Supporting Information

Hydrodeoxygenation of sulfoxides to sulfides by Pt and MoOx co-loaded TiO_2 catalyst

Abeda Sultana Touchy,[a] S. M. A. Hakim Siddiki,[b] Wataru Onodera,[a] Kenichi Kon,[a] Ken-ichi Shimizu [a,b,]*

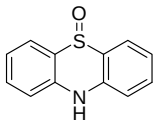
[a] Institute for Catalysis, Hokkaido University, N-21, W-10, Sapporo 001-0021, Japan
[b] Elements Strategy Initiative for Catalysts and Batteries, Kyoto University, Katsura, Kyoto 615-8520, Japan

*Corresponding author
Ken-ichi Shimizu
Institute for Catalysis, Hokkaido University, N-21, W-10, Sapporo 001-0021, Japan
E-mail: kshimizu@cat.hokudai.ac.jp, Fax: +81-11-706-9163

General procedure for oxidation of sulfides[1]

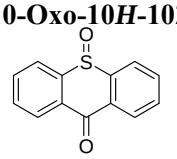
A solution of sulfide (1 mmol), and 35% hydrogen peroxide (3 mmol) in 5 mL of methanol was prepared. Oxalic acid dihydrate (0.5 mmol) was added to the solution and the reaction mixture was stirred for 12-15 h at room temperature until TLC analysis showed that no sulfide remained. Then the reaction mixture was extracted with ethylacetate (15 mL) and the combined organic fraction was washed with 3% $NaHSO_3$ and dried over Na_2SO_4. Evaporation of the solvent gave mixture of corresponding sulfoxide and sulfone. Sulfoxide was separated by column chromatography with silica gel 60 (spherical) 63-210 μm (Kanto Chemical Co. Ltd.) using eluting solvent hexane-ethylacetate (2:3). The products were identified by ^{1}H and ^{13}C NMR and GC-MS analyses.

10*H*-Phenothiazine 5-oxide:[2]



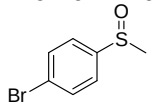
^{1}H NMR (600.17 MHz, CD_3CN, TMS): δ 8.96 (br s, 1H), 7.85 (d, J = 7.56 Hz, 2H), 7.73 (d, J = 7.56 Hz, 2H), 7.61-7.57 (m, 2H), 7.26-7.24 (m, 2H); ^{13}C NMR (150.92 MHz, CD_3CN) δ 137.44 (C×2), 132.27 (C×2), 130.10 (C×2), 121.57 (C×2), 120.82 (C×2), 115.95 (C×2); GC-MS m/e 215.045.

10-Oxo-10*H*-10λ^4-thioxanthen-9-one:[2]



^{1}H NMR (600.17 MHz, CD_3CN, TMS): δ 8.29 (d, *J* = 7.56 Hz, 2H), 8.14 (d, *J* = 7.56 Hz, 2H), 7.96 (t, *J* = 7.80 Hz, 2H), 7.88 (t, *J* = 6.90 Hz, 2H); ^{13}C NMR (150.92 MHz, CD_3CN) δ 177.48, 139.90 (C×2), 134.21 (C×2), 132.71 (C×2), 129.63 (C×2), 128.06 (C×2), 122.22 (C×2); GC-MS m/e 228.025.

1-Bromo-4-methanesulfinyl-benzene:[2]

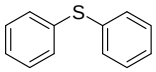


^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 6.85 (d, *J* = 8.94 Hz, 2H), 6.83 (d, *J* = 8.43 Hz, 2H), 2.10 (s, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 139.21, 131.63 (C×2), 128.19 (C×2), 127.16, 42.57 1 ; GC-MS m/e 217.950.

NMR and GC/MS analysis

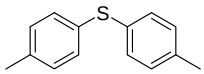
^{1}H and ^{13}C NMR spectra for amines of Table-2 were assigned and reproduced to the corresponding literature. ^{1}H and ^{13}C NMR spectra were recorded using at ambient temperature on JEOL-ECX 600 operating at 600.17 and 150.92 MHz, respectively with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane and *d*-solvent peaks 77.00 ppm chloroform. Abbreviations used in the NMR experiments: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. GC-MS spectra was taken by SHIMADZU QP2010.

Diphenyl sulfide:[3]



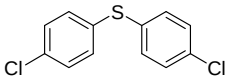
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.33-7.32 (m, 4H), 7.27 (t, *J* = 7.56 Hz, 4H), 7.22-7.19 (m, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 135.71 (C×2), 130.97 (C×4), 129.13 (C×4), 126.98 (C×2); GC-MS m/e 186.055.

Di-*p*-tolyl-sulfide:[4]



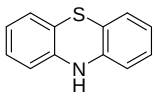
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.22 (d, *J* = 8.22 Hz, 4H), 7.09 (d, *J* = 8.22 Hz, 4H), 2.30 (s, 6H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 136.87 (C×2), 132.60 (C×2), 131.02 (C×4), 129.88 (C×4), 21.05 (C×2); GC-MS m/e 214.095.

Bis-(4-chloro-phenyl)-sfide:[3]



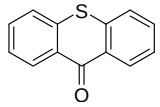
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.28-7.26 (m, 4H), 7.25-7.23 (m, 4H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 133.91 (C×2), 133.44 (C×2), 132.28 (C×4), 129.47 (C×4); GC-MS m/e 253.975.

10*H*-Phenothiazine:CAS registry No. [92-84-2]



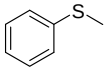
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 6.98-6.96 (m, 4H), 6.81 (s, 2H), 6.53 (d, *J* = 7.56 Hz, 2H), 5.77 (br s, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 141.53 (C×2), 127.30 (C×2) , 126.76 (C×2), 122.59 (C×2), 118.20 (C×2), 114.41 (C×2); GC-MS m/e 199.055.

Thioxanthen-9-one: CAS registry No.[492-22-8]



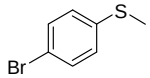
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 8.60 (d, *J* = 7.56 Hz, 2H), 7.61-7.58 (m, 2H), 7.55-7.52 (m, 2H), 7.48-7.45 (m, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 179.84, 137.18 (C×2), 132.16 (C×2), 129.74 (C×2), 129.11 (C×2), 126.20 (C×2), 125.88 (C×2); GC-MS m/e 212.025.

Methyl phenyl sulfide:[4]



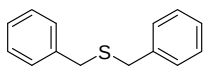
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.28-7.25 (m, 4H), 7.14-7.12 (m, 1H), 2.48 (s, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 138.35, 128.78 (C×2), 126.55 (C×2), 124.97, 15.80; GC-MS m/e 124.035.

1-Bromo-4-methylsulfanyl-benzene:[3]



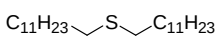
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.37 (d, *J* = 8.94 Hz, 2H), 7.09 (d, *J* = 8.28 Hz, 2H), 2.44 (s, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 137.62, 131.68 (C×2), 127.96 (C×2), 118.48, 15.79; GC-MS m/e 201.955.

Di-benzyl-sulfide:[3]



^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.38-7.32 (m, 6H), 7.29-7.25 (m, 4H), 3.91 (d, *J* = 12.08 Hz, 2H), 3.86 (d, *J* = 12.08 Hz, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 138.10 (C×2), 128.96 (C×4), 128.42 (C×4), 126.92 (C×2), 35.51 (C×2); GC-MS m/e 214.095.

Di-dodecyl-sulfide:[4]

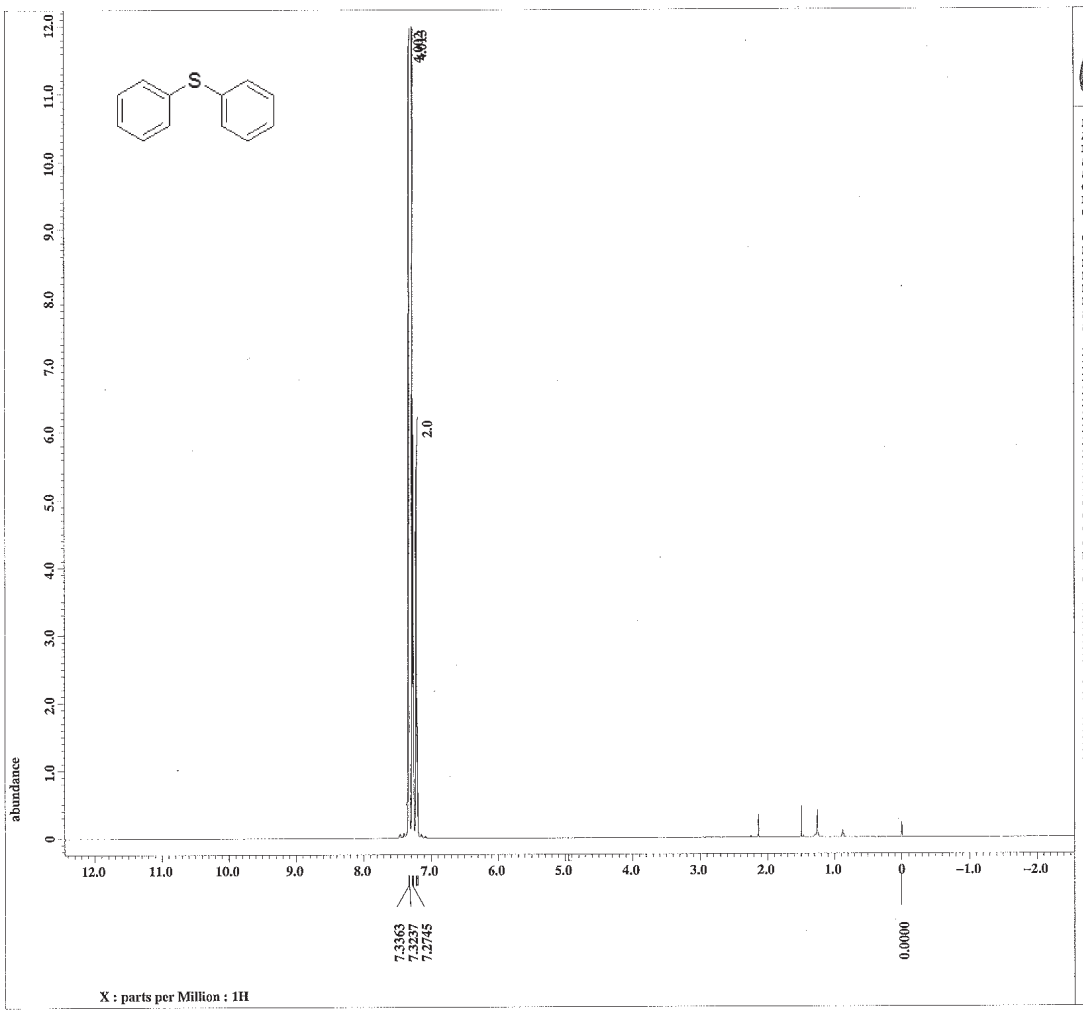


^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 2.49 (t, *J* = 6.84 Hz, 4H), 1.58-1.55 (m, 4H), 1.38-1.35 (m, 4H), 1.32-1.23 (m, 32H), 0.88 (t, *J* = 6.84 Hz, 6H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 32.15

(C×2), 31.90 (C×2), 29.71 (C×2), 29.64 (C×2), 29.62 (C×2), 29.59 (C×2), 29.52 (C×2), 29.34 (C×2), 29.25 (C×2), 28.95 (C×2), 22.68 (C×2), 14.11 (C×2); GC-MS m/e 370.365.

