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# Supporting Information

for

# A golden opportunity: benzofuranone modifications of aurones and their influence on optical properties, toxicity, and potential as dyes

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## Experimental methods and spectra for all new compounds

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## Instrumentation

The structures of the newly synthesized aurones were determined by NMR (JEOL ECA-500 and ECX-300) and GC–MS (Shimadzu QP2010S). UV-Vis analyses were performed on an HP 8452A Diode Array spectrophotometer. Spectrometry results from MTT assay were analyzed using Molecular Devices SpectraMax M5 Multi-Mode Microplate Reader.

## General procedure for the condensation of non-hydroxy-substituted benzofuranones

In a round bottom flask, 1 mmol of the appropriate benzofuranone was mixed with 2 mmol of *p*-tolualdehyde in approximately 3.5 g of neutral alumina and 5 mL of dichloromethane. The reaction was stirred at room temperature for 24 hours. The alumina was filtered out and the mixture concentrated *in vacuo*. Two equivalents of isonicotinic acid hydrazide was added to the concentrated mixture in 5 mL/mmol of both dichloromethane and dimethylformamide and left to stir at room temperature for another 24 hours to remove any excess aldehyde. In some cases, further purification methods were used and/or the ratio of the hydrazide scavenger and the solvents were changed to increase the scavenger's effectiveness after air exposure. Exceptions are noted below. The aurone product was extracted using a mixture of ethyl acetate and 1M HCl except where noted. The organic layer was concentrated and subjected to NMR and GC/MS analysis for purity.

## General synthetic procedure for hydroxyl-substituted benzofuranones

In a round bottom flask, 1 mmol of the appropriate benzofuranone was mixed with 2 mmol of *p*-tolualdehyde in 1.5 g/mmol of 1:1 (w:w) KOH/$H_2O$ and 5 mL/mmol methanol. The mixture was left to stir for 6 hours at room temperature. The mixture was triturated with 1 M HCl until acidic and precipitates formed. The crystals were filtered out and washed with 100% hexane followed by 25% EtOAc in hexane. Finally, 50% EtOAc in hexane was used as a final wash. The product was analyzed via NMR and GC/MS.

**2-(4-Methylbenzylidene)-1-(benzofuran-3-one) [1]:** used 4 equivalents of the hydrazide scavenger and 5 mL/0.5 mmol of DCM and DMF to produce 0.15 g (62%) of the desired product as a pale yellow solid (mp = 97-99 °C). $^1$H NMR (500 MHz, $CDCl_3$): δ= 2.29 (s, 3H), 6.76 (s, 1H), 7.08 (t, J=7.4 Hz, 1H), 7.13 (d, J=8.4 Hz, 2H), 7.19 (d, J=8.6 Hz, 1H), 7.51 (t, J=8.6 Hz, 1H), 7.68 (d, J=8.4 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.67, 112.93, 113.27, 121.74, 123.34, 124.48, 129.53, 129.70 (2C), 131.62 (2C), 136.68, 140.43, 146.47, 165.91, 184.51; GC RT: (24.81 minutes); MS (200 °C, 70 eV): 236 ($M^+$, 95.1), 235 (96.3), 221 (100.0), 92 (46.7), 76 (58.6).

**2-(4-Methylbenzylidene)-1-(5'-fluorobenzofuran-3-one) [2]:** purified via column chromatography using silica gel and 10:90 EtOAc/hexane as eluent followed by extraction in 1M HCl and EtOAc to give 0.15 g (59%) of the product as a pale yellow solid (mp = 122-124 °C). $^1$H NMR (500 MHz, $CDCl_3$): δ= 2.41 (s, 3H), 6.91 (s, 1H), 7.26 (d, *J*=8.2 Hz, 2H), 7.30 (dd, *J*=8.6, 3.4 Hz, 1H), 7.36 (td, *J*=8.6, 2.3 Hz, 1H), 7.45 (dd, *J*=6.3, 2.8 Hz, 1H), 7.80 (d, *J*=5.8 Hz, 2H); $^{13}$C NMR (125 MHz,

$CDCl_3$): δ= 21.69, 109.91 (d, J=95 Hz), 114.07 (d, J=33 Hz), 114.21, 122.39 (d, J=33 Hz), 123.93 (d, J=104 Hz), 129.47 (d, J=248 Hz), 129.77 (2C), 131.74 (2C), 140.85, 147.03, 157.78, 161.90, 183.86; GC RT: (24.00 minutes); MS (200 °C, 70 eV): 254 ($M^+$, 47.9), 253 (49.0), 239 (100.0), 126 (53.9), 115 (41.7).

**2-(4-Methylbenzylidene)-1-(6'-fluorobenzofuran-3-one) [3]:** a new batch of the hydrazide scavenger was reintroduced after column chromatography giving 0.1 g (39%) of the product as a pale yellow solid (mp = 172-174 °C). $^1$H NMR (500 MHz, $CDCl_3$): δ= 2.40 (s, 3H), 6.87 (s, 1H), 6.90 (td, *J*=14.9, 3.4 Hz, 1H), 7.0 (dd, *J*=14.3, 3.4 Hz, 1H), 7.25 (d, *J*=13.2 Hz, 2H), 7.77 (d, *J*=13.2 Hz, 2H), 7.80 (d, *J*=13.8 Hz, 1H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.74, 100.77(d, J=104 Hz), 111.86 (d, J=95 Hz), 113.75, 118.47, 126.45 (d, J=48 Hz), 129.55 (d, J=286 Hz), 129.83 (2C), 131.70 (2C), 140.83, 146.89, 167.14 (d, J=33 Hz), 169.25, 182.97; GC RT: (23.88 minutes); MS (200 °C, 70 eV): 254 ($M^+$, 56.3), 253 (65.4), 239 (100.0), 126 (61.2), 115 (50.6).

**2-(4-Methylbenzylidene)-1-(4'-chlorobenzofuran-3-one) [4]:** required 4 equivalents of the hydrazide scavenger and 5 mL/0.5 mmol of DCM and DMF to yield 0.2 g (74%) of the desired product as a pale yellow solid (mp = 172-173 °C). $^1$H NMR (500 MHz, $CDCl_3$): δ= 2.33 (s, 3H), 6.77 (s, 1H), 7.04 (d, *J*=8.0 Hz, 1H), 7.12 (d, *J*=8.0 Hz, 1H), 7.15 (d, *J*=8.0 Hz, 2H), 7.43 (t, *J*=8.0 Hz, 1H), 7.67 (d, *J*=8.1 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.75, 111.39, 113.95, 119.03, 124.56, 129.24, 129.79 (2C), 131.69 (2C), 132.27, 136.76, 140.83,

146.11, 166.43, 181.77; GC RT: (26.50 minutes); MS (200 °C, 70 eV): 272 (M+2, 12.2), 270 ($M^+$, 34.2), 269 (40.3), 257 (31.1), 255 (100.0), 134 (58.7), 115 (53.5).

**2-(4-Methylbenzylidene)-1-(5'-chlorobenzofuran-3-one) [5]:** another 2 equivalents of the hydrazide scavenger were reintroduced in 5 mL/mmol DCM and DMF to yield 0.2 g (72%) of the desired product as a pale yellow solid (mp = 143-146 °C). $^1$H NMR (500 MHz, $CDCl_3$): δ= 2.39 (s, 3H), 6.86 (s, 1H), 7.23 (d, *J*=6.8 Hz, 2H), 7.55 (dd, *J*=8.6, 2.3 Hz, 1H), 7.71 (d, *J*=2.3 Hz, 1H), 7.75 (d, *J*=8.0 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.76, 114.25, 114.41, 122.91, 124.09, 129.06, 129.17, 129.79 (2C), 131.78 (2C), 136.40, 140.98, 146.57, 164.05, 183.21; GC RT: (25.93 minutes); MS (200 °C, 70 eV): 272 (M+2, 14.2), 270 ($M^+$, 43.9), 269 (44.6), 257 (33.8), 255 (100.0), 134 (59.6), 115 (76.4).

**2-(4-Methylbenzylidene)-1-(6'-chlorobenzofuran-3-one) [6]:** another 2 equivalents of the hydrazide scavenger were reintroduced in 5 mL/mmol DCM and DMF to yield 0.19 g (70%) of the product as a pale yellow solid (mp = 165-166 °C). $^1$H NMR (500 MHz, $CDCl_3$): δ= 2.37 (s, 3H), 6.83 (s, 1H), 7.13 (dd, *J*=8.1, 1.7 Hz ,1H), 7.21 (d, *J*=8.0 Hz, 2H), 7.29 (s, 1H), 7.66 (d, *J*=8.2 Hz, 1H), 7.72 (d, *J*=8.6 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.76, 113.65, 114.23, 120.48, 124.30, 125.38, 129.24, 129.84 (2C), 131.75 (2C), 140.94, 142.72, 146.59, 166.06, 183.21; GC RT: (25.81 minutes); MS (200 °C, 70 eV): 272 (M+2, 15.3), 270 ($M^+$, 46.0), 269 (46.3), 257 (31.3), 255 (100.0), 134 (63.5), 115 (79.4).

**2-(4-Methylbenzylidene)-1-(7'-chlorobenzofuran-3-one) [7]:** another 2 equivalents of the hydrazide scavenger were reintroduced in 5 mL/mmol DCM and DMF to yield 0.14 g (50%) of the product as a pale yellow solid (mp = 172-174 °C). $^{1}$H NMR (500 MHz, $CDCl_3$): δ= 2.37 (s, 3H), 6.90 (s, 1H), 7.11 (t, *J*=7.7 Hz, 1H), 7.23 (d, *J*=8.0 Hz, 2H), 7.59 (d, *J*=7.4 Hz, 1H), 7.65 (d, *J*=7.4 Hz, 1H), 7.80 (d, *J*=8.1 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.80, 115.01, 118.62, 122.86, 123.65, 124.14, 129.17, 129.93 (2C), 132.02 (2C), 136.34, 141.16, 146.26, 161.53, 183.79; GC RT: (25.54 minutes); MS (200 °C, 70 eV): 272 (M+2, 16.6), 270 ($M^+$, 47.8), 269 (45.0), 257 (33.5), 255 (100.0), 134 (63.7), 115 (81.1).

**2-(4-Methylbenzylidene)-1-(5'-bromobenzofuran-3-one) [8]:** another 2 equivalents of the hydrazide scavenger were reintroduced in 5 mL/mmol DCM and DMF to yield 0.15 g (42%) of the desired product as a pale yellow solid (mp = 154-155 °C). $^{1}$H NMR (500 MHz, $CDCl_3$): δ= 2.41 (s, 3H), 6.90 (s, 1H), 7.23 (d, *J*=8.6 Hz, 1H), 7.25 (d, *J*=8.2 Hz, 2H), 7.71 (dd, *J*=8.6, 1.7 Hz, 1H), 7.78 (d, *J*=8.6 Hz, 2H), 7.90 (d, *J*=2.3 Hz, 1H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.63, 114.42, 114.57, 116.14, 123.36, 127.14, 129.04, 129.69 (2C), 131.66 (2C), 139.07, 140.91, 146.29, 164.41, 183.03; GC RT: (26.89 minutes); MS (200 °C, 70 eV): 316 ($[M+2-1]^+$, 31.8), 315 ($M^+$, 34.5), 314 ($[M-1]^+$, 32.4), 301 (69.9), 299 (74.2), 115 (100.0), 91 (14.8).

**2-(4-Methylbenzylidene)-1-(6'-bromobenzofuran-3-one) [9]:** scavenger was reintroduced three times to produce 0.26 g (83%) of the desired product as a

pale yellow solid (mp = 157-158 °C). $^{1}$H NMR (500 MHz, $CDCl_3$): δ= 2.37 (s, 3H), 6.84 (s, 1H), 7.20 (d, *J*=8.1 Hz, 2H), 7.29 (d, *J*=8.0 Hz, 1H), 7.47 (s, 1H), 7.59 (d, *J*=8.0 Hz, 1H), 7.72 (d, *J*=8.2 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.79, 114.30, 116.64, 120.86, 125.47, 127.11, 129.25, 129.85 (2C), 131.15, 131.77 (2C), 140.96, 146.45, 165.96, 183.36; GC RT: (26.79 minutes); MS (200 °C, 70 eV): 316 ($[M+2-1]^+$, 29.3), 315 ($M^+$, 35.2), 314 ($[M-1]^+$, 30.2), 301 (53.6), 299 (58.9), 115 (100.0), 91 (16.8).

**2-(4-Methylbenzylidene)-1-(7'-bromobenzofuran-3-one) [10]:** scavenger was reintroduced two times to yield 0.18 g (53%) of the product as a pale yellow solid (mp = 187-192 °C). $^{1}$H NMR (500 MHz, $CDCl_3$): δ= 2.40 (s, 3H), 6.93 (s, 1H), 7.08 (t, *J*=7.7 Hz, 1H), 7.26 (d, *J*=8.0 Hz, 2H), 7.72 (d, *J*=8.2 Hz, 1H), 7.77 (d, *J*=8.1 Hz, 1H), 7.84 (d, *J*=8.0 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.66, 106.08, 114.85, 123.39, 124.43, 129.06 129.81 (2C), 131.91 (2C), 139.11, 141.02, 146.15, 162.75, 183.85; GC RT: (26.41 minutes); MS (200 °C, 70 eV): 316 ($[M+2-1]^+$, 37.5), 315 ($M^+$, 38.2),314 ($[M-1]^+$, 38.6), 301 (72.2), 299 (75.4), 115 (79.6), 91 (25.2).

**2-(4-Methylbenzylidene)-1-(5'-methylbenzofuran-3-one) [11]:** scavenger was reintroduced one more time to produce 0.14 g (56%) of the desired product as a pale yellow solid (mp = 117-119 °C). $^{1}$H NMR (300 MHz, $CDCl_3$): δ= 2.37 (s, 3H), 2.38 (s, 3H), 6.83 (s, 1H), 7.17 (d, *J*=8.6 Hz, 1H), 7.22 (d, *J*=7.9 Hz, 2H), 7.41 (d, *J*=8.6 Hz, 1H), 7.55 (s, 1H), 7.77 (d, *J*=7.9 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$):

δ= 20.84, 21.71, 112.54, 113.04, 121.67, 124.20, 129.67, 129.73 (2C), 131.60 (2C), 133.09, 137.91, 140.39, 146.97, 164.51, 184.83; GC RT: (25.50 minutes); MS (200 °C, 70 eV): 250 ($M^+$, 69.2), 249 (74.0), 235 (100), 134 (54.5), 124 (77.5), 89 (60.2).

**2-(4-Methylbenzylidene)-1-(5',6'-dimethylbenzofuran-3-one) [12]:** another 2 equivalents of the hydrazide scavenger were reintroduced in 5 mL/mmol DCM and DMF to yield 0.11 g (40%) of the product as a pale yellow solid (mp = 181-183 °C). $^1$H NMR (500 MHz, $CDCl_3$): δ= 2.22 (s, 3H), 2.31 (s, 3H), 2.36 (s, 3H), 6.76 (s, 1H), 7.03 (s, 1H), 7.20 (d, *J*=8.0 Hz, 2H), 7.45 (s, 1H), 7.73 (d, *J*=8.1 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 19.28, 21.30, 21.48, 112.23, 113.18, 119.33, 124.16, 129.48 (2C), 129.59, 131.26 (2C), 132.01, 139.95, 146.93, 147.59, 164.98, 184.27; GC RT: (27.00 minutes); MS (200 °C, 70 eV): 264 ($M^+$, 68.2), 263 (84.2), 249 (91.45), 148 (42.3), 131 (55.9), 117 (53.7), 91 (100.0).

**2-(4-Methylbenzylidene)-1-(7'-methylbenzofuran-3-one) [13]:** 0.13 g (52%) yield as a pale yellow solid (mp = 107-108 °C). $^1$H NMR (500 MHz, CDCl3): δ= 2.37 (s, 3H), 2.44 (s, 3H), 6.83 (s, 1H), 7.05 (t, *J*=7.4 Hz, 1H), 7.22 (d, *J*=8.0 Hz, 2H), 7.39 (d, *J*=6.9 Hz, 1H), 7.58 (d, *J*=8.0 Hz, 1H), 7.76 (d, *J*=8.0 Hz, 2H); $^{13}$C NMR (125 MHz, CDCl3): δ= 14.38, 21.72, 113.11, 121.26, 121.90, 122.97, 123.29, 128.74 (3C- two are coincident), 131.62 (2C), 137.60, 140.42, 146.74, 164.79, 185.20; GC RT: (25.08 minutes); MS (200 °C, 70 eV): 250 ($M^+$, 58.9), 249 (66.0), 235 (100), 124 (61.0), 106 (70.0), 89 (59.3).

**2-(4-Methylbenzylidene)-1-(6'-hydroxybenzofuran-3-one) [14]:** 0.05 g (20%) yield as an off-white solid (mp > 210 °C). $^{1}$H NMR (500 MHz, $(CD_3)_2CO$): δ= 2.36 (s, 3H), 6.68 (s, 1H), 6.76 (dd, *J*=8.6, 2.3 Hz, 1H), 6.82 (d, *J*=1.7 Hz, 1H), 7.28 (d, *J*=8.0 Hz, 2H), 7.60 (d, *J*=8.0 Hz, 1H), 7.83 (d, *J*=8.6 Hz, 2H); $^{13}$C NMR (125 MHz, $(CD_3)_2CO$): δ= 20.67, 98.79, 110.41, 112.82, 113.96, 125.79, 129.65 (2C), 129.88, 131.22 (2C), 139.93, 147.49, 166.08, 168.39, 181.58; GC RT: (28.08 minutes); MS (200 °C, 70 eV): 252 ($M^+$, 58.5), 251 (54.8), 63 (76.1), 44 (100.0).

**2-(4-Methylbenzylidene)-1-(4'-hydroxybenzofuran-3-one) [15]:** 0.04 g (15%) yield, as a pale yellow solid (mp = 156-158 °C). $^{1}$H NMR (500 MHz, $CDCl_3$): δ= 2.41 (s, 3H), 6.61 (d, J=8.1 Hz, 1H), 6.79 (d, J=8.1 Hz, 1H), 6.83 (s,1H), 7.25 (d, J=7.4 Hz, 2H), 7.49 (t, J=8.3 Hz, 1H), 7.80 (d, J=8.0 Hz, 2H), 7.83 (x, 1H, OH); $^{13}$C NMR (125 MHz, $CDCl_3$): δ= 21.77, 103.71, 109.77, 109.81, 113.59, 129.32, 129.85 (2C), 131.75 (2C), 139.19, 140.89, 146.28, 156.72, 164.90, 185.75; GC RT: (25.09 minutes); MS (200 °C, 70 eV): 252 ($M^+$, 98.7), 251 (77.8), 237 (100.0).

**2-(4-Methylbenzylidene)-1-(6'-hydroxy-4'-methylbenzofuran-3-one) [16]:** 0.03 g (11%) yield as a pale yellow solid (mp >210 °C). $^{1}$H NMR (500 MHz, $CDCl_3$): δ= 2.39 (s, 3H), 2.62 (s, 3H), 6.44 (s, 1H), 6.57 (s, 1H), 6.75 (s, 1H), 7.23 (d, J=8.2 Hz, 2H), 7.76 (d, J=8.6 Hz, 2H); $^{13}$C NMR (500 MHz, $(CD_3)_2CO$): δ= 16.98, 20.66, 96.34, 109.58, 112.35, 113.43, 129.61 (2C), 130.01, 131.08 (2C), 139.67, 141.38, 147.65, 165.36, 168.61, 182.43; GC RT: (28.46 minutes); MS

(200 °C, 70 eV): 266 ($M^+$, 72.7), 265 (100.0), 251 (70.7), 132 (82.9), 77 (90.0), 51 (84.9).

**2-(4-Methylbenzylidene)-1-(6'-hydroxy-7'-methylbenzofuran-3-one) [17]:** 0.05 g (19%) yield as a pale yellow solid (mp >210 °C). $^1$H NMR (500 MHz, $(CD_3)_2CO$): δ= 2.25 (s, 3H), 2.43 (s, 3H), 6.42 (d, *J*=2.3 Hz, 1H), 6.52 (s, 1H), 6.52 (d, *J*=1.7 Hz, 1H), 7.17 (d, *J*=7.4 Hz, 2H), 7.71 (d, *J*=8.1 Hz, 2H); $^{13}$C NMR (125 MHz, $(CD_3)_2CO$): δ= 16.96, 20.65, 96.33, 109.55, 112.36, 113.43, 129.62 (2C), 130.02, 131.08 (2C), 139.67, 141.37, 148.57, 165.39, 168.49, 182.42; GC RT: (28.34 minutes); MS (200 °C, 70 eV): 266 ($M^+$, 31.9), 265 (48.9), 251 (43.6), 77 (53.6), 44 (100.0).

**General procedure for UV–vis spectroscopy**

Stock solutions ranging from 87 µM to 134 µM of the aurones were made by dissolving a known mass of the aurones in acetonitrile. Each stock solution was used for subsequent dilutions to achieve a final concentration ranging from 1.5 µM to 3.3 µM. Using five different known concentration points for each aurone, the molar extinction coefficient was determined by measurement of the absorbance at the $\lambda_{max}$ closest to the visible range. The absorbance measurement of the highest concentration point was used for the absorbance shift study. 2-(4-methylbenzylidene)-1-(6'-hydroxy-7'-methylbenzofuran-3-one (**17**) was the exception as the exact concentration of the solution could not be determined due to its poor solubility in acetonitrile.

## Hep G2 cytotoxicity assay

Hep G2 human liver cancer cells were cultured in phenol red Dulbecco's Modified Eagle Medium (DMEM) with 10% fetal bovine serum (FBS) and 1% Pen-strep (PSG) under 37°C and 5% $CO_2$ atmospheric conditions. The cells were washed with phosphate buffered saline (PBS) and diluted with phenol red free DMEM to a concentration between 200,000 to 250,000 cells/mL. The cells were distributed in a 96-well plate with each well containing 100 µL of the cell solution and were allowed to settle for 2 hours in the incubator. The cells were then treated with 200 µM of the aurones in DMSO and the known dyes in 10:90 (v:v) DMSO/ultrapure water in triplicates and were left in the incubator for 3 days. The vehicles were used as the negative controls (DMSO for the aurones and 10:90 DMSO:ultrapure water for the known dyes). 20 µL of 5 mg/mL MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) was added to each well containing treated cells after the 3-day incubation period and was allowed to settle in the incubator for 3.5 more hours. Finally, the media was removed from the wells and replaced by 100 µL of DMSO to lyse the cells and dissolve the formazan crystals that were formed. The absorbance was then recorded on the SpectraMax M5 Multi-Mode Microplate Reader. The procedure was repeated two more times using different batches of cultured Hep G2 cells on different days.

## Dyeing and mordanting processes [1]

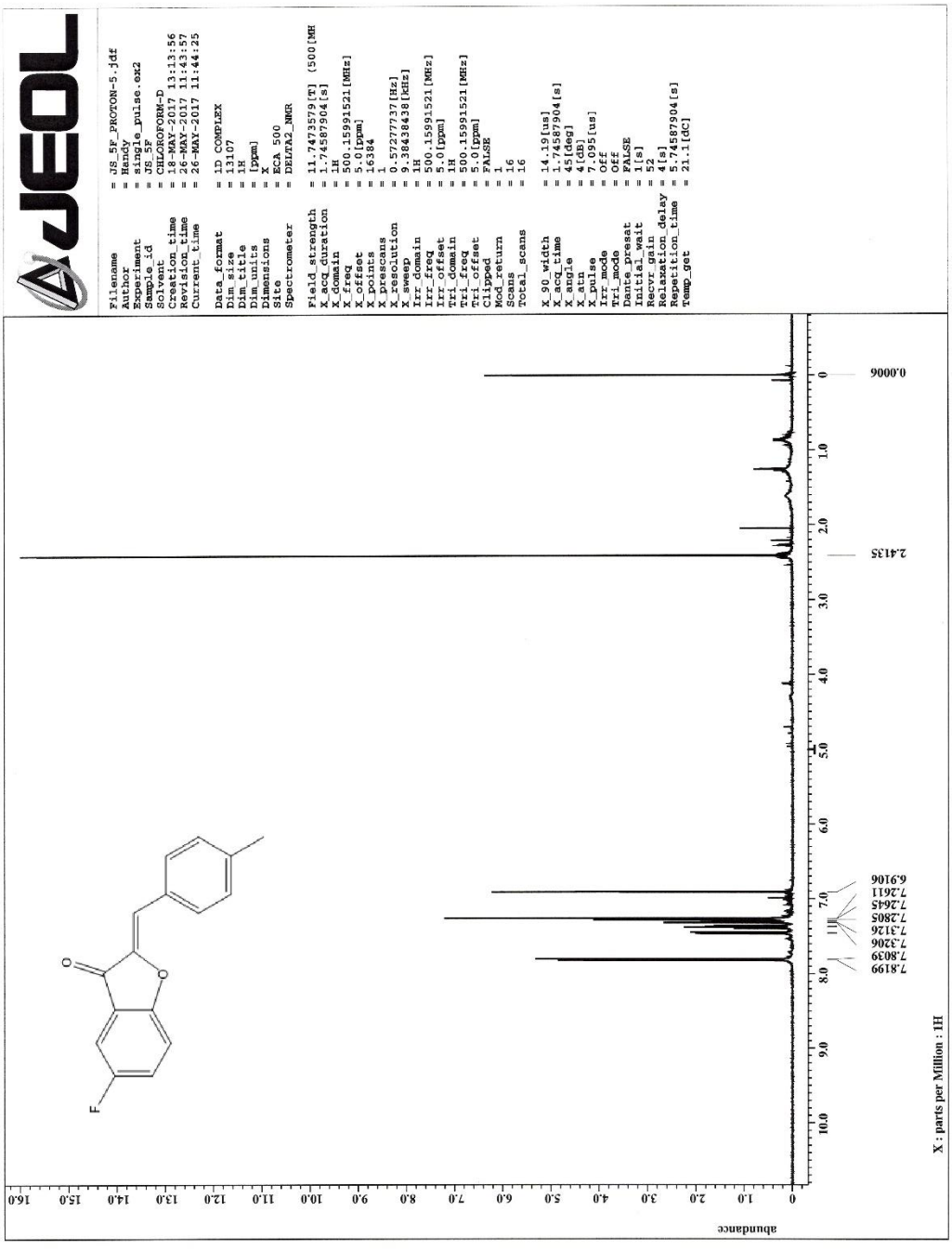
Multi-fiber fabric (Kimble 43, 13 fabric strip) was washed with warm water first then air dried. Mordanting solutions with $FeSO_4$ were made fresh at 1 g/25 mL of deionized $H_2O$ for both the pre-mordanting and post-mordanting processes. Both (**9**) (2-

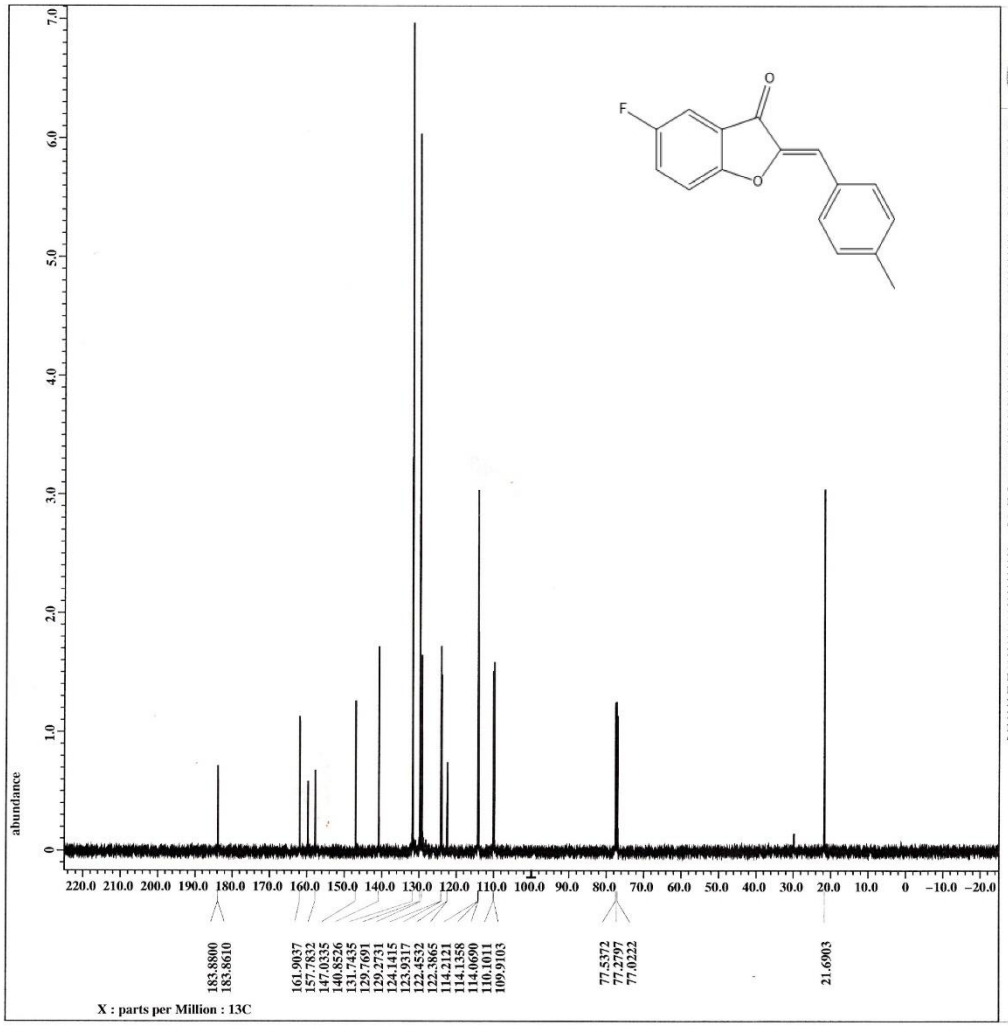
(4-methylbenzylidene)-1-(7'-bromobenzofuran-3-one)) and (**14**) (2-(4-methylbenzylidene)-1-(4'-hydroxybenzofuran-3-one)) were used as the dye samples with each undergoing pre, simultaneous, and post-mordanting processes.

