Supporting Information

Heterogeneous catalysts for cyclization of dicarboxylic acids to cyclic anhydrides as monomers for bioplastics

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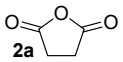
Characterization of Cyclic Anhydrides

^{1}H and ^{13}C NMR spectra for anhydrides of Table-3 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 40.42 ppm for dimethyl sulfoxide-d_6 and 77.00 ppm chloroform-d. Abbreviations used in the NMR experiments: s, singlet; d, doublet; dd, doublet of doublets, t, triplet; m, multiplet.

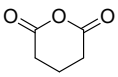
Dihydro-furan-2,5-dione:[1]



2a

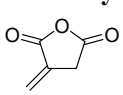
^{1}H NMR (600.17 MHz, DMSO-d_6): δ 2.93 (s, 4H); ^{13}C NMR (150.92 MHz, DMSO-d_6): δ 174.01 (C×2), 29.68 (C×2).

Dihydro-pyran-2,6-dione:[1]



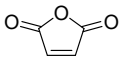
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 2.75 (t, *J* = 6.61 Hz, 4H), 2.05-2.01 (m, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$, TMS): δ 166.51 (C×2), 29.91 (C×2), 16.29.

3-Methylene-dihydro-furan-2,5-dione:[1]



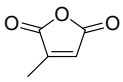
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 6.48 (t like, *J* = 2.52 Hz, 1H), 5.93 (t like, *J* = 2.52 Hz, 1H), 3.63 (t, *J* = 4.98 Hz, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$, TMS): δ 167.71, 164.46, 130.30, 126.63, 33.59.

Furan-2,5-dione:[1]



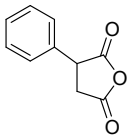
^{1}H NMR (600.17 MHz, DMSO-d_6): δ 6.67 (s, 2H); ^{13}C NMR (150.92 MHz, DMSO-d_6): δ 166.88 (C×2), 134.92 (C×2).

3-Methyl-furan-2,5-dione:[1]



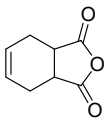
^{1}H NMR (600.17 MHz, DMSO-d_6): δ 7.10 (d like s, 1H), 2.12 (s, 3H); ^{13}C NMR (150.92 MHz, DMSO-d_6): δ 167.71, 165.90, 150.35, 130.63, 12.04.

3-Phenyl-dihydro-furan-2,5-dione:[1]



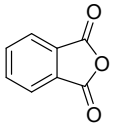
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.39 (t, *J* = 6.90 Hz, 2H), 7.34 (t, *J* = 7.56 Hz, 1H), 7.24 (d, *J* = 6.90 Hz, 2H), 4.32 (m, 1H), 3.42 (dd, *J* = 18.87, 10.32 Hz, 1H), 3.07 (dd, *J* = 18.87, 6.84 Hz, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$, TMS): δ 171.71, 169.62, 134.56, 129.36 (C×2), 128.55, 127.24 (C×2), 46.39, 36.49.

3a,4,7,7a-Tetrahydro-isobenzofuran-1,3-dione:[2]



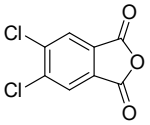
^{1}H NMR (600.17 MHz, DMSO-d_6): δ 5.99 (t, *J* = 1.38 Hz, 2H), 3.55 (t, *J* = 2.04 Hz, 2H), 2.43 (d, *J* = 15.31 Hz, 2H), 2.29 (d, *J* = 15.31 Hz, 2H); ^{13}C NMR (150.92 MHz, DMSO-d_6): δ 176.59 (C×2), 128.85 (C×2), 40.44 (C×2), 24.06 (C×2).

Isobenzofuran-1,3-dione:[1]



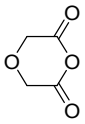
^{1}H NMR (600.17 MHz, DMSO-d_6): δ 8.13-8.12 (m, 2H), 8.06-8.04 (m, 2H); ^{13}C NMR (150.92 MHz, DMSO-d_6): δ 164.09 (C×2), 137.09 (C×2), 132.14 (C×2), 126.25 (C×2).

5,6-Dichloro-isobenzofuran-1,3-dione:[3]



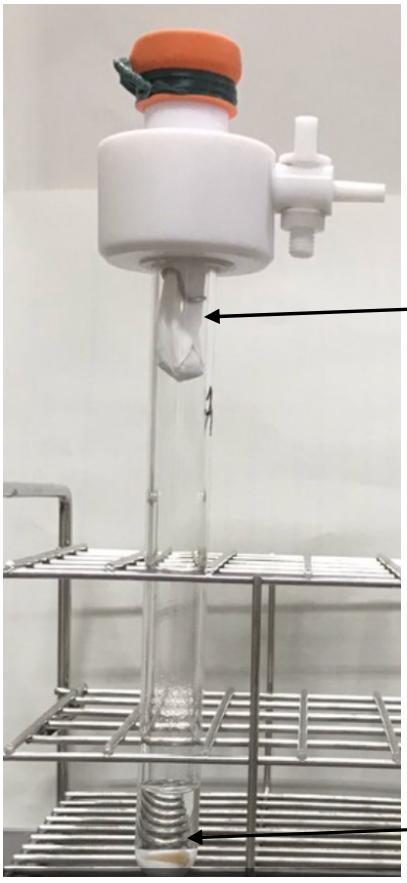
^{1}H NMR (600.17 MHz, DMSO-d_6): δ 8.52 (s, 2H); ^{13}C NMR (150.92 MHz, DMSO-d_6): δ 161.97 (C×2), 139.72 (C×2), 131.96 (C×2), 127.76 (C×2).

[1,4] Dioxane-2,6-dione:[2]



^{1}H NMR (600.17 MHz, DMSO-d_6): δ 4.13 (s, 4H); ^{13}C NMR (150.92 MHz, DMSO-d_6): δ 172.16 (C×2), 68.07 (C×2).

Picture of the reaction vessel with MS4Å

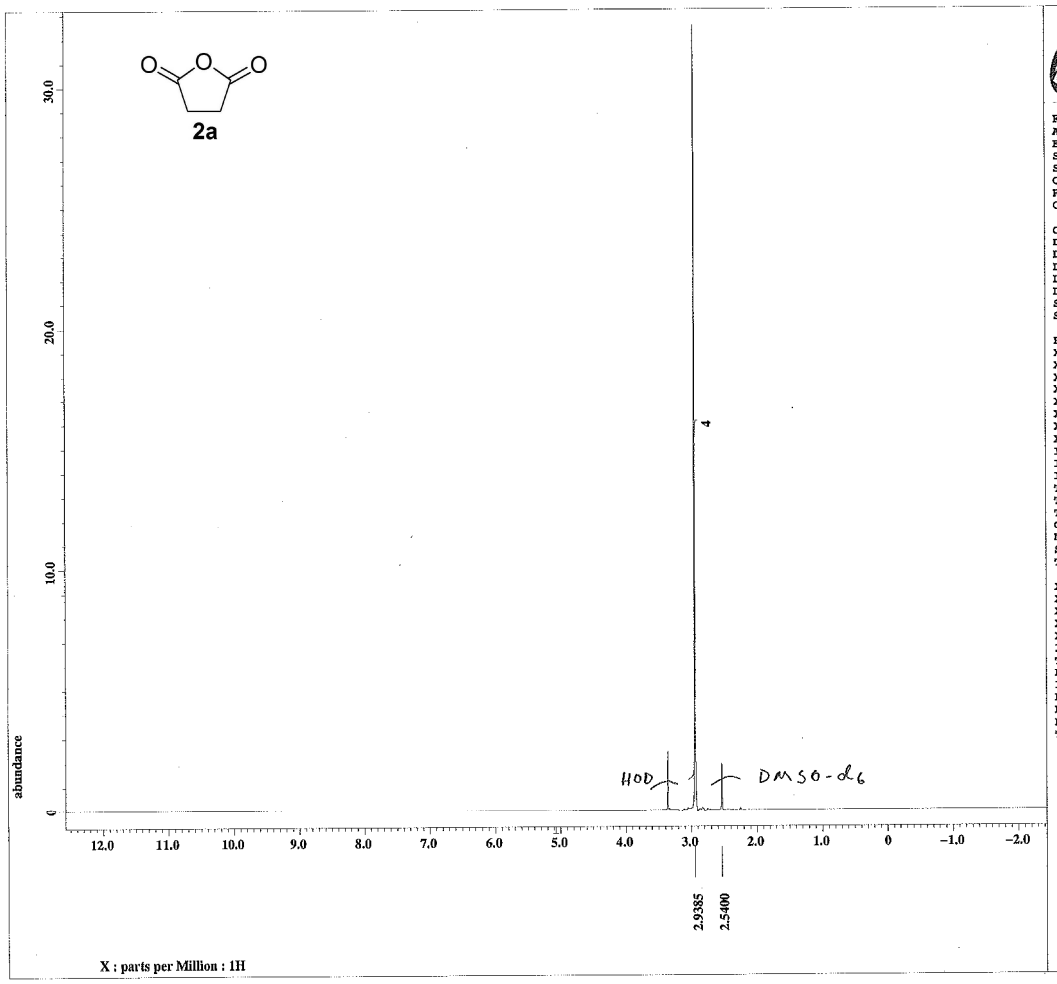


Molecular sieves (MS4Å, 0.2 or 2 g) wrapped in a filter paper was fixed at the upper part of the cylinder, which will be surmounted by a cooling part.

Catalyst powder and a stirrer bar in a reaction mixture.

References

1. C. Rober, F. de. Montigny, and C. M. Thomas, *ACS Catal,*, 2014, **4**, 3586–3589.
2. S. Paul, Y. Zhu, C. Romain, R. Brooks, P. K. Saini, and C. K. Williams, *Chem. Commun.*, 2015, **51**, 6459–6479.
3. D. Beck, W. Lv, M. Abdelmalak, C. B. Plescia, K. Agama, C. Marchand, Y. Pommier, and M. Cushman, *Bioorg. Med. Chem.*, 2016, **24**, 1469−1479.



