Electronic Supplementary Material (ESI) for RSC Advances.

Supplementary Information

Synthesis of novel Entecavir analogues having 4′-cyano-6″-fluoromethylenecyclopentene skeletons, as an aglycone moiety as highly potent and long-acting anti Hepatitis B virus agent

Hiroki Kumamoto[†], Nobuyo Higashi-Kuwata [‡], Sanae Hayashi[§], Debananda Das[#], Haydar Bulut[#], Ryoh Tokuda [∥], Shuhei Imoto [∥], Kengo Onitsuka [‡], Yuka Honda[⊥], Yuki Odanaka[⊥], Satoko Shinbara-Matsubayashi, Kazuhiro Haraguchi[†], Yasuhito Tanaka[§] and Hiroaki Mitsuya [‡,#,∞]

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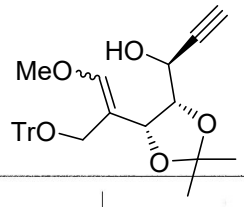
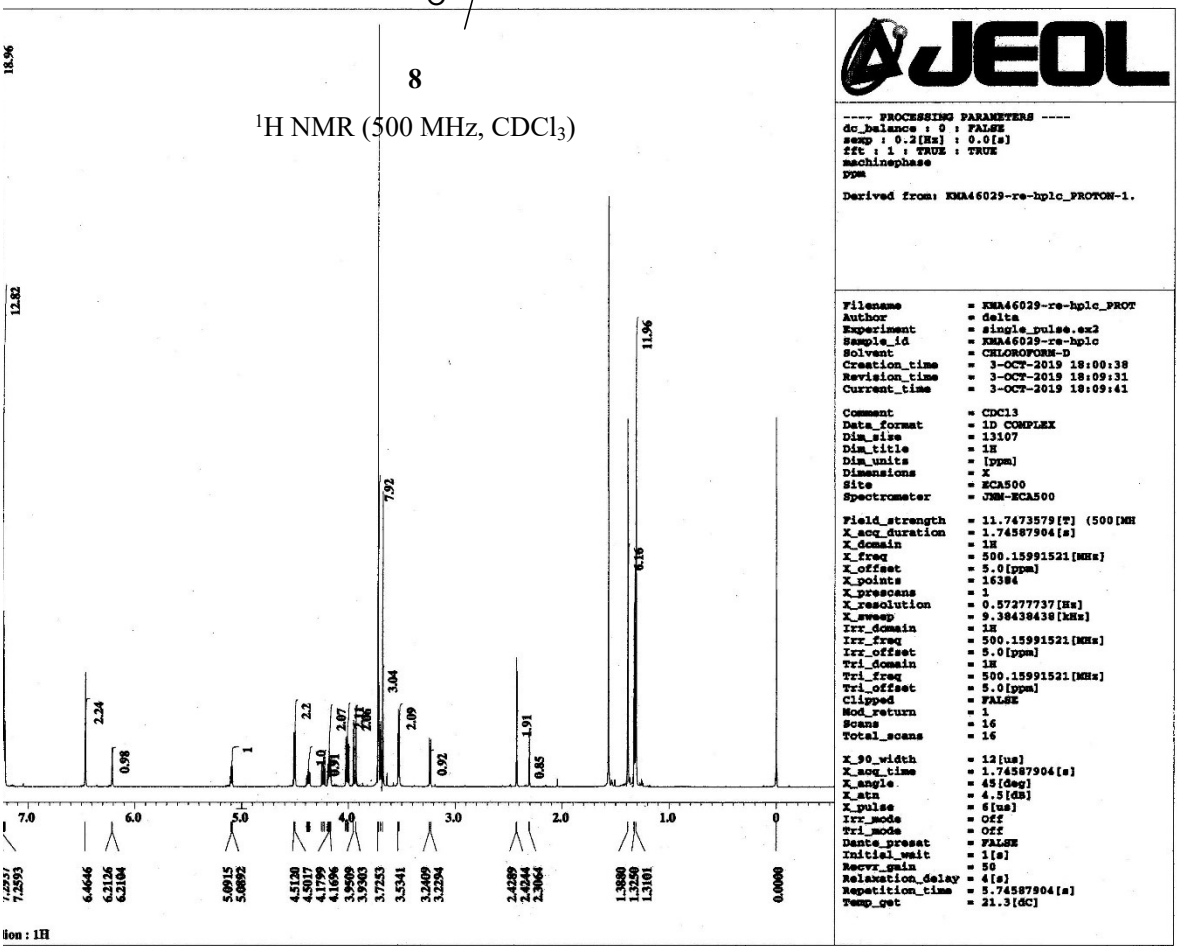
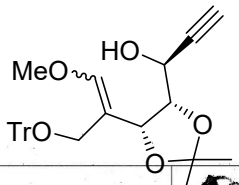


8

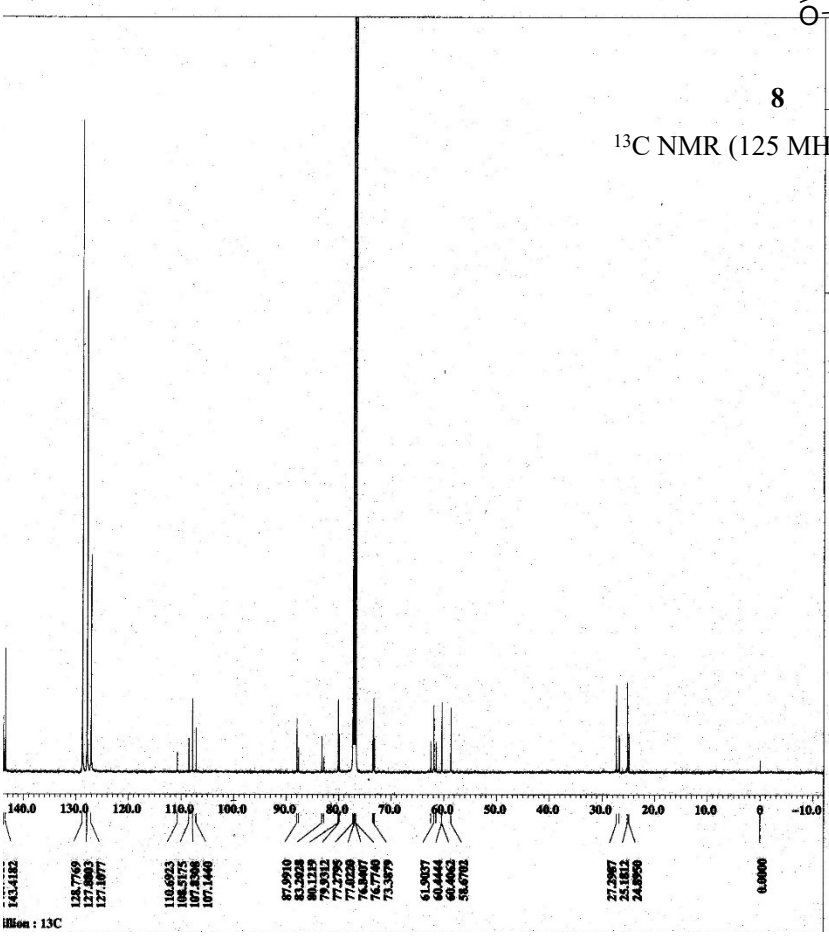
^{1}H NMR (500 MHz, $CDCl_3$)

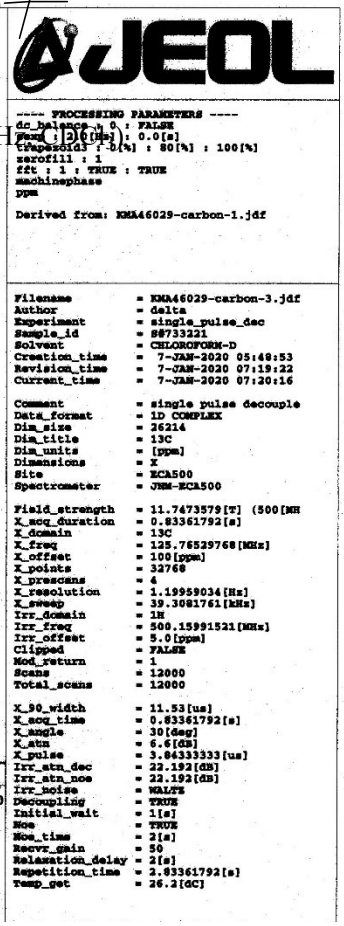


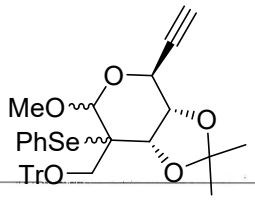


8

^{13}C NMR (125 MHz, CDCl$_3$)

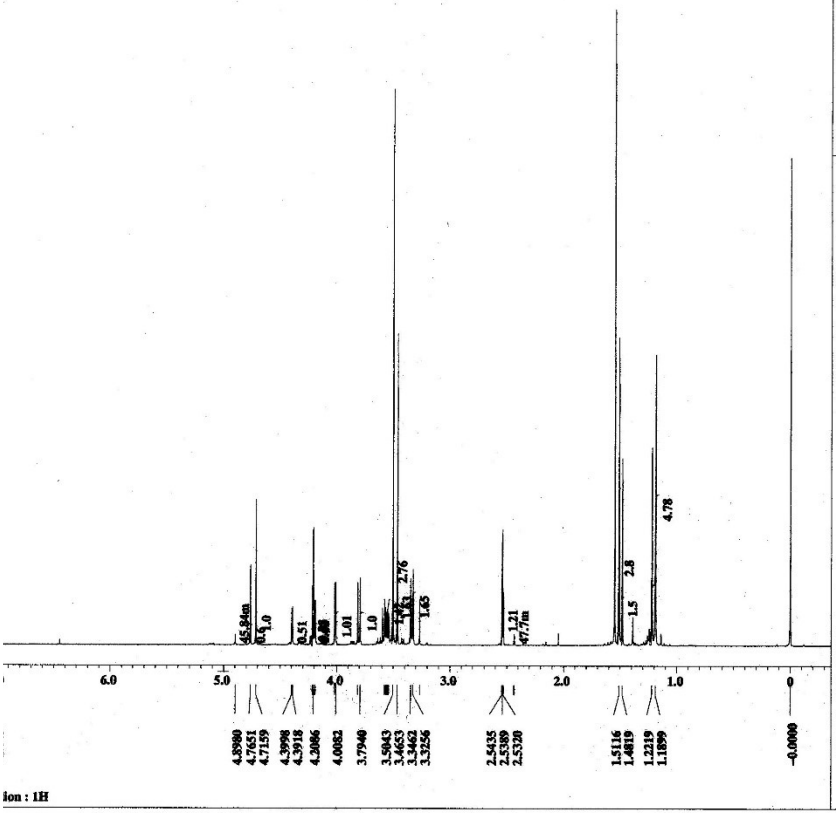


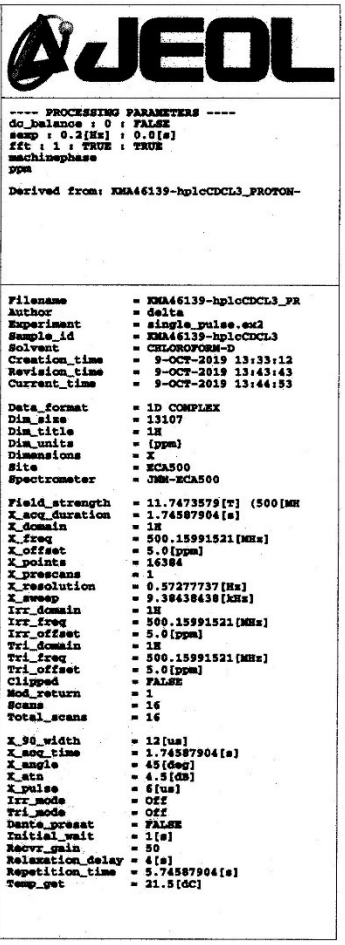


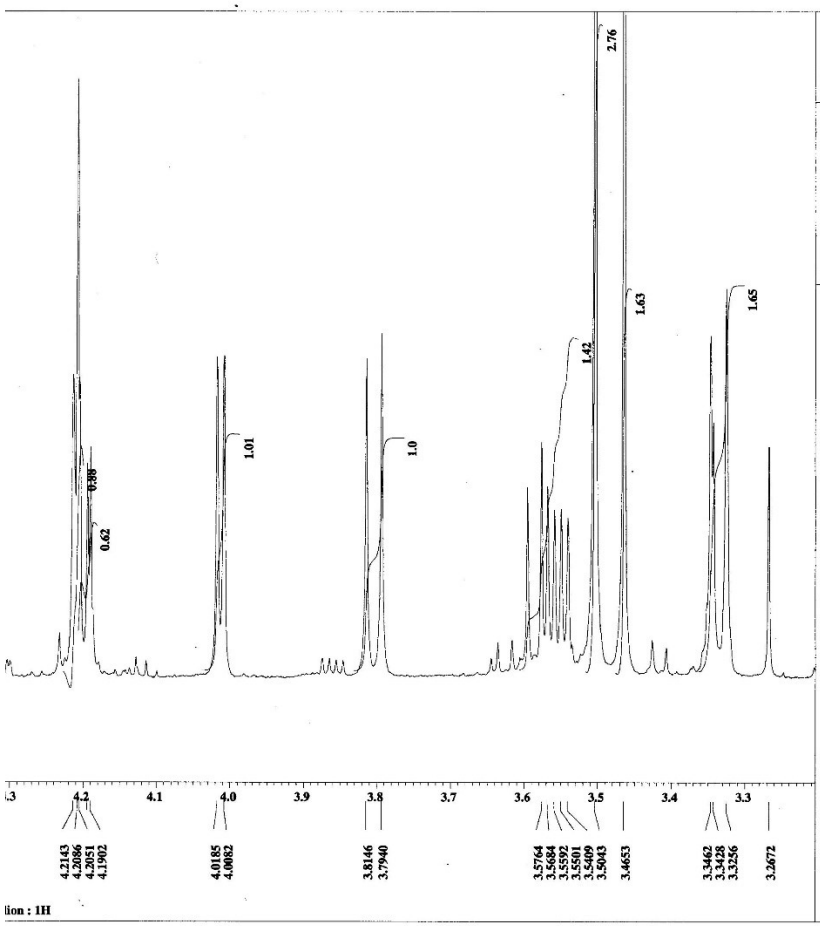


9

^{1}H NMR (500 MHz, $CDCl_3$)



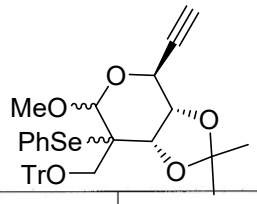




JEOL

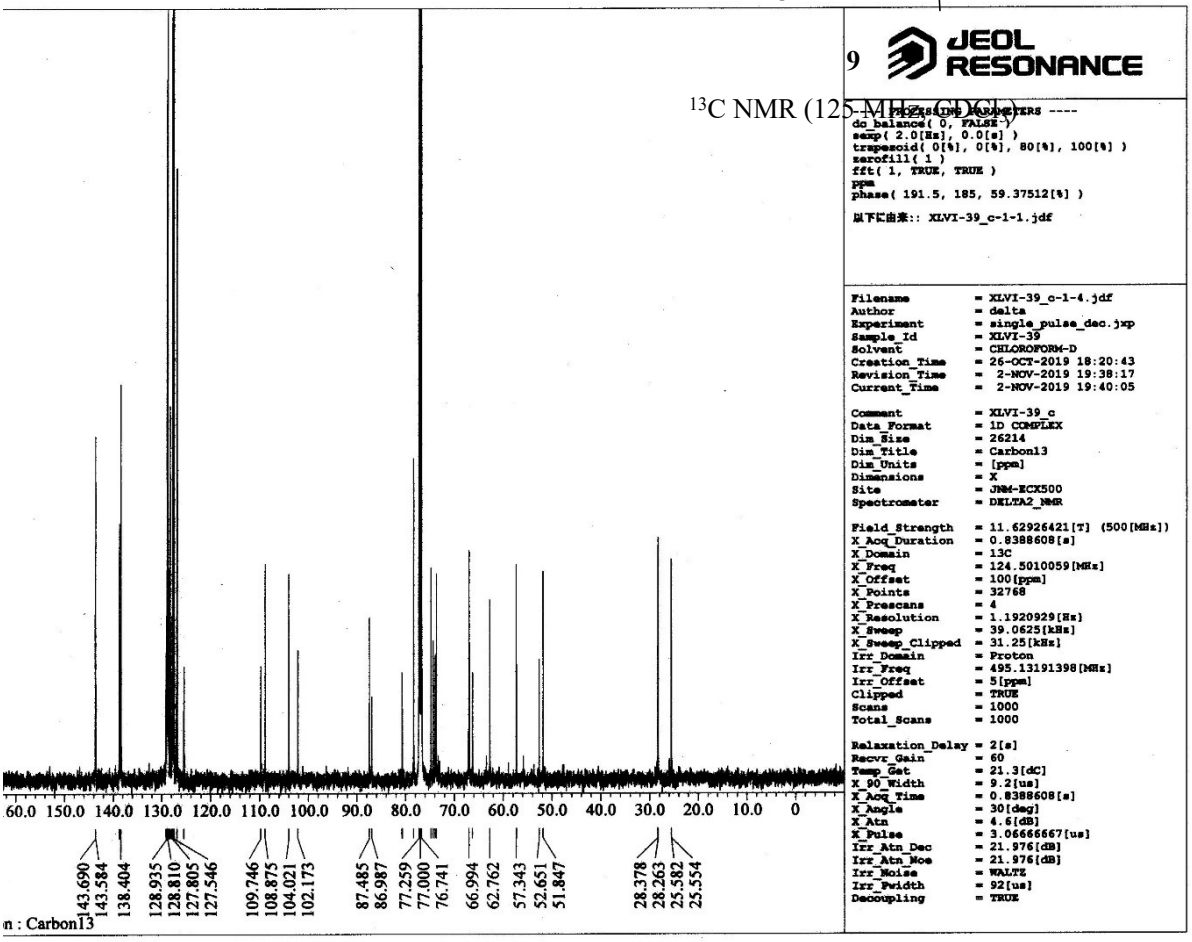
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46139-hplcCDCL3_PROTON-
```

```
Filename          = KMA46139-hplcCDCL3_PR
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = KMA46139-hplcCDCL3
Solvent           = CHLOROFORM-D
Creation_time     =  9-OCT-2019 13:33:12
Revision_time     =  9-OCT-2019 13:43:43
Current_time      =  9-OCT-2019 13:45:43

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 6[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 21.5[dC]
```



9

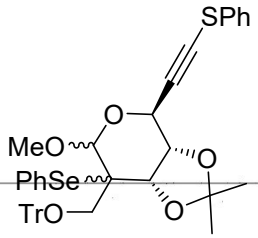
^{13}C NMR (125 MHz, CDCl$_3$)



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
expo(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
ppm
phase(191.5, 185, 59.37512[%])

以下に由来:: XLVI-39_c-1-1.jdf

Filename	= XLVI-39_c-1-4.jdf
Author	= delta
Experiment	= single_pulse_dec.jxp
Sample_Id	= XLVI-39
Solvent	= CHLOROFORM-D
Creation_Time	= 26-OCT-2019 18:20:43
Revision_Time	= 2-NOV-2019 19:38:17
Current_Time	= 2-NOV-2019 19:40:05
Comment	= XLVI-39_c
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECX500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
X_Sweep_Clipped	= 31.25[kHz]
Irr_Domain	= Proton
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 1000
Total_Scans	= 1000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 21.3[dC]
X_90_Width	= 9.2[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 4.6[dB]
X_Pulse	= 3.06666667[us]
Irr_Atn_Dec	= 21.976[dB]
Irr_Atn_Noe	= 21.976[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 92[us]
Decoupling	= TRUE



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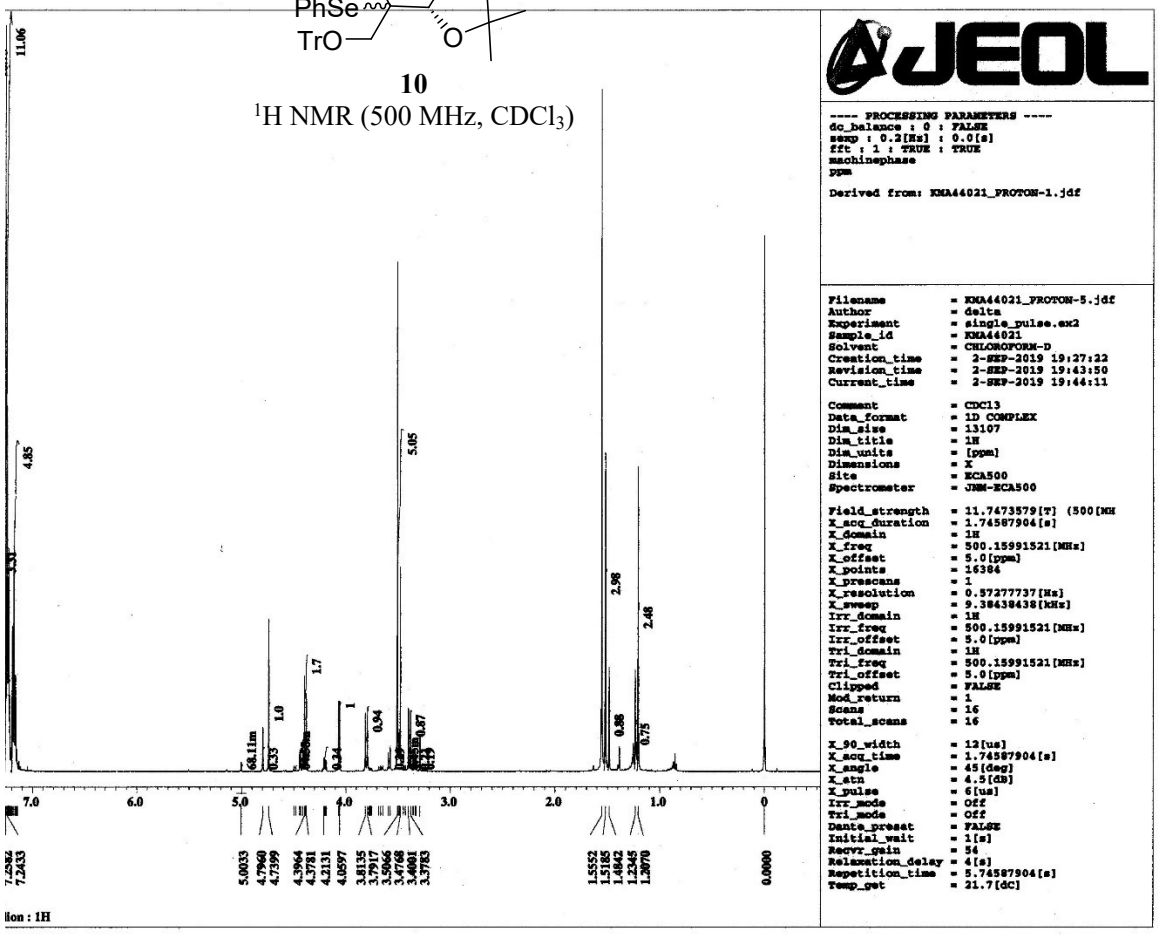
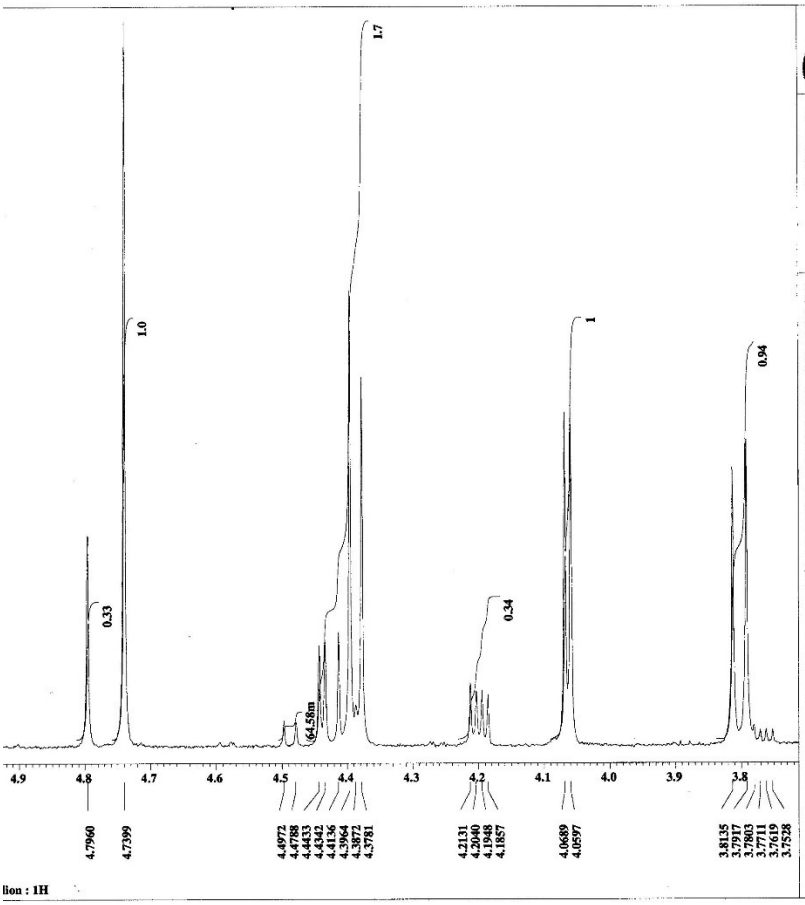
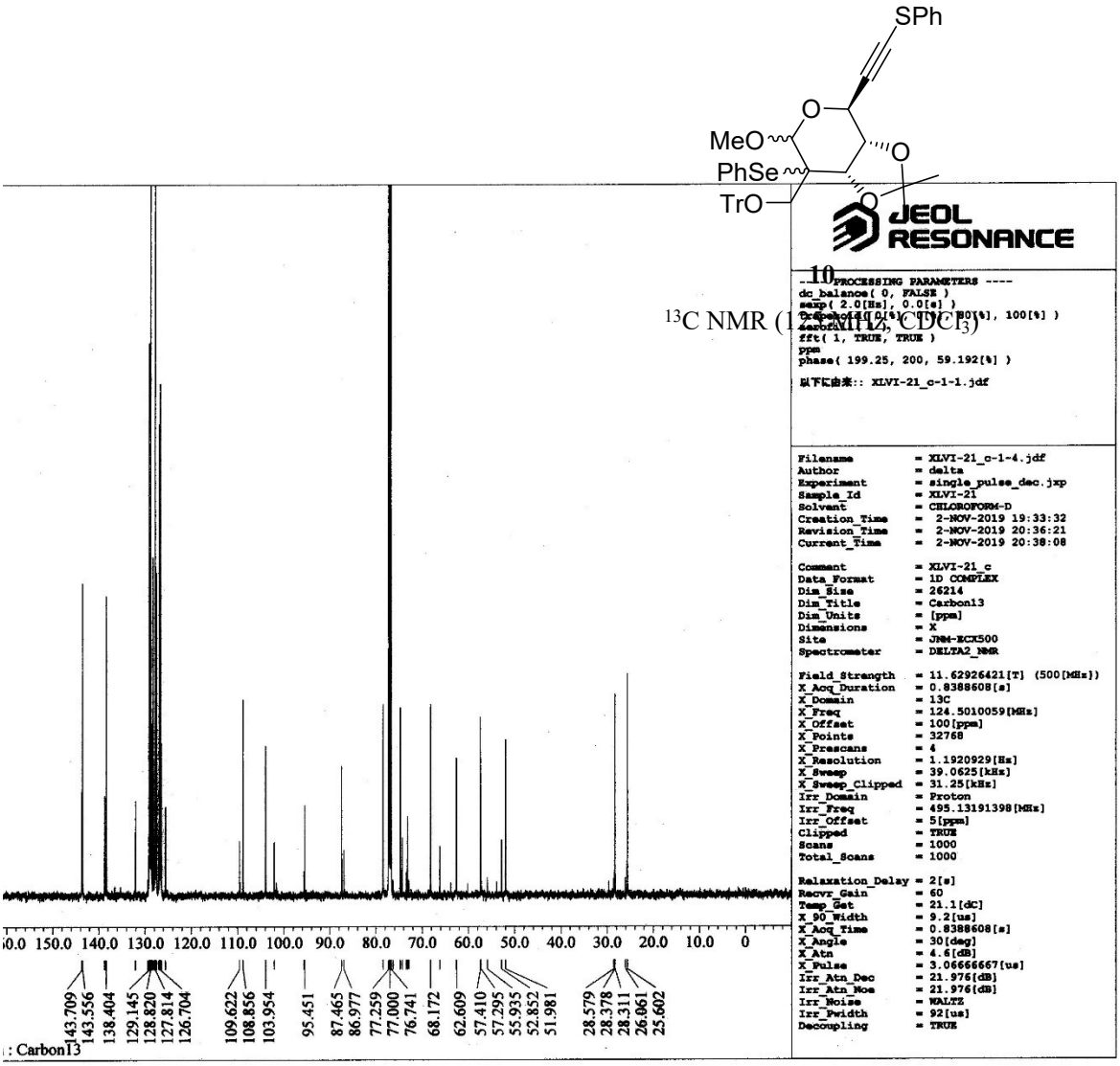
^{1}H NMR (500 MHz, $CDCl_3$)



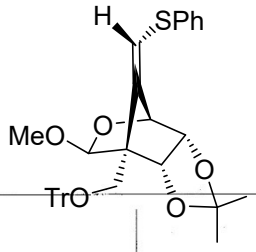



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA44021_PROTON-1.jdf
```

Filename	= KMA44021_PROTON-5.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= KMA44021
Solvent	= CHLOROFORM-D
Creation_time	= 2-SEP-2019 19:27:22
Revision_time	= 2-SEP-2019 19:43:50
Current_time	= 2-SEP-2019 19:44:51
Comment	= CDCl3
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 12[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4.5[dB]
X_pulse	= 6[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 54
Relaxation_delay	= 4[s]
Repetition_time	= 5.74587904[s]
Temp_get	= 21.7[dC]

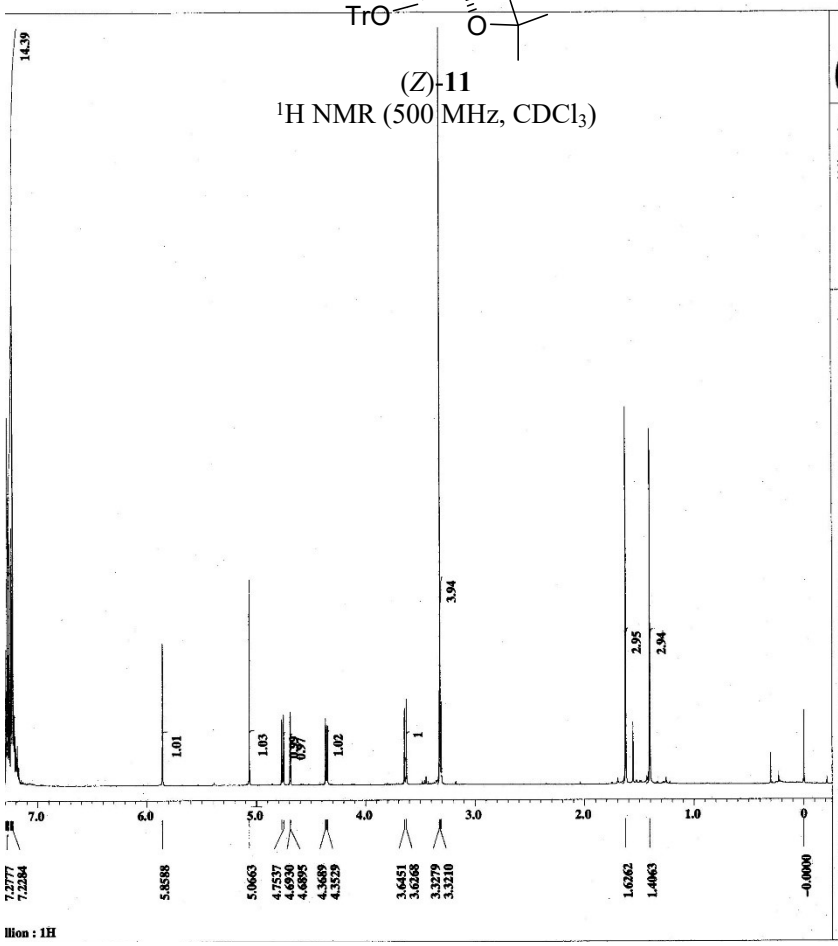


^{13}C NMR (125 MHz, $CDCl_3$)