## References


1. Praveena, R.; Deepha, V.; Sivakumar, R. Investigations on C-glycosyl flavonoids from Rhynchosia capitata, its antimicrobial and dyeing properties. *Fibers Polym.* **2014***, 15*, 525-533.

2-(4-methylbenzylidene)-1-(5’-fluorobenzofuran-3-one) [**2**]



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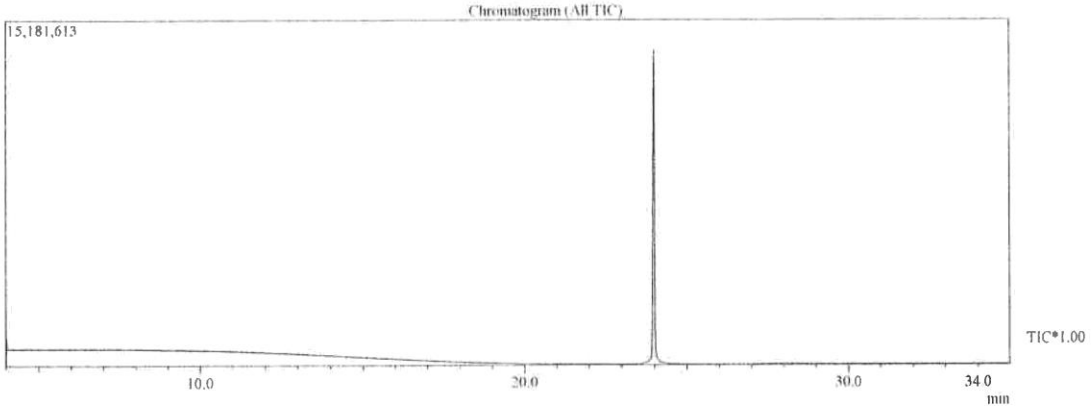
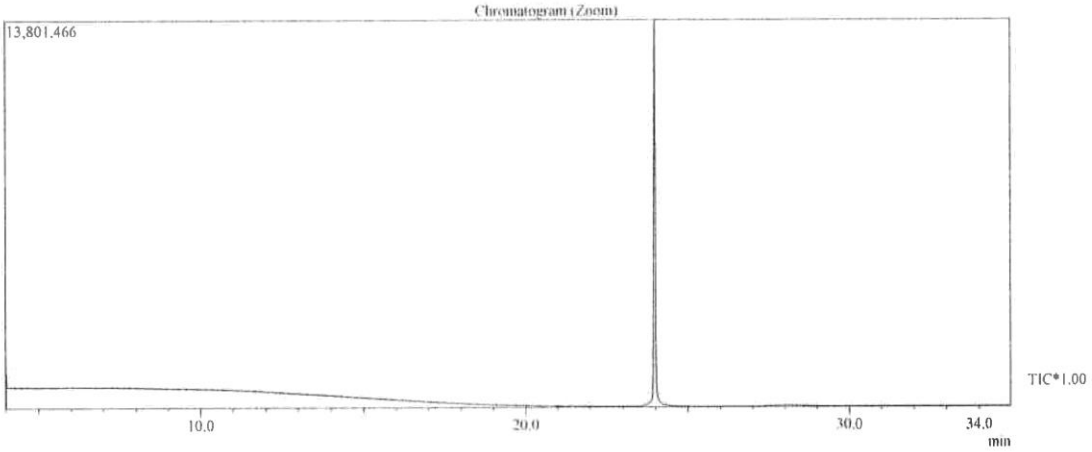
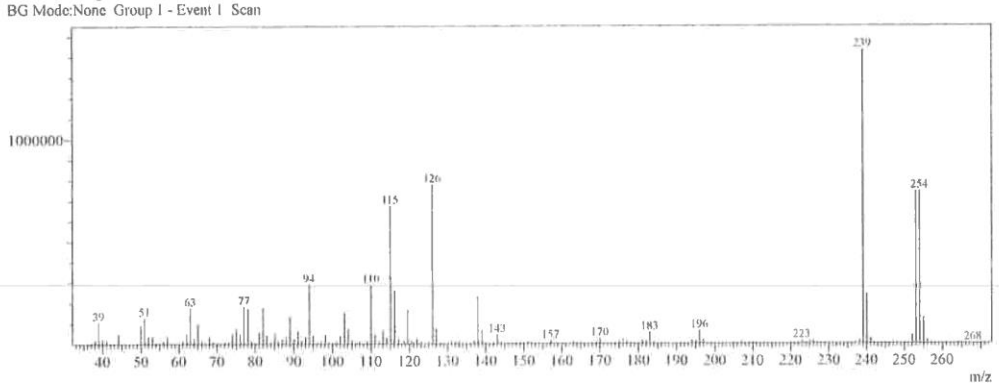
| Repetition_time | = 2.83361792[s] |
| Temp_get | = 21.3[dC] |



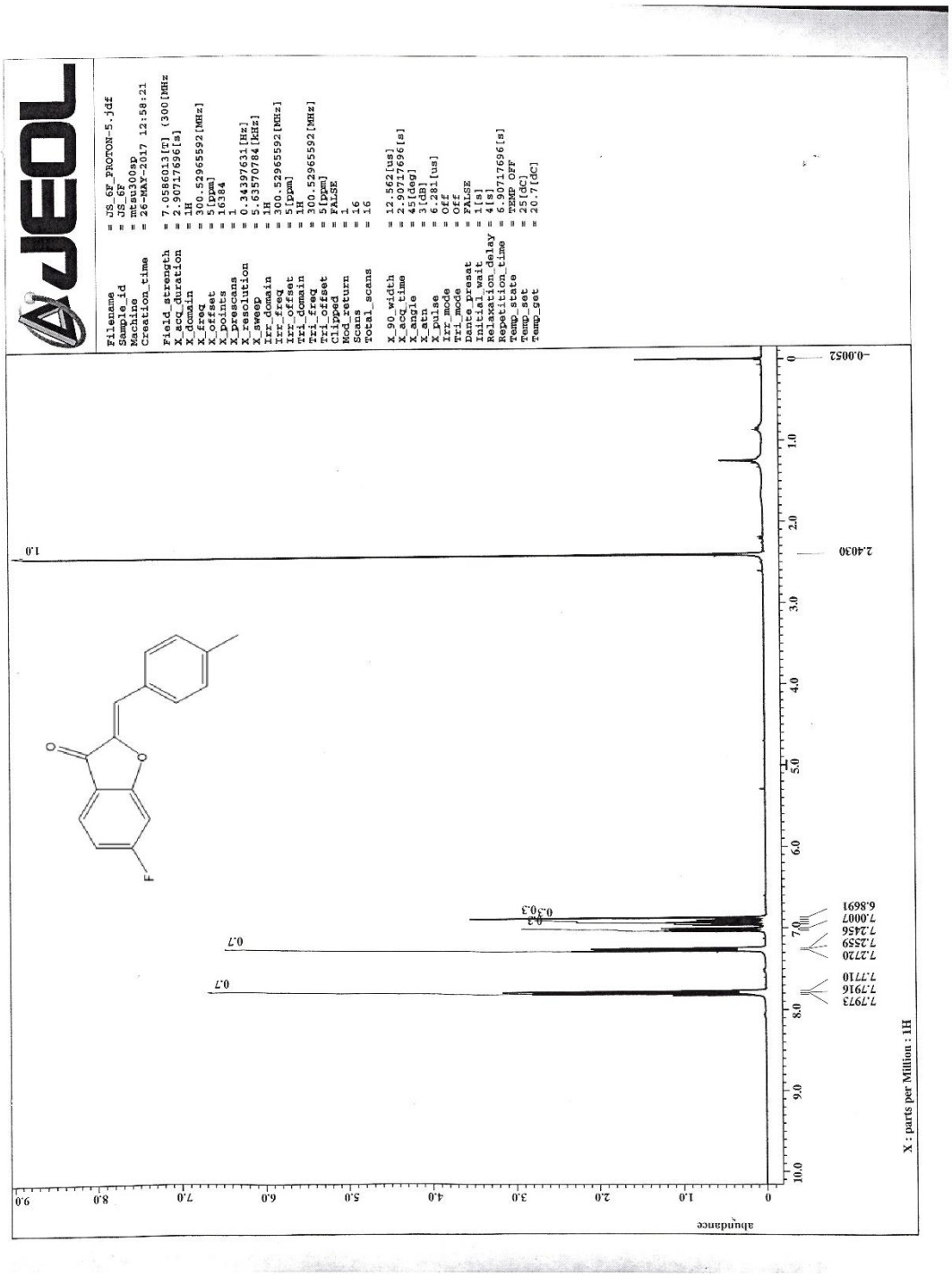



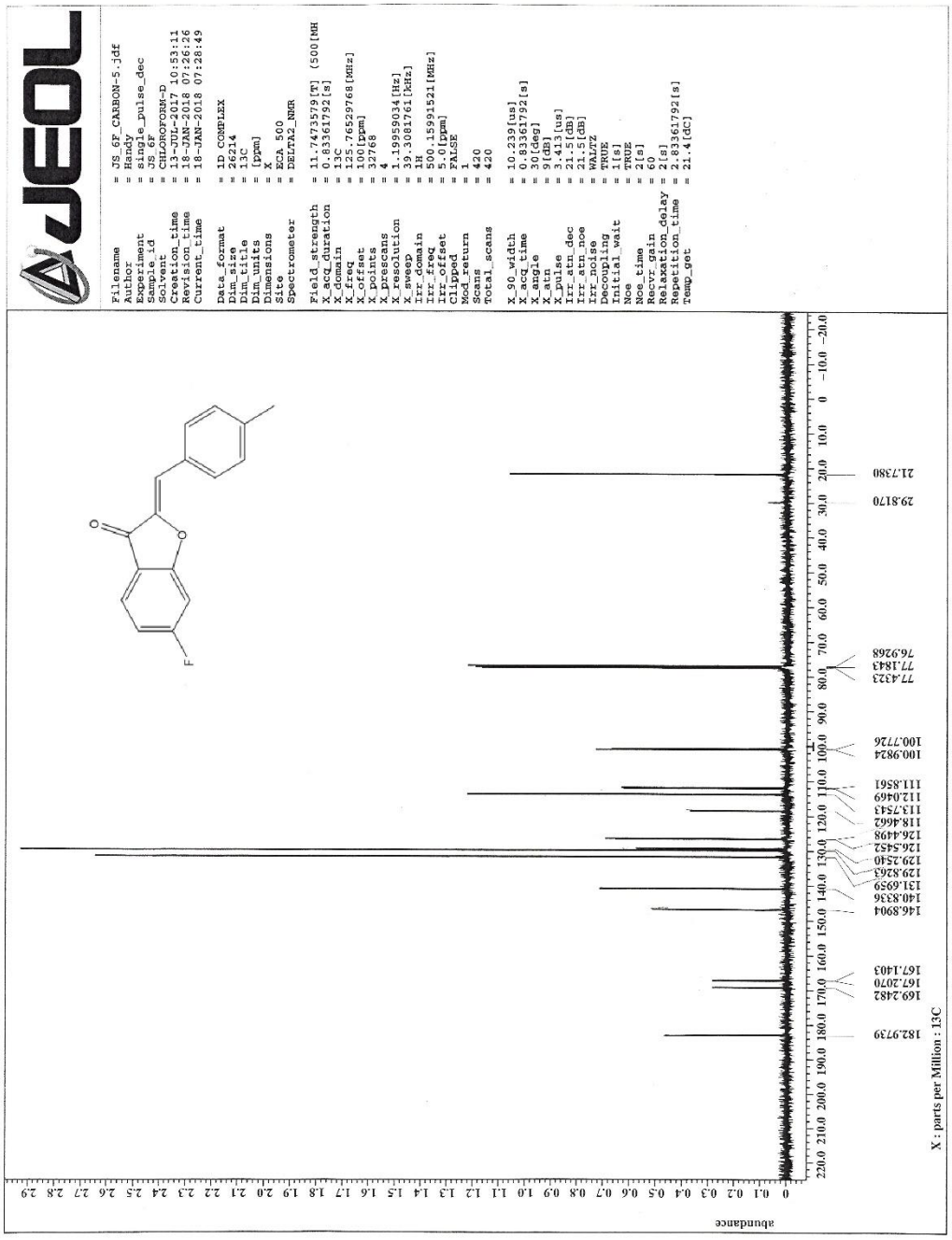

Spectrum

Line#:1 R.Time:23.975(Scan#:3996)
MassPeaks:188
RawMode:Single 23.975(3996) BasePeak:239.10(1431148)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(6’-fluorobenzofuran-3-one) [**3**]




JEOL
Filename = JS_6F_CARBON-5.jdf
Author = Handy
Experiment = single_pulse_dec
Sample_id = JS_6F
Solvent = CHLOROFORM-D
Creation_time = 13-JUL-2017 10:53:11
Revision_time = 18-JAN-2018 07:26:26
Current_time = 18-JAN-2018 07:28:49
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.74735579[T] (500[MH
X_acq_duration = 0.833661792[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 = 39.30817761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 420
Total_scans = 420
X_90_width = 10.239[us]
X_acq_time = 0.833661792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_mode = NAIUPZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recyc_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.833661792[s]
Temp_get = 21.4[dC]
O
O
F
21.7380
29.8170
76.9268
77.1843
77.4323
100.7726
100.9824
111.8561
112.0469
113.7543
118.4662
126.4498
126.5452
129.2540
129.8263
131.6959
140.8336
146.8904
167.1403
167.2070
169.2482
182.9739
abundance
X : parts per Million : 13C

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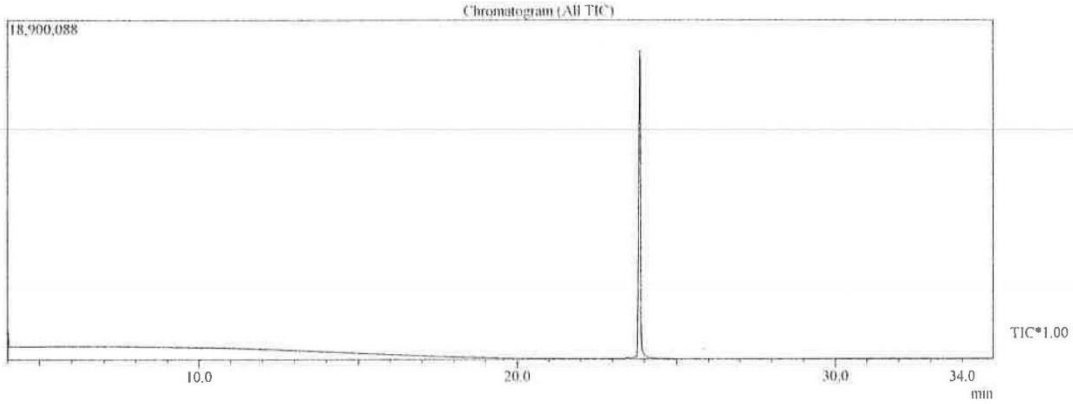
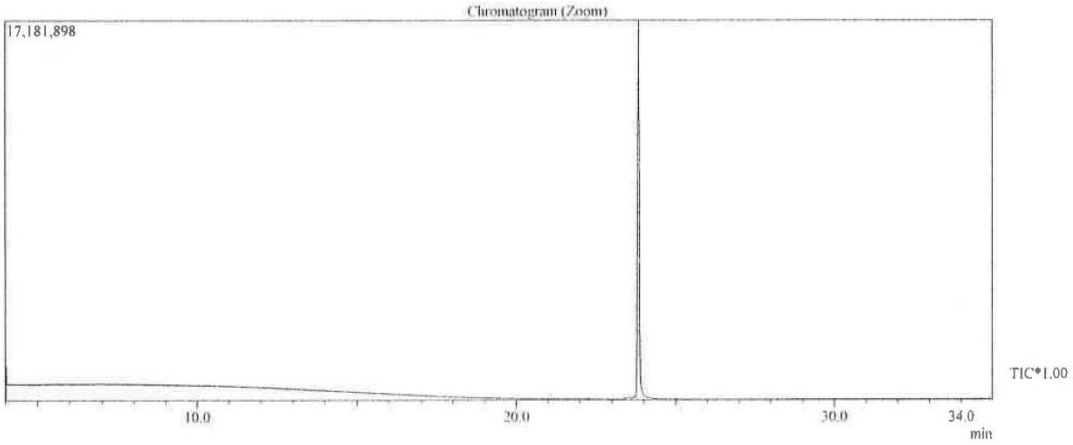
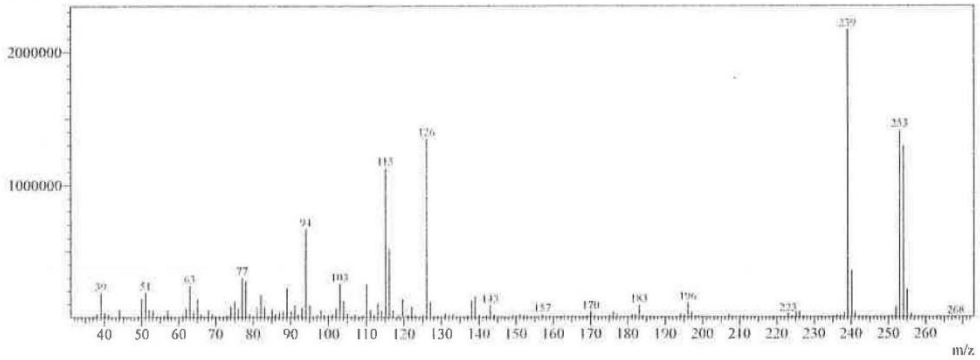


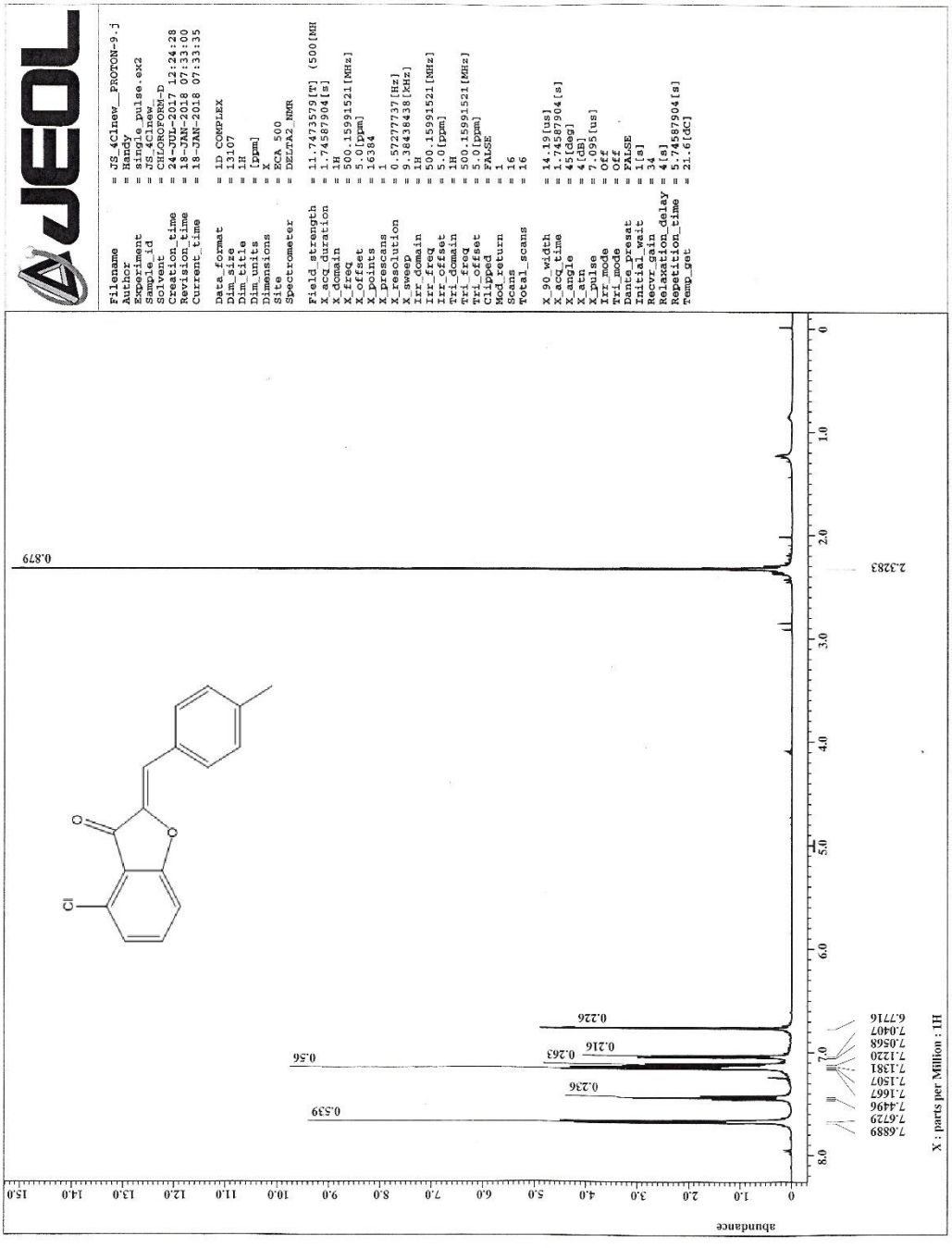



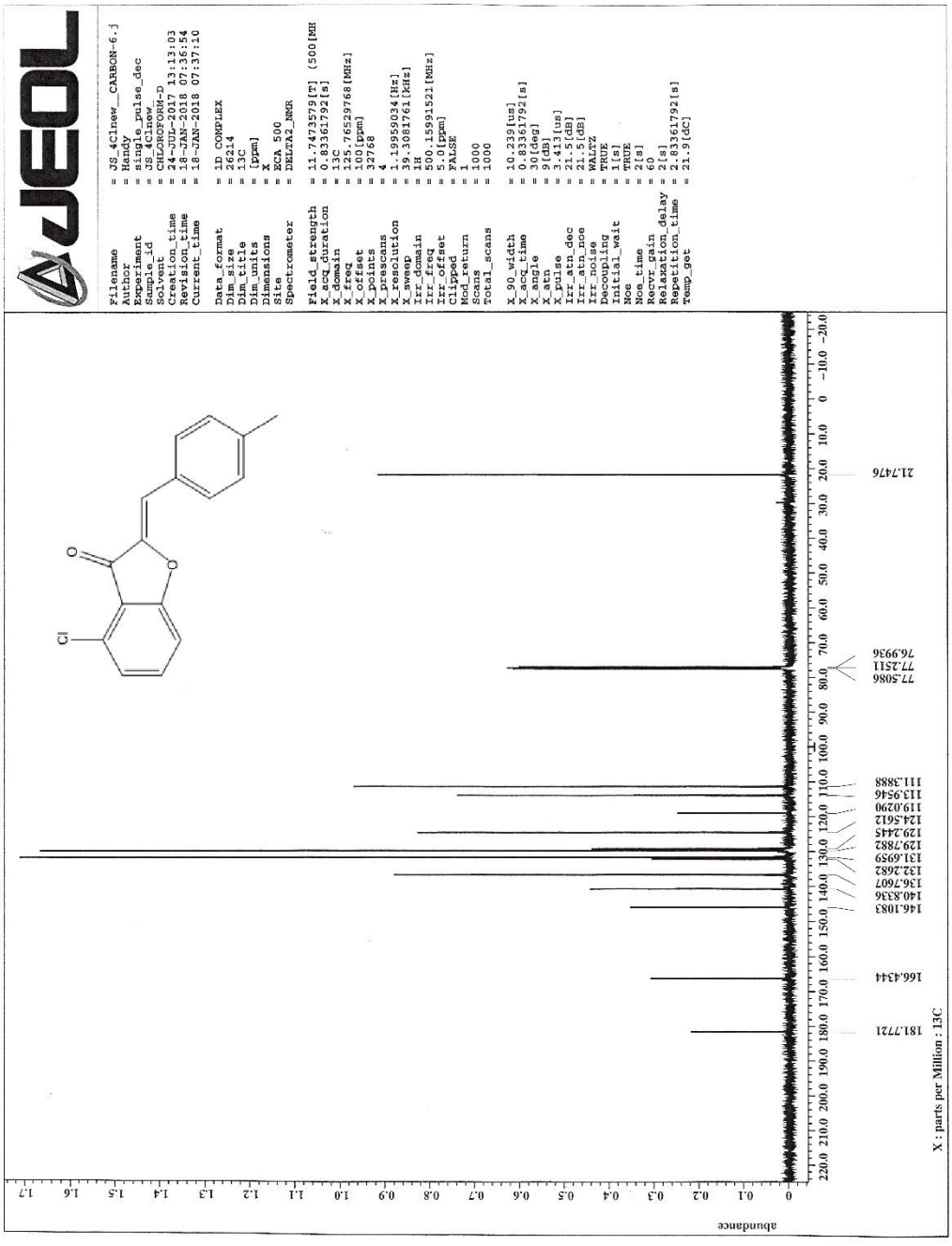

Spectrum

Line#:1 R.Time:23.865(Scan#:3974)
MassPeaks:197
RawMode:Single 23.865(3974) BasePeak:239.05(2166300)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(4’-chlorobenzofuran-3-one) [**4**]




JEOL
Filename = JS_4Clnew__CARBON-6.j
Author = Handy
Experiment = single_pulse_dec
Sample_id = 4Cl new
Solvent = CHLOROFORM-D
Creation_time = 24-JUL-2017 13:13:03
Revision_time = 18-JAN-2018 07:36:54
Current_time = 18-JAN-2018 07:37:10
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.74735791[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100.0[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
Model = 1
Scans = 1000
Total_scans = 1000
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[db]
X_pulse = 3.41[us]
X_pulse_dec = 21.5[dB]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = WALTZ
Irr_noise = TRUE
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recyr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 23.13361792[s]
Temp_get = 21.9[dC]
O
O
Cl
abundance
X : parts per Million : 13C
181.7721
166.4344
146.1083
140.8336
136.7607
132.2682
131.6959
129.7882
129.2445
124.5612
119.0290
113.9546
111.3888
77.5086
77.2511
76.9936
21.7476

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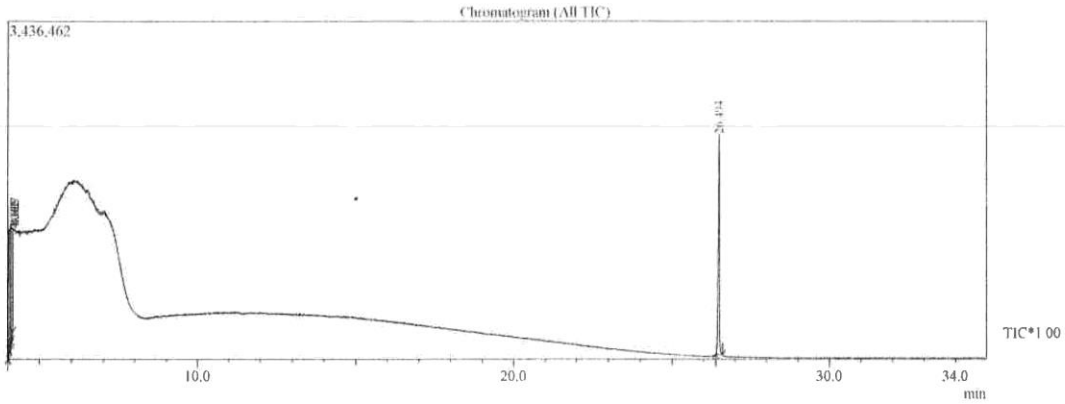
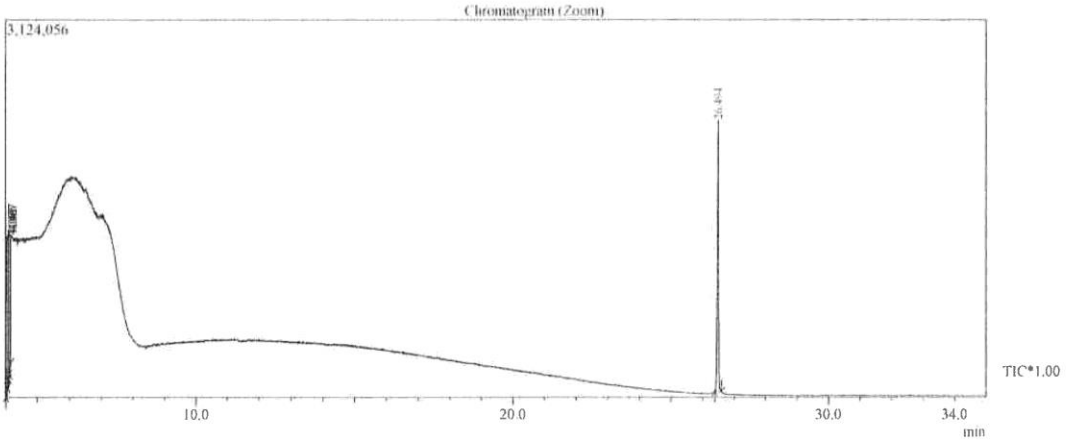
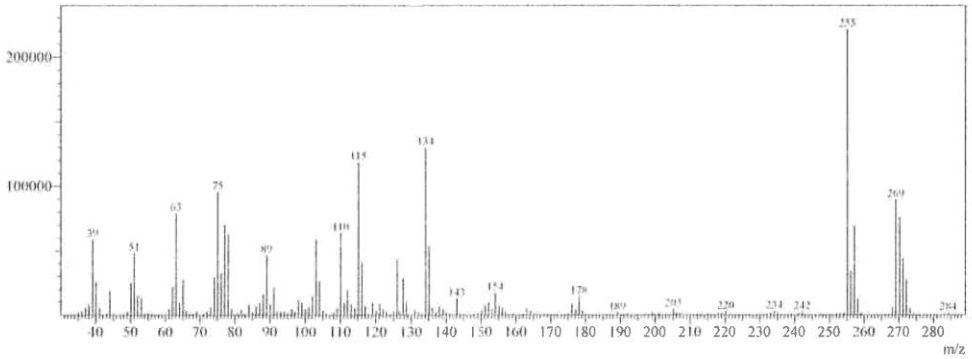


