Electronic Supplementary Material (ESI) for RSC Advances.

A concise route to functionalized benzofurans directly from *gem*-dibromoalkenes and phenols

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Supporting Information

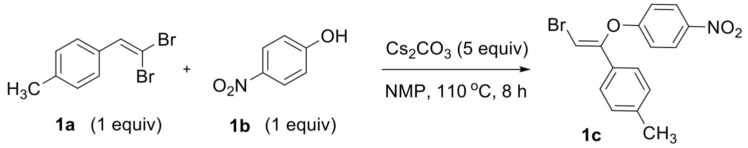
(Contents)

1. Preparation of (2,2-dibromovinyl)benzene:

To a round bottom flask, benzaldehyde (1 g, 9.42mmol, 1equiv) in 30 mL DCM and cooled at 0 °C under nitrogen atmosphere. Then, CBr_4 (6.2 g, 18.84 mmol, 2 equiv) and PPh_3 (9.9 g, 37.68 mmol, 4 equiv) were added successively in portions and stirred for 1 h under N_2. After that, the reaction mixture was quenched with hexane, filtered and the eluted part was concentrated for further purification by column chromatography (100-200 mesh silica-gel) using hexane as eluent. The desired product was obtained as pale yellow liquid (2.3 g, 93%) and is consistent with literature data.

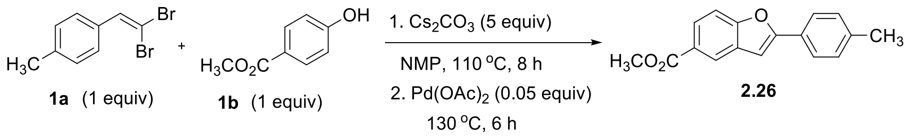
Ref. M. L. N. Rao, D. N. Jadhav and P. Dasgupta, *Org. Lett.*, 2010, 12, 2048-2051.

2. Preparation of 1c:



An oven-dried Schlenk tube under N_2 atmosphere was charged with 1-(2,2-dibromovinyl)-4-methylbenzene **1a** (0.104 g, 0.375 mmol, 1 equiv), *p*-nitrophenol **1b** (0.52 g, 0.375 mmol, 1equiv), Cs_2CO_3 (0.611 g, 1.875 mmol, 5 equiv) and NMP (3 mL) respectively. The reaction mixture was stirred at 110 °C in an oil bath for 8 h under N_2. The mixture was cooled to r.t. and followed by work-up using ethylacetate (50 mL). The organic extract was washed with water (10 mL), brine (10 mL), dried over anhydrous $MgSO_4$ and concentrated. The crude was purified by silica-gel column chromatography using 2% EtOAc in hexane as eluent. Product **1c** was isolated as yellow viscous liquid (0.109 g, 87%). This procedure was also followed for the preparation of intermediate **3c** with (*E*)-(4,4-dibromobuta-1,3-dienyl)benzene and *p*-cyanophenol.

3. Gram scale reaction (2.26):



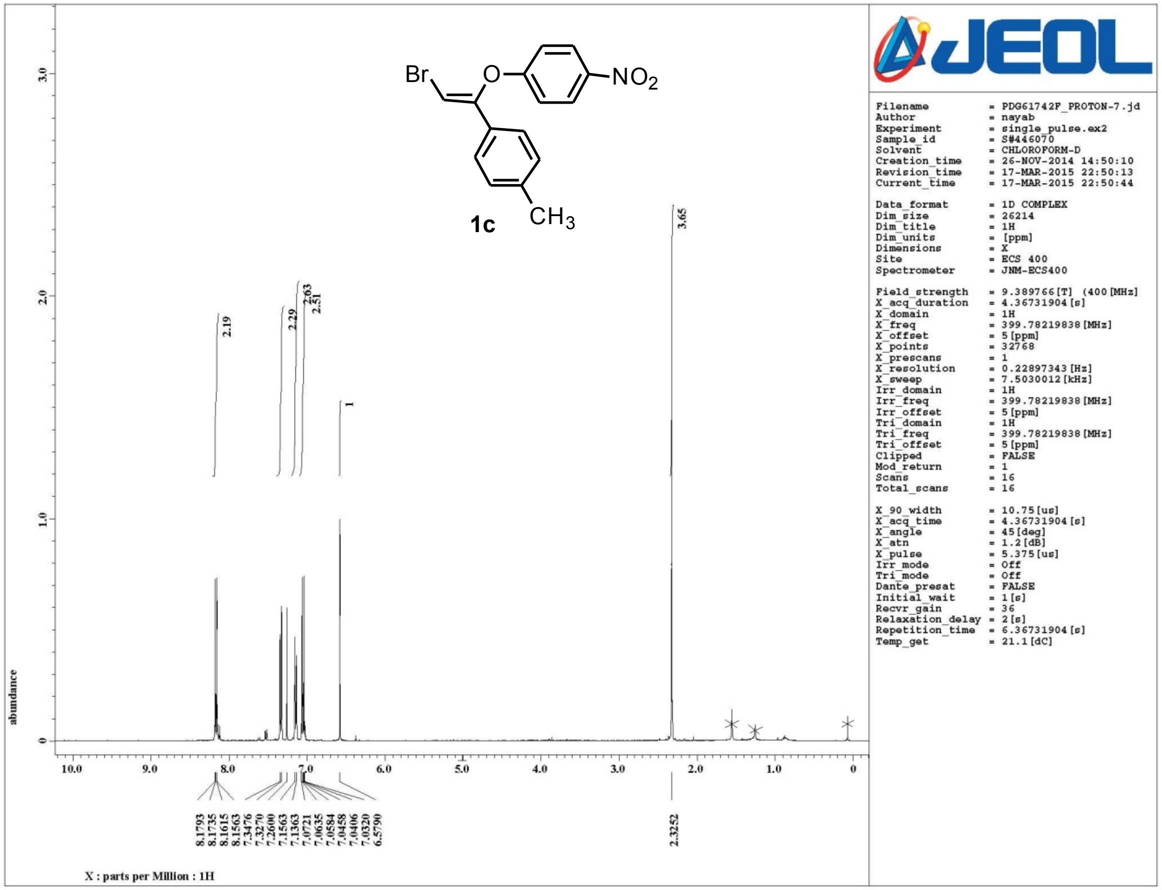
An oven-dried 100 mL Schlenk tube equipped with a magnetic pellet under N_2 atmosphere, added 1-(2,2-dibromovinyl)-4-methylbenzene **1a** (1 g, 3.62 mmol, 1 equiv), methyl 4-hydroxybenzoate (0.55 g, 3.62 mmol, 1 equiv), Cs_2CO_3 (5.9 g, 18.1 mmol, 5 equiv) and NMP (35 mL) respectively. The reaction mixture was stirred at 110 ^{o}C in an oil bath for 8 h under N_2. Then the mixture was cooled to r.t and added $Pd(OAc)_2$ (0.041 g, 0.181 mmol, 0.05 equiv) under N_2 and continued at 130 ^{o}C for 6 h. After cooling to r.t, the reaction mixture was transferred to a round-bottom flask to distill out NMP under reduced pressure. The obtained crude was diluted with ethyl acetate, passed through a column of silica-gel with repeated washings. The combined organic extract was concentrated to lowered volume and washed with water and brine. The ethyl acetate was concentrated and the crude was purified by using silica-gel column chromatography with 1% EtOAc in hexane to obtain **2.26** as a brown solid (0.405 g, 42%).

4. Spectral data of intermediates 4c and 5c:

4c. Pale yellow viscous liquid (0.095 g, 50%, as isomeric mixtures); R_f = 0.64 (EtOAc-hexane 1:9). ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.38 (d, 0.8H, J = 8.72 Hz, Ar-H), 7.28-7.25 (m, 6H, Ar-H), 7.16-7.13 (d, 0.4H, J = 8.72 Hz, Ar-H), 7.07-6.99 (m, 7H, Ar-H), 6.62 (m, 1H, Ar-H), 6.57-6.54 (m, 3H, Ar-H), 6.47-6.45 (m, 0.5H, Ar-H), 6.40 (d, 0.6H, J = 6.4 Hz, Ar-H), 6.36 (s, 2H, -C$\underline{H}_{olefin}$), 6.30 (s, 0.2H, -C$\underline{H}_{olefin}$), 2.30 (s, 8H, -C$\underline{H}_3$), 2.28 (s, 1H, -C$\underline{H}_3$). ^{13}C (100 MHz, CDCl$_3$): δ 157.0, 153.3 139.2, 130.6, 130.0, 129.4, 128.8, 125,8, 110.2, 105.2, 94.2, 21.3. IR (film): 3165, 2862, 1543, 1026, 667 cm^{-1}. HRMS (EI) calcd for $C_{24}H_{20}Br_2O_2$ [M$^+$] 497.9830; found 497.9839. The intermediate **4c** is susceptible for decomposition on standing at room temperature.

5c. Yellow viscous liquid (0.084 g, 38%, as isomeric mixtures); R_f = 0.20 (EtOAc-hexane 1:9). ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.42 (d, 0.4H, J = 9.16 Hz, Ar-H), 7.30-7.21 (m, 6H, Ar-H), 6.78-6.75 (m, 5H, Ar-H), 6.34 (s, 0.2H, -C$\underline{H}_{olefin}$), 6.32 (s, 2H, -C$\underline{H}_{olefin}$), 3.82 (s, 3H, -CO$_2$C$\underline{H}_3$), 3.77 (s, 6H, -C$\underline{H}_3$). ^{13}C (100 MHz, CDCl$_3$): δ 166.0, 160.4 156.9, 152.7, 132.5, 131.0, 127.3, 125.5, 114.2, 113.7, 112.2, 111.9, 111.4, 109.8, 109.3, 93.6, 93.4, 55.3, 52.3. IR (film): 3165, 2824, 1703, 1643, 1496, 1138, 1057, 733 cm^{-1}. HRMS (EI) calcd for $C_{26}H_{22}Br_2O_6$ [M$^+$] 587.9783; found 587.9788.

5. ^{1}H ,^{13}C and HRMS spectra of intermediate 1c:



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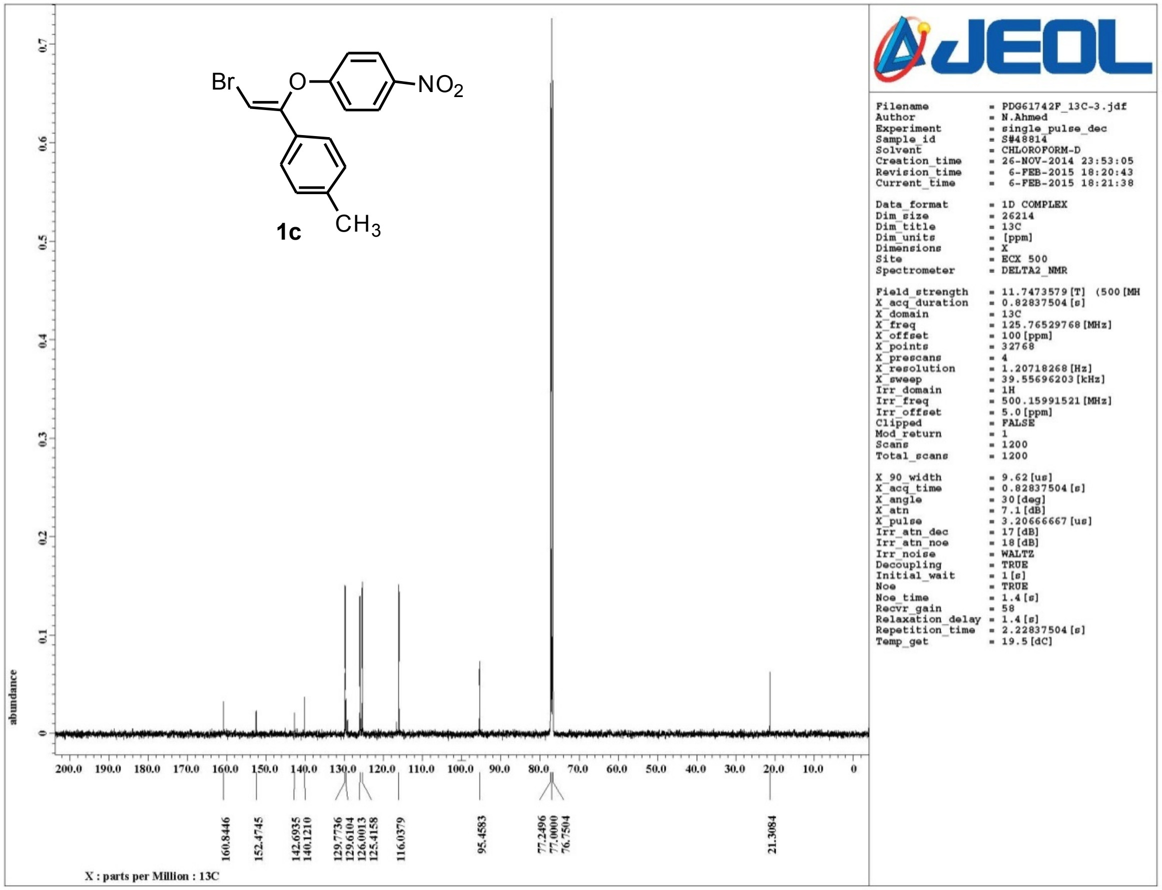
^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **1c**



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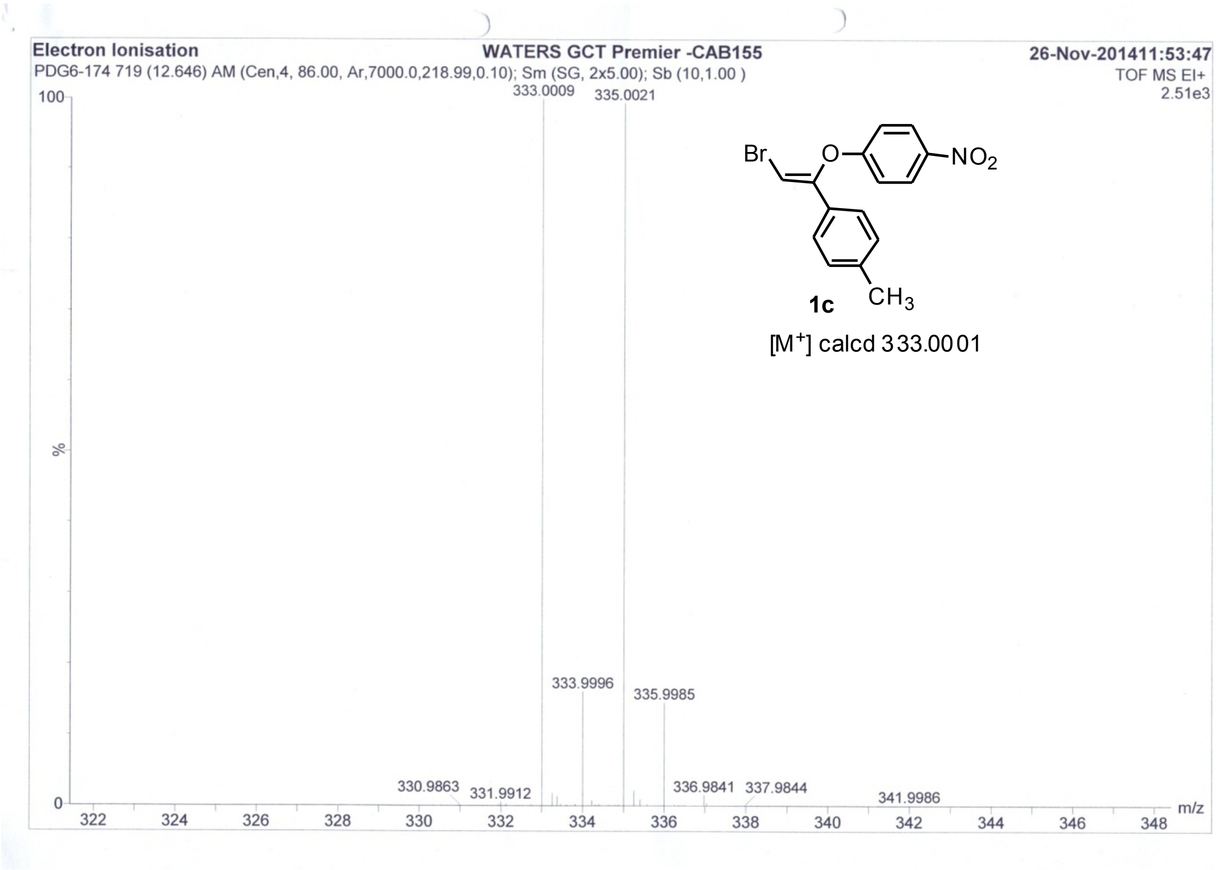
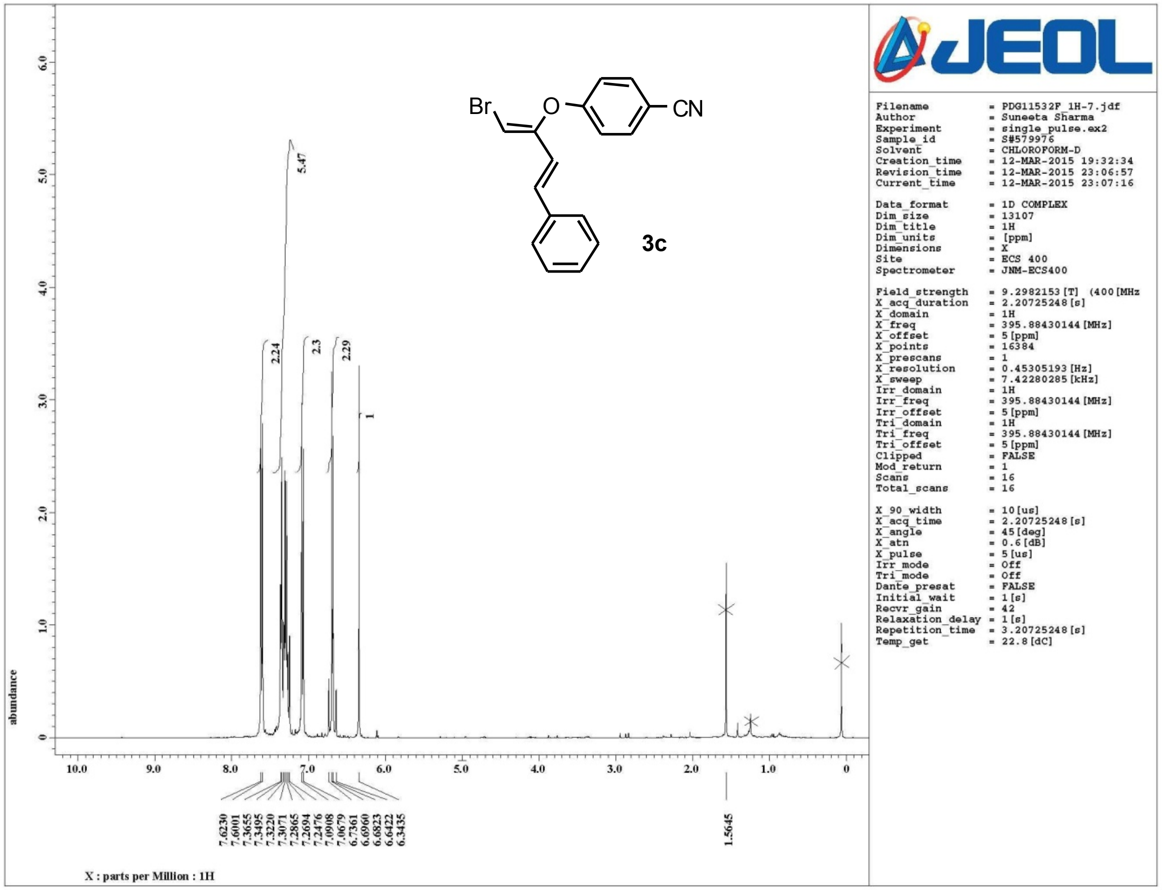


EI (HRMS) spectrum of **1c**

6. ^{1}H ,^{13}C and HRMS spectra of intermediate 3c:



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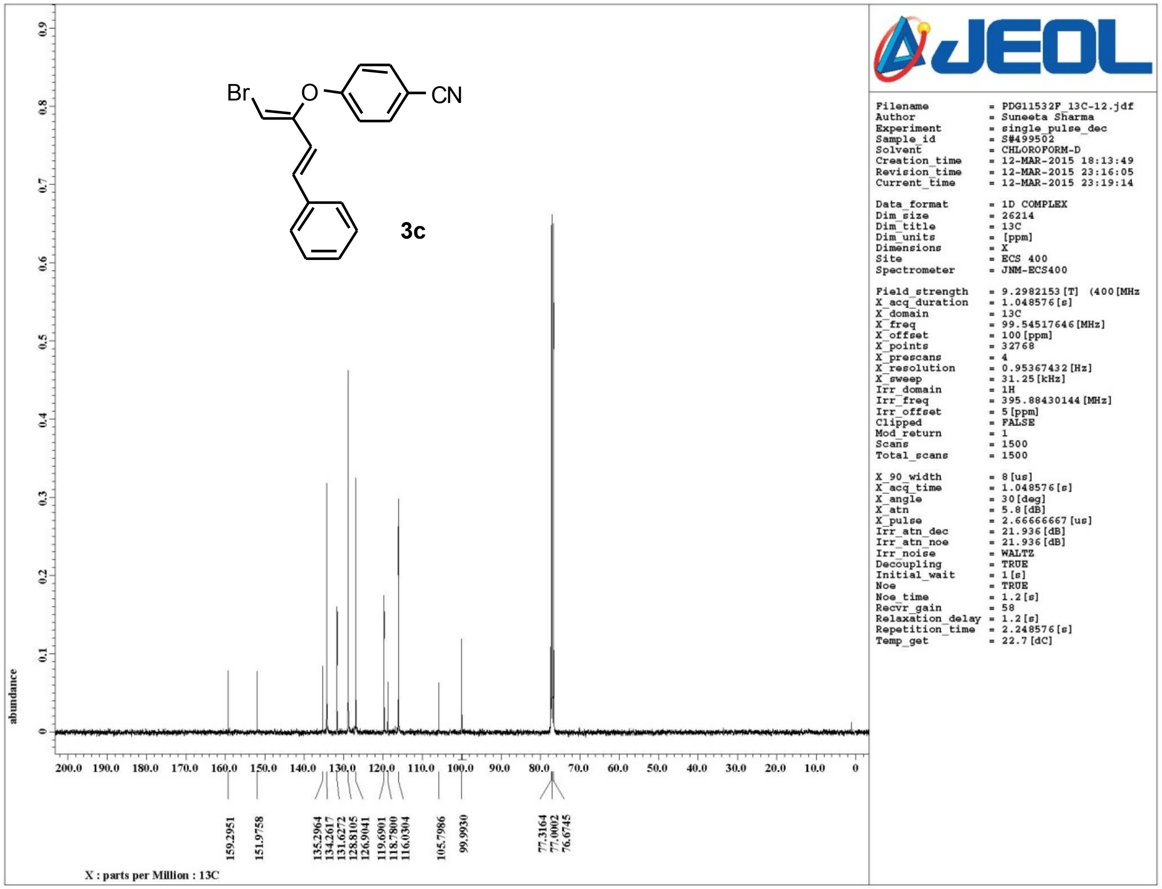
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **3c**



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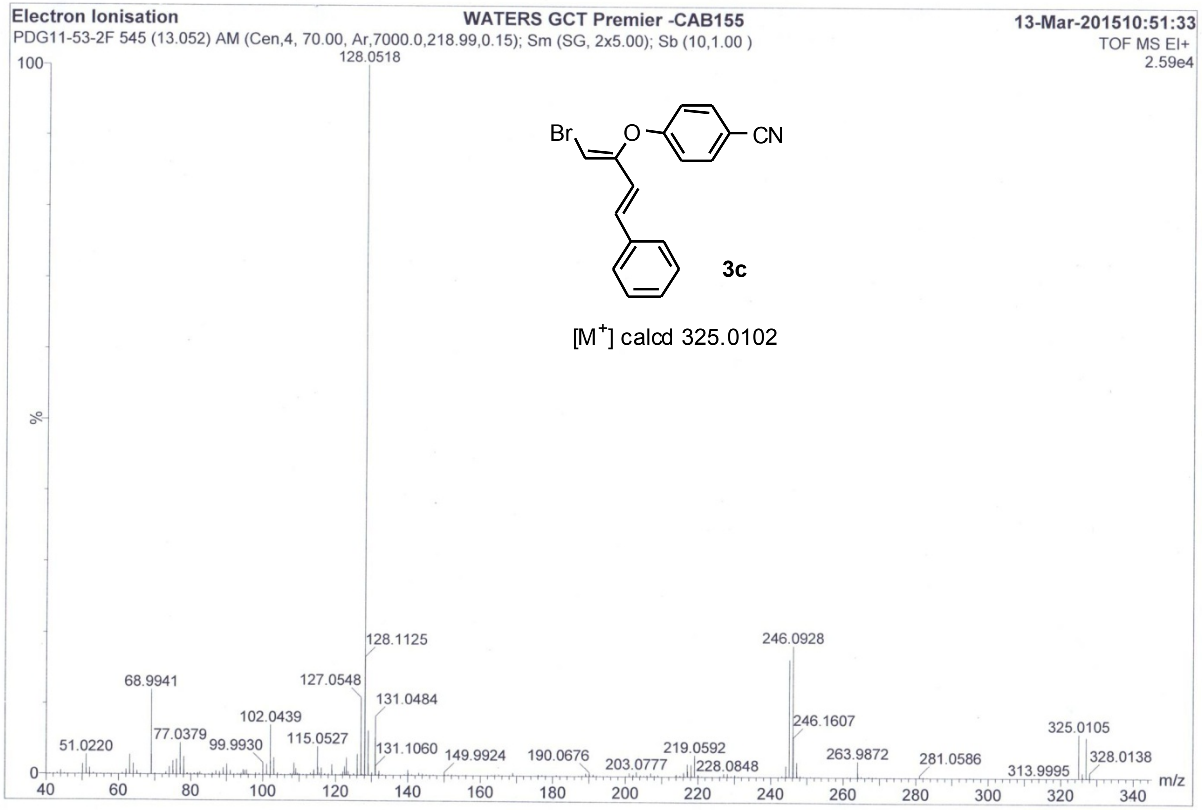
^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **3c**



EI (HRMS) spectrum of **3c**

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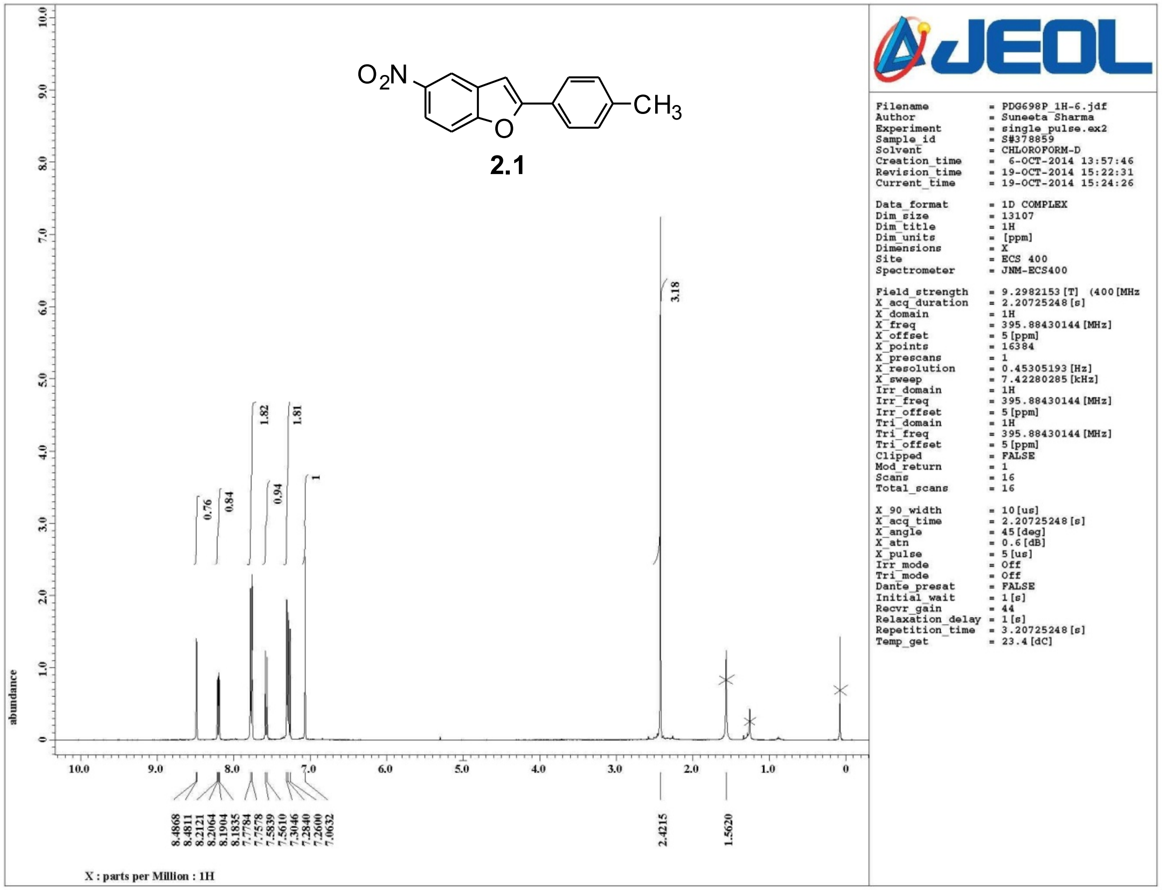

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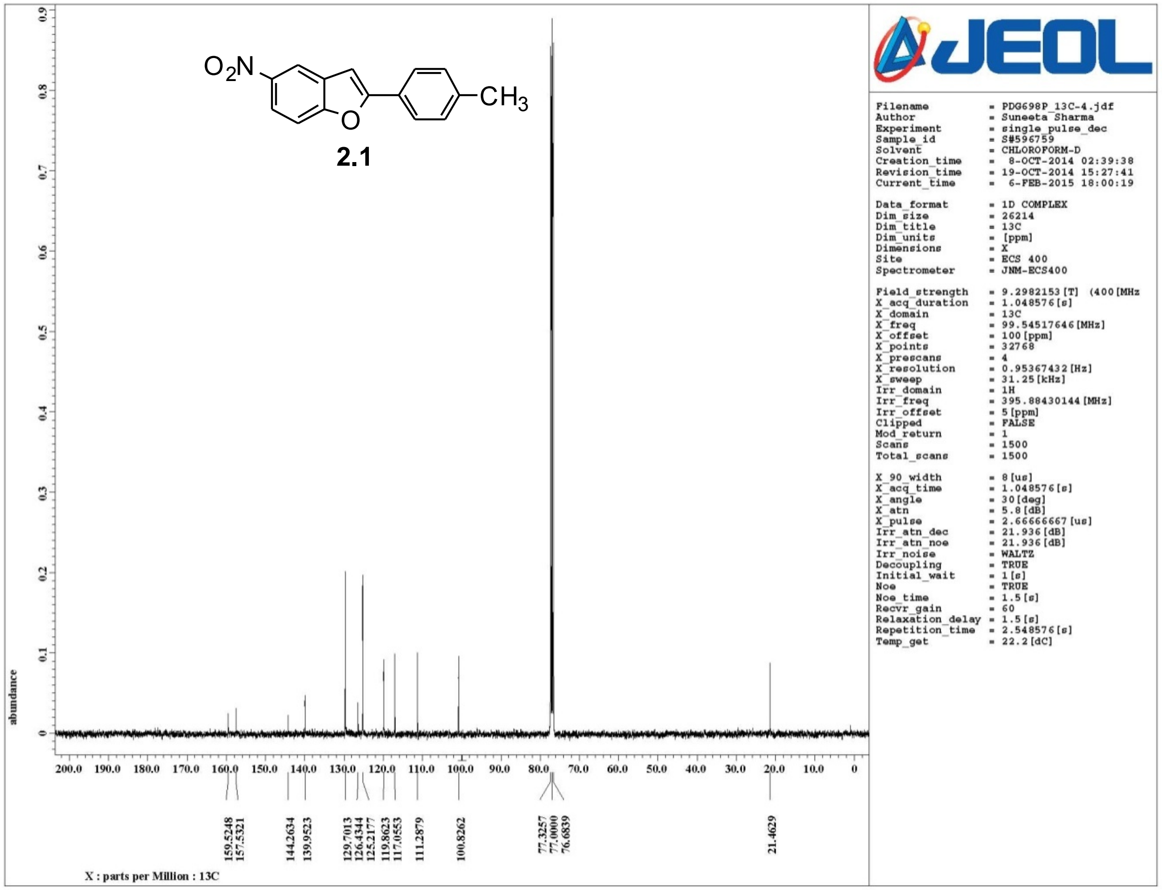
^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **2.1**



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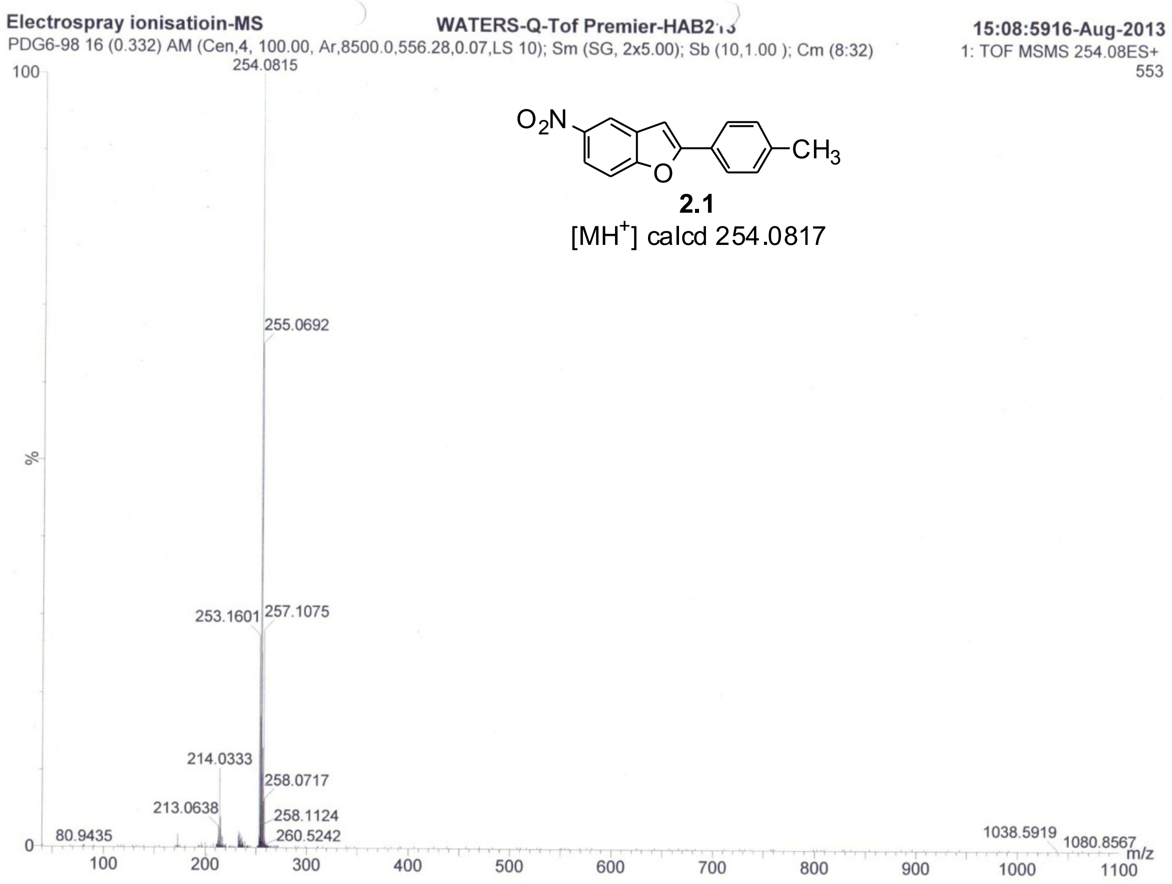
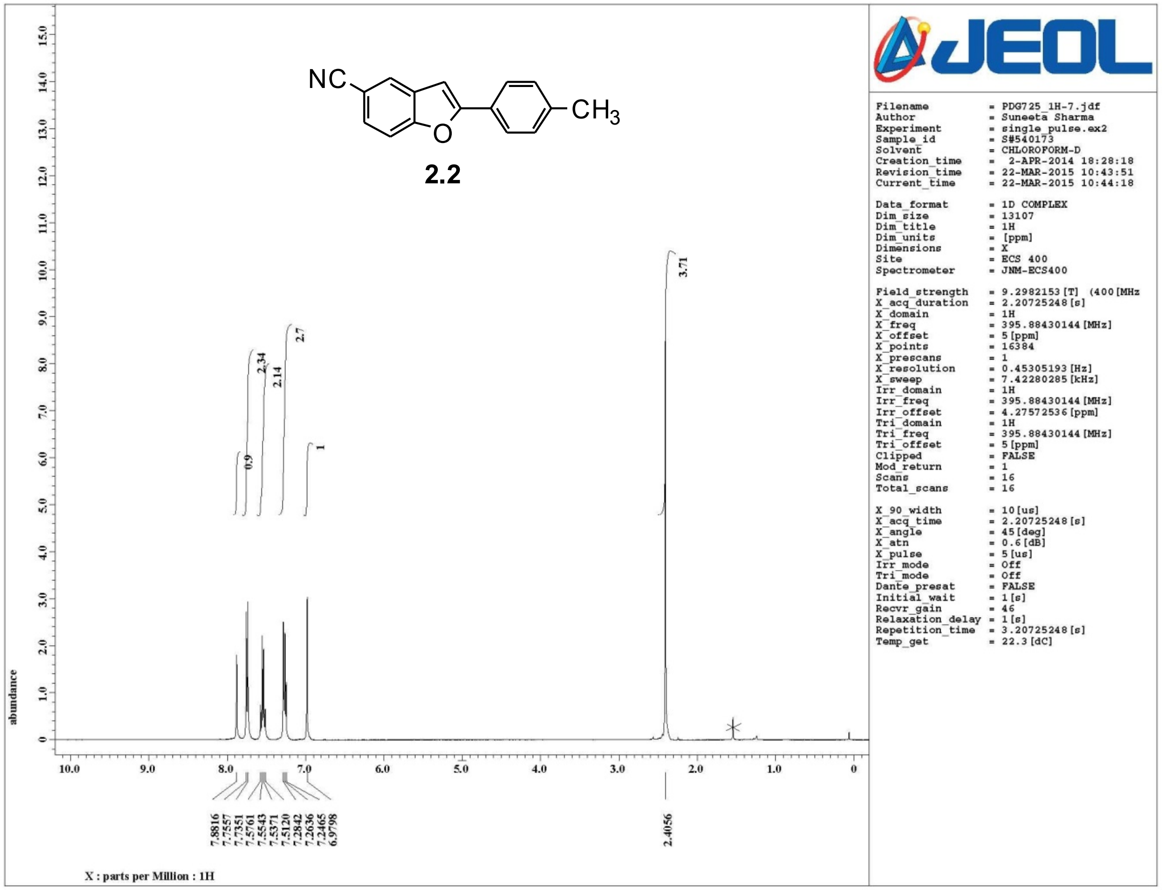
^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **2.1**



ESI (HRMS) spectrum of **2.1**



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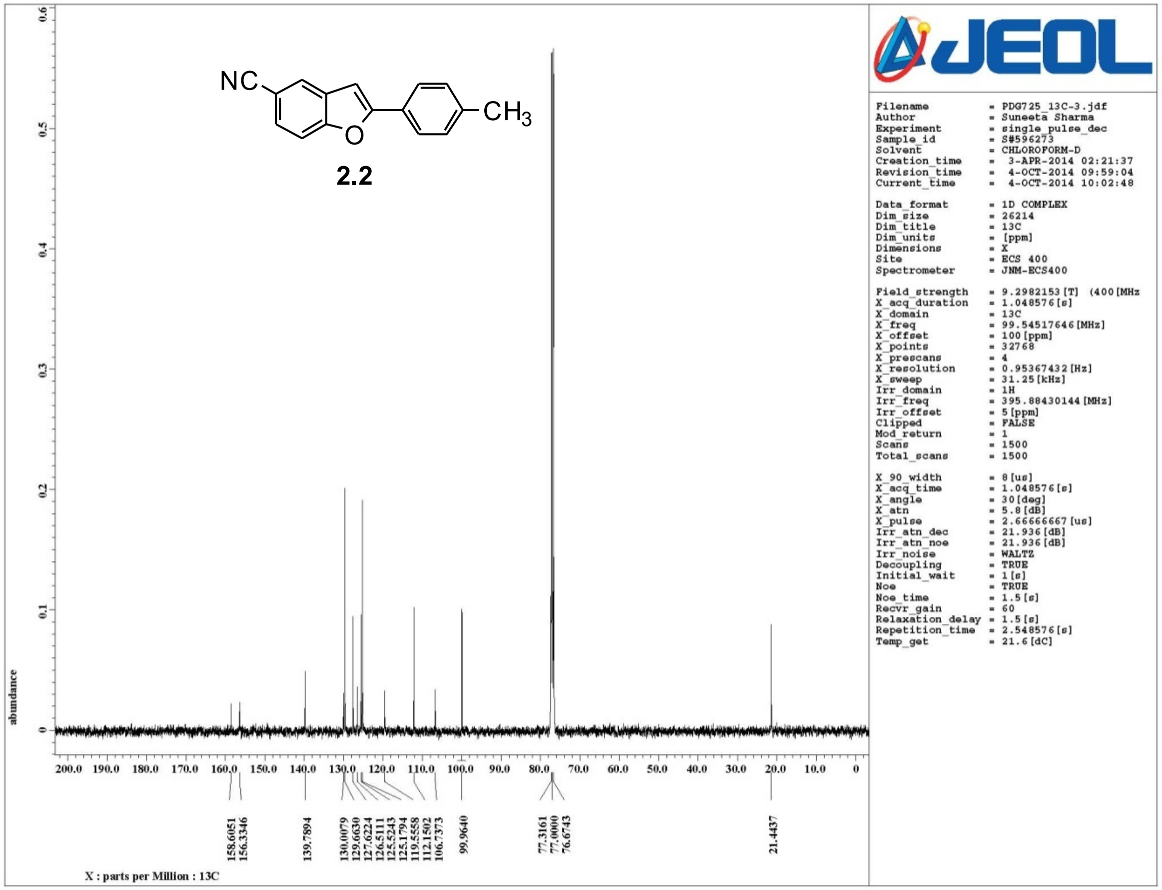
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **2.2**



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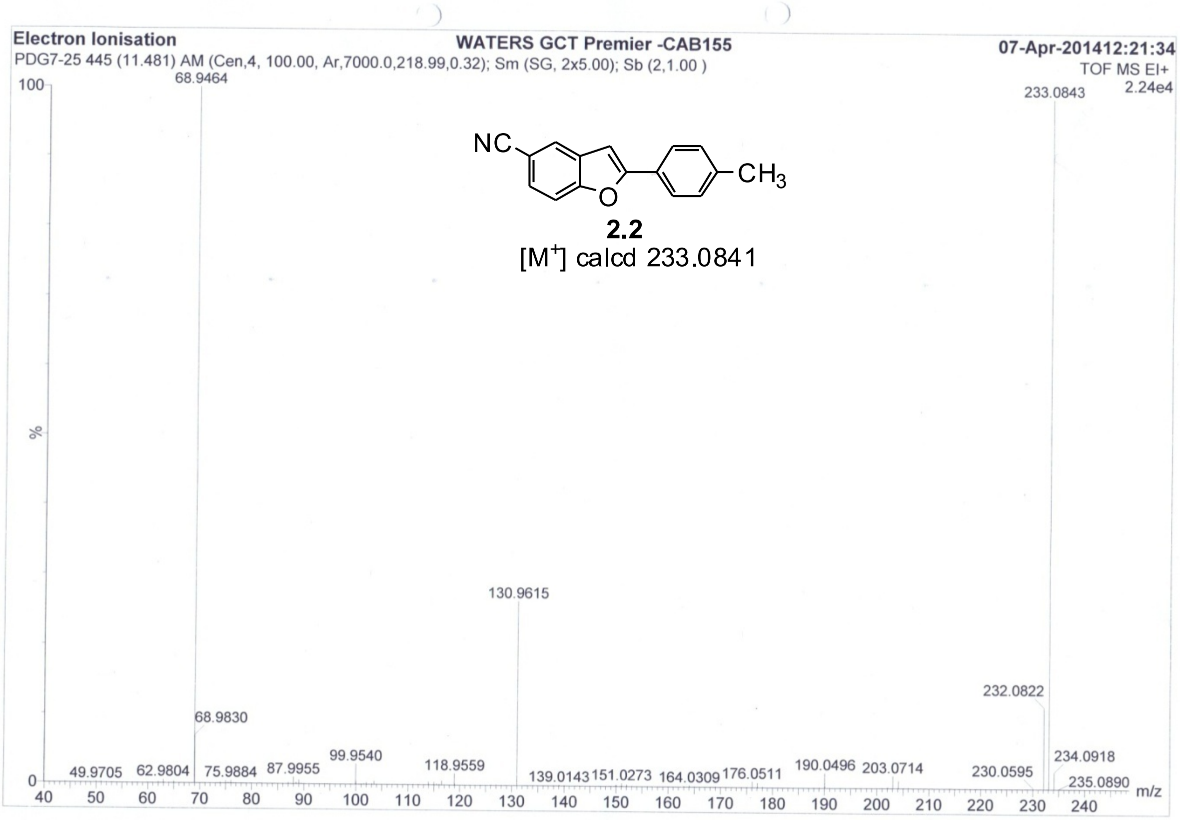
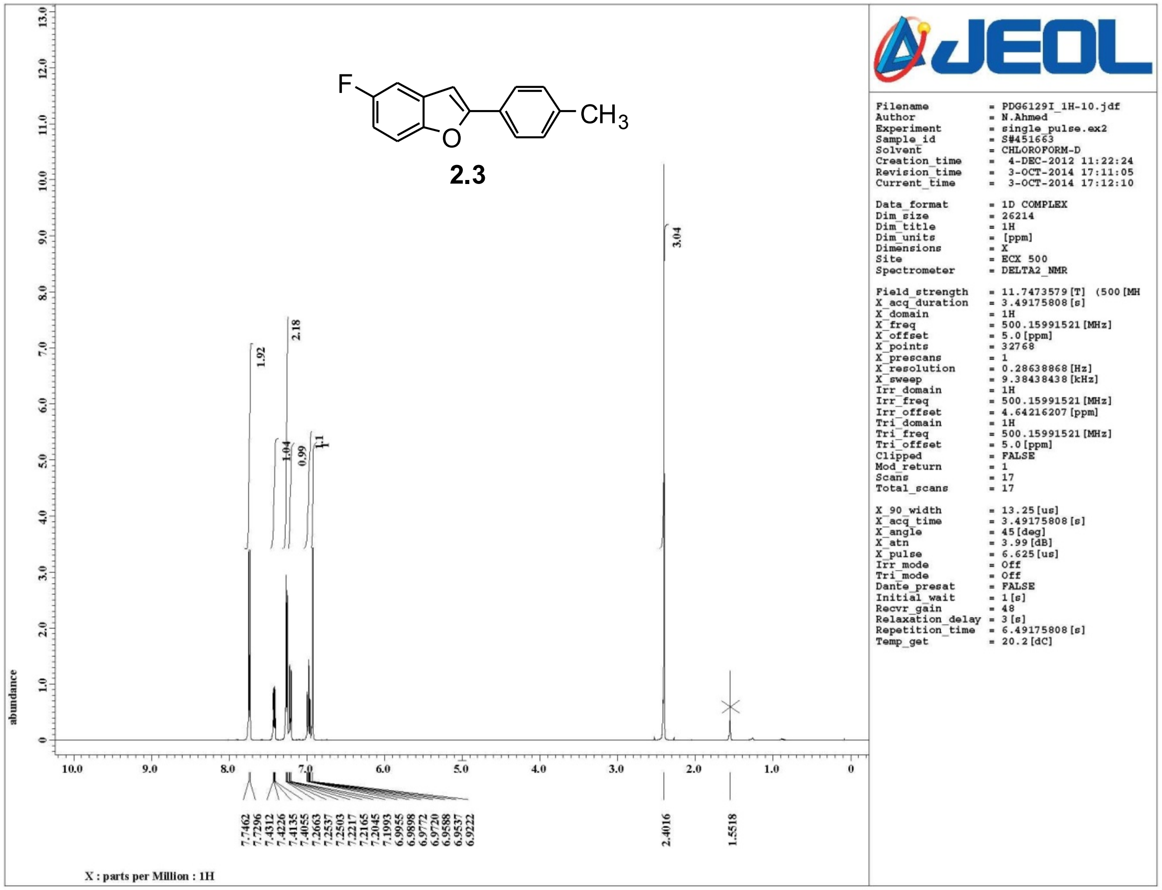
^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **2.2**

