

KO^tBu-Mediated annulation of acetonitrile with aldehyde: synthesis of substituted dihydropyridin-2(1*H*)-ones, pyridin-2(1*H*)-ones, and thiopyridin-2(1*H*)-ones

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pyridin-2(1 <i>H</i>)-ones, and thiopyridin-2(1 <i>H</i>)-ones	S2-S164

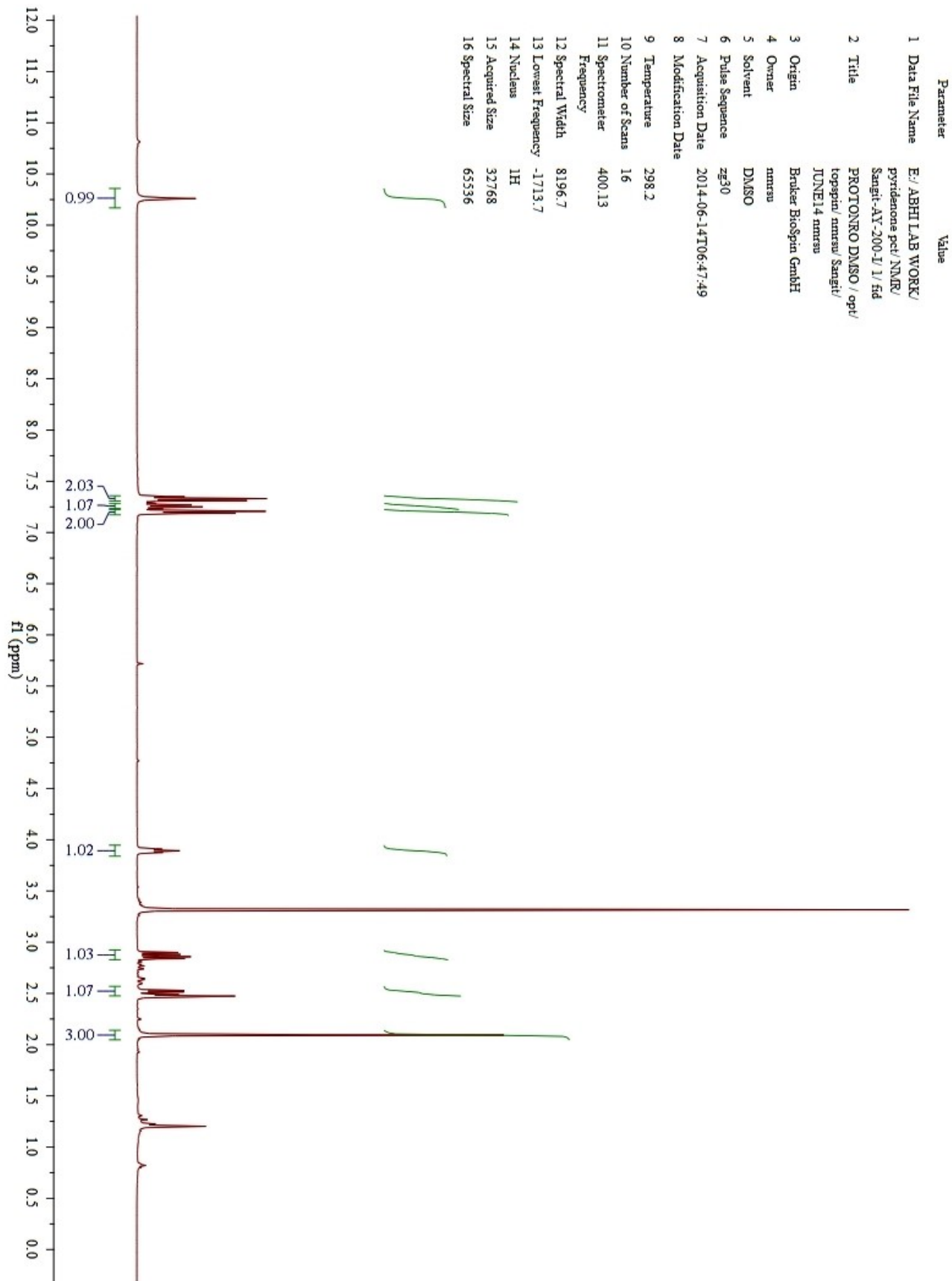
Figure S1 ^1H NMR spectra of **1**

Figure S2 ^{13}C NMR spectra of **1**

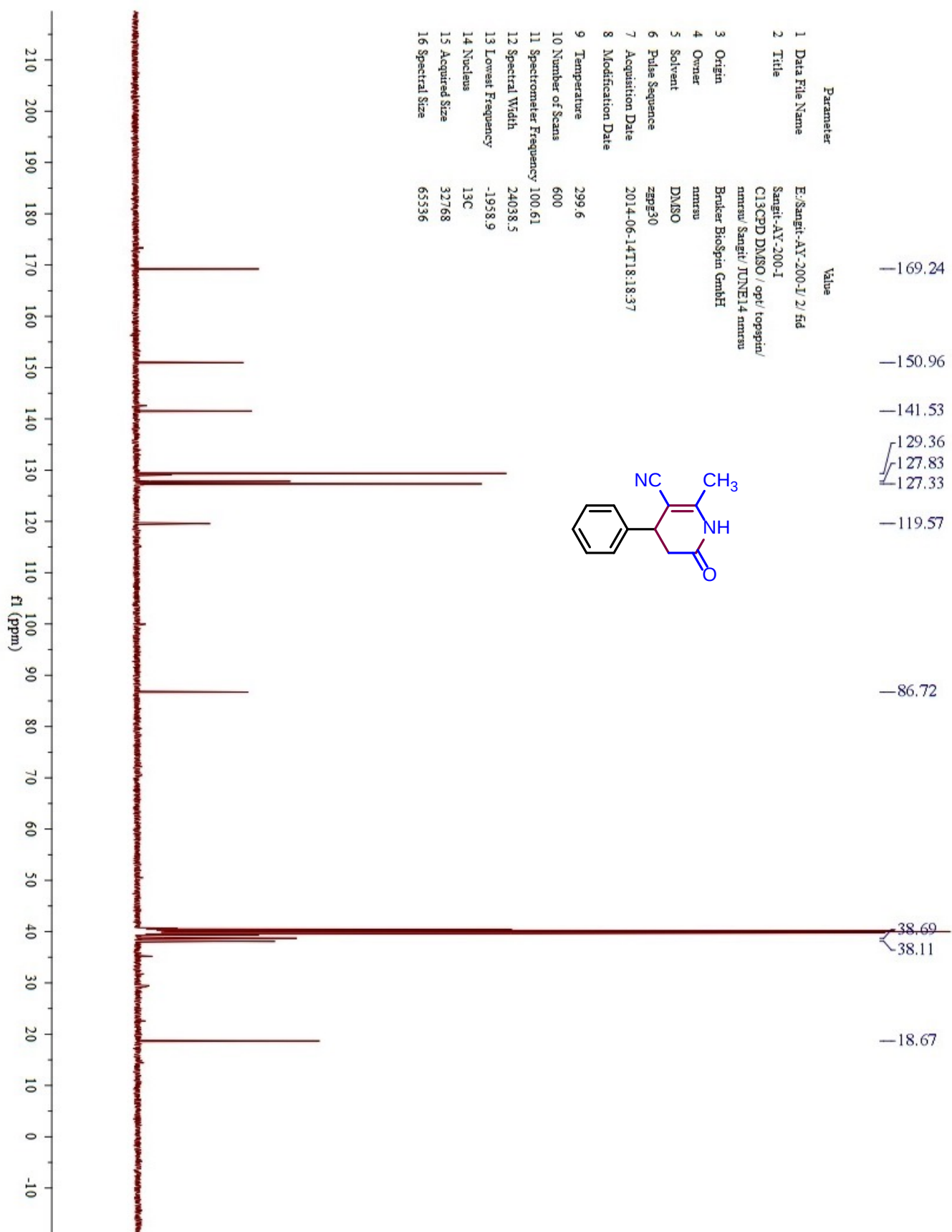


Figure S3 135-DEPT spectra NMR of 1

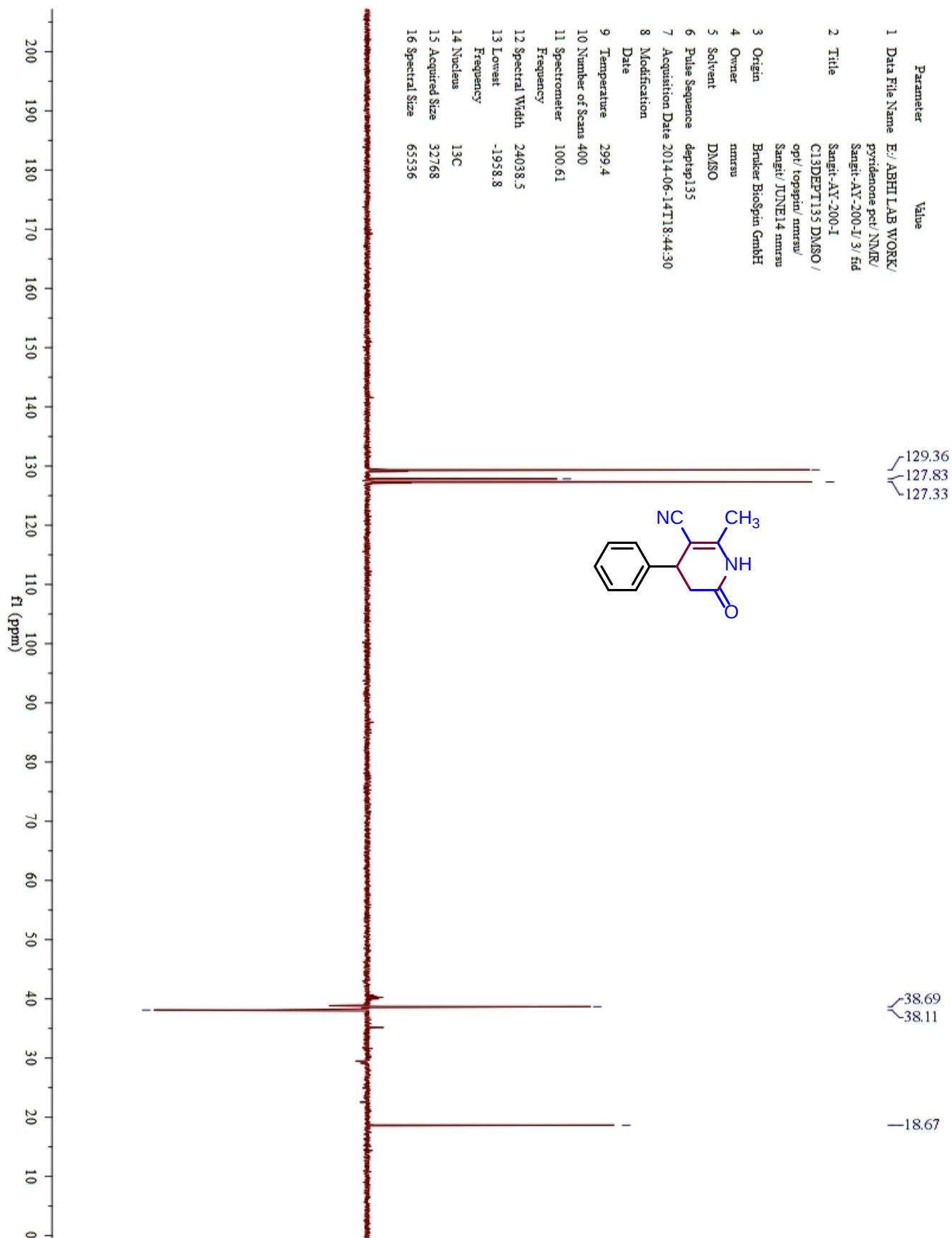


Figure S4 HRMS spectra of **1**

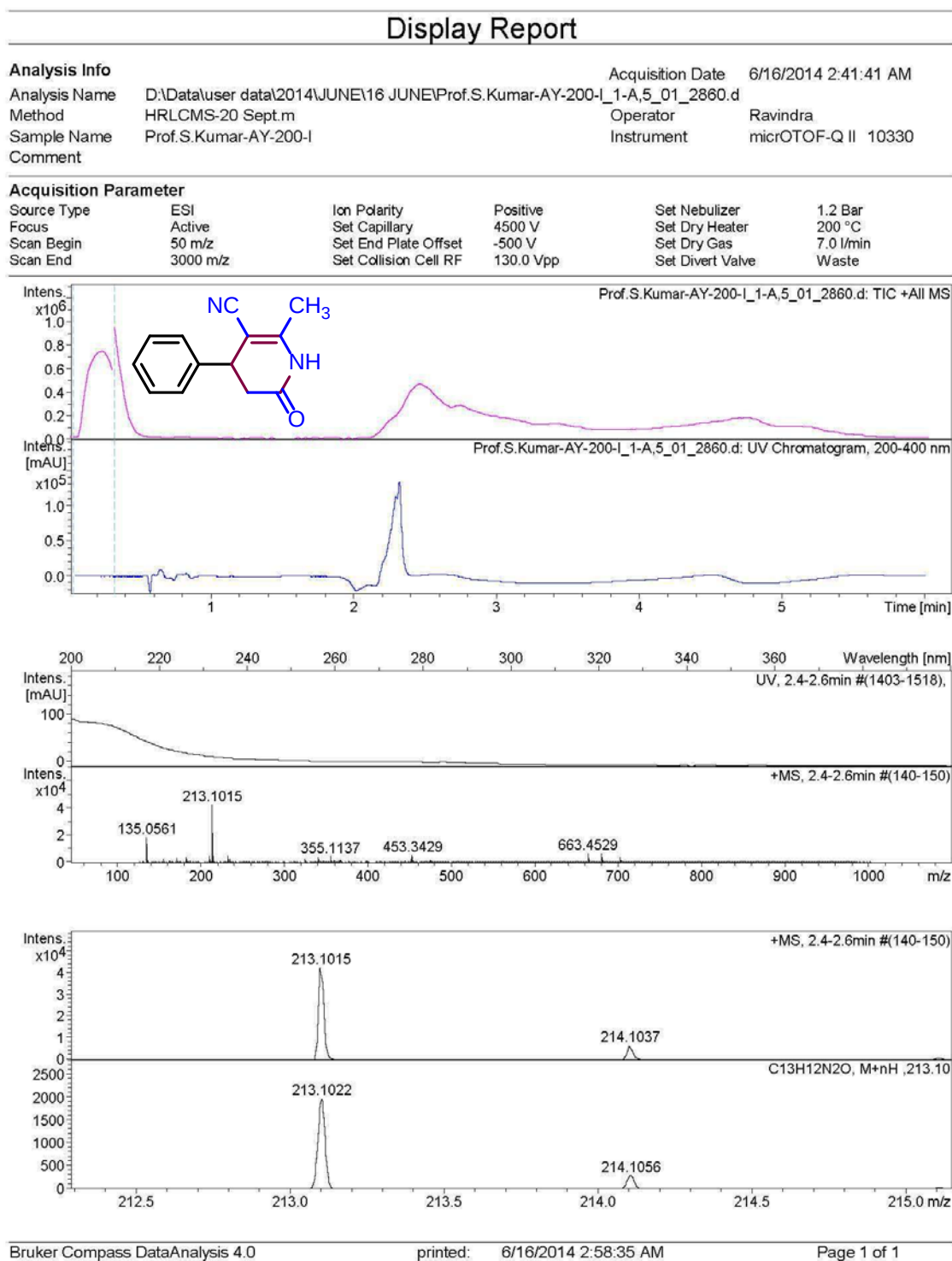


Figure S5 ^1H NMR spectra of **3**

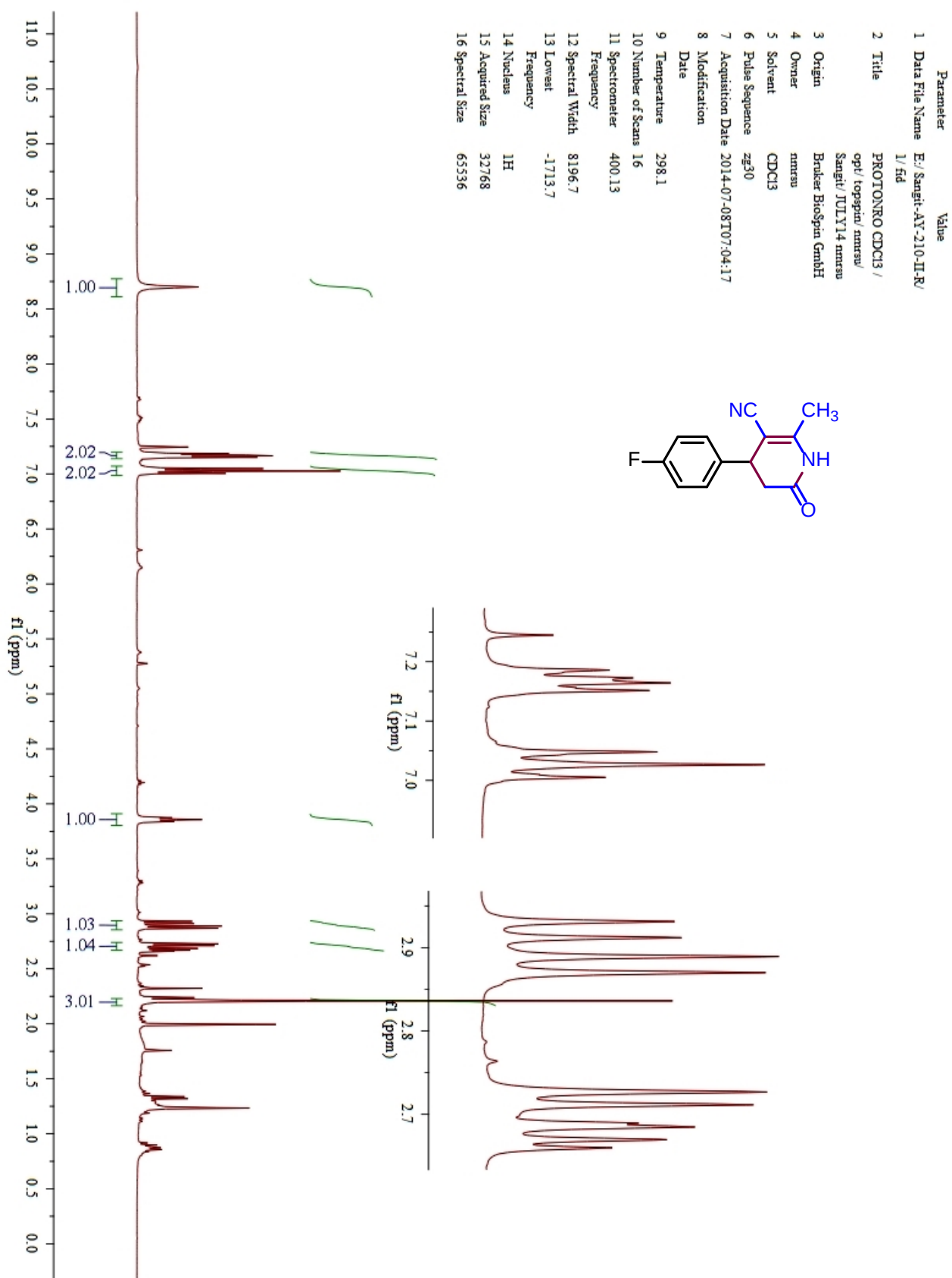


Figure S6 ¹³C NMR spectra of 3

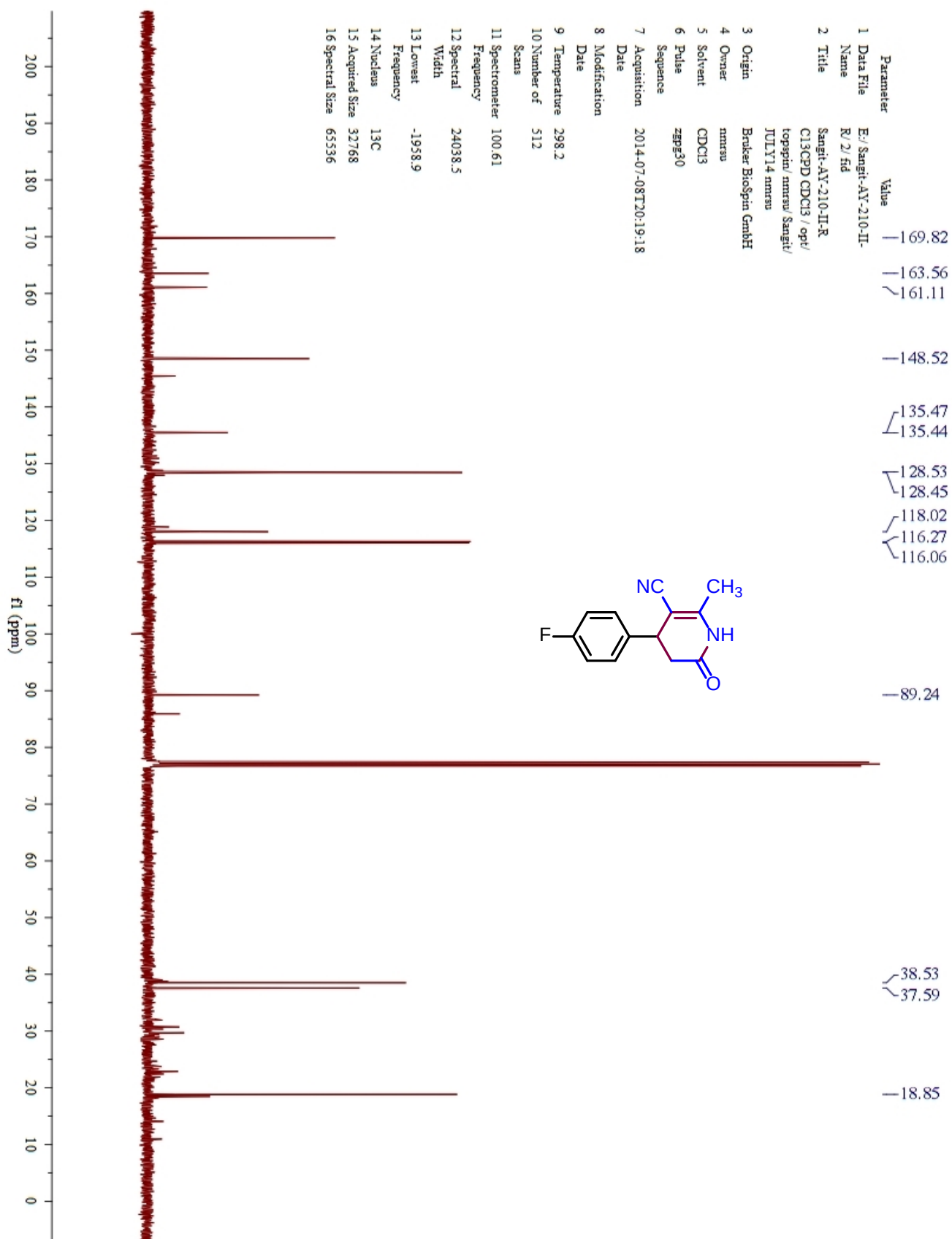


Figure S7 HRMS spectra of **3**

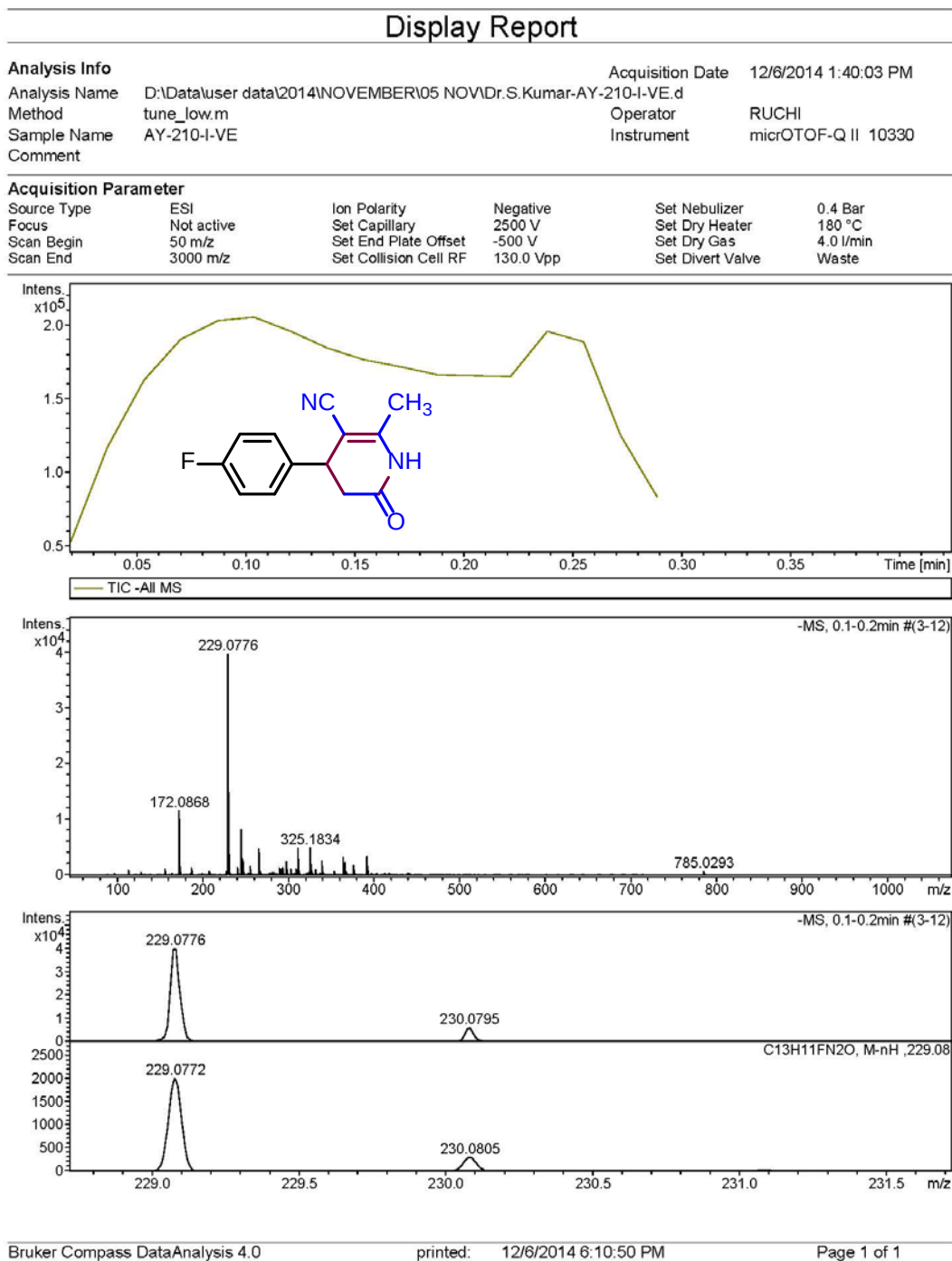


Figure S8 ^1H NMR spectra of **4**

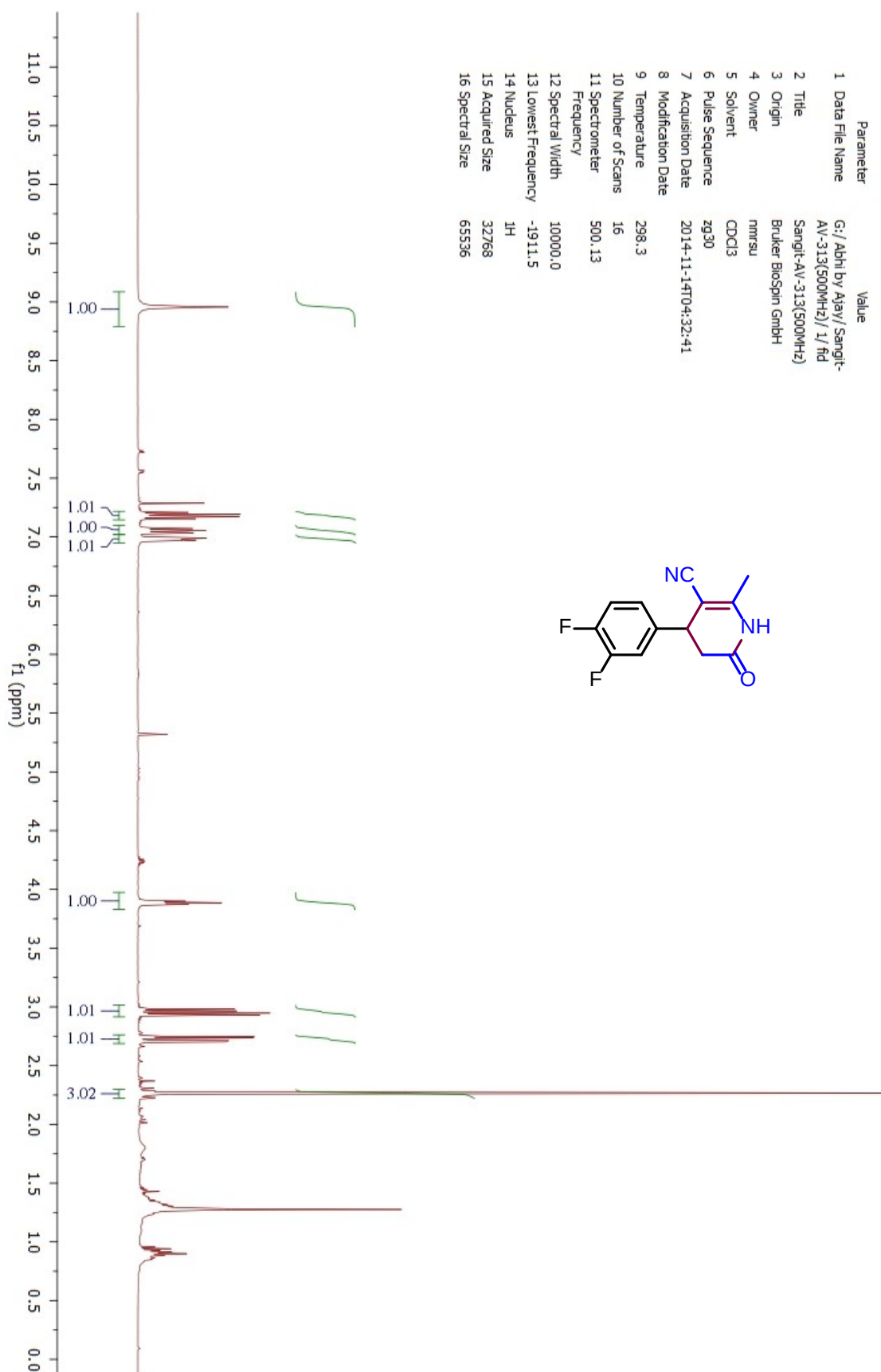


Figure S9 ^{13}C NMR spectra of **4**

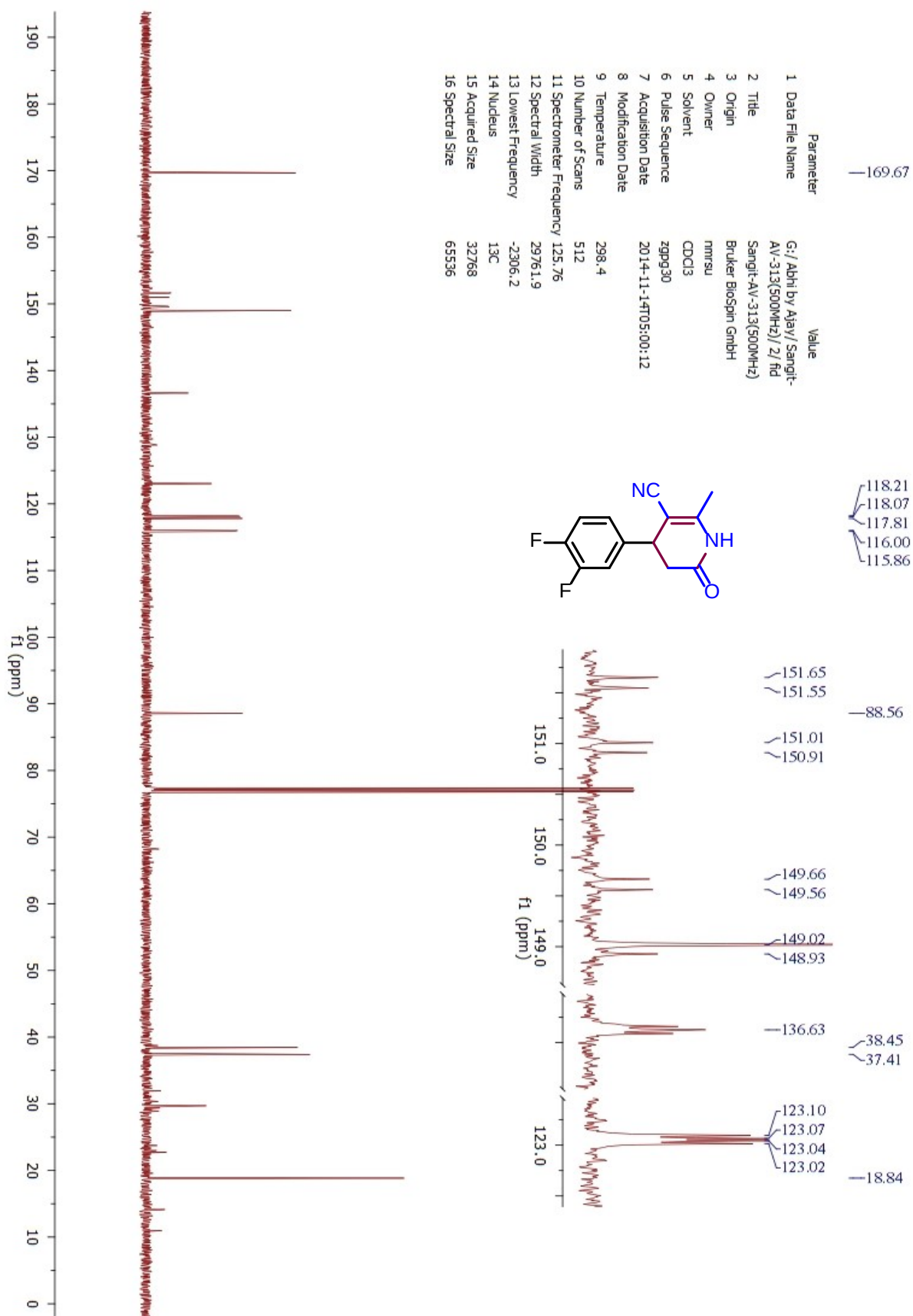


Figure S10 HRMS spectra of **4**