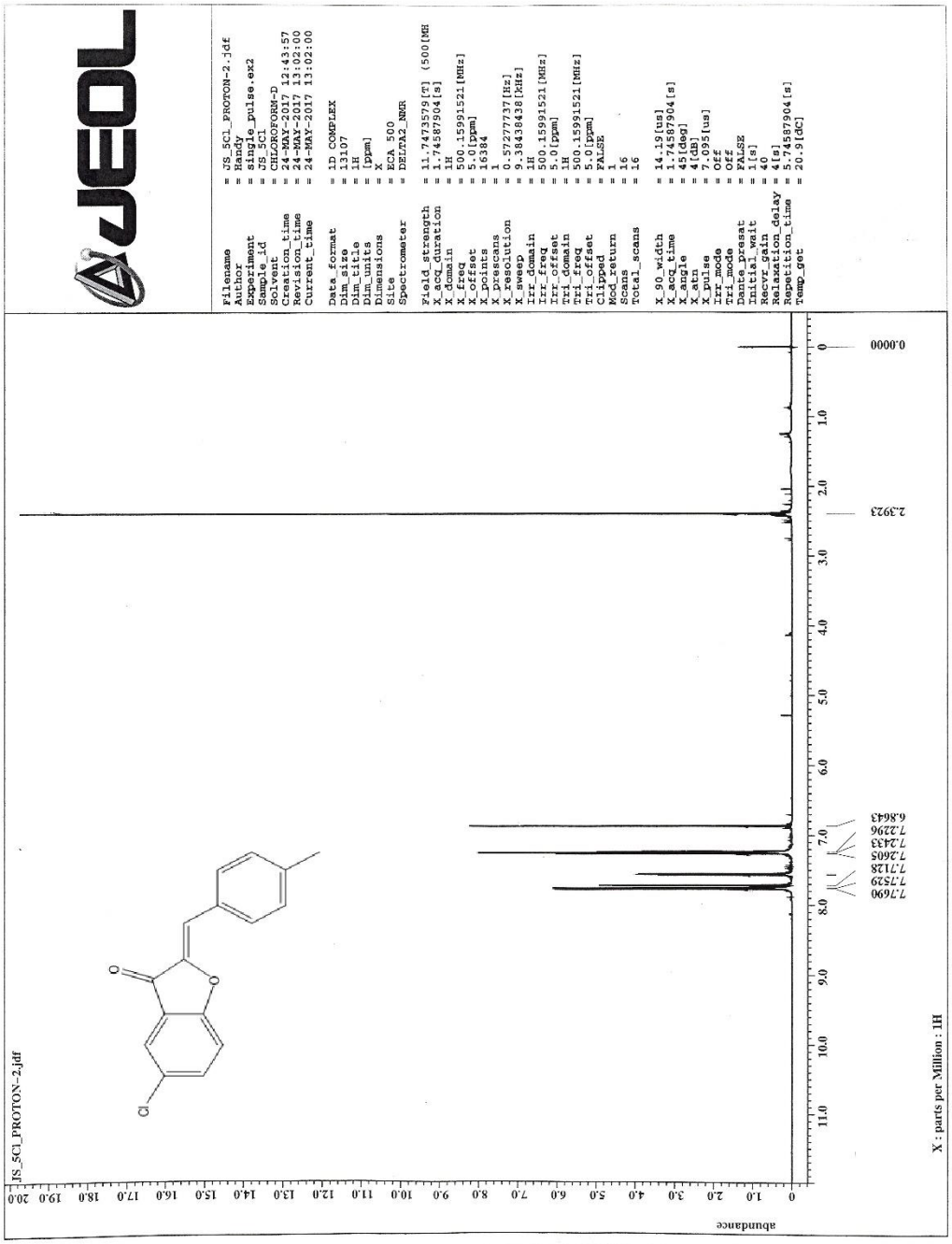



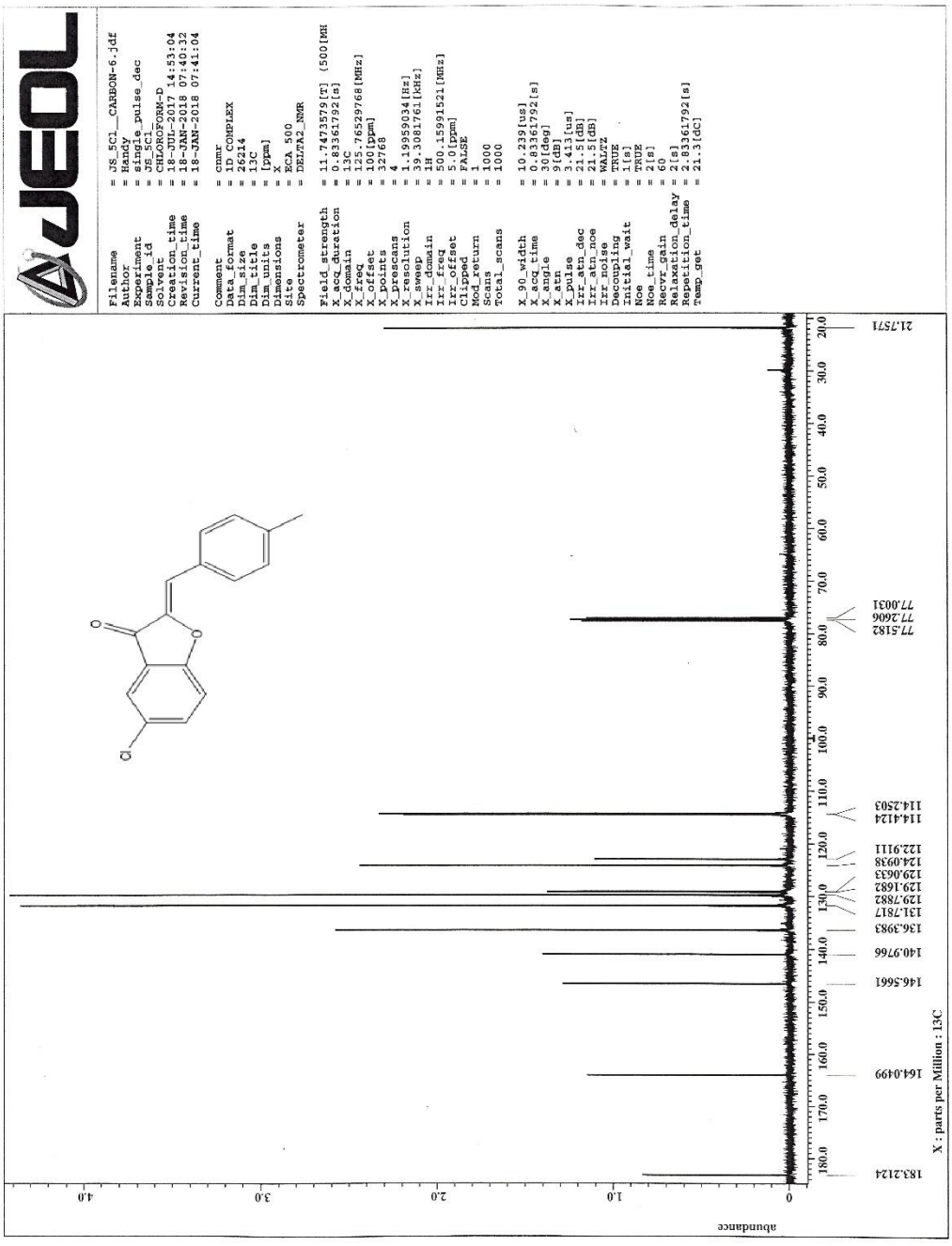

Spectrum

Line#:1 R.Time:26.495(Scan#:4500)
MassPeaks:156
RawMode:Single 26.495(4500) BasePeak:255.15(221250)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(5’-chlorobenzofuran-3-one) [**5**]




JEOL
Filename = JS_5Cl__CARBON-6.jdf
Author = Handy
Experiment = single_pulse_dec
Sample_id = JS_5Cl
Solvent = CHLOROFORM-D
Creation_time = 18-JUL-2017 14:53:04
Revision_time = 18-JAN-2018 07:40:32
Current_time = 18-JAN-2018 07:41:04
Comment = cmnr
Data format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.74735791[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.30817611[kHz]
Irr_domain = 1H
Irr_freq = 500.159931521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 1000
Total_scans = 1000
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_noise = WALTZ2
Decoupling = TRUE
Initial_wait = 1[s]
Mode = SUM
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 21.3[dC]
O
O
Cl
21.7571
77.5182
77.2606
77.0031
114.4124
114.2503
122.9111
124.0938
129.0633
129.1682
129.7882
131.7817
136.3983
140.9766
146.5661
164.0199
183.2124
abundance
X : parts per Million : 13C

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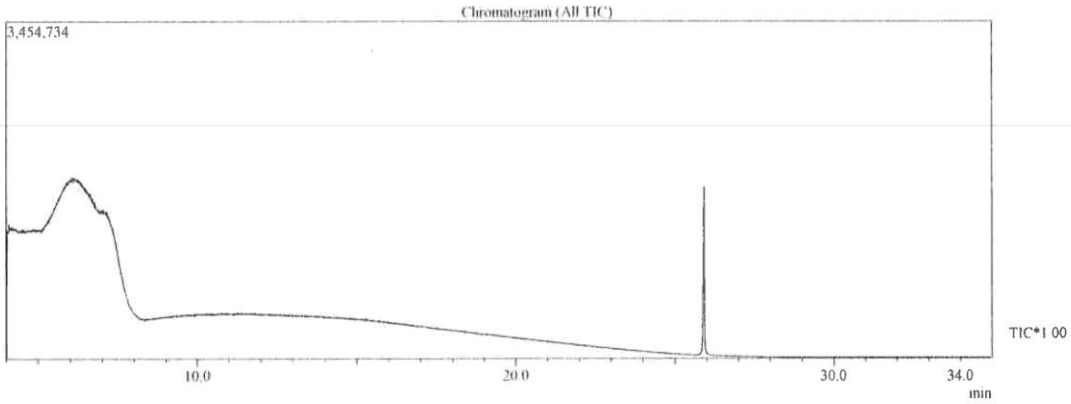
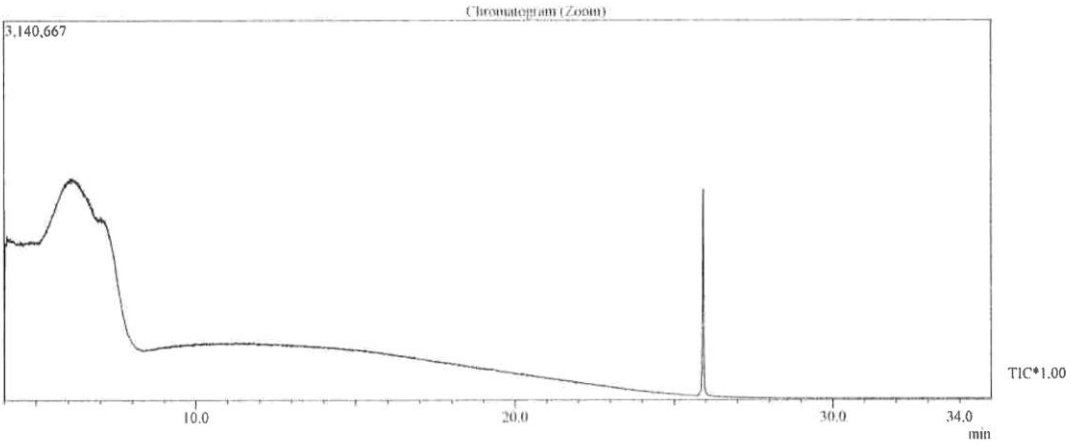
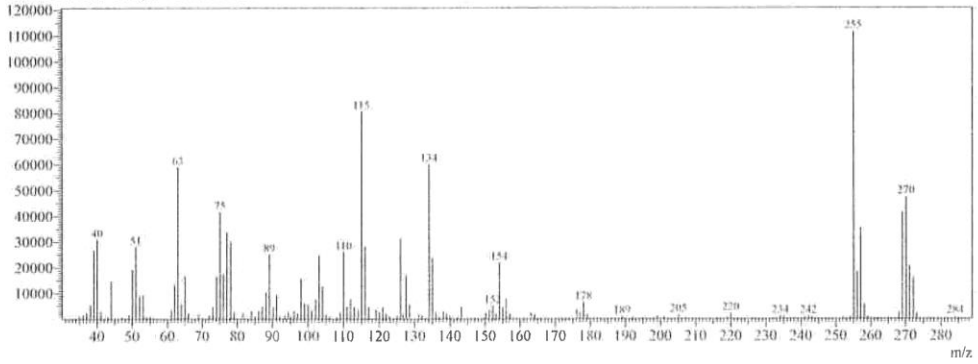


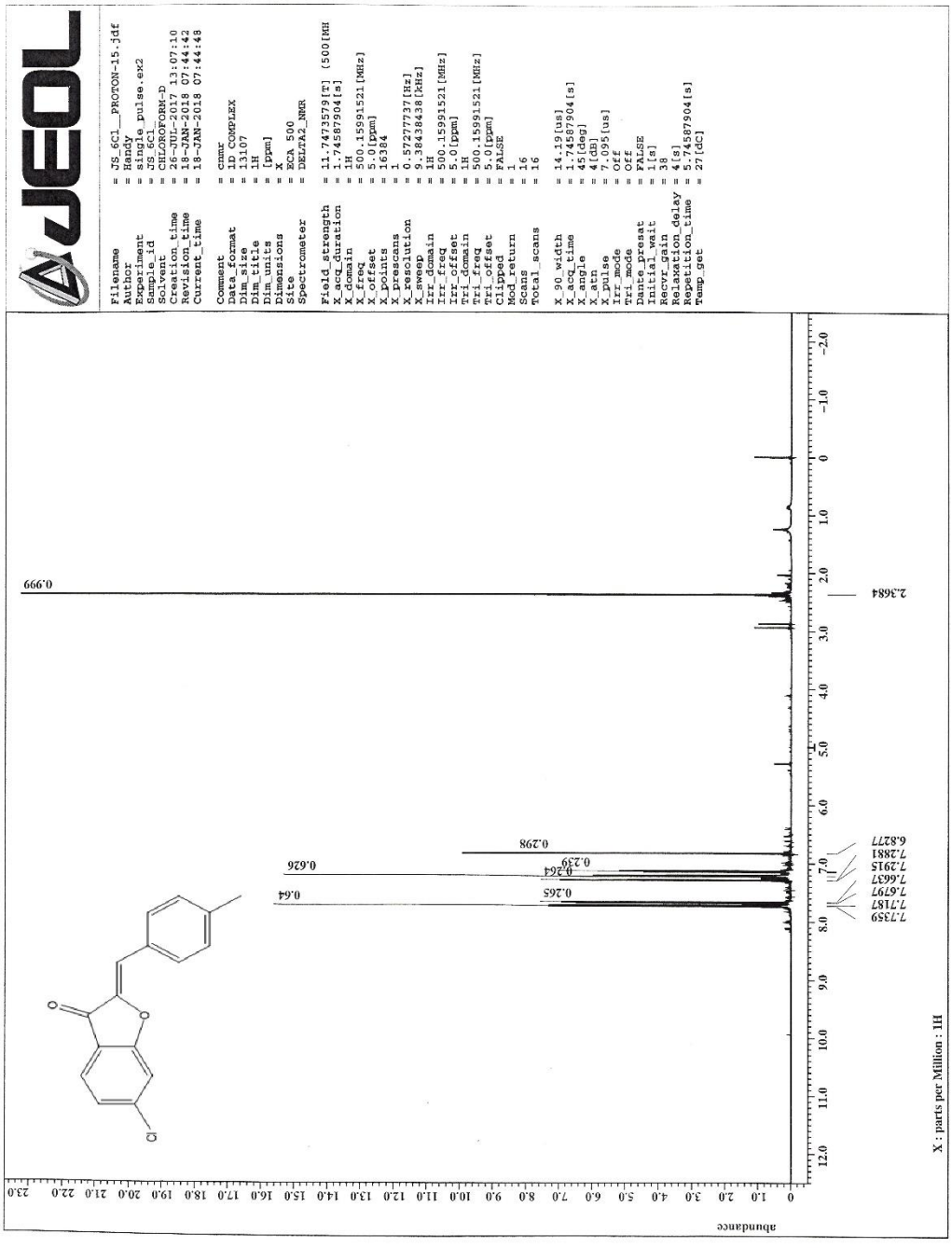



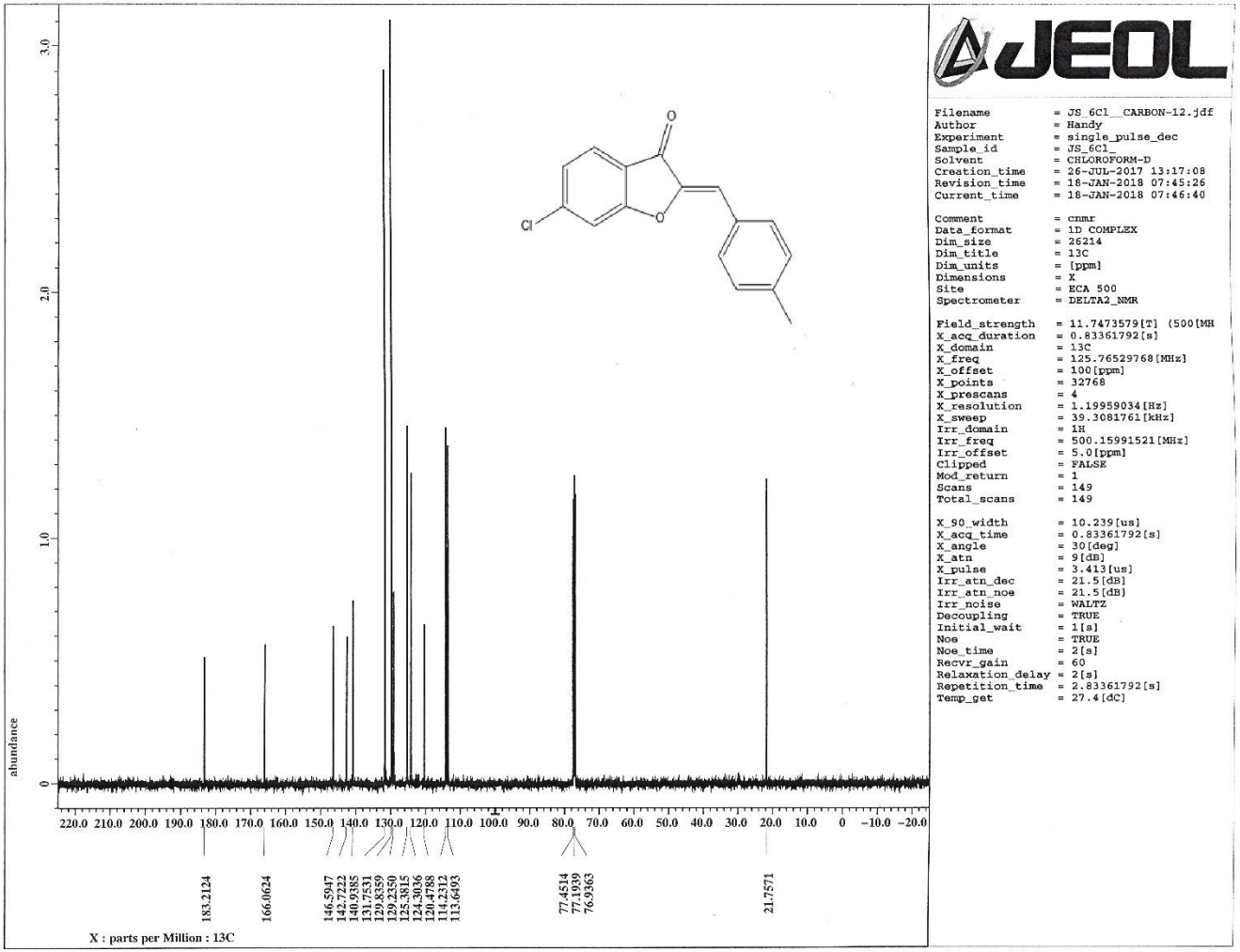

Spectrum

Line#:1 R.Time:25.910(Scan#:4383)
MassPeaks:128
RawMode:Single 25.910(4383) BasePeak:255.10(111482)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(6’-chlorobenzofuran-3-one) [**6**]




abundance
3.0
2.0
1.0
0
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
183.2124
166.0624
146.5947
142.7222
140.9385
131.7531
129.8359
129.2350
125.3815
124.3036
120.4788
114.2312
113.6493
77.4514
77.1939
76.9363
21.7571
X : parts per Million : 13C
O
Cl
O
JEOL
Filename = JS_6Cl__CARBON-12.jdf
Author = Handy
Experiment = single_pulse_dec
Sample_id = JS_6Cl_
Solvent = CHLOROFORM-D
Creation_time = 26-JUL-2017 13:17:08
Revision_time = 18-JAN-2018 07:45:26
Current_time = 18-JAN-2018 07:46:40
Comment = cnmr
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 = 149
Total_scans = 149
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 27.4[dC]

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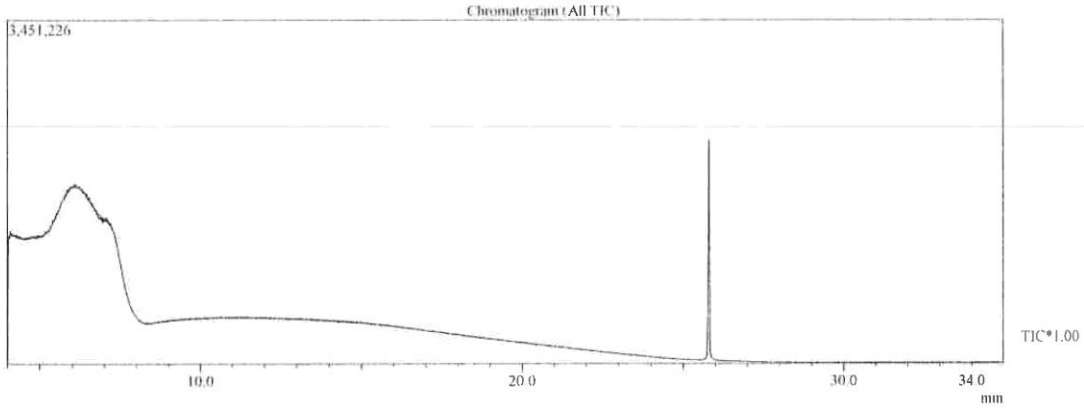
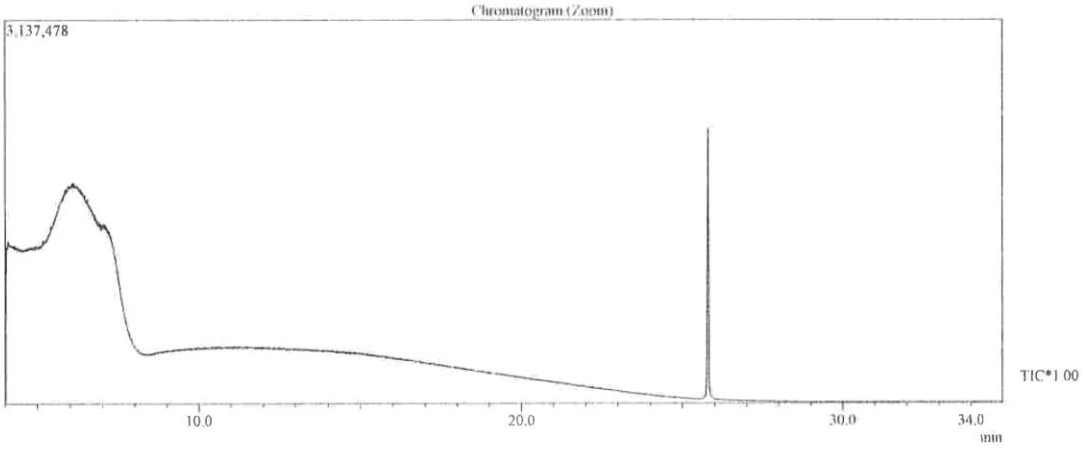
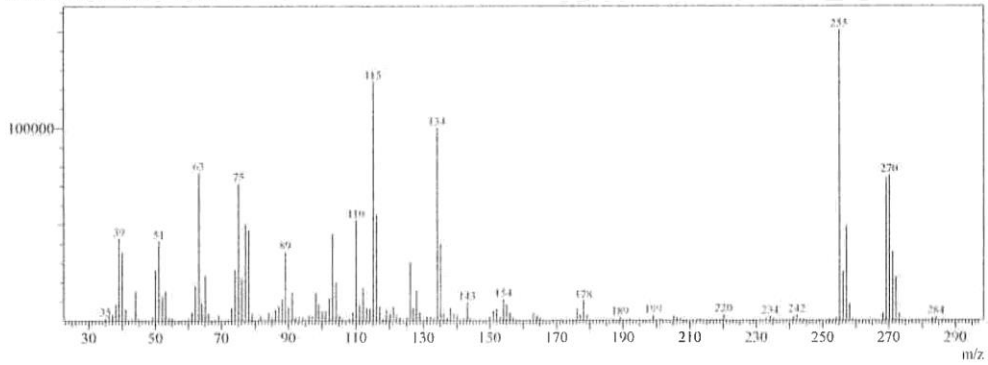