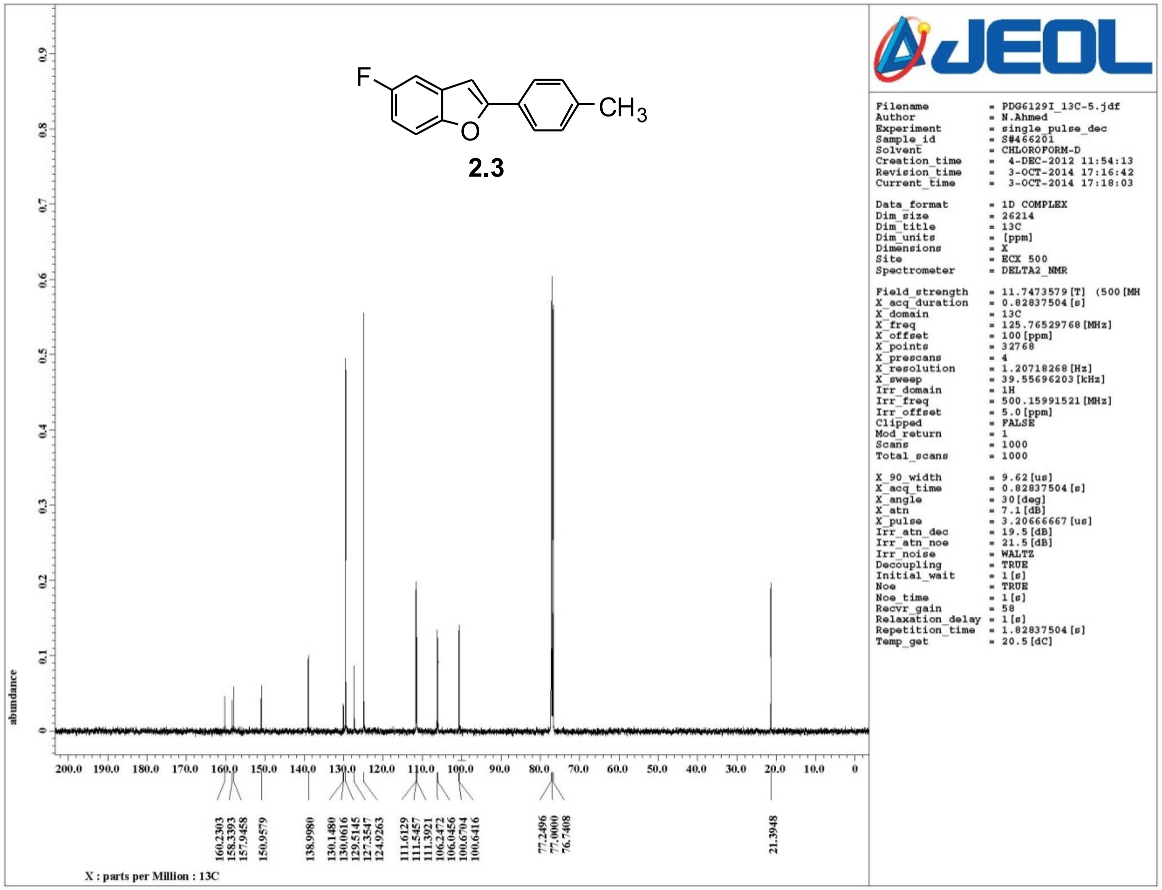


EI (HRMS) spectrum of **2.2**



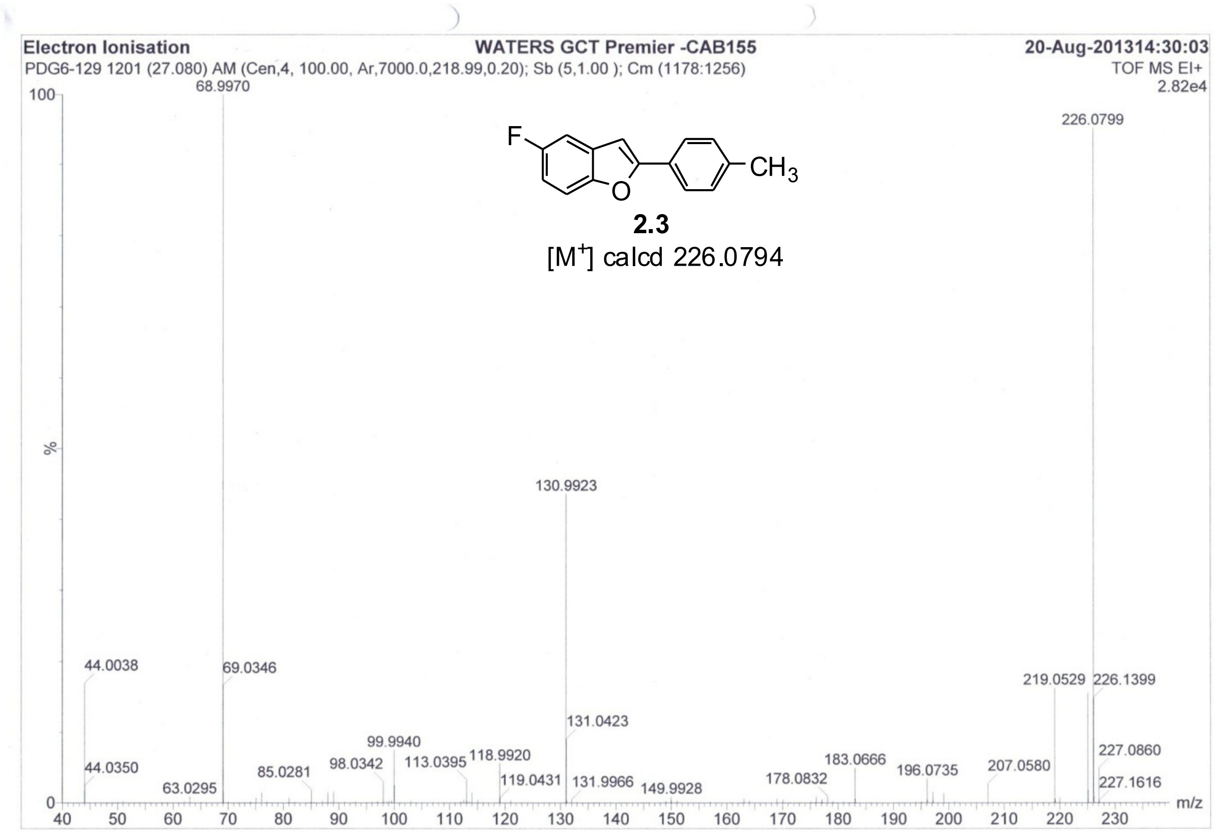
```
Filename          = PDG6129I_1H-10.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#451663
Solvent           = CHLOROFORM-D
Creation_time     =  4-DEC-2012 11:22:24
Revision_time     =  3-OCT-2014 17:11:05
Current_time      =  3-OCT-2014 17:12:10

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 3.49175808[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.28638868[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 4.64216207[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 17
Total_scans       = 17

X_90_width        = 13.25[us]
X_acq_time        = 3.49175808[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 3[s]
Repetition_time   = 6.49175808[s]
Temp_get          = 20.2[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.3**

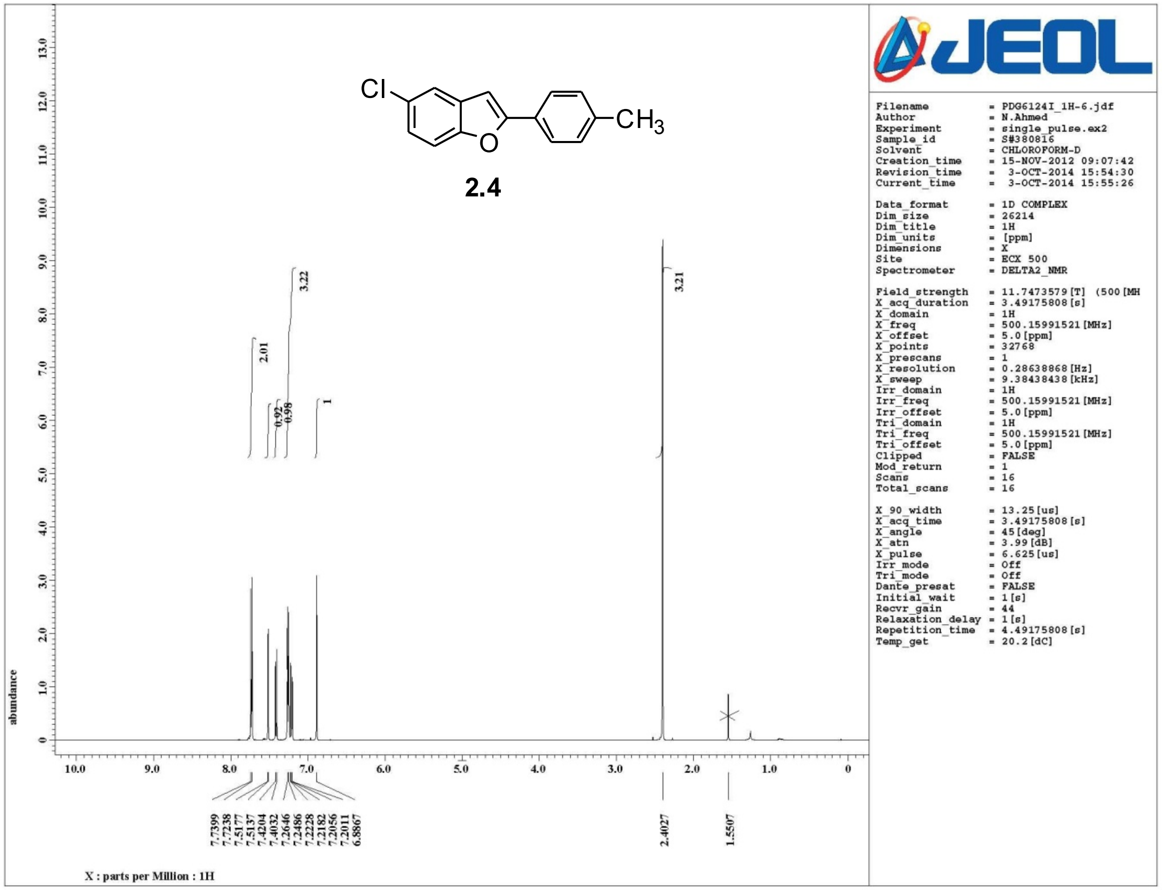


```
Filename          = PDG6129I_13C-5.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#466201
Solvent           = CHLOROFORM-D
Creation_time     =  4-DEC-2012 11:54:13
Revision_time     =  3-OCT-2014 17:16:42
Current_time      =  3-OCT-2014 17:18:03

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 20.5[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.3**

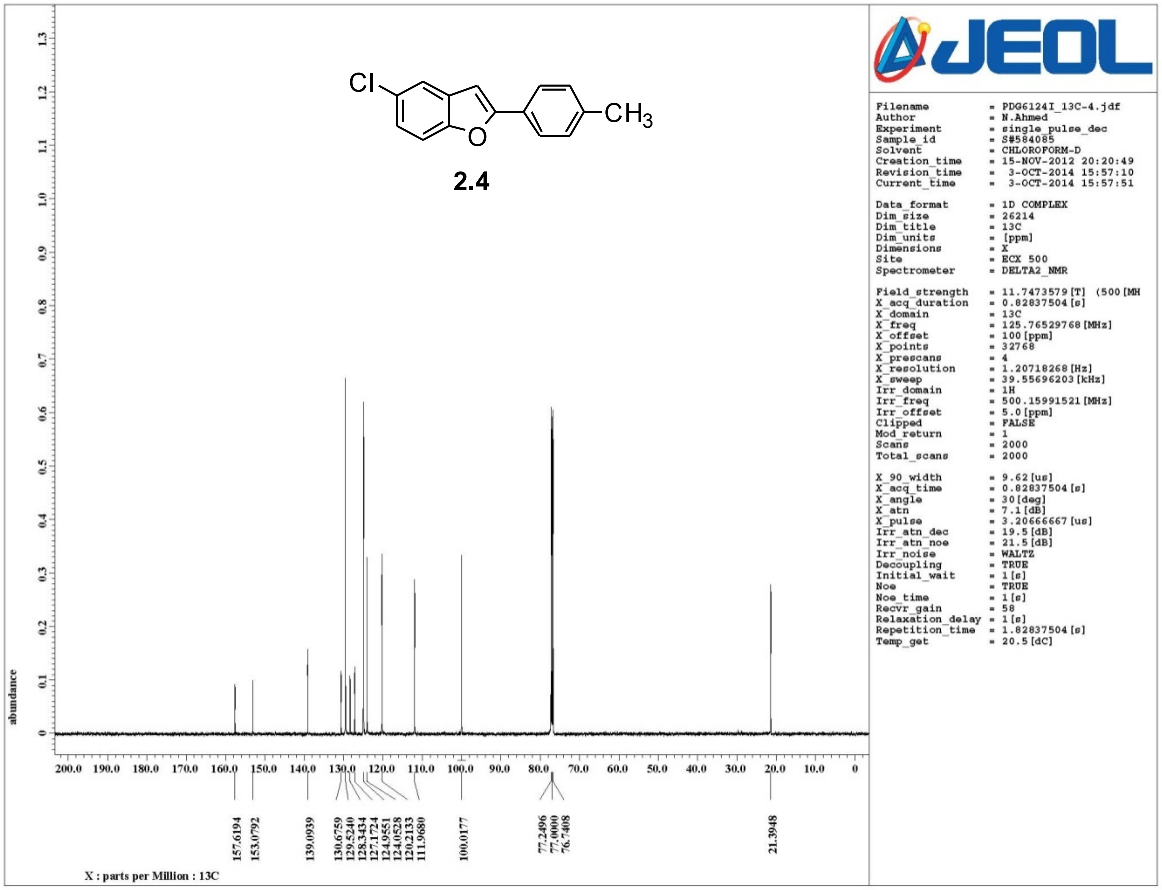


EI (HRMS) spectrum of **2.3**



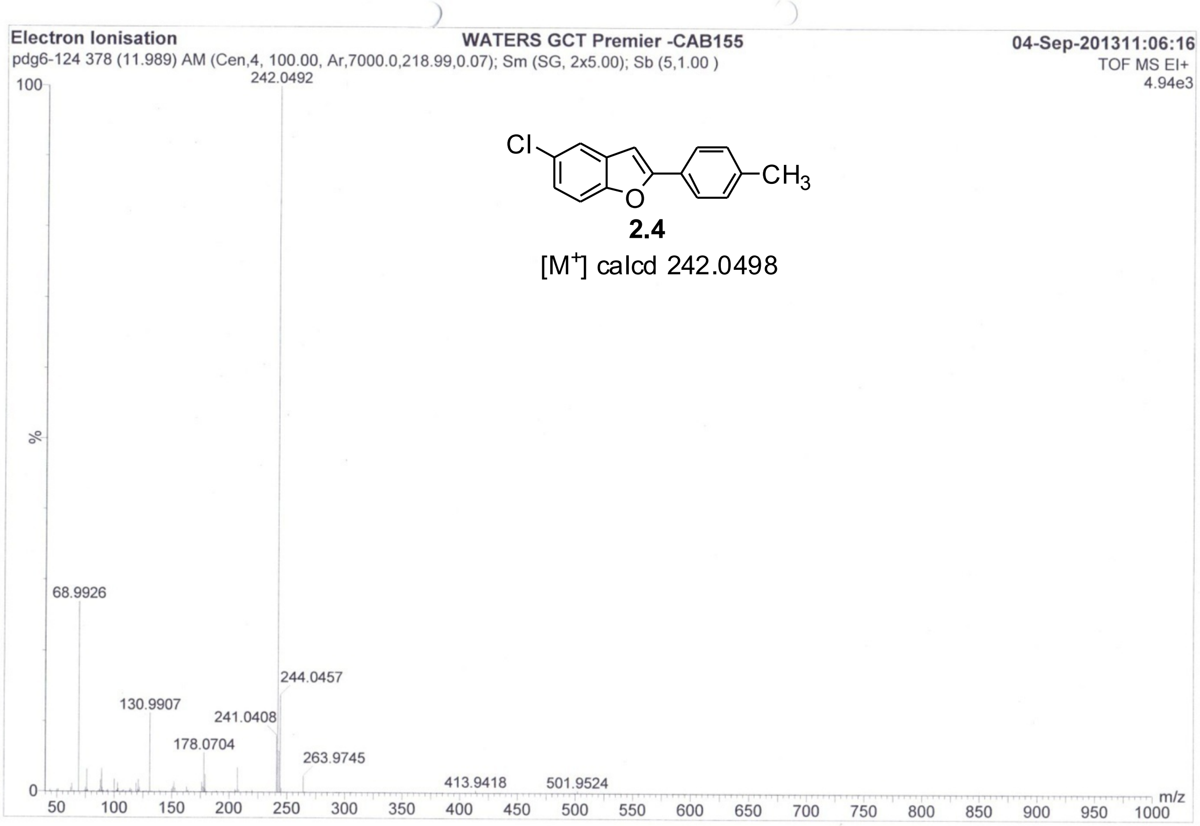
```
Filename            = PDG6124I_1H-6.jdf
Author              = N.Ahmed
Experiment          = single_pulse.ex2
Sample_id           = S#380816
Solvent             = CHLOROFORM-D
Creation_time       = 15-NOV-2012 09:07:42
Revision_time       =  3-OCT-2014 15:54:30
Current_time        =  3-OCT-2014 15:55:26

Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECX 500
Spectrometer        = DELTA2_NMR

Field_strength      = 11.7473579[T] (500[MH
X_acq_duration      = 3.49175808[s]
X_domain            = 1H
X_freq              = 500.15991521[MHz]
X_offset            = 5.0[ppm]
X_points            = 32768
X_prescans          = 1
X_resolution        = 0.28638868[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          = 13.25[us]
X_acq_time          = 3.49175808[s]
X_angle             = 45[deg]
X_atn               = 3.99[dB]
X_pulse             = 6.625[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 44
Relaxation_delay    = 1[s]
Repetition_time     = 4.49175808[s]
Temp_get            = 20.2[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.4**

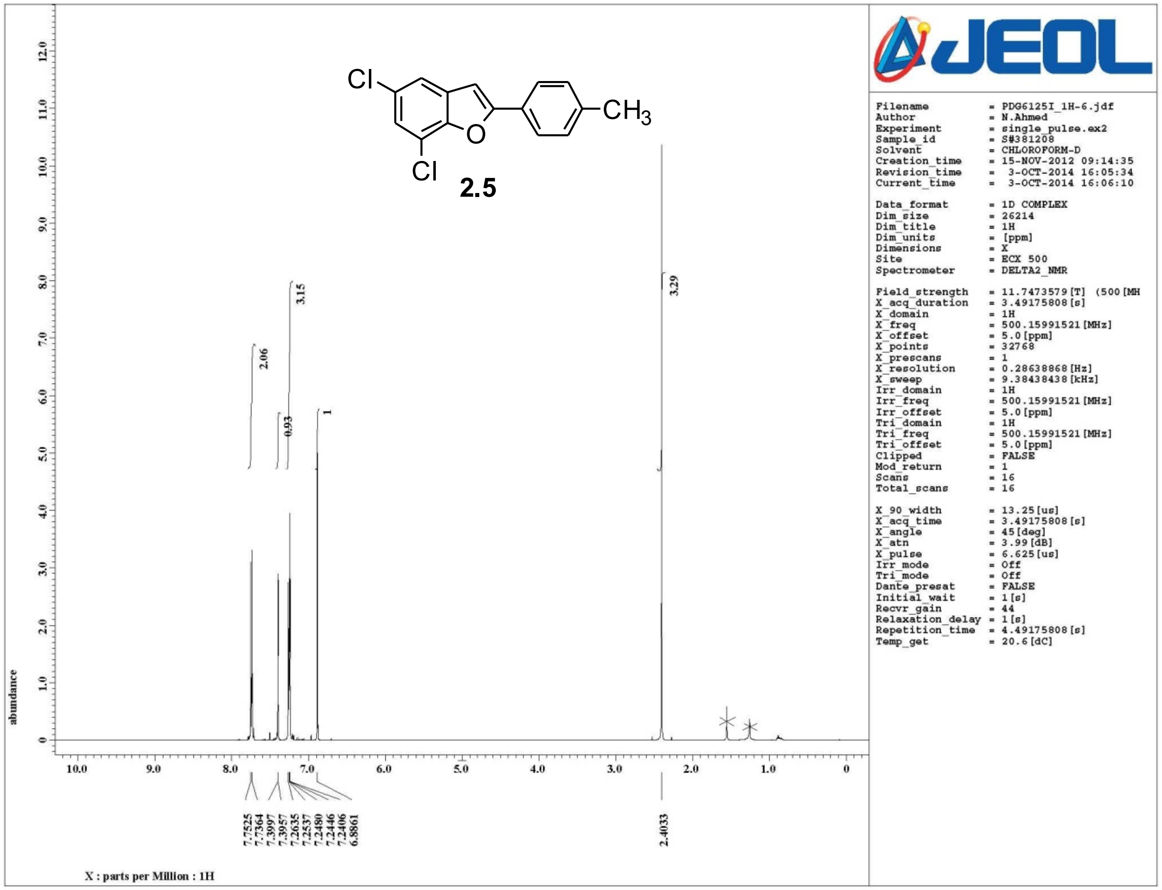


```
Filename           = PDG6124I_13C-4.jdf
Author             = N.Ahmed
Experiment         = single_pulse_dec
Sample_id          = S#584085
Solvent            = CHLOROFORM-D
Creation_time      = 15-NOV-2012 20:20:49
Revision_time      =  3-OCT-2014 15:57:10
Current_time       =  3-OCT-2014 15:57:51

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

Field_strength     = 11.7473579[T] (500[MH
X_acq_duration     = 0.82837504[s]
X_domain           = 13C
X_freq             = 125.76529768[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 1.20718268[Hz]
X_sweep            = 39.55696203[kHz]
Irr_domain         = 1H
Irr_freq           = 500.15991521[MHz]
Irr_offset         = 5.0[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 2000
Total_scans        = 2000

X_90_width         = 9.62[us]
X_acq_time         = 0.82837504[s]
X_angle            = 30[deg]
X_atn              = 7.1[dB]
X_pulse            = 3.20666667[us]
Irr_atn_dec        = 19.5[dB]
Irr_atn_noe        = 21.5[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 1[s]
Recvr_gain         = 58
Relaxation_delay   = 1[s]
Repetition_time    = 1.82837504[s]
Temp_get           = 20.5[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.4**

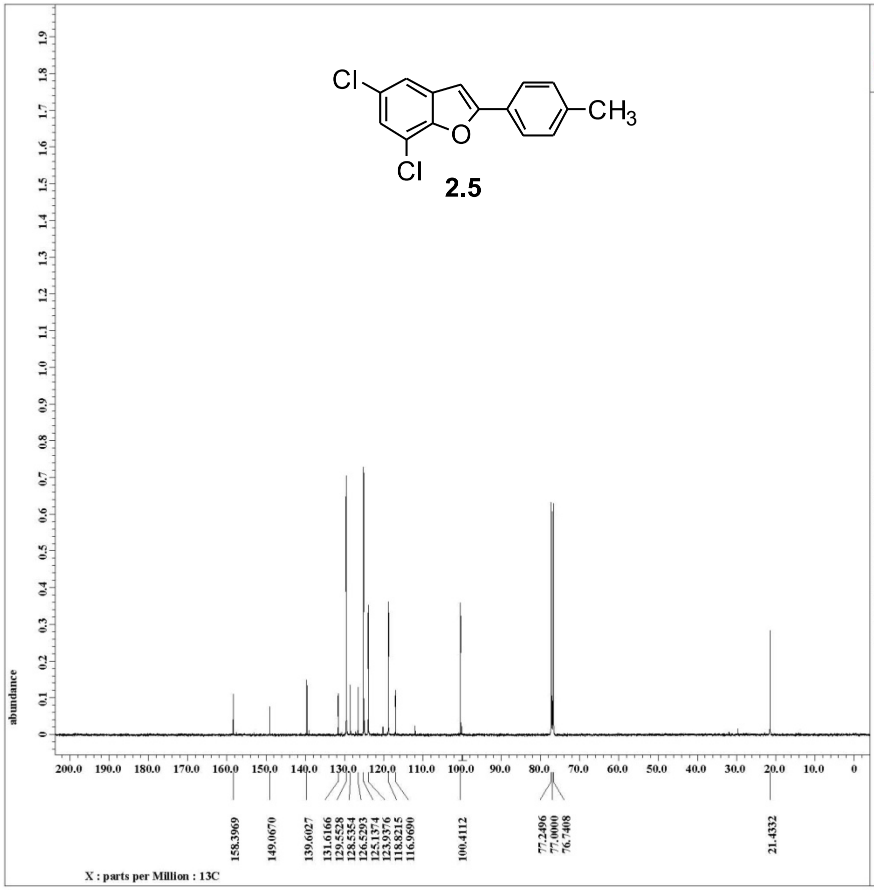


EI (HRMS) spectrum of **2.4**



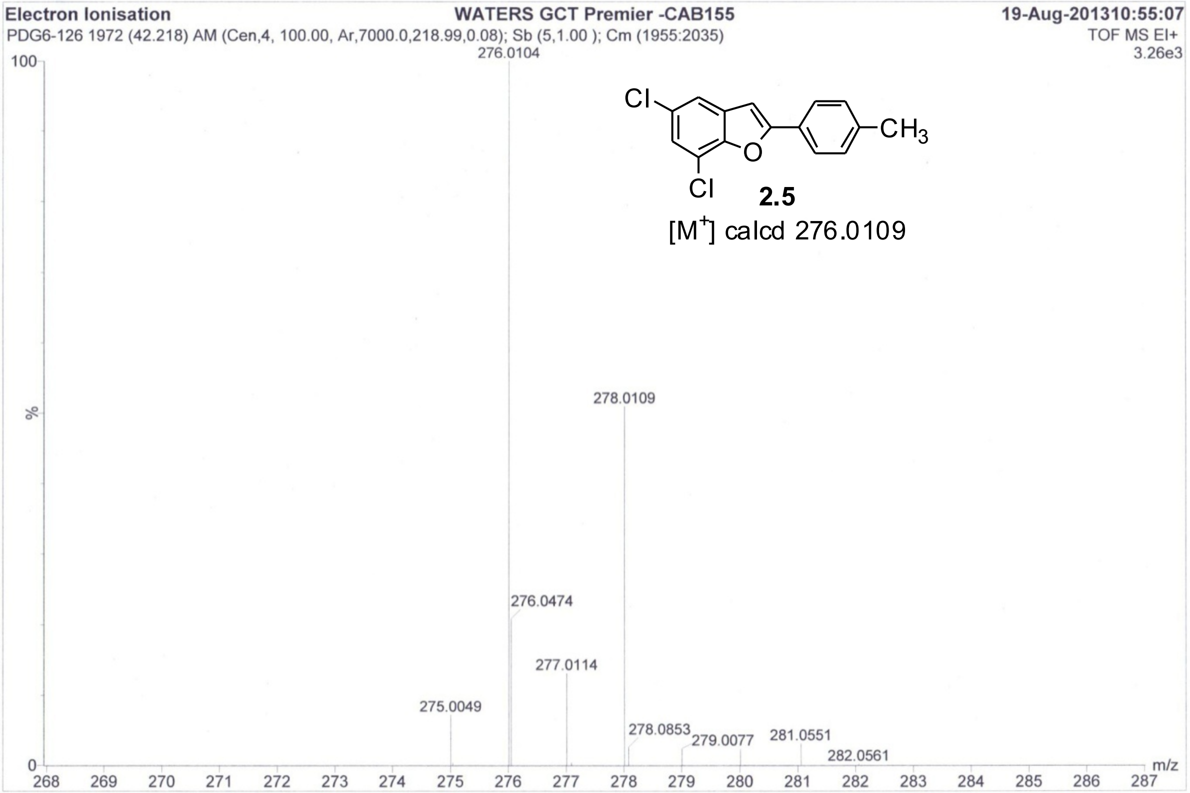
```
Filename          = PDG6125I_1H-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#381208
Solvent           = CHLOROFORM-D
Creation_time     = 15-NOV-2012 09:14:35
Revision_time     =  3-OCT-2014 16:05:34
Current_time      =  3-OCT-2014 16:06:10

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 3.49175808[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.28638868[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        = 13.25[us]
X_acq_time        = 3.49175808[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 1[s]
Repetition_time   = 4.49175808[s]
Temp_get          = 20.6[dC]
```

^{1}H NMR (500 MHz, CDCl$_3$) spectrum of **2.5**

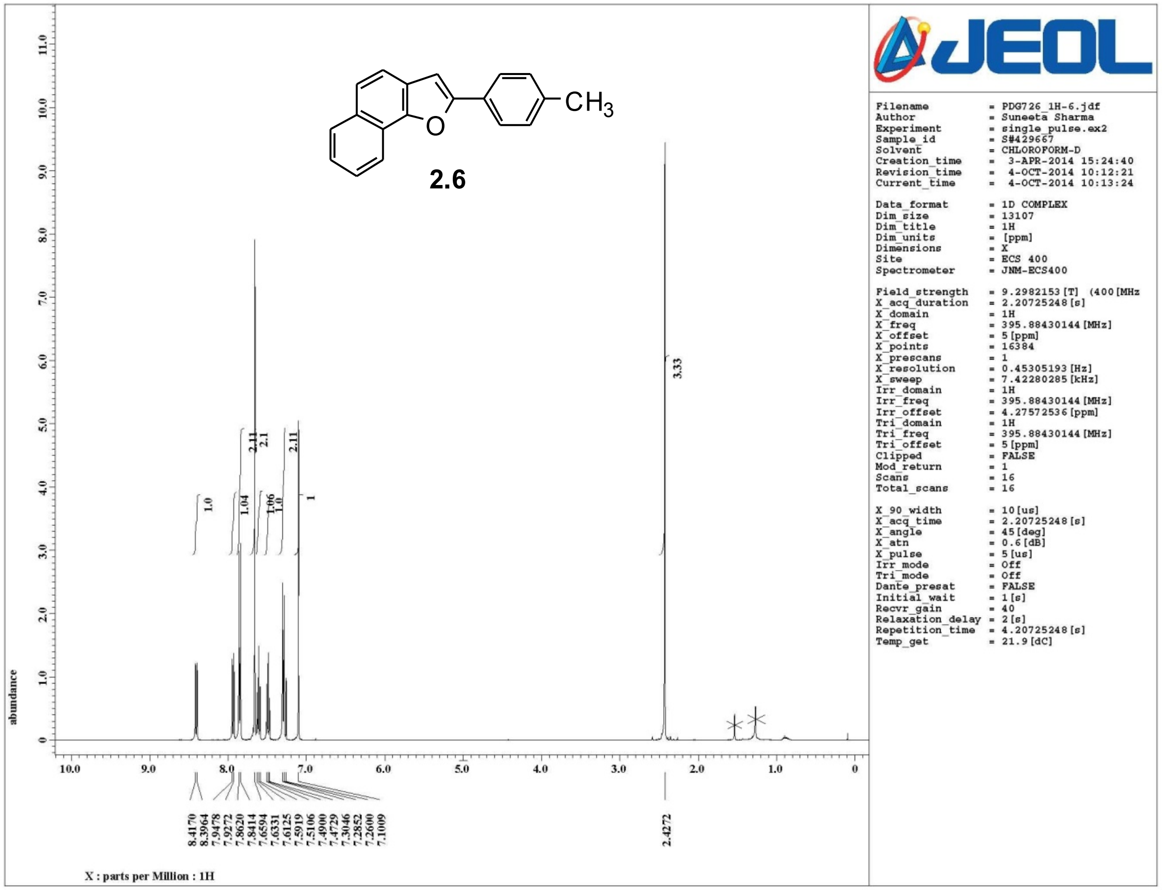


```
Filename          = PDG6125I_13C-4.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#584334
Solvent           = CHLOROFORM-D
Creation_time     = 15-NOV-2012 21:27:24
Revision_time     =  3-OCT-2014 16:08:00
Current_time      = 27-MAR-2015 11:14:43

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 2000
Total_scans       = 2000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 20.5[dC]
```

^{13}C NMR (125 MHz, CDCl$_3$) spectrum of **2.5**

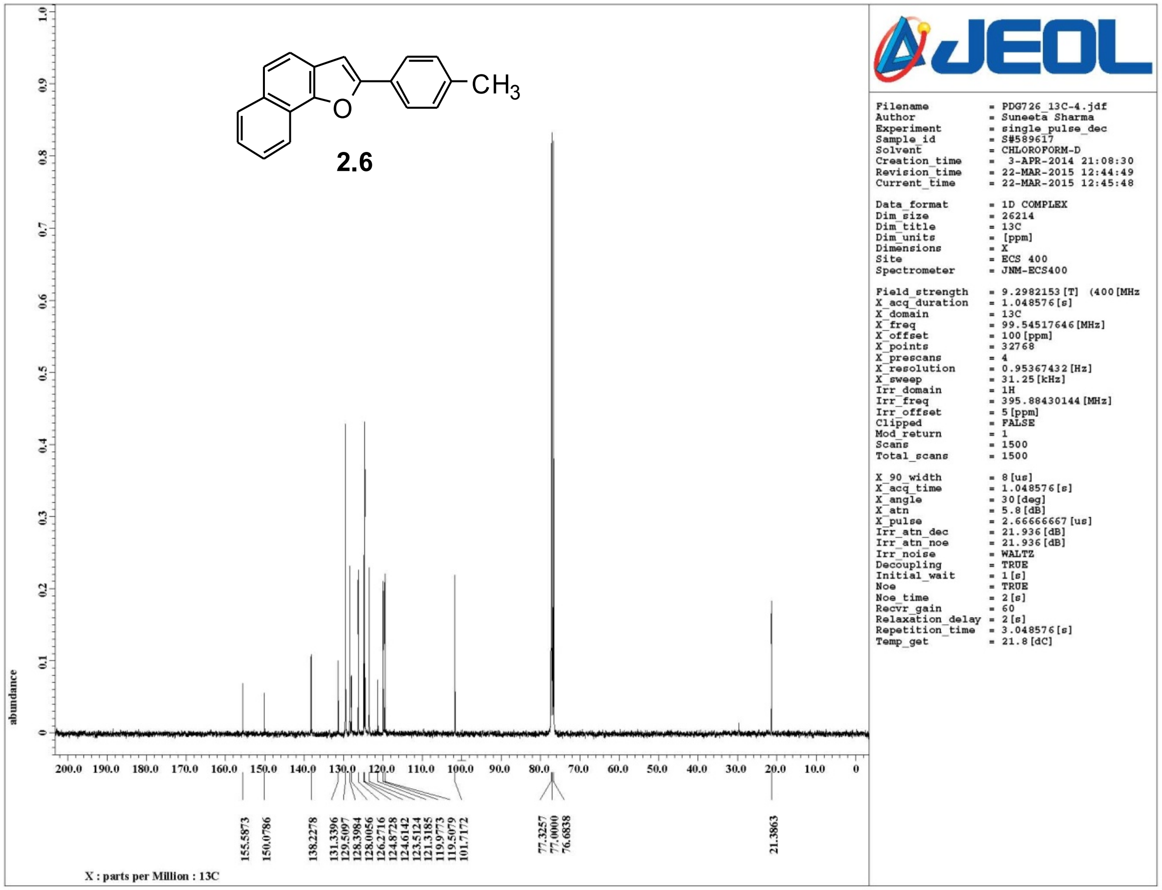


EI (HRMS) spectrum of **2.5**



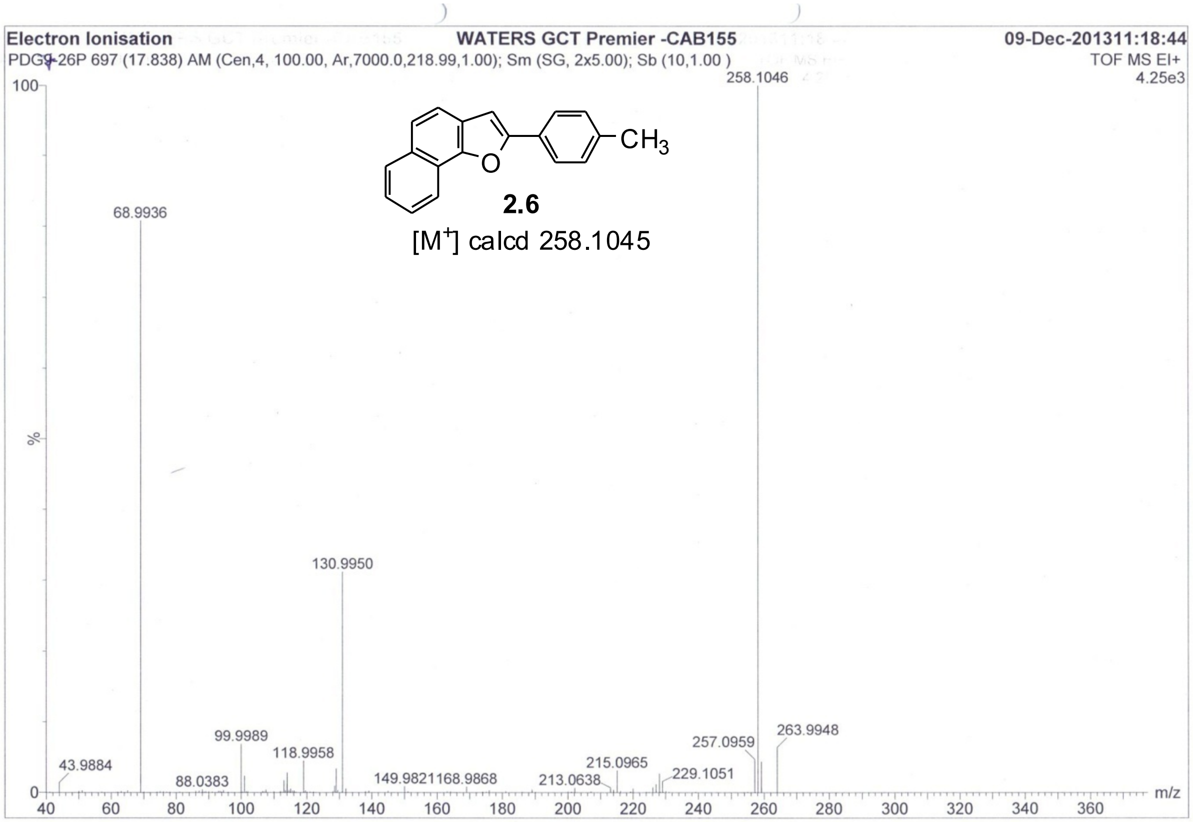
```
Filename          = PDG726_1H-6.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#429667
Solvent           = CHLOROFORM-D
Creation_time     =  3-APR-2014 15:24:40
Revision_time     =  4-OCT-2014 10:12:21
Current_time      =  4-OCT-2014 10:13:24

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 4.27572536[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 21.9[dC]
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **2.6**

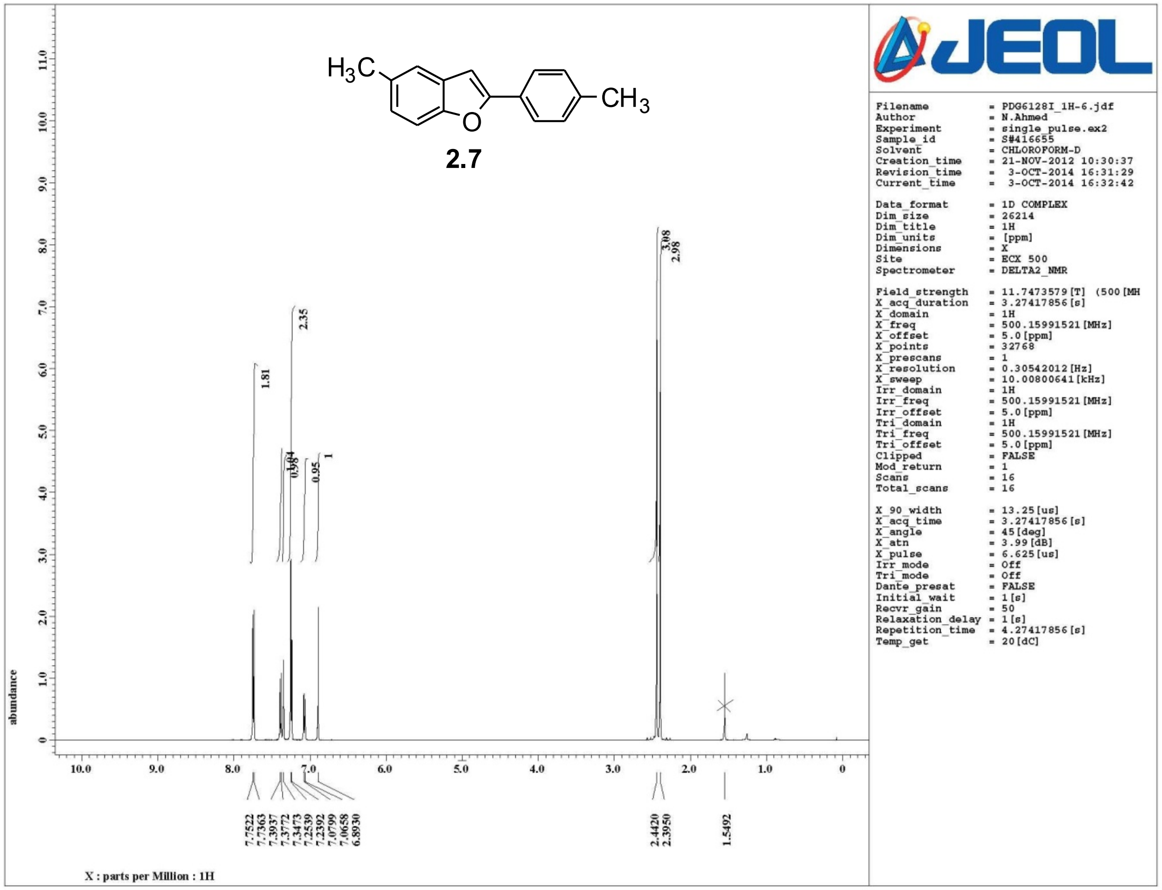


```
Filename          = PDG726_13C-4.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#589617
Solvent           = CHLOROFORM-D
Creation_time     =  3-APR-2014 21:08:30
Revision_time     = 22-MAR-2015 12:44:49
Current_time      = 22-MAR-2015 12:45:48

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1500
Total_scans       = 1500

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.048576[s]
Temp_get          = 21.8[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **2.6**

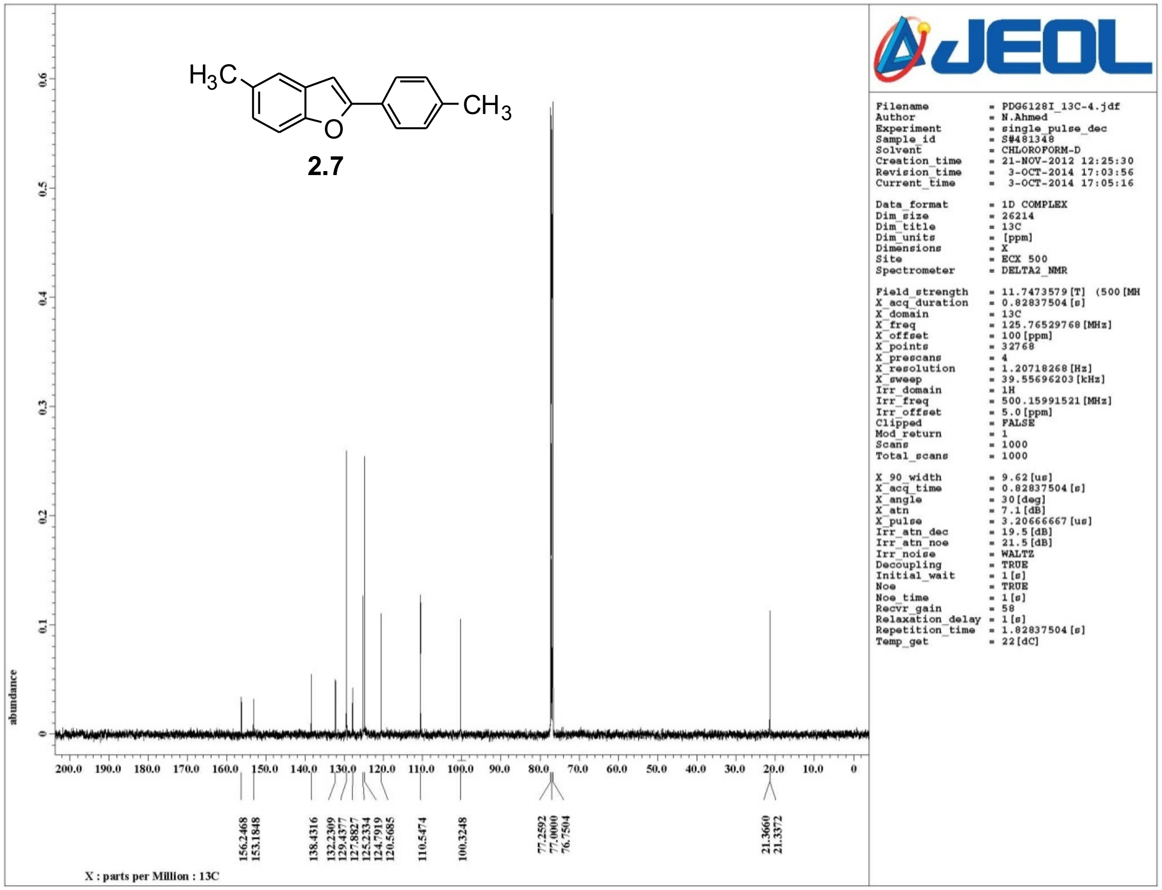


EI (HRMS) spectrum of **2.6**



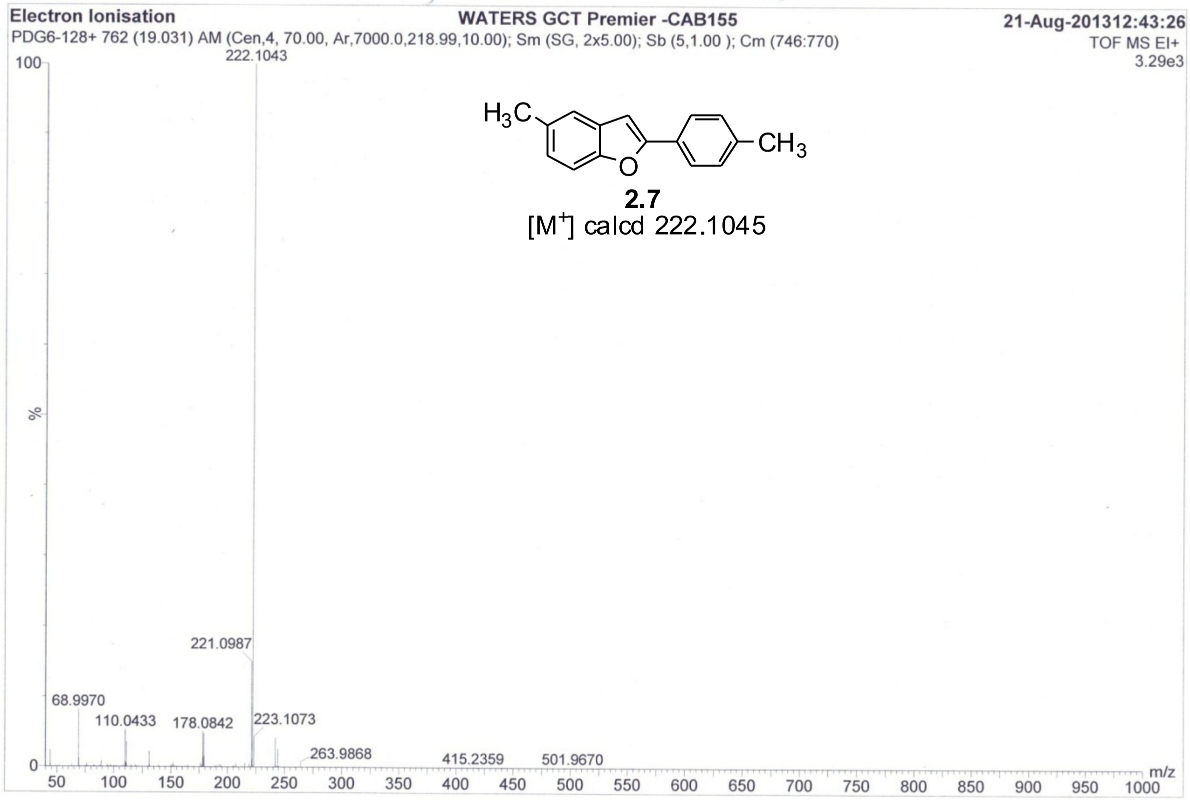
```
Filename            = PDG6128I_1H-6.jdf
Author              = N.Ahmed
Experiment          = single_pulse.ex2
Sample_id           = S#416655
Solvent             = CHLOROFORM-D
Creation_time       = 21-NOV-2012 10:30:37
Revision_time       =  3-OCT-2014 16:31:29
Current_time        =  3-OCT-2014 16:32:42

Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECX 500
Spectrometer        = DELTA2_NMR

Field_strength      = 11.7473579[T] (500[MH
X_acq_duration      = 3.27417856[s]
X_domain            = 1H
X_freq              = 500.15991521[MHz]
X_offset            = 5.0[ppm]
X_points            = 32768
X_prescans          = 1
X_resolution        = 0.30542012[Hz]
X_sweep             = 10.00800641[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          = 13.25[us]
X_acq_time          = 3.27417856[s]
X_angle             = 45[deg]
X_atn               = 3.99[dB]
X_pulse             = 6.625[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 50
Relaxation_delay    = 1[s]
Repetition_time     = 4.27417856[s]
Temp_get            = 20[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.7**

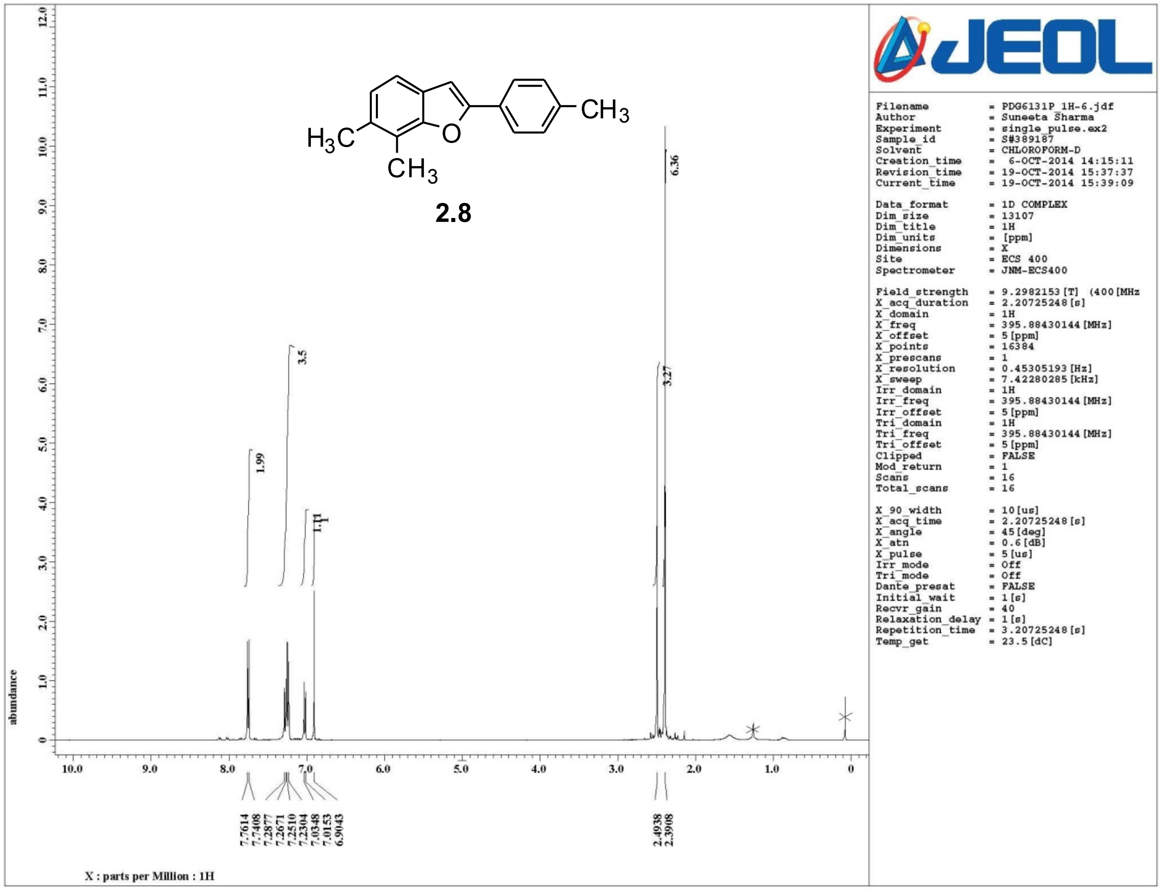


```
Filename          = PDG6128I_13C-4.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#481348
Solvent           = CHLOROFORM-D
Creation_time     = 21-NOV-2012 12:25:30
Revision_time     =  3-OCT-2014 17:03:56
Current_time      =  3-OCT-2014 17:05:16

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 22[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.7**

