Electronic Supplementary Material (ESI) for Dalton Transactions.

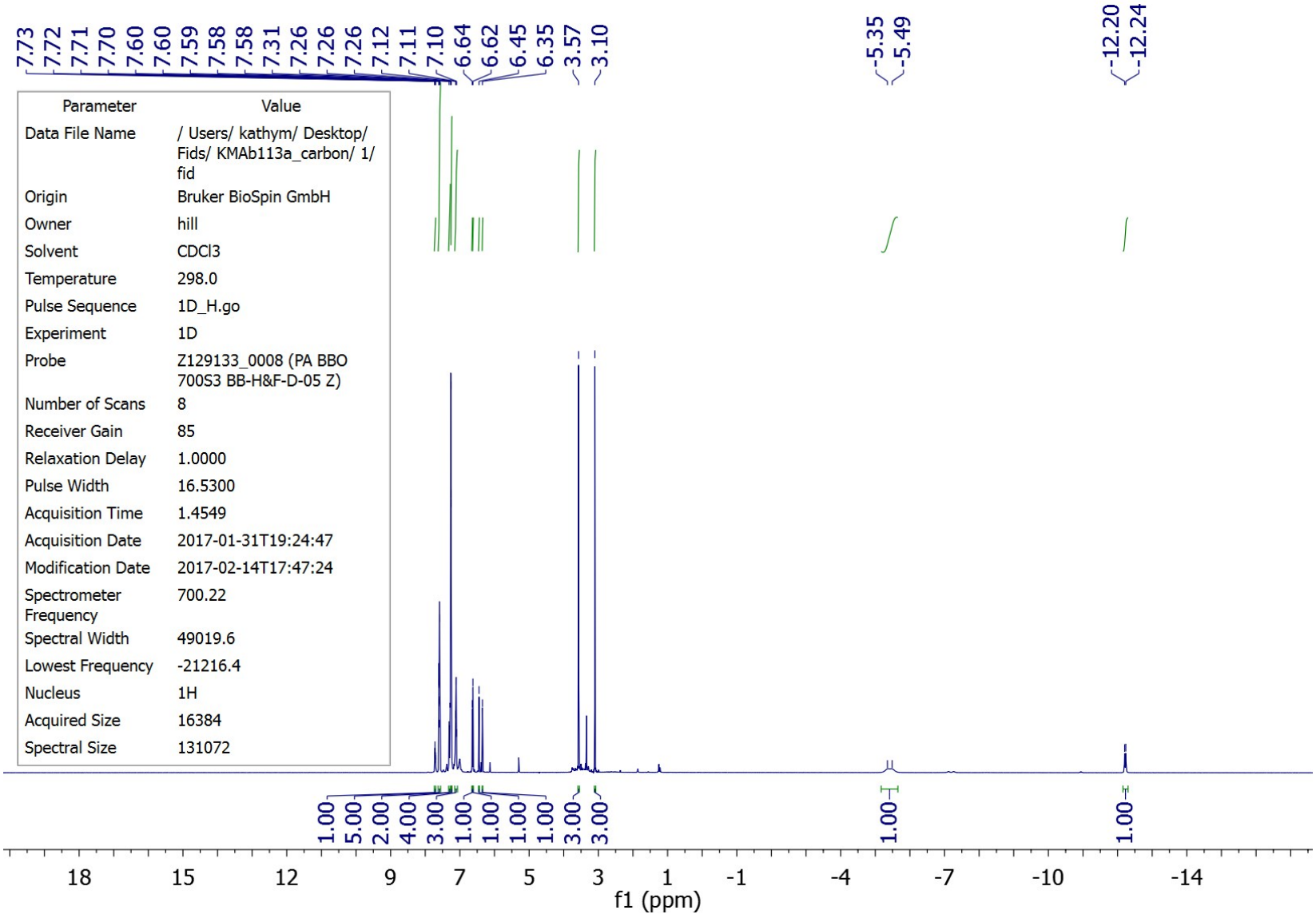
Characterisation of [RuH(CO)(PPh_3){$H_2B(mt)_2$}] (3a)

Commentary

The infrared spectrum reveals absorptions at ν = 1936 cm^{-1}, 2110 cm^{-1}, 2201 cm^{-1} and 2399 cm^{-1} in CH_2Cl_2 attributable to the carbonyl, Ru–H, B–H–Ru and terminal B–H groups, respectively. Consistent with C_1 symmetry, the ^{1}H NMR spectrum shows two distinct NCH_3 singlets (δ_H = 3.10, 3.57) and four doublets for the methimazolyl groups (δ_H = 6.35, 6.45, 6.62, 6.64; $^3J_{HH}$ = 2.0 Hz). The retention of a ruthenium hydride ligand in the resulting complex is indicated by the appearance in the ^{1}H NMR spectrum of a high frequency resonance (δ_H = –12.22) the doublet multiplicity of which confirms a monophosphine complex with hydride and phosphine ligands mutually *cis*-coordinated ($^2J_{PH}$ = 23.1 Hz).

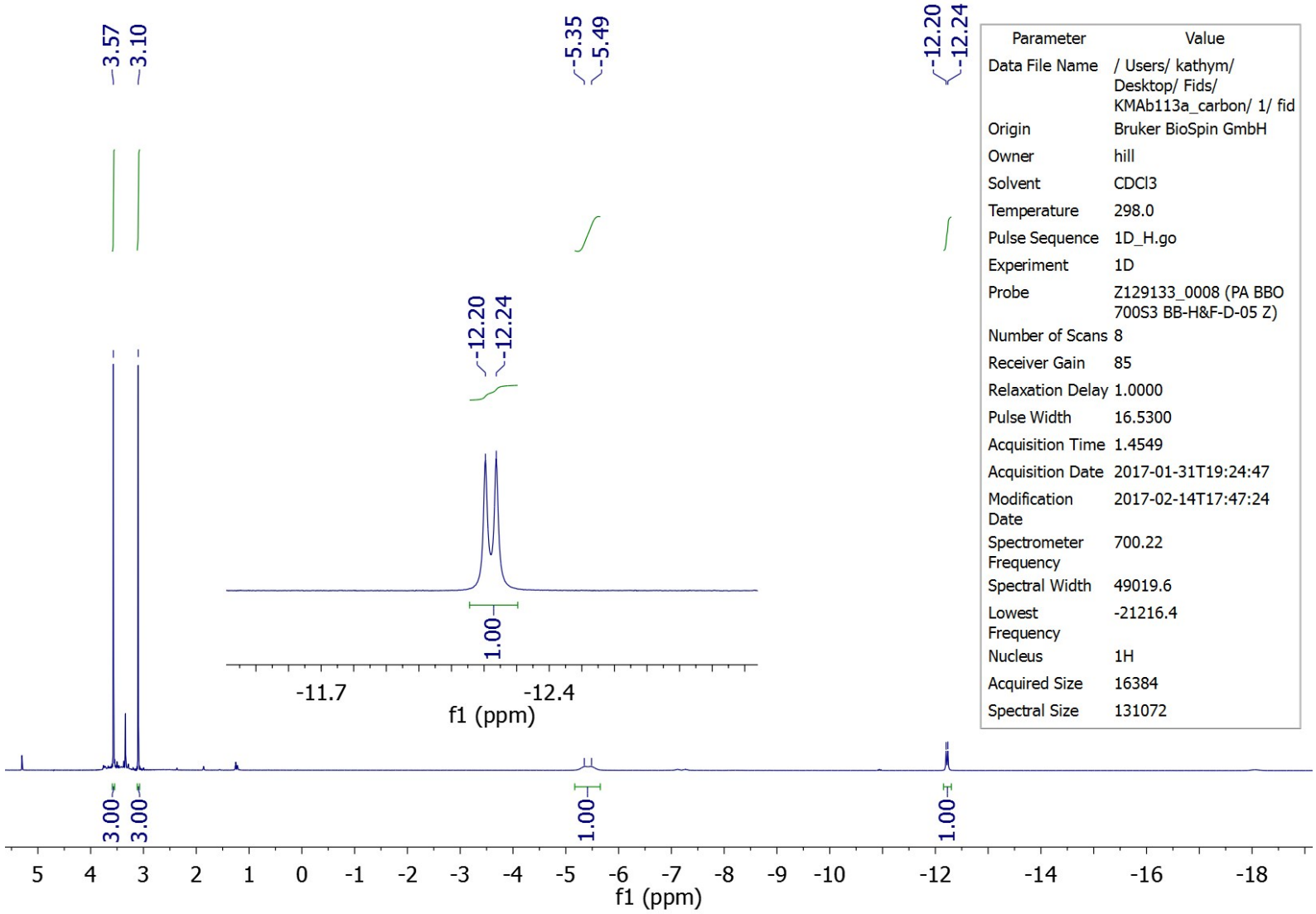
The 3c-2e Ru–H–B interaction gives rise to a resonance at δ_H = –5.45, the broadness of which is due to the coupling to the quadrupolar boron nuclei (^{10}B and ^{11}B). The terminal B–H resonance was not located, however there is no apparent site exchange between the terminal B–H and B–H–Ru groups over the temperature range 193 – 305 K, although at 305 K the ^{11}B quadrupole was sufficiently thermally decoupled that a $^1J_{BH}$ coupling of *ca* 85 Hz could be discerned. The $^{31}P\{^1H\}$ NMR spectrum shows a singlet at δ 56.7 ppm and the gross molecular composition was further confirmed by an APCI mass spectrum which included a molecular ion in addition to fragmentations attributable to loss of CO and PPh_3.

^{1}H NMR Spectrum of 3a



Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb113a_carbon/ 1/ fid
Origin	Bruker BioSpin GmbH
Owner	hill
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	1D_H.go
Experiment	1D
Probe	Z129133_0008 (PA BBO 700S3 BB-H&F-D-05 Z)
Number of Scans	8
Receiver Gain	85
Relaxation Delay	1.0000
Pulse Width	16.5300
Acquisition Time	1.4549
Acquisition Date	2017-01-31T19:24:47
Modification Date	2017-02-14T17:47:24
Spectrometer Frequency	700.22
Spectral Width	49019.6
Lowest Frequency	-21216.4
Nucleus	1H
Acquired Size	16384
Spectral Size	131072

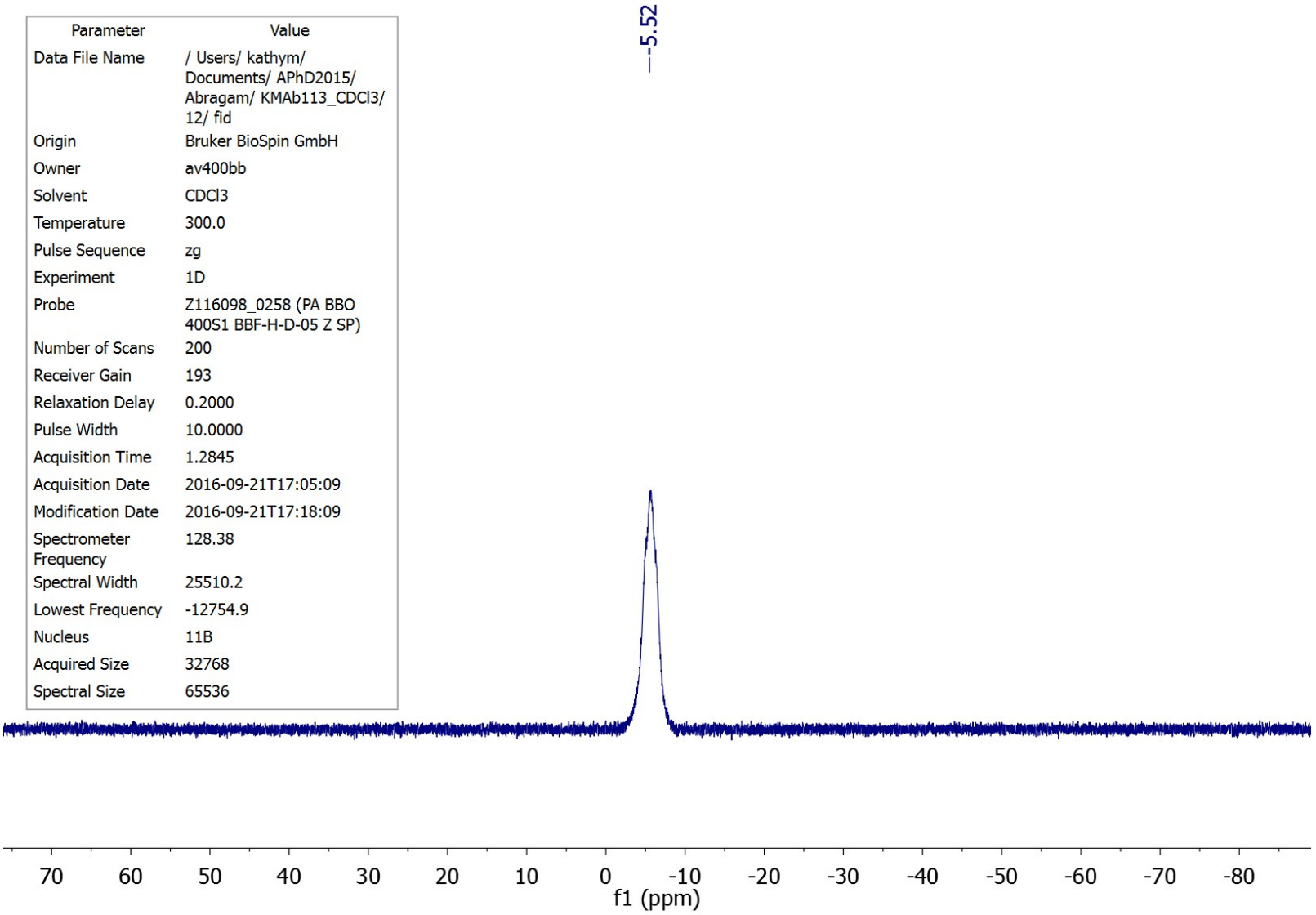
^{1}H NMR Spectrum of 3a (High Field Expansion)



Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb113a_carbon/ 1/ fid
Origin	Bruker BioSpin GmbH
Owner	hill
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	1D_H.go
Experiment	1D
Probe	Z129133_0008 (PA BBO 700S3 BB-H&F-D-05 Z)
Number of Scans	8
Receiver Gain	85
Relaxation Delay	1.0000
Pulse Width	16.5300
Acquisition Time	1.4549
Acquisition Date	2017-01-31T19:24:47
Modification Date	2017-02-14T17:47:24
Spectrometer Frequency	700.22
Spectral Width	49019.6
Lowest Frequency	-21216.4
Nucleus	1H
Acquired Size	16384
Spectral Size	131072

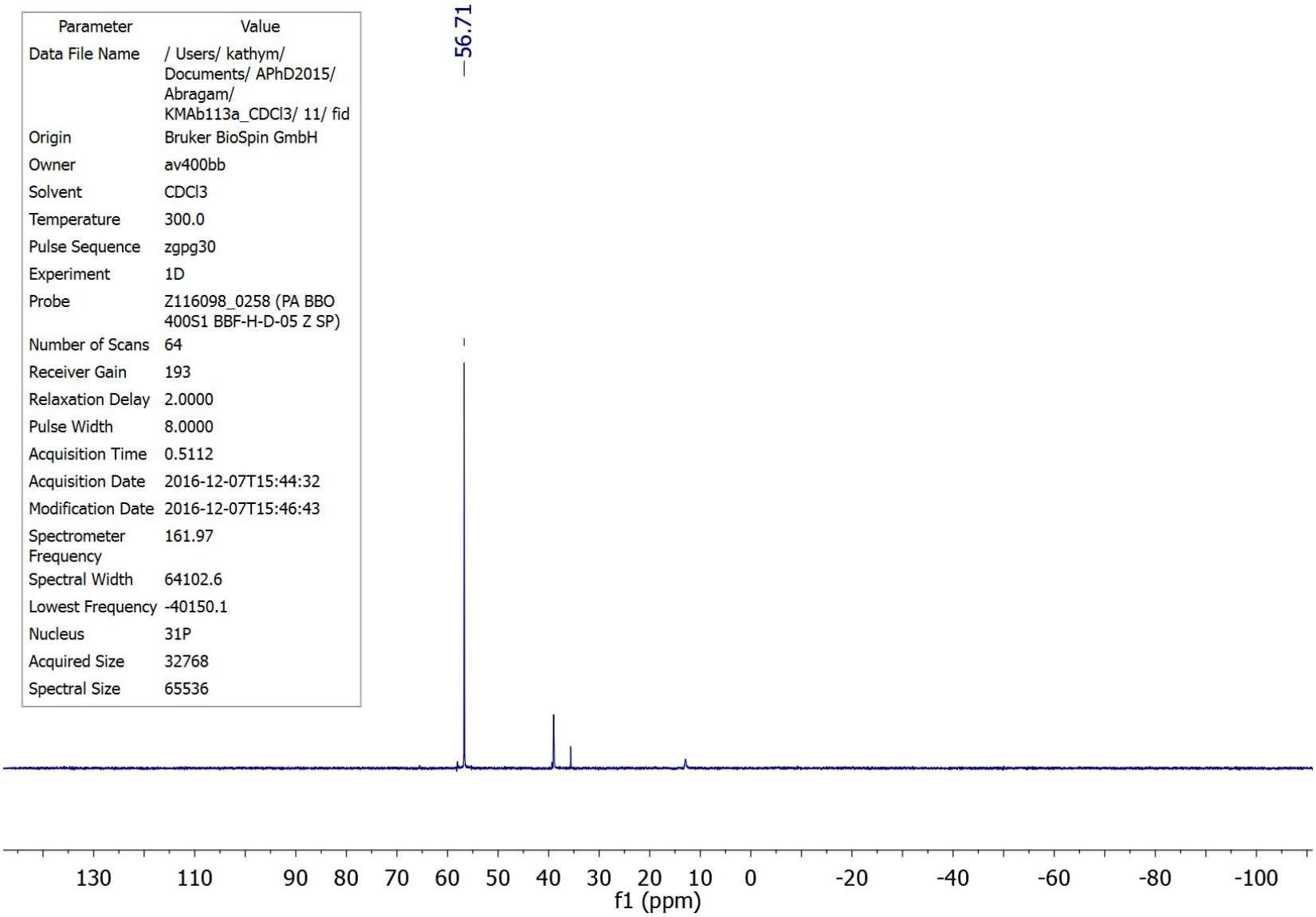
$^{11}B\{^{1}H\}$ NMR Spectrum of 3a

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb113_CDCl3/ 12/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zg
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2016-09-21T17:05:09
Modification Date	2016-09-21T17:18:09
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12754.9
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

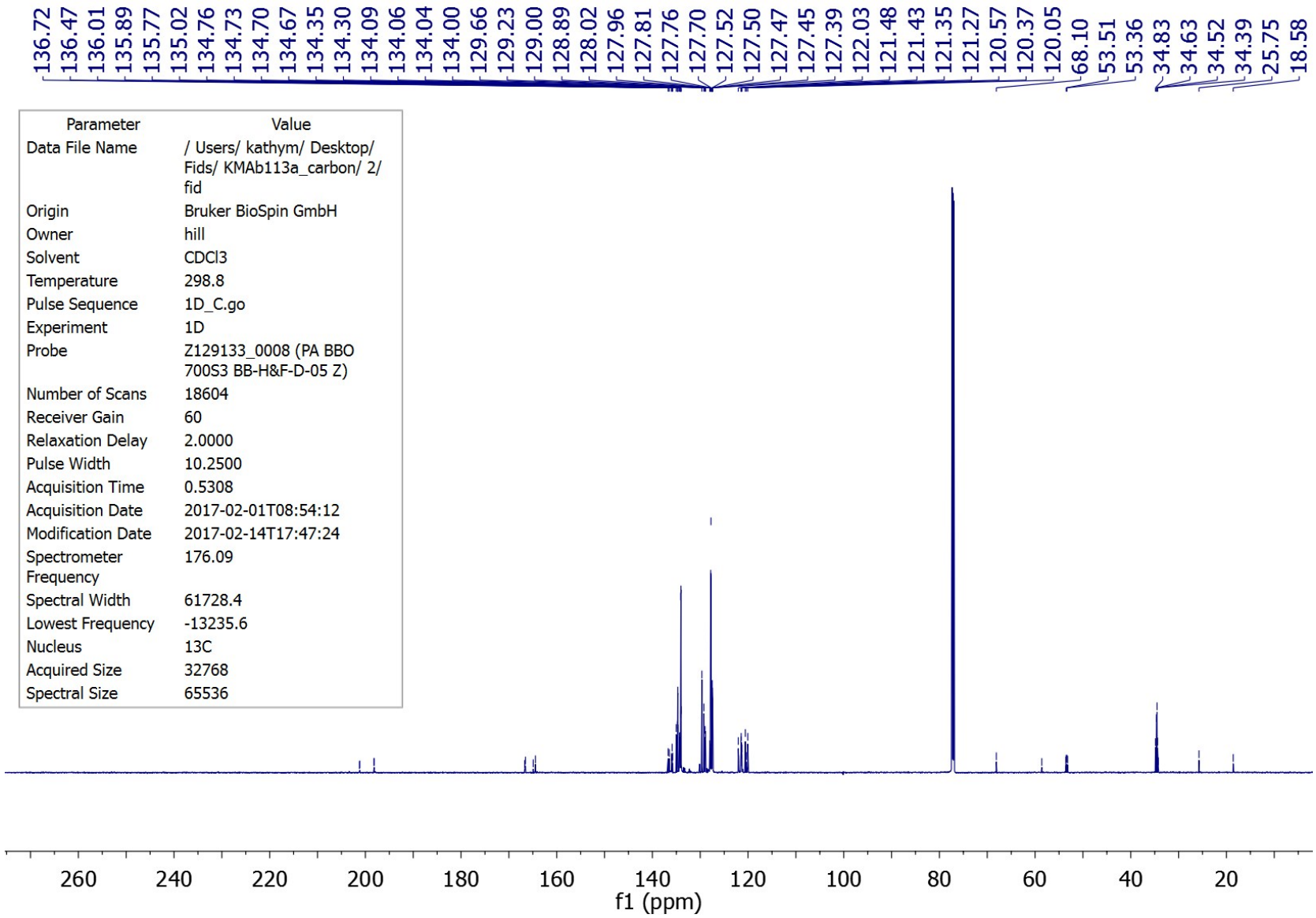


$^{31}P\{^{1}H\}$ NMR Spectrum of 3a

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb113a_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	64
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2016-12-07T15:44:32
Modification Date	2016-12-07T15:46:43
Spectrometer Frequency	161.97
Spectral Width	64102.6
Lowest Frequency	-40150.1
Nucleus	31P
Acquired Size	32768
Spectral Size	65536