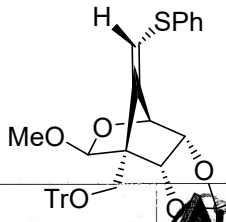
(*Z*)-**11**

^{1}H NMR (500 MHz, $CDCl_3$)



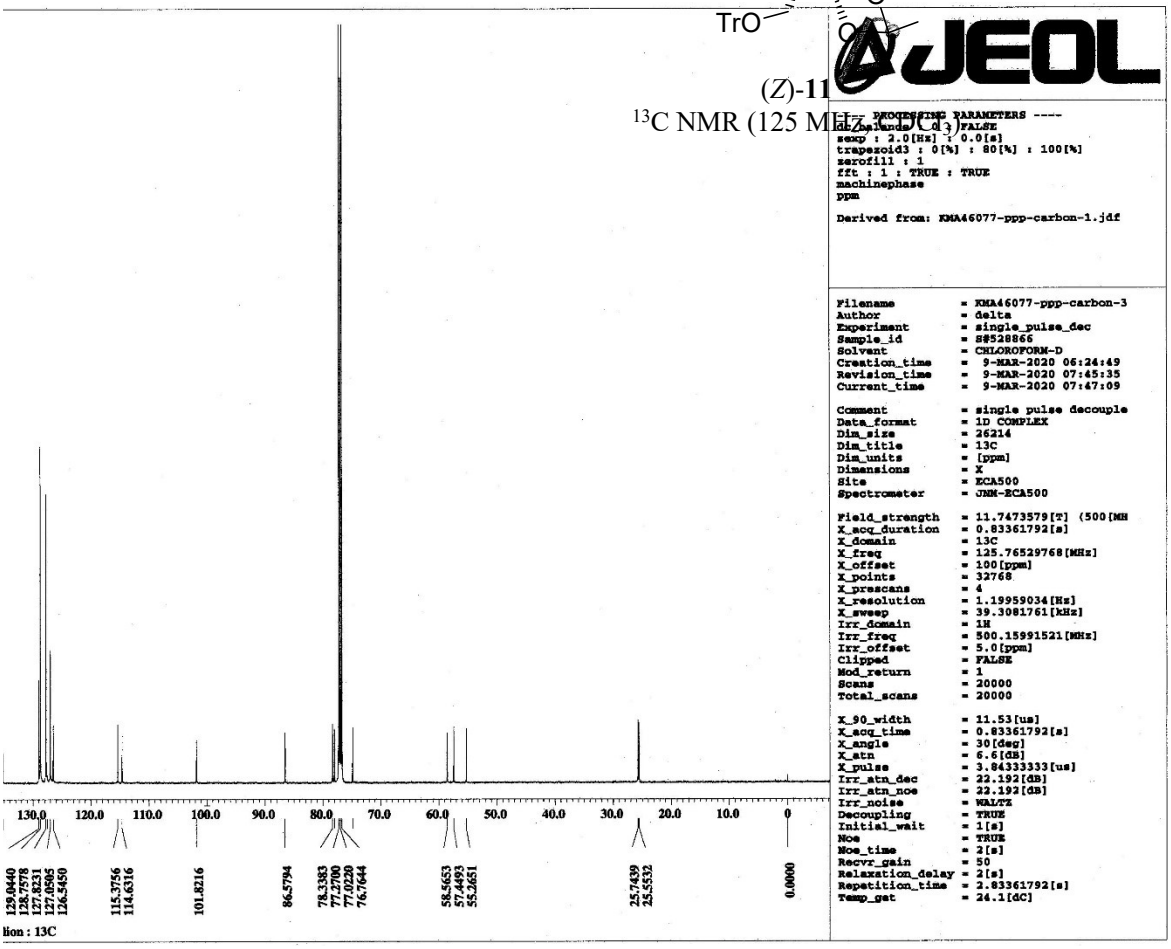
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46077-ppp_PROTON-1.jdf
```

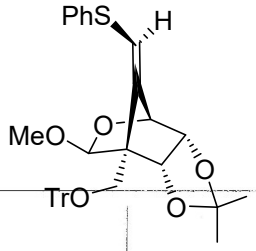
```
Filename          = KMA46077-ppp_PROTON-5
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = KMA46077-ppp
Solvent           = CHLOROFORM-D
Creation_time     =  8-MAR-2020 11:06:38
Revision_time     =  8-MAR-2020 11:14:05
Current_time      =  8-MAR-2020 11:14:25

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 6[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 23.4[dC]
```



(*Z*)-**11**

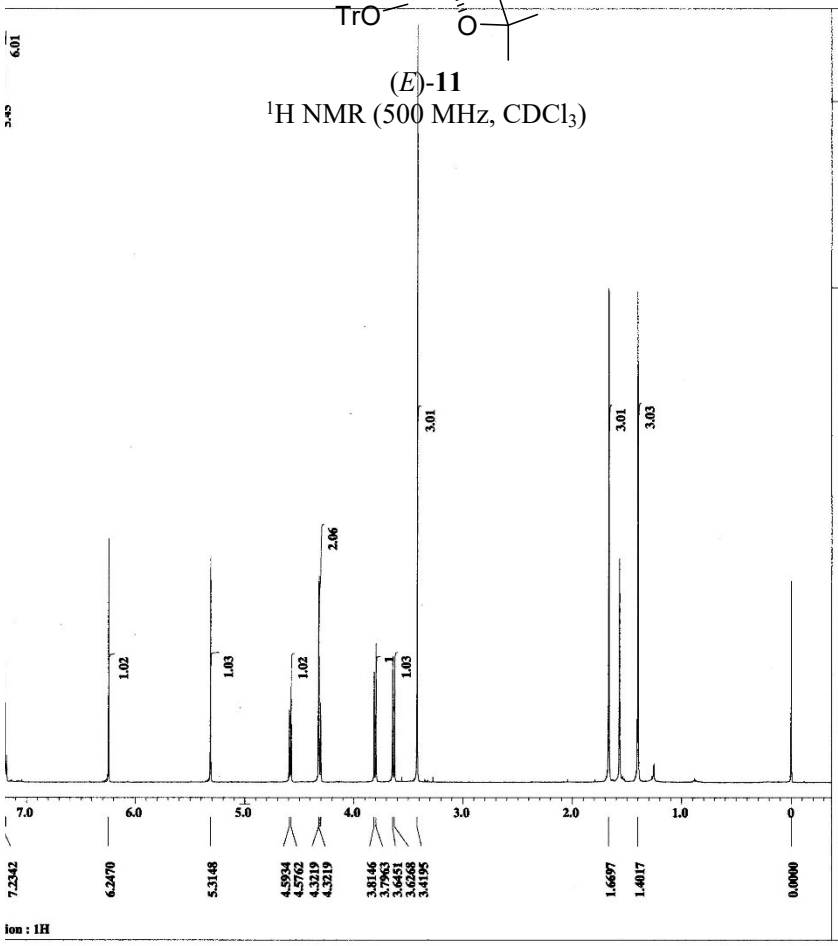
^{13}C NMR (125 MHz, $CDCl_3$)





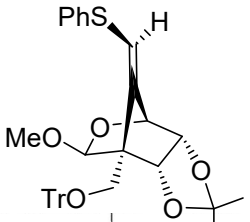
(*E*)-**11**

^{1}H NMR (500 MHz, $CDCl_3$)



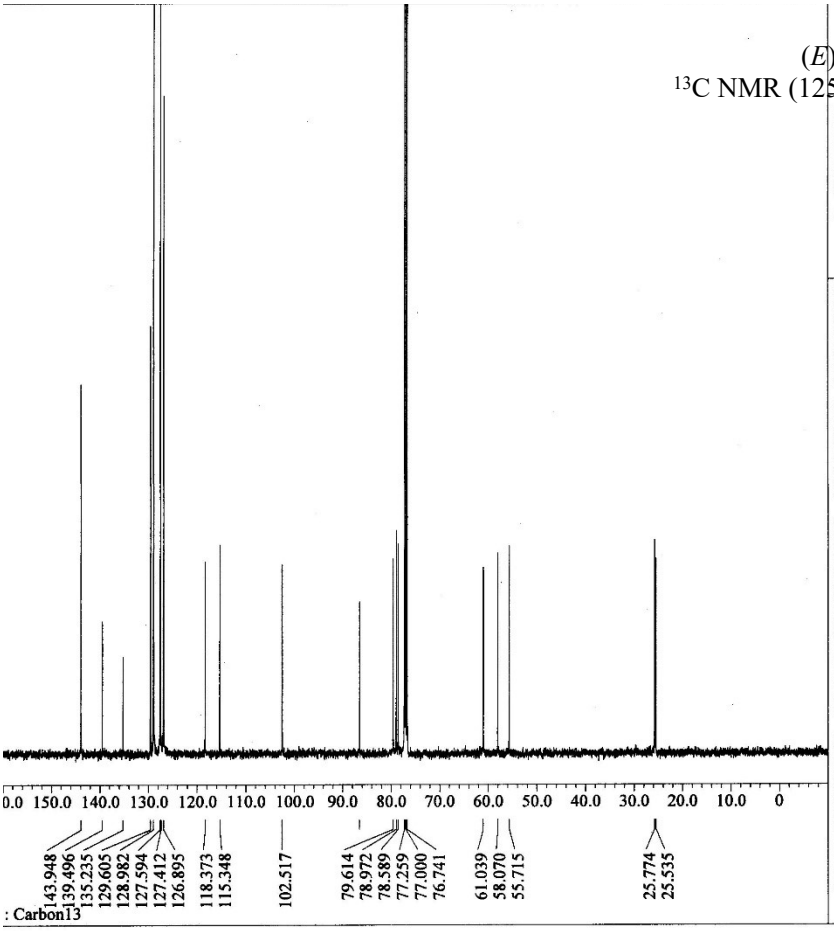
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46022-3_PROTON-1.jdf
```

```
Filename            = KMA46022-3_PROTON-5.j
Author              = delta
Experiment          = single_pulse.ex2
Sample_id           = KMA46022-3
Solvent             = CHLOROFORM-D
Creation_time       = 30-AUG-2019 15:15:50
Revision_time       = 30-AUG-2019 15:29:27
Current_time        = 30-AUG-2019 15:29:58

Comment             = CDCl3
Data_format         = 1D COMPLEX
Dim_size            = 13107
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA500
Spectrometer        = JNM-ECA500

Field_strength      = 11.7473579[T] (500[MH
X_acq_duration      = 1.74587904[s]
X_domain            = 1H
X_freq              = 500.15991521[MHz]
X_offset            = 5.0[ppm]
X_points            = 16384
X_prescans          = 1
X_resolution        = 0.57277737[Hz]
X_sweep             = 9.38438438[kHz]
Irr_domain          = 1H
Irr_freq            = 500.15991521[MHz]
Irr_offset          = 5.0[ppm]
Tri_domain          = 1H
Tri_freq            = 500.15991521[MHz]
Tri_offset          = 5.0[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 16
Total_scans         = 16

X_90_width          = 12[us]
X_acq_time          = 1.74587904[s]
X_angle             = 45[deg]
X_atn               = 4.5[dB]
X_pulse             = 6[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 48
Relaxation_delay    = 4[s]
Repetition_time     = 5.74587904[s]
Temp_get            = 21[dC]
```



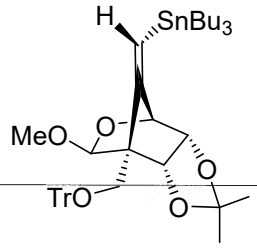
(*E*)-**11**

^{13}C NMR (125 MHz, $CDCl_3$)



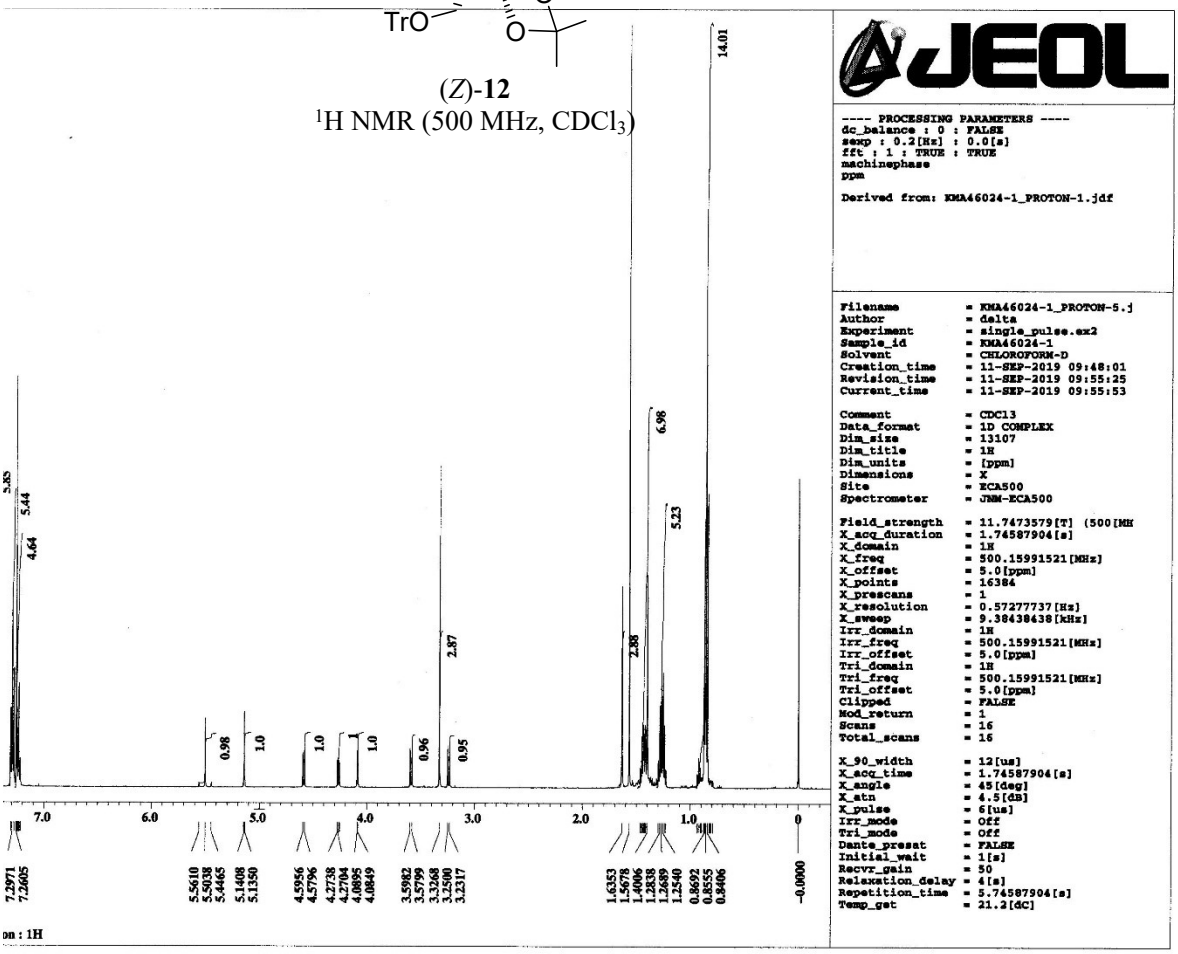

```
---- PROCESSING PARAMETERS ----
do_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
phase( 231, 220, 38.96921[%] )

以下EC由来:: XLVI-22-3_c-1-1.jdf
```

```
Filename          = XLVI-22-3_c-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = XLVI-22-3
Solvent           = CHLOROFORM-D
Creation_Time     =  2-NOV-2019 21:16:59
Revision_Time     = 13-NOV-2019 17:10:49
Current_Time      = 13-NOV-2019 17:15:21

Comment           = XLVI-22-3_c
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECX500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 0.8388608[s]
X_Domain          = 13C
X_Freq            = 124.5010059[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.1920929[Hz]
X_Sweep           = 39.0625[kHz]
X_Sweep_Clipped   = 31.25[kHz]
Irr_Domain        = Proton
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.2[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 0.8388608[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.976[dB]
Irr_Atn_Noe       = 21.976[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
```



(*Z*)-**12**

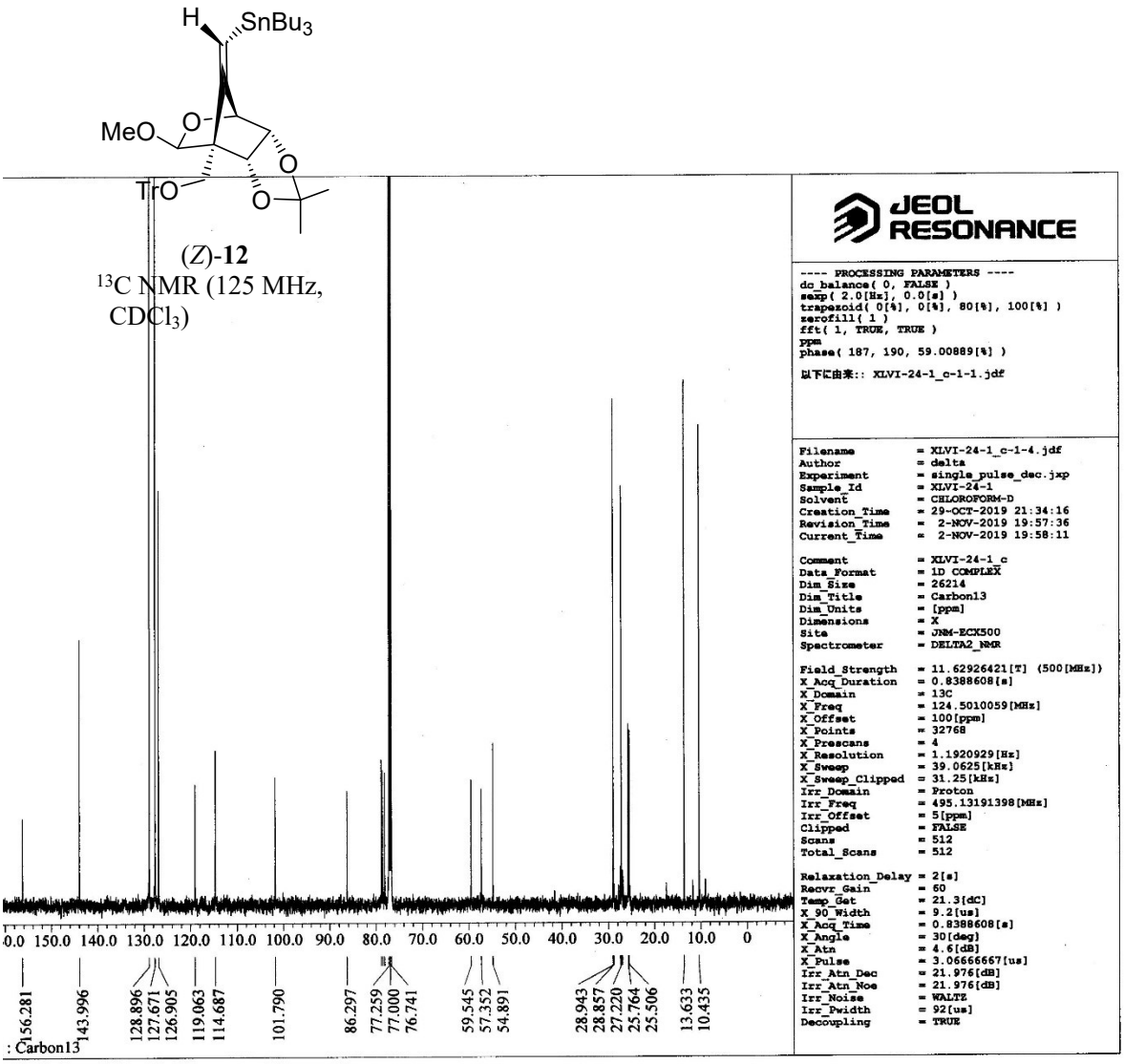
^{1}H NMR (500 MHz, $CDCl_3$)



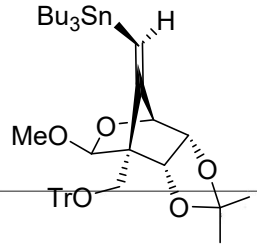
JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46024-1_PROTON-1.jdf
```

```
Filename            = KMA46024-1_PROTON-5.j
Author              = delta
Experiment          = single_pulse.ex2
Sample_id           = KMA46024-1
Solvent             = CHLOROFORM-D
Creation_time       = 11-SEP-2019 09:48:01
Revision_time       = 11-SEP-2019 09:55:25
Current_time        = 11-SEP-2019 09:55:53

Comment             = CDCl3
Data_format         = 1D COMPLEX
Dim_size            = 13107
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA500
Spectrometer        = JNM-ECA500

