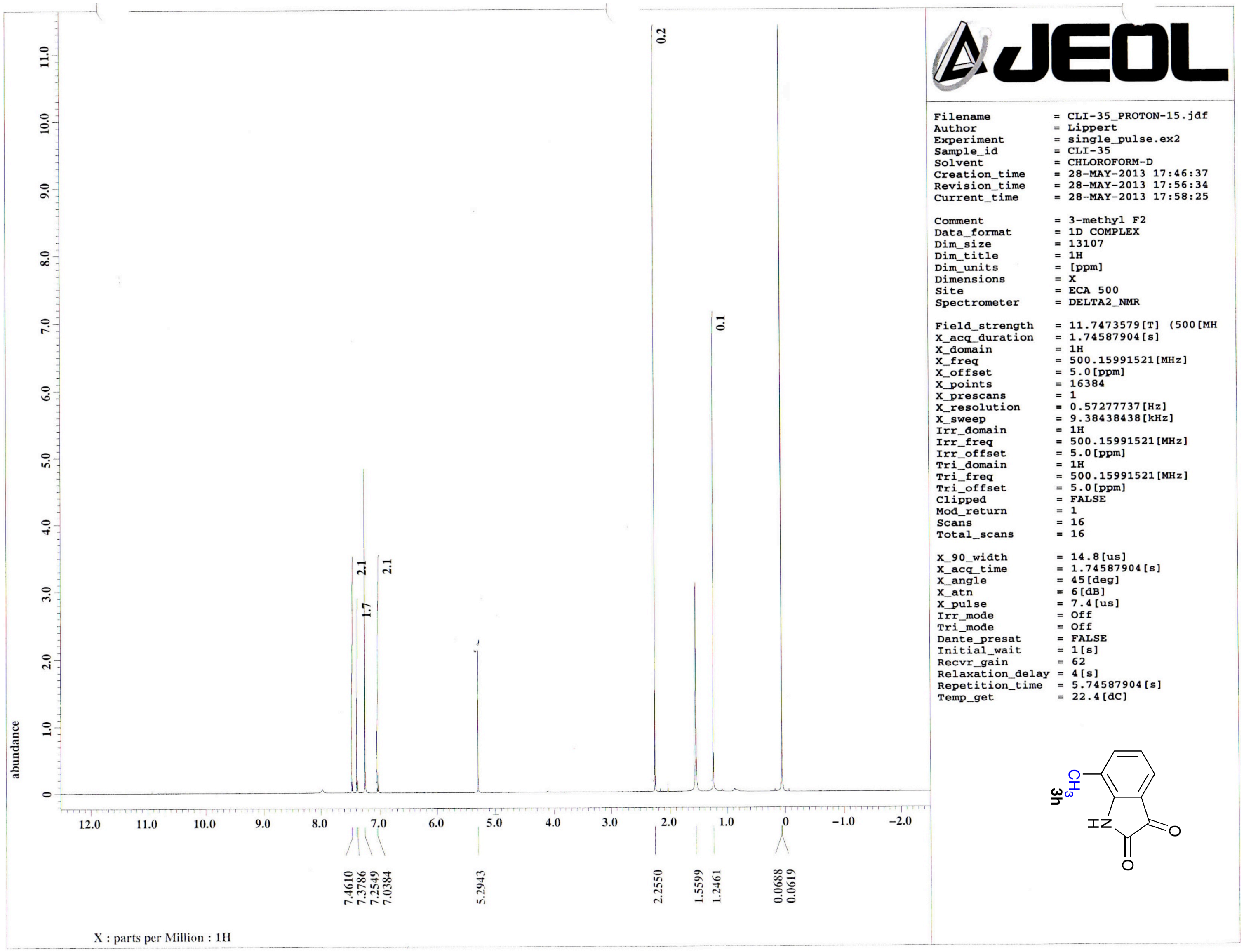
```
Filename           = CLI-35_PROTON-7.jdf
Author             = Lippert
Experiment         = single_pulse.ex2
Sample_id          = CLI-35
Solvent            = CHLOROFORM-D
Creation_time      = 27-MAY-2013 14:01:06
Revision_time      = 27-MAY-2013 14:14:54
Current_time       = 27-MAY-2013 14:15:01

Comment            = 3-methyl ylide
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
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              = 16
Total_scans        = 16

X_90_width         = 14.8[us]
X_acq_time         = 1.74587904[s]
X_angle            = 45[deg]
X_atn              = 6[dB]
X_pulse            = 7.4[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 58
Relaxation_delay   = 4[s]
Repetition_time    = 5.74587904[s]
Temp_get           = 23[dC]
```