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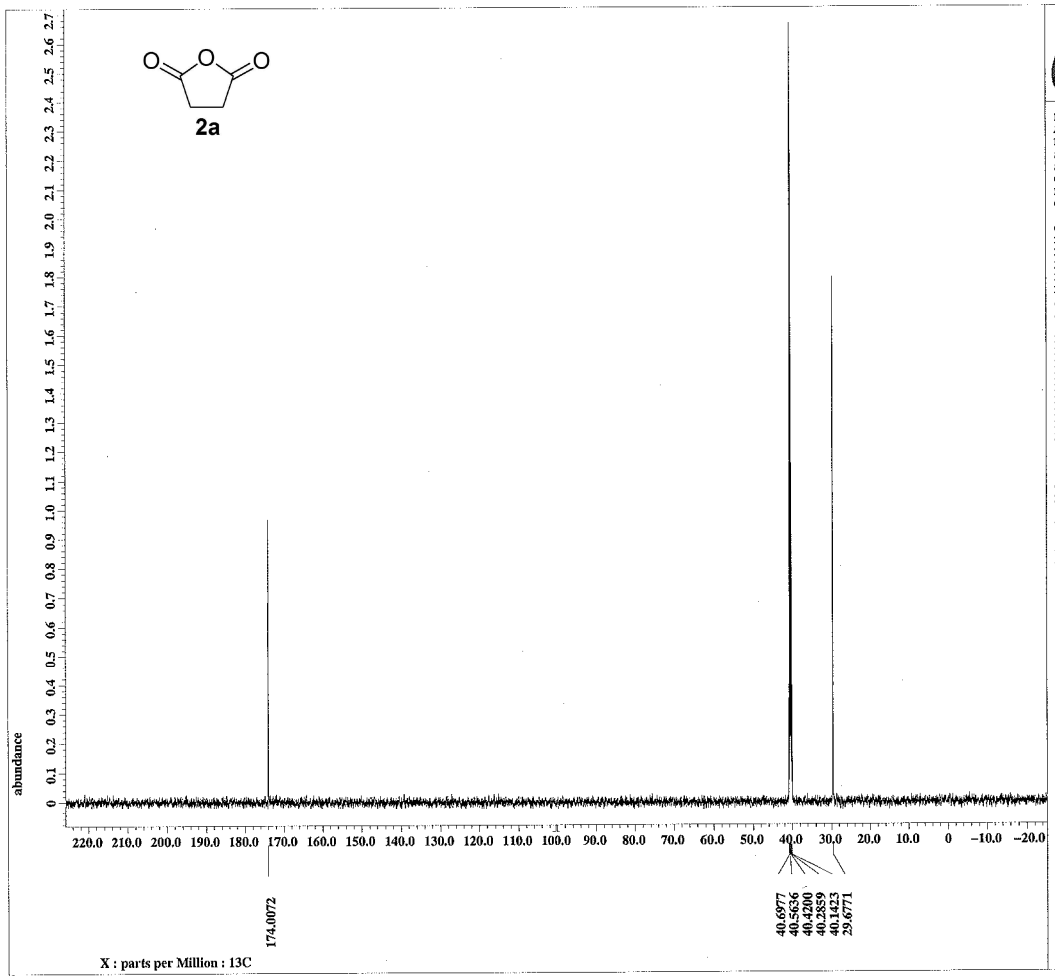

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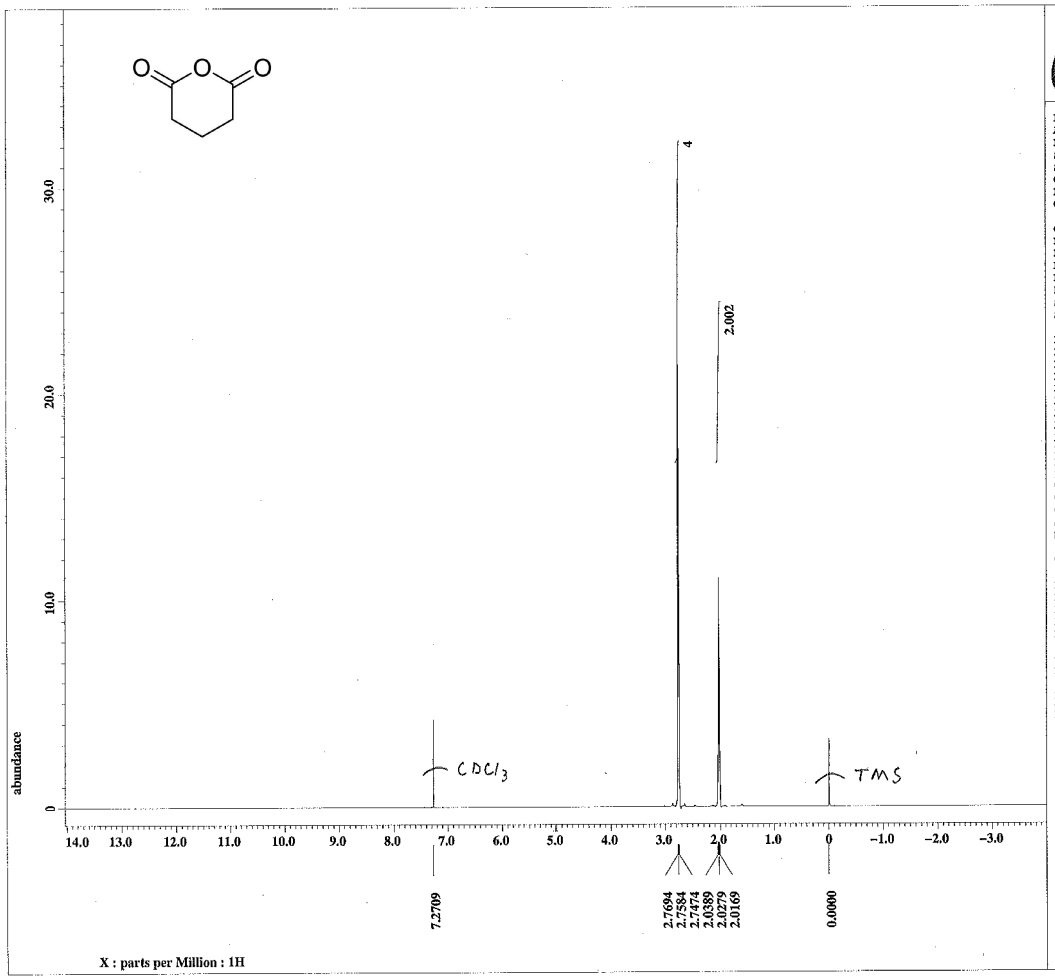

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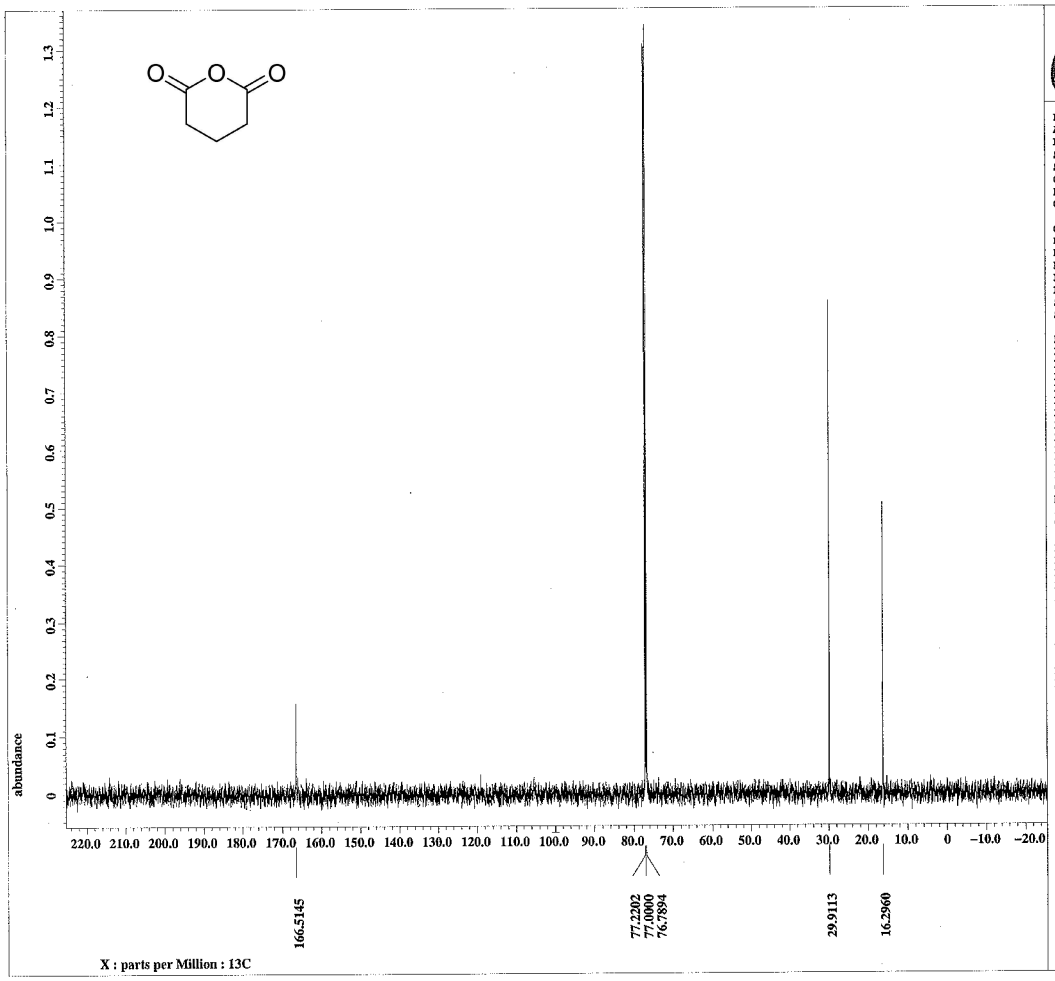

