

Appendix IV:

$^{119}\text{Sn}$  NMR

Spectra

Of

Ligands

And

Complexes



```

-- PROCESSING PARAMETERS ----
4c1n
sweep : 2.0 [Hz] : 0.0 [s]
trapezoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : TRUE
machinephase : TRUE
ppm

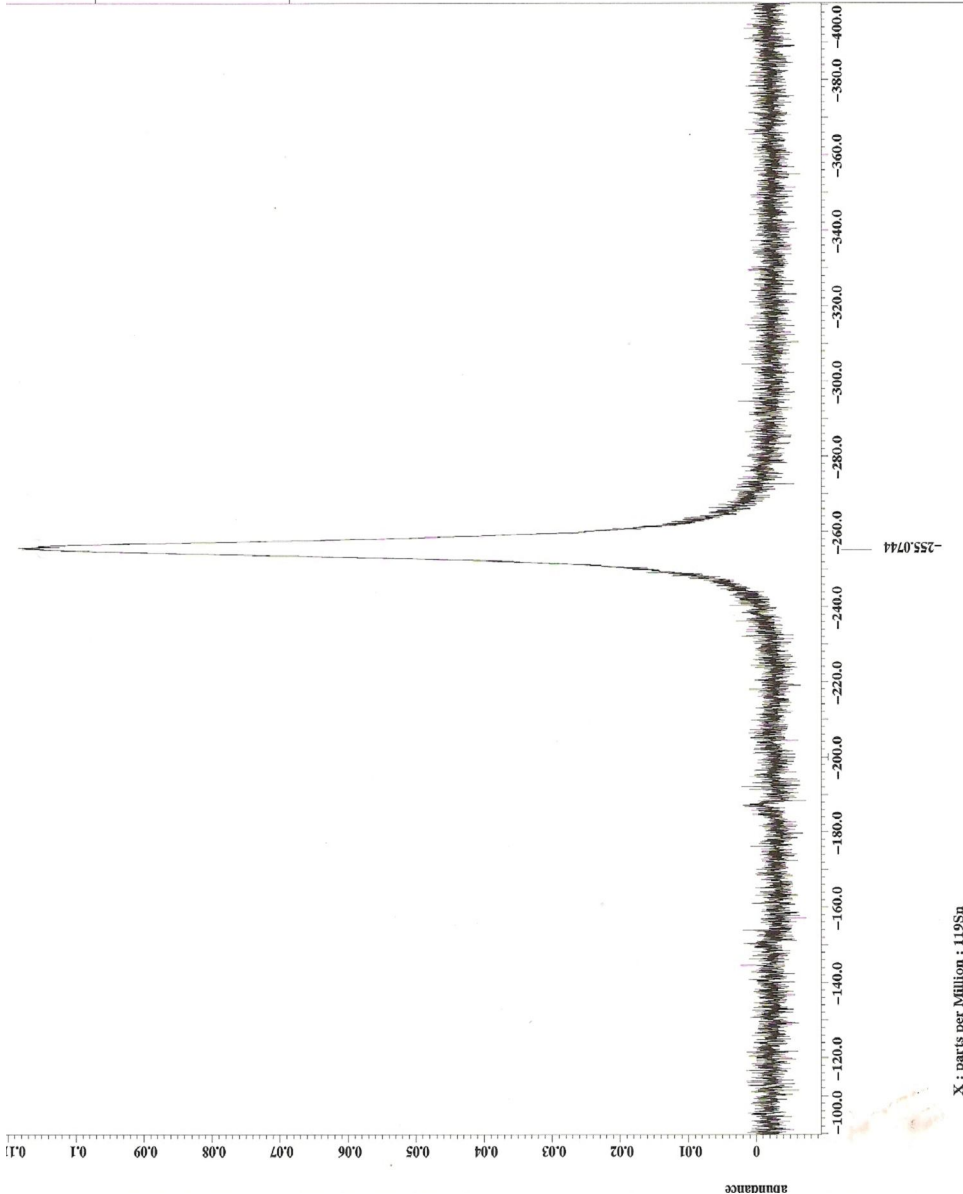
```