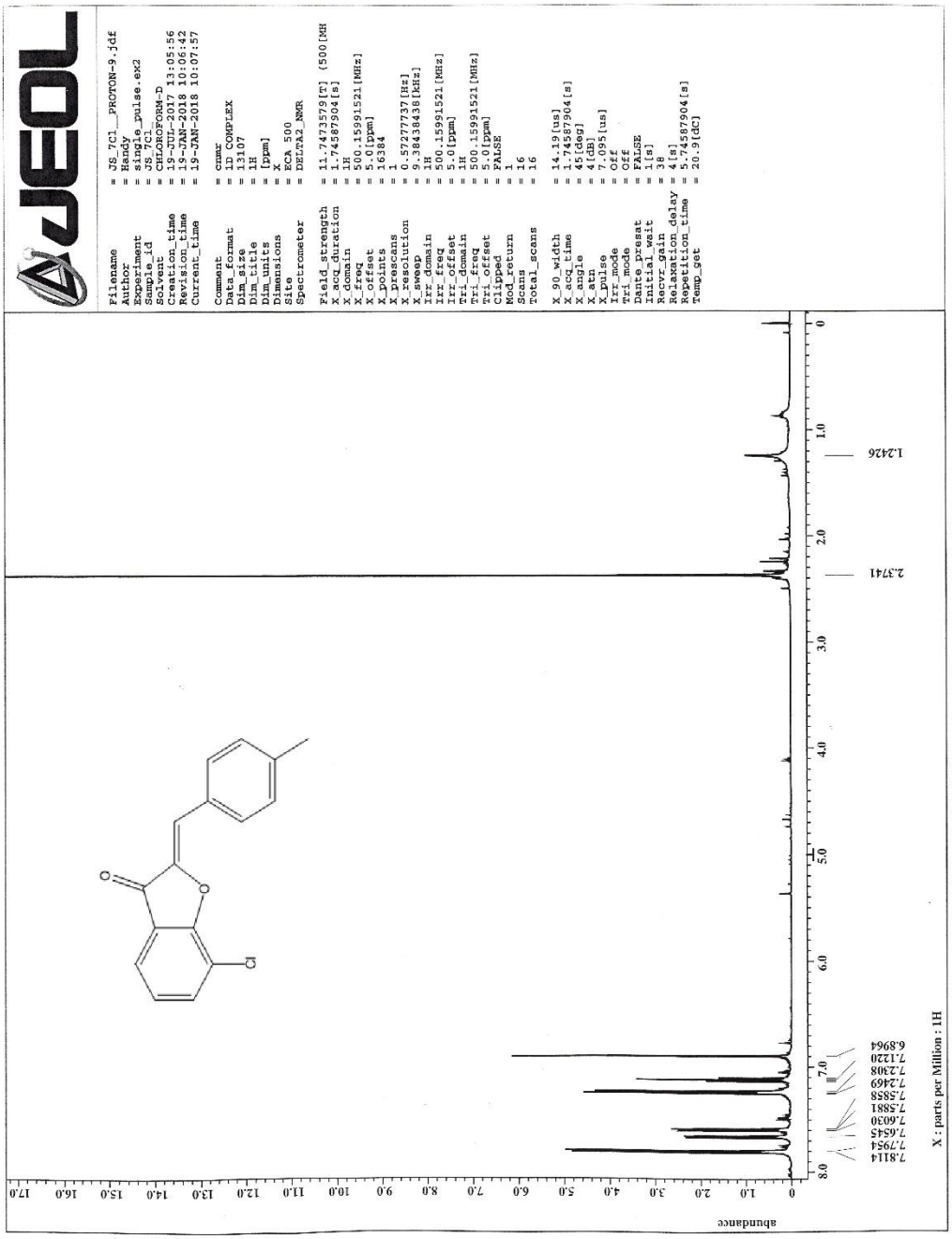



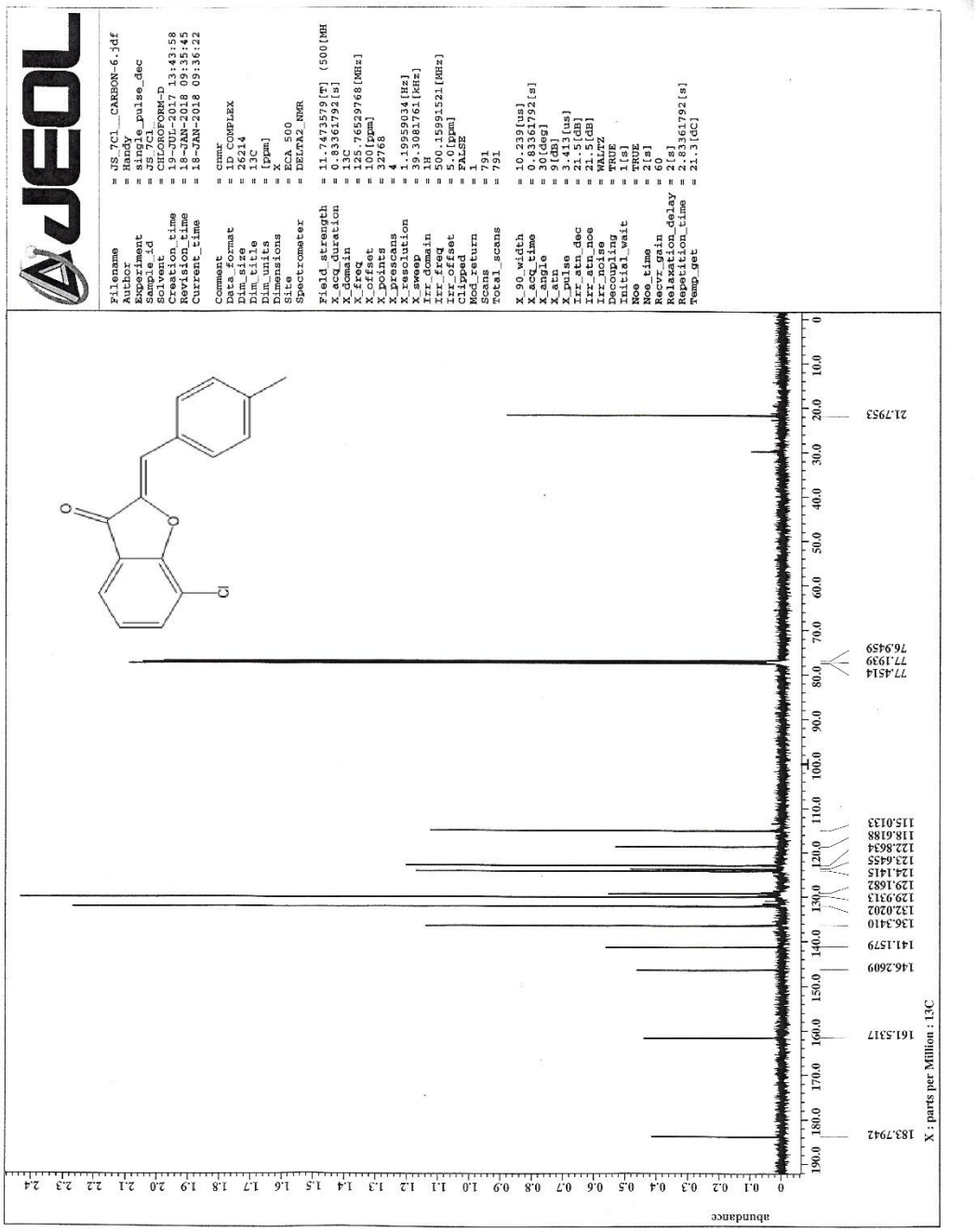

Spectrum

Line#:1 R.Time:25.800(Scan#:4361)
MassPeaks:147
RawMode:Single 25.800(4361) BasePeak:255.10(150625)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(7’-chlorobenzofuran-3-one) [**7**]




JEOL
Filename = JS_7Cl__CARBON-6.jdf
Author = Handy
Experiment = single_pulse_dec
Sample_id = JS_7Cl
Solvent = CHLOROFORM-D
Creation_time = 19-JUL-2017 13:43:58
Revision_time = 18-JAN-2018 09:35:45
Current_time = 18-JAN-2018 09:36:22
Comment = cmnr
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 = 1.19559034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 791
Scans = 791
Total_scans = 791
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Rcvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 22.3[dC]
Cl
O
O
21.7953
76.9459
77.1939
77.4514
115.0133
118.6188
122.8634
123.6455
124.1415
129.1682
129.9313
132.0202
136.3410
141.1579
146.2609
161.5317
183.7942
X : parts per Million : 13C
abundance

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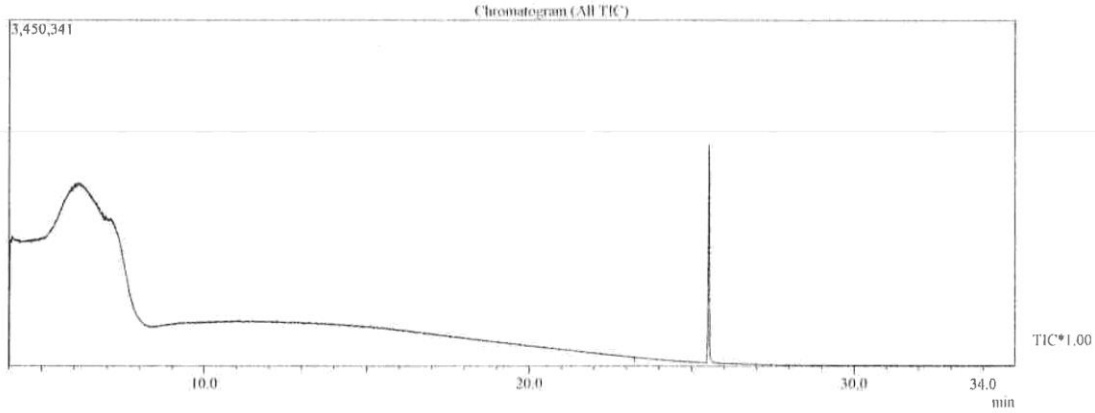
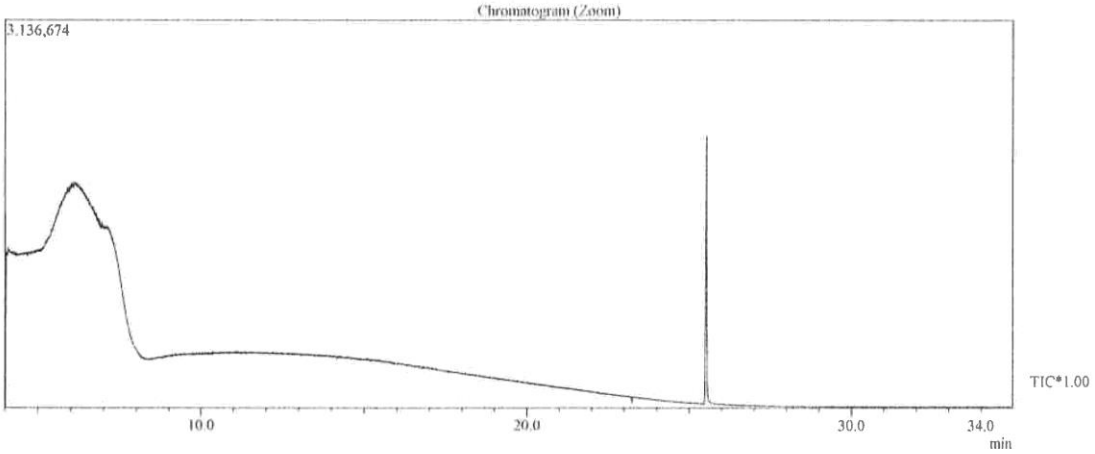
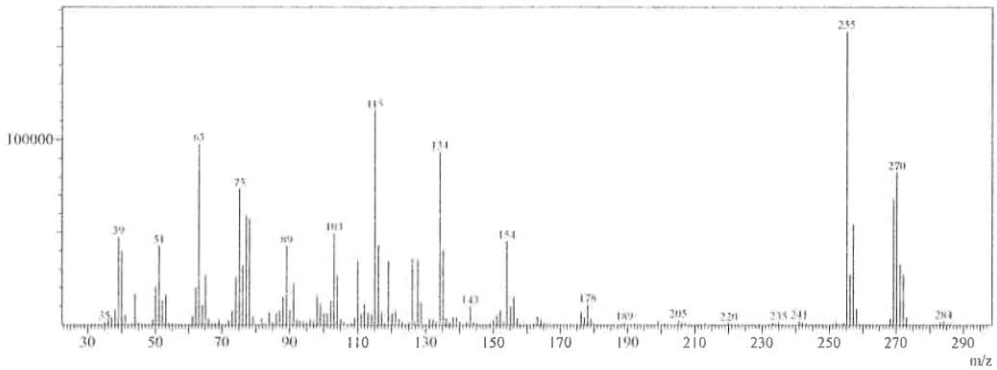


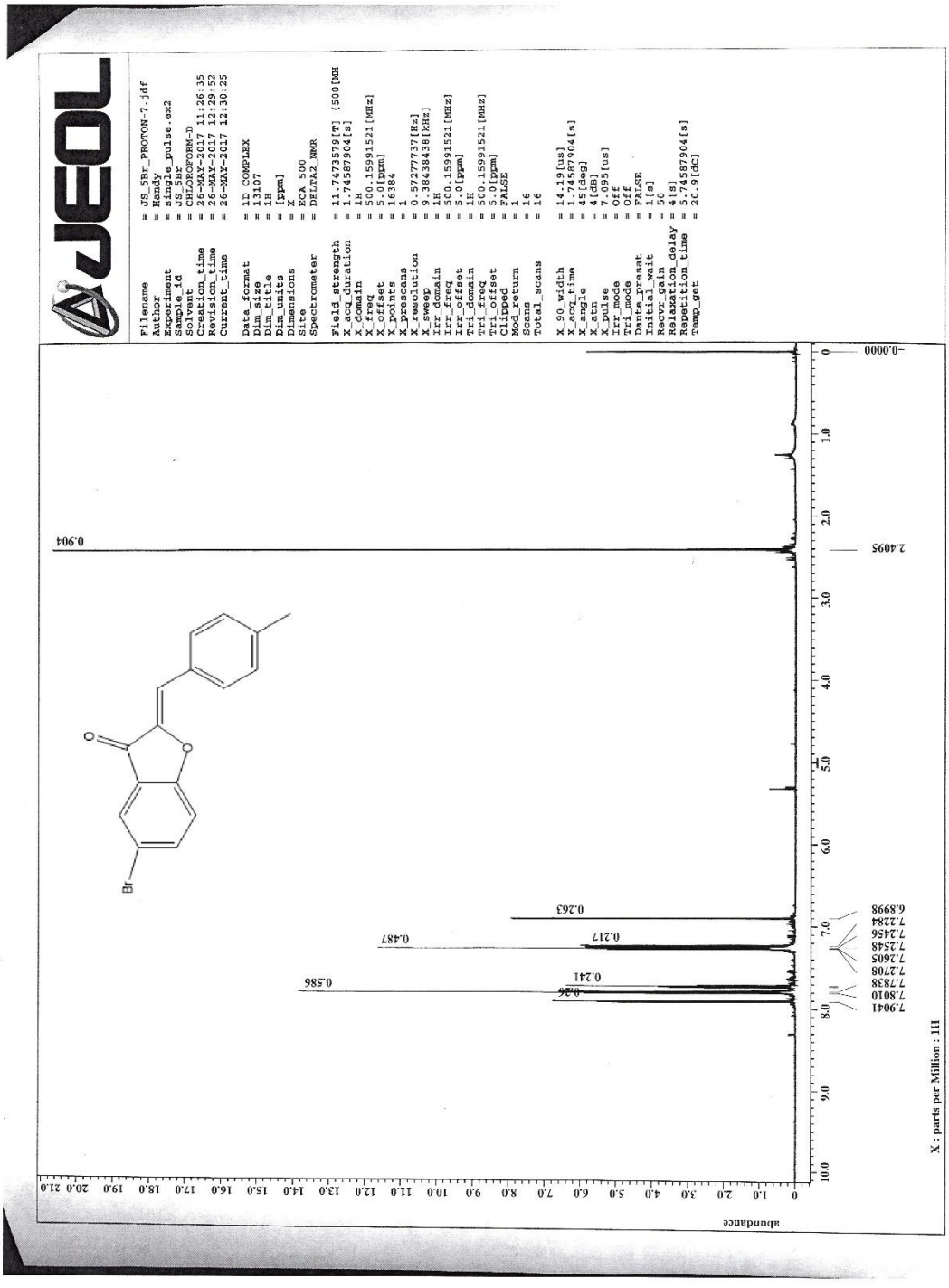



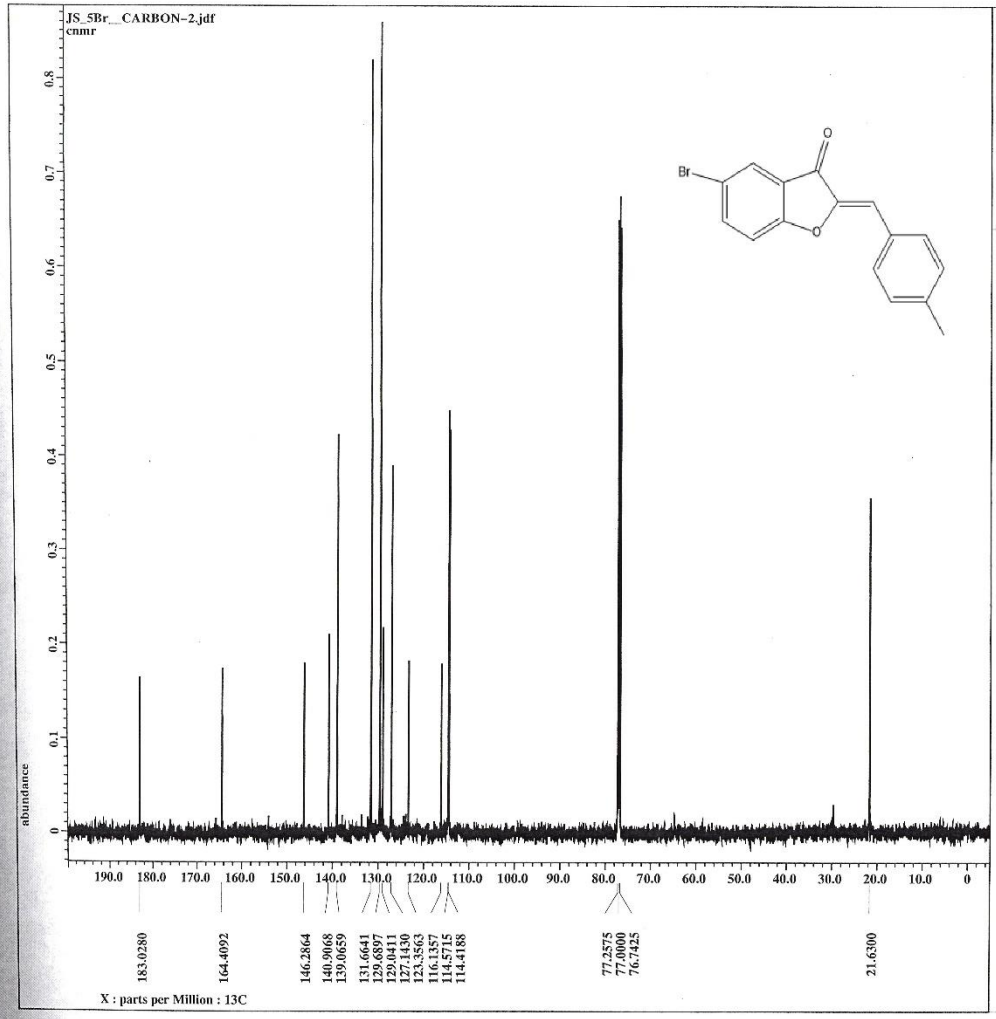

Spectrum

Line#:1 R.Time:25 530(Scan#:4307)
MassPeaks:145
RawMode:Single 25.530(4307) BasePeak:255.15(158383)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(5’-bromobenzofuran-3-one) [**8**]






JEOL

| | |
|---|---|
| Filename | = JS_5Br__CARBON-2.jdf |
| Author | = Handy |
| Experiment | = single_pulse_dec |
| Sample_id | = JS_5Br_ |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 20-JUL-2017 10:13:05 |
| Revision_time | = 20-JUL-2017 10:32:16 |
| Current_time | = 20-JUL-2017 10:32:16 |
| Comment | = cnmr |
| 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 | = 136 |
| Total_scans | = 136 |
| X_90_width | = 10.239[us] |
| X_acq_time | = 0.83361792[s] |
| X_angle | = 30[deg] |
| X_atn | = 9[dB] |
| X_pulse | = 3.413[us] |
| Irr_atn_dec | = 21.5[dB] |
| Irr_atn_noe | = 21.5[dB] |
| Irr_noise | = WALTZ |
| Decoupling | = TRUE |
| Initial_wait | = 1[s] |
| Noe | = TRUE |
| Noe_time | = 2[s] |
| Recvr_gain | = 60 |
| Relaxation_delay | = 2[s] |
| Repetition_time | = 2.83361792[s] |
| Temp_get | = 22.3[dC] |

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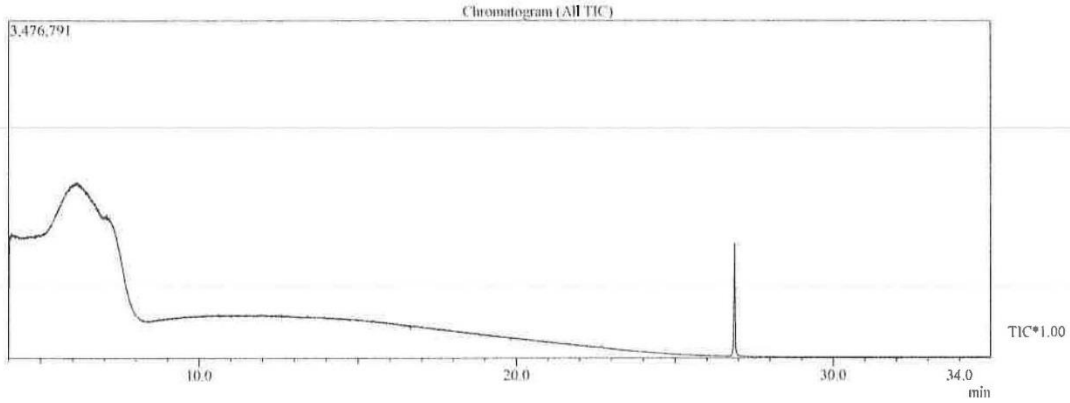
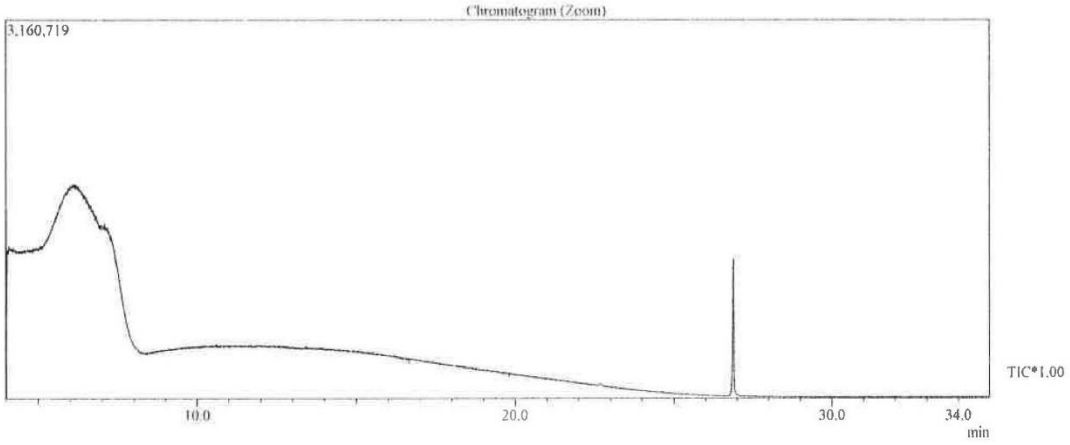
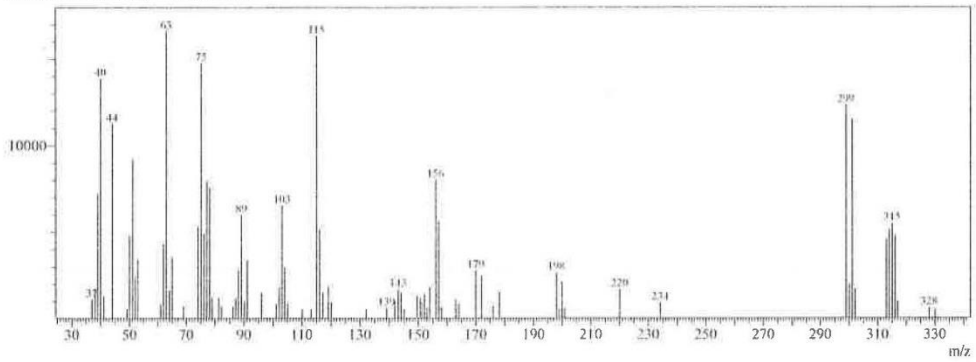


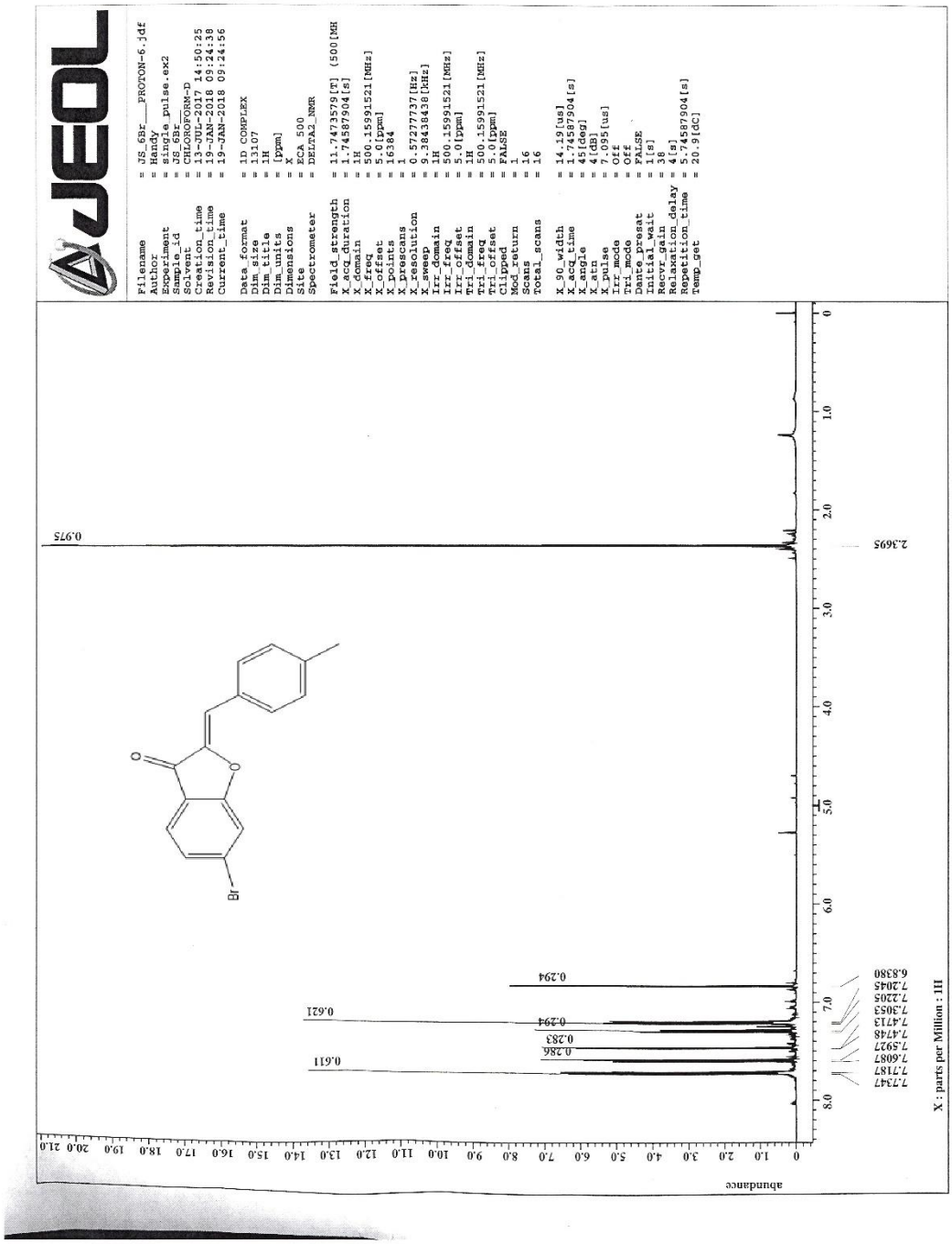



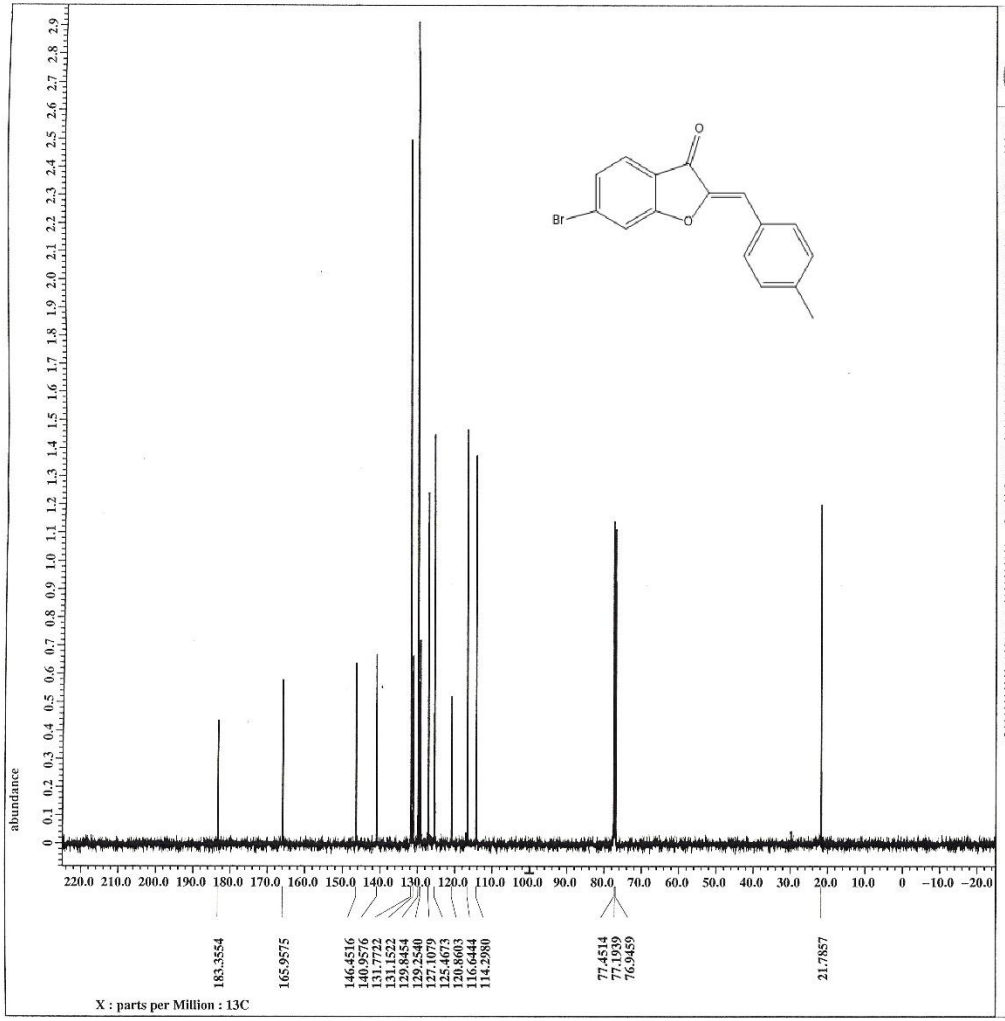

**Spectrum**

Line#:1 R.Time:26.900(Scan#:4581)
MassPeaks:82
RawMode:Single 26.900(4581) BasePeak:63.00(16604)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(6’-bromobenzofuran-3-one) [**9**]