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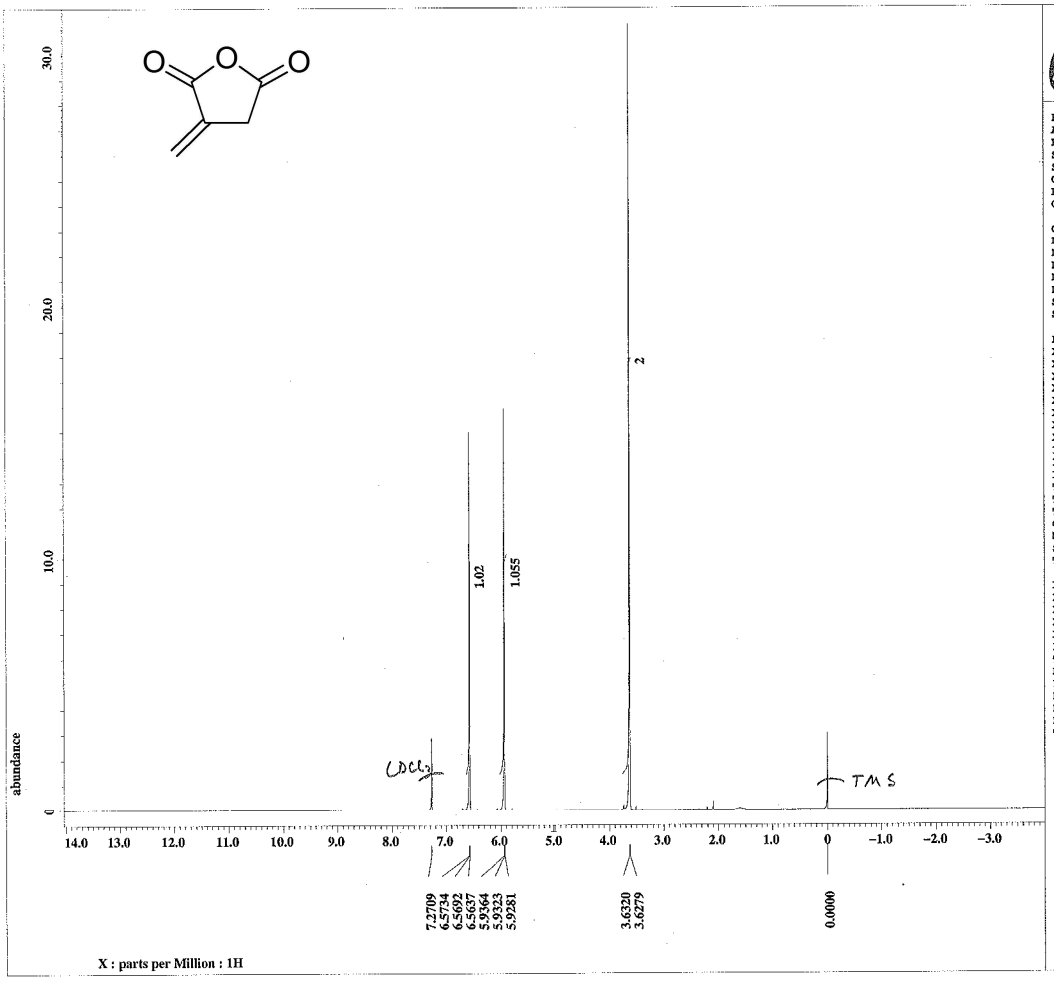

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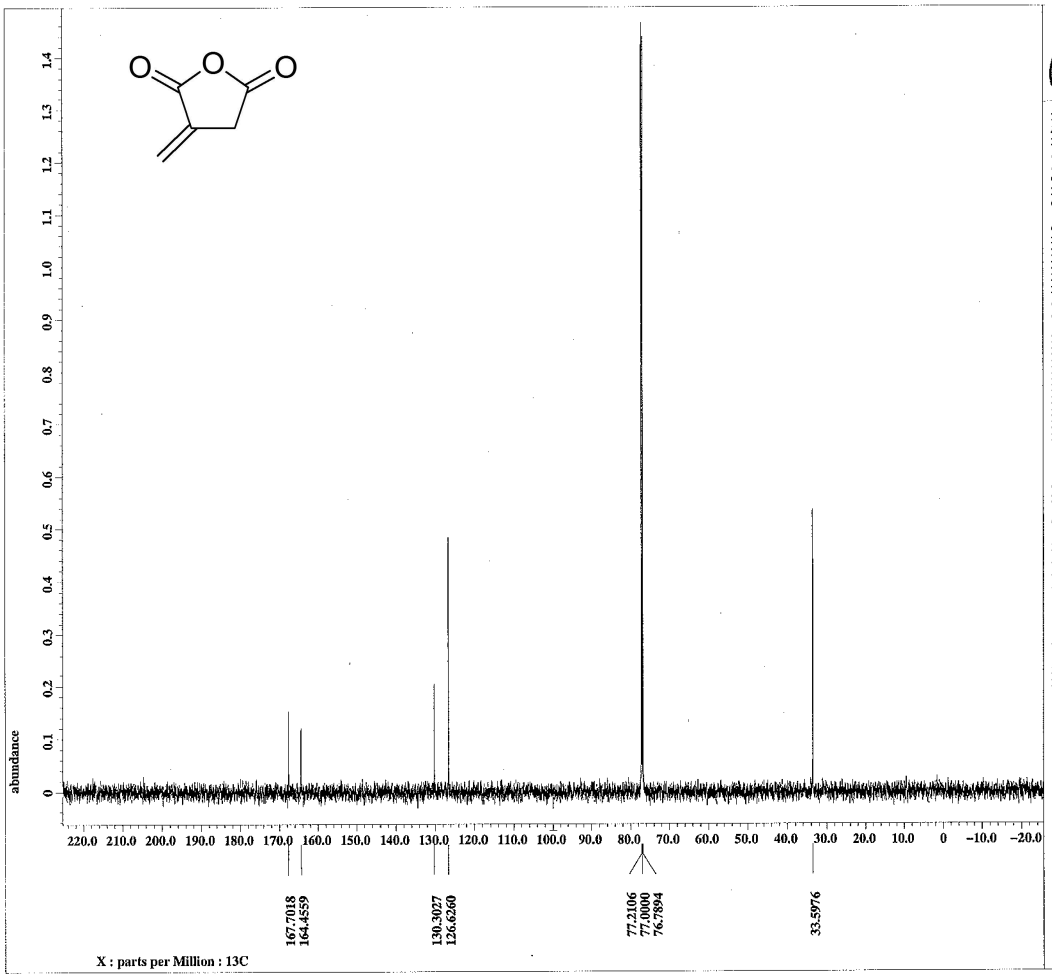

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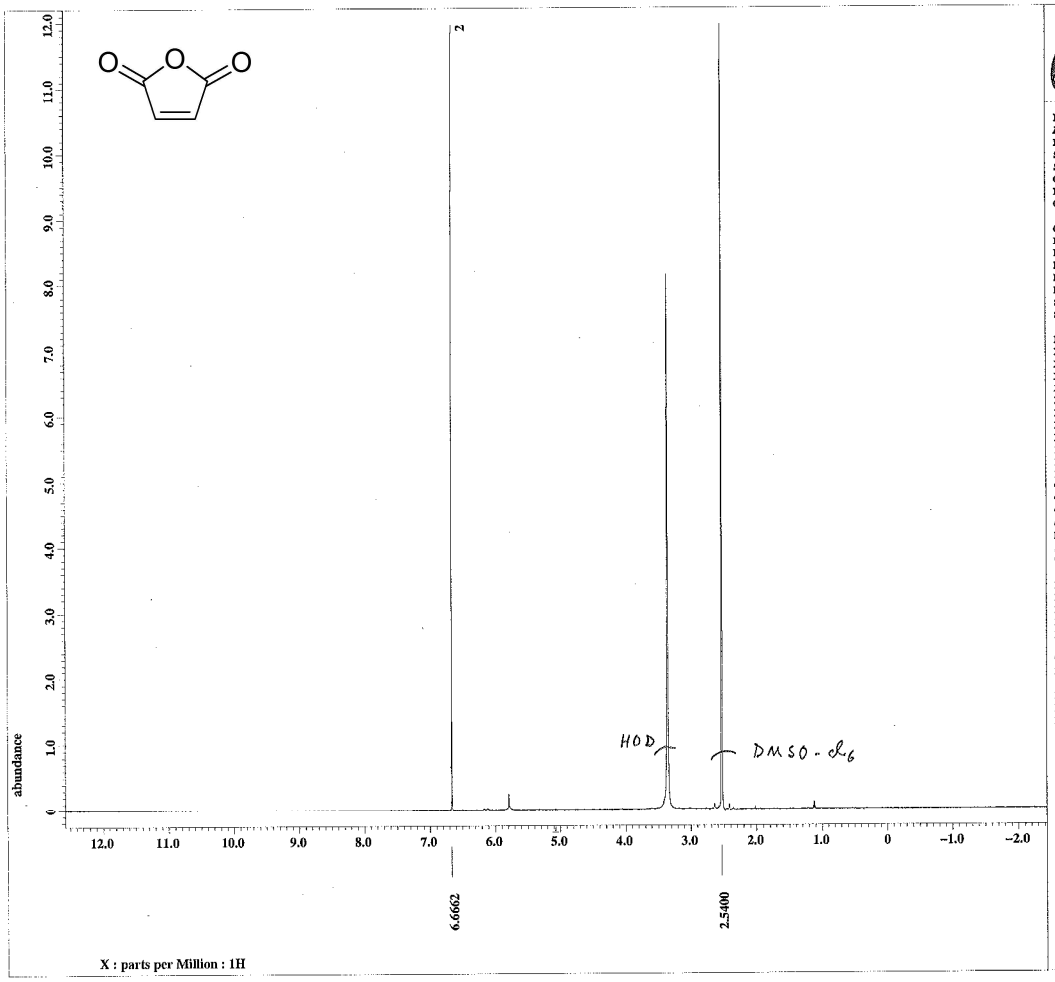