Derived from: tin119-ortho-22-8-07-1.jcf

```

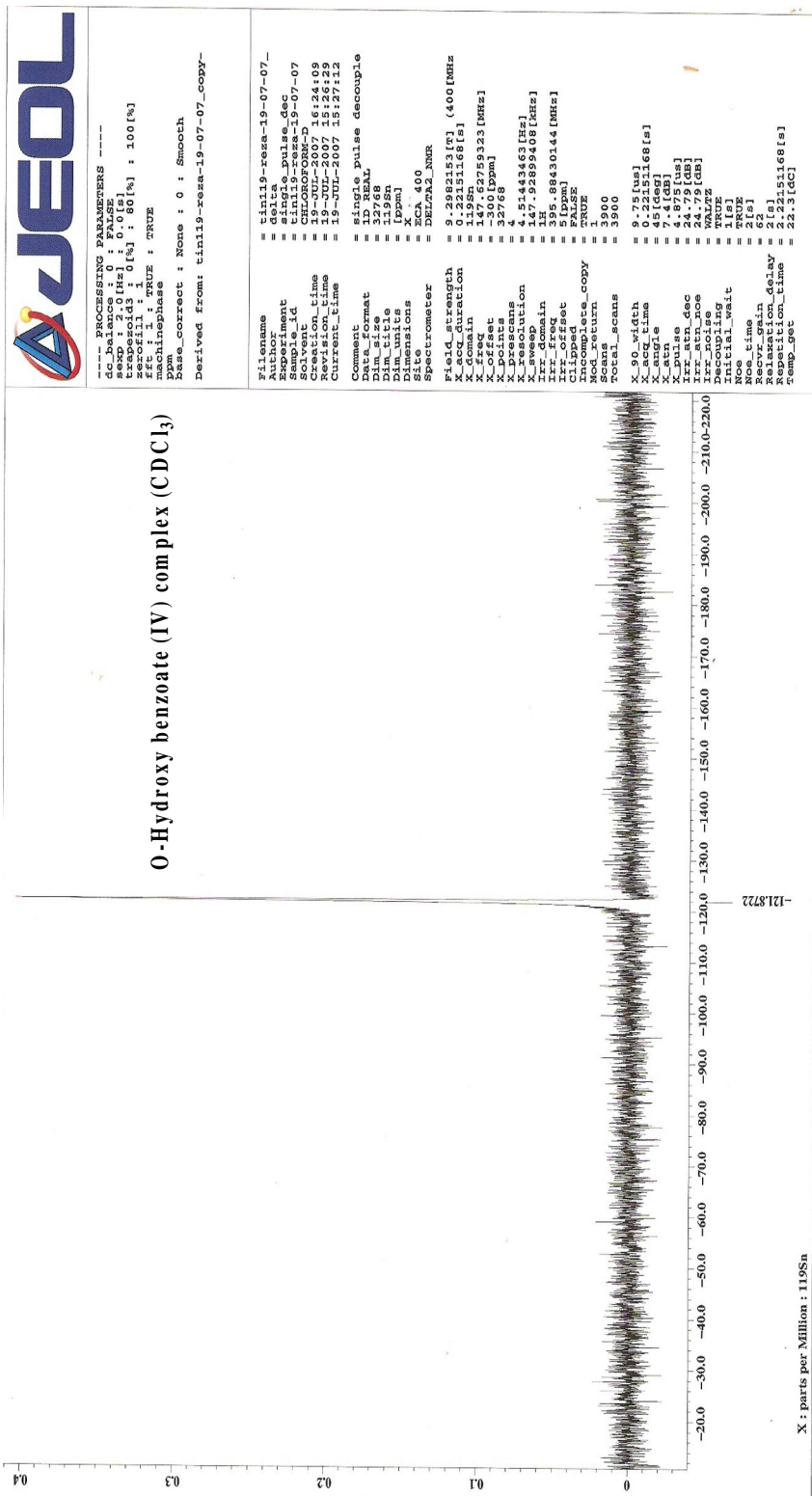
Filename = tin119-ortho-22-8-07-
Author = delta
Experiment = tin119_pulse_dec
Solved = tin119-ortho-22-8-07
Solvent = METHANOL-D3
Creation_time = 23-AUG-2007 09:06:15
Revision_time = 23-AUG-2007 09:50:26
Current_time = 23-AUG-2007 09:50:30
Comment = single pulse decouple
Date_format = DD-MMM-YY
Dim1 = 32768
Dim2 = 1998
Dim3 = 1998
Dim_units = [ppm]
Dimensions = X
Site = ECA_400
Spectrometer = DELTA2_RMR
Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 0.22151168 [s]
X_chan = 14986
X_center = 147.92759323 [MHz]
X_offset = -200 [ppm]
X_points = 32768
X_prescans = 4
X_resolution = 4.8143463 [Hz]
X_sweep = 147.92899468 [kHz]
Irr_domain = 1H
Irr_freq = 395.88430144 [MHz]
Irr_offset = [ppm]
Mod = PULSE
Mod_return = 1
Total_scans = 120000
X_90_width = 9.75 [us]
X_acq_time = 0.22151168 [s]
X_angle = 45 [deg]
X_cp = 4.87 [us]
X_pulse = 4.87 [us]
Irr_atn_dec = 24.75 [dB]
Irr_noise = WALTZ
Decoupling = TRUE
Noe_tidal_wait = FALSE
Noe_wait = FALSE
Recvr_gain = 62
Relaxation_delay = 2 [us]
Repetition_time = 0.22351168 [s]
Temp_set = 22.6 [dC]

```



X : parts per Million : 119Sn

Figure41.  $^{119}\text{Sn}$  NMR spectrum of dibutyltin(IV) bis(o-hydroxybenzoate) ( in methanol- $d^3$ )



```

----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sweep : 2.0 [Hz] : 0.0 [s]
zerofill : 1 : 0 [Hz] : 80 [%] : 100 [%]
nuc1 : 119 : TRUE : TRUE
nuc2 : 119 : TRUE : TRUE
ppm :
base_correct : None : 0 : Smooth
Derived from: tin119-reza-19-07-07_copy-
  