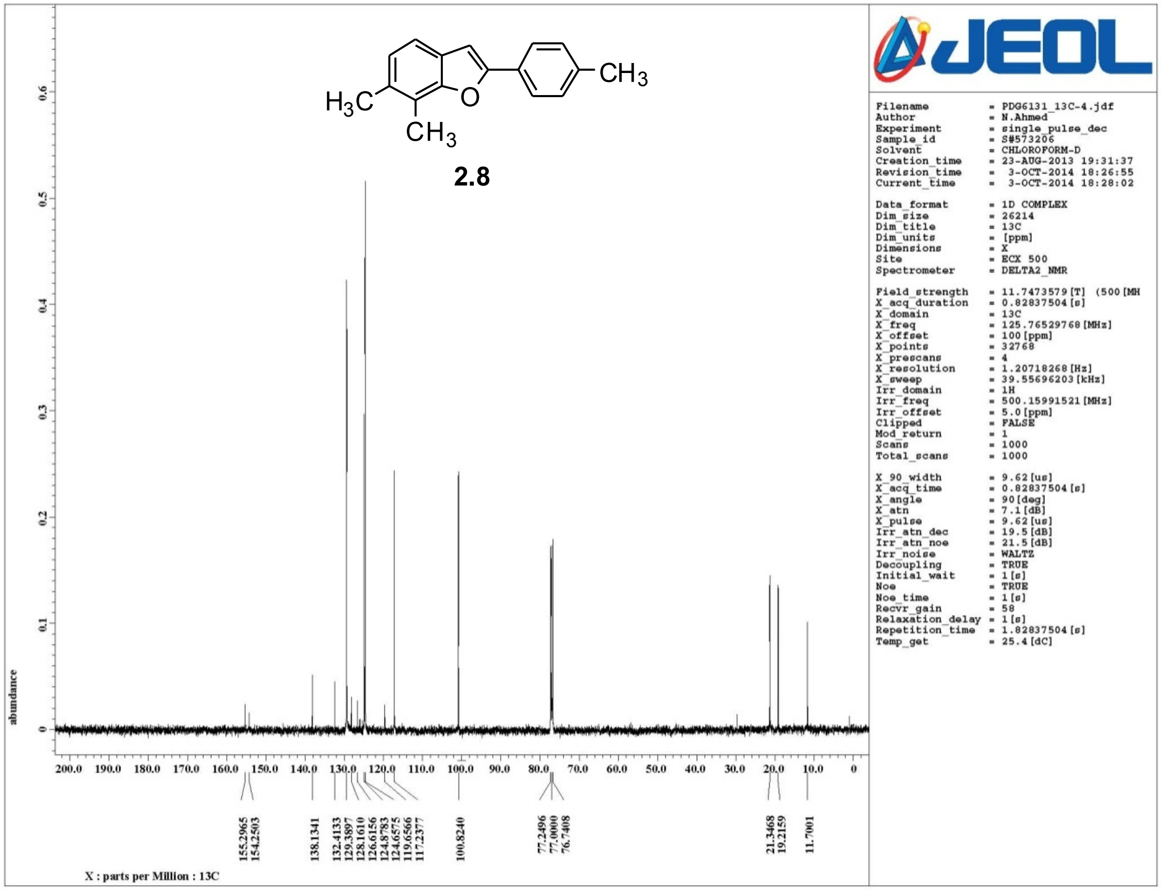


EI (HRMS) spectrum of **2.7**



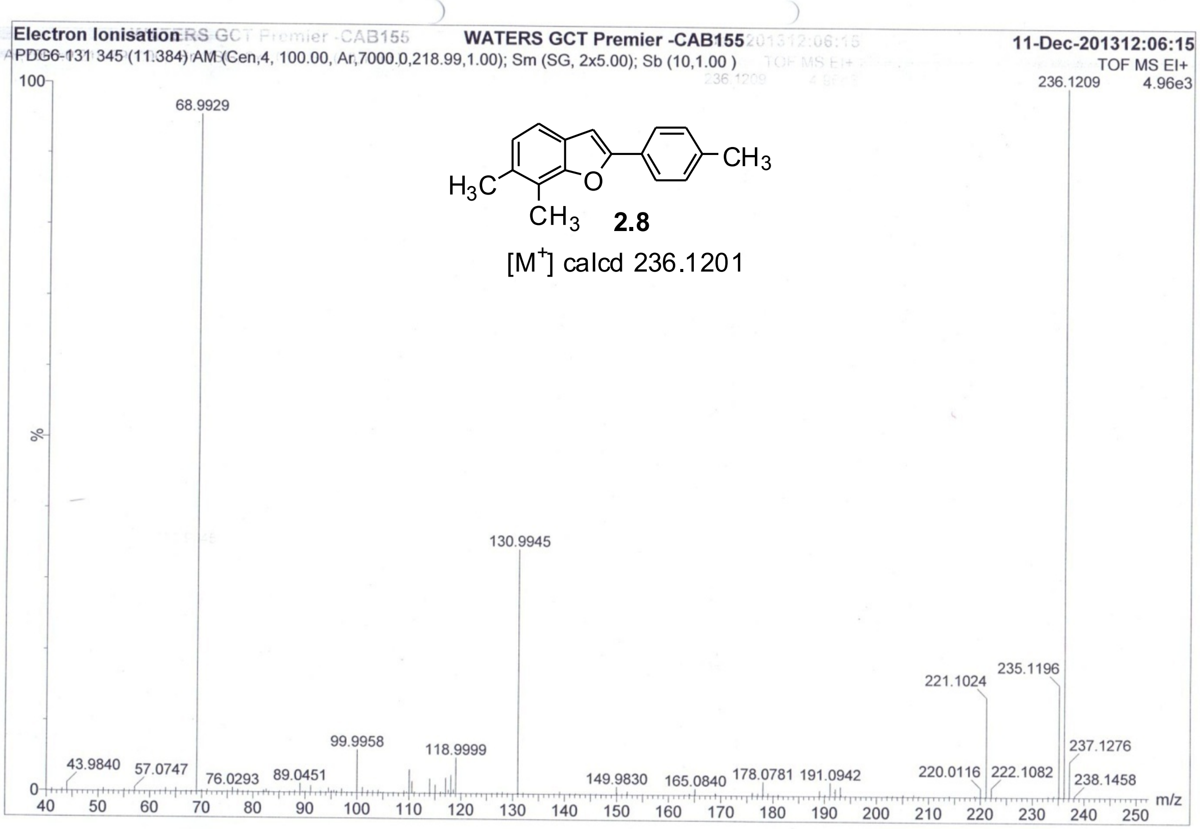
```
Filename          = PDG6131P_1H-6.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#389187
Solvent           = CHLOROFORM-D
Creation_time     =  6-OCT-2014 14:15:11
Revision_time     = 19-OCT-2014 15:37:37
Current_time      = 19-OCT-2014 15:39:09

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 1[s]
Repetition_time   = 3.20725248[s]
Temp_get          = 23.5[dC]
```

^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **2.8**

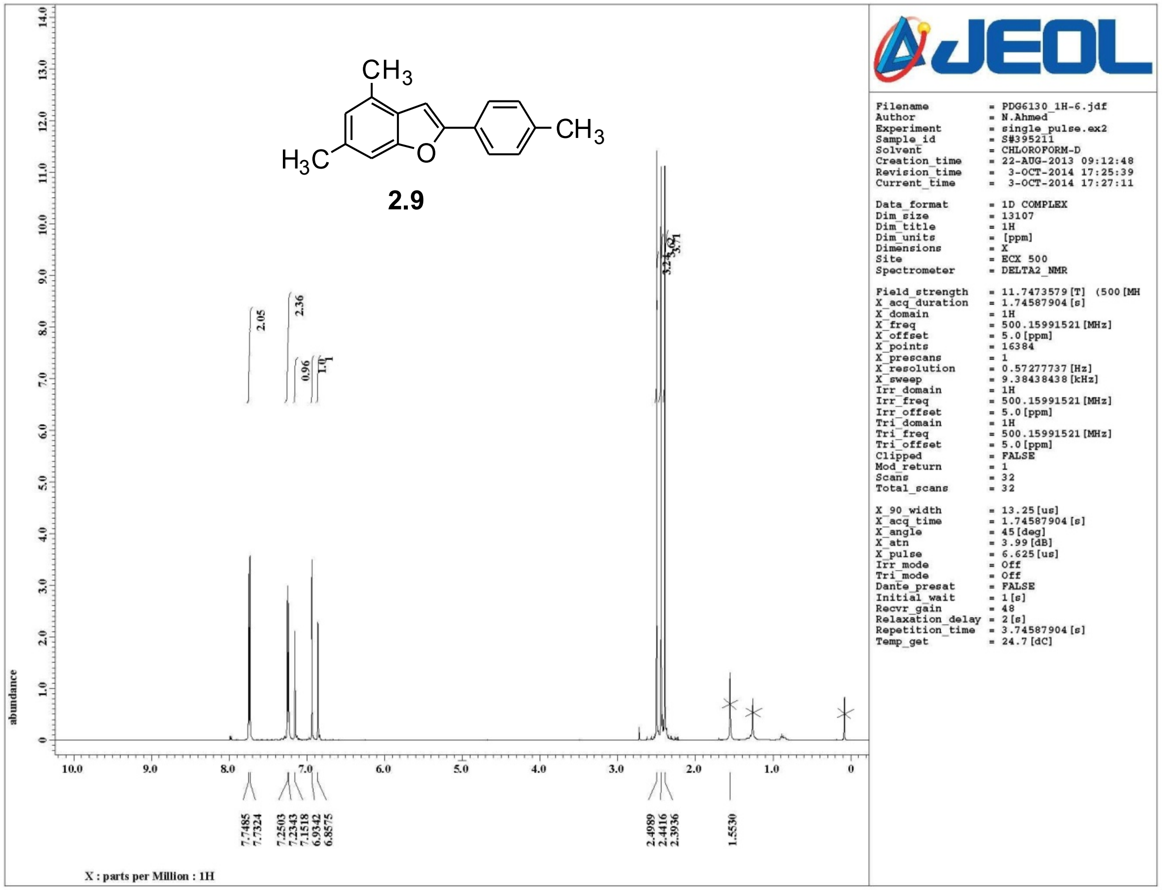


```
Filename          = PDG6131_13C-4.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#573206
Solvent           = CHLOROFORM-D
Creation_time     = 23-AUG-2013 19:31:37
Revision_time     =  3-OCT-2014 18:26:55
Current_time      =  3-OCT-2014 18:28:02

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 90[deg]
X_atn             = 7.1[dB]
X_pulse           = 9.62[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 25.4[dC]
```

^{13}C NMR (125 MHz, CDCl$_3$) spectrum of **2.8**

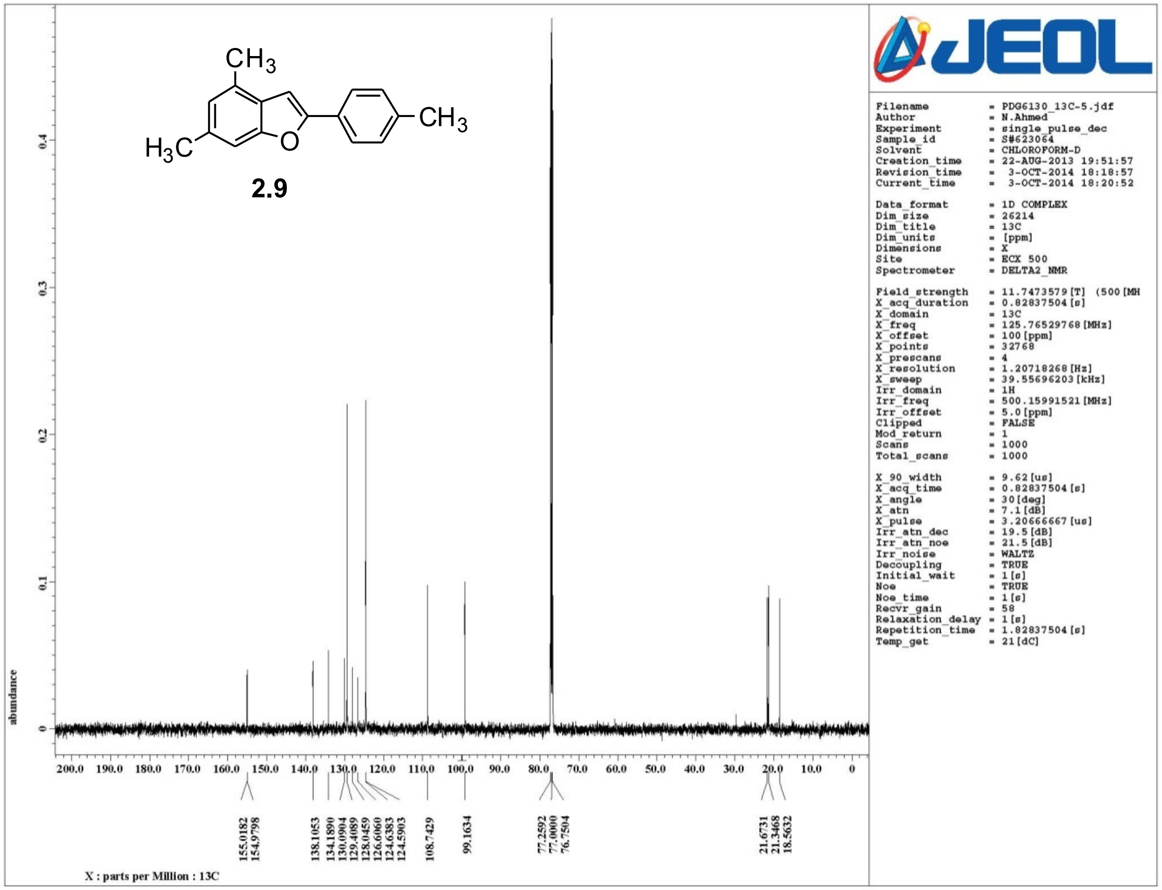


EI (HRMS) spectrum of **2.8**



```
Filename          = PDG6130_1H-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#395211
Solvent           = CHLOROFORM-D
Creation_time     = 22-AUG-2013 09:12:48
Revision_time     =  3-OCT-2014 17:25:39
Current_time      =  3-OCT-2014 17:27:11

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 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             = 32
Total_scans       = 32

X_90_width        = 13.25[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 2[s]
Repetition_time   = 3.74587904[s]
Temp_get          = 24.7[dC]
```

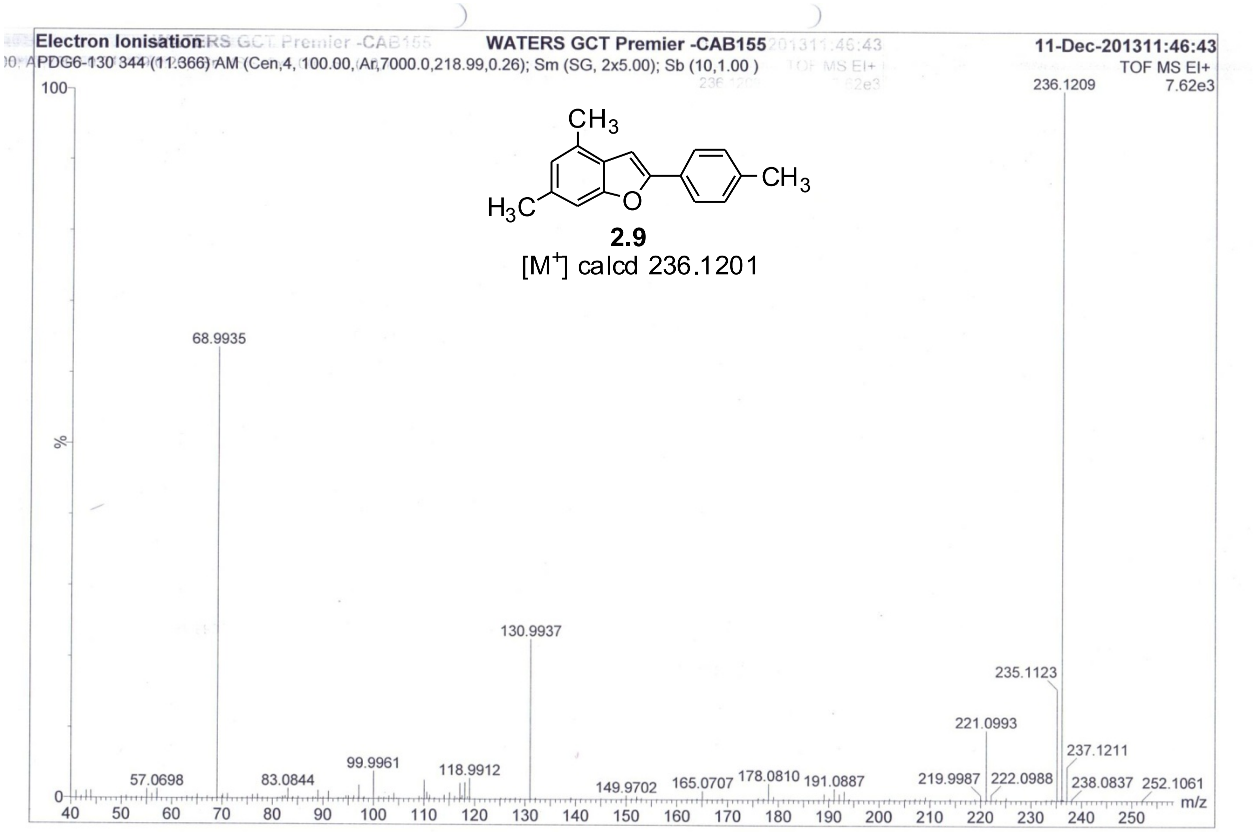
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.9**



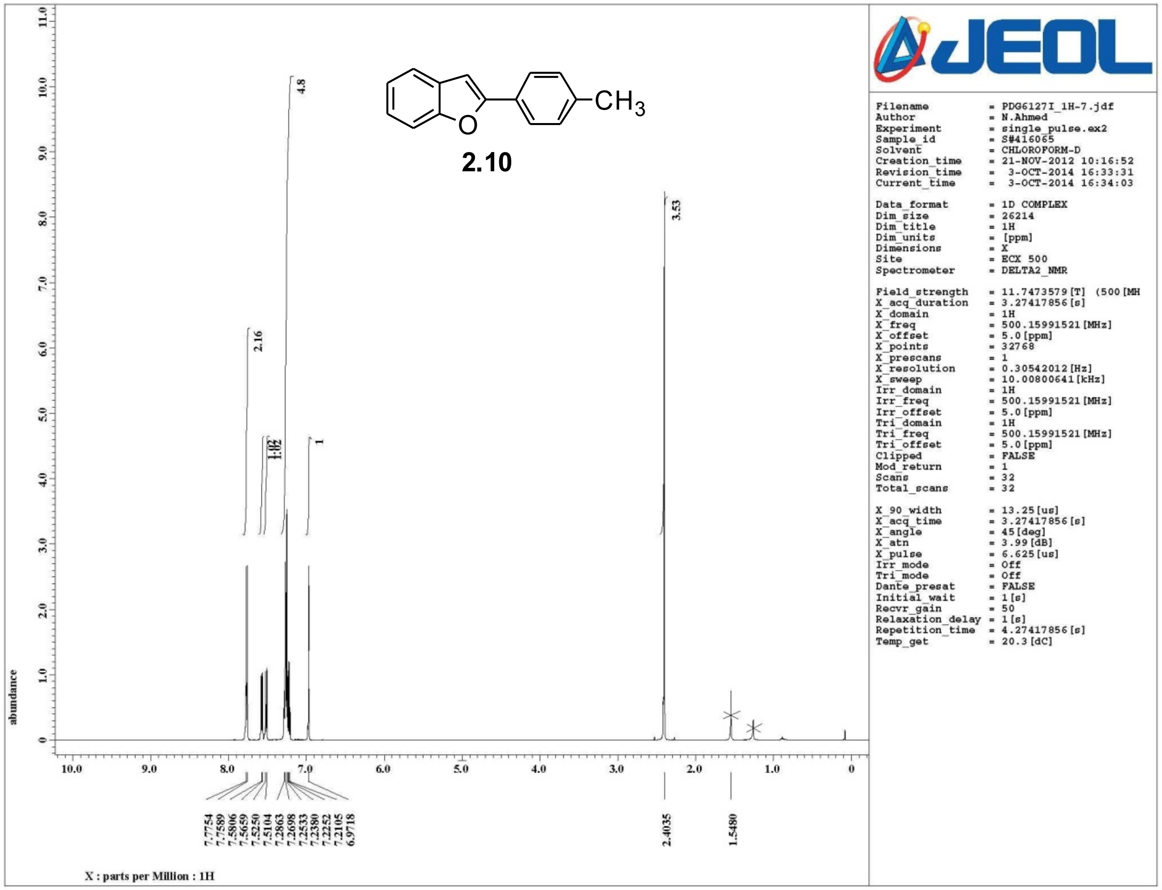
2.9

```
Filename            = PDG6130_13C-5.jdf
Author              = N.Ahmed
Experiment          = single_pulse_dec
Sample_id           = S#623064
Solvent             = CHLOROFORM-D
Creation_time       = 22-AUG-2013 19:51:57
Revision_time       =  3-OCT-2014 18:18:57
Current_time        =  3-OCT-2014 18:20:52

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

Field_strength      = 11.7473579[T] (500[MH
X_acq_duration      = 0.82837504[s]
X_domain            = 13C
X_freq              = 125.76529768[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 1.20718268[Hz]
X_sweep             = 39.55696203[kHz]
Irr_domain          = 1H
Irr_freq            = 500.15991521[MHz]
Irr_offset          = 5.0[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 1000
Total_scans         = 1000

X_90_width          = 9.62[us]
X_acq_time          = 0.82837504[s]
X_angle             = 30[deg]
X_atn               = 7.1[dB]
X_pulse             = 3.20666667[us]
Irr_atn_dec         = 19.5[dB]
Irr_atn_noe         = 21.5[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 1[s]
Recvr_gain          = 58
Relaxation_delay    = 1[s]
Repetition_time     = 1.82837504[s]
Temp_get            = 21[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.9**

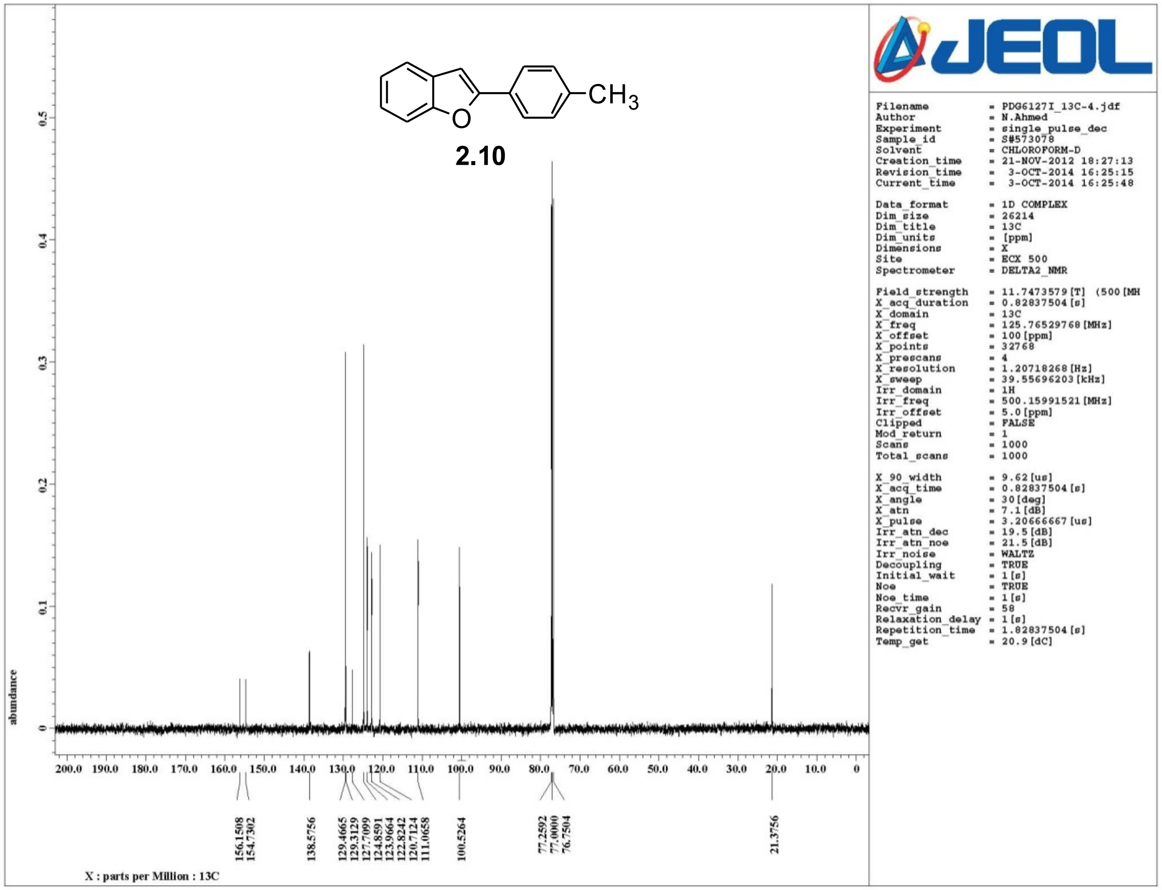


EI (HRMS) spectrum of **2.9**



```
Filename           = PDG6127I_1H-7.jdf
Author             = N.Ahmed
Experiment         = single_pulse.ex2
Sample_id          = S#416065
Solvent            = CHLOROFORM-D
Creation_time      = 21-NOV-2012 10:16:52
Revision_time      =  3-OCT-2014 16:33:31
Current_time       =  3-OCT-2014 16:34:03

Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECX 500
Spectrometer       = DELTA2_NMR

Field_strength     = 11.7473579[T] (500[MH
X_acq_duration     = 3.27417856[s]
X_domain           = 1H
X_freq             = 500.15991521[MHz]
X_offset           = 5.0[ppm]
X_points           = 32768
X_prescans         = 1
X_resolution       = 0.30542012[Hz]
X_sweep            = 10.00800641[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              = 32
Total_scans        = 32

X_90_width         = 13.25[us]
X_acq_time         = 3.27417856[s]
X_angle            = 45[deg]
X_atn              = 3.99[dB]
X_pulse            = 6.625[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 50
Relaxation_delay   = 1[s]
Repetition_time    = 4.27417856[s]
Temp_get           = 20.3[dC]
```

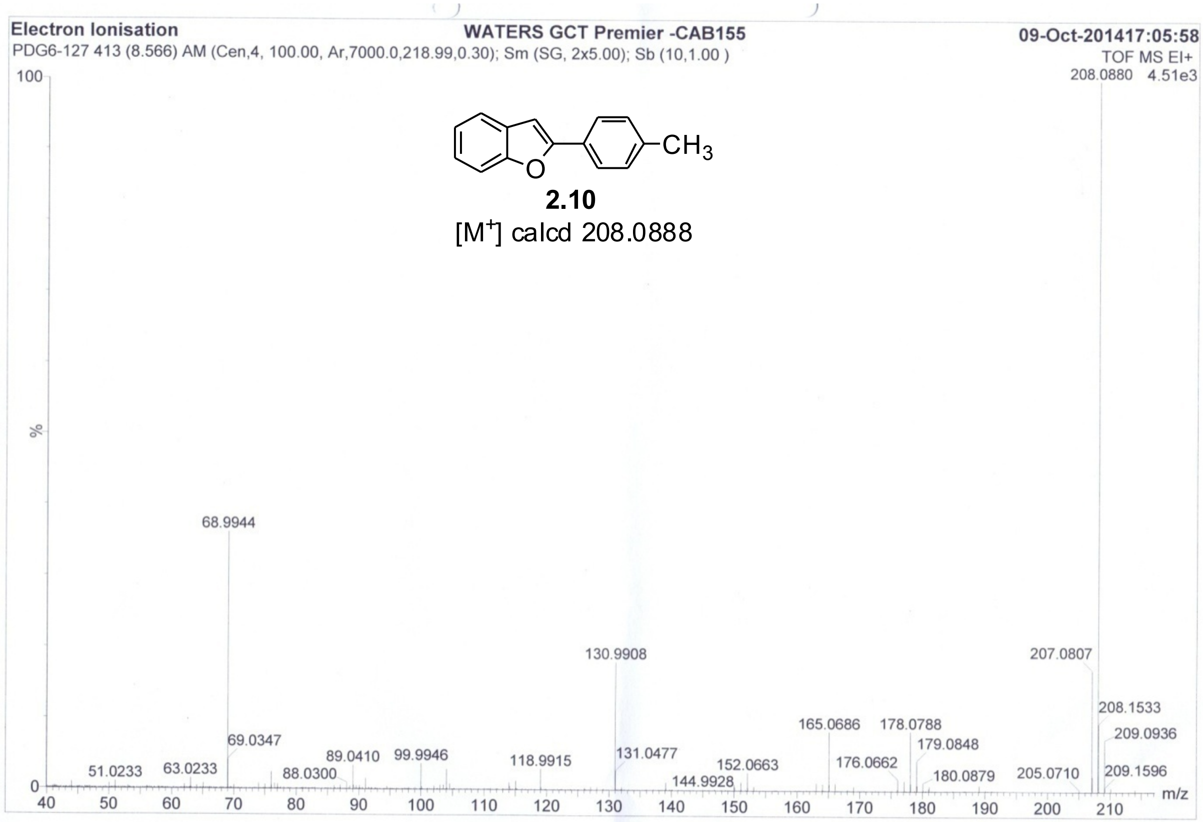
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.10**



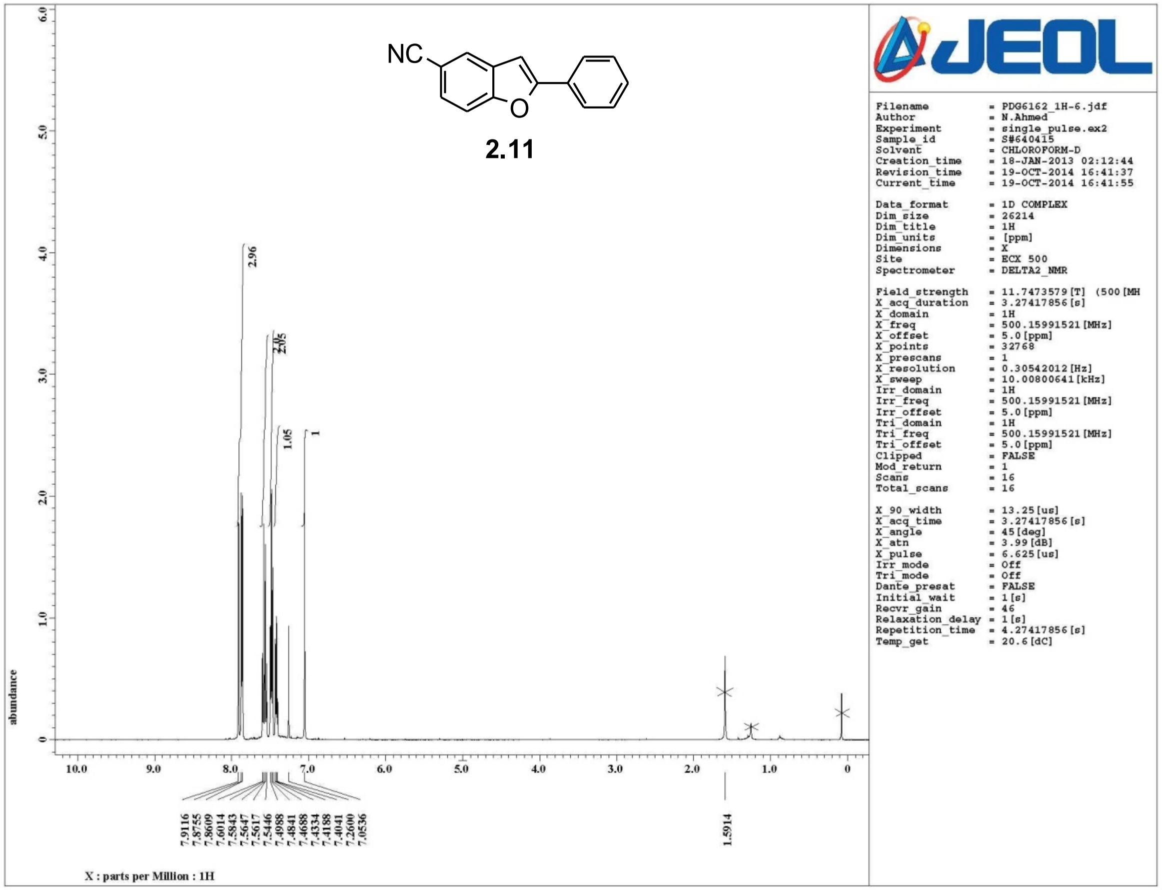
2.10

```
Filename          = PDG6127I_13C-4.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#573078
Solvent           = CHLOROFORM-D
Creation_time     = 21-NOV-2012 18:27:13
Revision_time     =  3-OCT-2014 16:25:15
Current_time      =  3-OCT-2014 16:25:48

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 20.9[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.10**

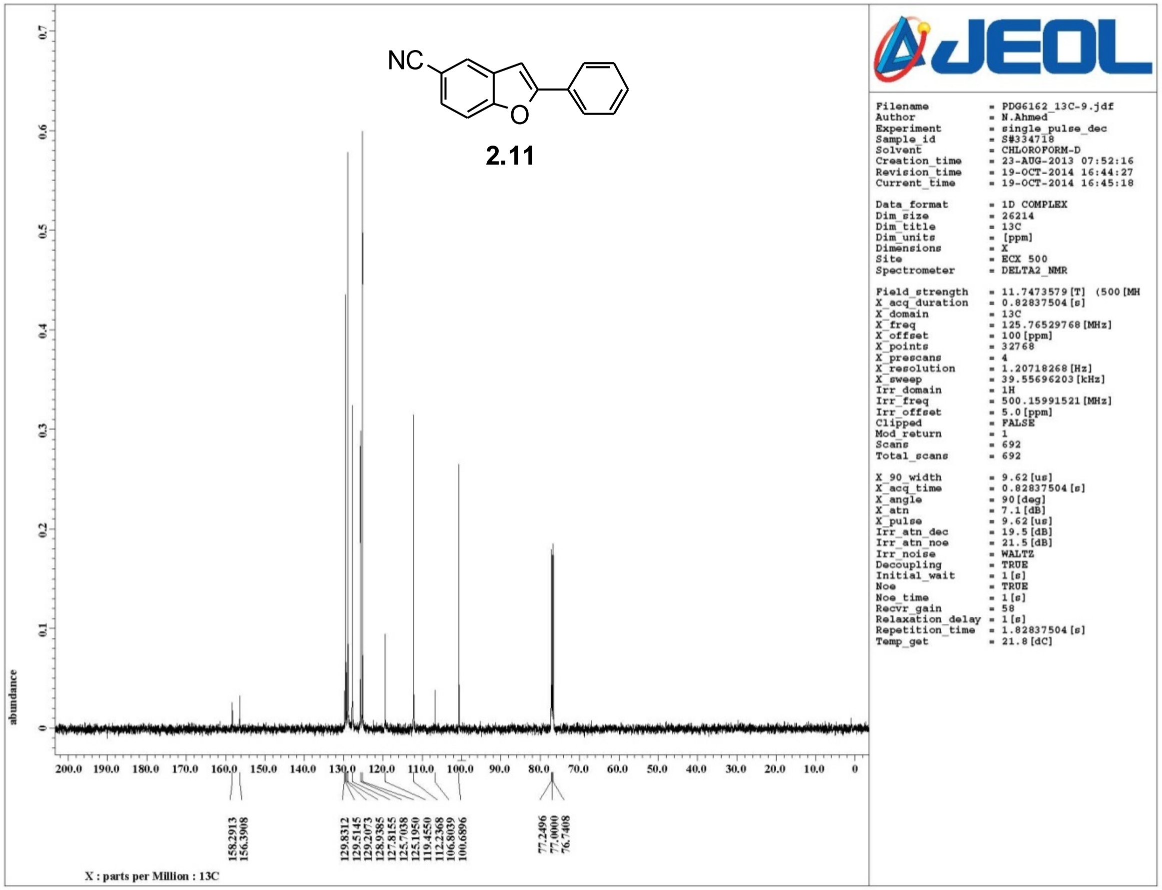


EI (HRMS) spectrum of **2.10**



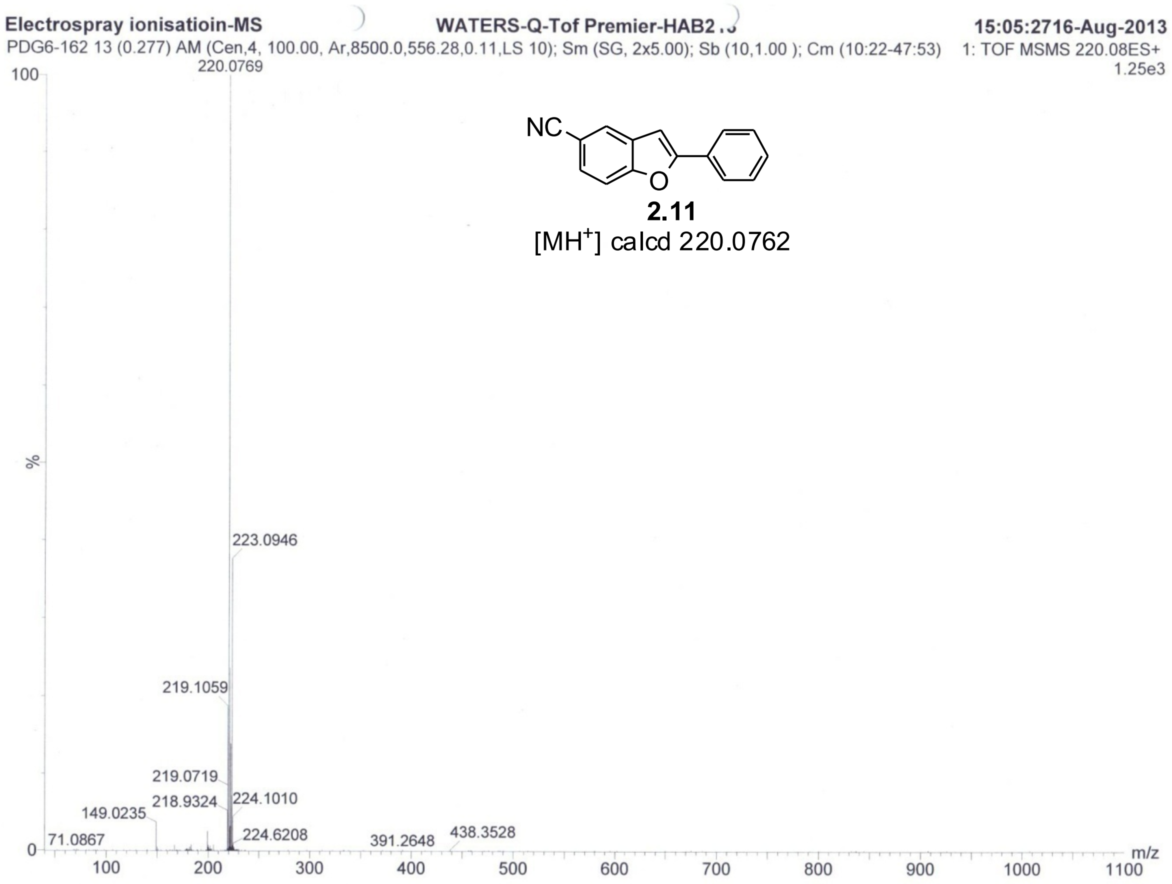
```
Filename          = PDG6162_1H-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#640415
Solvent           = CHLOROFORM-D
Creation_time     = 18-JAN-2013 02:12:44
Revision_time     = 19-OCT-2014 16:41:37
Current_time      = 19-OCT-2014 16:41:55

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 3.27417856[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.30542012[Hz]
X_sweep           = 10.00800641[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        = 13.25[us]
X_acq_time        = 3.27417856[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 1[s]
Repetition_time   = 4.27417856[s]
Temp_get          = 20.6[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.11**

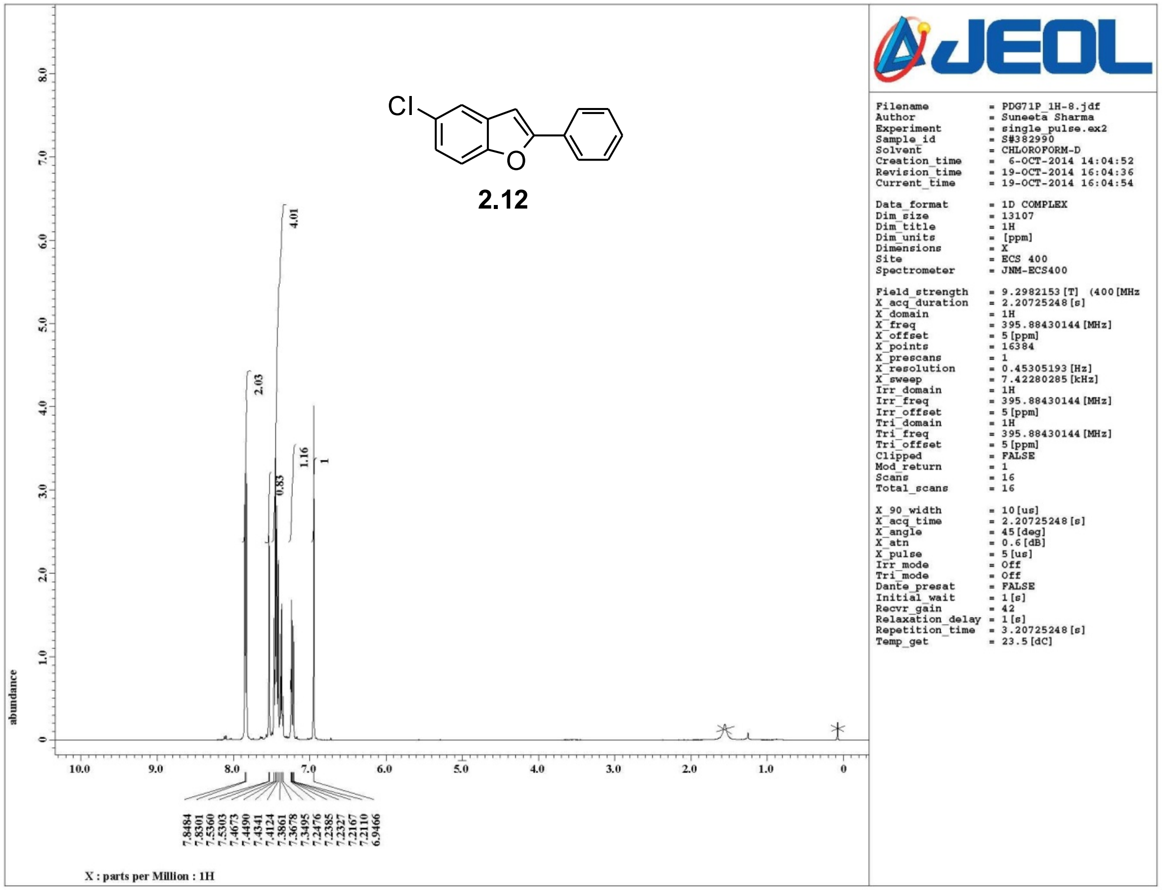


```
Filename          = PDG6162_13C-9.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#334718
Solvent           = CHLOROFORM-D
Creation_time     = 23-AUG-2013 07:52:16
Revision_time     = 19-OCT-2014 16:44:27
Current_time      = 19-OCT-2014 16:45:18

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 692
Total_scans       = 692

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 90[deg]
X_atn             = 7.1[dB]
X_pulse           = 9.62[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 21.8[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.11**

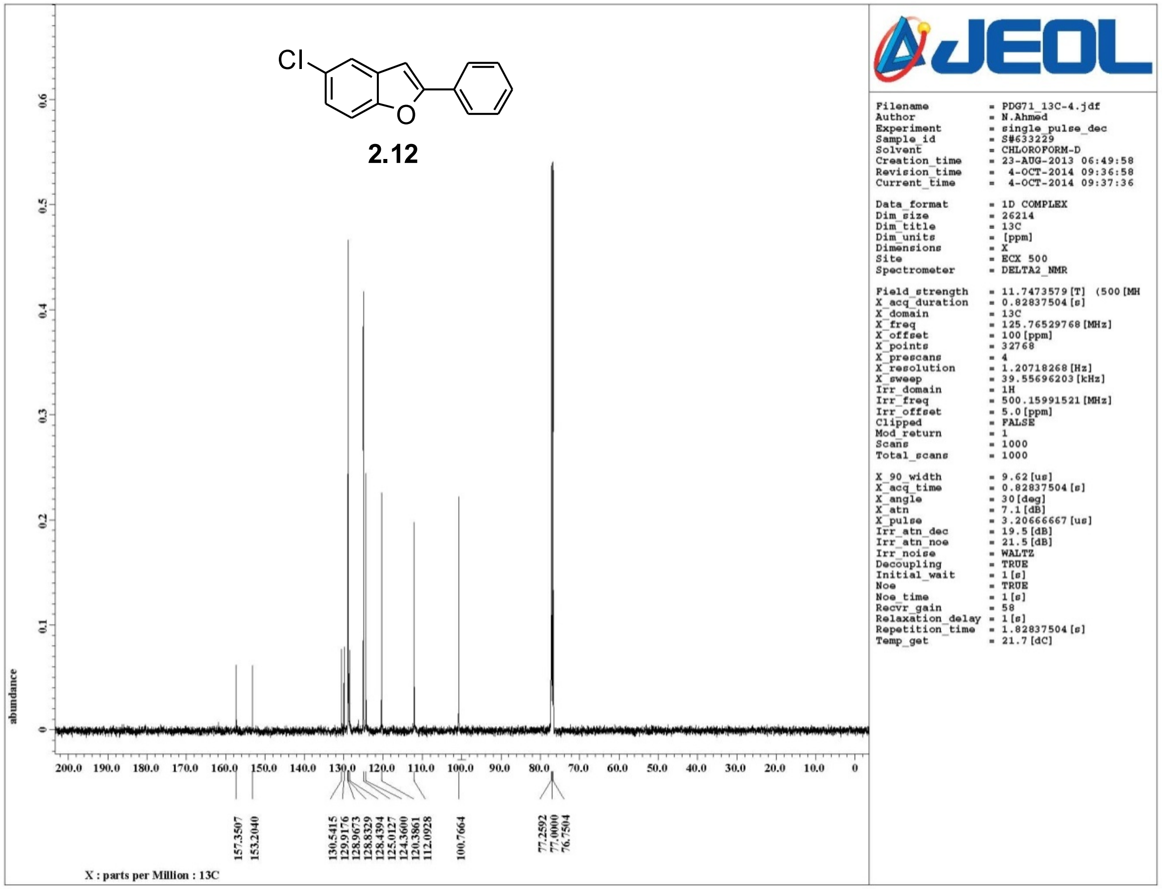


ESI (HRMS) spectrum of **2.11**



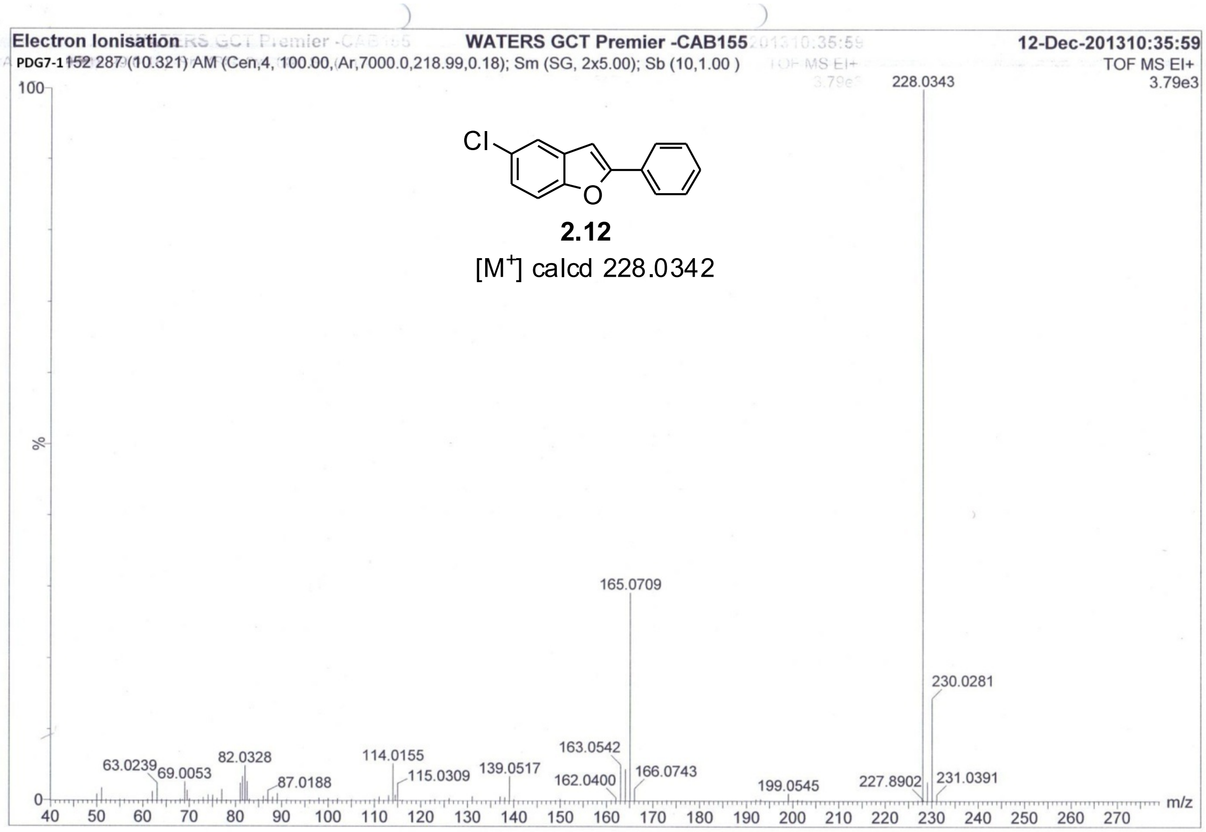
```
Filename          = PDG71P_1H-8.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#382990
Solvent           = CHLOROFORM-D
Creation_time     =  6-OCT-2014 14:04:52
Revision_time     = 19-OCT-2014 16:04:36
Current_time      = 19-OCT-2014 16:04:54

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 1[s]
Repetition_time   = 3.20725248[s]
Temp_get          = 23.5[dC]
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **2.12**



```
Filename          = PDG71_13C-4.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#633229
Solvent           = CHLOROFORM-D
Creation_time     = 23-AUG-2013 06:49:58
Revision_time     =  4-OCT-2014 09:36:58
Current_time      =  4-OCT-2014 09:37:36