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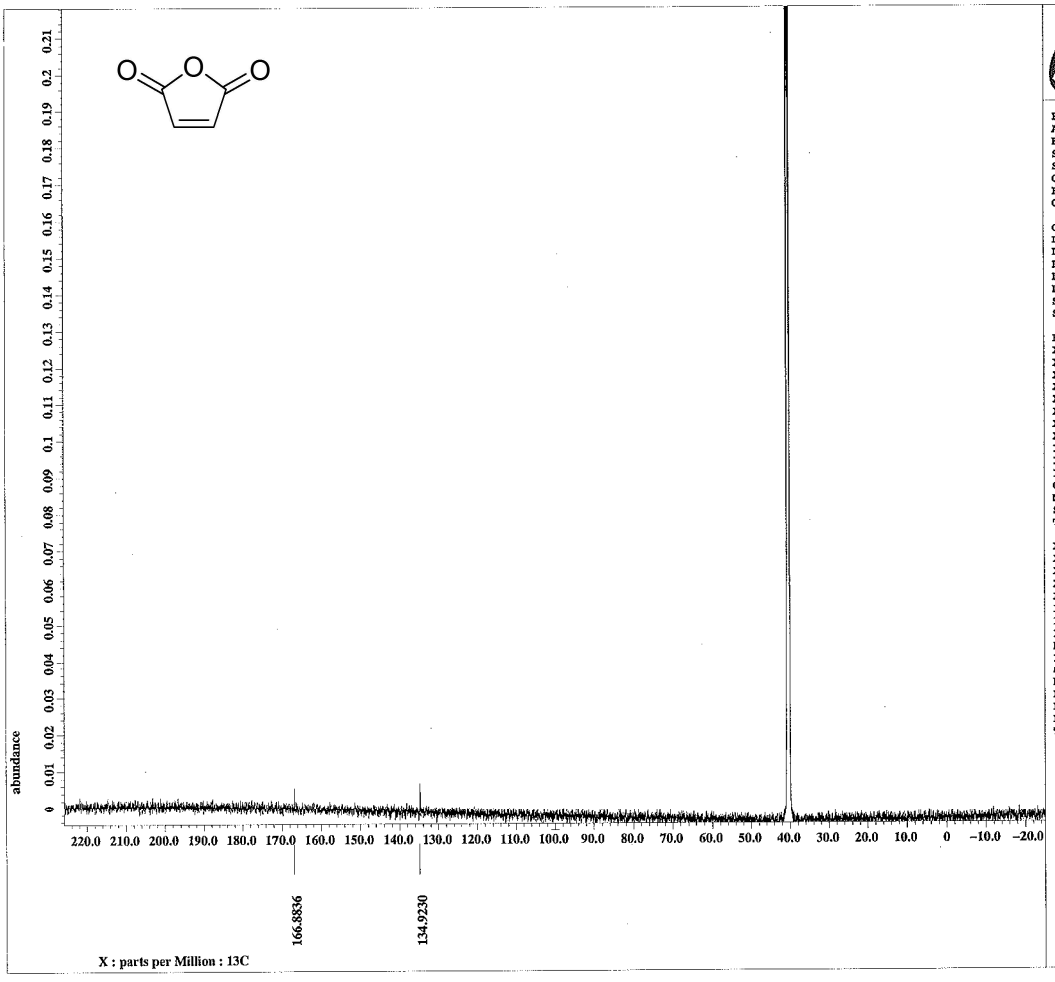
Relaxation_delay = 5[s]
Repetition_time = 6.4548992[s]
Temp_get = 23.1[dC]

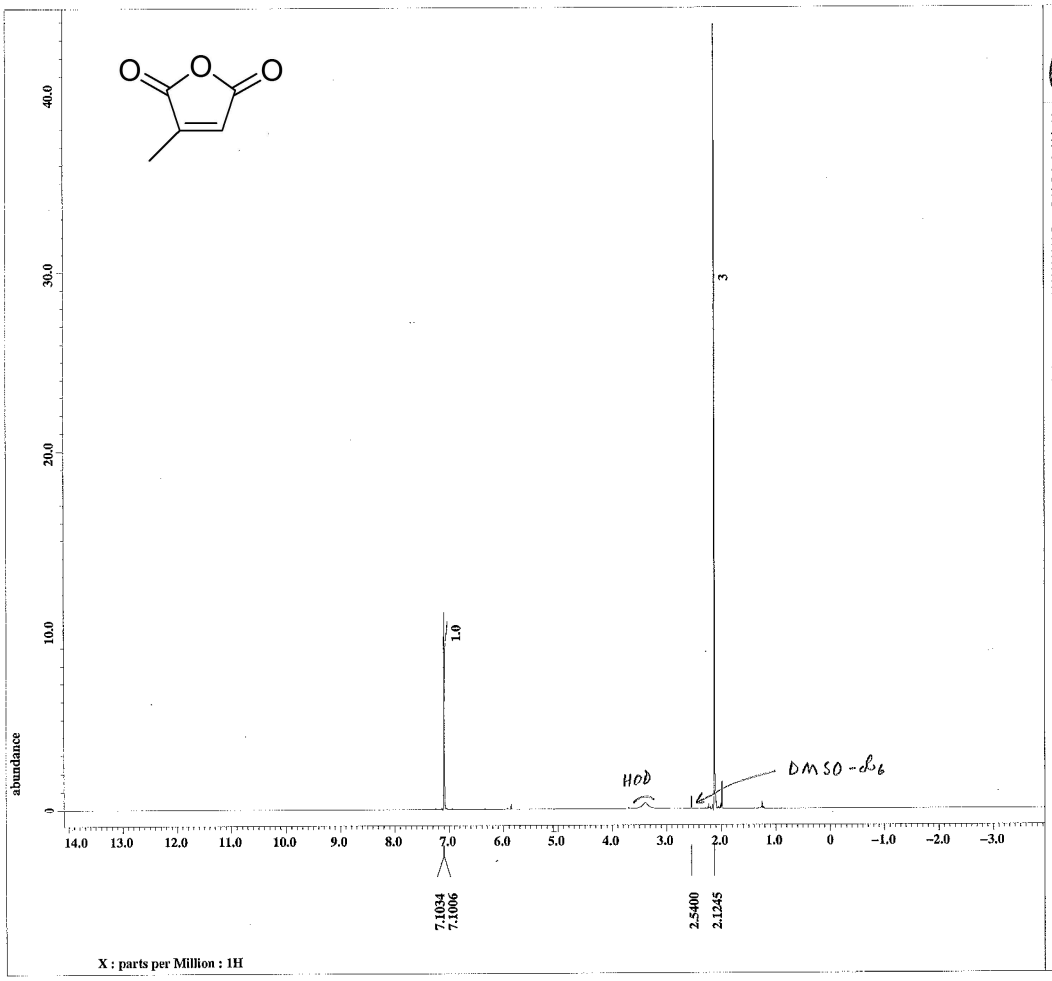


Filename = Exp-R-28-7b-carbon-3.
Author = delta
Experiment = single_pulse_dec
Sample_id = Exp-R-28-7b-carbon
Solvent = DMSO-D6
Creation_time = 8-FEB-2017 03:27:23
Revision_time = 17-FEB-2017 16:40:14
Current_time = 17-FEB-2017 16:40:19

Content = Exp-R-28-7b-carbon
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 600
Spectrometer = DELTA2_NMR

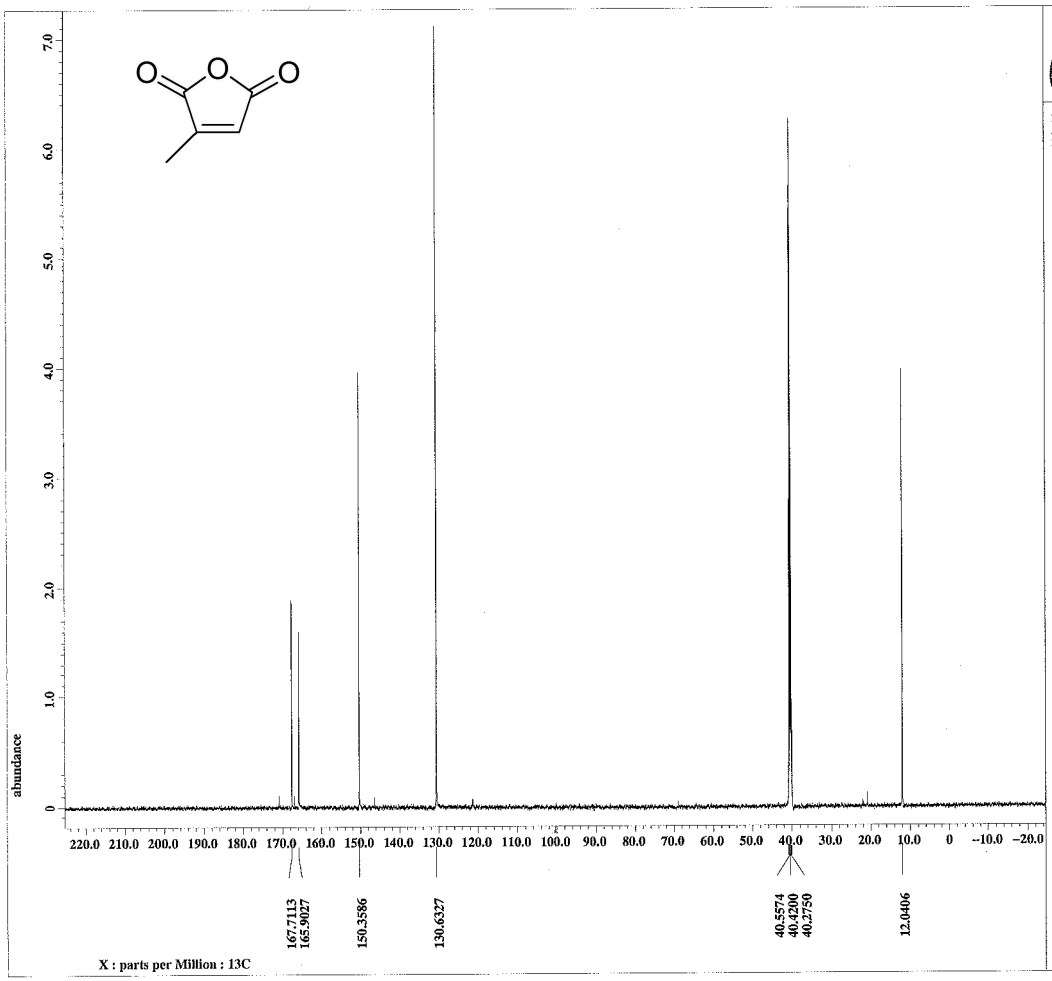
Field_strength = 14.09636928[T] (600[M
X_acq_duration = 0.69206016[s]
X_domain = 13C
X_freq = 150.91343039[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.44496109[Hz]
X_sweep = 47.34848485[kHz]
Irr_domain = 1H
Irr_freq = 600.1723046[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 10000
Total_scans = 10000