```

```

Filename      = tin119-reza-19-07-07_
Author       = delta_pulse_dec
Sample_id    = tin119-reza-19-07-07
Solvent      = CHLOROFORM-D
Acquisition_time = 16:24:09
Revision_time   = 19-JUL-2007 15:24:39
Current_time    = 19-JUL-2007 15:27:12
Comment       = single pulse decouple
Data_format   = 1D REAL
Dir          = 119SN
Dim_units    = [ppm]
Dimensions   = ECA 400
Spectrometer = DELTA2_NMR
Field_strength = 9.2882153 [T] (400 [MHz]
X_acq_duration = 0.22151168 [s]
X_sweep        = 17.62759333 [MHz]
X_offset       = -300 [ppm]
X_points       = 32768
X_resolution   = 4.51443463 [Hz]
X_sweep       = 147.92899408 [MHz]
X_freq        = 395.88430144 [MHz]
X_offset      = 5 [ppm]
Incomplete_copy = TRUE
Xdc_return    = 1
Total_scans   = 3500
X_90_width   = 9.75 [us]
X_acq_time    = 0.22151168 [s]
X_angle       = 45 [deg]
X_pulse       = 4.875 [us]
X_atn_dec     = 24.79 [dB]
X_noise      = WALZ [dB]
Decoupling   = TRUE
Noe_tial_wait = TRUE
Noe_time     = 2 [s]
Relaxation_delay = 2 [s]
Repetition_time = 2.22151168 [s]
Temp_set     = 22.3 [C]
  
```

Figure 42. <sup>119</sup>Sn NMR spectrum of dibutyltin(IV) bis(m-hydroxybenzoate) (chloroform-d)



```

---- PROCESSING PARAMETERS ----
dc_bai000 : 0 [Hz]
tempo1d3 : 0 [s]
tempo1d3 : 80 [%] : 100 [%]
zerofill : 1
machinename : TRUE
machinephase :
DPM
Derived from: tin119-meta-22-8-07-2.jdf

```

```

Filename = tin119-meta-22-8-07-6
Date_ = 20070807
Experiment = single_pulse_dec
Sample_id = tin119-meta-22-8-07
Solvent = METHANOL-D3 01:31:51
Revision_time = 23-AUG-2007 07:56:58
Current_time = 23-AUG-2007 07:57:19
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 32768
Dim_title = 119Sn
Dimensions = X
Site = ECA 400
Spectrometer = DELTA2_NMR
Field_strength = 9.2882153 [T] (400 [MHz]
X_acq_duration = 0.22151168 [s]
X_domain = 119Sn
X_offset = 2759323 [MHz]
X_ppm = -200 [ppm]
X_resolution = 32768
X_points = 4
X_prescans = 5143463 [Hz]
X_resolution = 147.92899408 [kHz]
X_sweep = 1H
X_gamma = 395.88430144 [MHz]
X_phase = 0 [deg]
X_clipset = FALSE
Mod_return = 1
Scans = 120000
Total_scans = 120000
X_90_width = 9.75 [us]
X_acq_time = 0.22151168 [s]
X_resolution = 7.4 [dB]
X_gain = 4.875 [us]
X_pulse = 24.79 [dB]
X_atn_dec = TRUE
Decoupling = TRUE
Initial_wait = 1 [s]
Noe = FALSE
Relaxation_delay = 2 [ms]
Repetition_time = 0.22351168 [s]
Temp_set = 22.7 [dC]