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 21.7[dC]
```

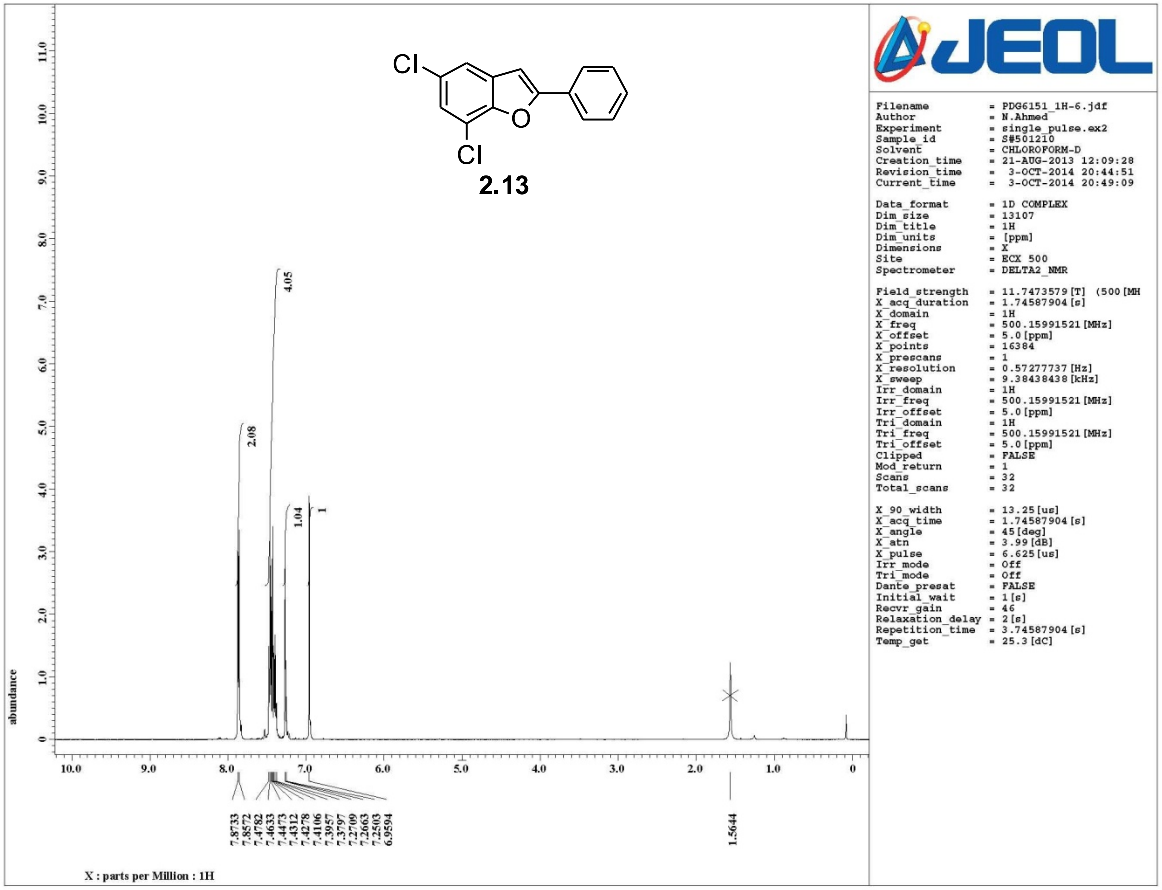
^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.12**



2.12

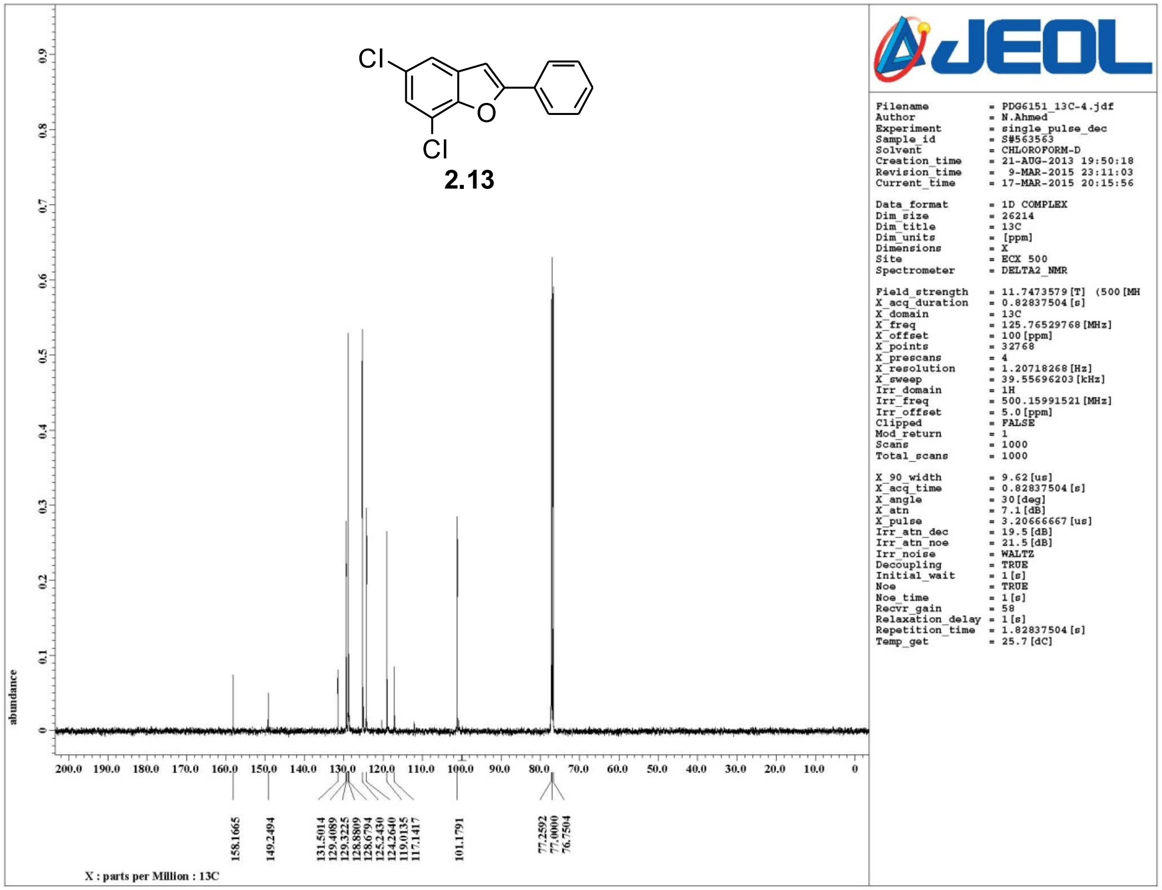
$[M^+]$ calcd 228.0342

EI (HRMS) spectrum of **2.12**



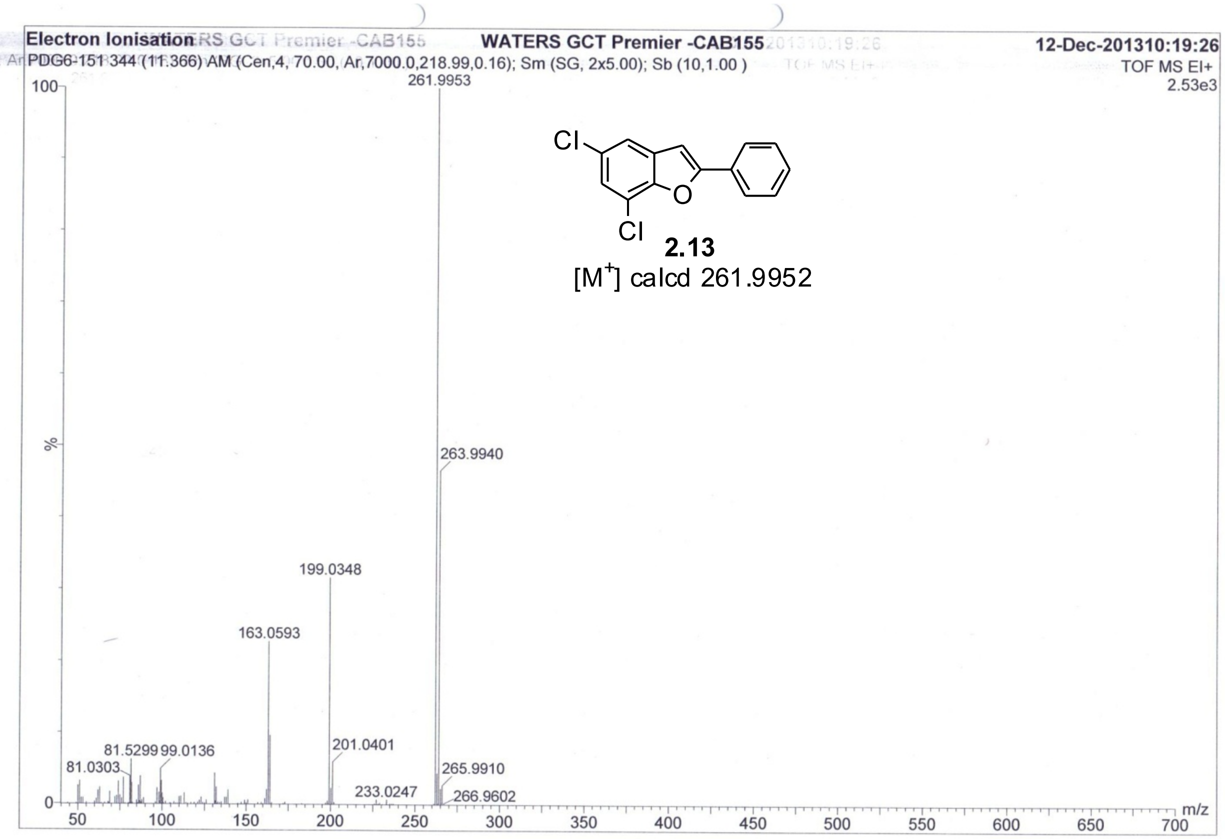
```
Filename          = PDG6151_1H-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#501210
Solvent           = CHLOROFORM-D
Creation_time     = 21-AUG-2013 12:09:28
Revision_time     =  3-OCT-2014 20:44:51
Current_time      =  3-OCT-2014 20:49:09

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 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             = 32
Total_scans       = 32

X_90_width        = 13.25[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 2[s]
Repetition_time   = 3.74587904[s]
Temp_get          = 25.3[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.13**

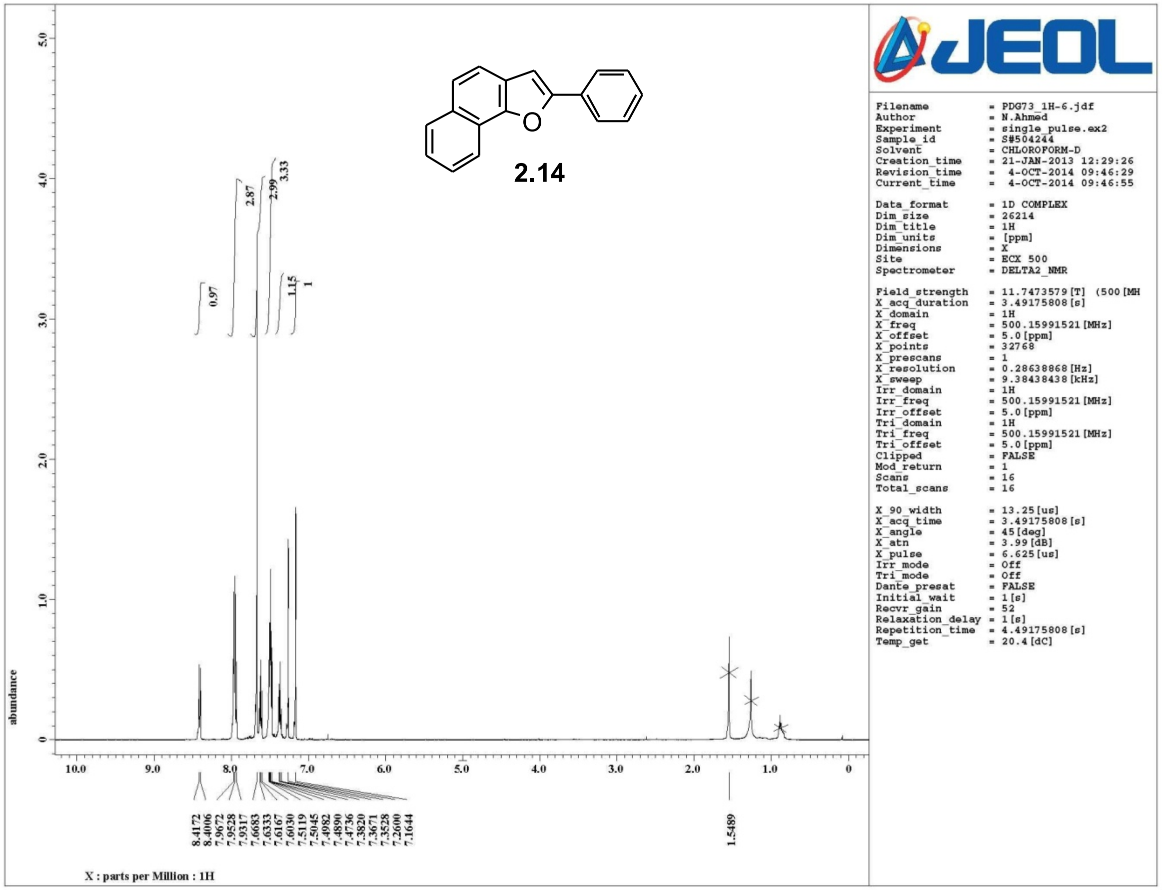


```
Filename          = PDG6151_13C-4.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#563563
Solvent           = CHLOROFORM-D
Creation_time     = 21-AUG-2013 19:50:18
Revision_time     =  9-MAR-2015 23:11:03
Current_time      = 17-MAR-2015 20:15:56

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 25.7[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.13**

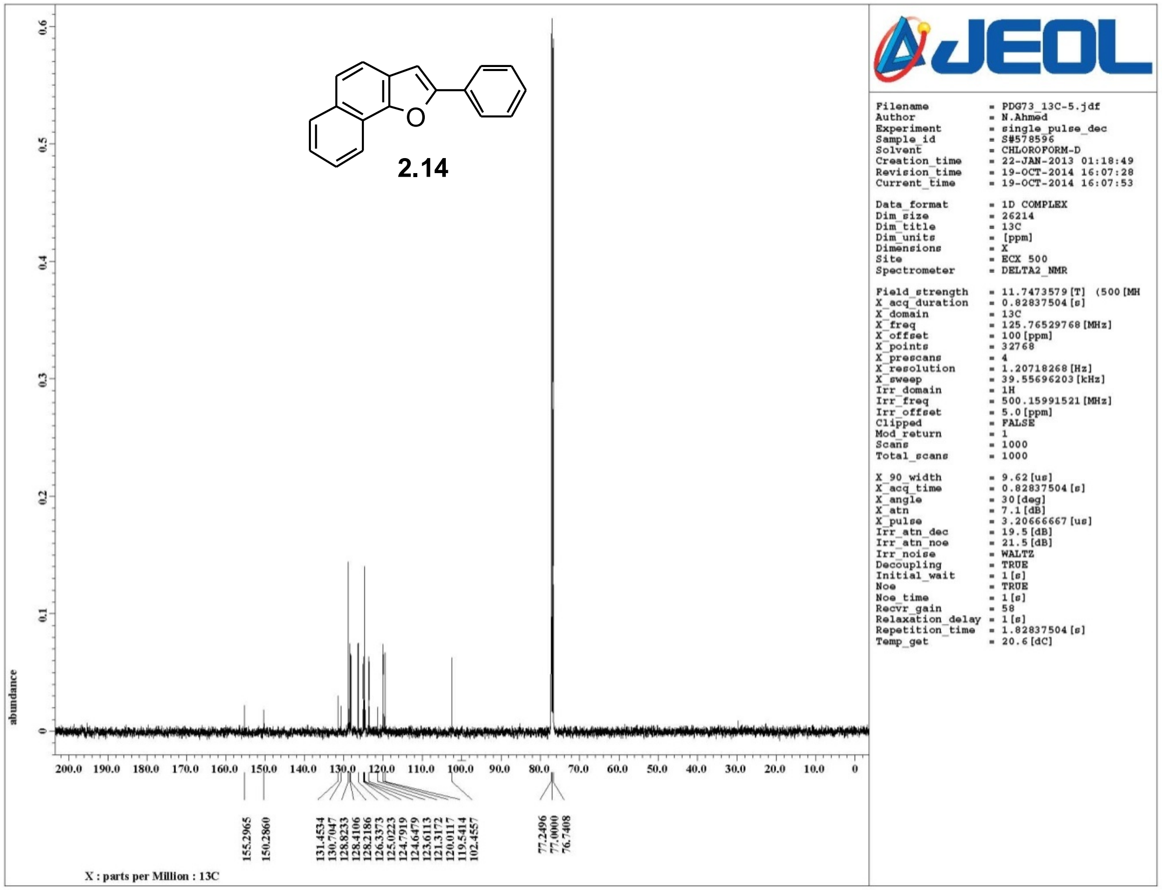


EI (HRMS) spectrum of **2.13**



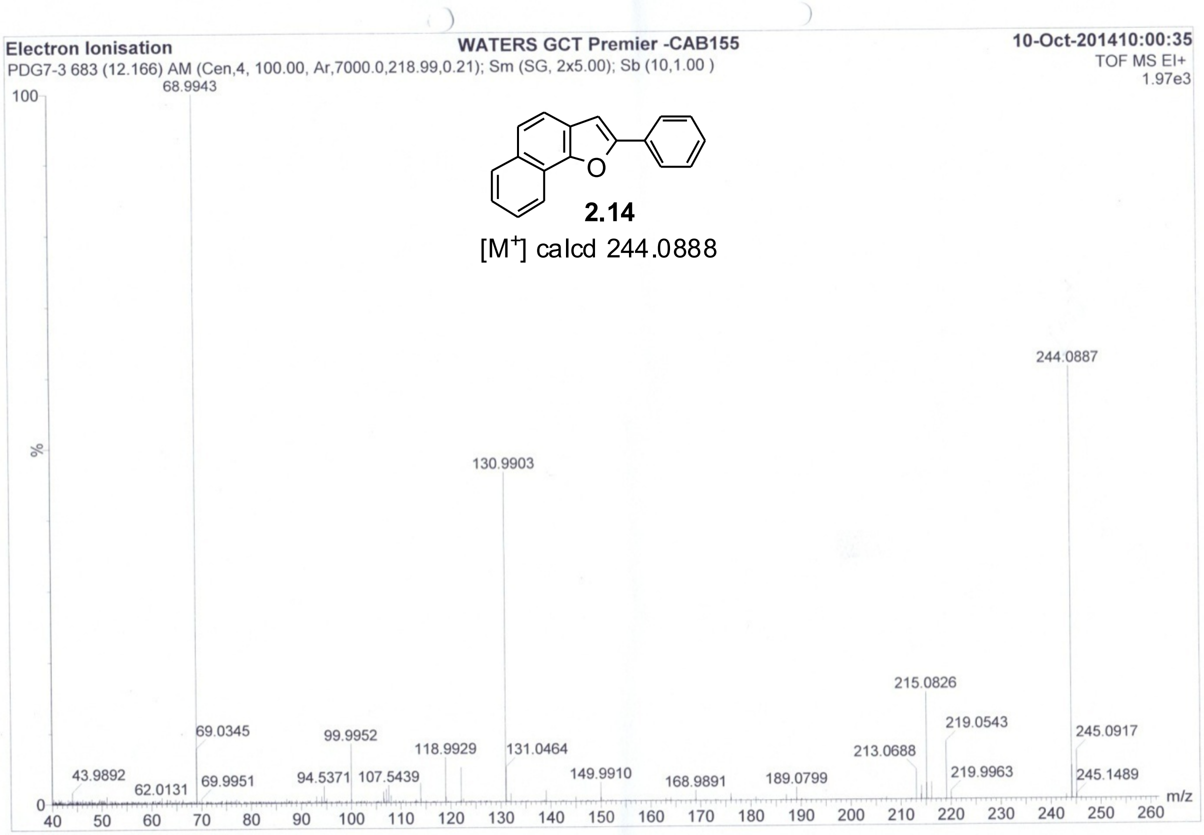
```
Filename           = PDG73_1H-6.jdf
Author             = N.Ahmed
Experiment         = single_pulse.ex2
Sample_id          = S#504244
Solvent            = CHLOROFORM-D
Creation_time      = 21-JAN-2013 12:29:26
Revision_time      =  4-OCT-2014 09:46:29
Current_time       =  4-OCT-2014 09:46:55

Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECX 500
Spectrometer       = DELTA2_NMR

Field_strength     = 11.7473579[T] (500[MH
X_acq_duration     = 3.49175808[s]
X_domain           = 1H
X_freq             = 500.15991521[MHz]
X_offset           = 5.0[ppm]
X_points           = 32768
X_prescans         = 1
X_resolution       = 0.28638868[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         = 13.25[us]
X_acq_time         = 3.49175808[s]
X_angle            = 45[deg]
X_atn              = 3.99[dB]
X_pulse            = 6.625[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 52
Relaxation_delay   = 1[s]
Repetition_time    = 4.49175808[s]
Temp_get           = 20.4[dC]
```

^{1}H NMR (500 MHz, CDCl$_3$) spectrum of **2.14**

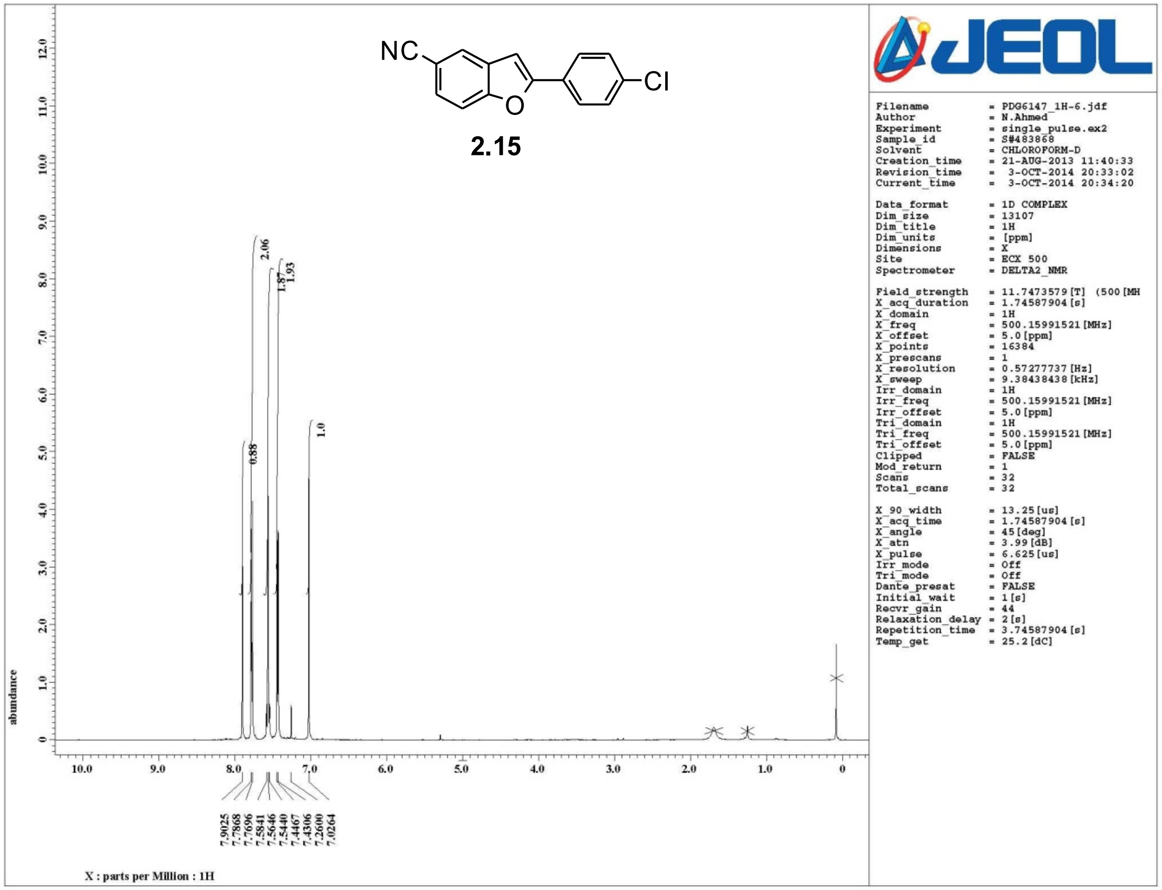


```
Filename           = PDG73_13C-5.jdf
Author             = N.Ahmed
Experiment         = single_pulse_dec
Sample_id          = S#578596
Solvent            = CHLOROFORM-D
Creation_time      = 22-JAN-2013 01:18:49
Revision_time      = 19-OCT-2014 16:07:28
Current_time       = 19-OCT-2014 16:07:53

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

Field_strength     = 11.7473579[T] (500[MH
X_acq_duration     = 0.82837504[s]
X_domain           = 13C
X_freq             = 125.76529768[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 1.20718268[Hz]
X_sweep            = 39.55696203[kHz]
Irr_domain         = 1H
Irr_freq           = 500.15991521[MHz]
Irr_offset         = 5.0[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 1000
Total_scans        = 1000

X_90_width         = 9.62[us]
X_acq_time         = 0.82837504[s]
X_angle            = 30[deg]
X_atn              = 7.1[dB]
X_pulse            = 3.20666667[us]
Irr_atn_dec        = 19.5[dB]
Irr_atn_noe        = 21.5[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 1[s]
Recvr_gain         = 58
Relaxation_delay   = 1[s]
Repetition_time    = 1.82837504[s]
Temp_get           = 20.6[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.14**



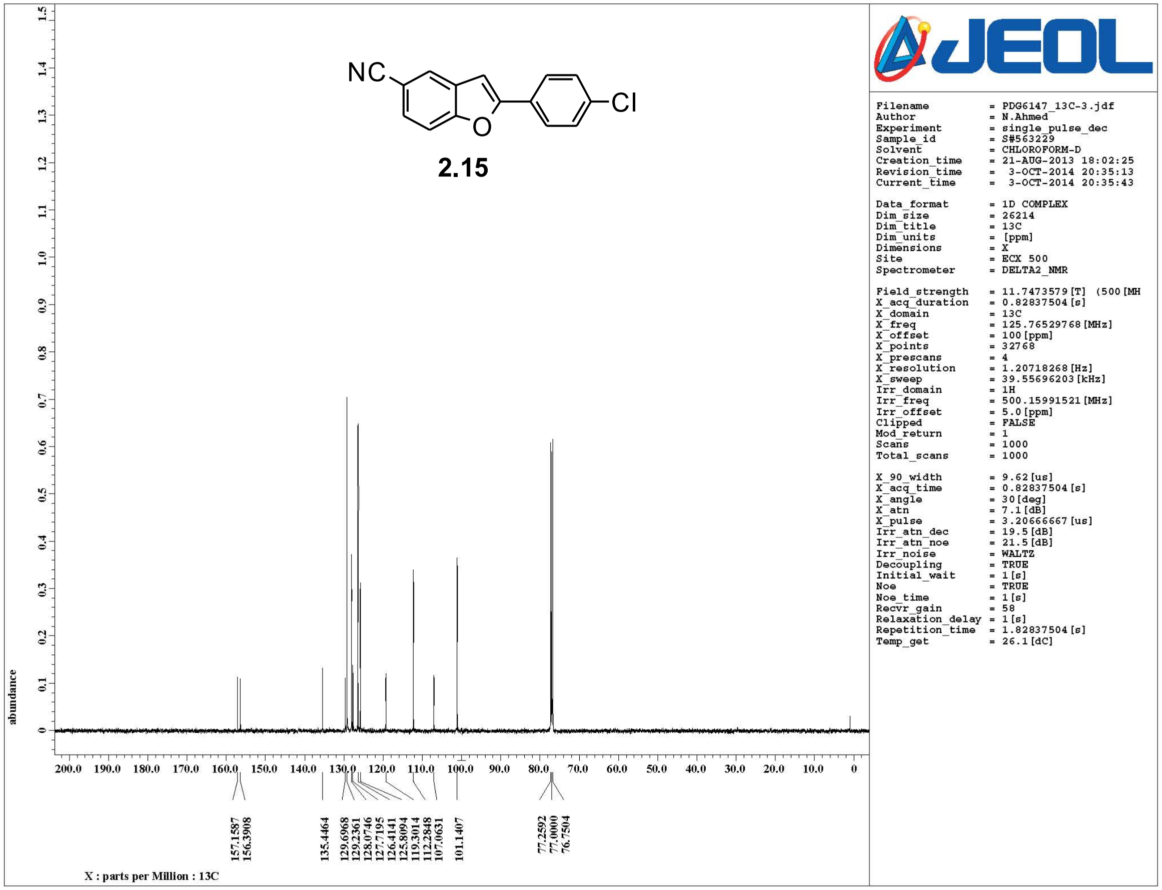
EI (HRMS) spectrum of **2.14**



2.15

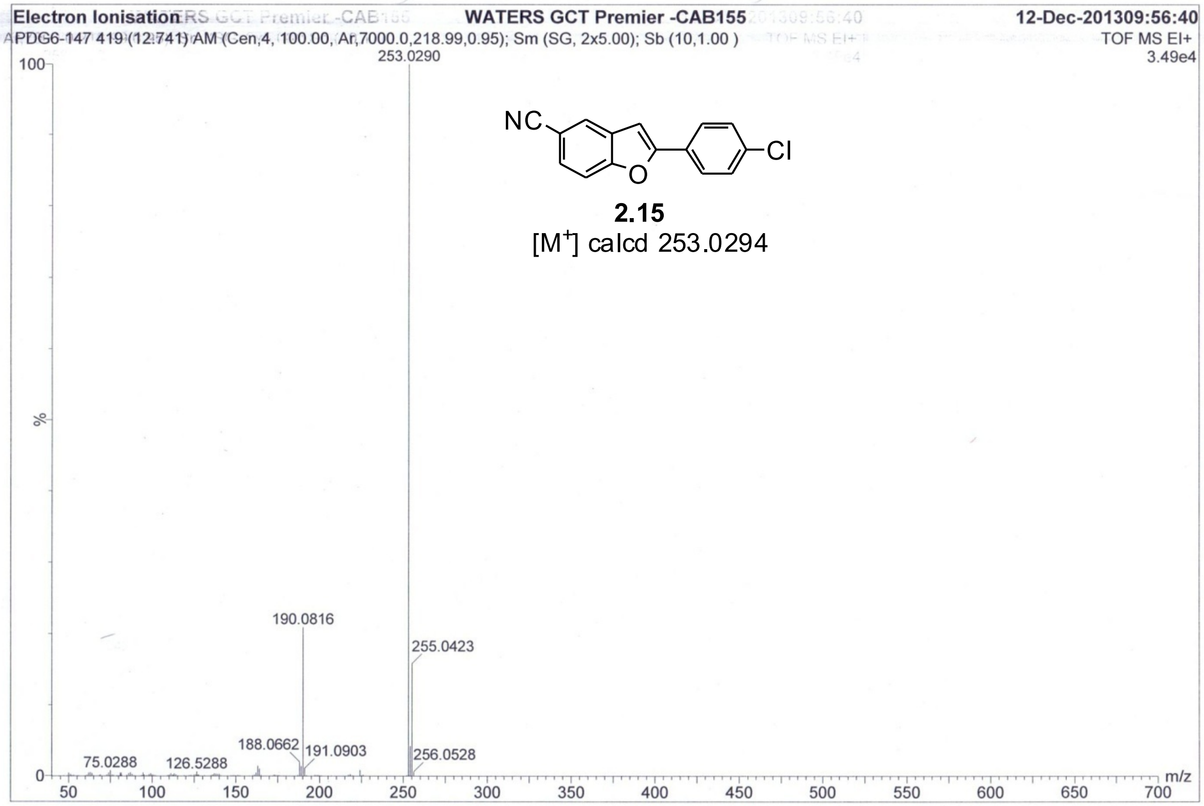
```
Filename          = PDG6147_1H-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#483868
Solvent           = CHLOROFORM-D
Creation_time     = 21-AUG-2013 11:40:33
Revision_time     =  3-OCT-2014 20:33:02
Current_time      =  3-OCT-2014 20:34:20

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 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             = 32
Total_scans       = 32

X_90_width        = 13.25[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 2[s]
Repetition_time   = 3.74587904[s]
Temp_get          = 25.2[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.15**

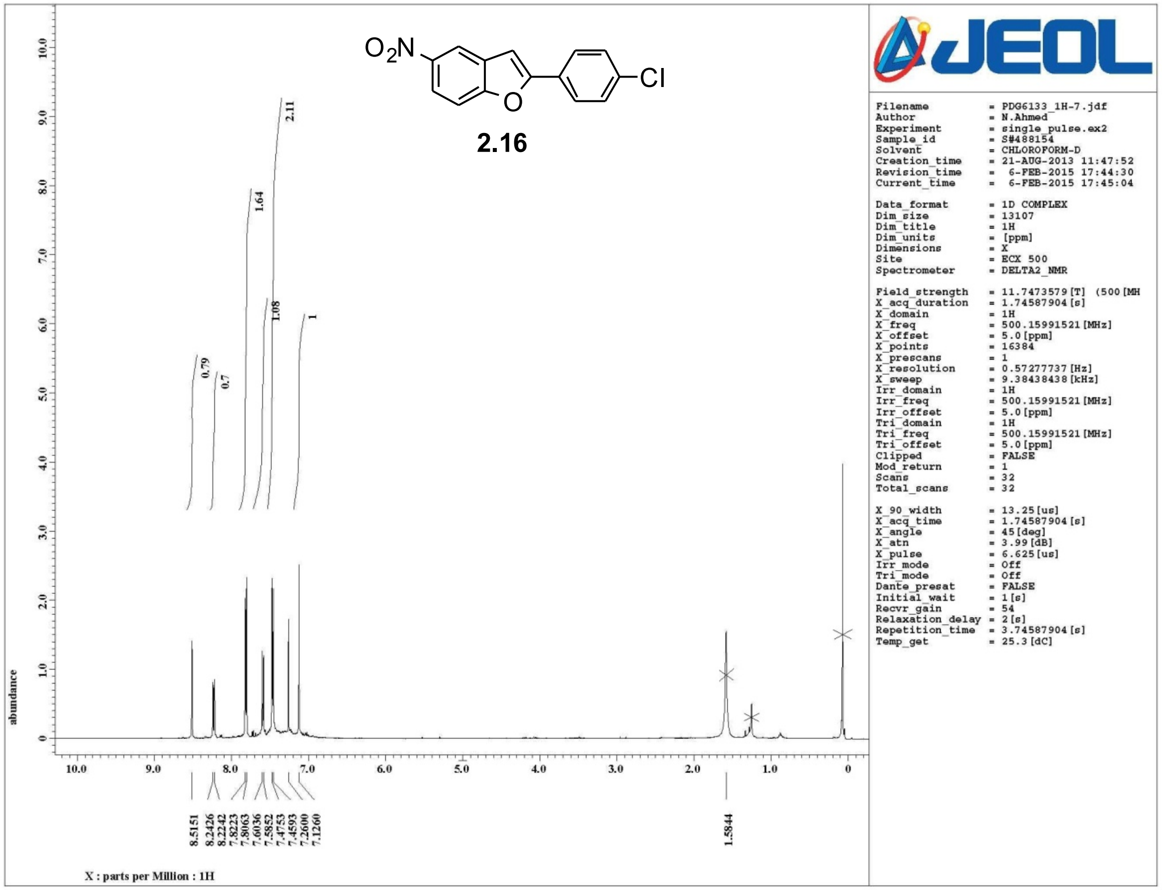


```
Filename          = PDG6147_13C-3.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#563229
Solvent           = CHLOROFORM-D
Creation_time     = 21-AUG-2013 18:02:25
Revision_time     =  3-OCT-2014 20:35:13
Current_time      =  3-OCT-2014 20:35:43

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 19.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 58
Relaxation_delay  = 1[s]
Repetition_time   = 1.82837504[s]
Temp_get          = 26.1[dC]
```

^{13}C NMR (125 MHz, CDCl$_3$) spectrum of **2.15**

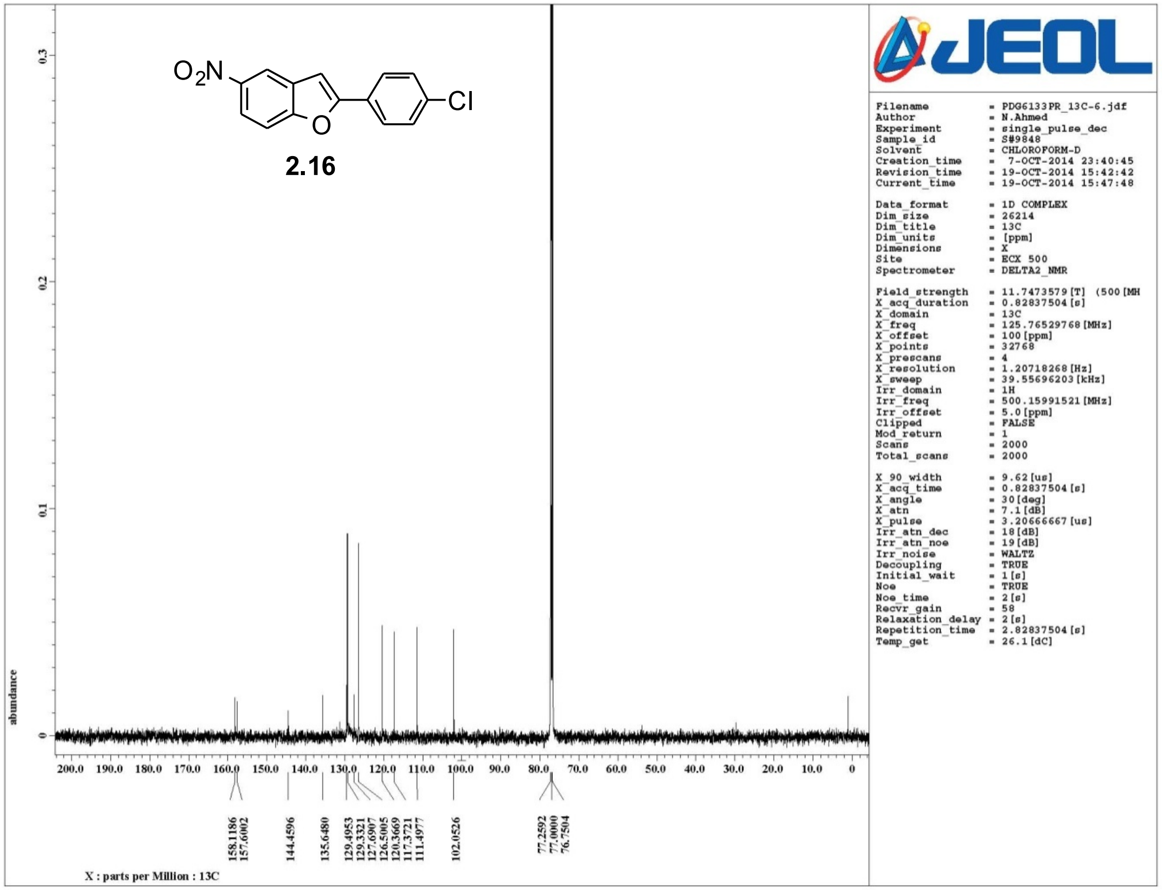


EI (HRMS) spectrum of **2.15**



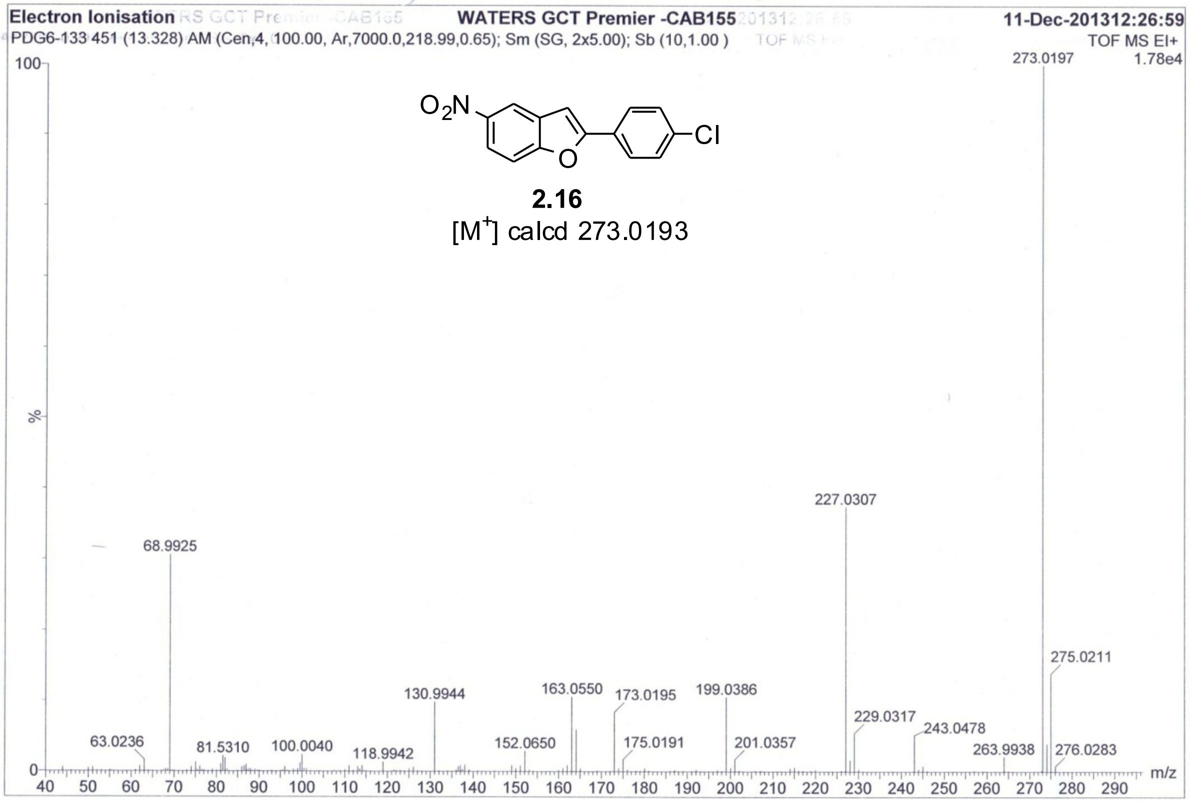
```
Filename          = PDG6133_1H-7.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#488154
Solvent           = CHLOROFORM-D
Creation_time     = 21-AUG-2013 11:47:52
Revision_time     =  6-FEB-2015 17:44:30
Current_time      =  6-FEB-2015 17:45:04

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 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             = 32
Total_scans       = 32

X_90_width        = 13.25[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 54
Relaxation_delay  = 2[s]
Repetition_time   = 3.74587904[s]
Temp_get          = 25.3[dC]
```

^{1}H NMR (500 MHz, CDCl$_3$) spectrum of **2.16**

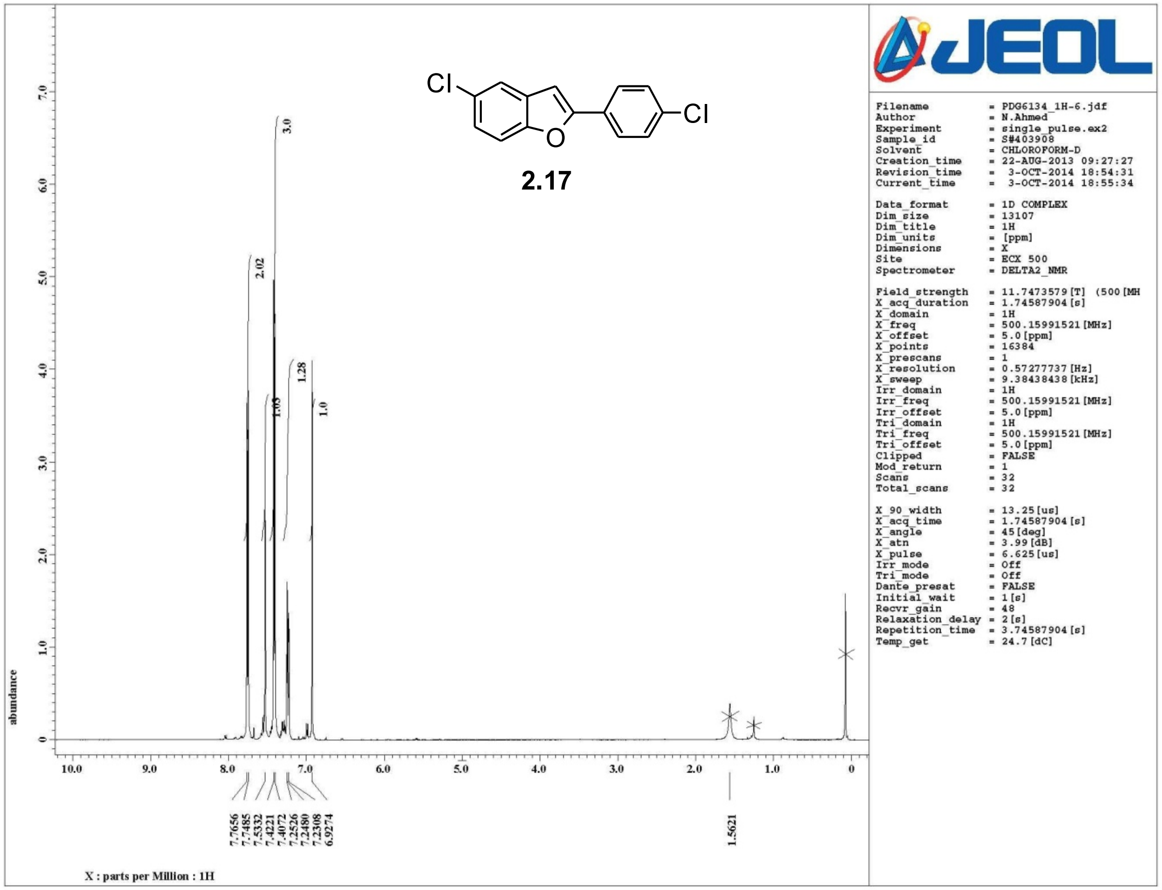


```
Filename          = PDG6133PR_13C-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#9848
Solvent           = CHLOROFORM-D
Creation_time     =  7-OCT-2014 23:40:45
Revision_time     = 19-OCT-2014 15:42:42
Current_time      = 19-OCT-2014 15:47:48

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 2000
Total_scans       = 2000

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 18[dB]
Irr_atn_noe       = 19[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   = 2.82837504[s]
Temp_get          = 26.1[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.16**



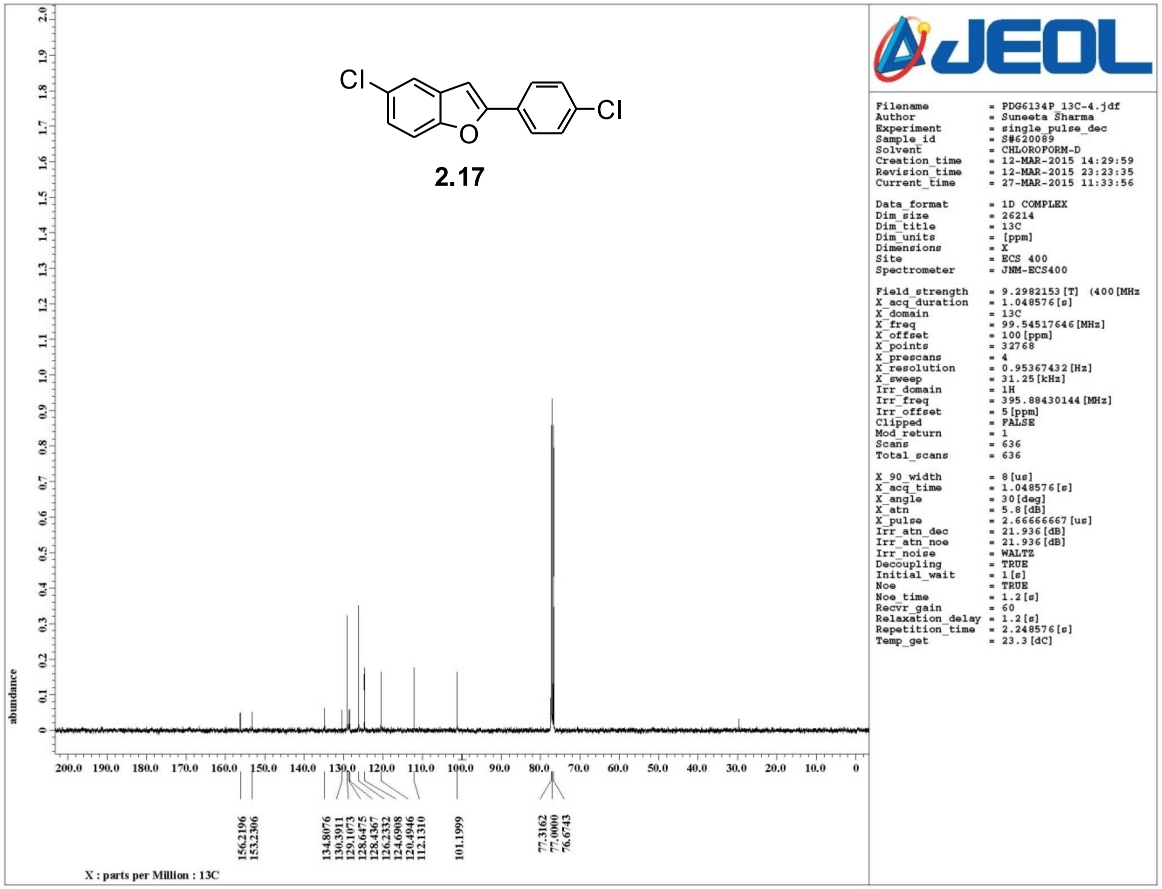
EI (HRMS) spectrum of **2.16**



2.17

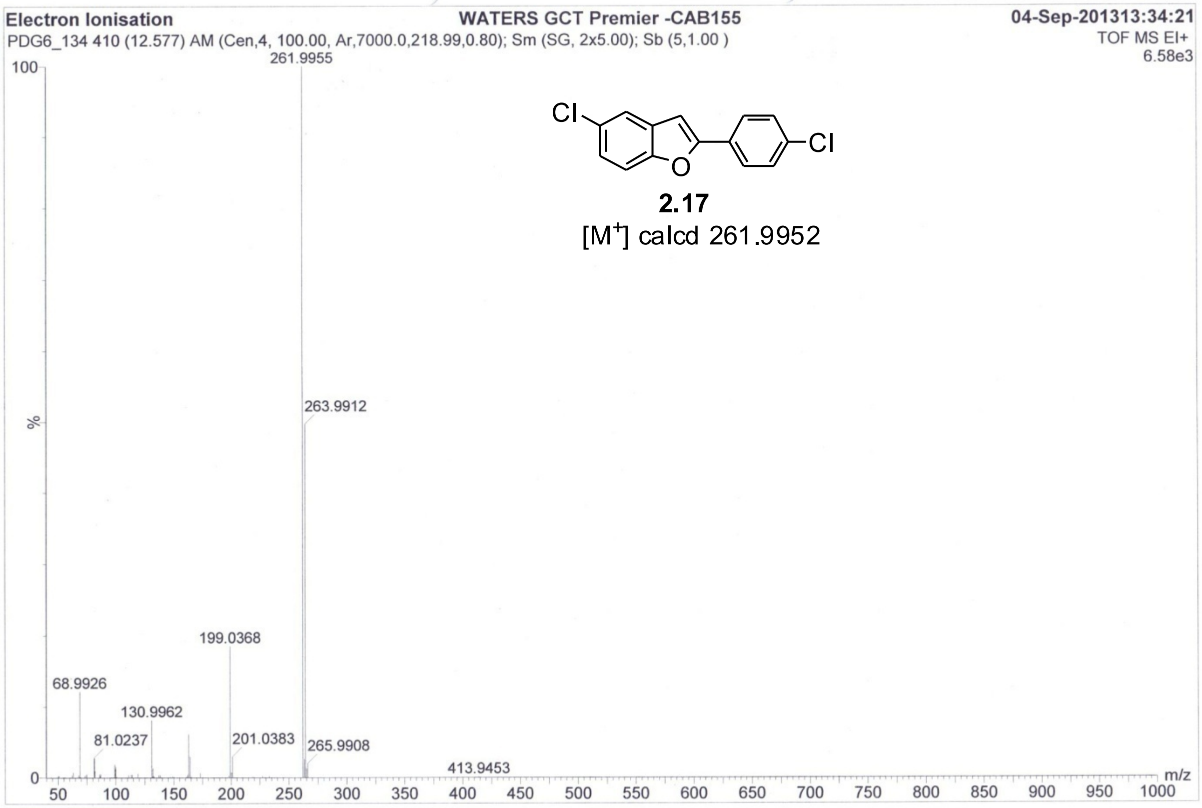
```
Filename          = PDG6134_1H-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#403908
Solvent           = CHLOROFORM-D
Creation_time     = 22-AUG-2013 09:27:27
Revision_time     =  3-OCT-2014 18:54:31
Current_time      =  3-OCT-2014 18:55:34

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 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             = 32
Total_scans       = 32

X_90_width        = 13.25[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 2[s]
Repetition_time   = 3.74587904[s]
Temp_get          = 24.7[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.17**

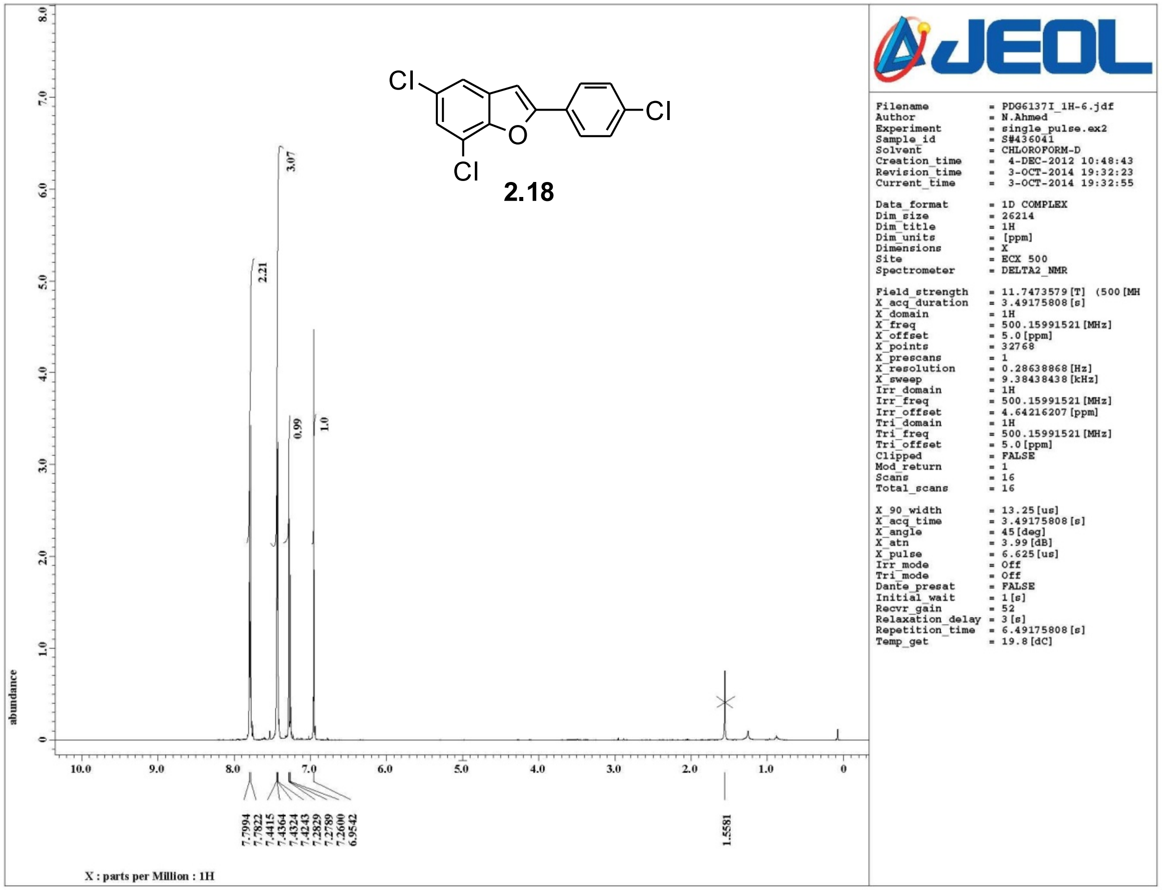