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[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.69206016[s]
Temp_get = 23.6[dC]



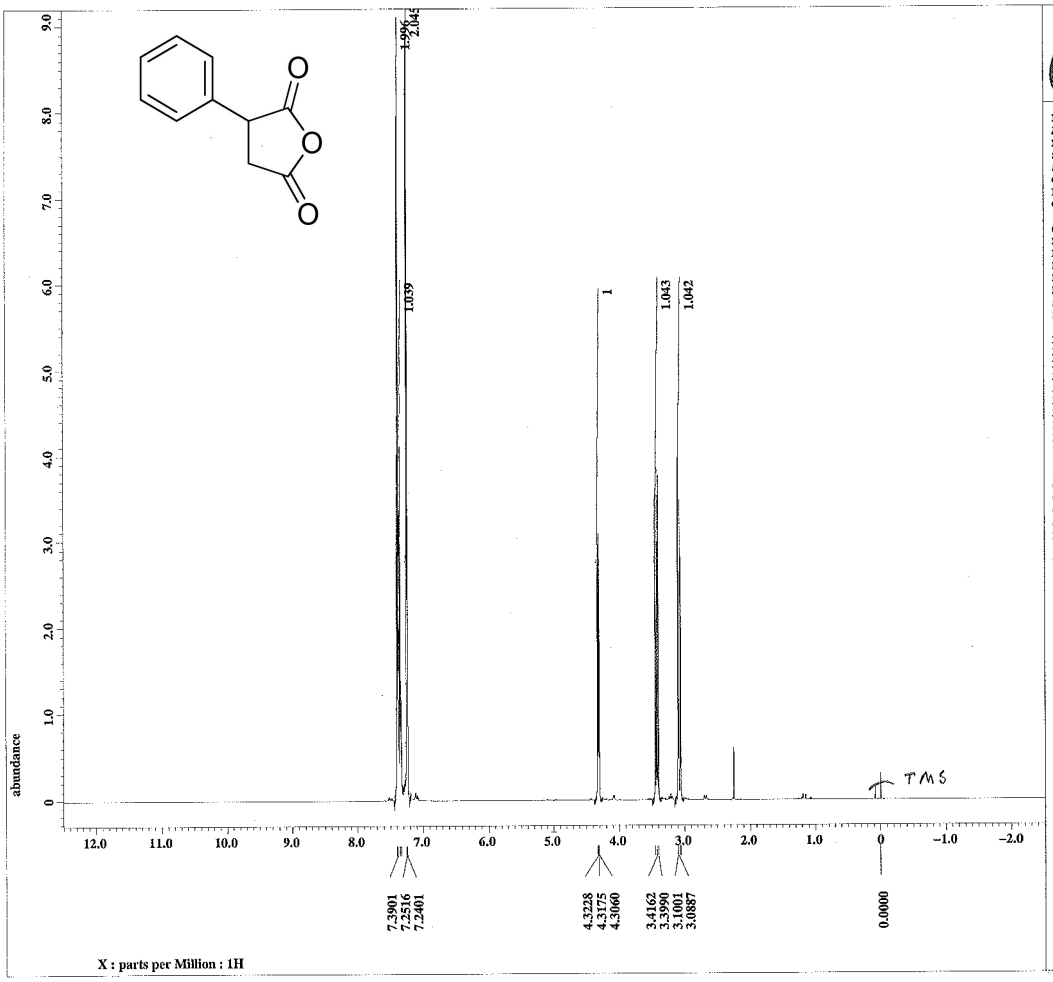
JEOL

```
Filename          = Exp-R-35-7a-proton-1.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-Ayub-375-6-proton
Solvent           = DMSO-D6
Creation_time     = 14-JUN-2016 11:36:34
Revision_time     = 20-FEB-2017 10:32:43
Current_time      = 20-FEB-2017 10:33:33

Content           = Exp-Ayub-375-6-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    = 1.21110528[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.82569205[Hz]
X_sweep           = 13.52813853[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        = 1.21110528[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        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 6.21110528[s]
Temp_get          = 21.3[dC]
```



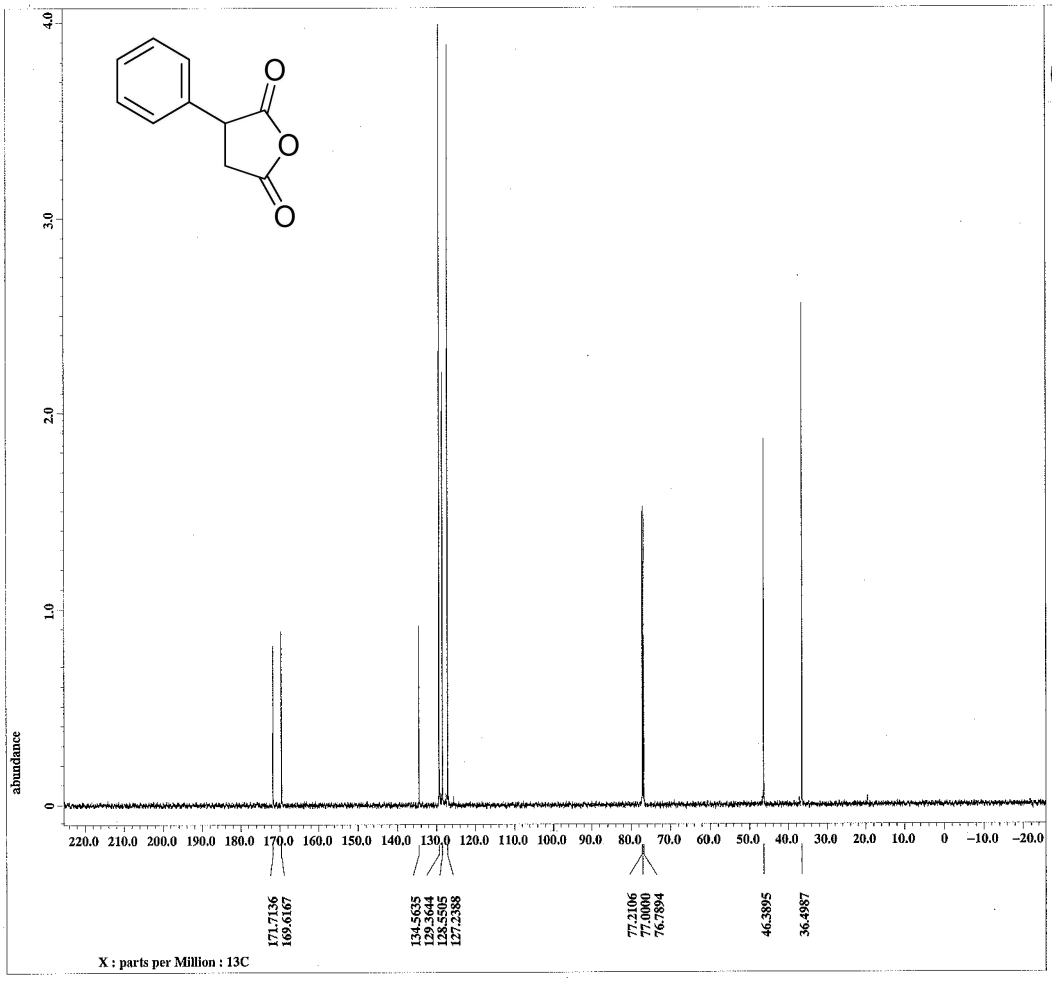
JEOL

```
Filename          = Exp-R-35-7a-carbon -1
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-Ayub-375-6-carbon
Solvent           = DMSO-D6
Creation_time     = 14-JUN-2016 12:01:46
Revision_time     = 20-FEB-2017 10:36:35
Current_time      = 20-FEB-2017 10:37:21

Content           = Exp-Ayub-375-6-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             = 430
Total_scans       = 430

X_90_width        = 10.9[us]
X_acq_time        = 0.868352[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        = 78
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 21.8[dC]
```