JEOL

| | |
|---|---|
| Filename | = JS_6Br___CARBON-5.jdf |
| Author | = Handy |
| Experiment | = single_pulse_dec |
| Sample_id | = JS_6Br__ |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 13-JUL-2017 15:04:52 |
| Revision_time | = 19-JAN-2018 09:25:22 |
| Current_time | = 19-JAN-2018 09:25:51 |
| 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 | = 292 |
| Total_scans | = 292 |
| X_90_width | = 10.239[us] |
| X_acq_time | = 0.83361792[s] |
| X_angle | = 30[deg] |
| X_atn | = 9[dB] |
| X_pulse | = 3.413[us] |
| Irr_atn_dec | = 21.5[dB] |
| Irr_atn_noe | = 21.5[dB] |
| Irr_noise | = WALTZ |
| Decoupling | = TRUE |
| Initial_wait | = 1[s] |
| Noe | = TRUE |
| Noe_time | = 2[s] |
| Recvr_gain | = 60 |
| Relaxation_delay | = 2[s] |
| Repetition_time | = 2.83361792[s] |
| Temp_get | = 21.3[dC] |

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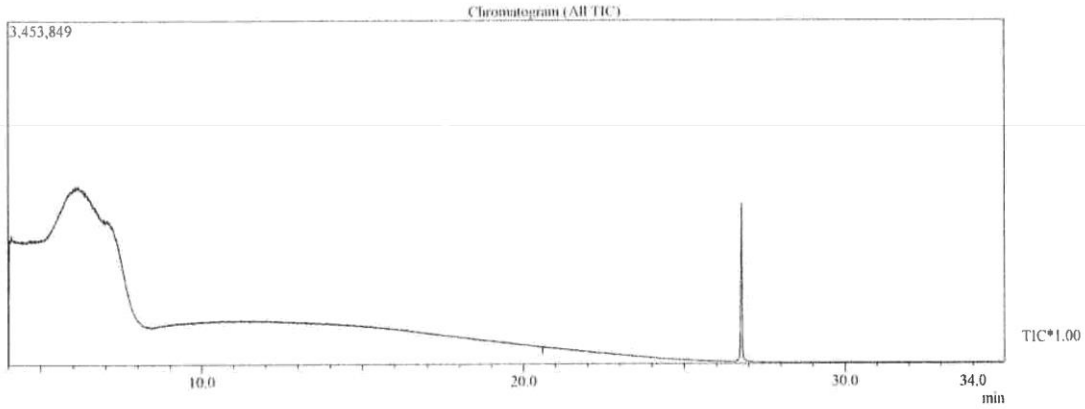
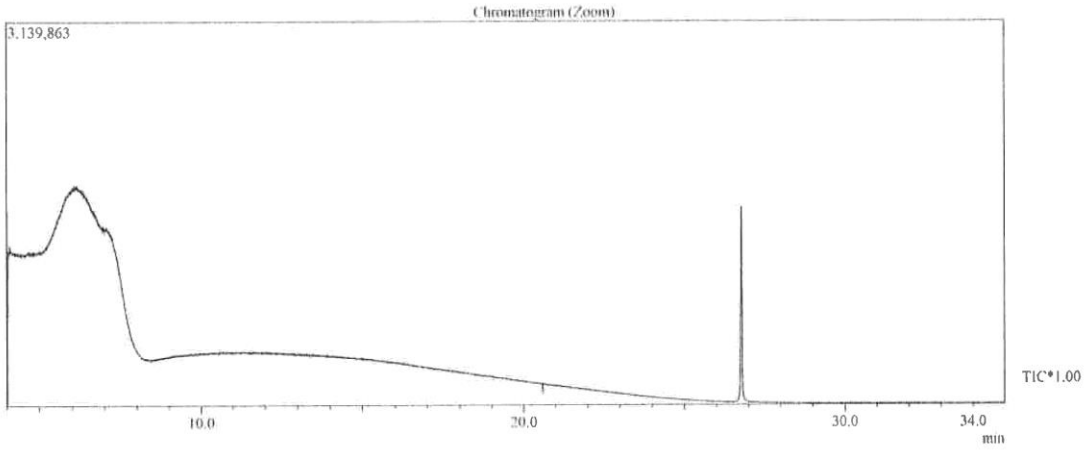
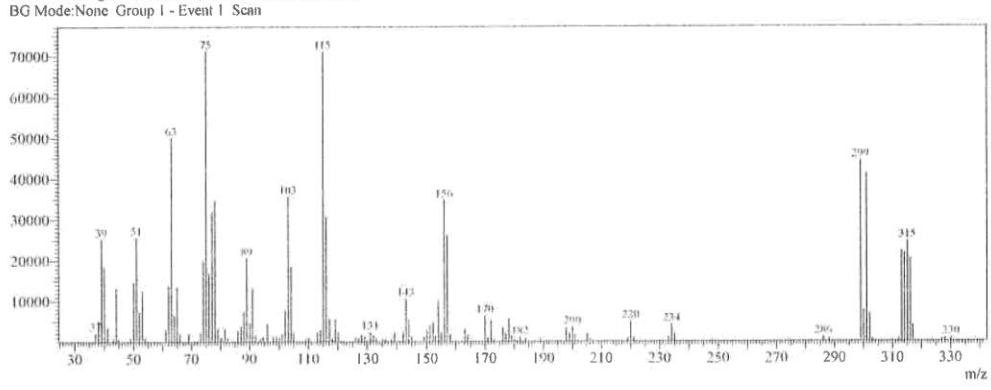


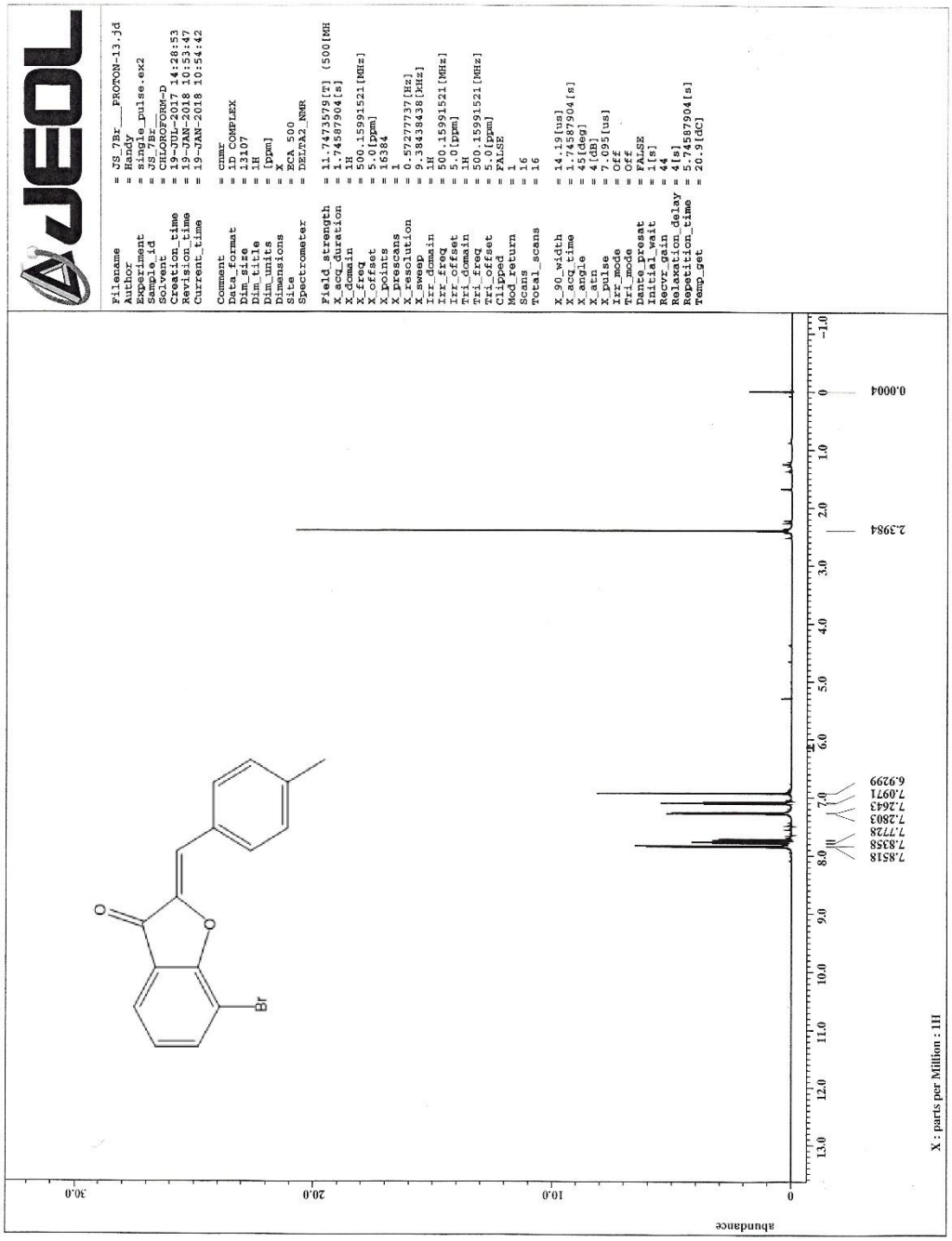



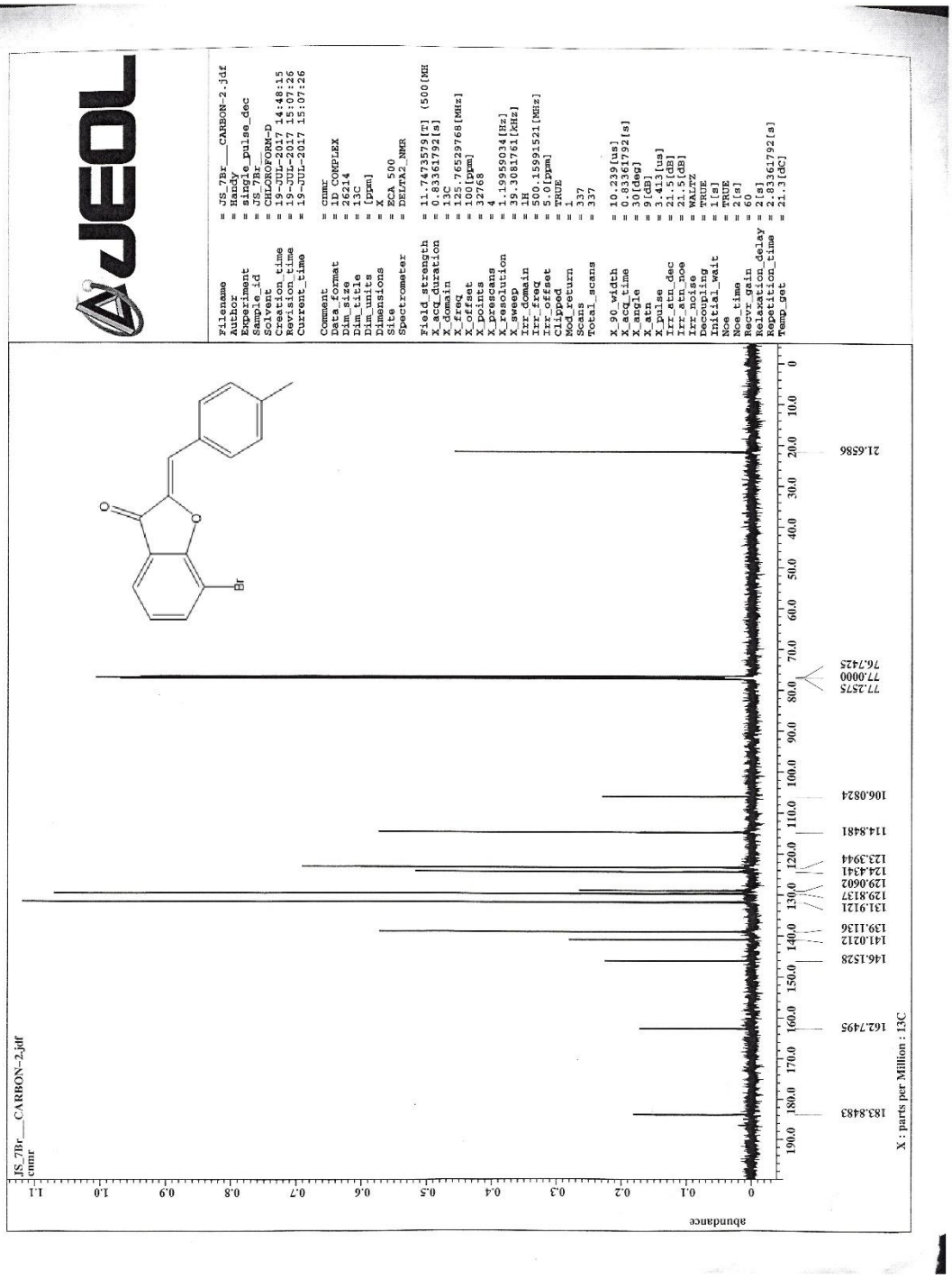

Spectrum

Line#:1 R.Time:26.765(Scan#:4554)
MassPeaks:127
RawMode:Single 26.765(4554) BasePeak:75.05(71248)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(7’-bromobenzofuran-3-one) [**10**]




JEOL
Filename = JS_7Br___CARBON-2.jdf
Author = Handy
Experiment = single_pulse_dec
Sample_id = JS_7Br__
Solvent = CHLOROFORM-D
Creation_time = 19-JUL-2017 14:48:15
Revision_time = 19-JUL-2017 15:07:26
Current_time = 19-JUL-2017 15:07:26
Comment = cnmr
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.74735791[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 = 337
total_scans = 337
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_mode = NNATT
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 = 21.3[dC]
Br
JS_7Br___CARBON-2.jdf
cnmr
abundance
X : parts per Million : 13C
183.8483
162.7495
146.1528
141.0212
139.1136
131.9121
129.8137
129.0602
124.4341
123.3944
114.8481
106.0824
77.2575
77.0000
76.7425
21.6586

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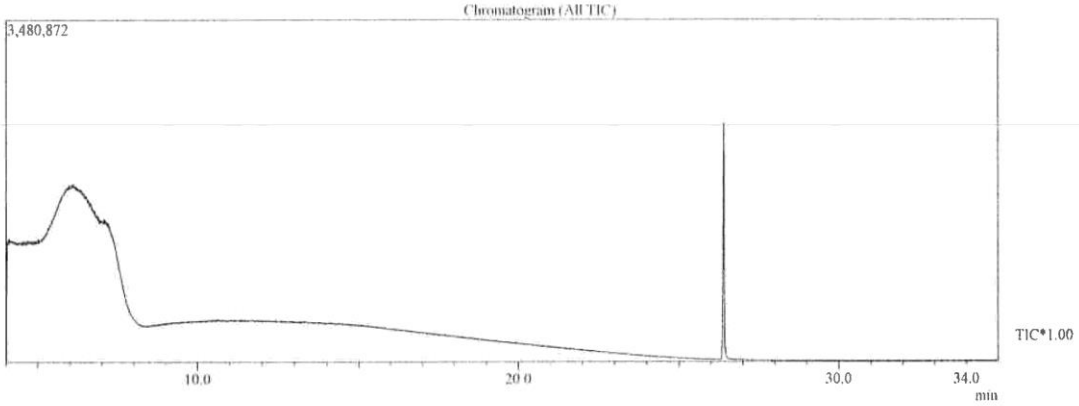
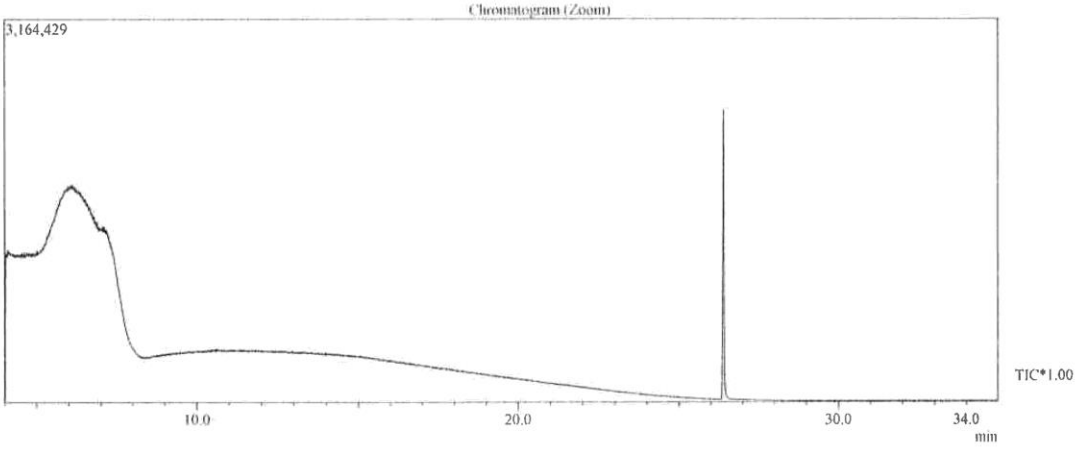
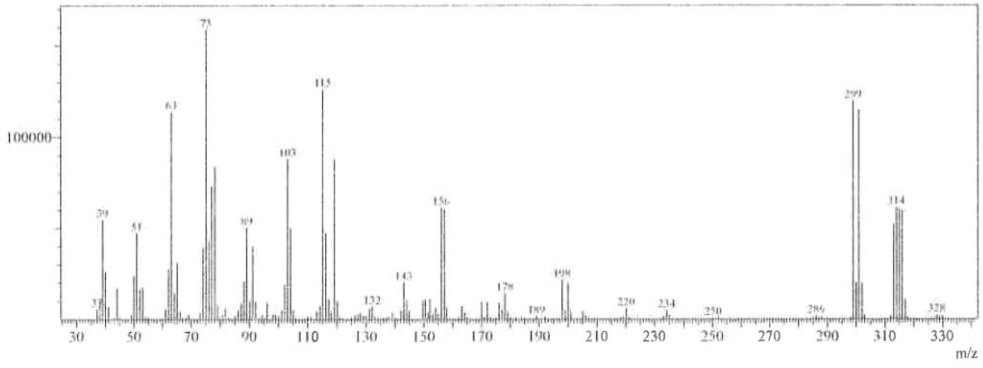


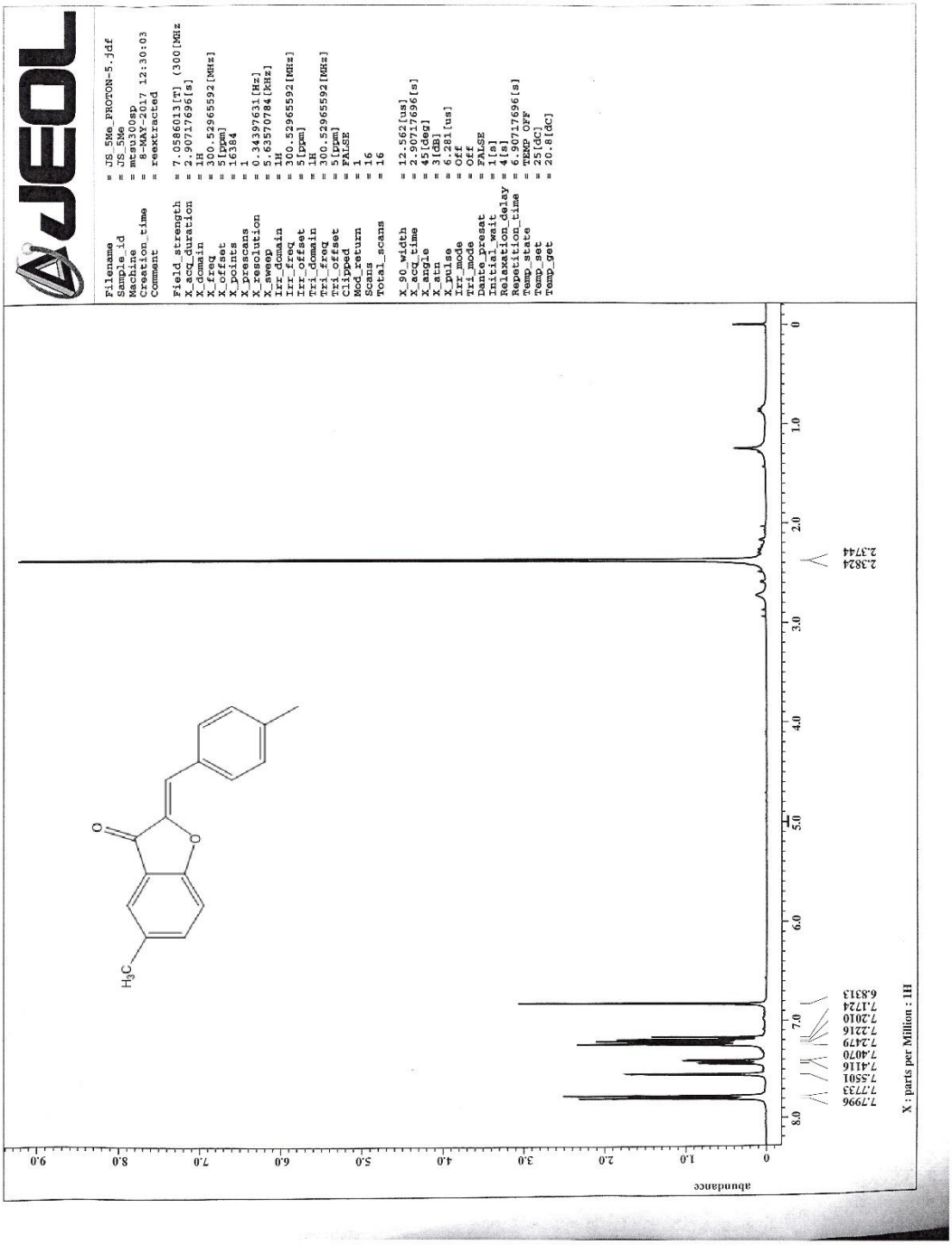



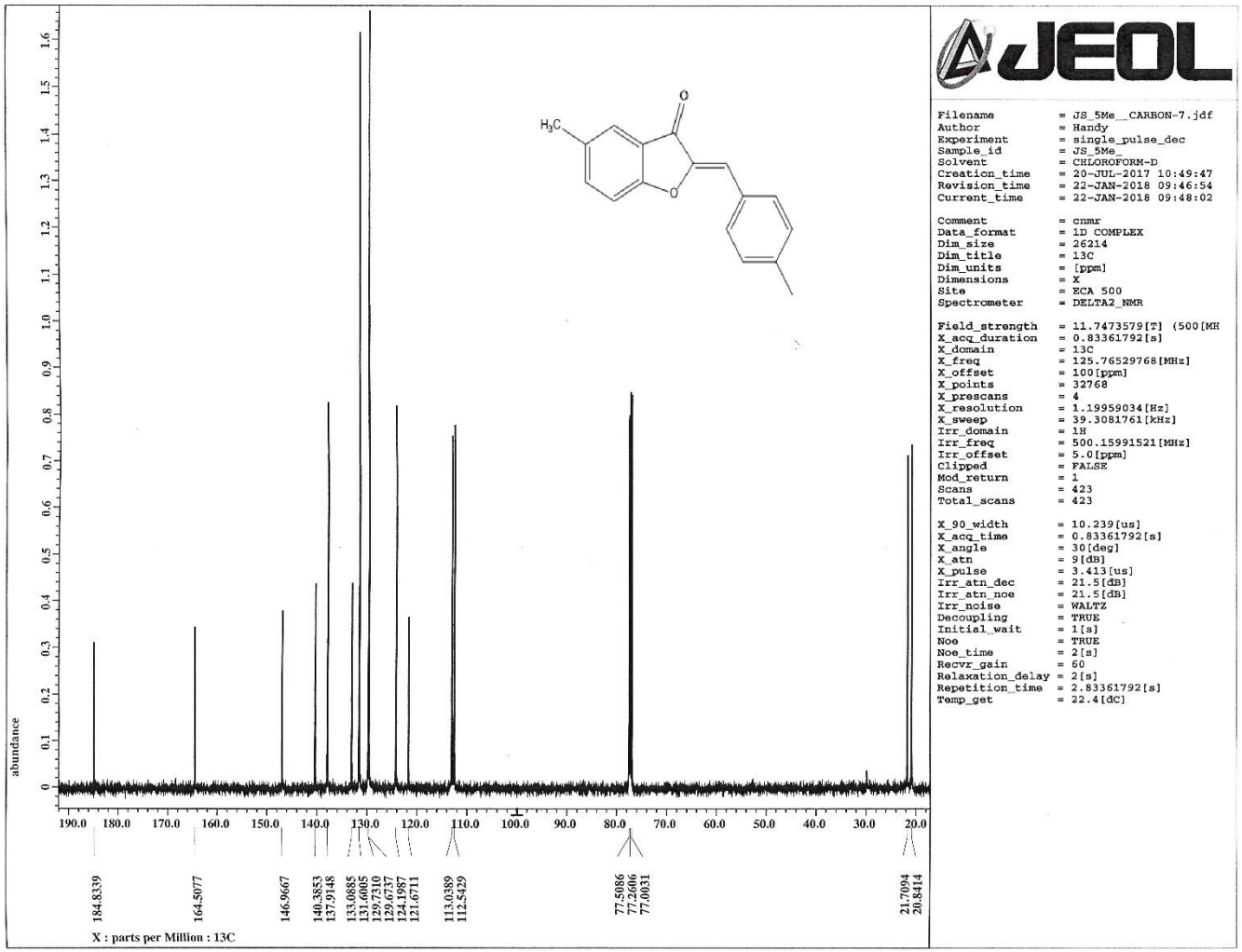

Spectrum

Line#:1 R.Time:26.405(Scan#:4482)
MassPeaks:157
RawMode:Single 26.405(4482) BasePeak:75.05(158668)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(5’-methylbenzofuran-3-one) [**11**]






JEOL

| | |
|---|---|
| Filename | = JS_5Me__CARBON-7.jdf |
| Author | = Handy |
| Experiment | = single_pulse_dec |
| Sample_id | = JS_5Me_ |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 20-JUL-2017 10:49:47 |
| Revision_time | = 22-JAN-2018 09:46:54 |
| Current_time | = 22-JAN-2018 09:48:02 |
| Comment | = cnmr |
| 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 | = 423 |
| Total_scans | = 423 |
| X_90_width | = 10.239[us] |
| X_acq_time | = 0.83361792[s] |
| X_angle | = 30[deg] |
| X_atn | = 9[dB] |
| X_pulse | = 3.413[us] |
| Irr_atn_dec | = 21.5[dB] |
| Irr_atn_noe | = 21.5[dB] |
| Irr_noise | = WALTZ |
| Decoupling | = TRUE |
| Initial_wait | = 1[s] |
| Noe | = TRUE |
| Noe_time | = 2[s] |
| Recvr_gain | = 60 |
| Relaxation_delay | = 2[s] |
| Repetition_time | = 2.83361792[s] |
| Temp_get | = 22.4[dC] |

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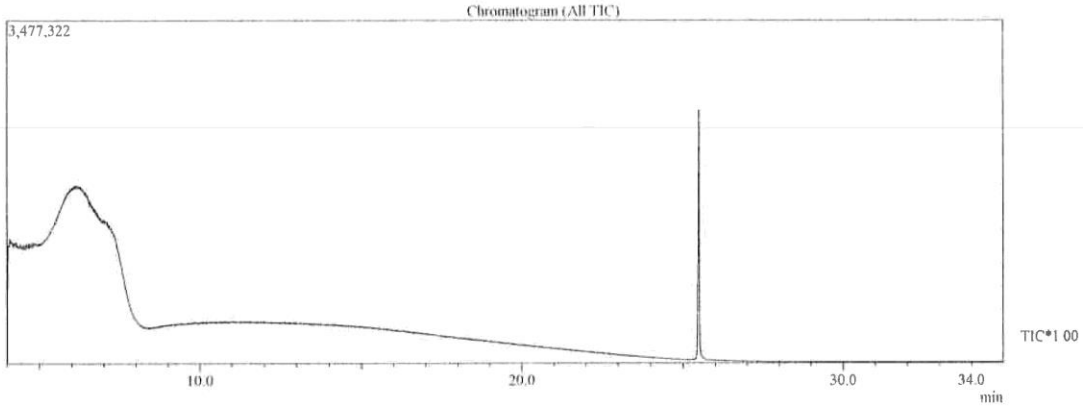
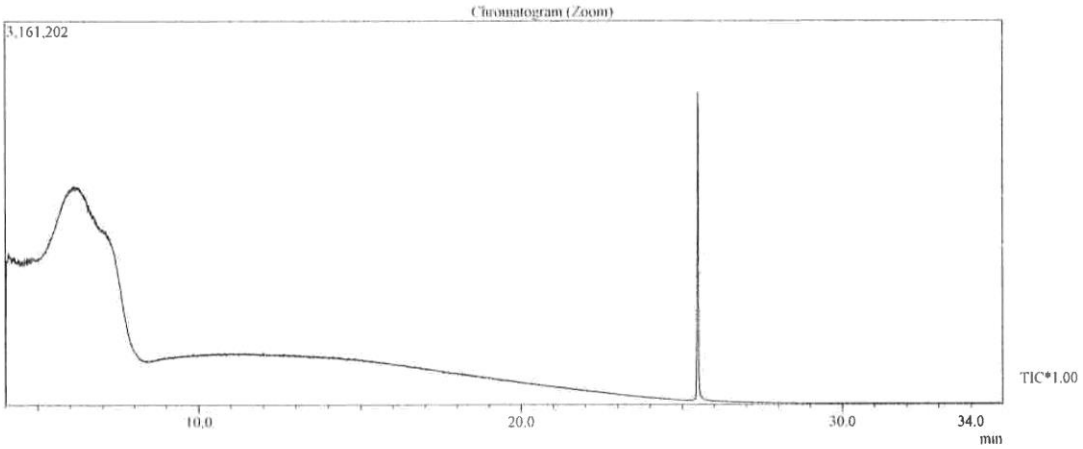
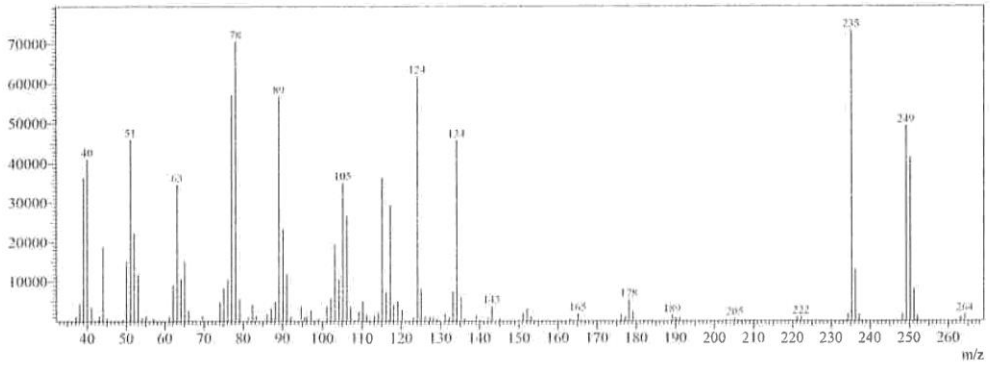