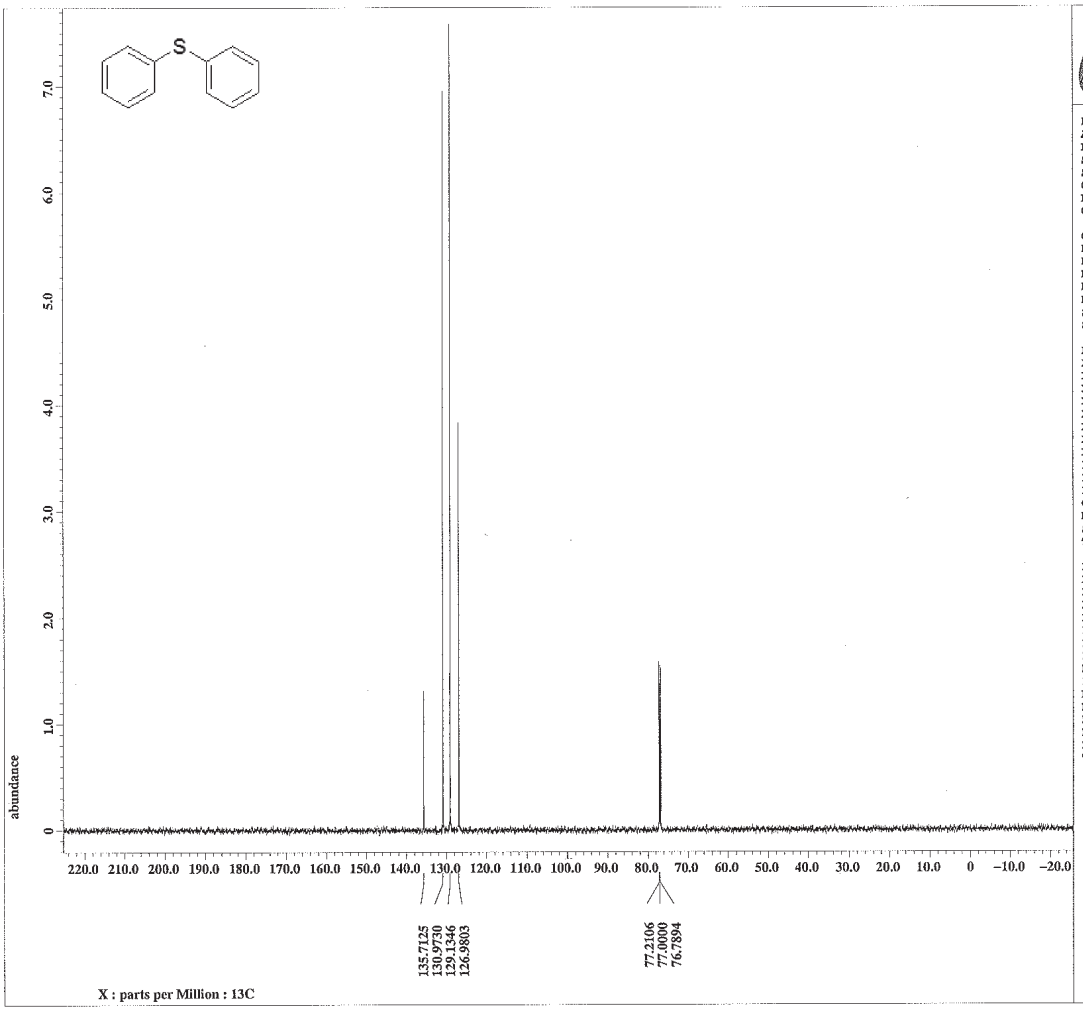
References

1. M. Mokhtary, M. Quandalee, M.R. Niaki, *E. J. Chem.*, **2012**, 863-868
2. J. Drabowicz, P. Łyżwa, M. Popielarczyk, M. Mikołajczyk, *Synthesis*, **1990**, 937–938.
3. T. Mitsudome, Y. Takahashi, T. Mizugaki, K. Jitsukawa, K. Kaneda, *Angew. Chem. Int. Ed.*, 2014, **53**, 8348–8351.
4. S. Enthaler, *ChemCatChem*, 2011, **3**, 666–670.



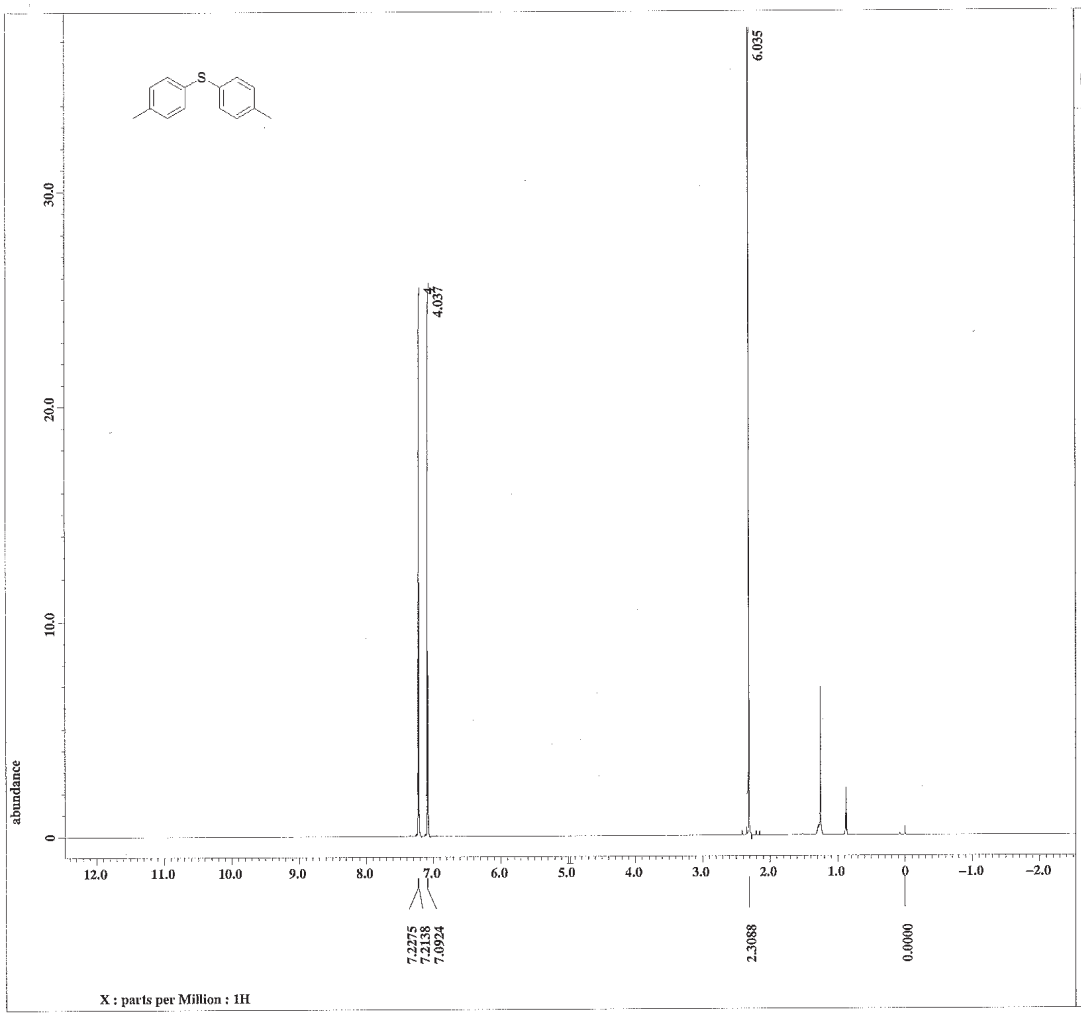
JEOL

Filename	= Exp-AB-210-1-2-proton
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-AB-210-1-2-proton
Solvent	= CHLOROFORM-D
Creation_time	= 28-MAY-2015 22:14:42
Revision_time	= 28-MAY-2015 22:46:34
Current_time	= 28-MAY-2015 22:46:43
Content	= Exp-AB-210-1-2-proton
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 1.4548992[s]
X_domain	= 1H
X_freq	= 600.1723046[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.68733284[Hz]
X_sweep	= 11.26126126[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 600.1723046[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13[us]
X_acq_time	= 1.4548992[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 36
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 20.6[dC]