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             = 9.38438638[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          = 12[us]
X_acq_time          = 1.74587904[s]
X_angle             = 45[deg]
X_atn               = 4.5[dB]
X_pulse             = 6[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 50
Relaxation_delay    = 4[s]
Repetition_time     = 5.74587904[s]
Temp_get            = 21.2[dC]
```

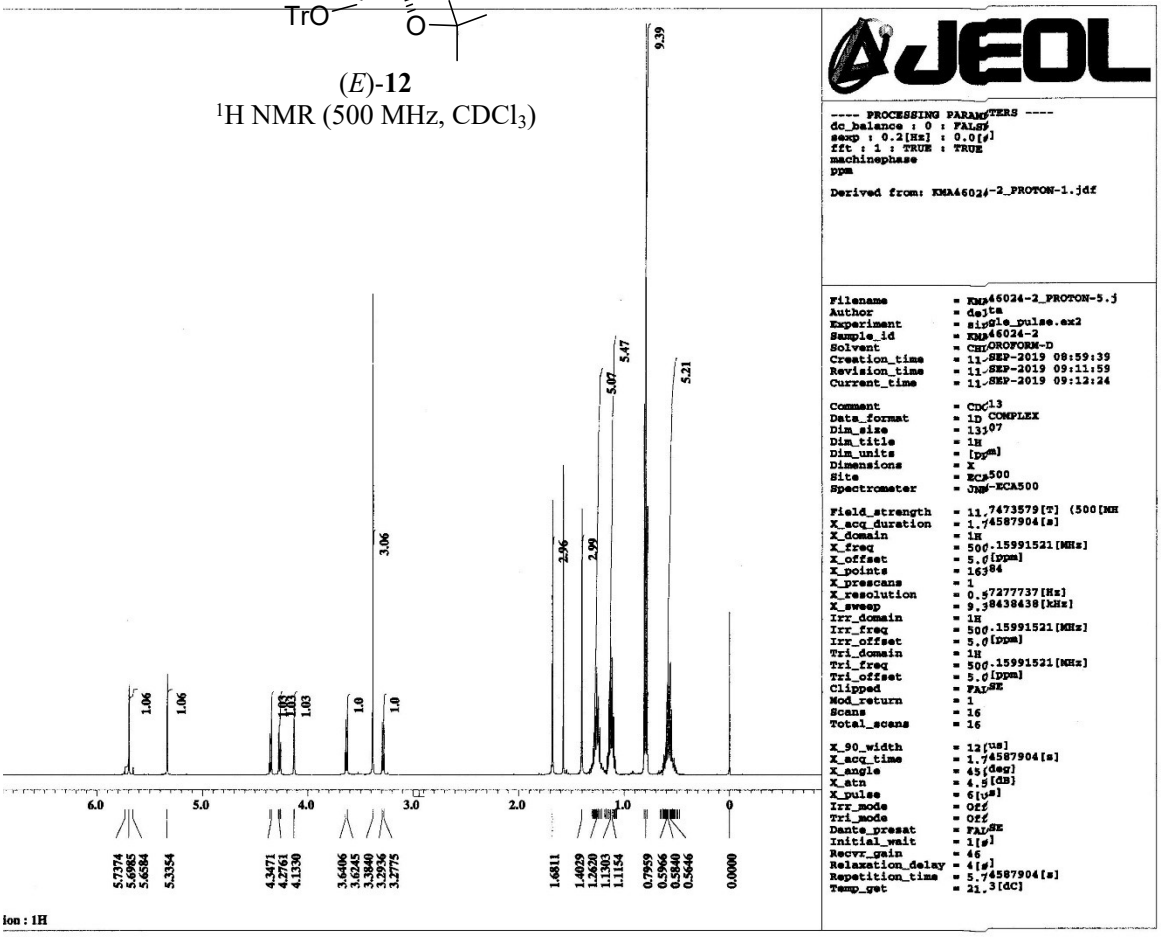


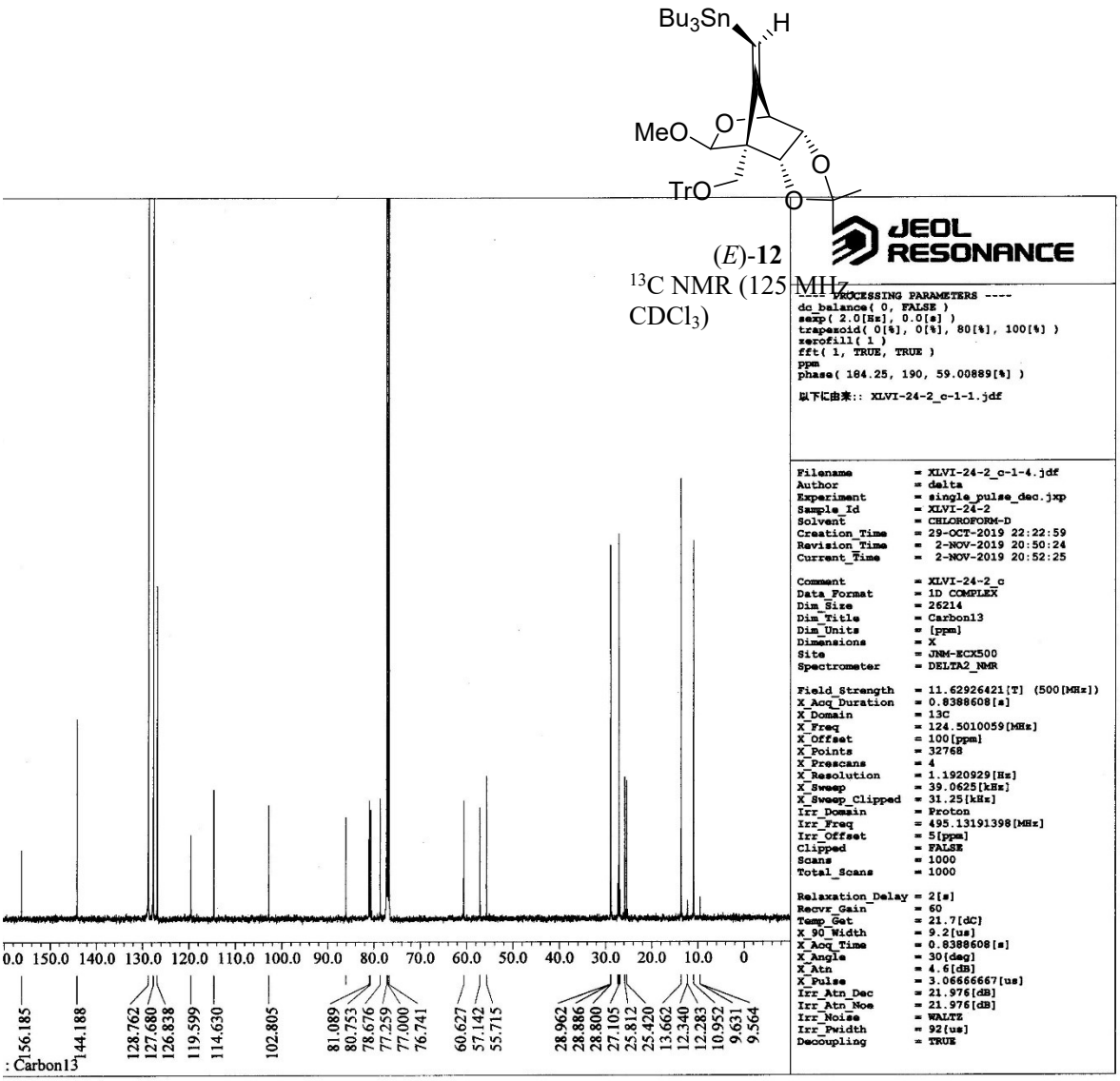
(*Z*)-**12**
^{13}C NMR (125 MHz, $CDCl_3$)



(*E*)-**12**

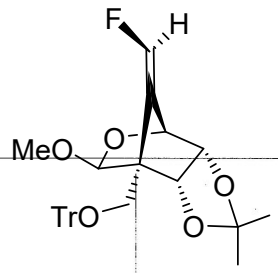
^{1}H NMR (500 MHz, $CDCl_3$)





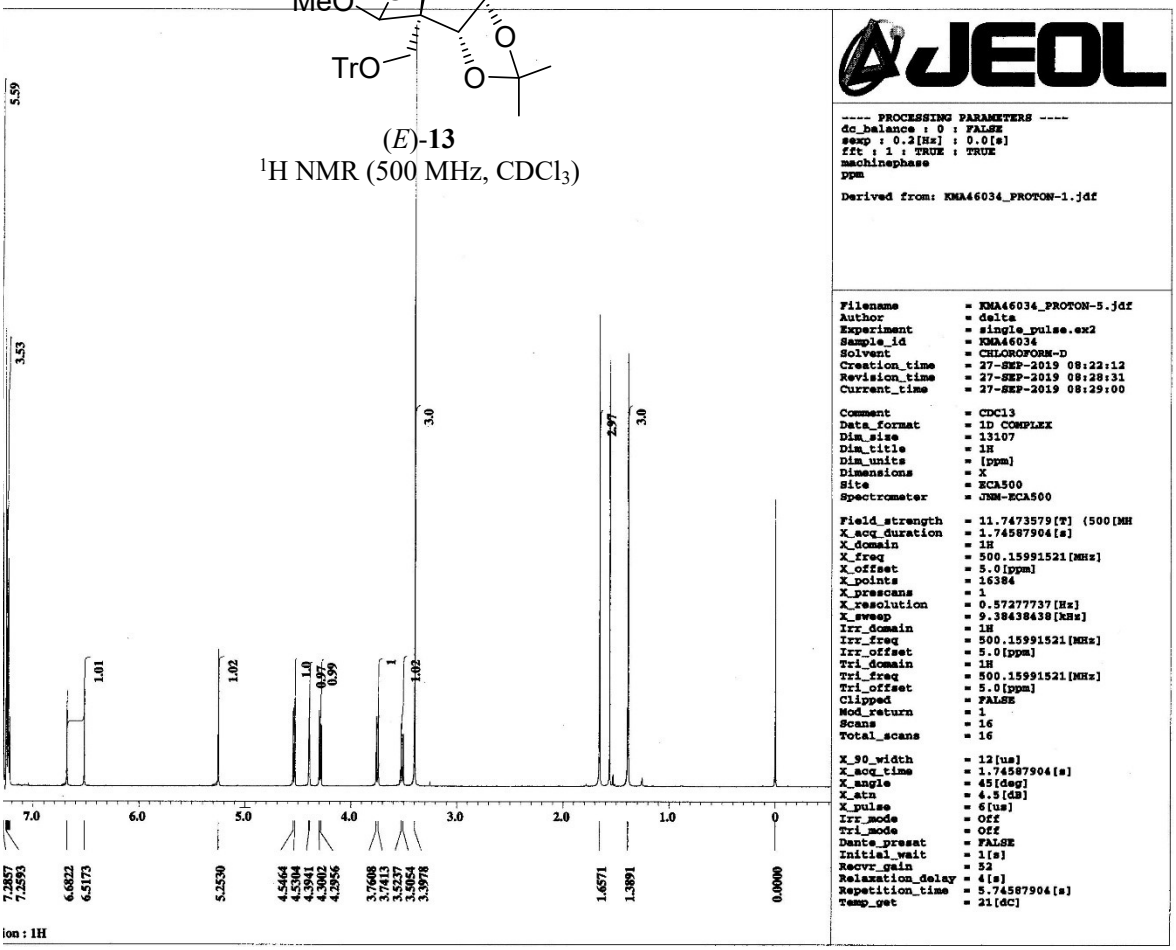
(*E*)-**12**

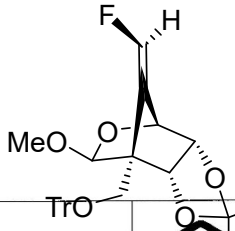
^{13}C NMR (125 MHz, $CDCl_3$)



(*E*)-**13**

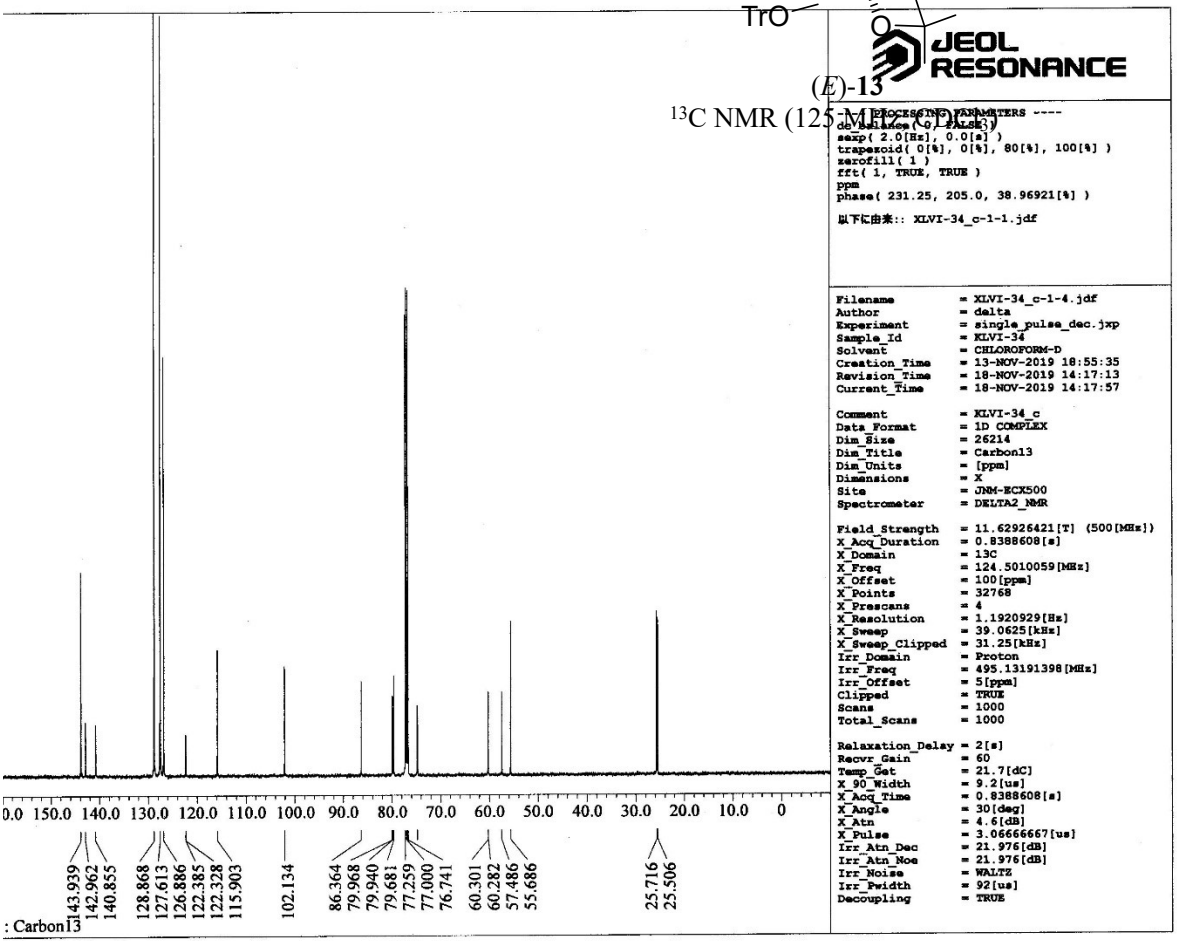
^{1}H NMR (500 MHz, $CDCl_3$)

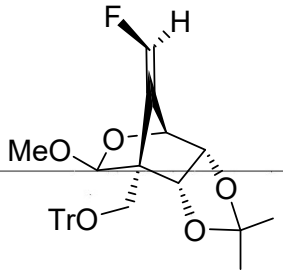




(*E*)-13

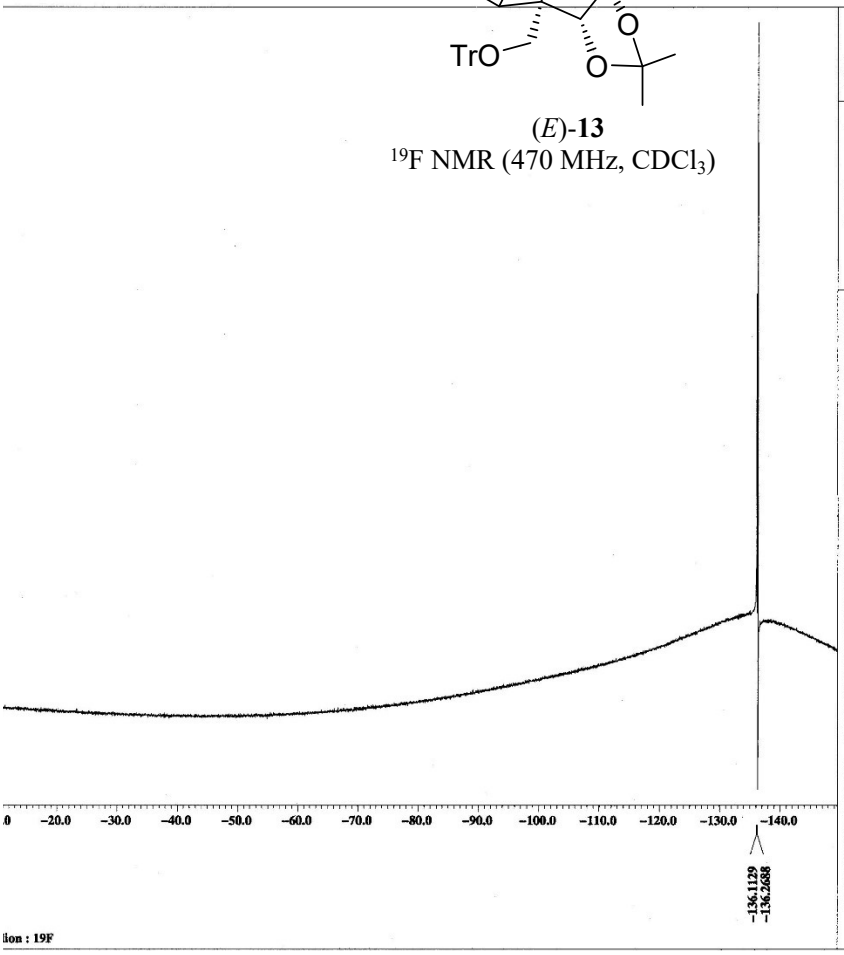
^{13}C NMR (125 MHz, CDCl$_3$)





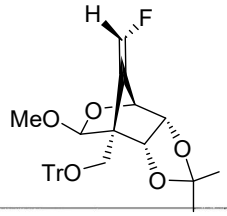
(*E*)-**13**

^{19}F NMR (470 MHz, $CDCl_3$)



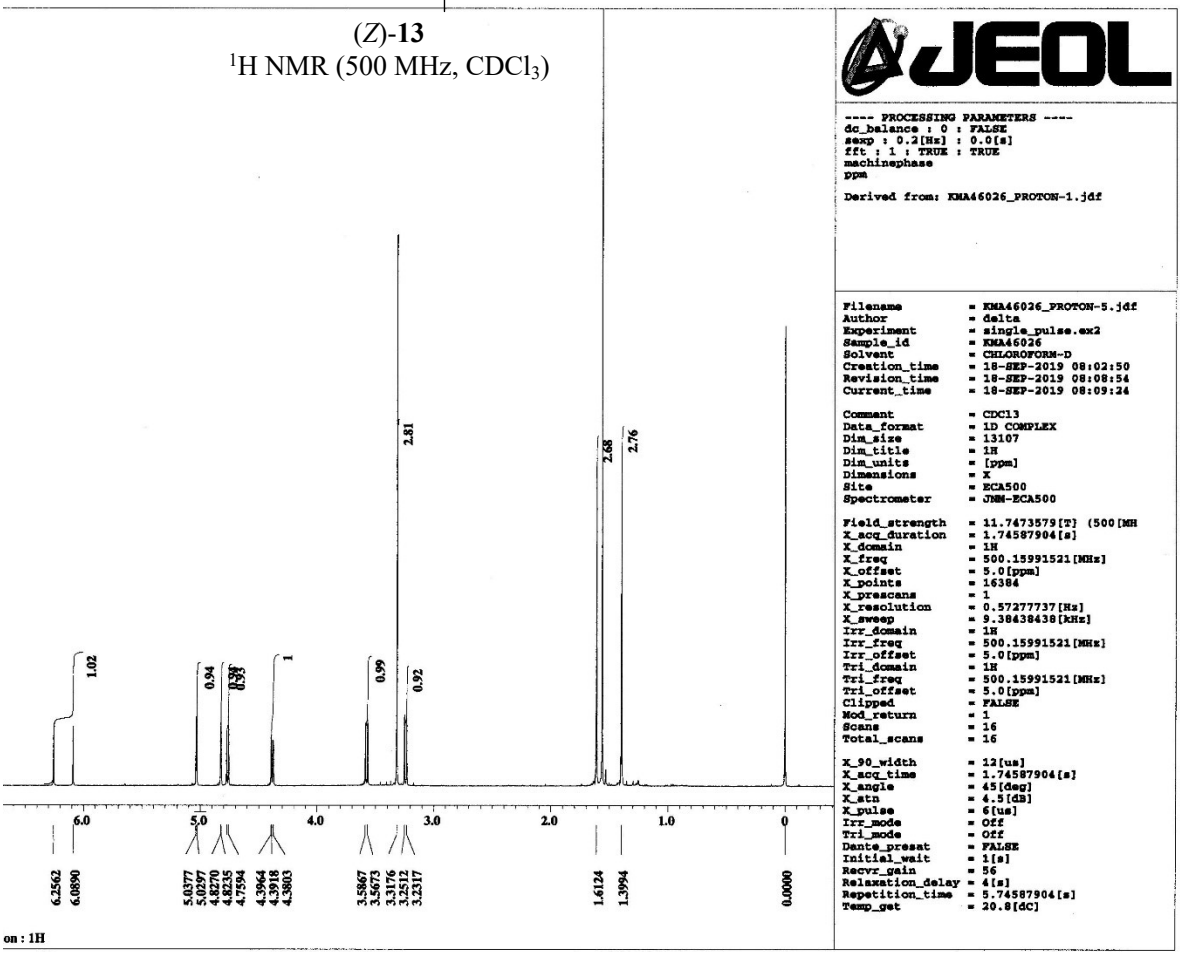

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA47088-19F-1.jdf
```

```
Filename          = KMA47088-19F-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#657660
Solvent           = CHLOROFORM-D
Creation_time     = 22-JAN-2021 18:20:31
Revision_time     = 22-JAN-2021 18:24:40
Current_time      = 22-JAN-2021 18:25:30

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 22.7[dC]
```

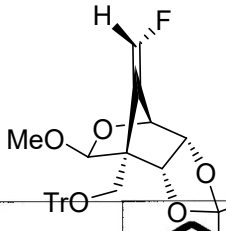


(*Z*)-**13**

^{1}H NMR (500 MHz, $CDCl_3$)

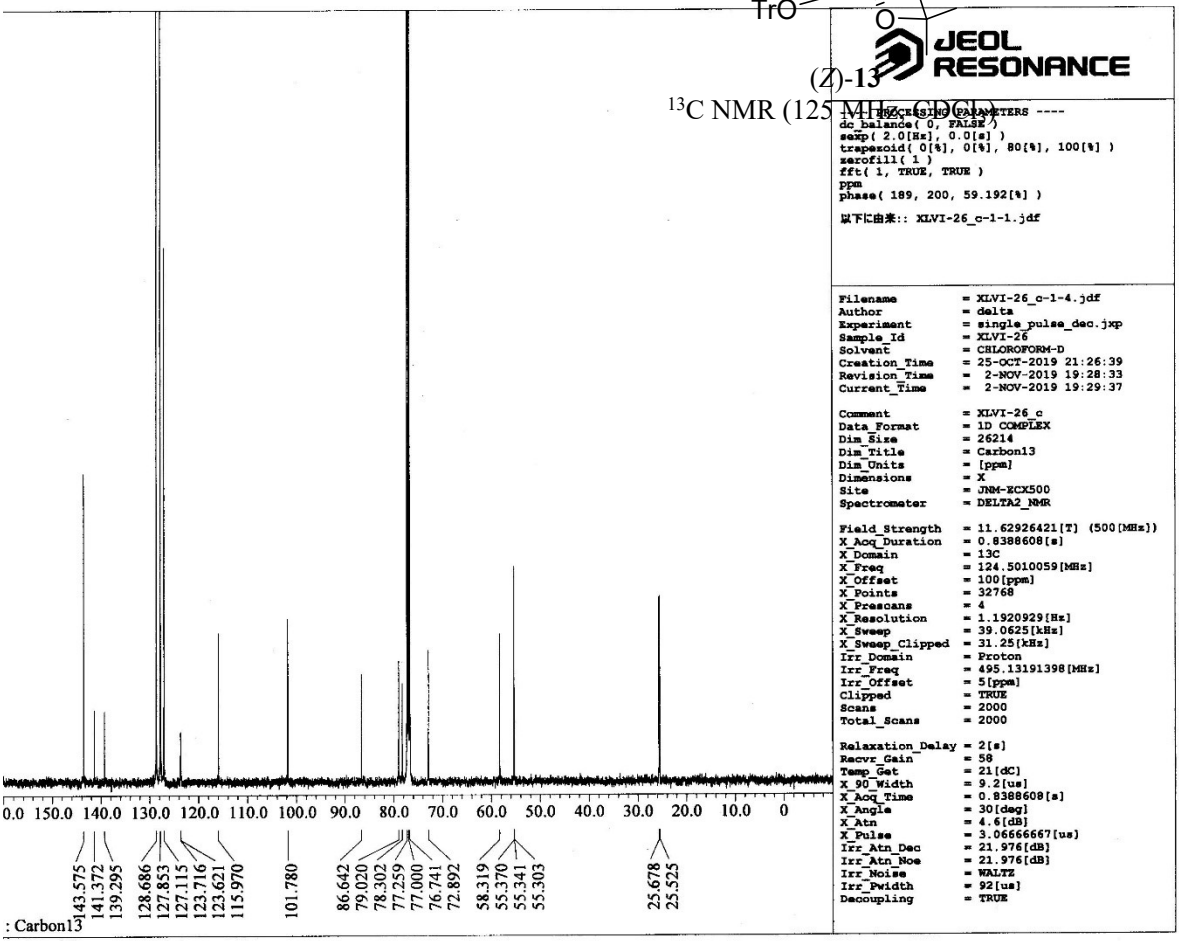


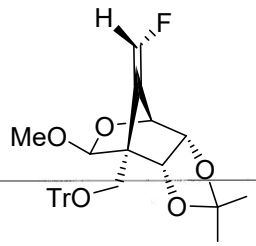

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46026_PROTON-1.jdf

Filename          = KMA46026_PROTON-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = KMA46026
Solvent           = CHLOROFORM-D
Creation_time     = 18-SEP-2019 08:02:50
Revision_time     = 18-SEP-2019 08:08:54
Current_time      = 18-SEP-2019 08:09:24

Comment           = CDCl3
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 6[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 56
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 20.8[dC]
```



(*Z*)-**13**

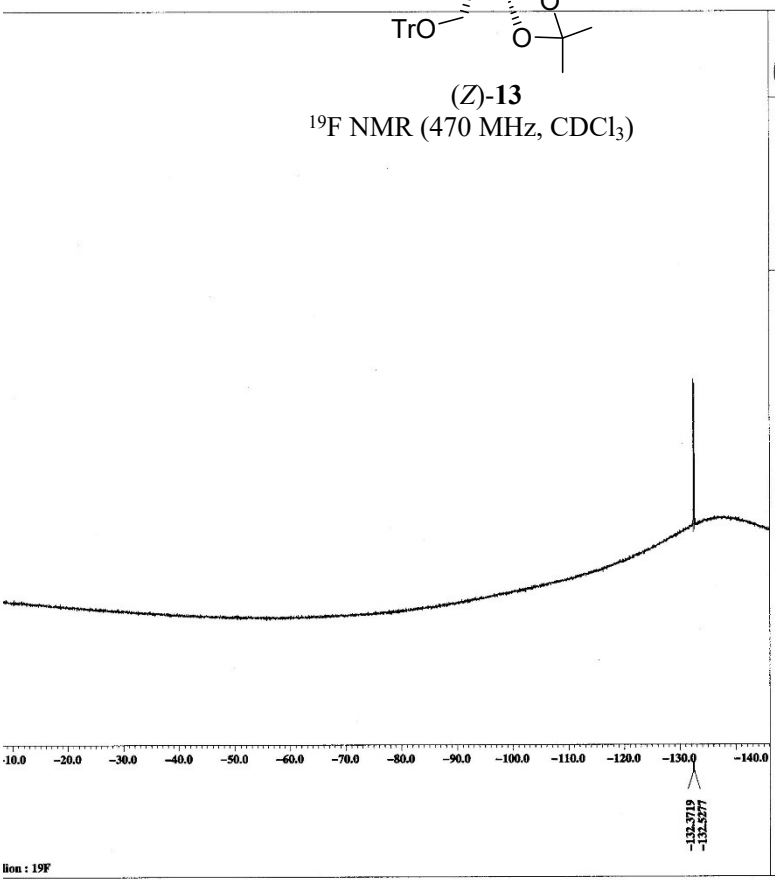
^{13}C NMR (125 MHz, CDCl$_3$)



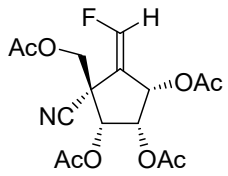


(*Z*)-**13**

^{19}F NMR (470 MHz, $CDCl_3$)

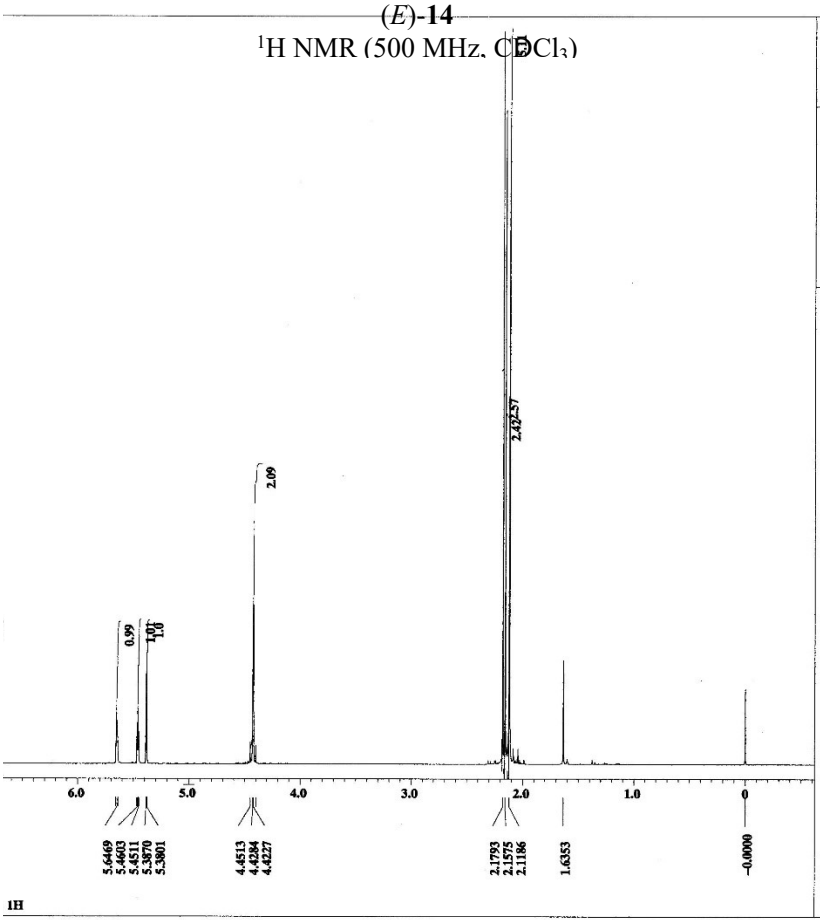


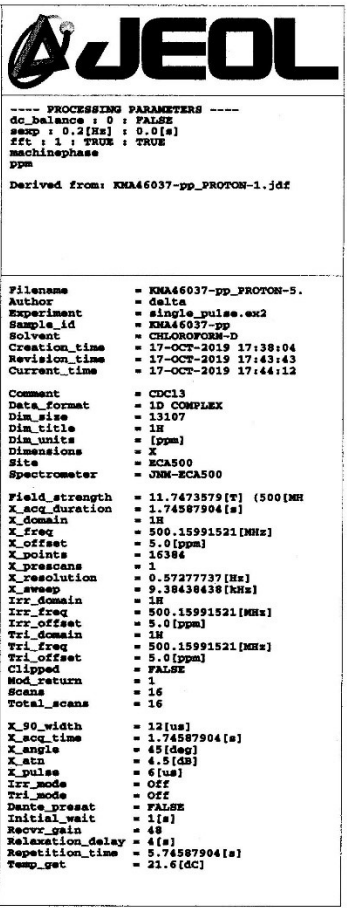

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46016-19F-1.jdf

Filename              = KMA46016-19F-4.jdf
Author                = delta
Experiment            = single_pulse.ex2
Sample_id             = S0569945
Solvent               = CHLOROFORM-D
Creation_time         = 13-MAR-2021 15:51:13
Revision_time         = 13-MAR-2021 15:58:15
Current_time          = 13-MAR-2021 15:58:36

Comment               = single_pulse
Data_format           = 1D COMPLEX
Dim_size              = 13107
Dim_title             = 19F
Dim_units             = [ppm]
Dimensions            = X
Site                  = ECA500
Spectrometer          = JNM-ECA500

Field_strength        = 11.7473579[T] (500[MH
X_acq_duration        = 68.15744[ms]
X_domain              = 19F
X_freq                = 470.62046084[MHz]
X_offset              = 0[ppm]
X_points              = 16384
X_prescans            = 1
X_resolution          = 14.67191256[Hz]
X_sweep               = 240.38461538[kHz]
Irr_domain            = 19F
Irr_freq              = 470.62046084[MHz]
Irr_offset            = 5[ppm]
Tri_domain            = 19F
Tri_freq              = 470.62046084[MHz]
Tri_offset            = 5[ppm]
Clipped               = TRUE
Mod_return            = 1
Scans                 = 64
Total_scans           = 64

X_90_width            = 11.7[us]
X_acq_time            = 68.15744[ms]
X_angle               = 45[deg]
X_atn                 = 4.5[dB]
X_pulse               = 5.85[us]
Irr_mode              = Off
Tri_mode              = Off
Dante_presat          = FALSE
Initial_wait          = 1[s]
Recvr_gain            = 50
Relaxation_delay      = 5[s]
Repetition_time       = 5.06815744[s]
Temp_get              = 22.6[dC]
```

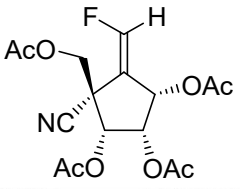


(*E*)-**14**

^{1}H NMR (500 MHz, $CDCl_3$)

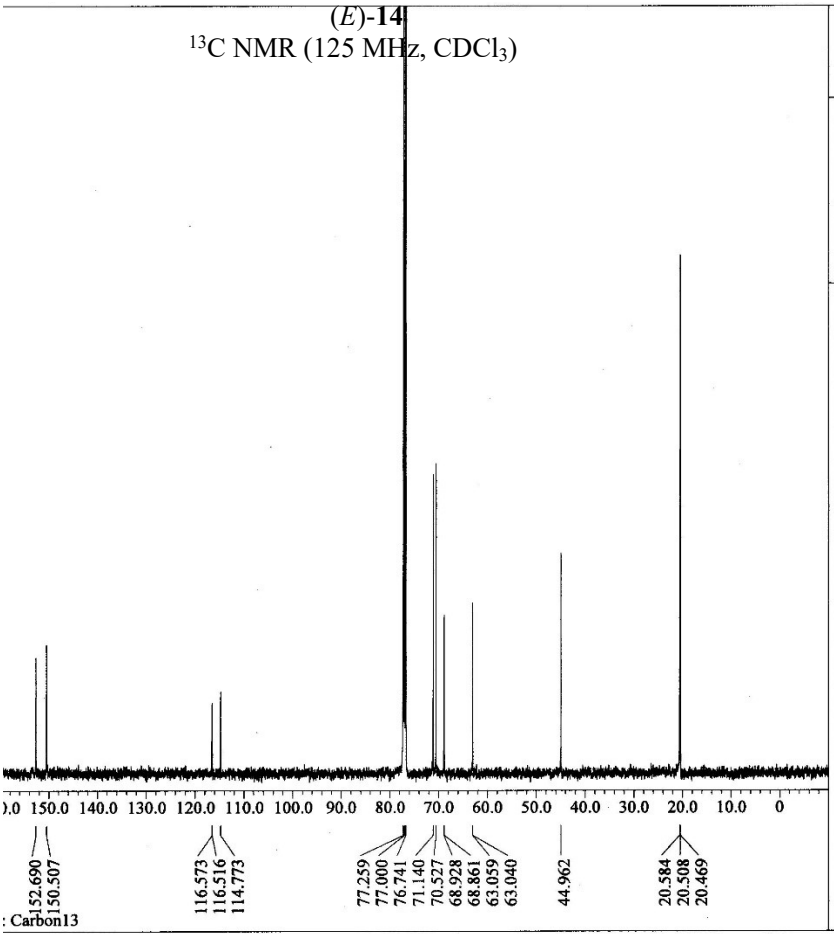






(*E*)-**14**

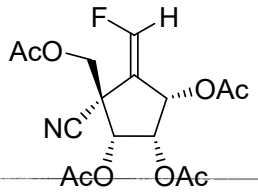
^{13}C NMR (125 MHz, $CDCl_3$)



JEOL RESONANCE

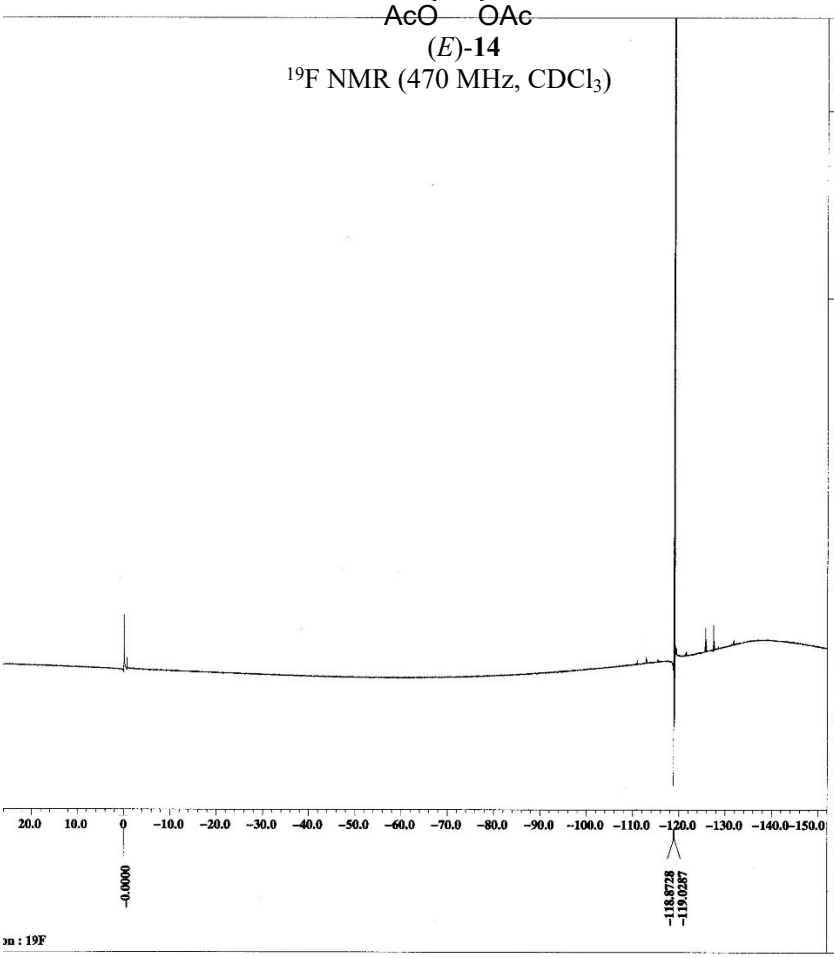
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
phase( 187.38406, 224.06905, 59.00889[%] )
phase( 0, 0, 0.91939[%] )
reference( 77.12541[ppm], 77[ppm] )

以下に由来:: XLVI-37_c-1-1.jdf
```

```
Filename          = XLVI-37_c-1-6.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = XLVI-37
Solvent           = CHLOROFORM-D
Creation_Time     = 18-NOV-2019 16:55:05
Revision_Time     = 19-NOV-2019 17:10:45
Current_Time      = 19-NOV-2019 17:13:38

Comment           = XLVI-37_c
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECX500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 0.8388608[s]
X_Domain          = 13C
X_Freq            = 124.5010059[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.1920929[Hz]
X_Sweep           = 39.0625[kHz]
X_Sweep_Clipped   = 31.25[kHz]
Irr_Domain        = Proton
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.2[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 0.8388608[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.976[dB]
Irr_Atn_Noe       = 21.976[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
```

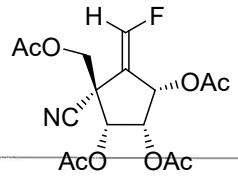


(*E*)-**14**

^{19}F NMR (470 MHz, $CDCl_3$)

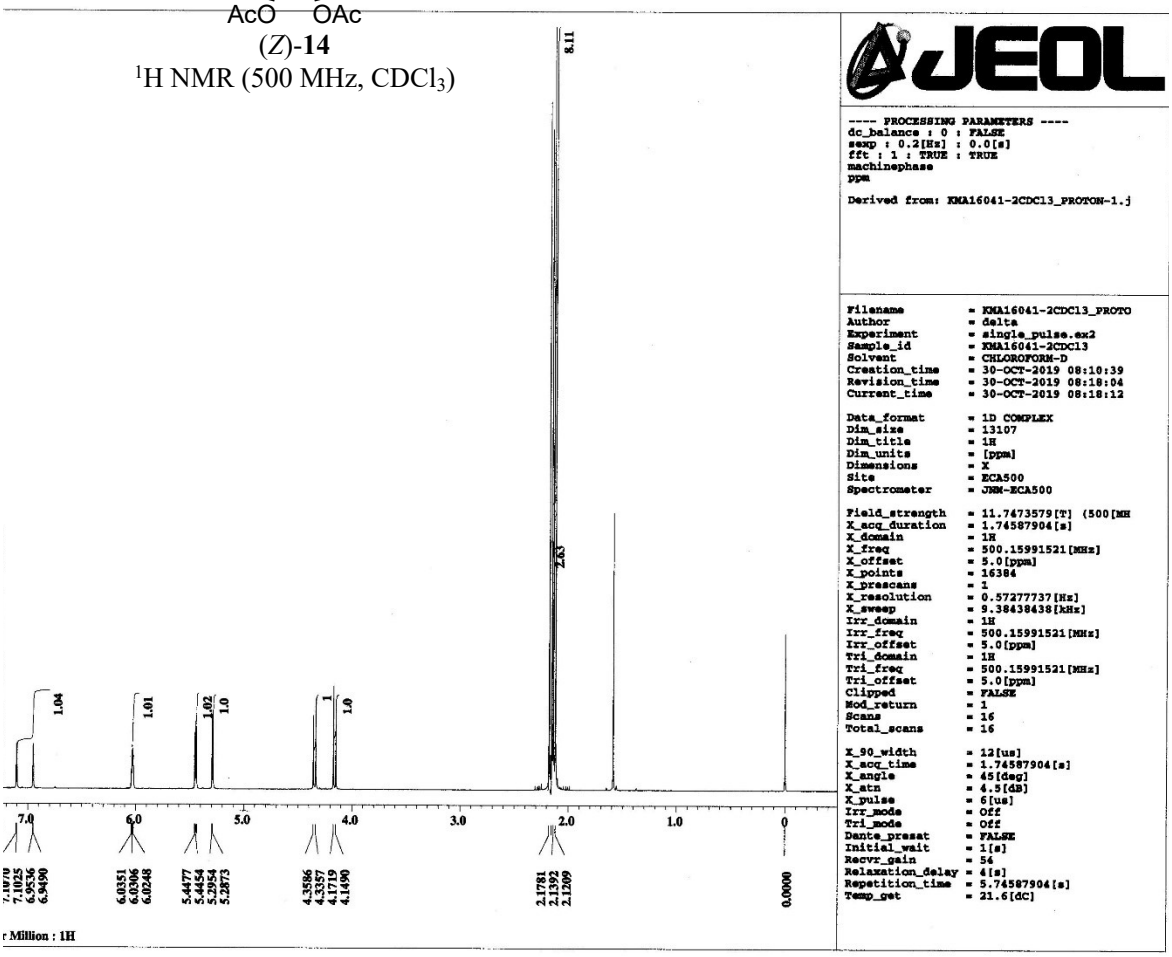



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA14092-19F-1.jdf

Filename          = KMA14092-19F-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#495689
Solvent           = CHLOROFORM-D
Creation_time     =  7-FEB-2021 13:49:37
Revision_time     = 15-AUG-2021 10:48:52
Current_time      = 15-AUG-2021 10:48:58

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 60
Total_scans       = 60

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 23.4[dC]
```