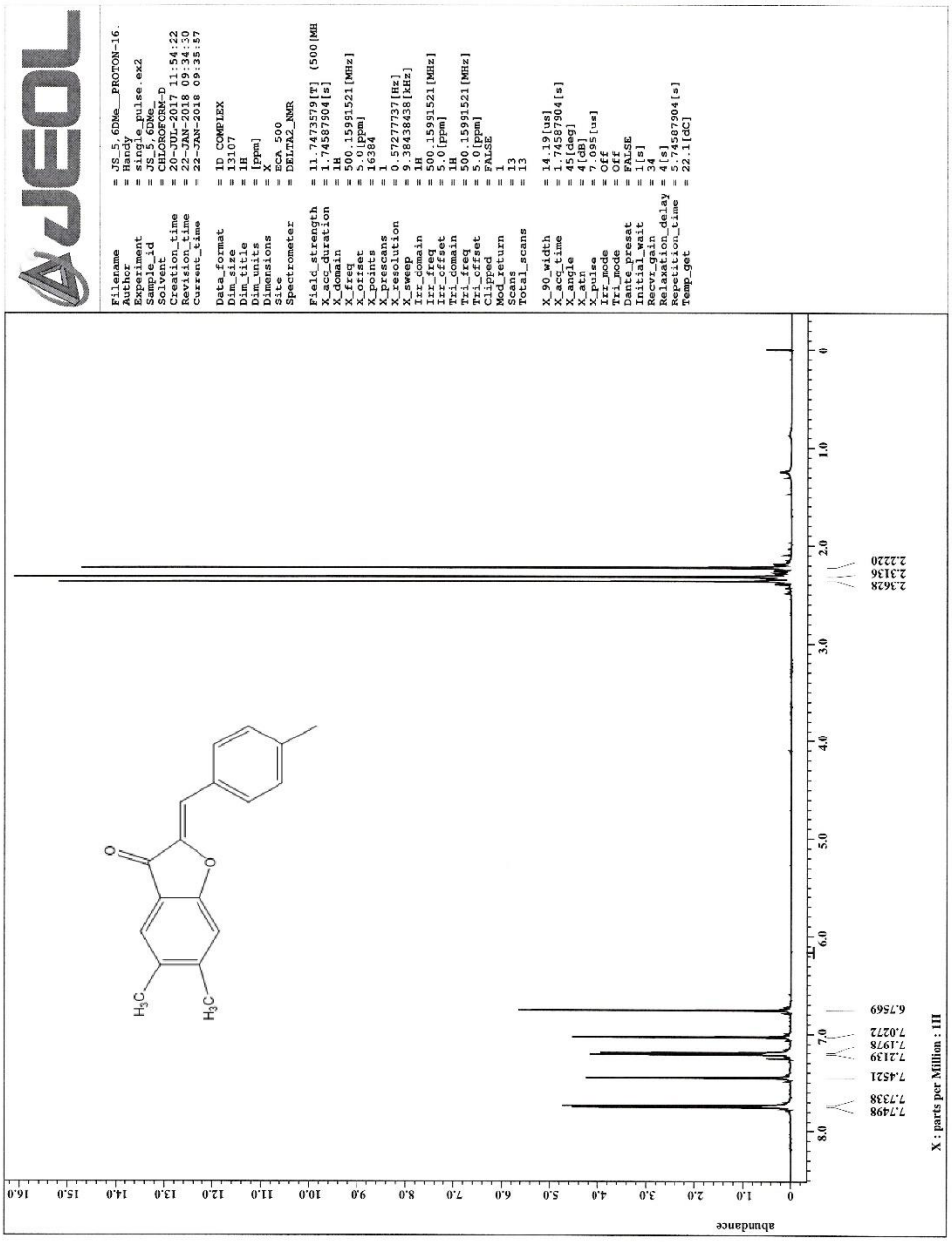



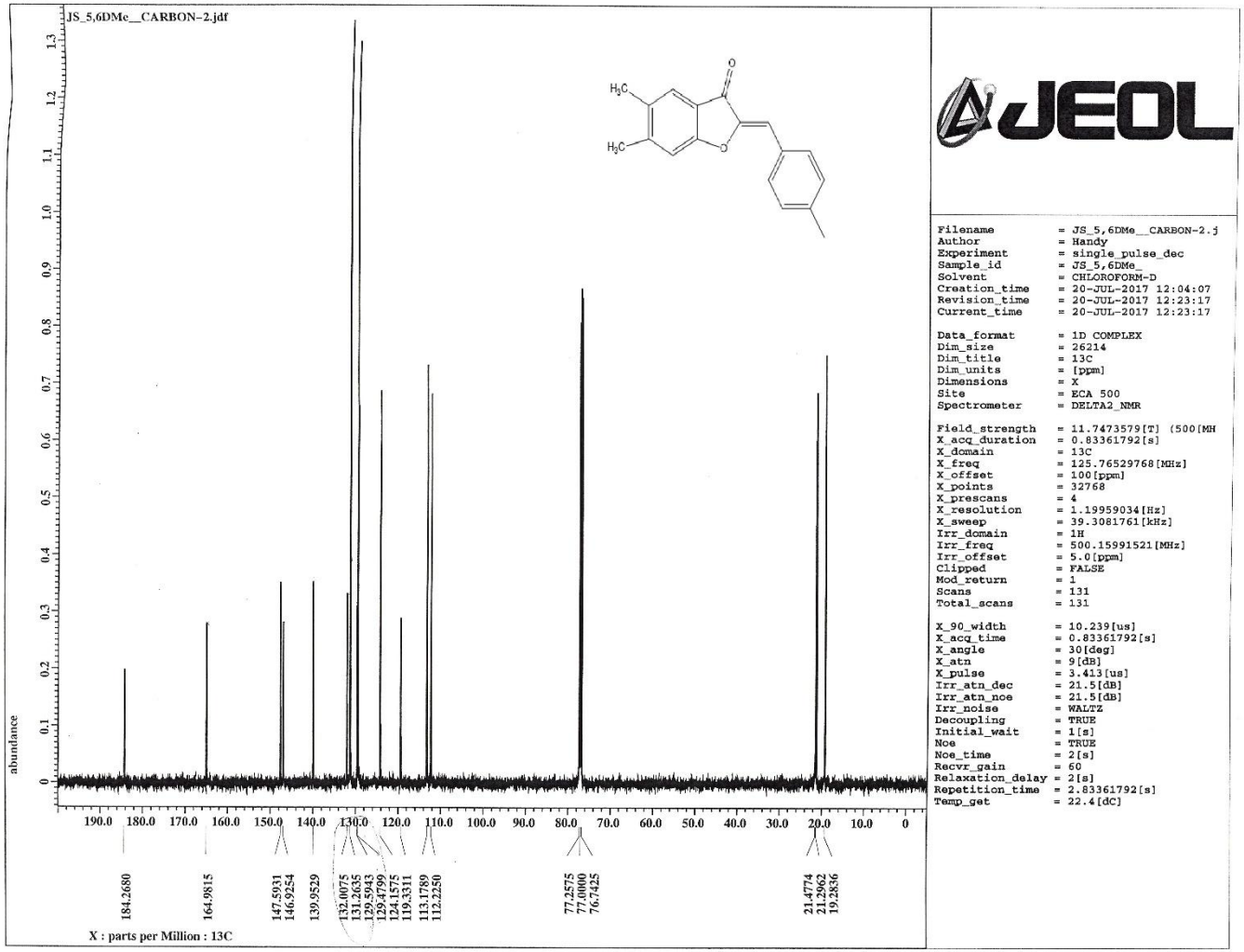

Spectrum

Line#:1 R.Time:25.505(Scan#:4302)
MassPeaks:105
RawMode:Single 25.505(4302) BasePeak:235.20(73345)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(5’,6’-dimethylbenzofuran-3-one) [**12**]




JS_5,6DMe__CARBON-2.jdf
abundance
X : parts per Million : 13C
184.2680
164.9815
147.5931
146.9254
139.9529
132.0075
131.2635
129.5943
129.4799
124.1575
119.3311
113.1789
112.2250
77.2575
77.0000
76.7425
21.4774
21.2962
19.2856
Filename = JS_5,6DMe__CARBON-2.j
Author = Handy
Experiment = single_pulse_dec
Sample_id = JS_5,6DMe_
Solvent = CHLOROFORM-D
Creation_time = 20-JUL-2017 12:04:07
Revision_time = 20-JUL-2017 12:23:17
Current_time = 20-JUL-2017 12:23:17
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 = 131
Total_scans = 131
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 22.4[dC]

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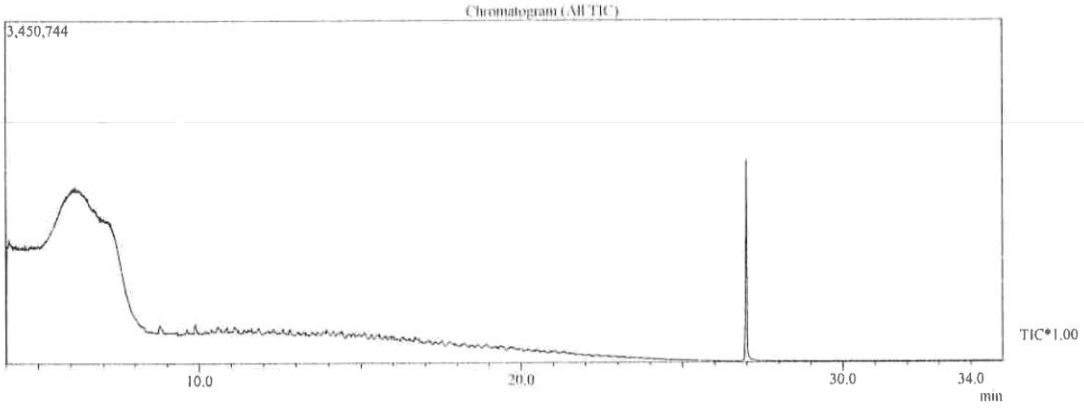
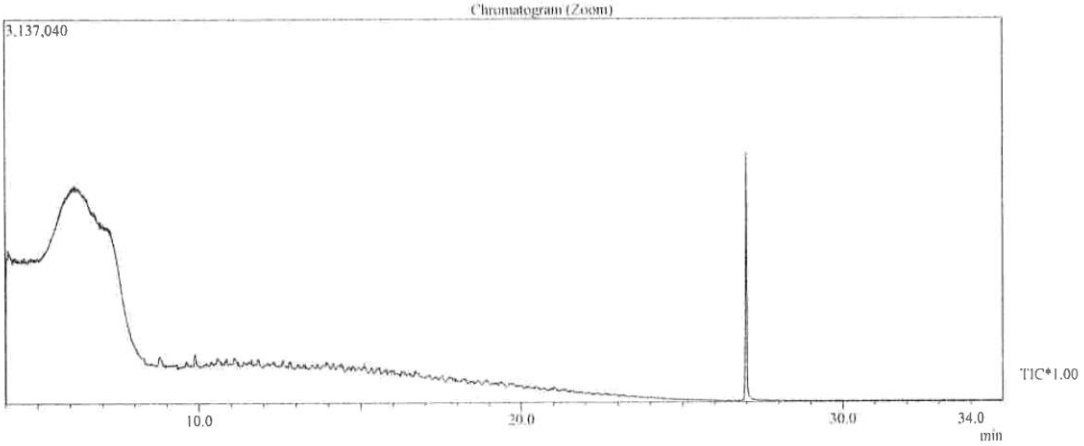
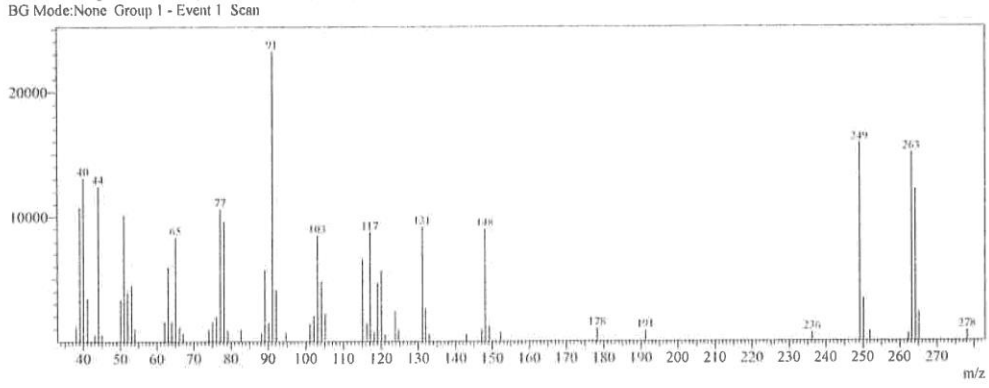


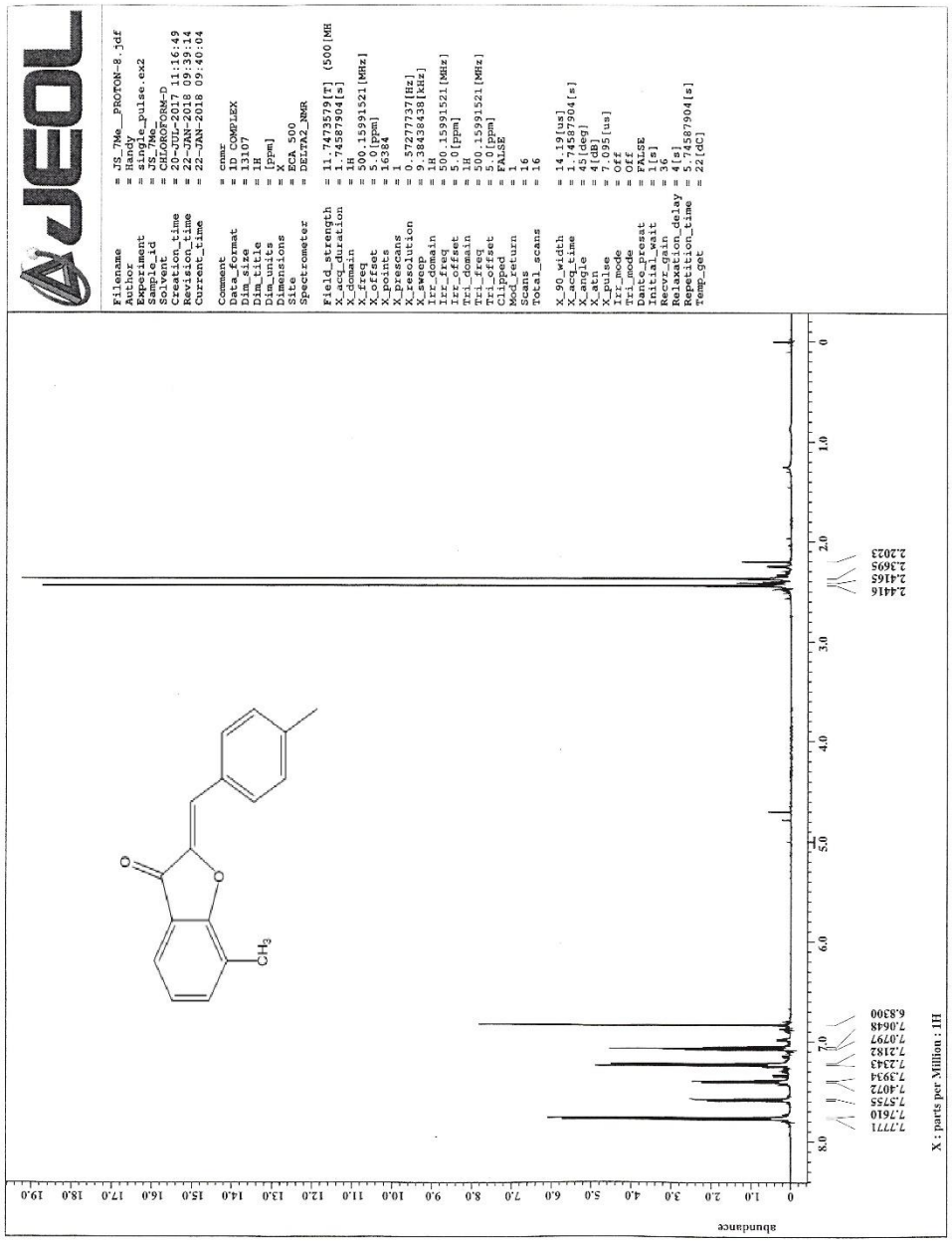



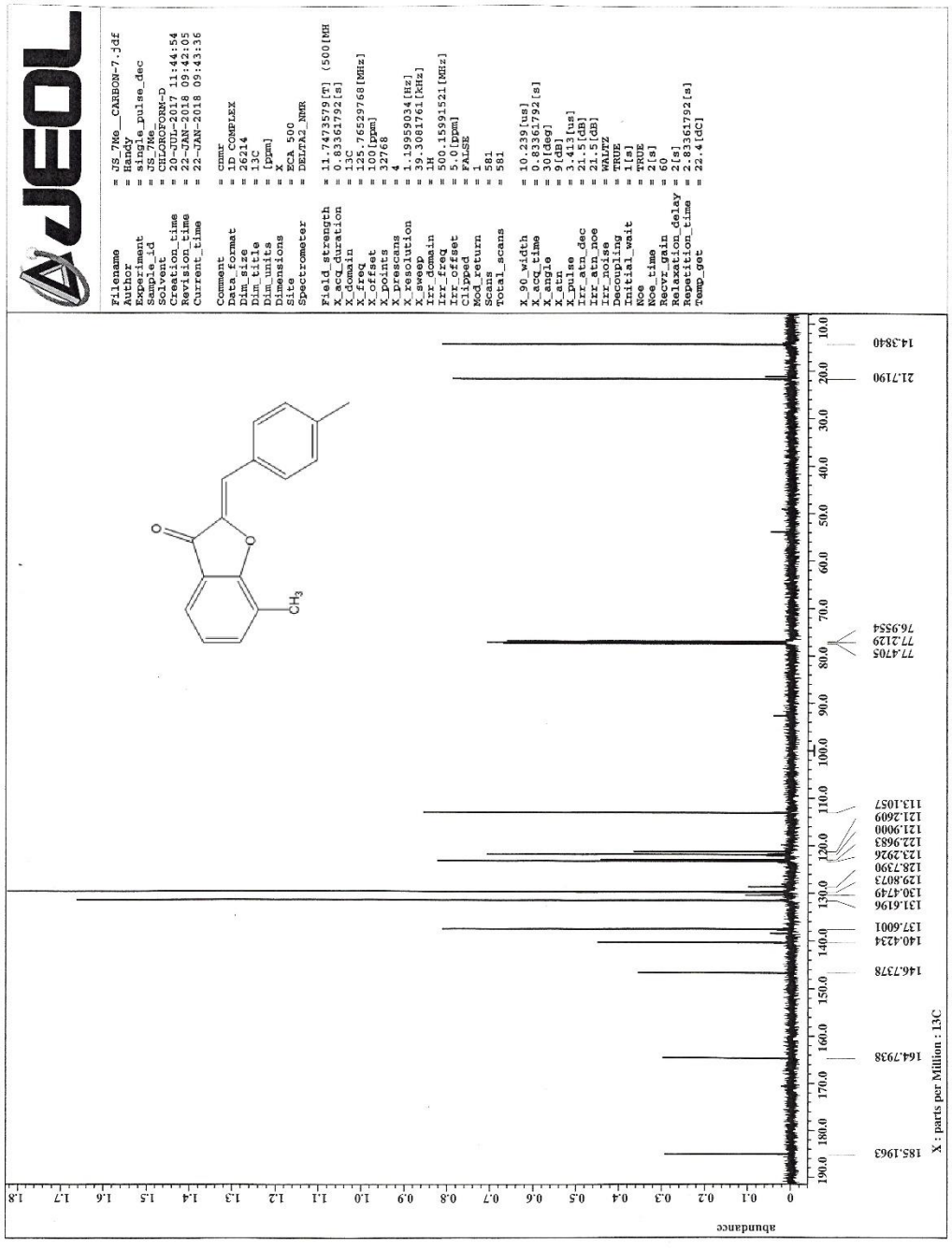

Spectrum

Line#:1 R.Time:27.015(Scan#:4604)
MassPeaks:64
RawMode:Single 27.015(4604) BasePeak:91.10(23252)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(7’-methylbenzofuran-3-one) [**13**]




JEOL
Filename = JS_786__CARBON-7.jdf
Author = Handy
Experiment = single_pulse_dec
Sample_id = JS_786
Solvent = CHLOROFORM-D
Creation_time = 20-JUL-2017 11:44:54
Revision_time = 22-JAN-2018 09:42:05
Current_time = 22-JAN-2018 09:43:36
Comment = cmnr
Data_format = 1D COMPLEX
Dim_size = 2214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.74735779[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 = 581
Total_scans = 581
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.41[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 20
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 22.4[dC]
14.3840
21.7190
76.9554
77.2129
77.4705
113.1057
121.2609
121.9000
122.9683
123.2926
128.7390
129.8073
130.4749
131.6196
137.6001
140.4234
146.7378
164.7938
185.1963
X : parts per Million : 13C
abundance

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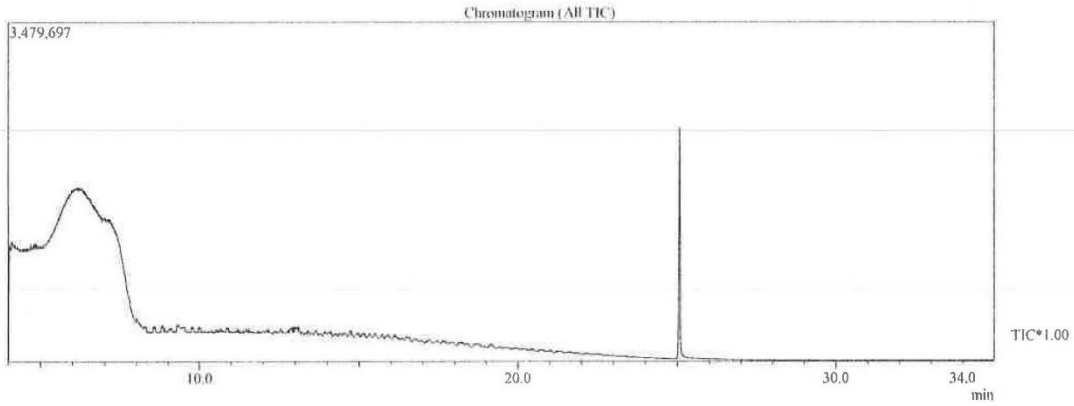
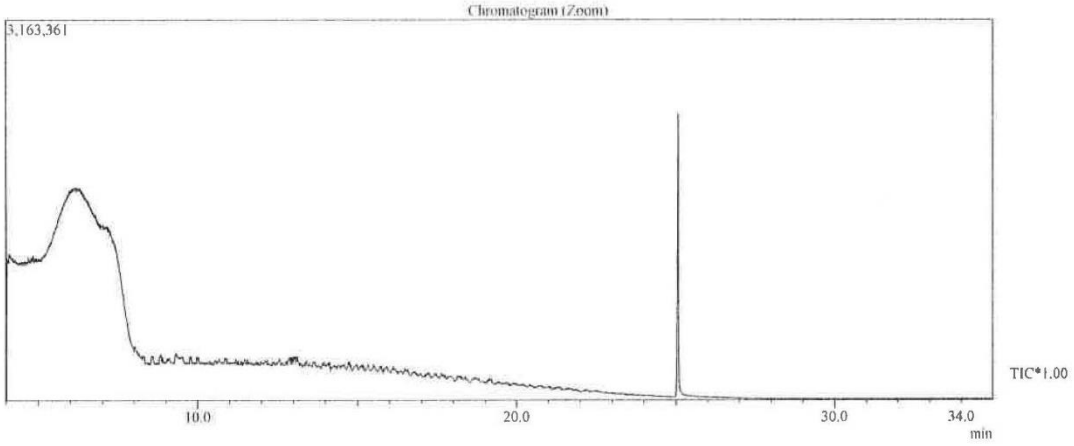
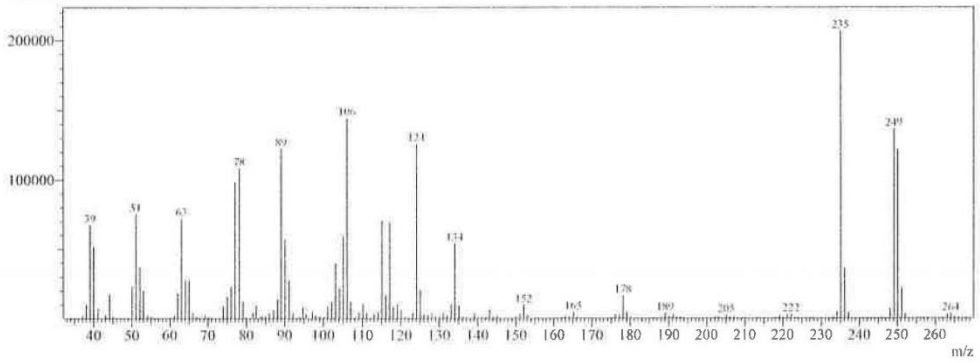