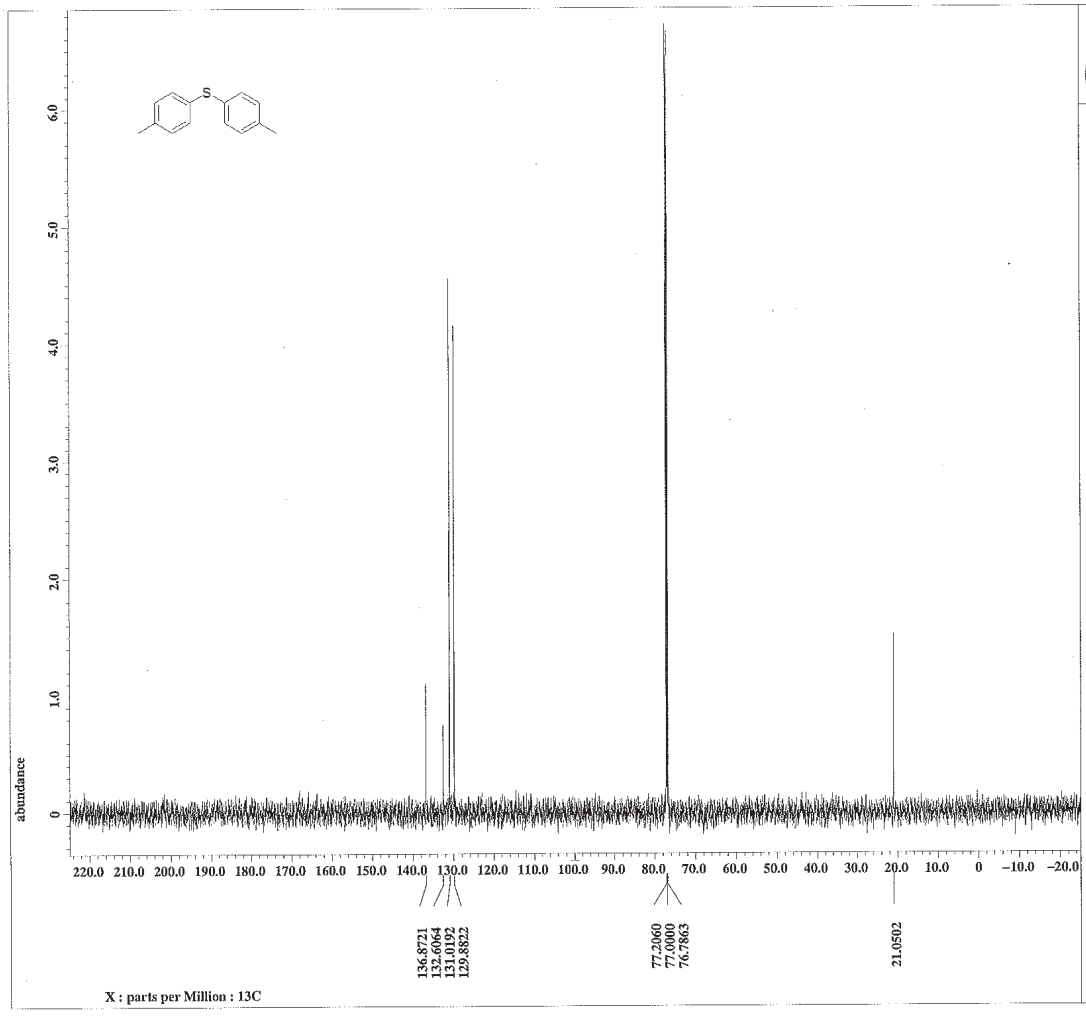
JEOL

Filename	= Exp-AB-210-1-2-carbon
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-AB-210-1-2-carbon
Solvent	= CHLOROFORM-D
Creation_time	= 28-MAY-2015 22:17:19
Revision_time	= 28-MAY-2015 22:47:53
Current_time	= 28-MAY-2015 22:47:58
Content	= Exp-AB-210-1-2-carbon
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 0.69206016[s]
X_domain	= 13C
X_freq	= 150.91343039[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.44496109[Hz]
X_sweep	= 47.34848485[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 34
Total_scans	= 34
X_90_width	= 11.4[us]
X_acq_time	= 0.69206016[s]
X_angle	= 30[deg]
X_atn	= 7.5[dB]
X_pulse	= 3.8[us]
Irr_atn_dec	= 19.391[dB]
Irr_atn_noe	= 19.391[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2.5[s]
Recvr_gain	= 60
Relaxation_delay	= 2.5[s]
Repetition_time	= 3.1920606016[s]
Temp_get	= 21[dC]



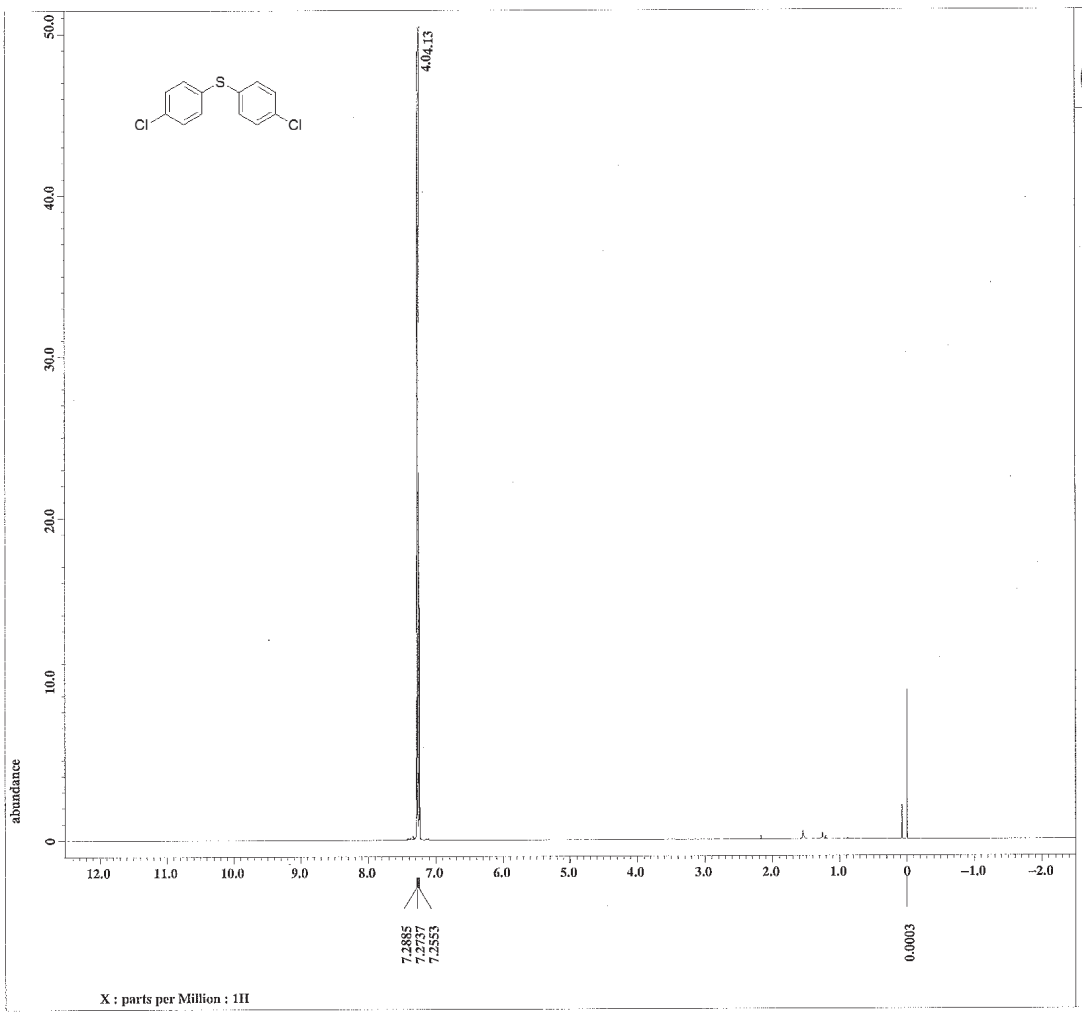
JEOL

Filename	= Exp-AB-207-2-proton-5
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-AB-207-2-proton
Solvent	= CHLOROFORM-D
Creation_time	= 11-MAY-2015 13:00:12
Revision_time	= 11-MAY-2015 13:30:32
Current_time	= 11-MAY-2015 13:30:43
Content	= Exp-AB-207-2-proton
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 1.4548992[s]
X_domain	= 1H
X_freq	= 600.1723046[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.68733284[Hz]
X_sweep	= 11.26126126[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 600.1723046[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13[us]
X_acq_time	= 1.4548992[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 36
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 21[dC]