```

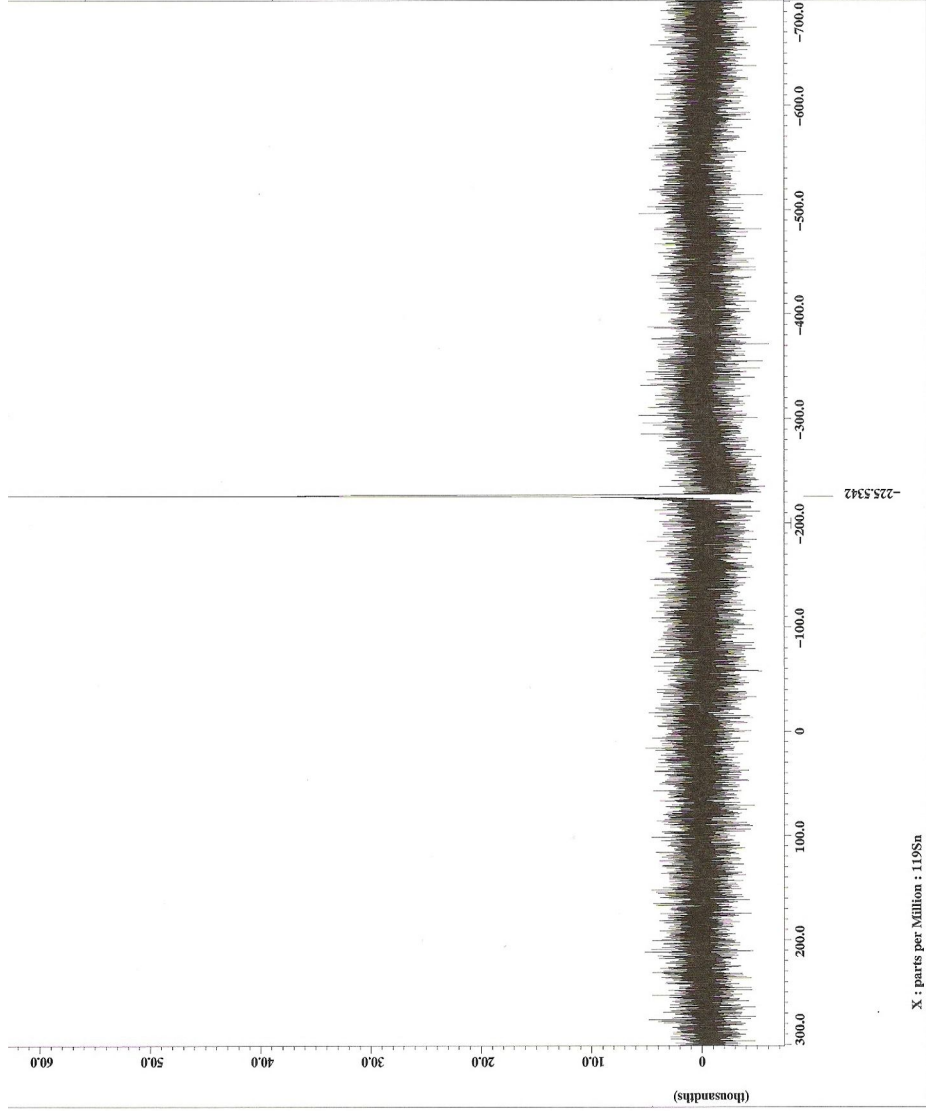


Figure 43. <sup>119</sup>Sn NMR spectrum of m-hydroxyl benzoate tin(IV) complex (methanol -d<sup>3</sup>)



```

---- PROCESSING PARAMETERS ----
gc_balance : 0 : FALSE
exp : 2.0 [Hz] : 0.0 [s]
zerofill : 1 : 0 [%] : 80 [%] : 100 [%]
fft : 1 : TRUE : TRUE
machinephase
ppm
Base_correct : None : 0 : Smooth
Derived from: tin119-reza-1-8.jdf

```

```

Filename = tin119-reza-1-10.jdf
Author = delta
Experiment = single pulse dec
Sample_id = Tin119-reza-1
Solvent = PYRIDINE-D5
Creation_Time = 4-JUL-2007 00:37:17
Revision_Time = 4-JUL-2007 08:09:08
Current_Time = 4-JUL-2007 08:09:59
Comment = single pulse decouple
Data_format = 1D REAL
Dir_size = 32768
Dir_title = 119Sn
Dim1_ticks = 1
Dimensions = 1
Site = ECA 400
Spectrometer = DELTA2_NMR
Field_strength = 9.2882153 [T] (400 [MHz]
X_acq_duration = 0.22151168 [s]
X_delay = 1.00000000 [s]
X_freq = 147.62759323 [MHz]
X_offset = -300 [ppm]
X_points = 32768
X_prescans = 4
X_resolution = 4.5143463 [Hz]
X_sweep = 147.92899408 [kHz]
X_time_domain = 1
X_tuning = 147.62759323 [MHz]
Irr_freq = 395.86430144 [MHz]
Irr_offset = 5 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 88000
Total_scans = 88000
X_90_width = 9.75 [us]
X_acq_time = 0.22151168 [s]
X_angle = 45 [deg]
X_atn = 7.4 [dB]
X_pulse = 4.875 [us]
Irr_pulse = 10 [us]
Irr_acq_dec = 24.79 [dB]
Decoupling = WURZ
Initial_wait = 1 [s]
Noe = FALSE
Recvr_gain = 62
Relaxation_delay = 90 [ms]
Repetition_time = 0.3151168 [s]
Temp_jsc = 20.1 [°C]

