Electronic Supplementary Information

Synthesis, Structure, and Reactivity of Pincer-type Iridium Complexes Having Gallyl- and Indyl-Metalloligands Utilizing 2,5-Bis(6-phosphino-2-pyridyl)pyrrolide as a New Scaffold for Metal–Metal Bonds

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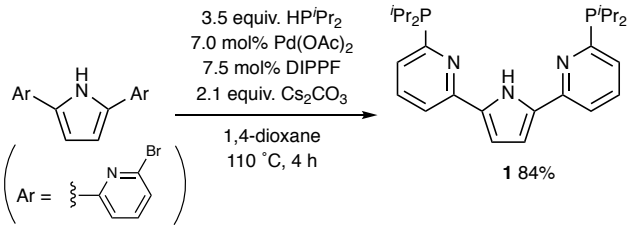
Table of Contents

General: All operations were performed under an argon atmosphere. ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded on an ECX-500 (500 MHz for ^{1}H, 125 MHz for ^{13}C and 202 MHz for ^{31}P), an ECZ-500 (500 MHz for ^{1}H, 125 MHz for ^{13}C and 202 MHz for ^{31}P), or an ECS-400 (400 MHz for ^{1}H, 100 MHz for ^{13}C and 160 MHz for ^{31}P) in $CDCl_3$, CD_2Cl_2, THF-d_8, and C_6D_6. Chemical shifts are expressed in parts per million (ppm) downfield from TMS (δ_H 0.00, δ_C 0.00) and H_3PO_4 aq. (δ_P 0.00) and referenced to residual solvents (δ_H 7.26 and δ_C 77.0 for chloroform, δ_H 5.32 and δ_C 53.1 for dichloromethane, δ_H 3.58 for tetrahydrofuran, and δ_H 7.15 and δ_C 128.6 for benzene). IR spectra were recorded on an FT/IR-VIR-200 (JASCO Co., Ltd.) with ATR PRO450-S accessory (JASCO Co., Ltd.). High resolution mass spectra (HRMS) were recorded on a JEOL JMS-T100 GSV mass spectrometer or a BRUKER micrOTOF II. Crystal data were collected by a Rigaku Saturn CCD system or a Rigaku XtaLAB Synergy R DW HyPix system equipped with a Rigaku GNNP low-temperature device. Silica Gel 60 (Kanto Chemical Co., Inc.) was used for flash column chromatography. Merck Kieselgel 60 F_{254} (0.25 mm thickness, coated on glass 20 × 20 cm^2) plate was used for analytical thin layer chromatography (TLC). THF, Et_2O, pentane and toluene were purified by solvent purification system of Glass-Contour. Benzene-d_6 and THF-d_8 were purchased from Kanto chemicals and dried and degassed by benzophenone ketyl. Other deuterated solvents were purchased from Kanto Chemicals and degassed by freeze-dry technique. 2,5-Bis(6-bromo-2-pyridyl)pyrrole was prepared according to a literature procedure.[1] CCDC 1915112 (**2**^{Ga}), 1915113 (**2**^{In}) , 1915114 (**3**^{Ga}) , 1915115 (**3**^{In}), and 1915116 (**4**^{In}) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

Preparation of 2,5-bis(6-phosphino-2-pyridyl)pyrrole derivative 1

Scheme S1

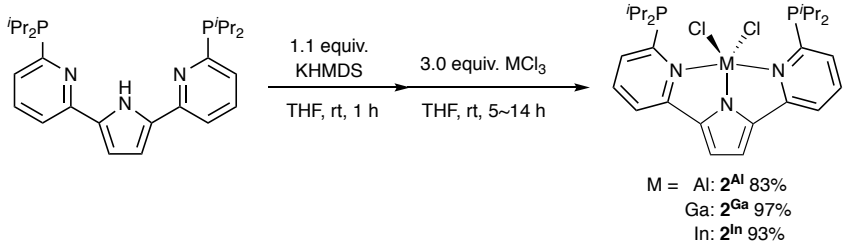


$Pd(OAc)_2$ (58.0 mg, 0.258 mmol) and DIPPF (116.0 mg, 0.277 mmol) were added to a two neck flask and dissolved in dry 1,4-dioxane (37.0 mL). 2,5-Bis(6-bromo-2-pyridyl)pyrrole (1.40 g, 3.69 mmol), Cs_2CO_3 (2.50 g, 7.75 mmol) and HP^{i}Pr$_2$ (1.50 g, 12.9 mmol) were added to the mixture, and the mixture was stirred at reflux untill complete consumption of the starting material. The solution was filtered through a silica gel, and the solvent was evaporated under reduced pressure to give a crude product. Recrystallization from Et_2O/pentane at −50°C gave **1** as pale yellow solids (1.40 g, 3.09 mmol) in 84% yield.

^{1}H NMR (500 MHz, C_6D_6) δ = 11.0 (s, 1H), 7.23 (t, *J* = 7.8 Hz, 2H), 7.09 (d, *J* = 7.8 Hz, 2H), 6.97 (td, *J* = 7.8, 2.0 Hz, 2H), 6.70 (d, *J* = 3.0 Hz, 2H), 2.54-2.46 (m, 4H), 1.24-1.20 (m, 12H), 1.13-1.08 (m, 12H); ^{13}C NMR (125 MHz, C_6D_6) δ = 161.0 (d, *J* = 18 Hz), 150.3, 135.2 (d, *J* = 13 Hz), 133.8, 129.4 (d, *J* = 38 Hz), 117.6, 109.4, 23.4 (d, *J* = 12 Hz), 20.0 (d, *J* = 18 Hz), 19.1 (d, *J* = 8.4 Hz); ^{31}P NMR (200 MHz, C_6D_6) δ = 14.2 (s); IR (ATR) 3449, 2951, 2924, 2863, 1580 cm^{-1}; HRMS (FD$^+$): Calcd for $C_{26}H_{37}N_3P_2$ [M$^+$]: 453.24627; Found: 453.24685.

Preparation of group 13 metal pyrrolides 2

Scheme S2



Preparation of **2^{Al}** (M = Al)

In a glovebox, KHMDS (0.5 M sol. in toluene, 1.45 mL, 0.73 mmol) was added to a solution of **1** (0.30 g, 0.66 mmol) in dry THF (40 mL) to form potassium pyrrolide. Then, $AlCl_3$ (0.26 g, 2.0 mmol) was added to the reaction mixture at room temperature. After 4.5 h, the mixture was concentrated under reduced pressure, and the crude product was purified by recrystallization from THF/Et_2O to give **2^{Al}** as yellow solids (0.30 g, 0.055 mmol) in 83% yield.

^{1}H NMR (500 MHz, C_6D_6) δ = 6.88-6.83 (m, 6H), 6.72 (s, 2H), 1.91-1.85 (m, 4H), 1.16-1.09 (m, 24H); ^{13}C NMR (125 MHz, C_6D_6) δ = 166.1 (d, *J* = 23 Hz), 151.1 (d, *J* = 12 Hz), 137.7, 136.5, 125.3, 117.7, 111.3, 27.2 (d, *J* = 17 Hz), 21.5 (d, *J* = 14 Hz), 20.2 (d, *J* = 20 Hz); ^{31}P NMR (200 MHz, C_6D_6) δ = 3.2 (s); IR (ATR) 2966, 2949, 2922, 2866, 1586, 1480 cm^{-1}. Satisfactory data for elemental analysis and high resolution mass spectroscopy were not obtained due to instability.

Preparation of **2^{Ga}** (M = Ga)

2^{Ga} was prepared according to the procedure described above using **1** (0.30 g, 0.66 mmol) and $GaCl_3$ (0.35 g, 2.0 mmol). The crude product was purified by recrystallization from THF/pentane to give **2^{Ga}** as yellow solids (0.38 g, 0.64 mmol) in 97% yield.

^{1}H NMR (500 MHz, THF-d_8) δ = 7.82 (t, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 8.0 Hz, 2H), 7.52 (d, *J* = 8.0 Hz, 2H), 6.92 (s, 2H), 2.25-2.15 (m, 4H), 1.25 (dd, *J* = 14.0, 7.0 Hz, 12H), 1.03 (dd, *J* = 13.0, 7.5 Hz, 12H); ^{13}C NMR (125 MHz, C_6D_6) δ = 165.2 (d, *J* = 9.5 Hz), 150.0 (d, *J* = 16 Hz), 137.4, 136.0, 125.3, 117.9, 111.5, 26.6 (d, *J* = 14 Hz), 21.1 (d, *J* = 12 Hz), 20.1 (d, *J* = 17 Hz); ^{31}P NMR (200 MHz, THF-d_8) δ = 7.4 (s); IR (ATR) 2951, 2866, 1585, 1438 cm^{-1}; HRMS (FD$^+$): Calcd for $C_{26}H_{37}N_3P_2GaCl_2$ [M$^+$]: 592.10956; Found: 592.10777.

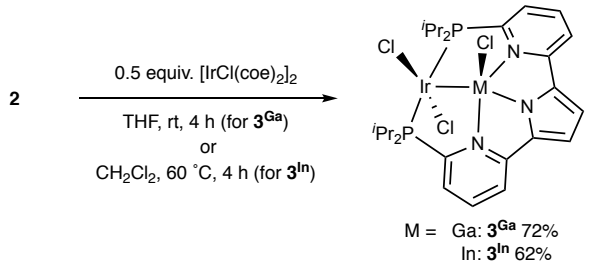
Preparation of **2^{In}** (M = In)

2^{In} was prepared according to the procedure described above using **1** (0.40 g, 0.88 mmol) and $InCl_3$ (0.39 g, 1.8 mmol). The crude product was purified by recrystallization from THF/pentane to give **2^{In}** as yellow solids (0.52 g, 0.82 mmol) in 93% yield.

^{1}H NMR (500 MHz, C_6D_6) δ = 6.95-6.93 (m, 2H), 6.86-6.82 (m, 4H), 6.71 (d, *J* = 7.5 Hz, 2H), 1.90-1.82 (m, 4H), 1.13-1.07 (m, 24H); ^{13}C NMR (125 MHz, C_6D_6) δ = 164.0 (d, *J* = 19 Hz), 150.8 (d, *J* = 12 Hz), 137.8, 135.5, 124.4, 118.7, 112.0, 26.5 (d, *J* = 16 Hz), 21.0 (d, *J* = 14 Hz), 20.0 (d, *J* = 19 Hz); ^{31}P NMR (200 MHz, C_6D_6) δ = 16.3 (s); IR (ATR) 2949, 2919, 2866, 1583, 1436 cm^{-1}; HRMS (FD$^+$): Calcd for $C_{26}H_{37}N_3P_2InCl_2$ [M$^+$]: 637.08003; Found: 637.07977.

Preparation of Ir^{III} dichloride complexes 3 having group 13 metalloligands

Scheme S3



Preparation of $\mathbf{3^{Ga}}$ (M = Ga)

In a glovebox, $\mathbf{2^{Ga}}$ (100 mg, 0.168 mmol) and $[IrCl(coe)_2]_2$ (75.4 mg, 0.0841 mmol) were placed in a round bottom flask and dissolved in dry THF (25.0 mL). After stirring for 4 h at room temperature, the mixture was filtered through a silica gel, and the solvent was evaporated in vacuo to give a crude product. The crude product was recrystallized from THF/Et_2O to give $\mathbf{3^{Ga}}$ as orange solids (99.1 mg, 0.121 mmol) in 72% yield.

^1H NMR (400 MHz, CD_2Cl_2) δ = 7.85 (t, *J* = 7.4 Hz, 2H), 7.56 (d, *J* = 7.4 Hz, 2H), 7.36 (d, *J* = 7.4 Hz, 2H), 6.78 (s, 2H), 3.36-3.18 (m, 4H), 1.58-1.48 (m, 6H), 1.42-1.36 (m, 6H), 1.28-1.20 (m, 12H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ = 149.7, 148.1, 138.6, 132.4, 123.8, 118.9, 111.4, 25.8 (t, *J* = 6 Hz), 20.2 (t, *J* = 14 Hz), 19.6, 19.5, 18.5 (t, *J* = 4 Hz), 16.1; ^{31}P NMR (200 MHz, CD_2Cl_2) δ = 50.1 (s); IR (ATR) 2957, 2916, 2864, 1578, 1440 cm^{-1}; HRMS (FD^+): Calcd for $C_{26}H_{37}N_3P_2GaIrCl_3$ [M^+]: 819.03351; Found: 819.03203.

Preparation of $\mathbf{3^{In}}$ (M = In)

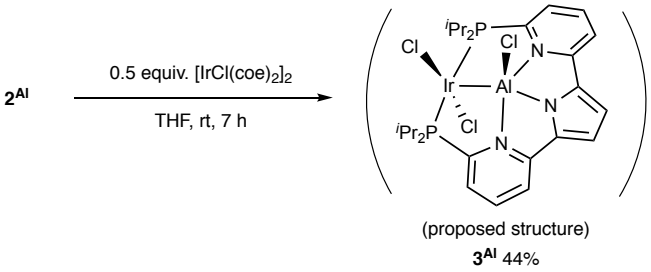
$\mathbf{3^{In}}$ was prepared according to the procedure described above using $\mathbf{2^{In}}$ (100.0 mg, 0.157 mmol) in dry CH_2Cl_2 at 60 ºC. The crude product was purified by recrystallization from THF/Et_2O to give $\mathbf{3^{In}}$ as brown solids (83.8 mg, 0.0968 mmol) in 62% yield.

^1H NMR (500 MHz, CD_2Cl_2) δ = 7.79 (t, *J* = 7.6 Hz, 2H), 7.50 (d, *J* = 7.6 Hz, 2H), 7.24 (d, *J* = 7.6 Hz, 2H), 6.73 (s, 2H), 3.29-3.20 (m, 2H), 3.19-3.11 (m, 2H), 1.56-1.51 (m, 6H), 1.37-1.33 (m, 6H), 1.30-1.22 (m, 12H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ = 151.8, 148.7 (t, *J* = 28 Hz), 138.4, 135.1, 120.5, 118.8, 113.6, 25.3 (t, *J* = 12 Hz), 20.0 (t, *J* = 14 Hz), 19.8, 19.7, 18.5, 15.8; ^{31}P NMR (200 MHz, CD_2Cl_2) δ = 58.1 (s); IR (ATR) 2956, 2912, 1586, 1440 cm^{-1}; HRMS (FD^+): Calcd for $C_{26}H_{37}N_3P_2InIrCl_3$ [M^+]: 865.01181; Found: 865.01369.

Concerning the preparation of $\mathbf{2^{Al}}$ (M = Al)

We also attempted to synthesize the corresponding Al–Ir complex $\mathbf{3^{Al}}$. However, the X-ray analysis was unsuccessful, and the structure was deduced by similarities of ^{1}H NMR spectra to those of $\mathbf{3^{Ga}}$ and $\mathbf{3^{In}}$.

Scheme S4



The reaction was carried out according to the procedure for $\mathbf{3^{Ga}}$ using $\mathbf{2^{Al}}$ (20.0 mg, 0.0363 mmol). Recrystallization of the crude product from THF/Et_2O afforded an Ir complex, which was deduced to be $\mathbf{3^{Al}}$, as orange solids (12.3 mg, 0.0158 mmol) in 44% yield.

^{1}H NMR (500 MHz, CD_2Cl_2) δ = 7.9 (t, *J* = 7.7 Hz, 2H), 7.60 (d, *J* = 7.7 Hz, 2H), 7.36 (d, *J* = 7.7 Hz, 2H), 6.77 (s, 2H), 3.33-3.25 (m, 2H), 3.10-3.00 (m, 2H), 1.55-1.49 (m, 6H), 1.40-1.36 (m, 6H), 1.20-1.13 (m, 12H); ^{31}P NMR (200 MHz, CD_2Cl_2) δ = 43.9 (s).

AIM analyses on Ir^{III} dichloride complexes 3

The structures were optimized by density functional theory (DFT) using the B3PW91 hybrid functional with tight SCF convergence and ultrafine integration grids (Gaussian 16, revision A.03).[2] Empirical dispersion correction was included. The LANL2DZ basis set, including a double-z valence basis set with the Hay and Wadt effective core potential (ECP), was used for aluminum, gallium and iridium and the 6-31G(d,p) basis set was used for carbon, hydrogen, phosphorous, nitrogen, and chlorine. Each of the stationary points was adequately characterized by normal coordinate analysis (no imaginary frequency for an equilibrium structure). In all calculations, the temperature was set to 298.15 K. A quantum theory of atoms in molecules (QTAIM) analyses were carried out using the AIMALL software package.[3,4]

The AIM analysis on $\mathbf{3^{Ga}}$ and $\mathbf{3^{In}}$ showed the bond critical points (bcp) for the Ir–Cl2 bonds, but not between group 13 metals and Cl2 as shown in Figure S1. These results clearly support that these are pincer-type Ir^{III} dichloride complexes bearing gallyl- and indyl-metalloligands (anionic Ga^{I} and In^{I}) in terms of formal oxidation state.