```
Filename          = PDG6134P_13C-4.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#620089
Solvent           = CHLOROFORM-D
Creation_time     = 12-MAR-2015 14:29:59
Revision_time     = 12-MAR-2015 23:23:35
Current_time      = 27-MAR-2015 11:33:56

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 636
Total_scans       = 636

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1.2[s]
Recvr_gain        = 60
Relaxation_delay  = 1.2[s]
Repetition_time   = 2.248576[s]
Temp_get          = 23.3[dC]
```

^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **2.17**

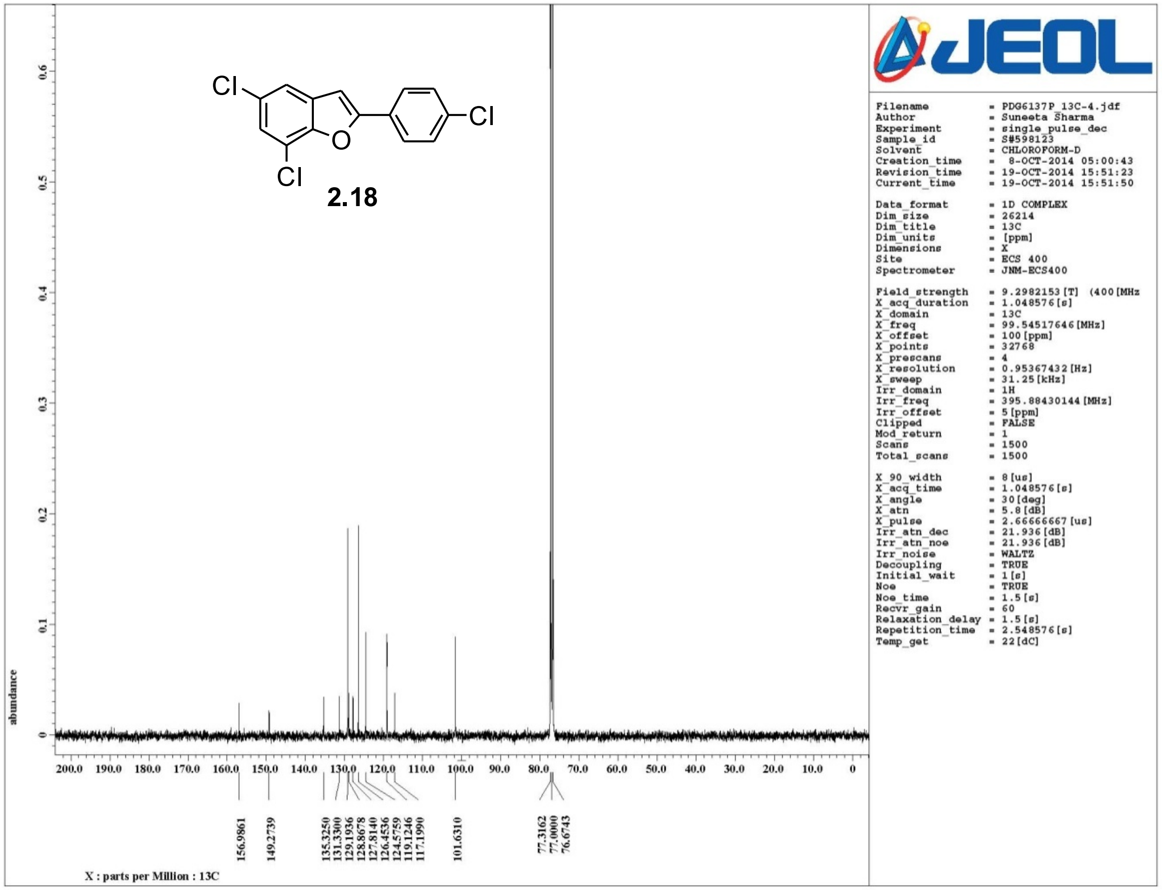


EI (HRMS) spectrum of **2.17**



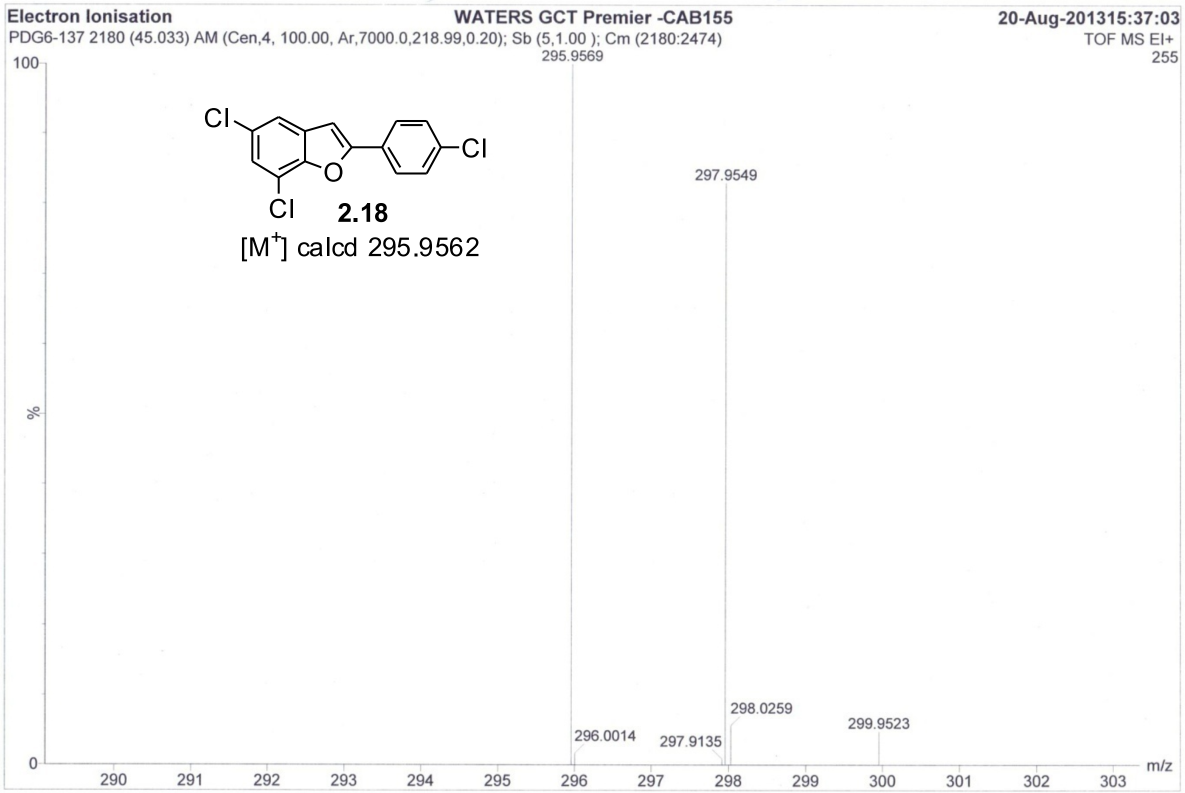
```
Filename          = PDG6137I_1H-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#436041
Solvent           = CHLOROFORM-D
Creation_time     =  4-DEC-2012 10:48:43
Revision_time     =  3-OCT-2014 19:32:23
Current_time      =  3-OCT-2014 19:32:55

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 3.49175808[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.28638868[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 4.64216207[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        = 13.25[us]
X_acq_time        = 3.49175808[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 52
Relaxation_delay  = 3[s]
Repetition_time   = 6.49175808[s]
Temp_get          = 19.8[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.18**

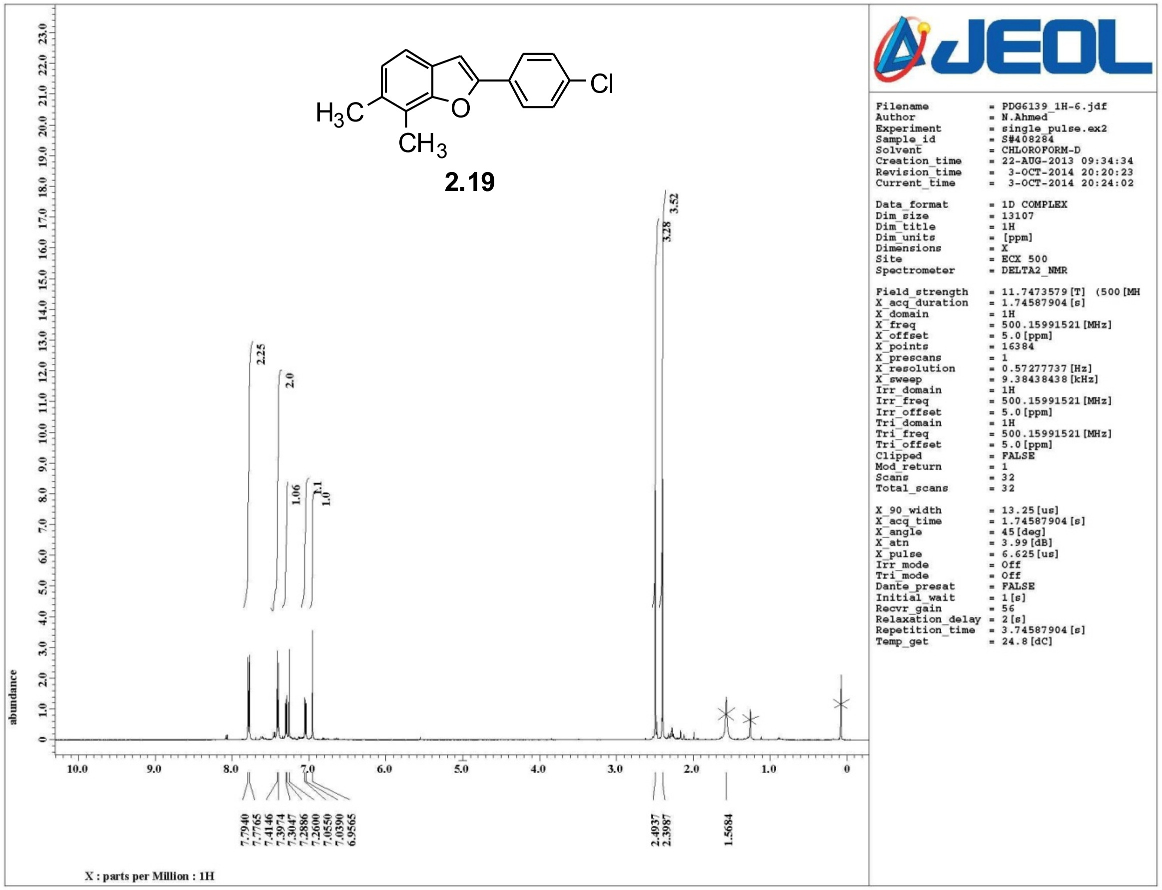


```
Filename          = PDG6137P_13C-4.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#598123
Solvent           = CHLOROFORM-D
Creation_time     =  8-OCT-2014 05:00:43
Revision_time     = 19-OCT-2014 15:51:23
Current_time      = 19-OCT-2014 15:51:50

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1500
Total_scans       = 1500

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1.5[s]
Recvr_gain        = 60
Relaxation_delay  = 1.5[s]
Repetition_time   = 2.548576[s]
Temp_get          = 22[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **2.18**



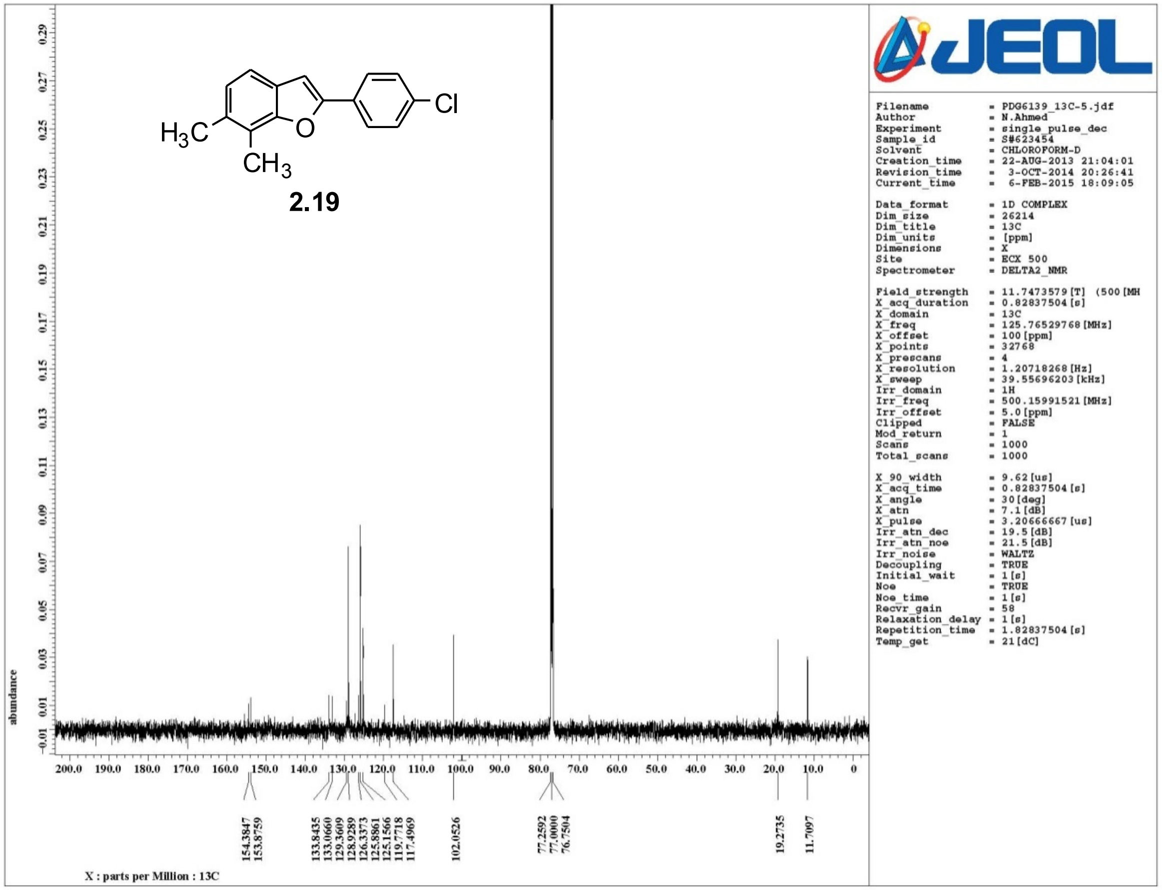
EI (HRMS) spectrum of **2.18**



2.19

```
Filename           = PDG6139_1H-6.jdf
Author             = N.Ahmed
Experiment         = single_pulse.ex2
Sample_id          = S#408284
Solvent            = CHLOROFORM-D
Creation_time      = 22-AUG-2013 09:34:34
Revision_time      =  3-OCT-2014 20:20:23
Current_time       =  3-OCT-2014 20:24:02

Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECX 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              = 32
Total_scans        = 32

X_90_width         = 13.25[us]
X_acq_time         = 1.74587904[s]
X_angle            = 45[deg]
X_atn              = 3.99[dB]
X_pulse            = 6.625[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 56
Relaxation_delay   = 2[s]
Repetition_time    = 3.74587904[s]
Temp_get           = 24.8[dC]
```

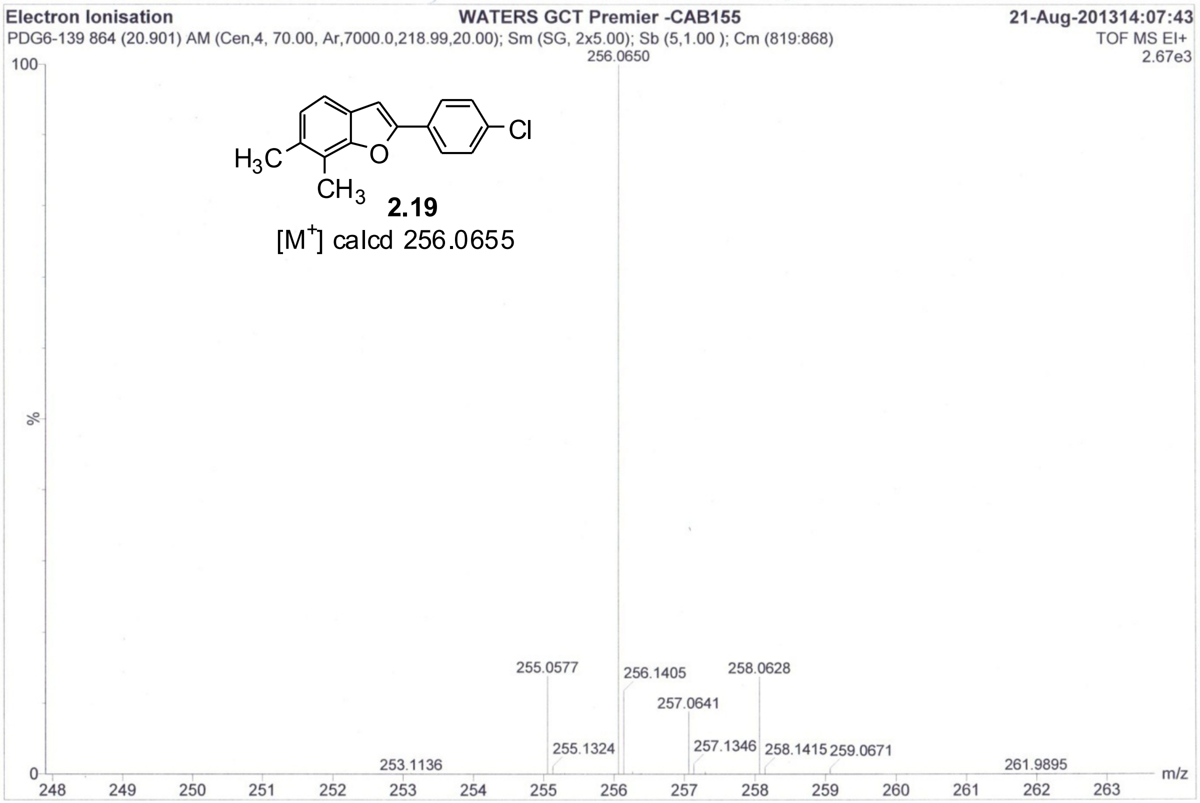
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.19**



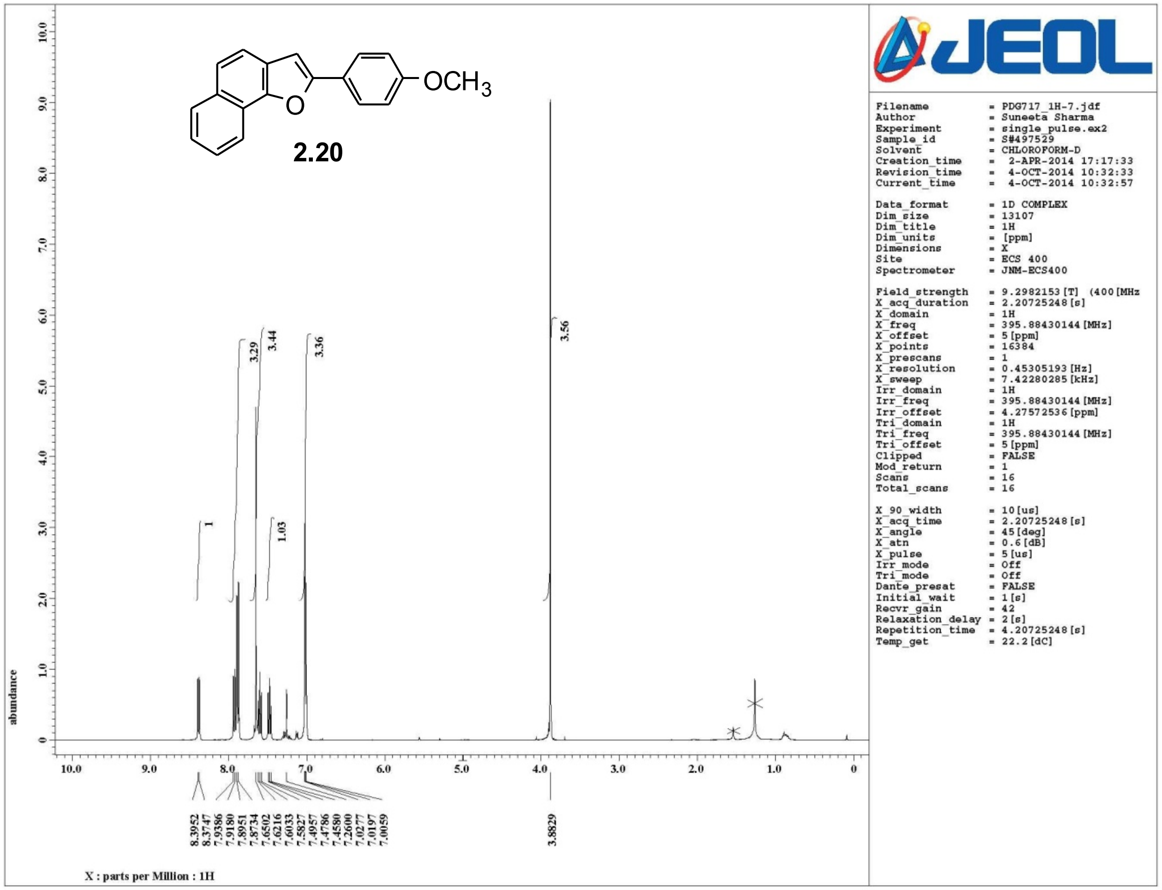
2.19

```
Filename           = PDG6139_13C-5.jdf
Author             = N.Ahmed
Experiment         = single_pulse_dec
Sample_id          = S#623454
Solvent            = CHLOROFORM-D
Creation_time      = 22-AUG-2013 21:04:01
Revision_time      =  3-OCT-2014 20:26:41
Current_time       =  6-FEB-2015 18:09:05

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

Field_strength     = 11.7473579[T]  (500[MH
X_acq_duration     = 0.82837504[s]
X_domain           = 13C
X_freq             = 125.76529768[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 1.20718268[Hz]
X_sweep            = 39.55696203[kHz]
Irr_domain         = 1H
Irr_freq           = 500.15991521[MHz]
Irr_offset         = 5.0[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 1000
Total_scans        = 1000

X_90_width         = 9.62[us]
X_acq_time         = 0.82837504[s]
X_angle            = 30[deg]
X_atn              = 7.1[dB]
X_pulse            = 3.20666667[us]
Irr_atn_dec        = 19.5[dB]
Irr_atn_noe        = 21.5[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 1[s]
Recvr_gain         = 58
Relaxation_delay   = 1[s]
Repetition_time    = 1.82837504[s]
Temp_get           = 21[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.19**

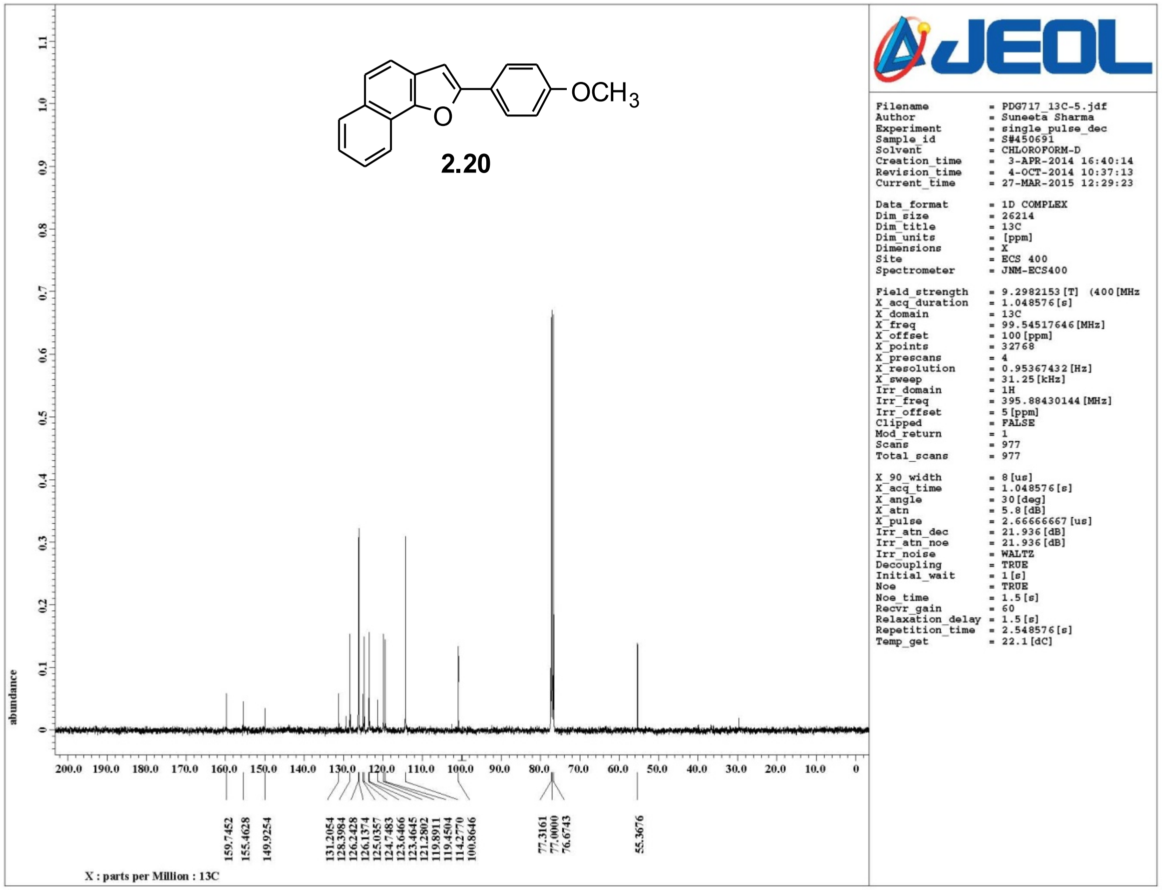


EI (HRMS) spectrum of **2.19**



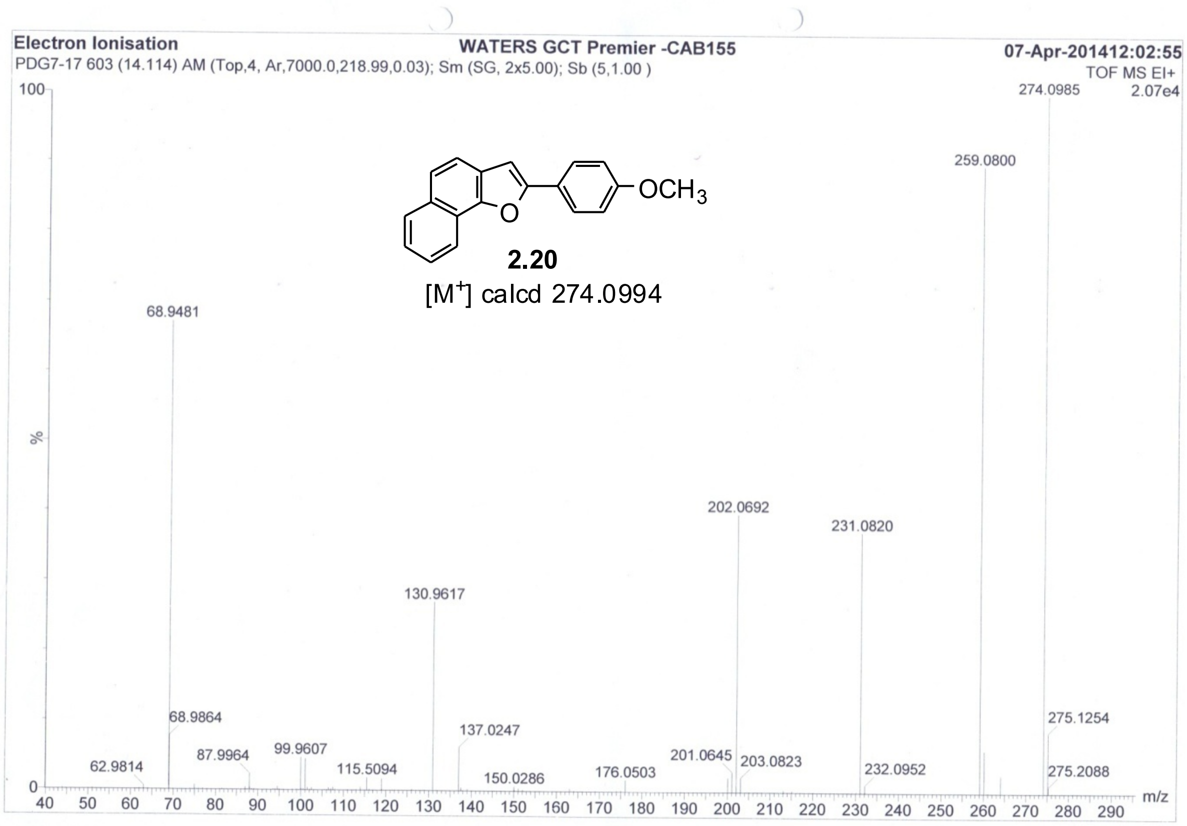
```
Filename          = PDG717_1H-7.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#497529
Solvent           = CHLOROFORM-D
Creation_time     =  2-APR-2014 17:17:33
Revision_time     =  4-OCT-2014 10:32:33
Current_time      =  4-OCT-2014 10:32:57

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 4.27572536[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 22.2[dC]
```

^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **2.20**

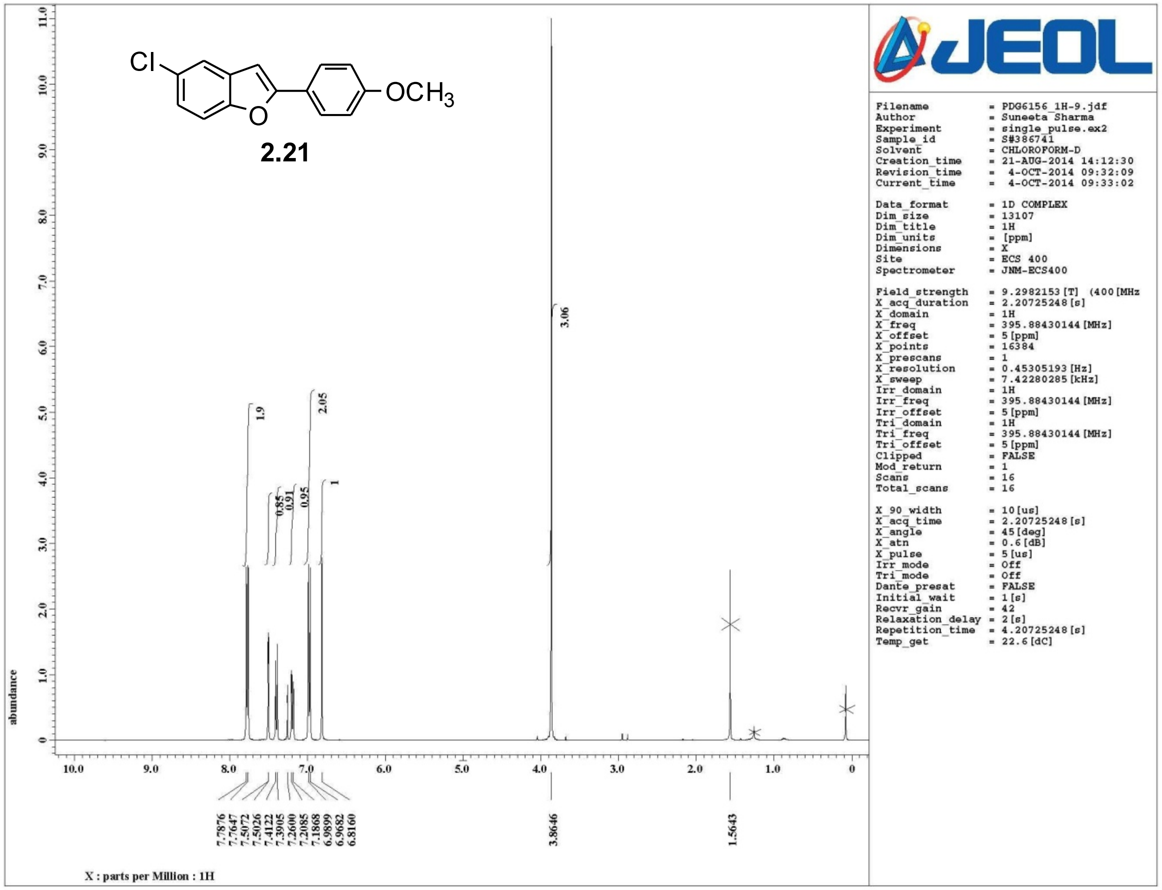


```
Filename          = PDG717_13C-5.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#450691
Solvent           = CHLOROFORM-D
Creation_time     =  3-APR-2014 16:40:14
Revision_time     =  4-OCT-2014 10:37:13
Current_time      = 27-MAR-2015 12:29:23

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 977
Total_scans       = 977

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1.5[s]
Recvr_gain        = 60
Relaxation_delay  = 1.5[s]
Repetition_time   = 2.548576[s]
Temp_get          = 22.1[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **2.20**

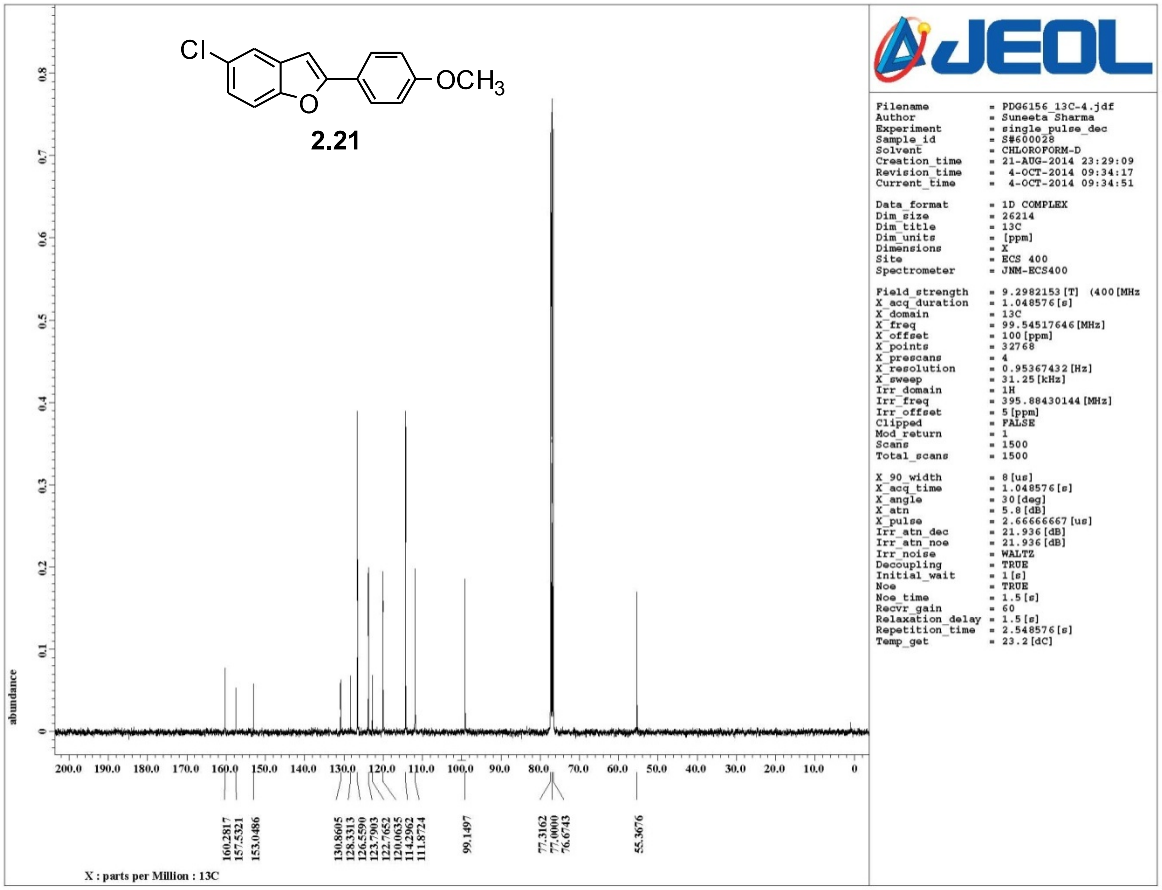


EI (HRMS) spectrum of **2.20**



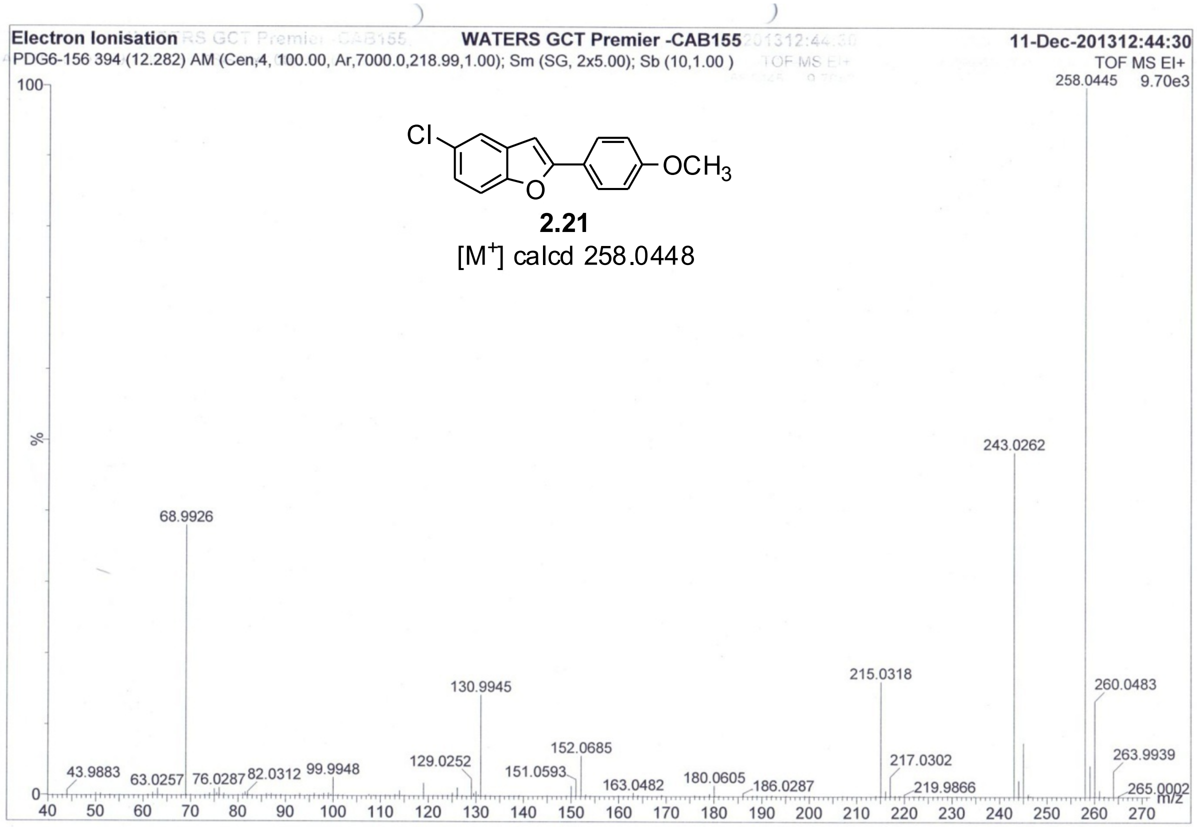
```
Filename          = PDG6156_1H-9.jdf
Author            = Suneeta_Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#386741
Solvent           = CHLOROFORM-D
Creation_time     = 21-AUG-2014 14:12:30
Revision_time     =  4-OCT-2014 09:32:09
Current_time      =  4-OCT-2014 09:33:02

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 22.6[dC]
```

^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **2.21**

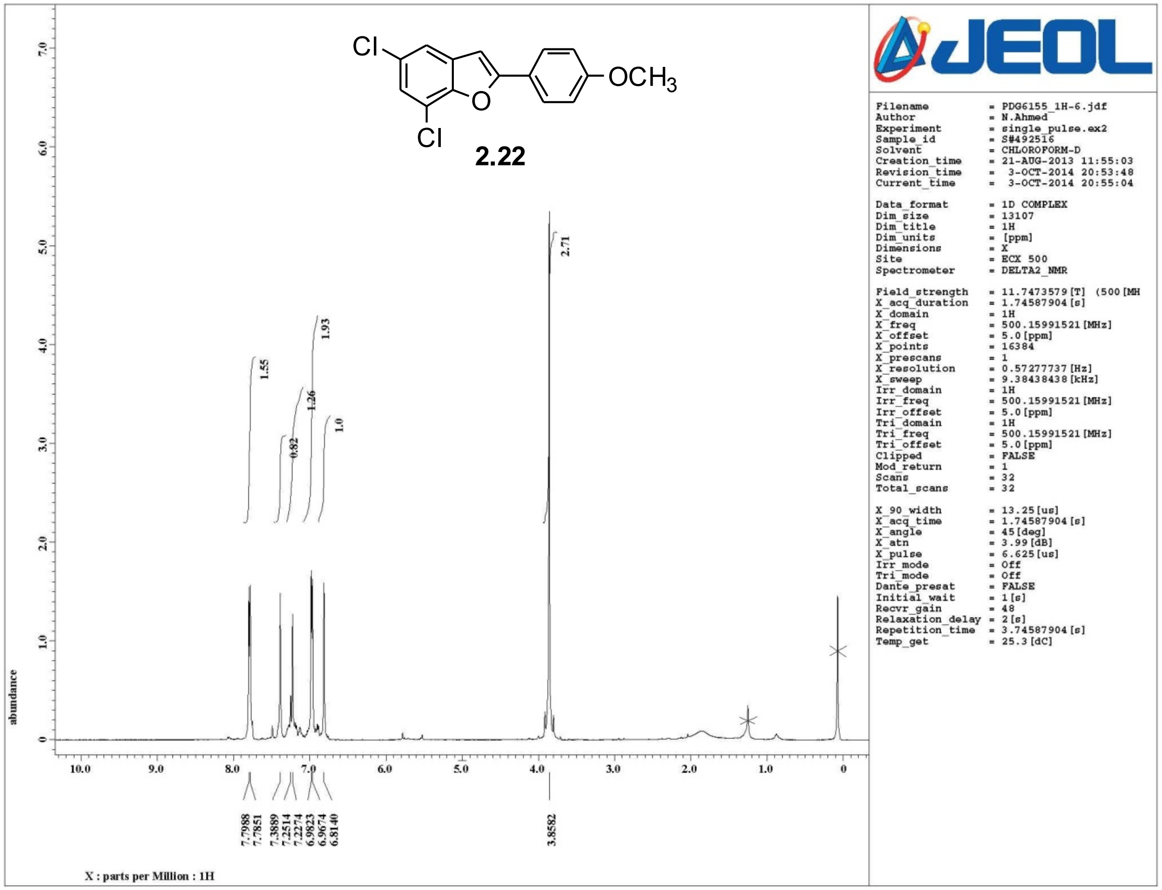


```
Filename          = PDG6156_13C-4.jdf
Author            = Suneeta_Sharma
Experiment        = single_pulse_dec
Sample_id         = S#600028
Solvent           = CHLOROFORM-D
Creation_time     = 21-AUG-2014 23:29:09
Revision_time     =  4-OCT-2014 09:34:17
Current_time      =  4-OCT-2014 09:34:51

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1500
Total_scans       = 1500

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1.5[s]
Recvr_gain        = 60
Relaxation_delay  = 1.5[s]
Repetition_time   = 2.548576[s]
Temp_get          = 23.2[dC]
```

^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **2.21**

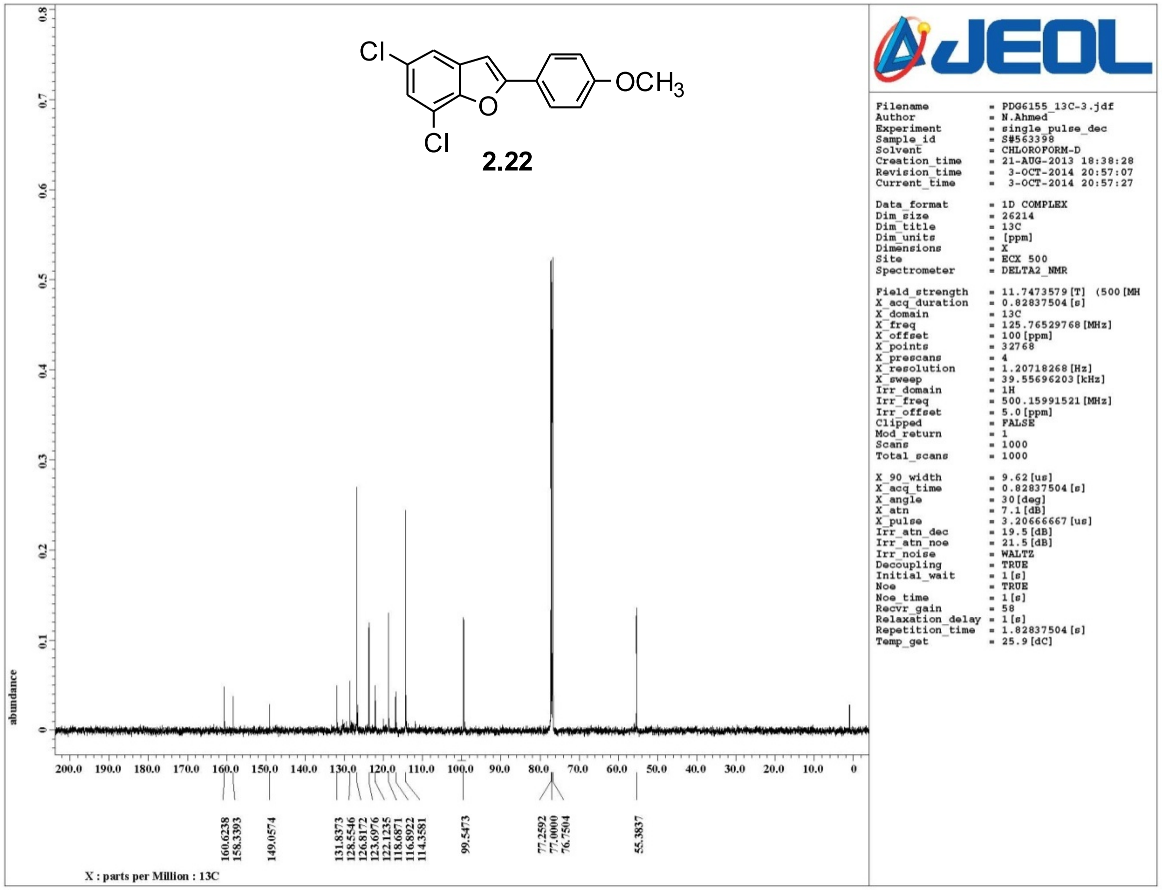


EI (HRMS) spectrum of **2.21**



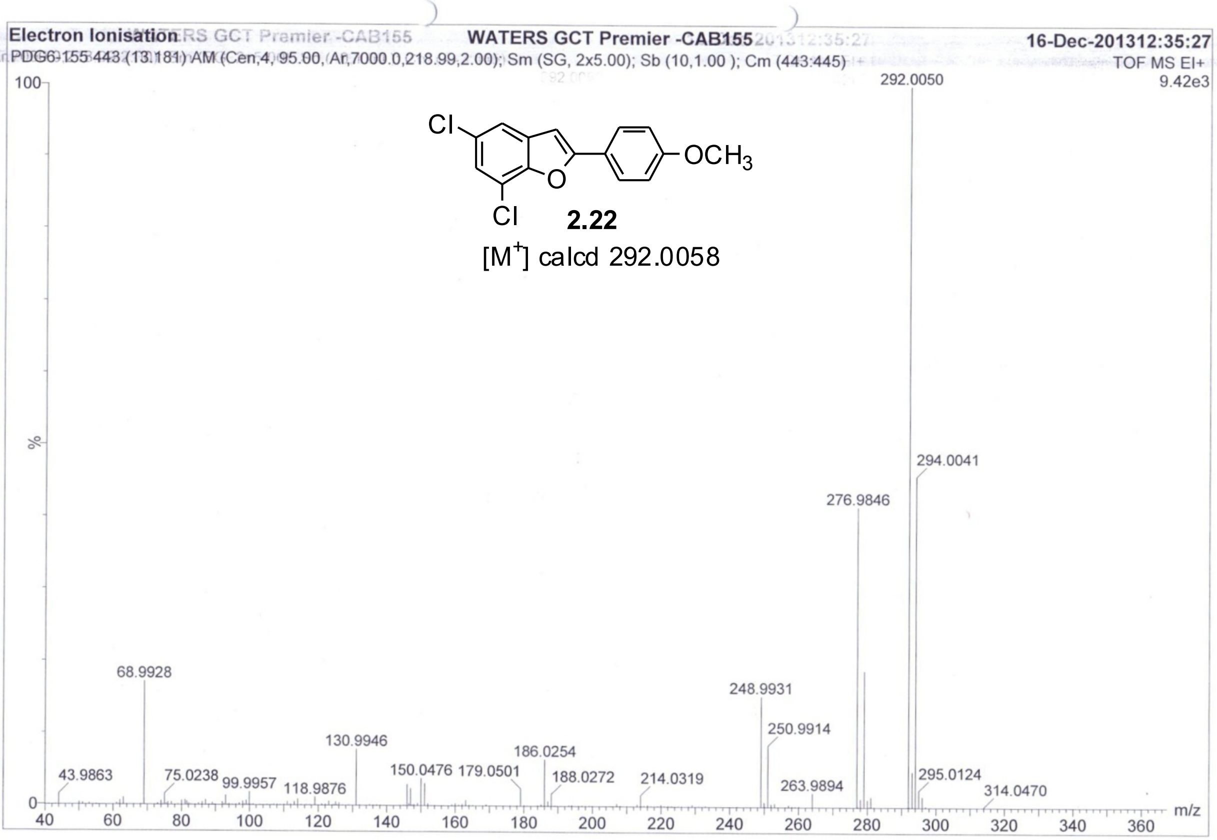
```
Filename          = PDG6155_1H-6.jdf
Author            = N.Ahmed
Experiment        = single_pulse.ex2
Sample_id         = S#492516
Solvent           = CHLOROFORM-D
Creation_time     = 21-AUG-2013 11:55:03
Revision_time     =  3-OCT-2014 20:53:48
Current_time      =  3-OCT-2014 20:55:04

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 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             = 32
Total_scans       = 32

X_90_width        = 13.25[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 2[s]
Repetition_time   = 3.74587904[s]
Temp_get          = 25.3[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **2.22**