```

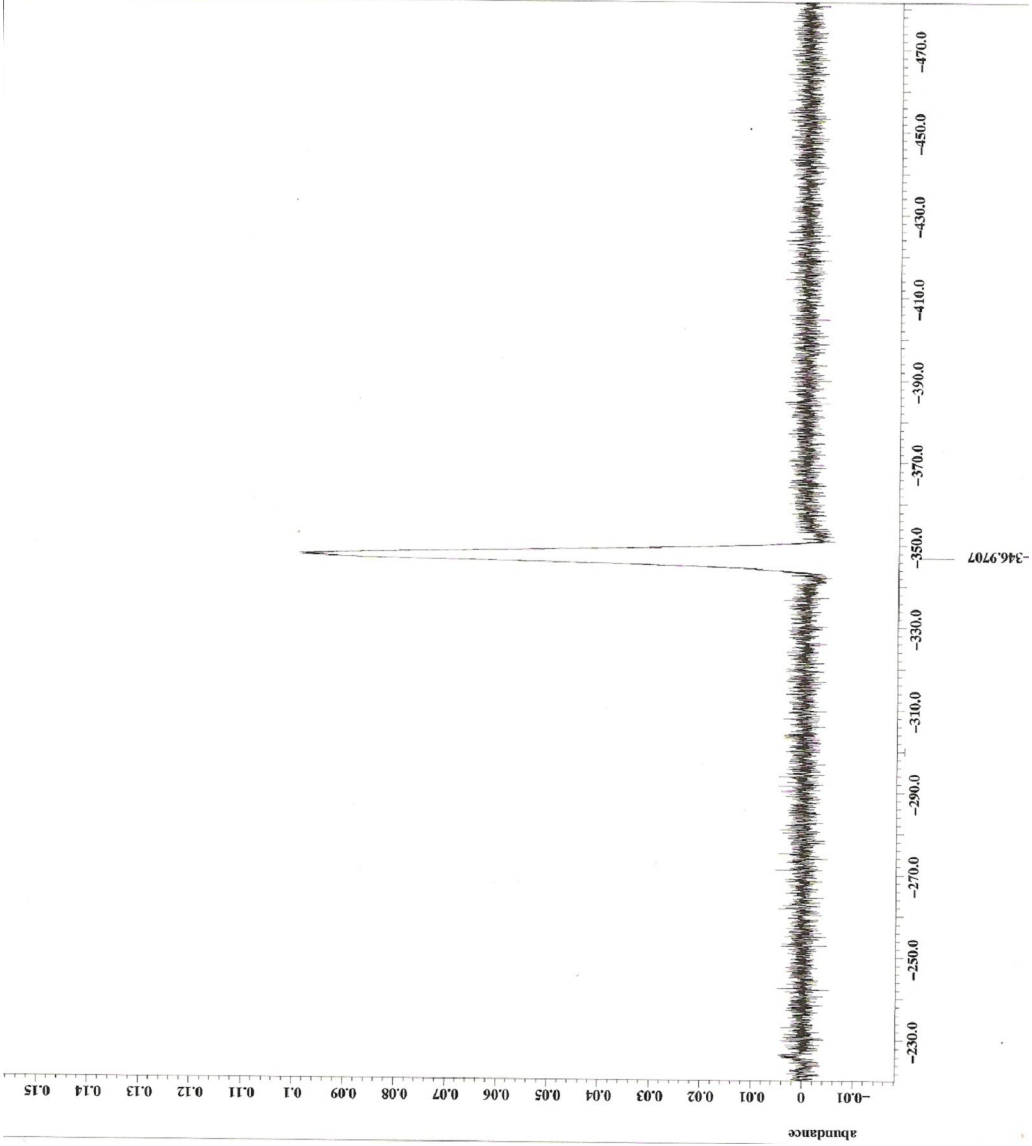
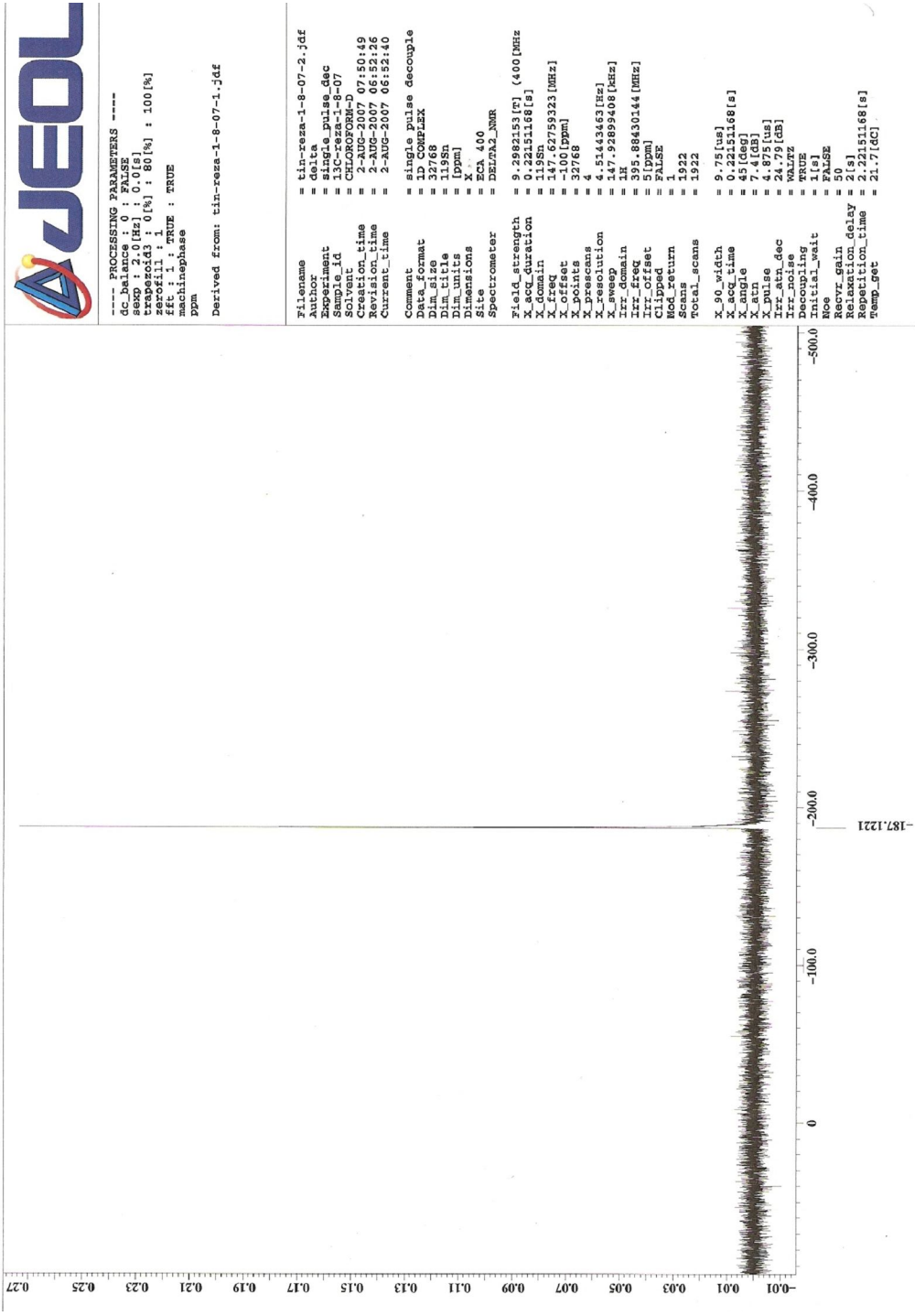