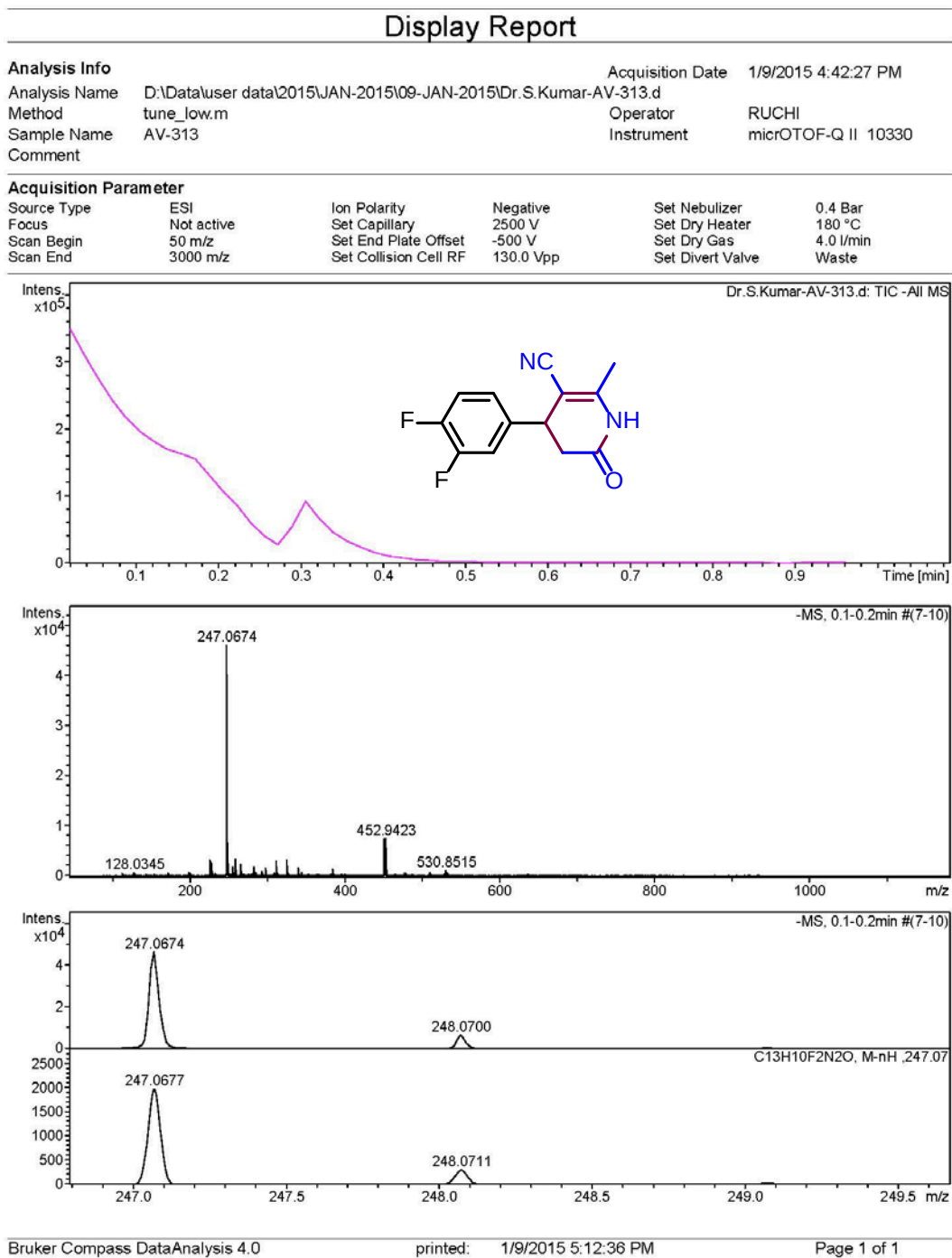


Figure S11 ¹H NMR spectra of 5

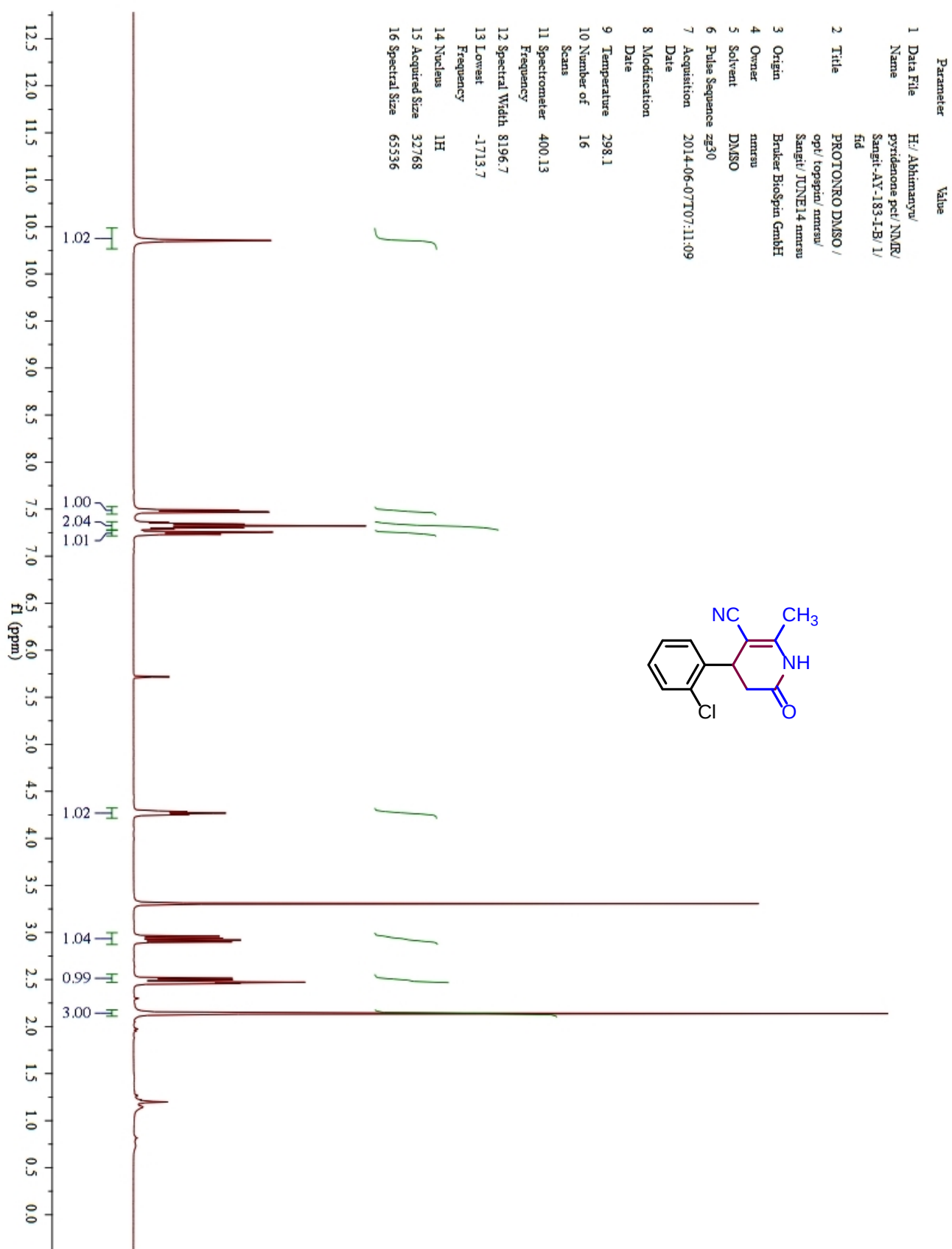


Figure S12 ^{13}C NMR spectra of 5

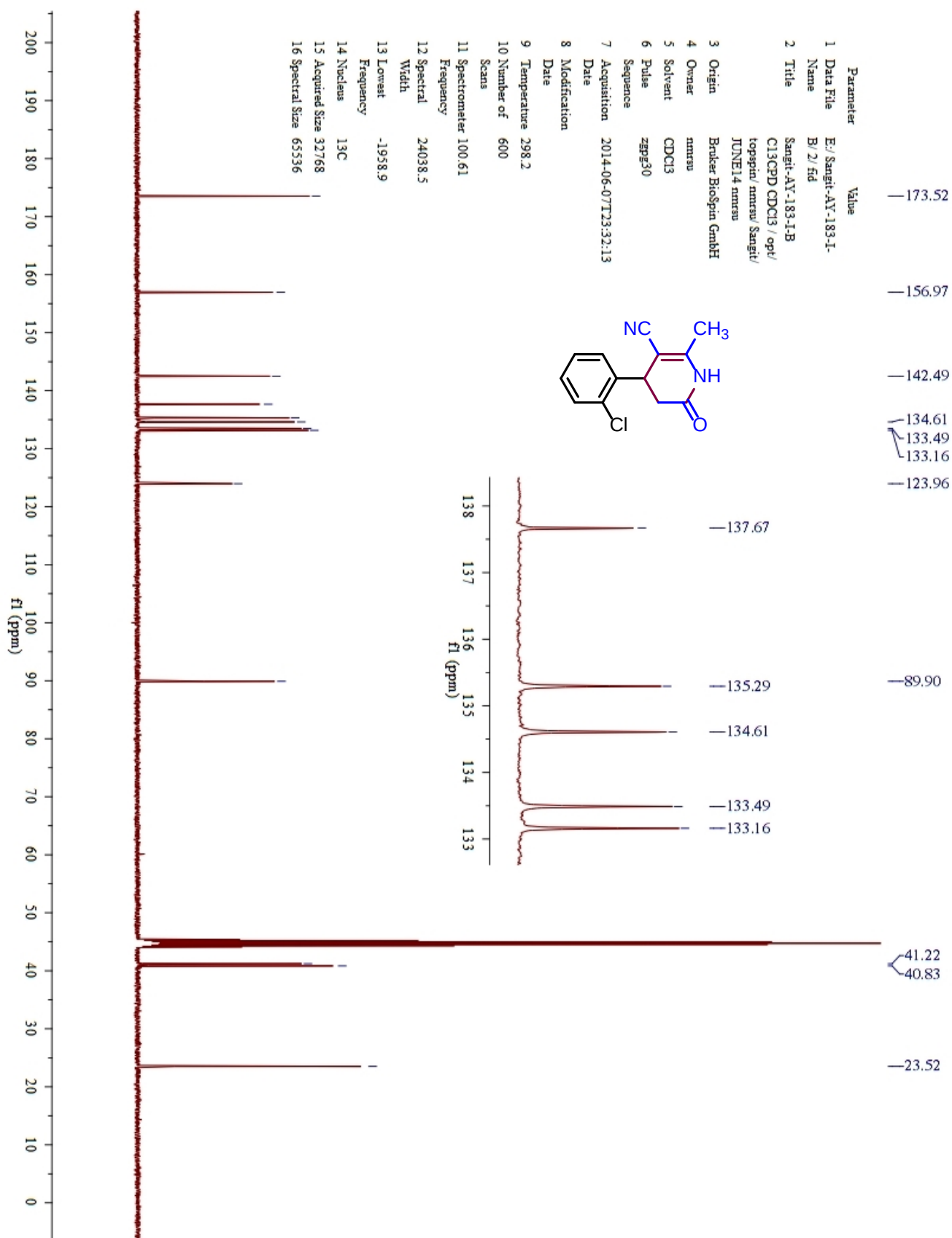


Figure S13 135-DEPT NMR spectra of 5

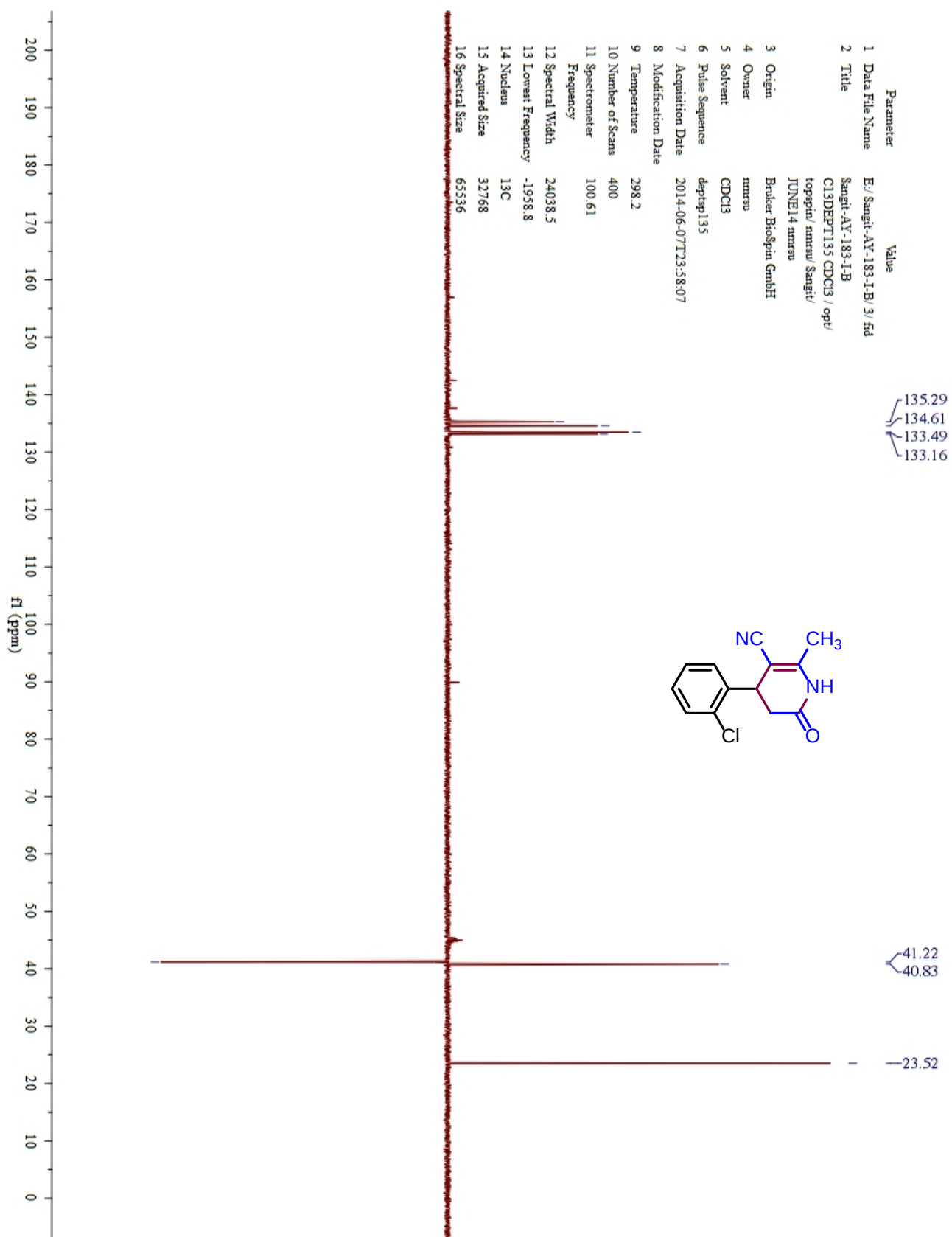


Figure S14 GCMS spectra of 5

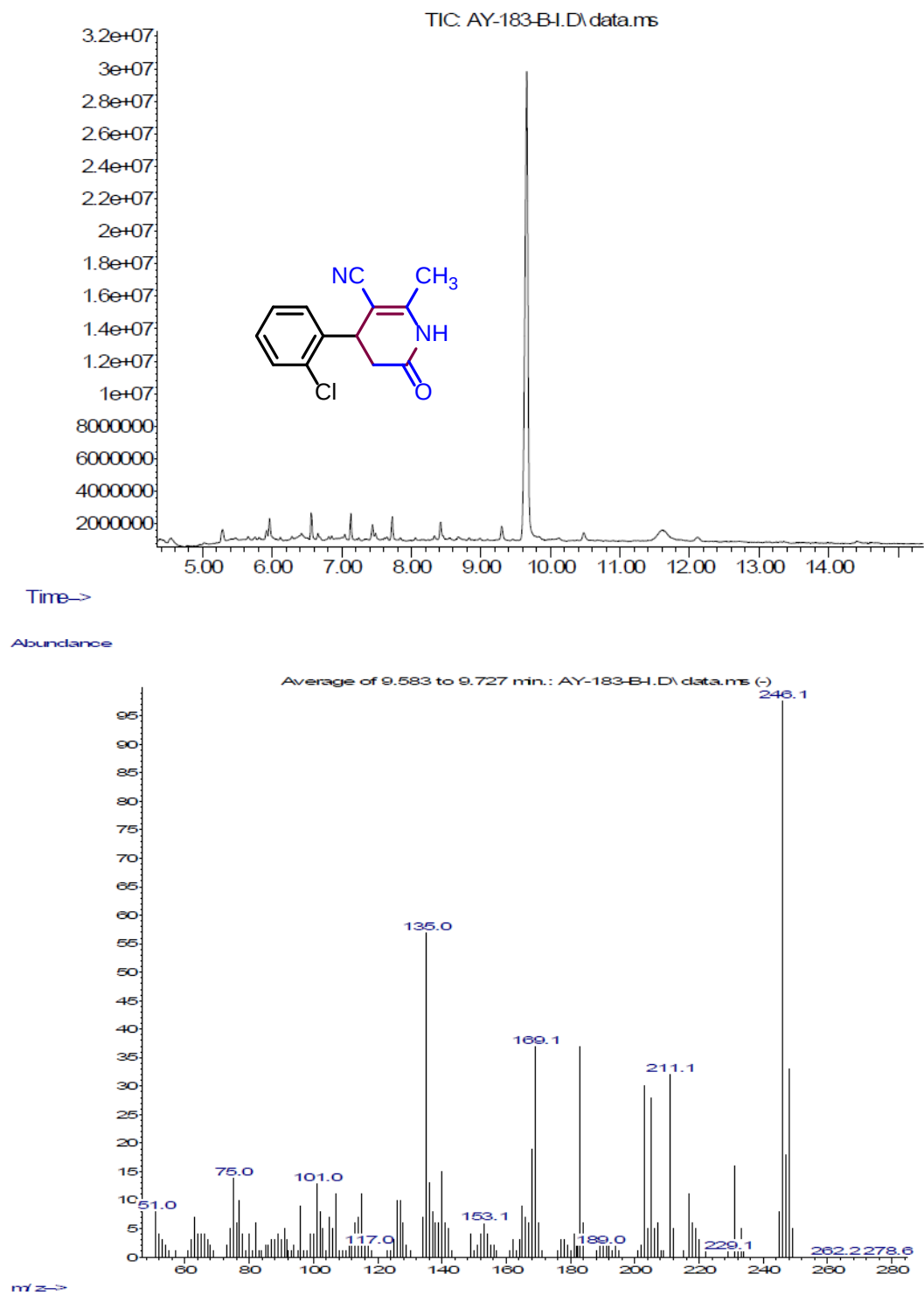


Figure S15 ¹H NMR spectra of **6**

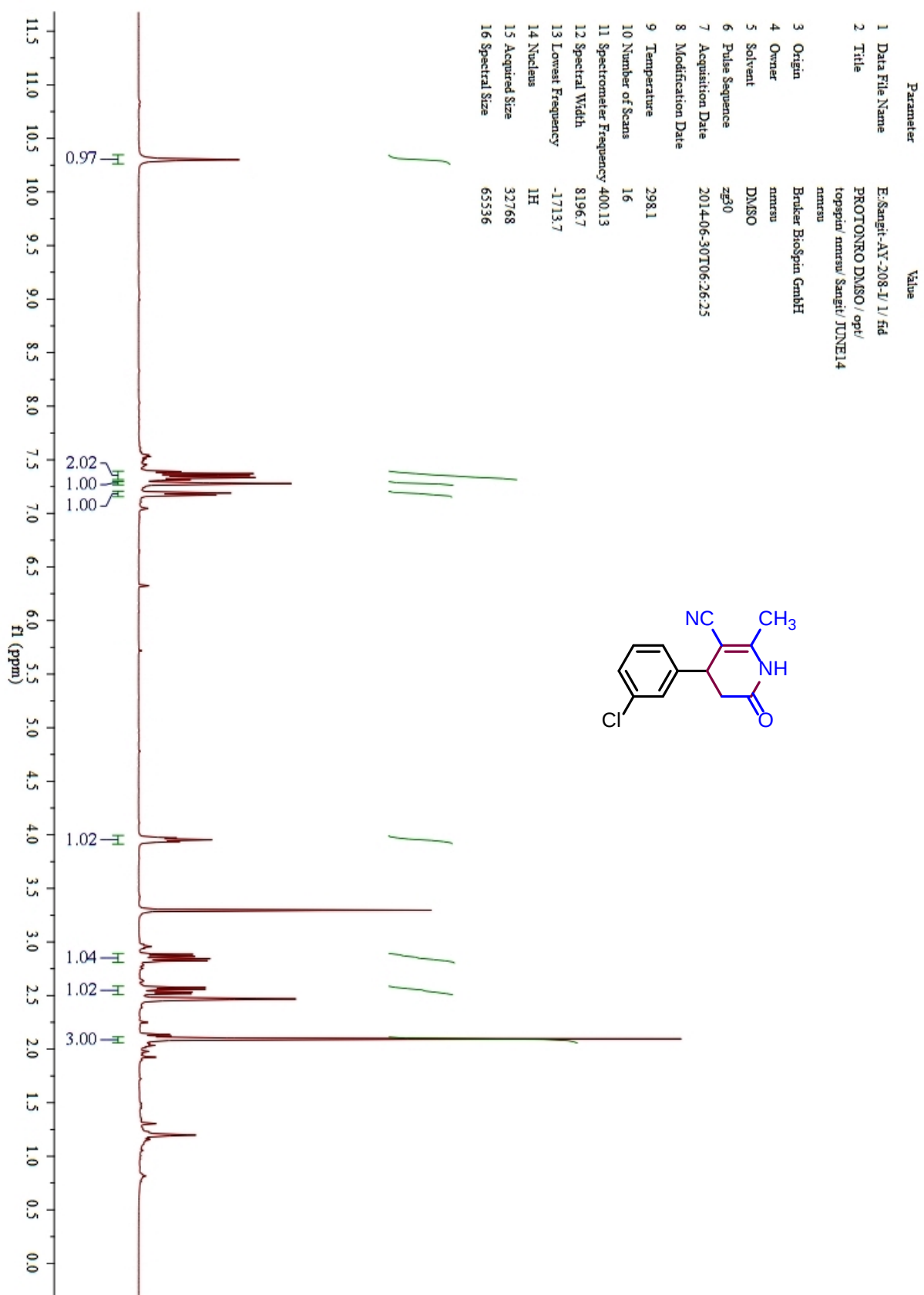


Figure S16 ¹³C NMR spectra of 6

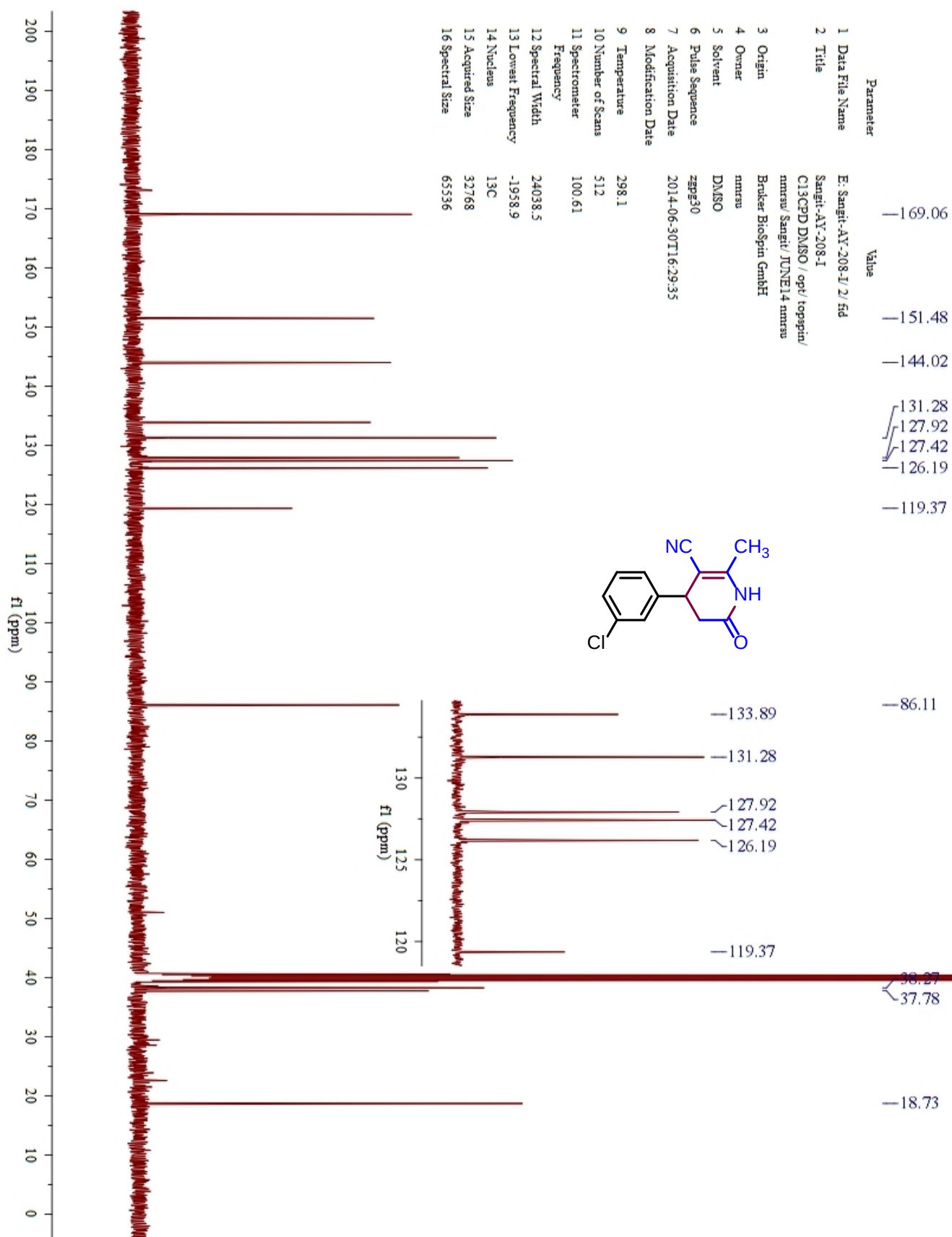


Figure S17 HRMS spectra of **6**

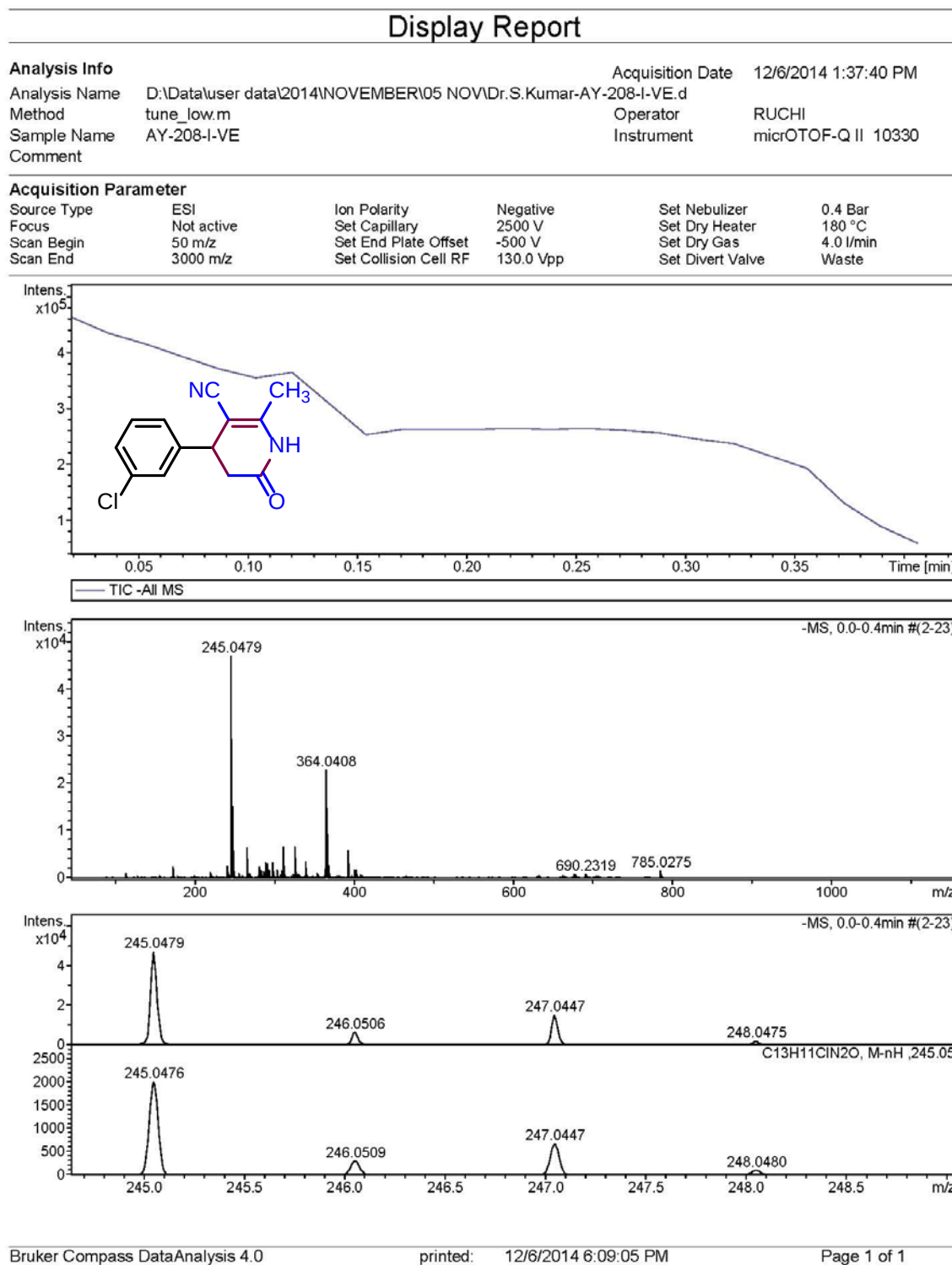


Figure S18 ^1H NMR spectra of 7

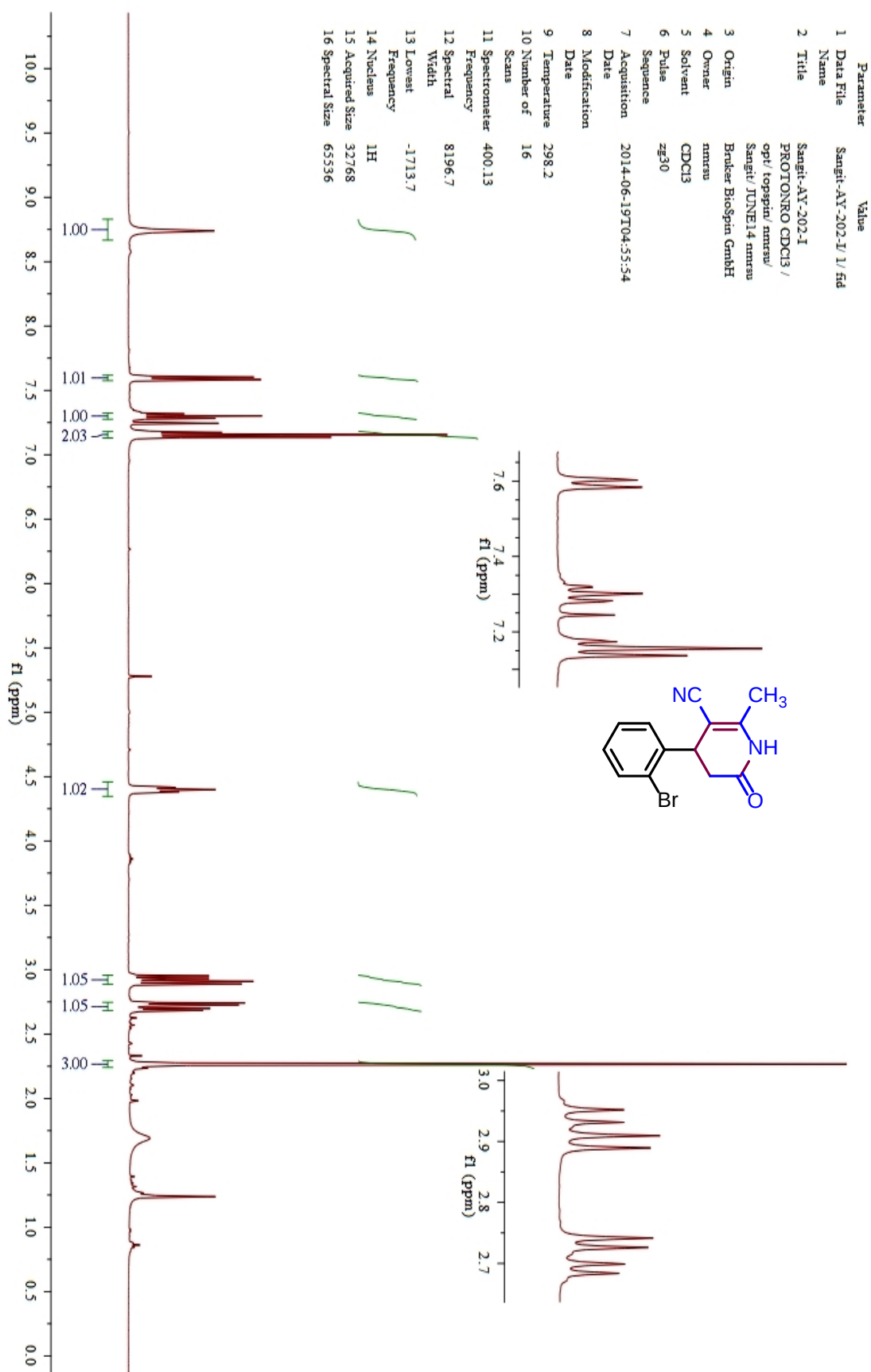


Figure S19 ^{13}C NMR spectra of 7

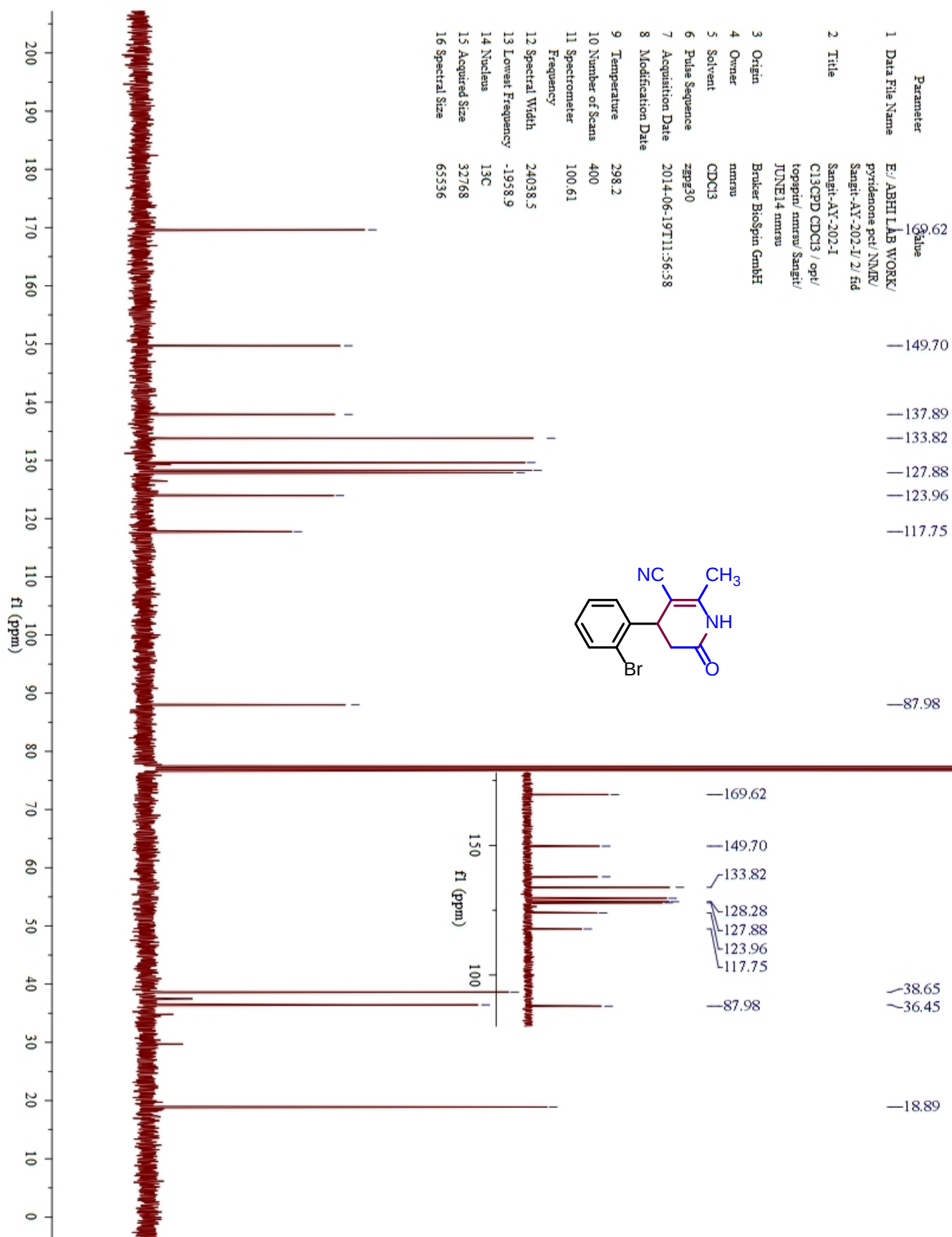


Figure S20 DEPT-135NMR spectra of 7

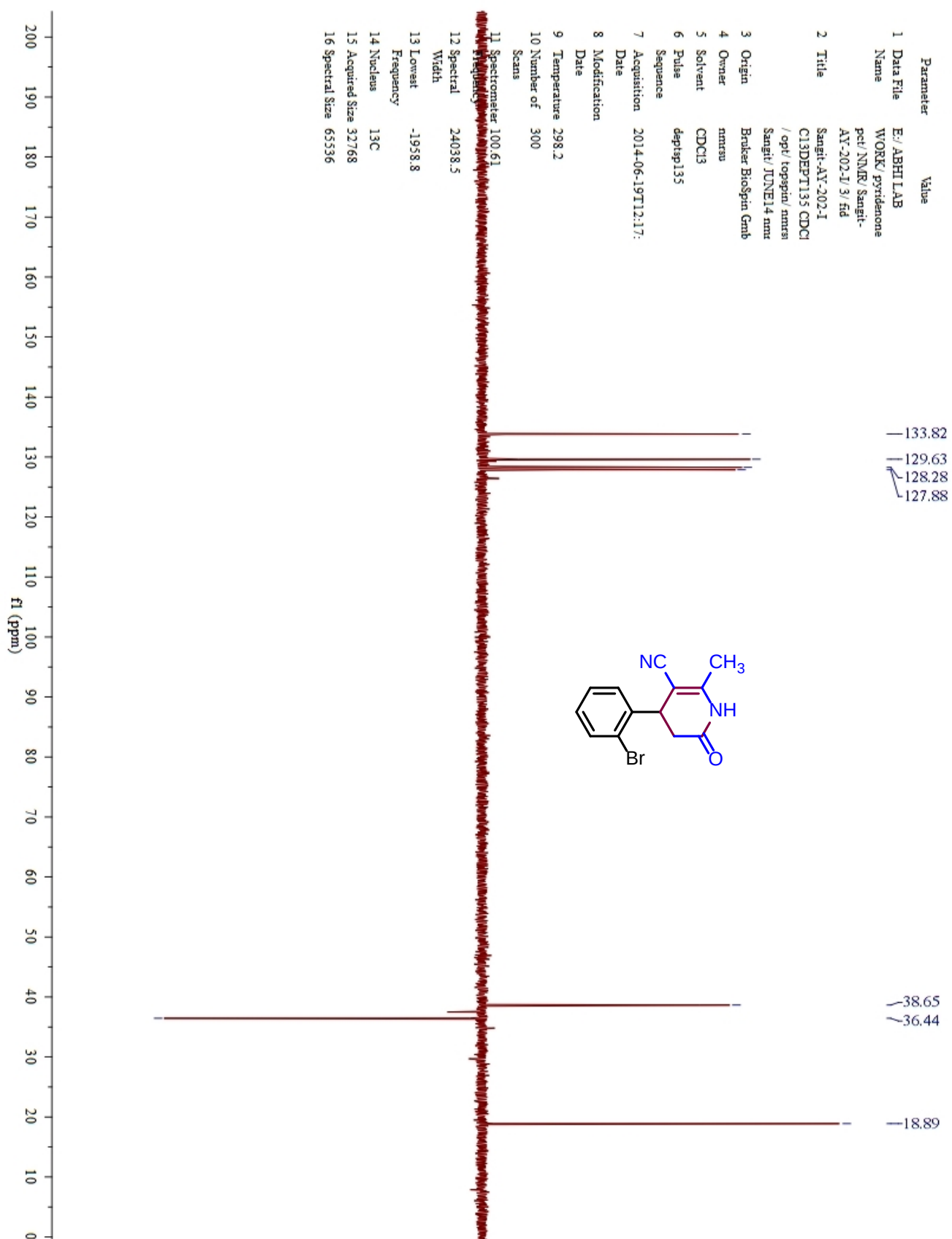


Figure S21 Mass spectra of 7

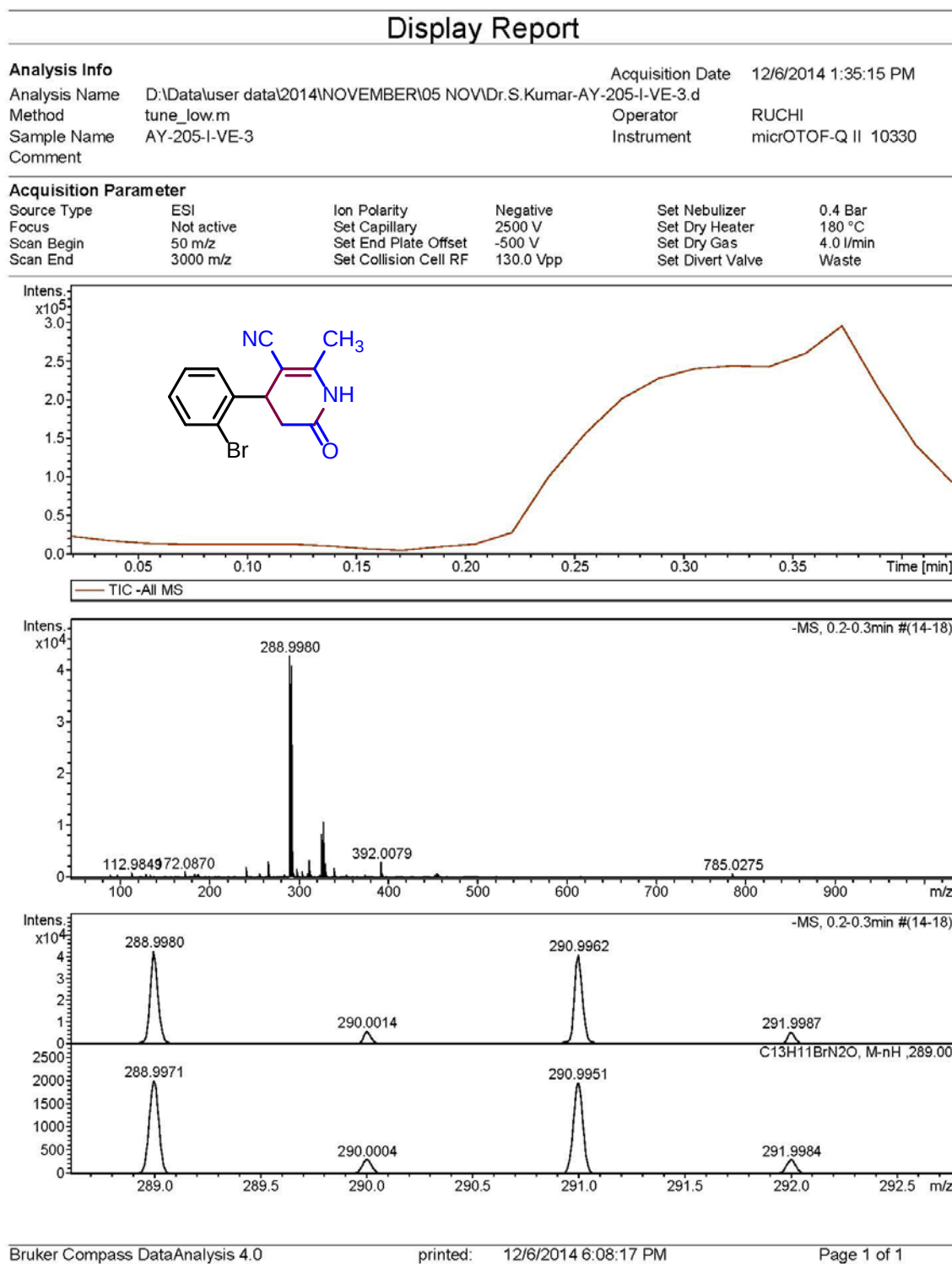


Figure S22 ^1H NMR spectra of **8**

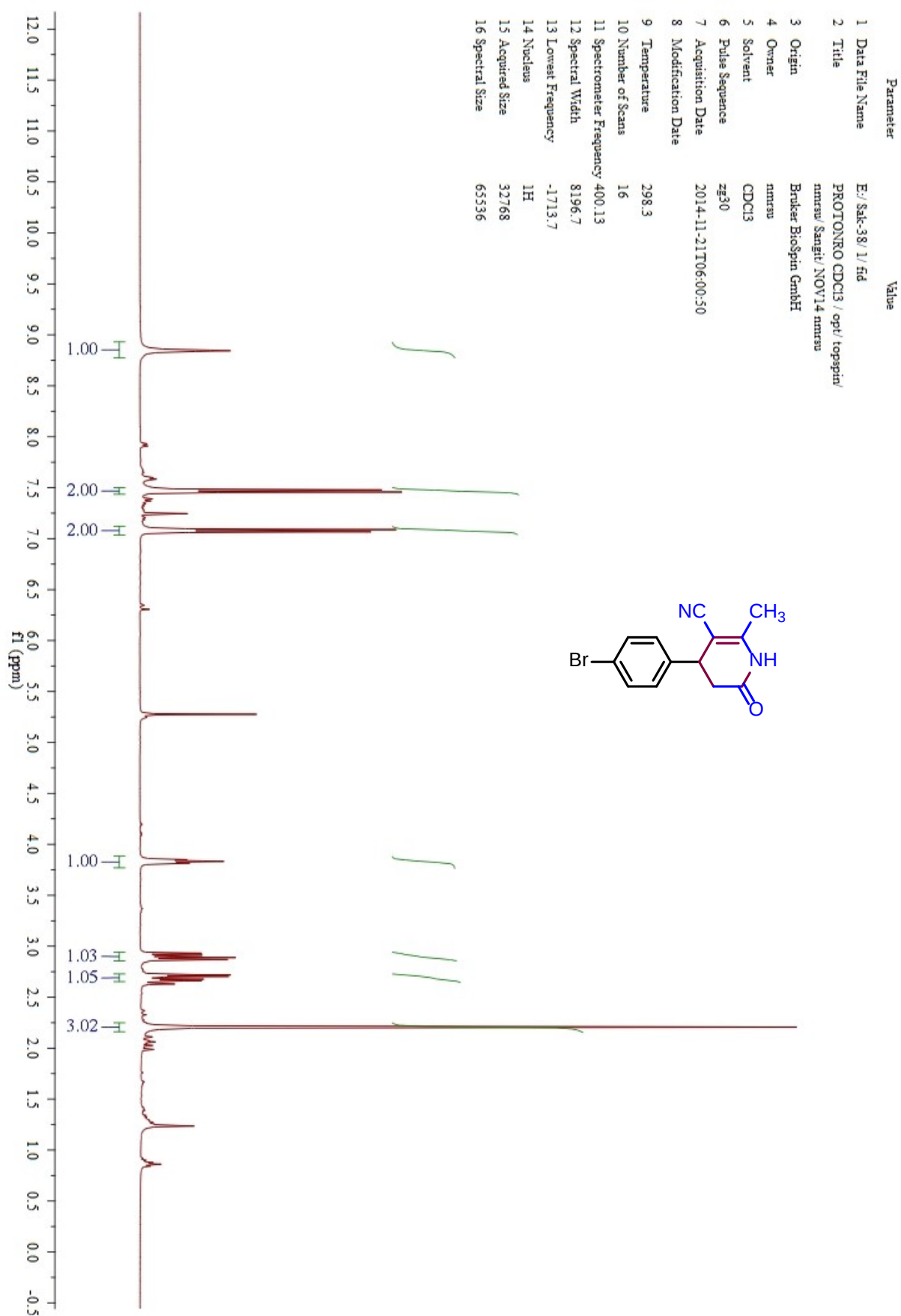
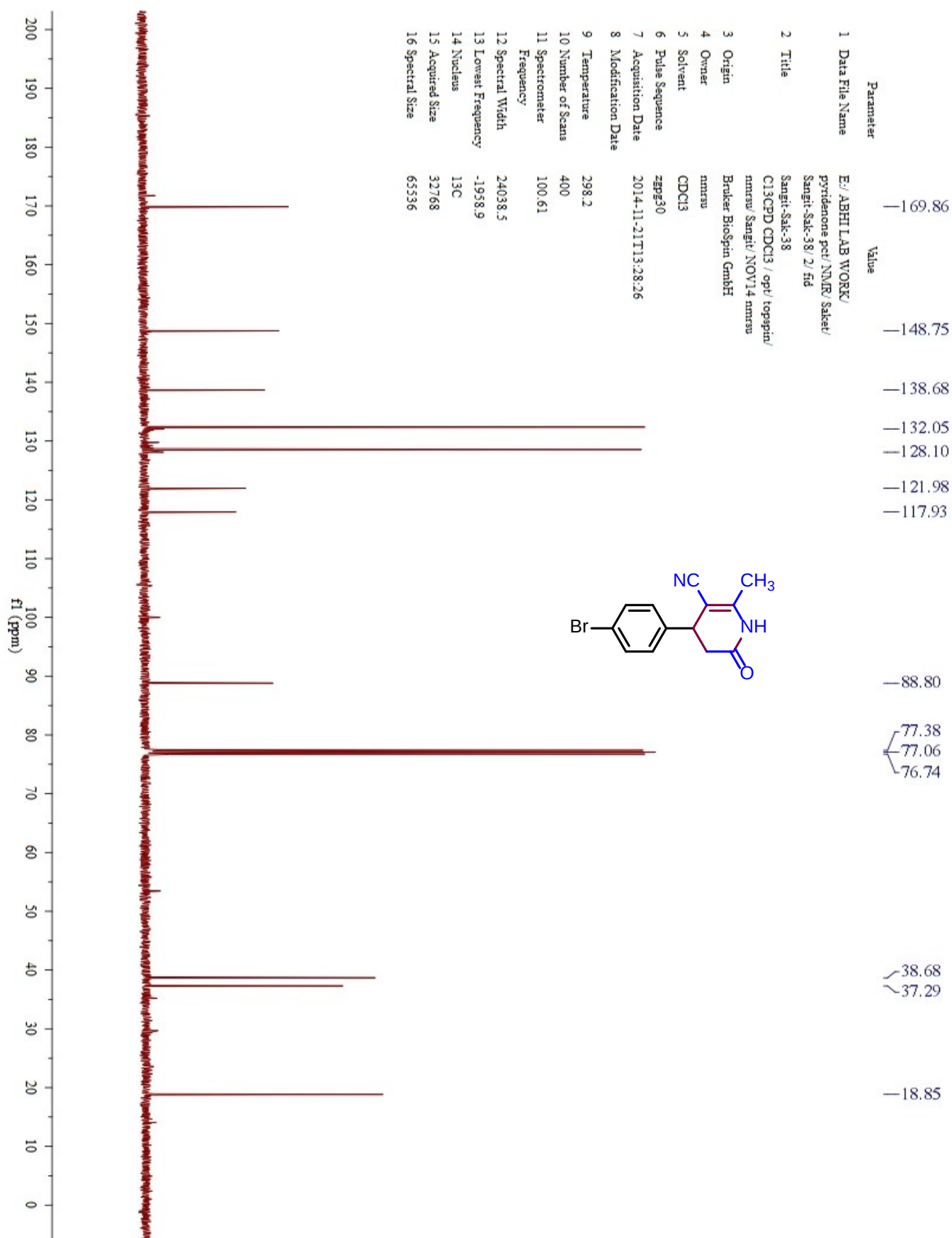