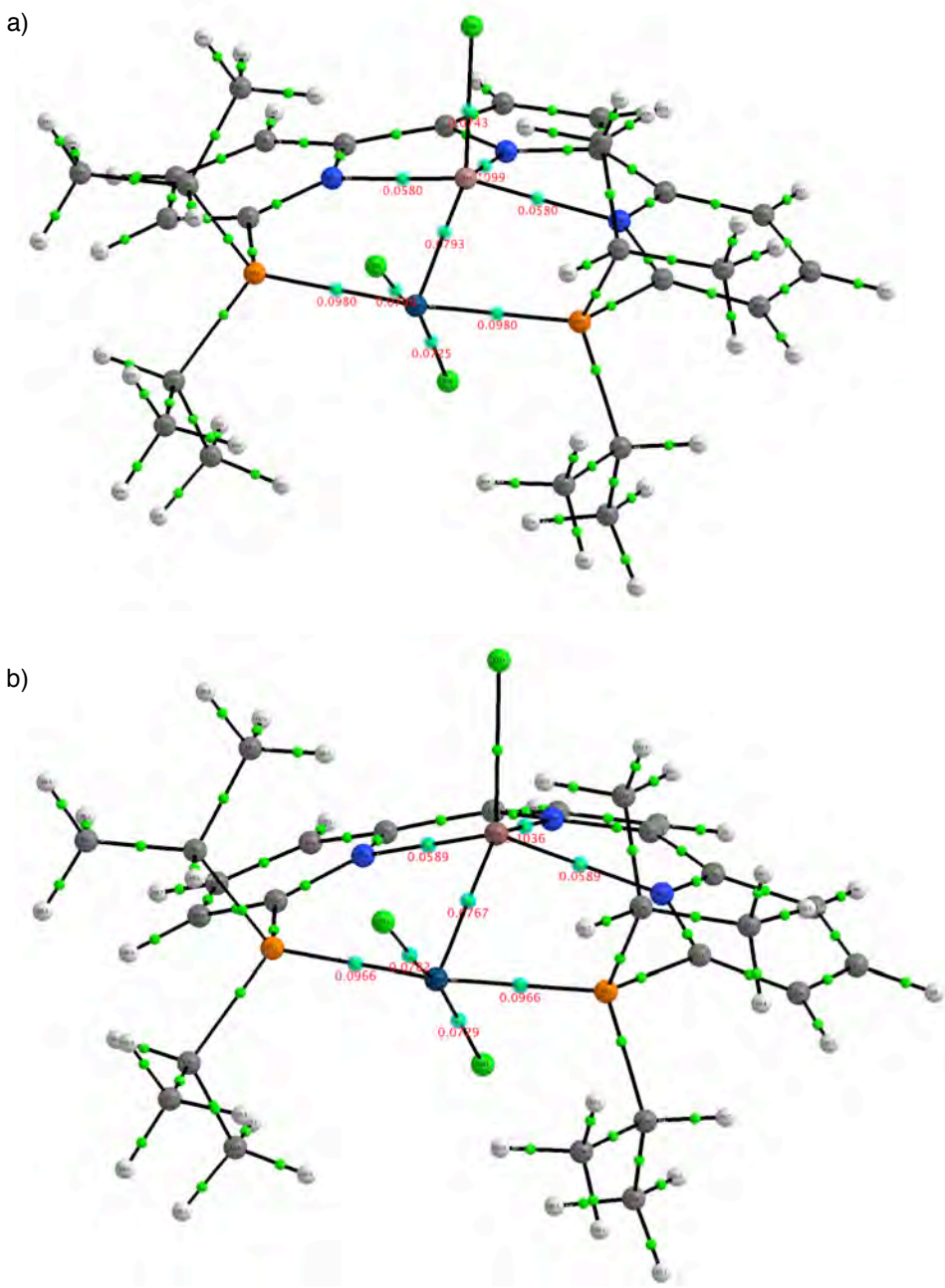
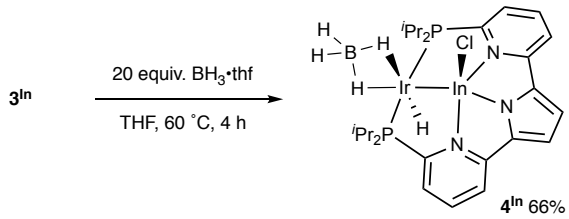


Figure S1. AIM analysis of a) $\mathbf{3}^{\mathbf{Ga}}$ and b) $\mathbf{3}^{\mathbf{In}}$. Selected bond critical points and bond paths with ρ(bcp) > 0.05 e bohr^{-3} are depicted as green spheres and black solid lines. The values of electron density at the selected bcp are depicted as red number.

Preparation of BH_3-coordinate In–Ir^{III} dihydride complex 4^{In}

Scheme S5



In a glovebox, a THF solution (50.0 mL) of **3^{In}** (100 mg, 0.115 mmol) was added to a solution of BH_3•thf in THF (0.9 M, 2.60 mL, 2.34 mmol) at room temperature, and then the mixture was stirred at 60 ºC for 4 h. The solution was concentrated in vacuo to give a crude product, which was recrystallized from THF/hexane to give **4^{In}** as pale brown solids (61.5 mg, 0.0758 mmol) in 66% yield.

^{1}H NMR (500 MHz, CD_2Cl_2) δ = 7.78 (t, *J* = 7.6Hz, 2H), 7.51 (d, *J* = 7.6 Hz, 2H), 7.46 (d, *J* = 7.6 Hz, 2H), 6.73 (s, 2H), 2.84-2.78 (m, 2H), 2.72-2.68 (m, 2H), 1.43-1.38 (m, 6H), 1.32-1.27 (m, 6H), 1.21-1.15 (m, 6H), 0.64-0.58 (m, 6H), –5.8 (br, 1H), –8.9, (br, 1H), –20.5, (td, *J* = 15.5, 8.5 Hz, 1H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ =139.7, 136.6, 122.7, 119.5, 113.4, 30.0, 26.0-25.4 (m), 21.3, 20.5, 17.1 (2C are missing due to gradual decomposition in the solution.); ^{31}P NMR (200 MHz, CD_2Cl_2) δ = 79.8 (s); IR (ATR) 2959, 2929, 2861, 2108, 1570, 1463 cm^{-1}; HRMS (FD^+): Calcd for $C_{26}H_{42}N_3P_2InIrBCl$ [M^+]: 811.12253; Found: 811.12189.

DFT calculation on 4^{In}

The structure was optimized according to the same calculation method for **3**. The structural data were almost identical to those of the X-ray diffraction analysis. Selected bond lengths and angles are depicted in Figure S2.

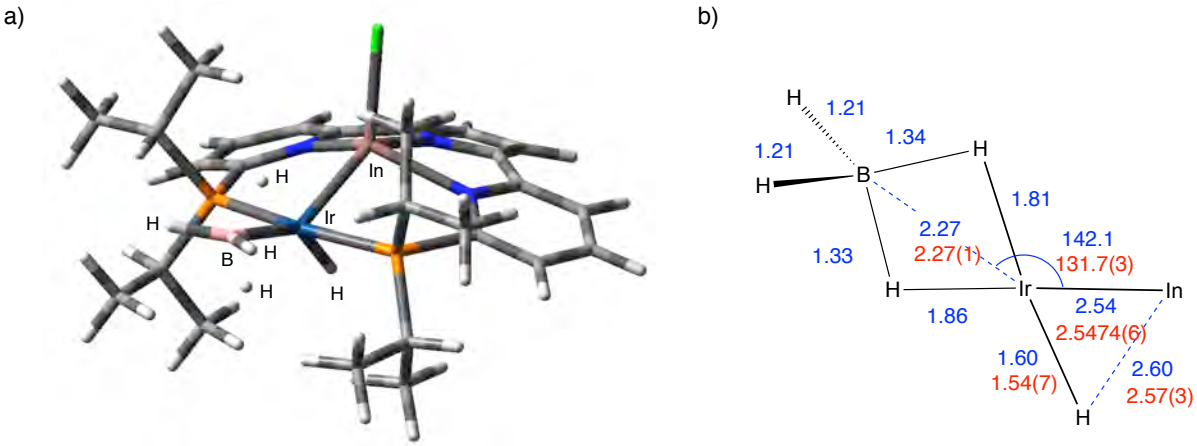
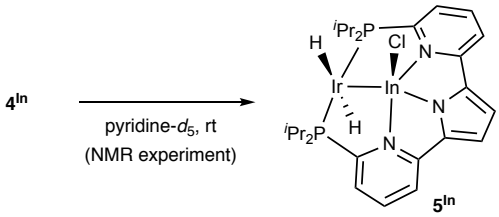


Figure S2. a) The optimized structure of **4^{In}**. b) Selected bond length and angles obtained from theoretical calculation (blue) and X-ray diffraction (red).

Formation of In–Ir^{III} dihydride complex 5^{In} (NMR experiment)

Scheme S6



The BH_3-coordinated In–IrH_2 complex **4^{In}** was converted to an iridium dihydride complex as a major product in pyridine-d_5 at room temperature. The characteristic three hydridic resonances of **4^{In}** (δ = –20.5, –8.9 and –5.8 in CD_2Cl_2) disappeared, and new resonances at δ = –10.4 (1H, td, J = 13.5, 3.5 Hz) and –26.8 (1H, td, J = 16.5, 3.5 Hz) appeared as a major product, which are attributed to H–Ir–H (Figure S3). These data are highly indicative of the formation of an In–IrH_2 complex **5^{In}** via dissociation of the BH_3 from **4^{In}**.

^{1}H NMR (500 MHz, Pyridine-d_5) δ = 7.62 (t, J = 7.6 Hz, 2H), 7.44 (d, J = 7.6 Hz, 2H), 7.41 (d, J = 7.6 Hz, 2H), 6.87 (s, 2H), 3.60-3.45 (m, 2H), 2.54-2.49 (m, 2H), 1.43-1.36 (m, 6H), 1.18-1.12 (m, 6H), 1.12-1.06 (m, 6H), 0.63-0.56 (m, 6H), −10.4 (td, J = 13.5, 3.5 Hz, 1H), −26.8 (td, J = 16.5, 3.5 Hz, 1H); ^{31}P NMR (200 MHz, Pyridine-d_5) δ = 88.1 (s).

Figure S3. ^{1}H NMR of **4^{In}** in pyridine-d_5, which was converted to **5^{In}** via dissociation of BH_3.

DFT calculation on 5^{In}

There are three possible isomers for the In–Ir dihydride as shown in Figure S3. DFT calculations suggested that the *trans* dihydride structure **A** having the In-metalloligand at an apical position is most stable among them.

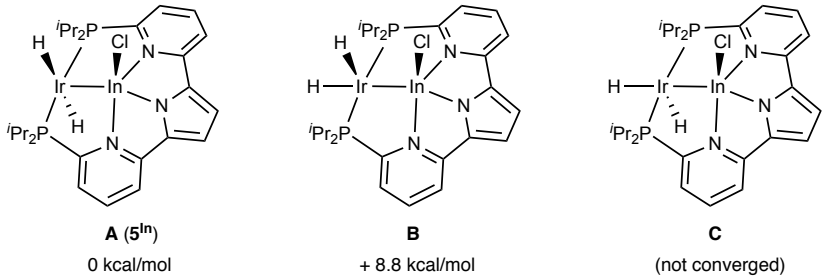


Figure S4. Optimized structures of three possible isomers of In–Ir dihydride complexes and their relative energy differences.

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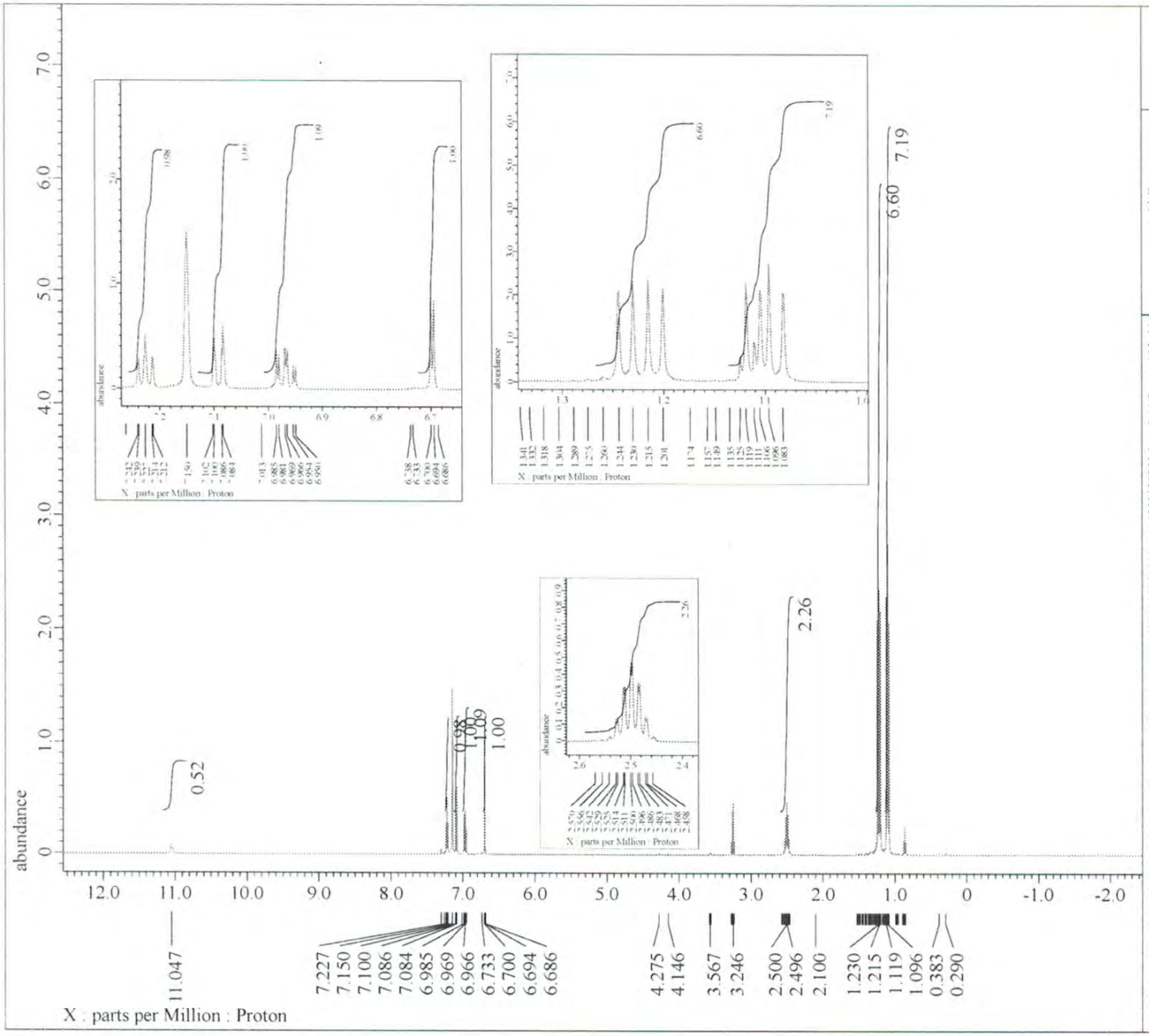
3) R. F. W. Bader, In *Atoms In Molecules -A Quantum Theory*; Oxford University Press: New York, **1990**.

4) AIMAll (Version 14.11.23), Todd A. Keith, TK Gristmill Software, Overland Park KS, USA, 2014 (aim.tkgristmill.com).



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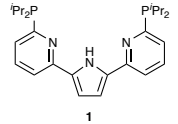

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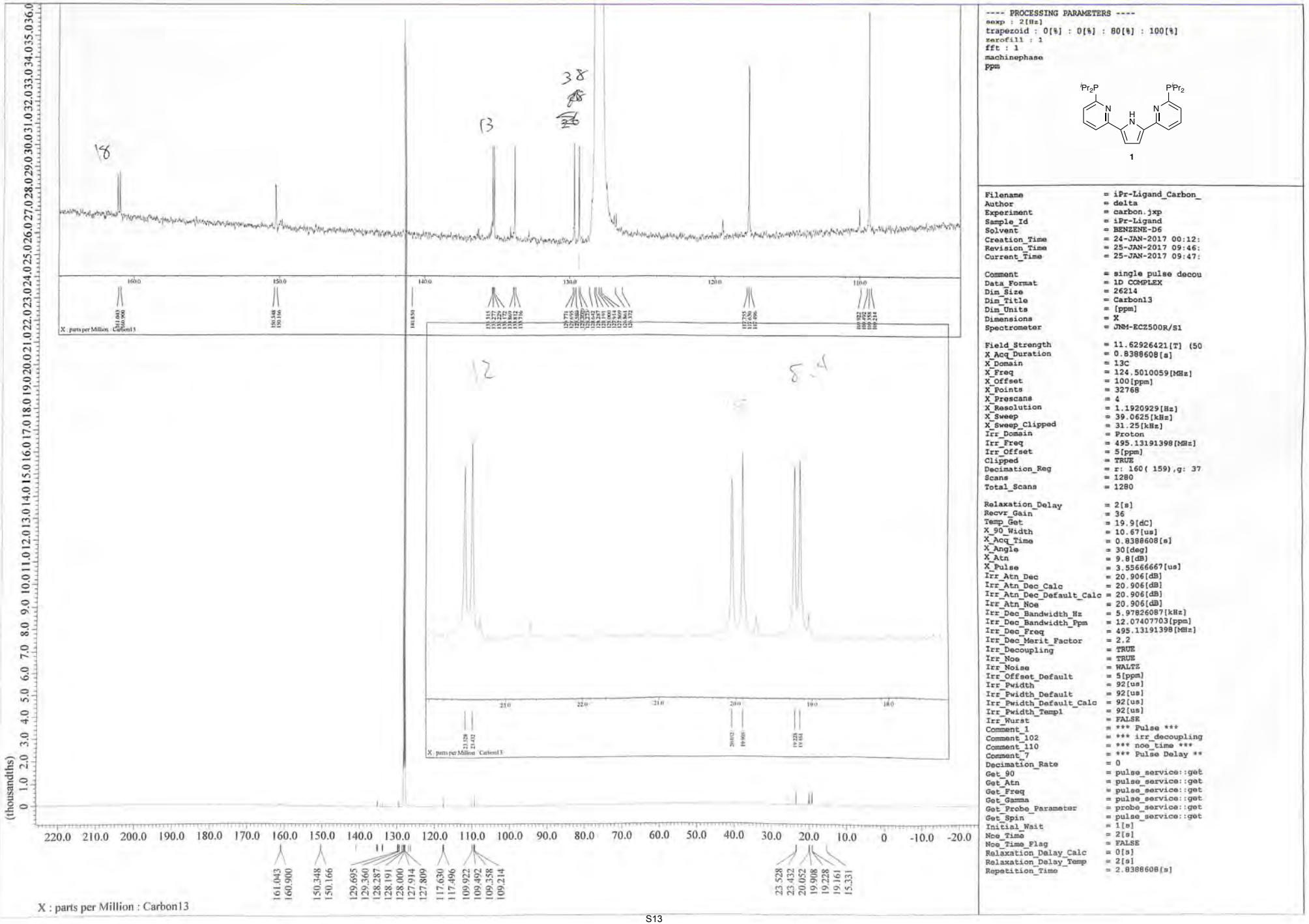
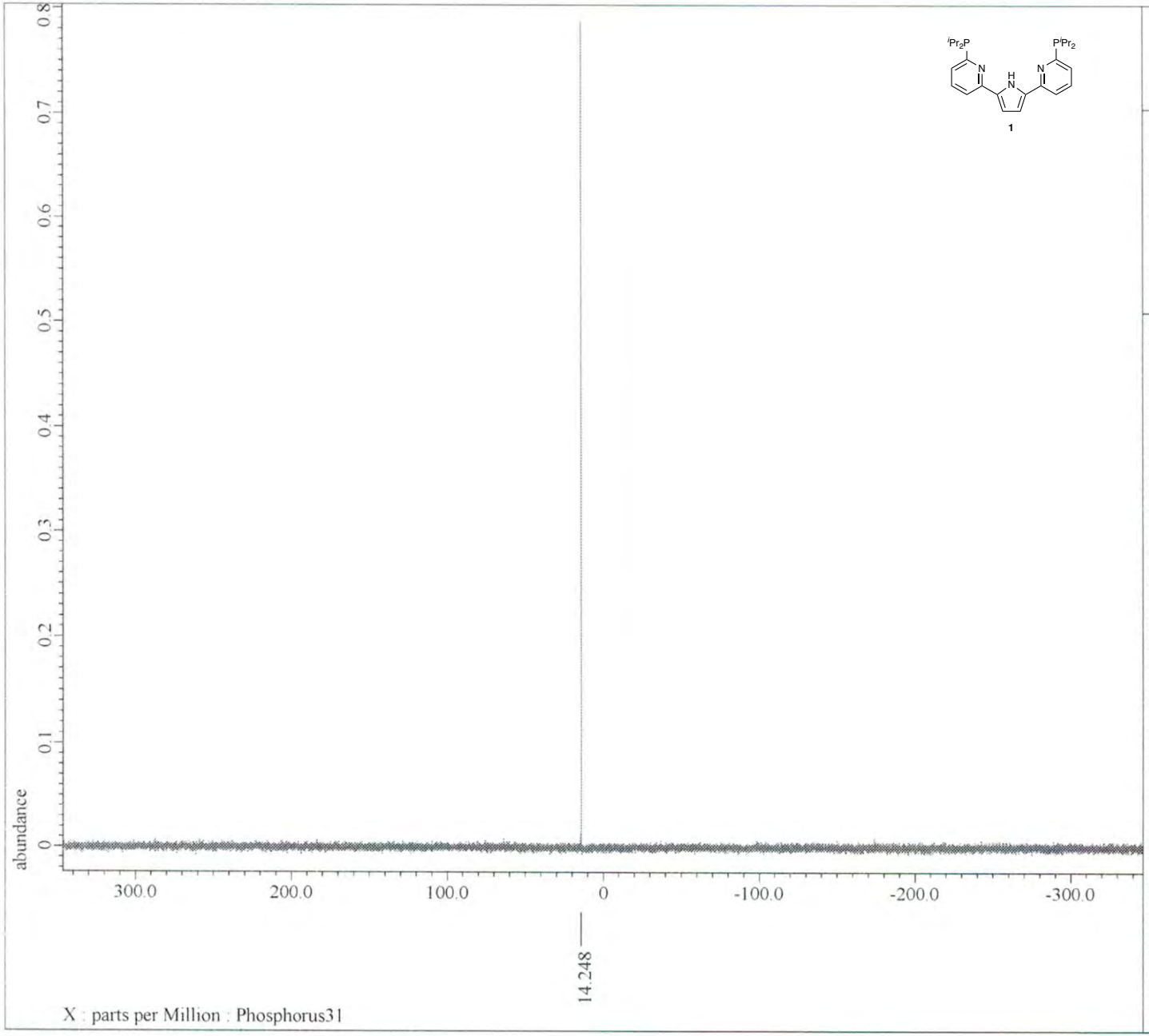
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Decimation_Reg	= r: 1346(1345),g: 52
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 56
Temp_Get	= 20.6[dC]
X_90_Width	= 12.12[us]
X_Acq_Time	= 1.76422912[s]
X_Angle	= 45[deg]
X_Atn	= 3.3[dB]
X_Pulse	= 6.06[us]
Irr_Mode	= Off
Tri_Mode	= Off
Comment_1	= *** Pulse ***
Comment_111	= *** presat_time ***
Comment_201	= *** obs_dante_presatu
Comment_202	= *** irr_ preaturation
Comment_203	= *** tri_ preaturation