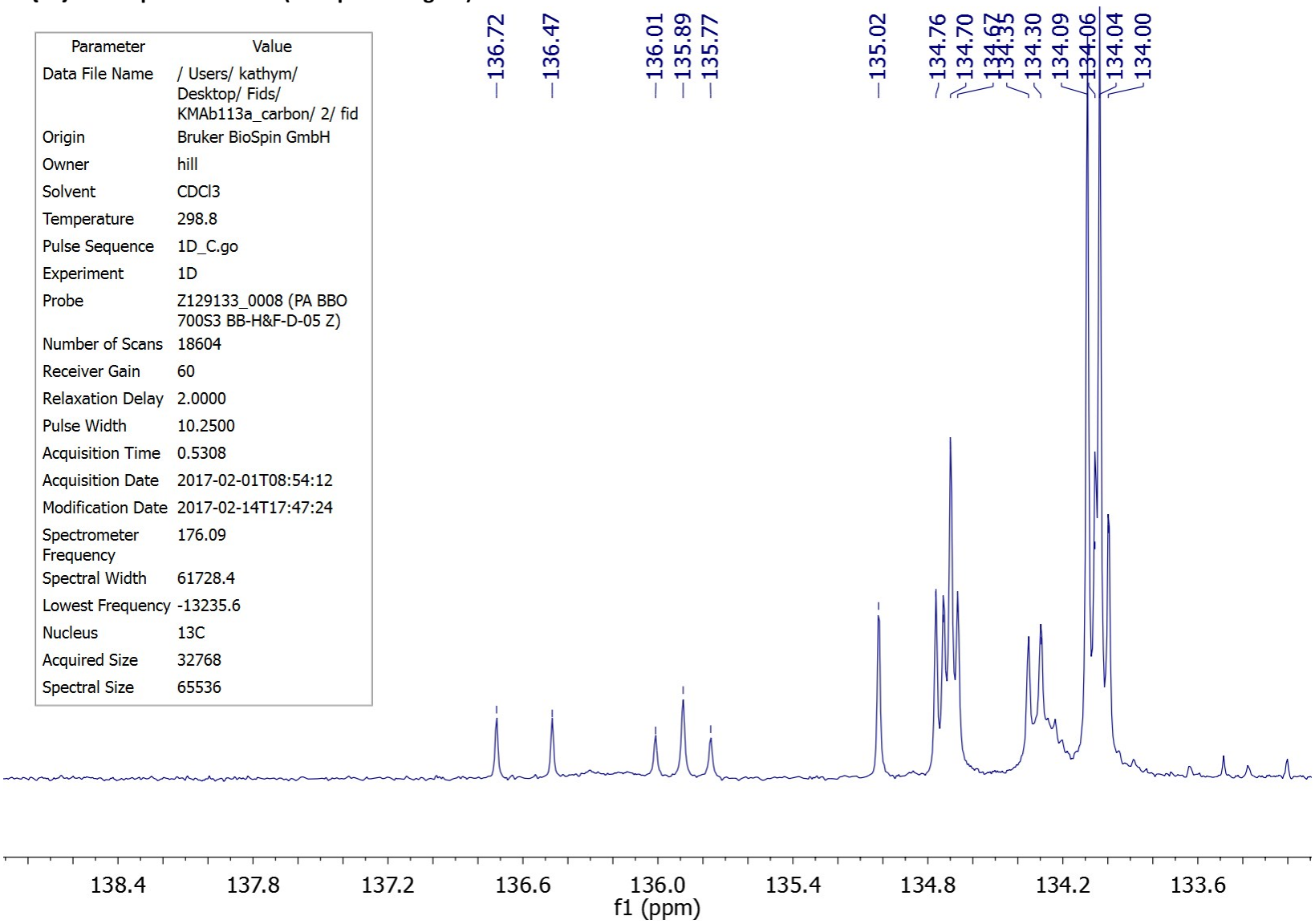
$^{13}C\{^{1}H\}$ NMR Spectrum of 3a



Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb113a_carbon/ 2/ fid
Origin	Bruker BioSpin GmbH
Owner	hill
Solvent	CDCl3
Temperature	298.8
Pulse Sequence	1D_C.go
Experiment	1D
Probe	Z129133_0008 (PA BBO 700S3 BB-H&F-D-05 Z)
Number of Scans	18604
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	10.2500
Acquisition Time	0.5308
Acquisition Date	2017-02-01T08:54:12
Modification Date	2017-02-14T17:47:24
Spectrometer Frequency	176.09
Spectral Width	61728.4
Lowest Frequency	-13235.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

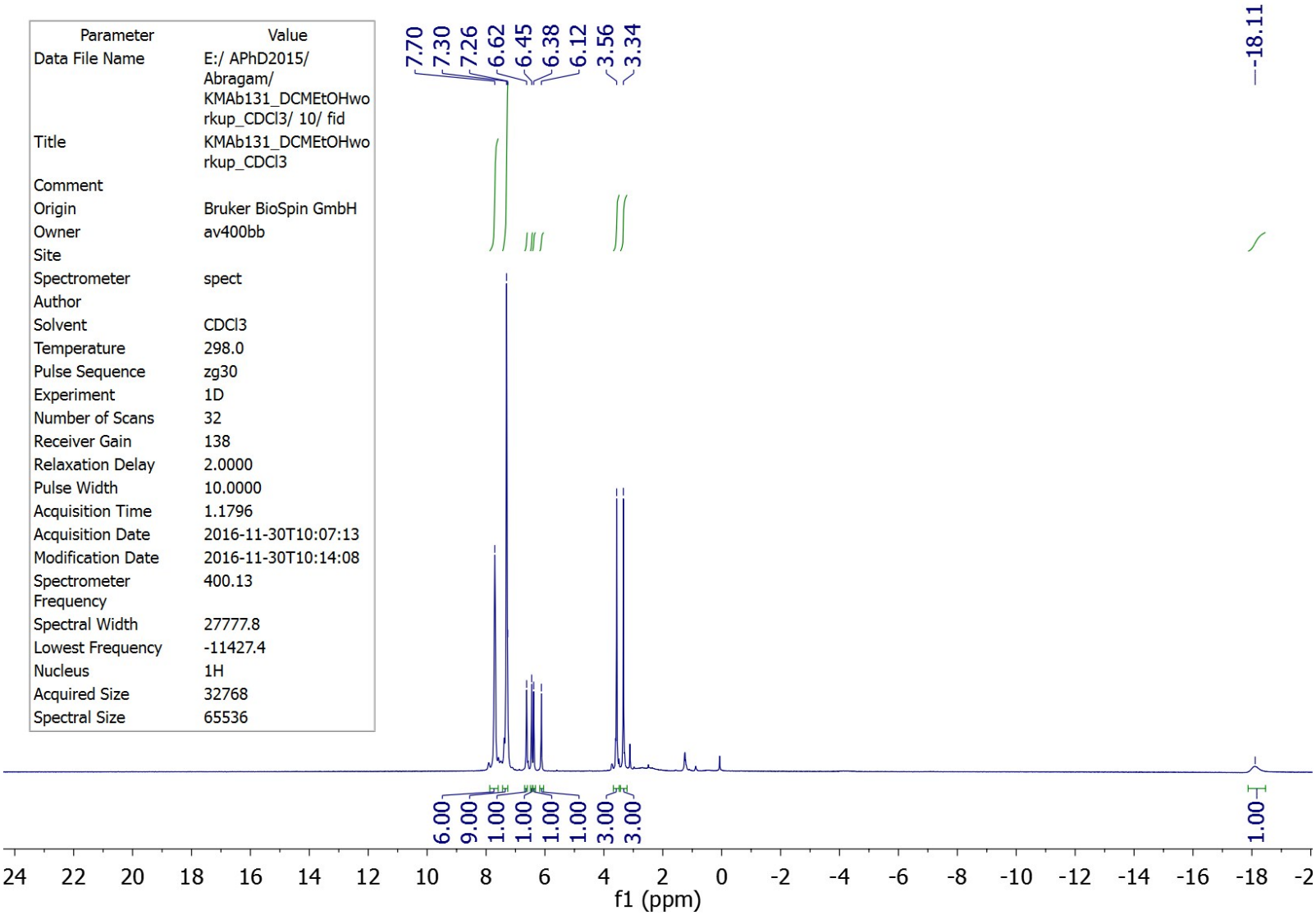
$^{13}C\{^{1}H\}$ NMR Spectrum of 3a (Phosphine Region)

Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb113a_carbon/ 2/ fid
Origin	Bruker BioSpin GmbH
Owner	hill
Solvent	CDCl3
Temperature	298.8
Pulse Sequence	1D_C.go
Experiment	1D
Probe	Z129133_0008 (PA BBO 700S3 BB-H&F-D-05 Z)
Number of Scans	18604
Receiver Gain	60
Relaxation Delay	2.0000
Pulse Width	10.2500
Acquisition Time	0.5308
Acquisition Date	2017-02-01T08:54:12
Modification Date	2017-02-14T17:47:24
Spectrometer Frequency	176.09
Spectral Width	61728.4
Lowest Frequency	-13235.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



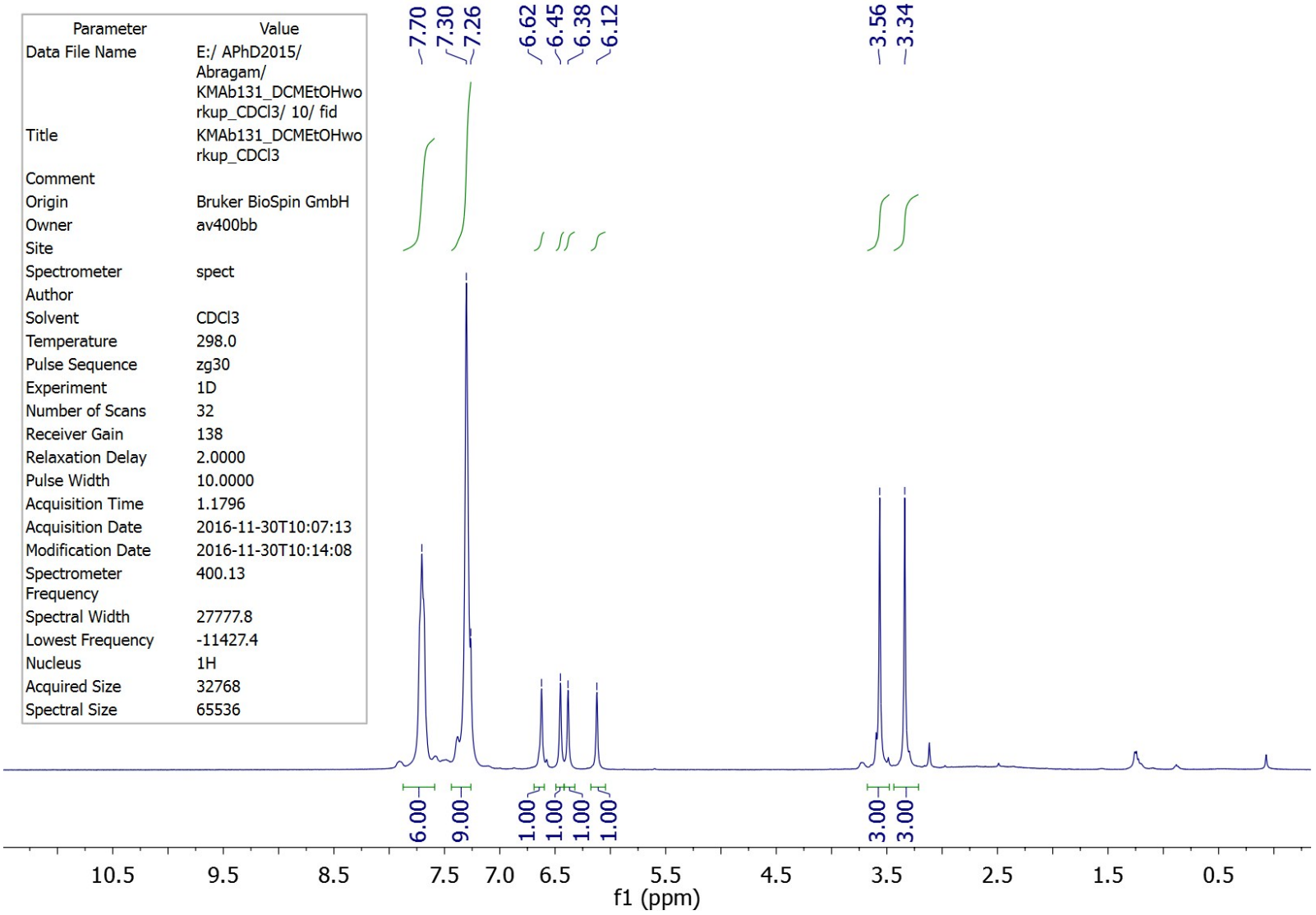
Characterisation of [RuCl(CO)(PPh_3){$H_2B(mt)_2$}] (3b)

^{1}H NMR Spectrum of 3b



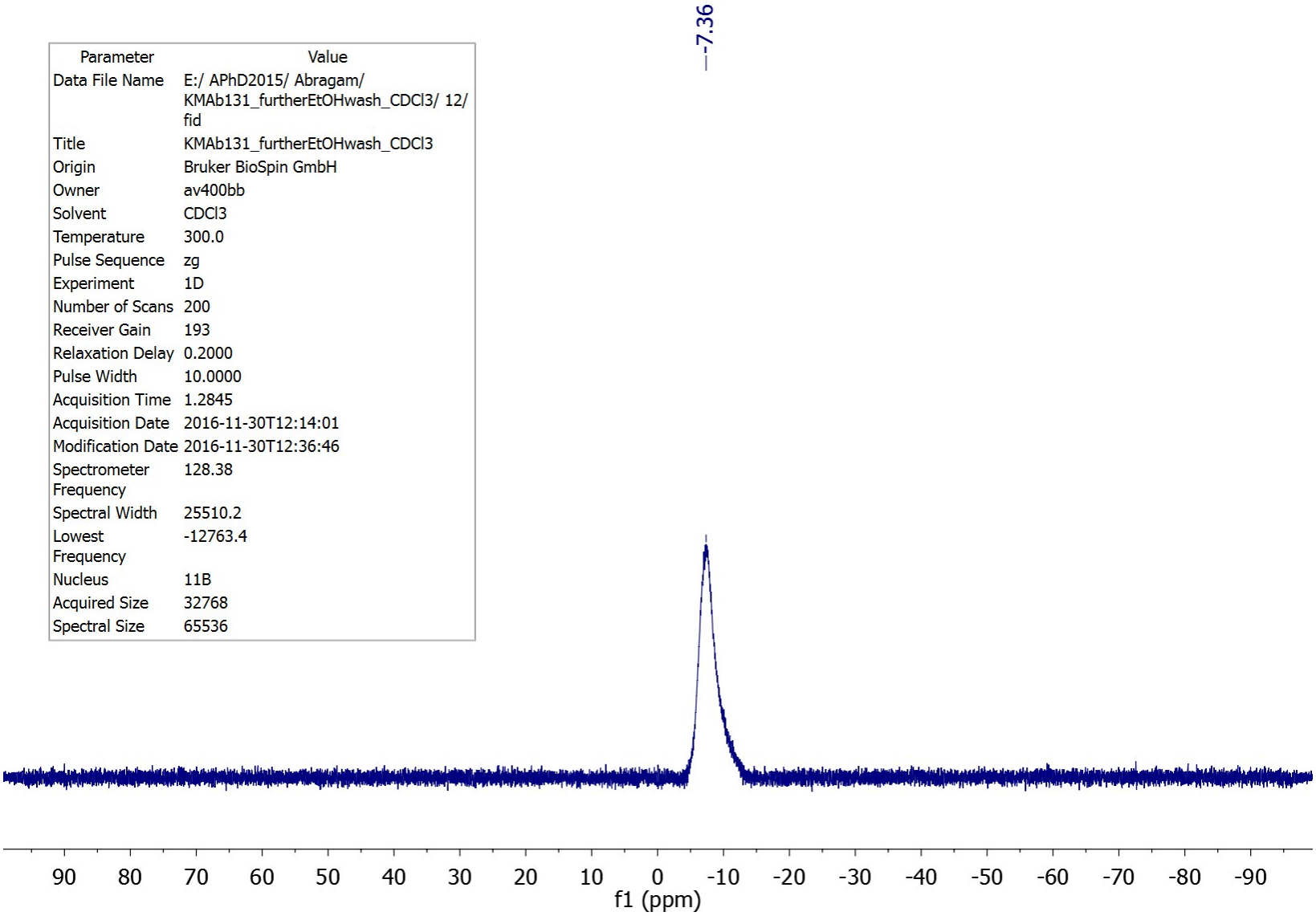
^{1}H NMR Spectrum of 3b (Expansion)

Parameter	Value
Data File Name	E:/ APhD2015/ Abragam/ KMAb131_DCMEtOHworkup_CDCl3/ 10/ fid
Title	KMAb131_DCMEtOHworkup_CDCl3
Comment	
Origin	Bruker BioSpin GmbH
Owner	av400bb
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	138
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1796
Acquisition Date	2016-11-30T10:07:13
Modification Date	2016-11-30T10:14:08
Spectrometer Frequency	400.13
Spectral Width	27777.8
Lowest Frequency	-11427.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



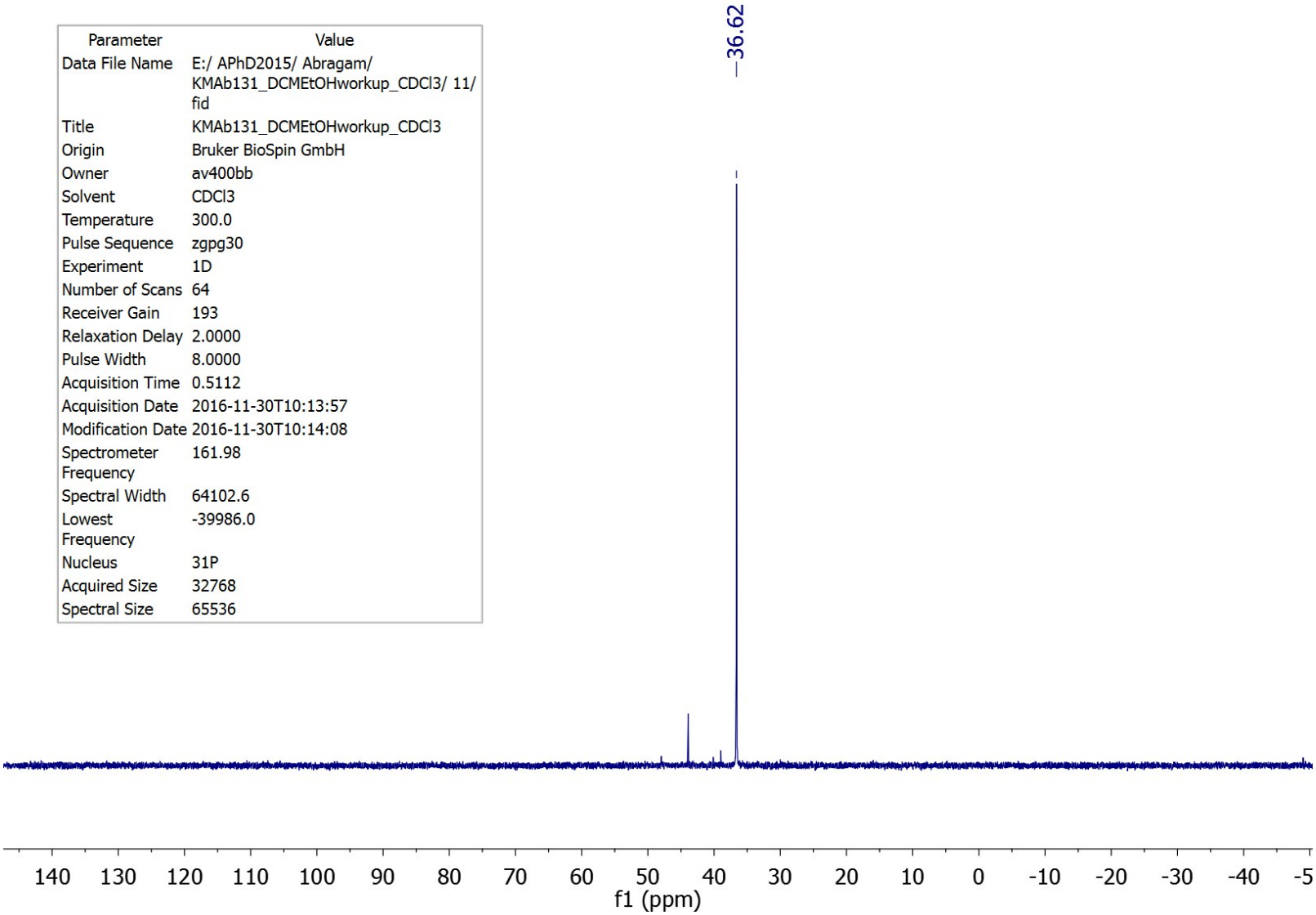
$^{11}B\{^{1}H\}$ NMR Spectrum of 3b

Parameter	Value
Data File Name	E:/ APhD2015/ Abragam/ KMAb131_furtherEtOHwash_CDCl3/ 12/ fid
Title	KMAb131_furtherEtOHwash_CDCl3
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zg
Experiment	1D
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2016-11-30T12:14:01
Modification Date	2016-11-30T12:36:46
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12763.4
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