Figure S23 ^{13}C NMR spectra of **8**



S25

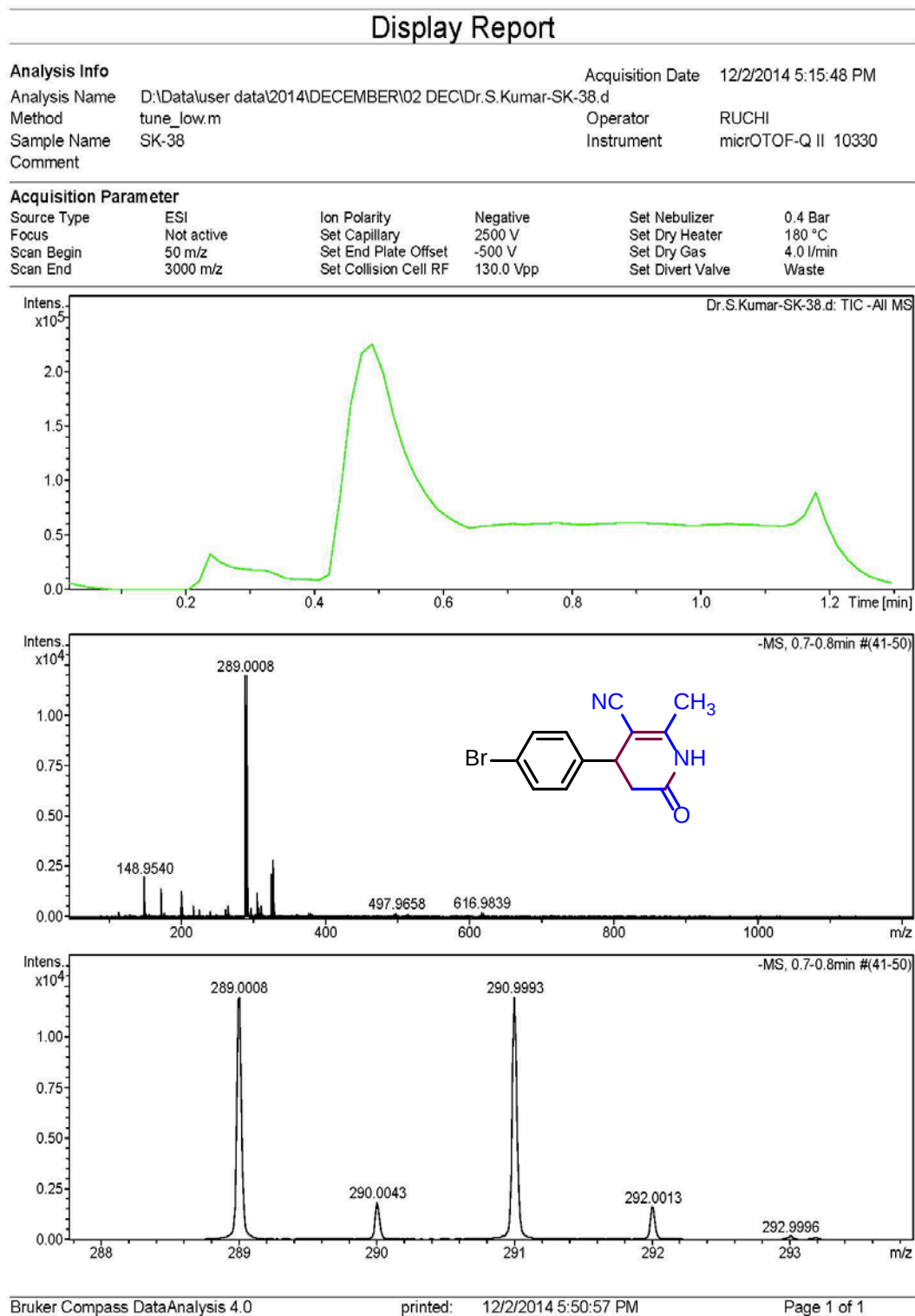


Figure S25 ¹H NMR spectra of **9**

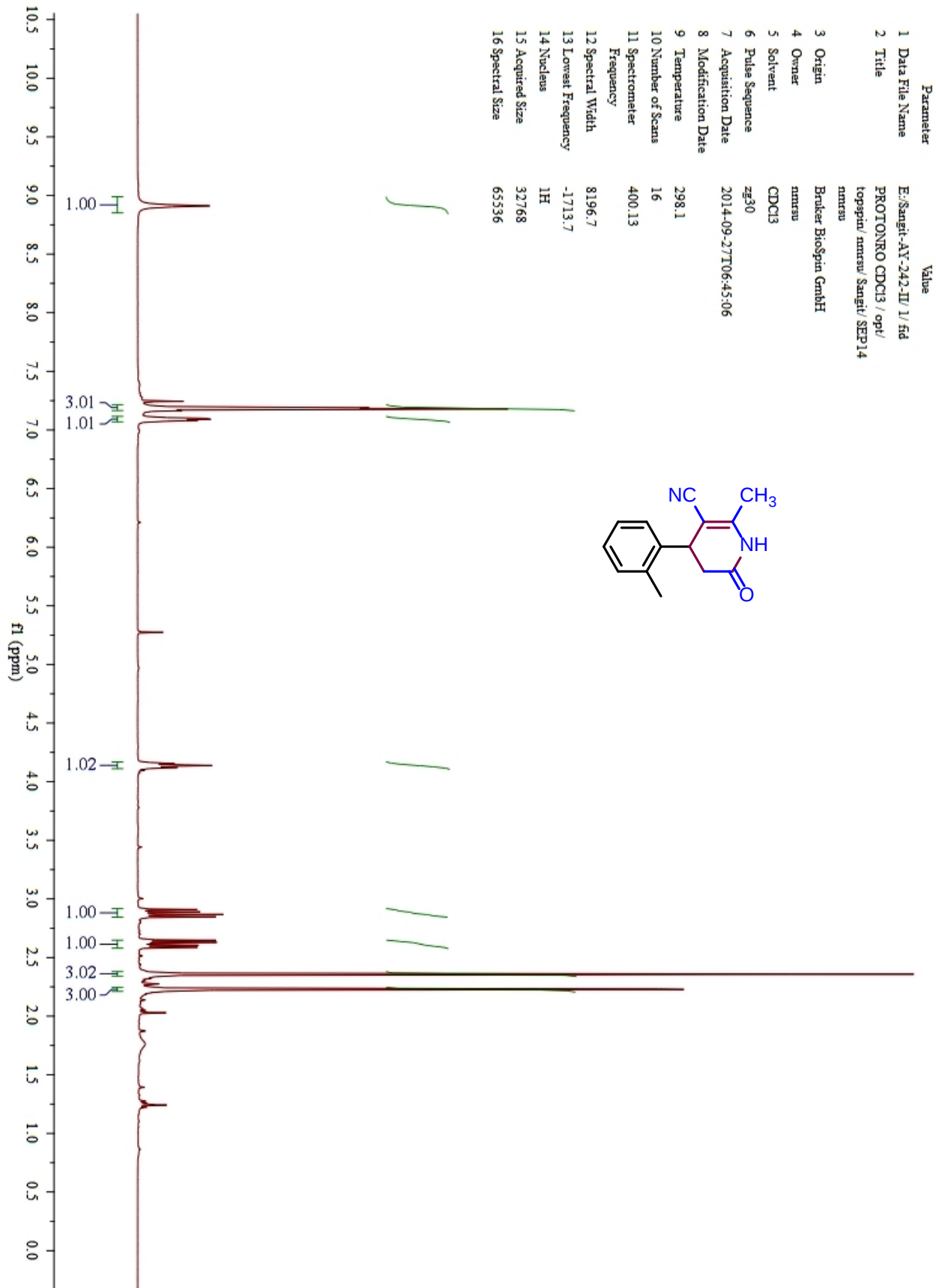


Figure S26 ^{13}C NMR spectra of **9**

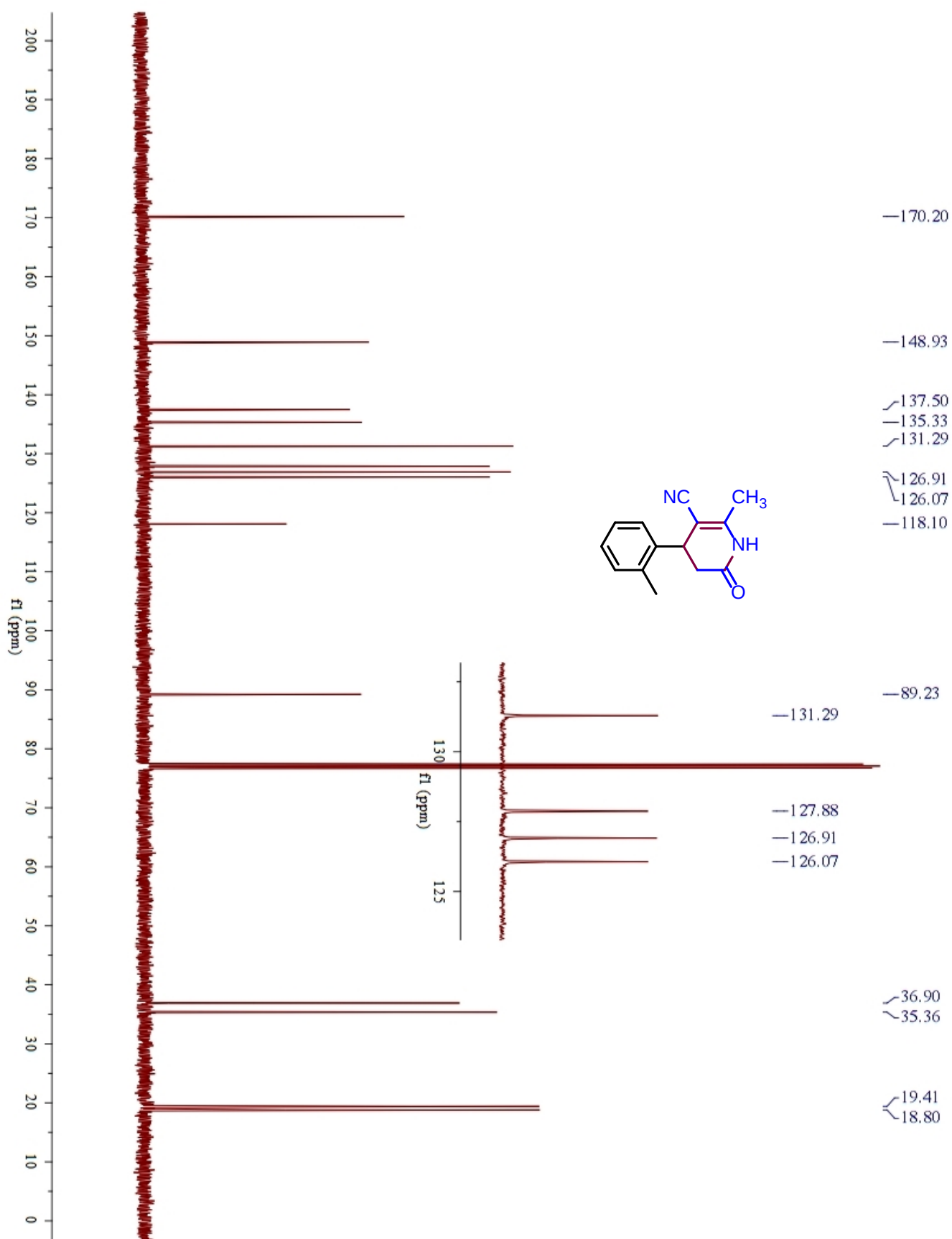


Figure S27 HRMS spectra of **9**

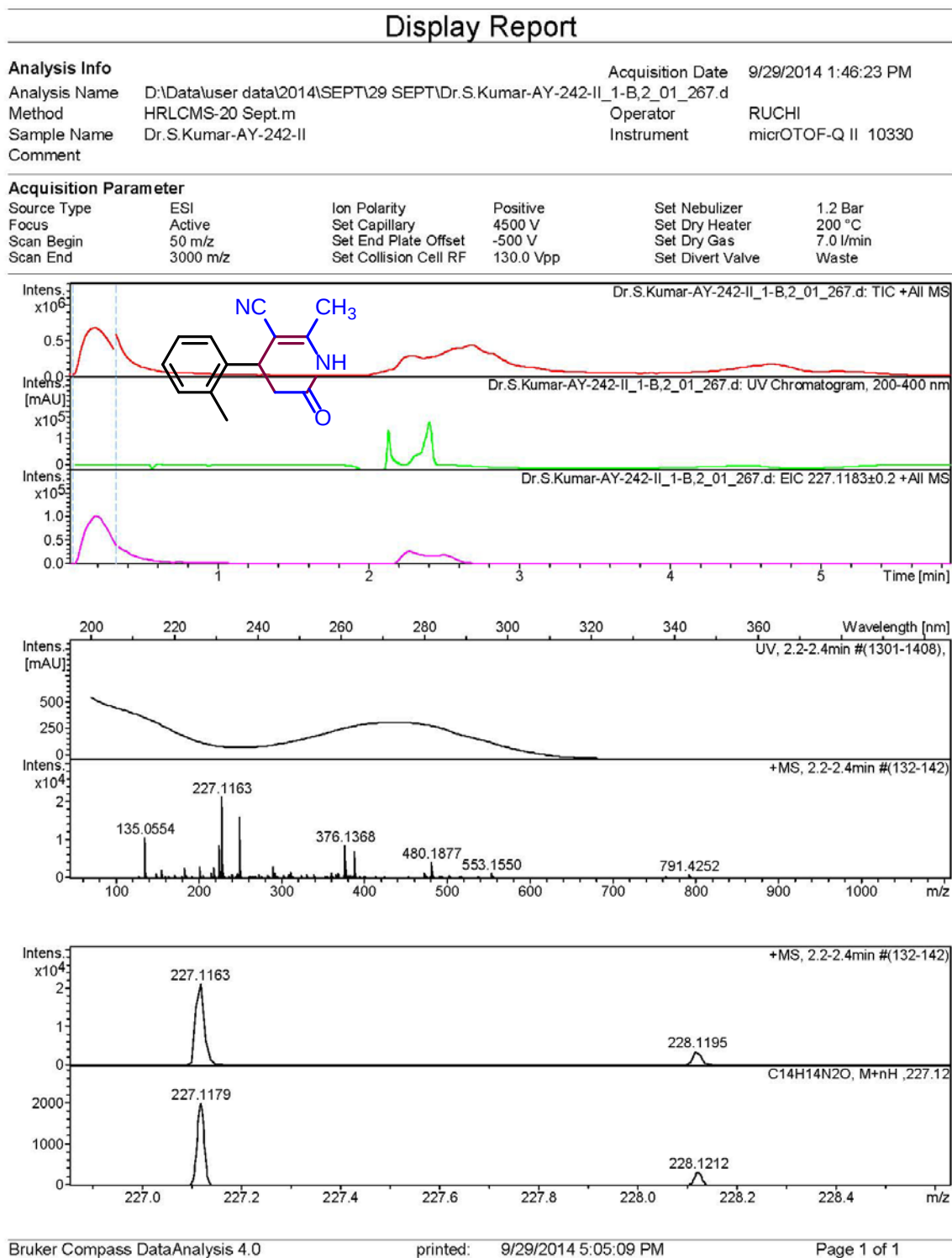


Figure S28 ¹H NMR spectra of **10**

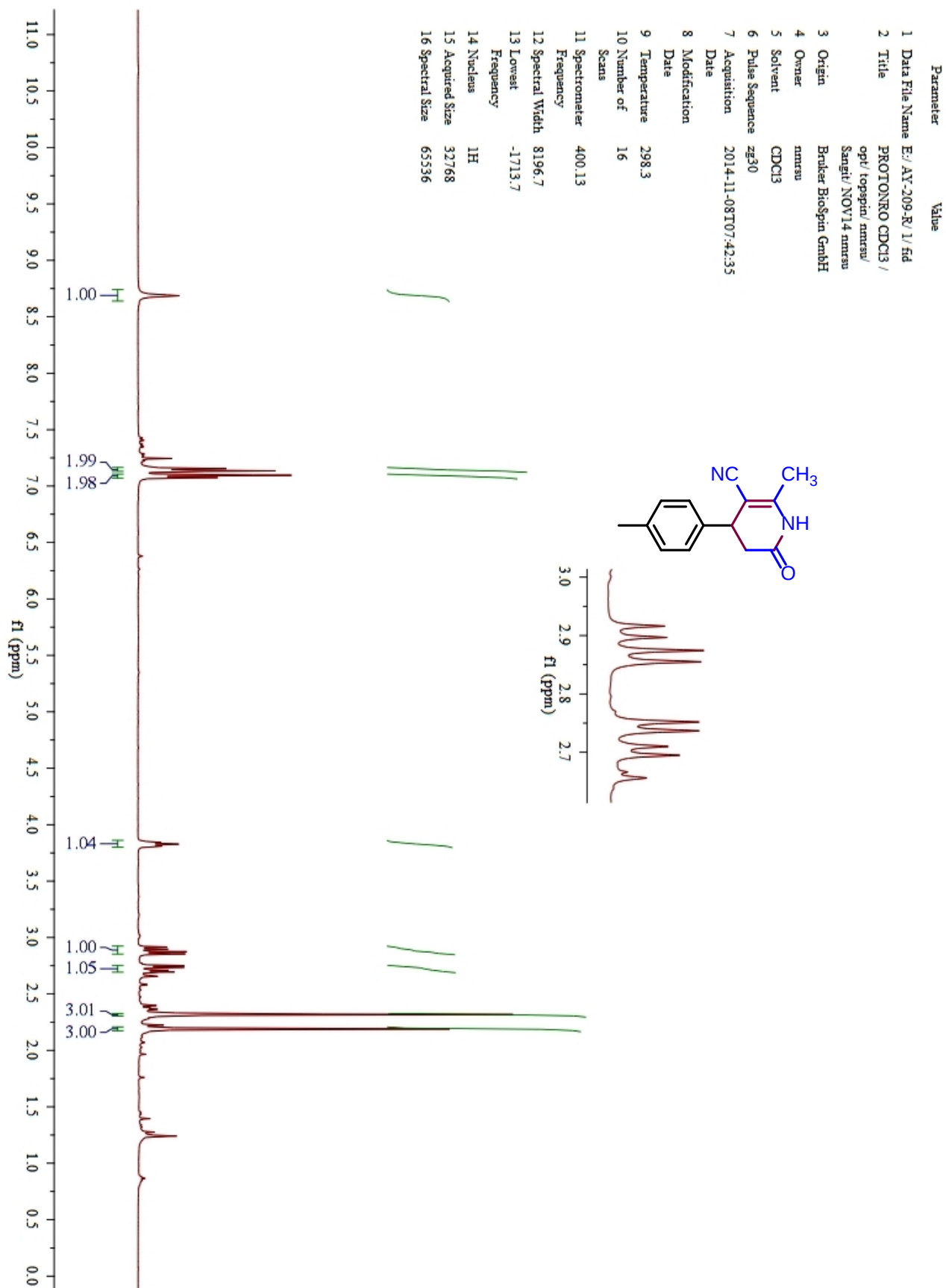


Figure S29 ^{13}C NMR spectra of **10**

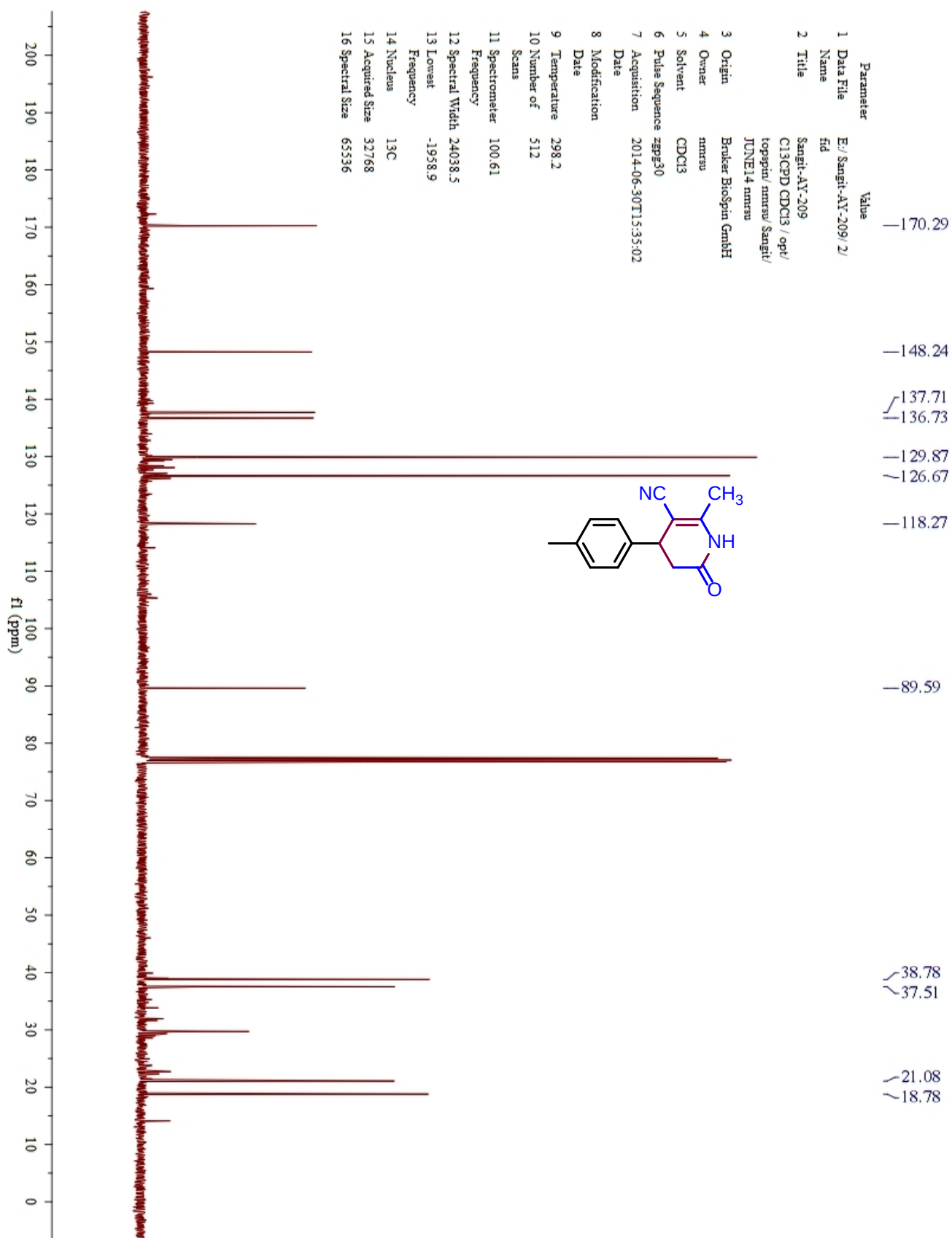


Figure S30 HRMS spectra of **10**



Sample ID: AY-209

Instrument: Agilent 7890A GC
with 5975C MS system

Supervisor: Dr. S. Kumar

Column: HP-5

Method: General_1_HP5_80_DEG.M

Acquisition date: 05/11/14

Operator: IISERB-CIF-Mass Facility

Ionization: EI (70 eV)

MSD: Single Quad.

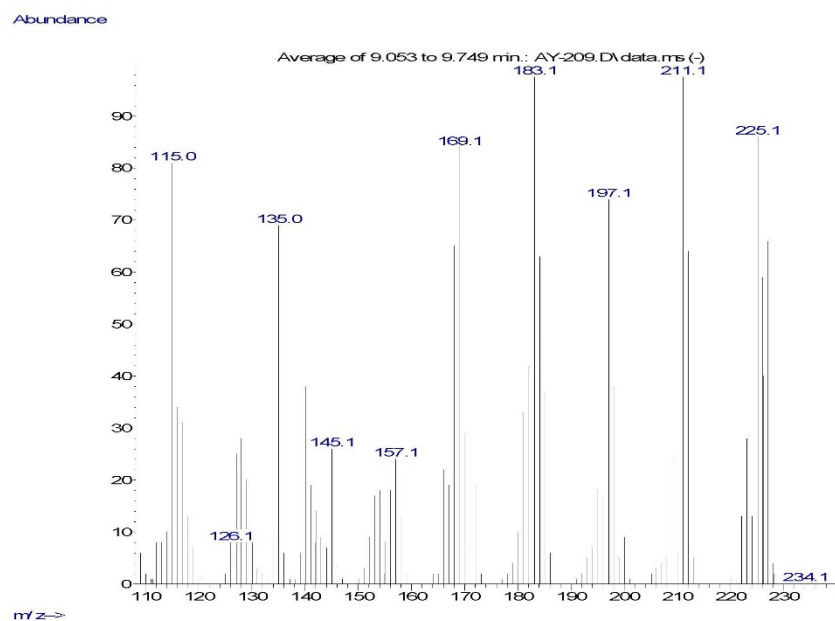
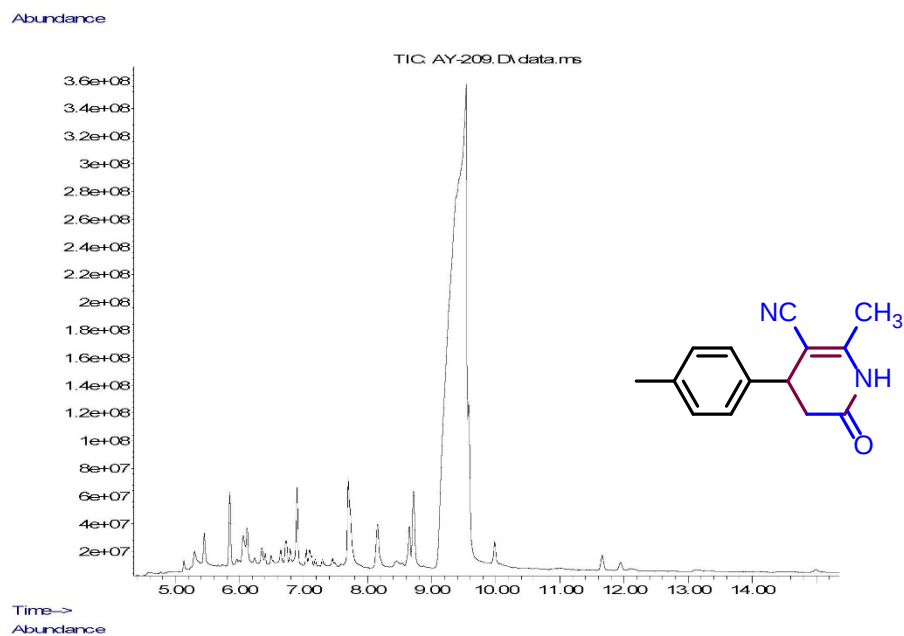