2h



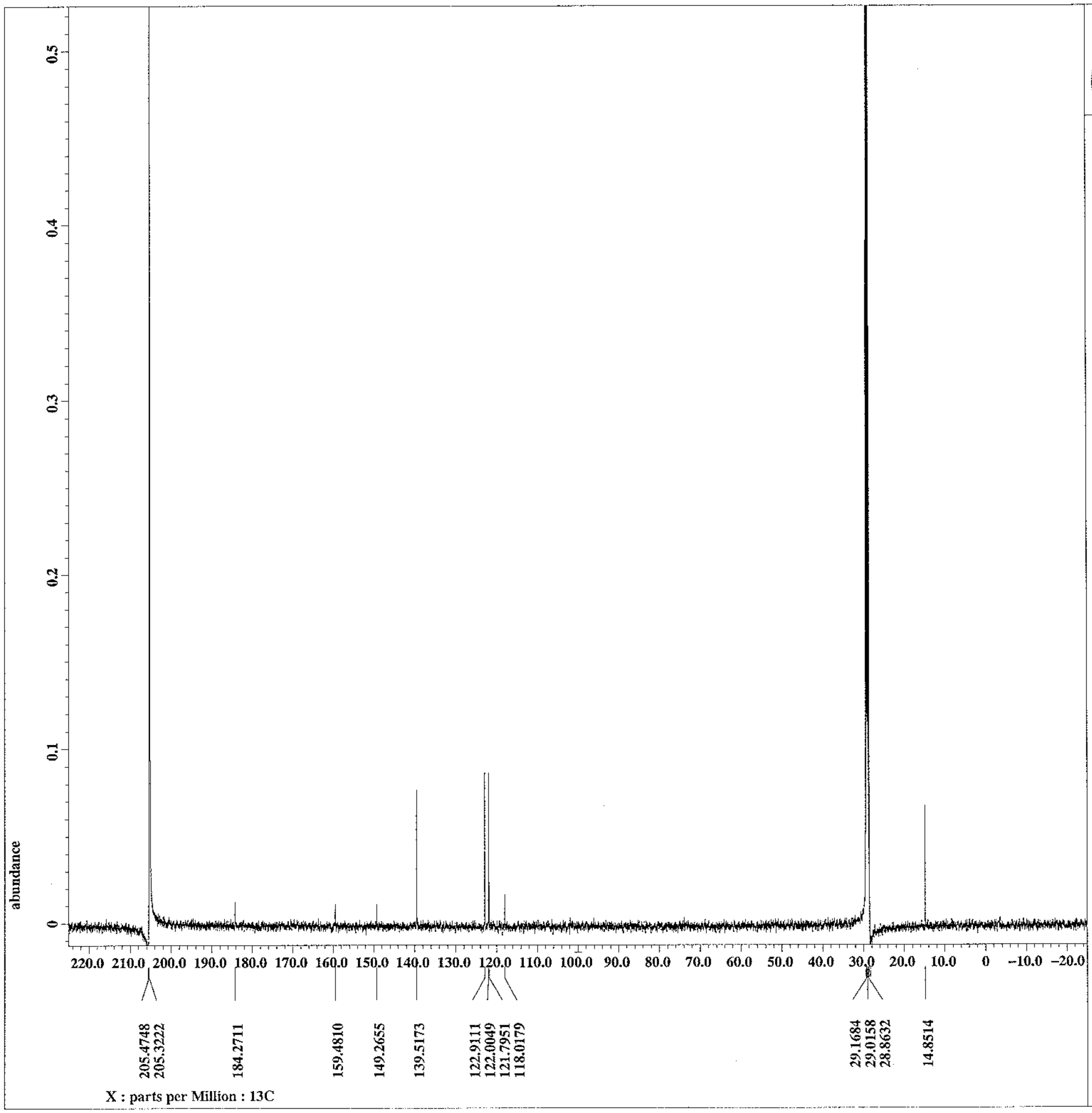
```
Filename          = CLI-35_PROTON-15.jdf
Author            = Lippert
Experiment        = single_pulse.ex2
Sample_id         = CLI-35
Solvent           = CHLOROFORM-D
Creation_time     = 28-MAY-2013 17:46:37
Revision_time     = 28-MAY-2013 17:56:34
Current_time      = 28-MAY-2013 17:58:25

Comment           = 3-methyl F2
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
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             = 16
Total_scans       = 16

X_90_width        = 14.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 6[dB]
X_pulse           = 7.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 62
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 22.4[dC]
```