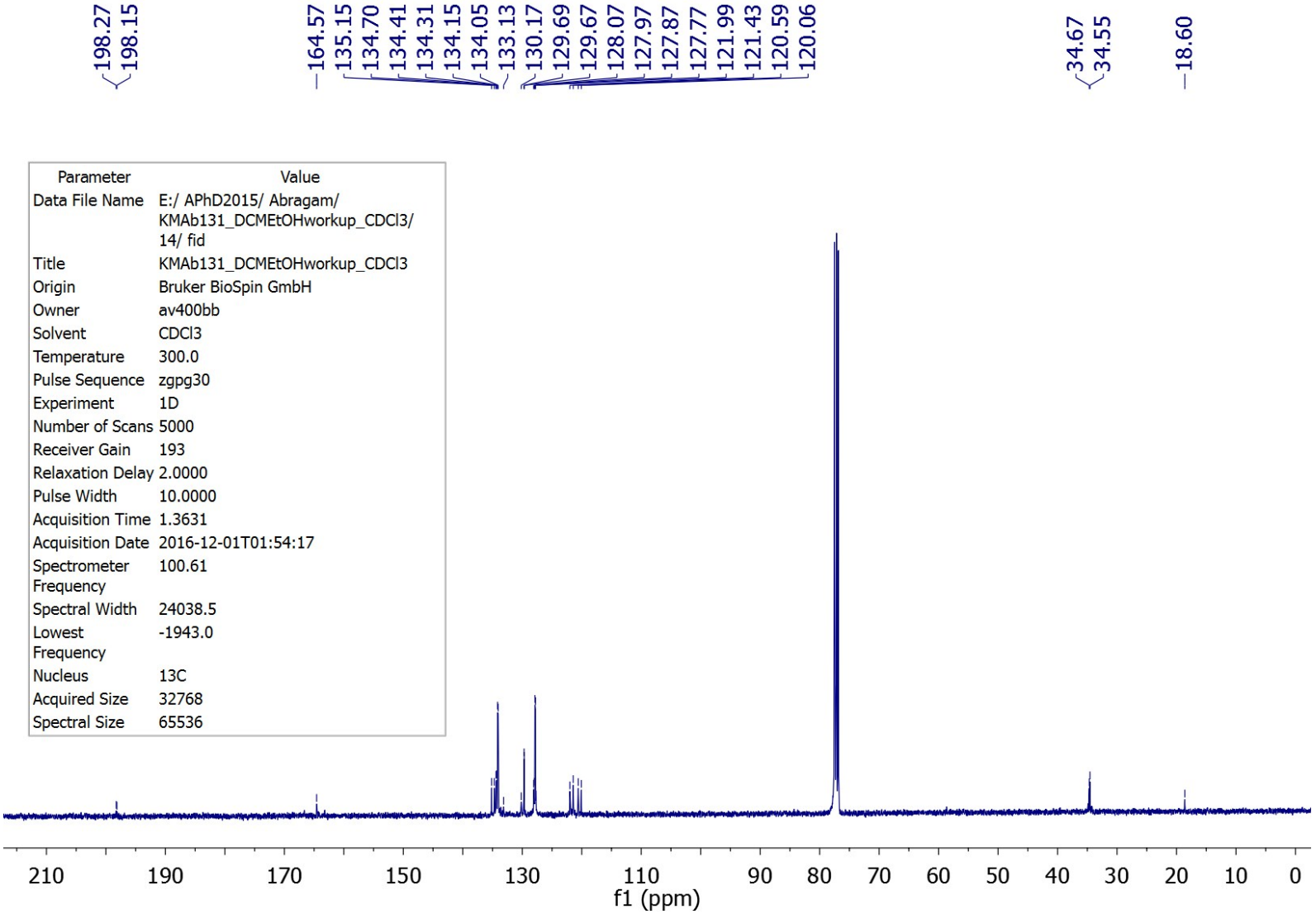


$^{31}P\{^{1}H\}$ NMR Spectrum of 3b

Parameter	Value
Data File Name	E:/ APhD2015/ Abragam/ KMAb131_DCMEtOHworkup_CDCl3/ 11/ fid
Title	KMAb131_DCMEtOHworkup_CDCl3
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	64
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2016-11-30T10:13:57
Modification Date	2016-11-30T10:14:08
Spectrometer Frequency	161.98
Spectral Width	64102.6
Lowest Frequency	-39986.0
Nucleus	31P
Acquired Size	32768
Spectral Size	65536

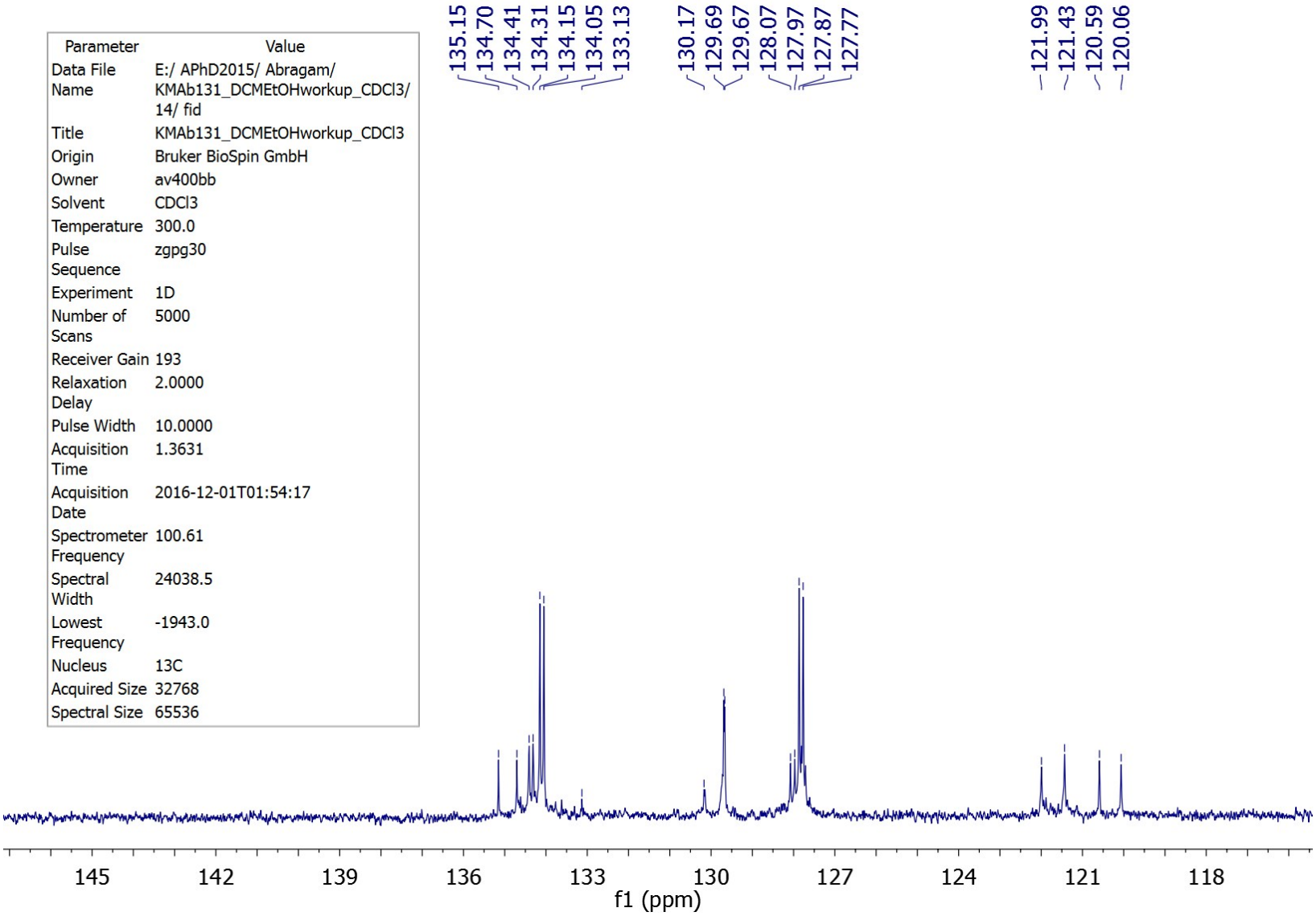


$^{13}C\{^{1}H\}$ NMR Spectrum of 3b



$^{13}C\{^{1}H\}$ NMR Spectrum of 3b (Expansion)

Parameter	Value
Data File Name	E:/ APhD2015/ Abragam/ KMAb131_DCMEtOHworkup_CDCl3/ 14/ fid
Title	KMAb131_DCMEtOHworkup_CDCl3
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	5000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-01T01:54:17
Spectrometer Frequency	100.61
Spectral Width	24038.5
Lowest Frequency	-1943.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

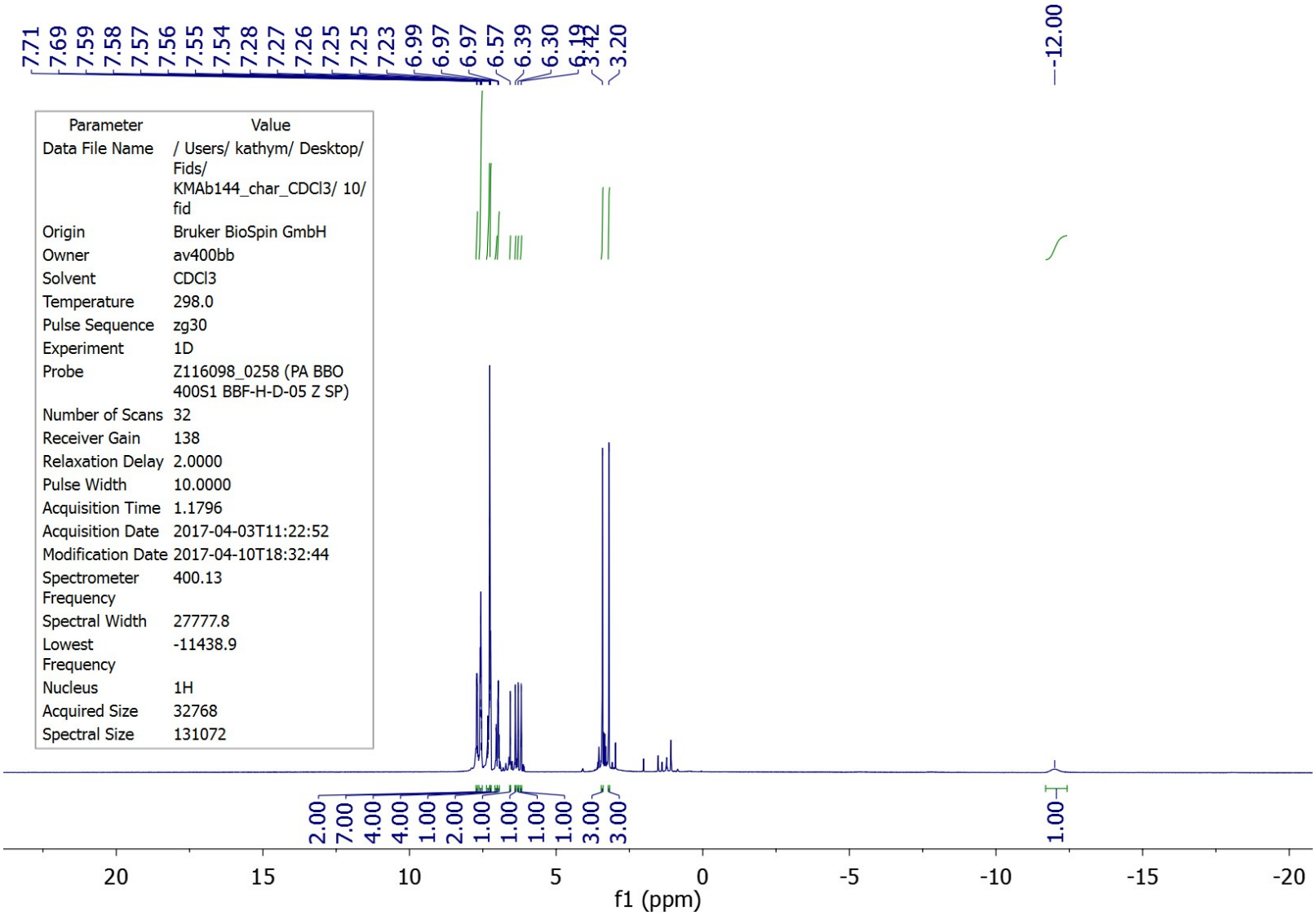


Characterisation of [Ru(SePh)(CO)(PPh_3){$H_2B(mt)_2$}] (3c)

Commentary

Spectroscopic data for **3b** call for little comment other than to note the conspicuous loss of the hydride doublet resonance in the ^1H NMR spectrum, whilst the broad B–H–Ru resonance shifts to δ_H = –18.11.

^{1}H NMR Spectrum of 3c



Parameter	Value
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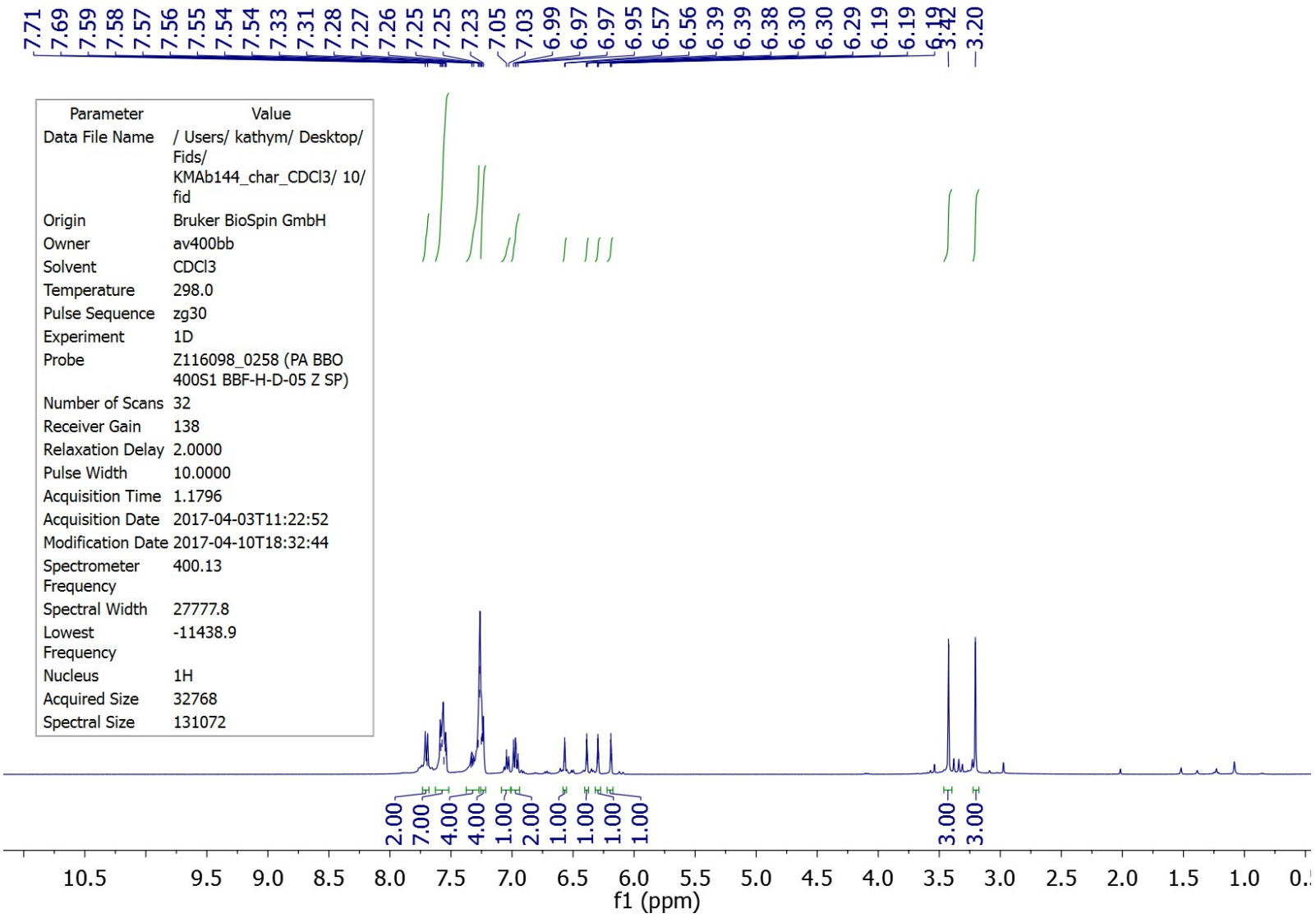
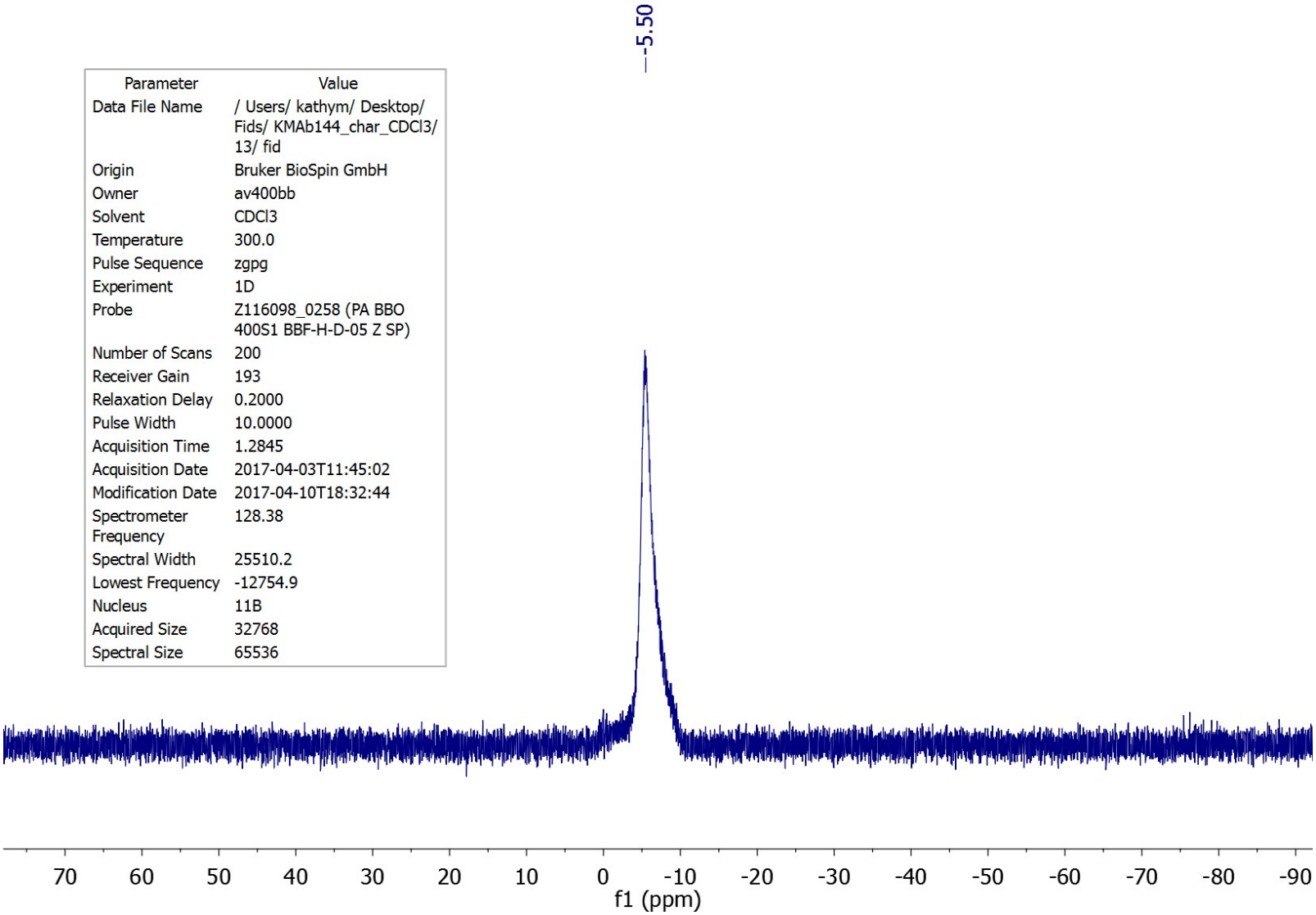
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	32
Receiver Gain	138
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1796
Acquisition Date	2017-04-03T11:22:52
Modification Date	2017-04-10T18:32:44
Spectrometer Frequency	400.13
Spectral Width	27777.8
Lowest Frequency	-11438.9
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

^{1}H NMR Spectrum of 3c (Expansion)



Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb144_char_CDCl3/ 10/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	32
Receiver Gain	138
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1796
Acquisition Date	2017-04-03T11:22:52
Modification Date	2017-04-10T18:32:44
Spectrometer Frequency	400.13
Spectral Width	27777.8
Lowest Frequency	-11438.9
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