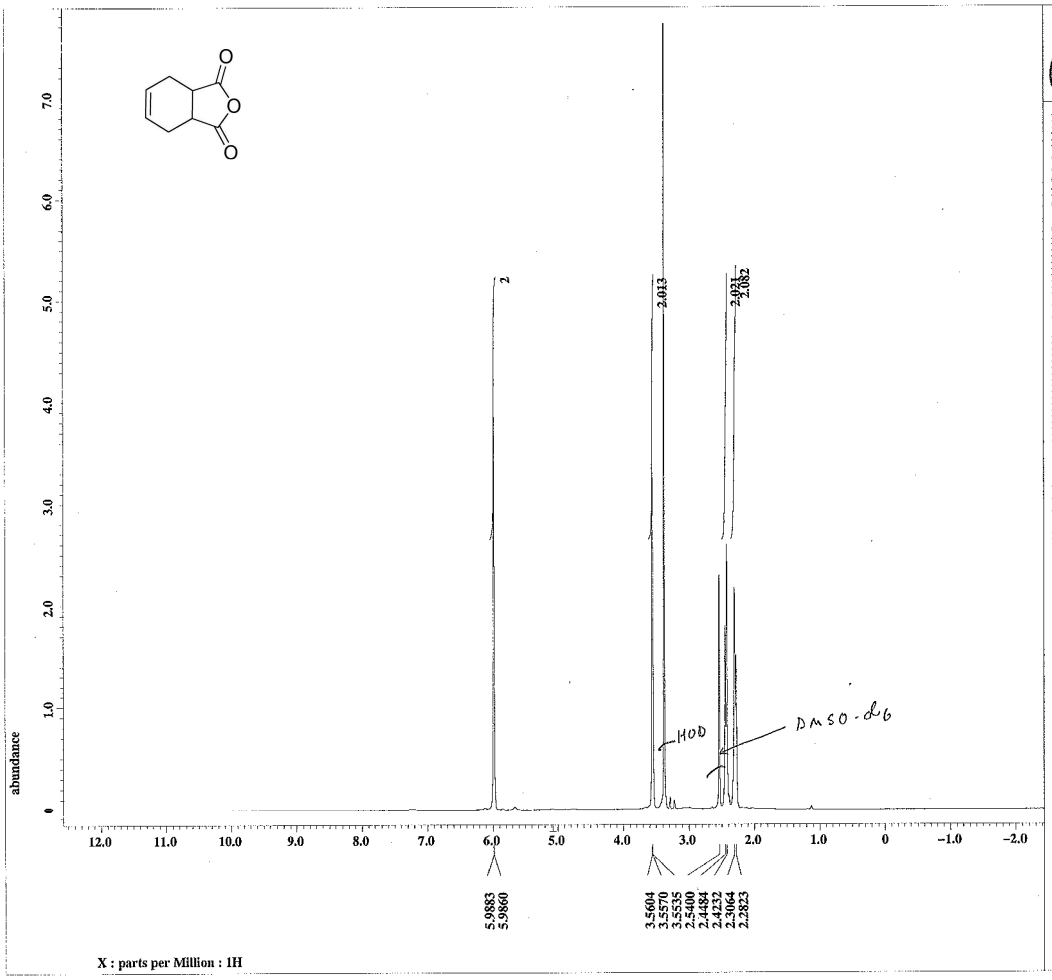
JEOL

```
Filename          = Exp-R-24-2-proton-5.j
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-R-24-2-proton
Solvent           = CHLOROFORM-D
Creation_time     =  7-FEB-2017 17:15:34
Revision_time     = 20-FEB-2017 10:40:31
Current_time      = 20-FEB-2017 10:40:52

Content           = Exp-R-24-2-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        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 23.1[dC]
```



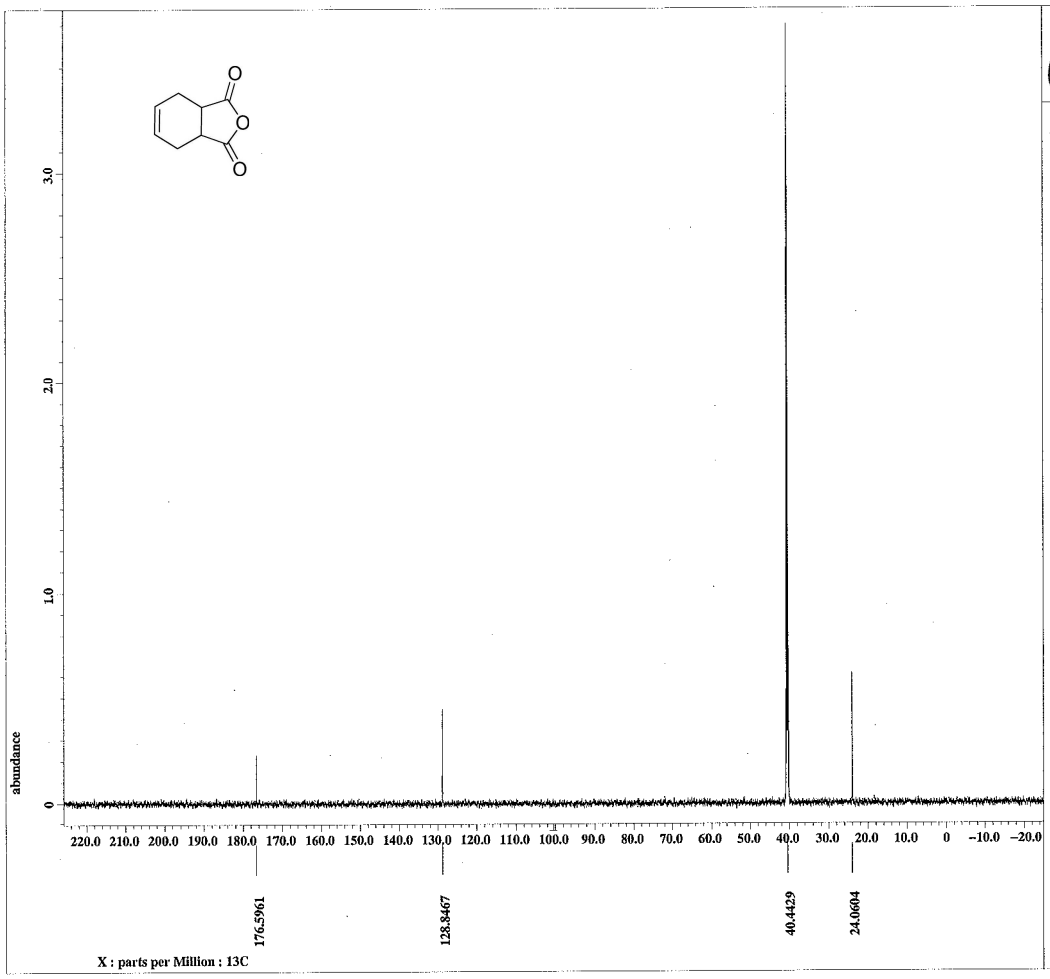
JEOL

```
Filename          = Exp-R-24-2-carbon-3.j
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-R-24-2-carbon
Solvent           = CHLOROFORM-D
Creation_time     =  7-FEB-2017 17:21:58
Revision_time     =  7-FEB-2017 18:26:57
Current_time      =  7-FEB-2017 18:27:49

Content           = Exp-R-24-2-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             = 127
Total_scans       = 127

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[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.69206016[s]
Temp_get          = 23.6[dC]
```



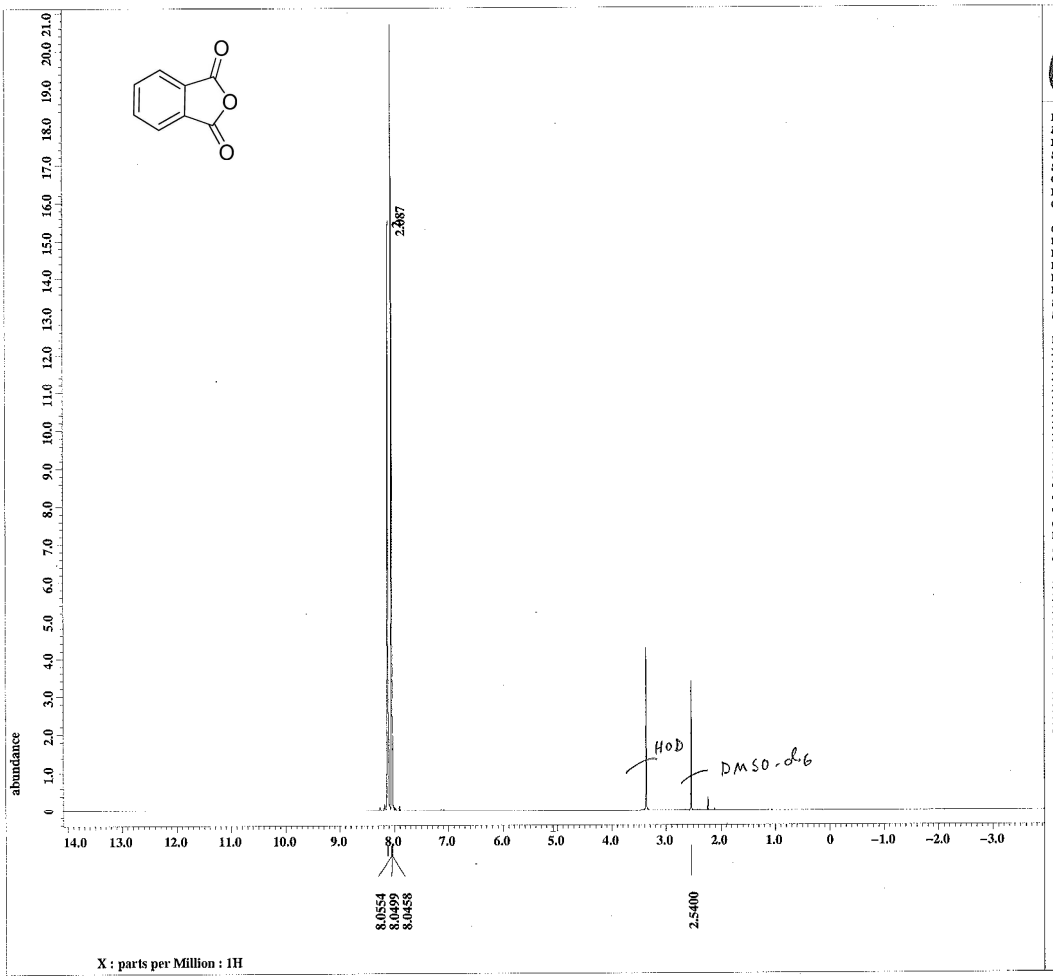
JEOL

Filename	= Exp-R-26-6a-proton-5.
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-R-26-6a-proton
Solvent	= DMSO-D6
Creation_time	= 6-JAN-2017 10:32:33
Revision_time	= 20-FEB-2017 10:46:10
Current_time	= 20-FEB-2017 10:46:19
Content	= Exp-R-26-6a-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	= 50
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 21[dC]