1

Filename	= iPr-Ligand_Carbon_
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= iPr-Ligand
Solvent	= BENZENE-D6
Creation_Time	= 24-JAN-2017 00:12:
Revision_Time	= 25-JAN-2017 09:46:
Current_Time	= 25-JAN-2017 09:47:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ500R/S1
Field_Strength	= 11.62926421[T] (50
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
Decimation_Reg	= r: 160(159),g: 37
Scans	= 1280
Total_Scans	= 1280
Relaxation_Delay	= 2[s]
Recvr_Gain	= 36
Temp_Get	= 19.9[dC]
X_90_Width	= 10.67[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 9.8[dB]
X_Pulse	= 3.55666667[us]
Irr_Atn_Dec	= 20.906[dB]
Irr_Atn_Dec_Calc	= 20.906[dB]
Irr_Atn_Dec_Default_Calc	= 20.906[dB]
Irr_Atn_Noe	= 20.906[dB]
Irr_Dec_Bandwidth_Hz	= 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.07407703[ppm]
Irr_Dec_Freq	= 495.13191398[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 92[us]
Irr_Pwidth_Default	= 92[us]
Irr_Pwidth_Default_Calc	= 92[us]
Irr_Pwidth_Templ	= 92[us]
Irr_Wurst	= FALSE
Comment_1	= *** Pulse ***
Comment_102	= *** irr_decoupling
Comment_110	= *** noe_time ***
Comment_7	= *** Pulse Delay **
Decimation_Rate	= 0
Get_90	= pulse_service::get
Get_Atn	= pulse_service::get
Get_Freq	= pulse_service::get
Get_Gamma	= pulse_service::get
Get_Probe_Parameter	= probe_service::get
Get_Spin	= pulse_service::get
Initial_Wait	= 1[s]
Noe_Time	= 2[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 2[s]
Repetition_Time	= 2.8388608[s]



1

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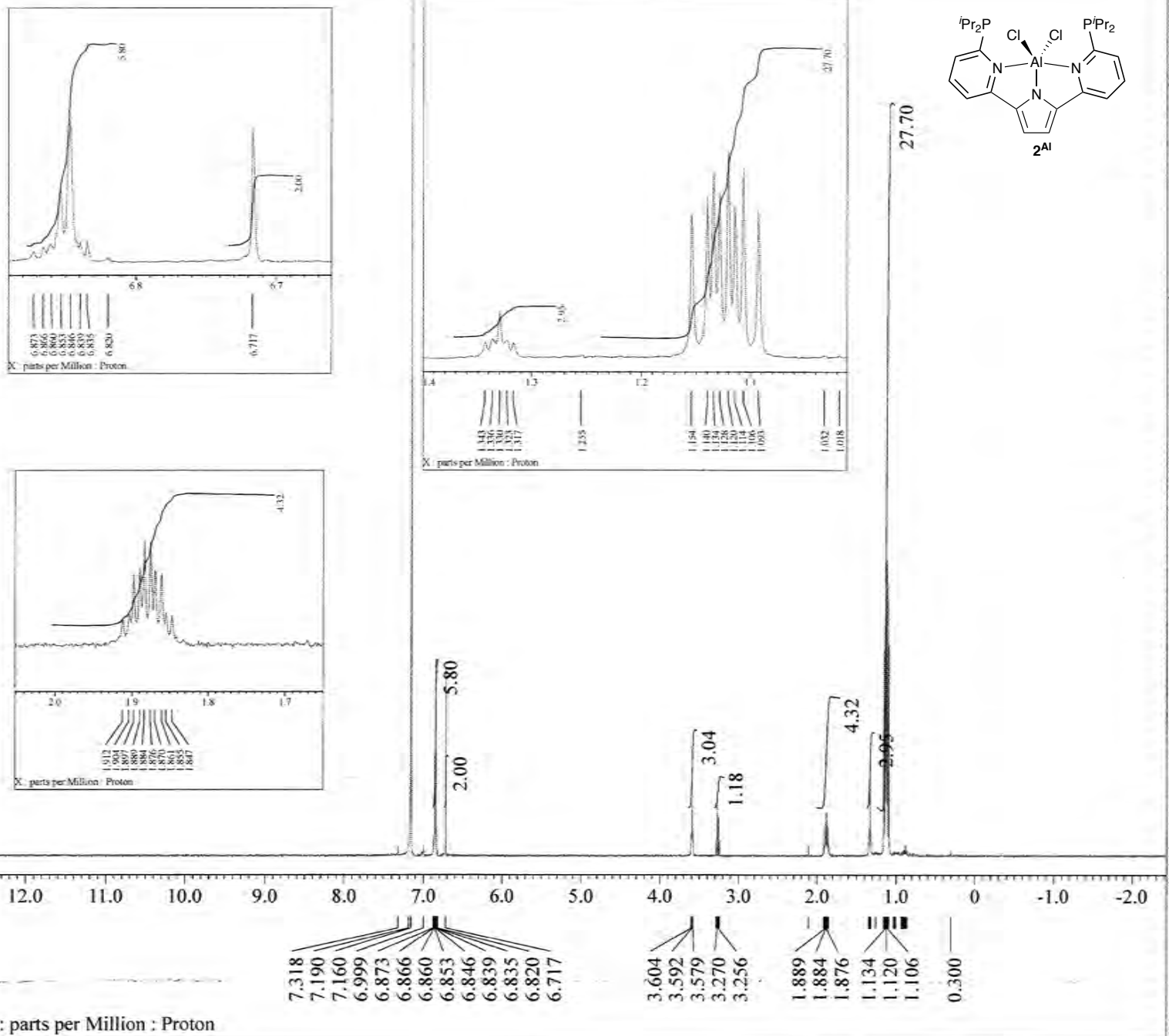
```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -51.09957, 0, 50[%] )
```

Filename	= UK-ligand-saiketu_
Author	= delta
Experiment	= single_pulse_dec.j
Sample_Id	= UK-ligand-saiketu
Solvent	= BENZENE-D6
Creation_Time	= 17-NOV-2016 16:35:
Revision_Time	= 25-JAN-2017 10:56:
Current_Time	= 25-JAN-2017 10:56:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Phosphorus31
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ500R/S1
Field_Strength	= 11.62926421[T] (50
X_Acq_Duration	= 0.18874368[s]
X_Domain	= 31P
X_Freq	= 200.43293989[MHz]
X_Offset	= 0[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 5.29819065[Hz]
X_Sweep	= 173.61111111[kHz]
X_Sweep_Clipped	= 138.88888889[kHz]
Irr_Domain	= Proton
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Decimation_Reg	= r: 36(35),g: 26
Scans	= 64
Total_Scans	= 64
Relaxation_Delay	= 2[s]
Recvr_Gain	= 66
Temp_Get	= 20.4[dC]
X_90_Width	= 31.6[us]
X_Acq_Time	= 0.18874368[s]
X_Angle	= 30[deg]
X_Atn	= 9[dB]
X_Pulse	= 10.53333333[us]
Irr_Atn_Dec	= 20.906[dB]
Irr_Atn_Dec_Calc	= 20.906[dB]
Irr_Atn_Dec_Default_Calc	= 20.906[dB]
Irr_Atn_Noe	= 20.906[dB]
Irr_Dec_Bandwidth_Hz	= 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.07407703[ppm]
Irr_Dec_Freq	= 495.13191398[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE



2Al

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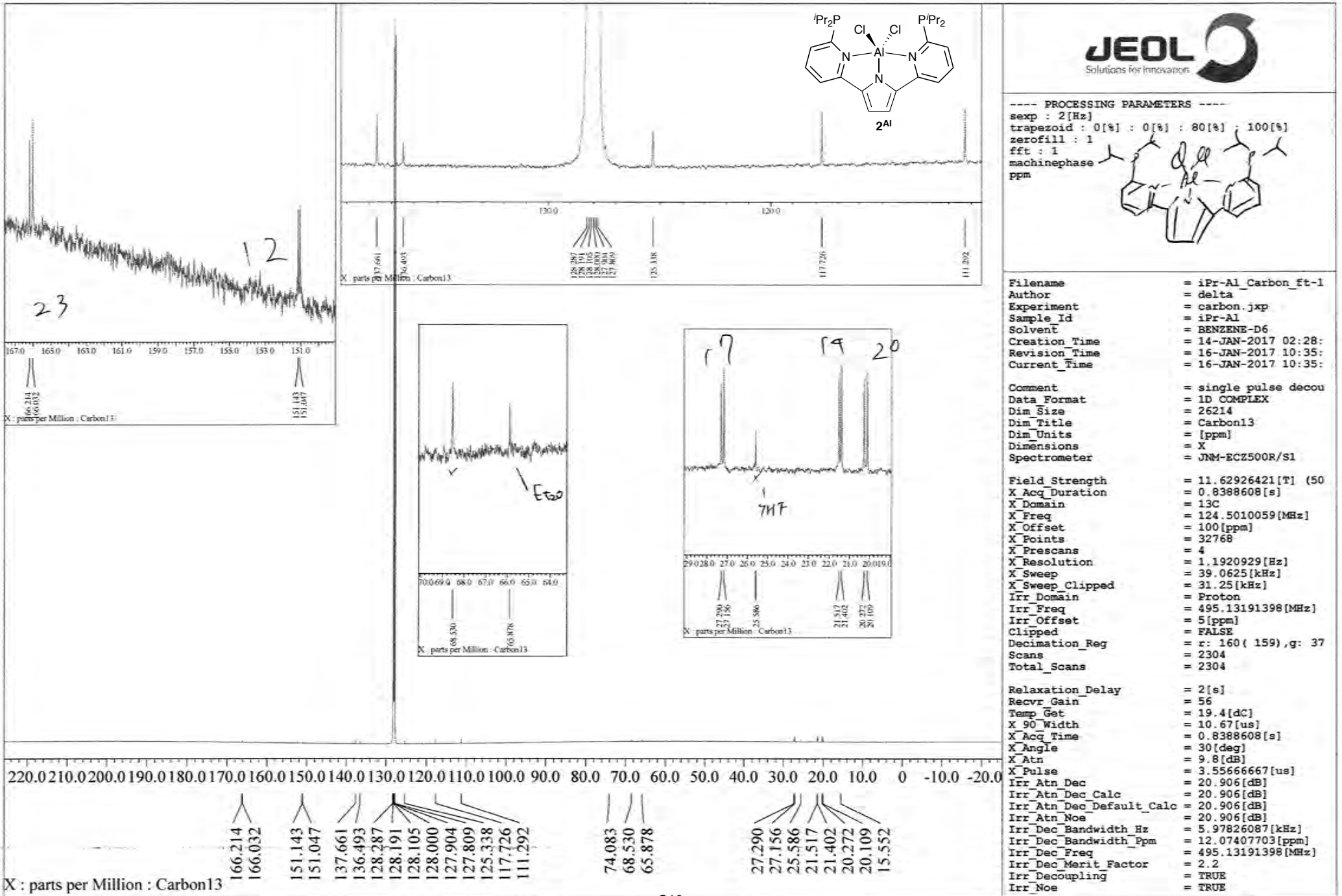
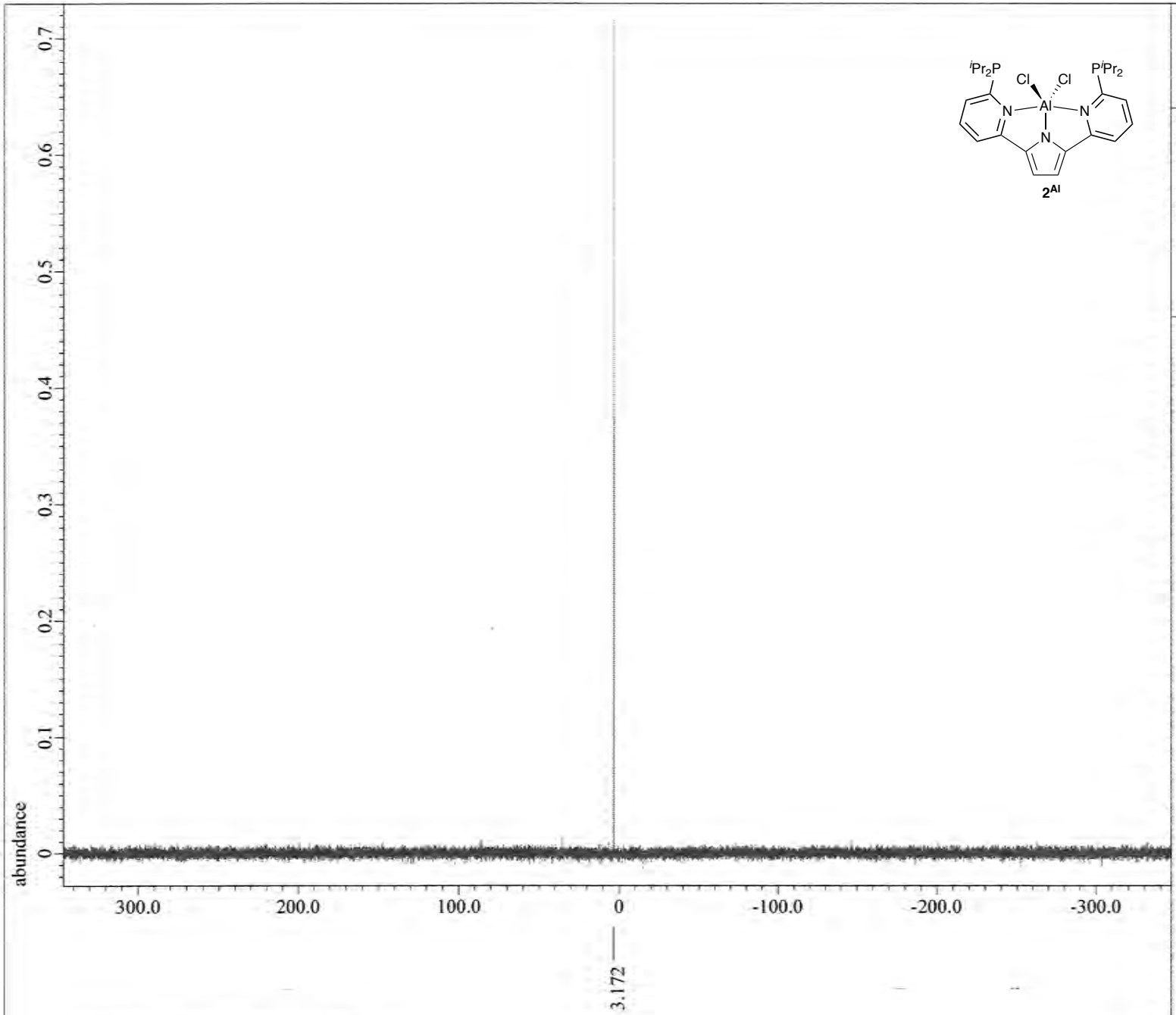
```
---- PROCESSING PARAMETERS ----
sexp : 0.2[Hz]
trapezoid : 0[%] : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1
machinephase
ppm
```

UK643

Filename	= iPr-Al_Proton_ft-1-4.
Author	= delta
Experiment	= proton.jxp
Sample_Id	= iPr-Al
Solvent	= BENZENE-D6
Creation_Time	= 14-JAN-2017 02:20:31
Revision_Time	= 16-FEB-2017 19:05:39
Current_Time	= 16-FEB-2017 19:07:22
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ500R/S1
Field_Strength	= 11.62926421[T] (500[M
X_Acq_Duration	= 1.76422912[s]
X_Domain	= 1H
X_Freq	= 495.13191398[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.5668198[Hz]
X_Sweep	= 9.28677563[kHz]
X_Sweep_Clipped	= 7.42942051[kHz]
Irr_Domain	= Proton
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 495.13191398[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Decimation_Reg	= r: 1346(1345),g: 52
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 66
Temp_Get	= 19.7[dC]
X_90_Width	= 12.12[us]
X_Acq_Time	= 1.76422912[s]
X_Angle	= 45[deg]
X_Atn	= 3.3[dB]
X_Pulse	= 6.06[us]
Irr_Mode	= Off
Tri_Mode	= Off
Comment_1	= *** Pulse ***
Comment_111	= *** presat_time ***
Comment_201	= *** obs_dante_presatu
Comment_202	= *** irr_ preaturation
Comment_203	= *** tri_ preaturation