Figure 44. <sup>119</sup>Sn NMR spectrum of dibutyltin(IV) bis(o-hydroxybenzoate) ( in pyridine-d<sup>5</sup>)



```

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sek0 : 2.0 [Hz] : 0.0 [s]
trabrfid3 : 0 [%] : 80 [%] : 100 [%]
sek1 : 0.0 [Hz] : 0.0 [s]
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: tin-reza-1-8-07-1.jdf

```

```

Filename = tin-reza-1-8-07-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 13C-reza-1-8-07
Operator = delta
Creation_time = 2-AUG-2007 07:50:49
Revision_time = 2-AUG-2007 06:52:26
Current_time = 2-AUG-2007 06:52:40
Comment = single pulse decouple
Data_format = DELTA2_NMR
Dim_size = 32768
Dim_title =
Dim_units = [ppm]
Dimensions = X, 400
Site = DELTA2_NMR
Spectrometer =
Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 0.22151168 [s]
X_domain = 119Sn
X_offset = 0.27789323 [MHz]
X_points = -100 [ppm]
X_prescans = 32768
X_resolution = 4
X_sweep = 4.5143463 [Hz]
X_start = 17.92899498 [MHz]
X_stop = 18.72899498 [MHz]
Irr_freq = 395.88430144 [MHz]
Irr_offset = 5 [ppm]
Clipped = FALSE
Mod_return = 1
Total_scans = 1922
X_90_width = 9.75 [us]
X_acq_time = 0.22151168 [s]
X_angle = 75 [deg]
X_pulse = 4.875 [us]
Irr_atn_dec = 24.79 [dB]
Irr_noise =
Decoupling = TRUE
Nopt_tial_wait =
Recvr_gain = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.22151168 [s]
Temp_set = 21.7 [dC]

```

Figure 45. <sup>119</sup>Sn NMR spectrum of dibutyltin(IV) [2-Salicylidenedimino-2-(hydroxymethyl)-1,3-dihydroxypropane]



```

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
seop : 2.0 [Hz] : 0.0 [s]
t2rhozd3 : 0 [%] : 80 [%] : 100 [%]
f2f1 : 1 : TRUE : TRUE
machinephase
ppm
Derived from: tin119-reza-br-complex-1.j

```

```

Filename = tin119-reza-br-comple
Author = delta
Experiment = single_pulse_dec
Sample_id = tin119-reza-br-comple
Solve_method =
Creation_time = 3-FEB-2008 02:19:19
Revision_time = 4-FEB-2008 12:43:22
Current_time = 4-FEB-2008 12:43:24
Comment = single pulse decouple
Data_format = in_chem3kx
Dim_size = 32768
Dim_title = 119Sn
Dim_units = [ppm]
Spectrum =
Site = Xn 400
Spectrometer = DELTA2_MNR