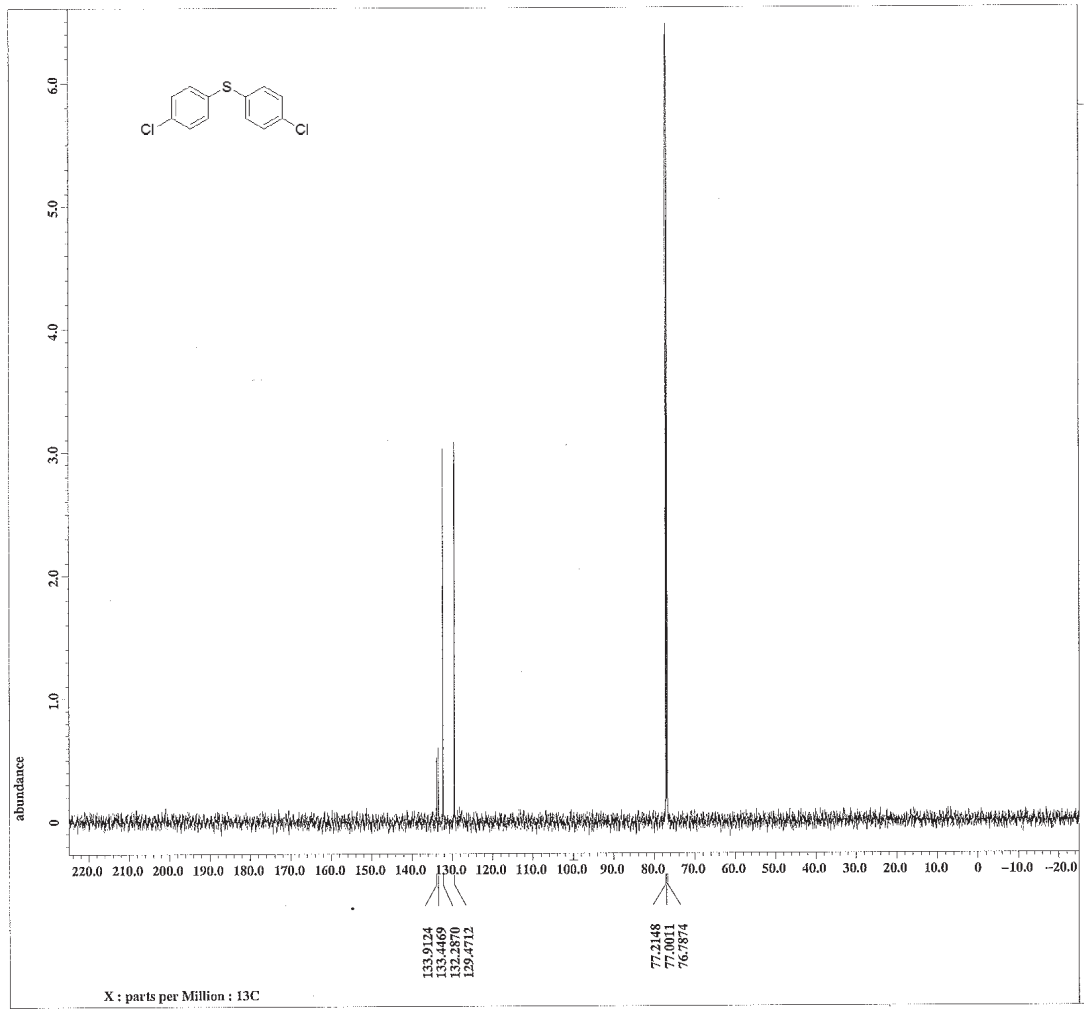


JEOL

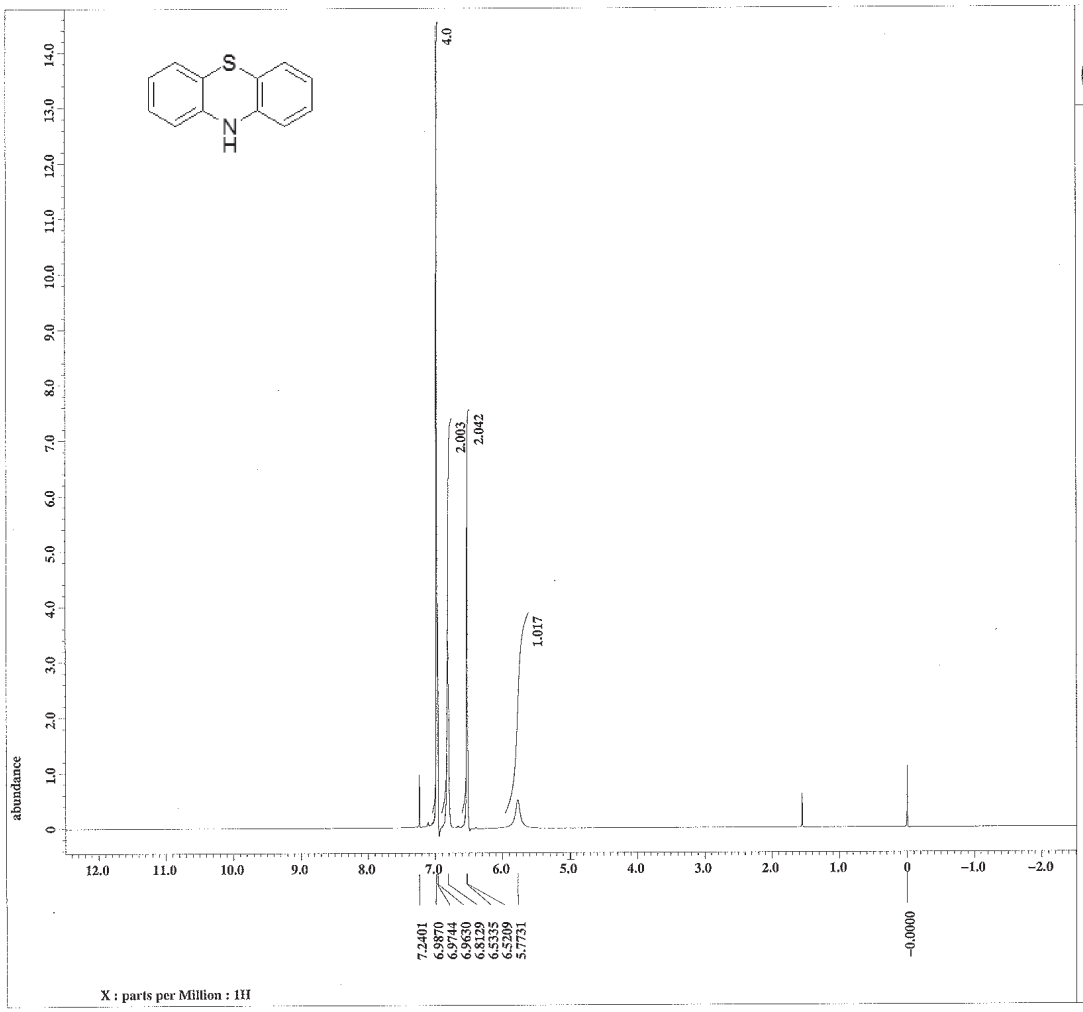
Filename	= Exp-AB-207-1-1-carbon
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-AB-207-1-1-carbon
Solvent	= CHLOROFORM-D
Creation_time	= 11-MAY-2015 13:11:02
Revision_time	= 11-MAY-2015 13:42:19
Current_time	= 11-MAY-2015 13:42:24
Content	= Exp-AB-207-1-1-carbon
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 0.868352[s]
X_domain	= 13C
X_freq	= 150.91343039[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.15160672[Hz]
X_sweep	= 37.73584906[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 31
Total_scans	= 31
X_90_width	= 11.4[us]
X_acq_time	= 0.868352[s]
X_angle	= 30[deg]
X_atn	= 7.5[dB]
X_pulse	= 3.8[us]
Irr_atn_dec	= 19.391[dB]
Irr_atn_noe	= 19.391[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2.5[s]
Recvr_gain	= 80
Relaxation_delay	= 2.5[s]
Repetition_time	= 3.368352[s]
Temp_get	= 21[dC]




```
Filename          = Exp-AB-213-1cr-proton
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-AB-213-1cr-proton
Solvent           = CHLOROFORM-D
Creation_time     =  9-APR-2015 16:30:56
Revision_time     =  9-APR-2015 16:58:57
Current_time      =  9-APR-2015 16:59:02

Content           = Exp-AB-213-1cr-proton
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 56
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.8[dC]
```

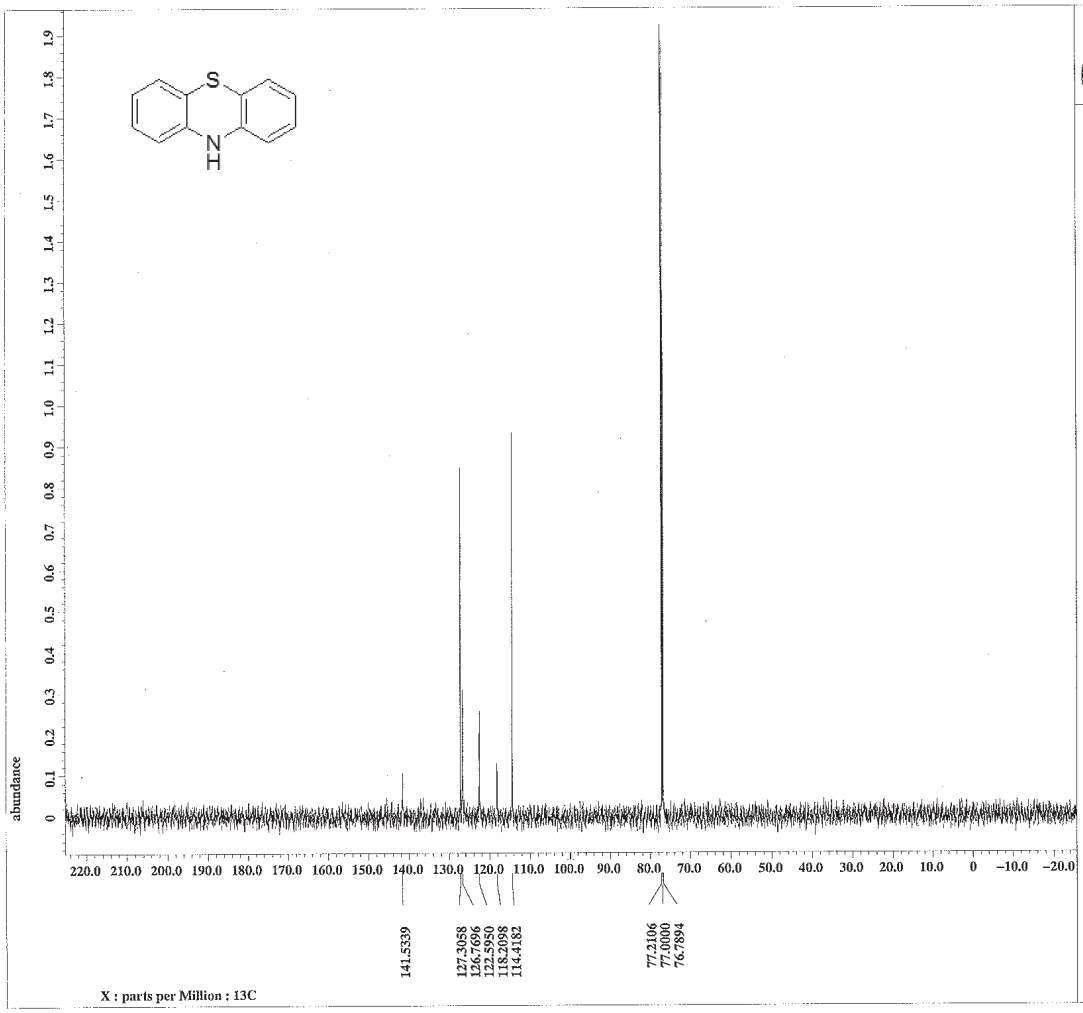



```
Filename          = Exp-AB-213-1cr-carbon
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-AB-213-1cr-carbon
Solvent           = CHLOROFORM-D
Creation_time     =  9-APR-2015 16:36:50
Revision_time     =  9-APR-2015 17:05:49
Current_time      =  9-APR-2015 17:05:58

Content           = Exp-AB-213-1cr-carbon
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 86
Total_scans       = 86

X_90_width        = 11.4[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 80
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 20.9[dC]
```