$^{i}Pr_2P$
Cl
Cl
$P^{i}Pr_2$
N
Al
N
N
2^{Al}
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
sexp : 2[Hz]
trapezoid : 0[%] : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1
machinephase
ppm
Filename = iPr-Al_Carbon_ft-1
Author = delta
Experiment = carbon.jxp
Sample_Id = iPr-Al
Solvent = BENZENE-D6
Creation_Time = 14-JAN-2017 02:28:
Revision_Time = 16-JAN-2017 10:35:
Current_Time = 16-JAN-2017 10:35:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (50
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
Decimation_Reg = r: 160(159),g: 37
Scans = 2304
Total_Scans = 2304
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 19.4[dC]
X_90_Width = 10.67[us]
X_Acq_Time = 0.8388608[s]
X_Angle = 30[deg]
X_Atn = 9.8[dB]
X_Pulse = 3.55666667[us]
Irr_Atn_Dec = 20.906[dB]
Irr_Atn_Dec_Calc = 20.906[dB]
Irr_Atn_Dec_Default_Calc = 20.906[dB]
Irr_Atn_Noe = 20.906[dB]
Irr_Dec_Bandwidth_Hz = 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm = 12.07407703[ppm]
Irr_Dec_Freq = 495.13191398[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
166.214
166.032
151.143
151.047
137.661
136.493
128.287
128.191
128.105
128.000
127.904
127.809
125.338
117.726
111.292
74.083
68.530
65.878
27.290
27.156
25.586
21.517
21.402
20.272
20.109
15.552
X : parts per Million : Carbon13



2^{Al}

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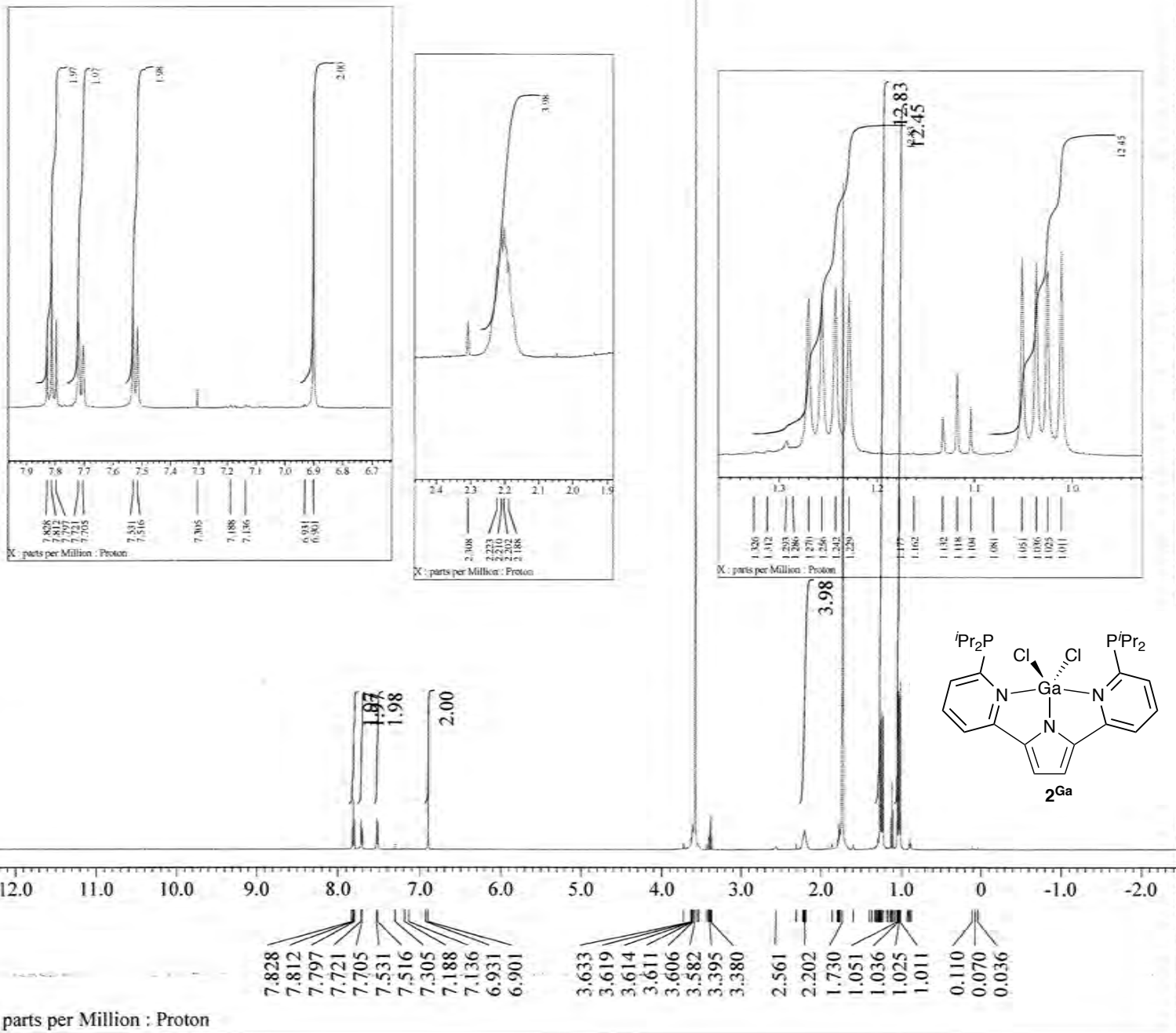
```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -30.93398, 0, 50[%] )
```

以下に由来：: iPr-Al_31P_1Hdec-1-1.jdf

Filename	= iPr-Al_31P_1Hdec-1
Author	= delta
Experiment	= single_pulse_dec.j
Sample_Id	= iPr-Al
Solvent	= BENZENE-D6
Creation_Time	= 14-JAN-2017 02:24:
Revision_Time	= 16-JAN-2017 11:01:
Current_Time	= 16-JAN-2017 11:02:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Phosphorus31
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ500R/S1
Field_Strength	= 11.62926421[T] (50
X_Acq_Duration	= 0.18874368[s]
X_Domain	= 31P
X_Freq	= 200.43293989[MHz]
X_Offset	= 0[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 5.29819065[Hz]
X_Sweep	= 173.61111111[kHz]
X_Sweep_Clipped	= 138.88888889[kHz]
Irr_Domain	= Proton
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Decimation_Reg	= r: 36(35),g: 26
Scans	= 64
Total_Scans	= 64
Relaxation_Delay	= 2[s]
Recvr_Gain	= 66
Temp_Get	= 19.8[dC]
X_90_Width	= 31.6[us]
X_Acq_Time	= 0.18874368[s]
X_Angle	= 30[deg]
X_Atn	= 9[dB]
X_Pulse	= 10.53333333[us]
Irr_Atn_Dec	= 20.906[dB]
Irr_Atn_Dec_Calc	= 20.906[dB]
Irr_Atn_Dec_Default_Calc	= 20.906[dB]
Irr_Atn_Noe	= 20.906[dB]
Irr_Dec_Bandwidth_Hz	= 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.07407703[ppm]
Irr_Dec_Freq	= 495.13191398[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE


JEOL
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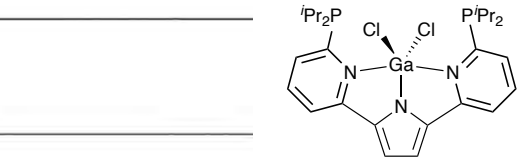
---- PROCESSING PARAMETERS ----
sexp : 0.2[Hz]
trapezoid : 0[%] : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1
machinephase
ppm
Filename = iPe-GaCl2-Et2O_Proton
Author = delta
Experiment = proton.jxp
Sample_Id = iPe-GaCl2-Et2O
Solvent = TETRAHYDROFURAN-D8
Creation_Time = 27-JAN-2017 05:07:23
Revision_Time = 21-FEB-2017 22:23:24
Current_Time = 21-FEB-2017 22:23:39
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (500[M
X_Acq_Duration = 1.76422912[s]
X_Domain = 1H
X_Freq = 495.13191398[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.5668198[Hz]
X_Sweep = 9.28677563[kHz]
X_Sweep_Clipped = 7.42942051[kHz]
Irr_Domain = Proton
Irr_Freq = 495.13191398[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 495.13191398[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Decimation_Reg = r: 1346(1345),g: 52
Scans = 32
Total_Scans = 32
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 19.8[dC]
X_90_Width = 12.12[us]
X_Acq_Time = 1.76422912[s]
X_Angle = 45[deg]
X_Atn = 3.3[dB]
X_Pulse = 6.06[us]
Irr_Mode = Off
Tri_Mode = Off
Comment_1 = *** Pulse ***
Comment_111 = *** presat_time ***
Comment_201 = *** obs_dante_presatu
Comment_202 = *** irr_ preaturation
Comment_203 = *** tri_ preaturation
iPr2P
Cl
Cl
PiPr2
N
Ga
N
N
2Ga
X : parts per Million : Proton



2^{Ga}

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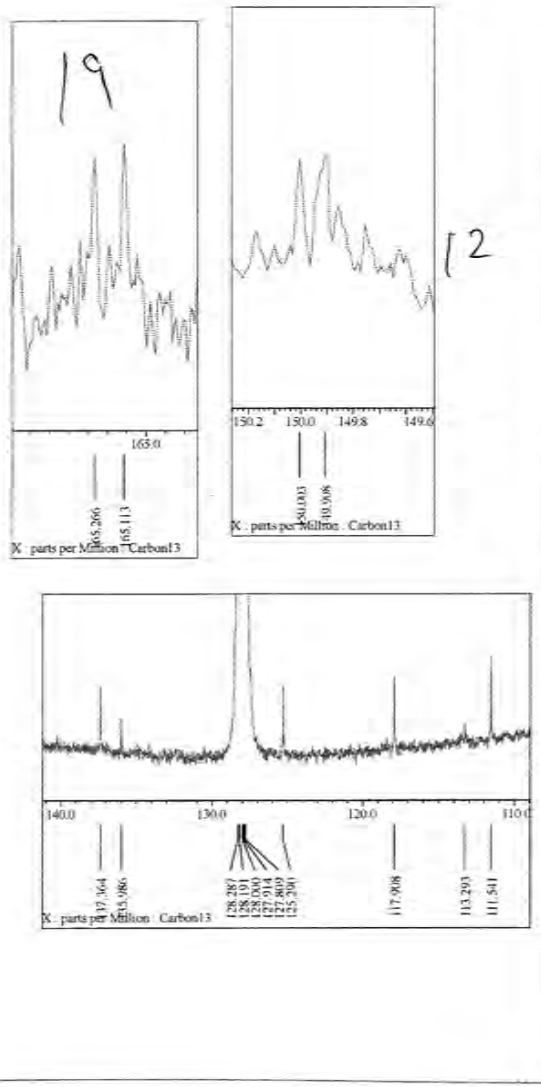
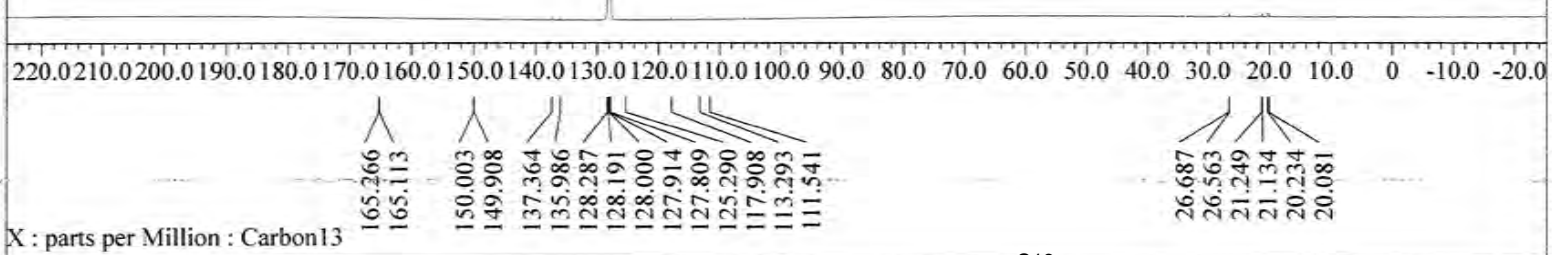
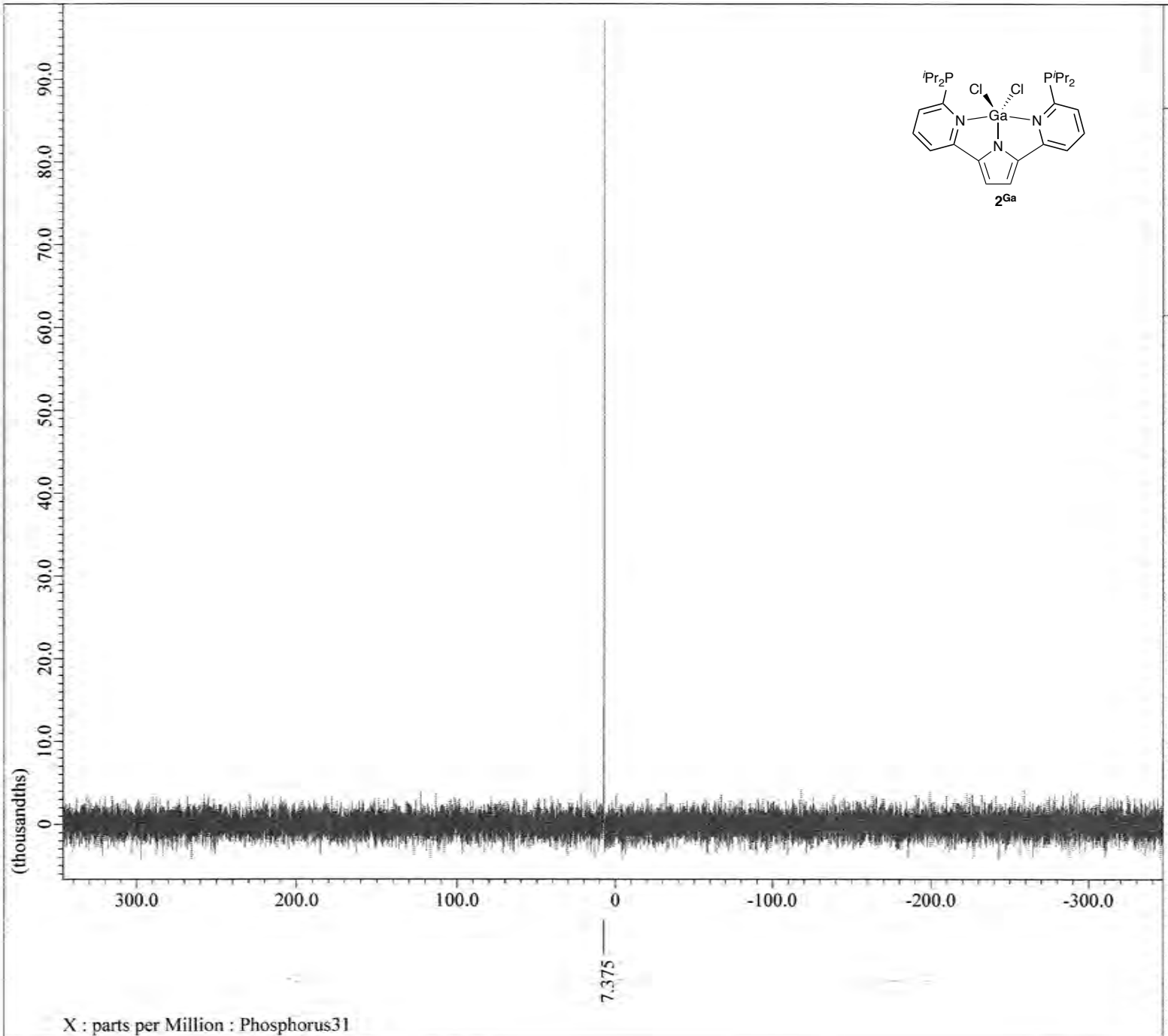



```
---- PROCESSING PARAMETERS ----
sexp : 2[Hz]
trapezoid : 0[%] : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1
machinephase
ppm
```

Filename	= iPr-Ga_Carbon_ft-1
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= iPr-Ga
Solvent	= BENZENE-D6
Creation_Time	= 14-JAN-2017 04:30:
Revision_Time	= 16-JAN-2017 10:49:
Current_Time	= 16-JAN-2017 10:49:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ500R/S1
Field_Strength	= 11.62926421[T] (50
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
Decimation_Reg	= r: 160(159),g: 37
Scans	= 2304
Total_Scans	= 2304
Relaxation_Delay	= 2[s]
Recvr_Gain	= 56
Temp_Get	= 19.9[dC]
X_90_Width	= 10.67[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 9.8[dB]
X_Pulse	= 3.55666667[us]
Irr_Atn_Dec	= 20.906[dB]
Irr_Atn_Dec_Calc	= 20.906[dB]
Irr_Atn_Dec_Default_Calc	= 20.906[dB]
Irr_Atn_Noe	= 20.906[dB]
Irr_Dec_Bandwidth_Hz	= 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.07407703[ppm]
Irr_Dec_Freq	= 495.13191398[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE





2^{Ga}

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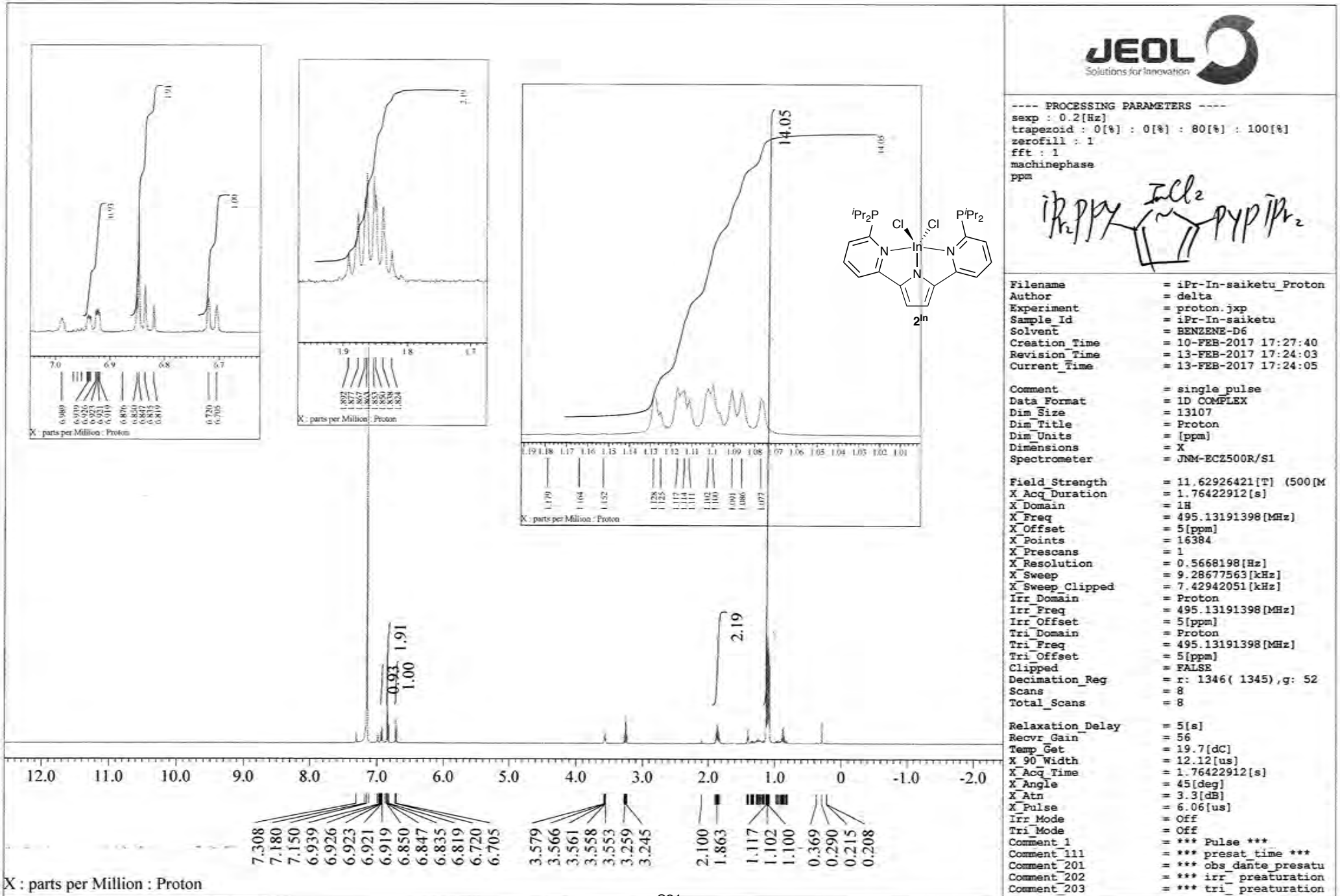
Solutions for Innovation

```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= iPe-GaCl2-Et2O_31P
Author	= delta
Experiment	= single_pulse_dec.j
Sample_Id	= iPe-GaCl2-Et2O
Solvent	= TETRAHYDROFURAN-D8
Creation_Time	= 27-JAN-2017 05:13:
Revision_Time	= 27-JAN-2017 09:38:
Current_Time	= 21-FEB-2017 22:21:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Phosphorus31
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-EC2500R/S1
Field_Strength	= 11.62926421[T] (50
X_Acq_Duration	= 0.18874368[s]
X_Domain	= 31P
X_Freq	= 200.43293989[MHz]
X_Offset	= 0[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 5.29819065[Hz]
X_Sweep	= 173.61111111[kHz]
X_Sweep_Clipped	= 138.88888889[kHz]
Irr_Domain	= Proton
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Decimation_Reg	= r: 36(35),g: 26
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 2[s]
Recvr_Gain	= 66
Temp_Get	= 19.8[dC]
X_90_Width	= 31.6[us]
X_Acq_Time	= 0.18874368[s]
X_Angle	= 30[deg]
X_Atn	= 9[dB]
X_Pulse	= 10.53333333[us]
Irr_Atn_Dec	= 20.906[dB]
Irr_Atn_Dec_Calc	= 20.906[dB]
Irr_Atn_Dec_Default_Calc	= 20.906[dB]
Irr_Atn_Noe	= 20.906[dB]
Irr_Dec_Bandwidth_Hz	= 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.07407703[ppm]
Irr_Dec_Freq	= 495.13191398[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE


JEOL
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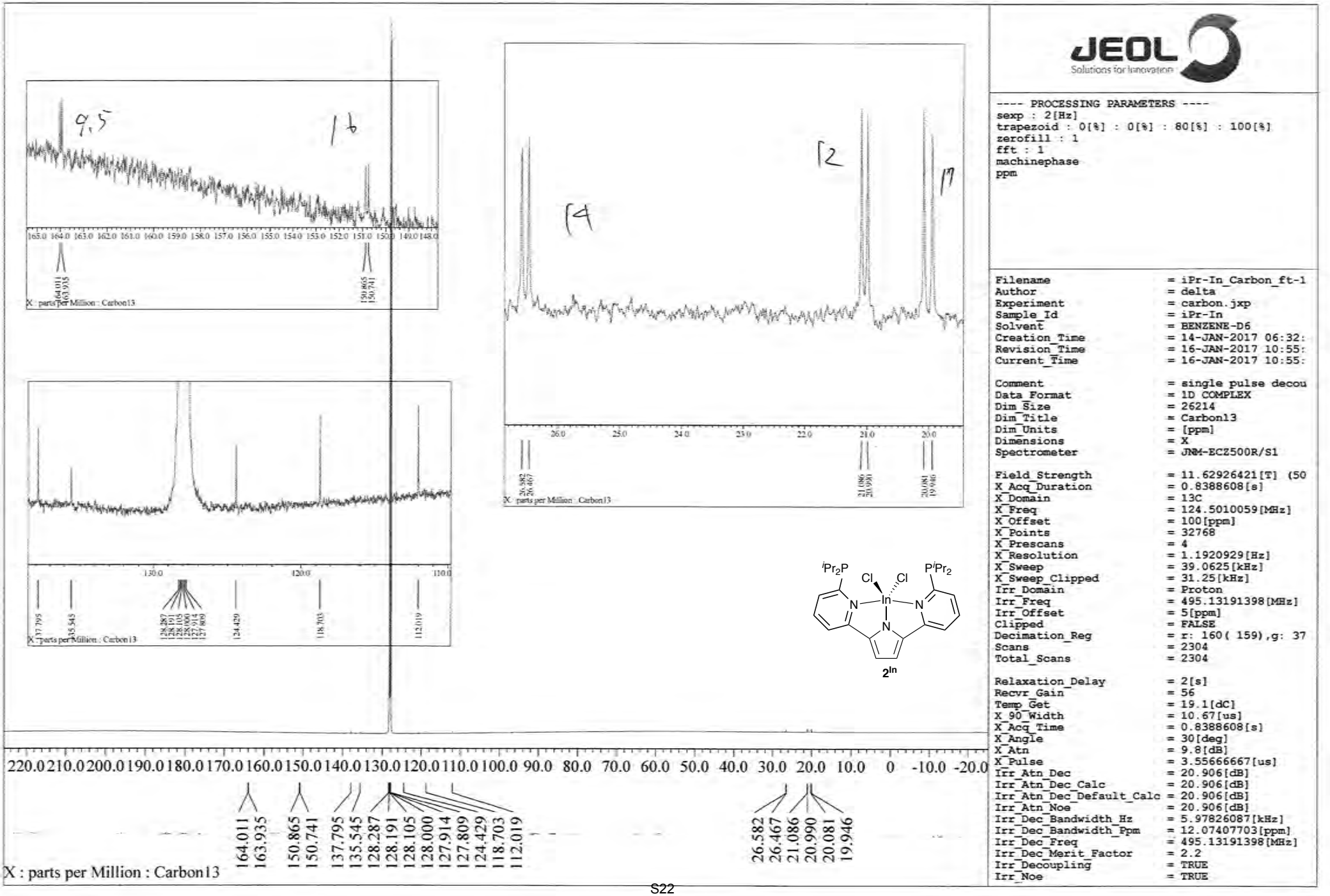
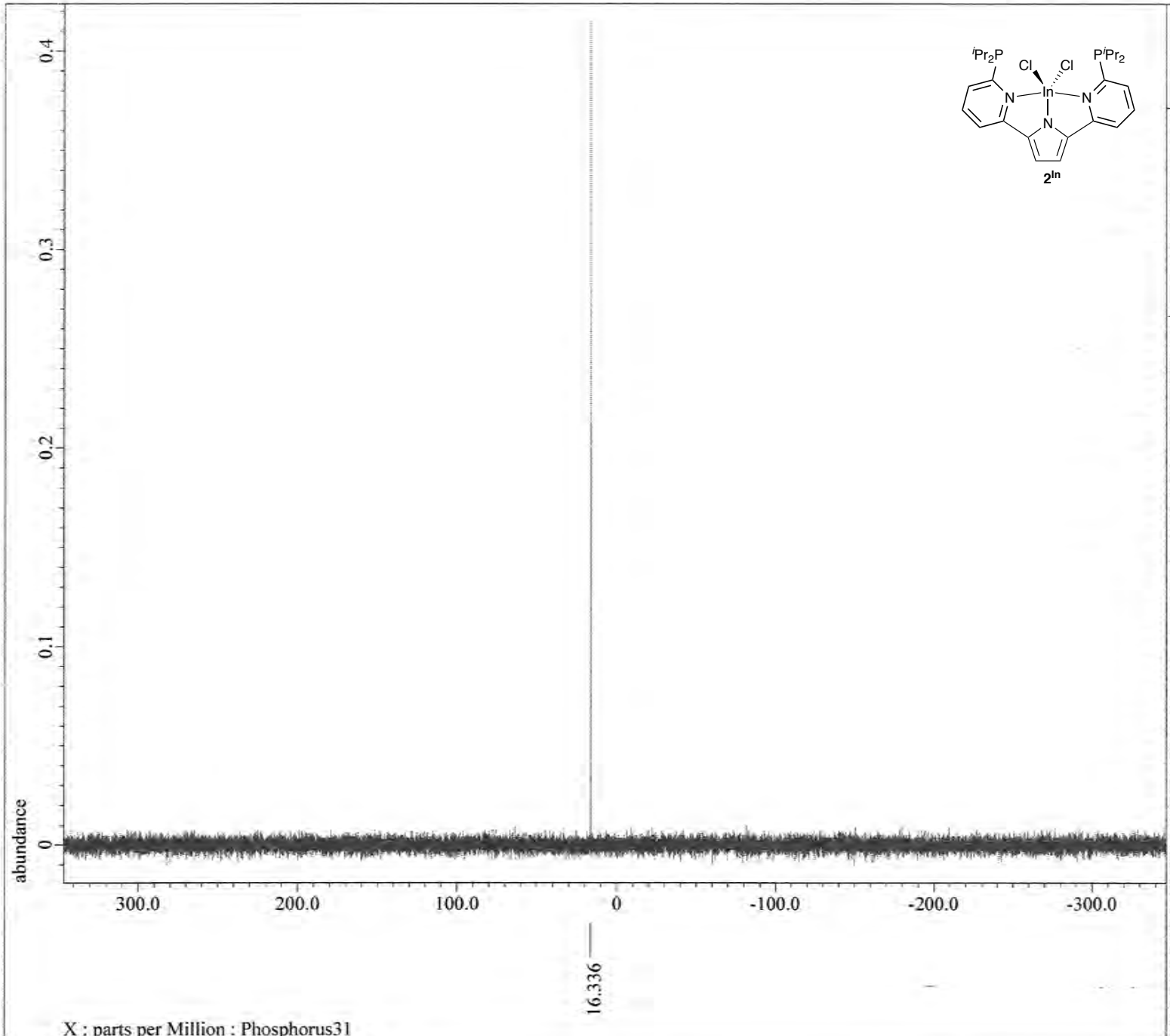
---- PROCESSING PARAMETERS ----
sexp : 0.2[Hz]
trapezoid : 0[%] : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1
machinephase
ppm
Filename = iPr-In-saiketu_Proton
Author = delta
Experiment = proton.jxp
Sample_Id = iPr-In-saiketu
Solvent = BENZENE-D6
Creation_Time = 10-FEB-2017 17:27:40
Revision_Time = 13-FEB-2017 17:24:03
Current_Time = 13-FEB-2017 17:24:05
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (500[M
X_Acq_Duration = 1.76422912[s]
X_Domain = 1H
X_Freq = 495.13191398[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.5668198[Hz]
X_Sweep = 9.28677563[kHz]
X_Sweep_Clipped = 7.42942051[kHz]
Irr_Domain = Proton
Irr_Freq = 495.13191398[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 495.13191398[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Decimation_Reg = r: 1346(1345),g: 52
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 19.7[dC]
X_90_Width = 12.12[us]
X_Acq_Time = 1.76422912[s]
X_Angle = 45[deg]
X_Atn = 3.3[dB]
X_Pulse = 6.06[us]
Irr_Mode = Off
Tri_Mode = Off
Comment_1 = *** Pulse ***
Comment_111 = *** presat_time ***
Comment_201 = *** obs_dante_presatu
Comment_202 = *** irr_ preaturation
Comment_203 = *** tri_ preaturation
2In
X : parts per Million : Proton


9,5
1b
12
14
19
X : parts per Million : Carbon13
164.011
163.935
150.865
150.741
137.795
135.545
128.287
128.191
128.105
128.000
127.914
127.809
124.429
118.703
112.019
26.582
26.467
21.086
20.990
20.081
19.946
$^{i}Pr_2P$
Cl
Cl
$P^{i}Pr_2$
In
N
N
N
2^{In}
---- PROCESSING PARAMETERS ----
sexp : 2[Hz]
trapezoid : 0[%] : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1
machinephase
ppm
Filename = iPr-In_Carbon_ft-1
Author = delta
Experiment = carbon.jxp
Sample_Id = iPr-In
Solvent = BENZENE-D6
Creation_Time = 14-JAN-2017 06:32:
Revision_Time = 16-JAN-2017 10:55:
Current_Time = 16-JAN-2017 10:55:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (50
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
Decimation_Reg = r: 160(159),g: 37
Scans = 2304
Total_Scans = 2304
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 19.1[dC]
X_90_Width = 10.67[us]
X_Acq_Time = 0.8388608[s]
X_Angle = 30[deg]
X_Atn = 9.8[dB]
X_Pulse = 3.55666667[us]
Irr_Atn_Dec = 20.906[dB]
Irr_Atn_Dec_Calc = 20.906[dB]
Irr_Atn_Dec_Default_Calc = 20.906[dB]
Irr_Atn_Noe = 20.906[dB]