Figure S31 ^1H NMR spectra of **11**

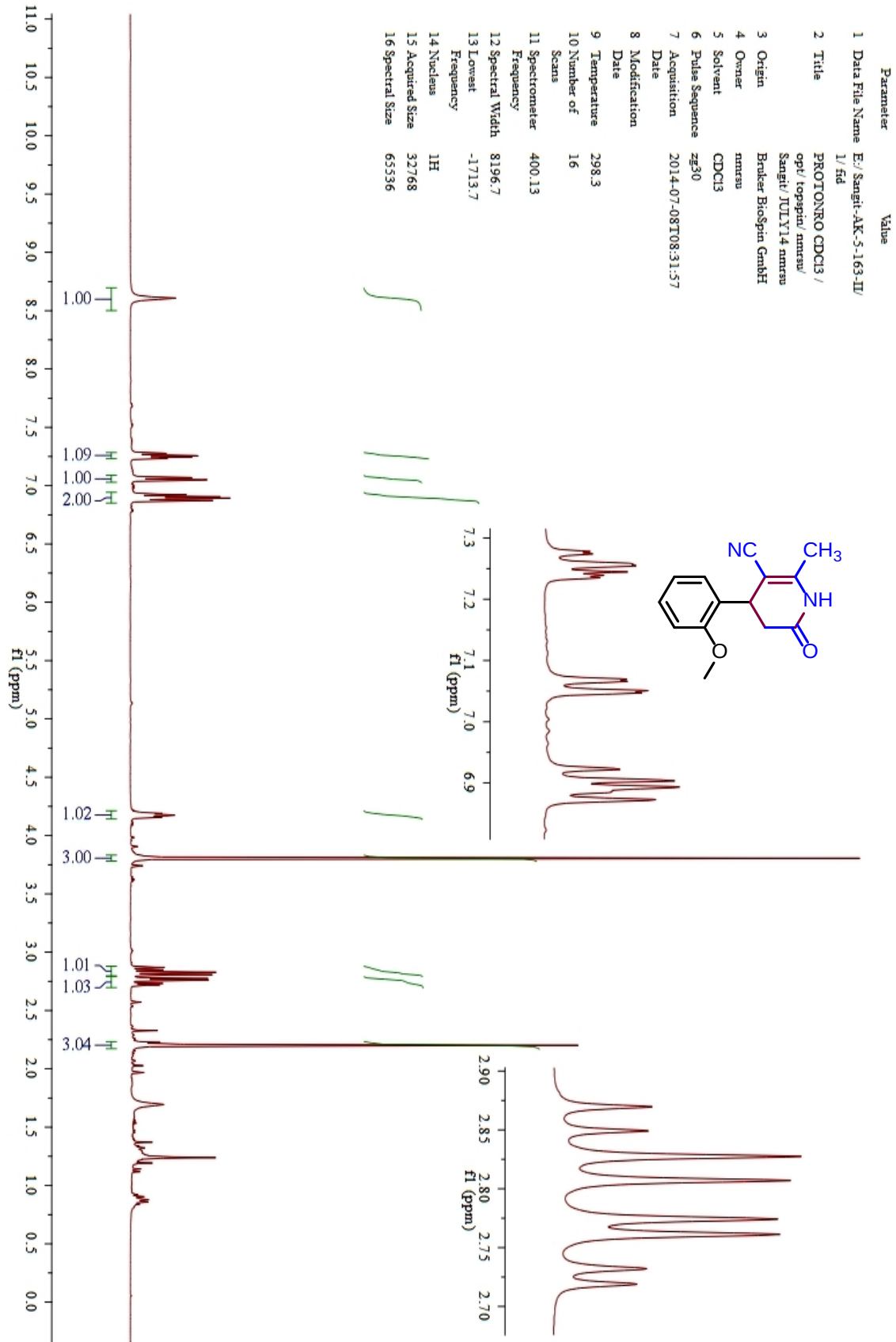


Figure S32 ^{13}C NMR spectra of **11**

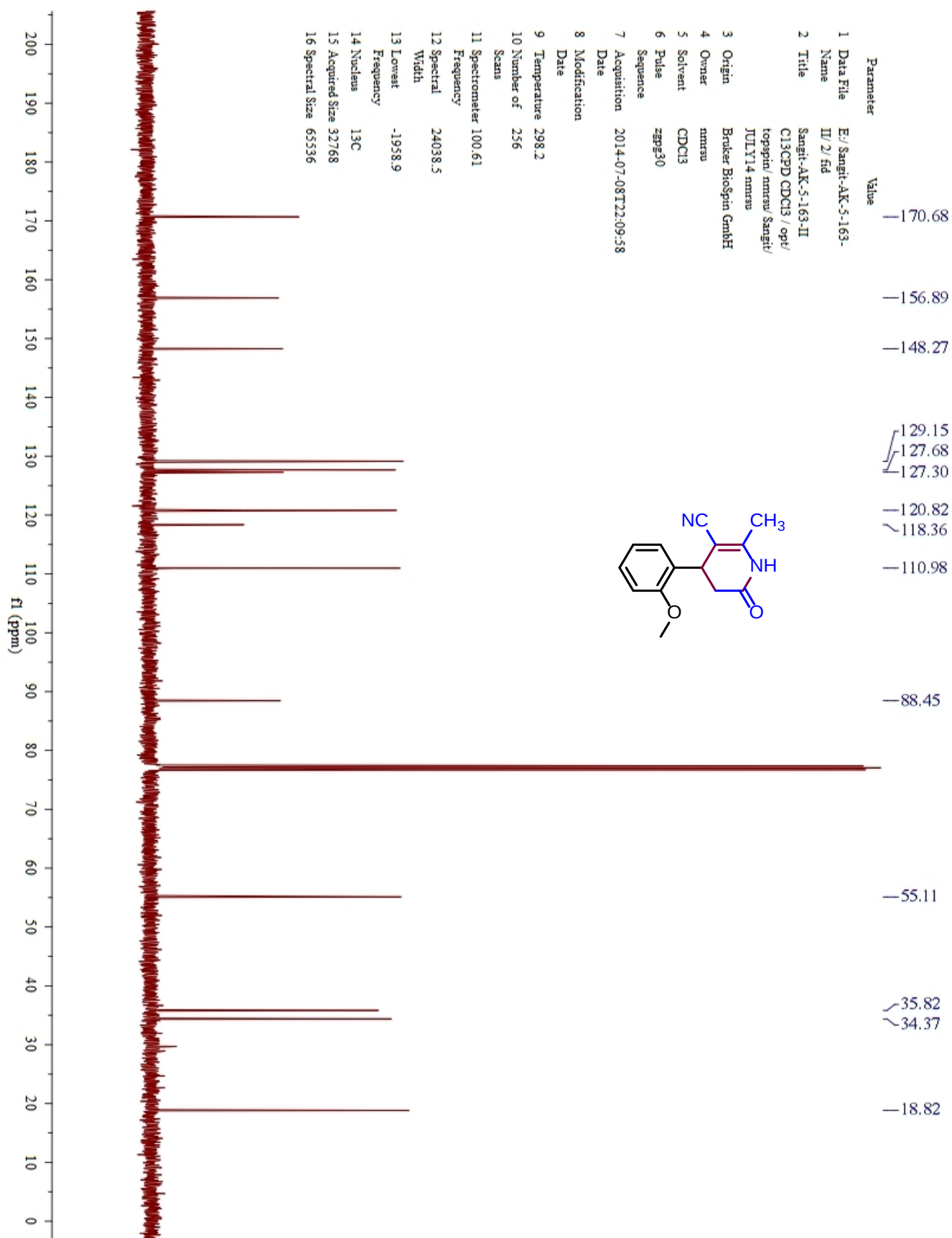


Figure S33 HRMS spectra of **11**

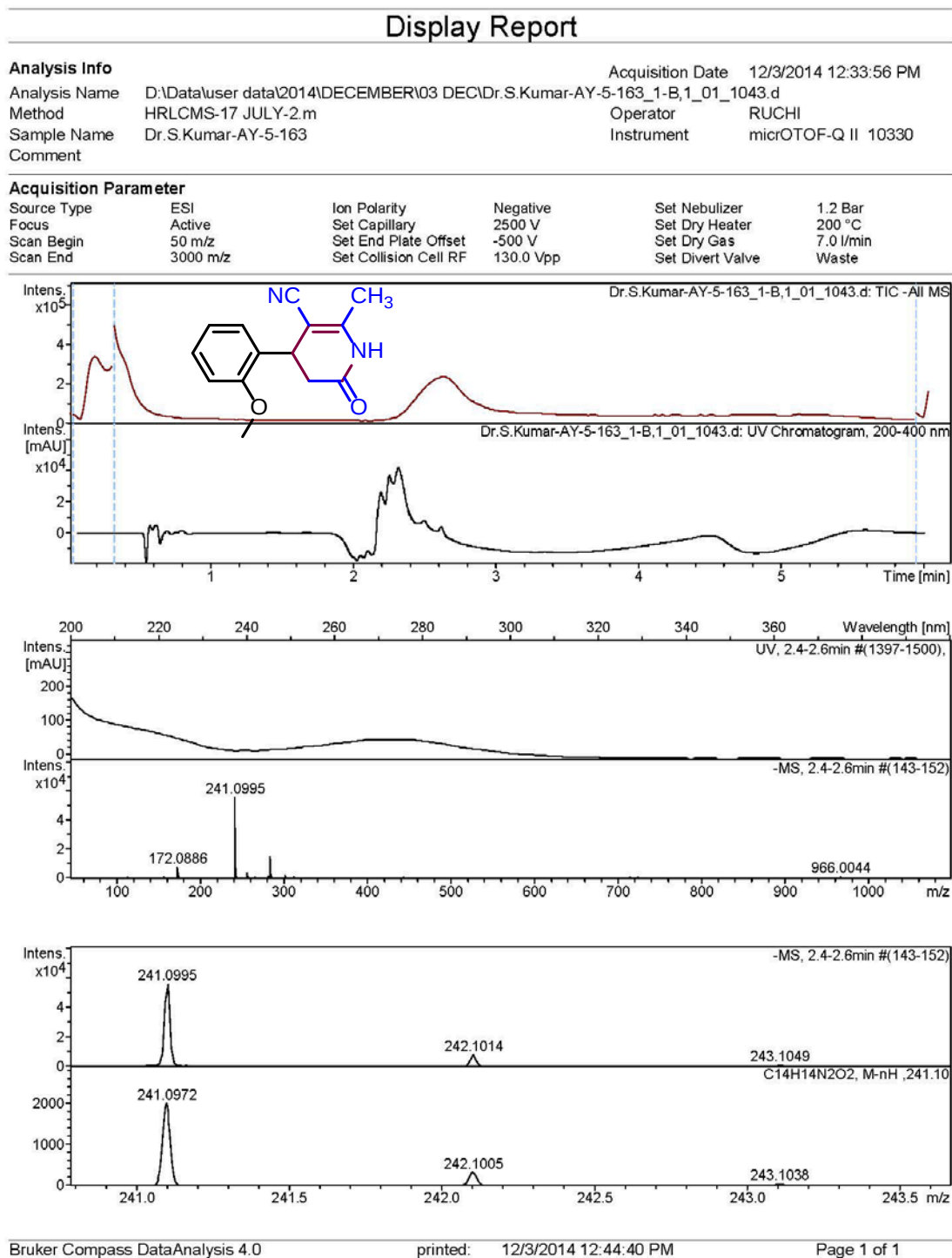


Figure S34 ¹H NMR spectra of **12**

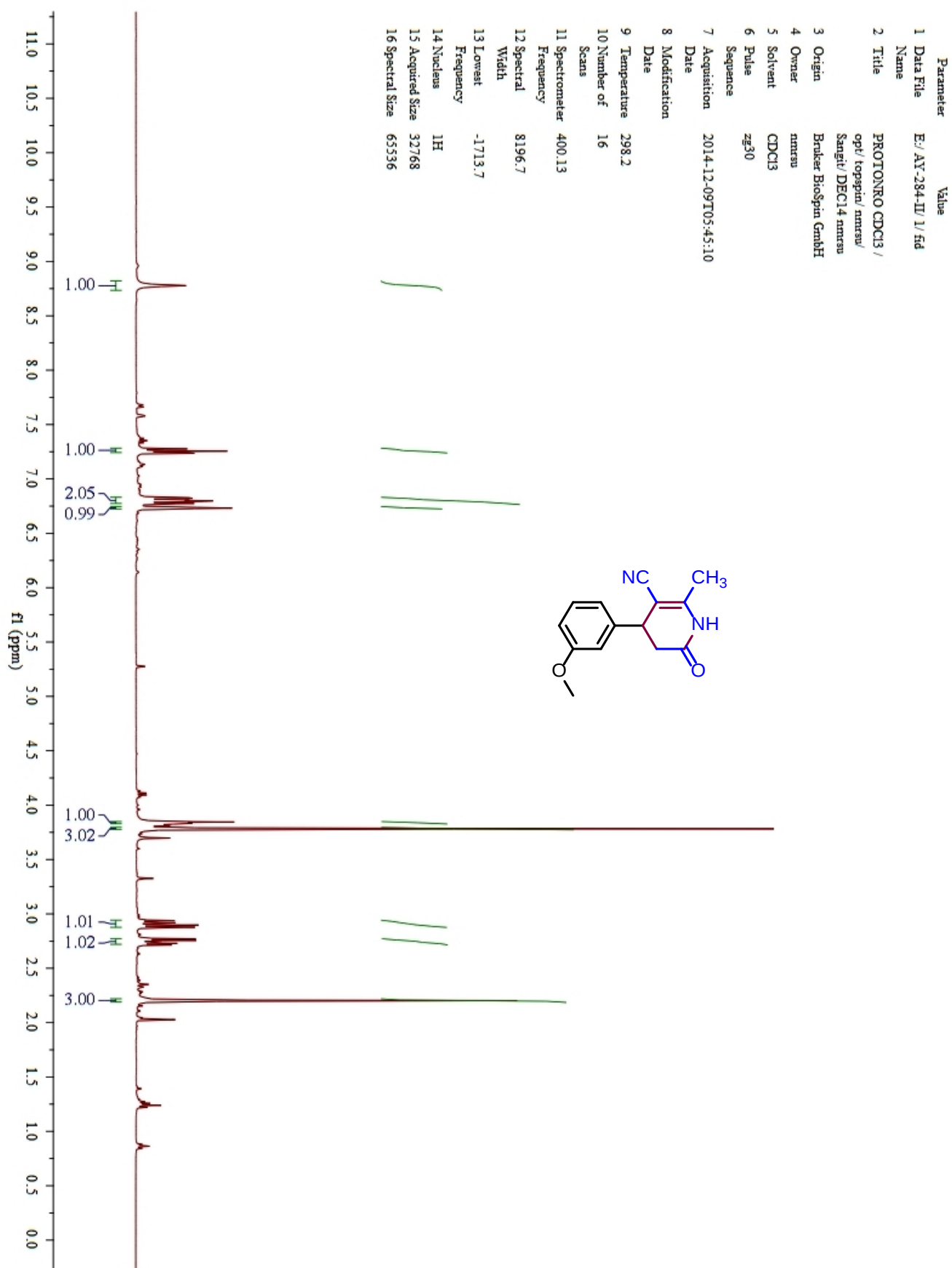


Figure S35 ^{13}C NMR spectra of **12**

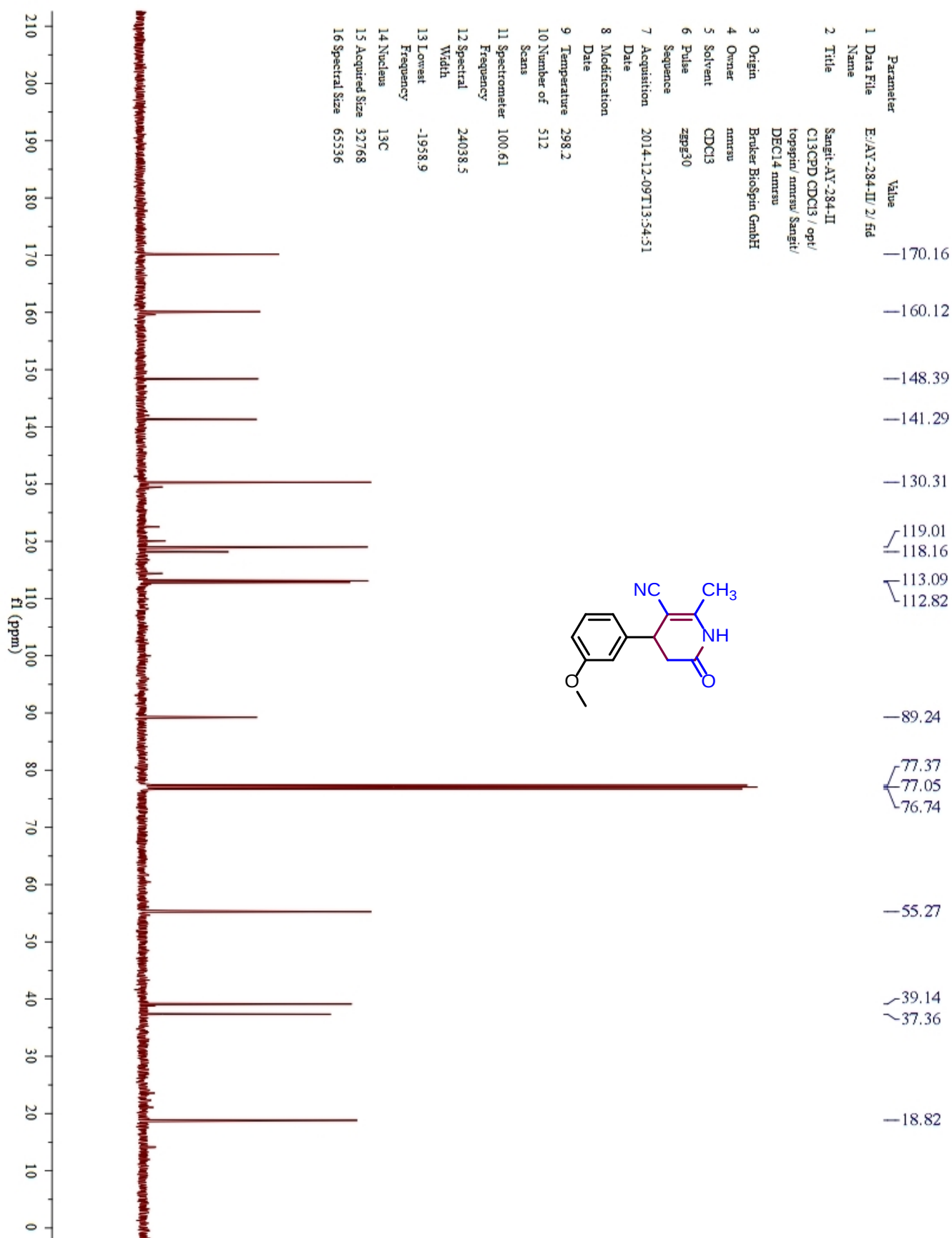


Figure S36 135-DEPT spectra of **12**

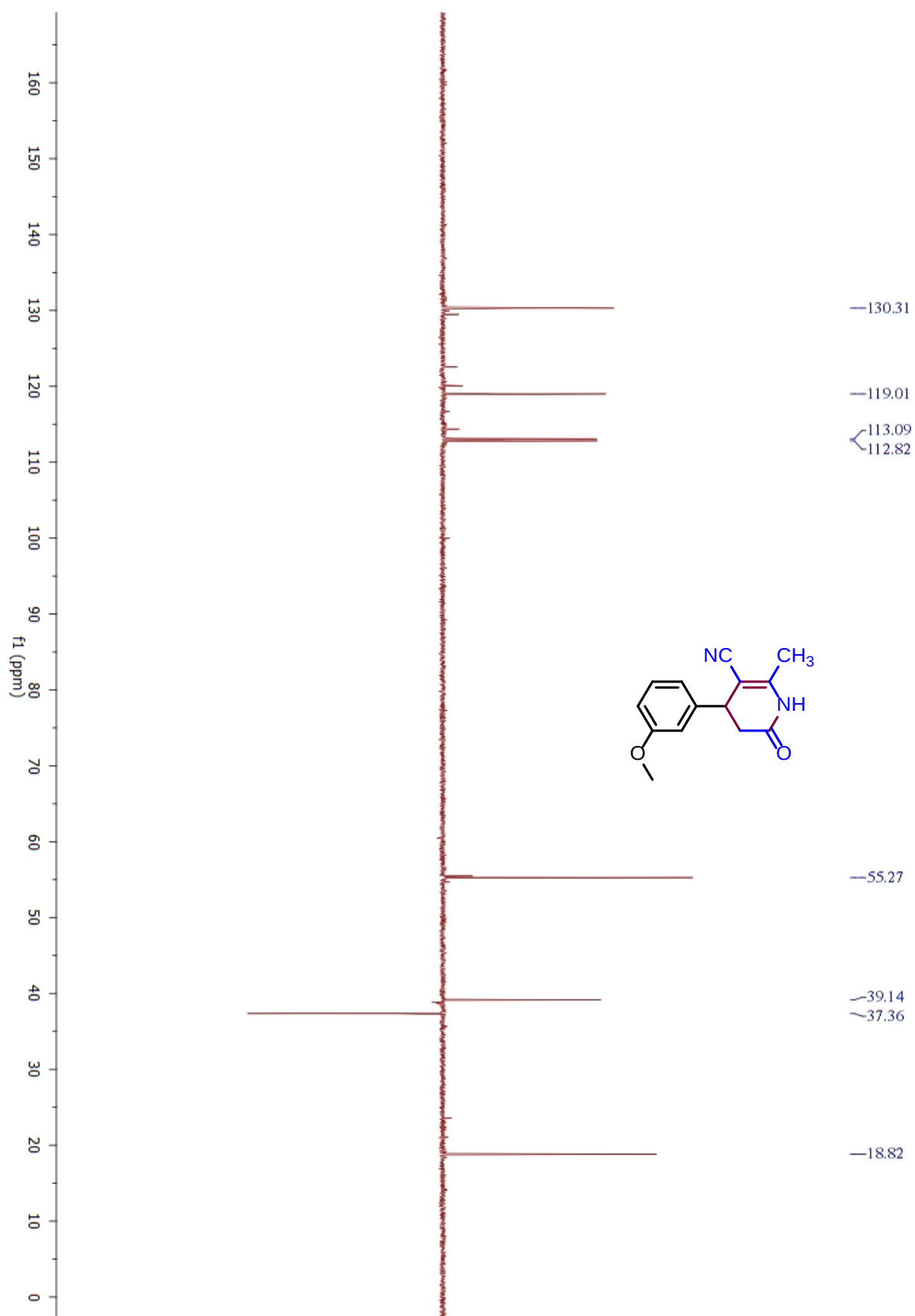


Figure S37 Mass spectra of **12**

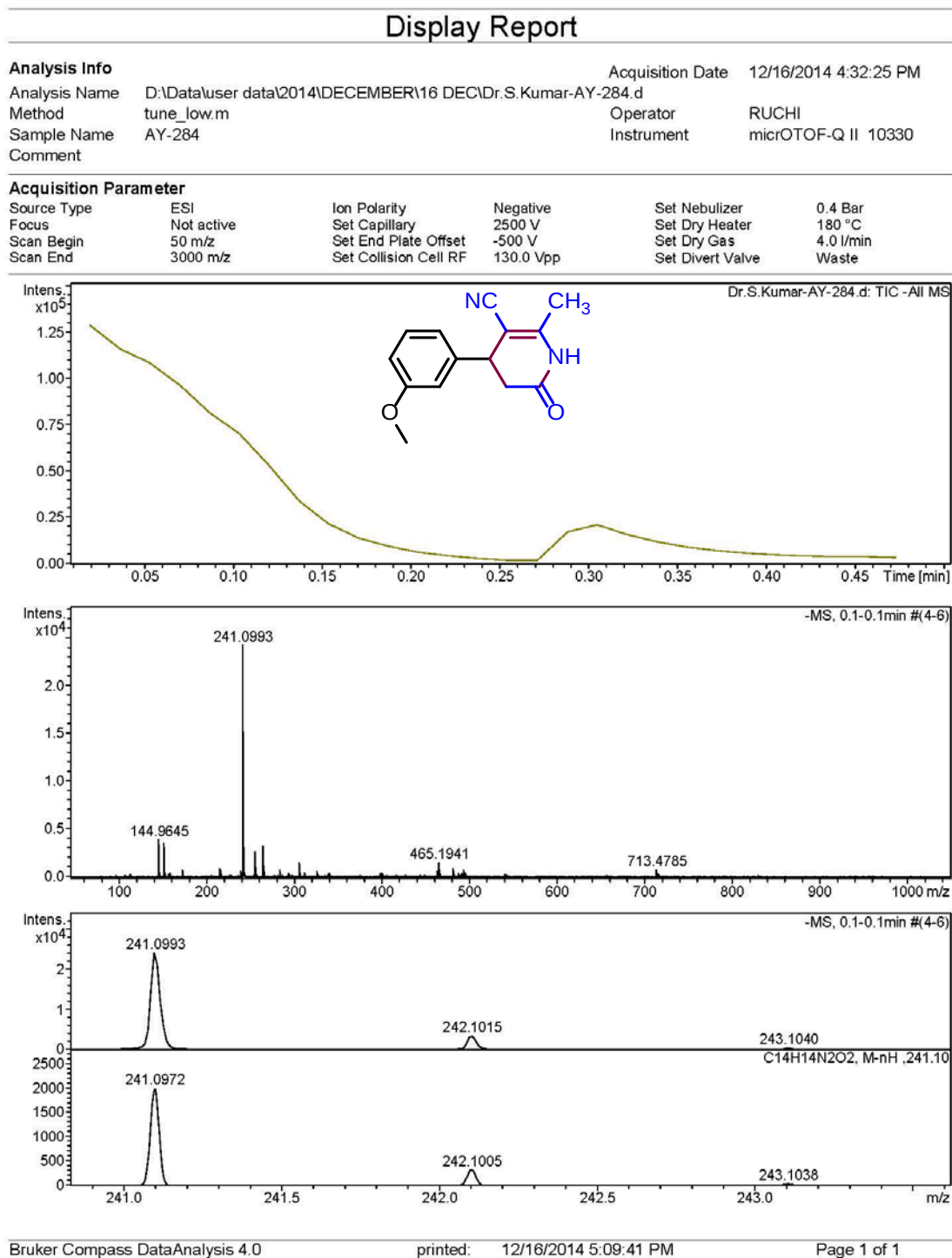


Figure S38 ¹H NMR spectra of **13**

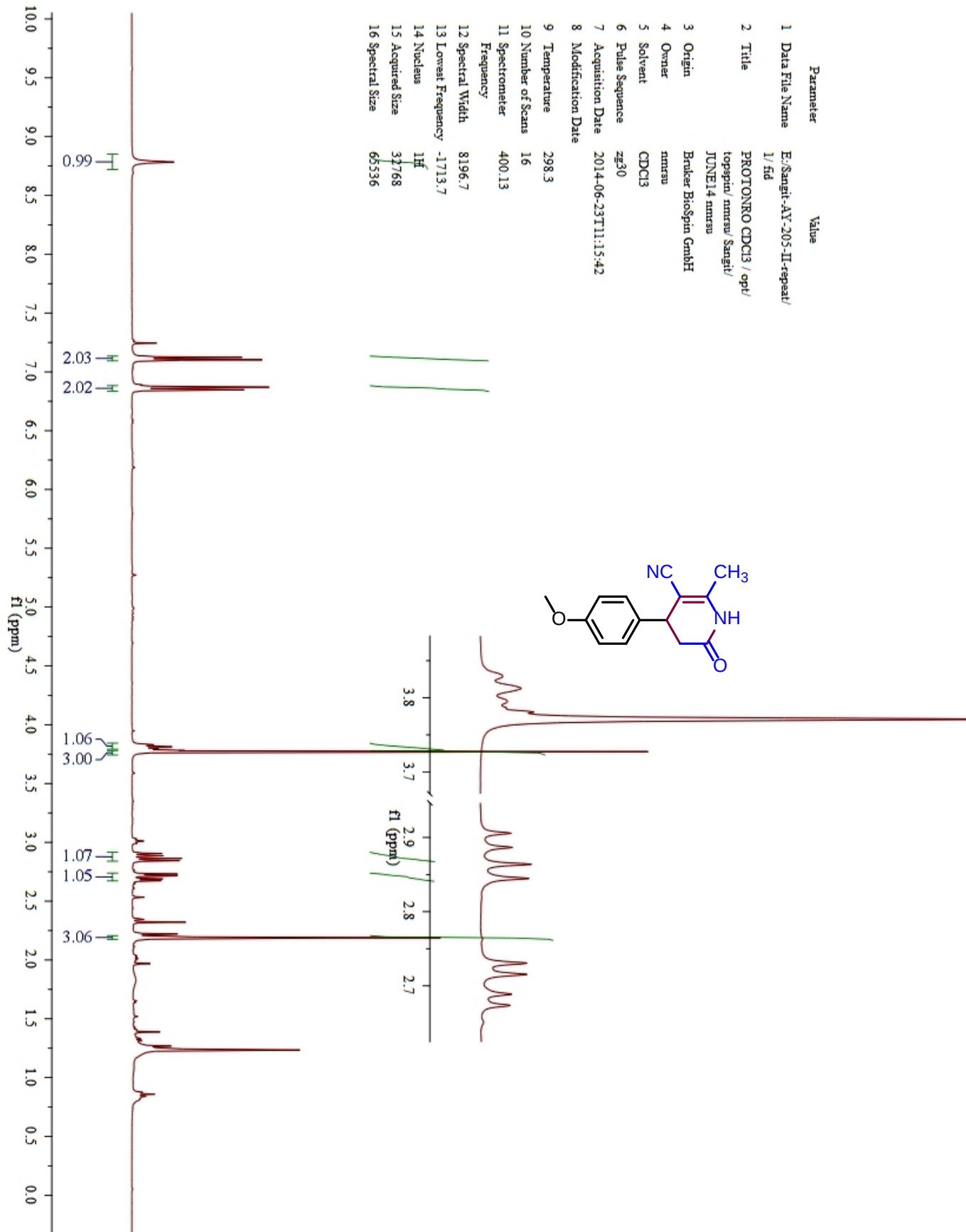


Figure S39 ¹³C NMR spectra of **13**

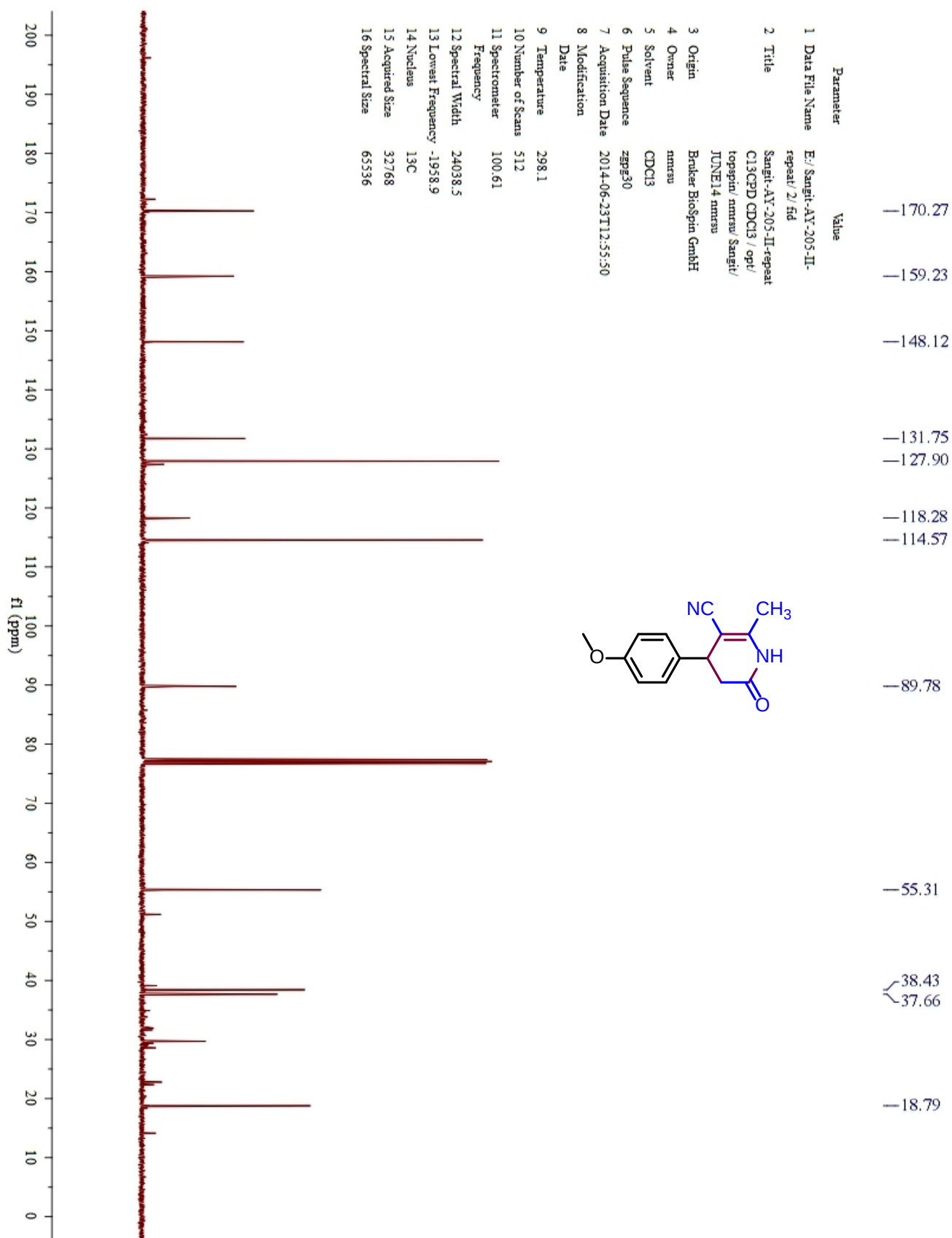


Figure 40 Mass spectra of 13

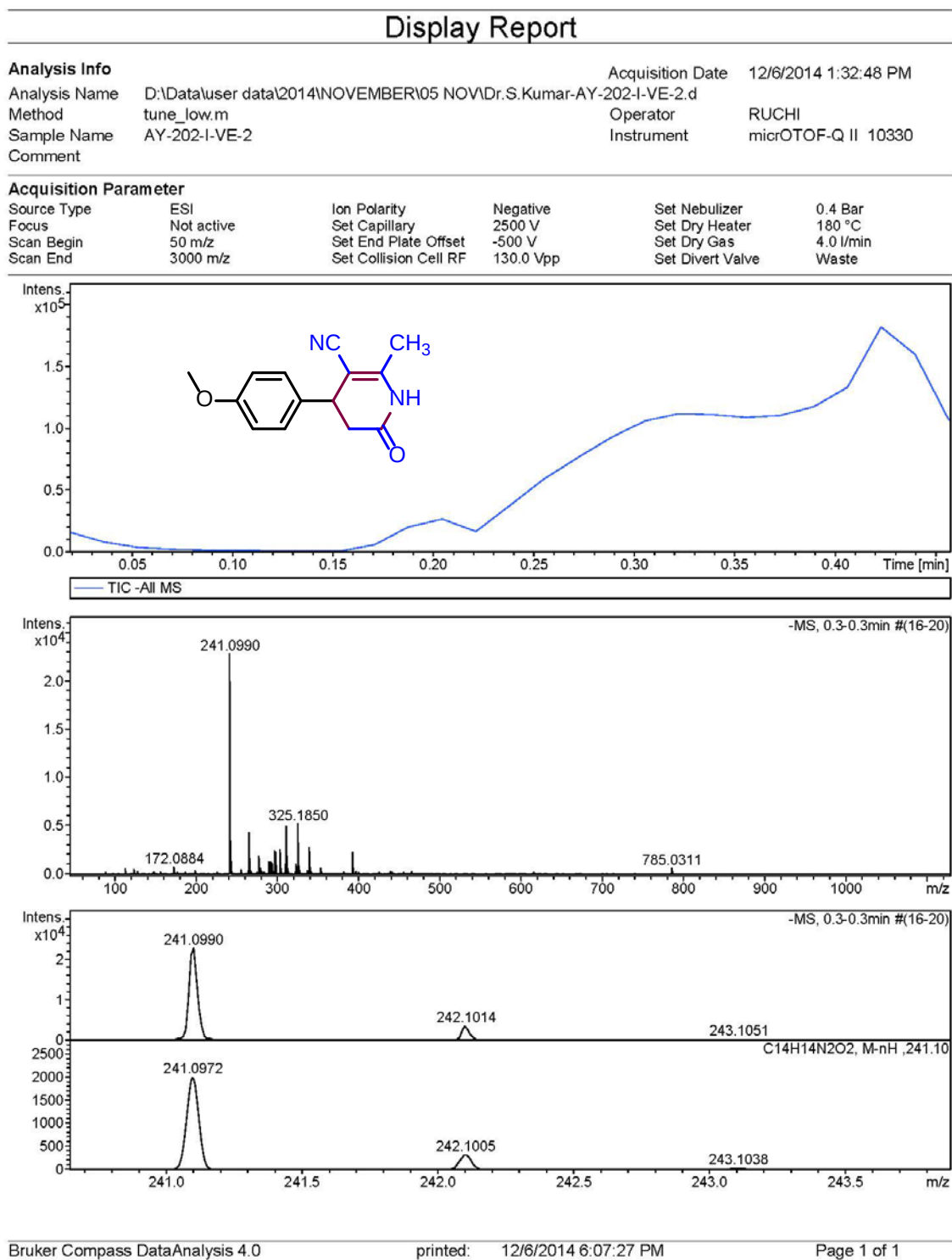


Figure S2 ¹³C NMR of AY-205

Parameter	Value
1 Data File Name	E:/ Sangit-AK-5-172-R/ 1/
2 Title	fid
3 Origin	Sangit-AK-5-172-R
4 Owner	Braker Biospin GmbH
5 Solvent	nmrso
6 Pulse Sequence	zg30
7 Acquisition Date	2014-07-21T10:47:16
8 Modification Date	
9 Temperature	298.2
10 Number of Scans	16
11 Spectrometer	400.13
12 Spectral Width	8223.7
13 Lowest Frequency	-1640.9
14 Nucleus	¹ H
15 Acquired Size	32768
16 Spectral Size	65536