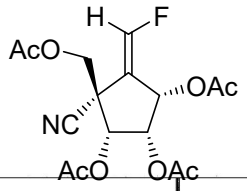


(*Z*)-**14**

^{1}H NMR (500 MHz, $CDCl_3$)

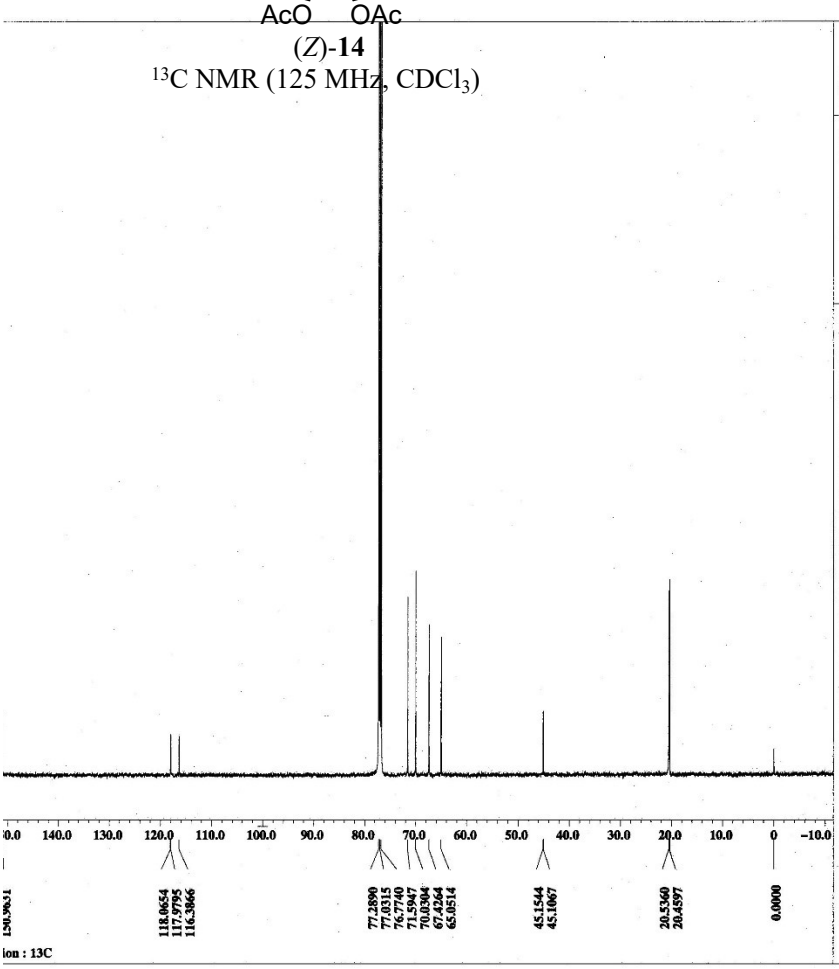


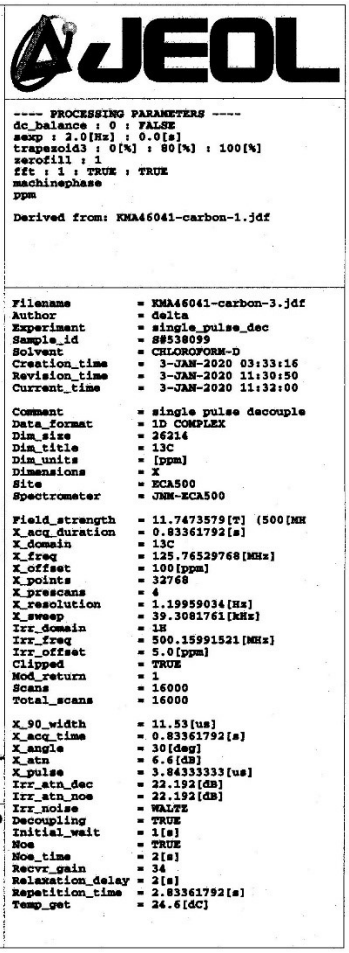

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA16041-2CDCl3_PROTON-1.j

Filename          = KMA16041-2CDCl3_PROTO
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = KMA16041-2CDCl3
Solvent           = CHLOROFORM-D
Creation_time     = 30-OCT-2019 08:10:39
Revision_time     = 30-OCT-2019 08:18:04
Current_time      = 30-OCT-2019 08:18:12

Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 6[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 56
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 21.6[dC]
```

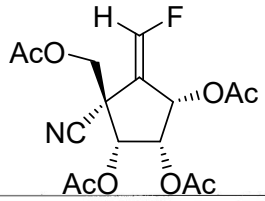


(*Z*)-**14**

^{13}C NMR (125 MHz, $CDCl_3$)

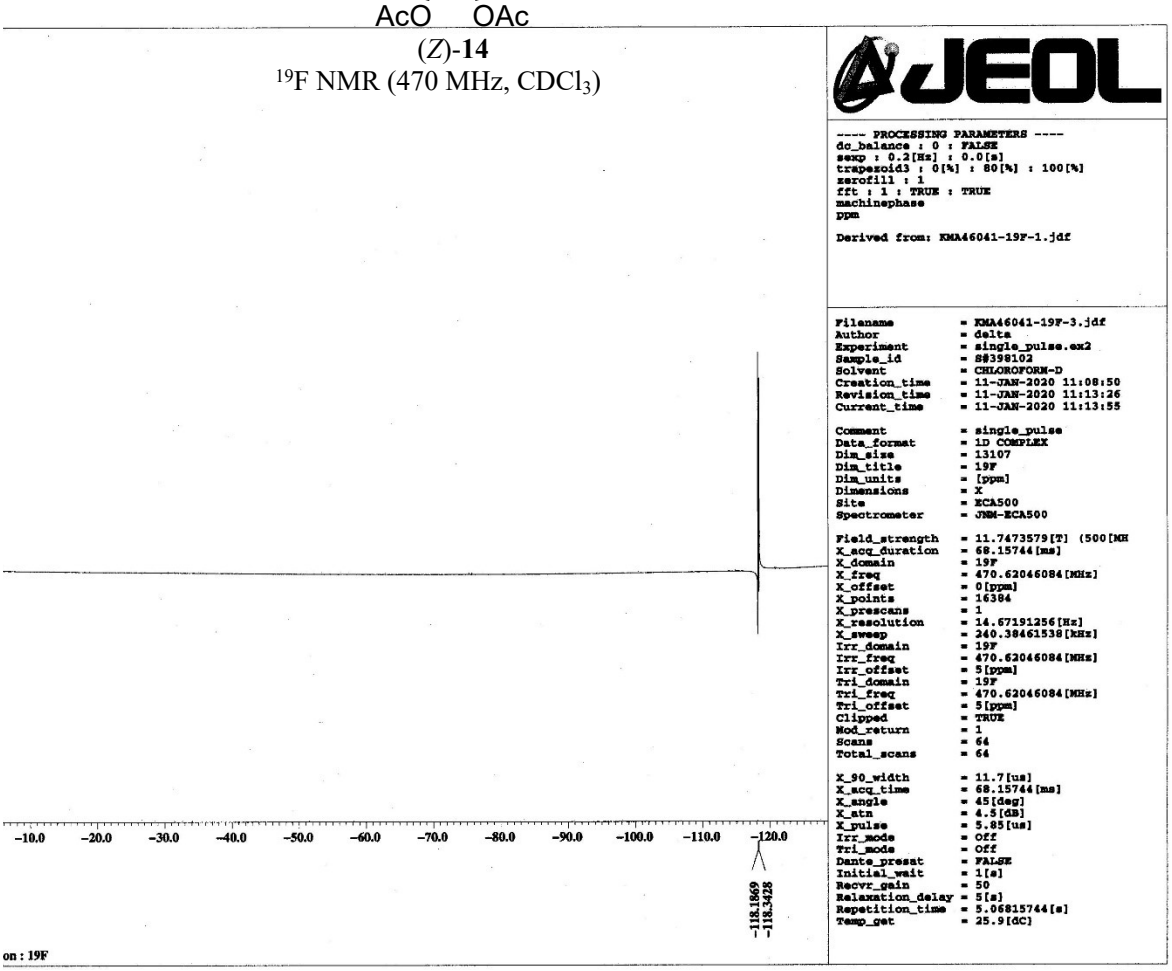




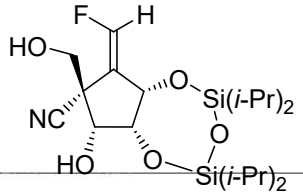


(*Z*)-**14**

^{19}F NMR (470 MHz, $CDCl_3$)

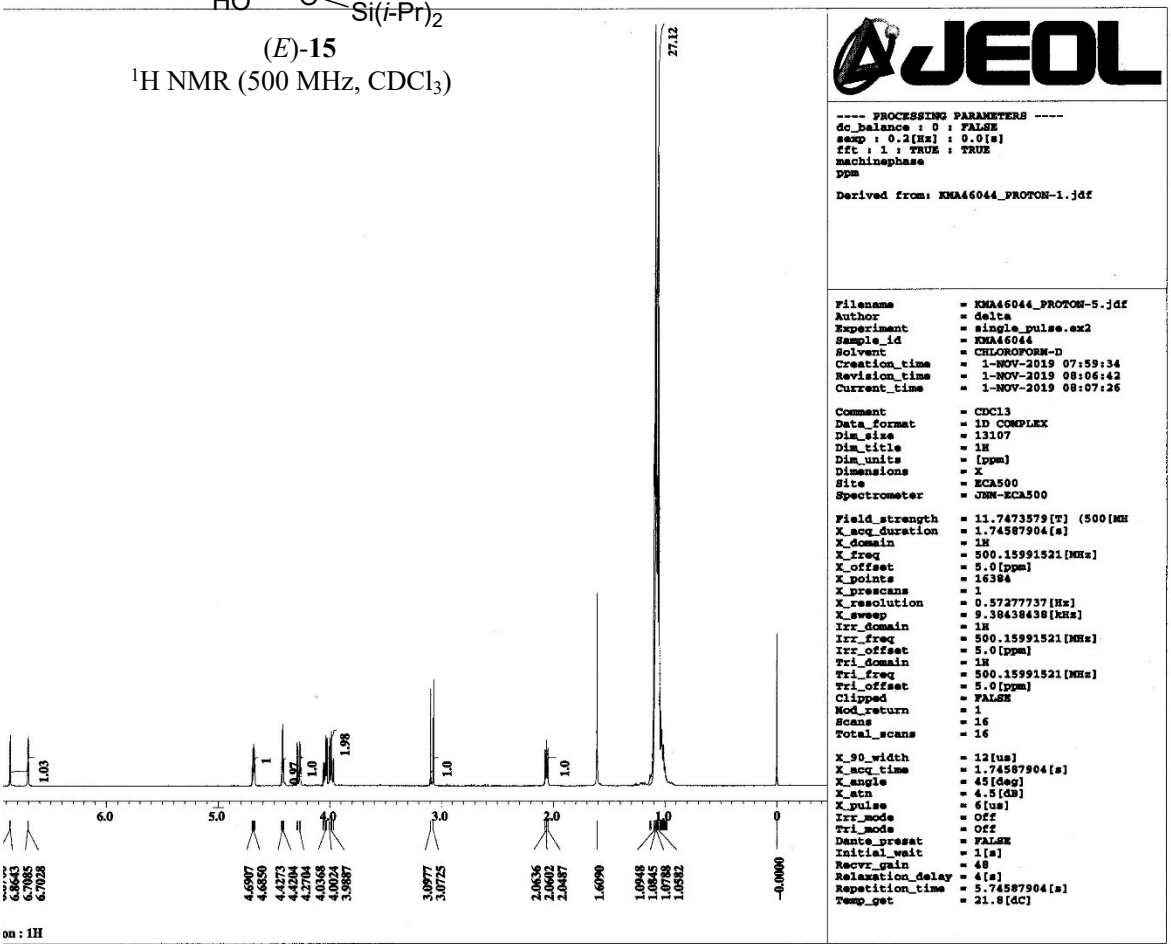


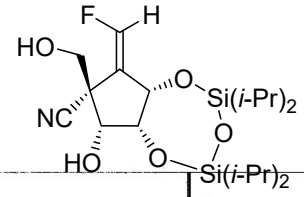

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46041-19F-1.jdf

Filename          = KMA46041-19F-3.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#398102
Solvent           = CHLOROFORM-D
Creation_time     = 11-JAN-2020 11:08:50
Revision_time     = 11-JAN-2020 11:13:26
Current_time      = 11-JAN-2020 11:13:55

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 25.9[dC]
```



(*E*)-**15**

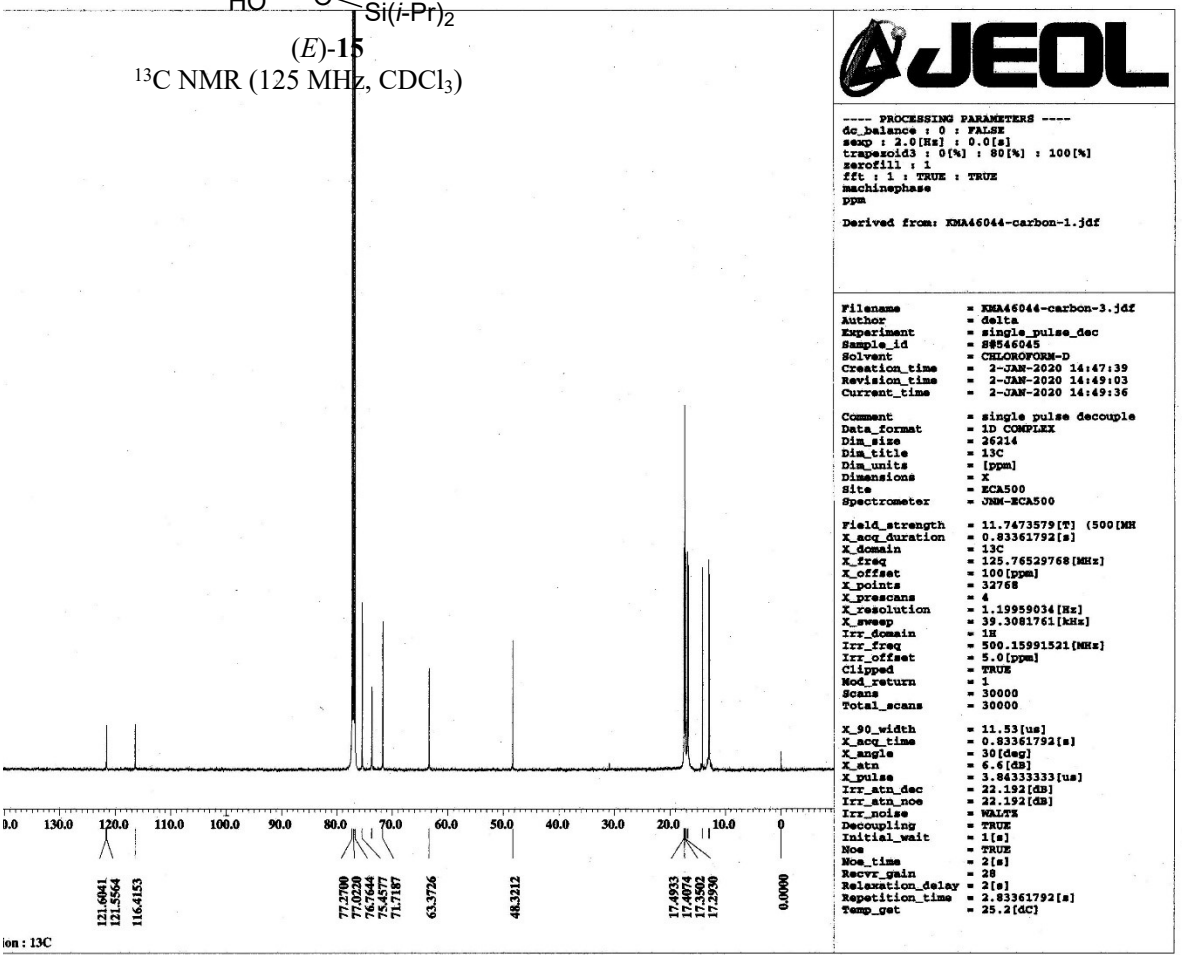
^{1}H NMR (500 MHz, $CDCl_3$)

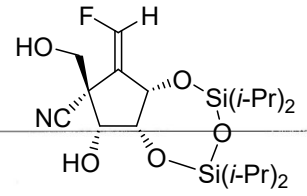




(*E*)-**15**

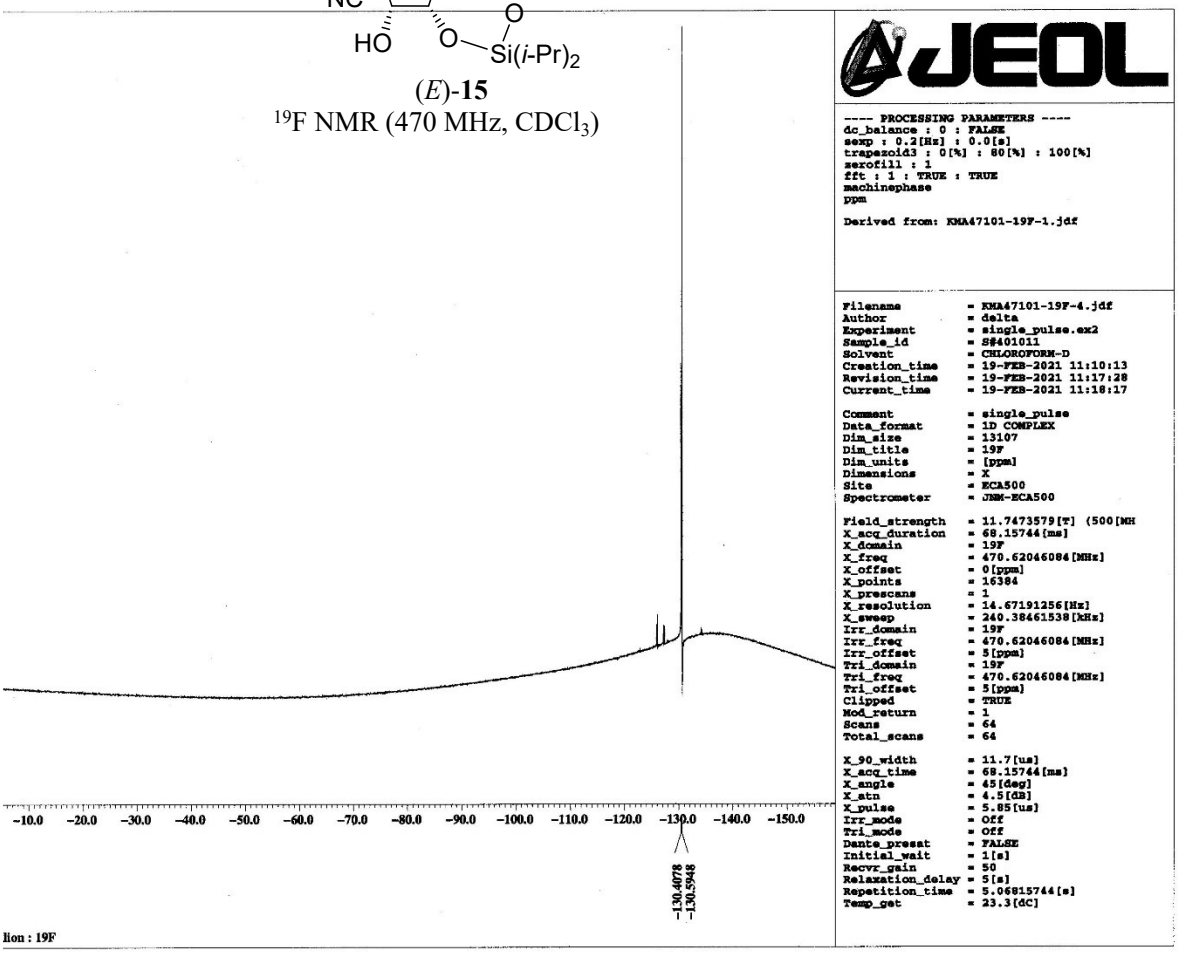
^{13}C NMR (125 MHz, $CDCl_3$)

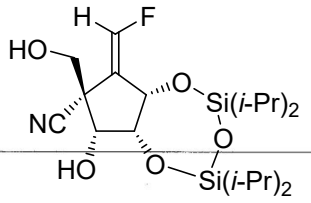




(*E*)-**15**

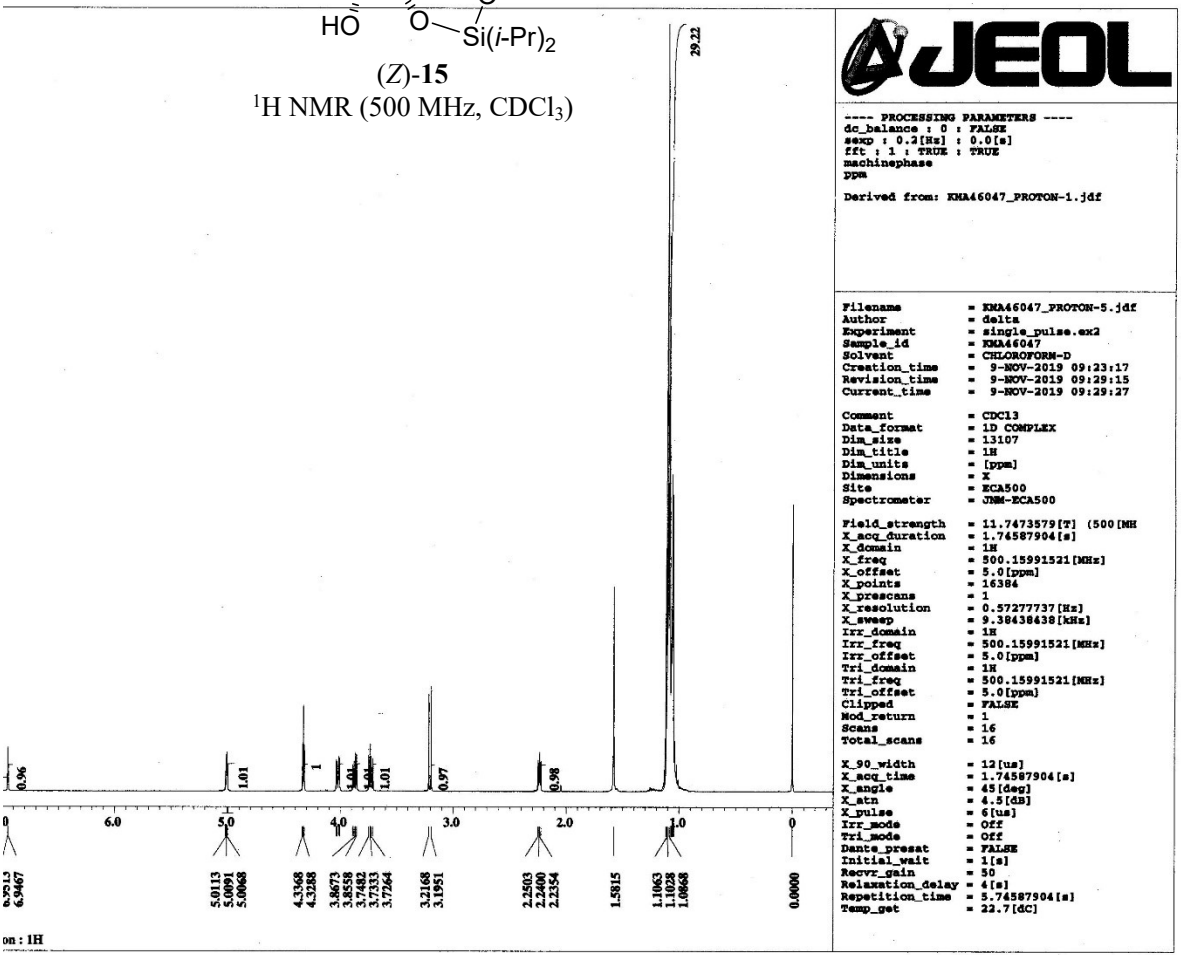
^{19}F NMR (470 MHz, $CDCl_3$)



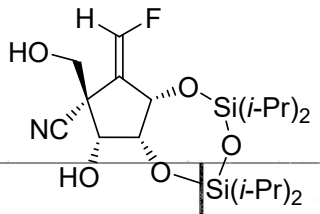


(*Z*)-**15**

^{1}H NMR (500 MHz, $CDCl_3$)

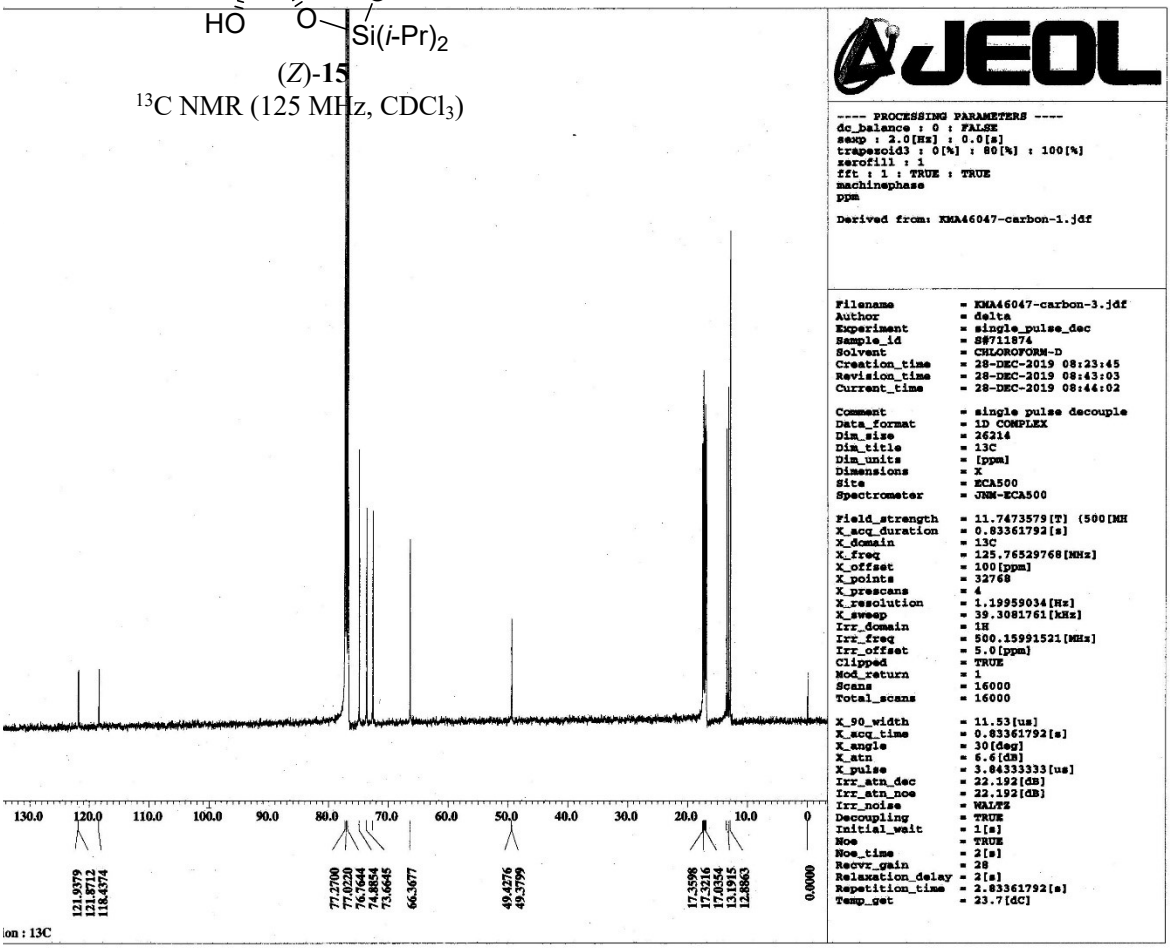


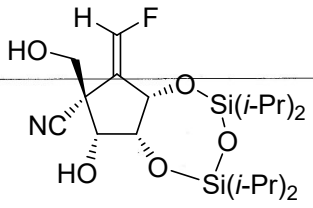

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46047_PROTON-1.jdf

Filename          = KMA46047_PROTON-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = KMA46047
Solvent           = CHLOROFORM-D
Creation_time     =  9-NOV-2019 09:23:17
Revision_time     =  9-NOV-2019 09:29:15
Current_time      =  9-NOV-2019 09:29:27