3h

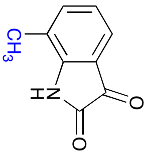


```
Filename          = KK473-CH3_CARBON-8.jd
Author            = Lippert
Experiment        = single_pulse_dec
Sample_id         = KK473-CH3
Solvent           = ACETONE-D6
Creation_time     = 20-APR-2013 16:55:21
Revision_time     = 20-APR-2013 17:04:22
Current_time      = 20-APR-2013 17:04:40

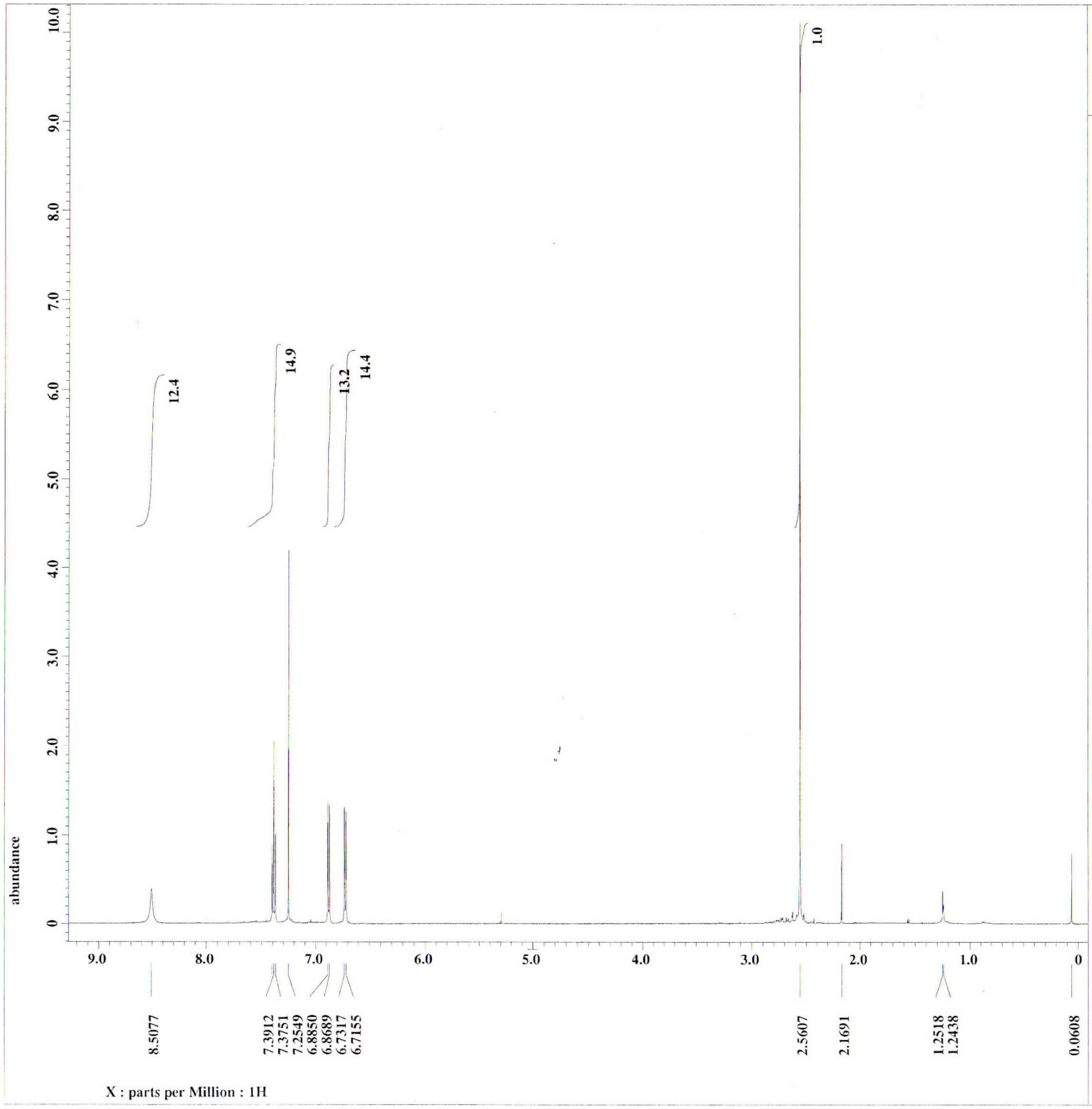
Comment           = 13C
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 2995
Total_scans       = 2995

X_90_width        = 14.49[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 4.83[us]
Irr_atn_dec       = 23[dB]
Irr_atn_noe       = 23[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 58
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 21.9[dC]
```