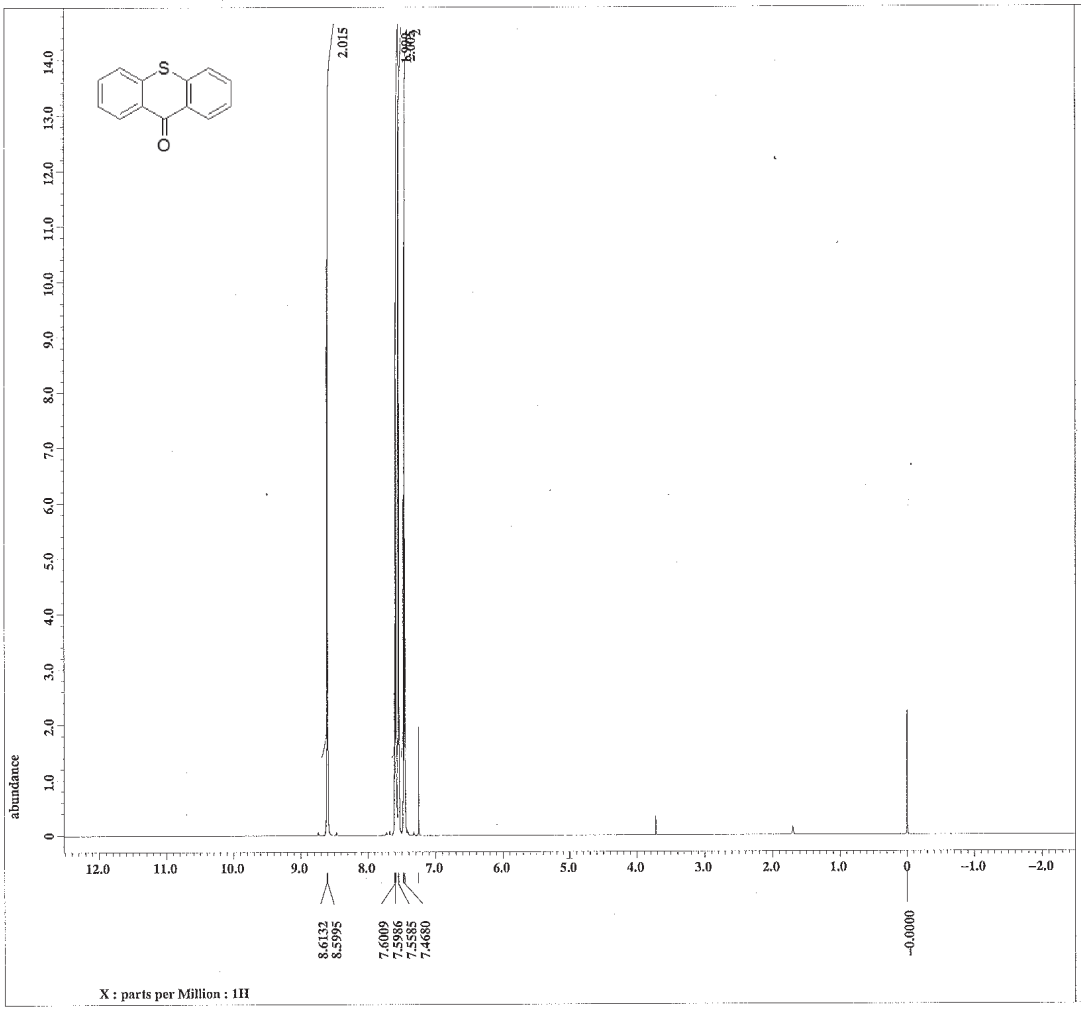
JEOL

```
Filename          = Exp-AB-benzothia-prot
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-AB-benzothia-prot
Solvent           = CHLOROFORM-D
Creation_time     = 13-NOV-2015 11:21:22
Revision_time     = 13-NOV-2015 22:00:31
Current_time      = 13-NOV-2015 22:00:40

Content           = Exp-AB-benzothia-prot
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Nod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 12.6[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.3[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.3[dC]
```



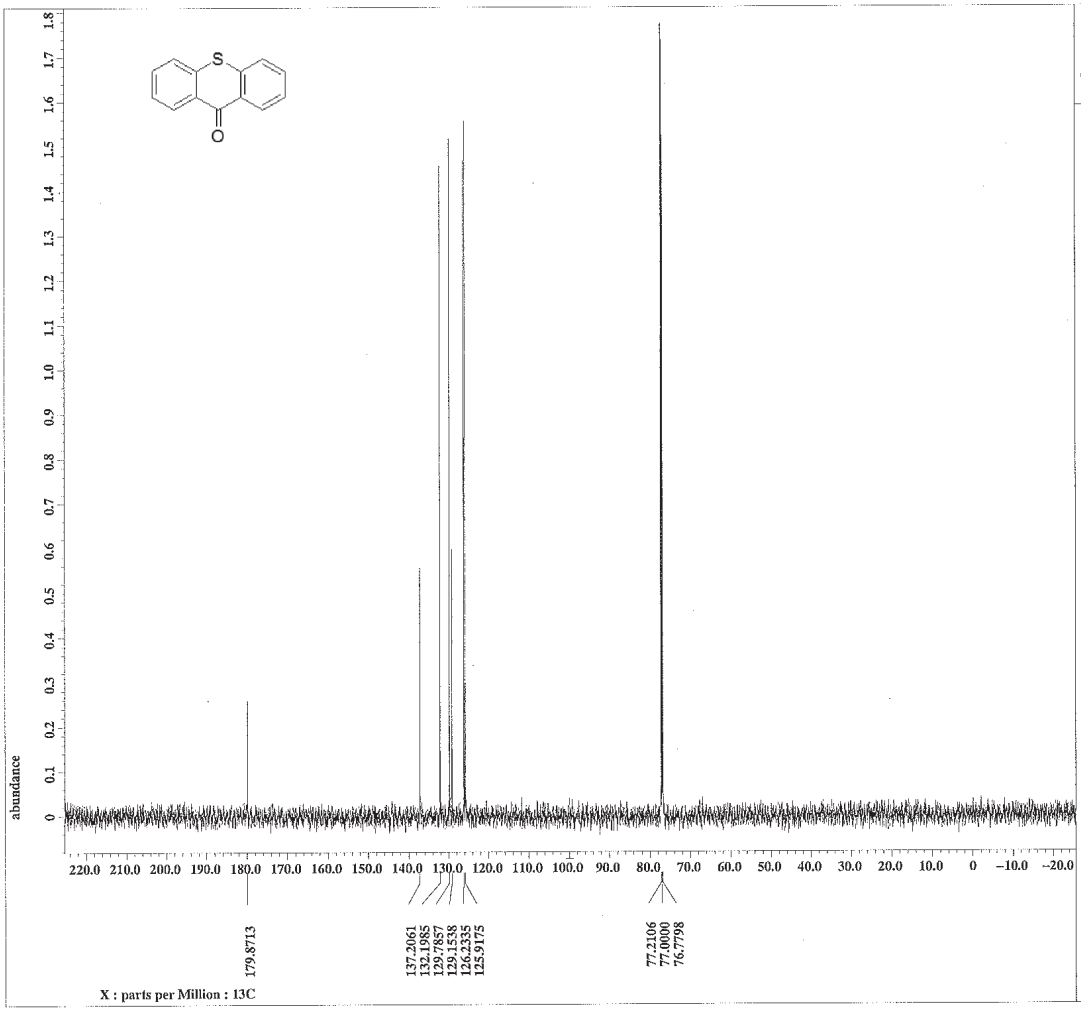
JEOL

```
Filename          = Exp-AB-benzothia-carb
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-AB-benzothia-carb
Solvent           = CHLOROFORM-D
Creation_time     = 13-NOV-2015 11:18:59
Revision_time     = 13-NOV-2015 12:06:48
Current_time      = 13-NOV-2015 12:07:01

Content           = Exp-AB-benzothia-carb
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.69206016[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.44496109[Hz]
X_sweep           = 47.34848485[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 44
Total_scans       = 44

X_90_width        = 10.9[us]
X_acq_time        = 0.69206016[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.63333333[us]
Irr_atn_dec       = 19.21[dB]
Irr_atn_noe       = 19.21[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 60
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.19206016[s]
Temp_get          = 21.6[dC]
```



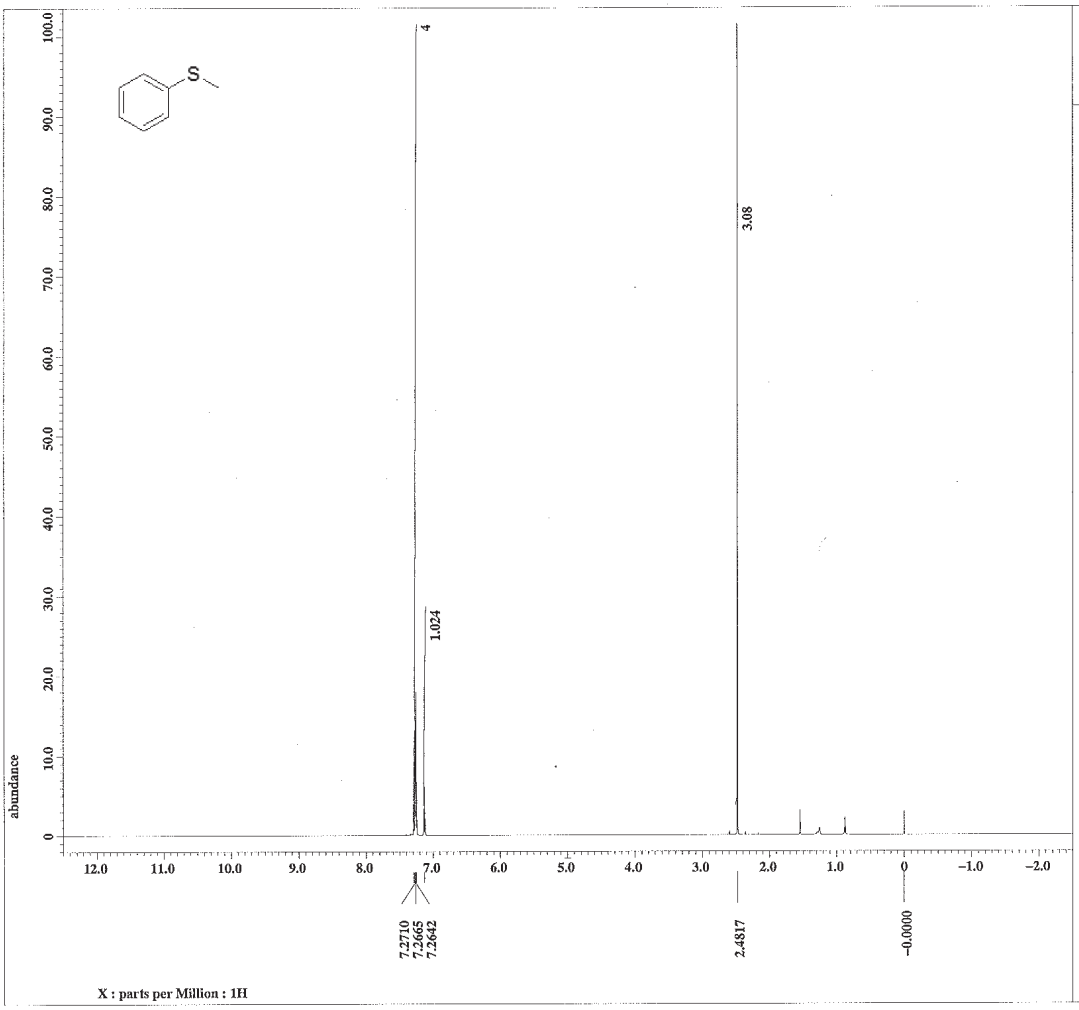
JEOL

```
Filename          = Exp-AB-final-2-proton
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-AB-final-2-proton
Solvent           = CHLOROFORM-D
Creation_time     = 13-NOV-2015 21:20:55
Revision_time     = 13-NOV-2015 22:10:05
Current_time      = 13-NOV-2015 22:10:11

Content           = Exp-AB-final-2-proton
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 12.6[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.3[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.7[dC]
```