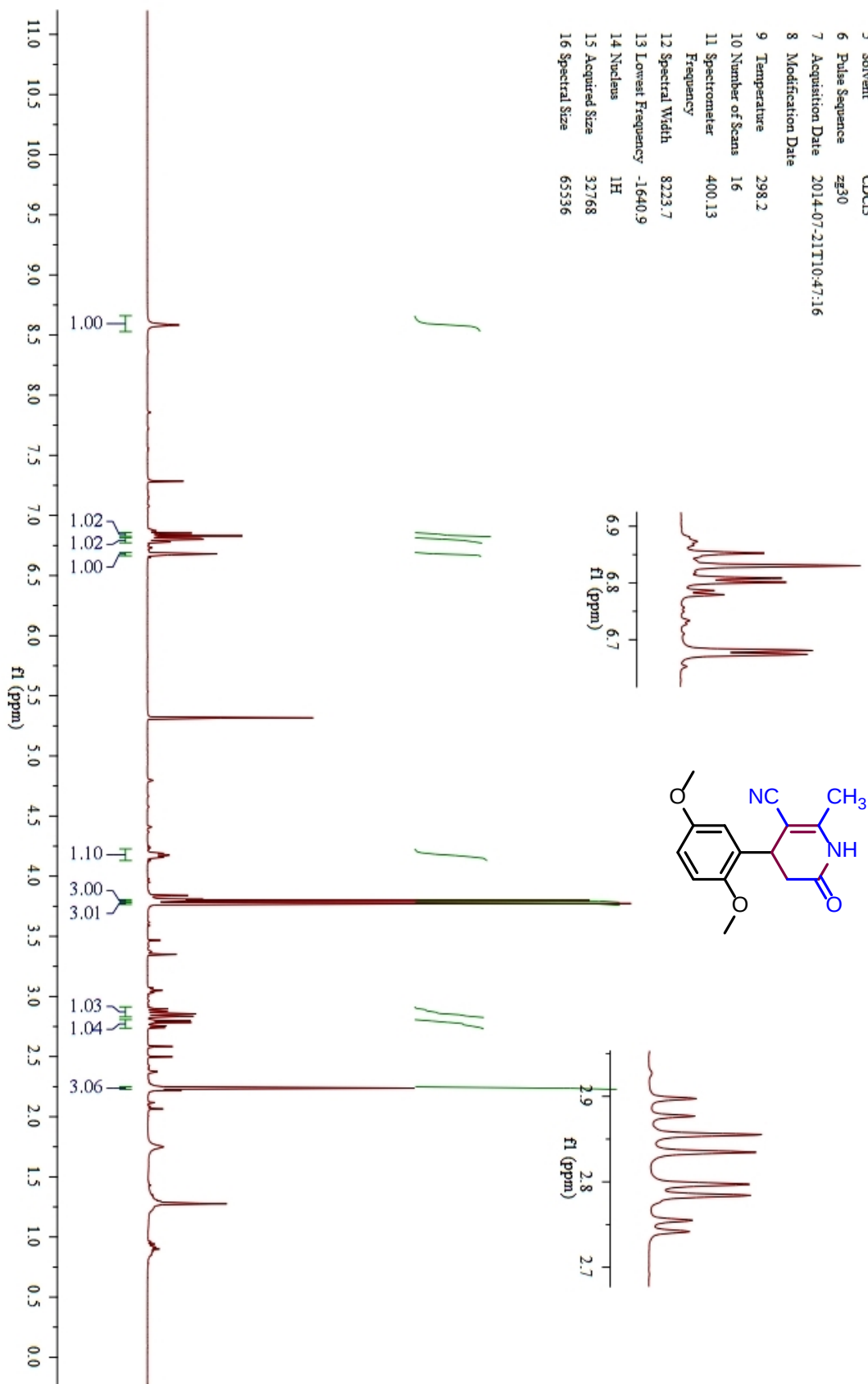


Figure S42 ^{13}C NMR spectra of **14**

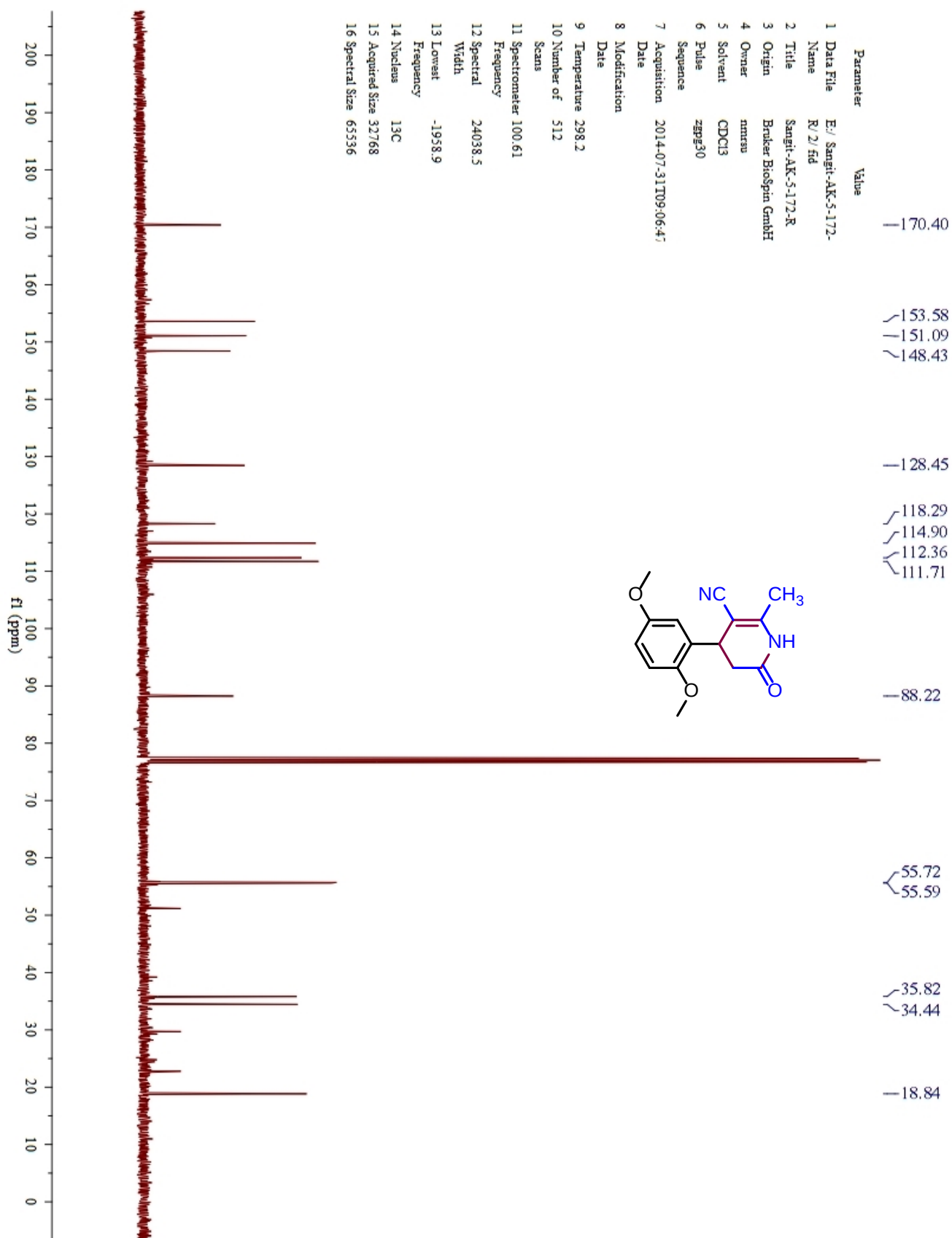


Figure S43 HRMS spectra of **14**

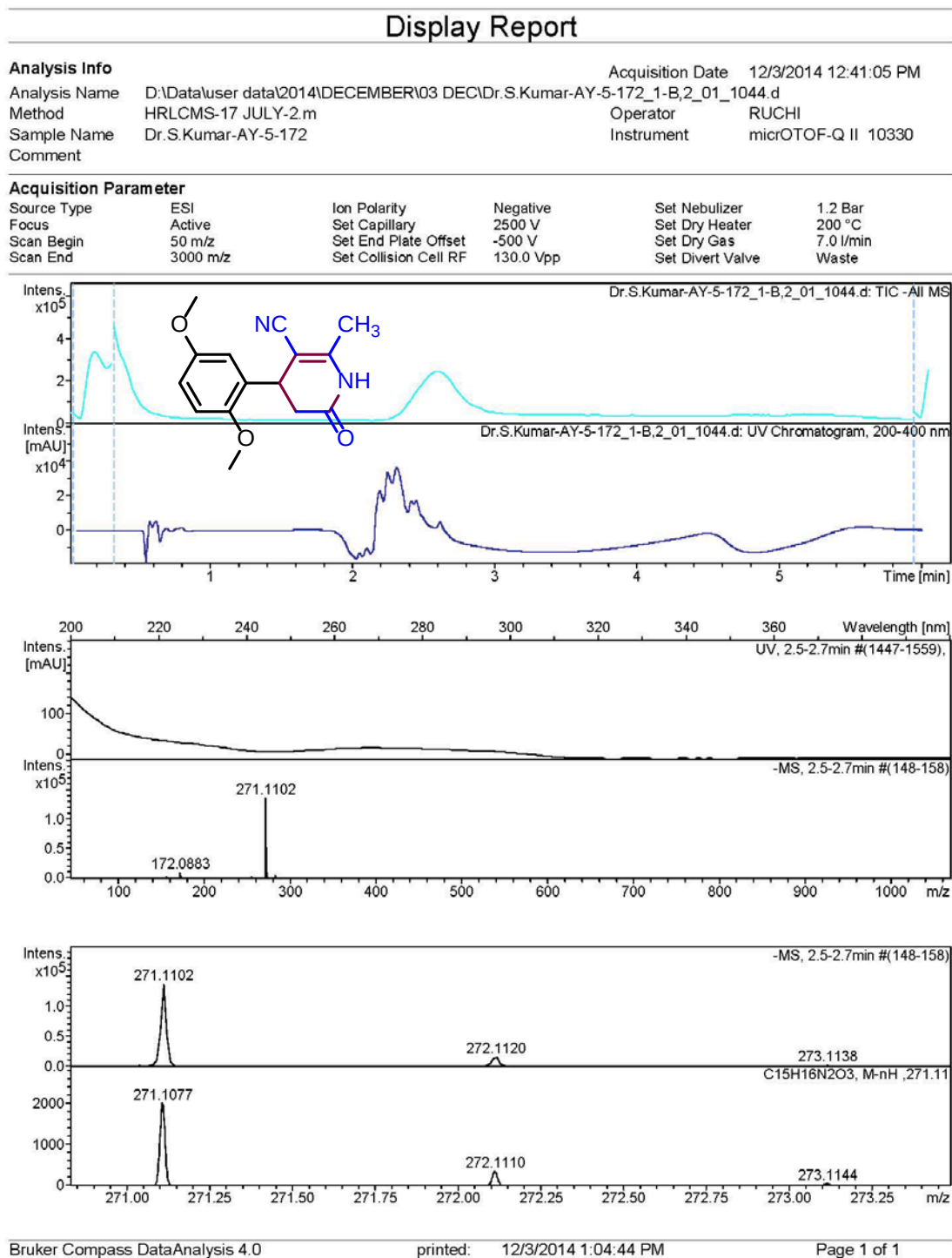


Figure S44 ¹H NMR spectra of **15**

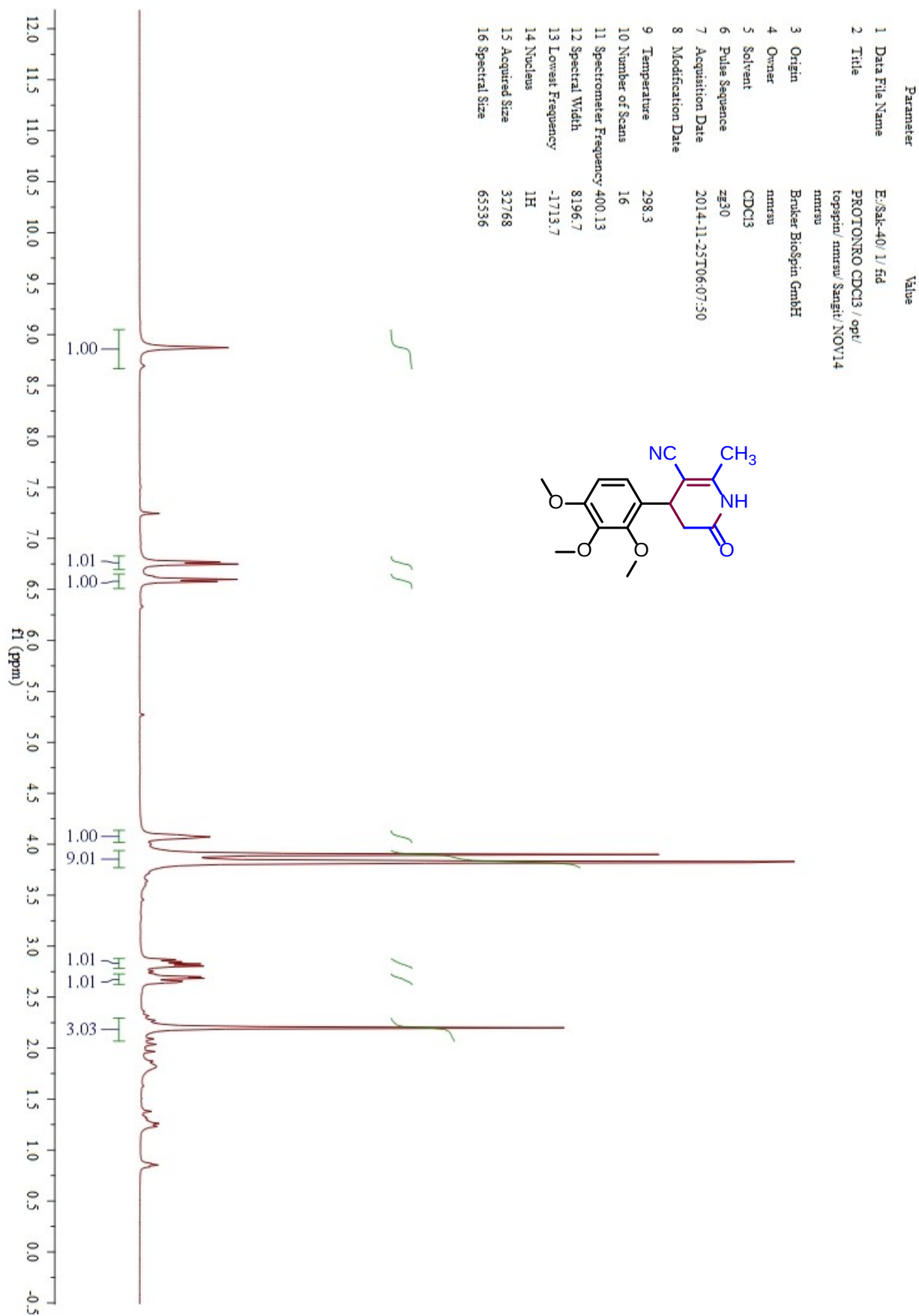


Figure S45 ¹³C NMR spectra of 15

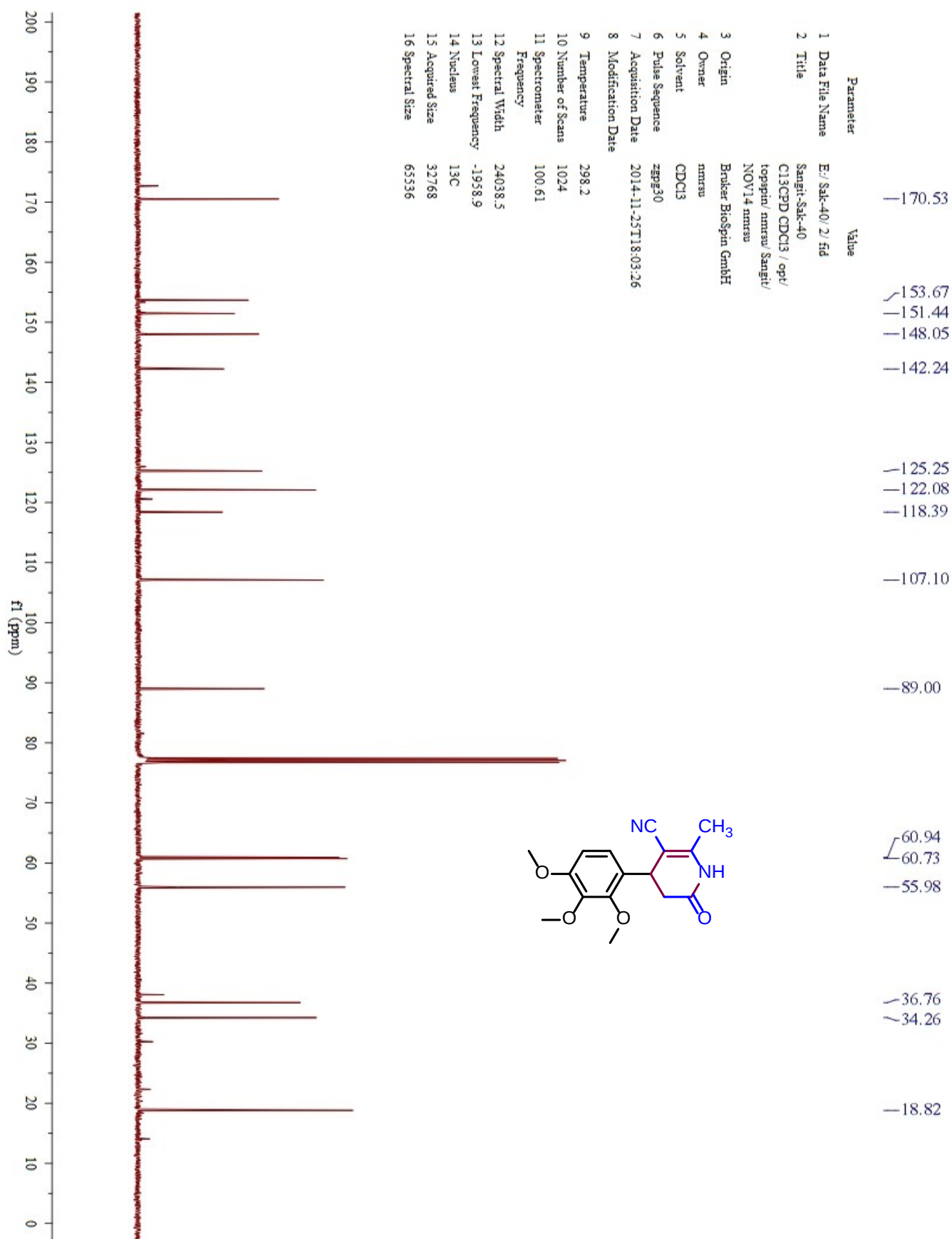


Figure S46 Mass spectra of 15

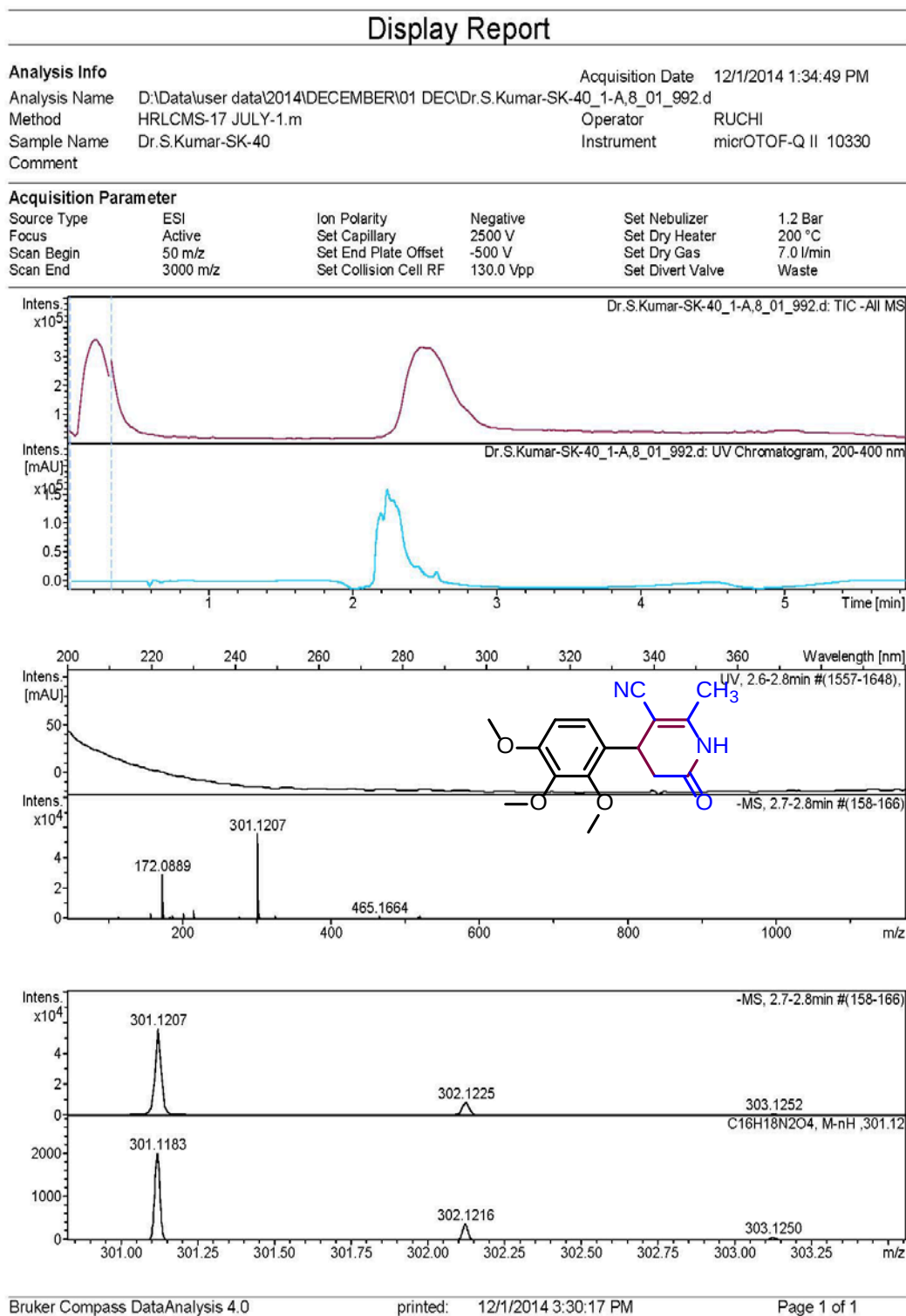


Figure S47 ^1H NMR spectra of **16**

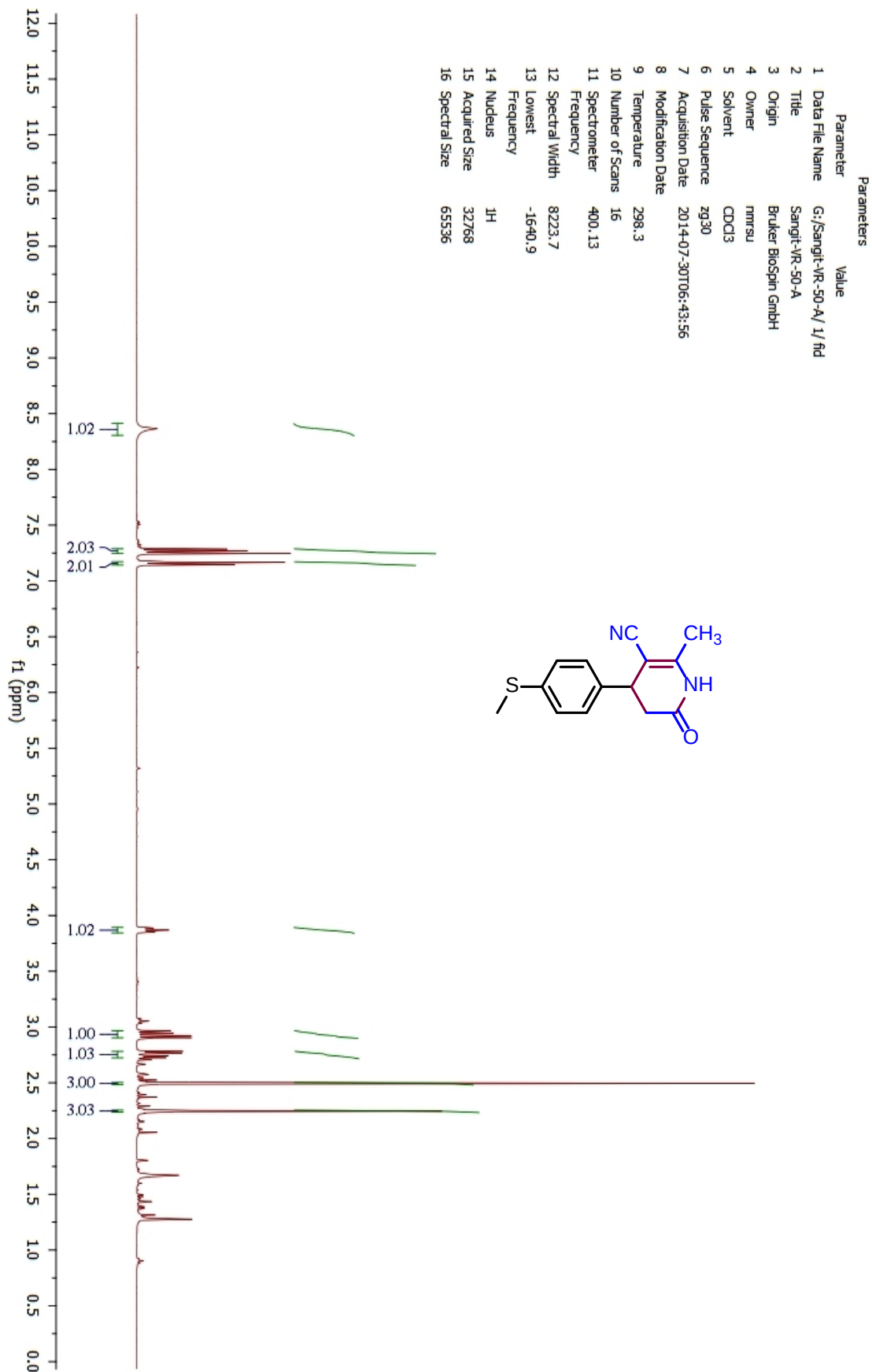


Figure S48 ¹³C NMR spectra of **16**

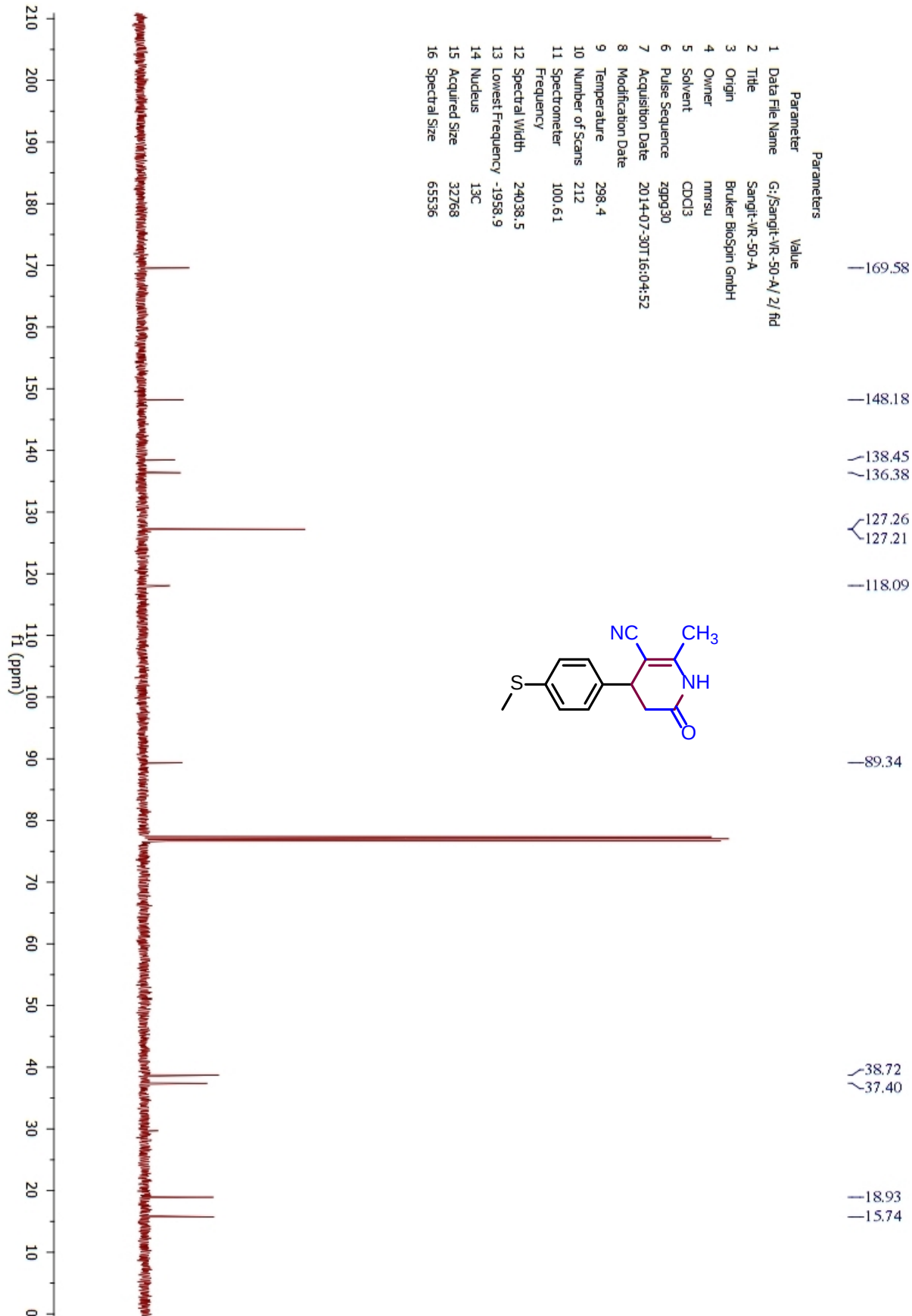


Figure S49 HRMS spectra of **16**

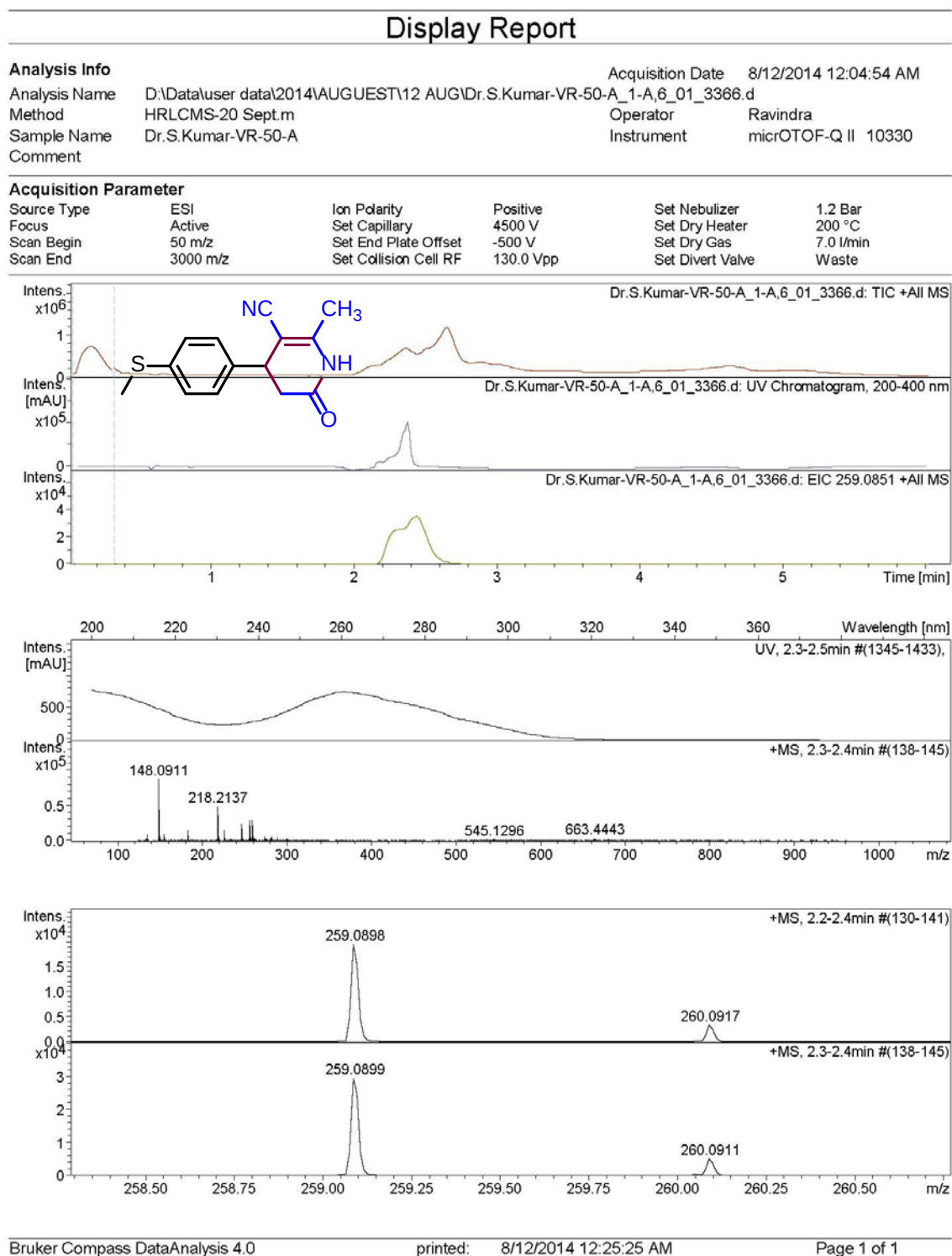


Figure S50 ¹H NMR spectra of **17**

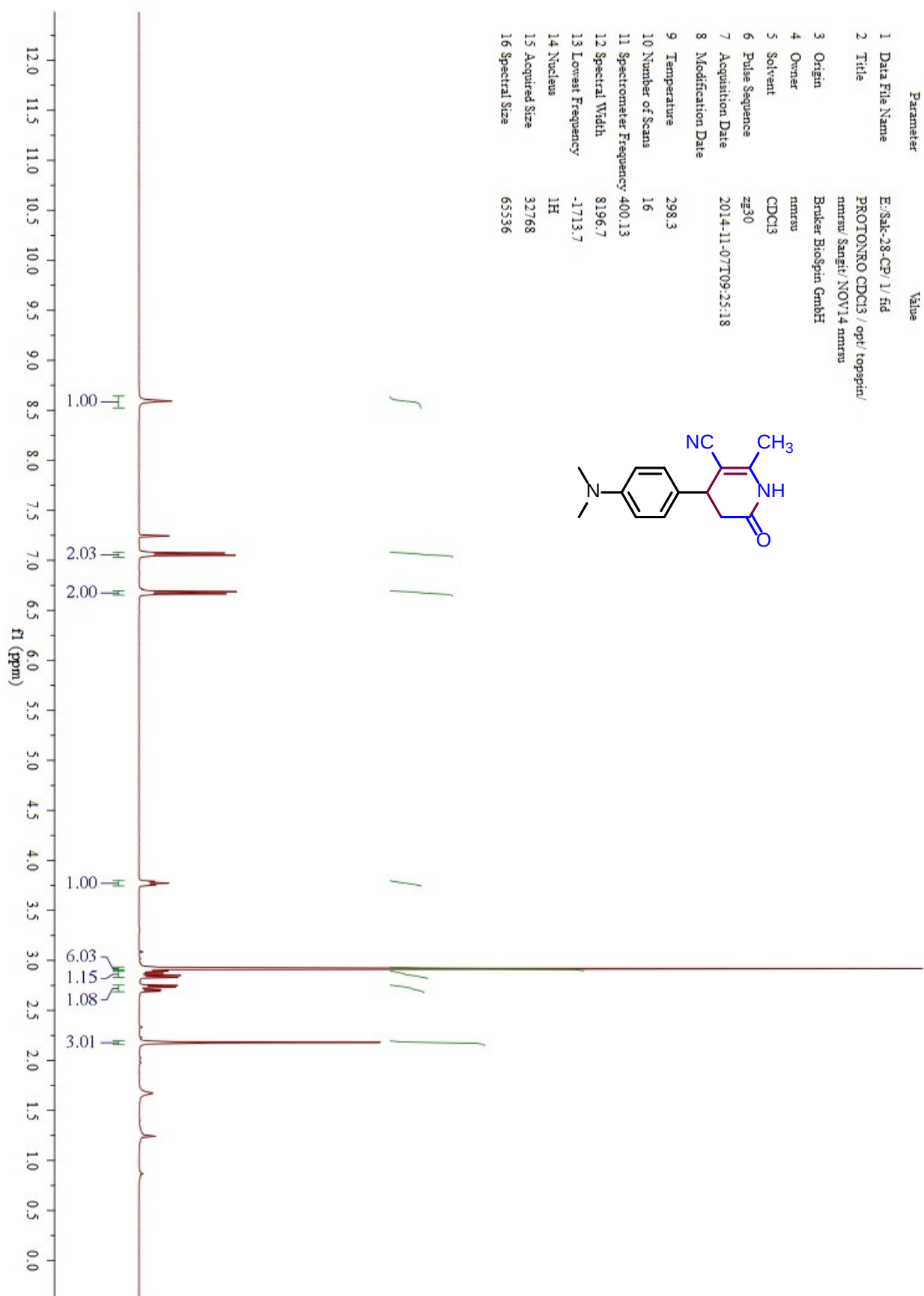


Figure S51 ^{13}C NMR spectra of 17

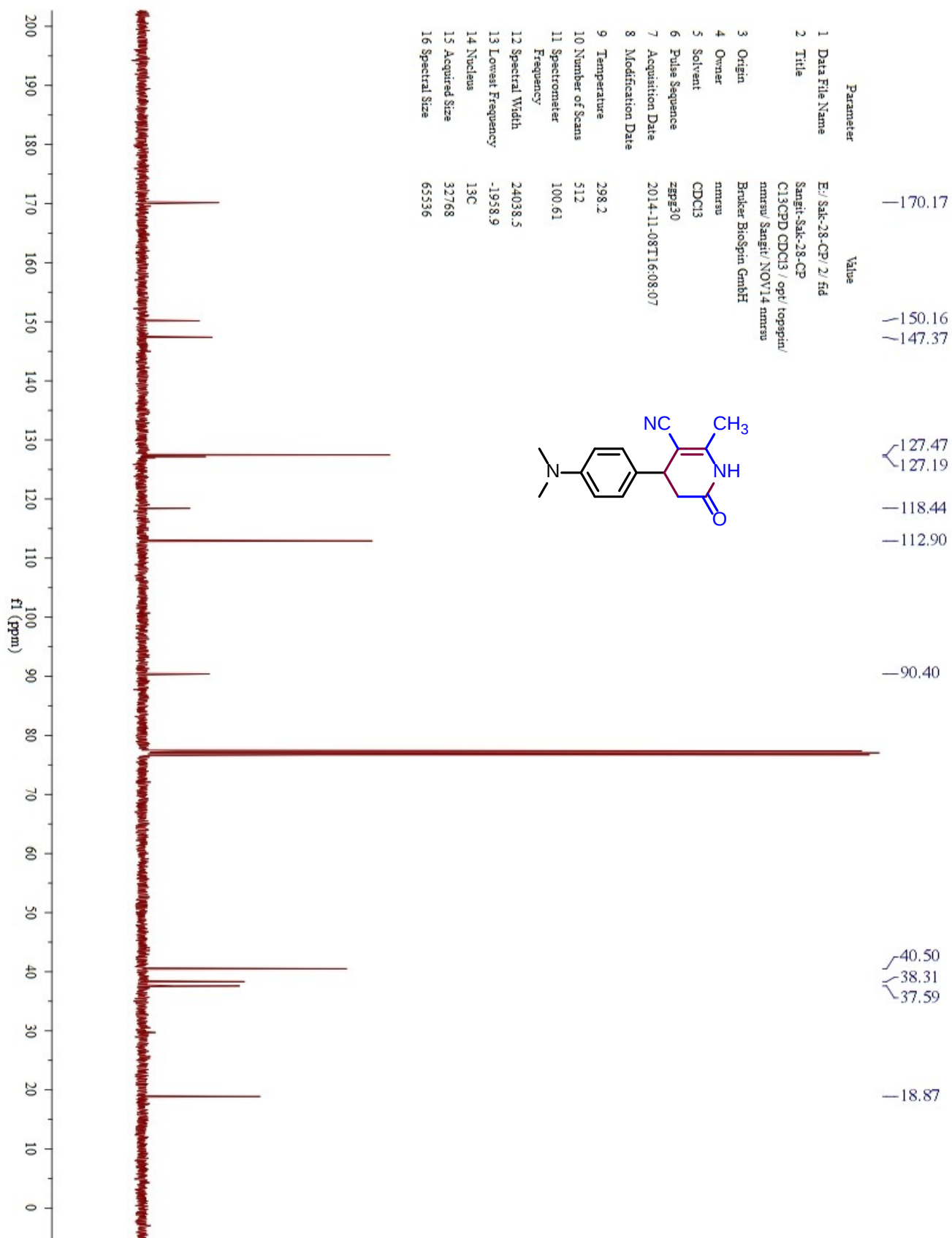


Figure S52 Mass spectra of **17**

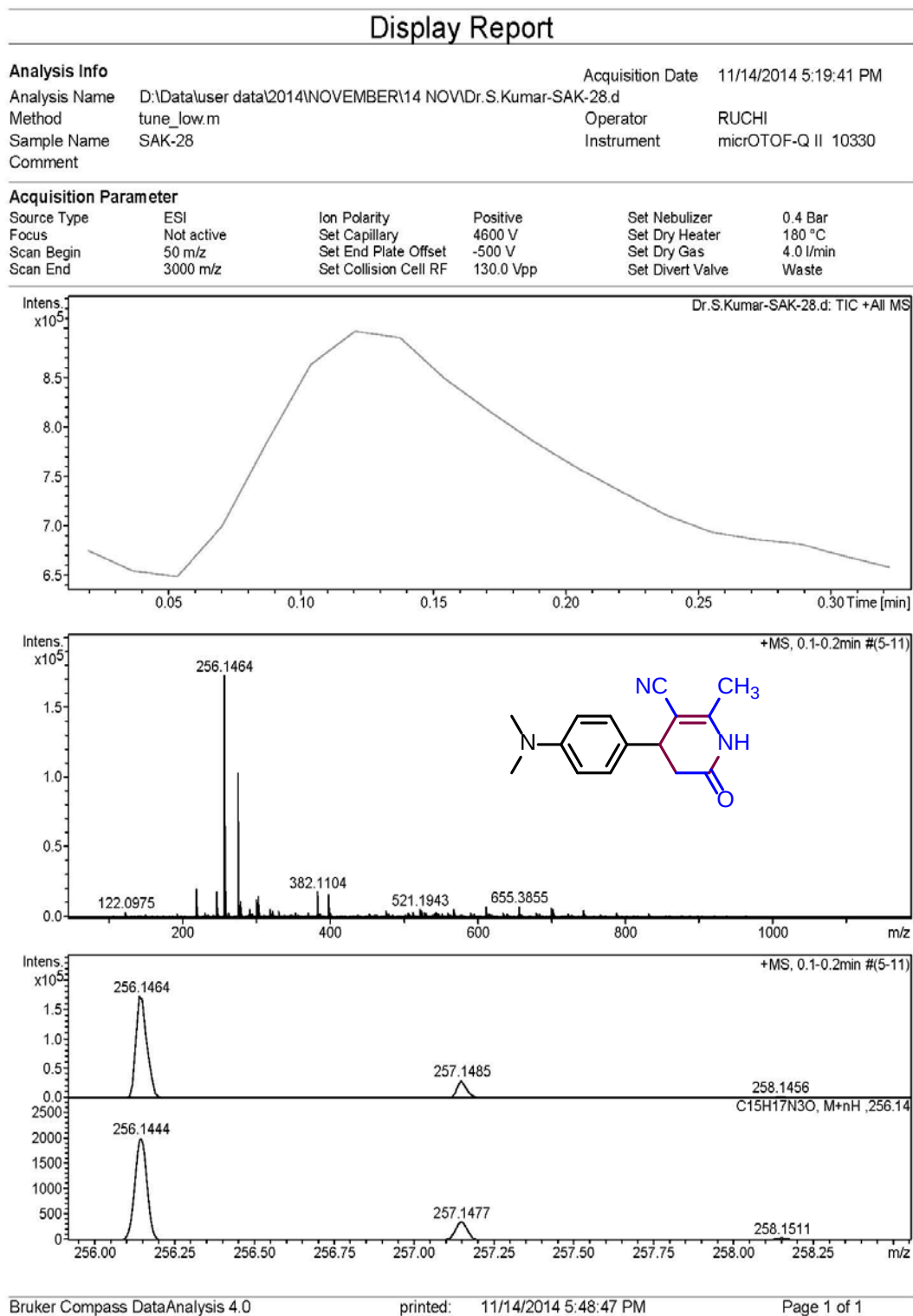


Figure S53 ¹H NMR spectra of **18**

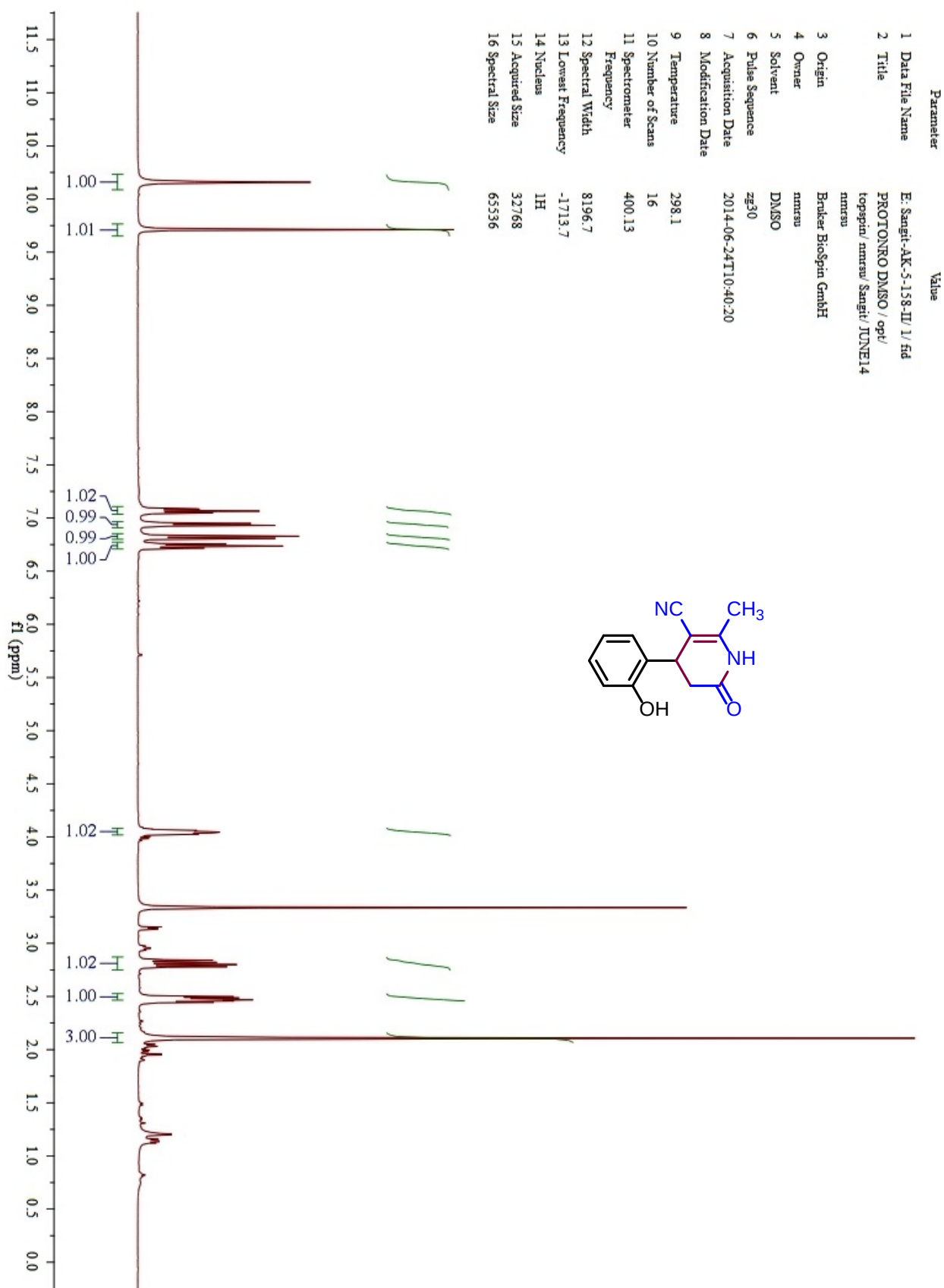


Figure S54 ^{13}C NMR spectra of **18**

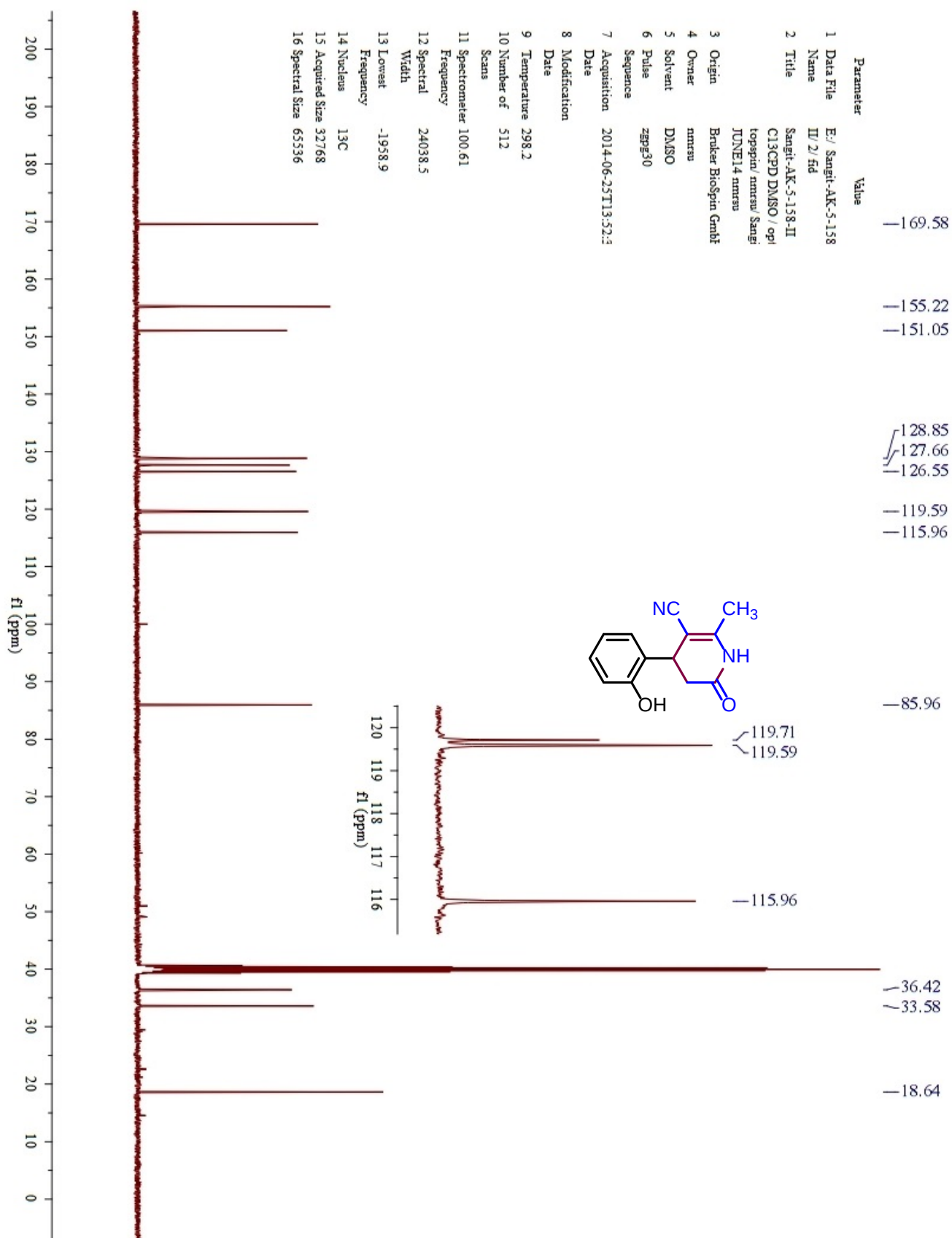