Comment           = CDCl3
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 6[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 22.7[dC]
```



(*Z*)-**15**

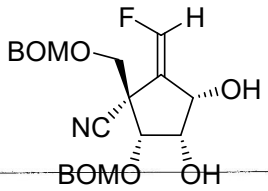
^{13}C NMR (125 MHz, $CDCl_3$)





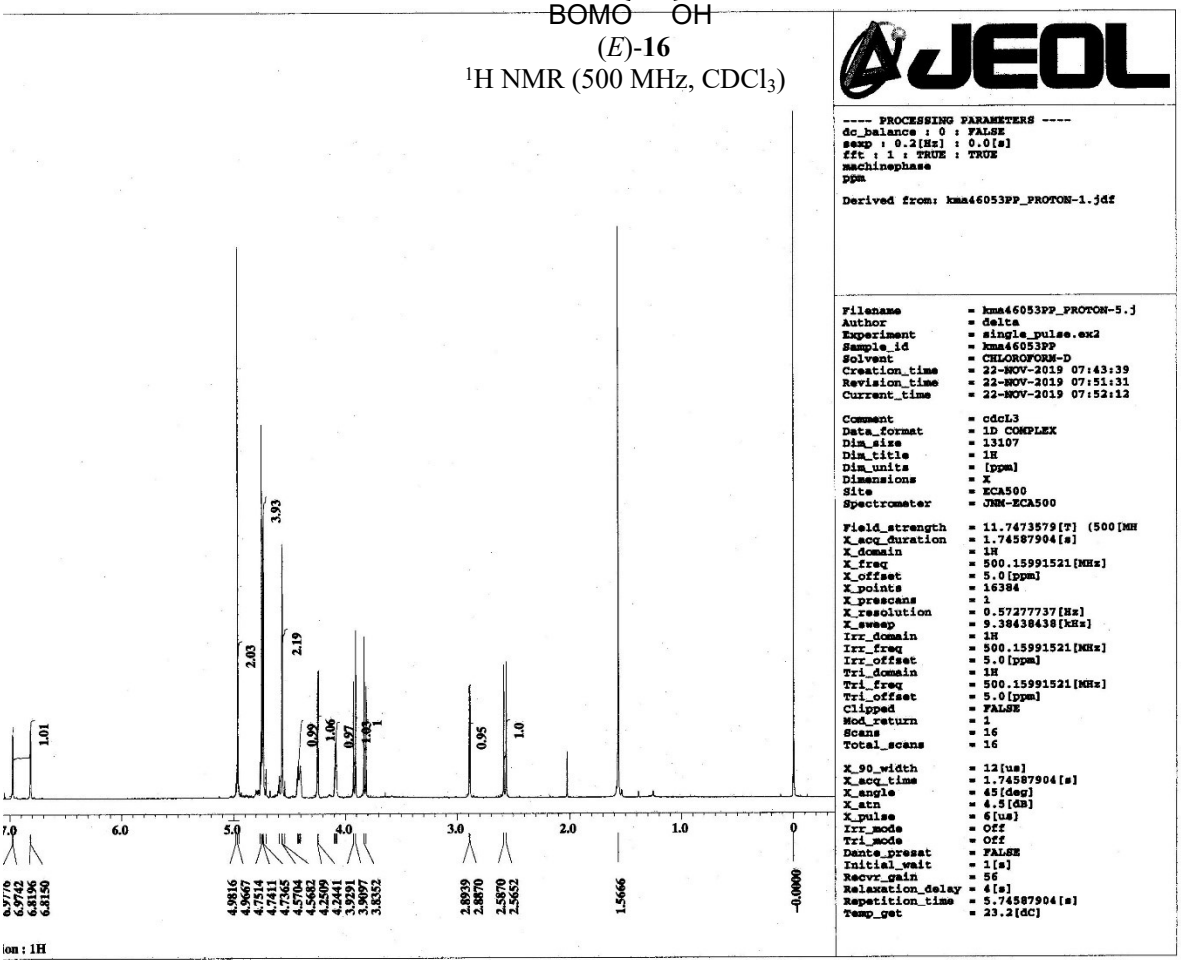
(*Z*)-**15**

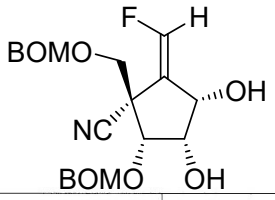
^{19}F NMR (470 MHz, $CDCl_3$)



(*E*)-**16**

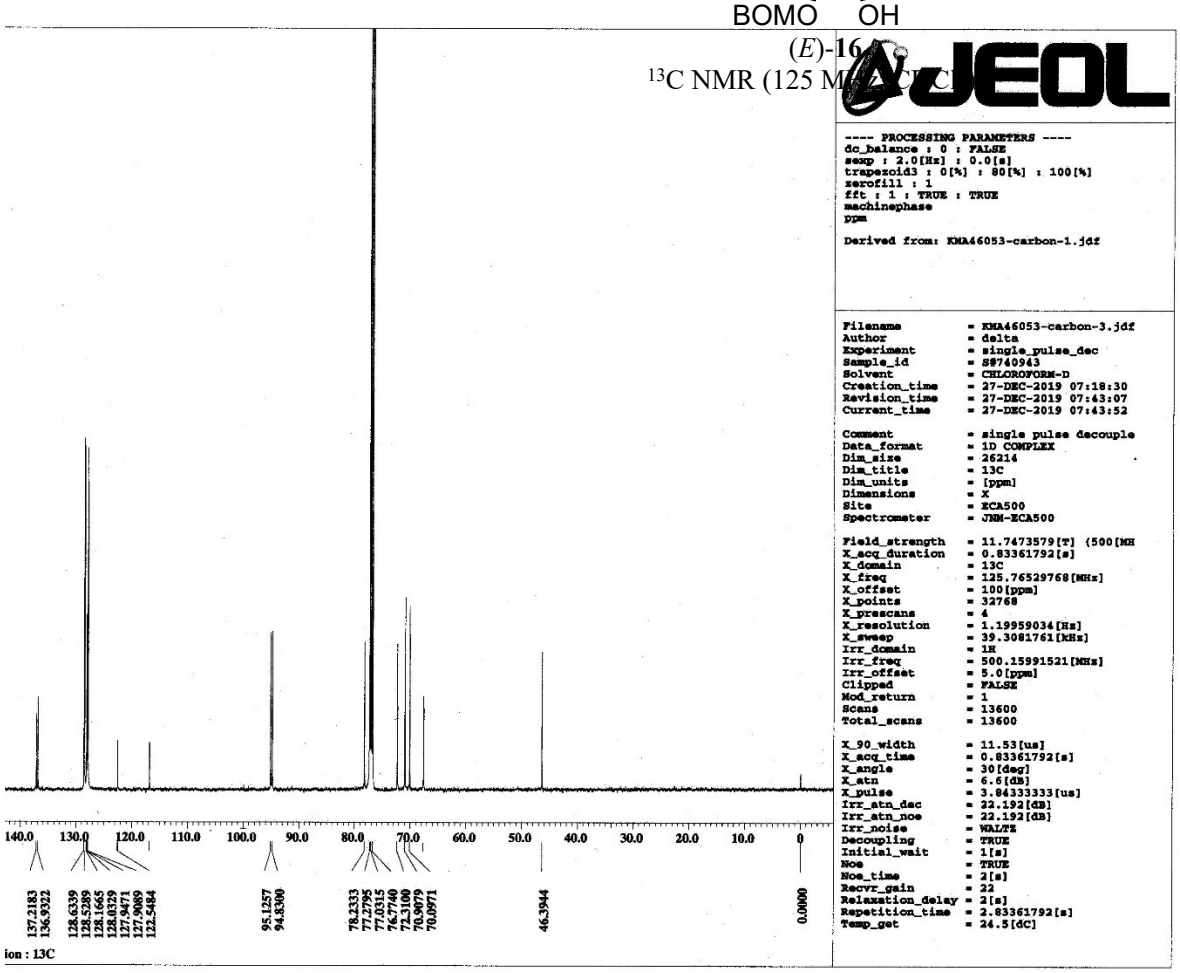
^{1}H NMR (500 MHz, $CDCl_3$)

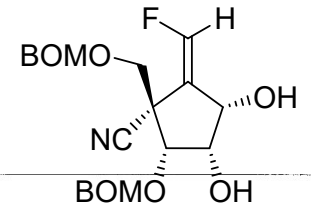




(*E*)-**16**

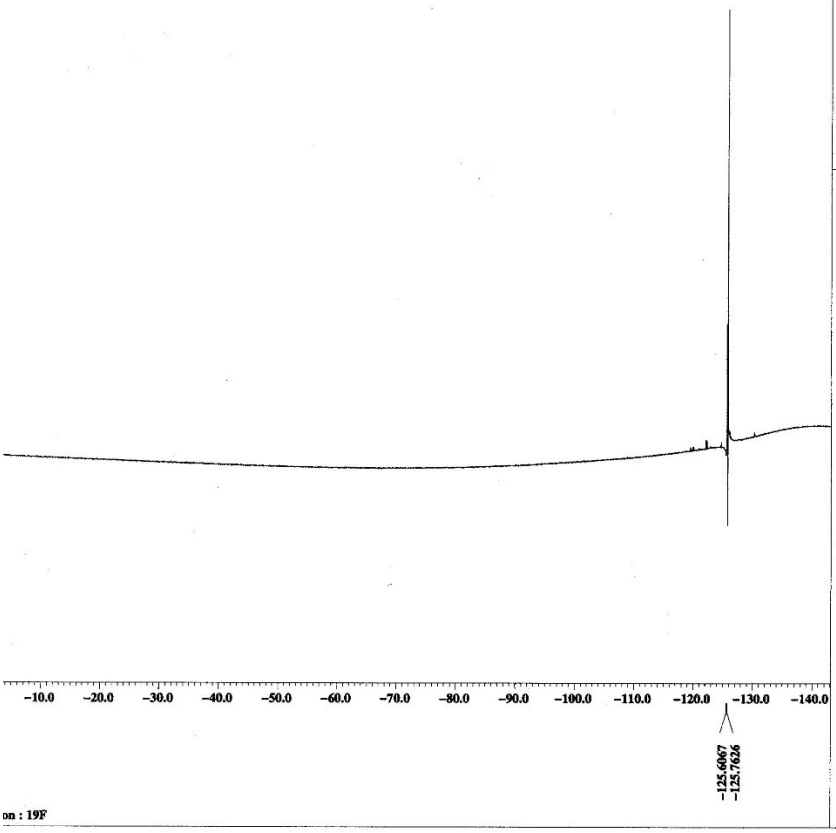
^{13}C NMR (125 MHz, CDCl$_3$)





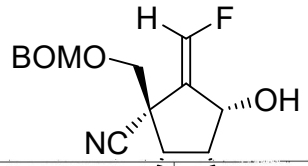
(*E*)-**16**

^{19}F NMR (470 MHz, $CDCl_3$)



JEOL

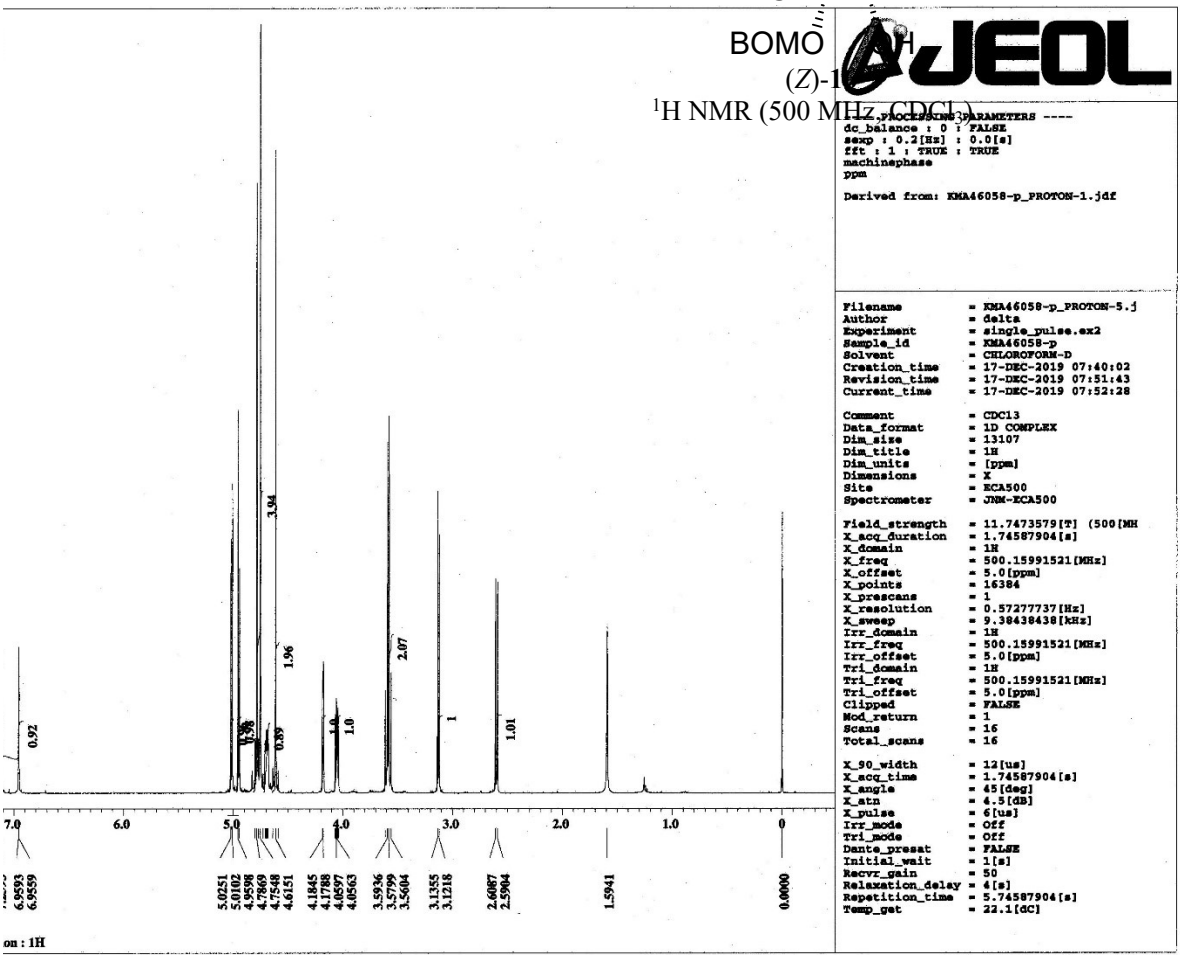
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA47108-19F-1.jdf

Filename          = KMA47108-19F-3.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#659829
Solvent           = CHLOROFORM-D
Creation_time     = 11-MAR-2021 18:21:05
Revision_time     = 11-MAR-2021 18:27:55
Current_time      = 11-MAR-2021 18:28:08

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 21.3[dC]
```



BOMO OH

(*Z*)-1

^{1}H NMR (500 MHz, CDCl$_3$)



---- ACCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

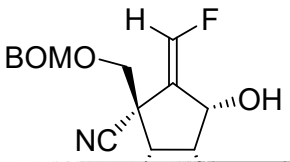
Derived from: KMA46058-p_PROTON-1.jdf

Filename = KMA46058-p_PROTON-5.j
Author = delta
Experiment = single_pulse.ex2
Sample_id = KMA46058-p
Solvent = CHLOROFORM-D
Creation_time = 17-DEC-2019 07:40:02
Revision_time = 17-DEC-2019 07:51:43
Current_time = 17-DEC-2019 07:52:28

Comment = CDCl3
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16

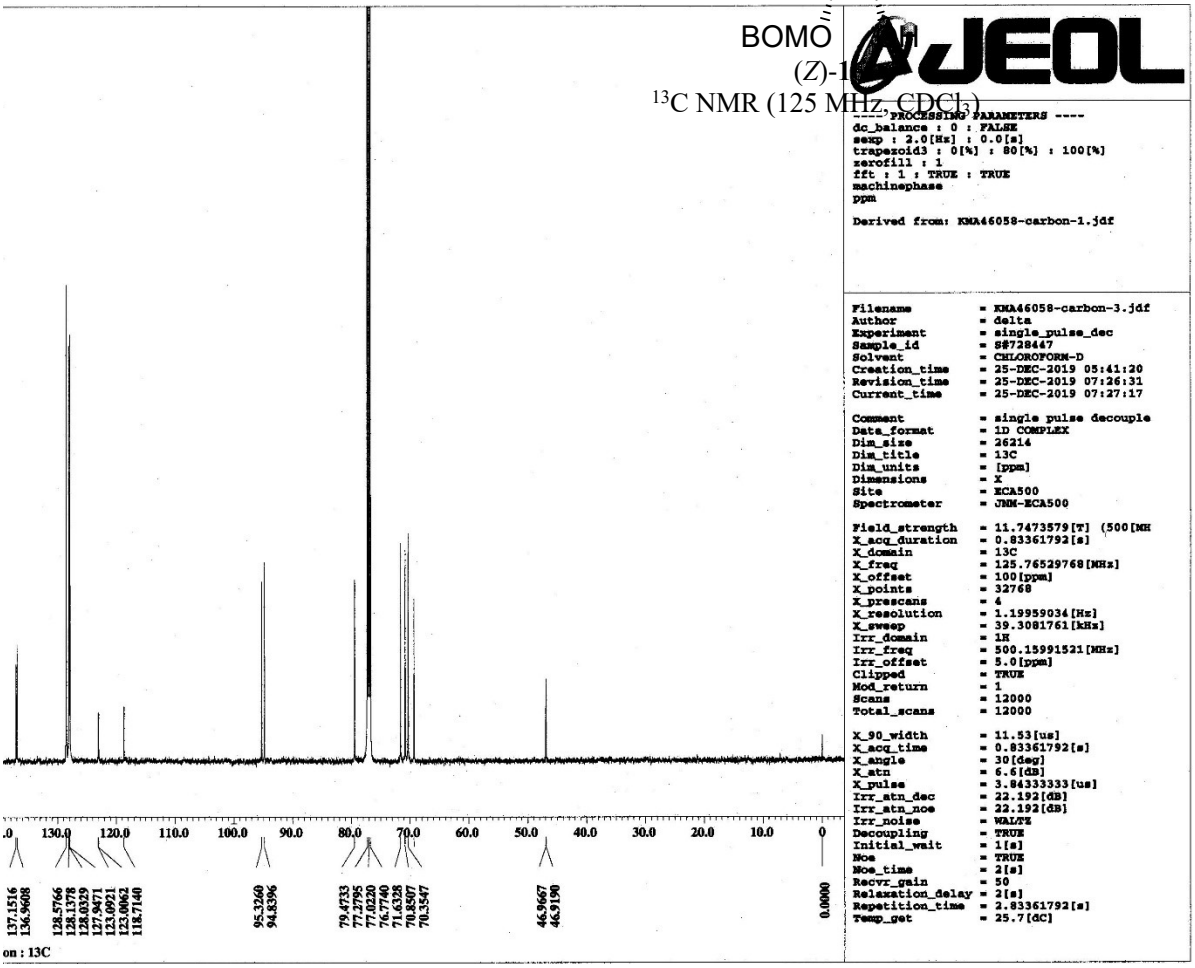
X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4.5[dB]
X_pulse = 6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 5.74587904[s]
Temp_get = 22.1[dC]

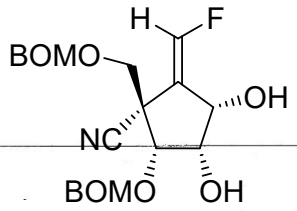


BOMO

(*Z*)-1

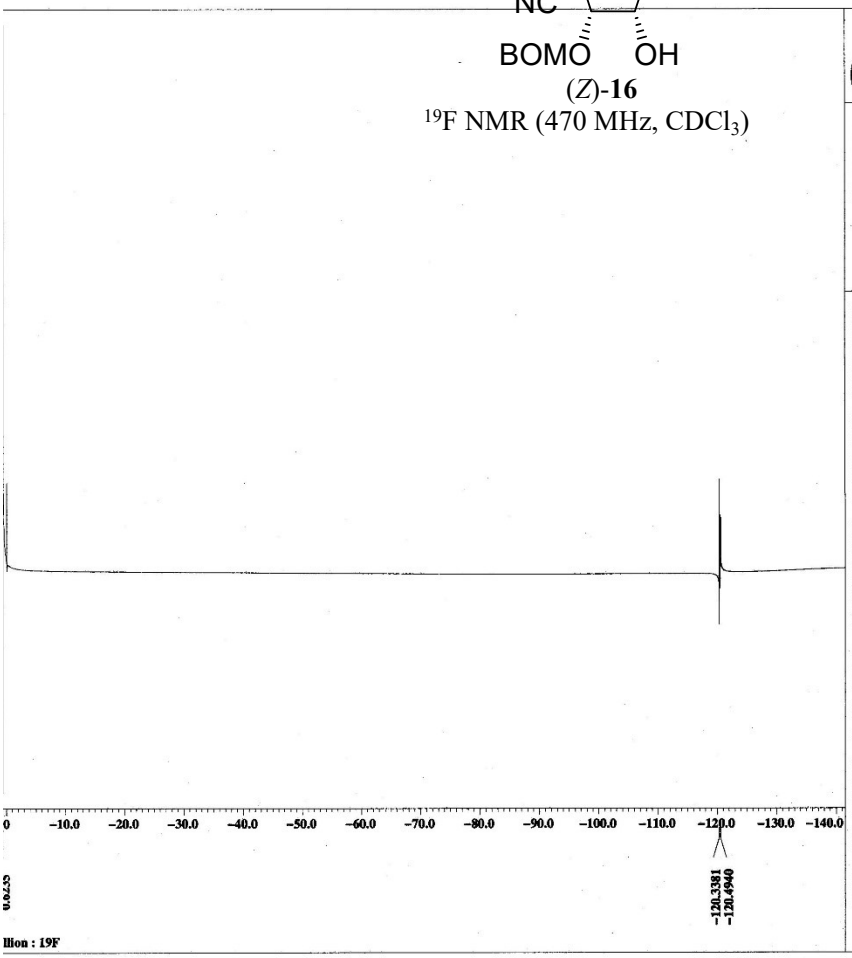
^{13}C NMR (125 MHz, $CDCl_3$)





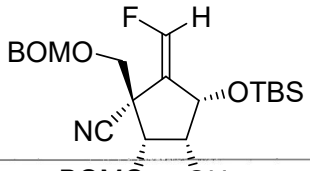
(*Z*)-**16**

^{19}F NMR (470 MHz, $CDCl_3$)



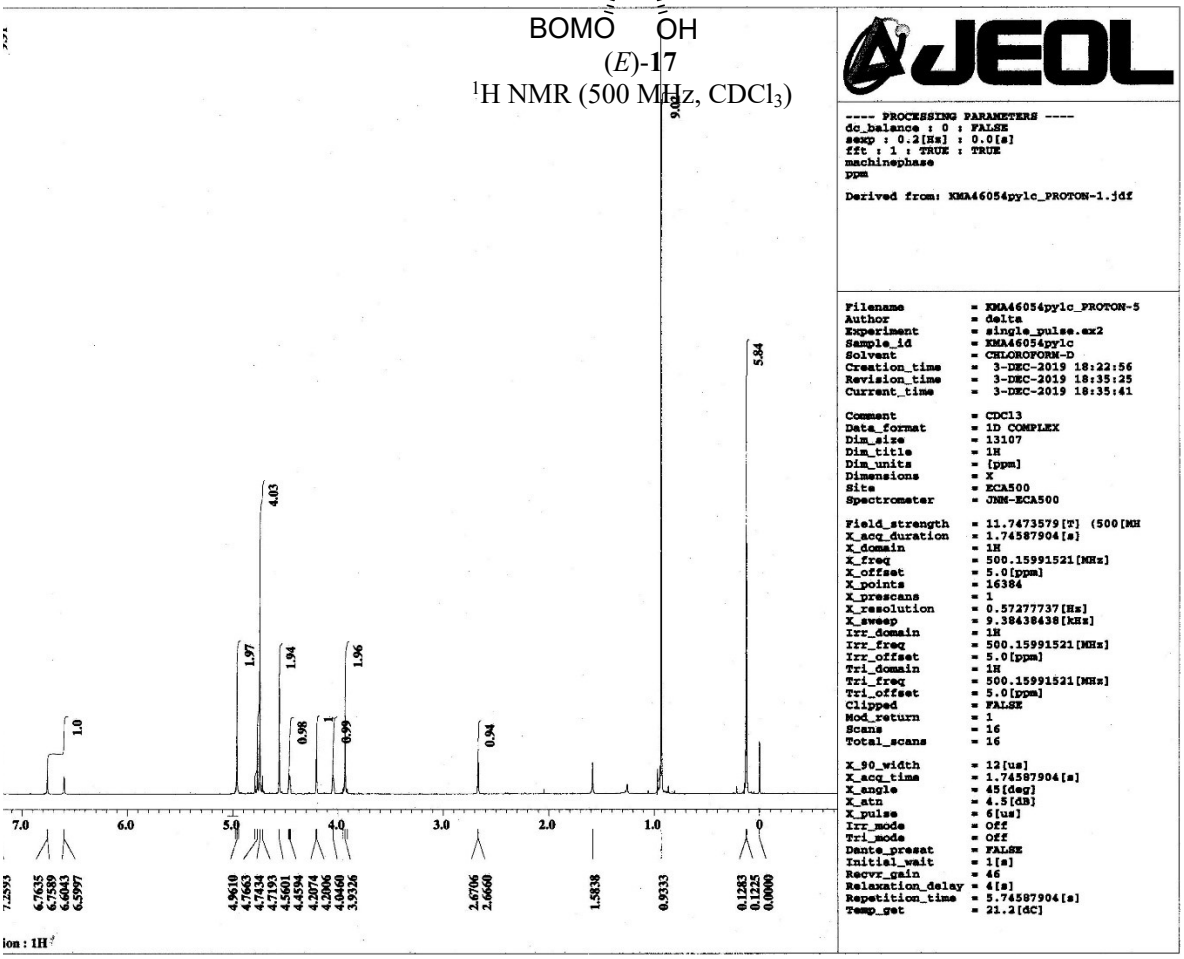

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA16058-19F-1.jdf
```

```
Filename          = KMA16058-19F-3.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#403382
Solvent           = CHLOROFORM-D
Creation_time     = 11-JAN-2020 11:17:37
Revision_time     = 11-JAN-2020 11:22:47
Current_time      = 11-JAN-2020 11:23:00

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 25.6[dC]
```



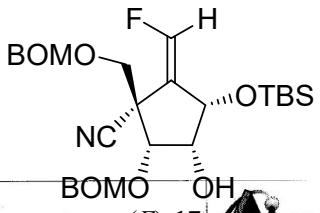
(*E*)-**17**

^{1}H NMR (500 MHz, $CDCl_3$)



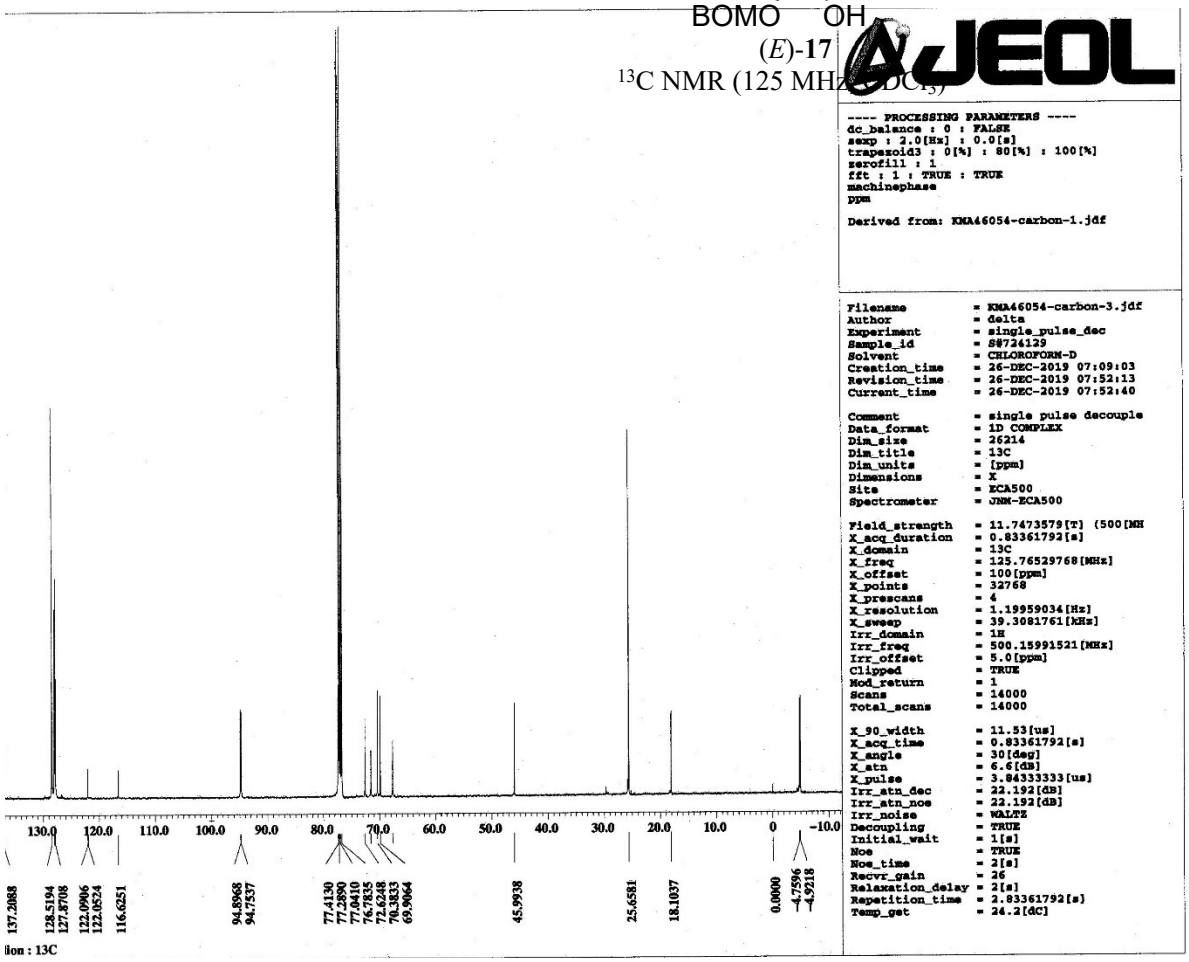
JEOL

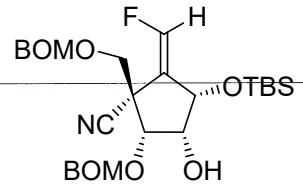
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46054pylc_PROTON-1.jdf

Filename          = KMA46054pylc_PROTON-5
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = KMA46054pylc
Solvent           = CHLOROFORM-D
Creation_time     =  3-DEC-2019 18:22:56
Revision_time     =  3-DEC-2019 18:35:25
Current_time      =  3-DEC-2019 18:35:41

Comment           = CDCl3
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 6[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 4[s]
Repetition_time   = 5.74587904[s]
Temp_get          = 21.2[dC]
```



(*E*)-**17**

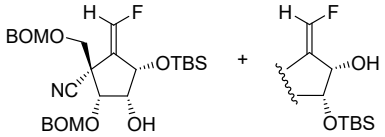
^{13}C NMR (125 MHz, $CDCl_3$)