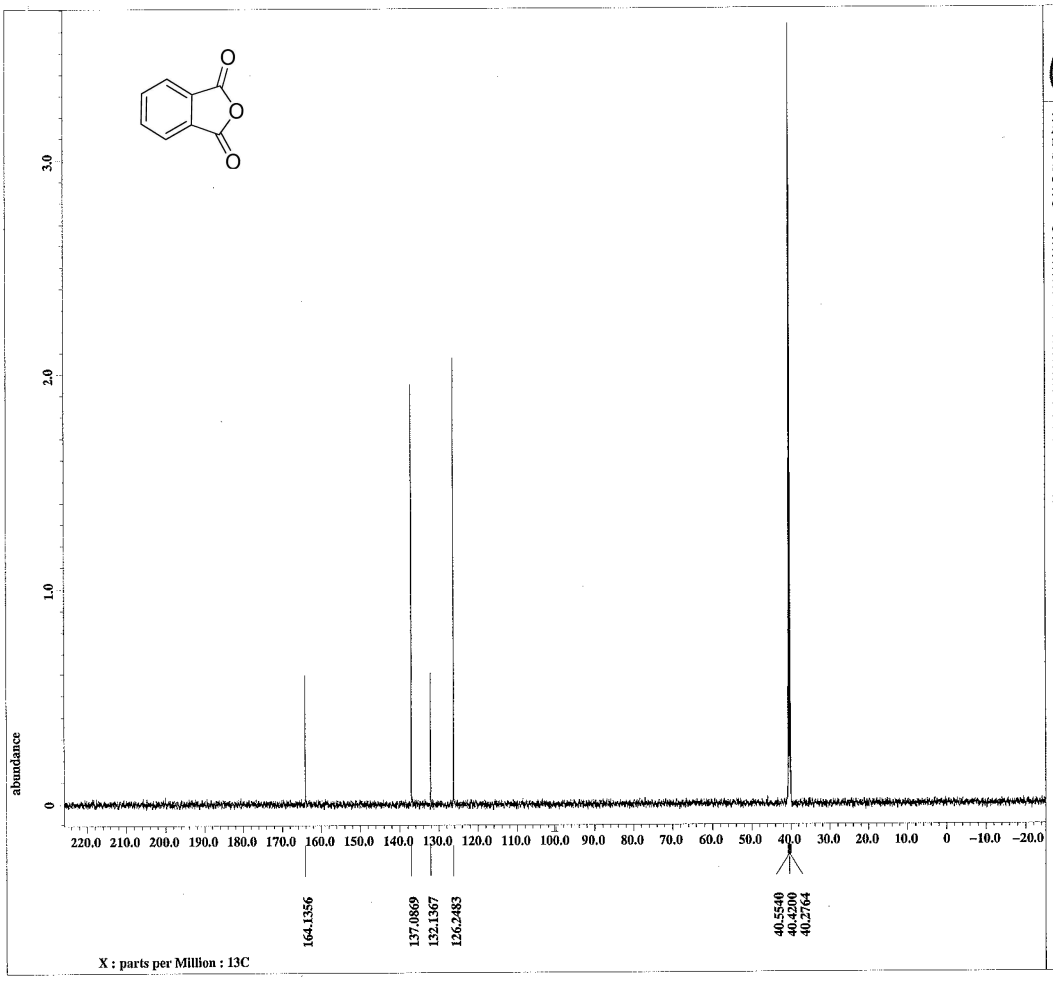
JEOL

Filename	= Exp-R-26-6a-carbon-4.
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-R-26-6a-carbon
Solvent	= DMSO-D6
Creation_time	= 6-JAN-2017 10:39:24
Revision_time	= 6-JAN-2017 11:50:25
Current_time	= 6-JAN-2017 11:50:30
Content	= Exp-R-26-6a-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	= 113.0
Total_scans	= 113.0
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]



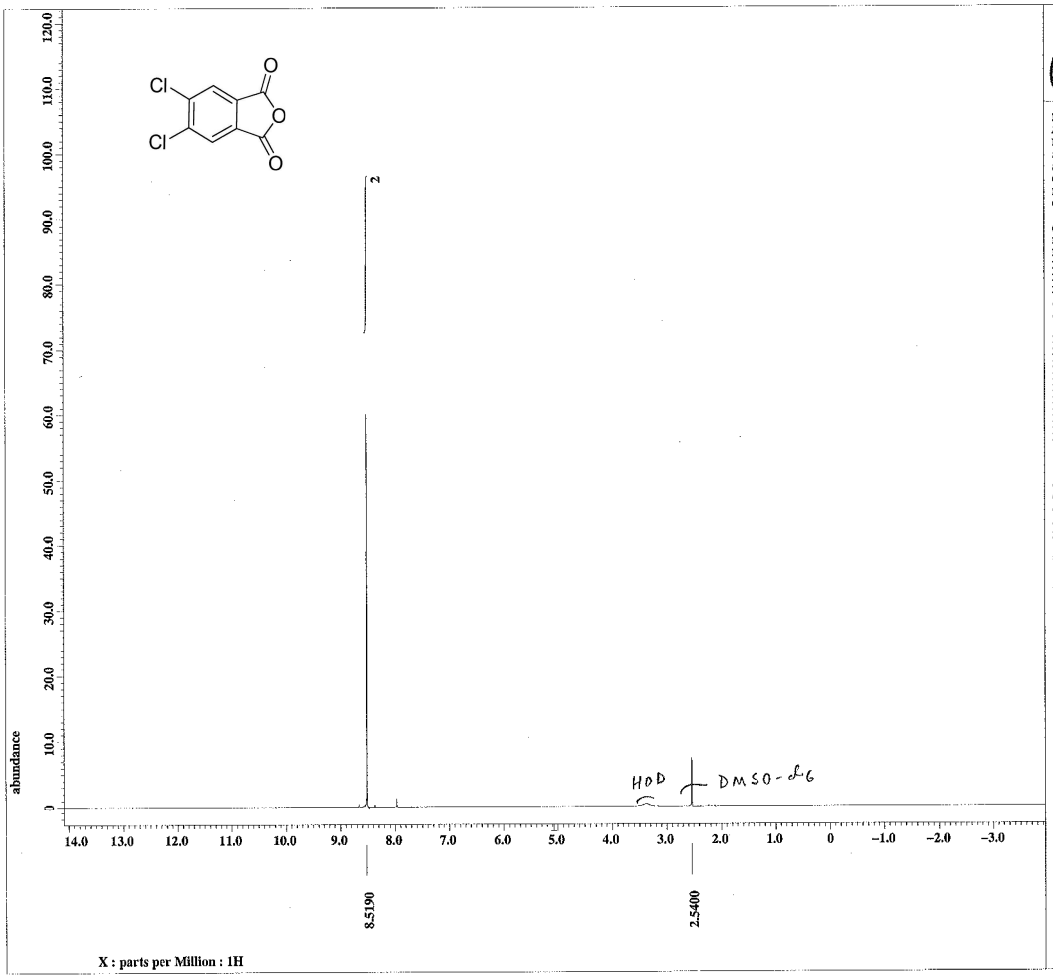
JEOL

```
Filename          = Exp-R-24-1a-proton-5.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-R-24-1a-proton
Solvent           = DMSO-D6
Creation_time     = 17-FEB-2017 13:19:03
Revision_time     = 20-FEB-2017 10:48:46
Current_time      = 20-FEB-2017 10:48:53

Content           = Exp-R-24-1a-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    = 1.21110528[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.82569205[Hz]
X_sweep           = 13.52813853[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        = 1.21110528[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        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 6.21110528[s]
Temp_get          = 23.9[dC]
```



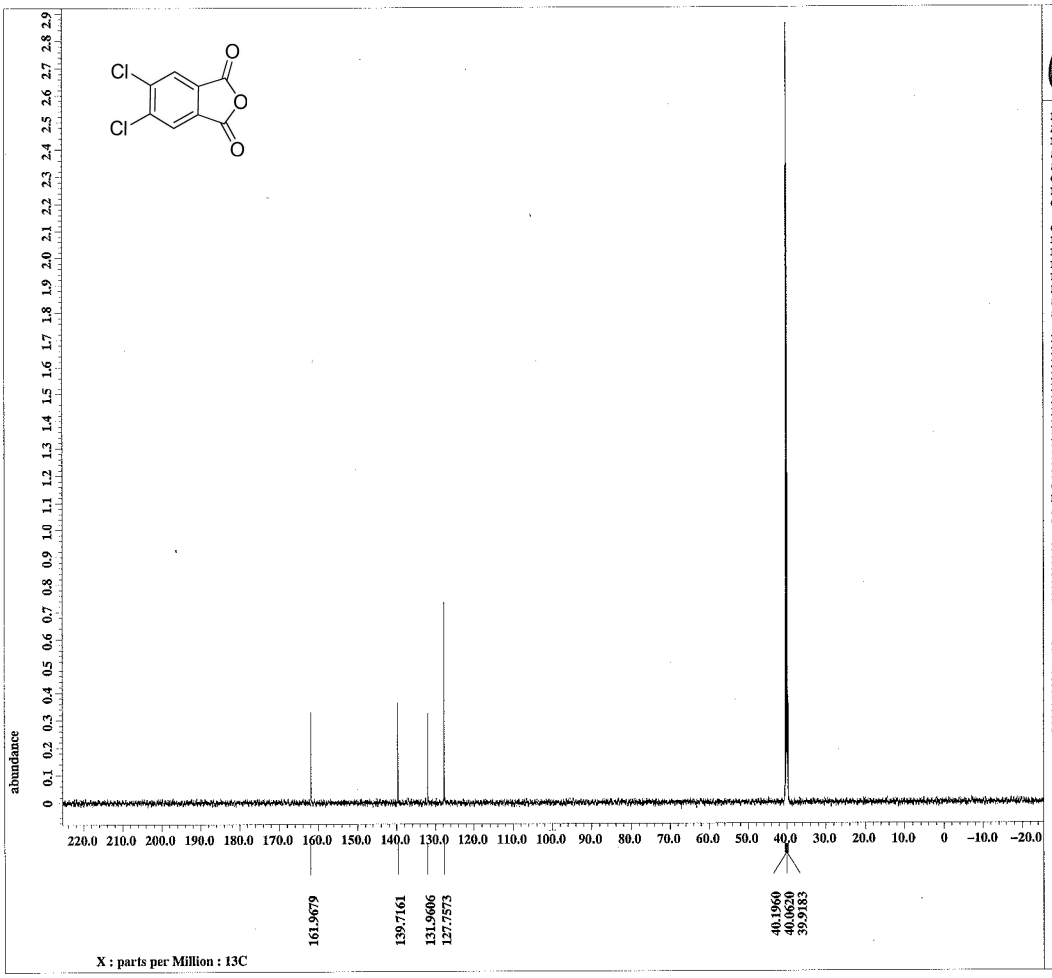
JEOL

```
Filename          = Exp-R-24-1a-carbon-3.
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-R-24-1a-carbon
Solvent           = DMSO-D6
Creation_time     = 17-FEB-2017 13:24:36
Revision_time     = 17-FEB-2017 14:23:38
Current_time      = 17-FEB-2017 14:23:48

Content           = Exp-R-24-1a-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             = 89
Total_scans       = 89

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          = 24.5[dC]
```