3h

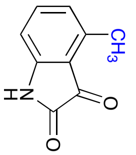


```
Filename           = CLI-30_PROTON-15.jdf
Author             = Lippert
Experiment         = single_pulse.ex2
Sample_id          = CLI-30
Solvent            = CHLOROFORM-D
Creation_time      = 19-APR-2013 18:35:41
Revision_time      = 19-APR-2013 21:13:11
Current_time       = 19-APR-2013 21:13:17

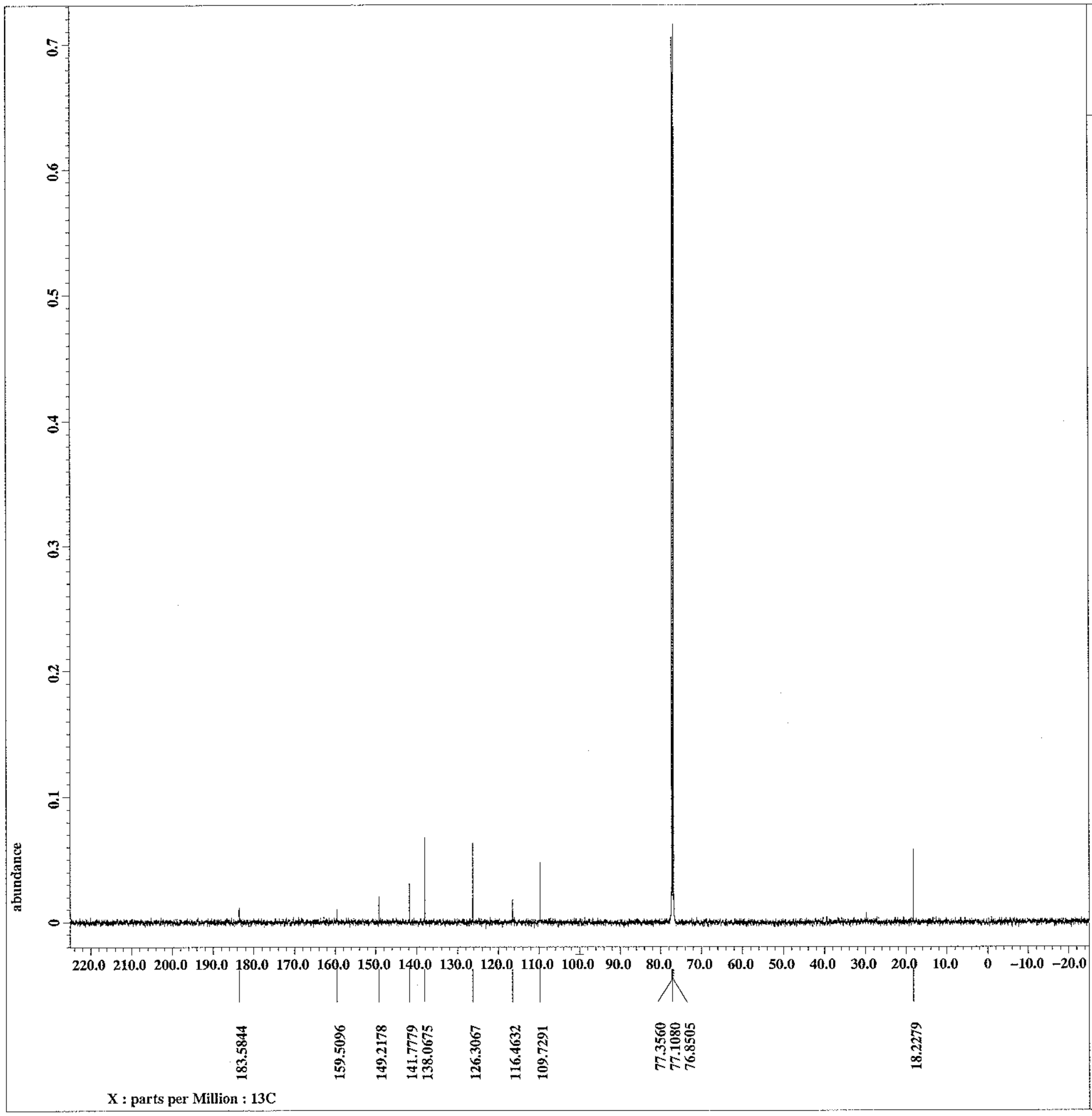
Comment            = 5-Methyl, somewhat im
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
Spectrometer       = DELTA2_NMR

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              = 16
Total_scans        = 16

X_90_width         = 14.8[us]
X_acq_time         = 1.74587904[s]
X_angle            = 45[deg]
X_atn              = 6[dB]
X_pulse            = 7.4[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 60
Relaxation_delay   = 4[s]
Repetition_time    = 5.74587904[s]
Temp_get           = 21.4[dC]
```



3i



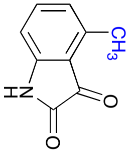
JEOL

```
Filename           = arlI76f1_CARBON-8.jdf
Author             = Lippert
Experiment         = single_pulse_dec
Sample_id          = arlI76f1
Solvent            = CHLOROFORM-D
Creation_time      = 28-FEB-2013 20:47:06
Revision_time      = 28-FEB-2013 20:48:00
Current_time       = 28-FEB-2013 20:48:18

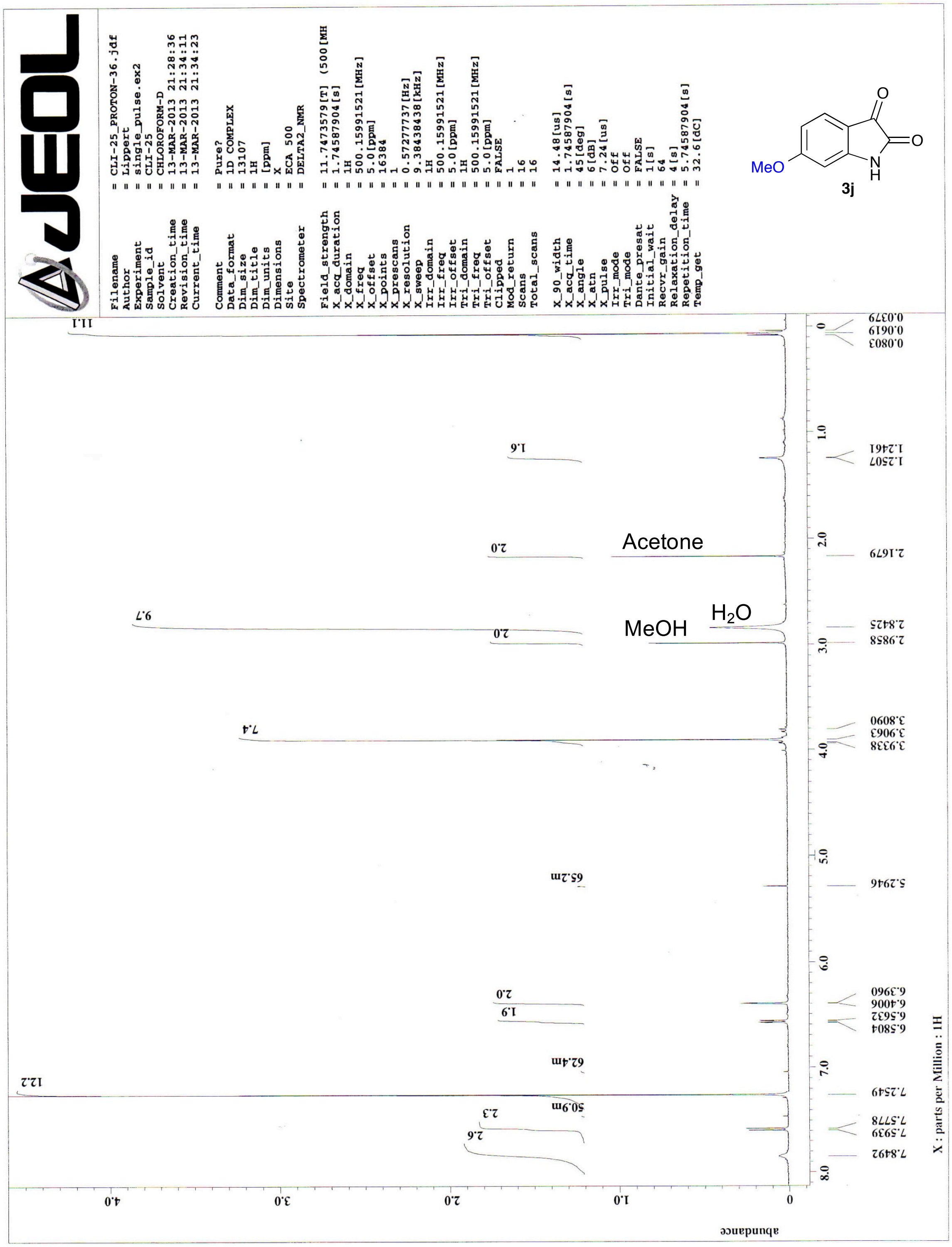
Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
Spectrometer       = DELTA2_NMR

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              = 2390
Total_scans        = 2390

X_90_width         = 14.11[us]
X_acq_time         = 0.83361792[s]
X_angle            = 30[deg]
X_atn              = 9[dB]
X_pulse            = 4.70333333[us]
Irr_atn_dec        = 23[dB]
Irr_atn_noe        = 23[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           = 32.4[dC]
```