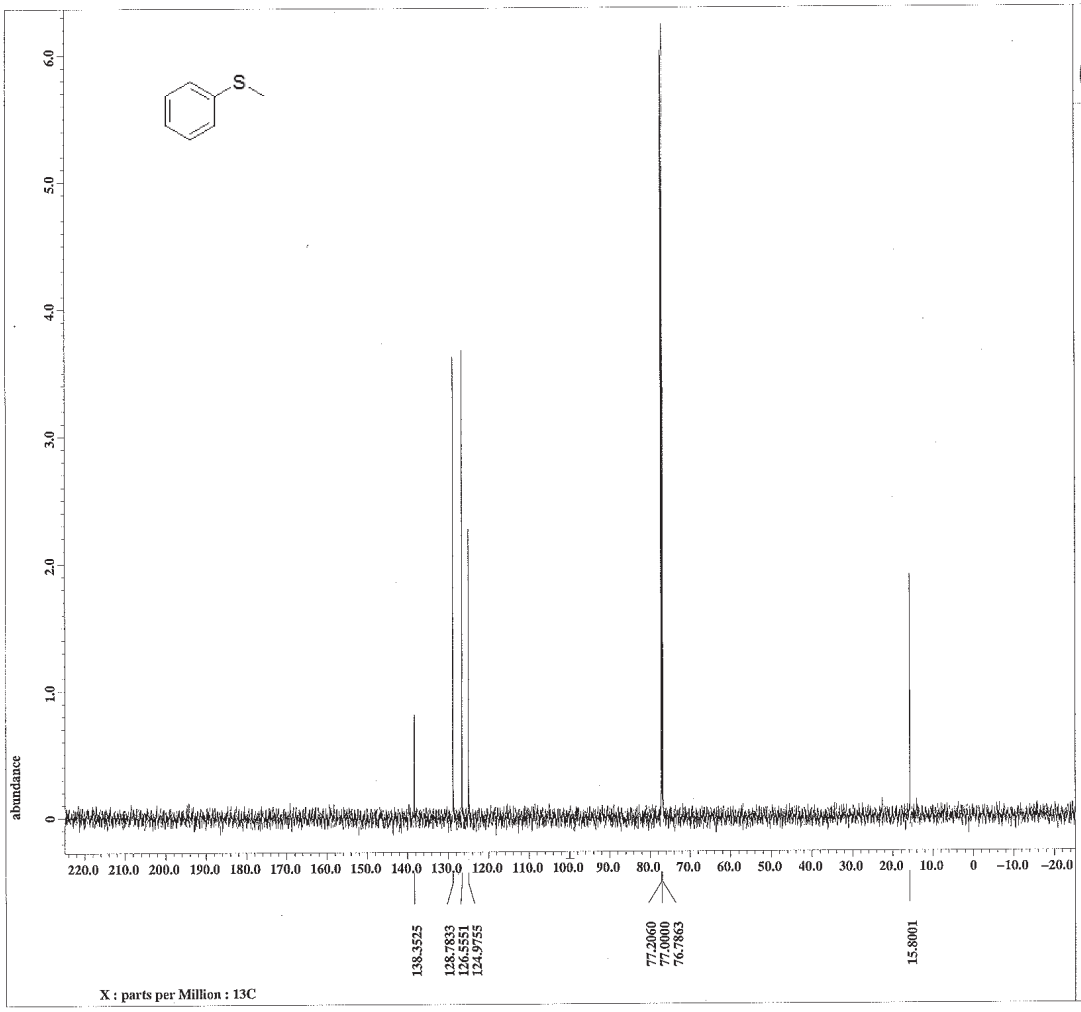
JEOL

```
Filename          = Exp-AB-final-2-carbon
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-AB-final-2-carbon
Solvent           = CHLOROFORM-D
Creation_time     = 13-NOV-2015 21:24:34
Revision_time     = 13-NOV-2015 22:12:50
Current_time      = 13-NOV-2015 22:12:53

Content           = Exp-AB-final-2-carbon
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.69206016[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.44496109[Hz]
X_sweep           = 47.34848485[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 53
Total_scans       = 53

X_90_width        = 10.9[us]
X_acq_time        = 0.69206016[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.63333333[us]
Irr_atn_dec       = 19.21[dB]
Irr_atn_noe       = 19.21[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 60
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.19206016[s]
Temp_get          = 21.7[dC]
```



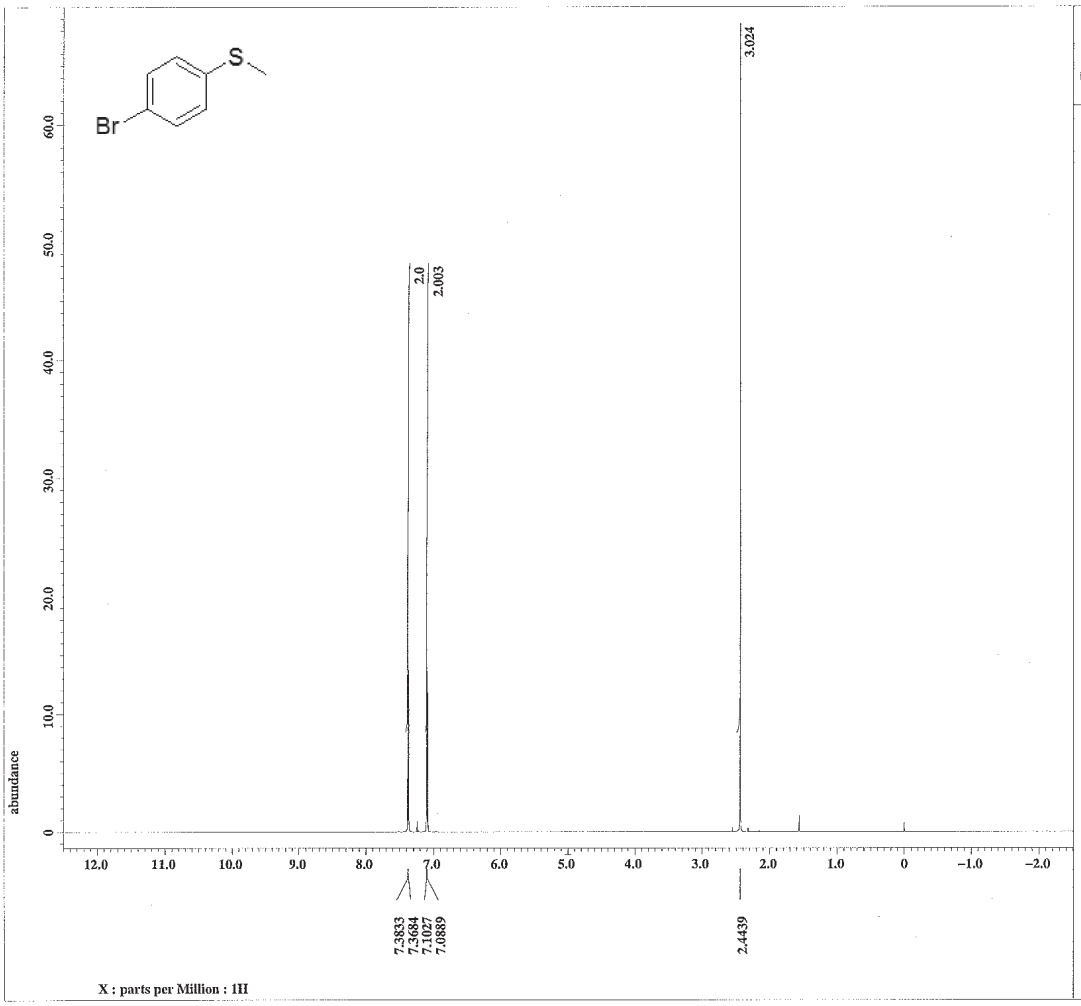
JEOL

```
Filename          = Exp-AB-201-4-proton-5
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-AB-201-4-proton
Solvent           = CHLOROFORM-D
Creation_time     = 28-MAY-2015 12:18:24
Revision_time     = 28-MAY-2015 12:50:09
Current_time      = 28-MAY-2015 12:50:16

Content           = Exp-AB-201-4-proton
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.3[dC]
```



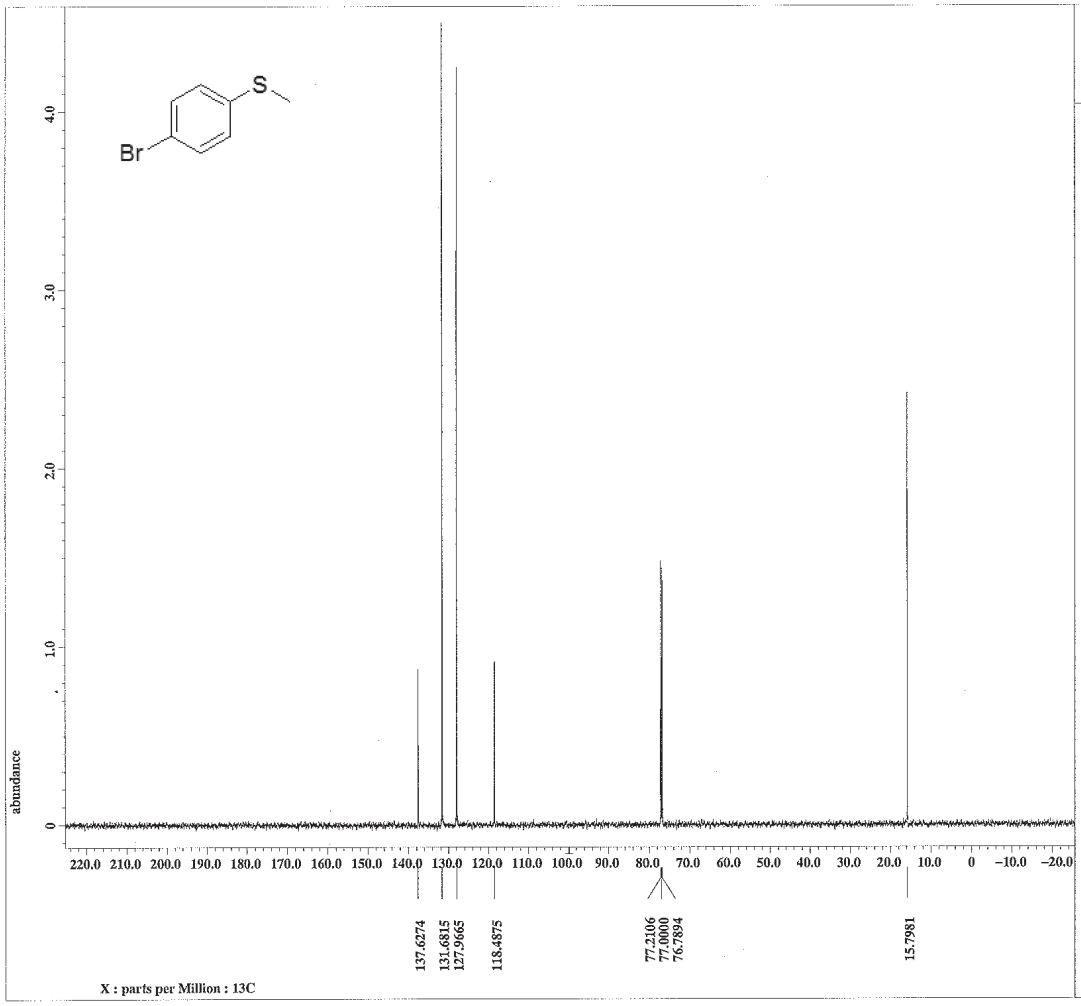
JEOL

```
Filename          = Exp-AB-201-4-carbon-3
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-AB-201-4-carbon
Solvent           = CHLOROFORM-D
Creation_time     = 28-MAY-2015 12:23:01
Revision_time     = 28-MAY-2015 12:57:47
Current_time      = 28-MAY-2015 12:57:49

Content           = Exp-AB-201-4-carbon
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 67
Total_scans       = 67

X_90_width        = 11.4[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 80
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 21[dC]
```