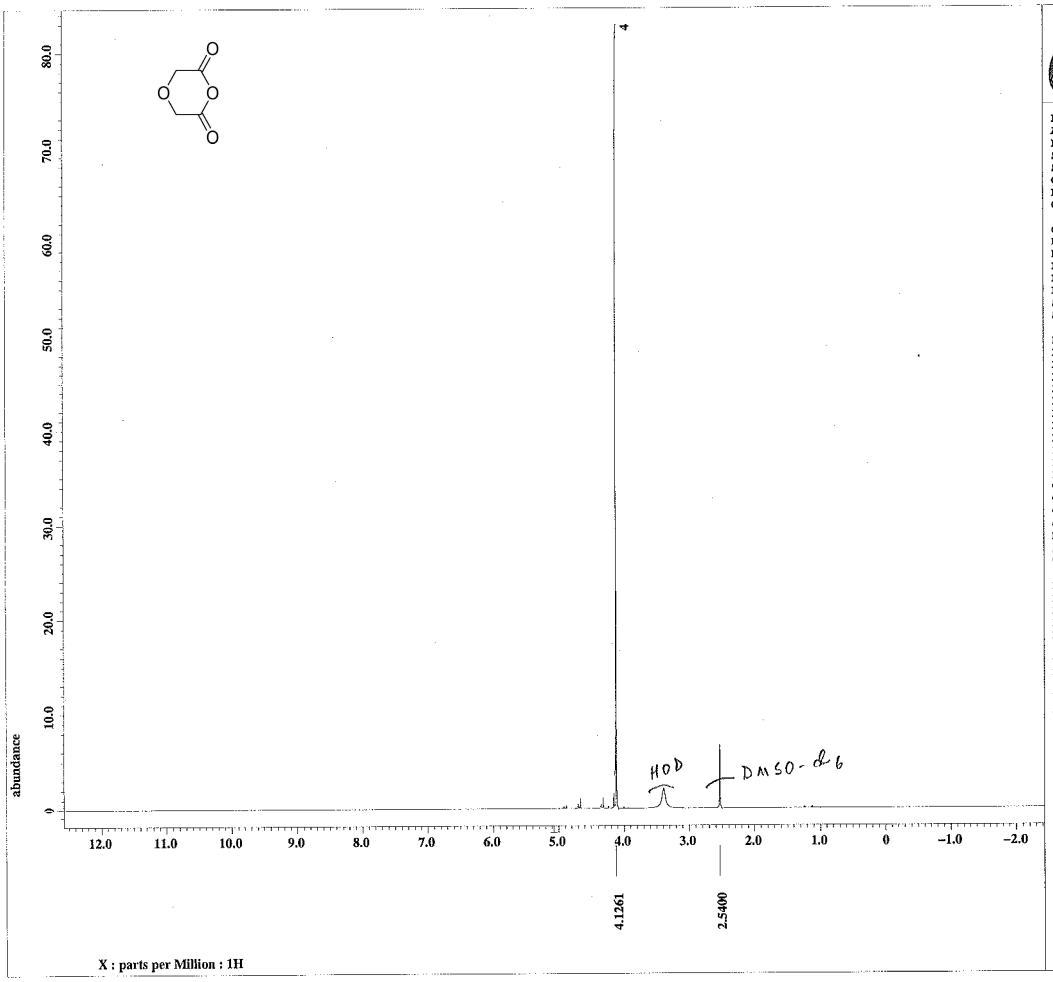
JEOL

```
Filename          = Exp-R-26-2d-proton-5.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-R-26-2d-proton
Solvent           = DMSO-D6
Creation_time     = 17-FEB-2017 13:46:55
Revision_time     = 17-FEB-2017 19:38:31
Current_time      = 17-FEB-2017 19:38:43

Content           = Exp-R-26-2d-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    = 1.21110528[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.82569205[Hz]
X_sweep           = 13.52813853[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        = 1.21110528[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        = 56
Relaxation_delay  = 5[s]
Repetition_time   = 6.21110528[s]
Temp_get          = 23.8[dC]
```



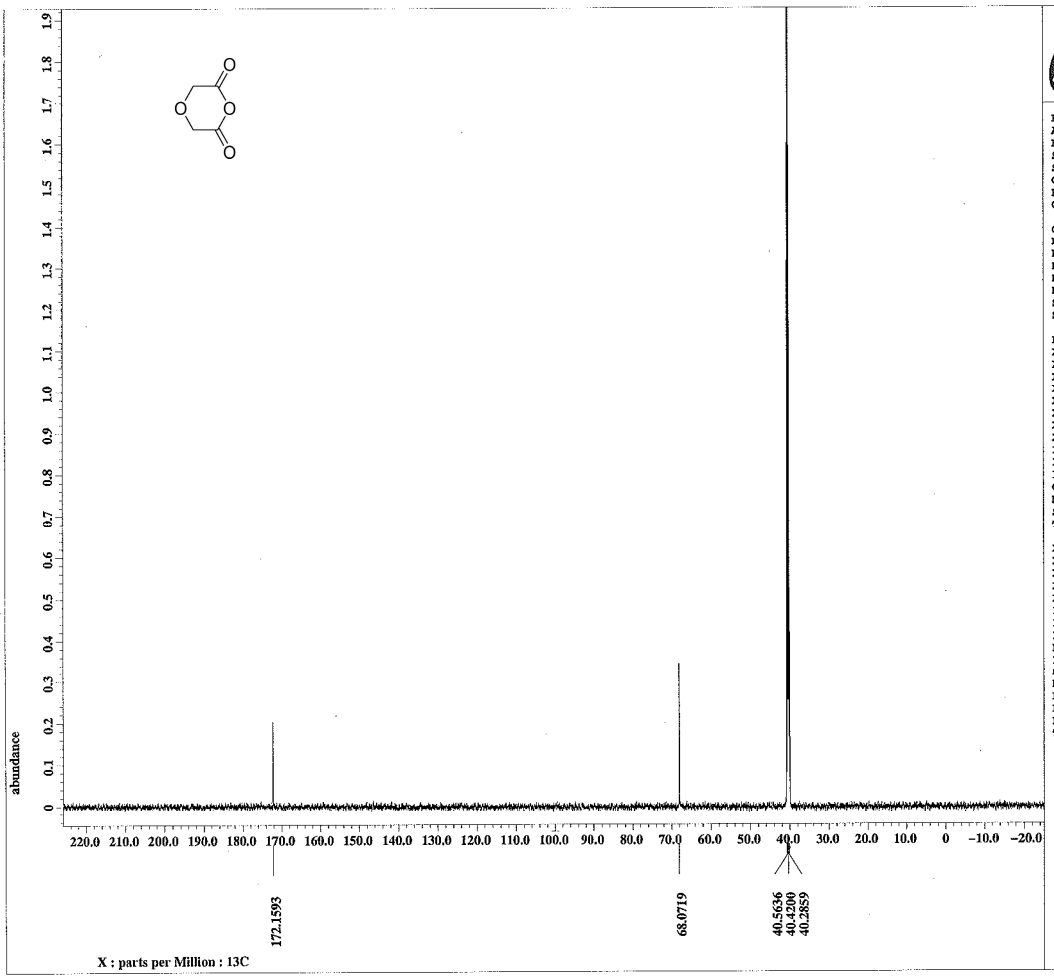
JEOL

```
Filename          = Exp-R-26-2d-carbon-2.
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-R-26-2d-carbon
Solvent           = DMSO-D6
Creation_time     = 17-FEB-2017 13:53:55
Revision_time     = 17-FEB-2017 14:52:27
Current_time      = 17-FEB-2017 14:52:33

Content           = Exp-R-26-2d-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           = TRUE
Mod_return        = 1
Scans             = 116.0
Total_scans       = 116.0

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        = 58
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.19206016[s]
Temp_get          = 24.5[dC]
```



JEOL

```
Filename          = Exp-R-26-4a-proton-5.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-R-26-4a-proton
Solvent           = DMSO-D6
Creation_time     = 12-JAN-2017 10:06:35
Revision_time     = 20-FEB-2017 10:51:39
Current_time      = 20-FEB-2017 10:52:35

Content           = Exp-R-26-4a-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        = 56
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.5[dC]
```



JEOL

```
Filename          = Exp-R-26-4a-carbon-3.
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-R-26-4a-carbon
Solvent           = DMSO-D6
Creation_time     = 12-JAN-2017 10:29:29
Revision_time     = 17-FEB-2017 19:43:32
Current_time      = 17-FEB-2017 19:43:50

Content           = Exp-R-26-4a-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             = 415
Total_scans       = 415

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          = 21.2[dC]
```