3i

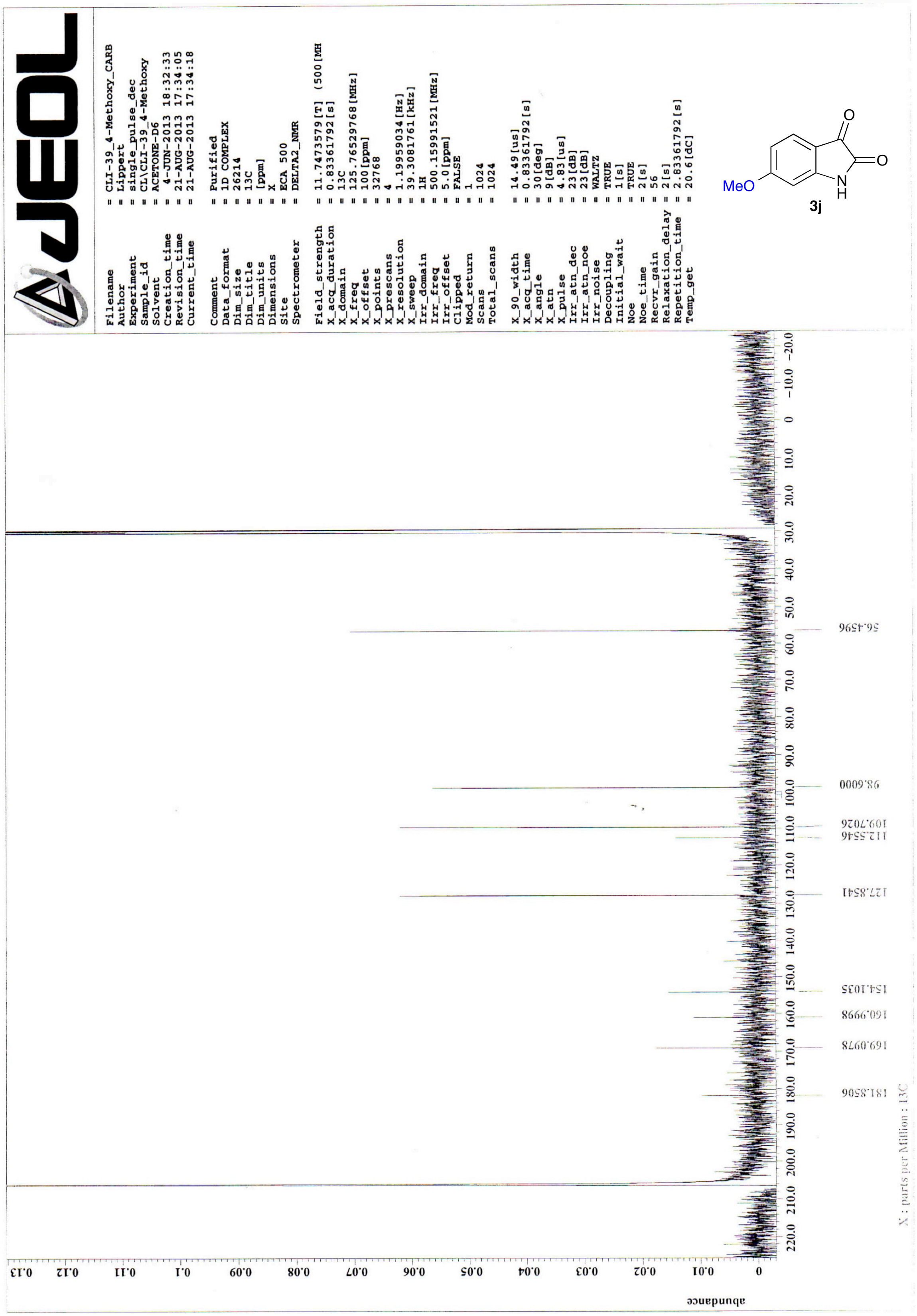


```
Filename           = CLI-25_PROTON-36.jdf
Author             = Lippert
Experiment         = single_pulse.ex2
Sample_id          = CLI-25
Solvent            = CHLOROFORM-D
Creation_time      = 13-MAR-2013 21:28:36
Revision_time      = 13-MAR-2013 21:34:11
Current_time       = 13-MAR-2013 21:34:23

Comment            = Pure?
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
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              = 16
Total_scans        = 16

X_90_width         = 14.48[us]
X_acq_time         = 1.74587904[s]
X_angle            = 45[deg]
X_atn              = 6[dB]
X_pulse            = 7.24[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 64
Relaxation_delay   = 4[s]
Repetition_time    = 5.74587904[s]
Temp_get           = 32.6[dC]
```