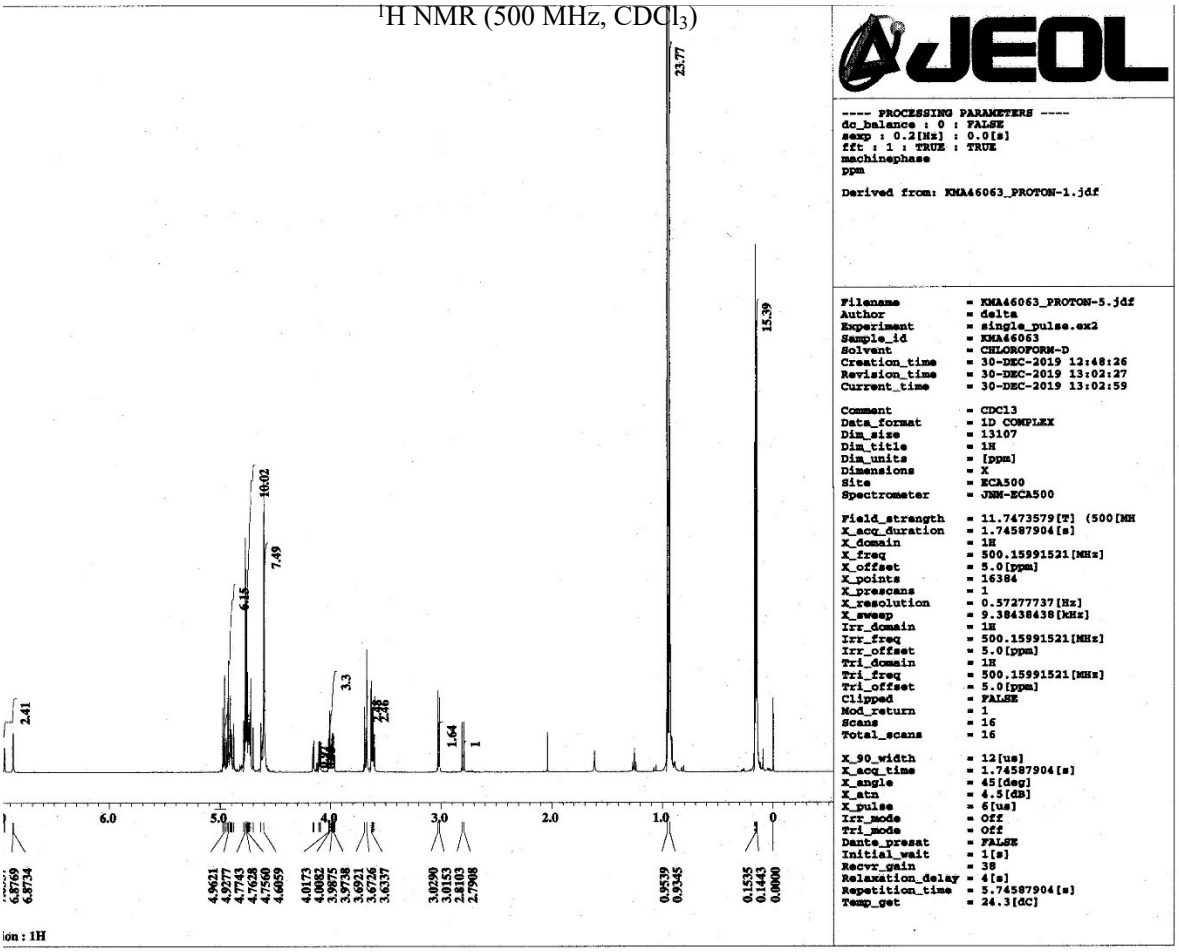
(*E*)-**17**

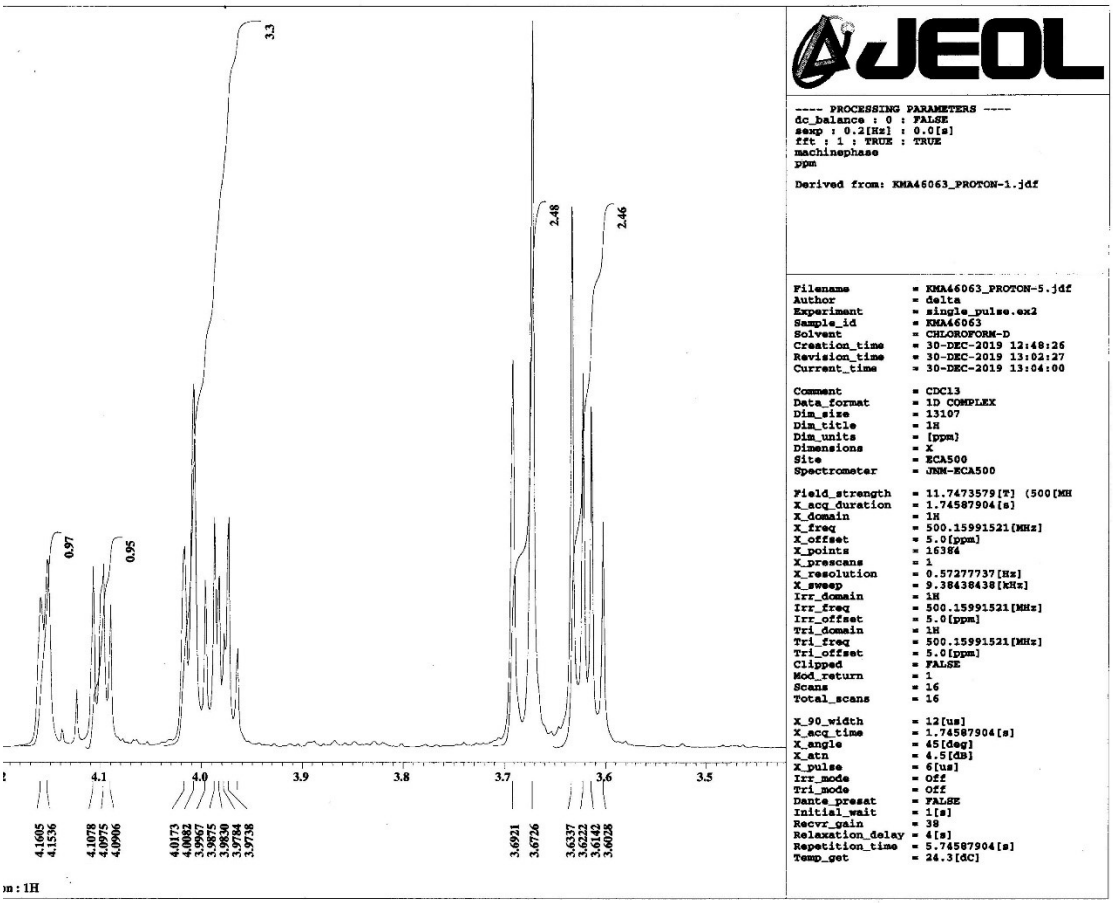
^{19}F NMR (470 MHz, $CDCl_3$)



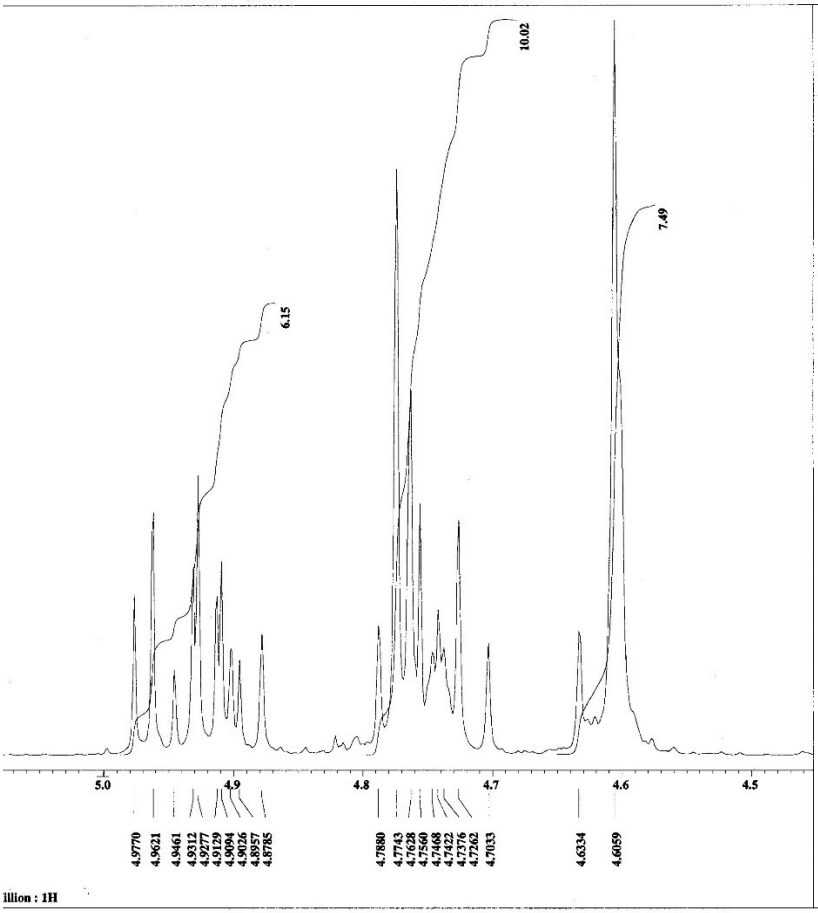
(*Z*)-**17** and (*Z*)-**17a**

^{1}H NMR (500 MHz, $CDCl_3$)



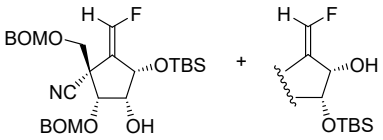

3.3
2.48
2.46
0.97
0.95
4.1
4.0
3.9
3.8
3.7
3.6
3.5
4.1605
4.1536
4.1078
4.0975
4.0906
4.0173
4.0082
3.9967
3.9875
3.9830
3.9784
3.9738
3.6921
3.6726
3.6337
3.6222
3.6127
3.6028
on : 1H

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: KMA46063_PROTON-1.jdf
Filename = KMA46063_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = KMA46063
Solvent = CHLOROFORM-D
Creation_time = 30-DEC-2019 12:48:26
Revision_time = 30-DEC-2019 13:02:27
Current_time = 30-DEC-2019 13:04:00
Comment = CDCl3
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4.5[dB]
X_pulse = 6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 4[s]
Repetition_time = 5.74587904[s]
Temp_get = 24.3[dC]



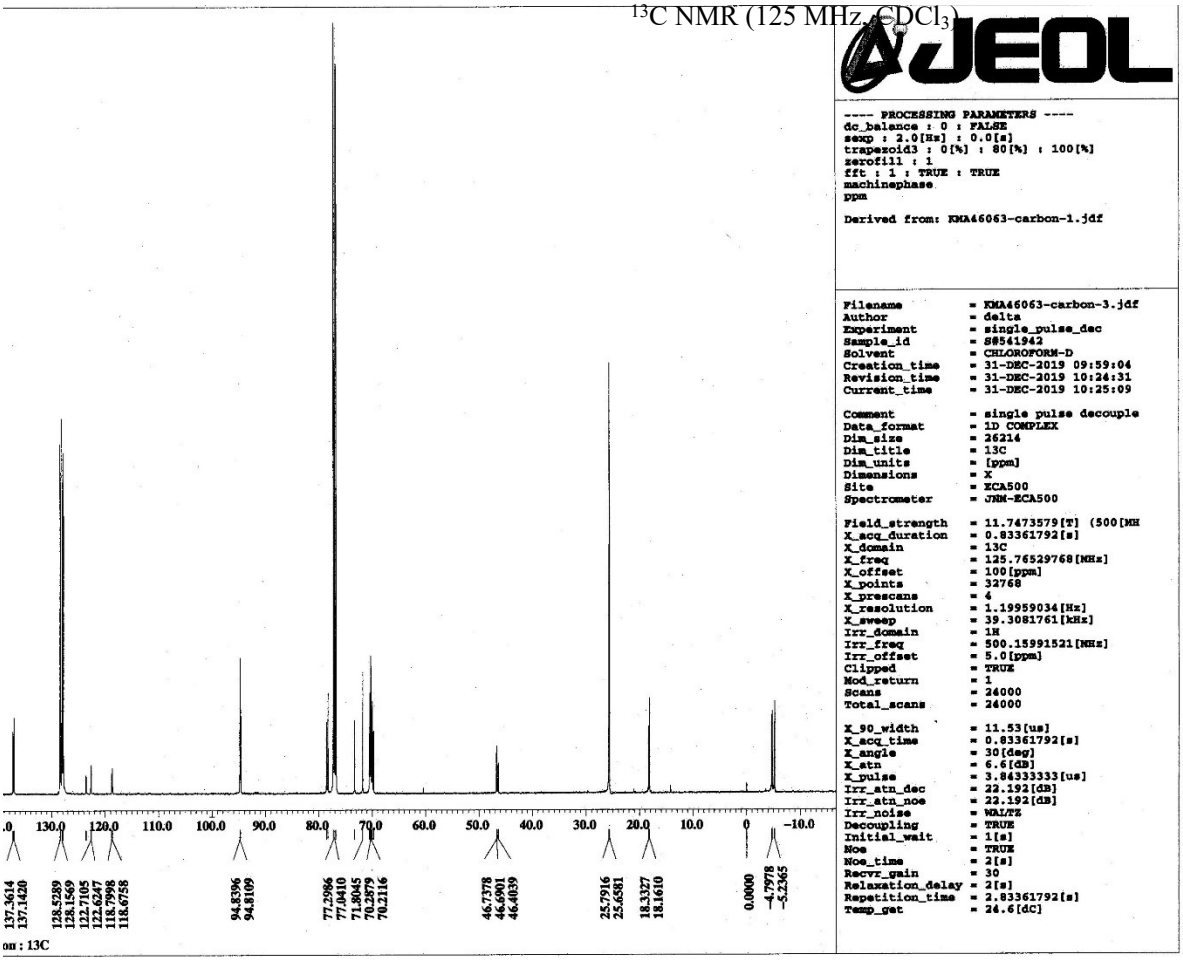
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46063_PROTON-1.jdf
```

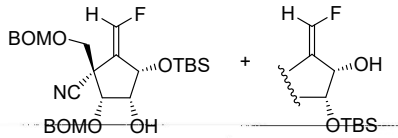
Filename	= KMA46063_PROTON-5.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= KMA46063
Solvent	= CHLOROFORM-D
Creation_time	= 30-DEC-2019 12:48:26
Revision_time	= 30-DEC-2019 13:02:27
Current_time	= 30-DEC-2019 13:03:36
Comment	= CDCl3
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 12[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4.5[dB]
X_pulse	= 6[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 4[s]
Repetition_time	= 5.74587904[s]
Temp_get	= 24.3[dC]



(*Z*)-**17** and (*Z*)-**17a**

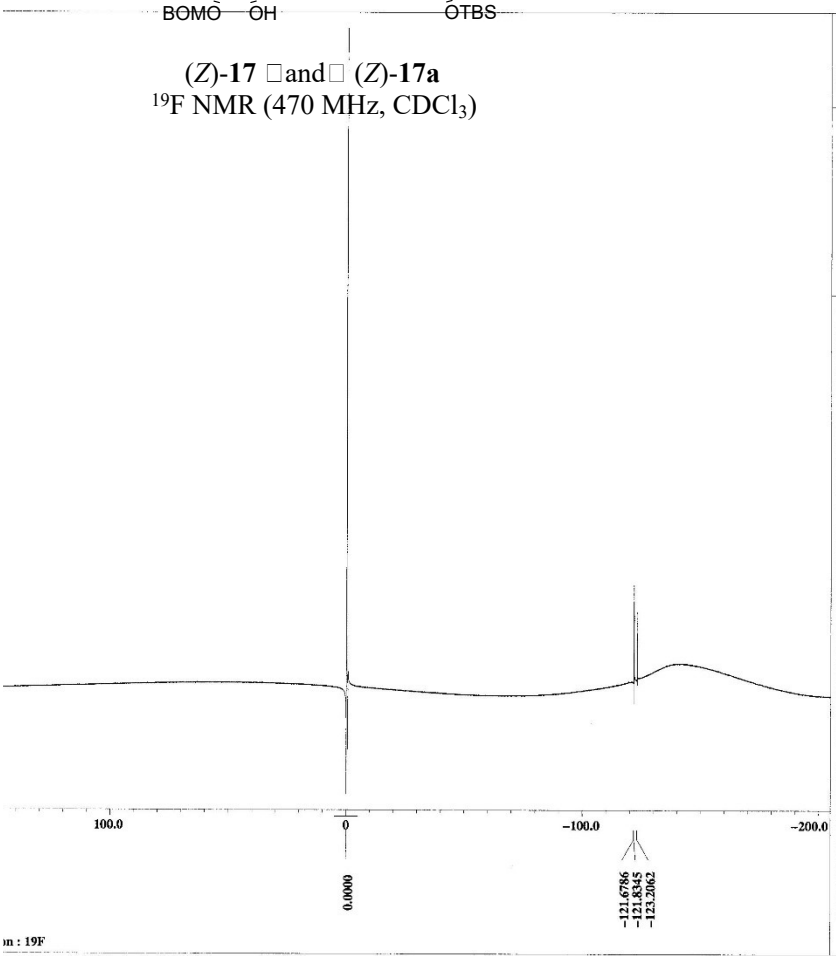
^{13}C NMR (125 MHz, $CDCl_3$)





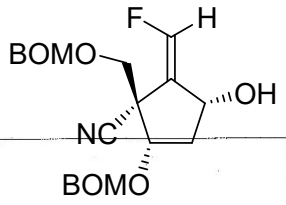
(*Z*)-**17** and (*Z*)-**17a**

^{19}F NMR (470 MHz, $CDCl_3$)



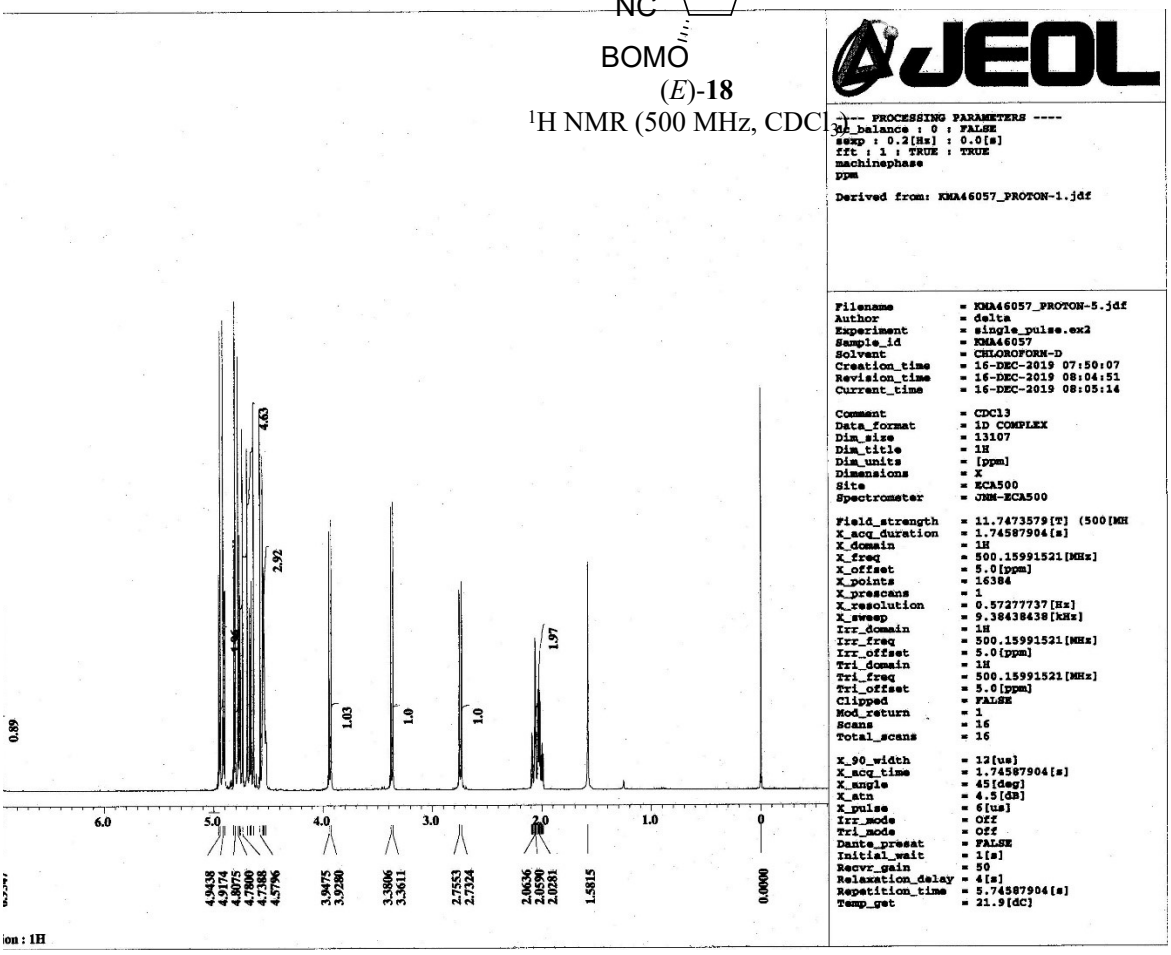
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46063-CFCl3-1.jdf
```

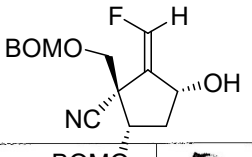
```
Filename          = KMA46063-CFCl3-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#486078
Solvent           = CHLOROFORM-D
Creation_time     = 17-AUG-2021 13:23:21
Revision_time     = 17-AUG-2021 13:41:39
Current_time      = 17-AUG-2021 13:41:50

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 60
Total_scans       = 60

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 20.7[dC]
```



(*E*)-**18**

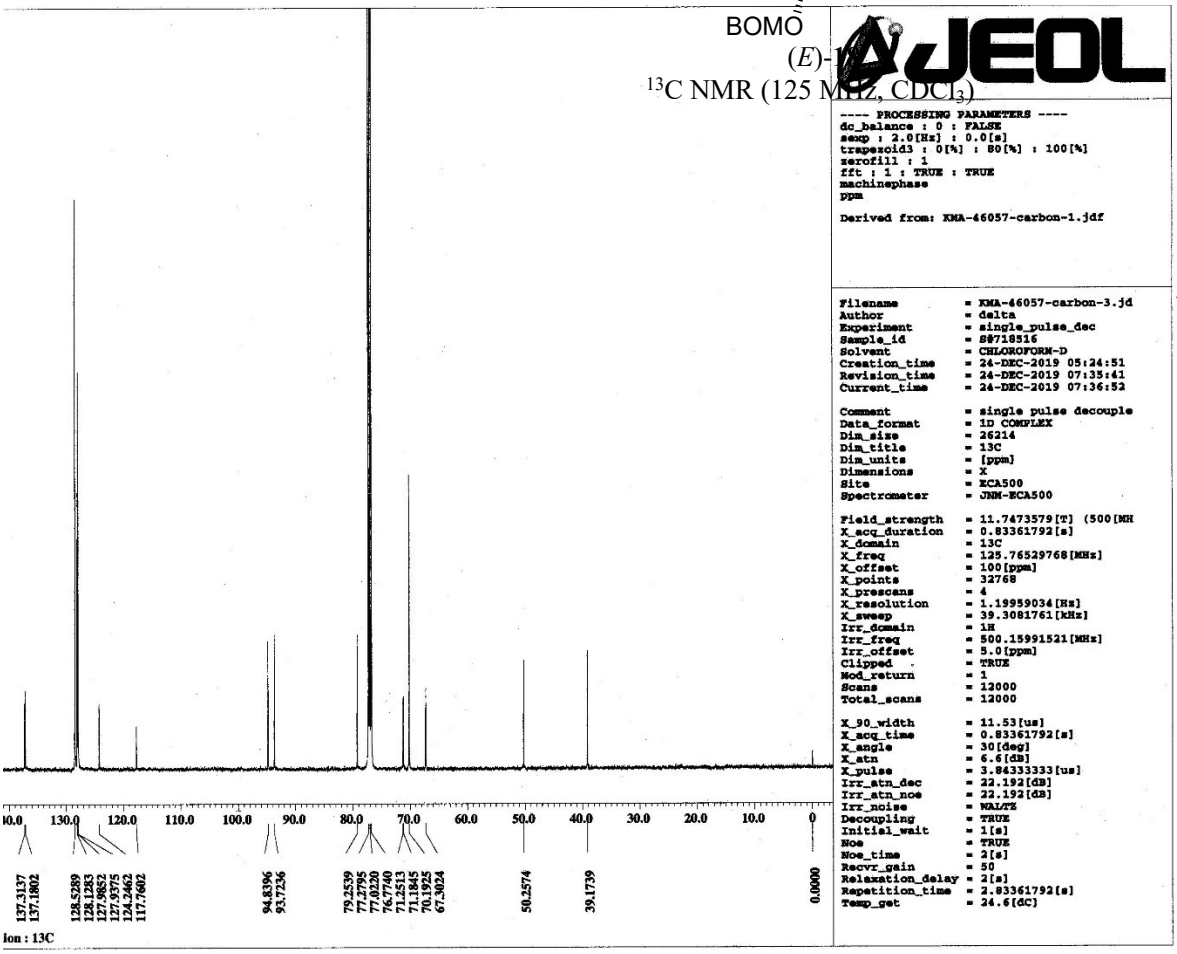
^{1}H NMR (500 MHz, $CDCl_3$)

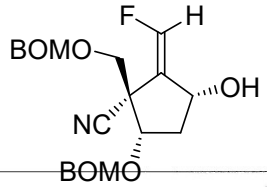




(*E*)-

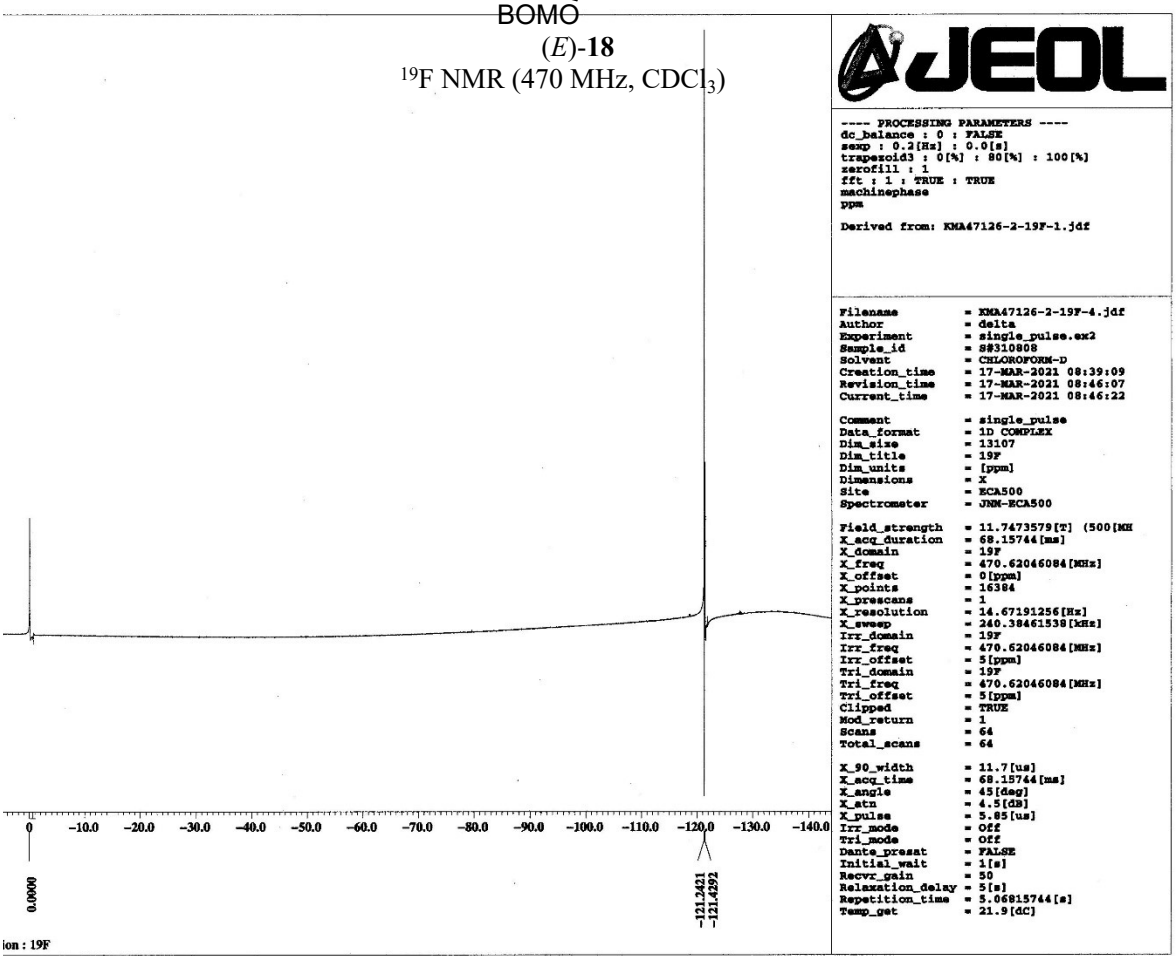
^{13}C NMR (125 MHz, $CDCl_3$)





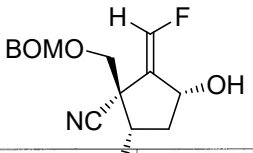
(*E*)-**18**

^{19}F NMR (470 MHz, $CDCl_3$)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA47126-2-19F-1.jdf
```

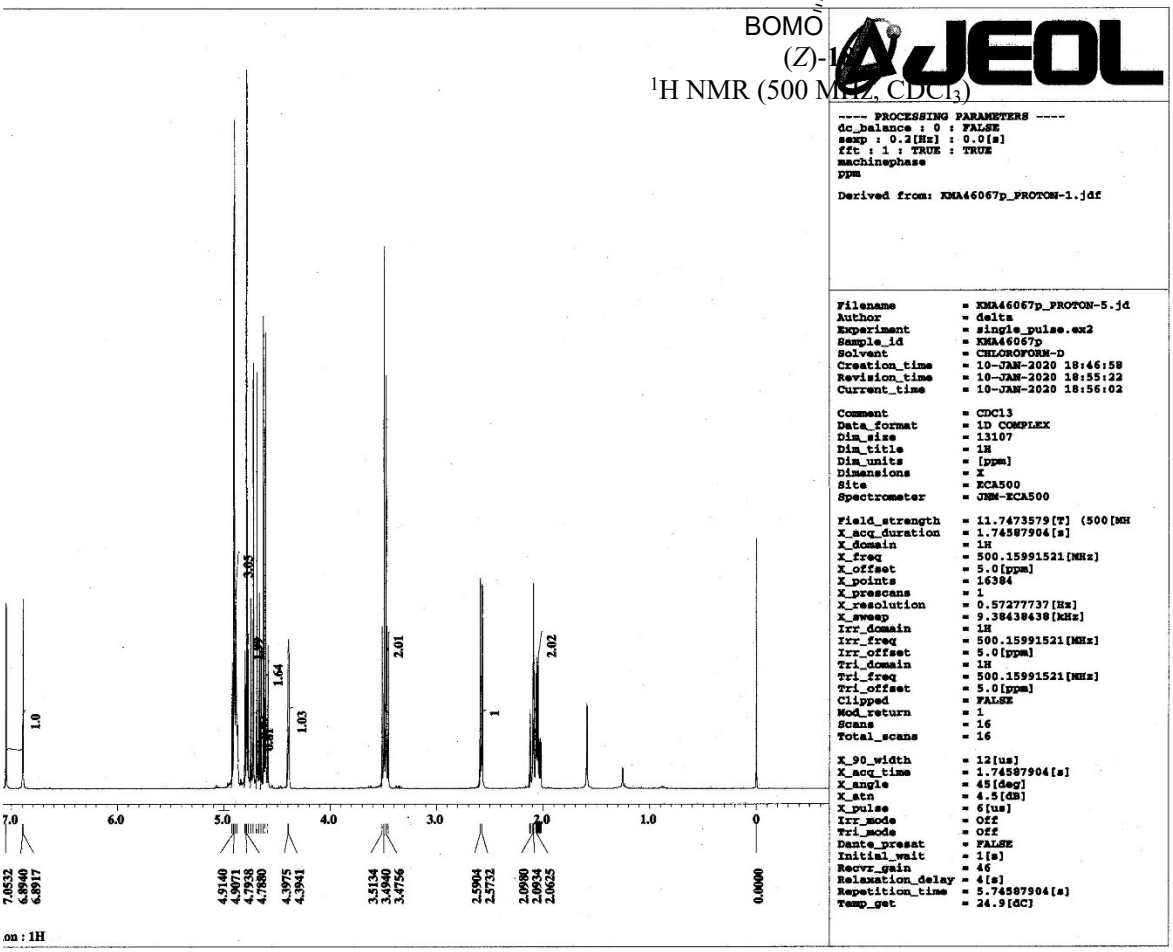
```
Filename          = KMA47126-2-19F-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#3108008
Solvent           = CHLOROFORM-D
Creation_time     = 17-MAR-2021 08:39:09
Revision_time     = 17-MAR-2021 08:46:07
Current_time      = 17-MAR-2021 08:46:22