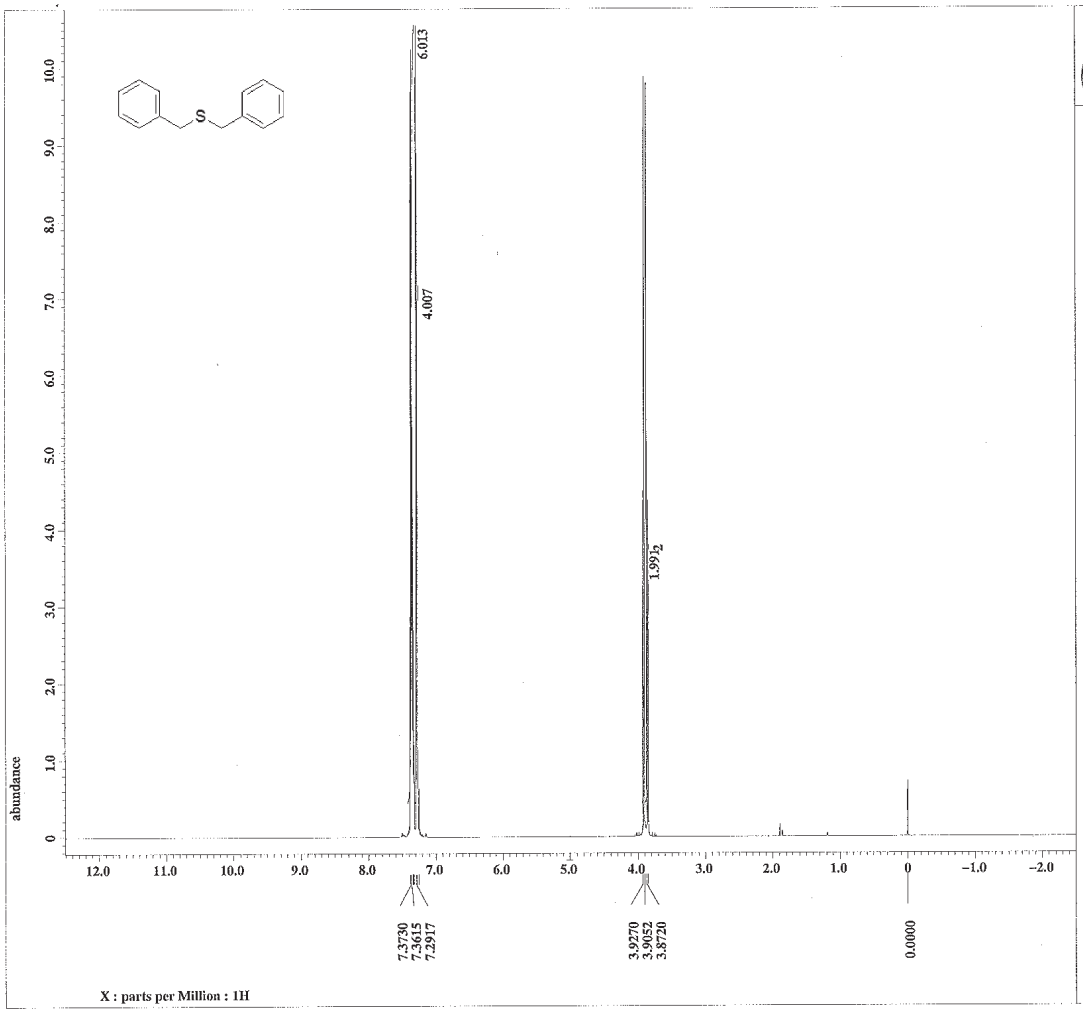
JEOL

```
Filename          = Exp-AB-4-Br-Ph-Me-sul
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-AB-4-Br-Ph-Me-sul
Solvent           = CHLOROFORM-D
Creation_time     = 24-JUL-2015 15:42:53
Revision_time     = 24-JUL-2015 16:24:55
Current_time      = 24-JUL-2015 16:25:00

Content           = Exp-AB-4-Br-Ph-Me-sul
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.8[dC]
```



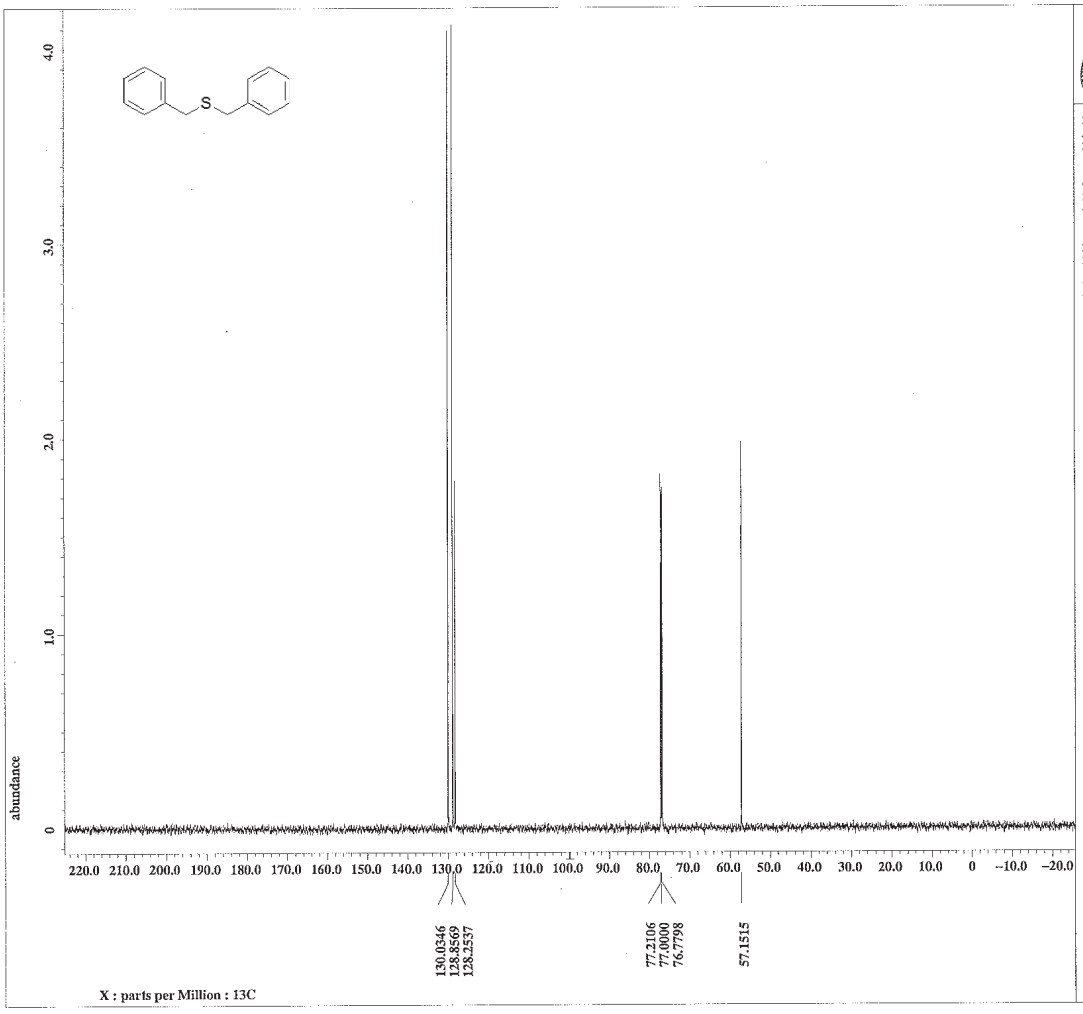
JEOL

```
Filename          = Exp-AB-4-Br-Ph-Me-sul
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-AB-4-Br-Ph-Me-sul
Solvent           = CHLOROFORM-D
Creation_time     = 24-JUL-2015 15:48:01
Revision_time     = 24-JUL-2015 16:26:02
Current_time      = 24-JUL-2015 16:26:08

Content           = Exp-AB-4-Br-Ph-Me-sul
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.69206016[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.44496109[Hz]
X_sweep           = 47.34848485[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 81
Total_scans       = 81

X_90_width        = 11.4[us]
X_acq_time        = 0.69206016[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 60
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.19206016[s]
Temp_get          = 21.3[dC]
```