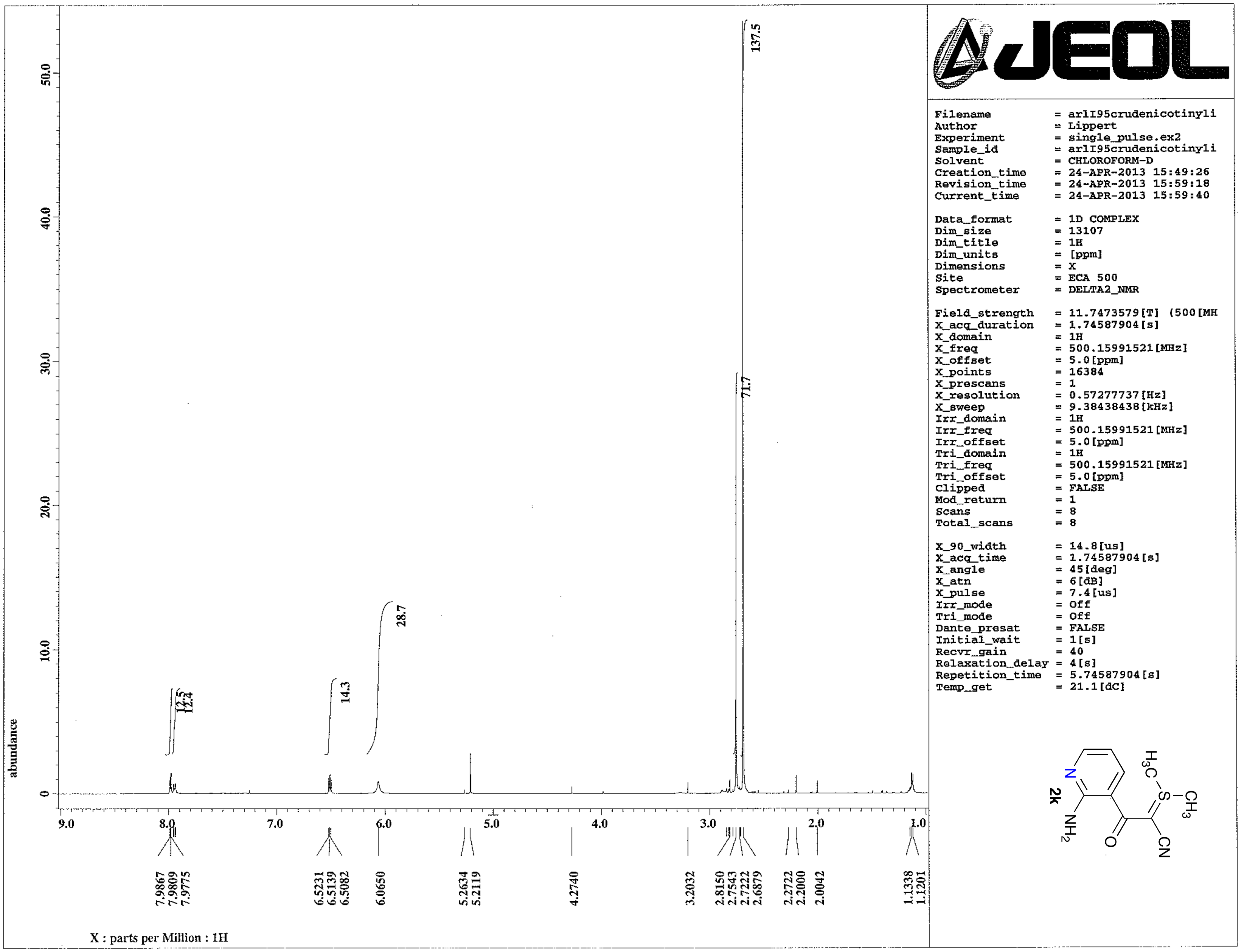


Filename = CLI-39_4-Methoxy_CARB
Author = Lippert
Experiment = single_pulse_dec
Sample_id = CL\CLI-39_4-Methoxy
Solvent = ACETONE-D6
Creation_time = 4-JUN-2013 18:32:33
Revision_time = 21-AUG-2013 17:34:05
Current_time = 21-AUG-2013 17:34:18