$^{11}B\{^{1}H\}$ NMR Spectrum of 3c



Parameter	Value
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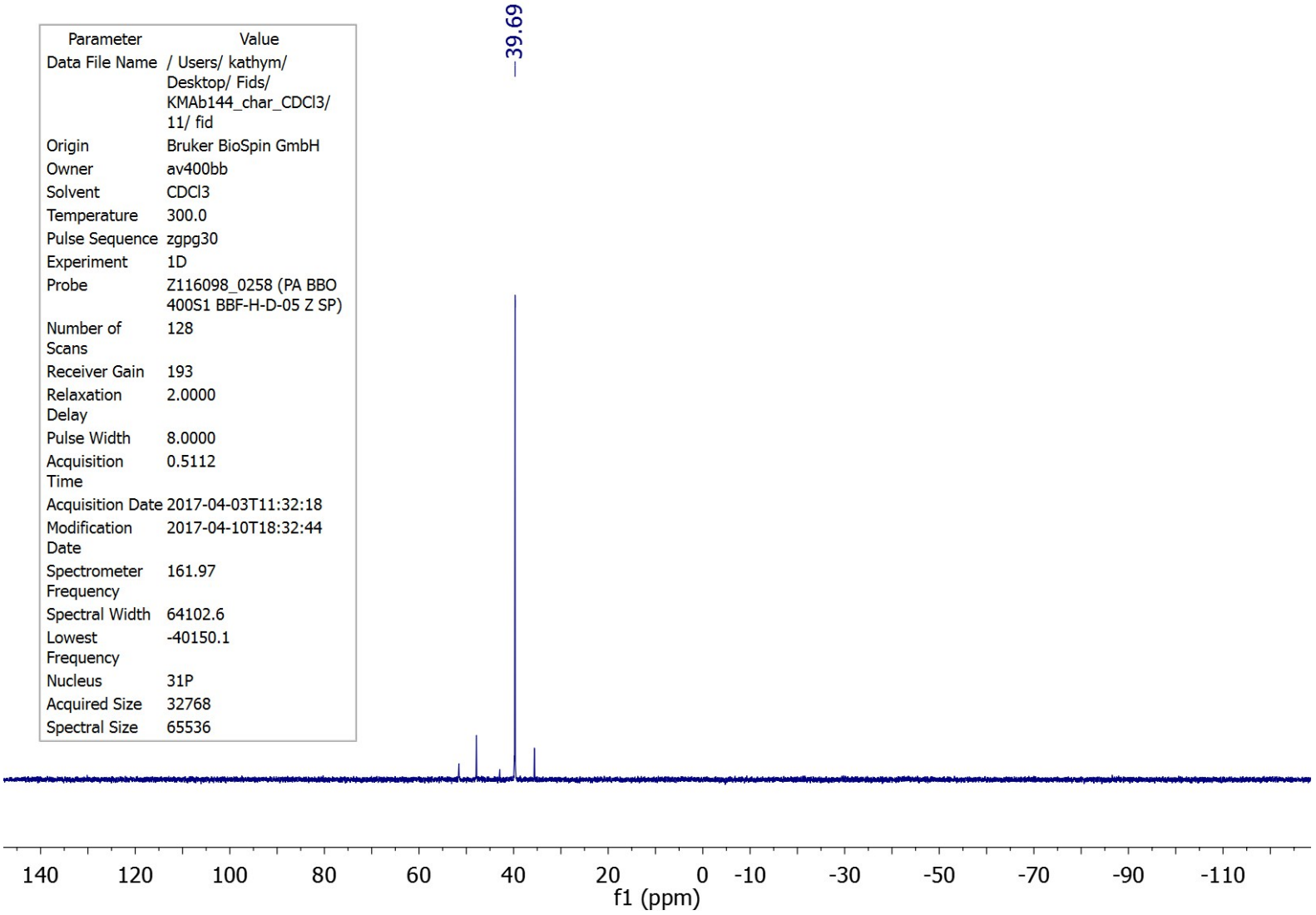
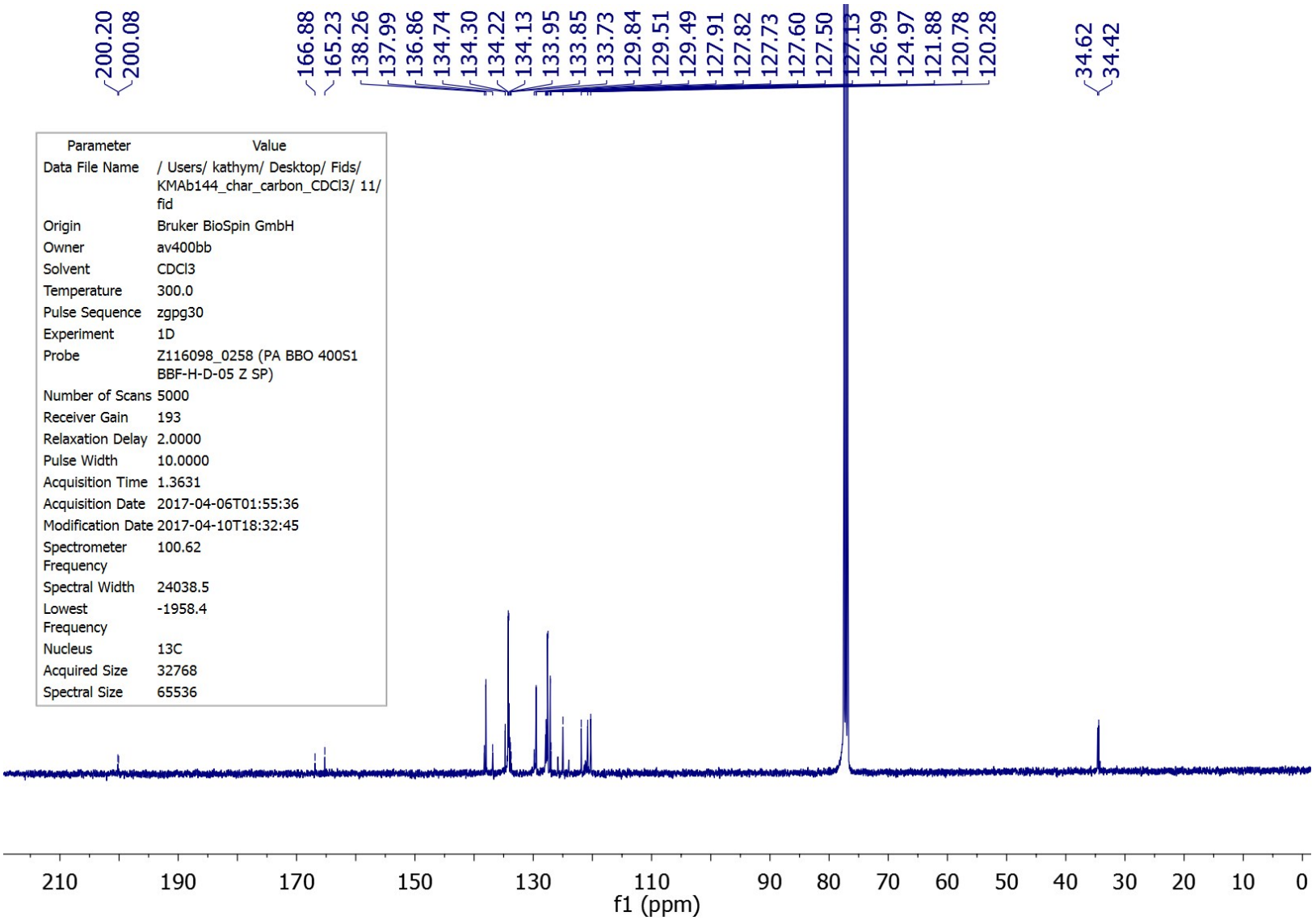
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2017-04-03T11:45:02
Modification Date	2017-04-10T18:32:44
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12754.9
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

$^{31}P\{^{1}H\}$ NMR Spectrum of 3c

Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb144_char_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	128
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2017-04-03T11:32:18
Modification Date	2017-04-10T18:32:44
Spectrometer Frequency	161.97
Spectral Width	64102.6
Lowest Frequency	-40150.1
Nucleus	31P
Acquired Size	32768
Spectral Size	65536

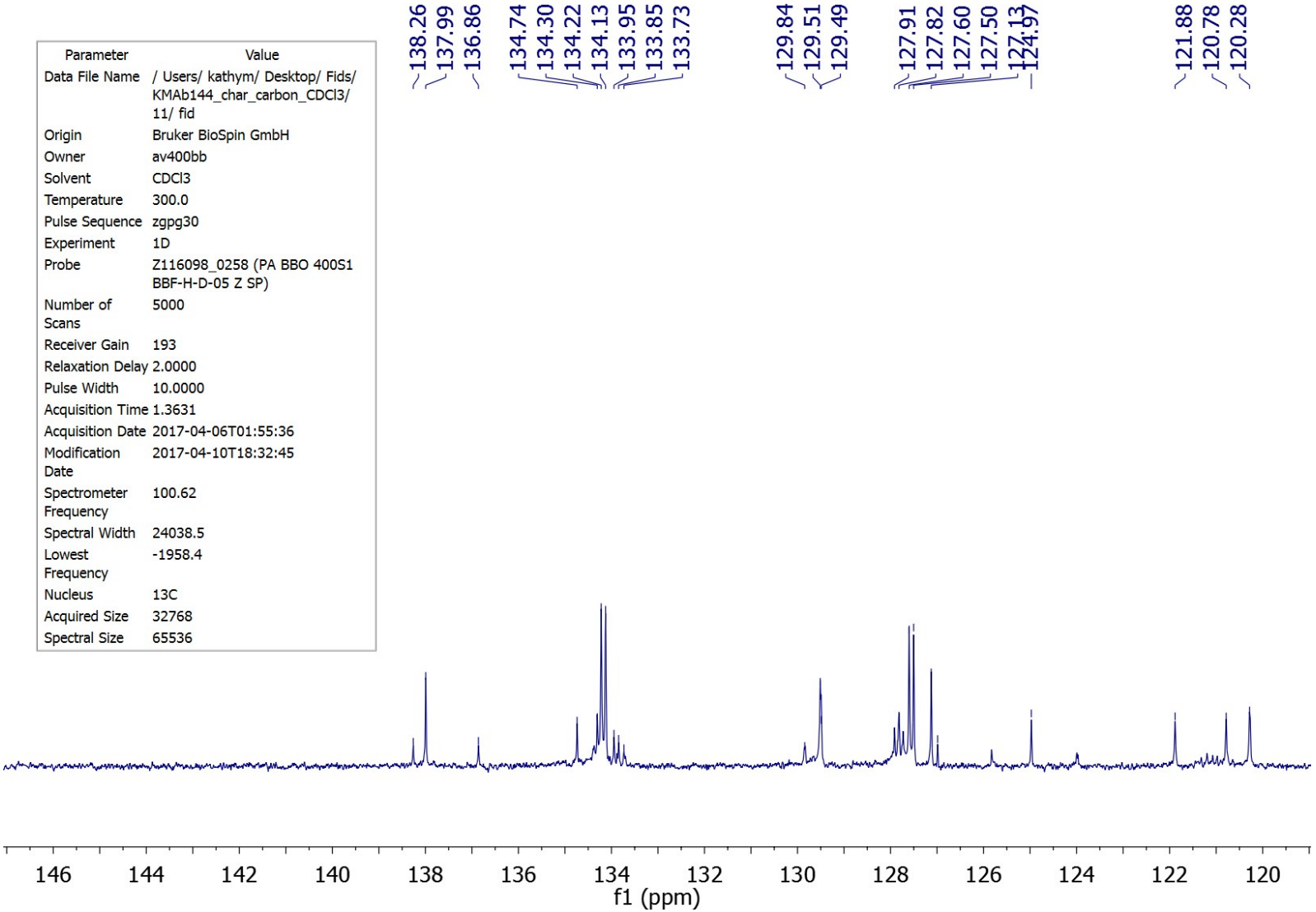


$^{13}C\{^{1}H\}$ NMR Spectrum of 3c

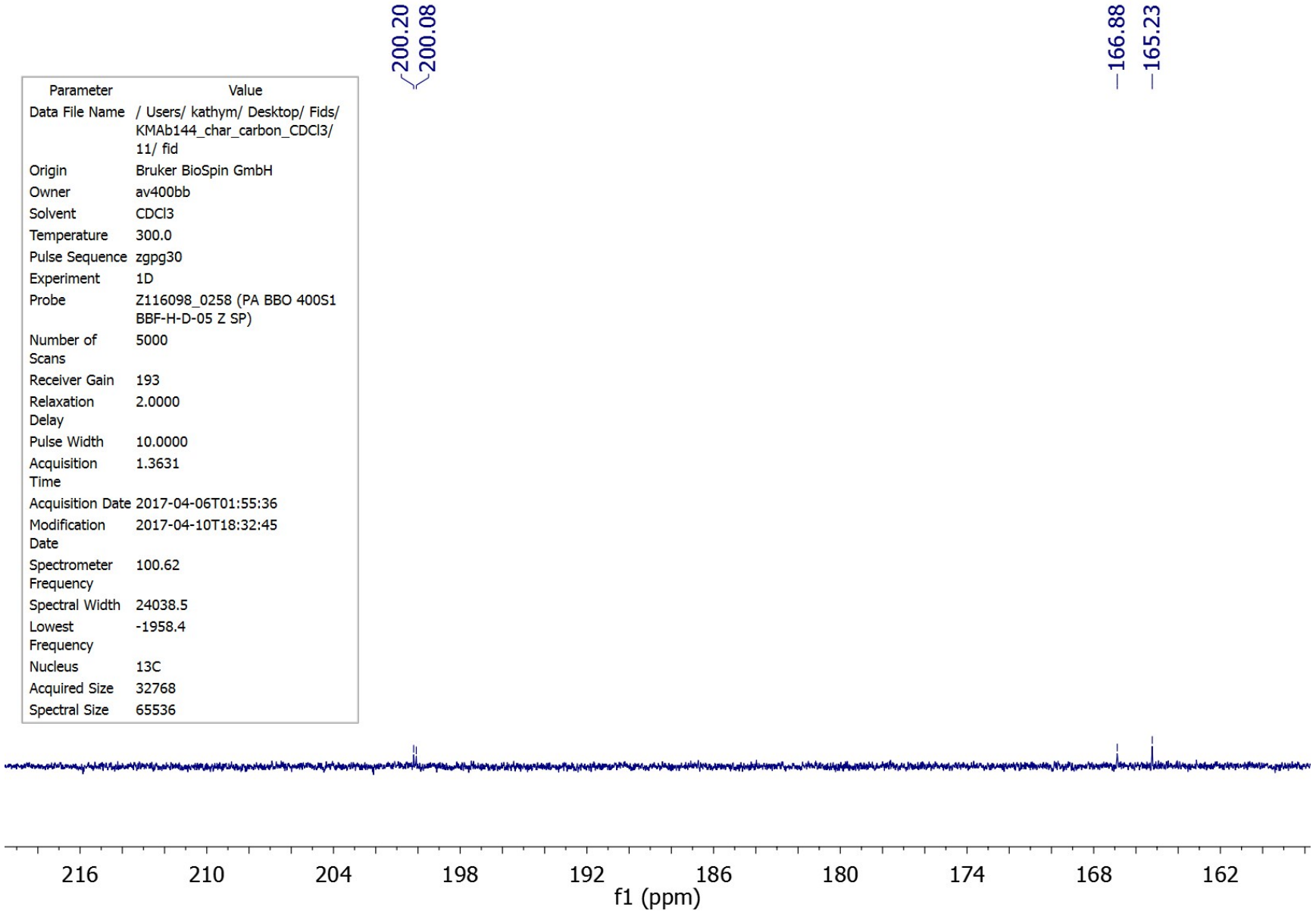


Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb144_char_carbon_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	5000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-06T01:55:36
Modification Date	2017-04-10T18:32:45
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

$^{13}C\{^{1}H\}$ NMR Spectrum of 3c (Expansions)

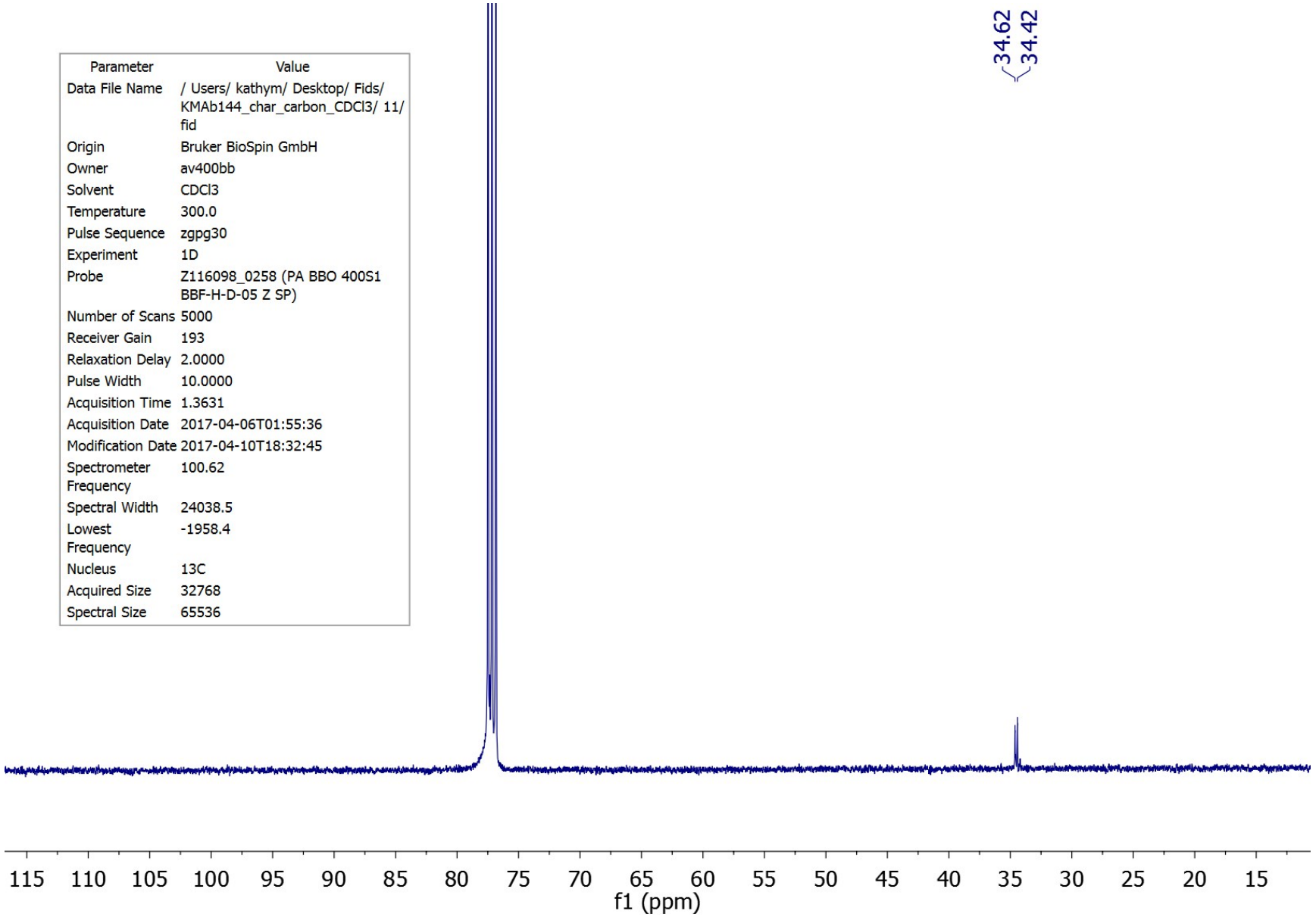


Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb144_char_carbon_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	5000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-06T01:55:36
Modification Date	2017-04-10T18:32:45
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb144_char_carbon_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	5000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-06T01:55:36
Modification Date	2017-04-10T18:32:45
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb144_char_carbon_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z116098_0258 (PA BBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	5000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-06T01:55:36
Modification Date	2017-04-10T18:32:45
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

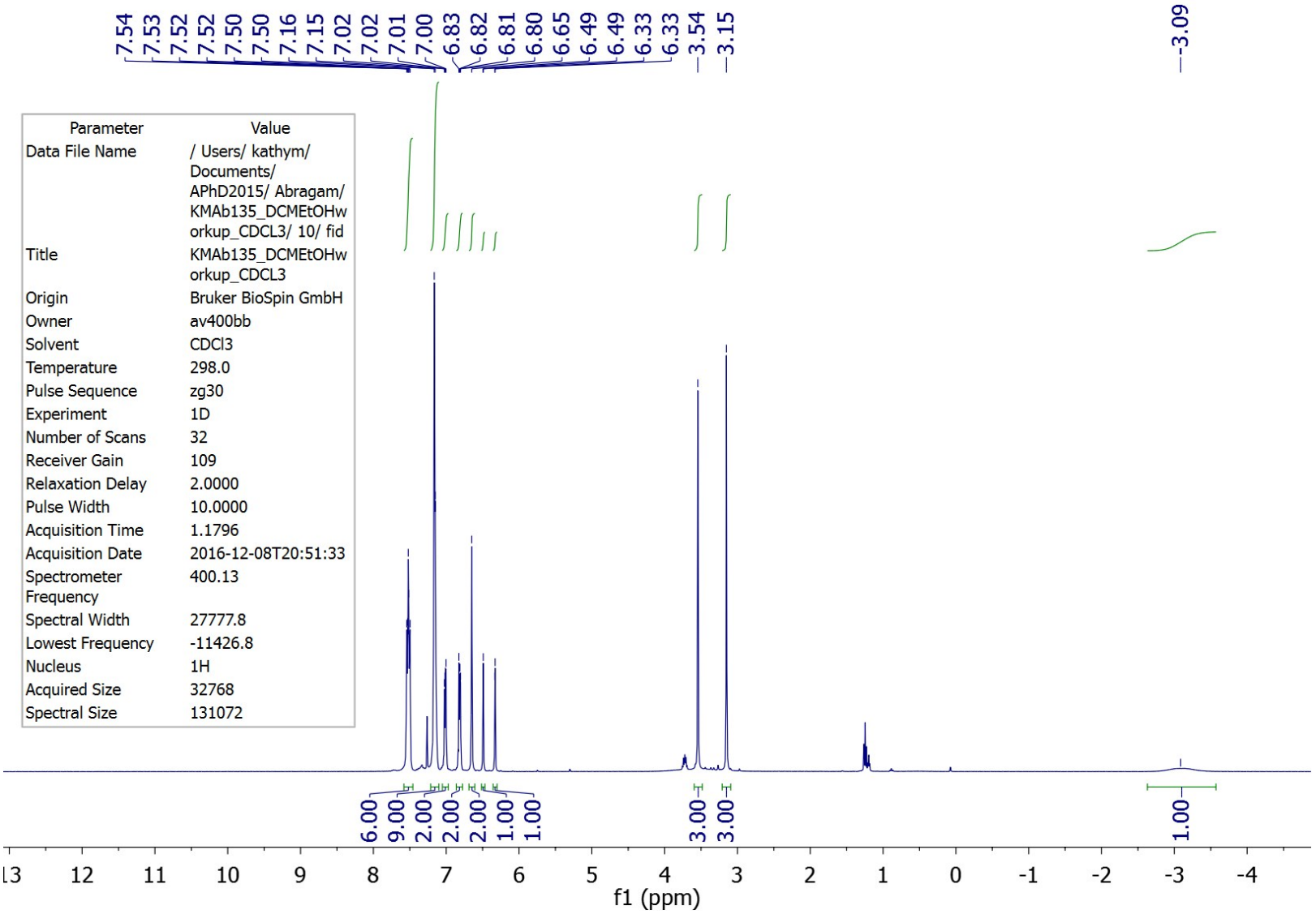


Characterisation of [Ru(BCat)(CO)(PPh_3){$H_2B(mt)_2$}] (3e)

Commentary

The formulation of **3e** follows from spectroscopic data akin to those for **3a-c** in addition to the appearance of two resonances in the ^{11}B NMR spectrum. The resonance at δ_{11B} = –5.44 corresponds to the BH_2 group (*cf*. δ_{11B} = – 5.5 for **3a**) whilst that at δ_{11B} = 52.3 corresponds to the trigonal boron of the BCat ligand. The $^{31}P\{^1H\}$ NMR spectrum comprises a single *sharp* resonance (δ_P = 47.2) confirming that the boryl and phosphine ligands are mutually *cis*.