```

```

Field_strength = 9.2982153 [T] (400 [MHz]
X_acq_duration = 0.22151168 [s]
X_acq_time = 9.725168 [s]
X_freq = 147.6275933 [MHz]
X_offset = 1 [ppm]
X_points = 32768
X_prescans = 4
X_resolution = 4.5144363 [Hz]
X_sweep = 47.2899408 [MHz]
Irr_domain =
Irr_freq = 395.88430144 [MHz]
Irr_offset = 5 [ppm]
Xapped =
X_return =
Scans = 3600
Total_scans = 3600
X_90_width = 9.75 [us]
X_acq_time = 9.725168 [s]
X_angle = 7.4 [dB]
X_atn =
X_pulse = 3.25 [us]
Irr_atn_dec = 24 [dB]
Irr_pulse = 24 [dB]
Decoupling = TRUE
Initial_wait = 1 [s]
Noe_time = TRUE
Noe_time = 2 [s]
Relaxation_delay = 2 [s]
Repetition_time = 2.22151168 [s]
Temp_get = 20.2 [dC]

```

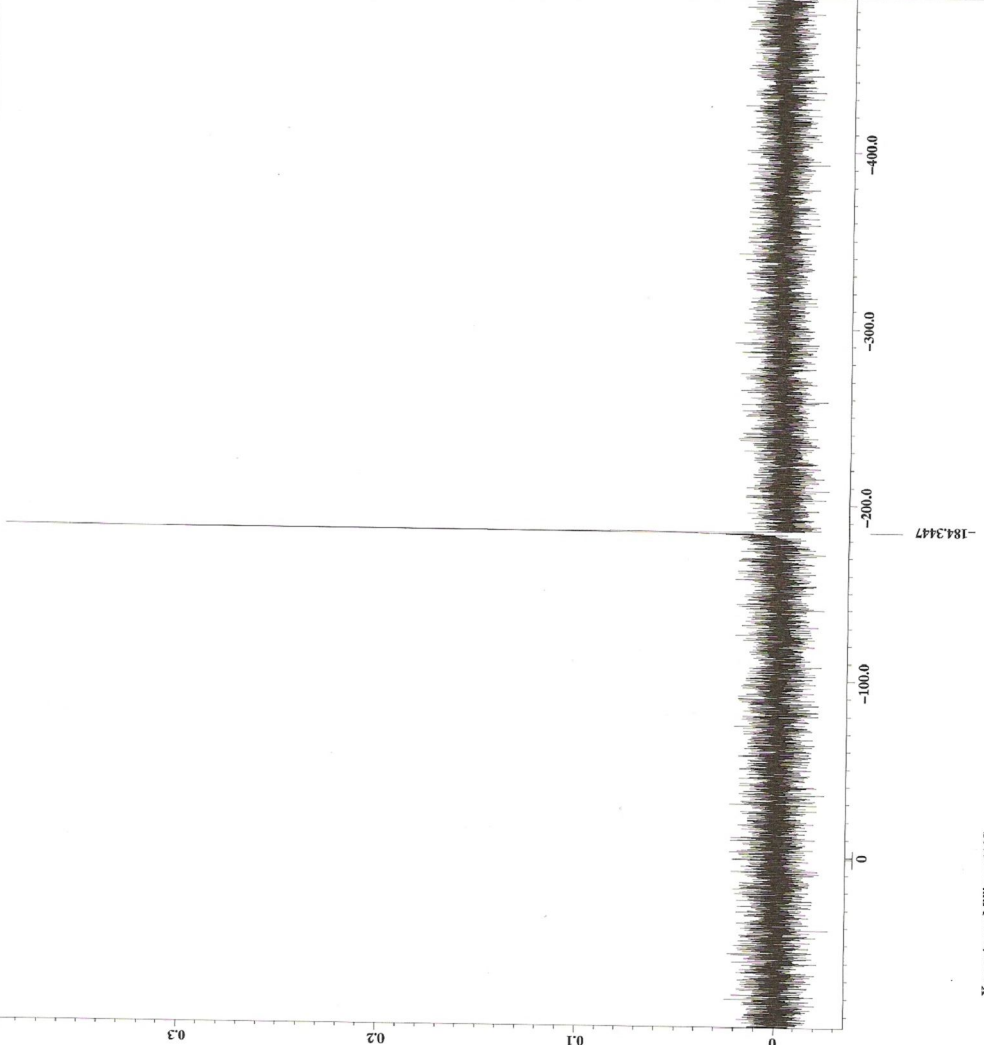


Figure 46. <sup>119</sup>Sn NMR spectrum of 5-chloro dibutyltin(IV) [2-Salicylidenedimino-2-(hydroxymethyl)-1,3-dihydroxypropane] (in chloroform-d)





```

--- PROCESSING PARAMETERS ---
dc_balance : 0 : FALSE
sexp : 2.0 [Hz] : 0.0 [s]
crapsoid3 : 0 [%] : 80 [%] : 100 [%]
ft_off1 : TRUE : TRUE
machinphase
ppm