Irr_Dec_Bandwidth_Hz = 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm = 12.07407703[ppm]
Irr_Dec_Freq = 495.13191398[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE



2^{In}

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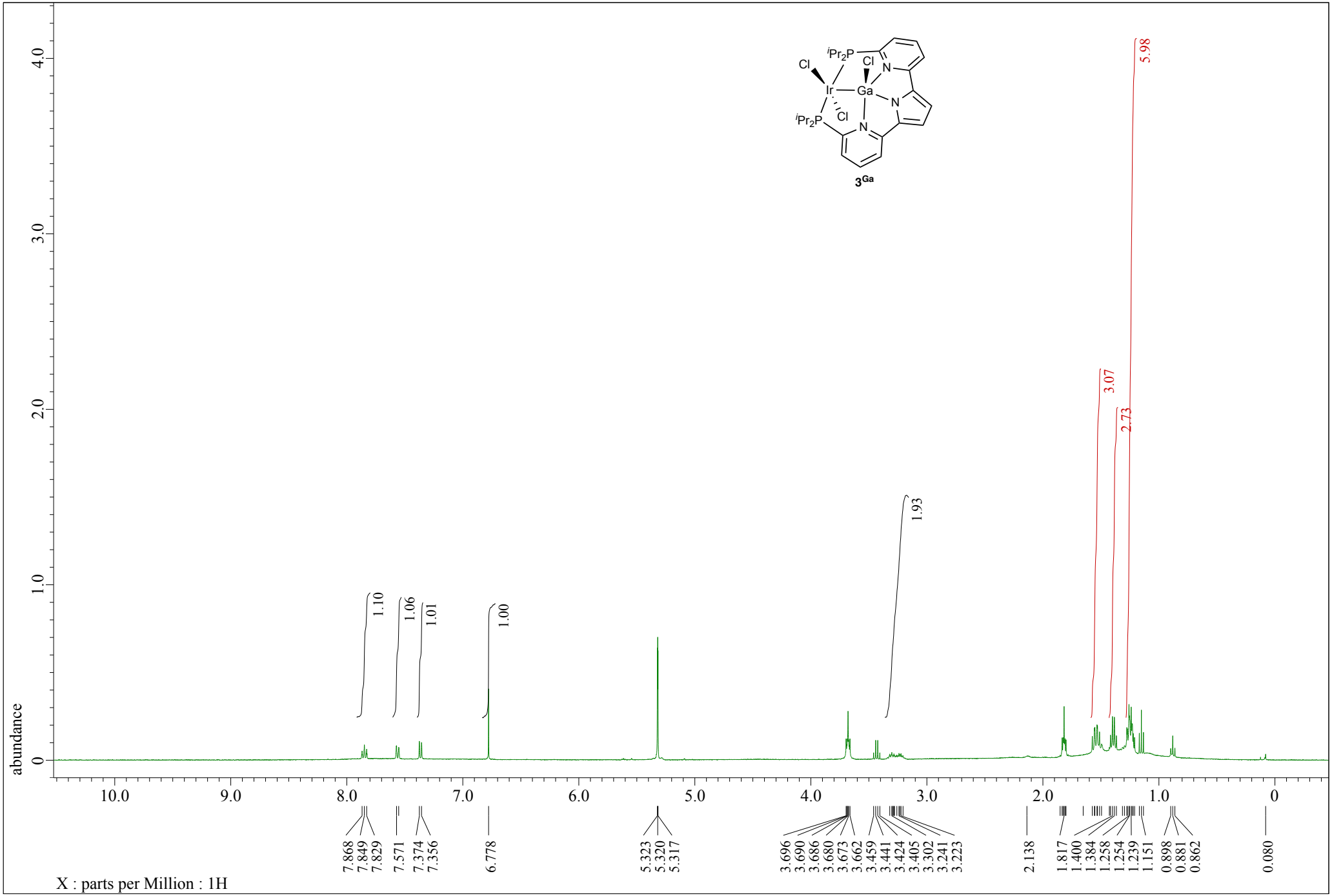
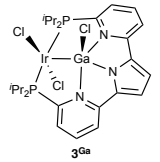
```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -18.35636, 0, 50[%] )
```

以下に由来：: iPr-In_31P_1Hdec-1-1.jdf

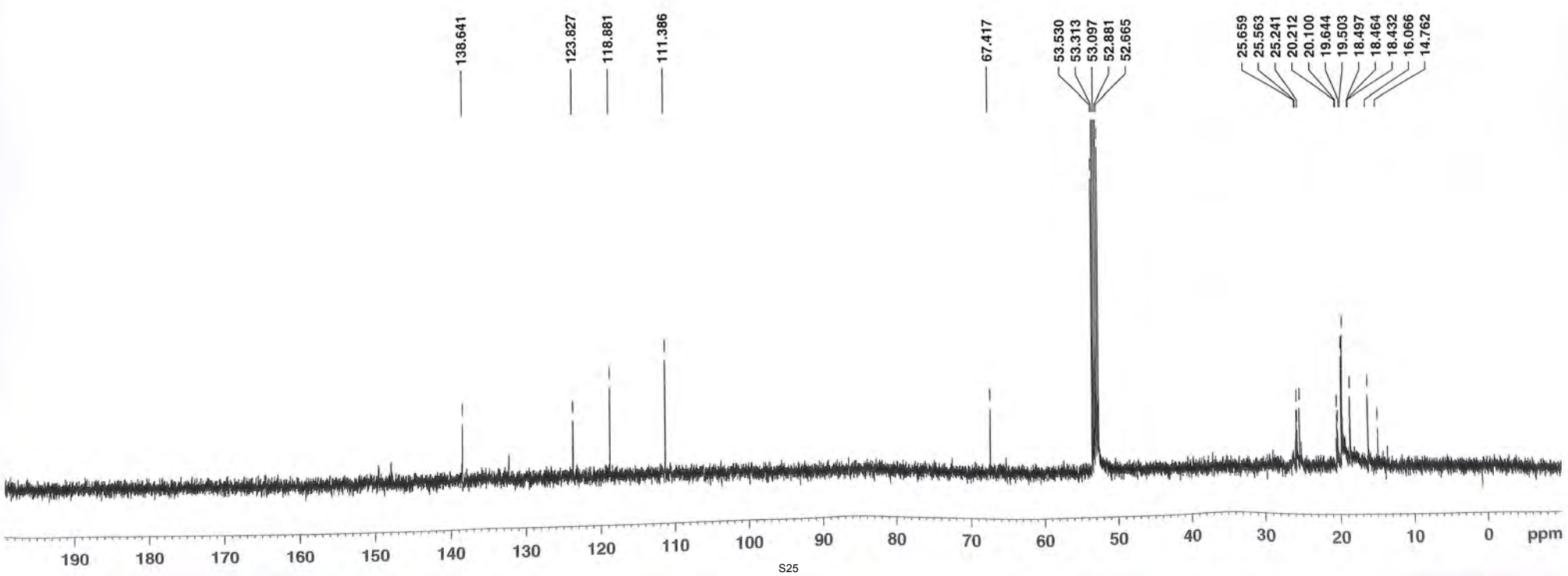
Filename	= iPr-In_31P_1Hdec-1
Author	= delta
Experiment	= single_pulse_dec.j
Sample_Id	= iPr-In
Solvent	= BENZENE-D6
Creation_Time	= 14-JAN-2017 06:27:
Revision_Time	= 14-JAN-2017 06:30:
Current_Time	= 16-JAN-2017 11:01:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Phosphorus31
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ500R/S1
Field_Strength	= 11.62926421[T] (50
X_Acq_Duration	= 0.18874368[s]
X_Domain	= 31P
X_Freq	= 200.43293989[MHz]
X_Offset	= 0[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 5.29819065[Hz]
X_Sweep	= 173.61111111[kHz]
X_Sweep_Clipped	= 138.88888889[kHz]
Irr_Domain	= Proton
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Decimation_Reg	= r: 36(35),g: 26
Scans	= 64
Total_Scans	= 64
Relaxation_Delay	= 2[s]
Recvr_Gain	= 66
Temp_Get	= 20.4[dC]
X_90_Width	= 31.6[us]
X_Acq_Time	= 0.18874368[s]
X_Angle	= 30[deg]
X_Atn	= 9[dB]
X_Pulse	= 10.53333333[us]
Irr_Atn_Dec	= 20.906[dB]
Irr_Atn_Dec_Calc	= 20.906[dB]
Irr_Atn_Dec_Default_Calc	= 20.906[dB]
Irr_Atn_Noe	= 20.906[dB]
Irr_Dec_Bandwidth_Hz	= 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.07407703[ppm]
Irr_Dec_Freq	= 495.13191398[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

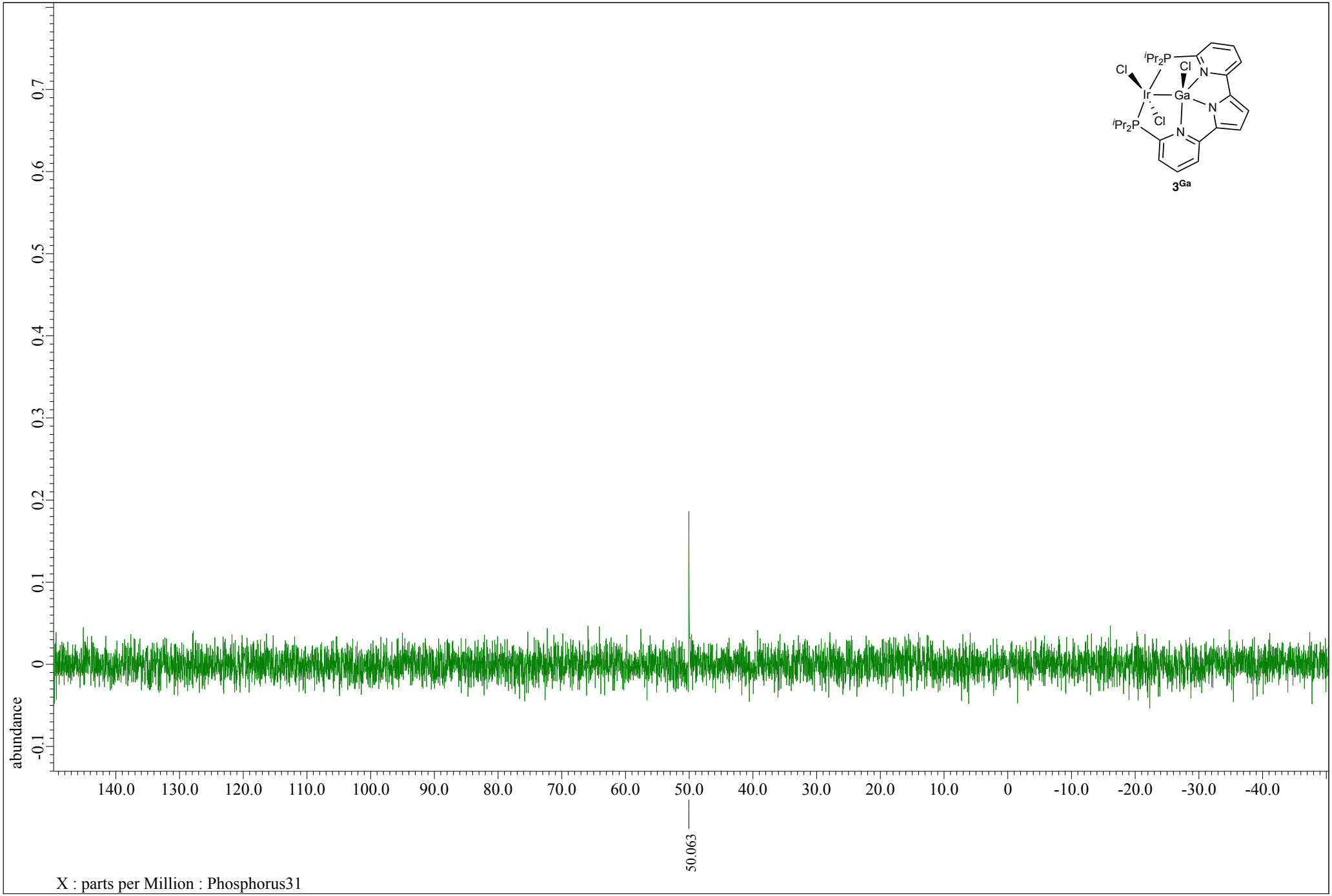

abundance
X : parts per Million : 1H
3Ga
1.10
1.06
1.01
1.00
1.93
3.07
2.73
5.98
7.868
7.849
7.829
7.571
7.374
7.356
6.778
5.323
5.320
5.317
3.696
3.690
3.686
3.680
3.673
3.662
3.459
3.441
3.424
3.405
3.302
3.241
3.223
2.138
1.817
1.400
1.384
1.258
1.254
1.239
1.151
0.898
0.881
0.862
0.080

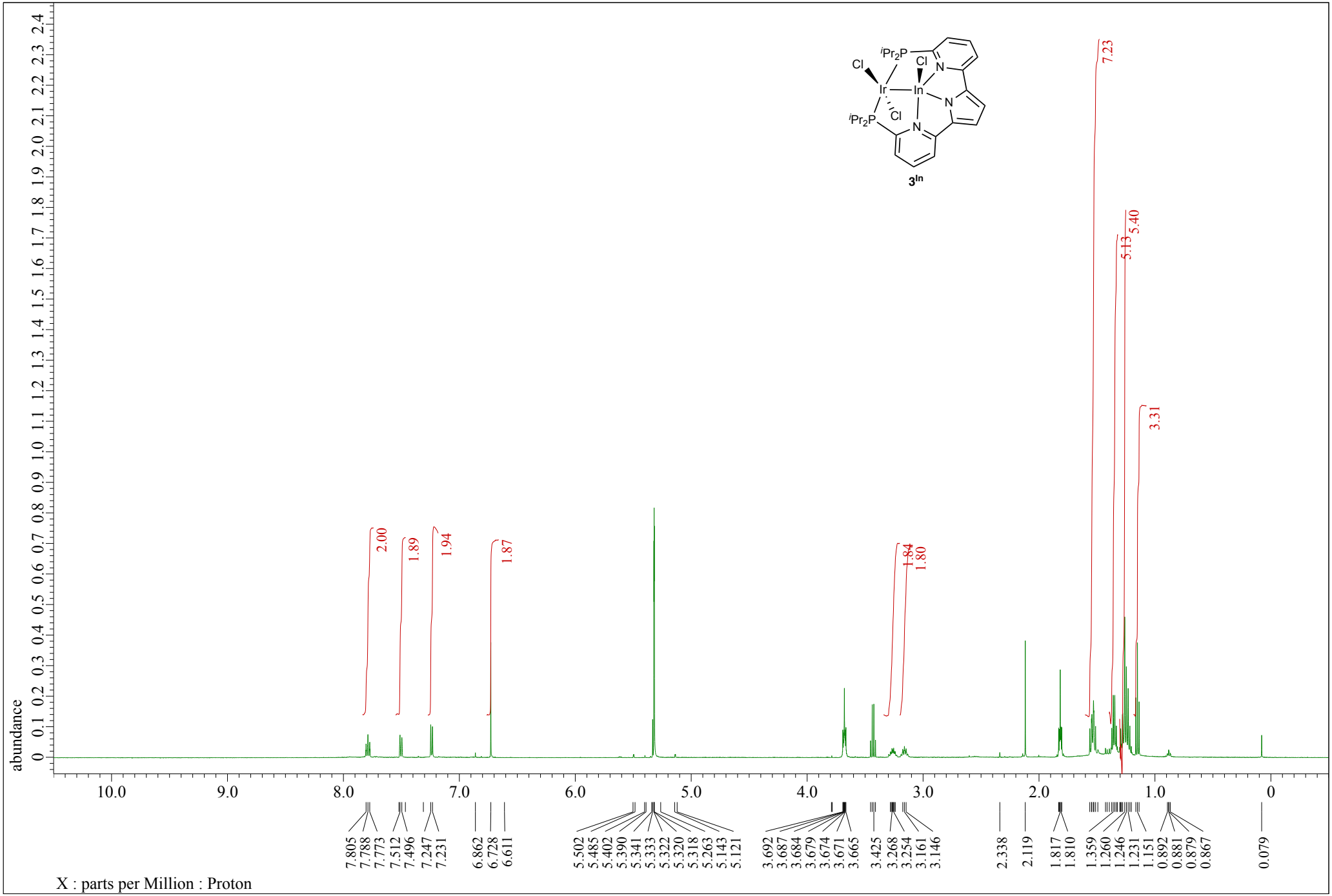
BPPP_GaIrCl2

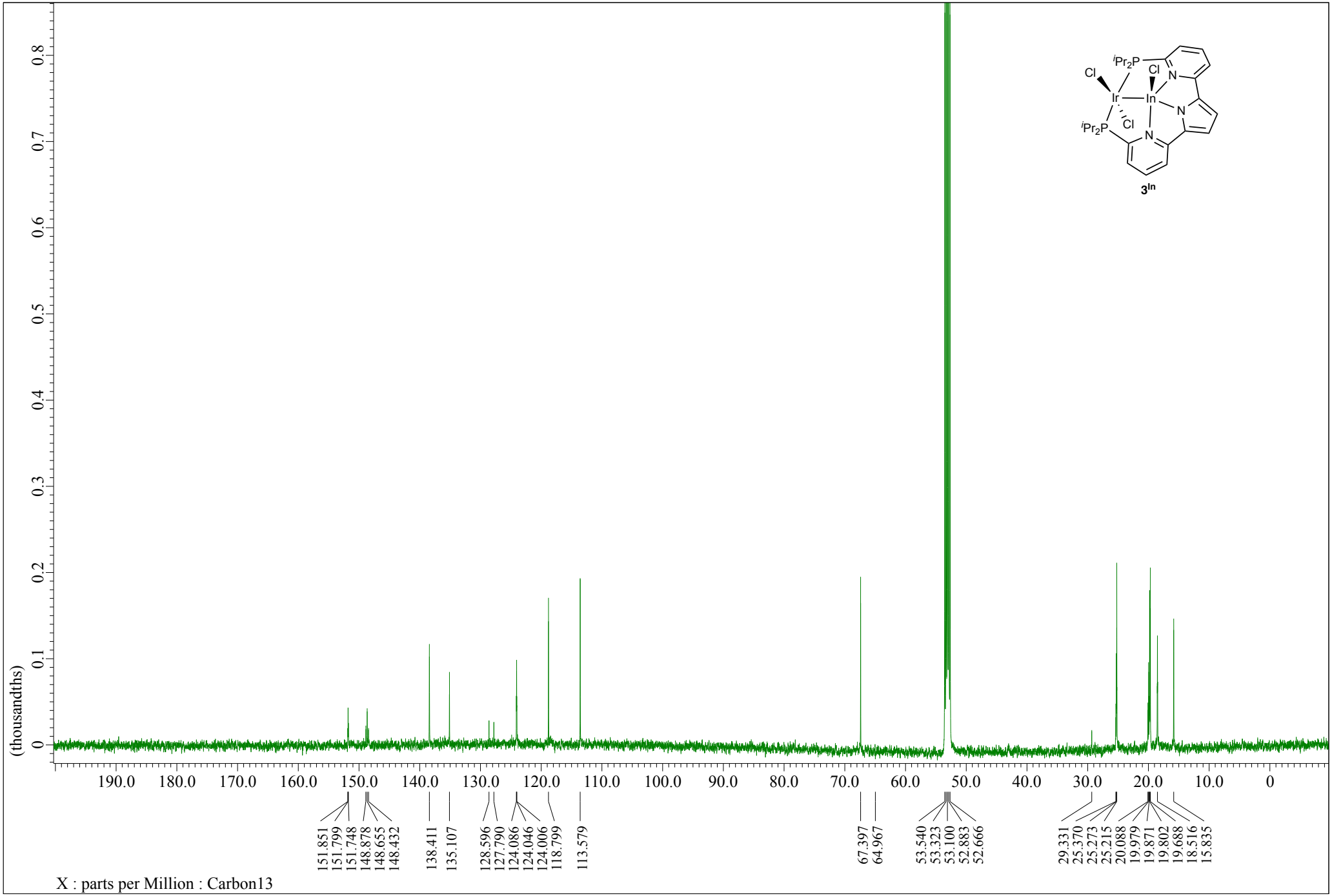


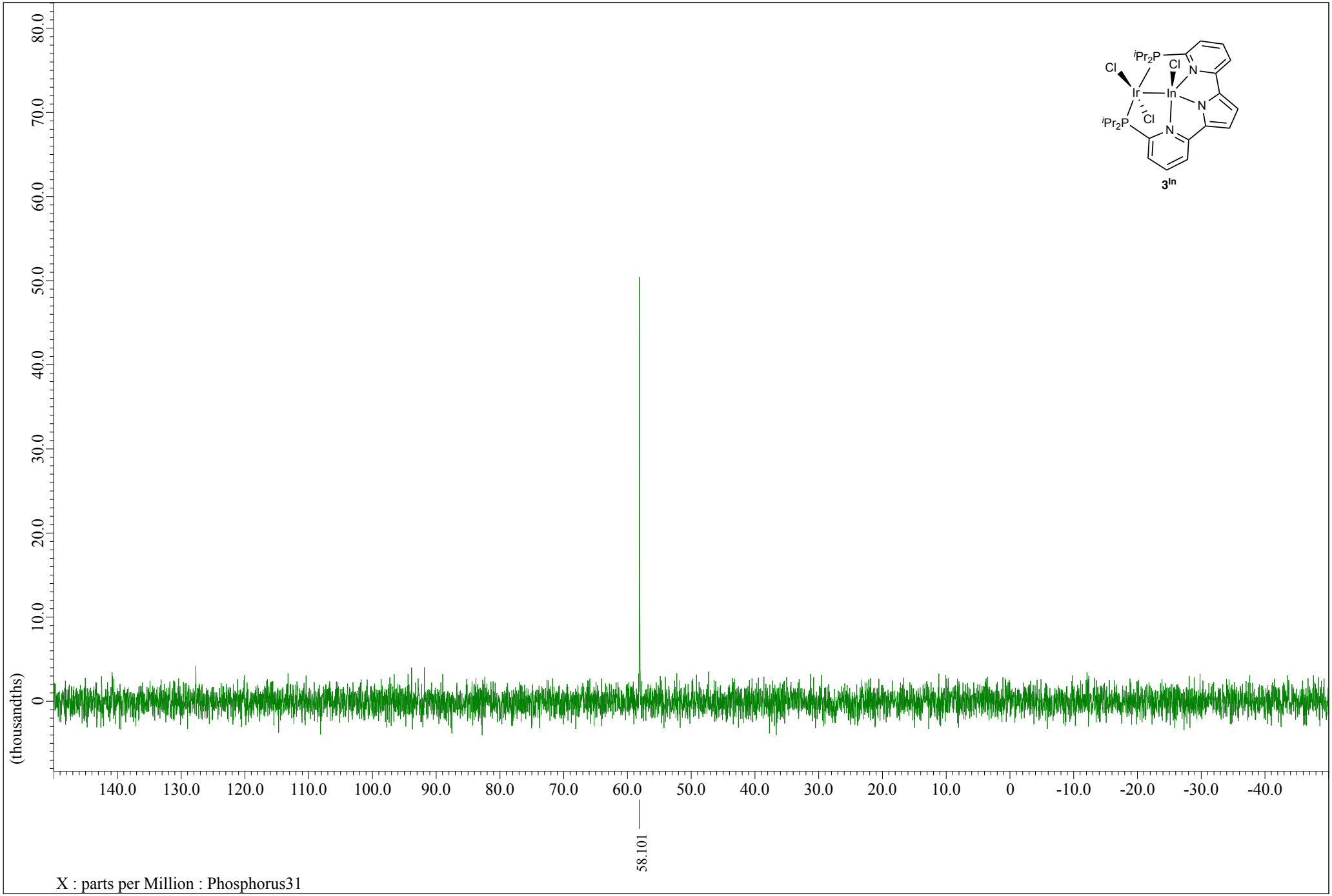
3Ga

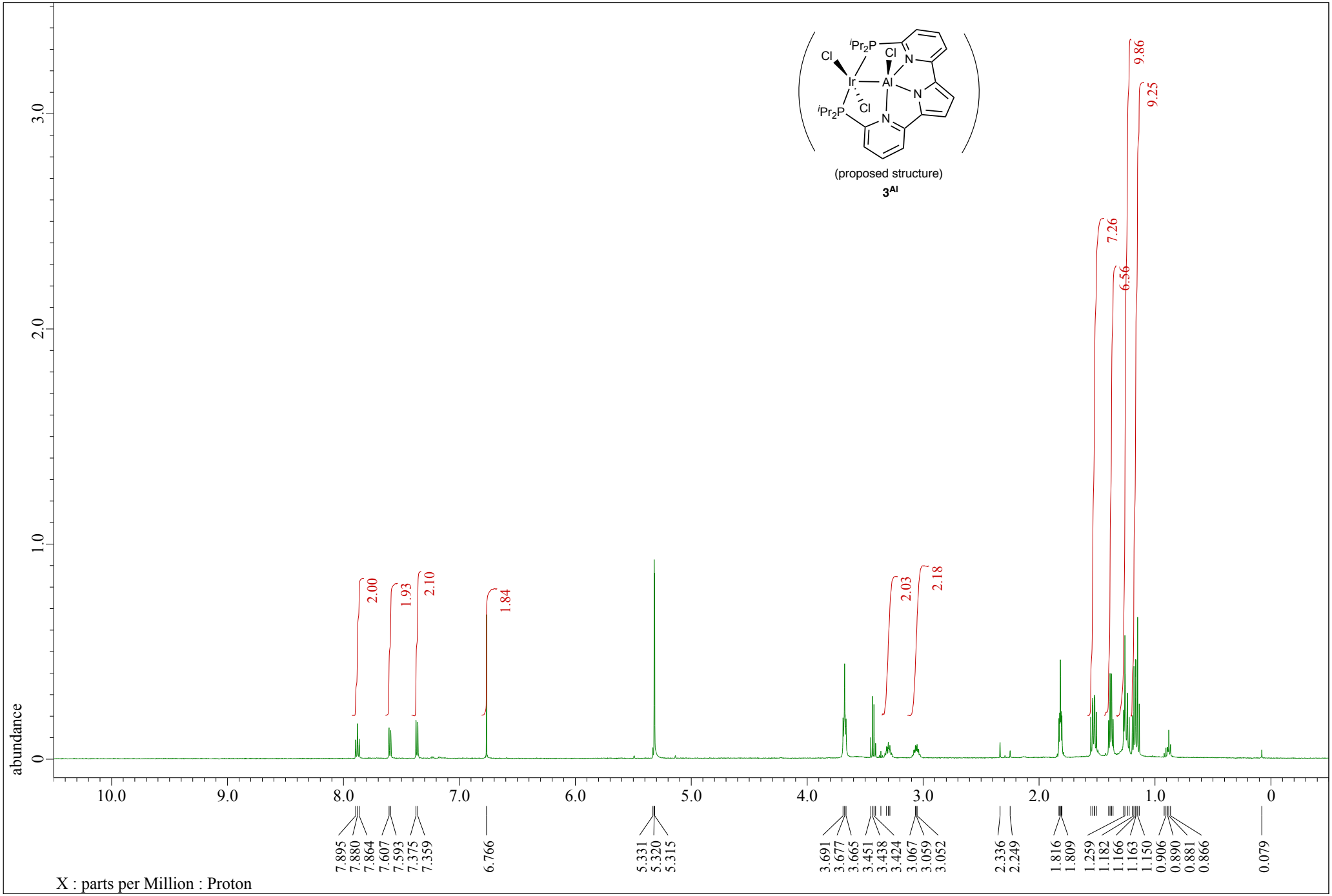


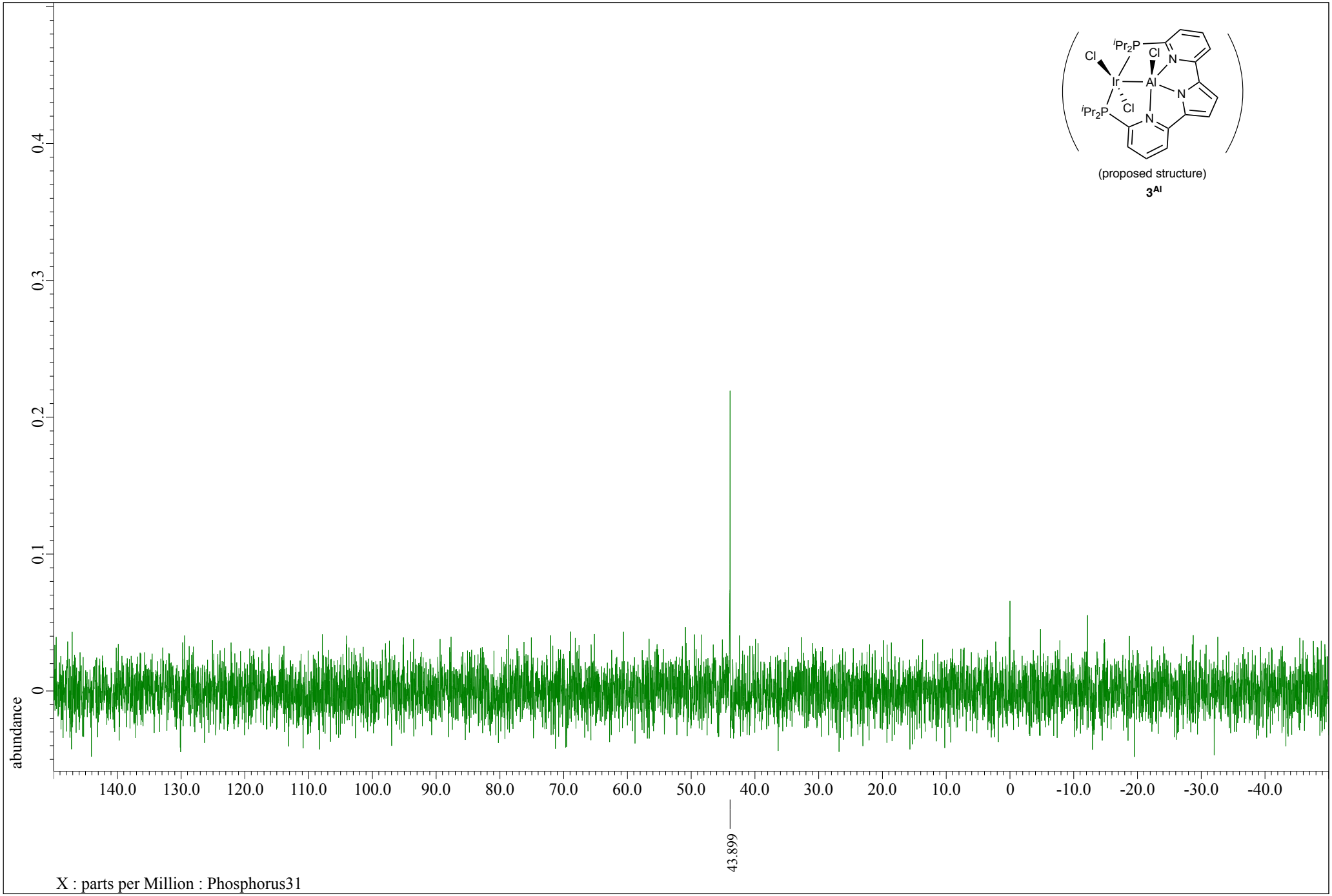

abundance
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-0.1
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
-40.0
50.063
X : parts per Million : Phosphorus31
Cl
iPr2P
Cl
N
Ir
Ga
N
iPr2P
Cl
N
3Ga

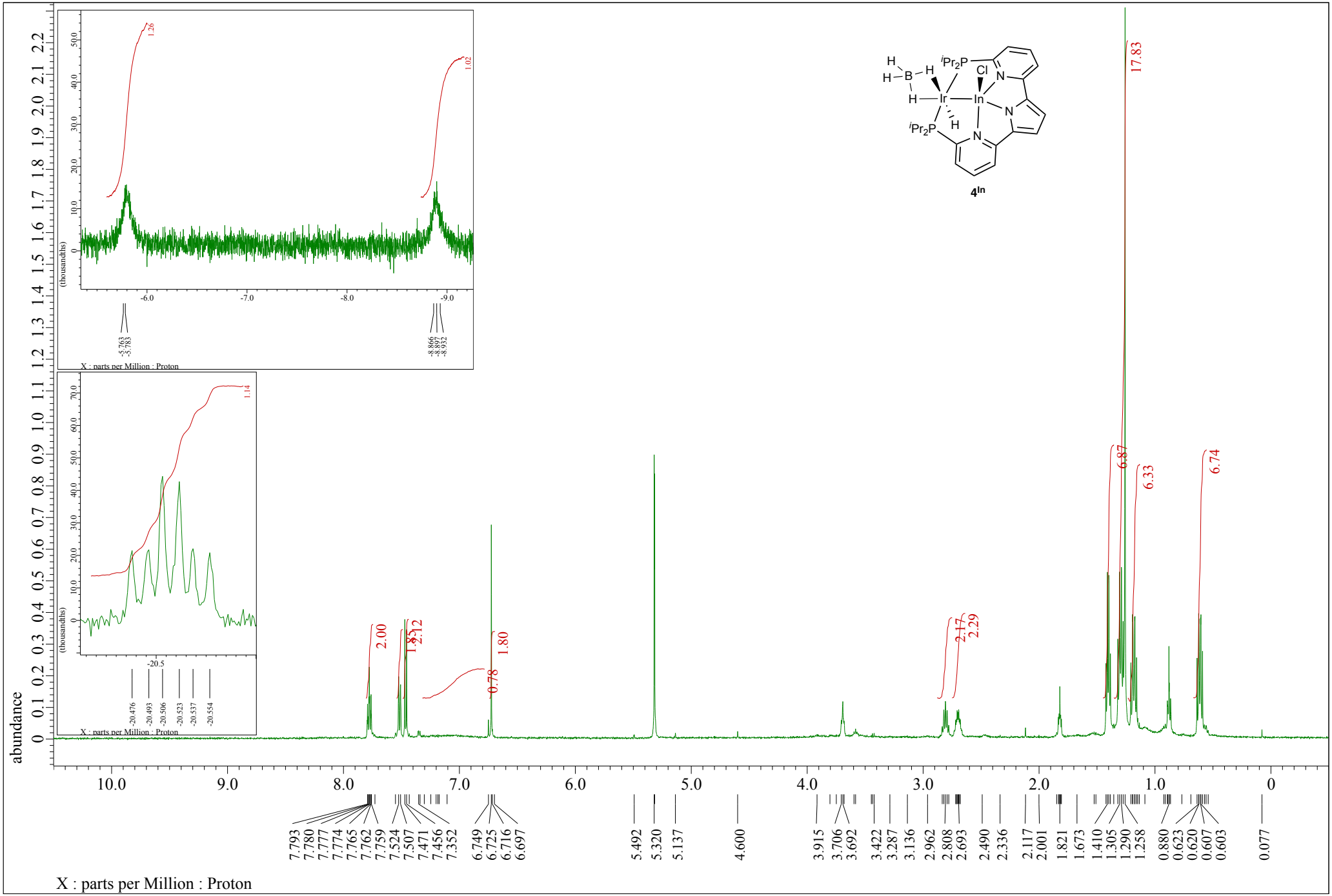

abundance
X : parts per Million : Proton
3In
7.23
5.40
5.13
3.31
2.00
1.89
1.94
1.87
1.84
1.80


(thousandths)
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
151.851
151.799
151.748
148.878
148.655
148.432
138.411
135.107
128.596
127.790
124.086
124.046
124.006
118.799
113.579
67.397
64.967
53.540
53.323
53.100
52.883
52.666
29.331
25.370
25.273
25.215
20.088
19.979
19.871
19.802
19.688
18.516
15.835
X : parts per Million : Carbon13
Cl
iPr2P
Cl
N
Ir
In
N
iPr2P
Cl
N
3In

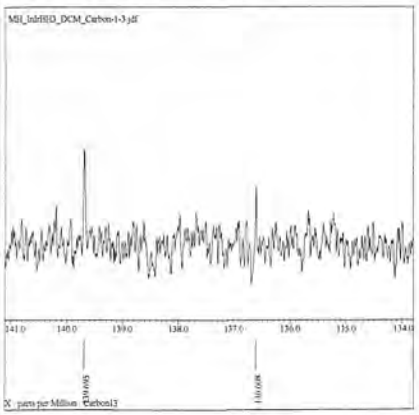

80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
(thousandths)
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
-40.0
58.101
X : parts per Million : Phosphorus31
Cl
iPr2P
Cl
Ir
In
N
N
iPr2P
Cl
N
3In

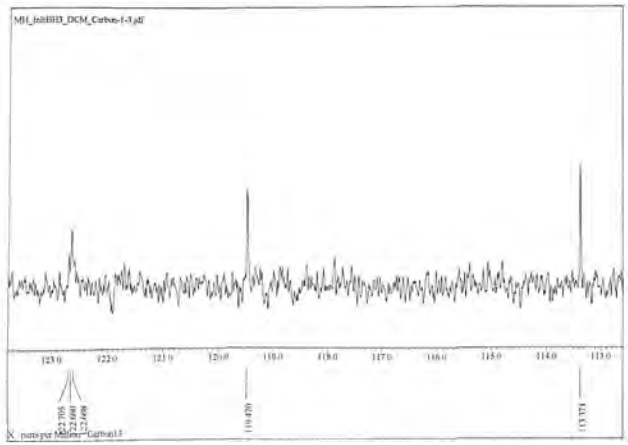

(proposed structure)
3Al
abundance
X : parts per Million : Proton
2.00
1.93
2.10
1.84
2.03
2.18
7.26
6.56
9.86
9.25
7.895
7.880
7.864
7.607
7.593
7.375
7.359
6.766
5.331
5.320
5.315
3.691
3.677
3.665
3.451
3.438
3.424
3.067
3.059
3.052
2.336
2.249
1.816
1.809
1.259
1.182
1.166
1.163
1.150
0.906
0.890
0.881
0.866
0.079

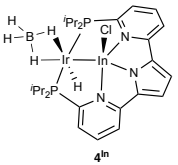

Cl
$^{i}Pr_2P$
Cl
N
Ir
Al
N
Cl
$^{i}Pr_2P$
N
(proposed structure)
3Al
0.4
0.3
0.2
0.1
0
abundance
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
-40.0
43.899
X : parts per Million : Phosphorus31


abundance
X : parts per Million : Proton