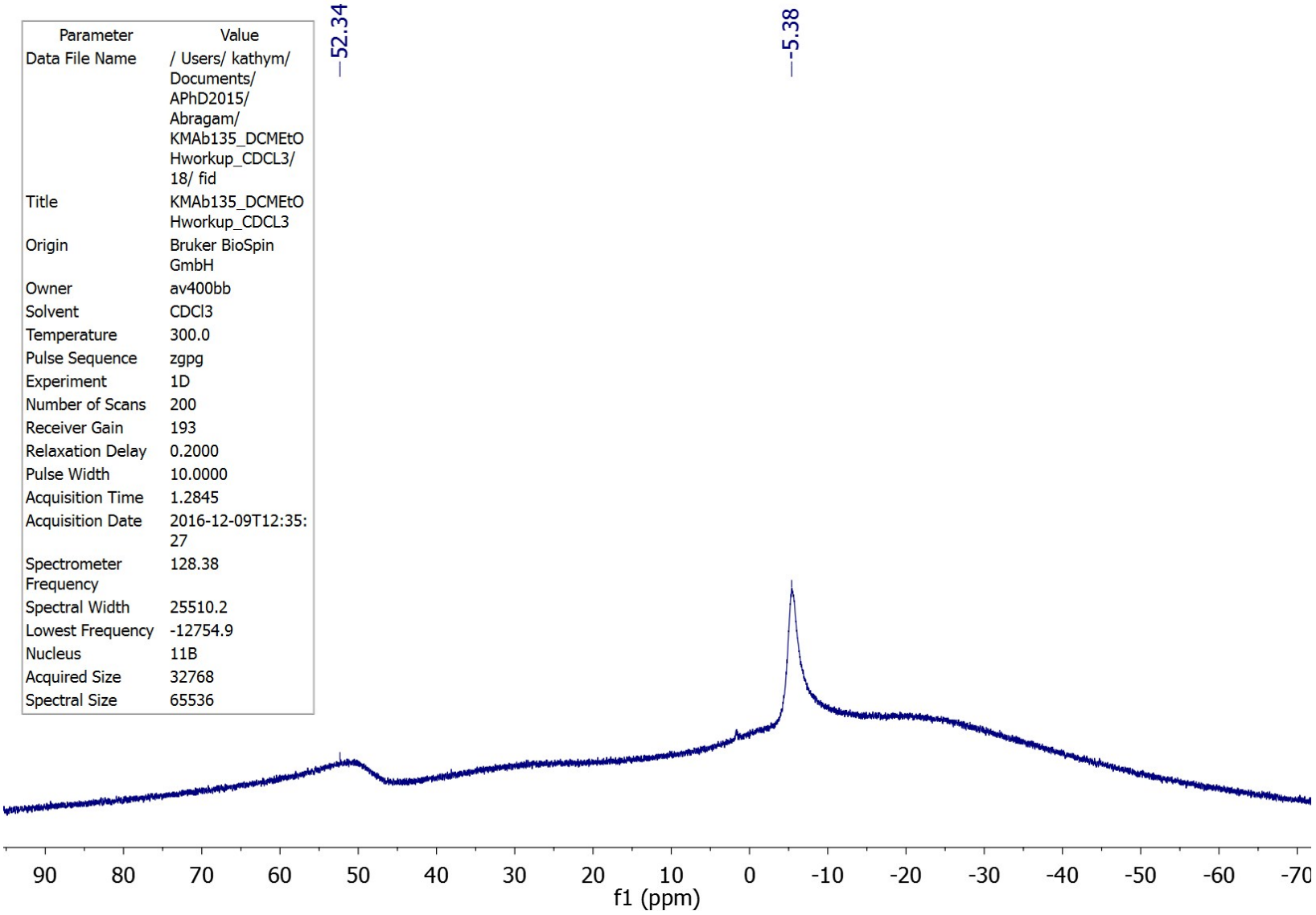
^{1}H NMR Spectrum of 3e



Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb135_DCMEtOHworkup_CDCL3/ 10/ fid
Title	KMAb135_DCMEtOHworkup_CDCL3
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	109
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1796
Acquisition Date	2016-12-08T20:51:33
Spectrometer Frequency	400.13
Spectral Width	27777.8
Lowest Frequency	-11426.8
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

$^{11}B\{^{1}H\}$ NMR Spectrum of 3e

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb135_DCMEtOHworkup_CDCL3/ 18/ fid
Title	KMAb135_DCMEtOHworkup_CDCL3
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg
Experiment	1D
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2016-12-09T12:35:27
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12754.9
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



$^{31}P\{^{1}H\}$ NMR Spectrum of 3e

Parameter	Value
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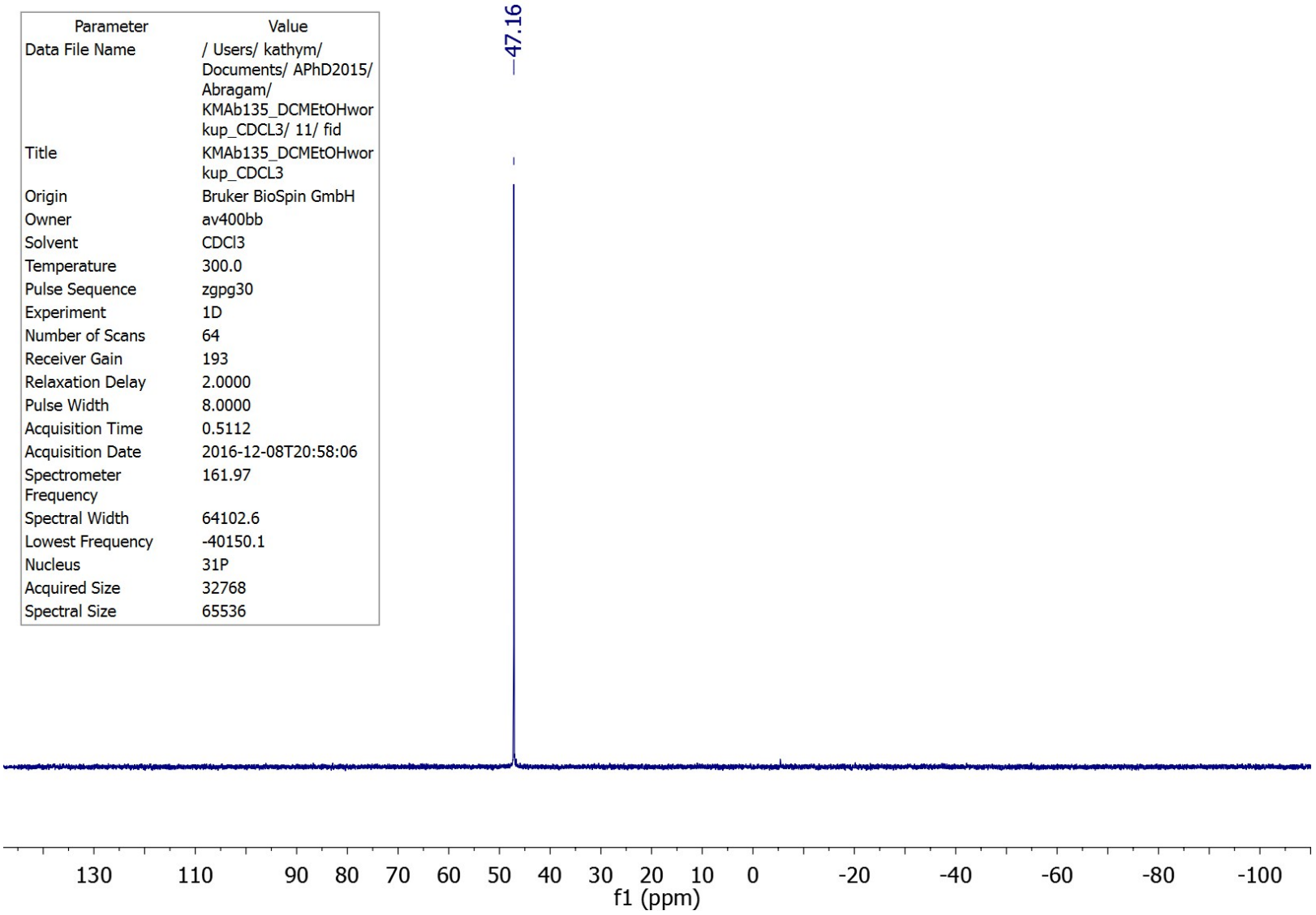
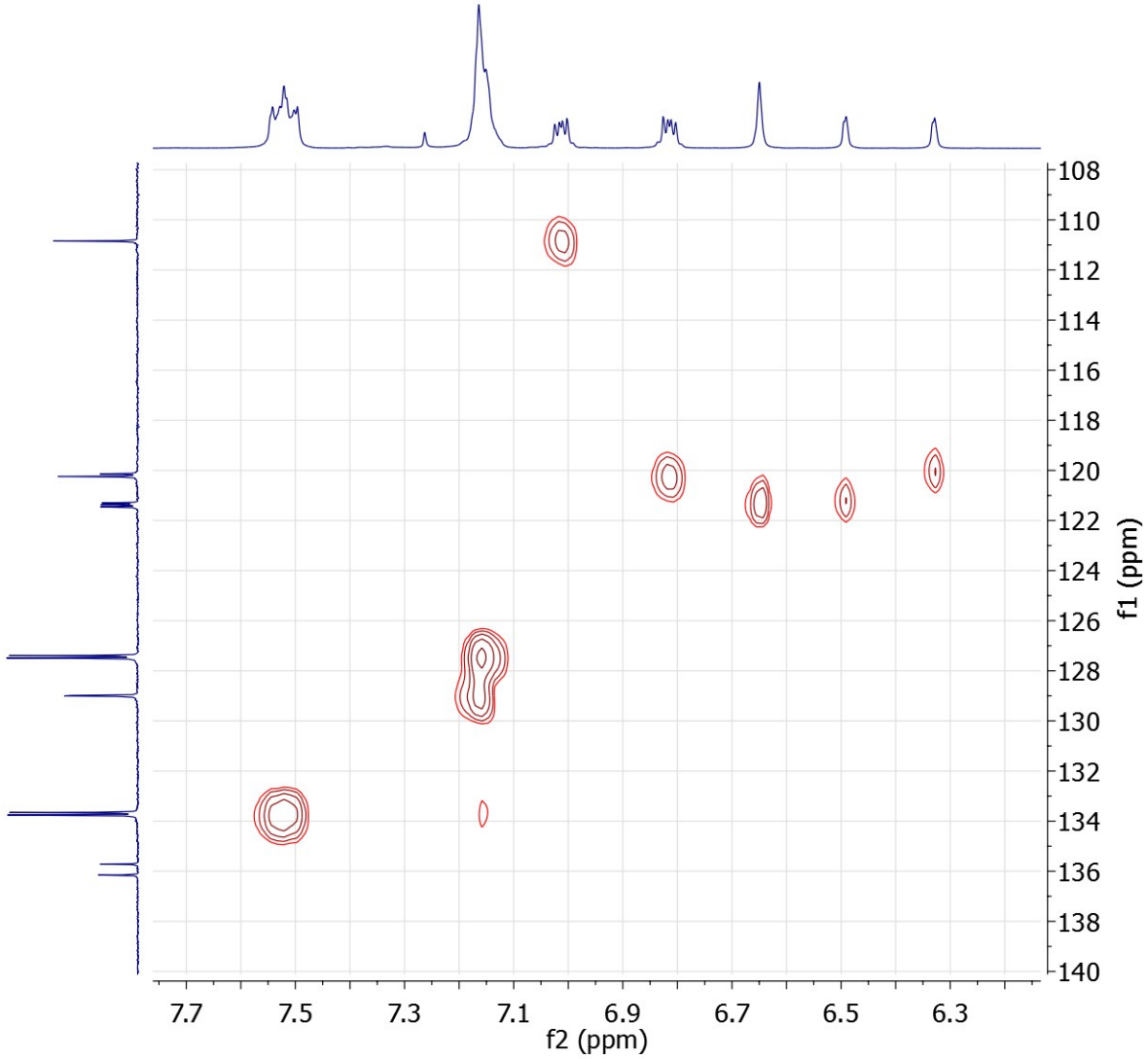
Title	KMAb135_DCMEtOHworkup_CDCL3
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	64
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2016-12-08T20:58:06
Spectrometer Frequency	161.97
Spectral Width	64102.6
Lowest Frequency	-40150.1
Nucleus	31P
Acquired Size	32768
Spectral Size	65536

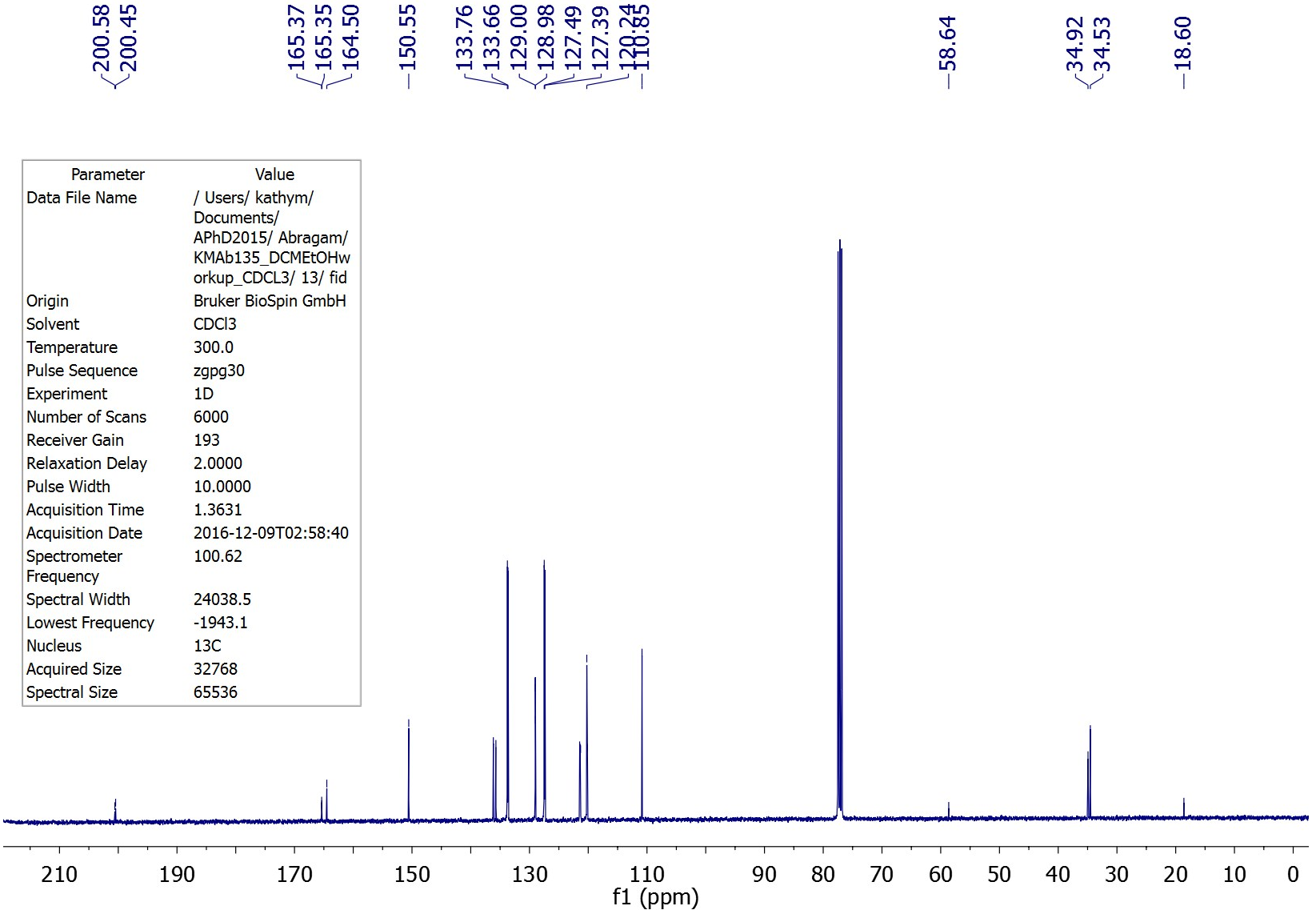


HSQC ^{13}C-^{1}H NMR Spectrum of 3e

Parameter	Value (f2, f1)
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb135_DCMEtOH workup_CDCL3/ 14/ ser
Title	KMAb135_DCMEtOH workup_CDCL3
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	hsqcedetgpsisp2.3
Experiment	2D-HSQC-EDITED
Number of Scans	4
Receiver Gain	193
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.2135
Acquisition Date	2016-12-09T03:00:46
Spectrometer Frequency	(400.13, 100.62)
Spectral Width	(4795.4, 16611.3)
Lowest Frequency	(-526.3, -1245.9)
Nucleus	(1H, 13C)
Acquired Size	(1024, 256)
Spectral Size	(1024, 1024)



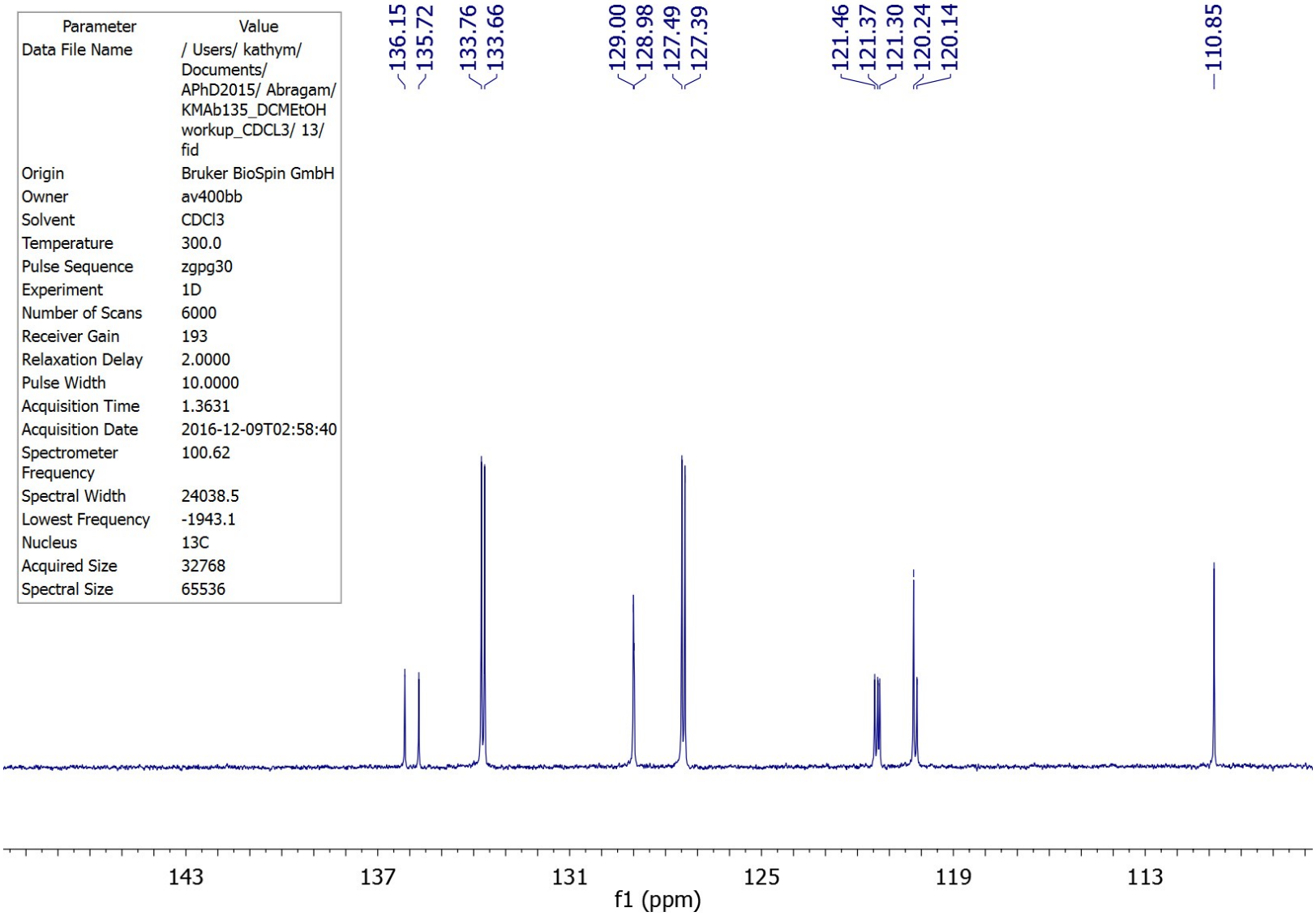
$^{13}C\{^{1}H\}$ NMR Spectrum of 3e



Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb135_DCMEtOHworkup_CDCL3/ 13/ fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	6000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-09T02:58:40
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1943.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

$^{13}C\{^{1}H\}$ NMR Spectrum of 3e (Expansion)