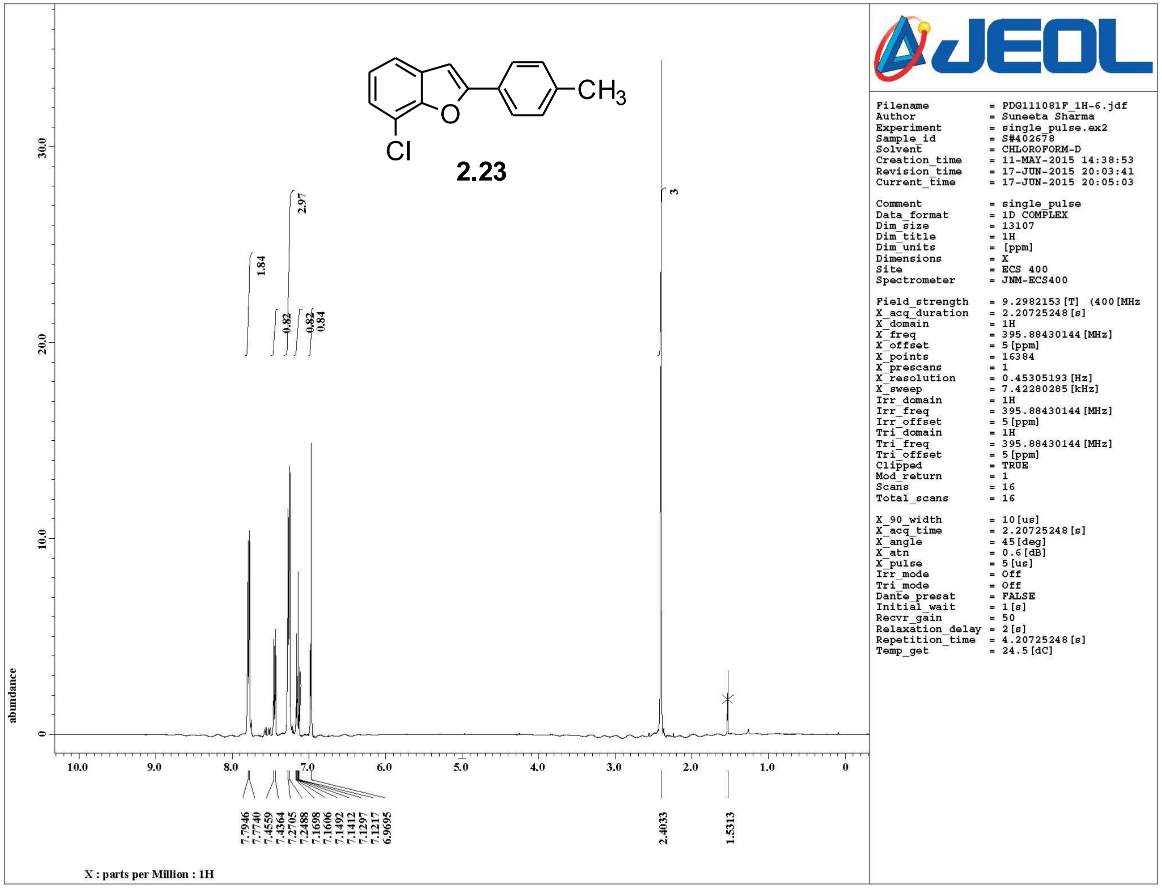


```
Filename           = PDG6155_13C-3.jdf
Author             = N.Ahmed
Experiment         = single_pulse_dec
Sample_id          = S#563398
Solvent            = CHLOROFORM-D
Creation_time      = 21-AUG-2013 18:38:28
Revision_time      =  3-OCT-2014 20:57:07
Current_time       =  3-OCT-2014 20:57:27

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

Field_strength     = 11.7473579[T] (500[MH
X_acq_duration     = 0.82837504[s]
X_domain           = 13C
X_freq             = 125.76529768[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 1.20718268[Hz]
X_sweep            = 39.55696203[kHz]
Irr_domain         = 1H
Irr_freq           = 500.15991521[MHz]
Irr_offset         = 5.0[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 1000
Total_scans        = 1000

X_90_width         = 9.62[us]
X_acq_time         = 0.82837504[s]
X_angle            = 30[deg]
X_atn              = 7.1[dB]
X_pulse            = 3.20666667[us]
Irr_atn_dec        = 19.5[dB]
Irr_atn_noe        = 21.5[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 1[s]
Recvr_gain         = 58
Relaxation_delay   = 1[s]
Repetition_time    = 1.82837504[s]
Temp_get           = 25.9[dC]
```

^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **2.22**

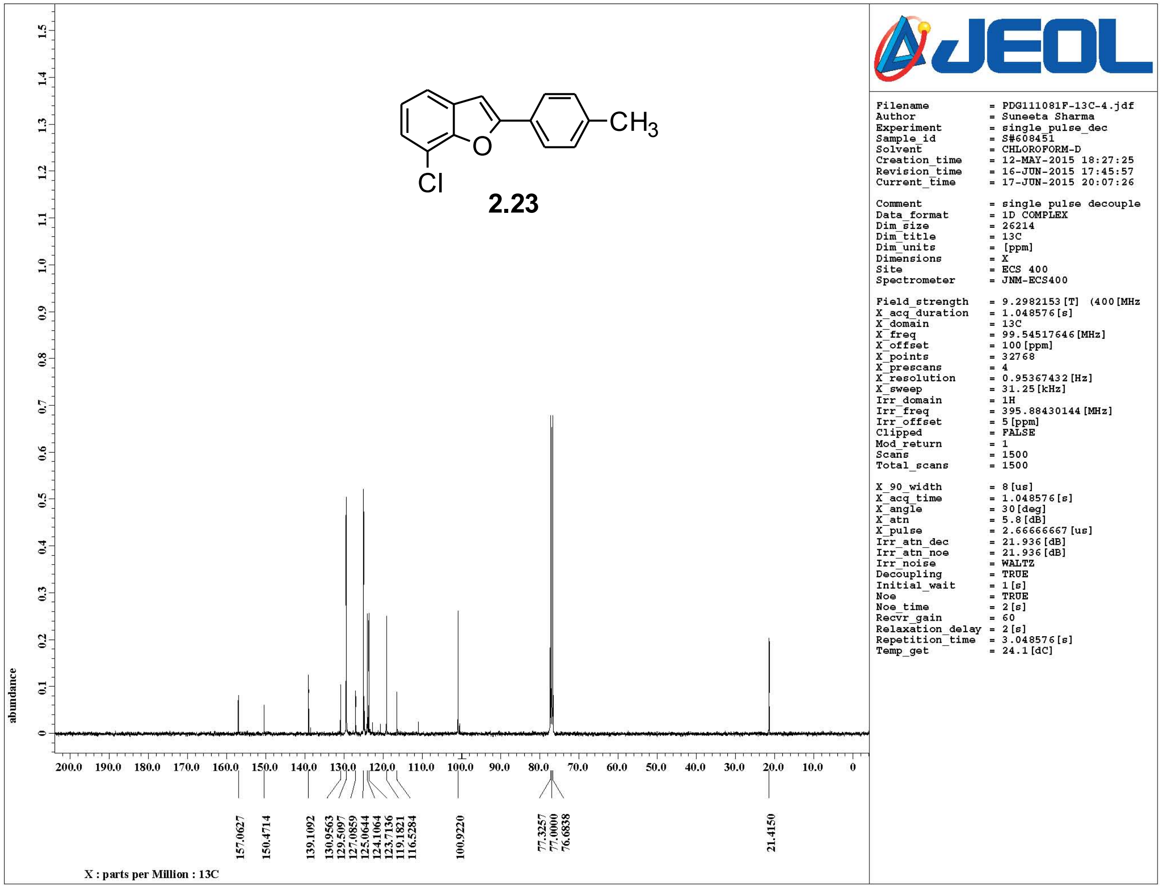


EI (HRMS) spectrum of **2.22**



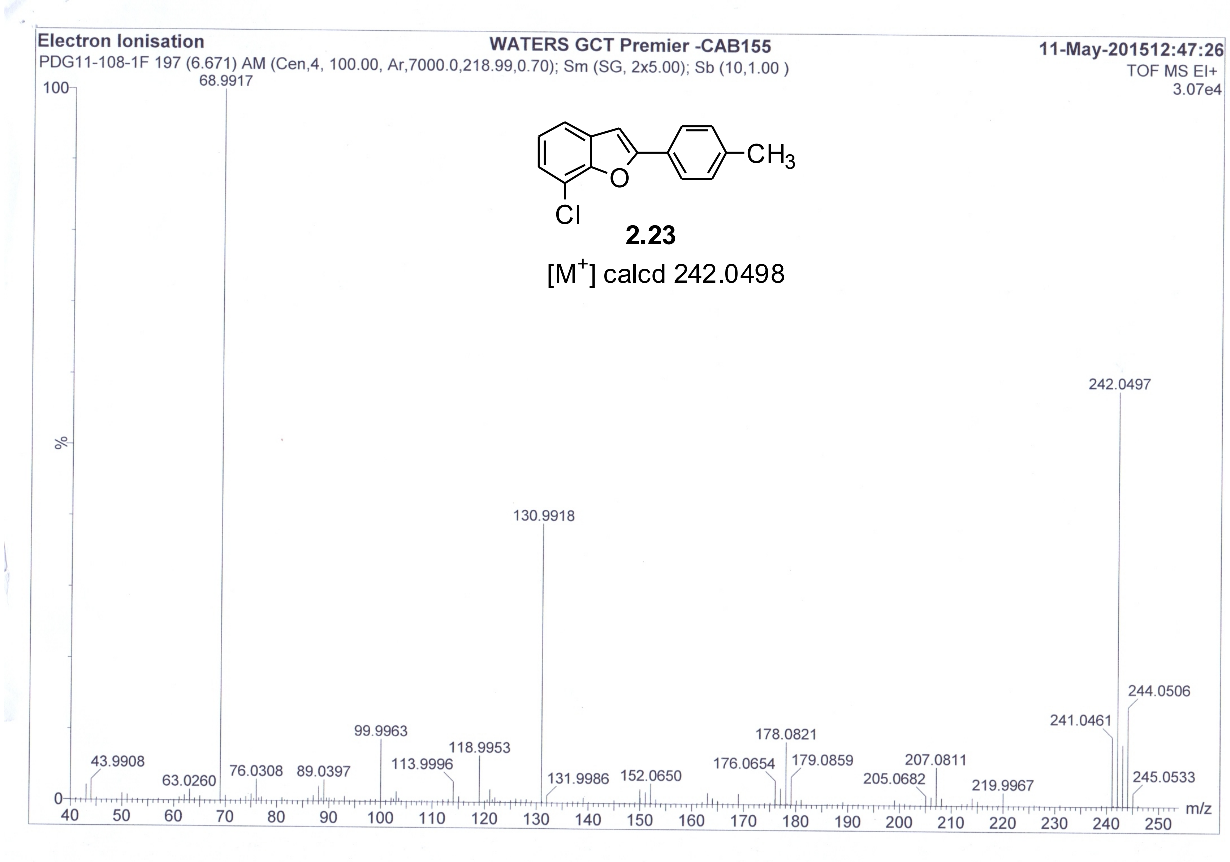
```
Filename          = PDG111081F_1H-6.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#402678
Solvent           = CHLOROFORM-D
Creation_time     = 11-MAY-2015 14:38:53
Revision_time     = 17-JUN-2015 20:03:41
Current_time      = 17-JUN-2015 20:05:03

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 24.5[dC]
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **2.23**

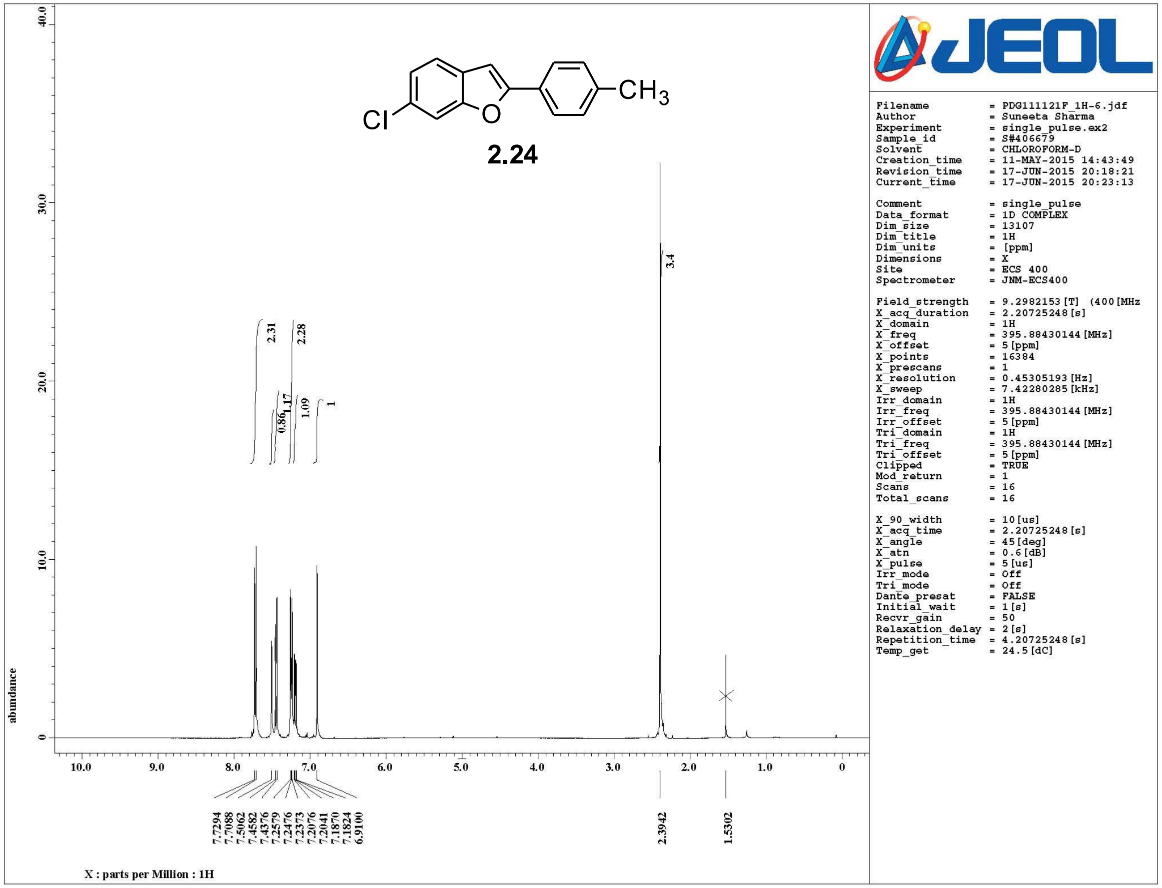


```
Filename           = PDG111081F-13C-4.jdf
Author             = Suneeta Sharma
Experiment         = single_pulse_dec
Sample_id          = S#608451
Solvent            = CHLOROFORM-D
Creation_time      = 12-MAY-2015 18:27:25
Revision_time      = 16-JUN-2015 17:45:57
Current_time       = 17-JUN-2015 20:07:26

Comment            = single pulse decouple
Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.2982153[T] (400[MHz
X_acq_duration     = 1.048576[s]
X_domain           = 13C
X_freq             = 99.54517646[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 0.95367432[Hz]
X_sweep            = 31.25[kHz]
Irr_domain         = 1H
Irr_freq           = 395.88430144[MHz]
Irr_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 1500
Total_scans        = 1500

X_90_width         = 8[us]
X_acq_time         = 1.048576[s]
X_angle            = 30[deg]
X_atn              = 5.8[dB]
X_pulse            = 2.66666667[us]
Irr_atn_dec        = 21.936[dB]
Irr_atn_noe        = 21.936[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 60
Relaxation_delay   = 2[s]
Repetition_time    = 3.048576[s]
Temp_get           = 24.1[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **2.23**



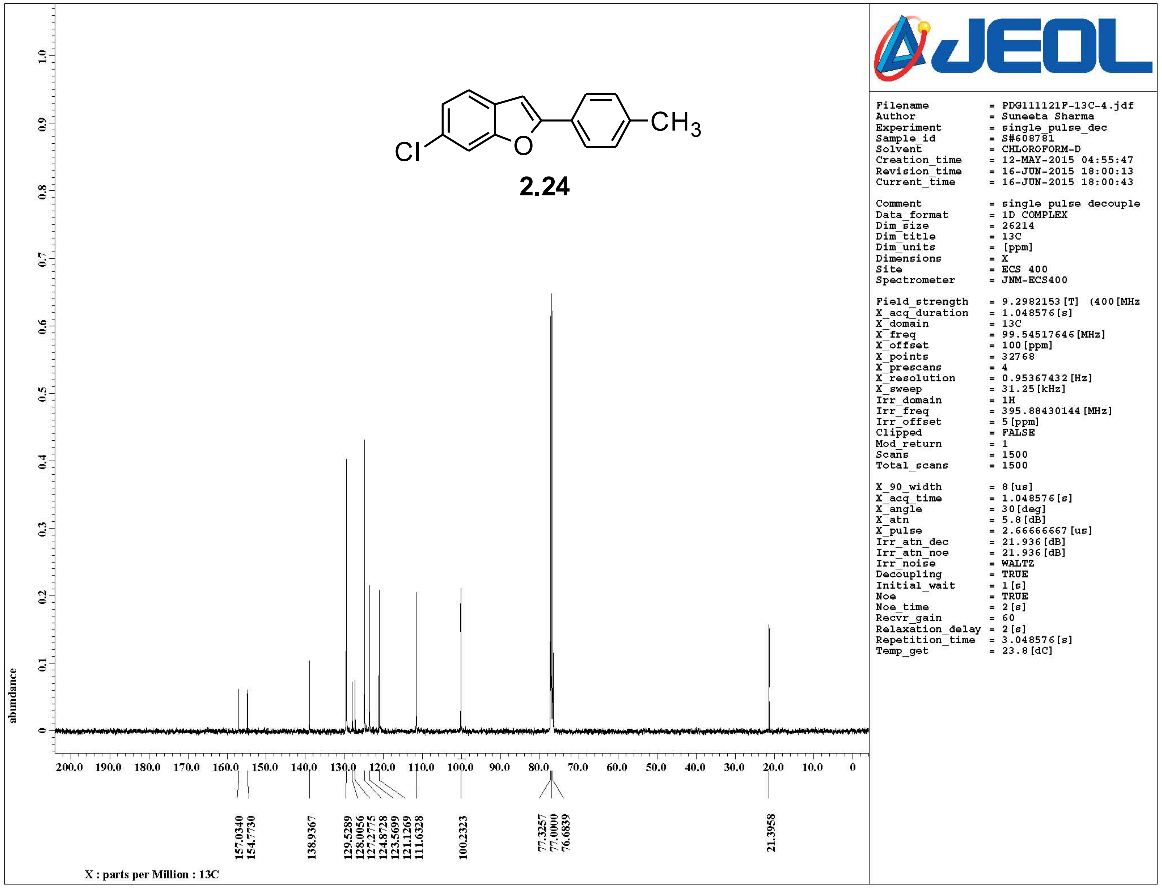
EI (HRMS) spectrum of **2.23**



2.24

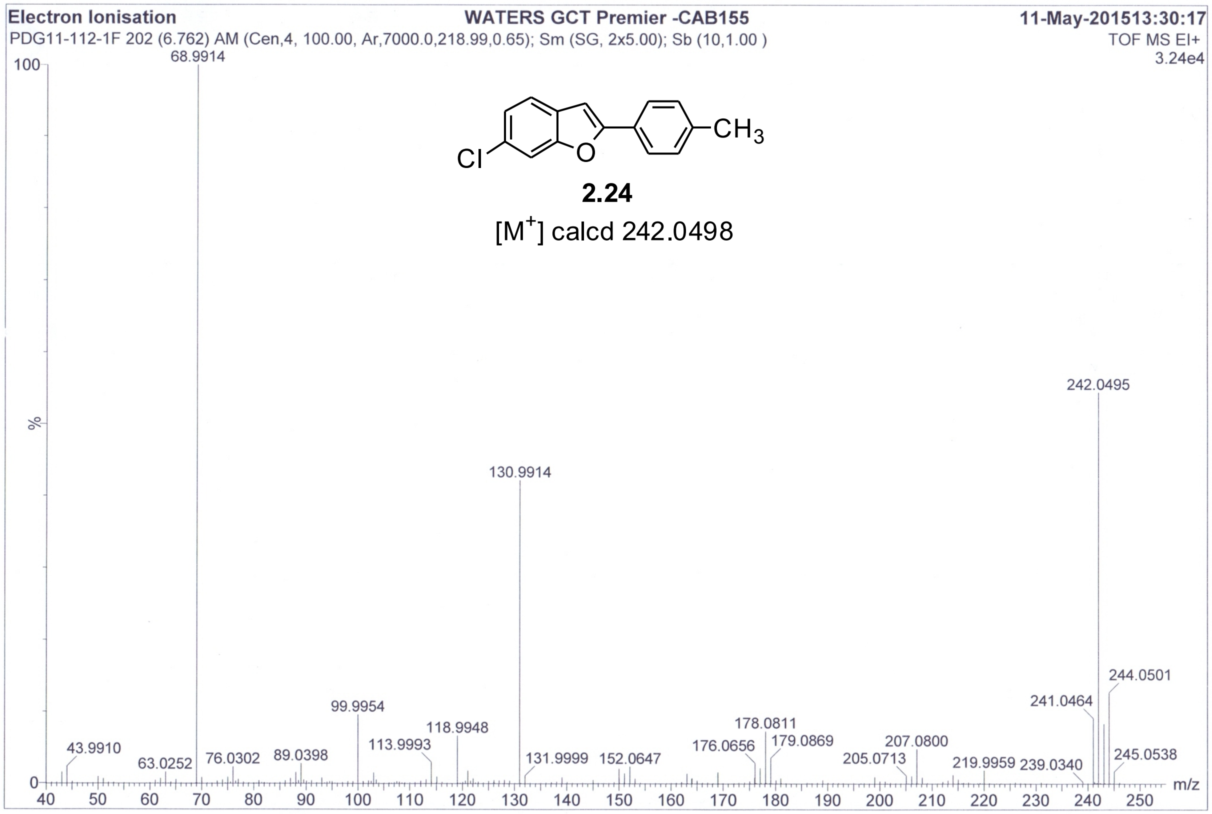
```
Filename           = PDG111121F_1H-6.jdf
Author             = Suneeta Sharma
Experiment         = single_pulse.ex2
Sample_id          = S#406679
Solvent            = CHLOROFORM-D
Creation_time      = 11-MAY-2015 14:43:49
Revision_time      = 17-JUN-2015 20:18:21
Current_time       = 17-JUN-2015 20:23:13

Comment            = single_pulse
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.2982153[T] (400[MHz
X_acq_duration     = 2.20725248[s]
X_domain           = 1H
X_freq             = 395.88430144[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 1
X_resolution       = 0.45305193[Hz]
X_sweep            = 7.42280285[kHz]
Irr_domain         = 1H
Irr_freq           = 395.88430144[MHz]
Irr_offset         = 5[ppm]
Tri_domain         = 1H
Tri_freq           = 395.88430144[MHz]
Tri_offset         = 5[ppm]
Clipped            = TRUE
Mod_return         = 1
Scans              = 16
Total_scans        = 16

X_90_width         = 10[us]
X_acq_time         = 2.20725248[s]
X_angle            = 45[deg]
X_atn              = 0.6[dB]
X_pulse            = 5[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 50
Relaxation_delay   = 2[s]
Repetition_time    = 4.20725248[s]
Temp_get           = 24.5[dC]
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **2.24**

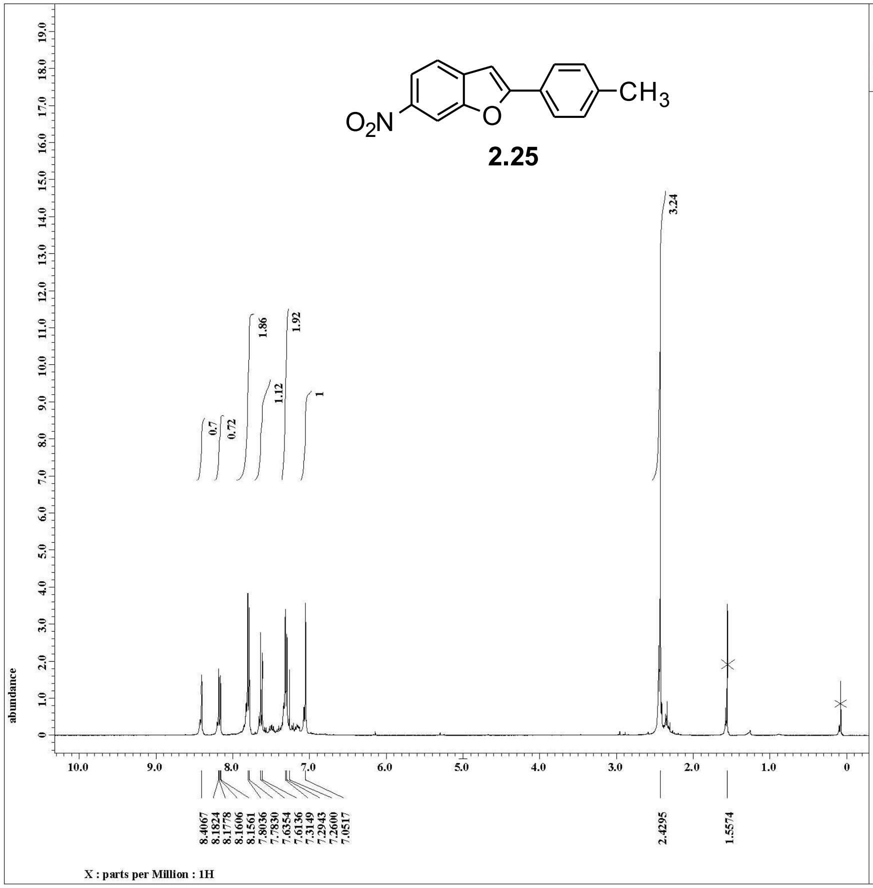


```
Filename          = PDG111121F-13C-4.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#608781
Solvent           = CHLOROFORM-D
Creation_time     = 12-MAY-2015 04:55:47
Revision_time     = 16-JUN-2015 18:00:13
Current_time      = 16-JUN-2015 18:00:43

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1500
Total_scans       = 1500

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.048576[s]
Temp_get          = 23.8[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **2.24**

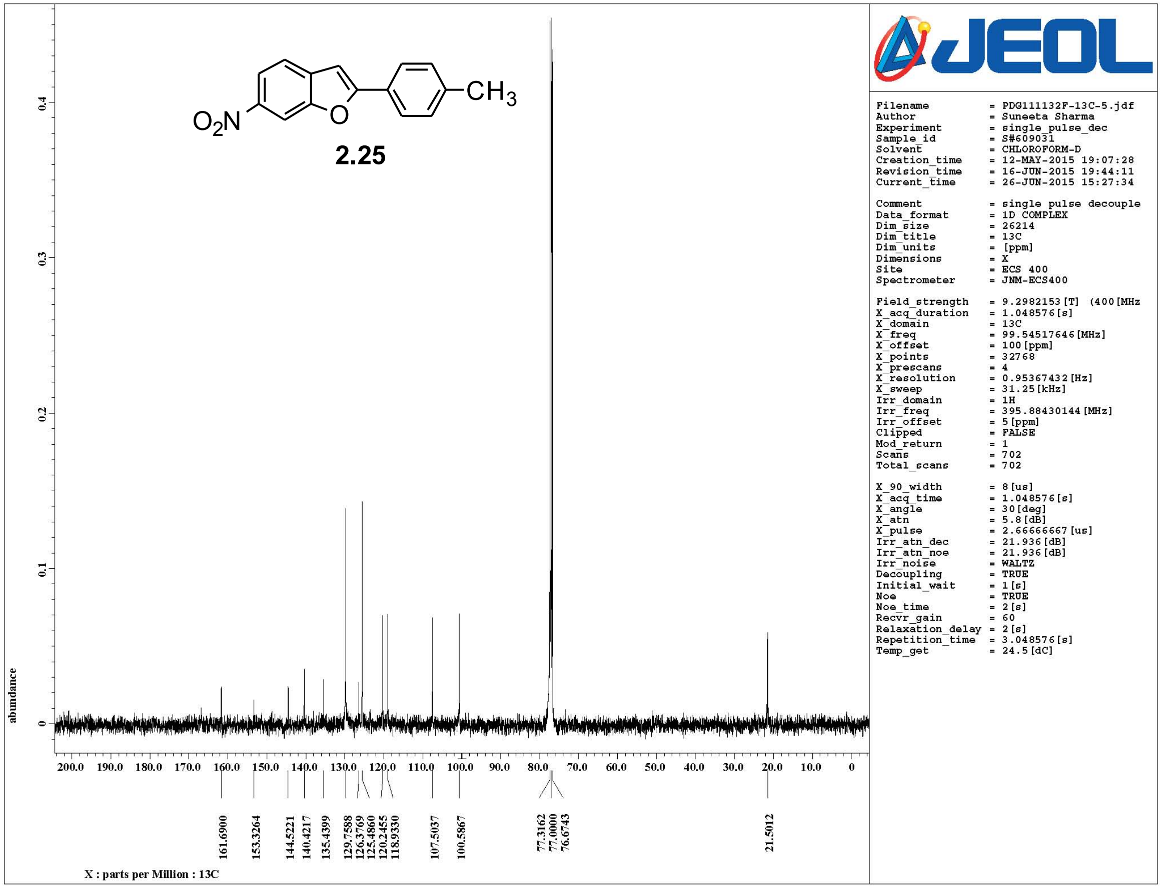


EI (HRMS) spectrum of **2.24**



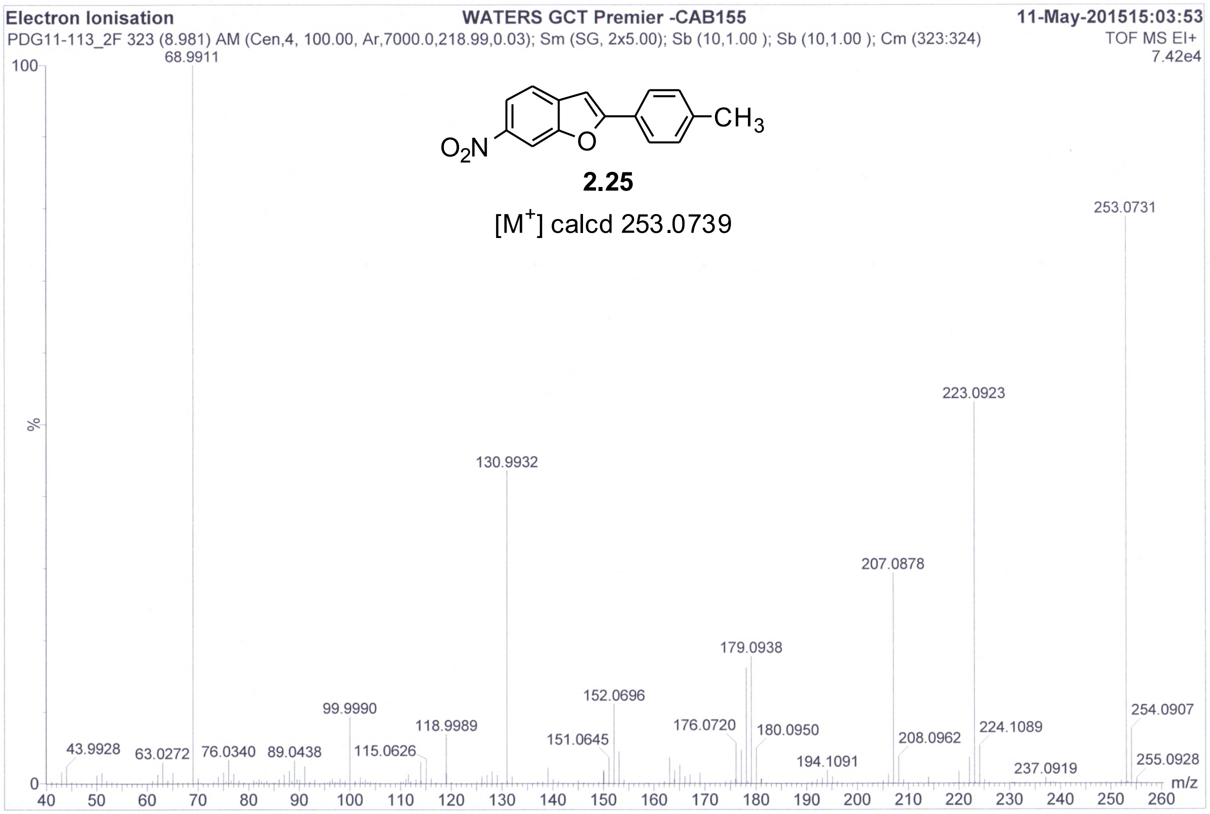
```
Filename           = PDG111132F_1H-6.jdf
Author             = Suneeta Sharma
Experiment         = single_pulse.ex2
Sample_id          = S#409920
Solvent            = CHLOROFORM-D
Creation_time      = 11-MAY-2015 14:49:19
Revision_time      = 17-JUN-2015 20:38:36
Current_time       = 17-JUN-2015 20:43:35

Comment            = single_pulse
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.2982153[T] (400[MHz
X_acq_duration     = 2.20725248[s]
X_domain           = 1H
X_freq             = 395.88430144[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 1
X_resolution       = 0.45305193[Hz]
X_sweep            = 7.42280285[kHz]
Irr_domain         = 1H
Irr_freq           = 395.88430144[MHz]
Irr_offset         = 5[ppm]
Tri_domain         = 1H
Tri_freq           = 395.88430144[MHz]
Tri_offset         = 5[ppm]
Clipped            = TRUE
Mod_return         = 1
Scans              = 16
Total_scans        = 16

X_90_width         = 10[us]
X_acq_time         = 2.20725248[s]
X_angle            = 45[deg]
X_atn              = 0.6[dB]
X_pulse            = 5[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 50
Relaxation_delay   = 2[s]
Repetition_time    = 4.20725248[s]
Temp_get           = 24.5[dC]
```

^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **2.25**

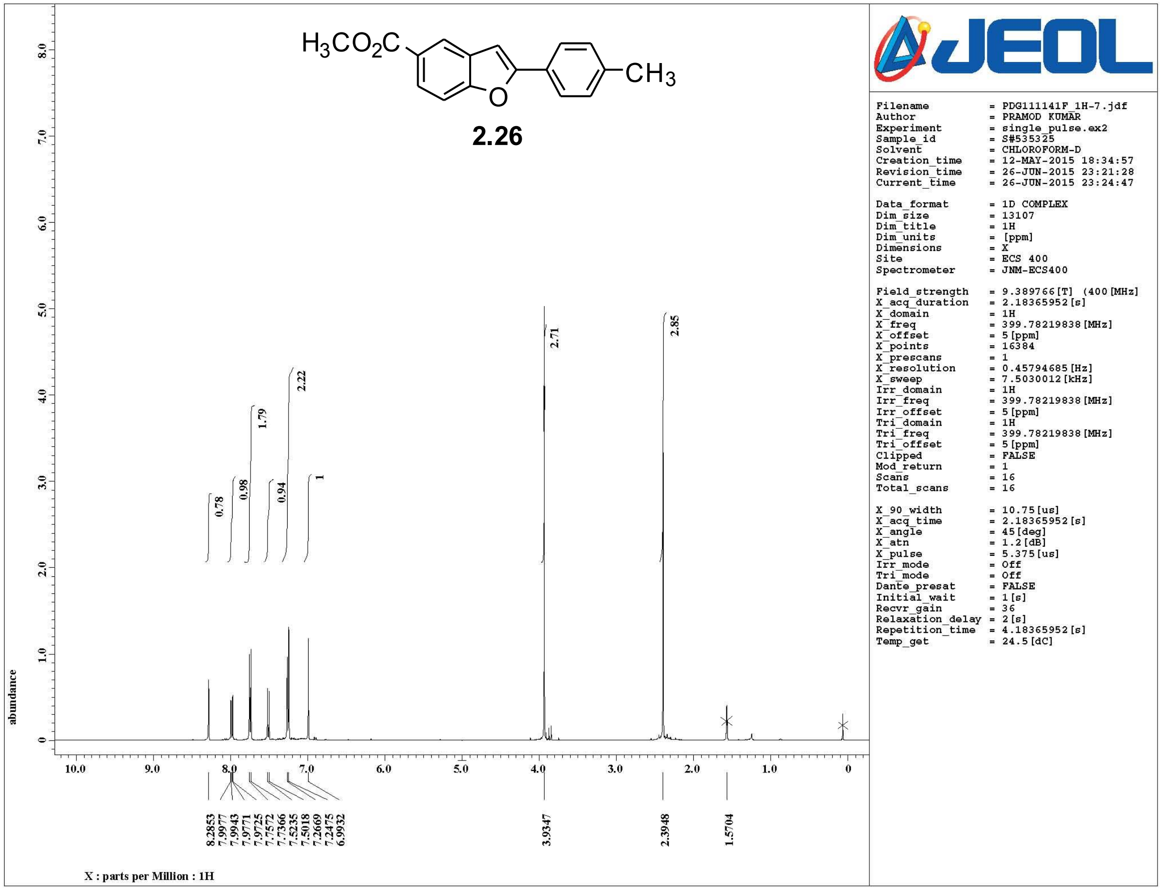


```
Filename          = PDG111132F-13C-5.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#609031
Solvent           = CHLOROFORM-D
Creation_time     = 12-MAY-2015 19:07:28
Revision_time     = 16-JUN-2015 19:44:11
Current_time      = 26-JUN-2015 15:27:34

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 702
Total_scans       = 702

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.048576[s]
Temp_get          = 24.5[dC]
```

^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **2.25**

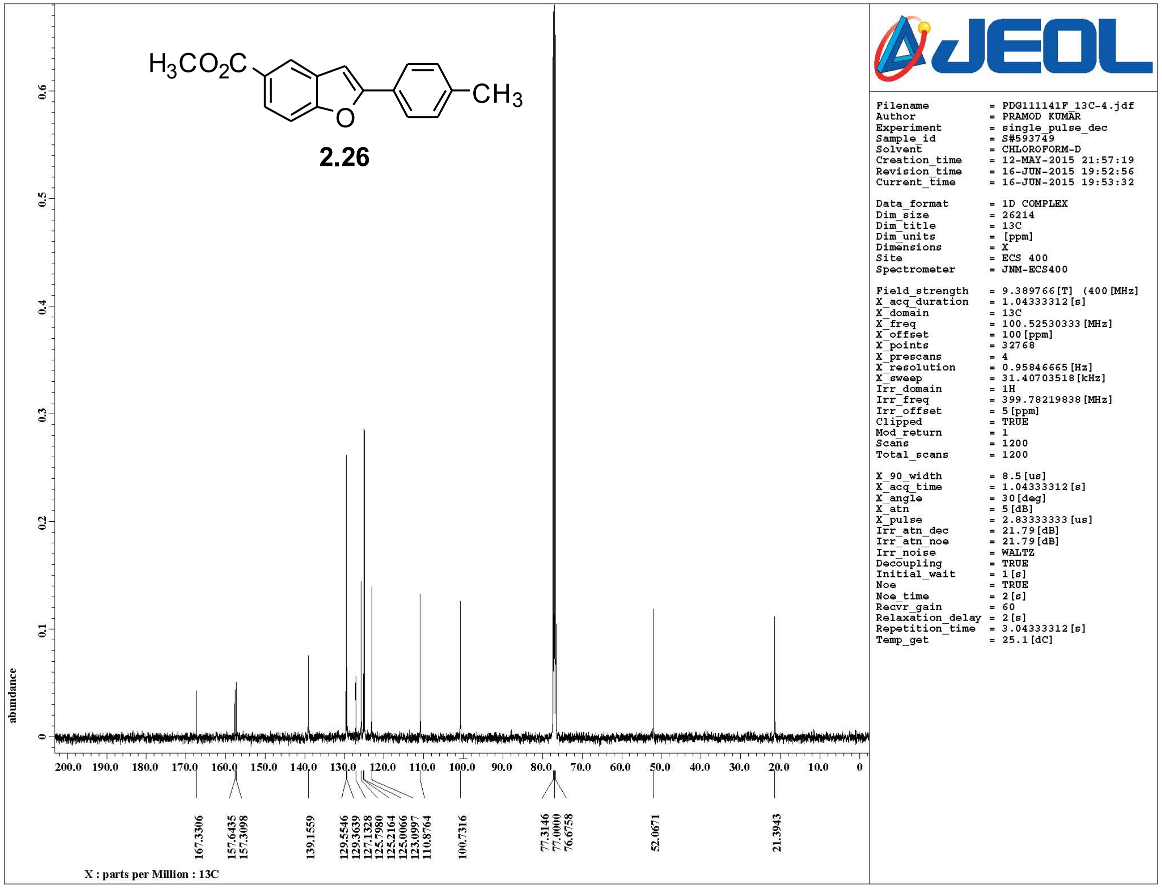


EI (HRMS) spectrum of **2.25**



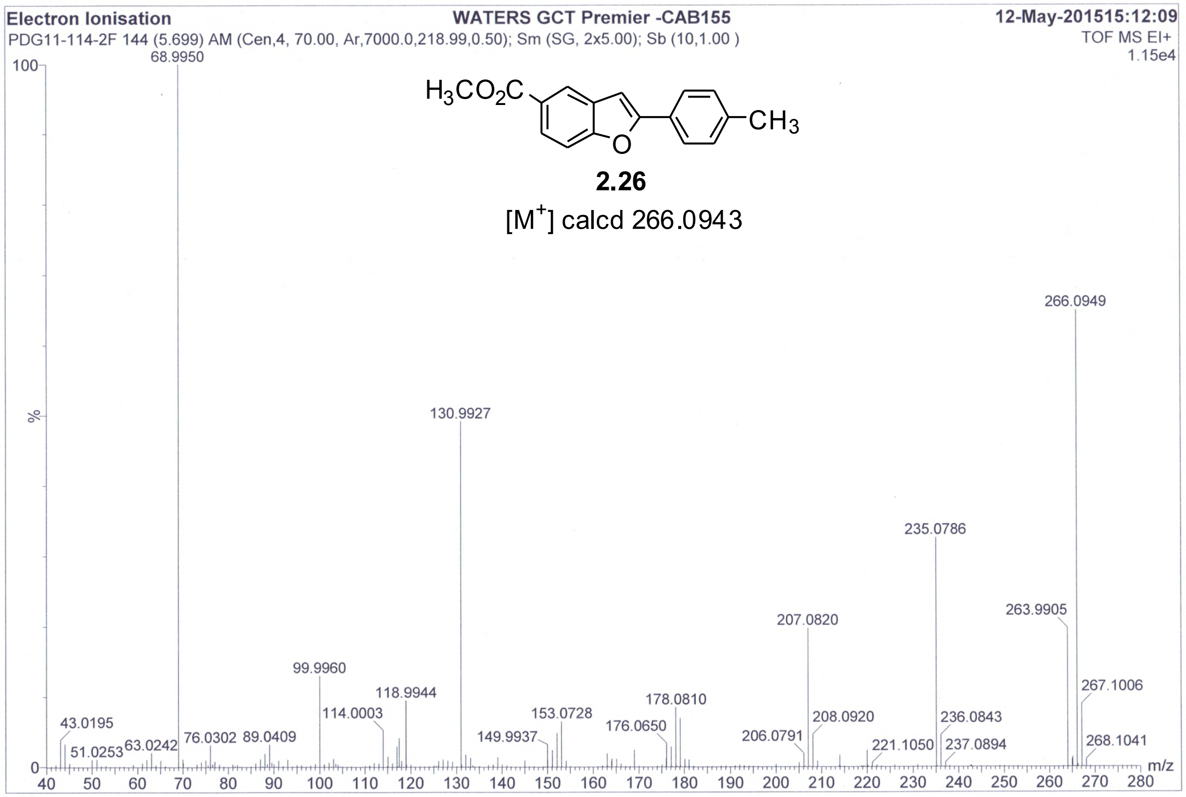
```
Filename          = PDG111141F_1H-7.jdf
Author            = PRAMOD KUMAR
Experiment        = single_pulse.ex2
Sample_id         = S#535325
Solvent           = CHLOROFORM-D
Creation_time     = 12-MAY-2015 18:34:57
Revision_time     = 26-JUN-2015 23:21:28
Current_time      = 26-JUN-2015 23:24:47

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10.75[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 1.2[dB]
X_pulse           = 5.375[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 2[s]
Repetition_time   = 4.18365952[s]
Temp_get          = 24.5[dC]
```

^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **2.26**

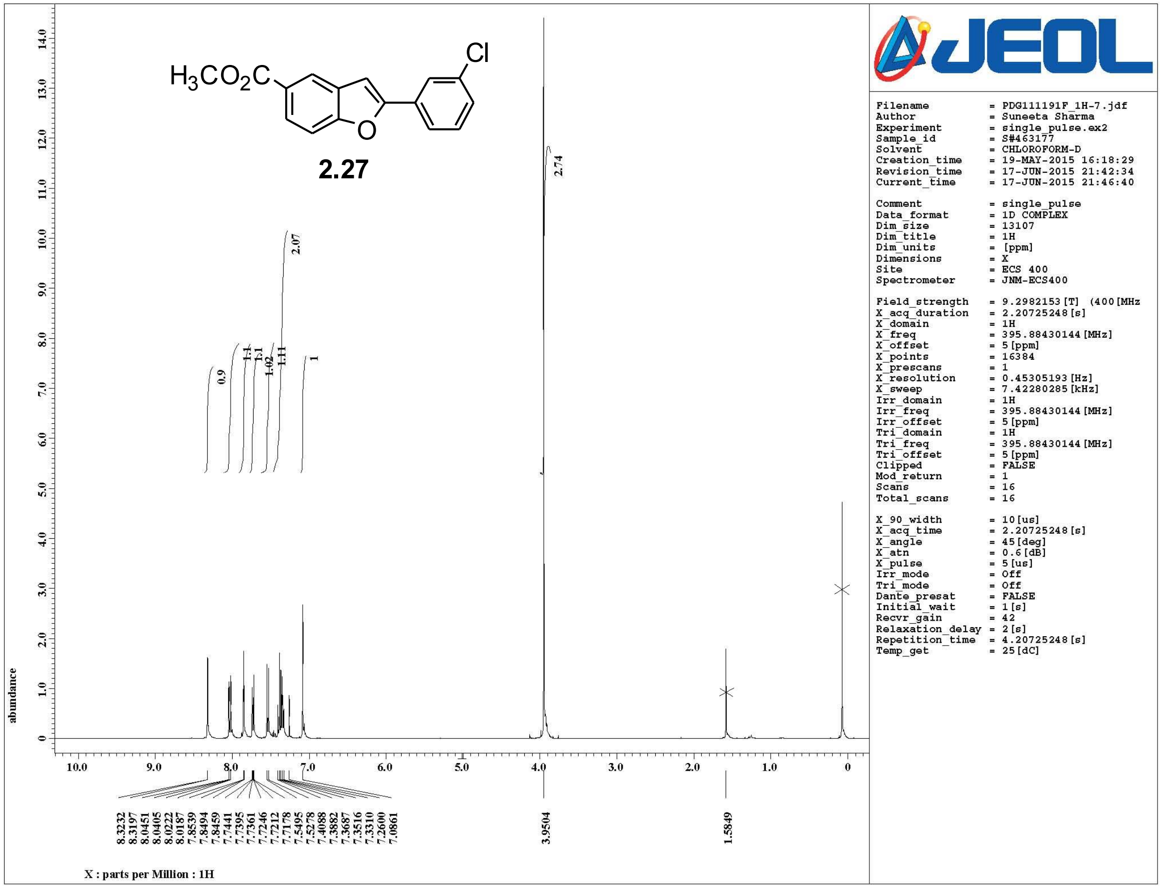


```
Filename          = PDG111141F_13C-4.jdf
Author            = PRAMOD KUMAR
Experiment        = single_pulse_dec
Sample_id         = S#593749
Solvent           = CHLOROFORM-D
Creation_time     = 12-MAY-2015 21:57:19
Revision_time     = 16-JUN-2015 19:52:56
Current_time      = 16-JUN-2015 19:53:32

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 1200
Total_scans       = 1200

X_90_width        = 8.5[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 5[dB]
X_pulse           = 2.83333333[us]
Irr_atn_dec       = 21.79[dB]
Irr_atn_noe       = 21.79[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 25.1[dC]
```

^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **2.26**

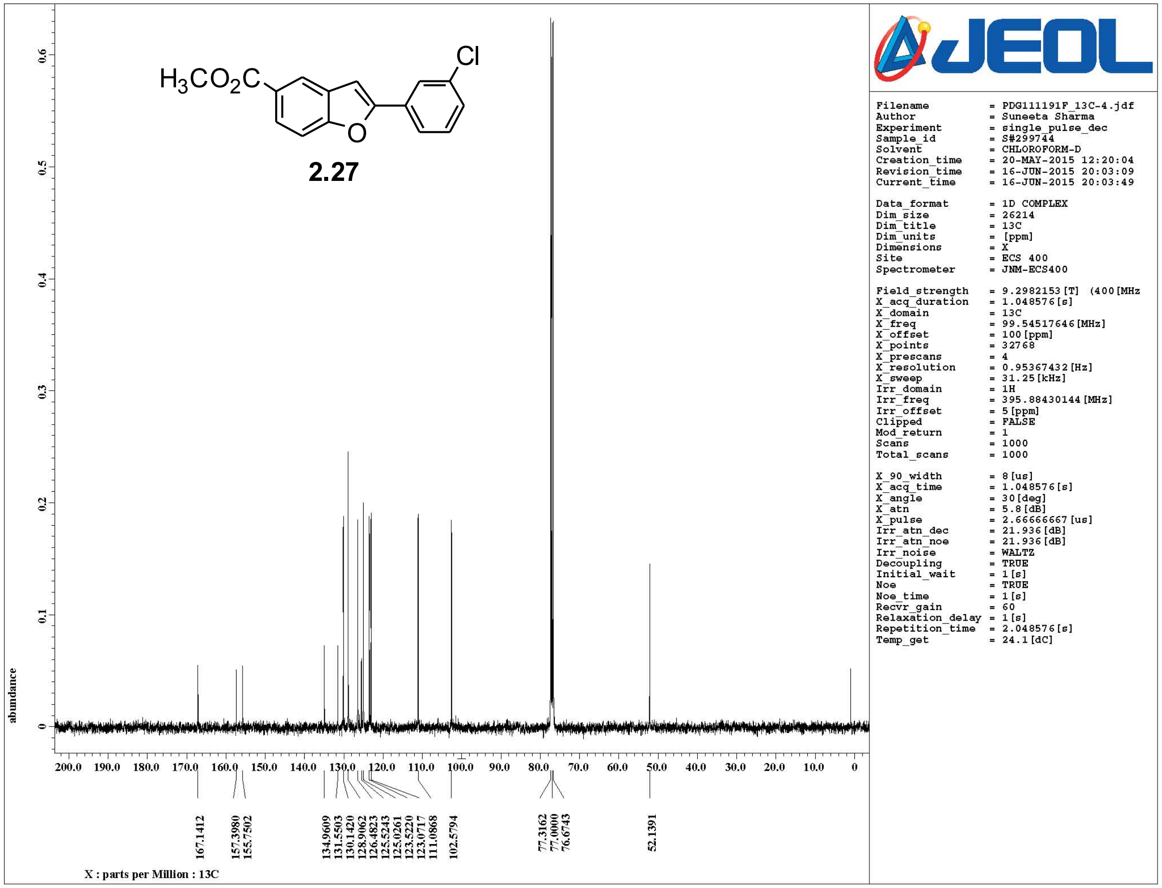


EI (HRMS) spectrum of **2.26**



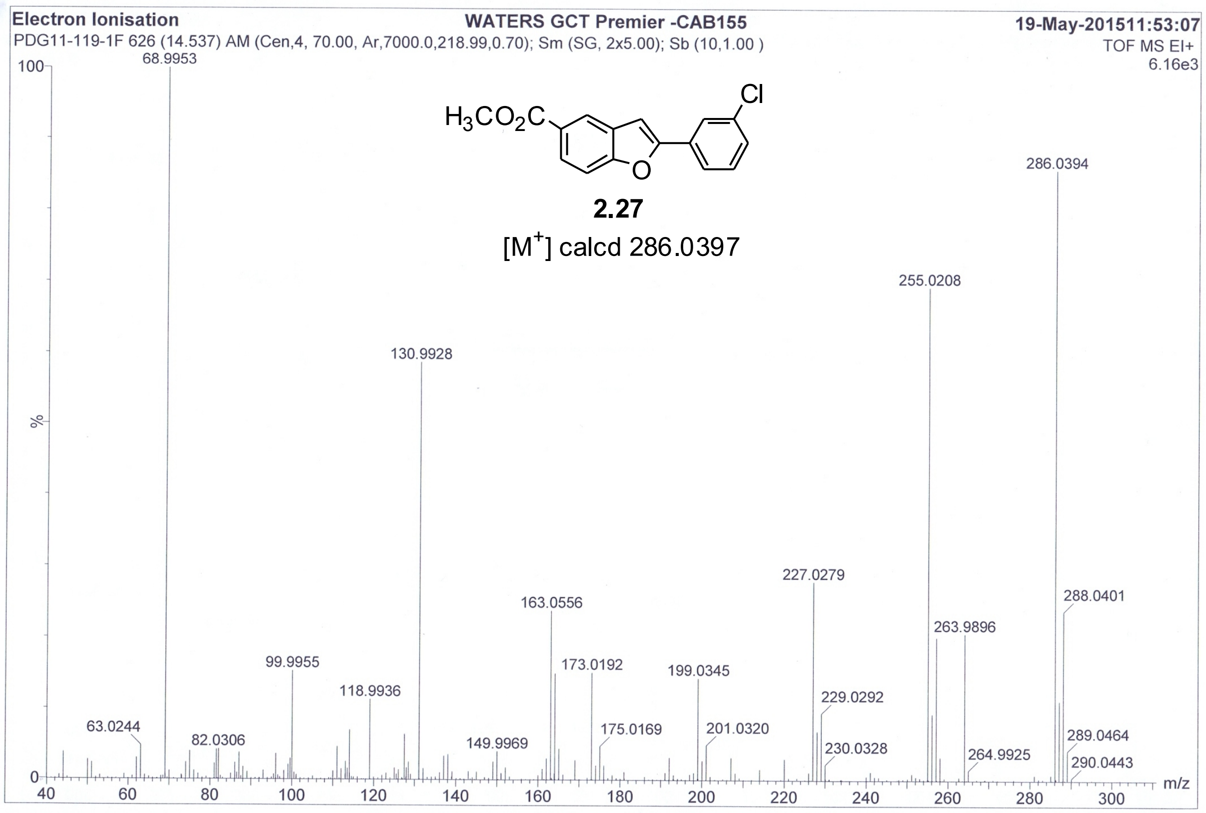
```
Filename            = PDG111191F_1H-7.jdf
Author              = Suneeta Sharma
Experiment          = single_pulse.ex2
Sample_id           = S#463177
Solvent             = CHLOROFORM-D
Creation_time       = 19-MAY-2015 16:18:29
Revision_time       = 17-JUN-2015 21:42:34
Current_time        = 17-JUN-2015 21:46:40

Comment             = single_pulse
Data_format         = 1D COMPLEX
Dim_size            = 13107
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = JNM-ECS400

Field_strength      = 9.2982153[T] (400[MHz
X_acq_duration      = 2.20725248[s]
X_domain            = 1H
X_freq              = 395.88430144[MHz]
X_offset            = 5[ppm]
X_points            = 16384
X_prescans          = 1
X_resolution        = 0.45305193[Hz]
X_sweep             = 7.42280285[kHz]
Irr_domain          = 1H
Irr_freq            = 395.88430144[MHz]
Irr_offset          = 5[ppm]
Tri_domain          = 1H
Tri_freq            = 395.88430144[MHz]
Tri_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 16
Total_scans         = 16

X_90_width          = 10[us]
X_acq_time          = 2.20725248[s]
X_angle             = 45[deg]
X_atn               = 0.6[dB]
X_pulse             = 5[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 42
Relaxation_delay    = 2[s]
Repetition_time     = 4.20725248[s]
Temp_get            = 25[dC]
```