4in
17.83
6.87
6.33
6.74
2.00
2.12
1.80
0.78
2.17
2.29
1.26
1.02
1.14
7.793
7.780
7.777
7.774
7.765
7.762
7.759
7.524
7.507
7.471
7.456
7.352
6.749
6.725
6.716
6.697
5.492
5.320
5.137
4.600
3.915
3.706
3.692
3.422
3.287
3.136
2.962
2.808
2.693
2.490
2.336
2.117
2.001
1.821
1.673
1.410
1.305
1.290
1.258
0.880
0.623
0.620
0.607
0.603
0.077

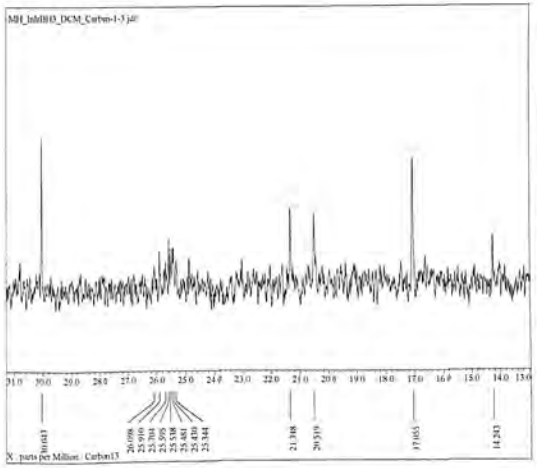
MH_InIrBH3_DCM_Carbon-1-3.jdf

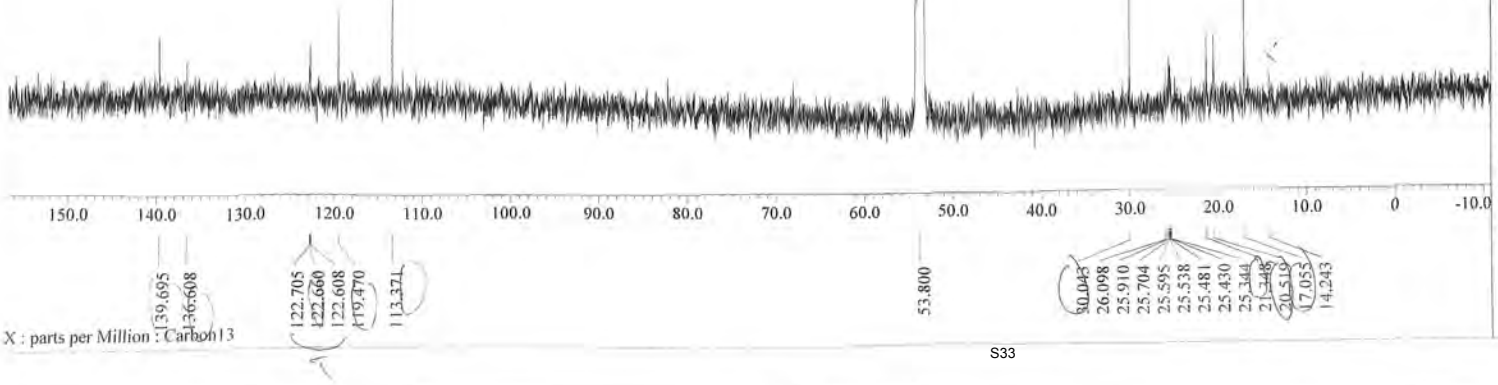






4[In]



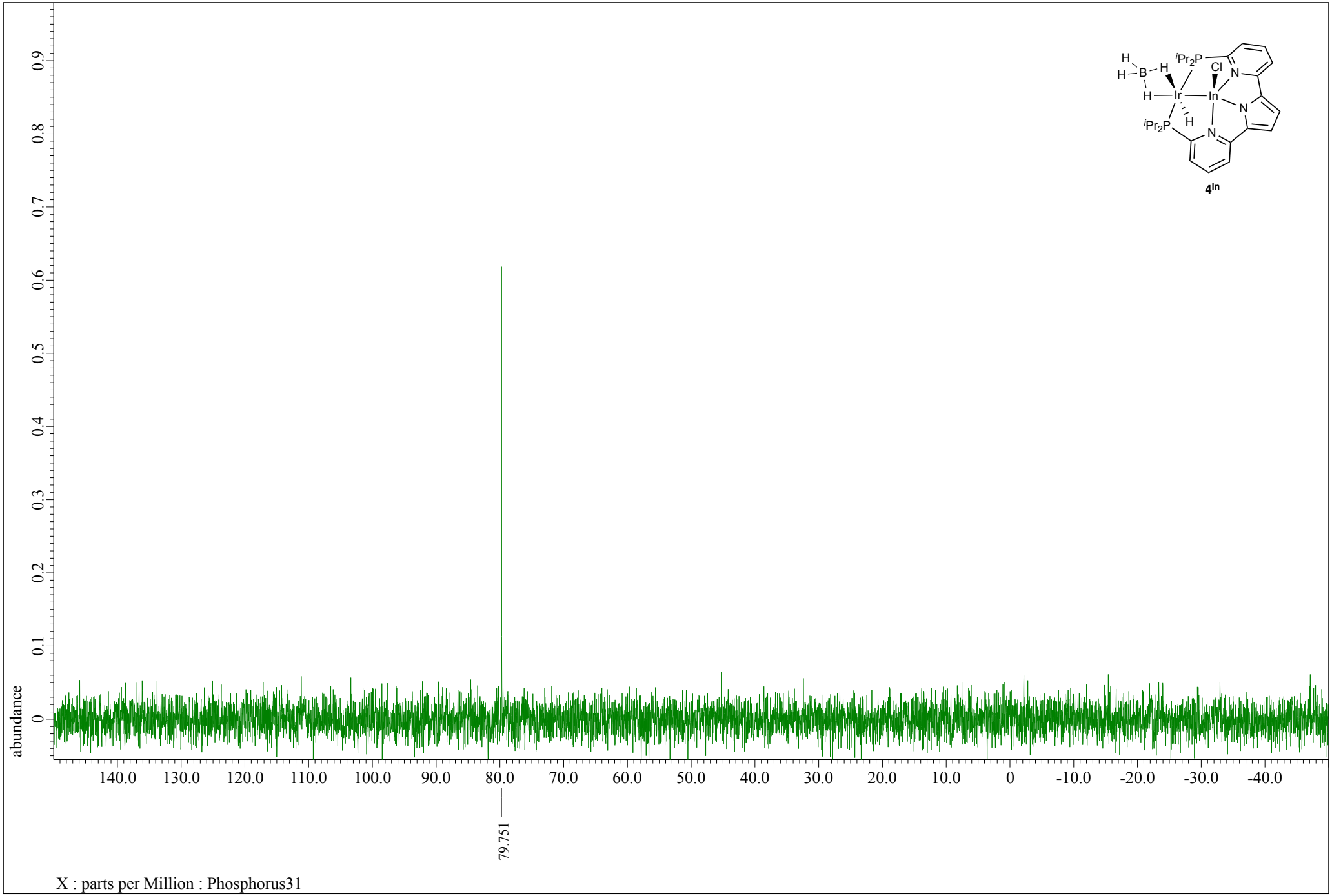


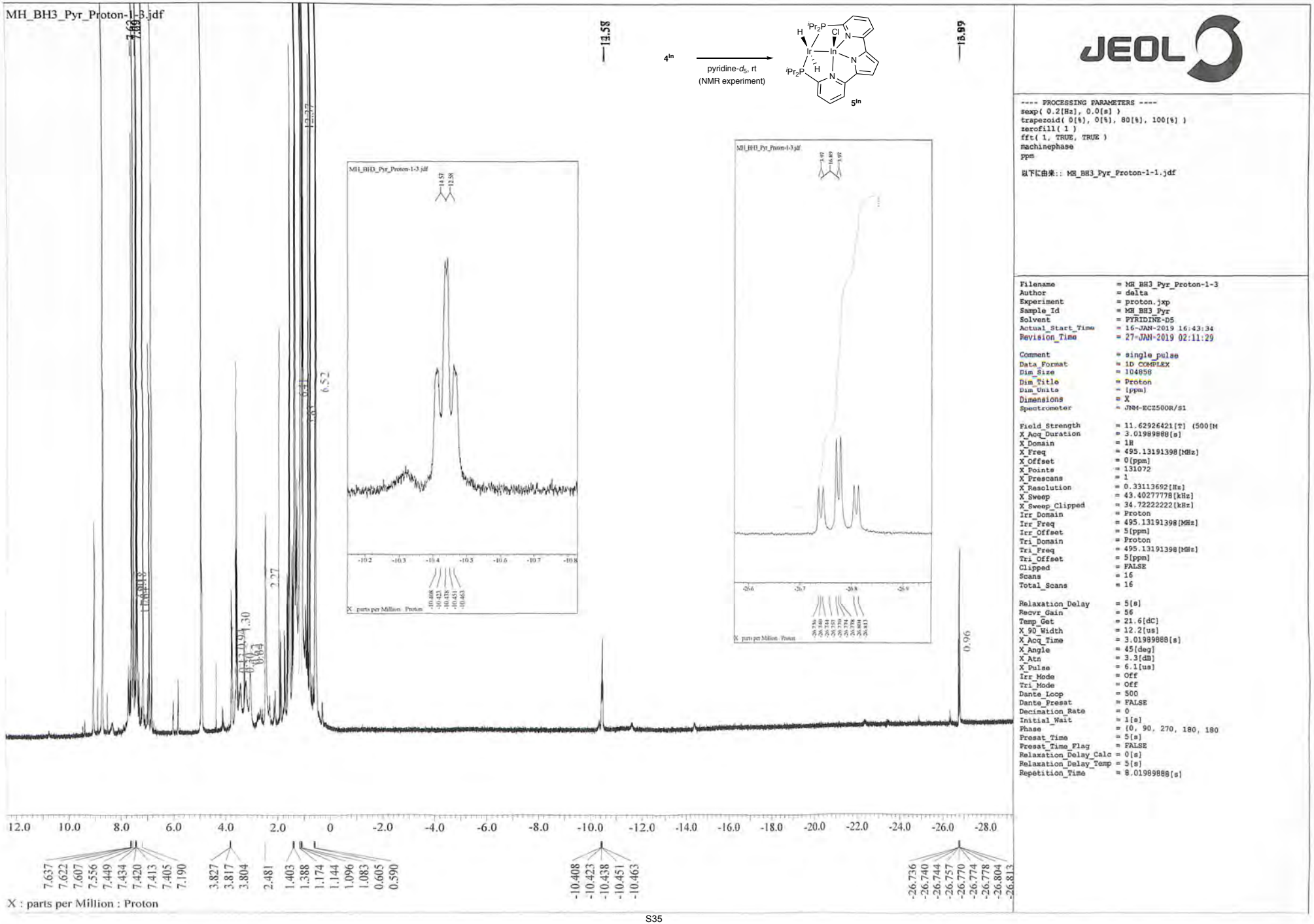
JEOL

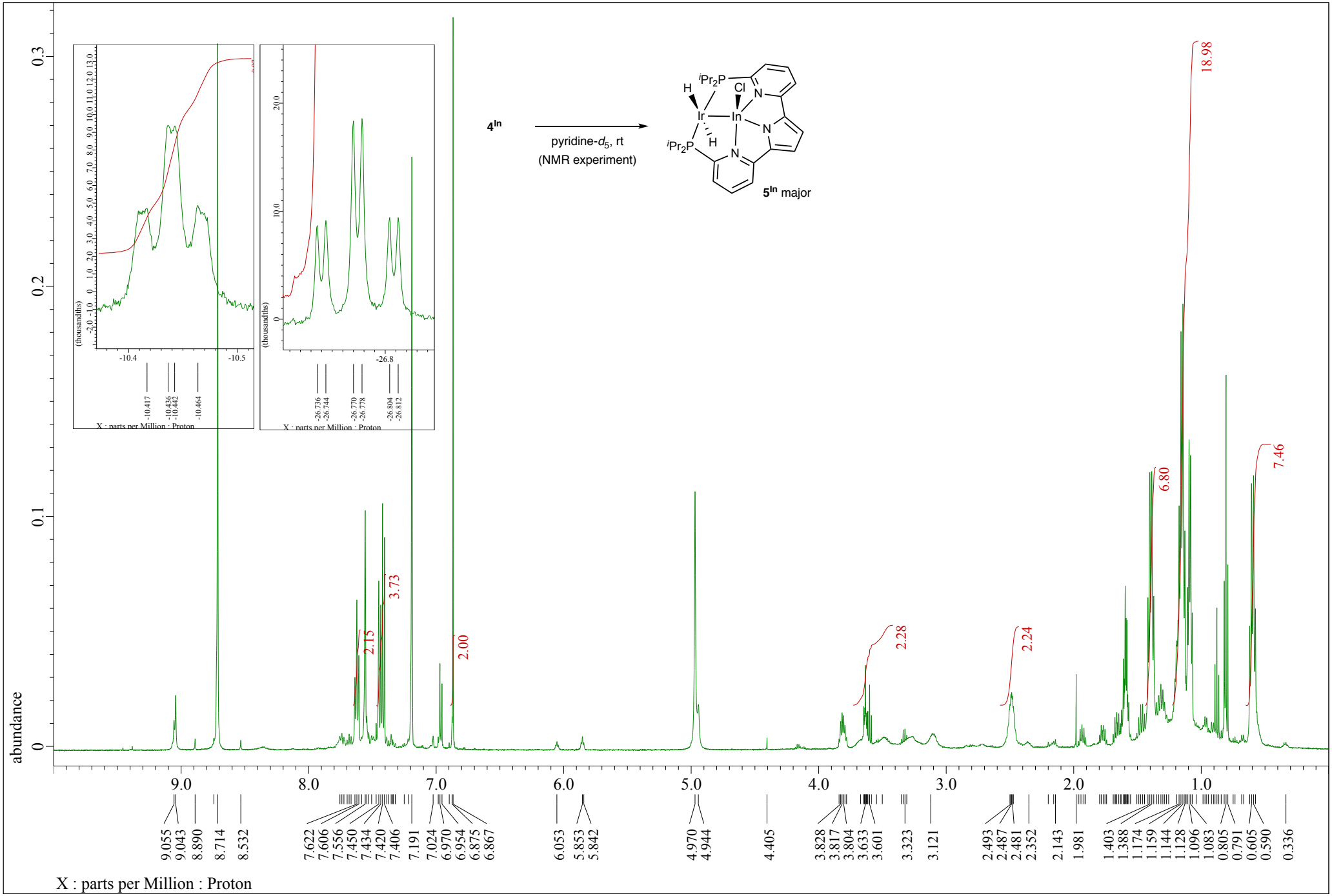
```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: MH_InIrBH3_DCM_Carbon-1-1.jdf
```

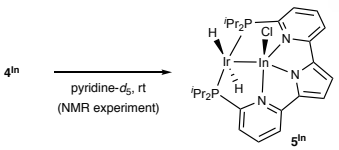
Filename	= MH_InIrBH3_DCM_Car
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= MH_InIrBH3_DCM
Solvent	= METHYLENE-CHLORIDE
Actual_Start_Time	= 10-JAN-2019 04:29:
Revision_Time	= 10-JAN-2019 15:04:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ500R/S1
Field_Strength	= 11.62926421[T] (50
X_Acq_Duration	= 1.40509184[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 0[ppm]
X_Points	= 131072
X_Prescans	= 4
X_Resolution	= 0.71169725[Hz]
X_Sweep	= 93.28358209[kHz]
X_Sweep_Clipped	= 74.62686567[kHz]
Irr_Domain	= Proton
Irr_Freq	= 495.1319139[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1000
Total_Scans	= 1000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 56
Temp_Get	= 22.4[dC]
X_90_Width	= 11.1[us]
X_Acq_Time	= 1.40509184[s]
X_Angle	= 30[deg]
X_Atn	= 10[dB]
X_Pulse	= 3.7[us]
Irr_Atn_Dec	= 21.6[dB]
Irr_Atn_Dec_Calc	= 21.6[dB]
Irr_Atn_Dec_Default_Calc	= 21.6[dB]
Irr_Atn_Noe	= 21.6[dB]
Irr_Dec_Bandwidth_Hz	= 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.07407703[ppm]
Irr_Dec_Freq	= 495.1319139[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 92[us]
Irr_Pwidth_Default	= 92[us]
Irr_Pwidth_Default_Calc	= 92[us]
Irr_Pwidth_Temp1	= 92[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Initial_Wait	= 1[s]
Noe_Time	= 2[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 2[s]
Repetition_Time	= 3.405091841[s]

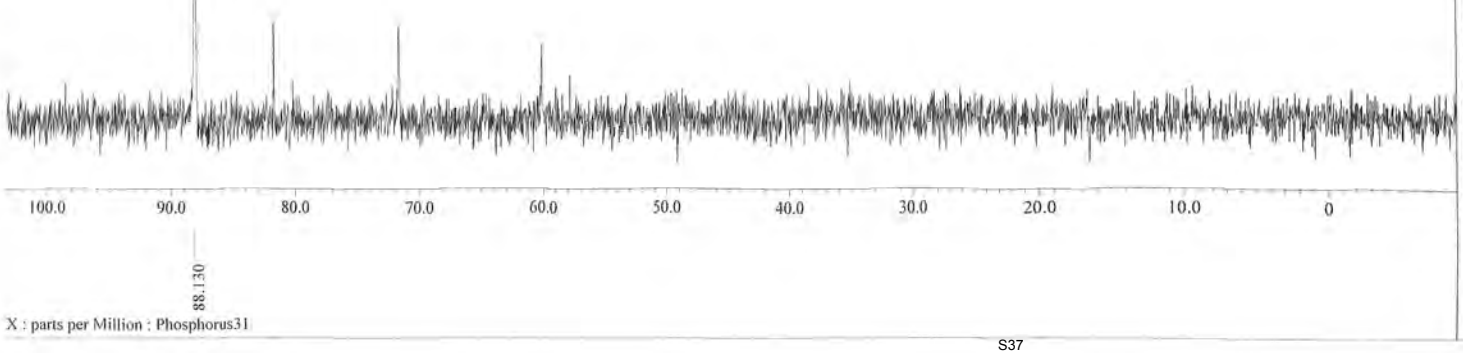

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
-40.0
79.751
X : parts per Million : Phosphorus31
H
H–B–H
H–Ir–In
iPr2P
iPr2P
H
Cl
N
N
N
4In


MH_BH3_Pyr_Proton-1-3.jdf
4In
pyridine-d5, rt
(NMR experiment)
5In
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: MH_BH3_Pyr_Proton-1-1.jdf
Filename = MH_BH3_Pyr_Proton-1-3
Author = delta
Experiment = proton.jxp
Sample_Id = MH_BH3_Pyr
Solvent = PYRIDINE-D5
Actual_Start_Time = 16-JAN-2019 16:43:34
Revision_Time = 27-JAN-2019 02:11:29
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 104858
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (500[M
X_Acq_Duration = 3.01989888[s]
X_Domain = 1H
X_Freq = 495.13191398[MHz]
X_Offset = 0[ppm]
X_Points = 131072
X_Prescans = 1
X_Resolution = 0.33113692[Hz]
X_Sweep = 43.40277778[kHz]
X_Sweep_Clipped = 34.72222222[kHz]
Irr_Domain = Proton
Irr_Freq = 495.13191398[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 495.13191398[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 21.6[dC]
X_90_Width = 12.2[us]
X_Acq_Time = 3.01989888[s]
X_Angle = 45[deg]
X_Atn = 3.3[dB]
X_Pulse = 6.1[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 8.01989888[s]


4In
pyridine-d5, rt
(NMR experiment)
5In major
iPr2P
H
Cl
Ir
In
N
H
iPr2P
abundance
X : parts per Million : Proton
18.98
7.46
6.80
2.28
2.24
2.15
3.73
2.00
9.055
9.043
8.890
8.714
8.532
7.622
7.606
7.556
7.450
7.434
7.420
7.406
7.191
7.024
6.970
6.954
6.875
6.867
6.053
5.853
5.842
4.970
4.944
4.405
3.828
3.817
3.804
3.633
3.601
3.323
3.121
2.493
2.487
2.481
2.352
2.143
1.981
1.403
1.388
1.174
1.159
1.144
1.128
1.096
1.083
0.805
0.791
0.605
0.590
0.336

MH_BH3_Pyr_31P_1Hdec-1-4.jdf





X : parts per Million : Phosphorus31