Figure S55 HRMS spectra of **18**

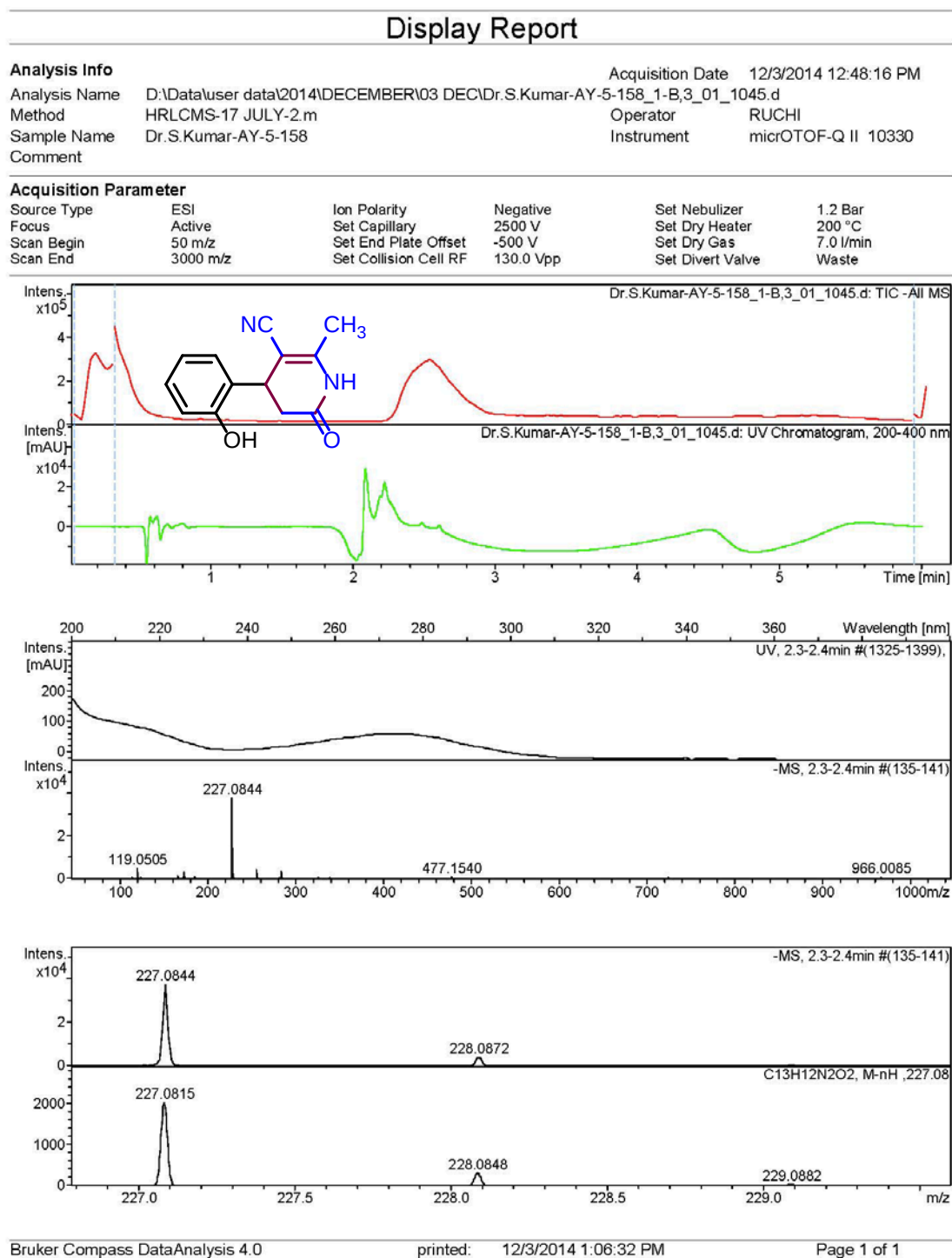


Figure S56 ^1H NMR spectra of **19**

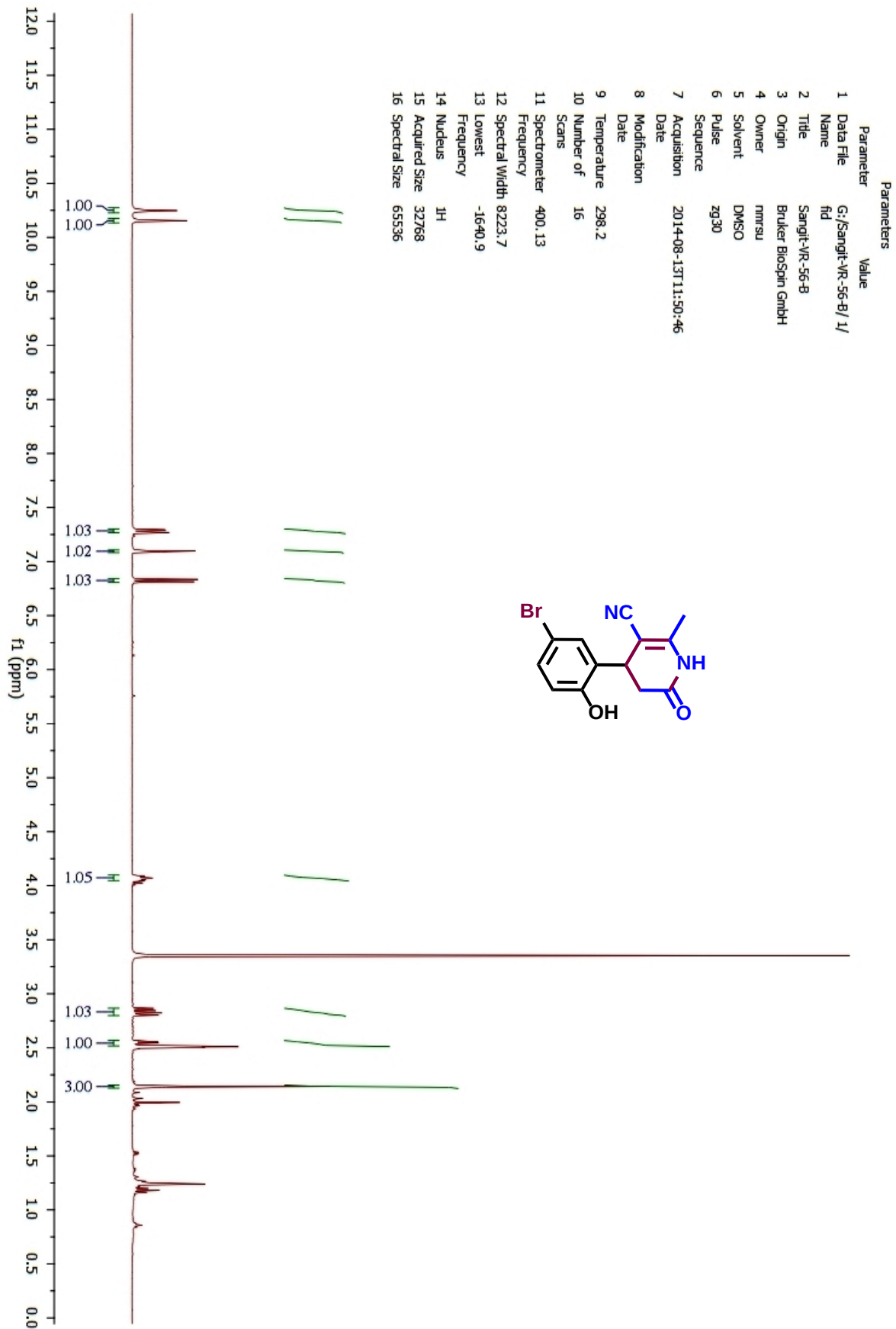


Figure S57 ^{13}C NMR spectra of **19**

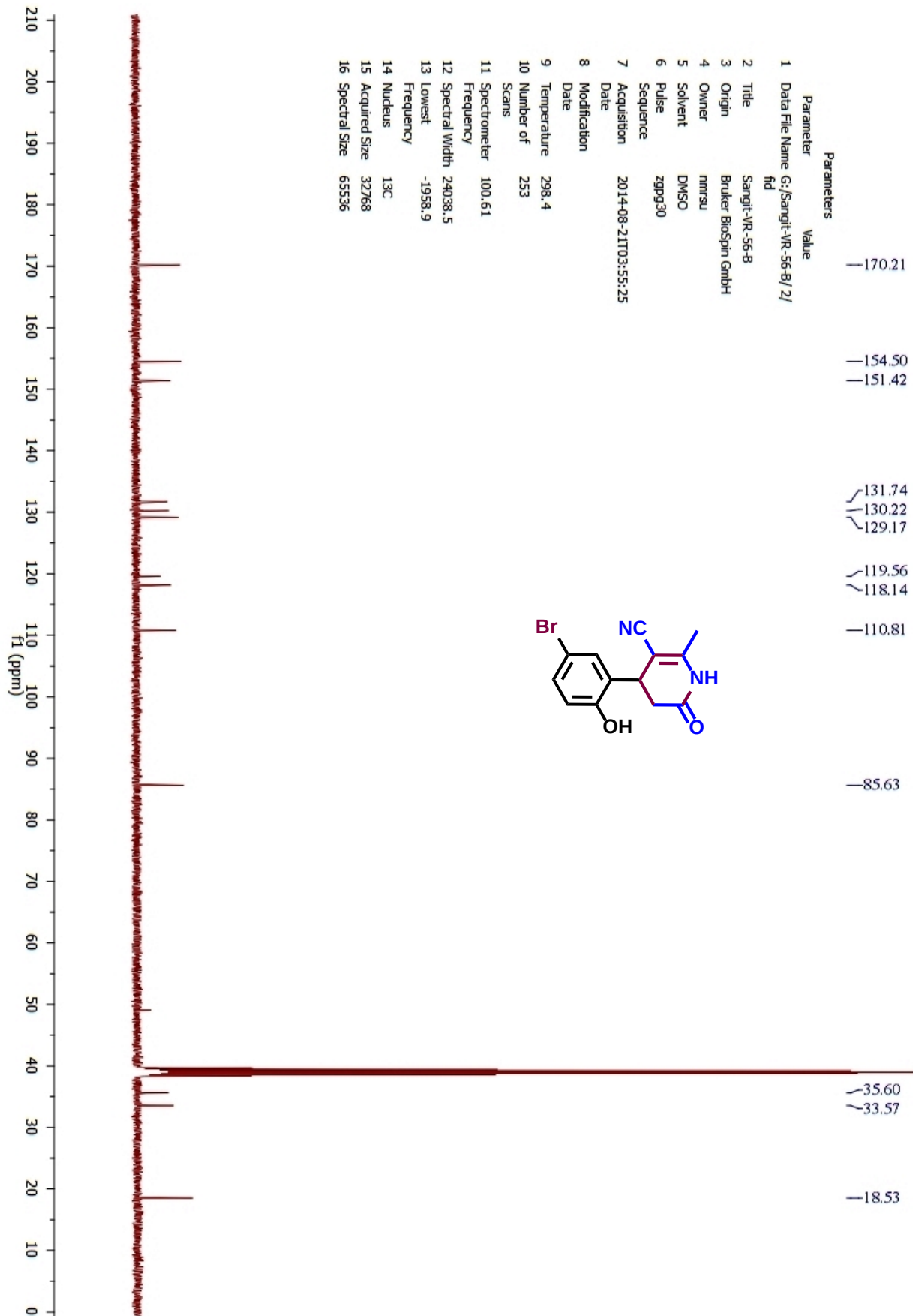


Figure S58 HRMS spectra of **19**

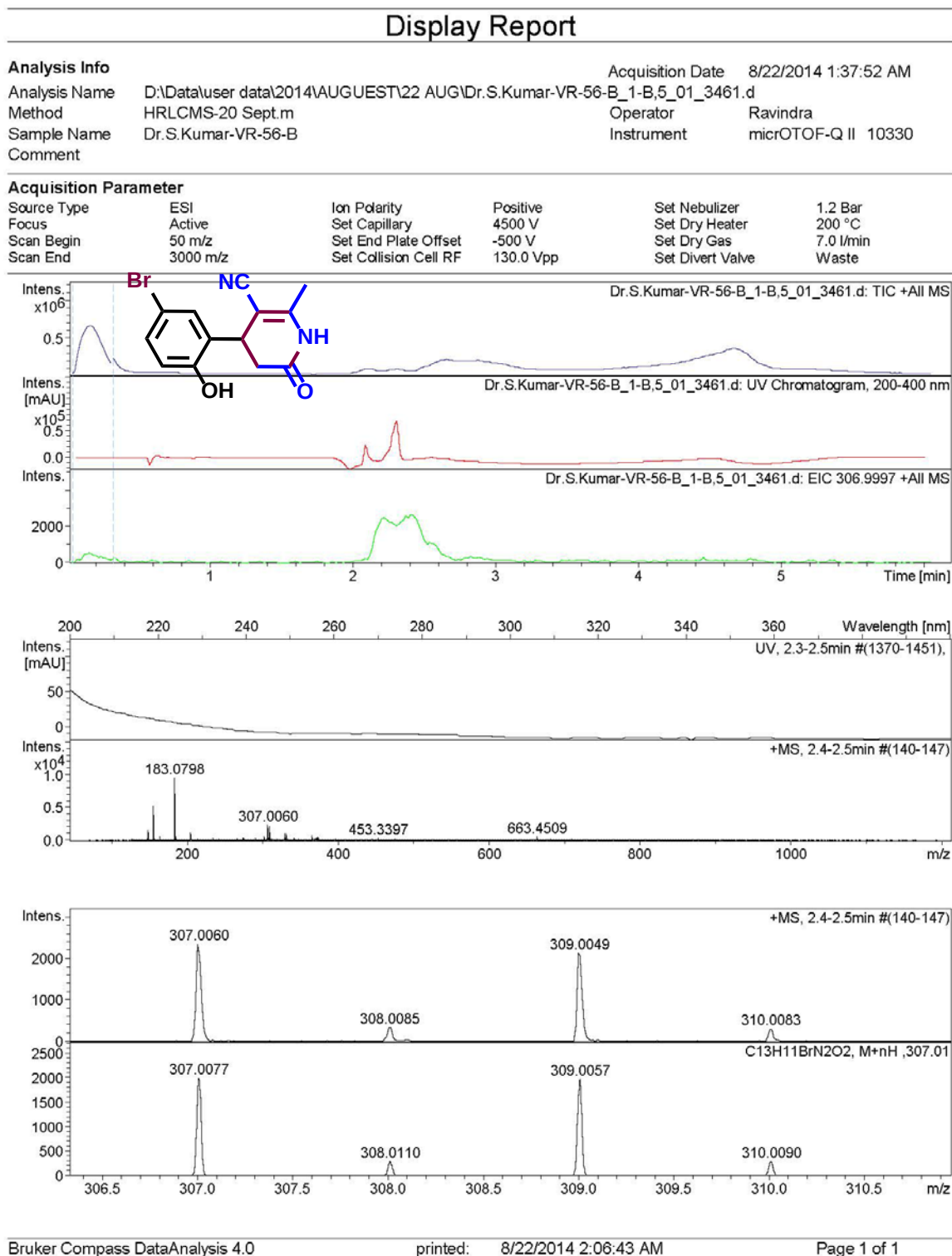


Figure S59 ¹H NMR spectra of **20**

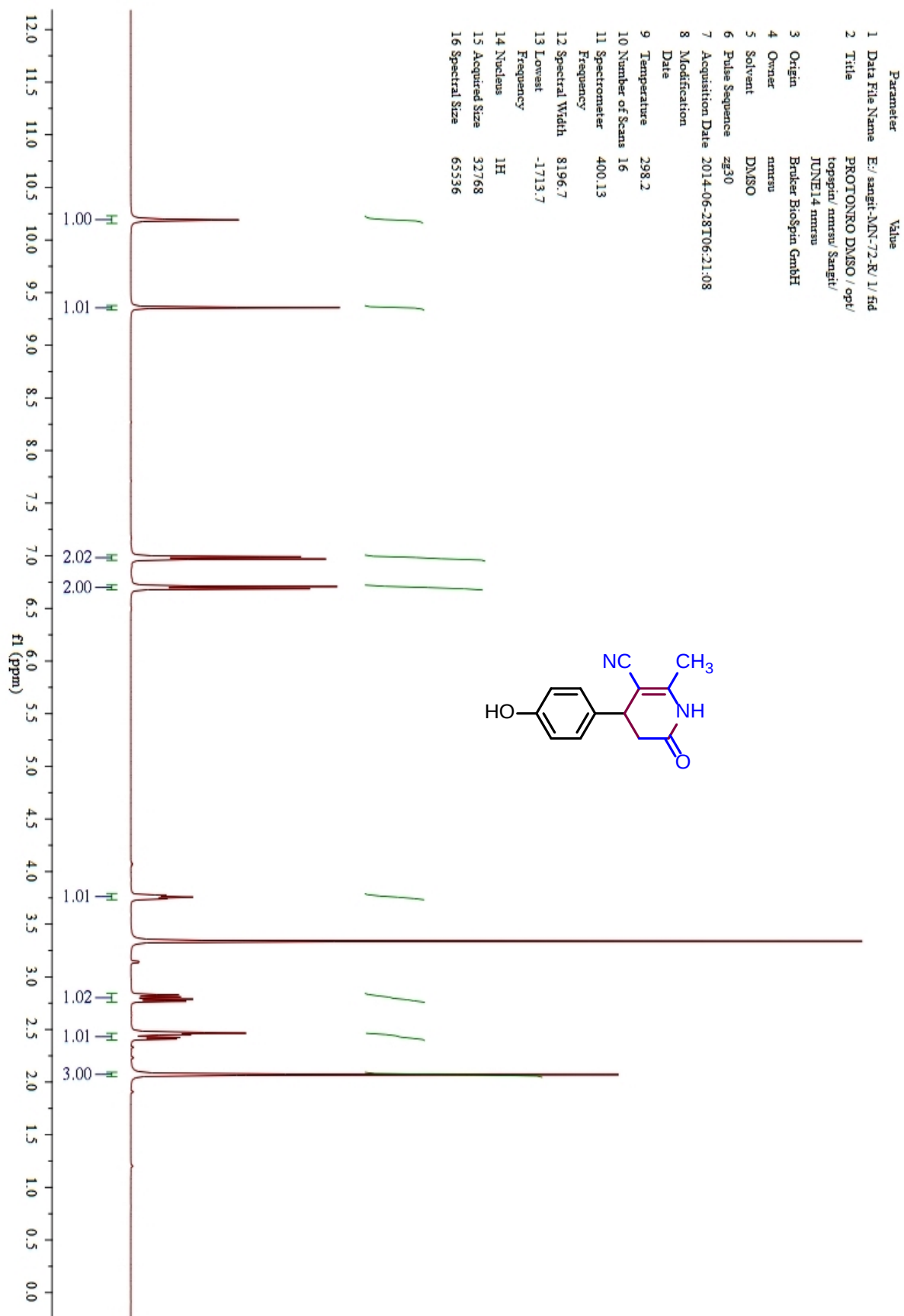


Figure S60 ^{13}C NMR spectra of **20**

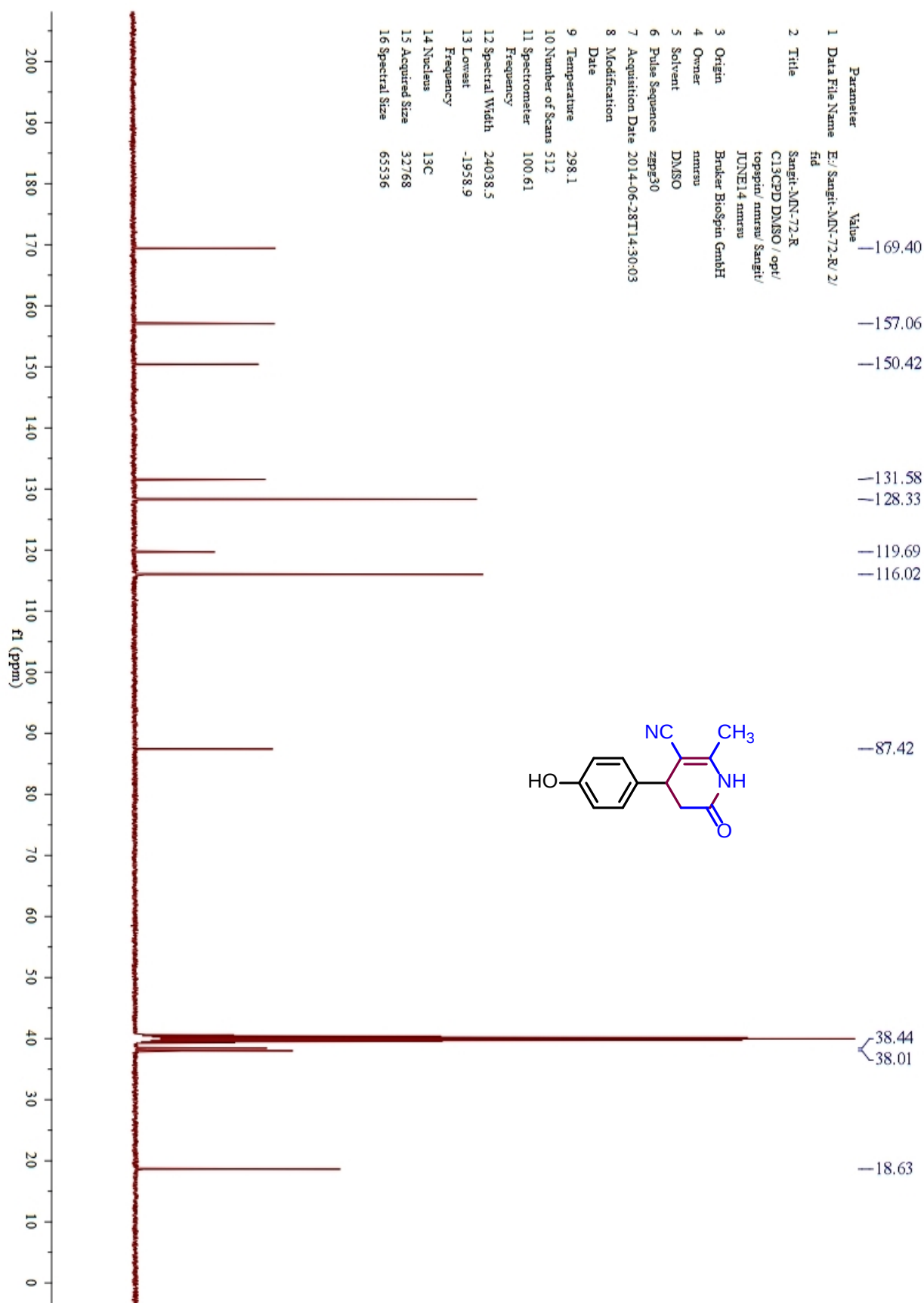


Figure S61 Mass spectra of **20**

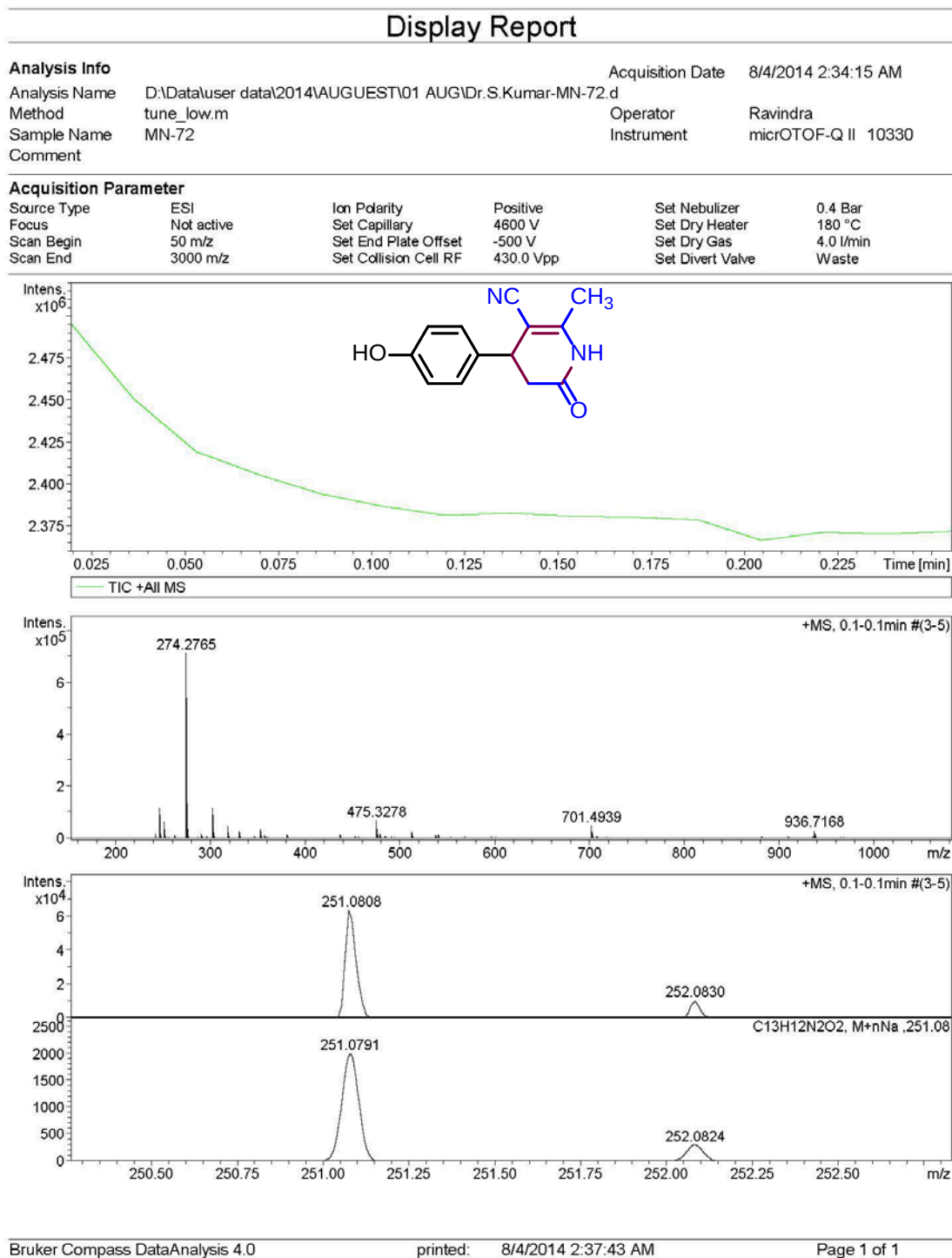


Figure S62 ¹H NMR spectra of **21**

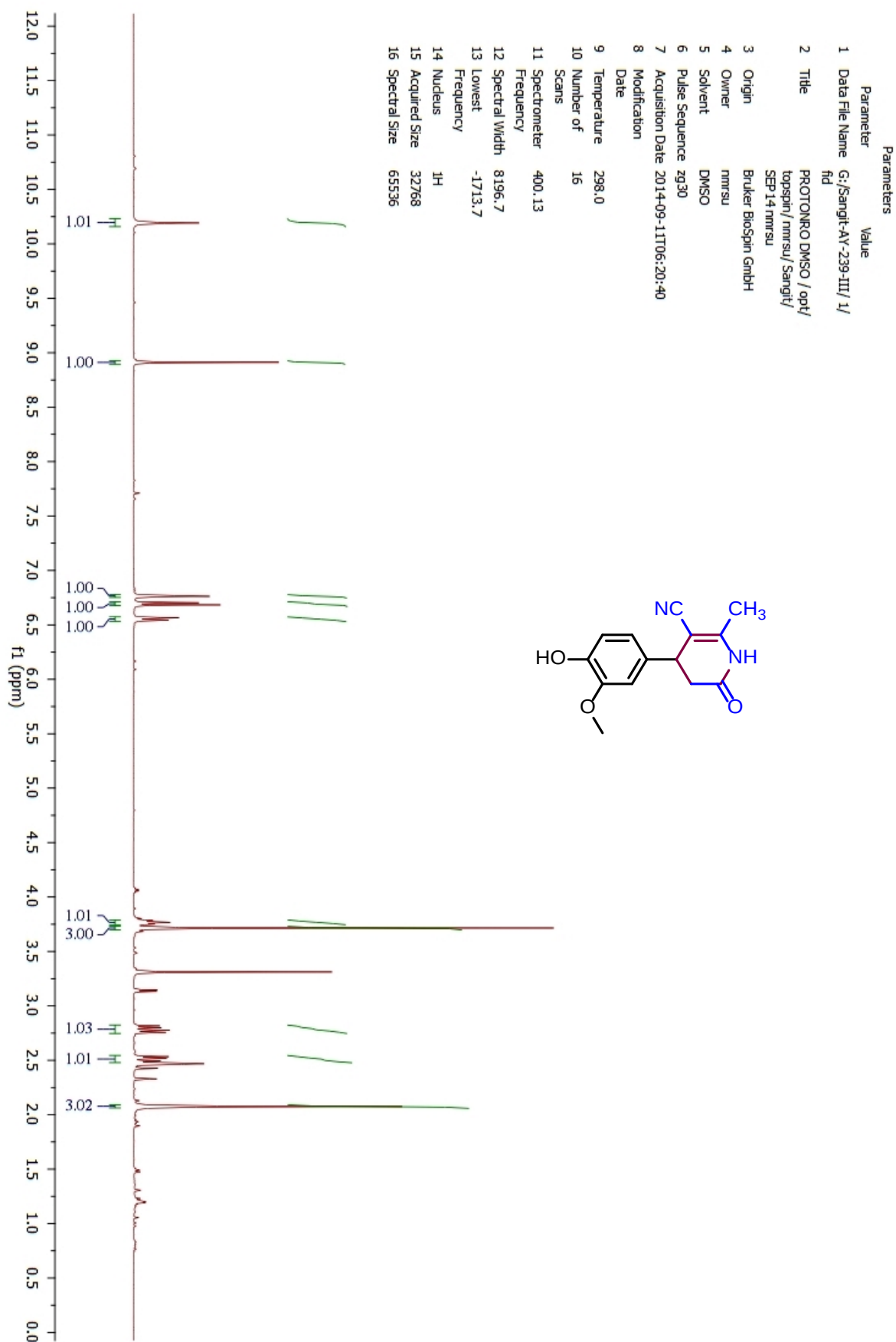


Figure S63 ¹³C NMR spectra of **21**

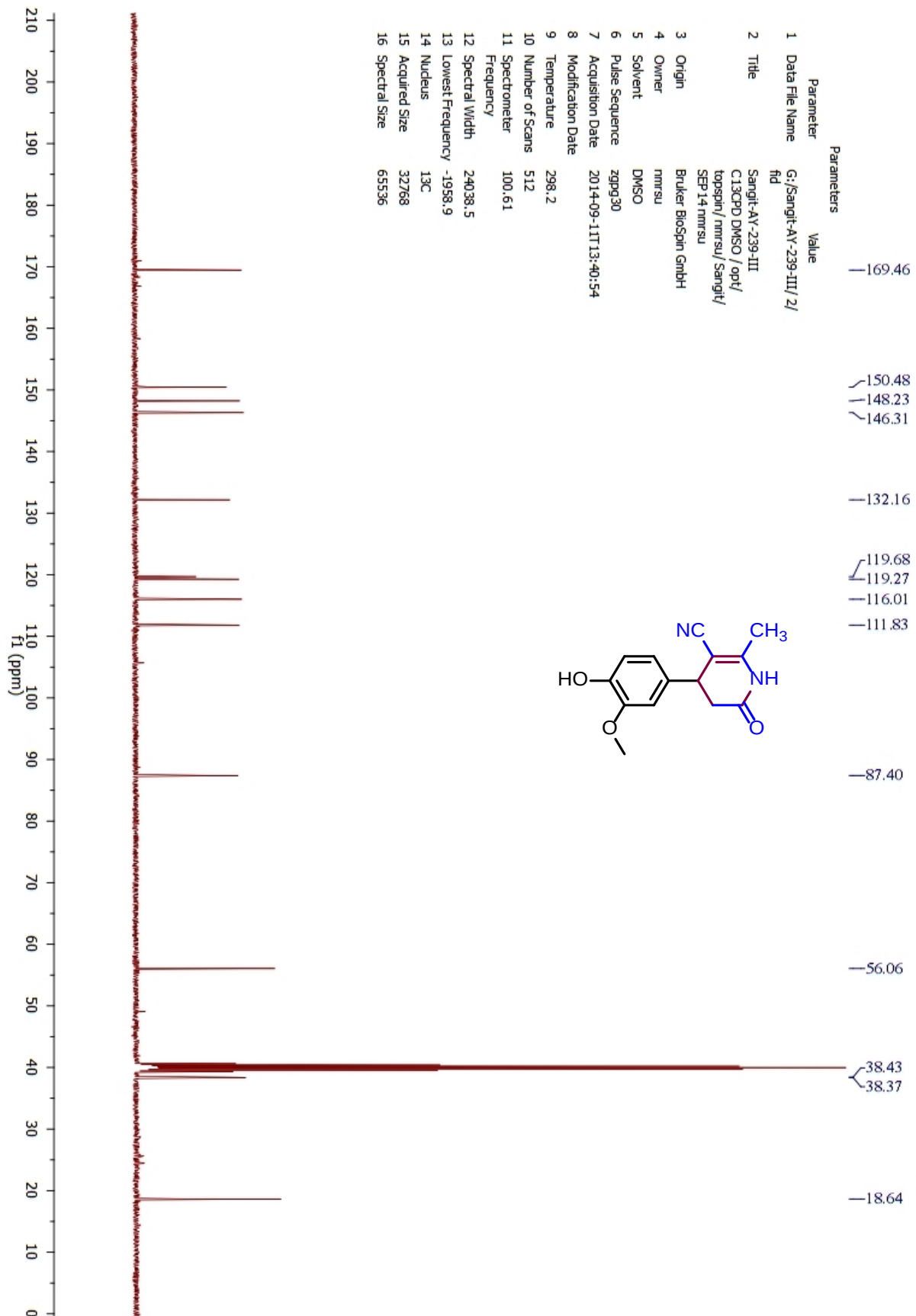


Figure S64 HRMS spectra of **21**

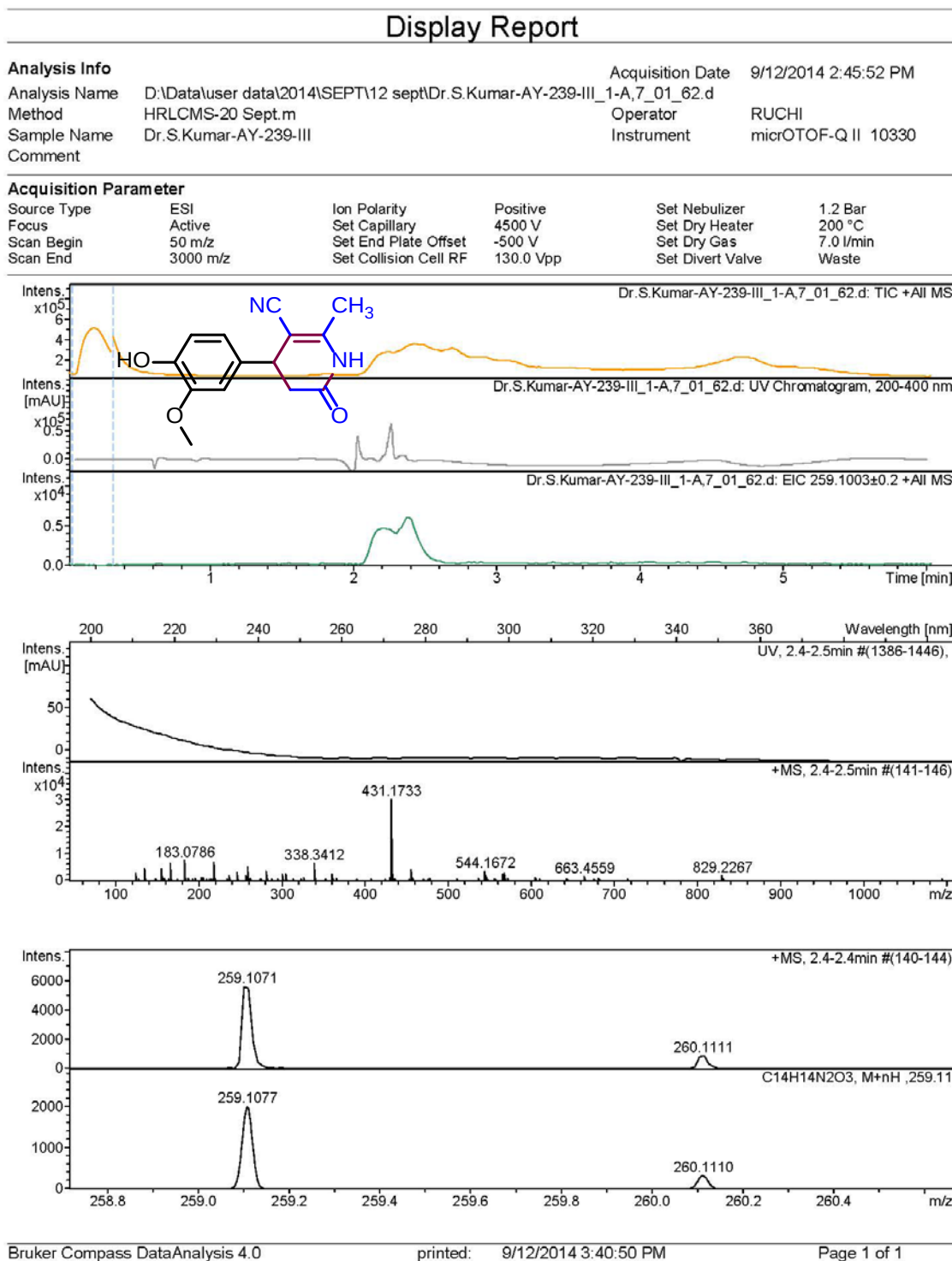


Figure S65 ¹H NMR spectra of 22

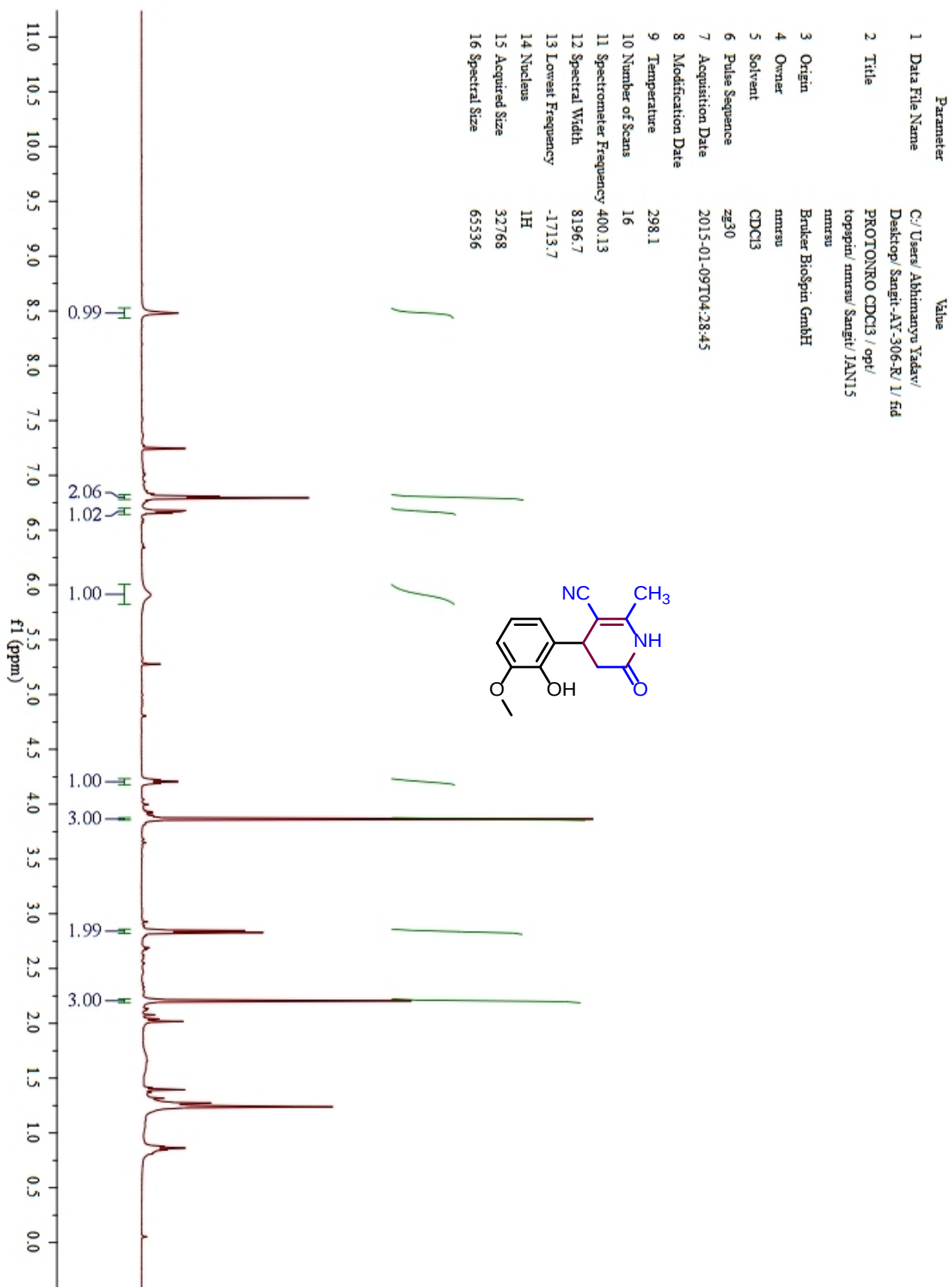


Figure S66 ^{13}C NMR spectra of 22

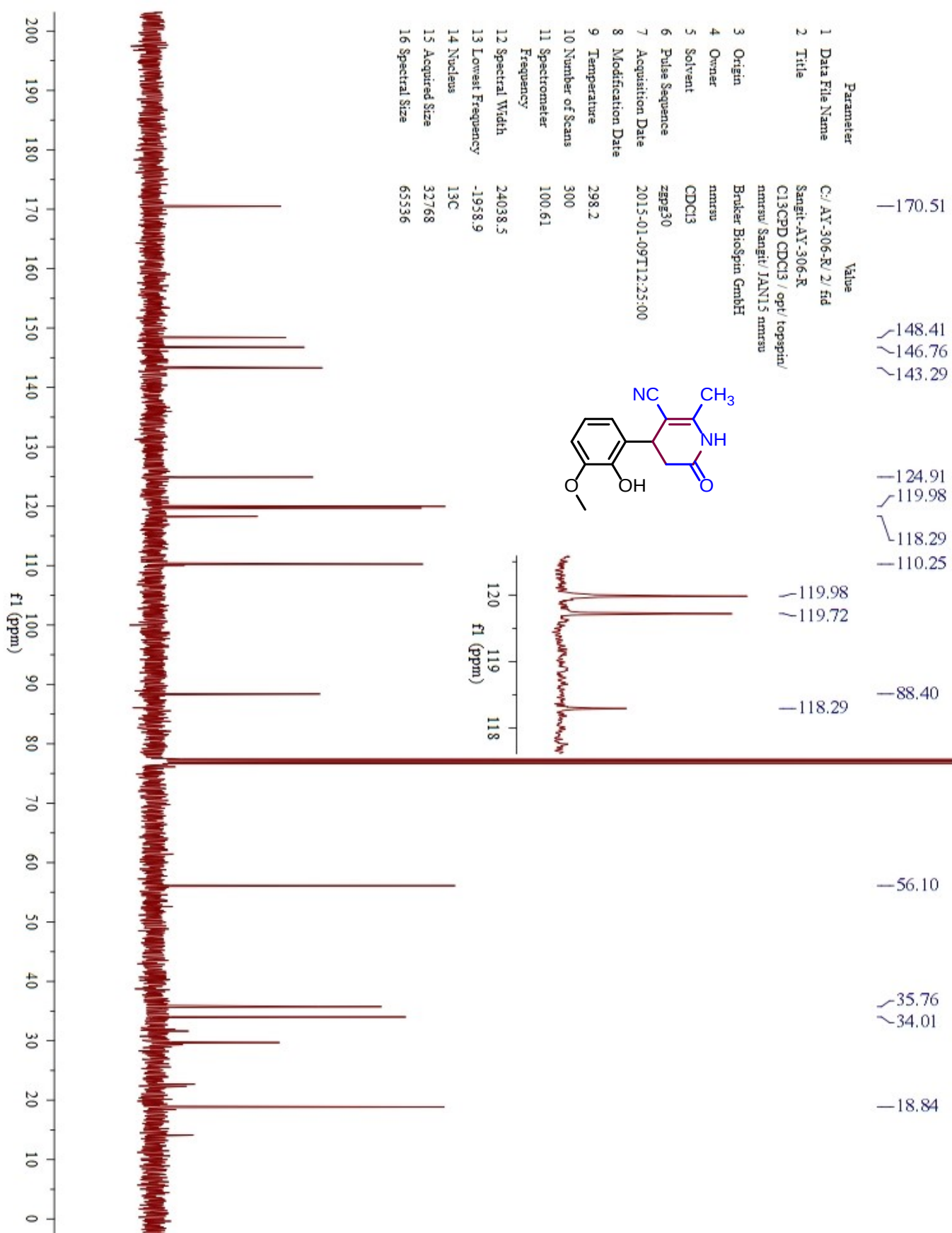


Figure S67 HRMS spectra of 22

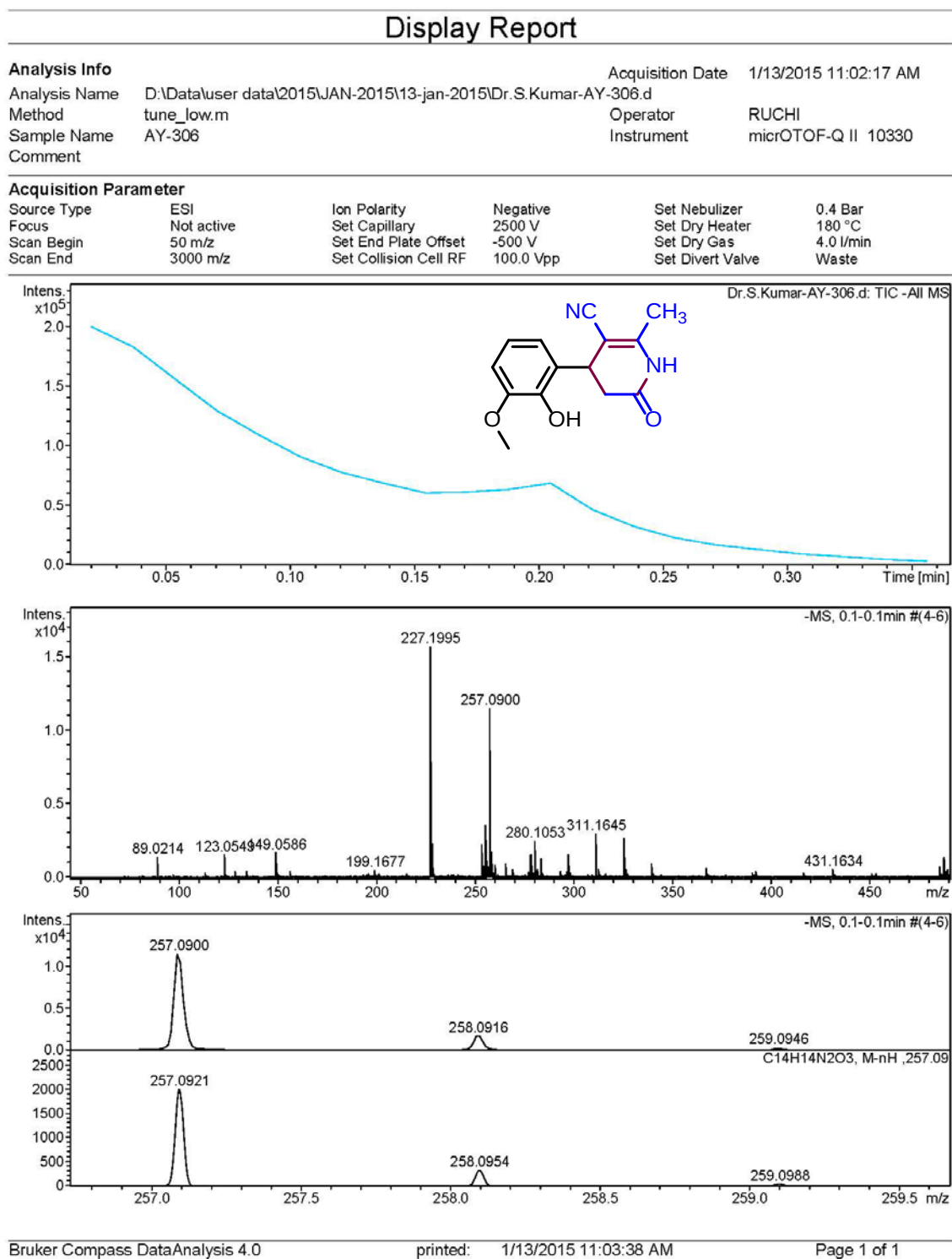


Figure S68 ¹H NMR spectra of 23