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb135_DCMEtOH workup_CDCL3/ 13/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	6000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-09T02:58:40
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1943.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

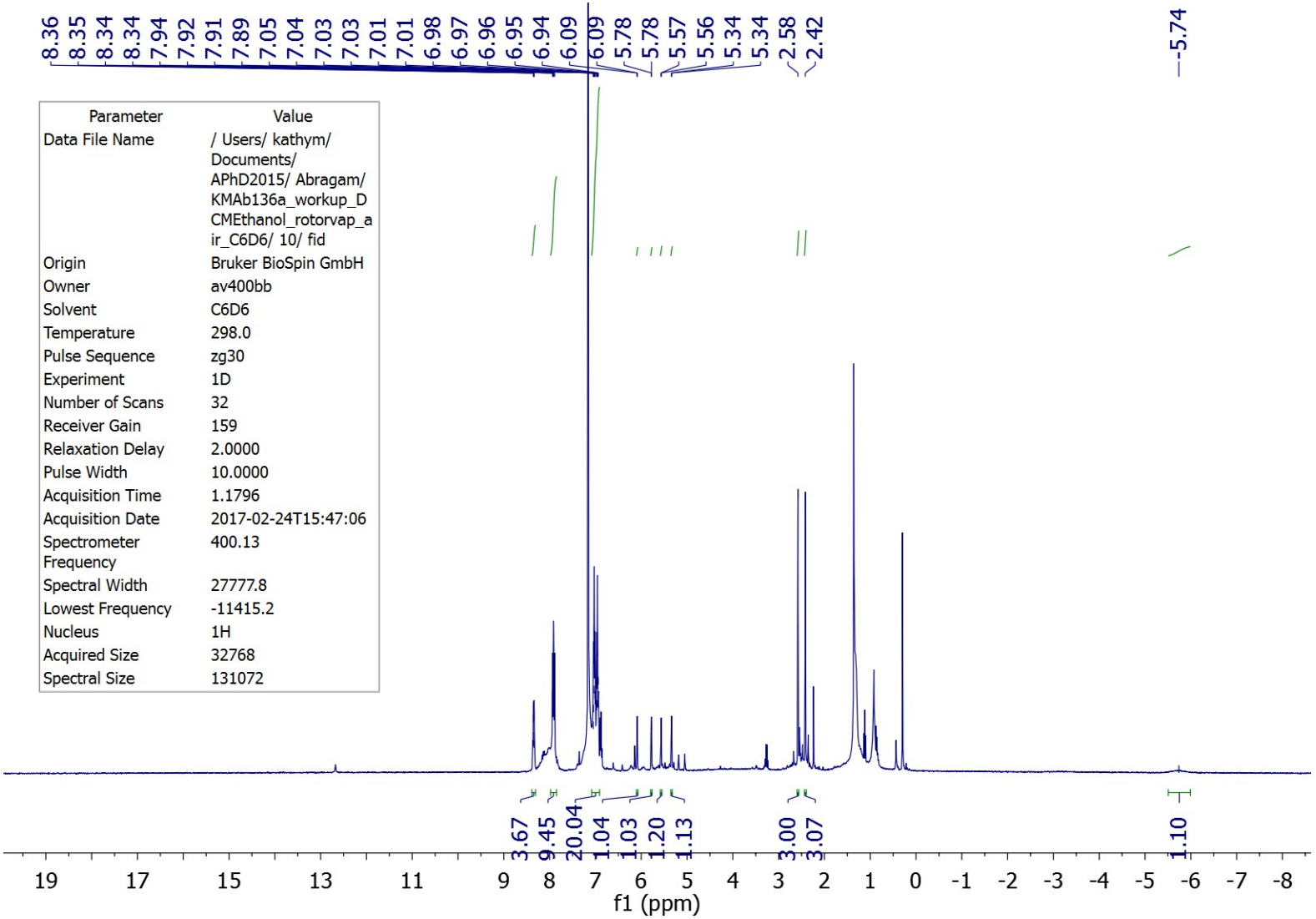


Characterisation of $[Ru(SiCl_3)(CO)(PPh_3)\{H_2B(mt)_2\}]$ (3f)

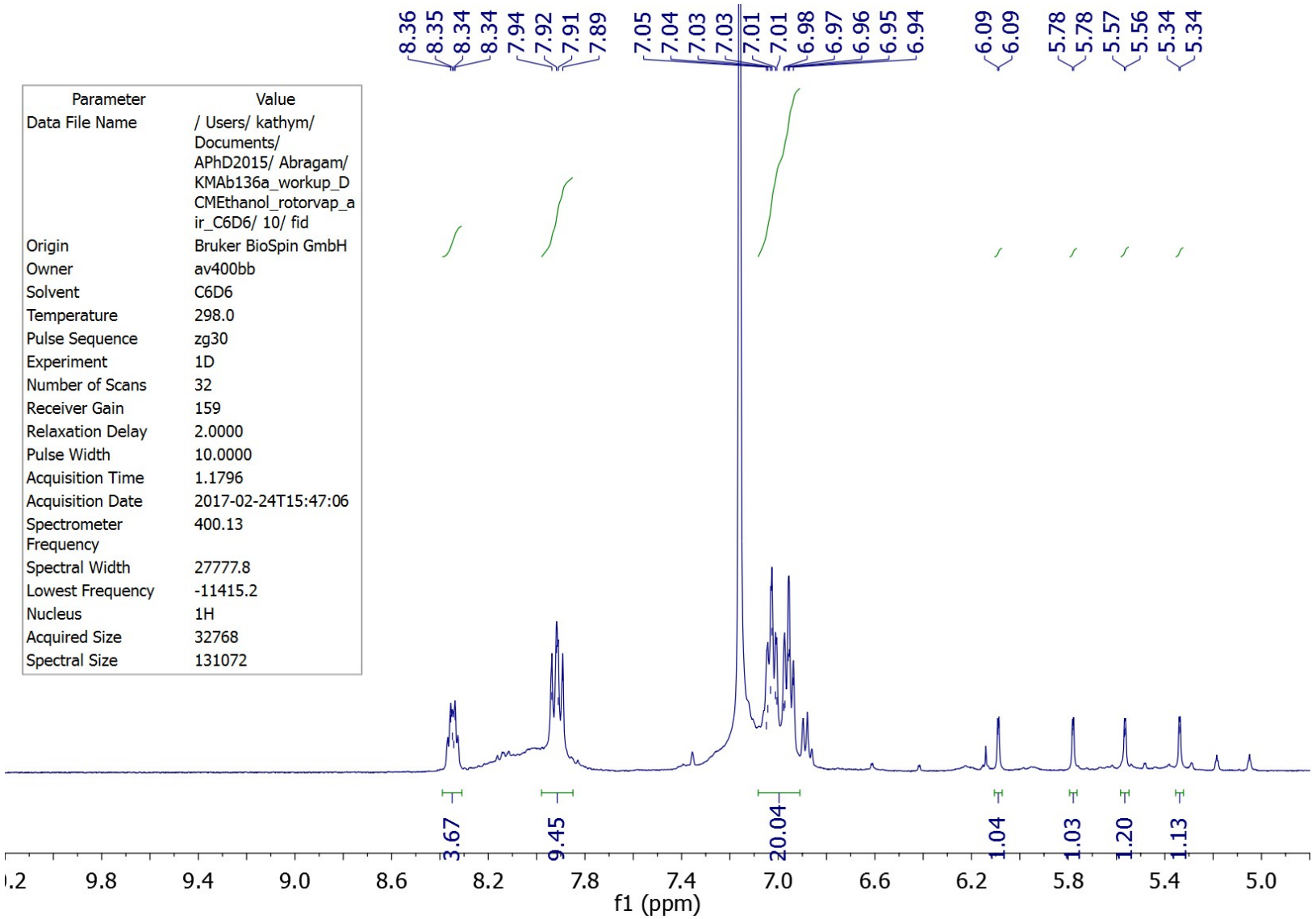
Commentary

The infrared spectrum of **3f** included weak absorptions at 2430 and 2046 cm^{-1} consistent with κ^3-*H,S,S'* coordination of the borate which was further supported by a broad resonance at –5.74 ppm in the ^1H NMR spectrum.

^{1}H NMR Spectrum of 3f



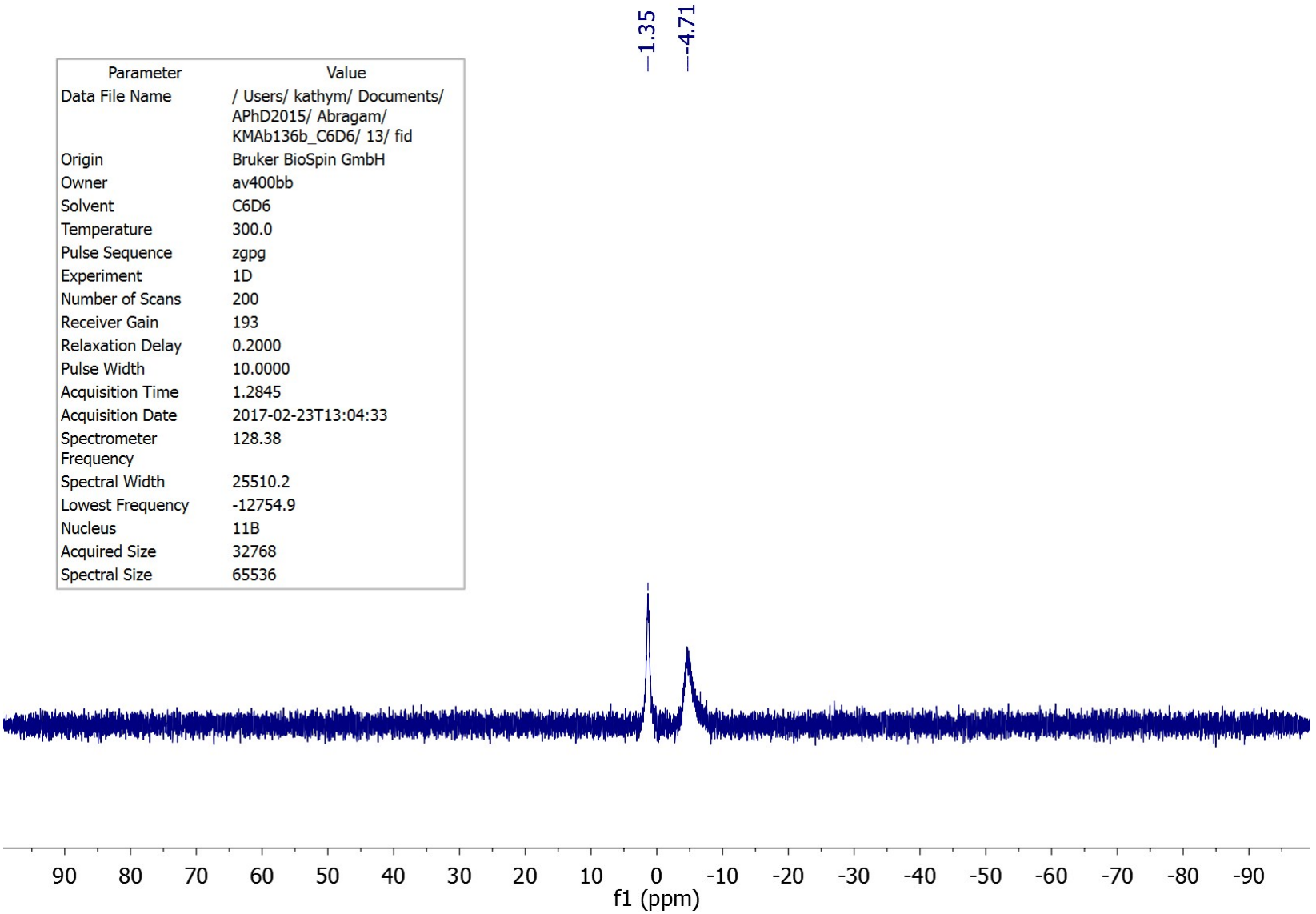
^{1}H NMR Spectrum of 3f (Expansion)



Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb136a_workup_DCMEthanol_rotorvap_air_C6D6/ 10/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	C6D6
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	159
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1796
Acquisition Date	2017-02-24T15:47:06
Spectrometer Frequency	400.13
Spectral Width	27777.8
Lowest Frequency	-11415.2
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

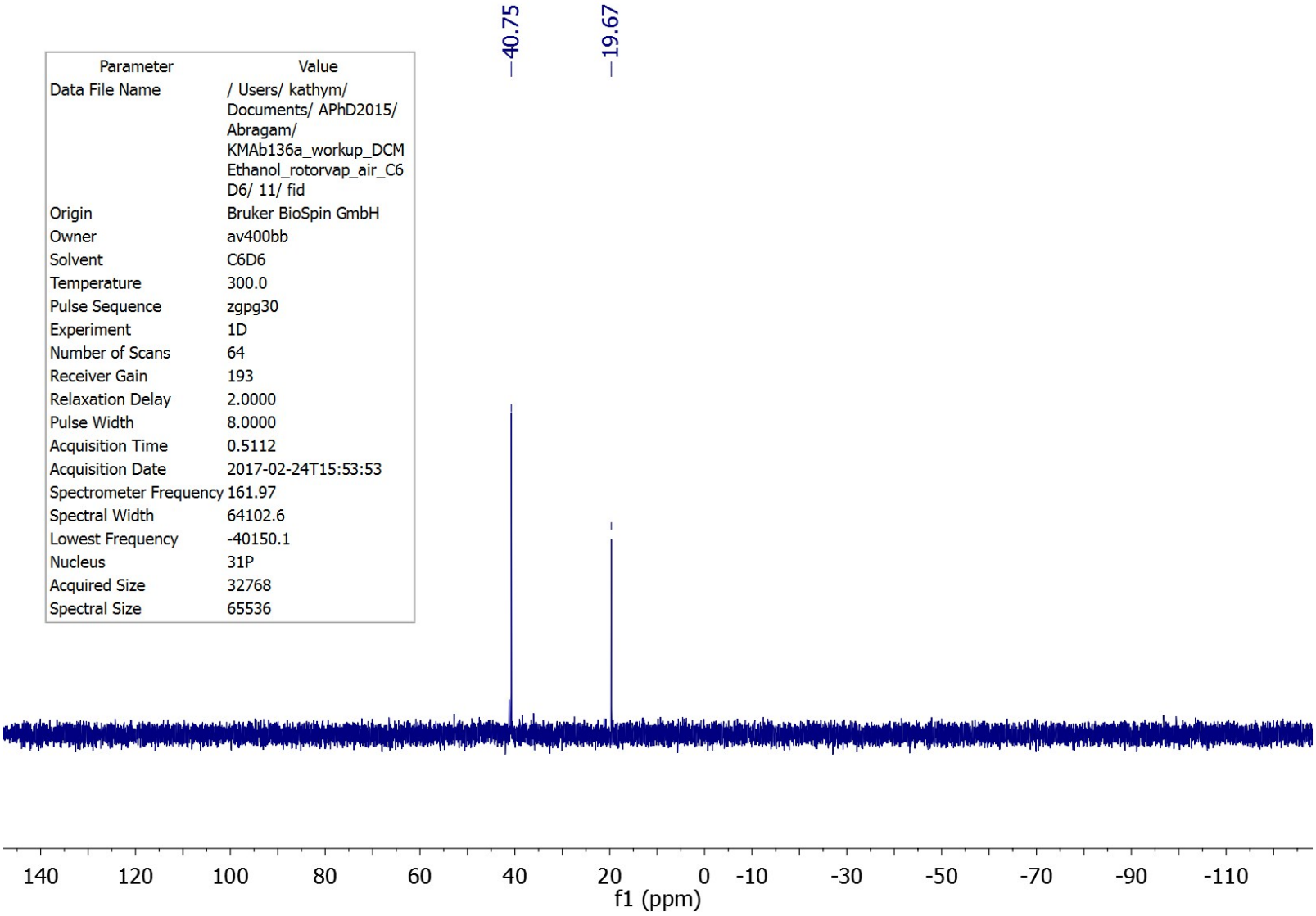
$^{11}B\{^{1}H\}$ NMR Spectrum of 3f

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb136b_C6D6/ 13/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	C6D6
Temperature	300.0
Pulse Sequence	zgpg
Experiment	1D
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2017-02-23T13:04:33
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12754.9
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



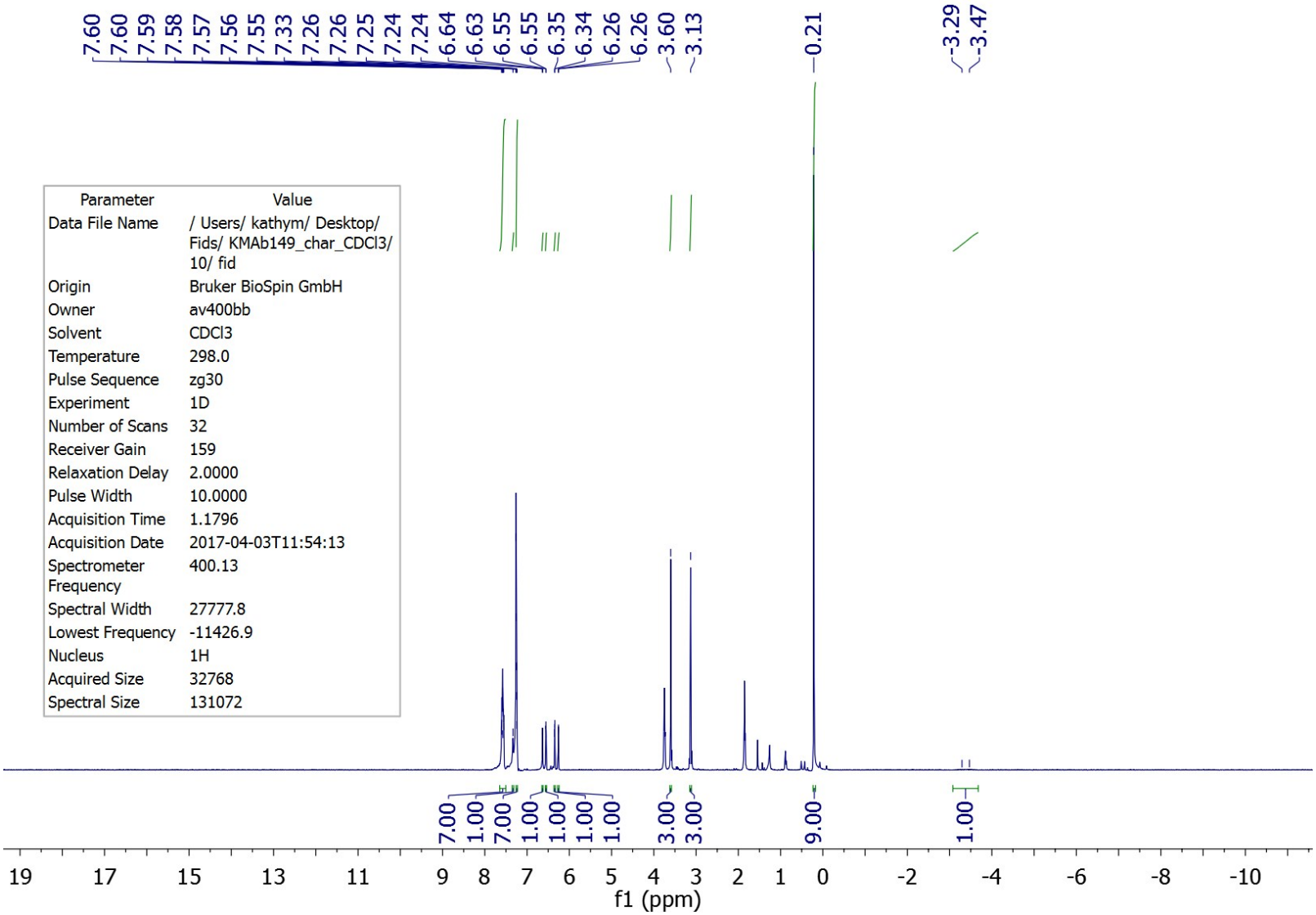
$^{31}P\{^{1}H\}$ NMR Spectrum of 3f

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb136a_workup_DCM Ethanol_rotorvap_air_C6 D6/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	C6D6
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	64
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2017-02-24T15:53:53
Spectrometer Frequency	161.97
Spectral Width	64102.6
Lowest Frequency	-40150.1
Nucleus	31P
Acquired Size	32768
Spectral Size	65536