```

Derived from: tin119-reza-br-complex-1.j

```

Filename = tin119-reza-br-comple
Author = delta
Experiment = single_pulse_dec
Procfile_id = tin119-reza-br-comple
Solvent =
Creation_time = 3-FEB-2008 02:19:19
Revision_time = 4-FEB-2008 12:43:22
Current_time = 4-FEB-2008 12:43:24
Comment = single pulse decouple
File_format = ID_COMPLEX
Dim_units = 11986
Dim_title = [ppm]
Dimensions = X
Site = ECA 400
Spectrometer = DELTA2_NMR
Field_strength = 9.282153 [T] (400 [MHz]
X_domain = 0.22151168 [s]
X_duration = 11986
X_freq = 147.62759323 [MHz]
X_offset = 1 [ppm]
X_points = 32768
P_points = 4
P_resolution = 51443.63 [Hz]
X_sweep = 147.32899408 [MHz]
Irr_domain =
Irr_freq = 395.88430144 [MHz]
Irr_offset = 5 [ppm]
Clipped = FALSE
Acq_return = 1
Total_scans = 3600
X_90_width = 9.75 [us]
X_acq_time = 0.22151168 [s]
X_angle = 30 [deg]
X_atn = 7.4 [dB]
X_pulse = 2.25 [us]
Irr_atn_dec = 24 [dB]
Irr_atn_noe = WALTZ
Decoupling = TRUZ
Initial_wait = 1 [s]
Noe_time = TRUE
Recovery = 2 [s]
Relaxation_delay = 2 [s]

```

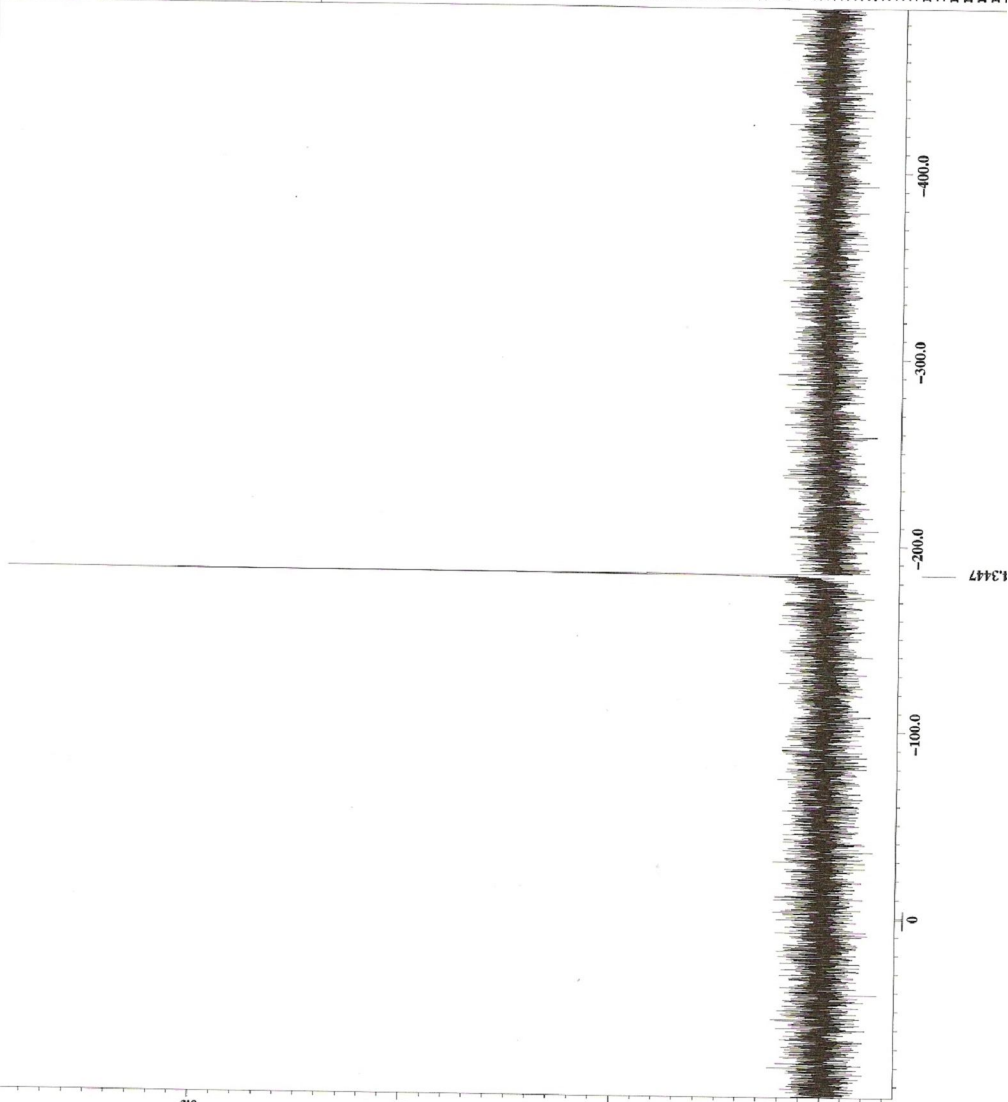


Figure 47.  $^{119}\text{Sn}$  NMR spectra of dibutyltin(IV) 5-bromo [2-Salicylidenediminato-2-(hydroxymethyl)-1,3-dihydroxypropane] (chloroform-d)