```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 53.54325, 0, 50[%] )

以下に由来:: MH_BH3_Pyr_31P_1Hdec-1-1.jdf
```

Filename	= MH_BH3_Pyr_31P_1Hd
Author	= delta
Experiment	= single_pulse_dec.j
Sample_Id	= MH_BH3_Pyr
Solvent	= PYRIDINE-D5
Actual_Start_Time	= 16-JAN-2019 14:01:
Revision_Time	= 27-JAN-2019 02:12:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Phosphorus31
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ500R/S1
Field_Strength	= 11.62926421[T] (50
X_Acq_Duration	= 0.18874368[s]
X_Domain	= 31P
X_Freq	= 200.43293989[MHz]
X_Offset	= 0[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 5.29819065[Hz]
X_Sweep	= 173.61111111[kHz]
X_Sweep_Clipped	= 138.88888889[kHz]
Irr_Domain	= Proton
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 21.7[dC]
X_90_Width	= 34.2[us]
X_Acq_Time	= 0.18874368[s]
X_Angle	= 30[deg]
X_Atn	= 9.4[dB]
X_Pulse	= 11.4[us]
Irr_Atn_Dec	= 21.6[dB]
Irr_Atn_Dec_Calc	= 21.6[dB]
Irr_Atn_Dec_Default_Calc	= 21.6[dB]
Irr_Atn_Noe	= 21.6[dB]
Irr_Dec_Bandwidth_Hz	= 5.97026087[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.07407703[ppm]
Irr_Dec_Freq	= 495.13191398[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 92[us]
Irr_Pwidth_Default	= 92[us]
Irr_Pwidth_Default_Calc	= 92[us]
Irr_Pwidth_Templ	= 92[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.18874368[s]