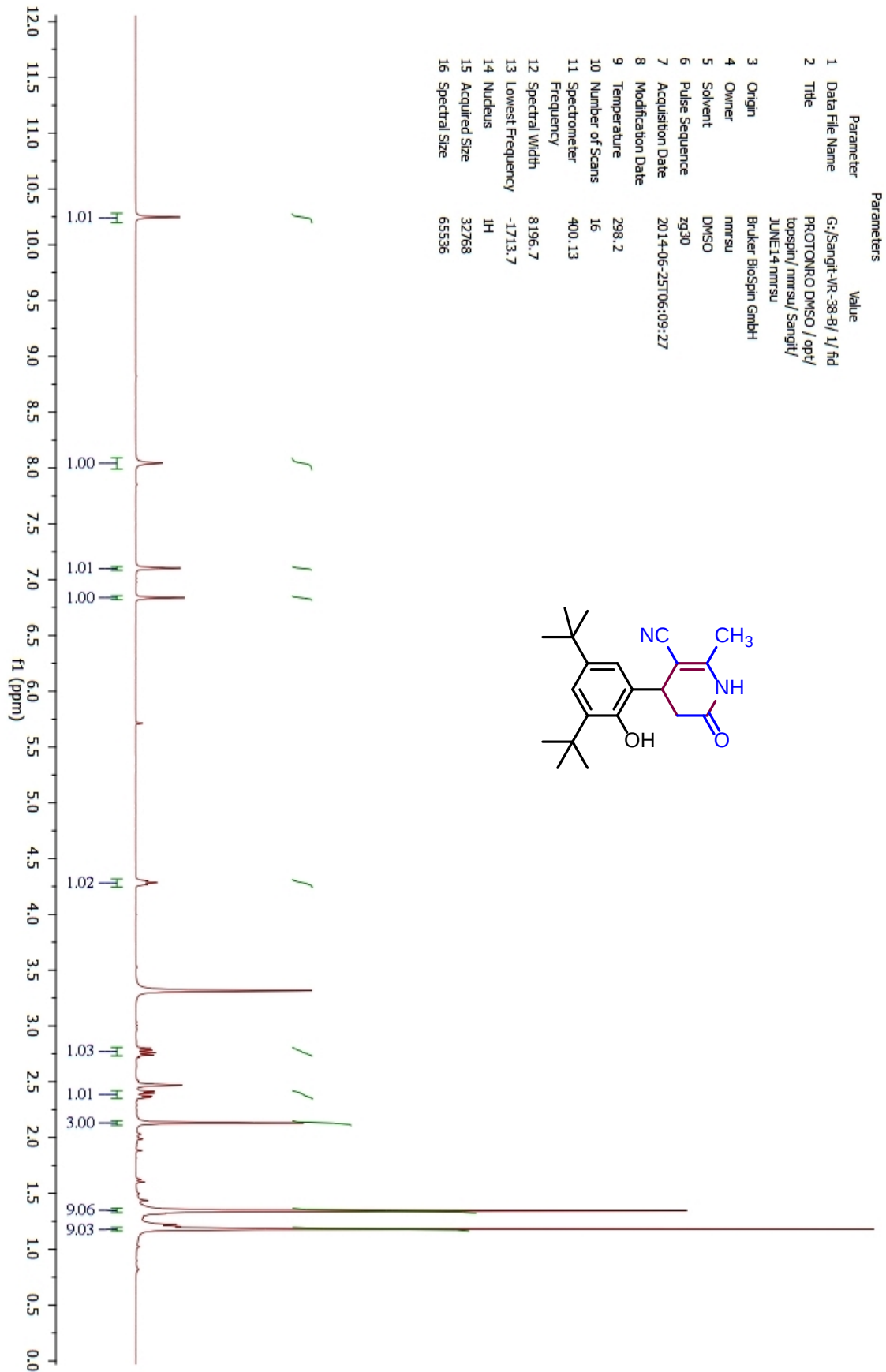


Figure S69 ^{13}C NMR spectra of **23**

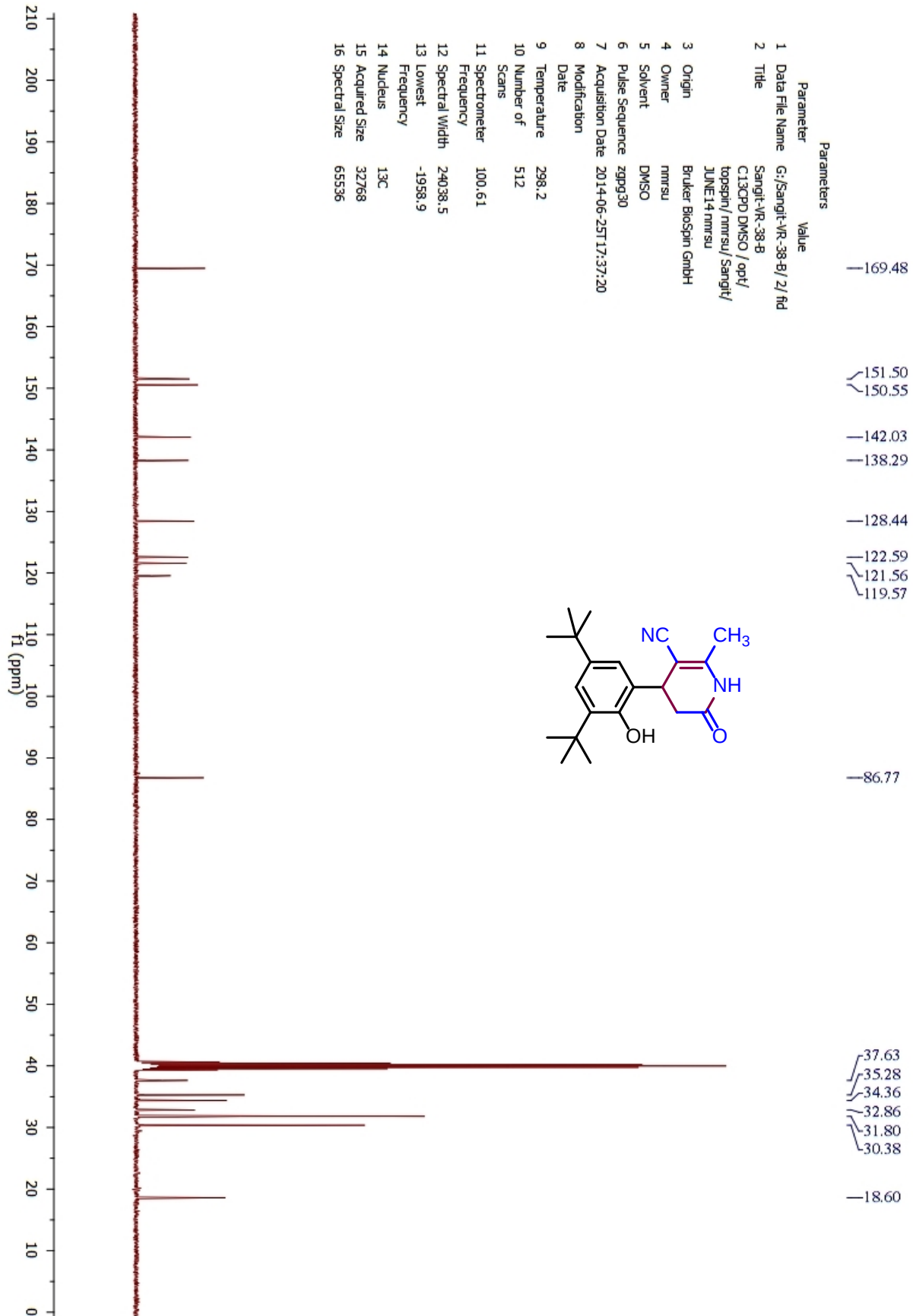


Figure S70 HRMS spectra of 23

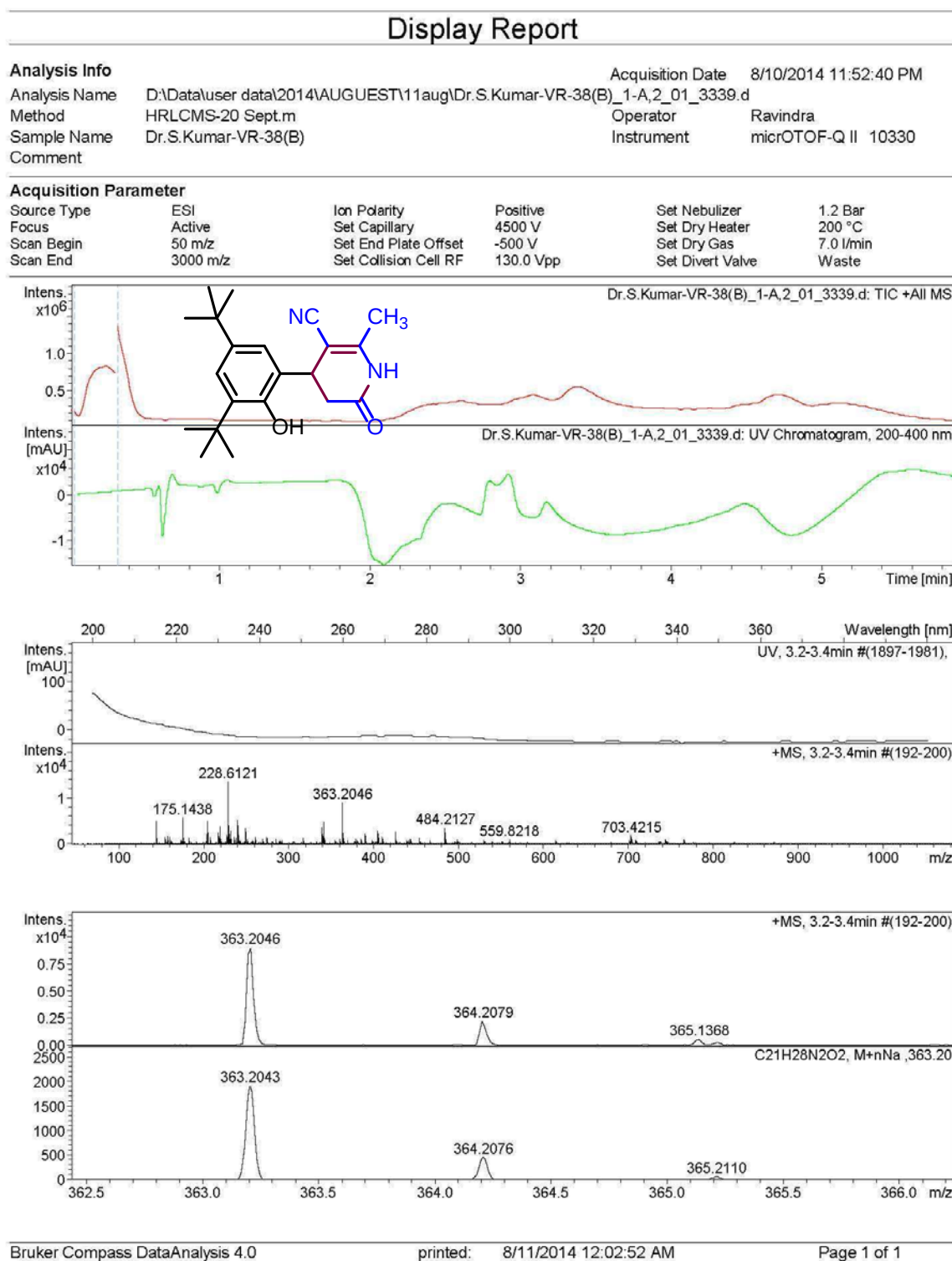


Figure S71 ¹H NMR spectra of **24**

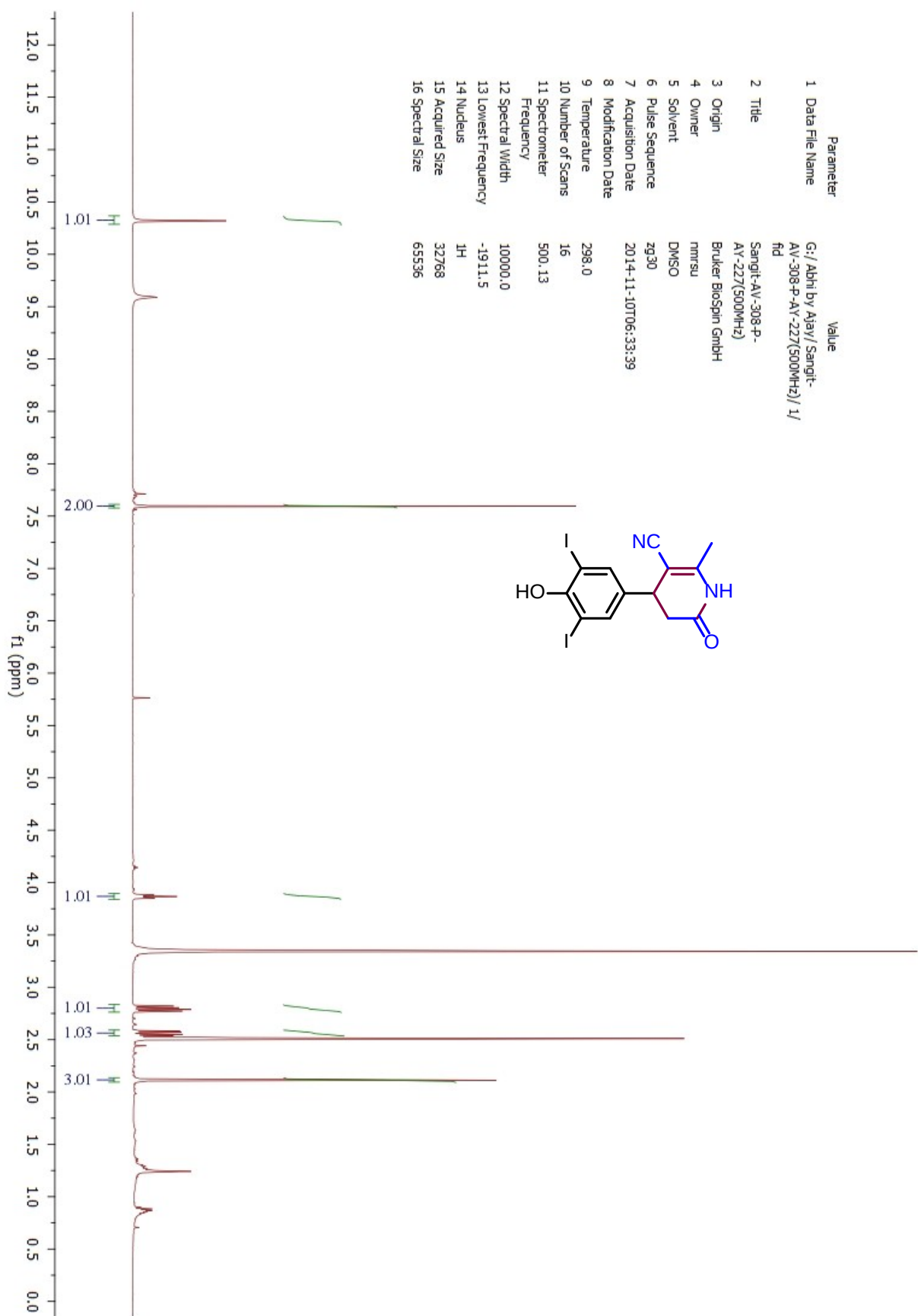


Figure S72 ¹³C NMR spectra of **24**

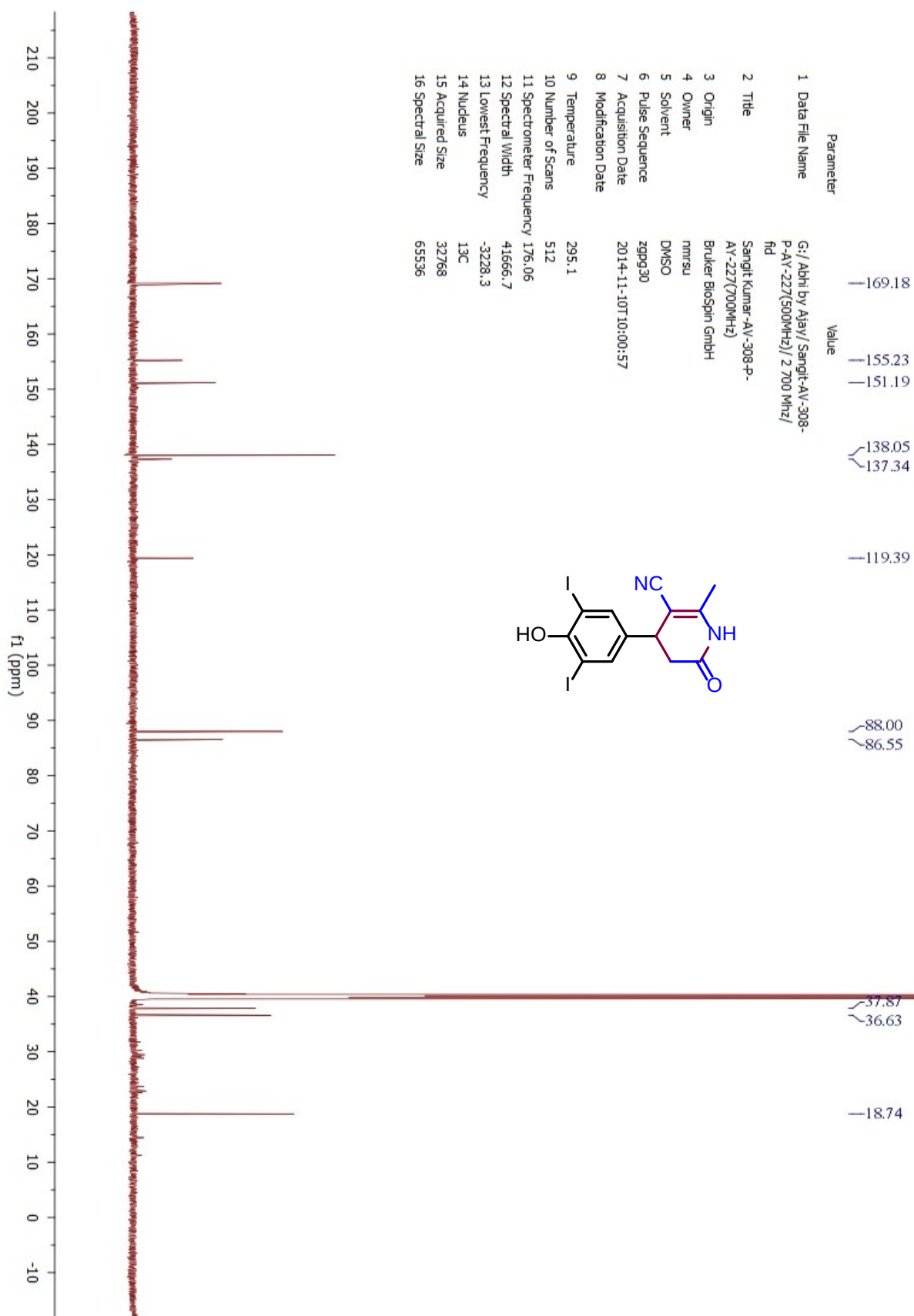


Figure S73 HRMS spectra of **24**

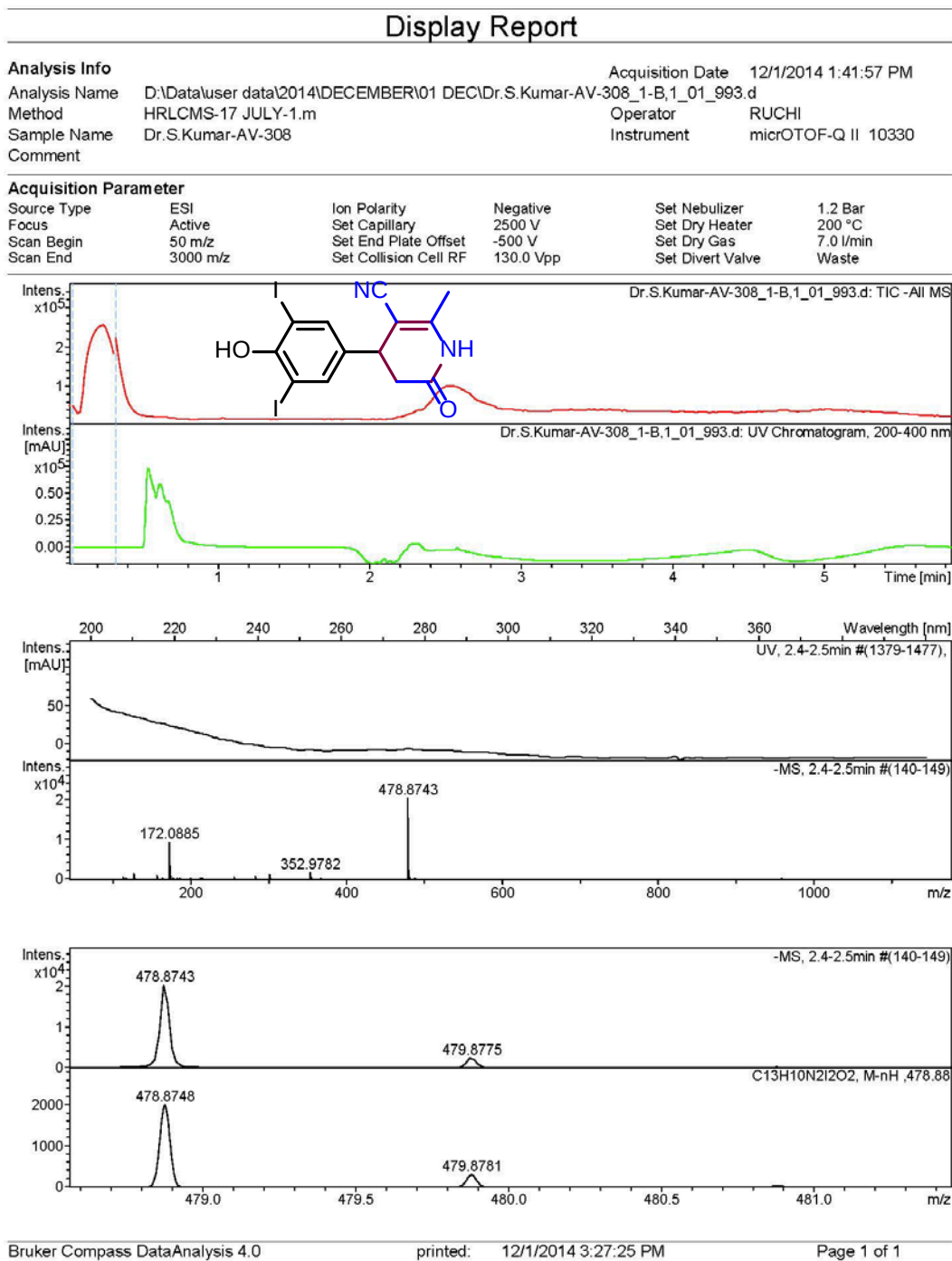


Figure S74 ^1H NMR spectra of 25

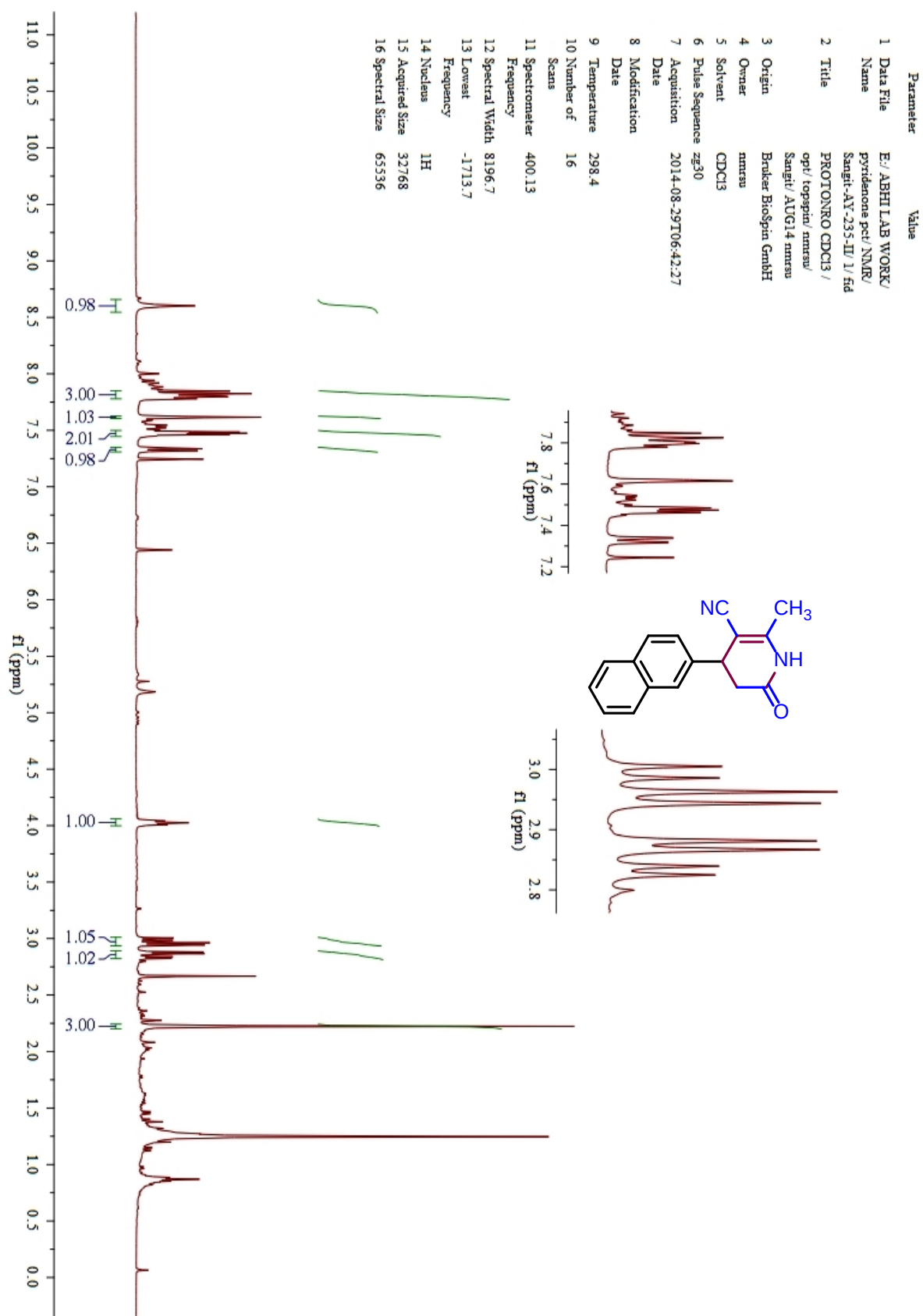


Figure S75 ¹³C NMR spectra of 25

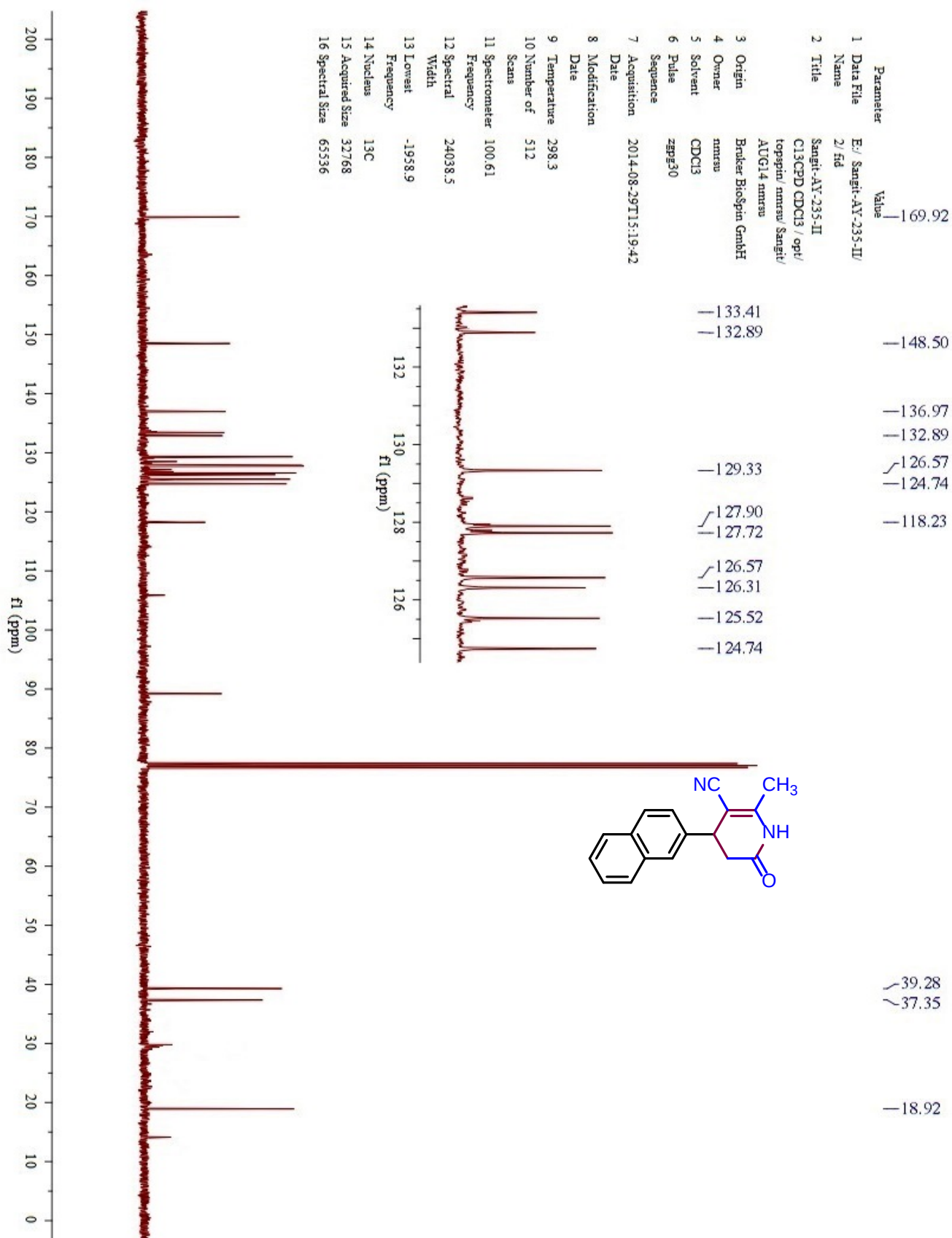
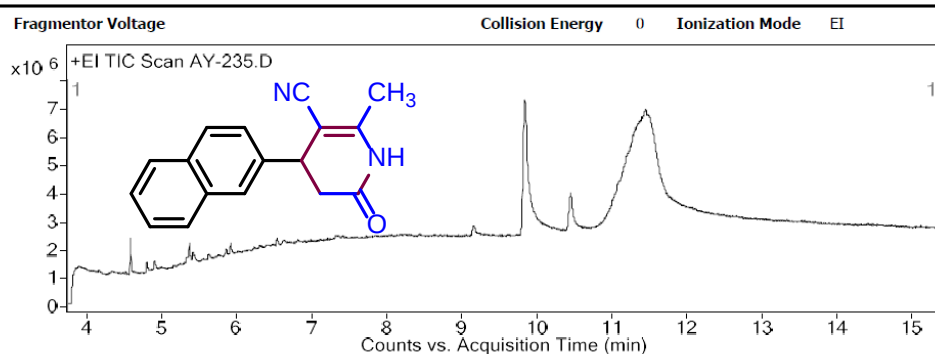


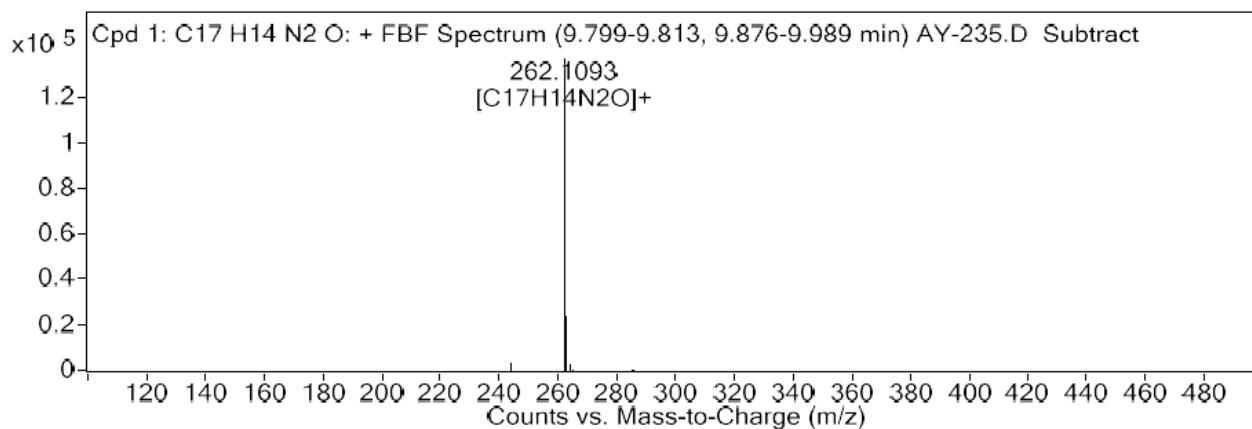
Figure S76 GC HRMS spectra of 25

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IRM Calibration Status	Success	DA Method	iiSER13MAY 2013.m
Comment			
Expected Barcode		Sample Amount	
Dual Inj Vol	0.1	TuneName	Install_tune.ei.tune_1_4_13.tune.xml
TunePath	D:\MassHunter\GCMS\1\7200	TuneDateStamp	41421.21309
MSFirmwareVersion	G.7200.01.09	OperatorName	Agilent-PC\admin
RunCompletedFlag	TRUE		