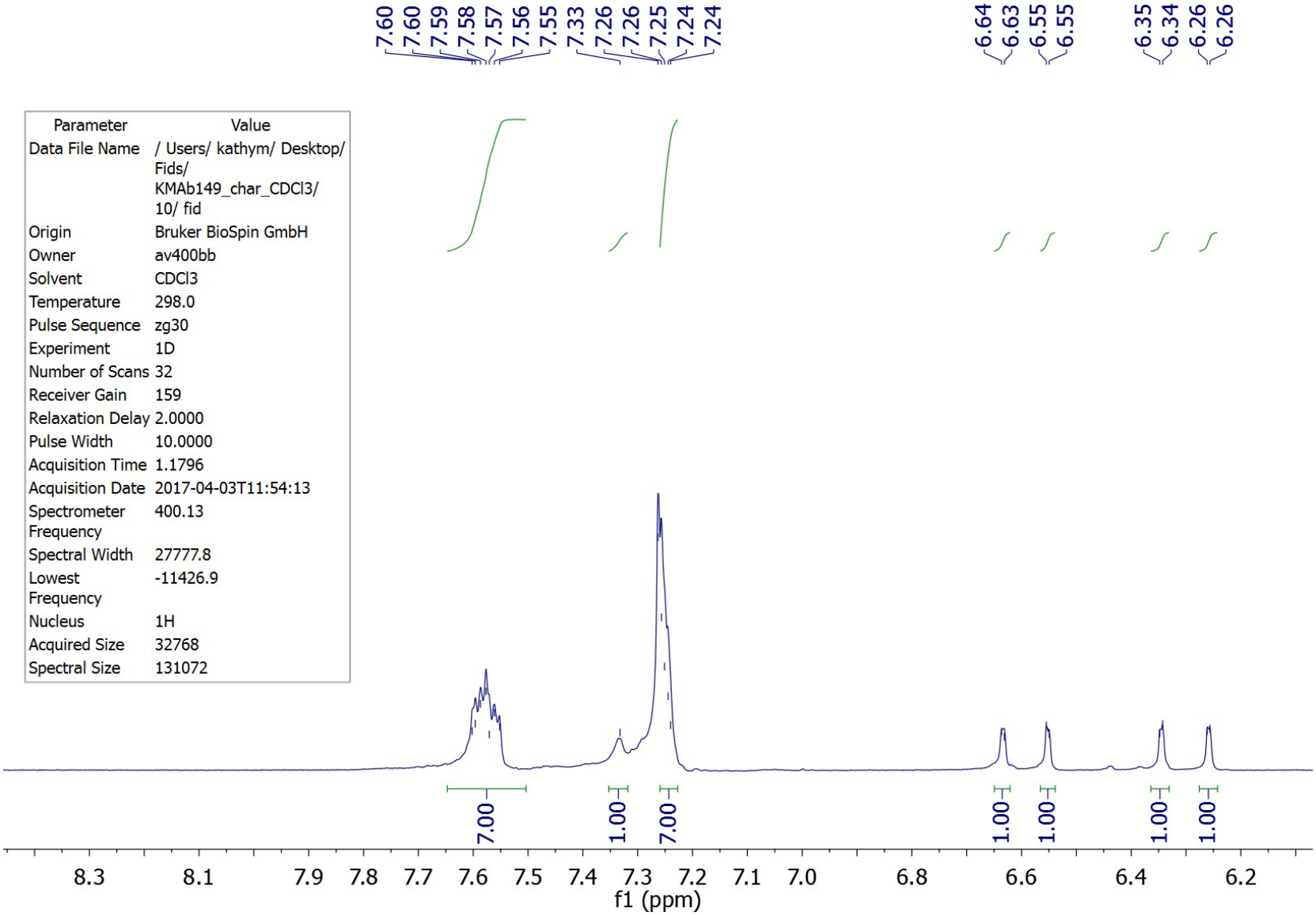
Characterisation of [Ru(SiMe$_3$)(CO)(PPh$_3$){H$_2$B(mt)$_2$}] (3f)

^{1}H NMR Spectrum of 3g



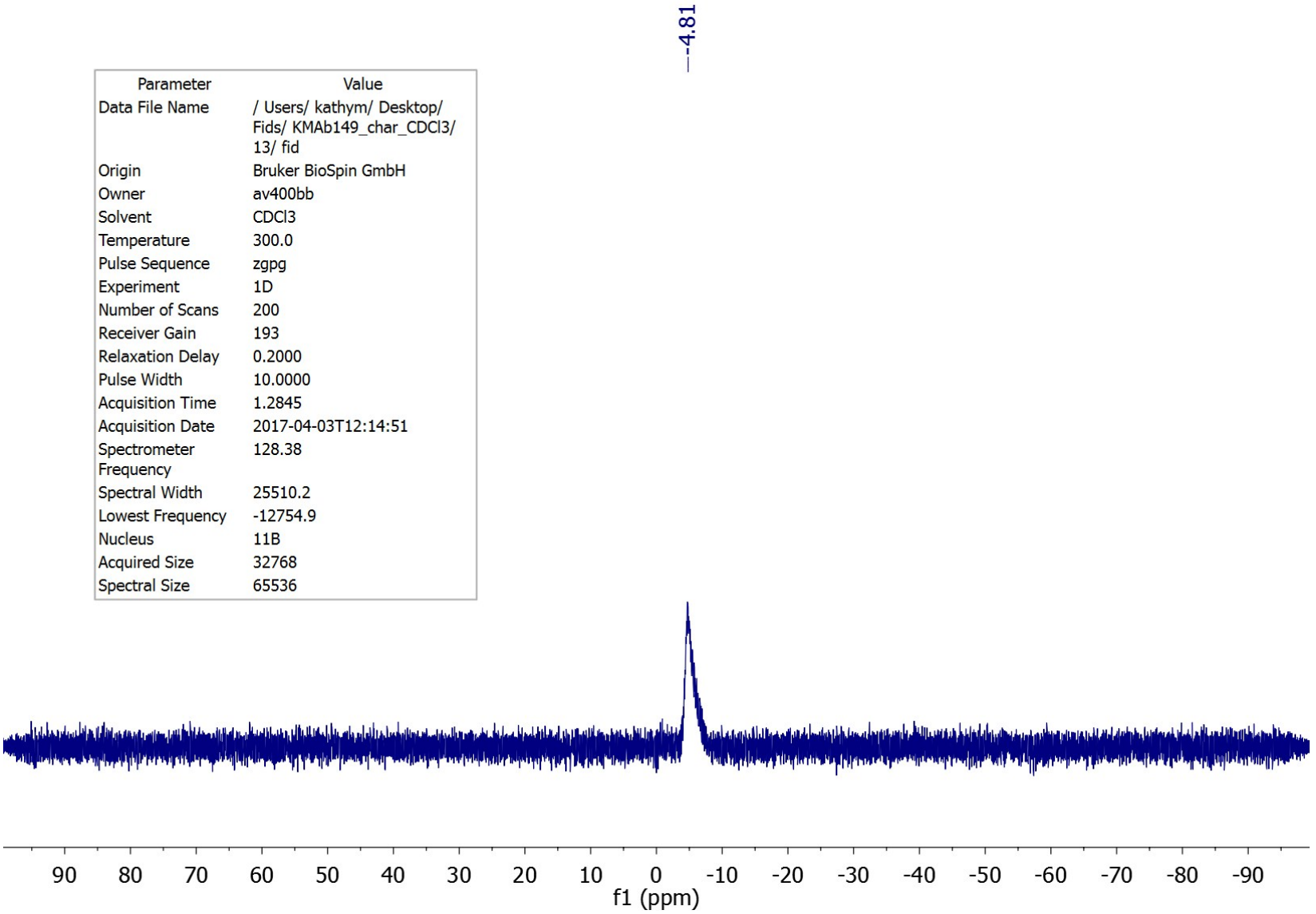
Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb149_char_CDCl3/ 10/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	159
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1796
Acquisition Date	2017-04-03T11:54:13
Spectrometer Frequency	400.13
Spectral Width	27777.8
Lowest Frequency	-11426.9
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

^{1}H NMR Spectrum of 3g (Expansion)



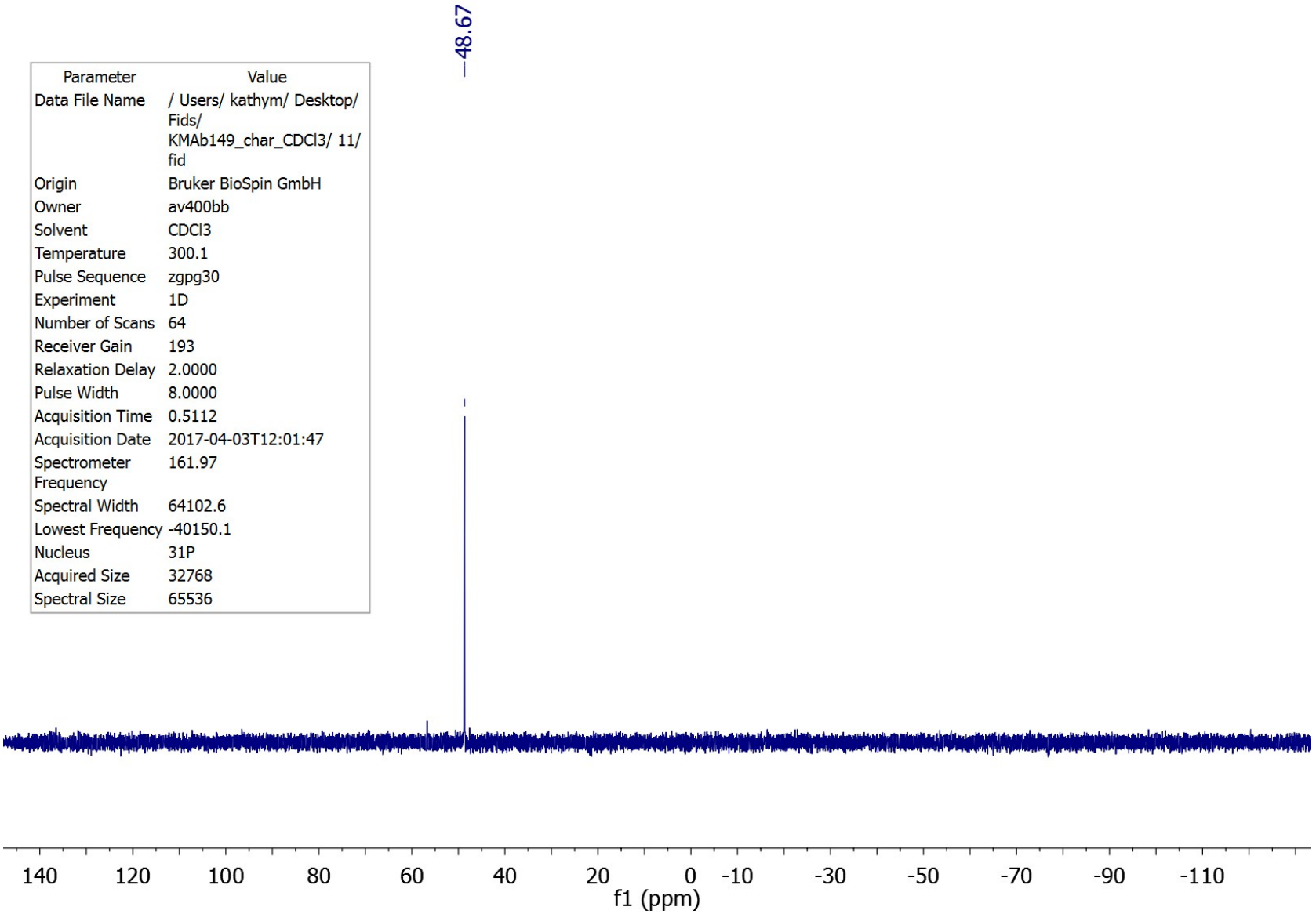
$^{11}B\{^{1}H\}$ NMR Spectrum of 3g

Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb149_char_CDCl3/ 13/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg
Experiment	1D
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2017-04-03T12:14:51
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12754.9
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

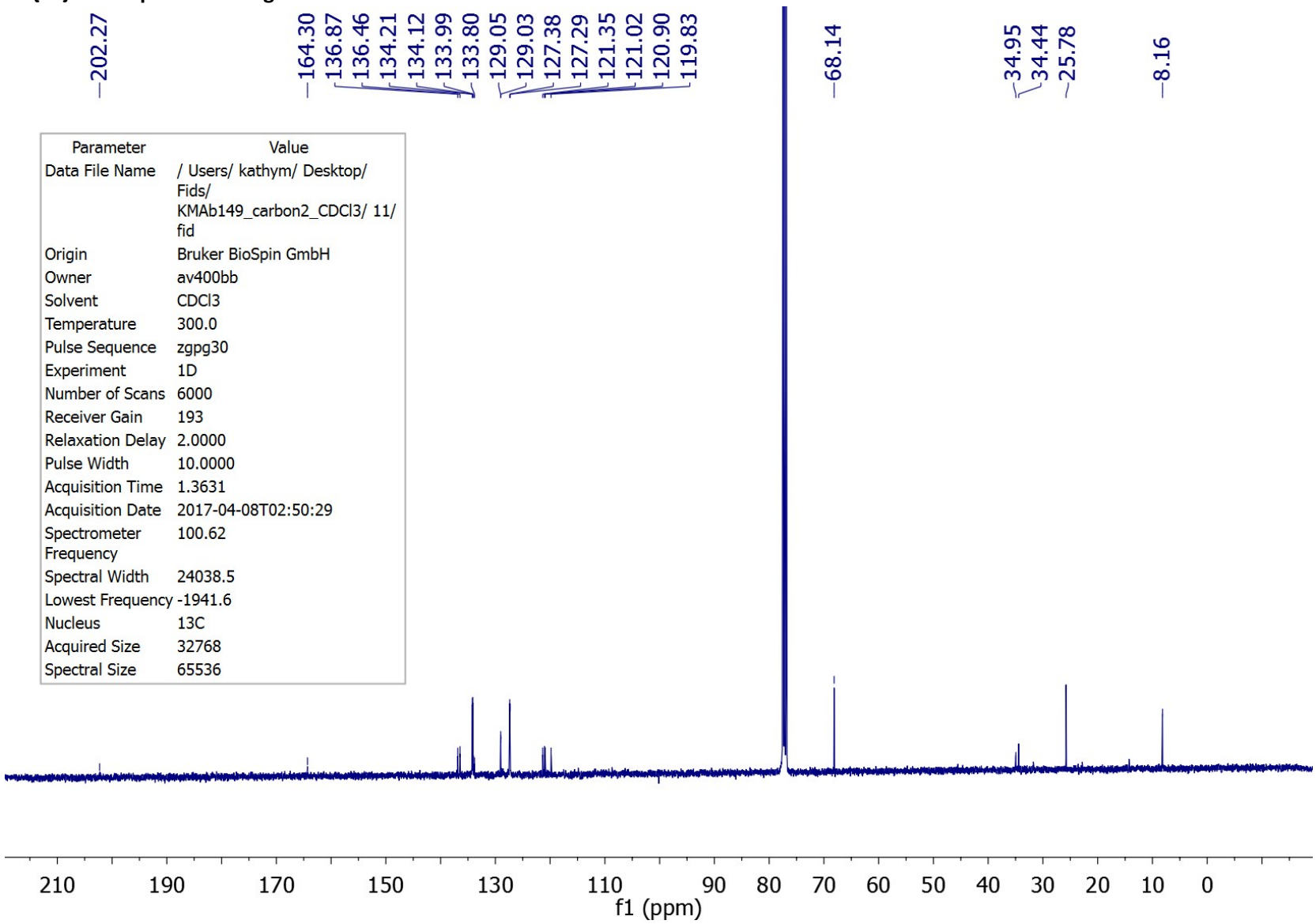


$^{31}P\{^{1}H\}$ NMR Spectrum of 3g

Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb149_char_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.1
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	64
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2017-04-03T12:01:47
Spectrometer Frequency	161.97
Spectral Width	64102.6
Lowest Frequency	-40150.1
Nucleus	31P
Acquired Size	32768
Spectral Size	65536

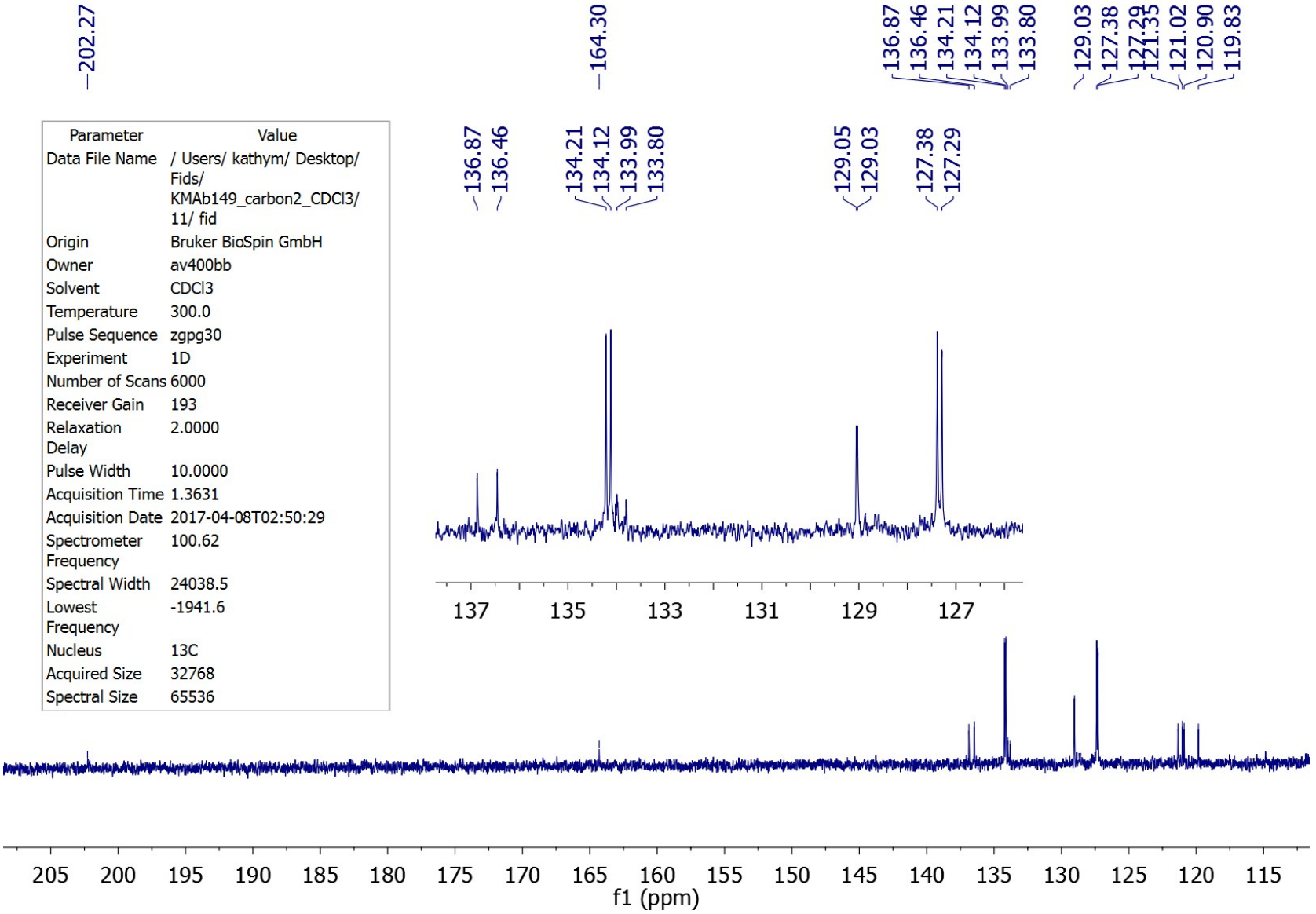


$^{13}C\{^{1}H\}$ NMR Spectrum of 3g



Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb149_carbon2_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	6000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-08T02:50:29
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1941.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

$^{13}C\{^{1}H\}$ NMR Spectrum of 3g (Expansion)



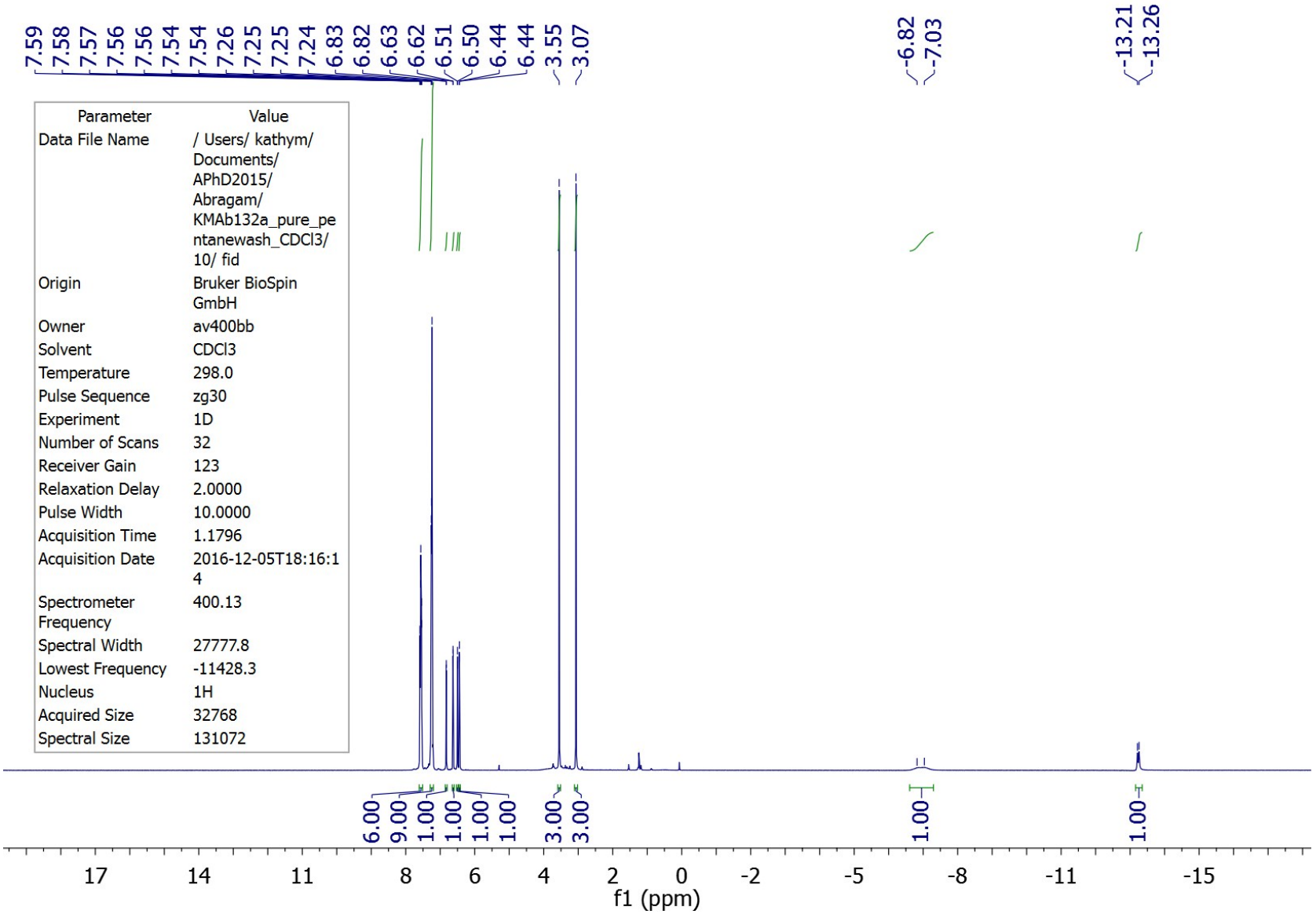
Parameter	Value
Data File Name	/ Users/ kathym/ Desktop/ Fids/ KMAb149_carbon2_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	6000
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-08T02:50:29
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1941.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Characterisation of [Os(H)(CO)(PPh_3){$H_2B(mt)_2$}] (3h)

Commentary

As for **3a**, two weak infrared absorptions may be assigned to the terminal ν_{BH} (KBr: 2398 cm^{-1}) and 3c-2e ν_{BHOs} (2100 cm^{-1}) groups. The anticipated ν_{OsH} absorption appeared as a shoulder (1949 cm^{-1}) on the more intense ν_{CO} absorption (1907 cm^{-1}). The ^1H NMR spectrum includes a broad (h.h.w. = 0.44 ppm) resonance centred at –7.02 ppm due to the B–H–Os interaction in addition to a doublet at –13.23 ppm ($^2J_{PH}$ = 18.2 Hz) due to the terminal osmium hydride.