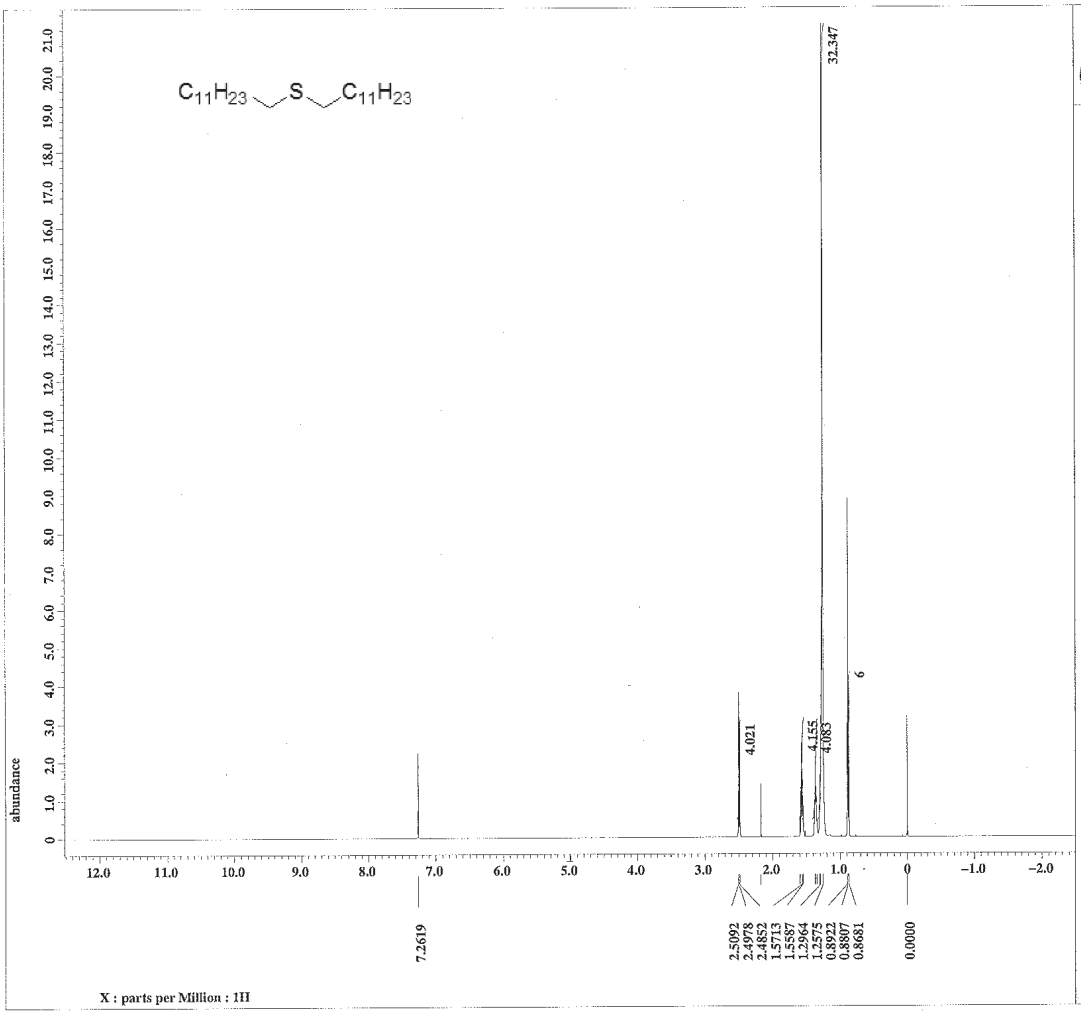
JEOL

```
Filename            = Exp-AB-final-3-proton
Author              = delta
Experiment          = single_pulse.ex2
Sample_id           = Exp-AB-final-3-proton
Solvent             = CHLOROFORM-D
Creation_time       = 13-NOV-2015 21:36:44
Revision_time       = 13-NOV-2015 22:25:09
Current_time        = 13-NOV-2015 22:25:12

Content             = Exp-AB-final-3-proton
Data_format         = 1D COMPLEX
Dim_size            = 13107
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA 600
Spectrometer        = DELTA2_NMR

Field_strength      = 14.09636928[T] (600[M
X_acq_duration      = 1.4548992[s]
X_domain            = 1H
X_freq              = 600.1723046[MHz]
X_offset            = 5[ppm]
X_points            = 16384
X_prescans          = 1
X_resolution        = 0.68733284[Hz]
X_sweep             = 11.26126126[kHz]
Irr_domain          = 1H
Irr_freq            = 600.1723046[MHz]
Irr_offset          = 5[ppm]
Tri_domain          = 1H
Tri_freq            = 600.1723046[MHz]
Tri_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 8
Total_scans         = 8

X_90_width          = 12.6[us]
X_acq_time          = 1.4548992[s]
X_angle             = 45[deg]
X_atn               = 3.6[dB]
X_pulse             = 6.3[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 40
Relaxation_delay    = 5[s]
Repetition_time     = 6.4548992[s]
Temp_get            = 20.3[dC]
```



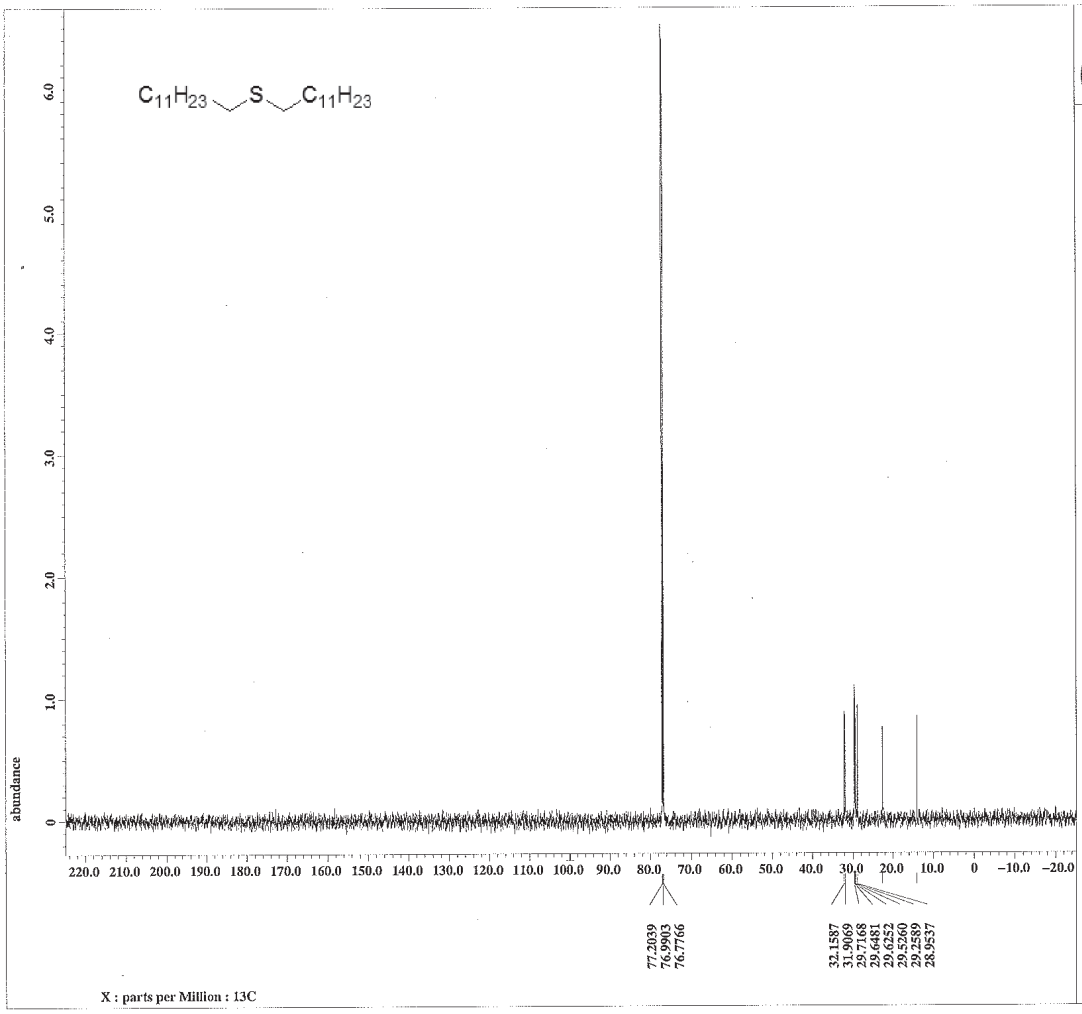
JEOL

```
Filename            = Exp-AB-final-3-carbon
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = Exp-AB-final-3-carbon
Solvent             = CHLOROFORM-D
Creation_time       = 13-NOV-2015 21:41:13
Revision_time       = 13-NOV-2015 22:31:28
Current_time        = 13-NOV-2015 22:31:32

Content             = Exp-AB-final-3-carbon
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA 600
Spectrometer        = DELTA2_NMR

Field_strength      = 14.09636928[T] (600[M
X_acq_duration      = 0.69206016[s]
X_domain            = 13C
X_freq              = 150.91343039[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 1.44496109[Hz]
X_sweep             = 47.34848485[kHz]
Irr_domain          = 1H
Irr_freq            = 600.1723046[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 69
Total_scans         = 69

X_90_width          = 10.9[us]
X_acq_time          = 0.69206016[s]
X_angle             = 30[deg]
X_atn               = 7.5[dB]
X_pulse             = 3.63333333[us]
Irr_atn_dec         = 19.21[dB]
Irr_atn_noe         = 19.21[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 2.5[s]
Recvr_gain          = 60
Relaxation_delay    = 2.5[s]
Repetition_time     = 3.19206016[s]
Temp_get            = 21.4[dC]
```



JEOL

Filename	= Exp-AB-207-3cr-proton
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-AB-207-3cr-proton
Solvent	= CHLOROFORM-D
Creation_time	= 9-APR-2015 16:17:37
Revision_time	= 9-APR-2015 16:47:40
Current_time	= 9-APR-2015 16:47:43
Content	= Exp-AB-207-3cr-proton
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 1.4548992[s]
X_domain	= 1H
X_freq	= 600.1723046[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.68733284[Hz]
X_sweep	= 11.26126126[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 600.1723046[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13[us]
X_acq_time	= 1.4548992[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 46
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 20.5[dC]



JEOL

Filename	= Exp-AB-207-3cr-carbon
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-AB-207-3cr-carbon
Solvent	= CHLOROFORM-D
Creation_time	= 9-APR-2015 16:23:44
Revision_time	= 9-APR-2015 16:52:49
Current_time	= 9-APR-2015 16:52:55
Content	= Exp-AB-207-3cr-carbon
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 0.868352[s]
X_domain	= 13C
X_freq	= 150.91343039[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.15160672[Hz]
X_sweep	= 37.73584906[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 94
Total_scans	= 94
X_90_width	= 11.4[us]
X_acq_time	= 0.868352[s]
X_angle	= 30[deg]
X_atn	= 7.5[dB]
X_pulse	= 3.8[us]
Irr_atn_dec	= 19.391[dB]
Irr_atn_noe	= 19.391[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2.5[s]
Recvr_gain	= 80
Relaxation_delay	= 2.5[s]
Repetition_time	= 3.368352[s]
Temp_get	= 20.7[dC]