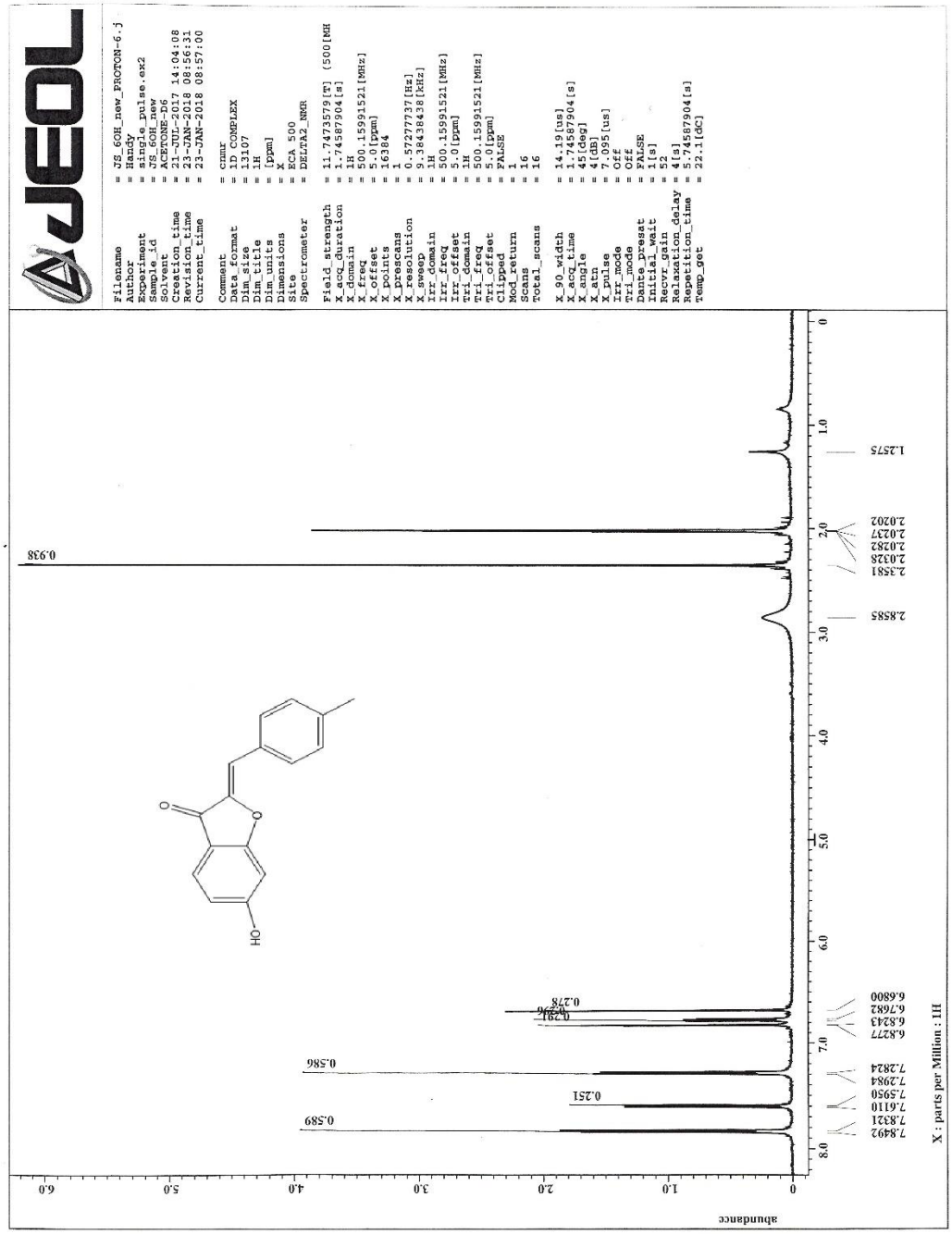



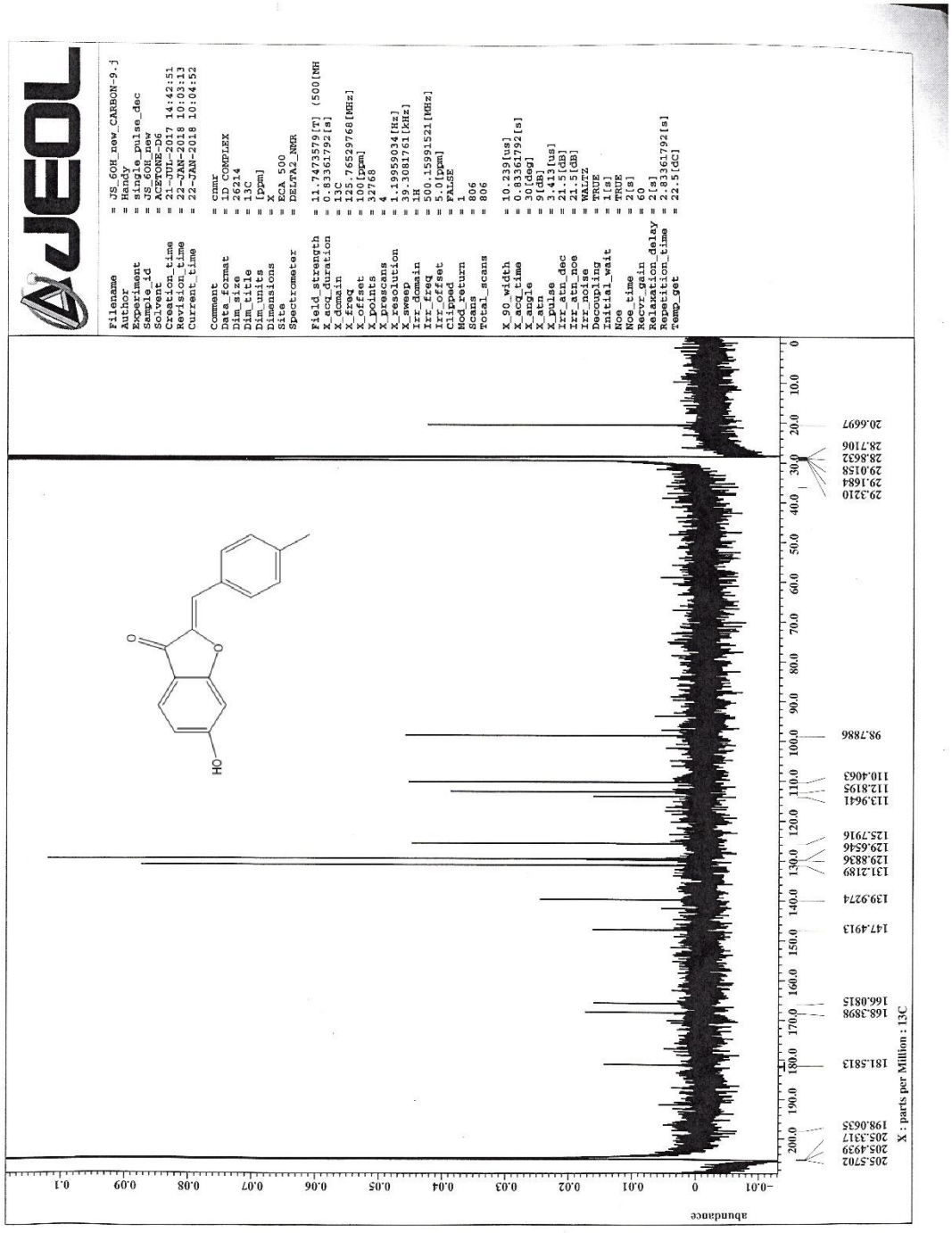

Spectrum

Line#:1 R.Time:25.080(Scan#:4217)
MassPeaks:135
RawMode:Single 25.080(4217) BasePeak:235.10(206370)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(6’-hydroxybenzofuran-3-one) [**14**]




JEOL
Filename = JS_60H_new_CARBON-9.j
Author = Handy
Experiment = single_pulse_dec
Sample_id = JS60H_new
Solvent = ACETONE-D6
Creation_time = 21-JUL-2017 14:42:51
Revision_time = 22-JAN-2018 10:03:13
Current_time = 22-JAN-2018 10:04:52
Comment = cmnr
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 = 806
Total_scans = 806
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_coupling = 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 = 22.5[dC]
abundance
X : parts per Million : 13C
205.5702
205.4939
205.3317
198.0635
181.5813
168.3898
166.0815
147.4913
139.9274
131.2189
129.8836
129.6546
125.7916
113.9641
112.8195
110.4063
98.7886
29.3210
29.1684
29.0158
28.8632
28.7106
20.6697
O
O
O
HO

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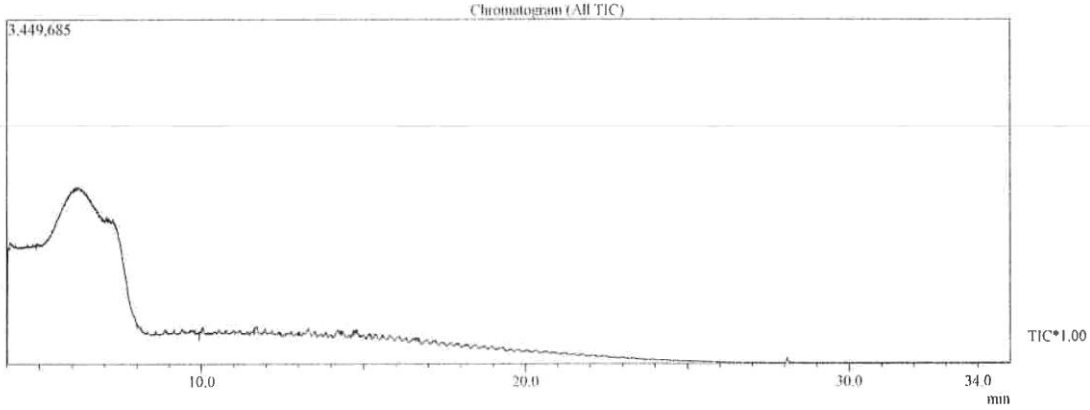
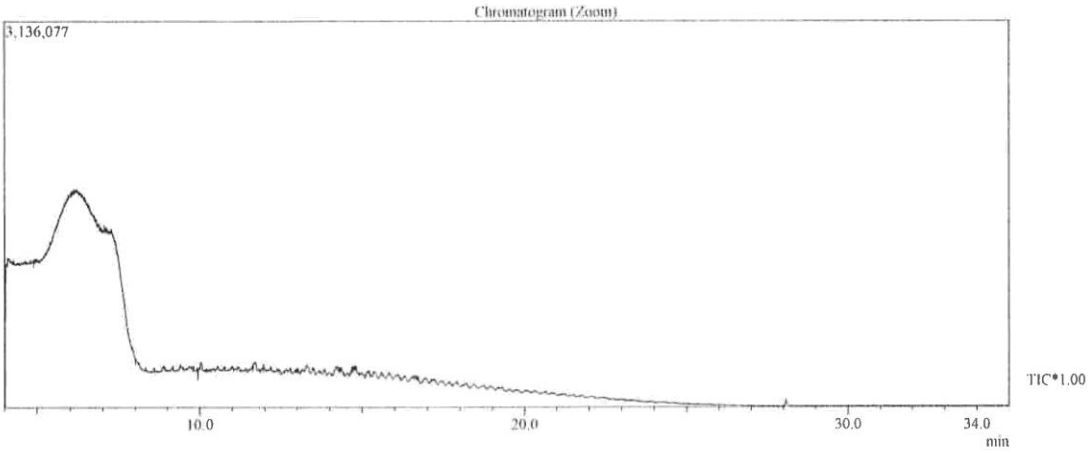
Chromatogram (All TIC)

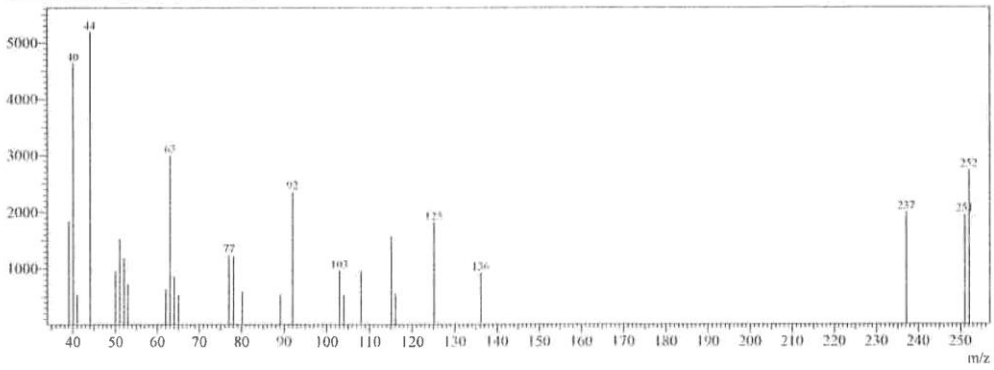



Chromatogram (Zoom)

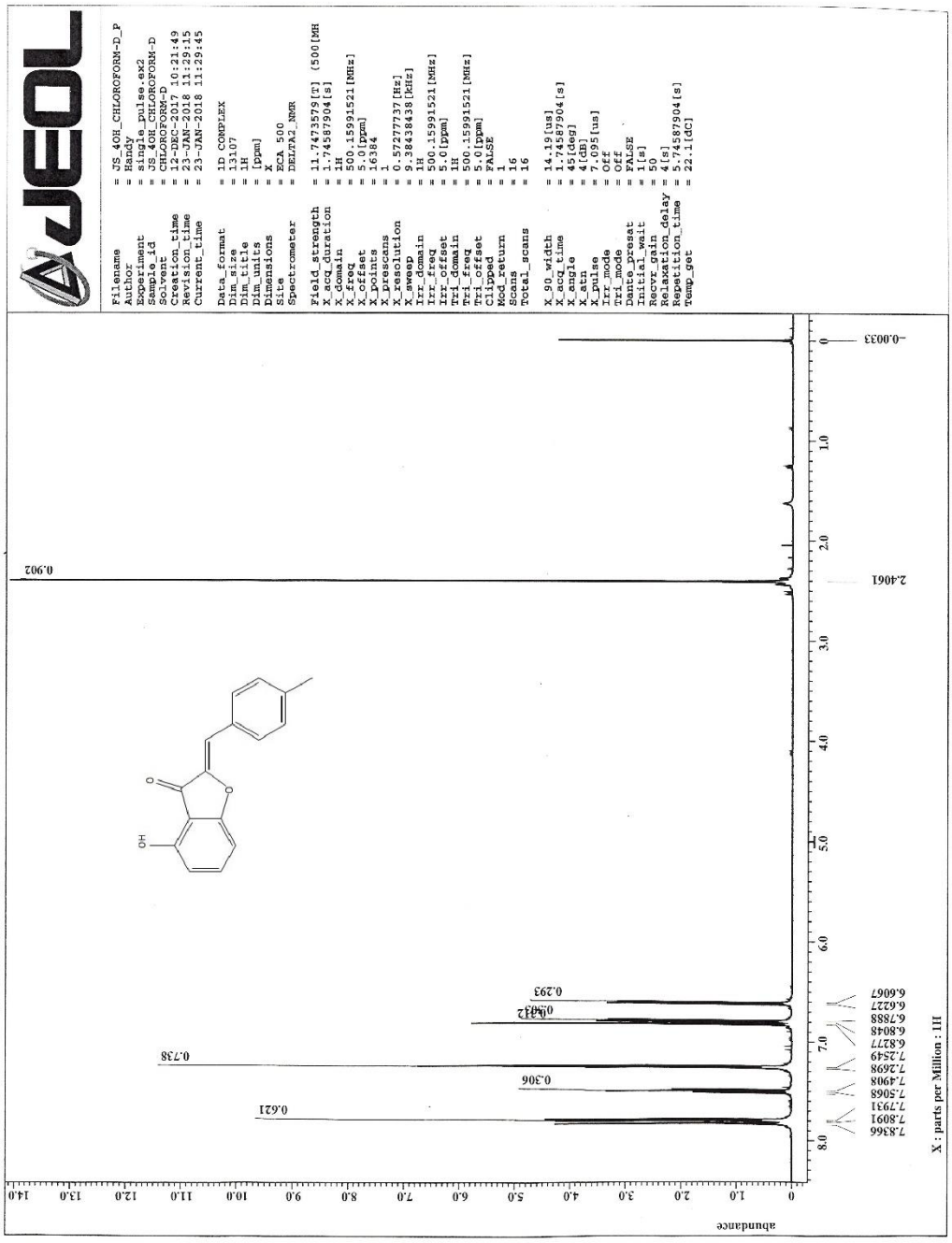


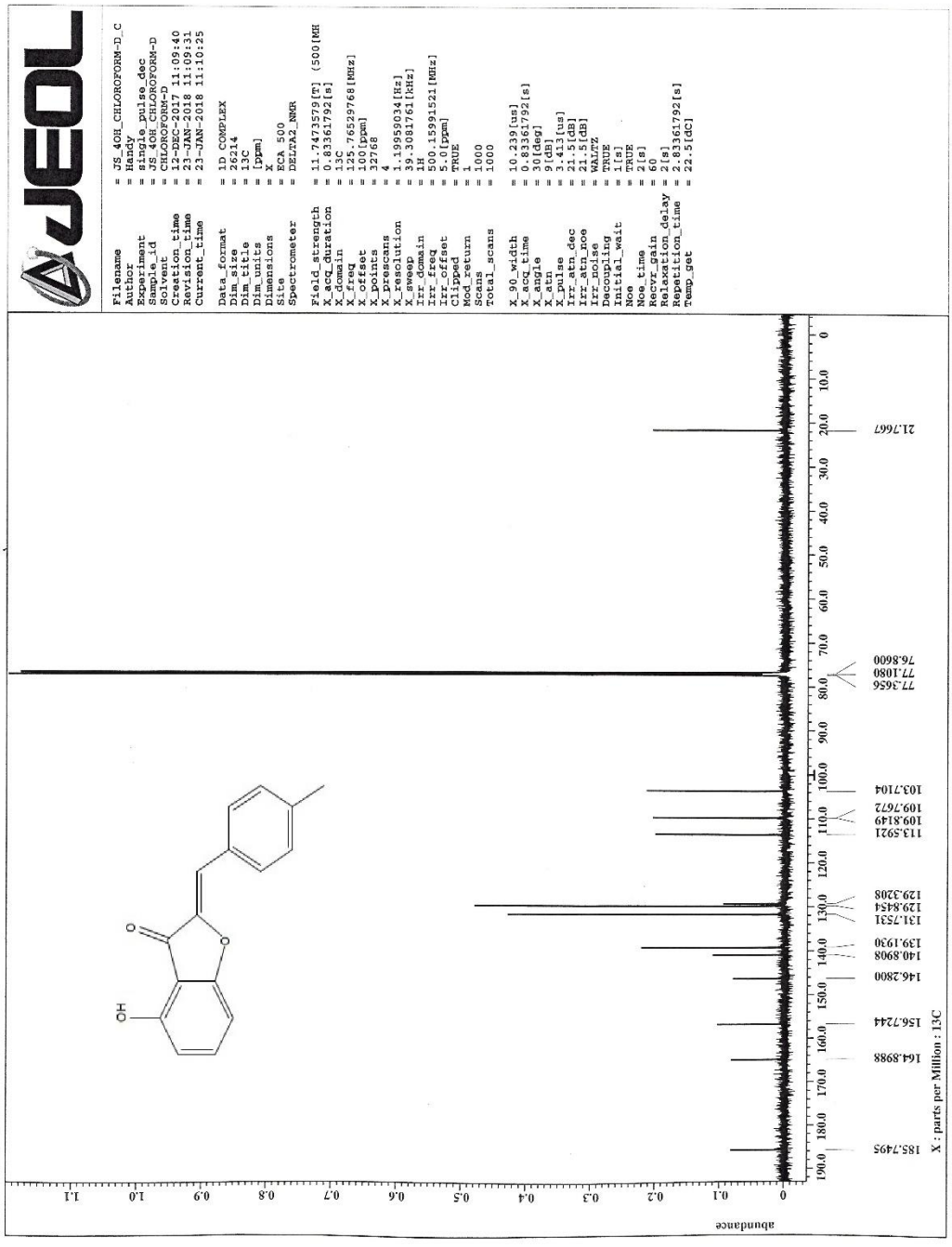

Spectrum

Line#:1 R.Time:28.090(Scan#:4819)
MassPeaks:27
RawMode:Single 28.090(4819) BascPeak 44.05(5186)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(4’-hydroxybenzofuran-3-one) [**15**]




JEOL
Filename = JS_4OH_CHLOROFORM-D_C
Author = Handy
Experiment = single_pulse.dec
Sample_id = JS_4OH_CHLOROFORM-D
Solvent = CHLOROFORM-D
Creation_time = 12-DEC-2017 11:09:40
Revision_time = 23-JAN-2018 11:09:31
Current_time = 23-JAN-2018 11:10:25
Data_format = 1D COMPLEX
Dim_size = 32k
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.74735797[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 = 39.30817611[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = TRUE
Mod_return = 1000
Scans = 1000
total_scans = 1000
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_set = 22.5[dC]
OH
O
O
21.7667
76.8600
77.1080
77.3656
103.7104
109.7672
109.8149
113.5921
129.3208
129.8454
131.7531
139.1930
140.8908
146.2800
156.7244
164.8988
185.7495
X : parts per Million : 13C
abundance

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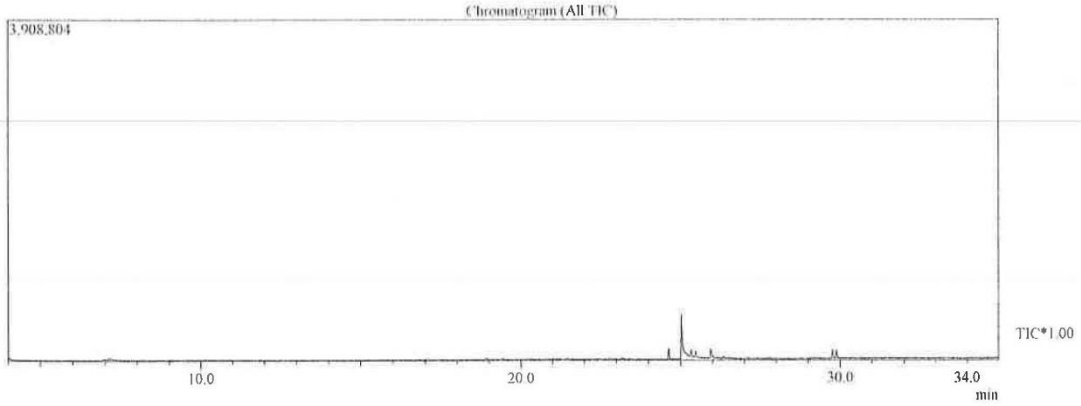
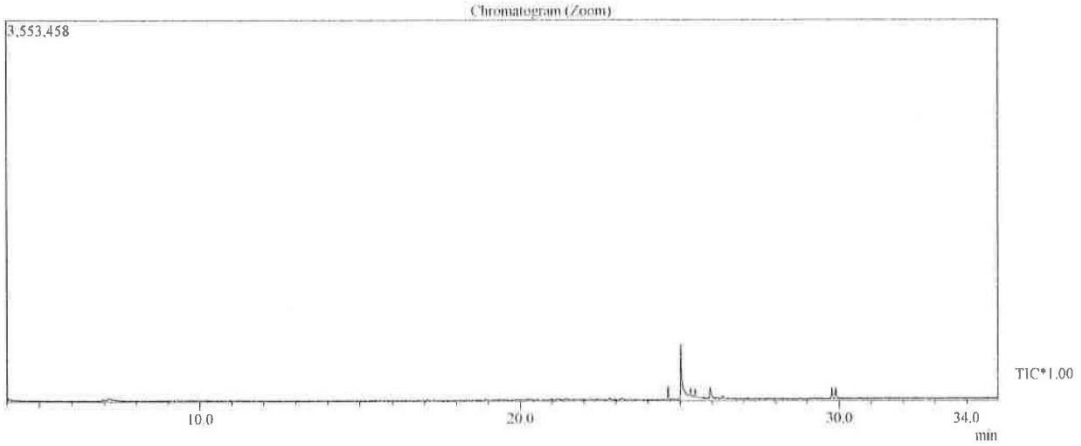
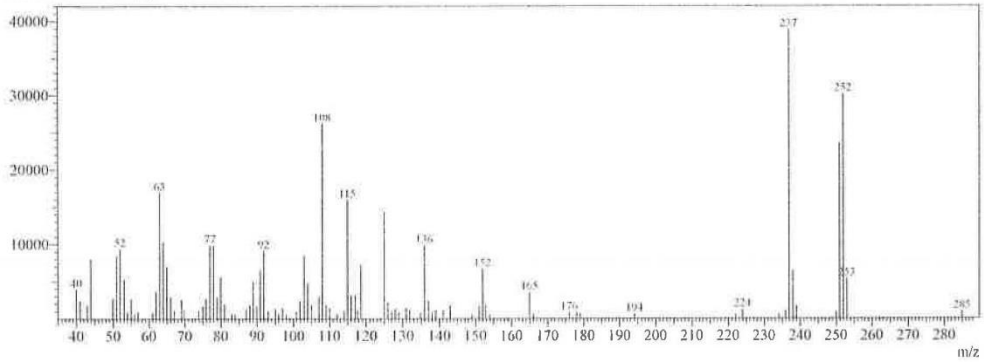


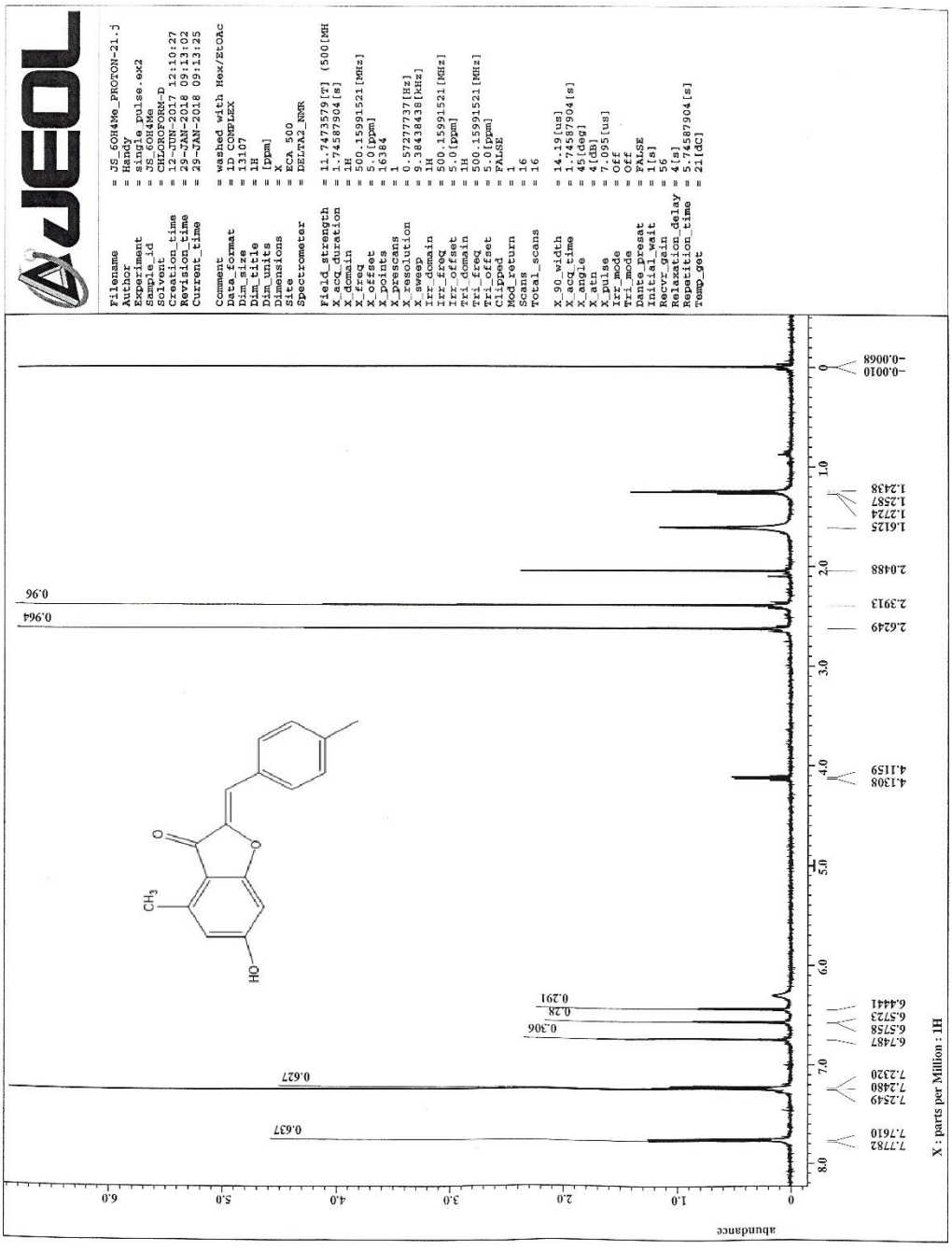



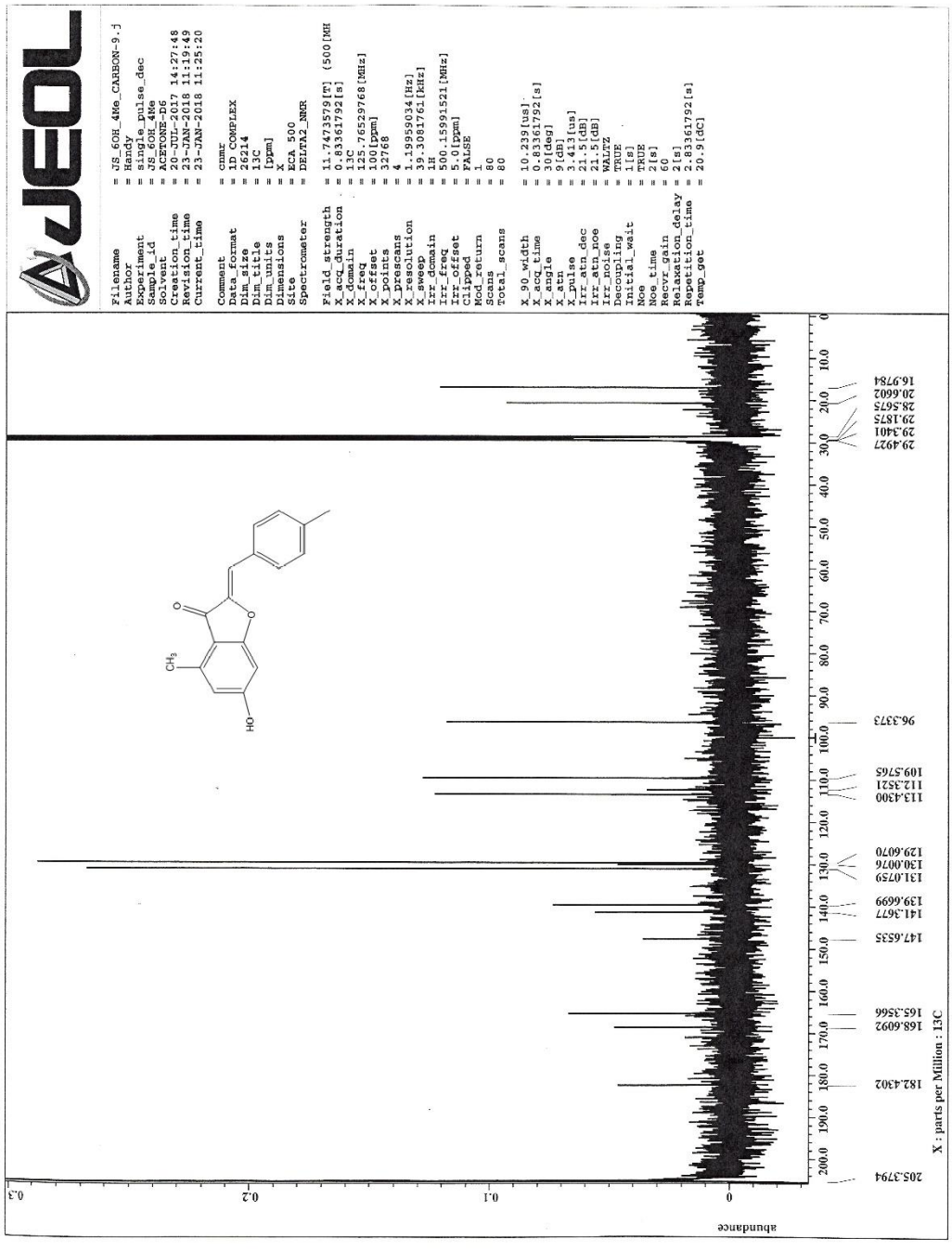

Spectrum

Line#:1 R.Time:25.035(Scan#:4208)
MassPeaks:95
RawMode:Single 25.035(4208) BasePeak:236.90(38782)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(6’-hydroxy-4’-methylbenzofuran-3-one) [**16**]