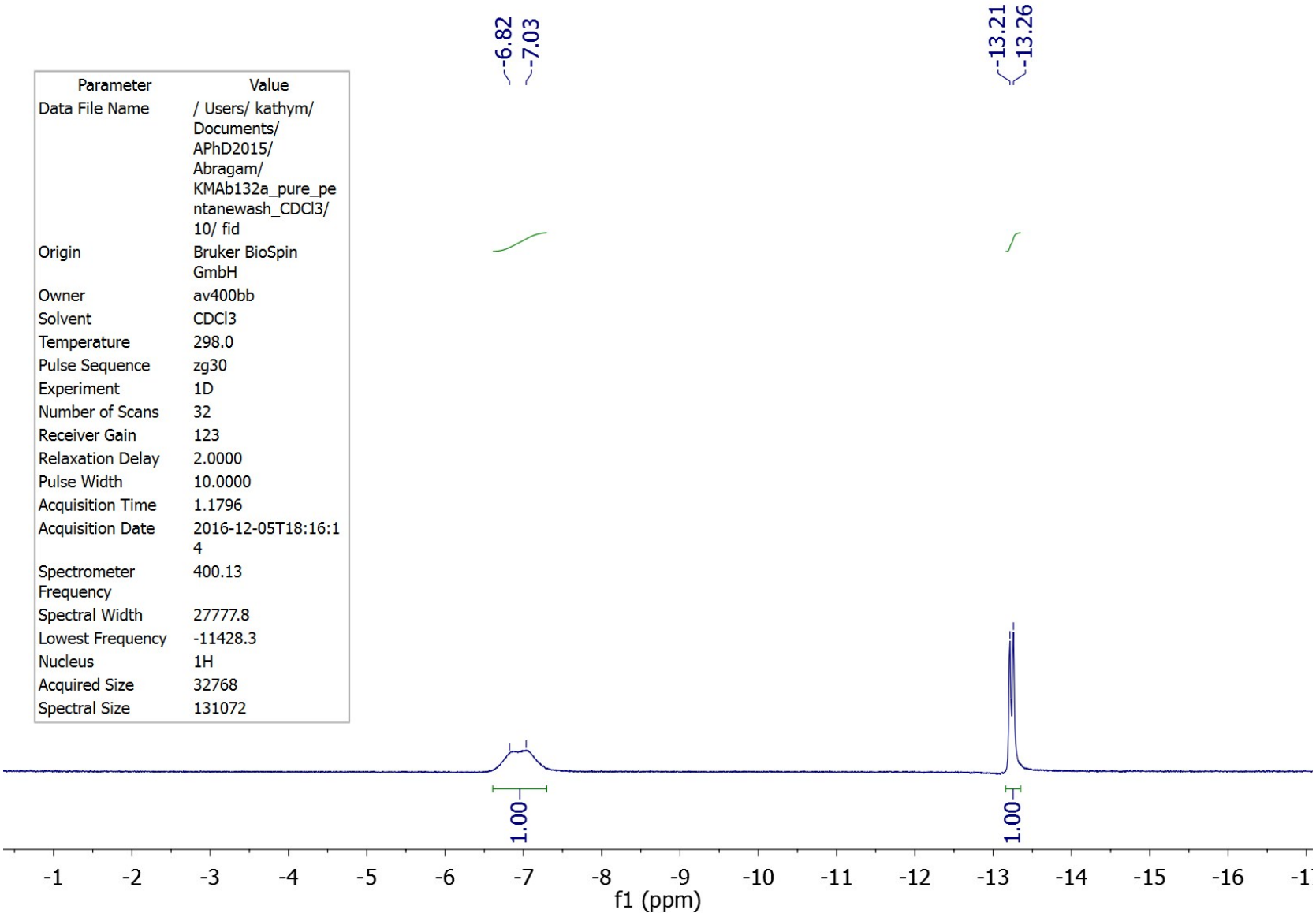
^{1}H NMR Spectrum of 3h



Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb132a_pure_pentanewash_CDCl3/ 10/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	123
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1796
Acquisition Date	2016-12-05T18:16:14
Spectrometer Frequency	400.13
Spectral Width	27777.8
Lowest Frequency	-11428.3
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

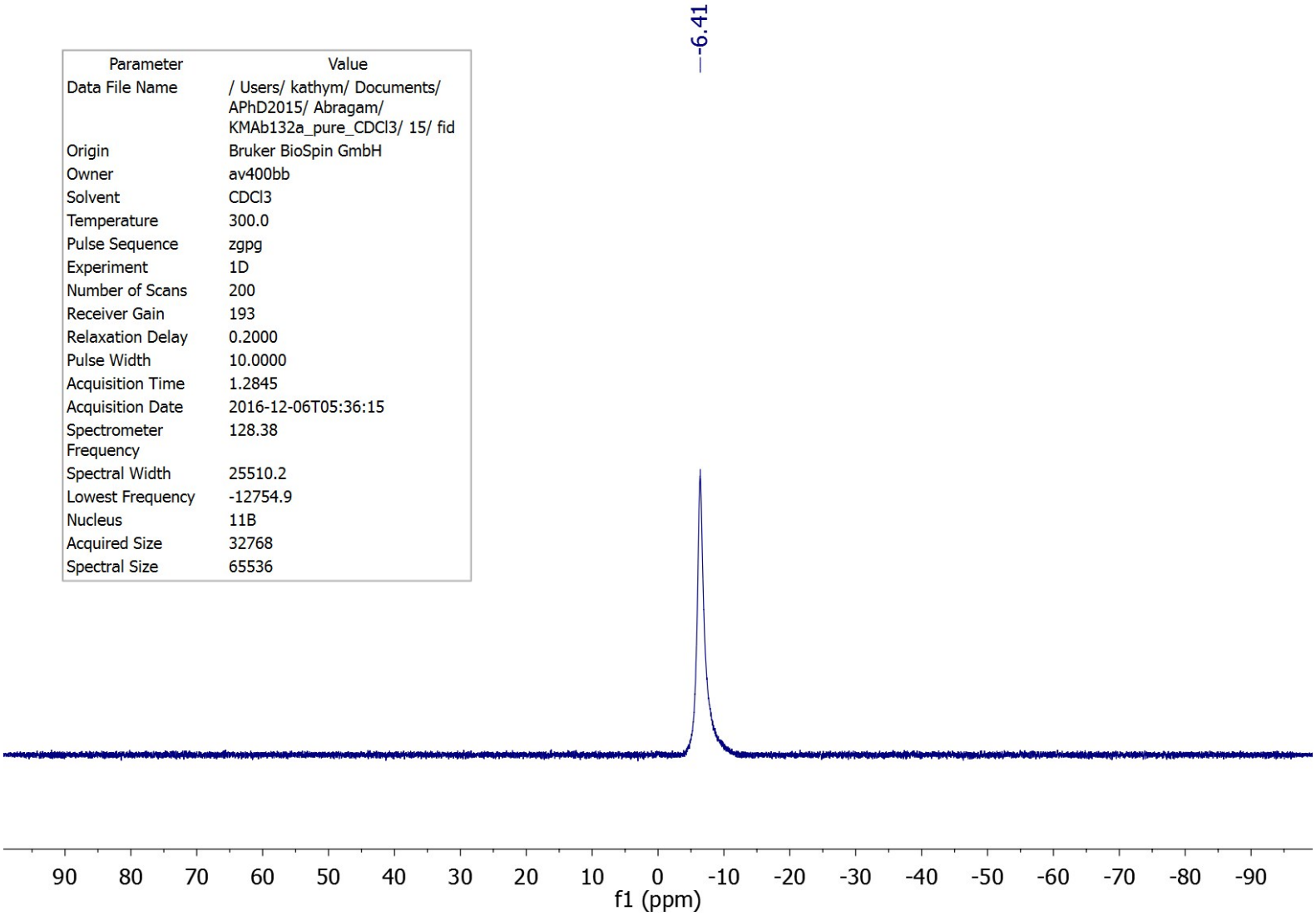
^{1}H NMR Spectrum of 3h (Expansion)

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb132a_pure_pentanewash_CDCl3/ 10/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	123
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1796
Acquisition Date	2016-12-05T18:16:14
Spectrometer Frequency	400.13
Spectral Width	27777.8
Lowest Frequency	-11428.3
Nucleus	1H
Acquired Size	32768
Spectral Size	131072



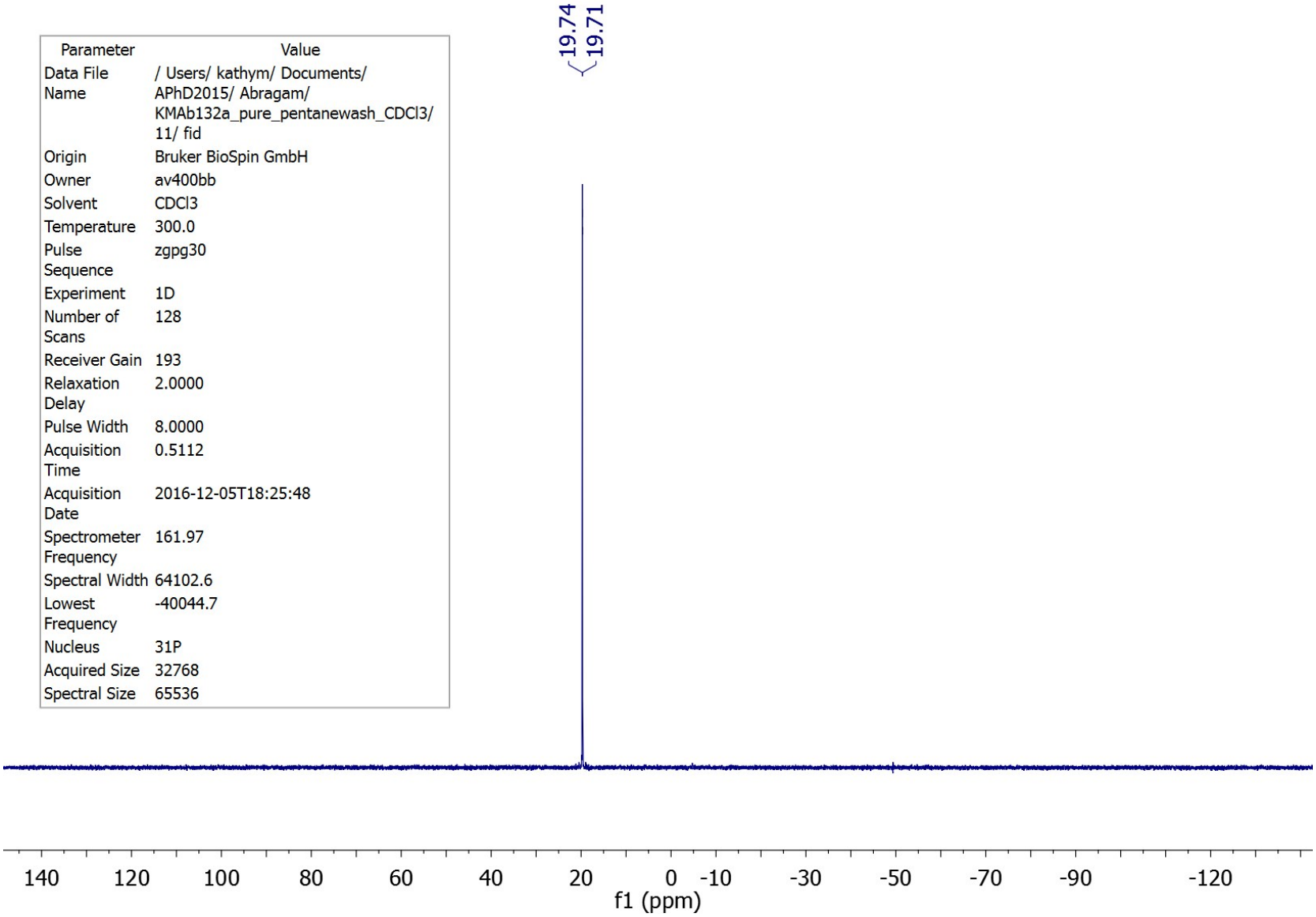
$^{11}B\{^{1}H\}$ NMR Spectrum of 3h

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb132a_pure_CDCl3/ 15/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg
Experiment	1D
Number of Scans	200
Receiver Gain	193
Relaxation Delay	0.2000
Pulse Width	10.0000
Acquisition Time	1.2845
Acquisition Date	2016-12-06T05:36:15
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12754.9
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

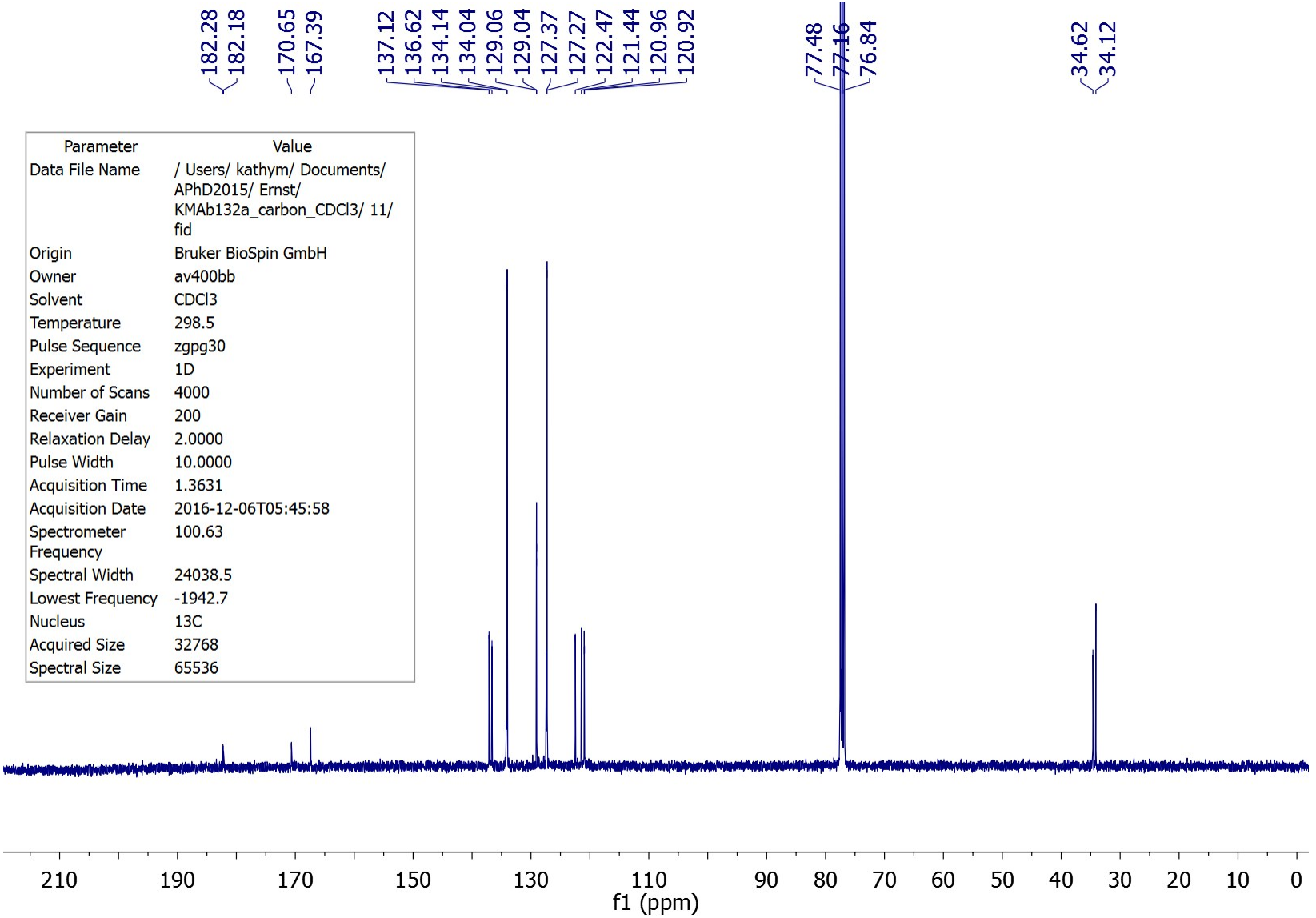


$^{31}P\{^{1}H\}$ NMR Spectrum of 3h

Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Abragam/ KMAb132a_pure_pentanewash_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	193
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2016-12-05T18:25:48
Spectrometer Frequency	161.97
Spectral Width	64102.6
Lowest Frequency	-40044.7
Nucleus	31P
Acquired Size	32768
Spectral Size	65536

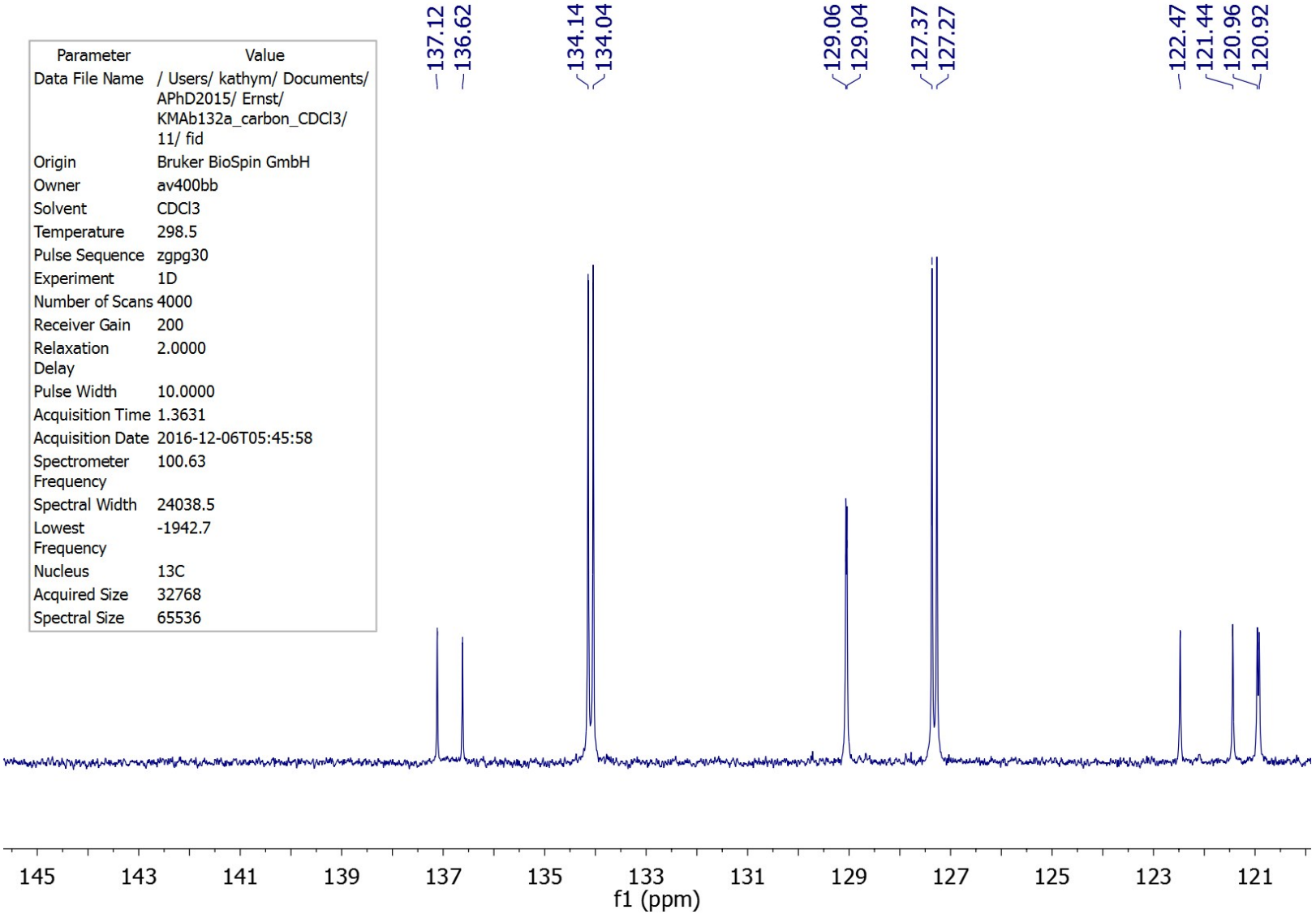


$^{13}C\{^{1}H\}$ NMR Spectrum of 3h



Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Ernst/ KMAb132a_carbon_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	298.5
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	4000
Receiver Gain	200
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-06T05:45:58
Spectrometer Frequency	100.63
Spectral Width	24038.5
Lowest Frequency	-1942.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

$^{13}C\{^{1}H\}$ NMR Spectrum of 3h (Expansion)



Parameter	Value
Data File Name	/ Users/ kathym/ Documents/ APhD2015/ Ernst/ KMAb132a_carbon_CDCl3/ 11/ fid
Origin	Bruker BioSpin GmbH
Owner	av400bb
Solvent	CDCl3
Temperature	298.5
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	4000
Receiver Gain	200
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-06T05:45:58
Spectrometer Frequency	100.63
Spectral Width	24038.5
Lowest Frequency	-1942.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536