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 21.9[dC]
```

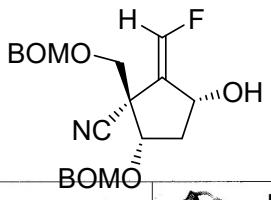


BOMO

(*Z*)-1

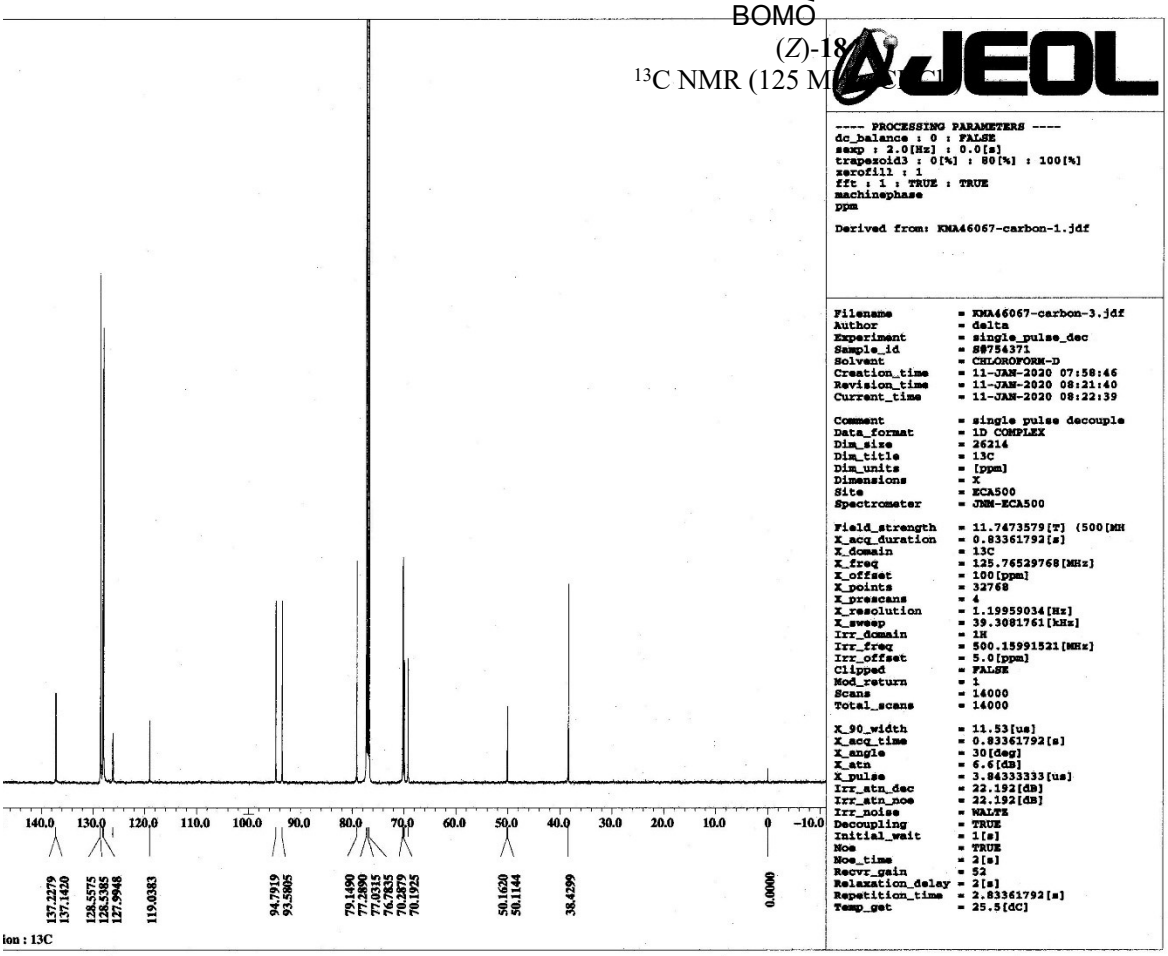
^{1}H NMR (500 MHz, $CDCl_3$)

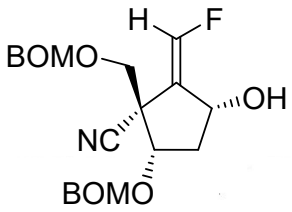




(*Z*)-18

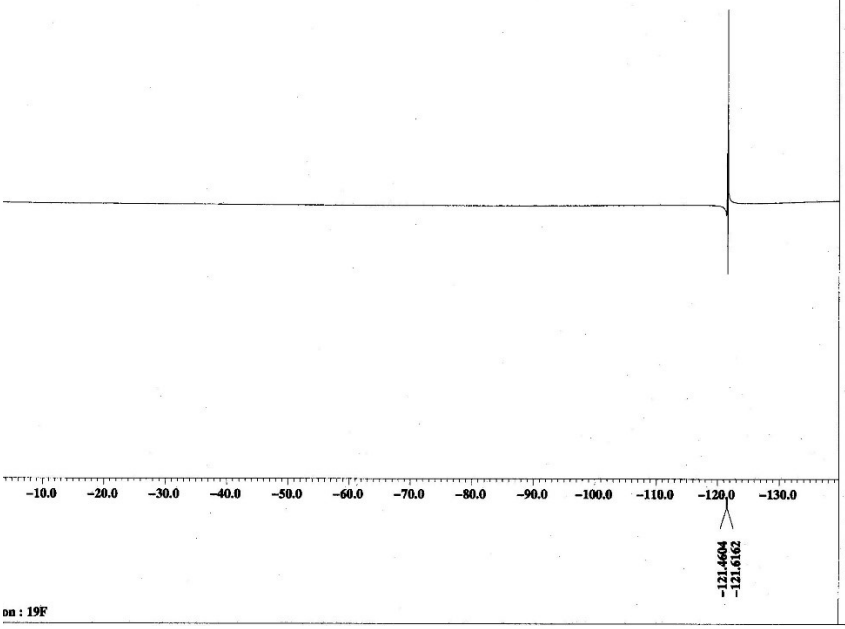
^{13}C NMR (125 M





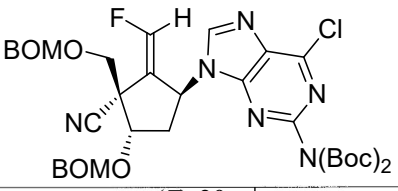
(*Z*)-**18**

^{19}F NMR (470 MHz, $CDCl_3$)



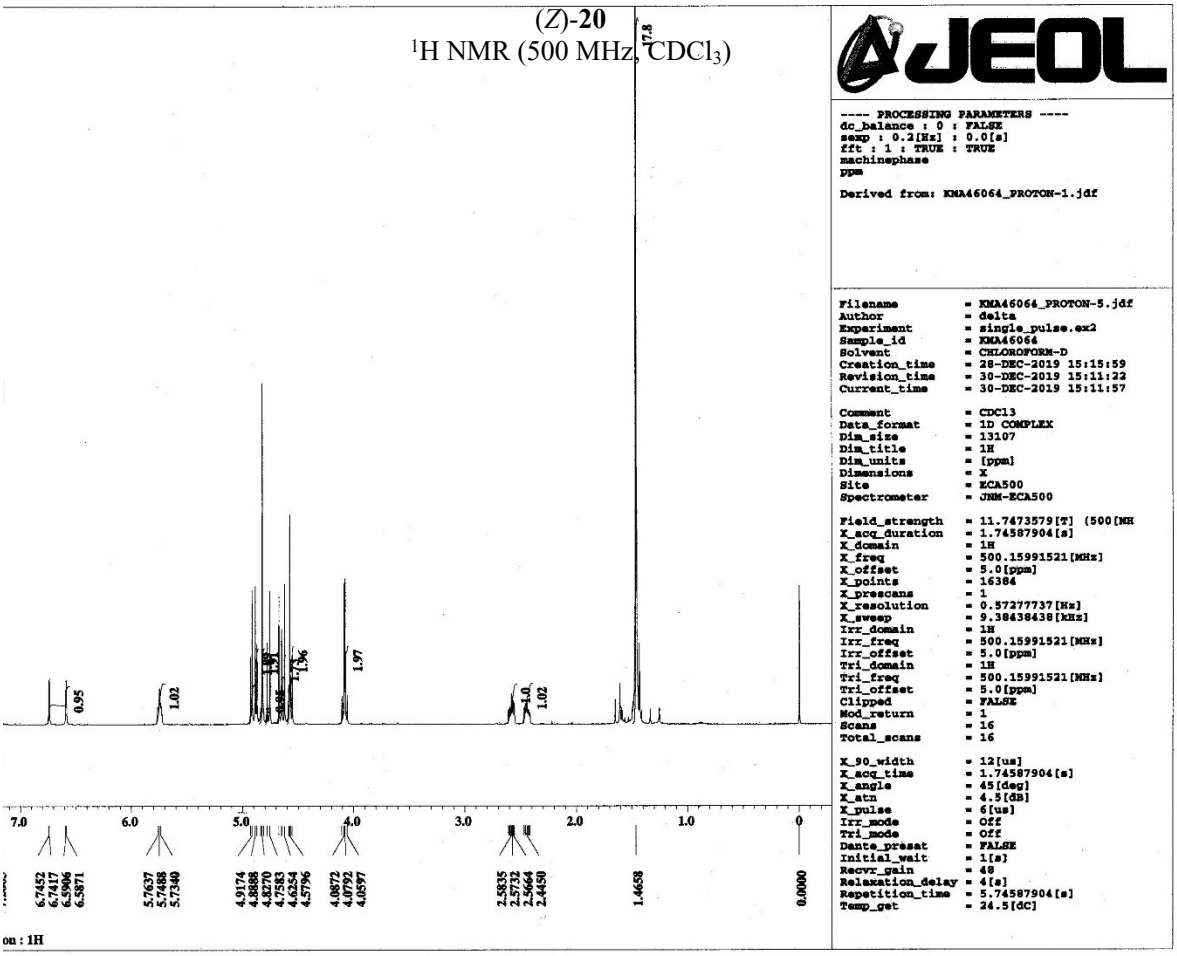

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46067-19F-1.jdf
```

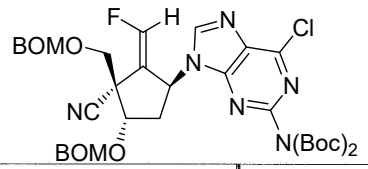
```
Filename          = KMA46067-19F-3.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S8409054
Solvent           = CHLOROFORM-D
Creation_time     = 11-JAN-2020 11:27:39
Revision_time     = 11-JAN-2020 11:30:22
Current_time      = 11-JAN-2020 11:30:49

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 25.6[dC]
```



(*Z*)-**20**

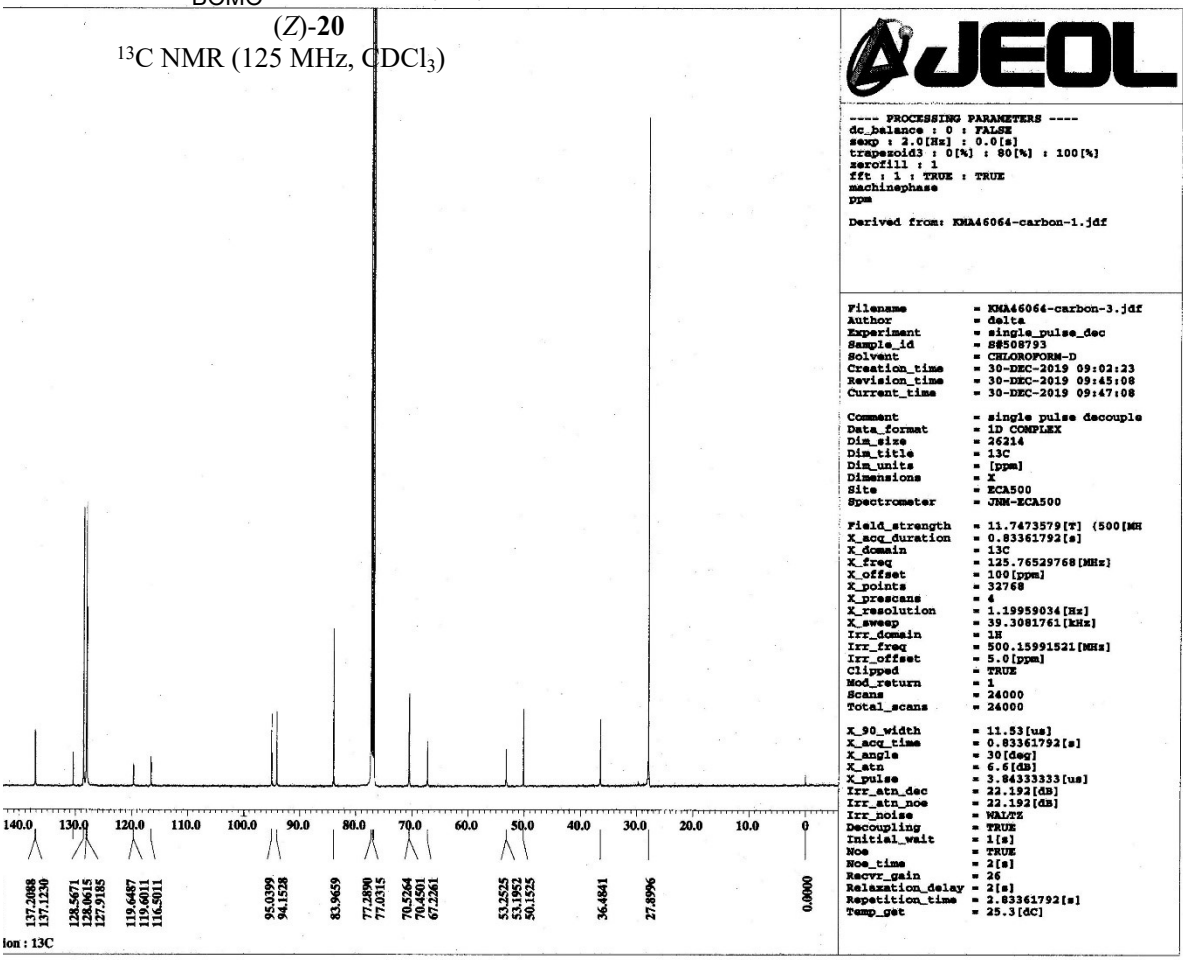
^{1}H NMR (500 MHz, $CDCl_3$)

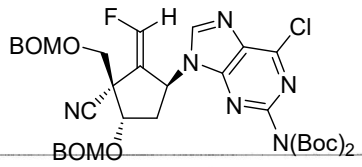




(*Z*)-**20**

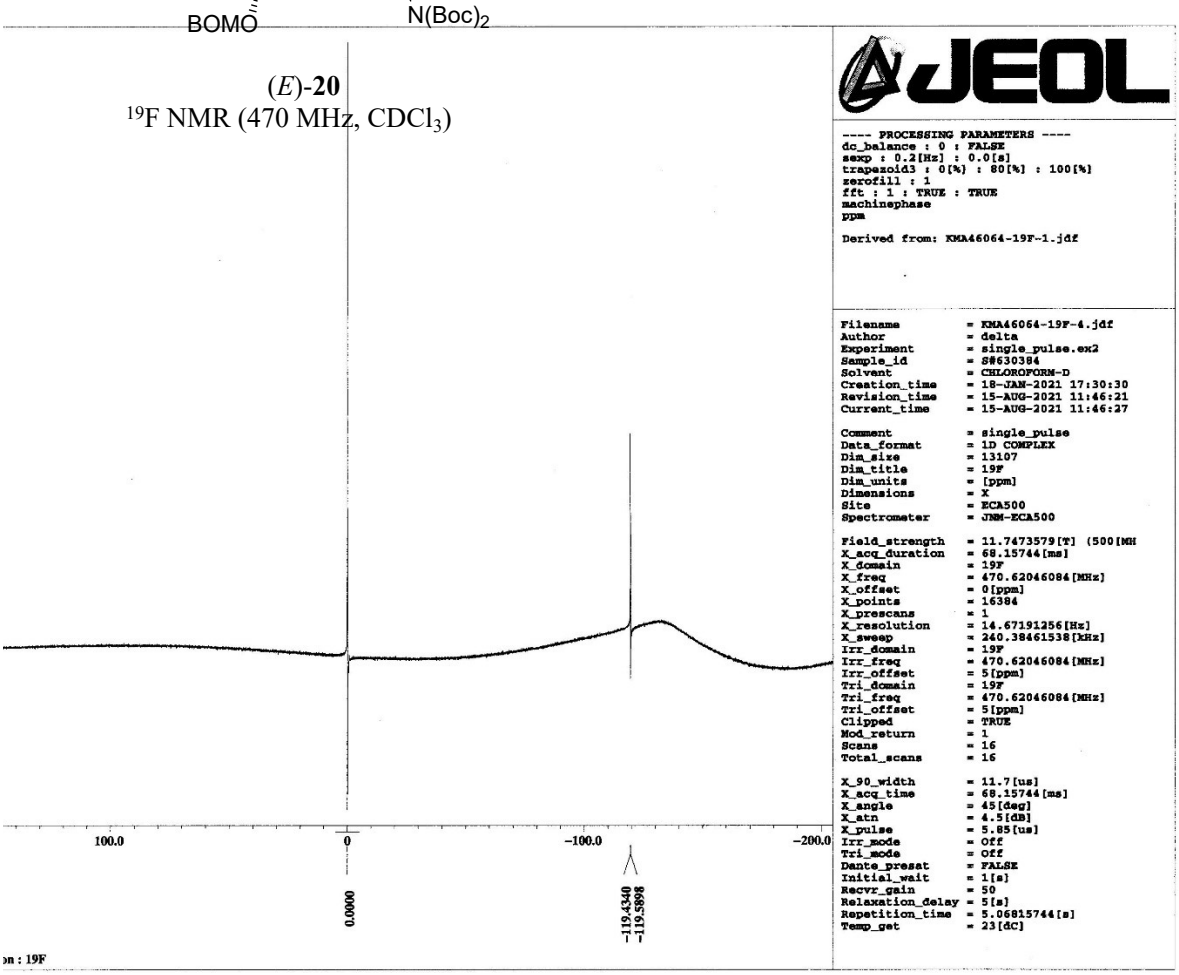
^{13}C NMR (125 MHz, $CDCl_3$)





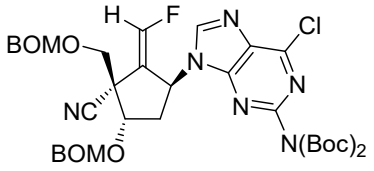
(*E*)-**20**

^{19}F NMR (470 MHz, $CDCl_3$)



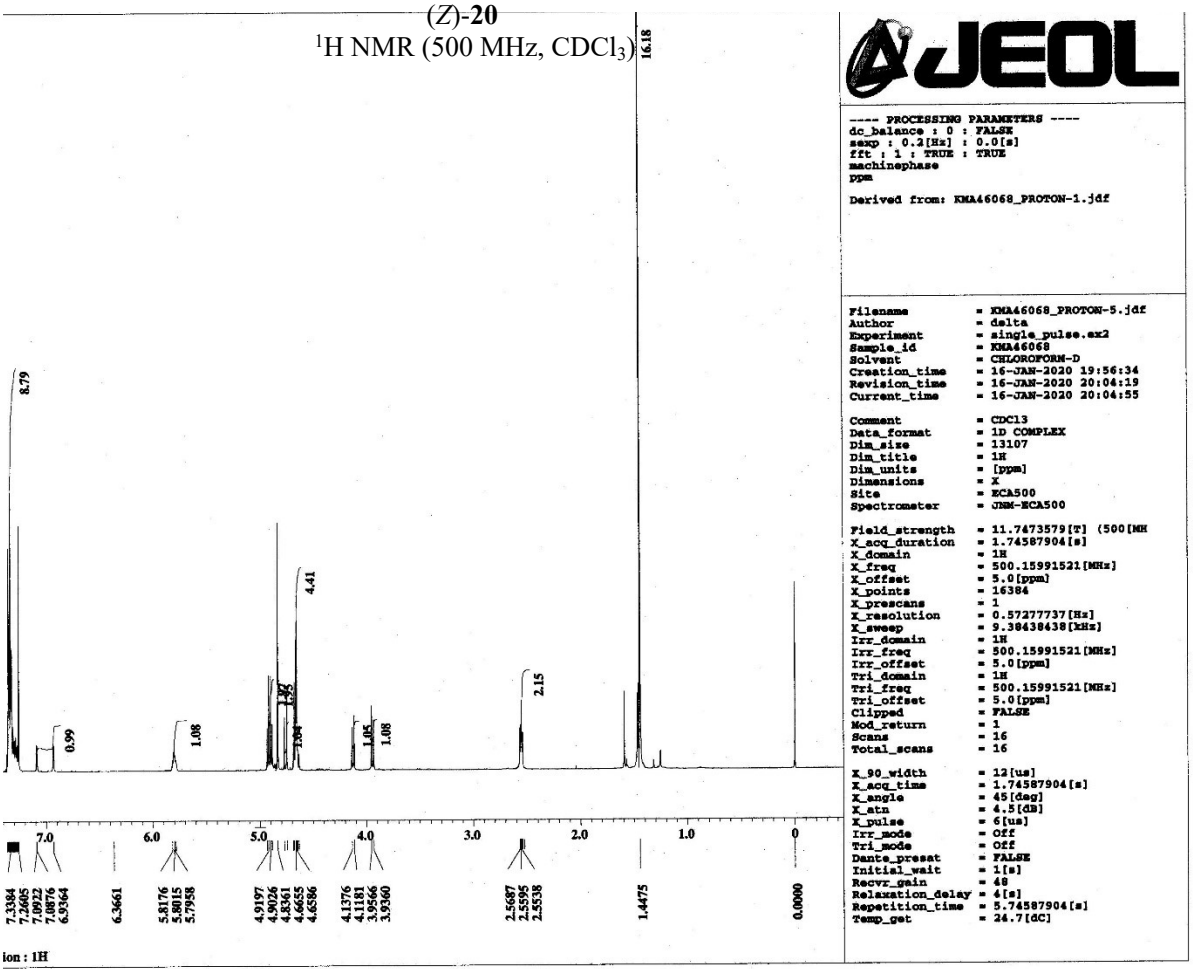

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46064-19F-1.jdf
```

```
Filename          = KMA46064-19F-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#630384
Solvent           = CHLOROFORM-D
Creation_time     = 18-JAN-2021 17:30:30
Revision_time     = 15-AUG-2021 11:46:21
Current_time      = 15-AUG-2021 11:46:27

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 23[dC]
```



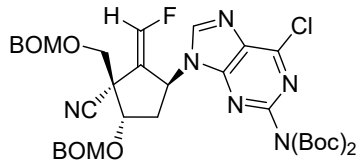
(*Z*)-**20**

^{1}H NMR (500 MHz, $CDCl_3$)



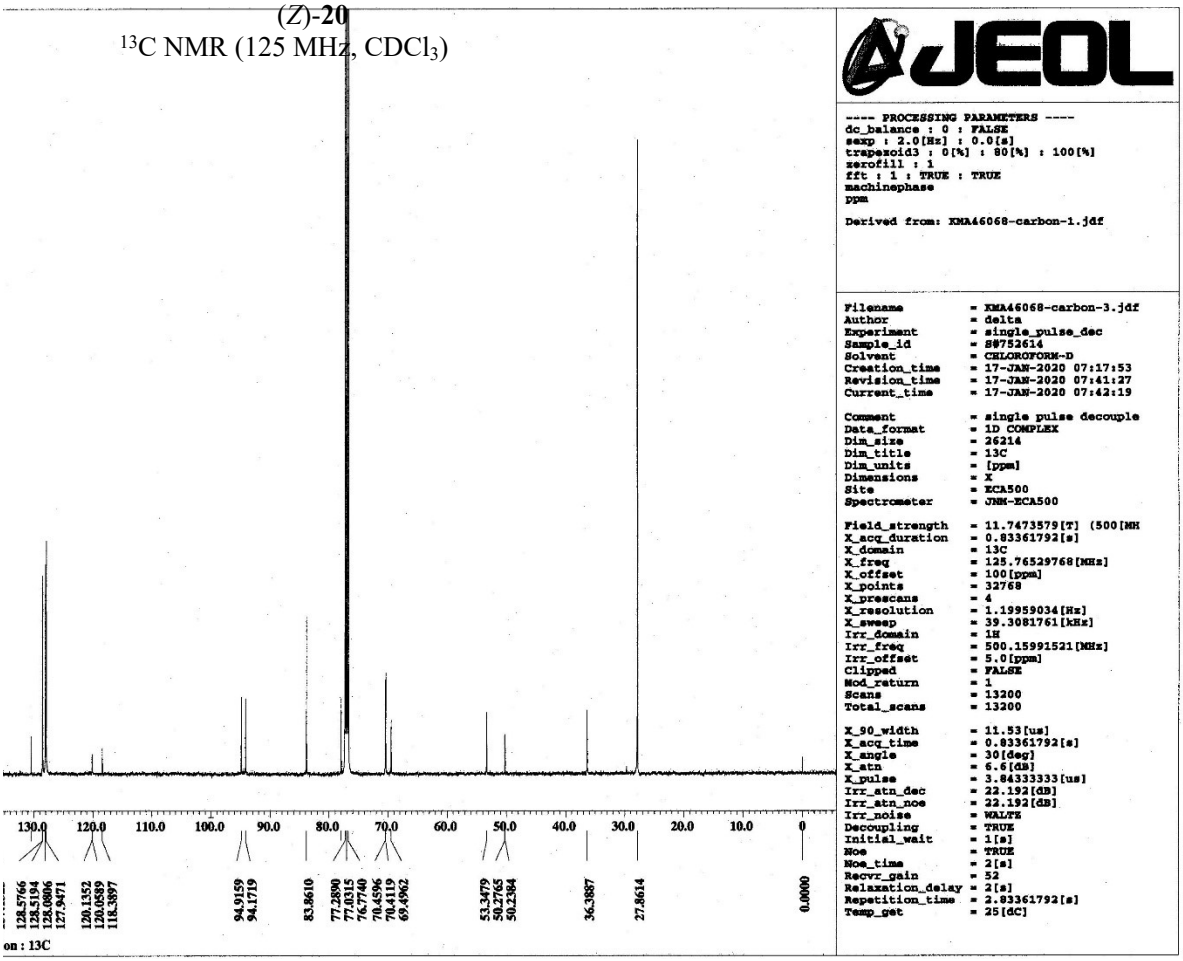

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA46068_PROTON-1.jdf
```

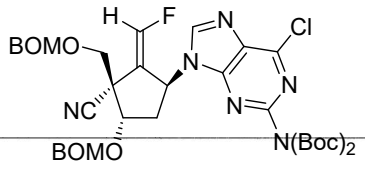
Filename	= KMA46068_PROTON-5.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= KMA46068
Solvent	= CHLOROFORM-D
Creation_time	= 16-JAN-2020 19:56:34
Revision_time	= 16-JAN-2020 20:04:19
Current_time	= 16-JAN-2020 20:04:55
Comment	= CDCl3
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 12[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4.5[dB]
X_pulse	= 6[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 48
Relaxation_delay	= 4[s]
Repetition_time	= 5.74587904[s]
Temp_get	= 24.7[dC]



(*Z*)-**20**

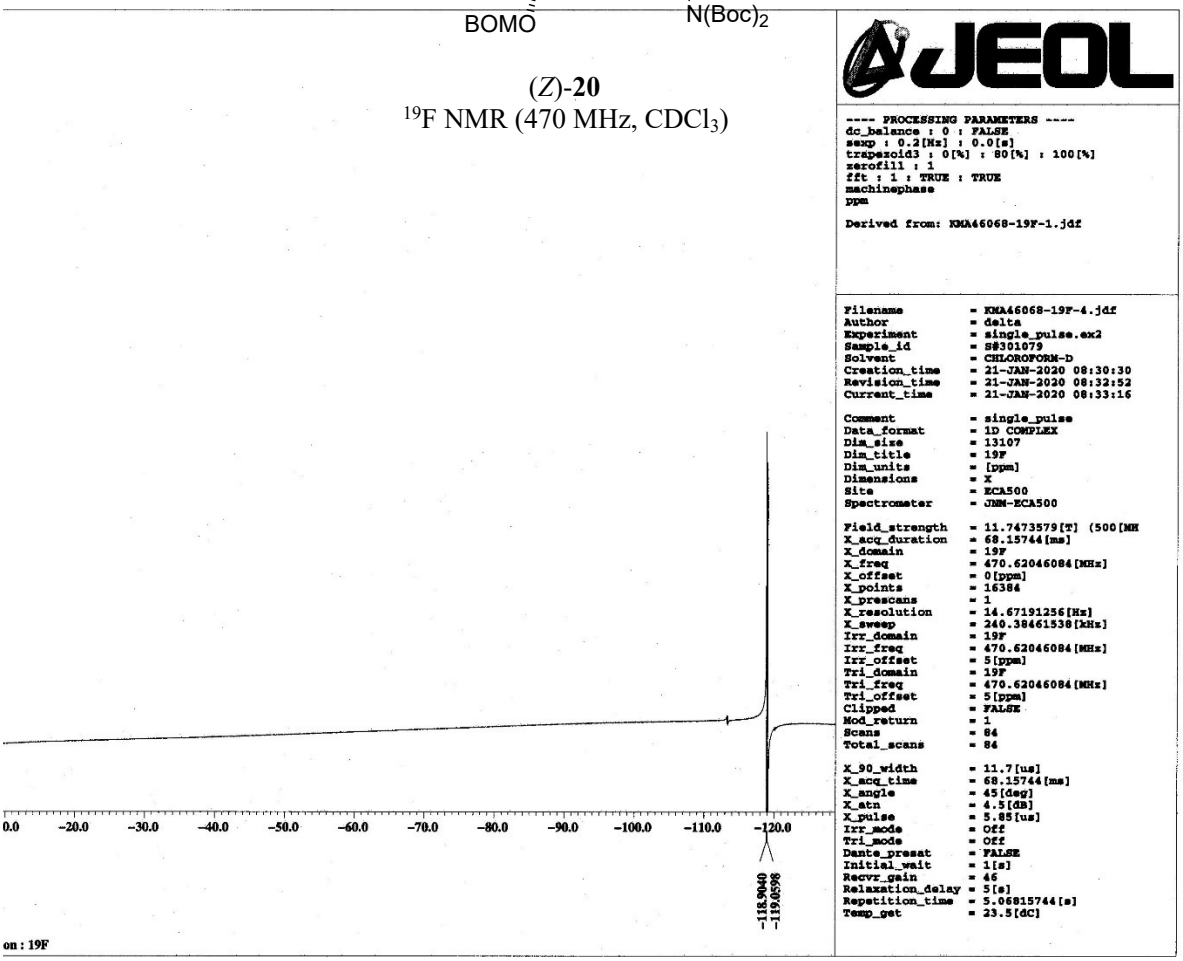
^{13}C NMR (125 MHz, $CDCl_3$)

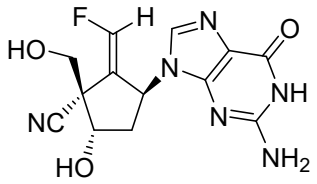




(*Z*)-**20**

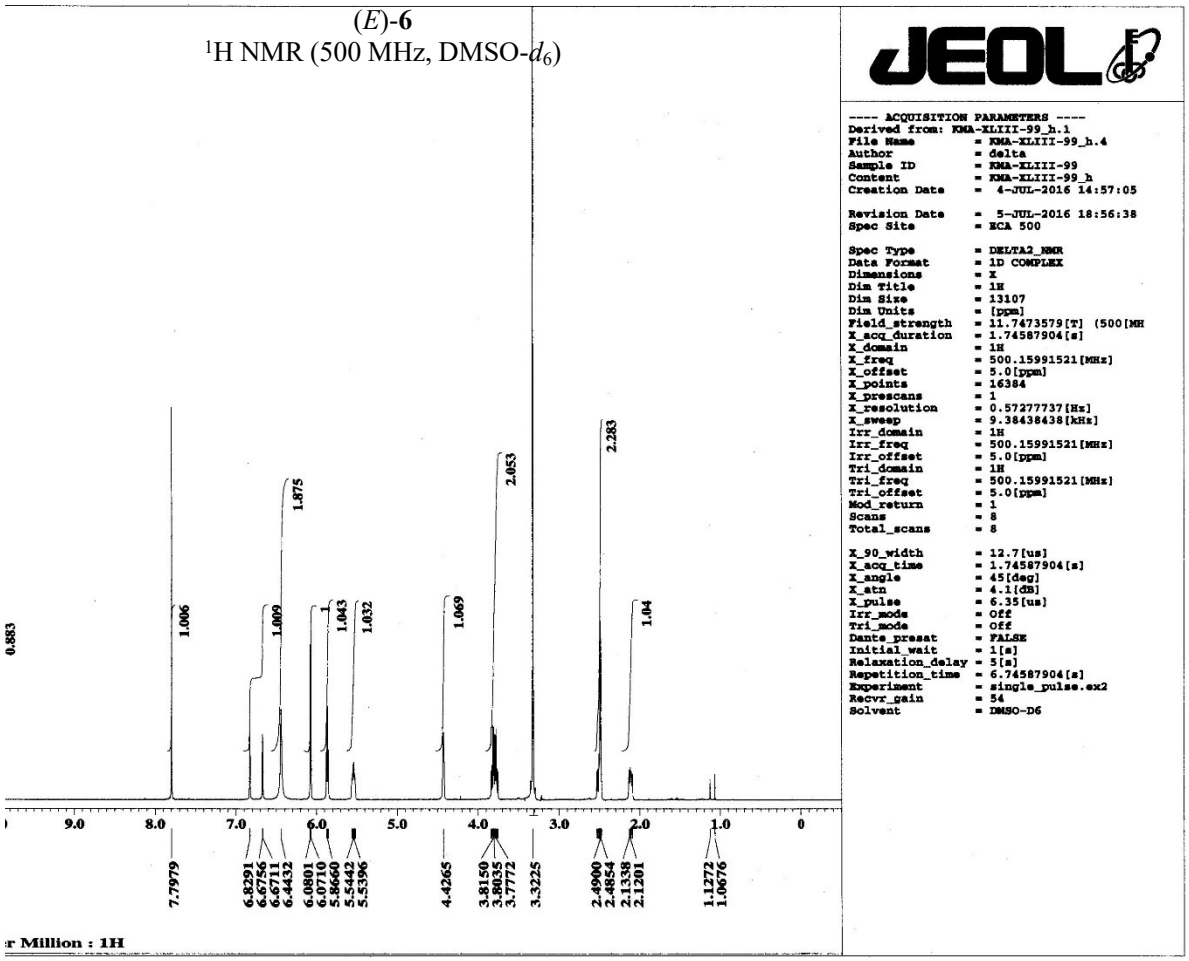
^{19}F NMR (470 MHz, $CDCl_3$)