Comment = Purified
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
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 = 1024
Total_scans = 1024

X_90_width = 14.49[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 4.83[us]
Irr_atn_dec = 23[dB]
Irr_atn_noe = 23[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 = 20.6[dC]



2k

```
Filename          = arlI95crudenicotinyli
Author            = Lippert
Experiment        = single_pulse.ex2
Sample_id         = arlI95crudenicotinyli
Solvent           = CHLOROFORM-D
Creation_time     = 24-APR-2013 15:49:26
Revision_time     = 24-APR-2013 15:59:18
Current_time      = 24-APR-2013 15:59:40

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

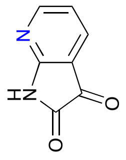
X_90_width        = 14.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 6[dB]
X_pulse           = 7.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 21.1[dC]
```

```
Filename           = CLI-51_Pyridine_Pure_
Author             = Lippert
Experiment         = single_pulse.ex2
Sample_id          = CL/CLI-51_Pyridine_Pu
Solvent            = METHANOL-D3
Creation_time      =  3-JUL-2013 11:22:25
Revision_time      =  7-SEP-2013 17:03:01
Current_time       =  7-SEP-2013 17:03:10

Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECA 500
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              = 16
Total_scans        = 16

X_90_width         = 14.8[us]
X_acq_time         = 1.74587904[s]
X_angle            = 45[deg]
X_atn              = 6[dB]
X_pulse            = 7.4[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 60
Relaxation_delay   = 4[s]
Repetition_time    = 5.74587904[s]
Temp_get           = 20.8[dC]
```