^1H NMR (400 MHz, $CDCl_3$) spectrum of **2.27**

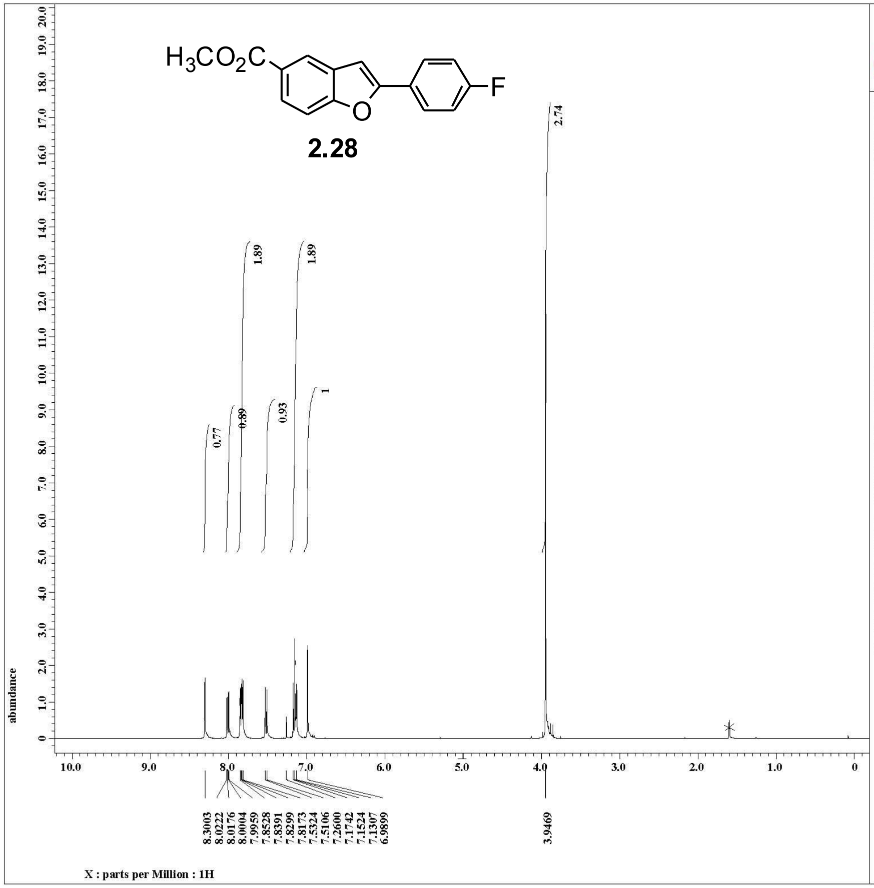


```
Filename          = PDG111191F_13C-4.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#299744
Solvent           = CHLOROFORM-D
Creation_time     = 20-MAY-2015 12:20:04
Revision_time     = 16-JUN-2015 20:03:09
Current_time      = 16-JUN-2015 20:03:49

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.048576[s]
Temp_get          = 24.1[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **2.27**

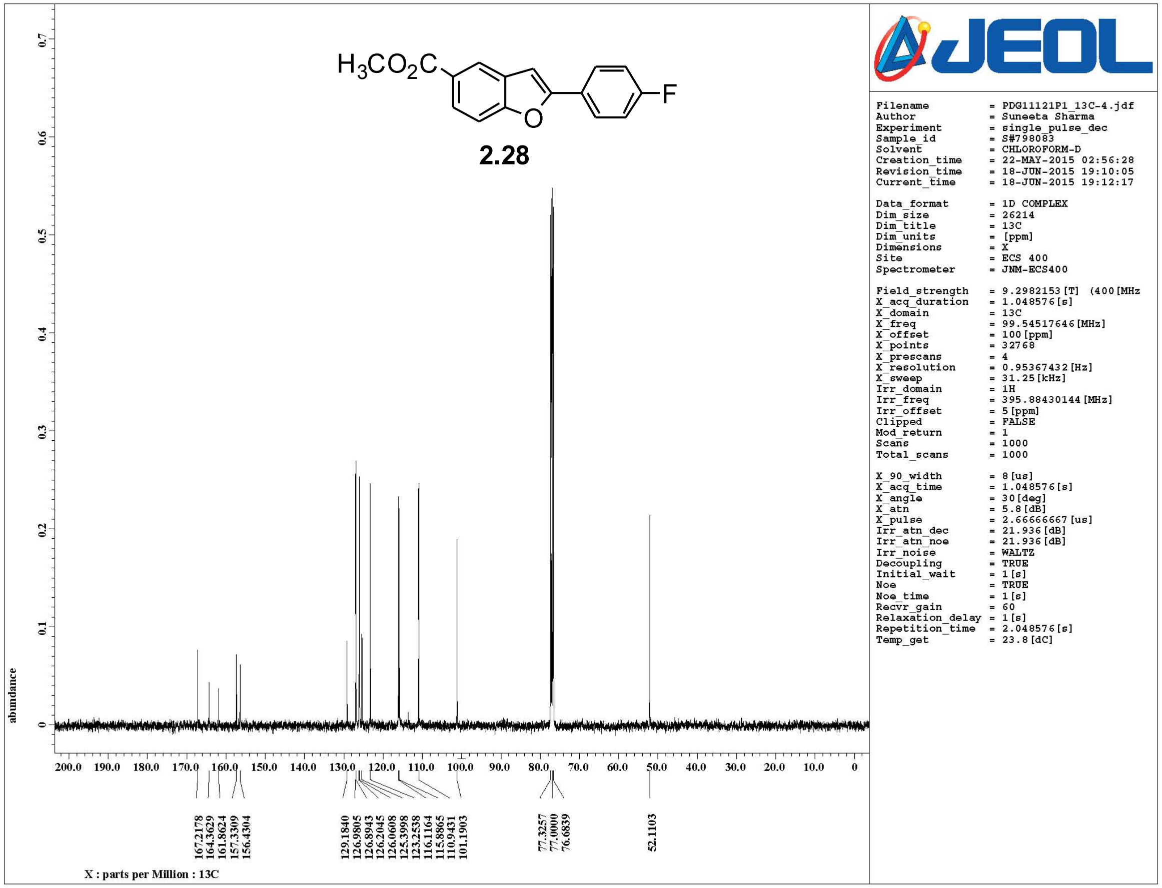


EI (HRMS) spectrum of **2.27**



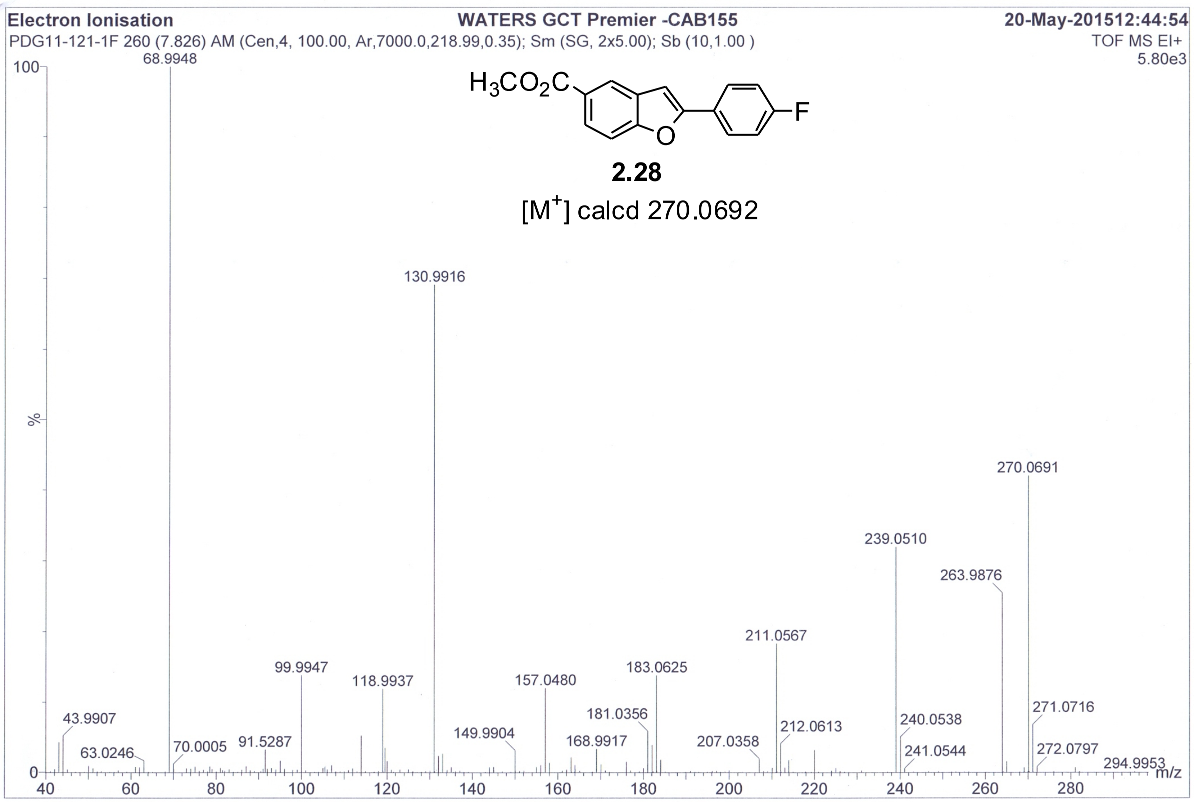
```
Filename          = PDG11121P1_1H-7.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#564401
Solvent           = CHLOROFORM-D
Creation_time     = 21-MAY-2015 19:06:41
Revision_time     = 17-JUN-2015 21:52:07
Current_time      = 17-JUN-2015 21:56:23

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 24.8[dC]
```

^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **2.28**

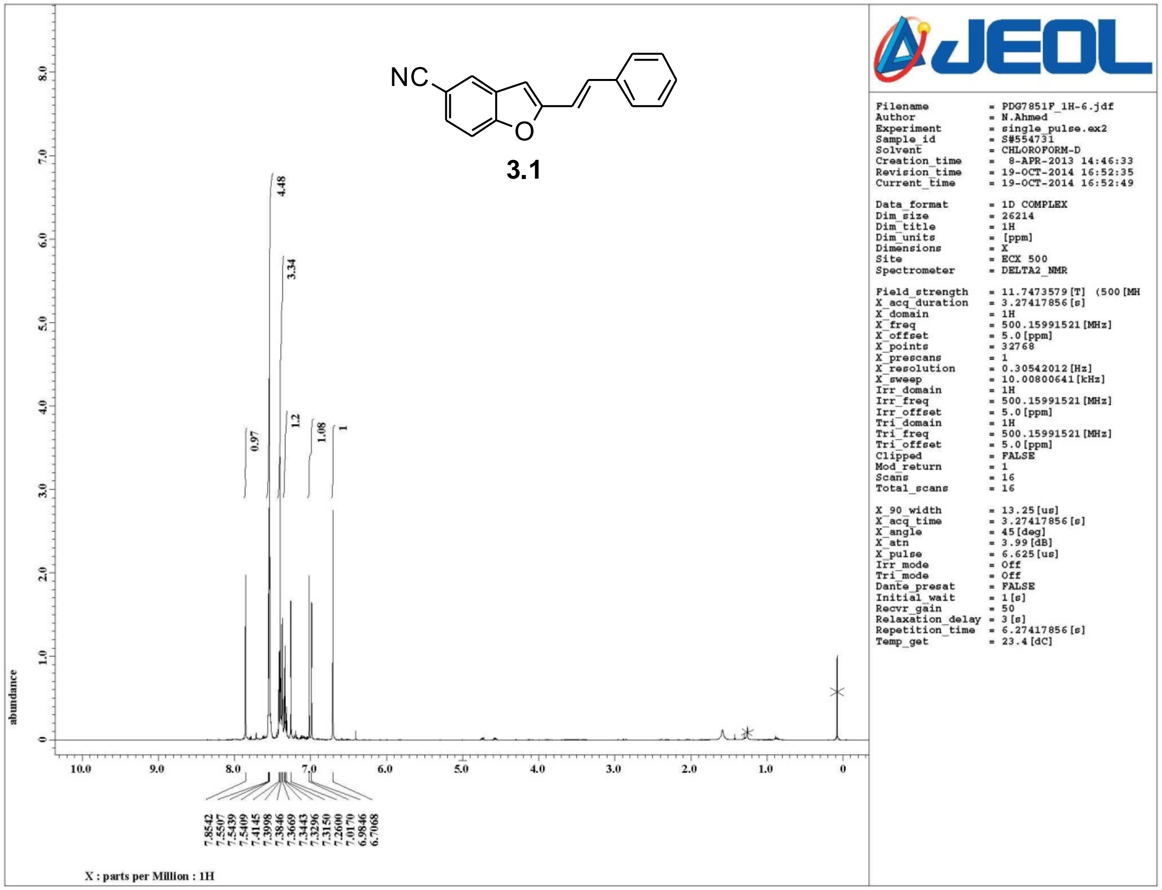


```
Filename          = PDG11121P1_13C-4.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#798083
Solvent           = CHLOROFORM-D
Creation_time     = 22-MAY-2015 02:56:28
Revision_time     = 18-JUN-2015 19:10:05
Current_time      = 18-JUN-2015 19:12:17

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.048576[s]
Temp_get          = 23.8[dC]
```

^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **2.28**

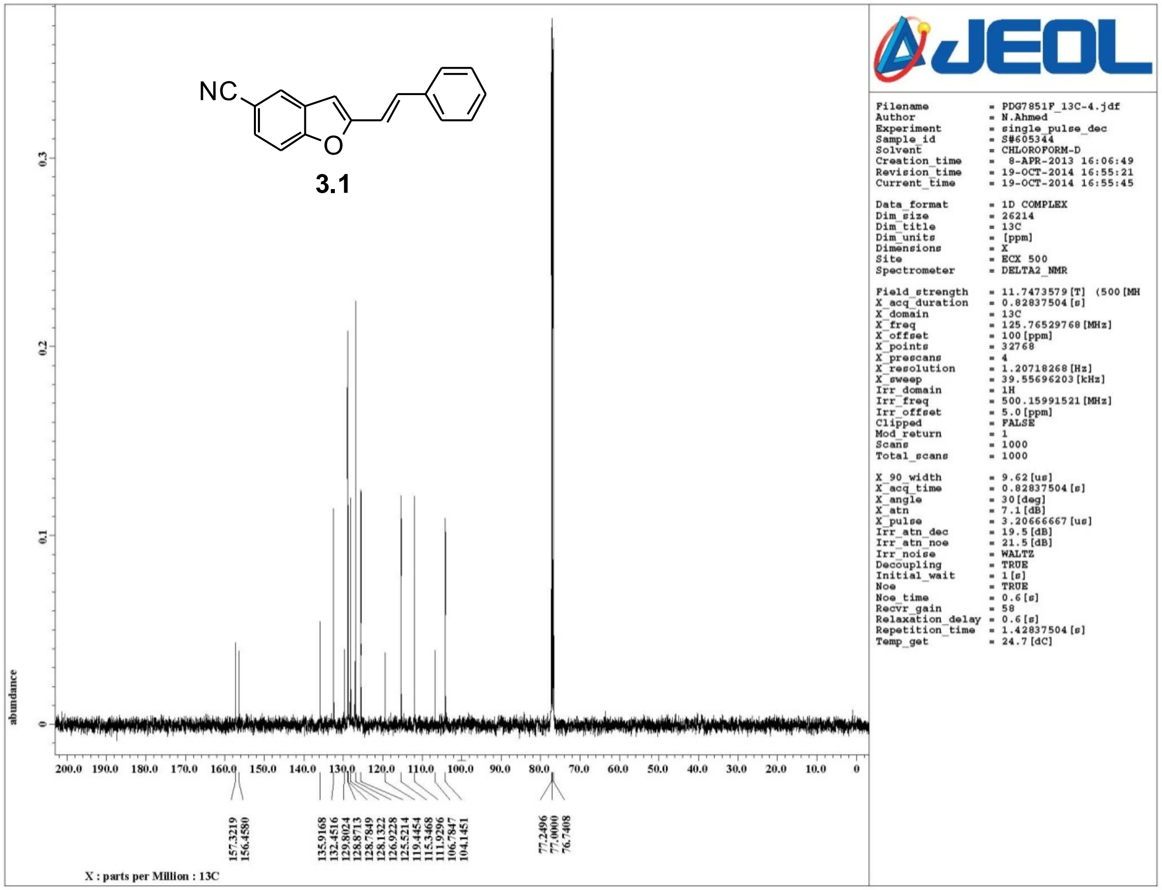


EI (HRMS) spectrum of **2.28**



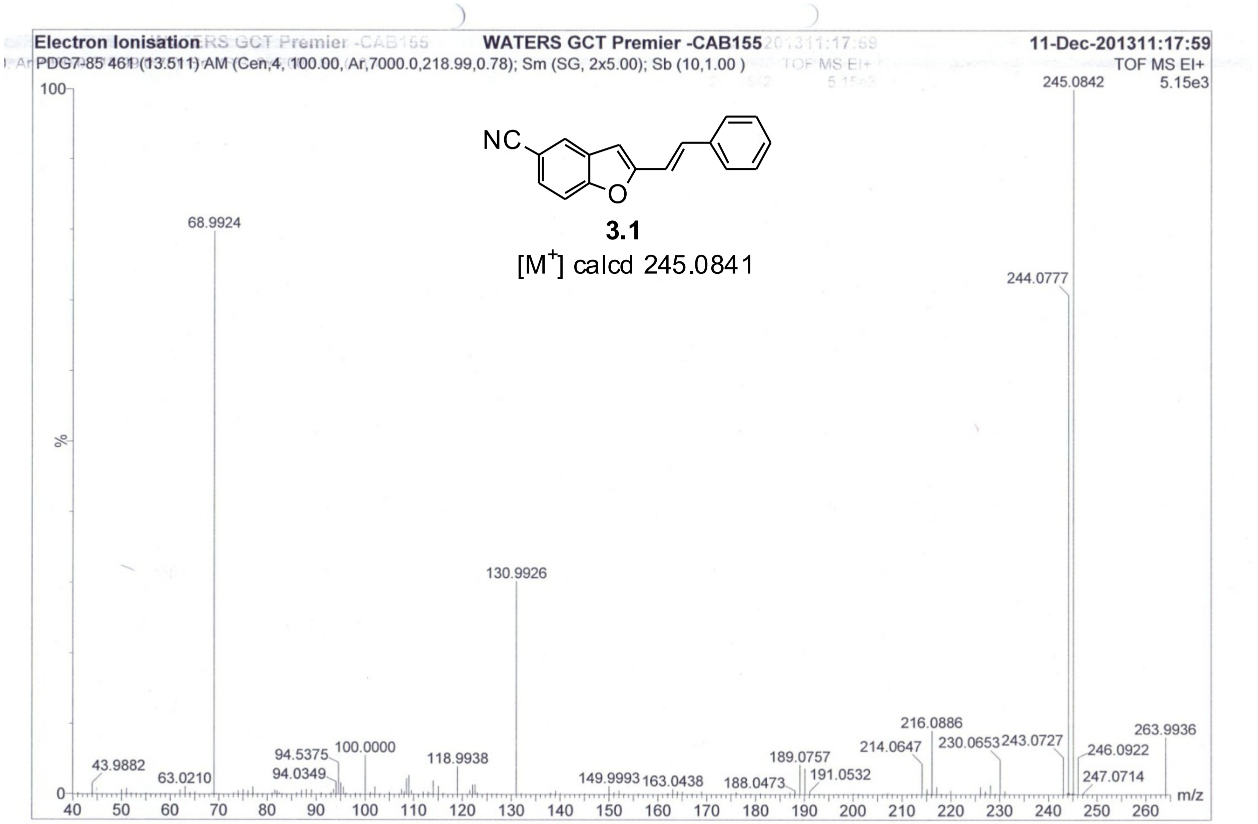
```
Filename            = PDG7851F_1H-6.jdf
Author              = N.Ahmed
Experiment          = single_pulse.ex2
Sample_id           = S#554731
Solvent             = CHLOROFORM-D
Creation_time       =  8-APR-2013 14:46:33
Revision_time       = 19-OCT-2014 16:52:35
Current_time        = 19-OCT-2014 16:52:49

Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECX 500
Spectrometer        = DELTA2_NMR

Field_strength      = 11.7473579[T] (500[MH
X_acq_duration      = 3.27417856[s]
X_domain            = 1H
X_freq              = 500.15991521[MHz]
X_offset            = 5.0[ppm]
X_points            = 32768
X_prescans          = 1
X_resolution        = 0.30542012[Hz]
X_sweep             = 10.00800641[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          = 13.25[us]
X_acq_time          = 3.27417856[s]
X_angle             = 45[deg]
X_atn               = 3.99[dB]
X_pulse             = 6.625[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 50
Relaxation_delay    = 3[s]
Repetition_time     = 6.27417856[s]
Temp_get            = 23.4[dC]
```

^{1}H NMR (500 MHz, $CDCl_3$) spectrum of **3.1**



```
Filename         = PDG7851F_13C-4.jdf
Author           = N.Ahmed
Experiment       = single_pulse_dec
Sample_id        = S#605344
Solvent          = CHLOROFORM-D
Creation_time    =  8-APR-2013 16:06:49
Revision_time    = 19-OCT-2014 16:55:21
Current_time     = 19-OCT-2014 16:55:45

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

Field_strength   = 11.7473579[T] (500[MH
X_acq_duration   = 0.82837504[s]
X_domain         = 13C
X_freq           = 125.76529768[MHz]
X_offset         = 100[ppm]
X_points         = 32768
X_prescans       = 4
X_resolution     = 1.20718268[Hz]
X_sweep          = 39.55696203[kHz]
Irr_domain       = 1H
Irr_freq         = 500.15991521[MHz]
Irr_offset       = 5.0[ppm]
Clipped          = FALSE
Mod_return       = 1
Scans            = 1000
Total_scans      = 1000

X_90_width       = 9.62[us]
X_acq_time       = 0.82837504[s]
X_angle          = 30[deg]
X_atn            = 7.1[dB]
X_pulse          = 3.20666667[us]
Irr_atn_dec      = 19.5[dB]
Irr_atn_noe      = 21.5[dB]
Irr_noise        = WALTZ
Decoupling       = TRUE
Initial_wait     = 1[s]
Noe              = TRUE
Noe_time         = 0.6[s]
Recvr_gain       = 58
Relaxation_delay = 0.6[s]
Repetition_time  = 1.42837504[s]
Temp_get         = 24.7[dC]
```

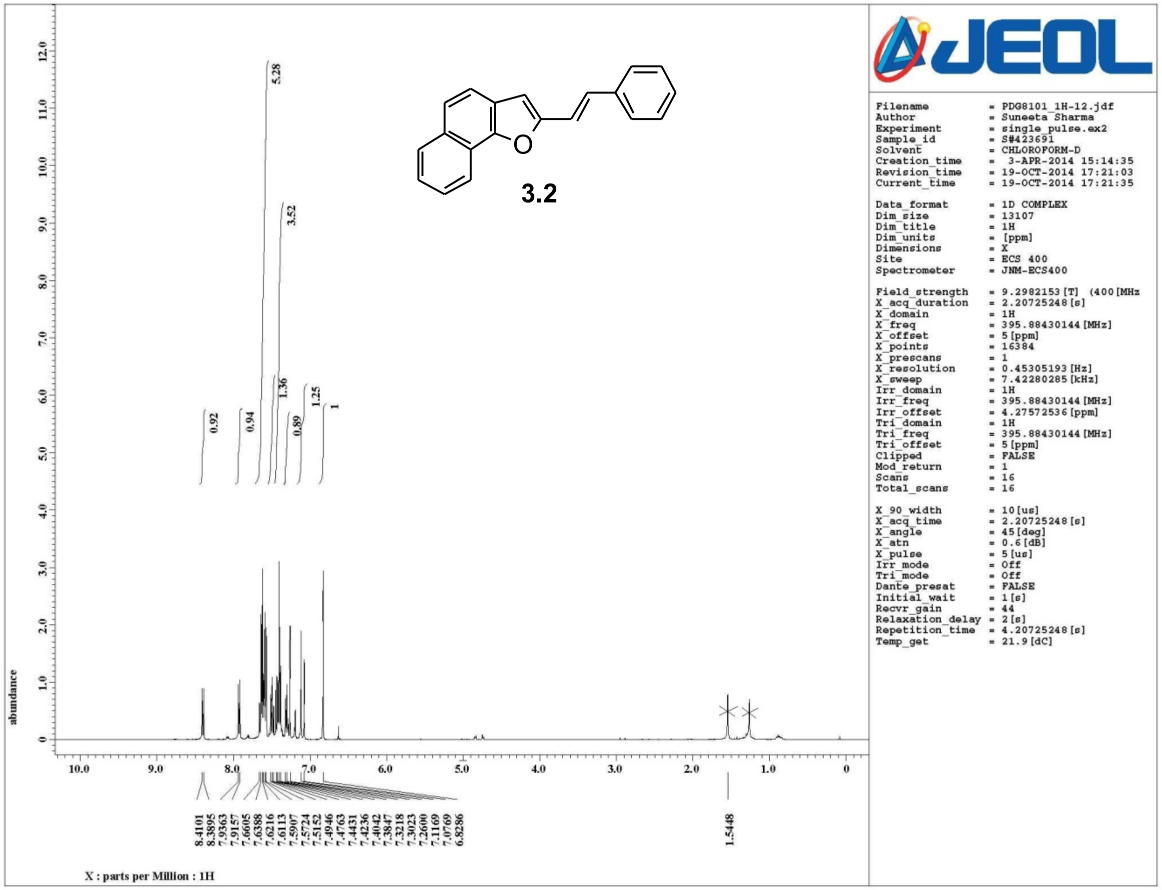
^{13}C NMR (125 MHz, $CDCl_3$) spectrum of **3.1**



3.1

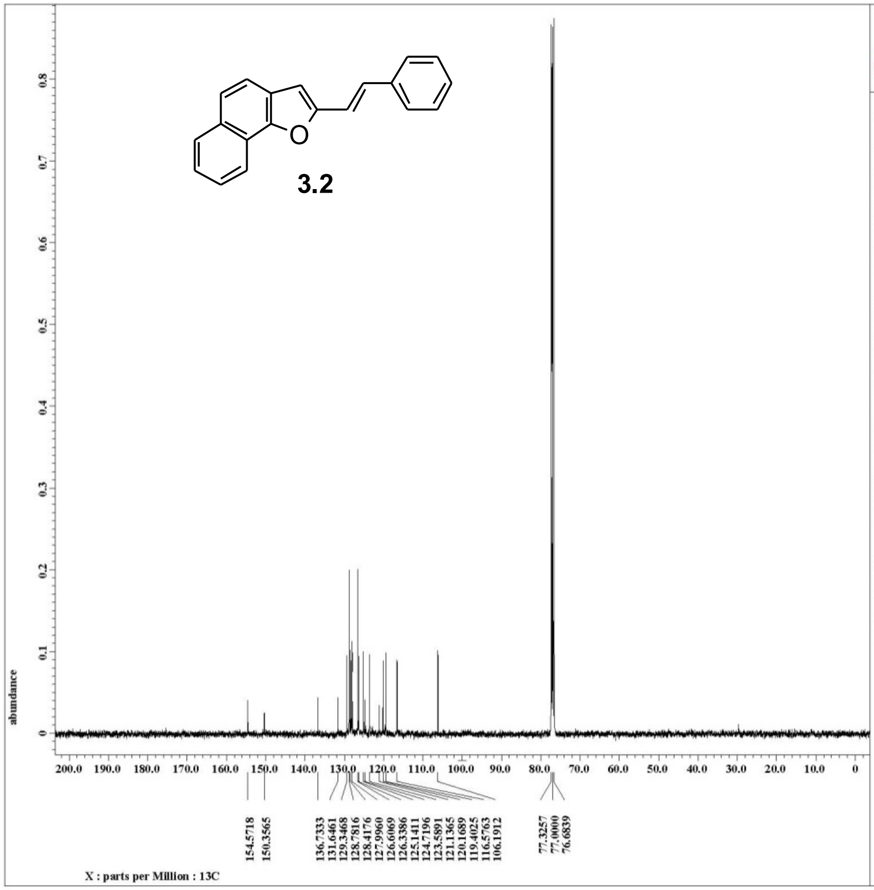
[M$^+$] calcd 245.0841

EI (HRMS) spectrum of **3.1**



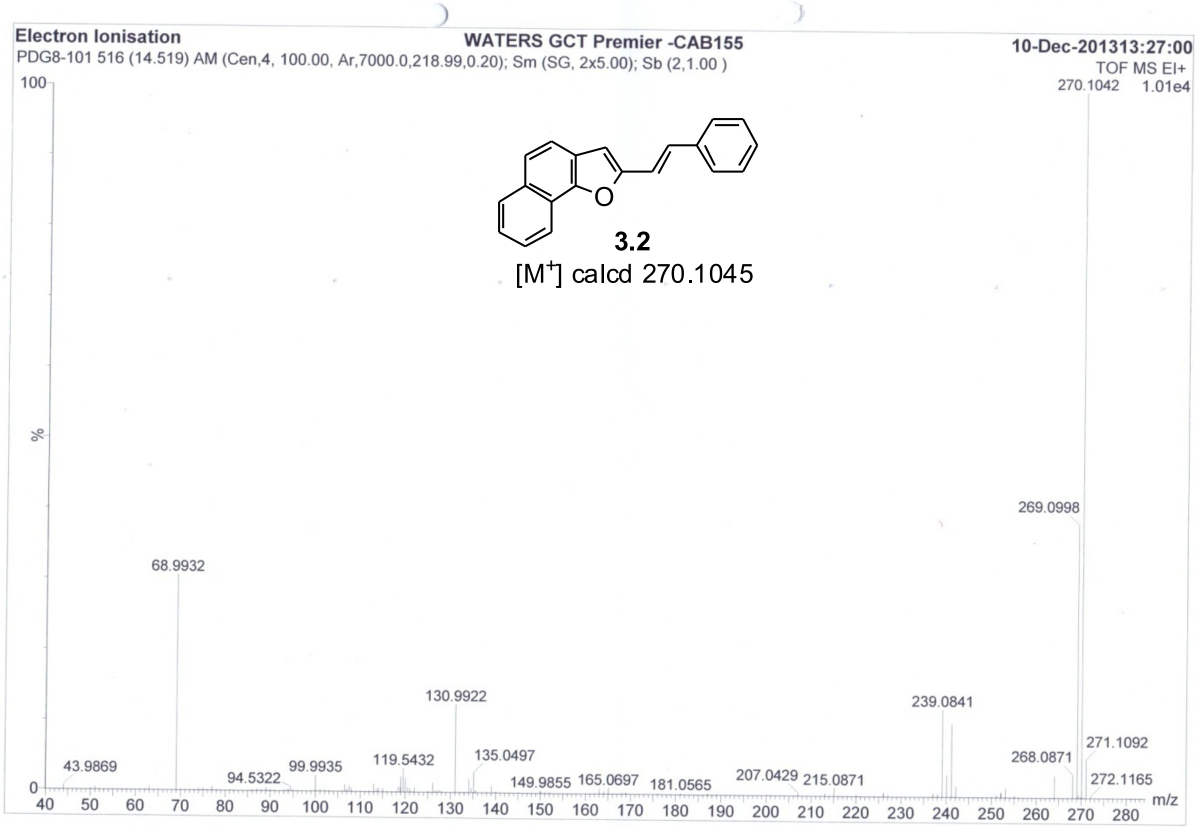
```
Filename          = PDG8101_1H-12.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#423691
Solvent           = CHLOROFORM-D
Creation_time     =  3-APR-2014 15:14:35
Revision_time     = 19-OCT-2014 17:21:03
Current_time      = 19-OCT-2014 17:21:35

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 4.27572536[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 21.9[dC]
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **3.2**

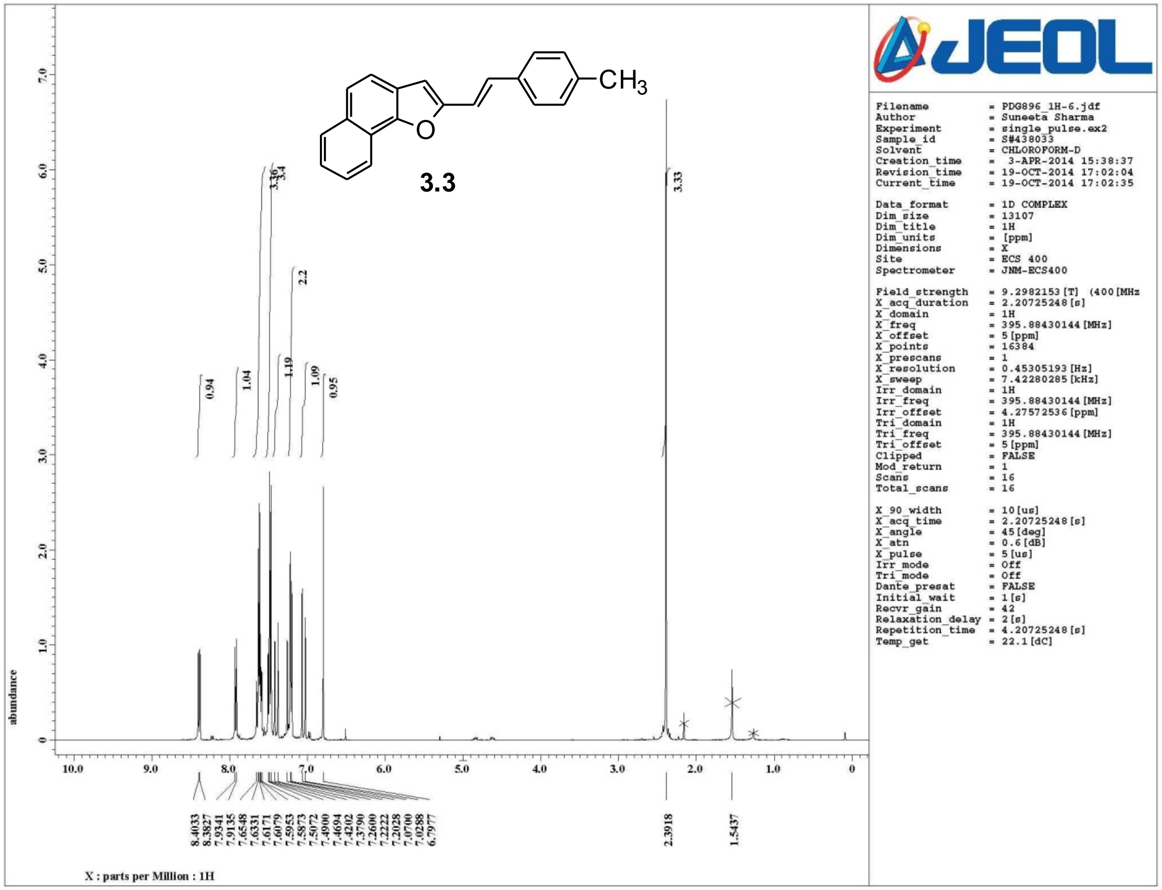


```
Filename          = PDG8101_13C-4.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#590623
Solvent           = CHLOROFORM-D
Creation_time     =  3-APR-2014 23:49:46
Revision_time     = 19-OCT-2014 17:24:09
Current_time      = 19-OCT-2014 17:24:46

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1500
Total_scans       = 1500

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.048576[s]
Temp_get          = 21.7[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **3.2**

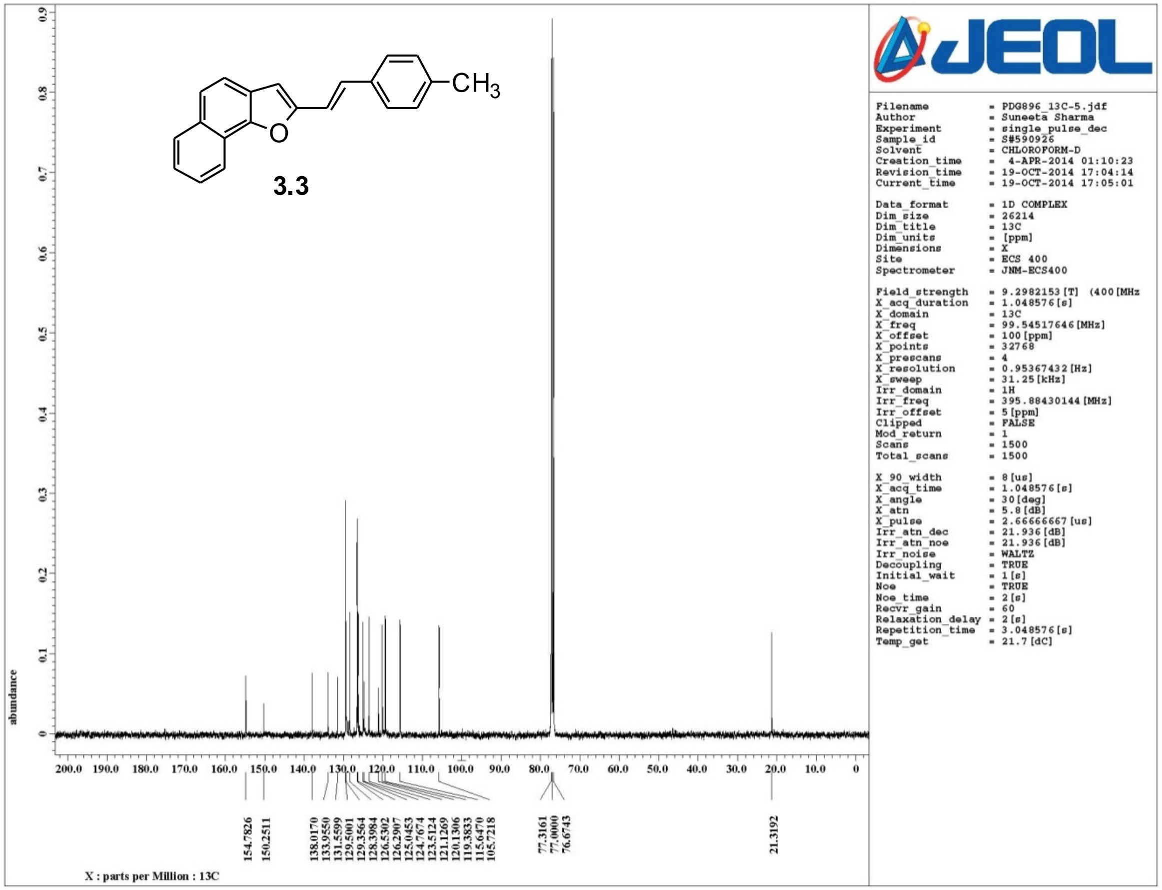


EI (HRMS) spectrum of **3.2**



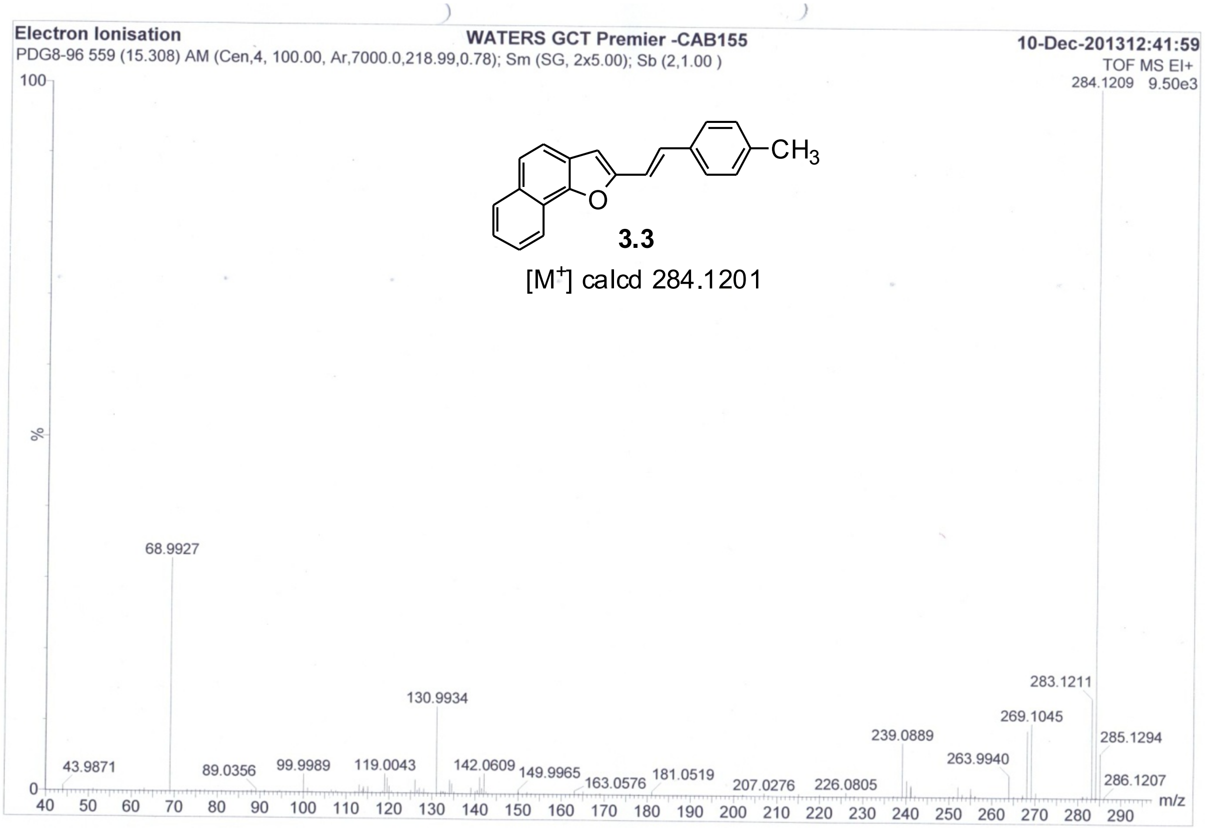
```
Filename          = PDG896_1H-6.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#438033
Solvent           = CHLOROFORM-D
Creation_time     =  3-APR-2014 15:38:37
Revision_time     = 19-OCT-2014 17:02:04
Current_time      = 19-OCT-2014 17:02:35

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T]  (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 4.27572536[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 22.1[dC]
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **3.3**

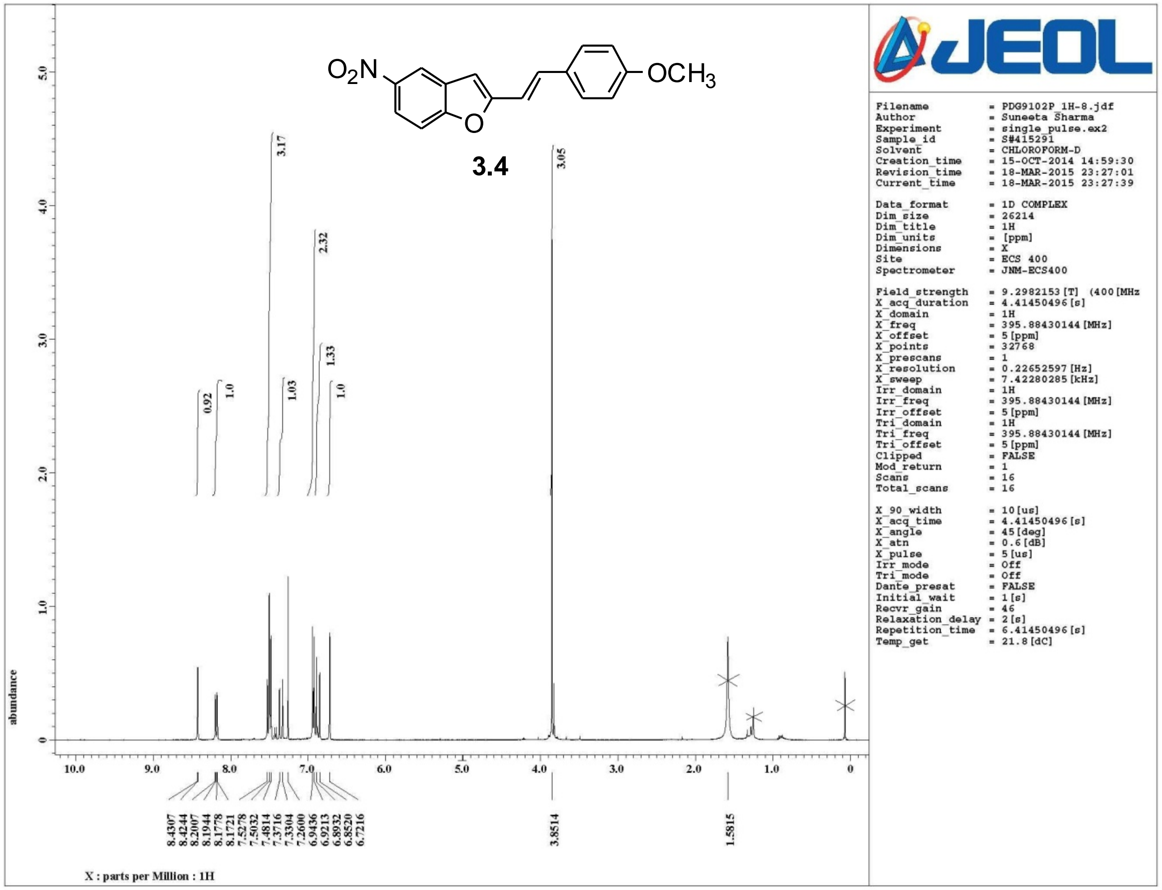


```
Filename           = PDG896_13C-5.jdf
Author             = Suneeta Sharma
Experiment         = single_pulse_dec
Sample_id          = S#590926
Solvent            = CHLOROFORM-D
Creation_time      =  4-APR-2014 01:10:23
Revision_time      = 19-OCT-2014 17:04:14
Current_time       = 19-OCT-2014 17:05:01

Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.2982153[T] (400[MHz
X_acq_duration     = 1.048576[s]
X_domain           = 13C
X_freq             = 99.54517646[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 0.95367432[Hz]
X_sweep            = 31.25[kHz]
Irr_domain         = 1H
Irr_freq           = 395.88430144[MHz]
Irr_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 1500
Total_scans        = 1500

X_90_width         = 8[us]
X_acq_time         = 1.048576[s]
X_angle            = 30[deg]
X_atn              = 5.8[dB]
X_pulse            = 2.66666667[us]
Irr_atn_dec        = 21.936[dB]
Irr_atn_noe        = 21.936[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 60
Relaxation_delay   = 2[s]
Repetition_time    = 3.048576[s]
Temp_get           = 21.7[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **3.3**

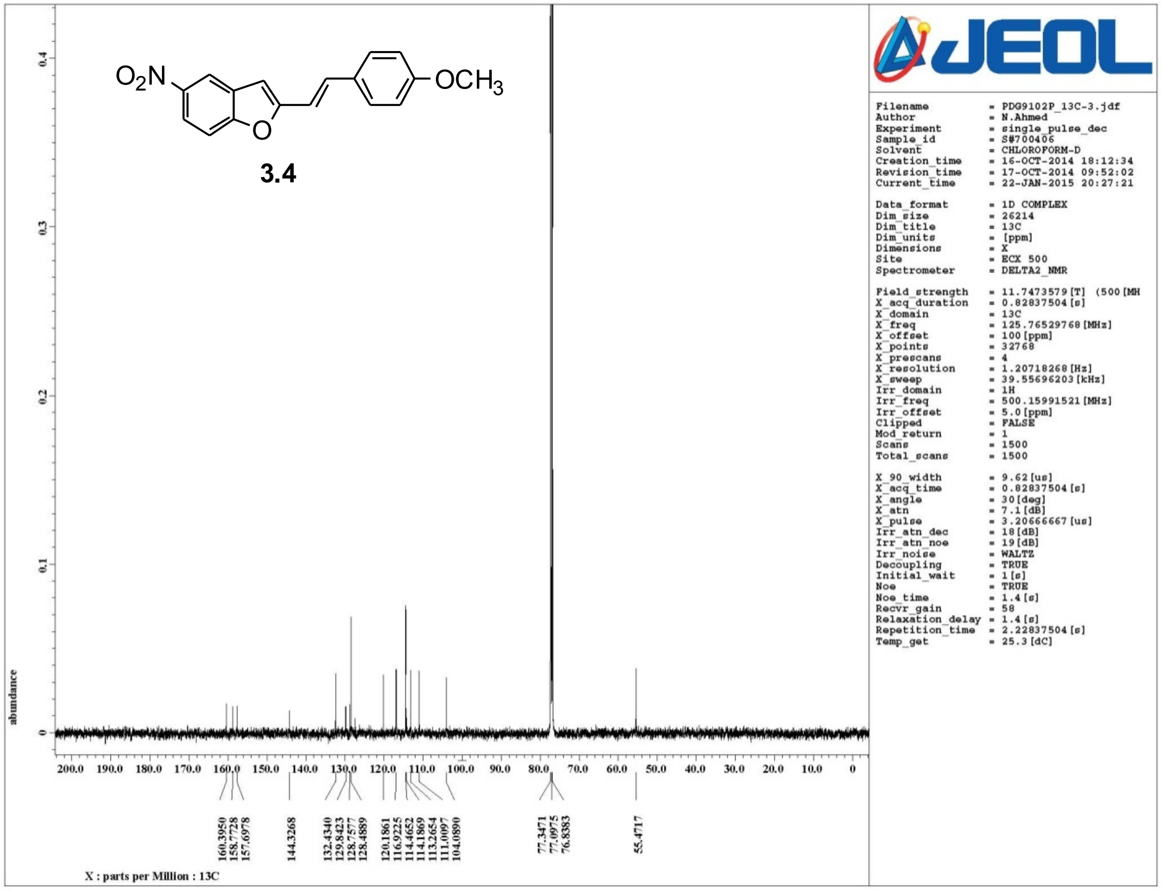


EI (HRMS) spectrum of **3.3**



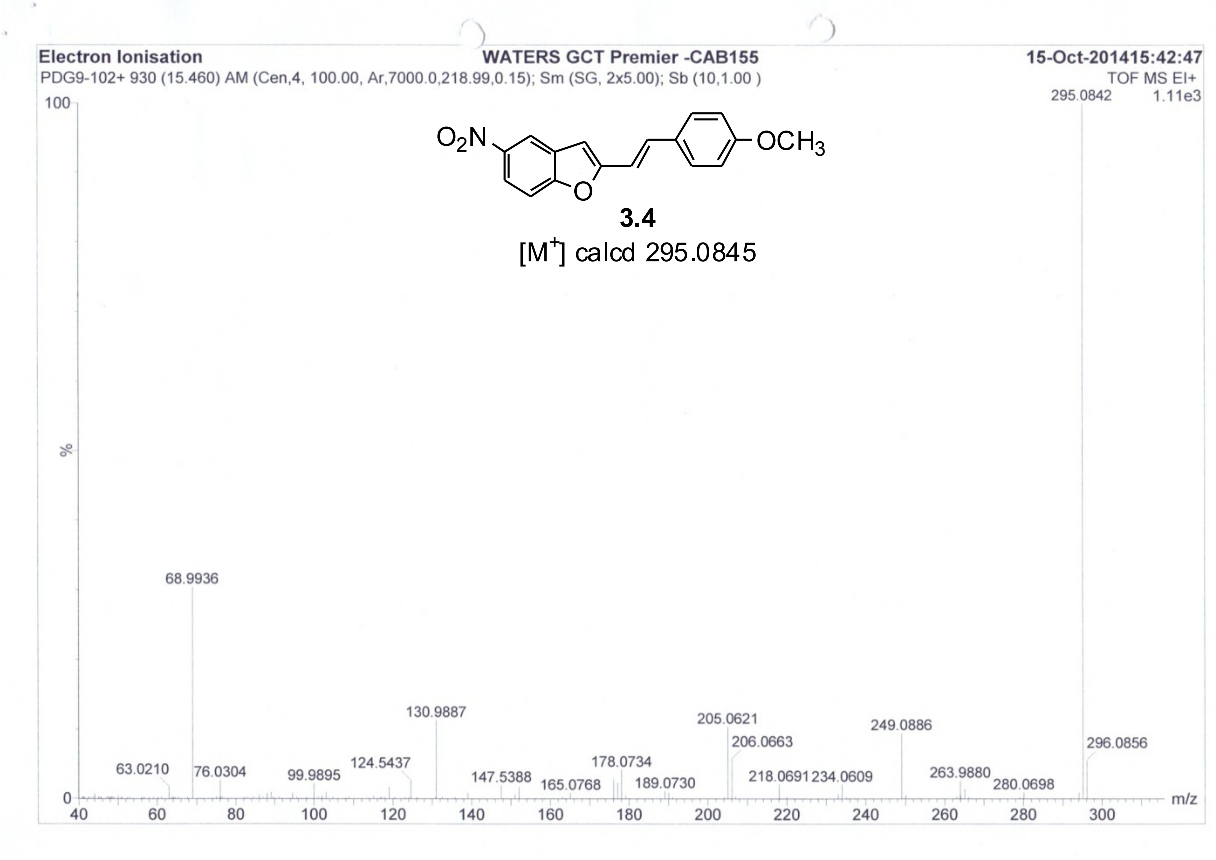
```
Filename            = PDG9102P_1H-8.jdf
Author              = Suneeta Sharma
Experiment          = single_pulse.ex2
Sample_id           = S#415291
Solvent             = CHLOROFORM-D
Creation_time       = 15-OCT-2014 14:59:30
Revision_time       = 18-MAR-2015 23:27:01
Current_time        = 18-MAR-2015 23:27:39

Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = JNM-ECS400

Field_strength      = 9.2982153[T] (400[MHz
X_acq_duration      = 4.41450496[s]
X_domain            = 1H
X_freq              = 395.88430144[MHz]
X_offset            = 5[ppm]
X_points            = 32768
X_prescans          = 1
X_resolution        = 0.22652597[Hz]
X_sweep             = 7.42280285[kHz]
Irr_domain          = 1H
Irr_freq            = 395.88430144[MHz]
Irr_offset          = 5[ppm]
Tri_domain          = 1H
Tri_freq            = 395.88430144[MHz]
Tri_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 16
Total_scans         = 16

X_90_width          = 10[us]
X_acq_time          = 4.41450496[s]
X_angle             = 45[deg]
X_atn               = 0.6[dB]
X_pulse             = 5[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 46
Relaxation_delay    = 2[s]
Repetition_time     = 6.41450496[s]
Temp_get            = 21.8[dC]
```

^1H NMR (400 MHz, $CDCl_3$) spectrum of **3.4**



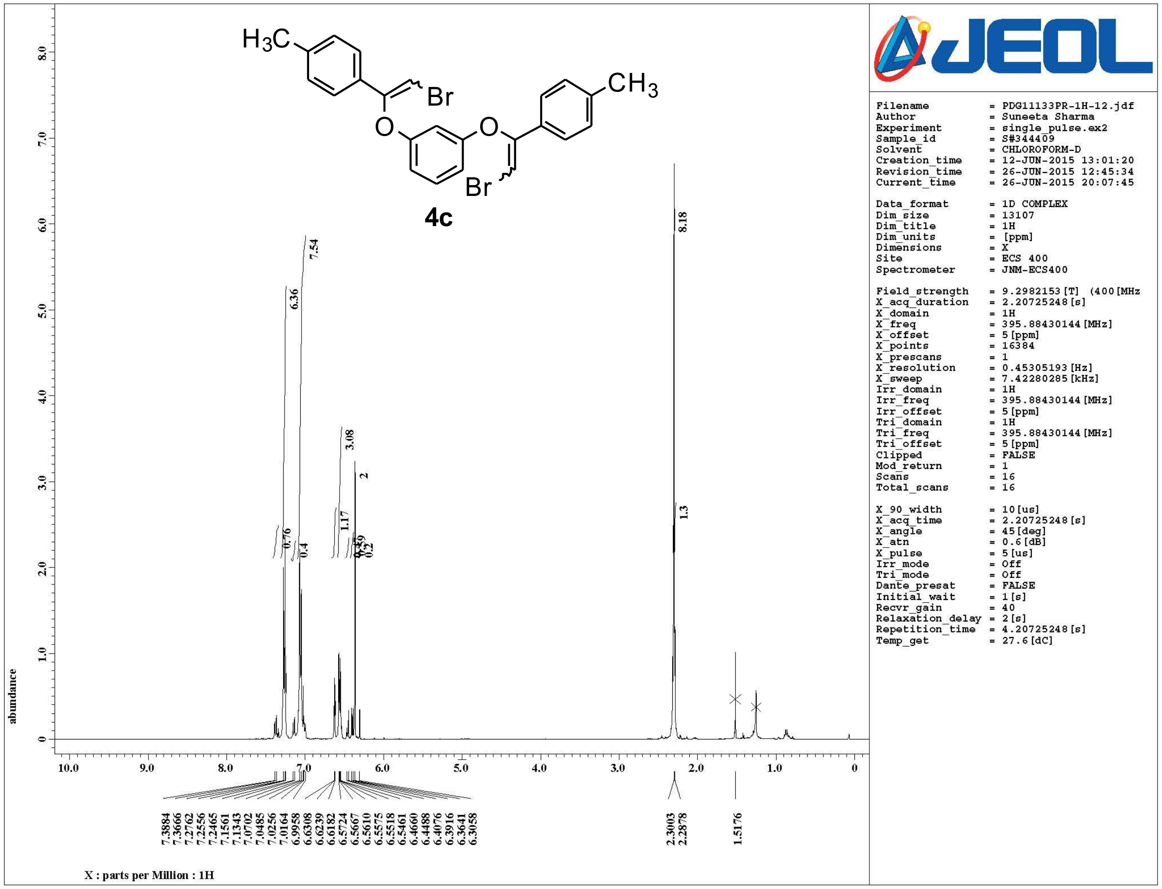
```
Filename          = PDG9102P_13C-3.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#700406
Solvent           = CHLOROFORM-D
Creation_time     = 16-OCT-2014 18:12:34
Revision_time     = 17-OCT-2014 09:52:02
Current_time      = 22-JAN-2015 20:27:21

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

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.82837504[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.20718268[Hz]
X_sweep           = 39.55696203[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1500
Total_scans       = 1500

X_90_width        = 9.62[us]
X_acq_time        = 0.82837504[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 18[dB]
Irr_atn_noe       = 19[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1.4[s]
Recvr_gain        = 58
Relaxation_delay  = 1.4[s]
Repetition_time   = 2.22837504[s]
Temp_get          = 25.3[dC]
```

^{13}C NMR (125 MHz, CDCl$_3$) spectrum of **3.4**



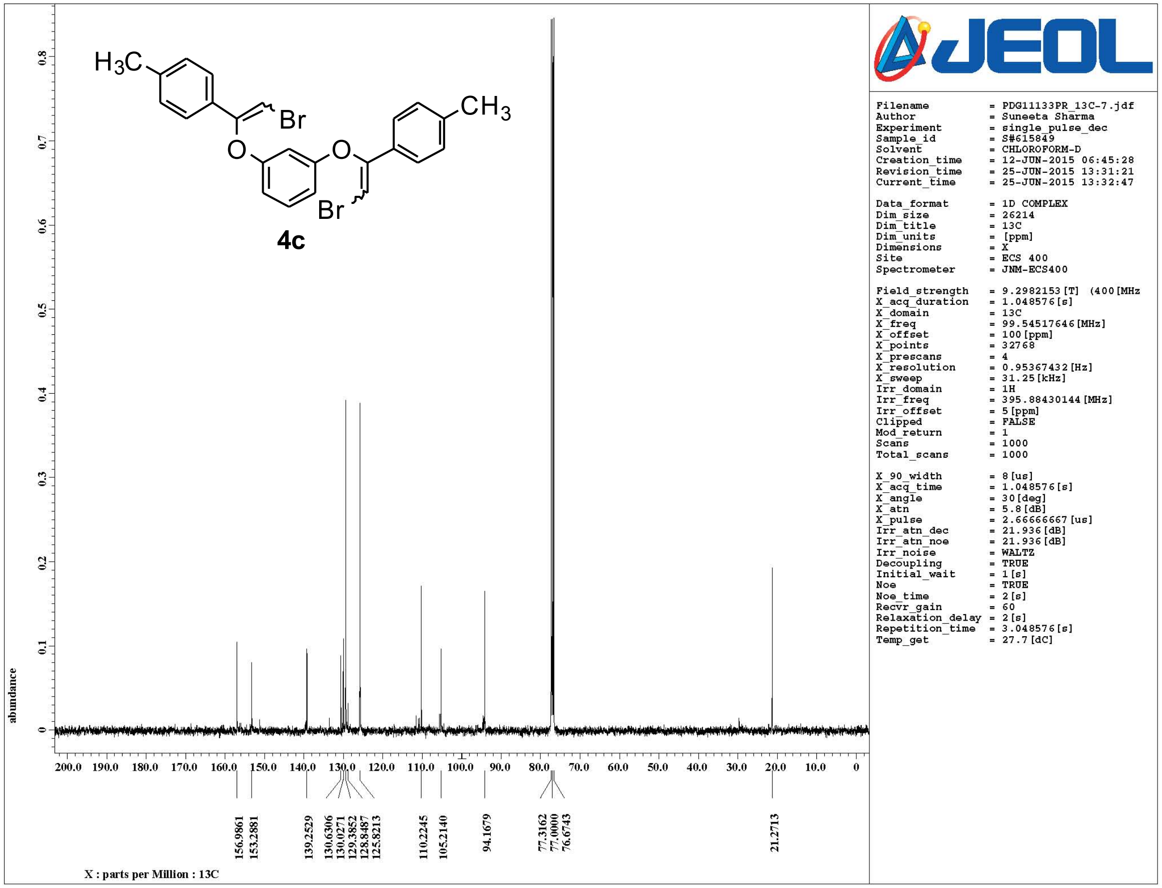
EI (HRMS) spectrum of **3.4**

8. ^{1}H ,^{13}C and HRMS spectra of intermediates 4c and 5c:



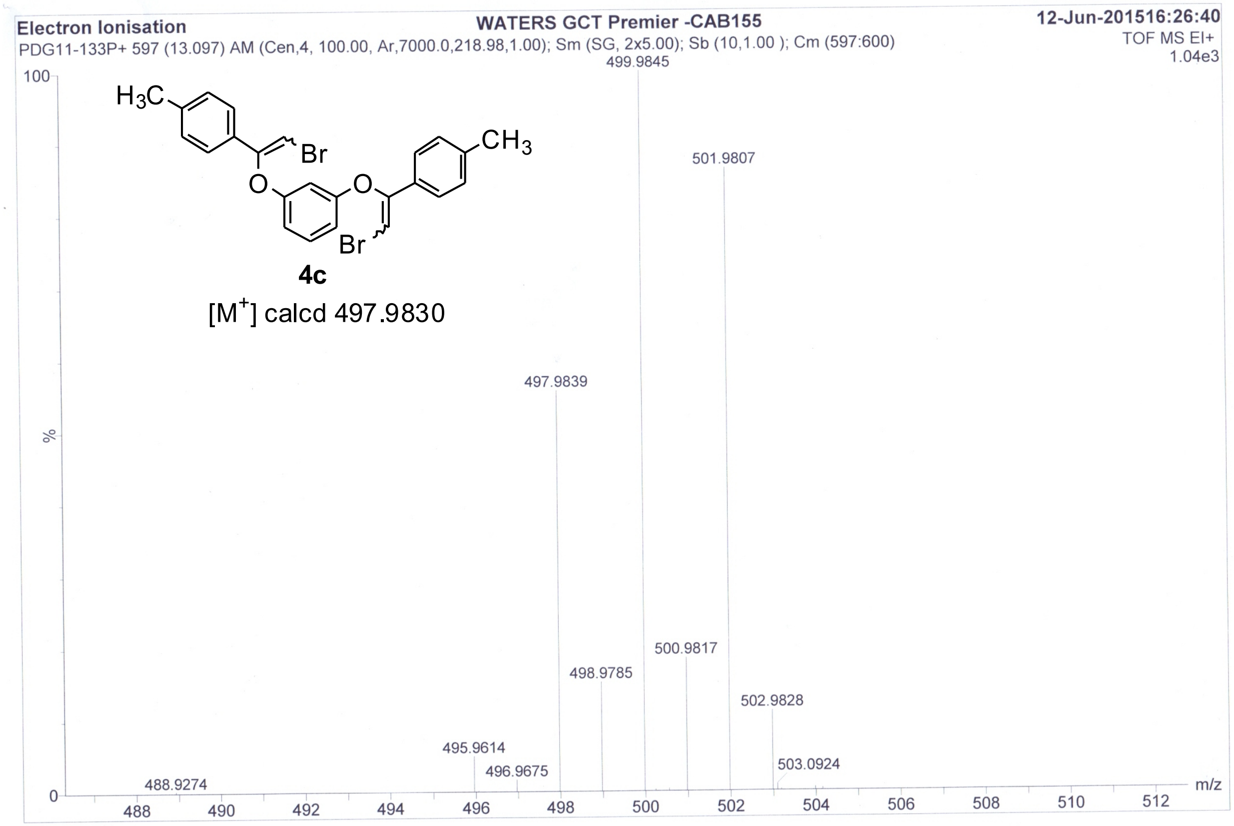
```
Filename           = PDG11133PR-1H-12.jdf
Author             = Suneeta Sharma
Experiment         = single_pulse.ex2
Sample_id          = S#344409
Solvent            = CHLOROFORM-D
Creation_time      = 12-JUN-2015 13:01:20
Revision_time      = 26-JUN-2015 12:45:34
Current_time       = 26-JUN-2015 20:07:45

Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.2982153[T] (400[MHz
X_acq_duration     = 2.20725248[s]
X_domain           = 1H
X_freq             = 395.88430144[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 1
X_resolution       = 0.45305193[Hz]
X_sweep            = 7.42280285[kHz]
Irr_domain         = 1H
Irr_freq           = 395.88430144[MHz]
Irr_offset         = 5[ppm]
Tri_domain         = 1H
Tri_freq           = 395.88430144[MHz]
Tri_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 16
Total_scans        = 16

X_90_width         = 10[us]
X_acq_time         = 2.20725248[s]
X_angle            = 45[deg]
X_atn              = 0.6[dB]
X_pulse            = 5[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 40
Relaxation_delay   = 2[s]
Repetition_time    = 4.20725248[s]
Temp_get           = 27.6[dC]
```

^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **4c**

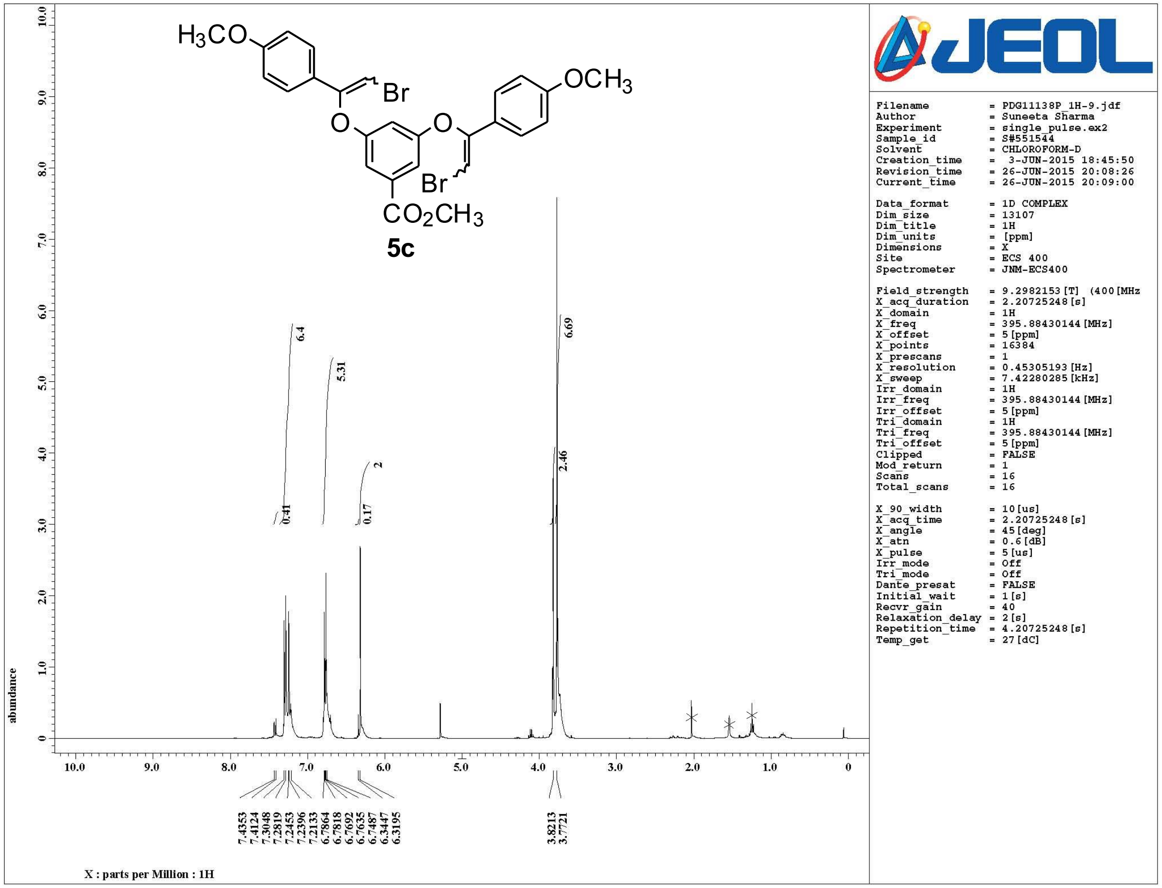


```
Filename          = PDG11133PR_13C-7.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#615849
Solvent           = CHLOROFORM-D
Creation_time     = 12-JUN-2015 06:45:28
Revision_time     = 25-JUN-2015 13:31:21
Current_time      = 25-JUN-2015 13:32:47

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.048576[s]
Temp_get          = 27.7[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **4c**

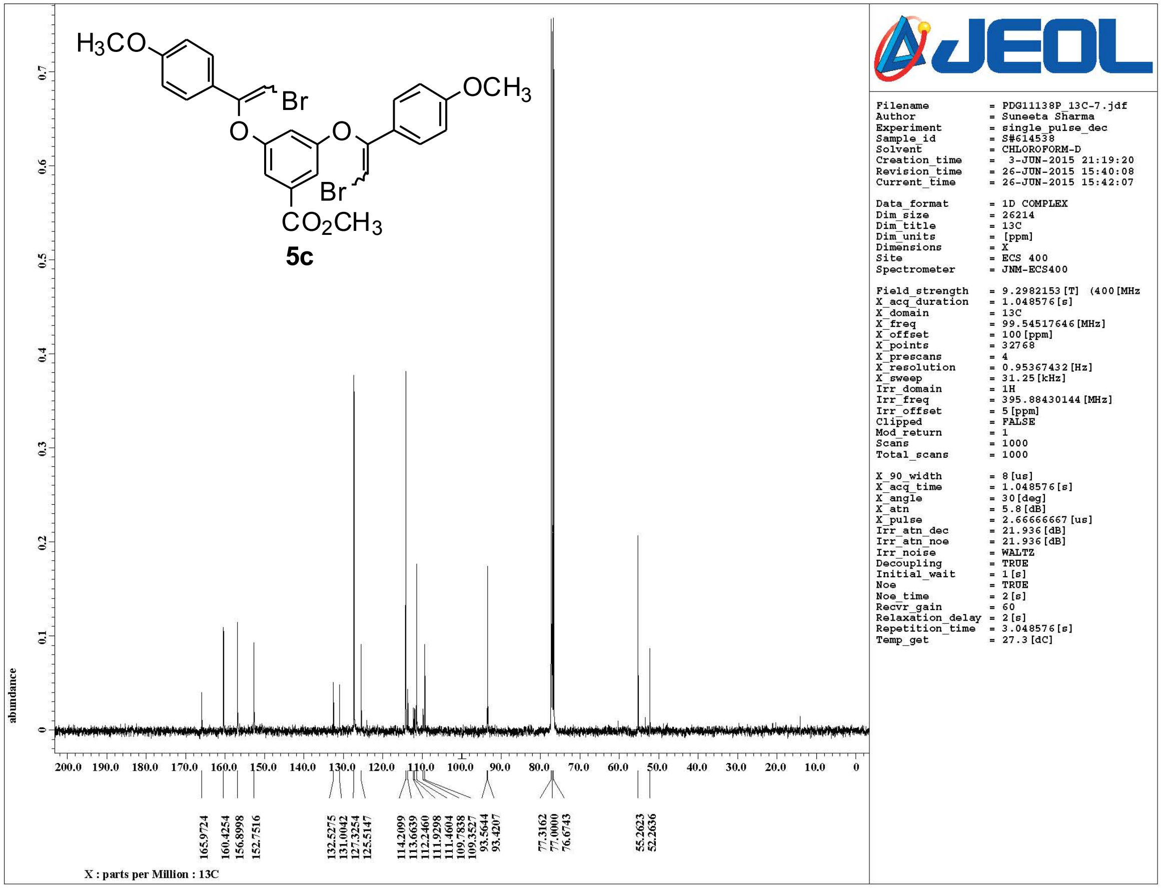


EI (HRMS) spectrum of **4c**



```
Filename          = PDG11138P_1H-9.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#551544
Solvent           = CHLOROFORM-D
Creation_time     =  3-JUN-2015 18:45:50
Revision_time     = 26-JUN-2015 20:08:26
Current_time      = 26-JUN-2015 20:09:00

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 27[dC]
```

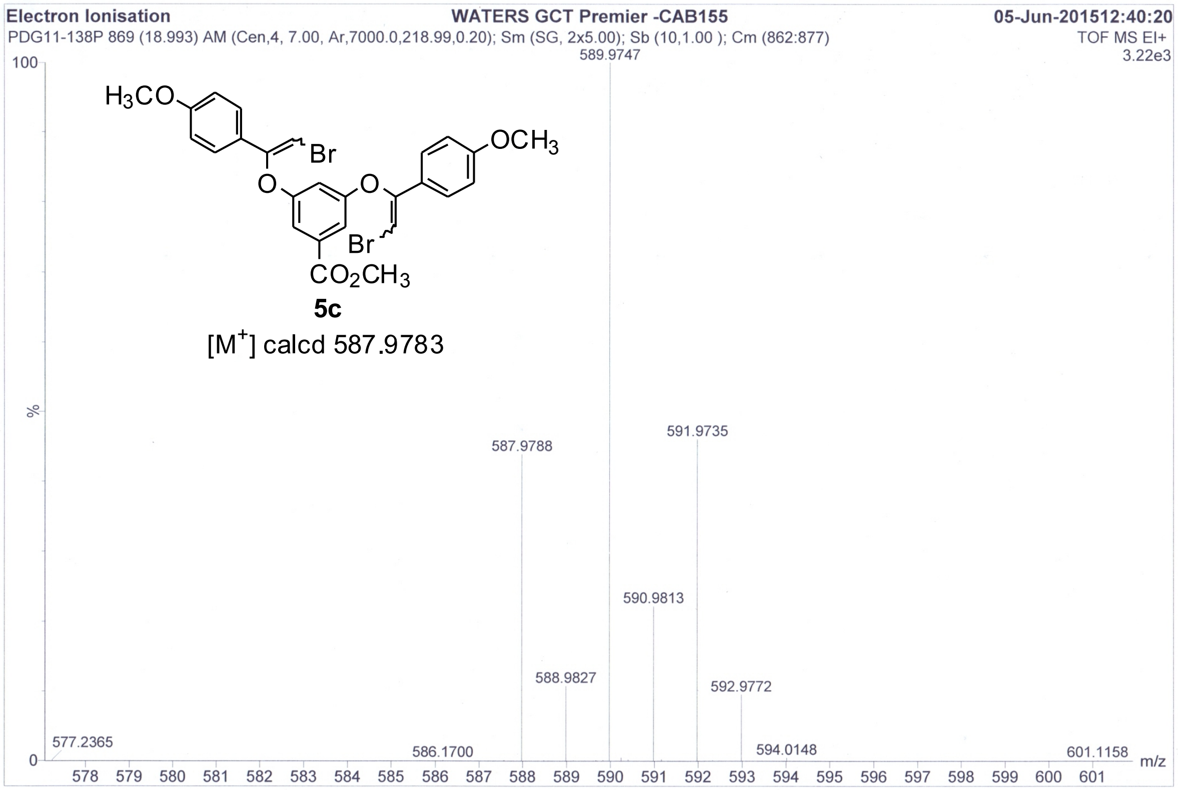
^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **5c**



5c

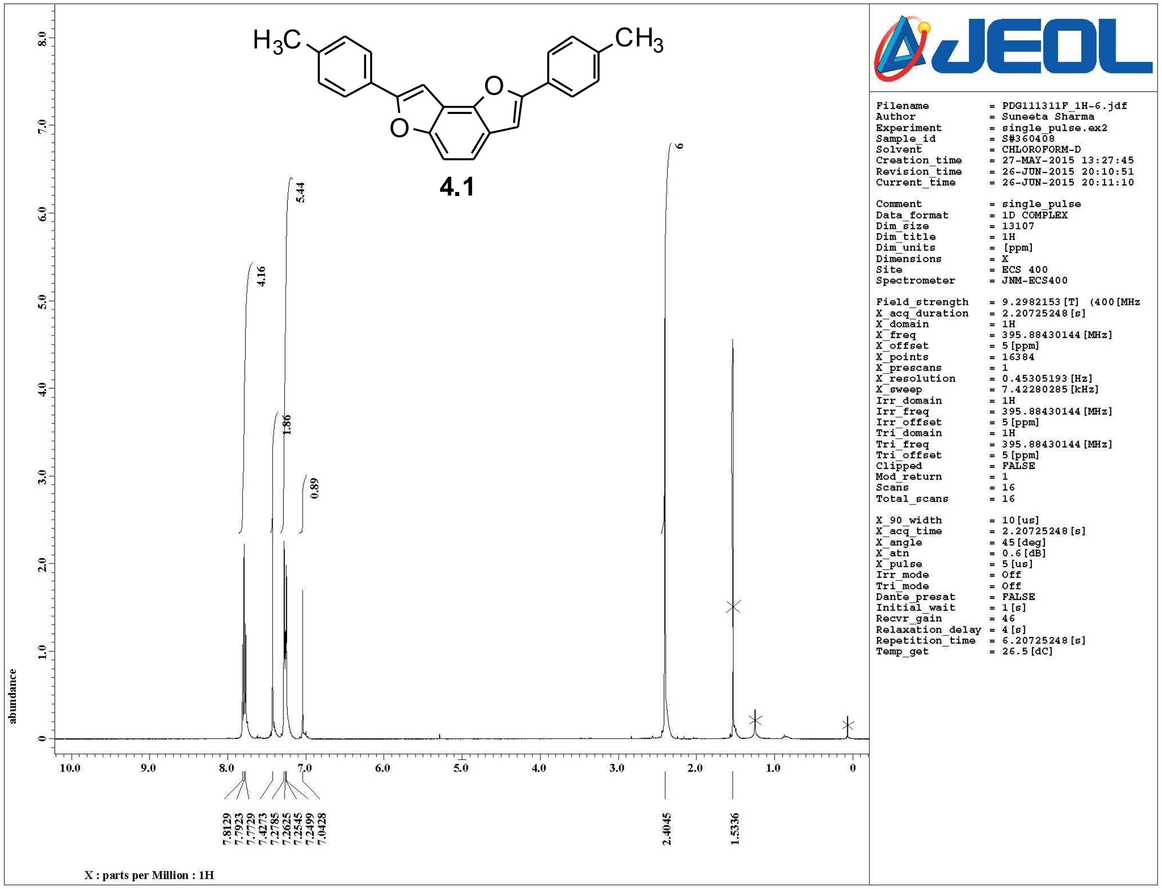
```
Filename          = PDG11138P_13C-7.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#614538
Solvent           = CHLOROFORM-D
Creation_time     =  3-JUN-2015 21:19:20
Revision_time     = 26-JUN-2015 15:40:08
Current_time      = 26-JUN-2015 15:42:07

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.048576[s]
Temp_get          = 27.3[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **5c**



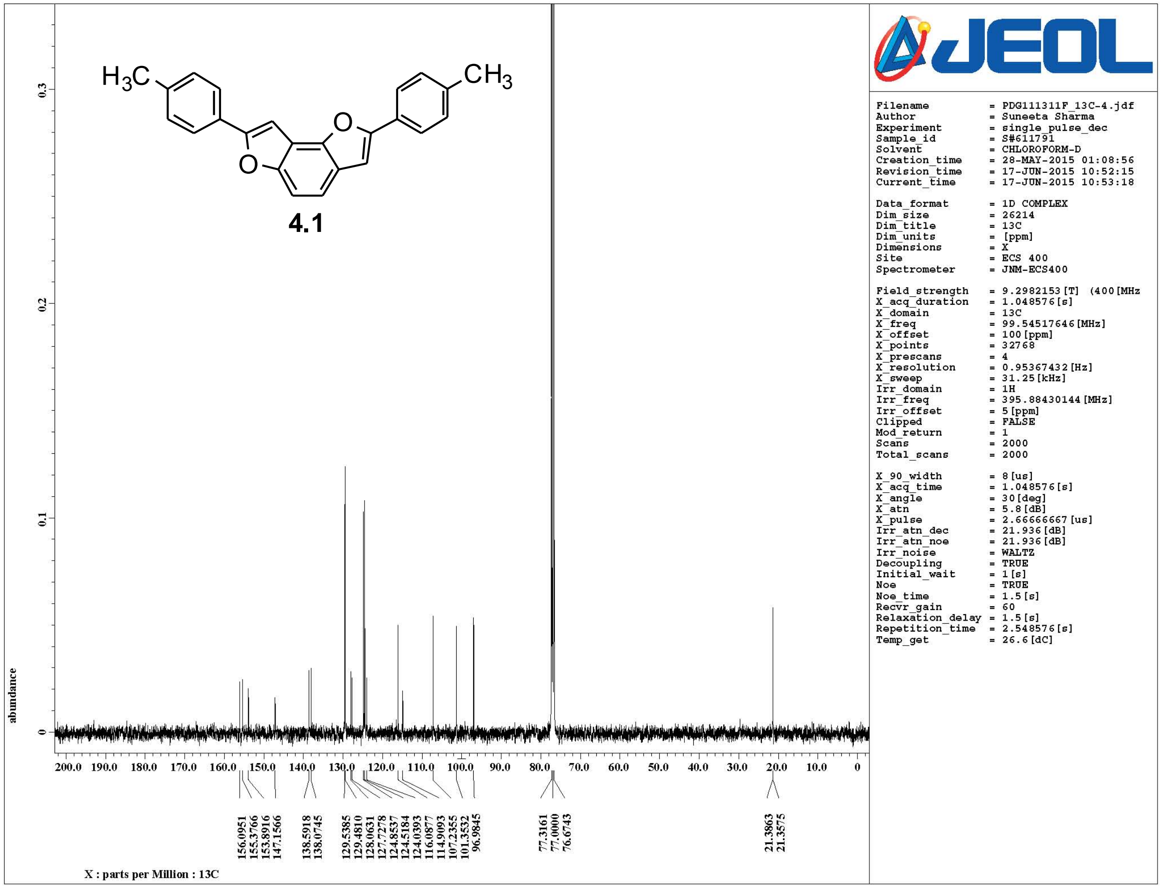
EI (HRMS) spectrum of **5c**

9. ^{1}H ,^{13}C and HRMS spectra of benzodifurans 4.1 and 5.1:



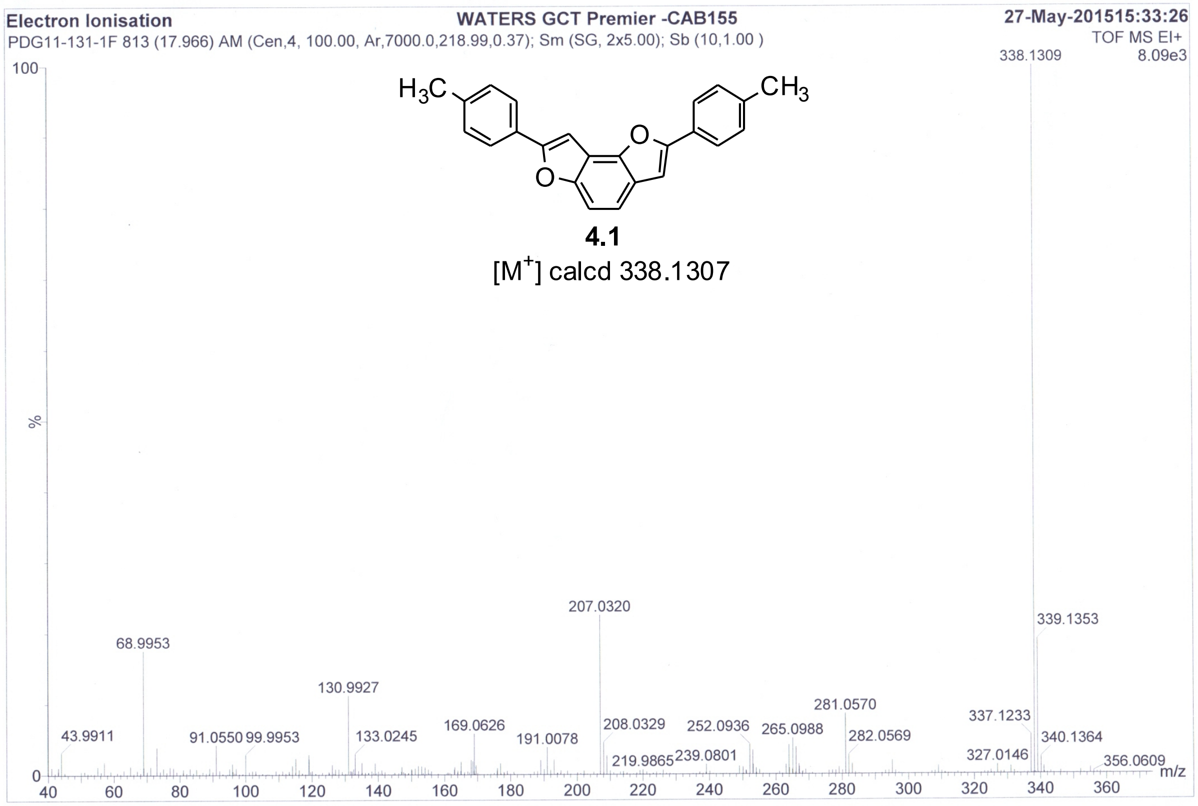
```
Filename          = PDG111311F_1H-6.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#360408
Solvent           = CHLOROFORM-D
Creation_time     = 27-MAY-2015 13:27:45
Revision_time     = 26-JUN-2015 20:10:51
Current_time      = 26-JUN-2015 20:11:10

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T]  (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 4[s]
Repetition_time   = 6.20725248[s]
Temp_get          = 26.5[dC]
```

^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **4.1**

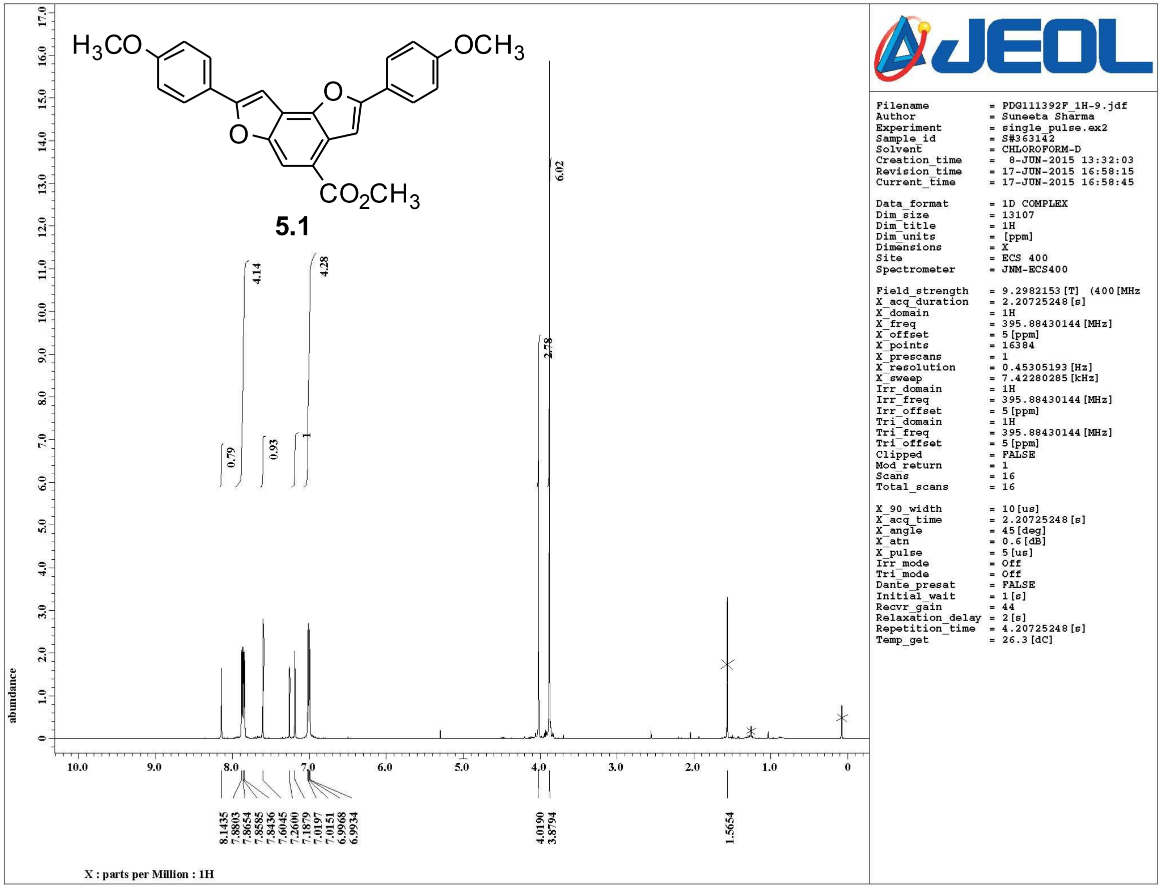


```
Filename          = PDG111311F_13C-4.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse_dec
Sample_id         = S#611791
Solvent           = CHLOROFORM-D
Creation_time     = 28-MAY-2015 01:08:56
Revision_time     = 17-JUN-2015 10:52:15
Current_time      = 17-JUN-2015 10:53:18

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 2000
Total_scans       = 2000

X_90_width        = 8[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 5.8[dB]
X_pulse           = 2.66666667[us]
Irr_atn_dec       = 21.936[dB]
Irr_atn_noe       = 21.936[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1.5[s]
Recvr_gain        = 60
Relaxation_delay  = 1.5[s]
Repetition_time   = 2.548576[s]
Temp_get          = 26.6[dC]
```

^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **4.1**

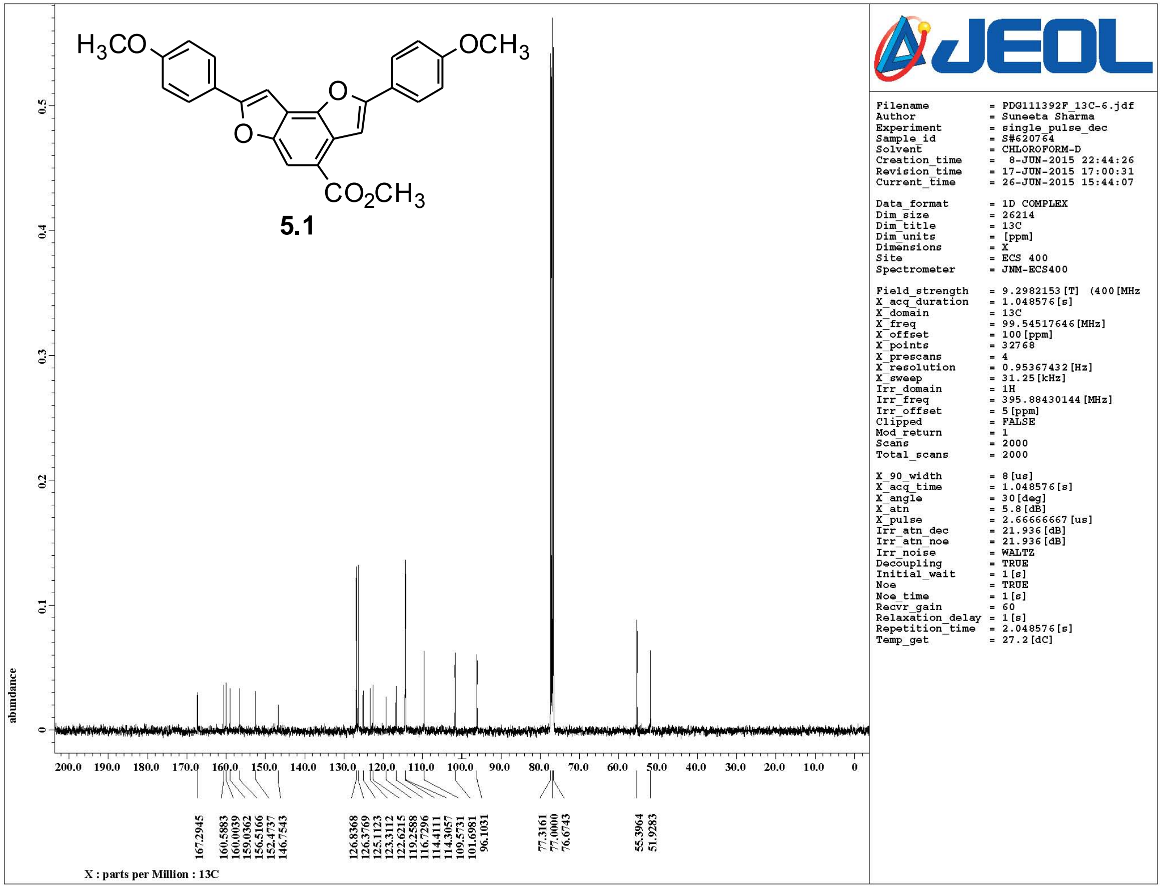


EI (HRMS) spectrum of **4.1**



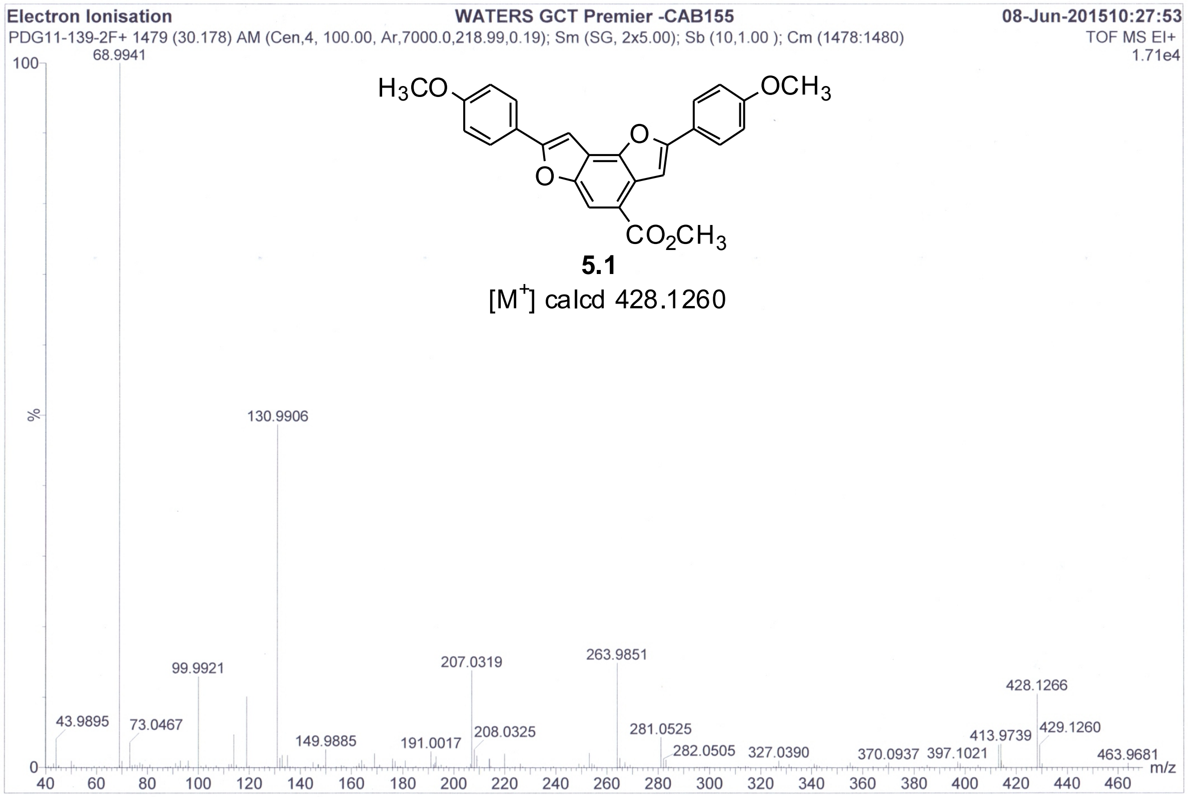
```
Filename          = PDG111392F_1H-9.jdf
Author            = Suneeta Sharma
Experiment        = single_pulse.ex2
Sample_id         = S#363142
Solvent           = CHLOROFORM-D
Creation_time     =  8-JUN-2015 13:32:03
Revision_time     = 17-JUN-2015 16:58:15
Current_time      = 17-JUN-2015 16:58:45

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 0.6[dB]
X_pulse           = 5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 2[s]
Repetition_time   = 4.20725248[s]
Temp_get          = 26.3[dC]
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **5.1**



```
Filename            = PDG111392F_13C-6.jdf
Author              = Suneeta Sharma
Experiment          = single_pulse_dec
Sample_id           = S#620764
Solvent             = CHLOROFORM-D
Creation_time       =  8-JUN-2015 22:44:26
Revision_time       = 17-JUN-2015 17:00:31
Current_time        = 26-JUN-2015 15:44:07

Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = JNM-ECS400

Field_strength      = 9.2982153[T] (400[MHz
X_acq_duration      = 1.048576[s]
X_domain            = 13C
X_freq              = 99.54517646[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 0.95367432[Hz]
X_sweep             = 31.25[kHz]
Irr_domain          = 1H
Irr_freq            = 395.88430144[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 2000
Total_scans         = 2000

X_90_width          = 8[us]
X_acq_time          = 1.048576[s]
X_angle             = 30[deg]
X_atn               = 5.8[dB]
X_pulse             = 2.66666667[us]
Irr_atn_dec         = 21.936[dB]
Irr_atn_noe         = 21.936[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 1[s]
Recvr_gain          = 60
Relaxation_delay    = 1[s]
Repetition_time     = 2.048576[s]
Temp_get            = 27.2[dC]
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **5.1**



EI (HRMS) spectrum of **5.1**

10. Crystal structure of benzodifuran 4.1:

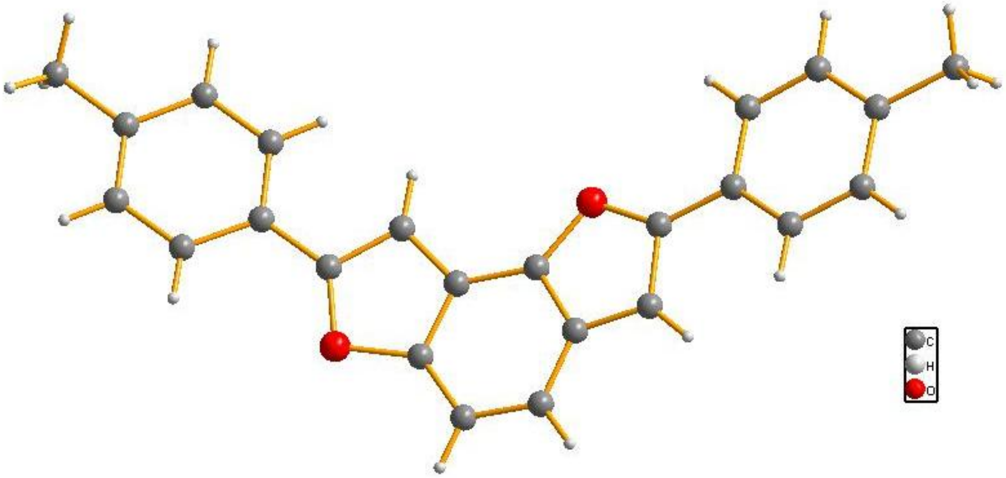


Fig. X-ray structure of **4.1**

The single crystal X-ray was recorded in Bruker SMART APEX-II CCD diffractometer. Data was collected at 273 K using graphite-monochromated MoKα radiation (λ = 0.71073 Å). The crystal structure was solved by direct methods and was refined by full matrix least squares on F^2 using SHELXL-97 and SHELXTL. All hydrogen atoms were included in idealized positions using a riding model. Anisotropic displacement parameters were used for refining non-hydrogen atoms.

Ref: (a) G. M. Sheldrick, *SHELXL-97*, *Program for Crystal Structure Solution and Refinement*; University of Göttingen, Göttingen, Germany, 1997; (b) G. M. Sheldrick, *Acta Cryst.*, 2008, **A64**, 112.

Crystal data for compound **4.1**: $C_{48}H_{36}O_4$, M = 676.77, Monoclinic, a = 18.012(2) Å, b = 6.5373(8) Å, c = 14.3077(19) Å, α = 90.00°, β = 98.302(4)°, γ = 90.00°, V = 1667.1(4) Å^3, T = 273(2) K, space group Cc, Z = 2, μ(MoKα) = 0.085 mm^{-1}, 6737 reflections measured, 3191 independent reflections (R_{int} = 0.0642). The final R_1 values were 0.0978 ($I>2\sigma(I)$). The final $wR(F^2)$ values were 0.2589 ($I>2\sigma(I)$). The final R_1 values were 0.1541 (all data). The final $wR(F^2)$ values were 0.3025 (all data). The goodness of fit on F^2 was 1.065. CCDC number CCDC 1406195.