JEOL
Filename = US_6OH_4Me_CARBON-9.J
Author = Handy
Experiment = single_pulse_dec
Sample_id = 6OH-4Me
Solvent = ACETONE-D6
Creation_time = 20-JUL-2017 14:27:48
Revision_time = 23-JAN-2018 11:19:49
Current_time = 23-JAN-2018 11:25:20
Comment = cnmr
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 = 80
Total_scans = 80
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.413[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_noise = WALTZ
Decoupling = WALTZ
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 20.9[dC]
abundance
X : parts per Million : 13C
205.3794
182.4302
168.6092
165.3566
147.6535
141.3677
139.6699
131.0759
130.0076
129.6070
113.4300
112.3521
109.5765
96.3373
29.4927
29.3401
29.1875
28.5675
20.6602
16.9784
CH3
HO
O
O

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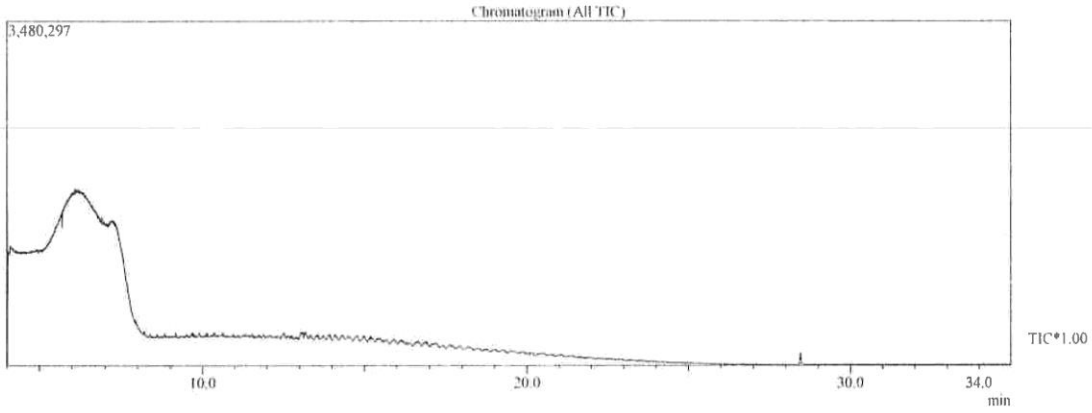
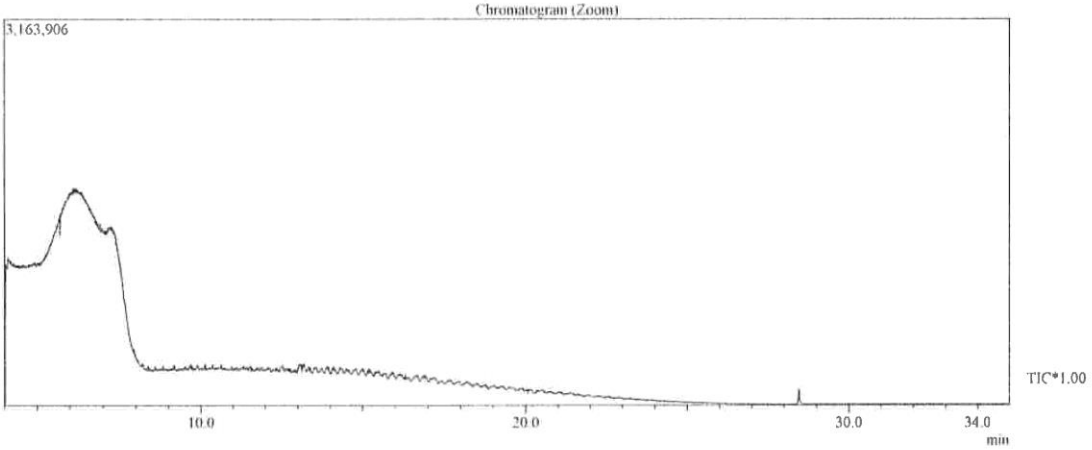
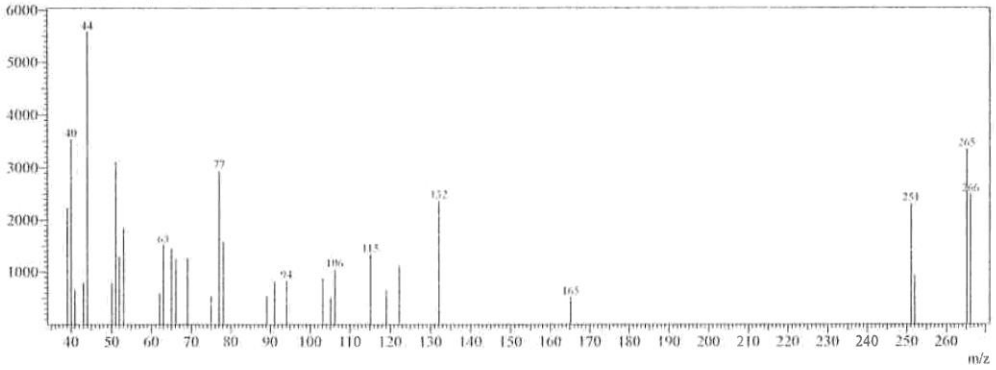


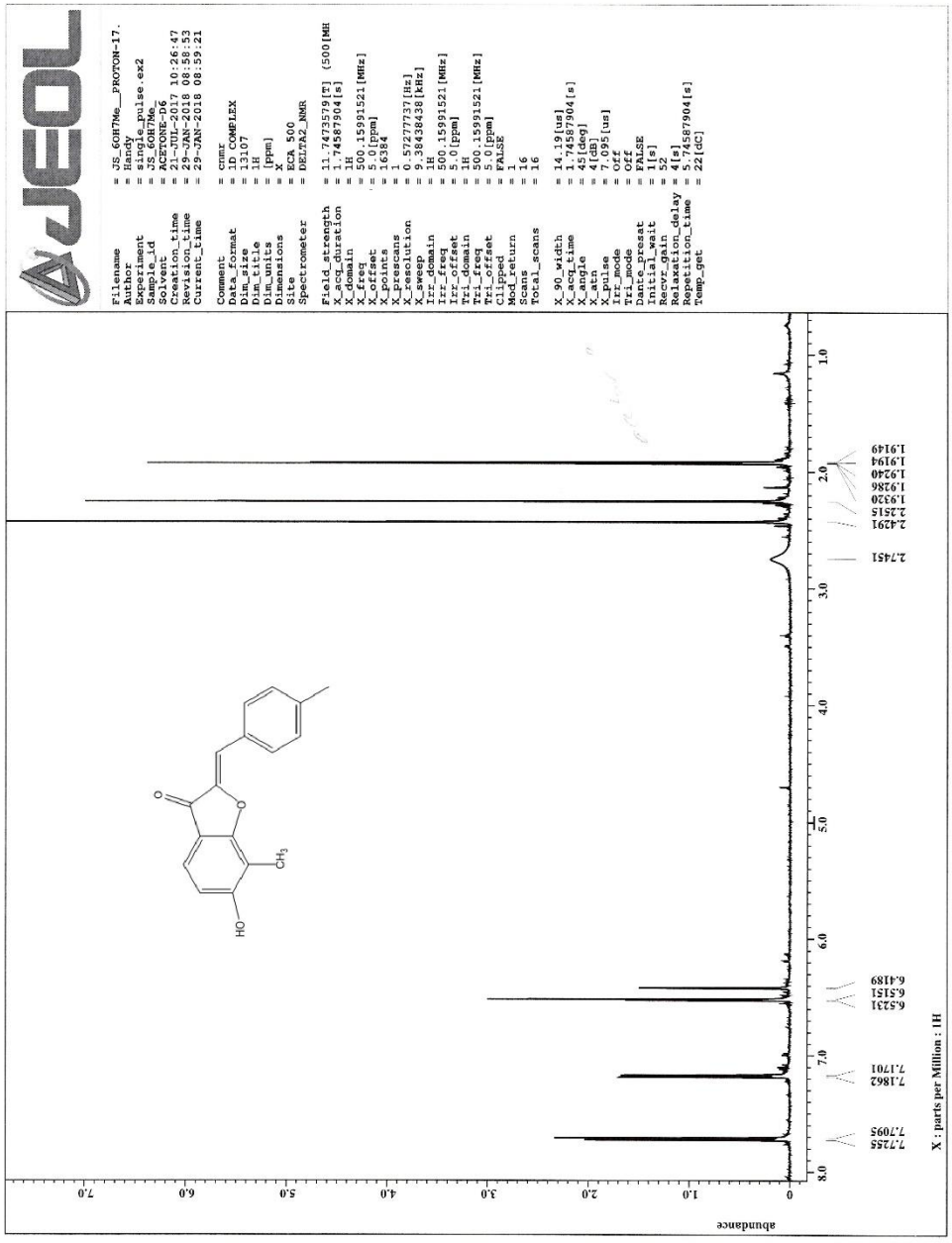



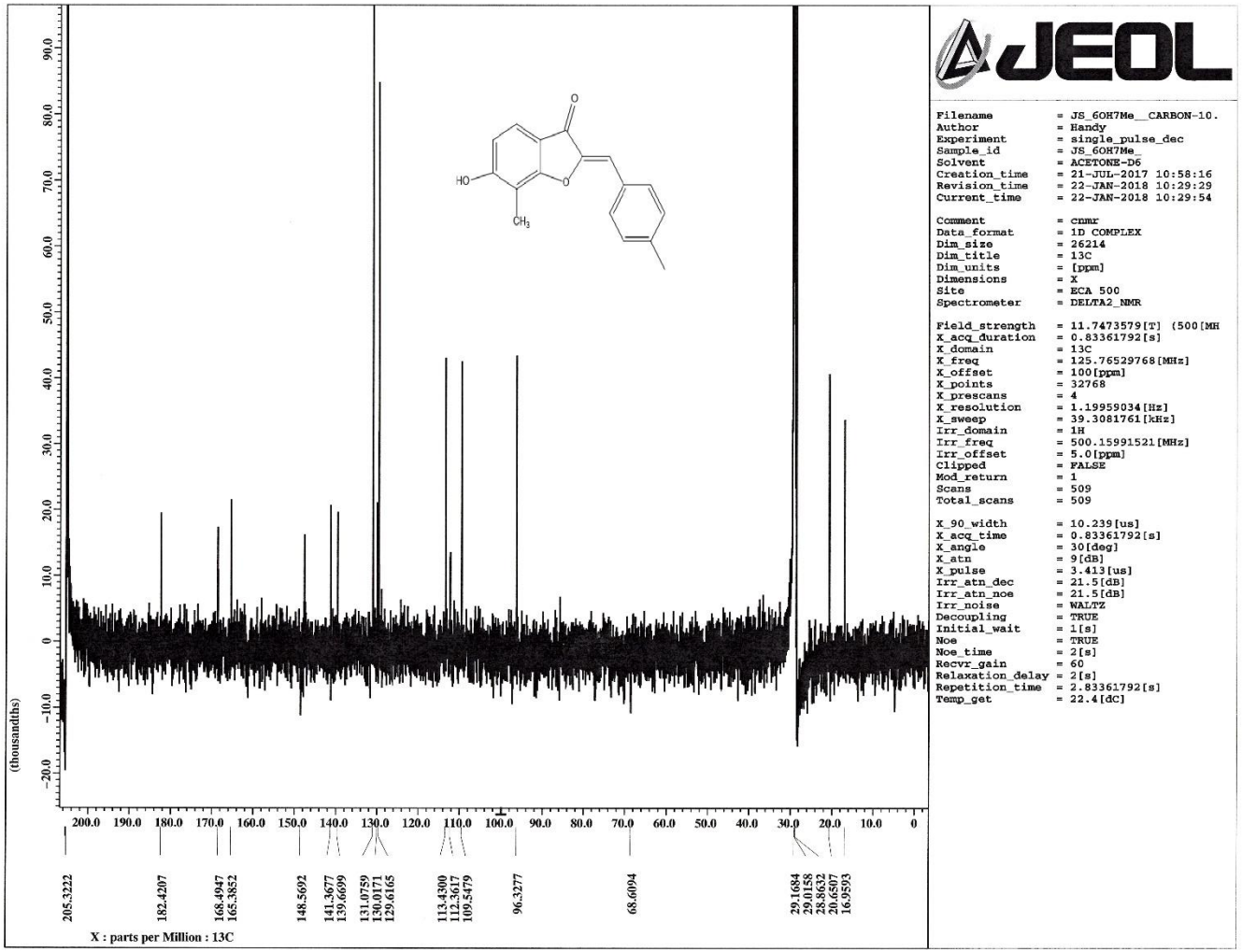

Spectrum

Line#:1 R.Time:28.475(Scan#:4896)
MassPeaks:32
RawMode:Single 28.475(4896) BasePeak:44.05(5575)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(6’-hydroxy-7’-methylbenzofuran-3-one) [**17**]






JEOL

```
Filename          = JS_6OH7Me__CARBON-10.
Author            = Handy
Experiment        = single_pulse_dec
Sample_id         = JS_6OH7Me_
Solvent           = ACETONE-D6
Creation_time     = 21-JUL-2017 10:58:16
Revision_time     = 22-JAN-2018 10:29:29
Current_time      = 22-JAN-2018 10:29:54

Comment           = cnmr
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             = 509
Total_scans       = 509

X_90_width        = 10.239[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.413[us]
Irr_atn_dec       = 21.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 22.4[dC]
```

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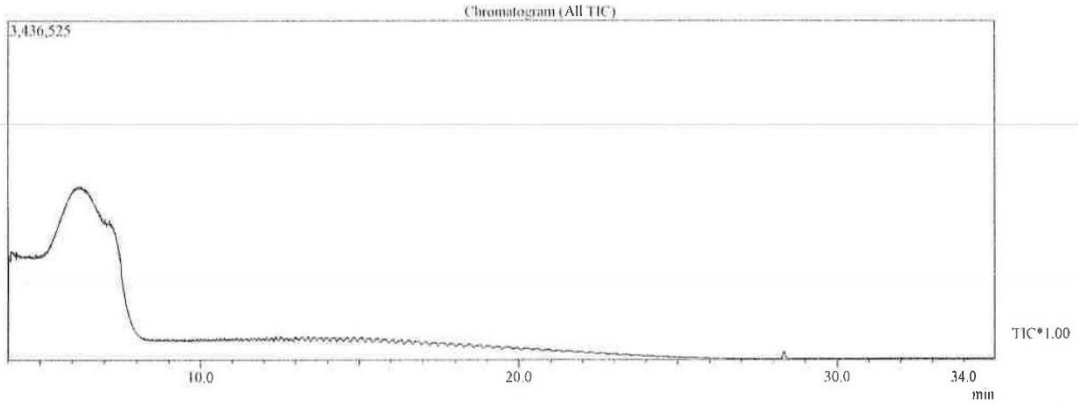
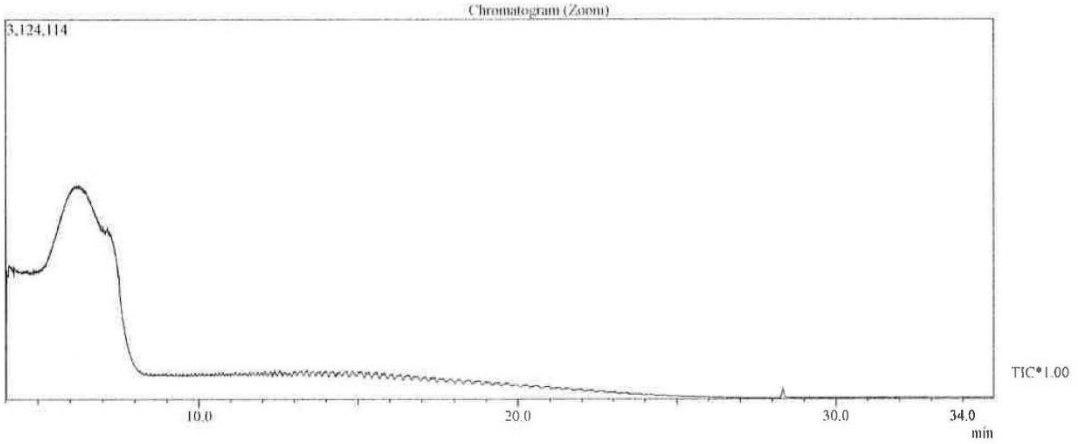
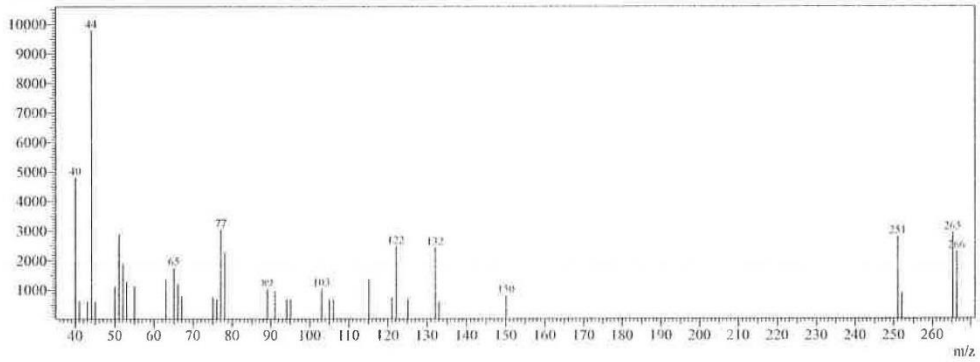


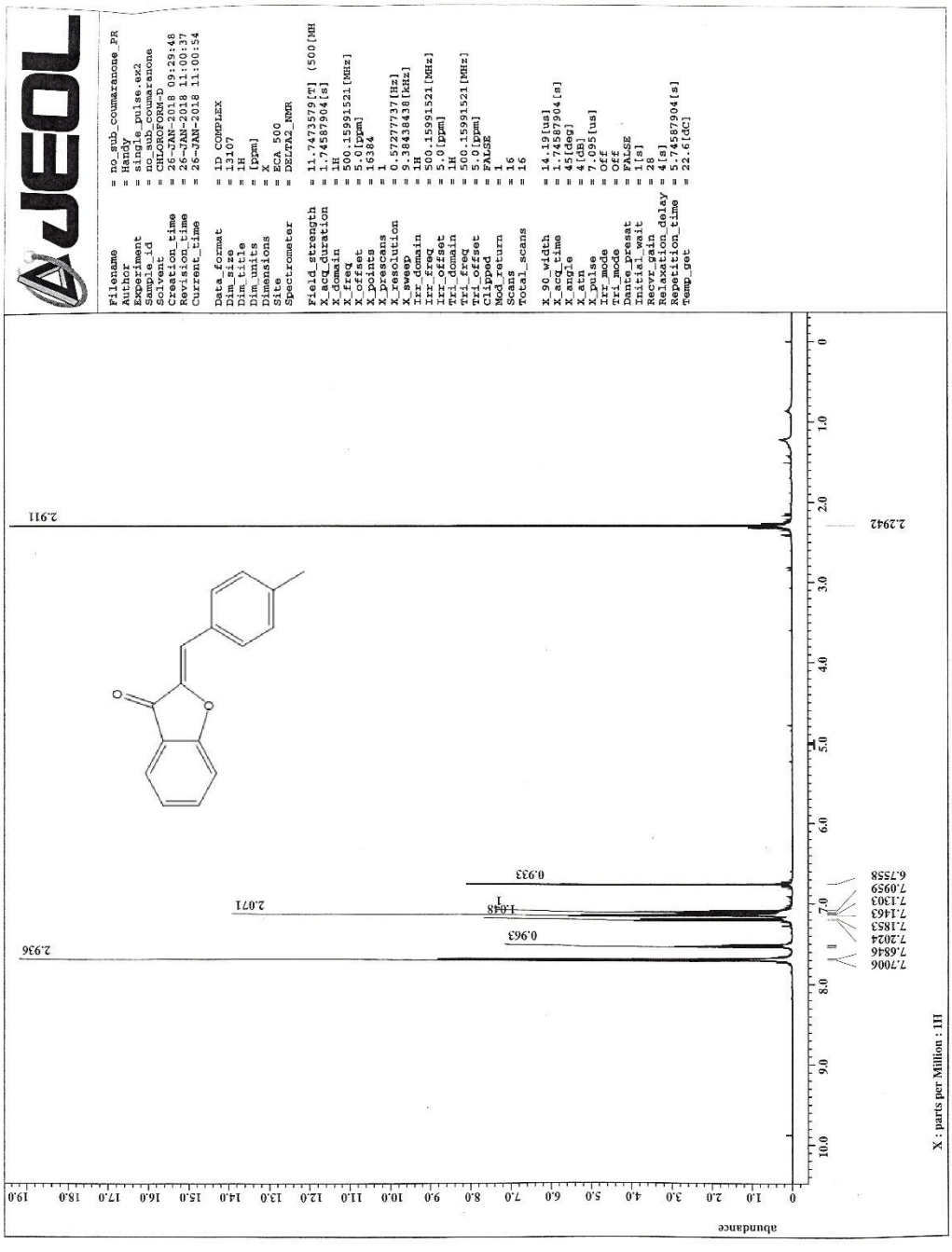



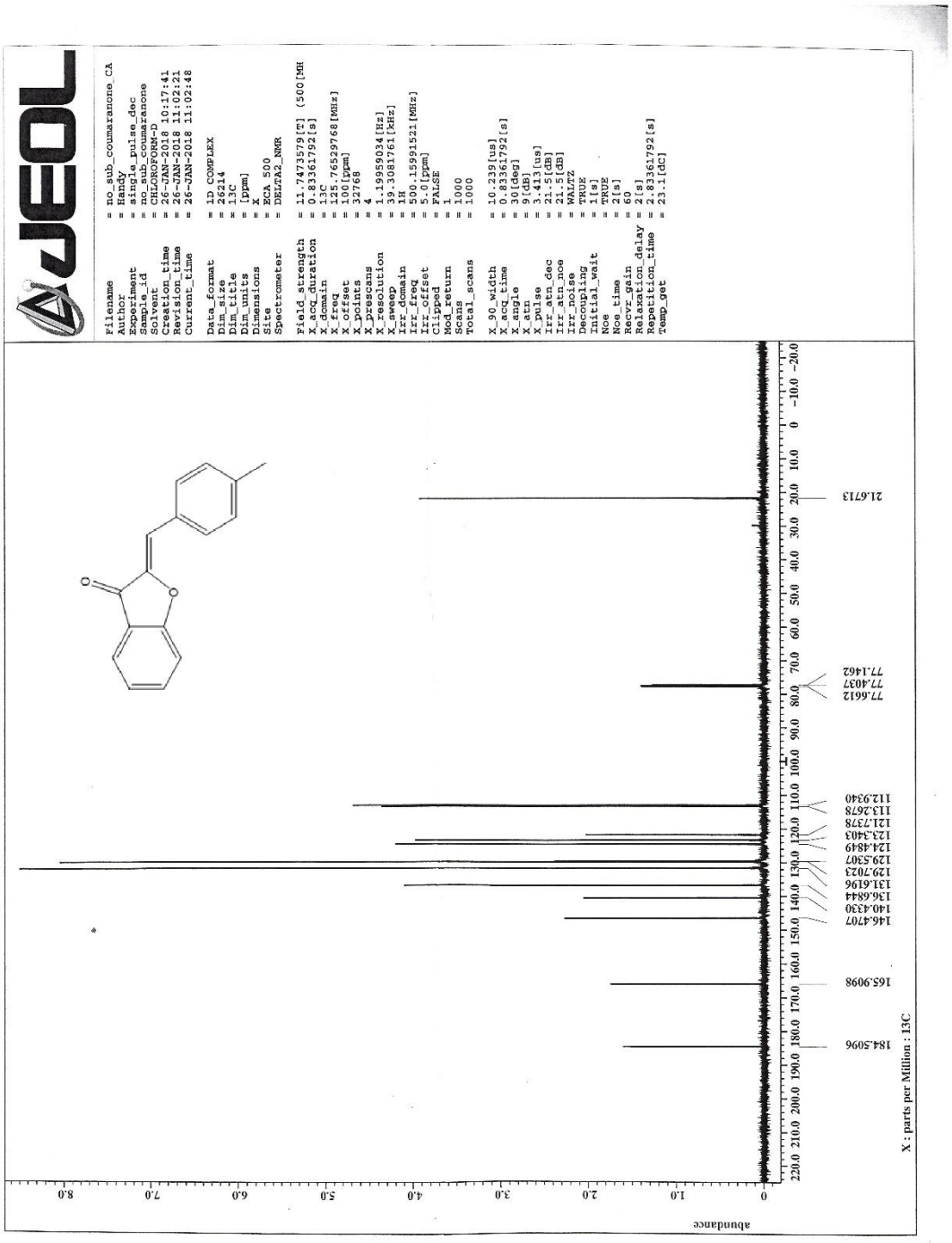

Spectrum

Line#:1 R.Time:28.360(Scan#:4873)
MassPeaks:36
RawMode:Single 28.360(4873) BasePeak:44.05(9778)
BG Mode:None Group 1 - Event 1 Scan



2-(4-methylbenzylidene)-1-(benzofuran-3-one) [**1**]




JEOL
Filename = no_sub_coumarancne_CA
Author = Hindy
Experiment = single_pulse_dec
Sample_id = no_sub_coumarancne
Solvent = CHLOROFORM-D
Creation_time = 26-JAN-2018 10:17:41
Revision_time = 26-JAN-2018 11:02:21
Current_time = 26-JAN-2018 11:02:48
Data_format = 1D COMPLEX
Dim_size = 2214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.74735579[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 = 39.30881761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = PAUSE
Mod_return = 1000
Scans = 1000
Total_scans = 1000
X_90_width = 10.239[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.41[us]
Irr_atn_dec = 21.5[dB]
Irr_atn_noe = 21.5[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.1[dC]
21.6713
77.6612
77.4037
77.1462
112.9340
113.2678
121.7378
123.3403
124.4849
129.5307
129.7023
131.6196
136.6844
140.4330
146.4707
165.9098
184.5096
abundance
X : parts per Million : 13C

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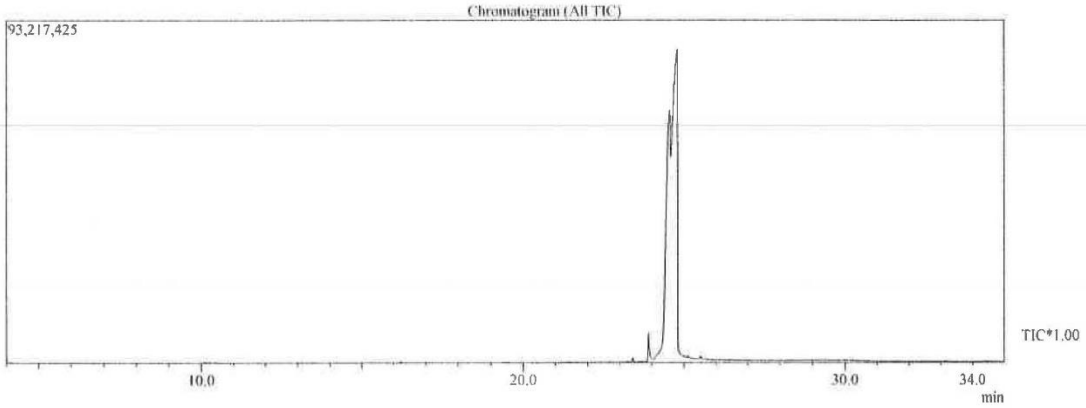


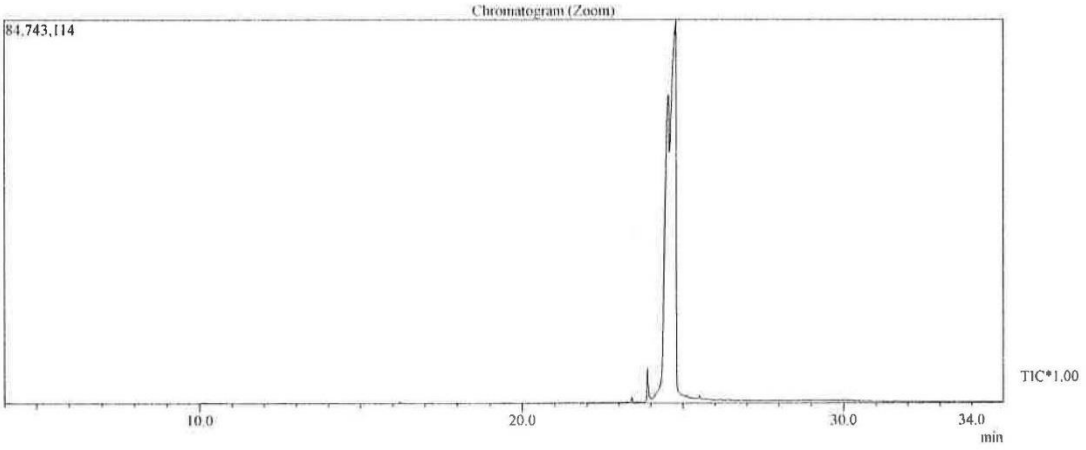


Spectrum

Line#:1 R.Time:24.745(Scan#:4150)
MassPeaks:185
RawMode:Single 24.745(4150) BasePeak:220.95(8428992)
BG Mode:None Group 1 - Event 1 Scan