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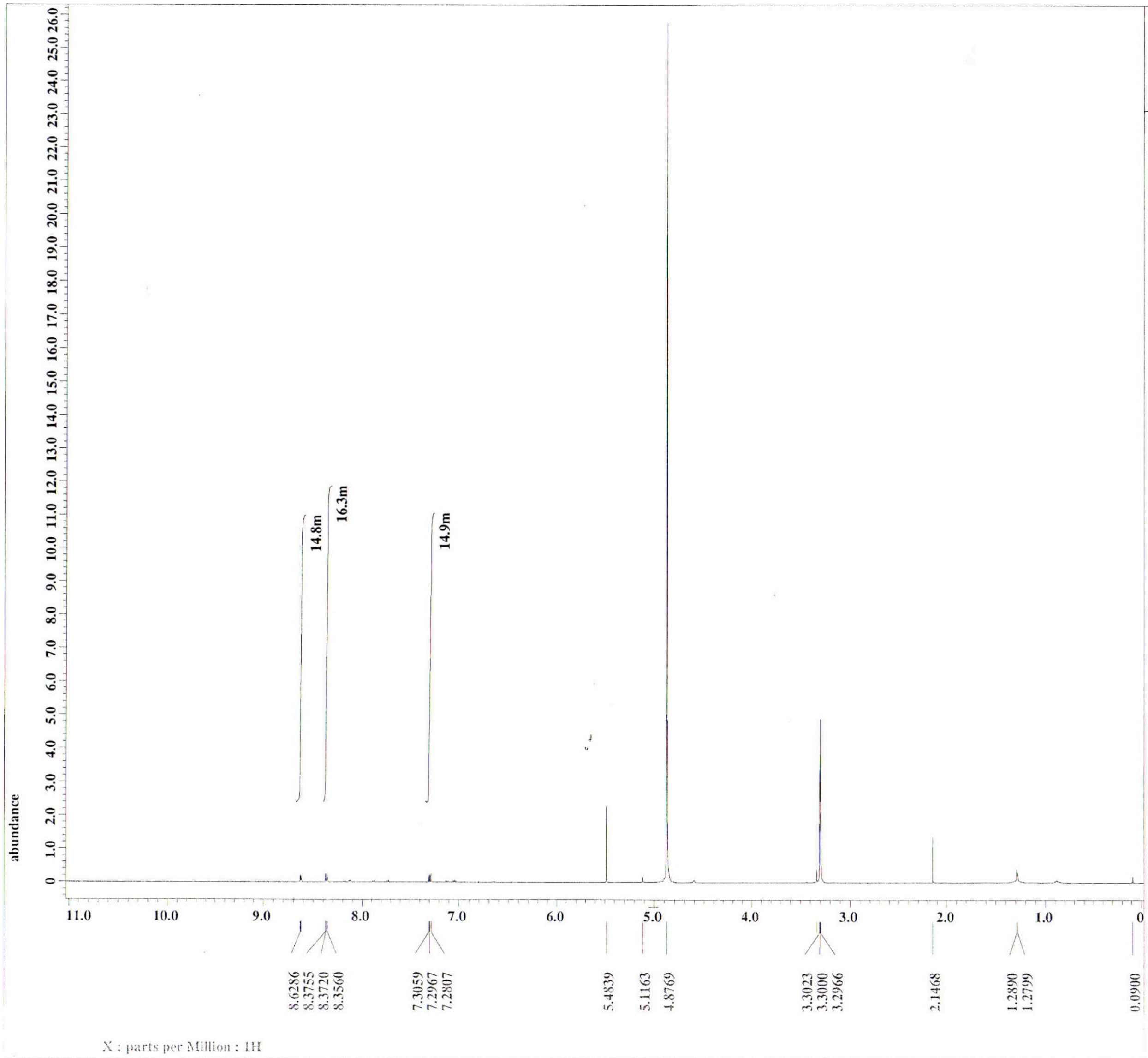
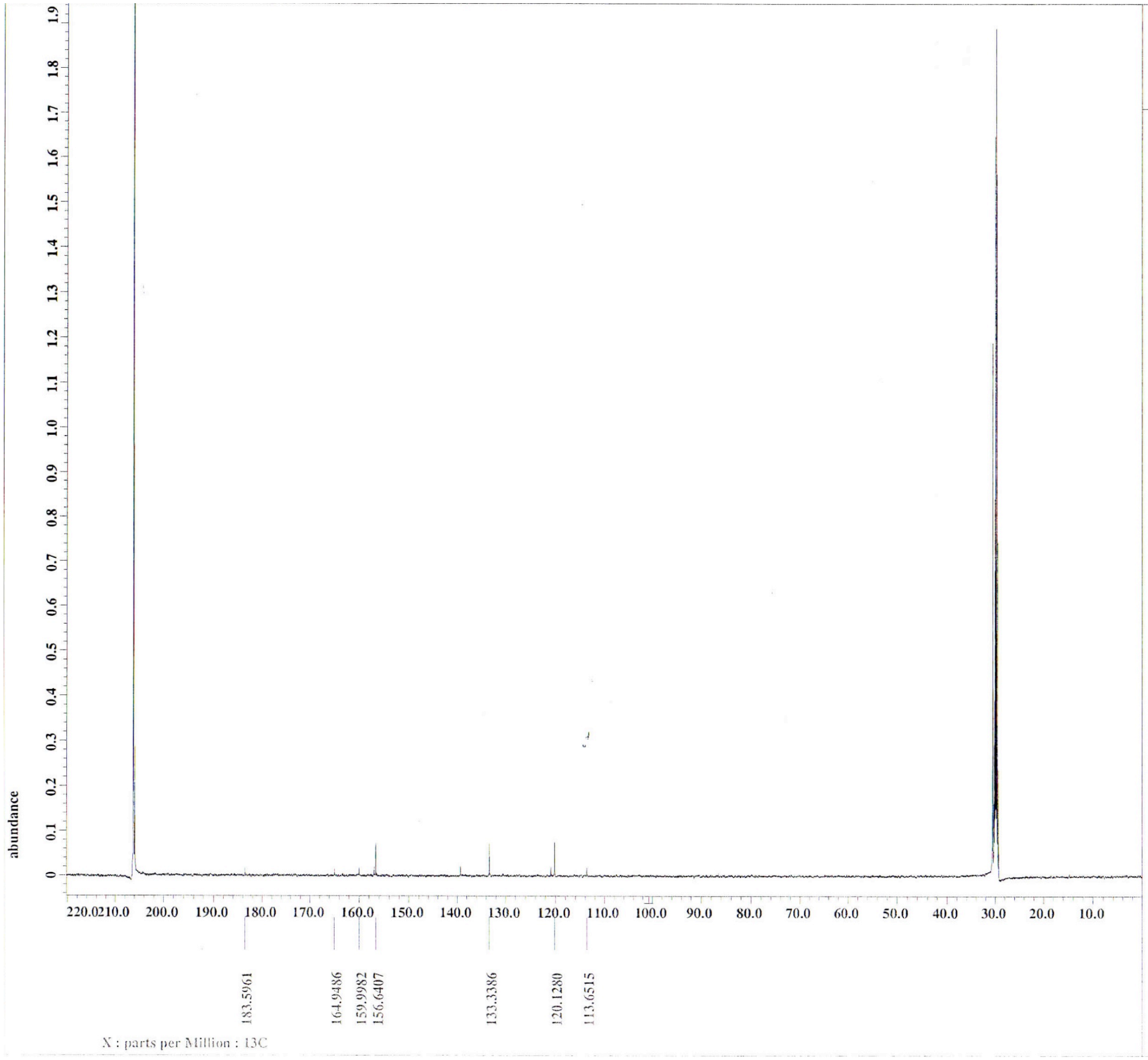


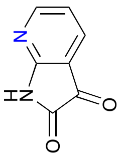


```
Filename        = CLI-39_Pyridine_isati
Author          = Lippert
Experiment      = single_pulse_dec
Sample_id       = CL\CLI-39_Pyridine_is
Solvent         = ACETONE-D6
Creation_time   =  5-JUN-2013 19:17:43
Revision_time   =  4-NOV-2013 12:21:18
Current_time    =  4-NOV-2013 12:23:39

Comment         = Pure
Data_format     = 1D COMPLEX
Dim_size        = 26214
Dim_title       = 13C
Dim_units       = [ppm]
Dimensions      = X
Site            = ECA 500
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           = 1455
Total_scans     = 1455

X_90_width      = 14.49[us]
X_acq_time      = 0.83361792[s]
X_angle         = 30[deg]
X_atn           = 9[dB]
X_pulse         = 4.83[us]
Irr_atn_dec     = 23[dB]
Irr_atn_noe     = 23[dB]
Irr_noise       = WALTZ
Decoupling      = TRUE
Initial_wait    = 1[s]
Noe             = TRUE
Noe_time        = 2[s]
Recvr_gain      = 58
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get        = 20.4[dC]
```



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