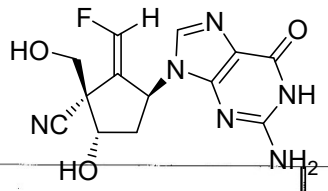


(*E*)-**6**

^{1}H NMR (500 MHz, DMSO-d_6)

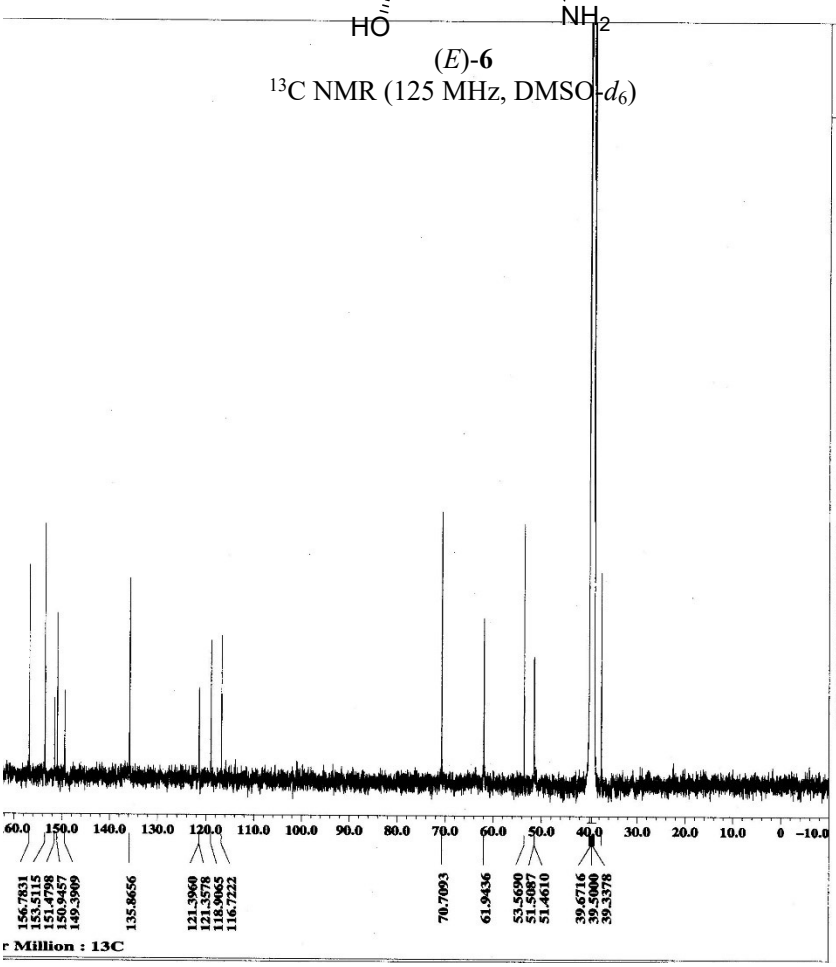


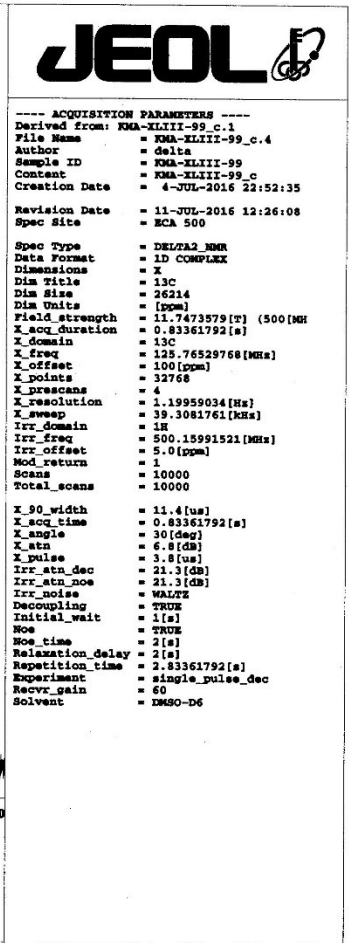

```
---- ACQUISITION PARAMETERS ----
Derived from: KMA-XLIII-99_h.1
File Name          = KMA-XLIII-99_h.4
Author             = delta
Sample ID          = KMA-XLIII-99
Content            = KMA-XLIII-99_h
Creation Date      =  4-JUL-2016 14:57:05

Revision Date      =  5-JUL-2016 18:56:38
Spec Site          = ECA 500

Spec Type          = DELTA2_NMR
Data Format        = 1D COMPLEX
Dimensions         = X
Dim Title          = 1H
Dim Size           = 13107
Dim Units          = [ppm]
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]
Mod_return         = 1
Scans              = 8
Total_scans        = 8

X_90_width         = 12.7[us]
X_acq_time         = 1.74587904[s]
X_angle            = 45[deg]
X_atn              = 4.1[dB]
X_pulse            = 6.35[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Relaxation_delay   = 5[s]
Repetition_time    = 6.74587904[s]
Experiment         = single_pulse.ex2
Recvr_gain         = 54
Solvent            = DMSO-D6
```

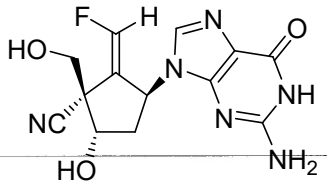


(*E*)-**6**

^{13}C NMR (125 MHz, DMSO-d_6)

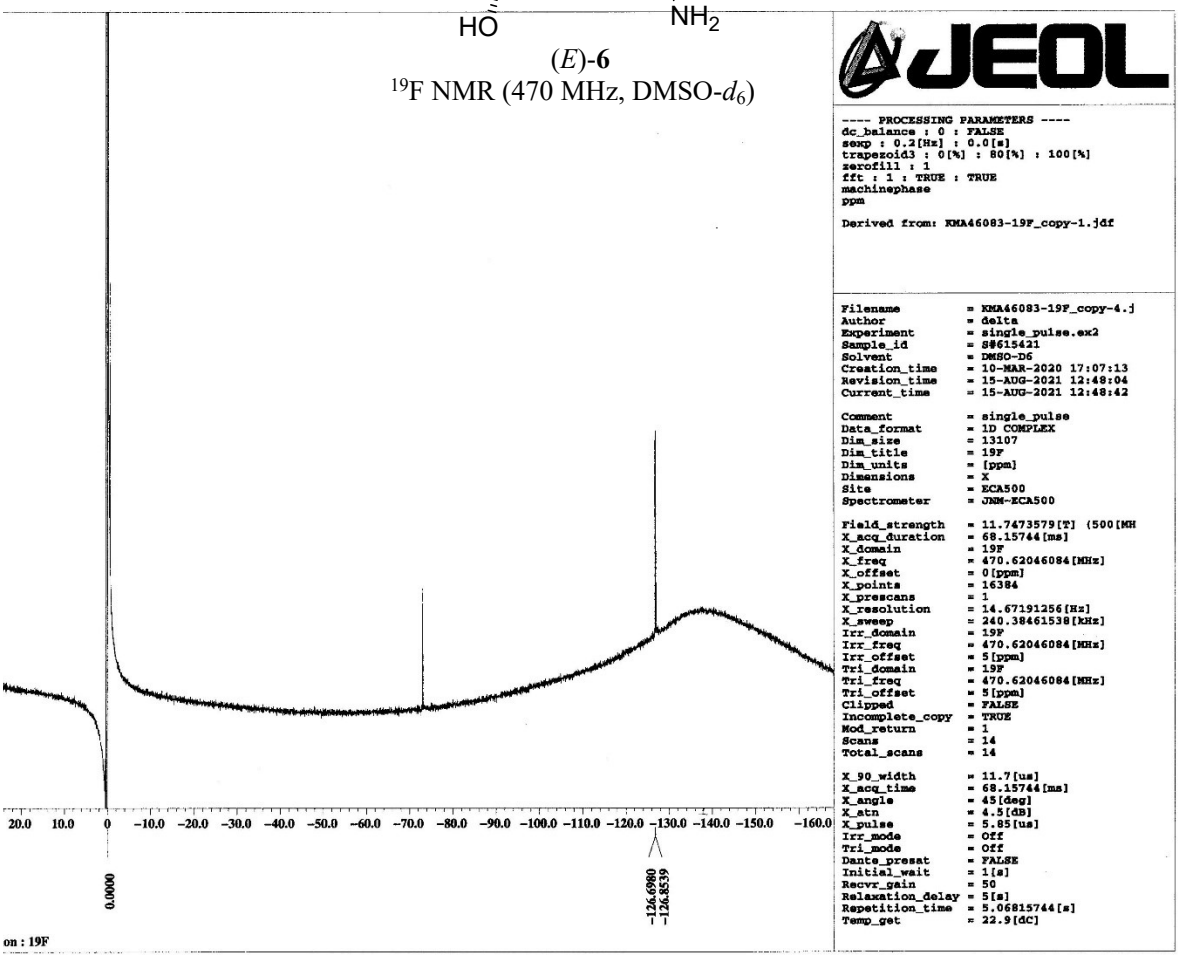






(*E*)-**6**

^{19}F NMR (470 MHz, DMSO-d_6)



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

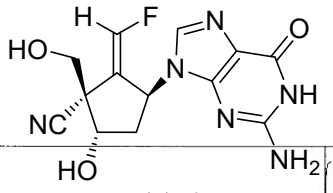
Derived from: KMA46083-19F_copy-1.jdf

Filename = KMA46083-19F_copy-4.j
Author = delta
Experiment = single_pulse.ex2
Sample_id = S0615421
Solvent = DMSO-D6
Creation_time = 10-MAR-2020 17:07:13
Revision_time = 15-AUG-2021 12:48:04
Current_time = 15-AUG-2021 12:48:42

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 19F
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

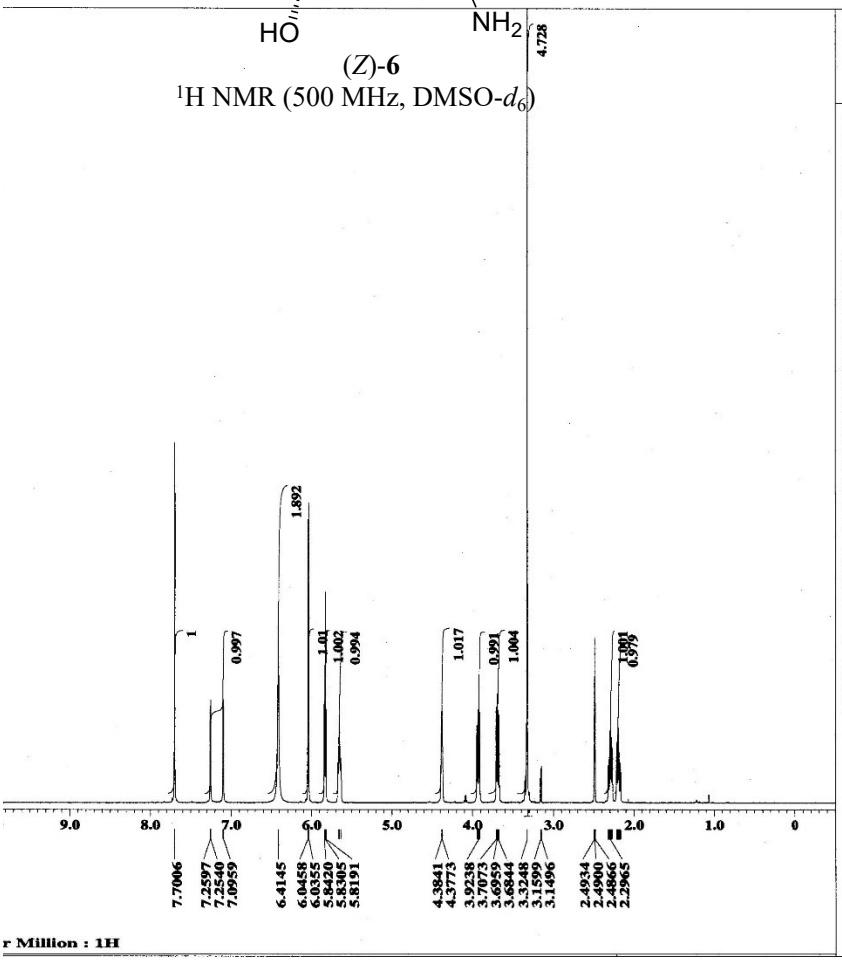
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 68.15744[ms]
X_domain = 19F
X_freq = 470.62046084[MHz]
X_offset = 0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 14.67191256[Hz]
X_sweep = 240.38461538[kHz]
Irr_domain = 19F
Irr_freq = 470.62046084[MHz]
Irr_offset = 5[ppm]
Tri_domain = 19F
Tri_freq = 470.62046084[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Incomplete_copy = TRUE
Mod_return = 1
Scans = 14
Total_scans = 14

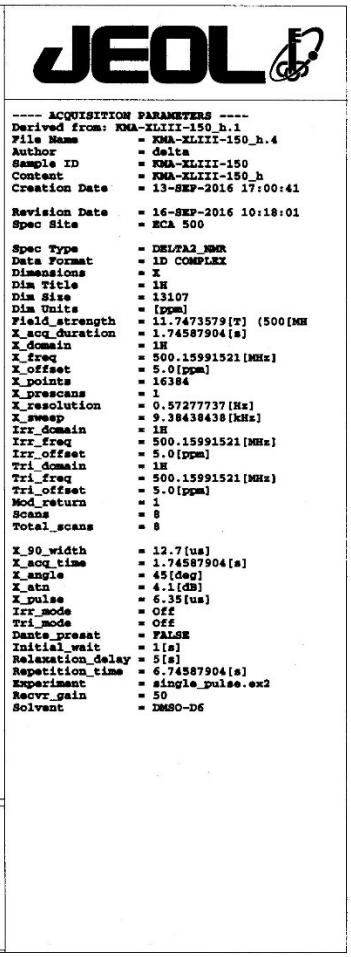
X_90_width = 11.7[us]
X_acq_time = 68.15744[ms]
X_angle = 45[deg]
X_atn = 4.5[dB]
X_pulse = 5.85[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 5.06815744[s]
Temp_get = 22.9[dC]

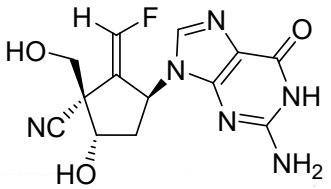


(*Z*)-**6**

^{1}H NMR (500 MHz, DMSO-d_6)

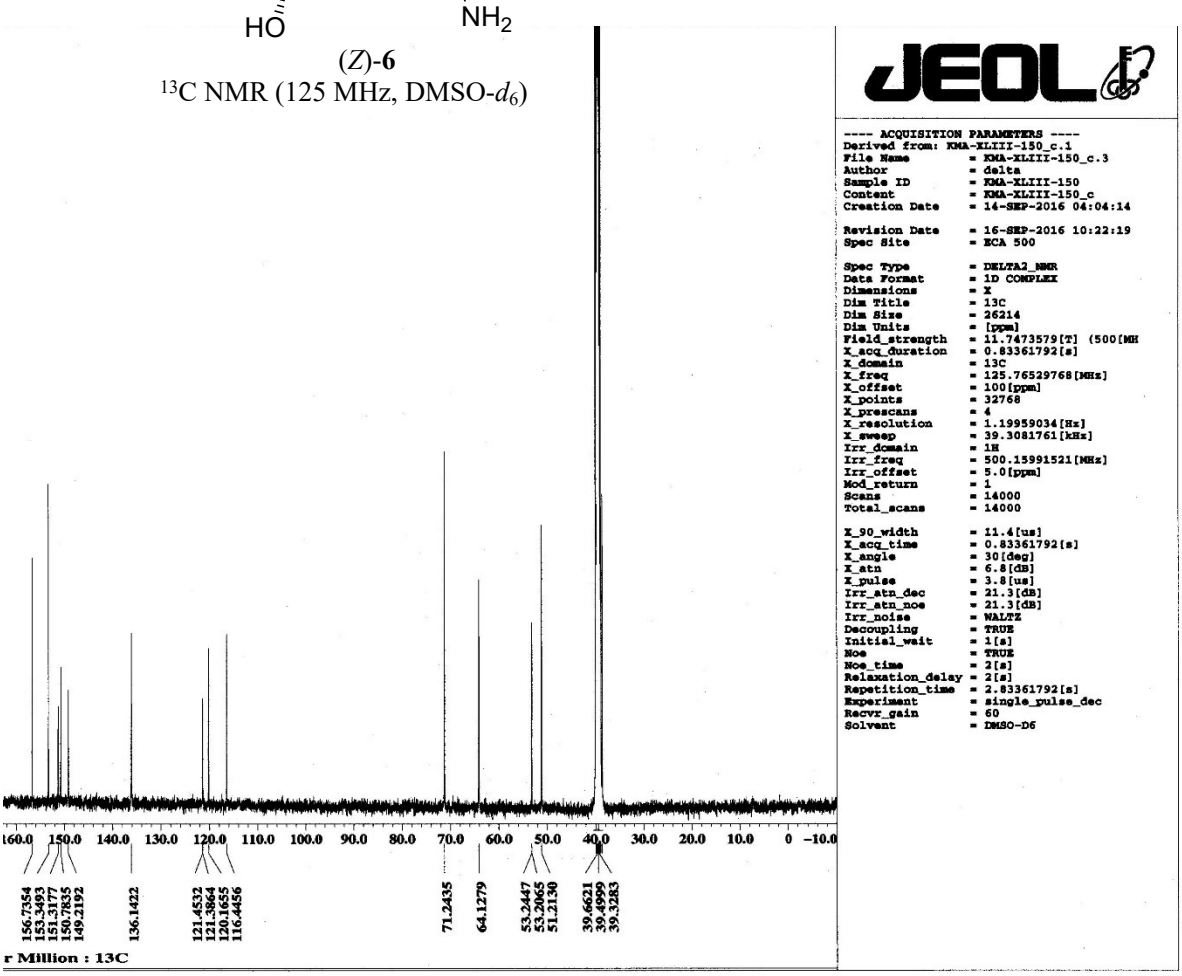






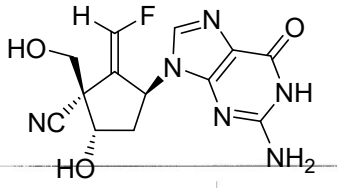
(*Z*)-**6**

^{13}C NMR (125 MHz, DMSO-d_6)



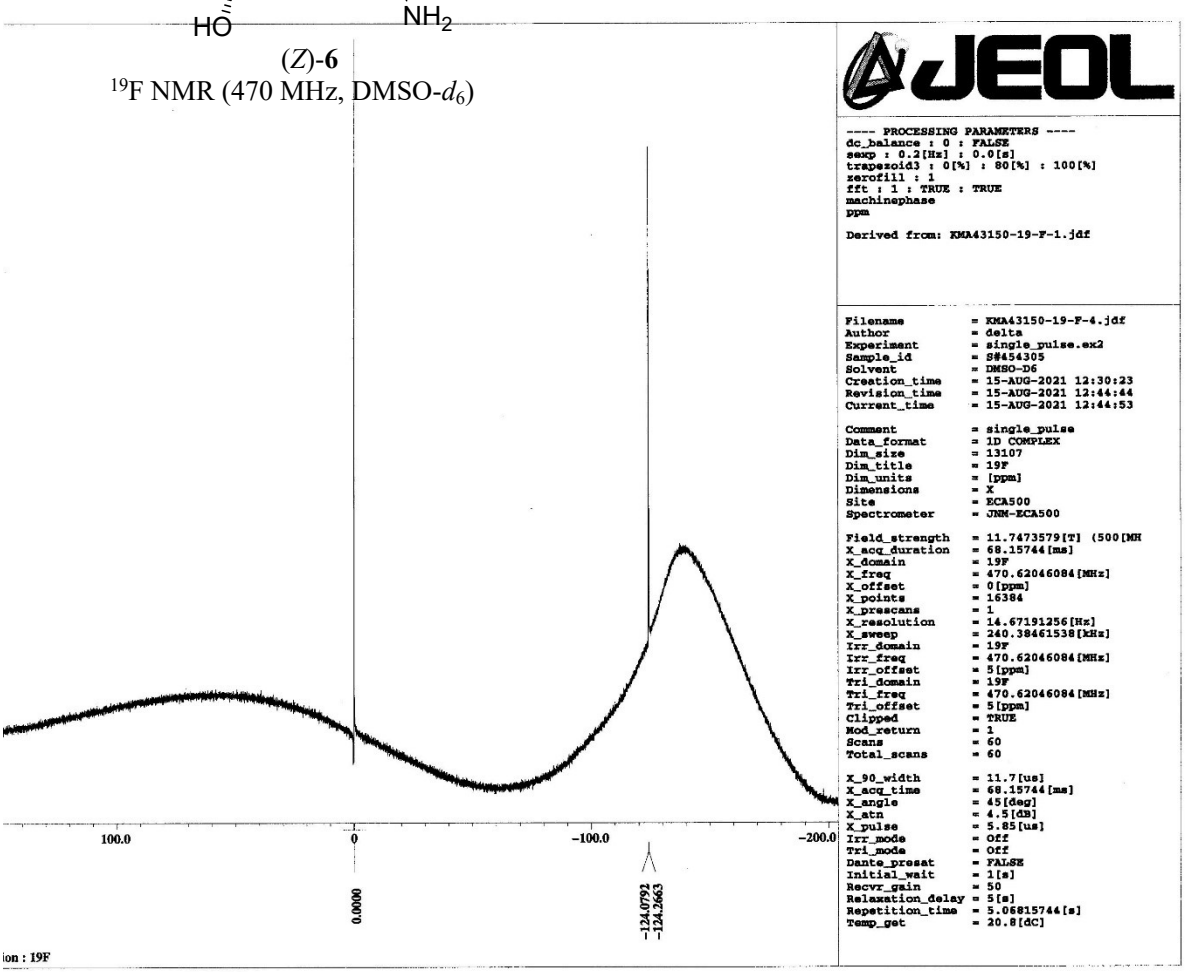
JEOL

```
---- ACQUISITION PARAMETERS ----
Derived from: KMA-XLIII-150_c.1
File Name          = KMA-XLIII-150_c.3
Author             = delta
Sample ID          = KMA-XLIII-150
Content            = KMA-XLIII-150_c
Creation Date      = 14-SEP-2016 04:04:14

Revision Date      = 16-SEP-2016 10:22:19
Spec Site          = ECA 500

Spec Type          = DELTA2_NMR
Data Format        = 1D COMPLEX
Dimensions         = X
Dim Title          = 13C
Dim Size           = 26214
Dim Units          = [ppm]
Field_strength     = 11.7473579[T] (500[MH
X_acq_duration     = 0.83361792[s]
X_domain           = 13C
X_freq             = 125.76529768[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 1.19959034[Hz]
X_sweep            = 39.3081761[kHz]
Irr_domain         = 1H
Irr_freq           = 500.15991521[MHz]
Irr_offset         = 5.0[ppm]
Mod_return         = 1
Scans              = 14000
Total_scans        = 14000

X_90_width         = 11.4[us]
X_acq_time         = 0.83361792[s]
X_angle            = 30[deg]
X_atn              = 6.8[dB]
X_pulse            = 3.8[us]
Irr_atn_dec        = 21.3[dB]
Irr_atn_noe        = 21.3[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Relaxation_delay   = 2[s]
Repetition_time    = 2.83361792[s]
Experiment         = single_pulse_dec
Recvr_gain         = 60
Solvent            = DMSO-D6
```



(*Z*)-**6**

^{19}F NMR (470 MHz, DMSO-d_6)

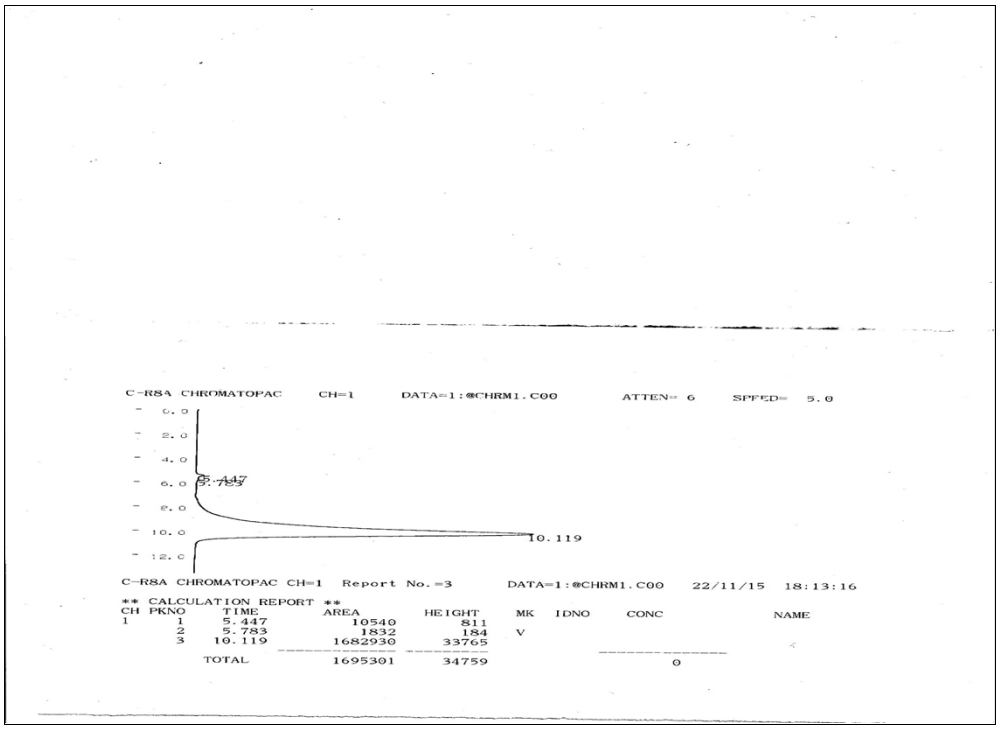



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: KMA43150-19-F-1.jdf

Filename          = KMA43150-19-F-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#454305
Solvent           = DMSO-D6
Creation_time     = 15-AUG-2021 12:30:23
Revision_time     = 15-AUG-2021 12:44:44
Current_time      = 15-AUG-2021 12:44:53

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 68.15744[ms]
X_domain          = 19F
X_freq            = 470.62046084[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 14.67191256[Hz]
X_sweep           = 240.38461538[kHz]
Irr_domain        = 19F
Irr_freq          = 470.62046084[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 470.62046084[MHz]
Tri_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 60
Total_scans       = 60

X_90_width        = 11.7[us]
X_acq_time        = 68.15744[ms]
X_angle           = 45[deg]
X_atn             = 4.5[dB]
X_pulse           = 5.85[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.06815744[s]
Temp_get          = 20.8[dC]
```

HPLC traces of biological assay samples (*E*)- and (*Z*)-6

Method: 20% MeOH in H_2O (contained 0.5% of AcOH); 10 mL/mn.

For (*E*)-6; R_t = 10.1 min. Area; 99.3%.



C-R8A CHROMATOPAC CH=1 DATA=1:@CHRM1.C00 ATTEN= 6 SPEED= 5.0

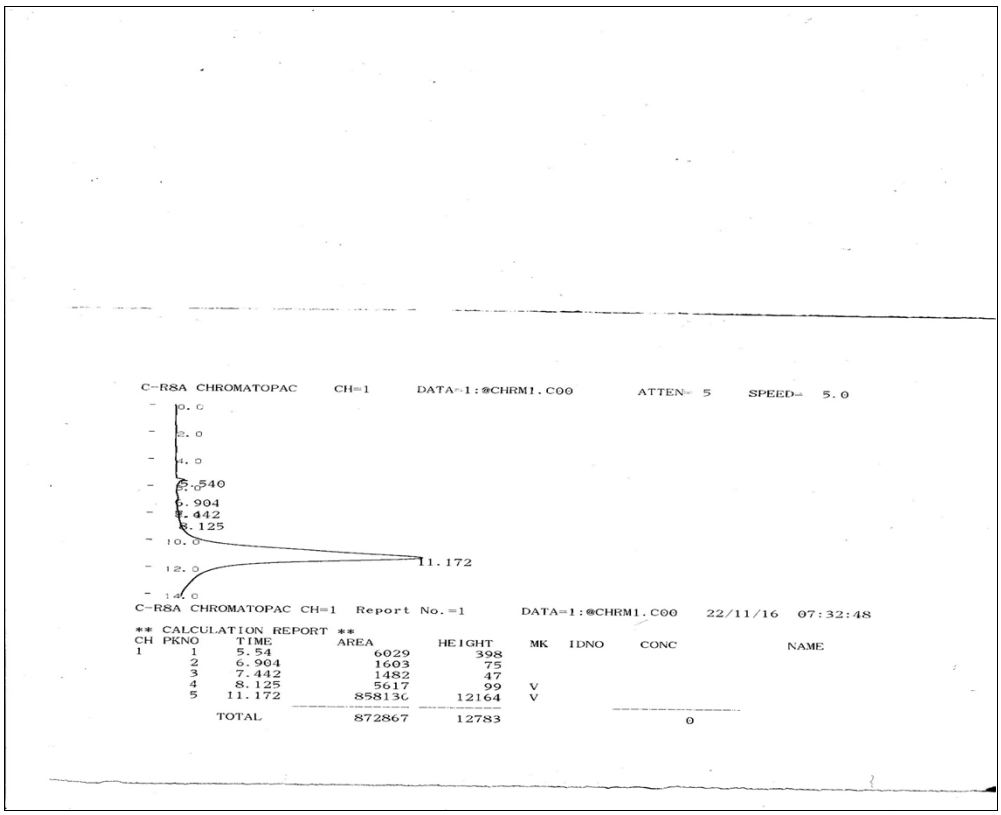
5.447
5.783
10.119

C-R8A CHROMATOPAC CH=1 Report No.=3 DATA=1:@CHRM1.C00 22/11/15 18:13:16

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1	5.447	10540	811				
	2	5.783	1832	184	V			
	3	10.119	1682930	33765				
	TOTAL		1695301	34759			0	

For (*Z*)-6; R_t = 11.2 min. Area; 98.3%.



C-R8A CHROMATOPAC CH=1 DATA=1:@CHRM1.C00 ATTEN= 5 SPEED= 5.0

5.540
6.904
7.442
8.125
11.172

C-R8A CHROMATOPAC CH=1 Report No.=1 DATA=1:@CHRM1.C00 22/11/16 07:32:48

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1	5.54	6029	398				
	2	6.904	1603	75				
	3	7.442	1482	47				
	4	8.125	5617	99	V			
	5	11.172	858136	12164	V			
	TOTAL		872867	12783			0	