User Chromatograms



Qualitative Analysis Report



--- End Of Report ---

Figure S77 ¹H NMR spectra of **26**

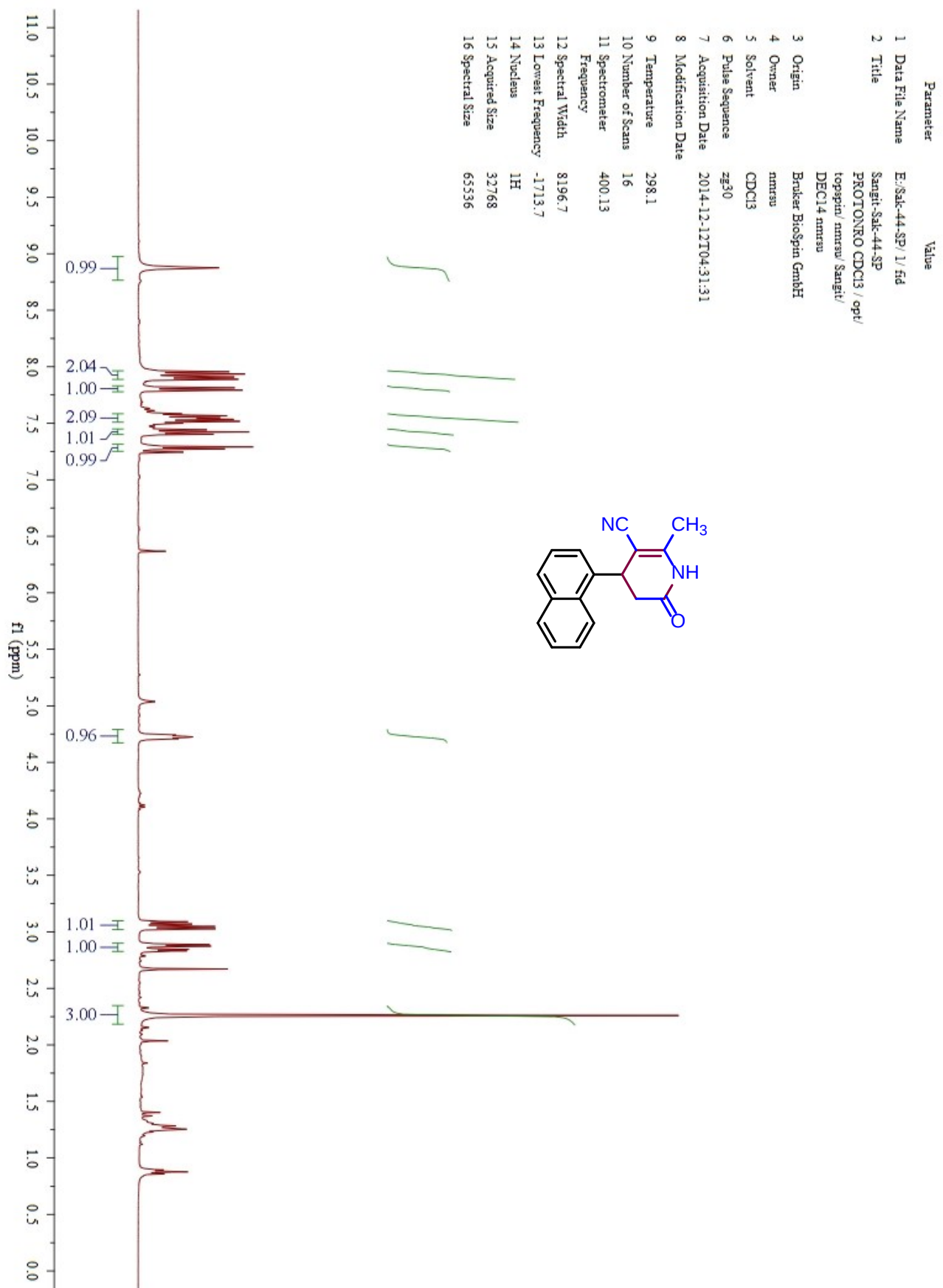


Figure S78 ¹³C NMR spectra of **26**

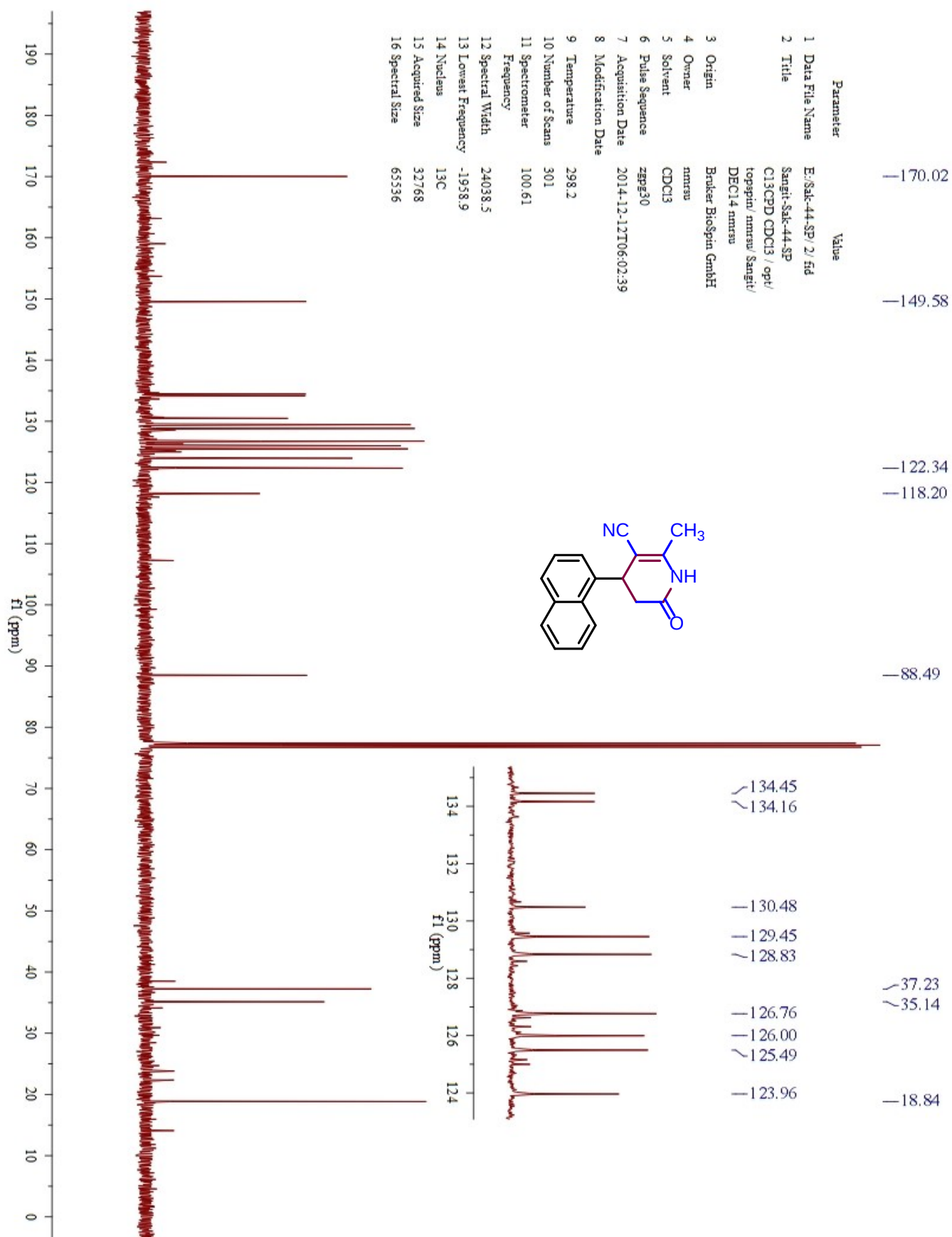


Figure S79 Mass spectra of **26**

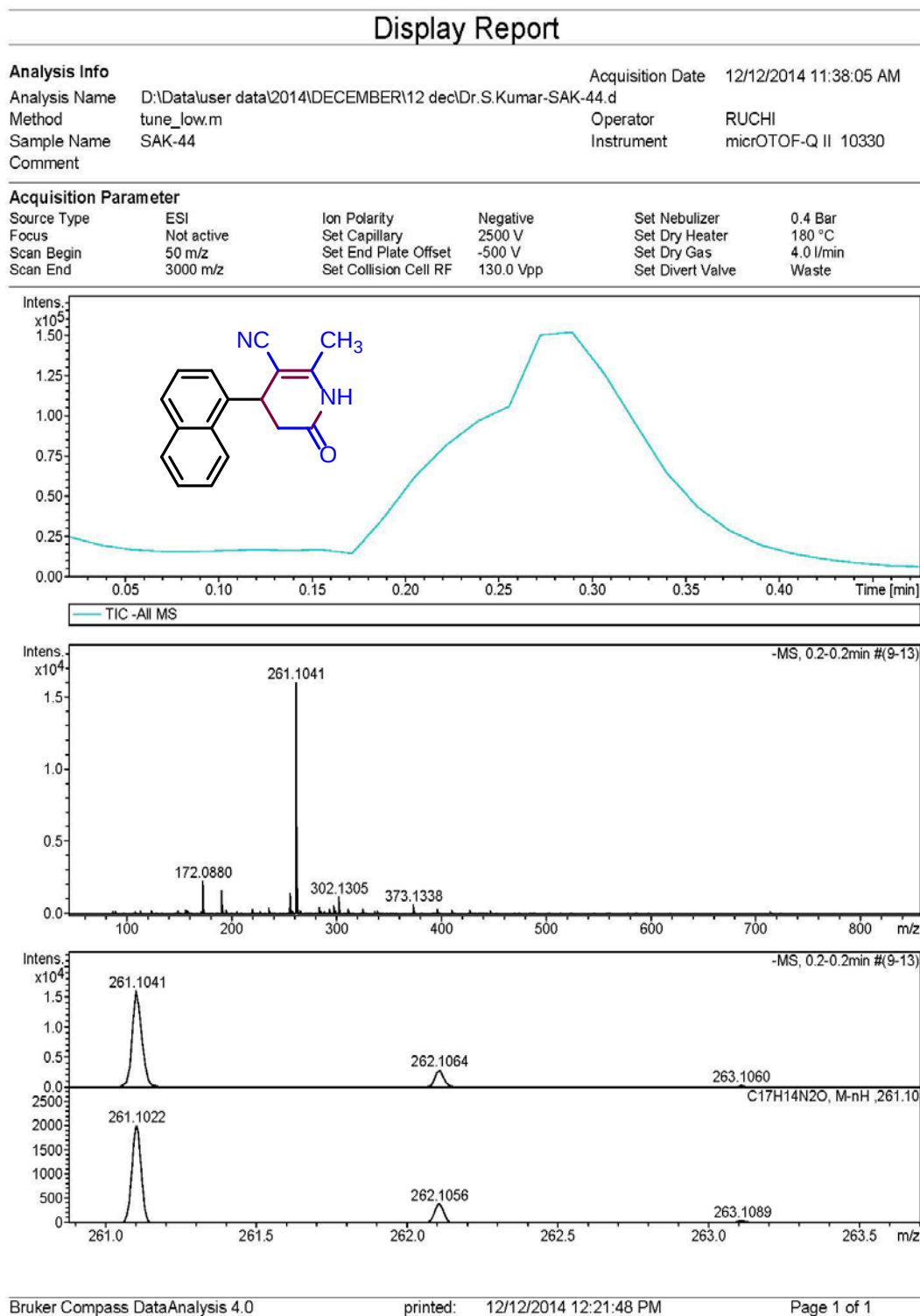


Figure S80 ¹H NMR spectra of 27

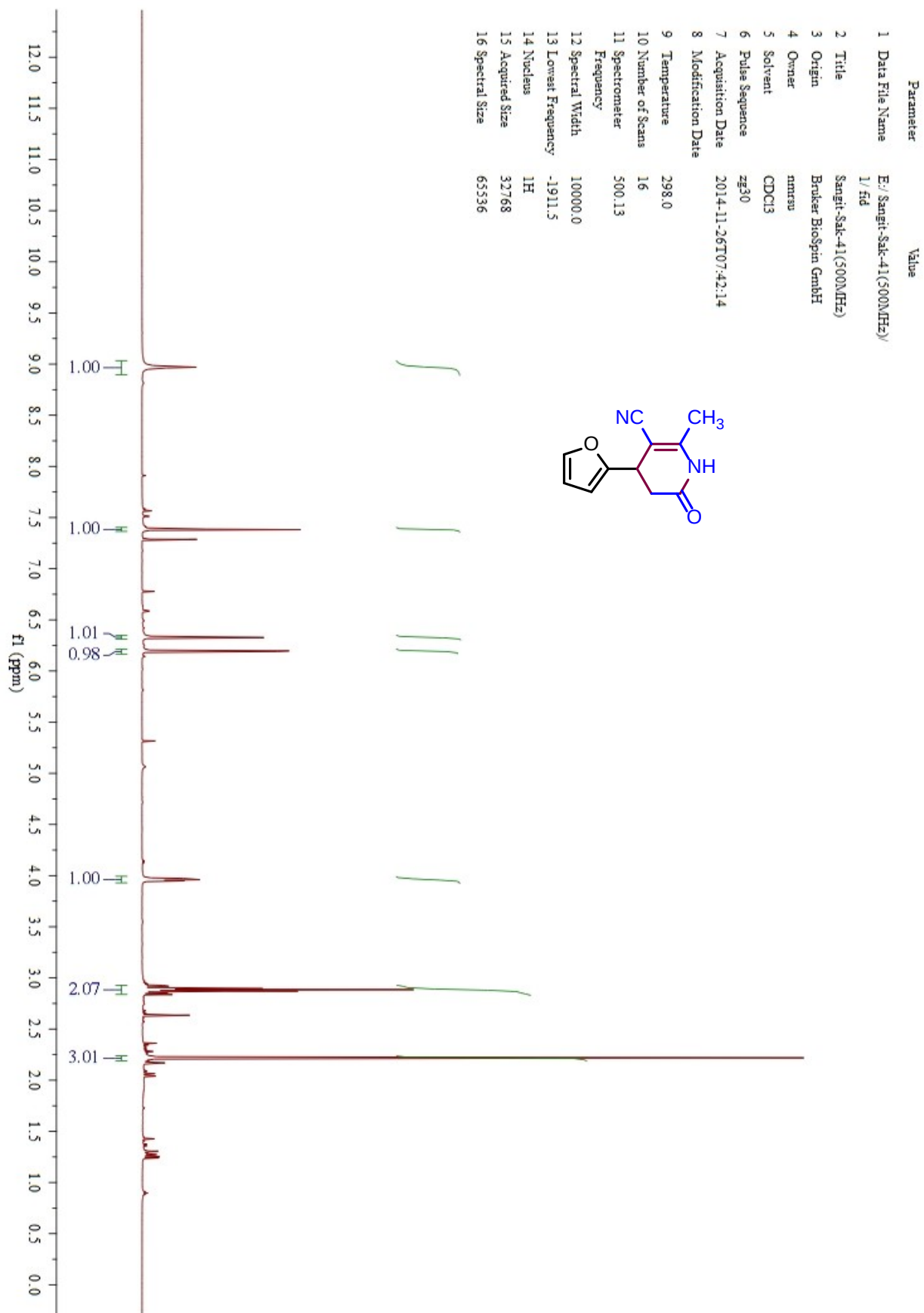


Figure S81 ^{13}C NMR spectra of 27

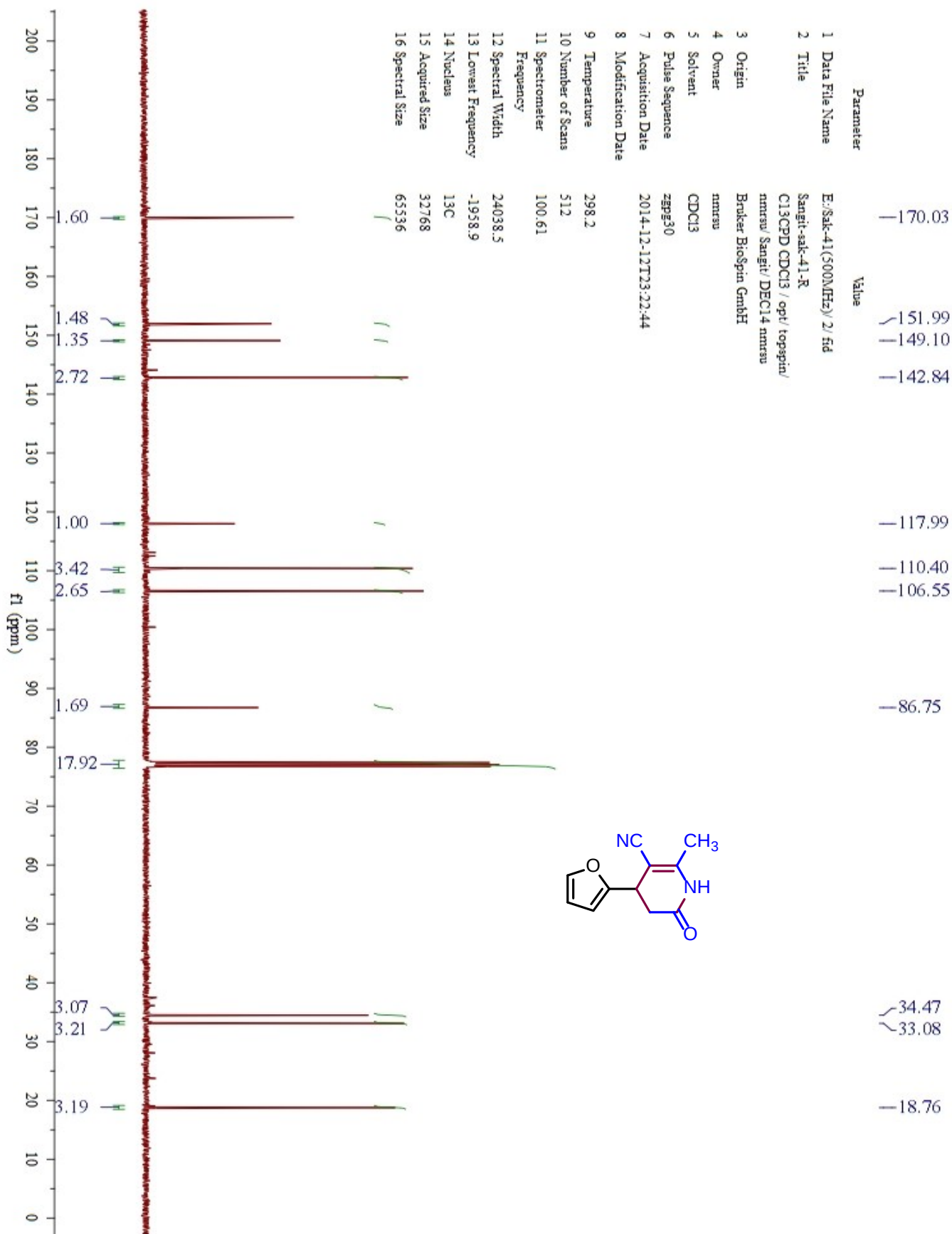


Figure S82 Mass spectra of 27

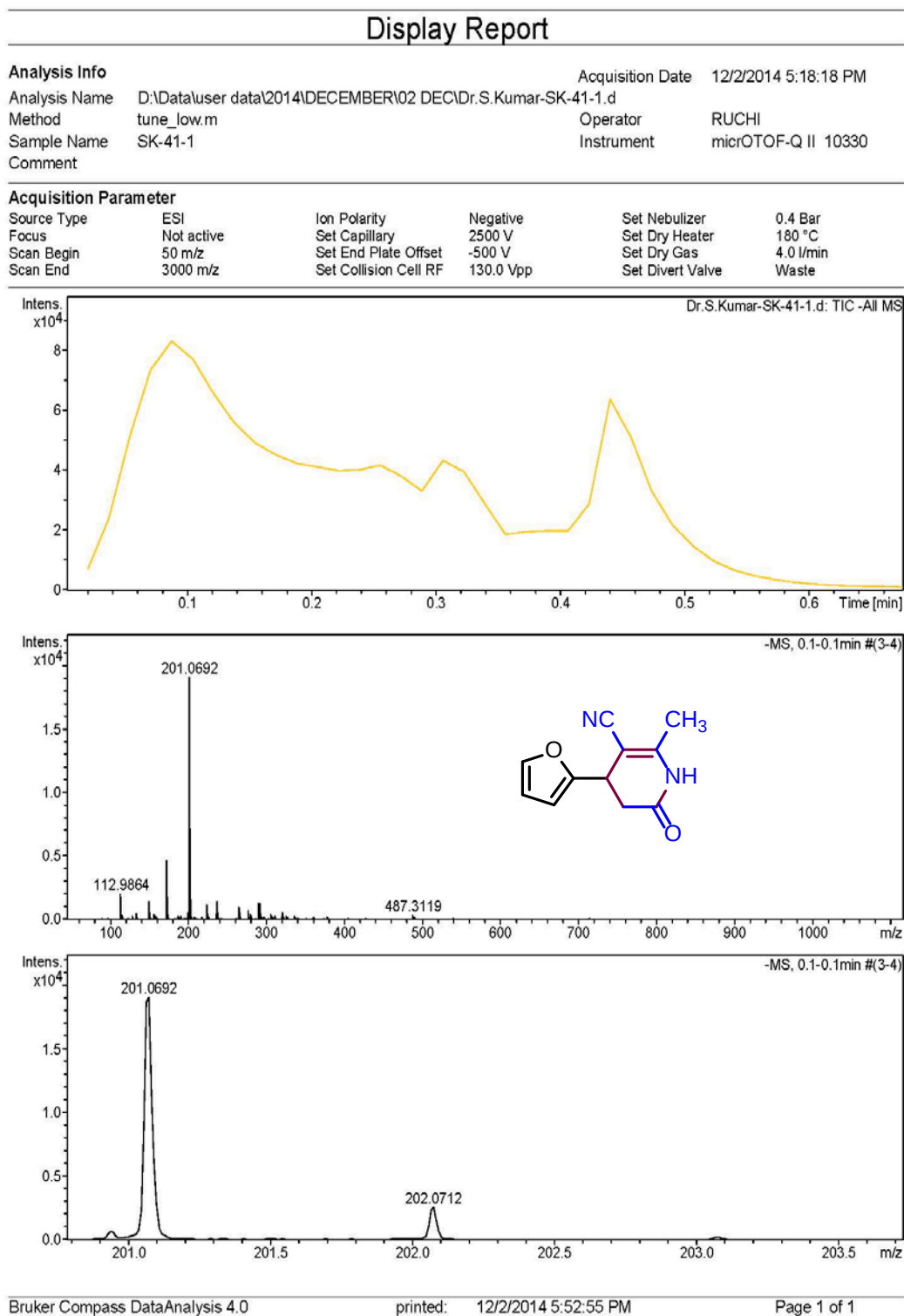


Figure S83 ¹H NMR spectra of **28**

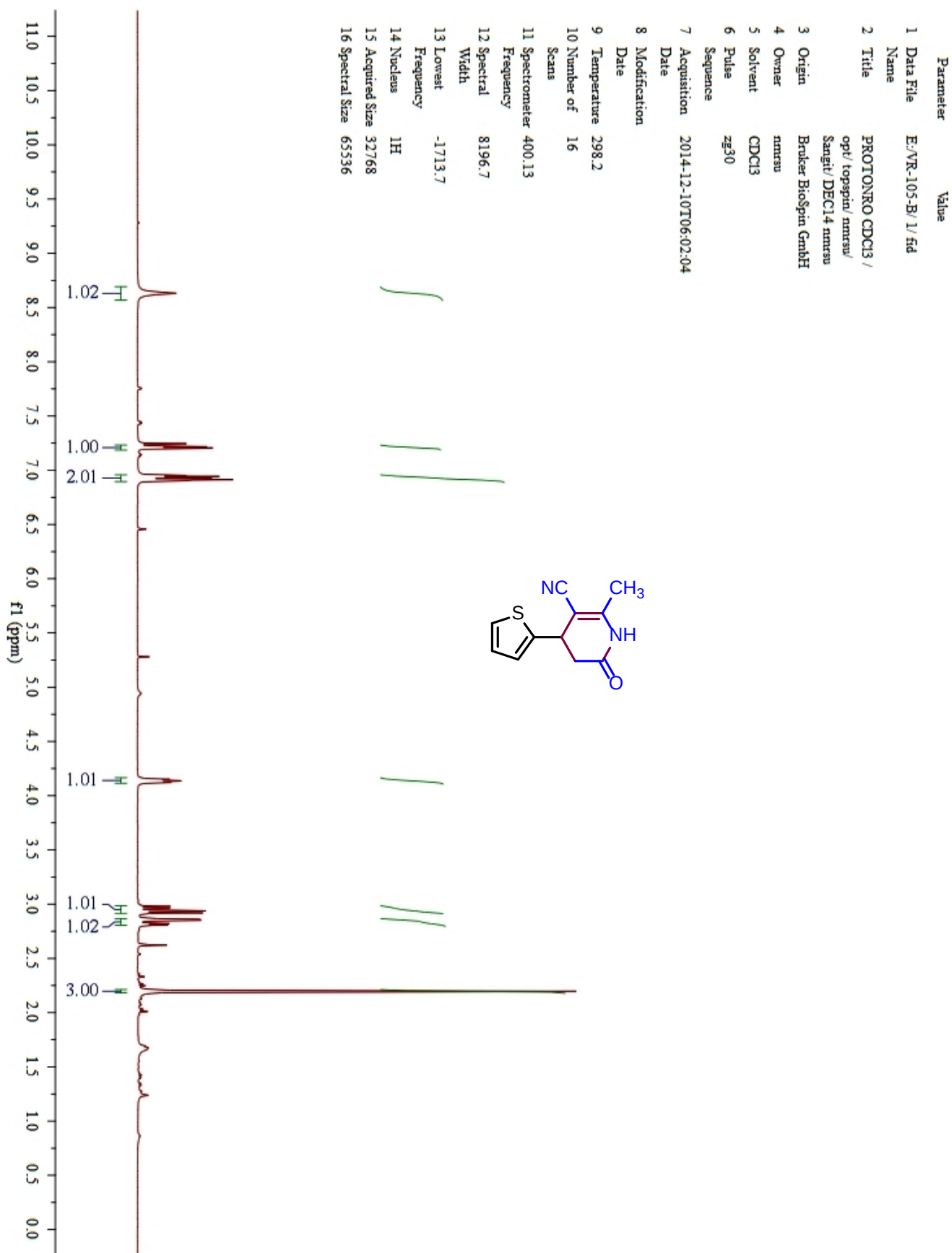


Figure S84 ^{13}C NMR spectra of **28**

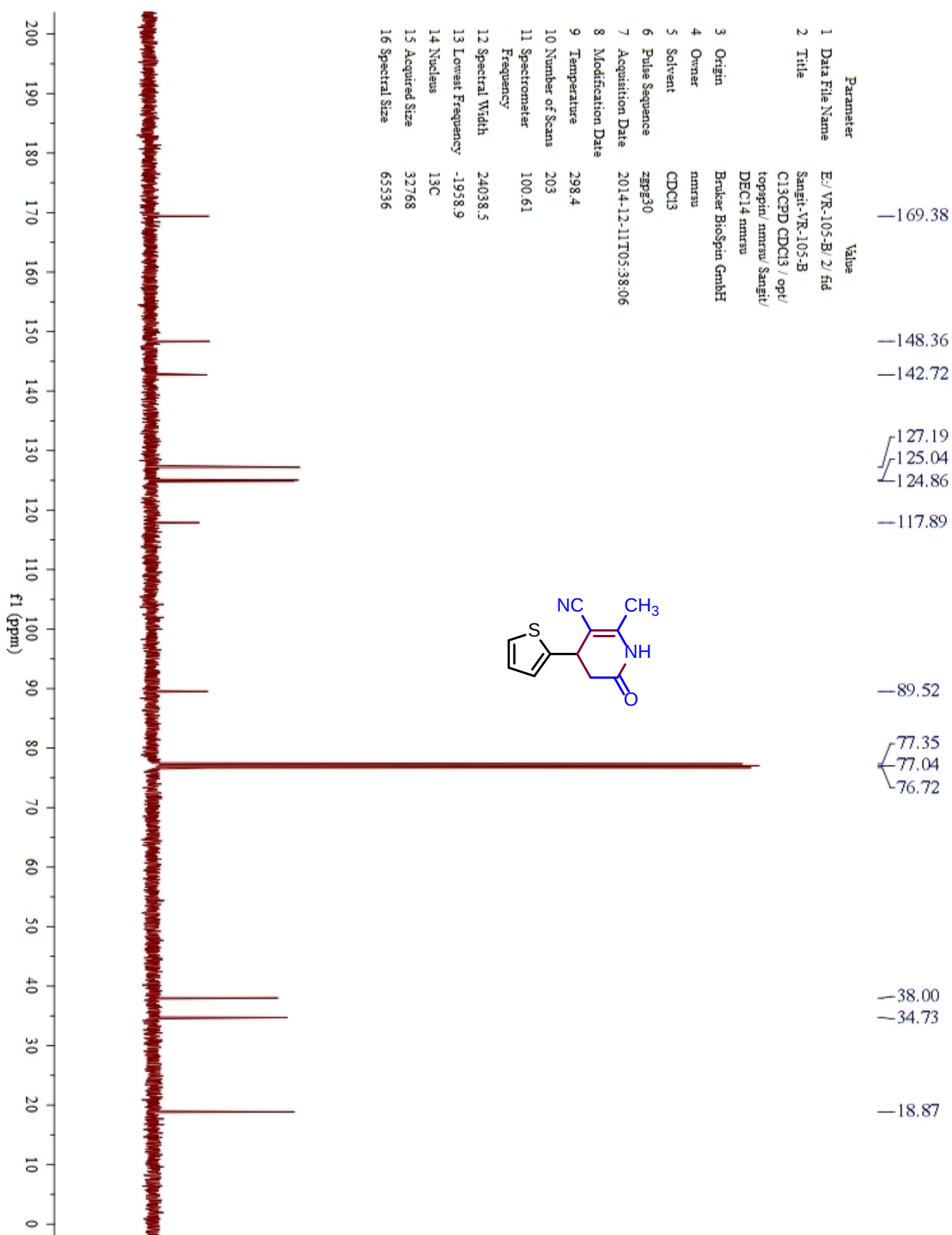


Figure S85 Mass spectra of **28**

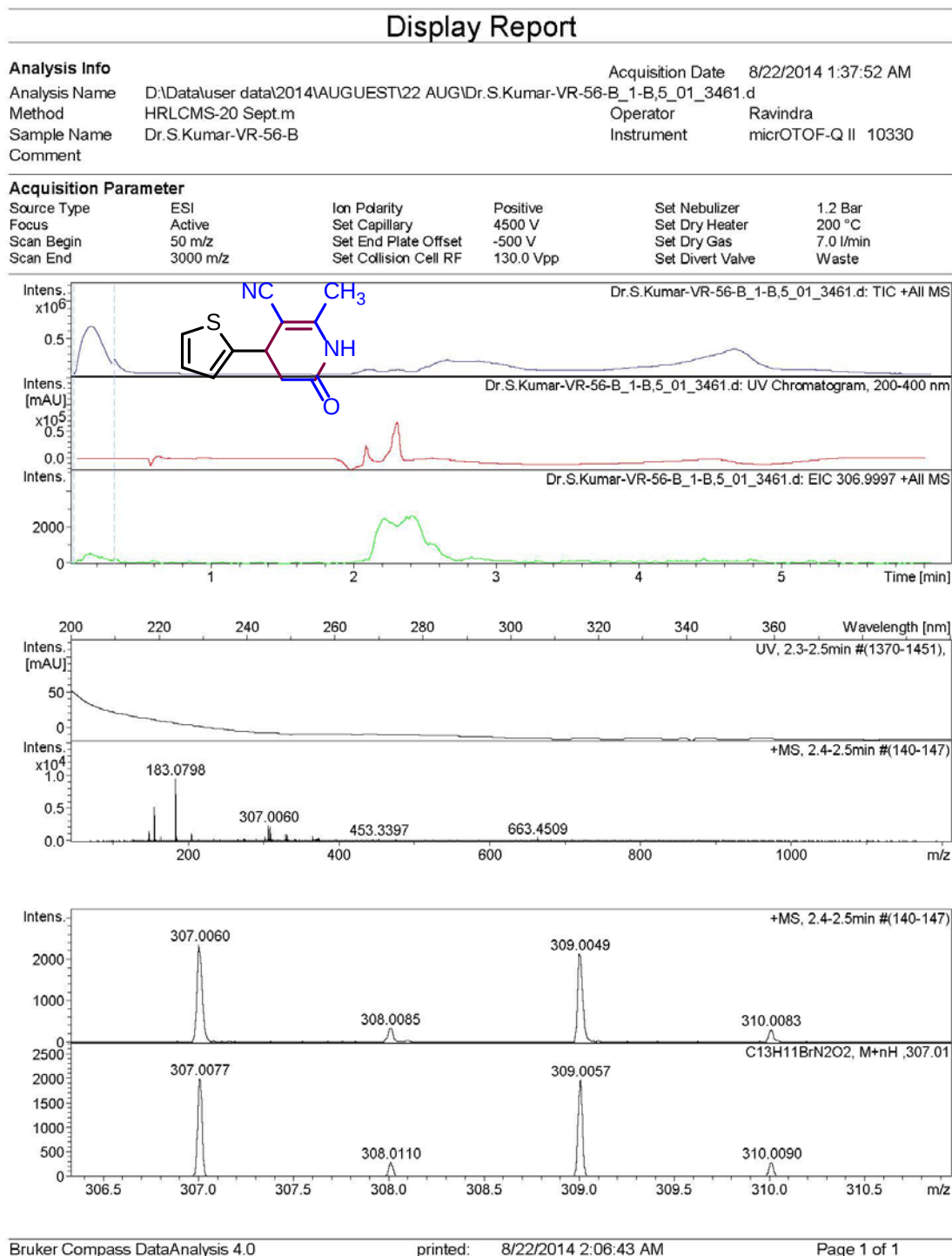


Figure S86 ^1H NMR spectra of **29**

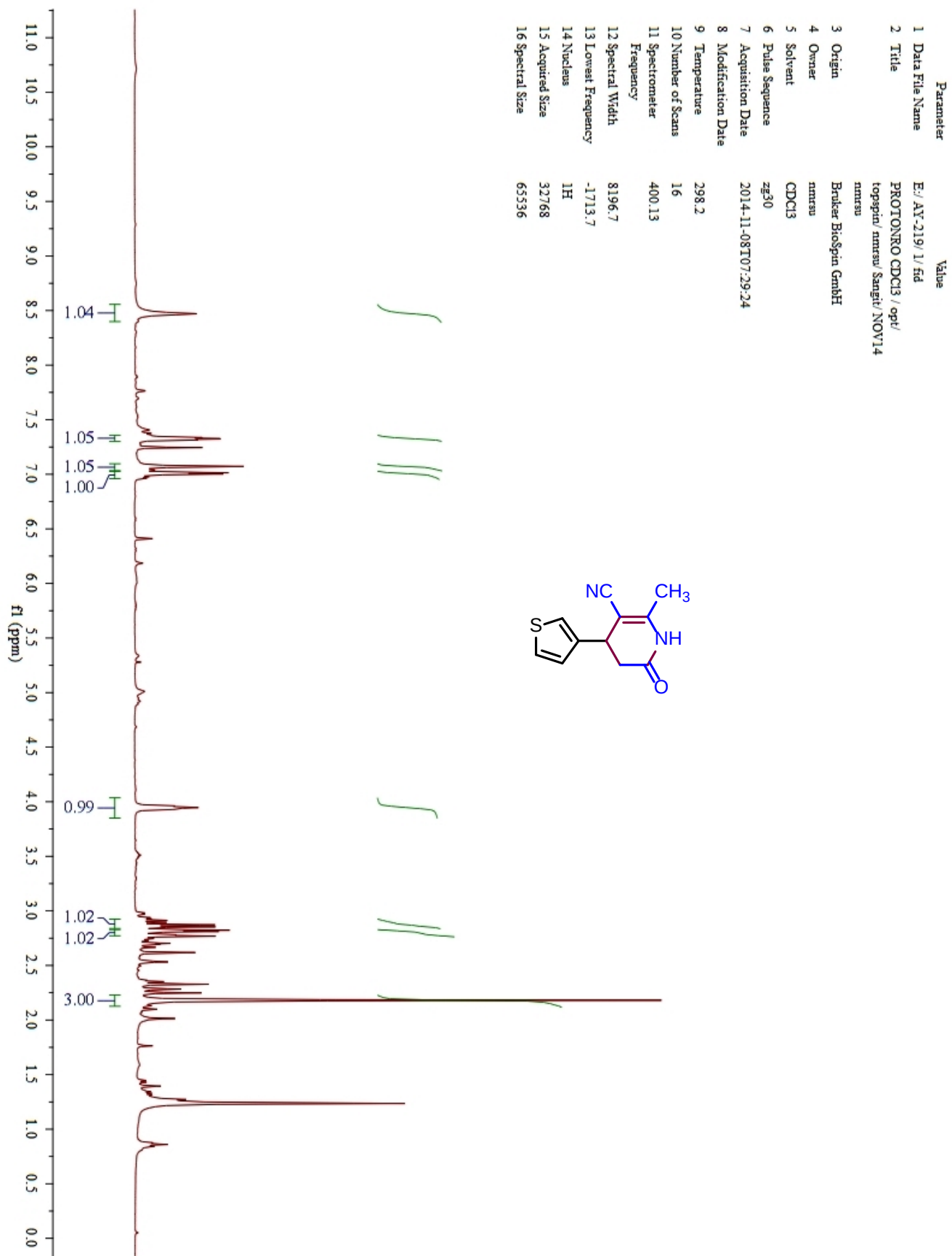


Figure S87 ^{13}C NMR spectra of **29**

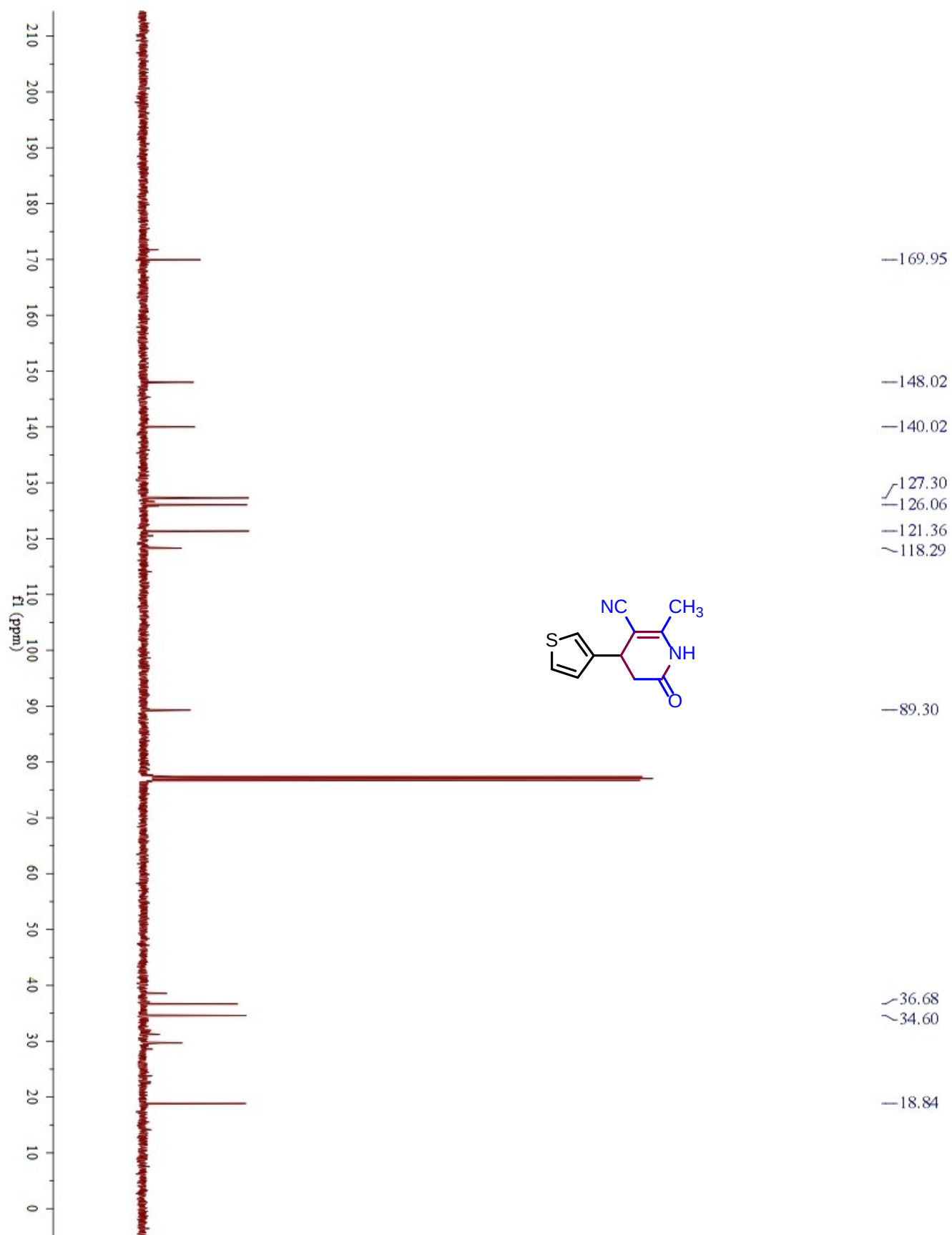


Figure S88 Mass spectra of **29**

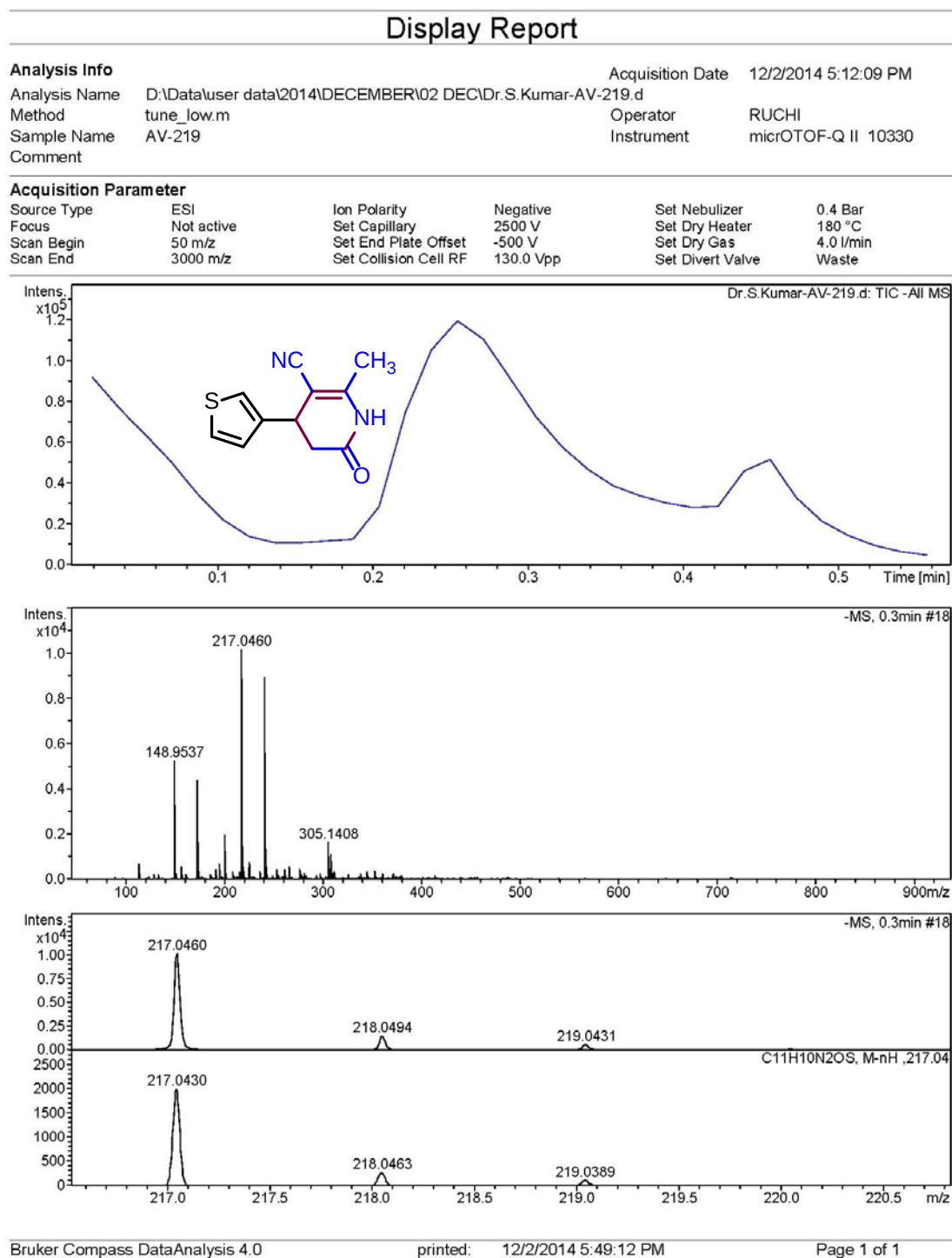


Figure S89 ¹H NMR spectra of **30**

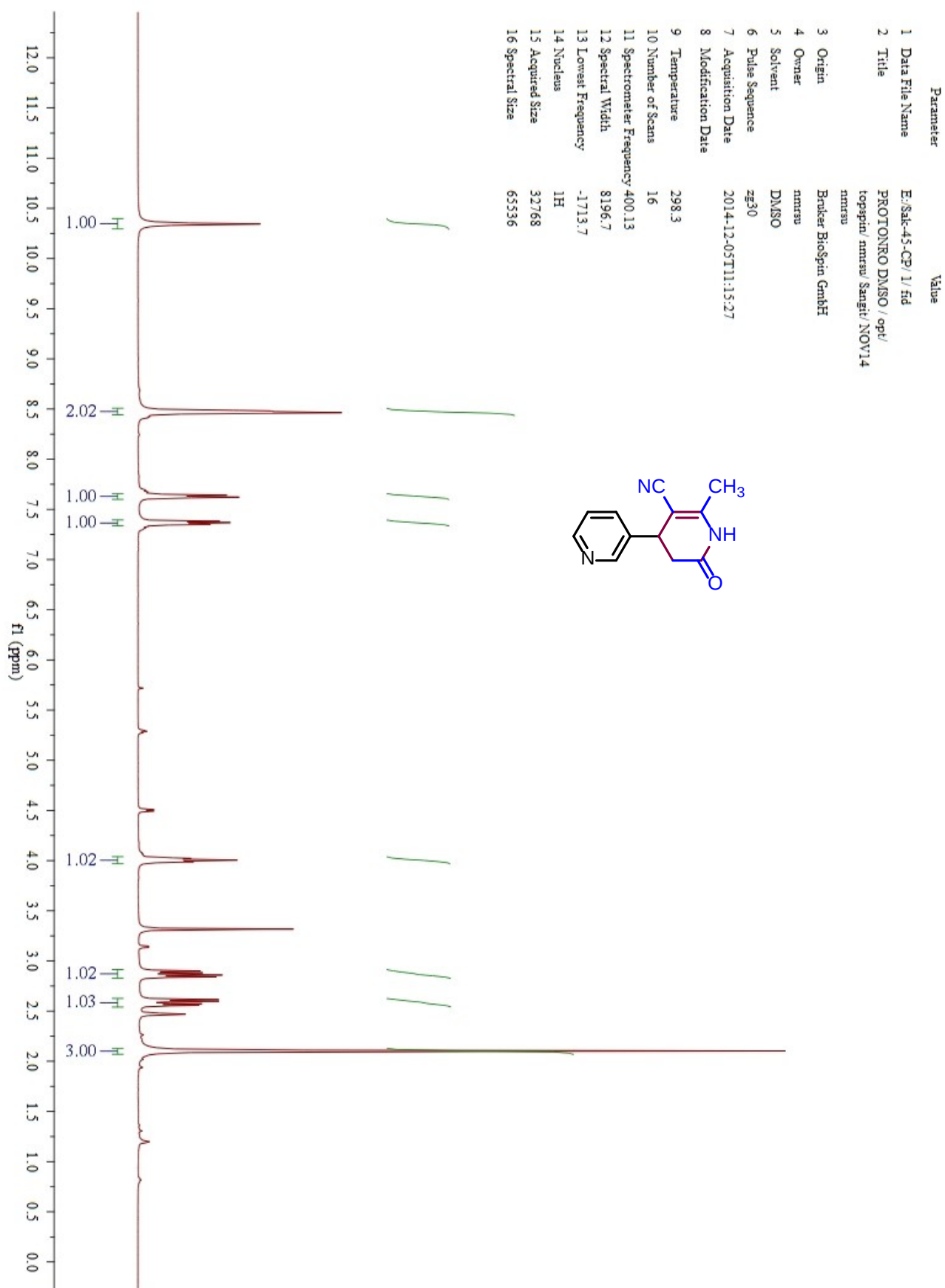


Figure S90 ^{13}C NMR spectra of **30**

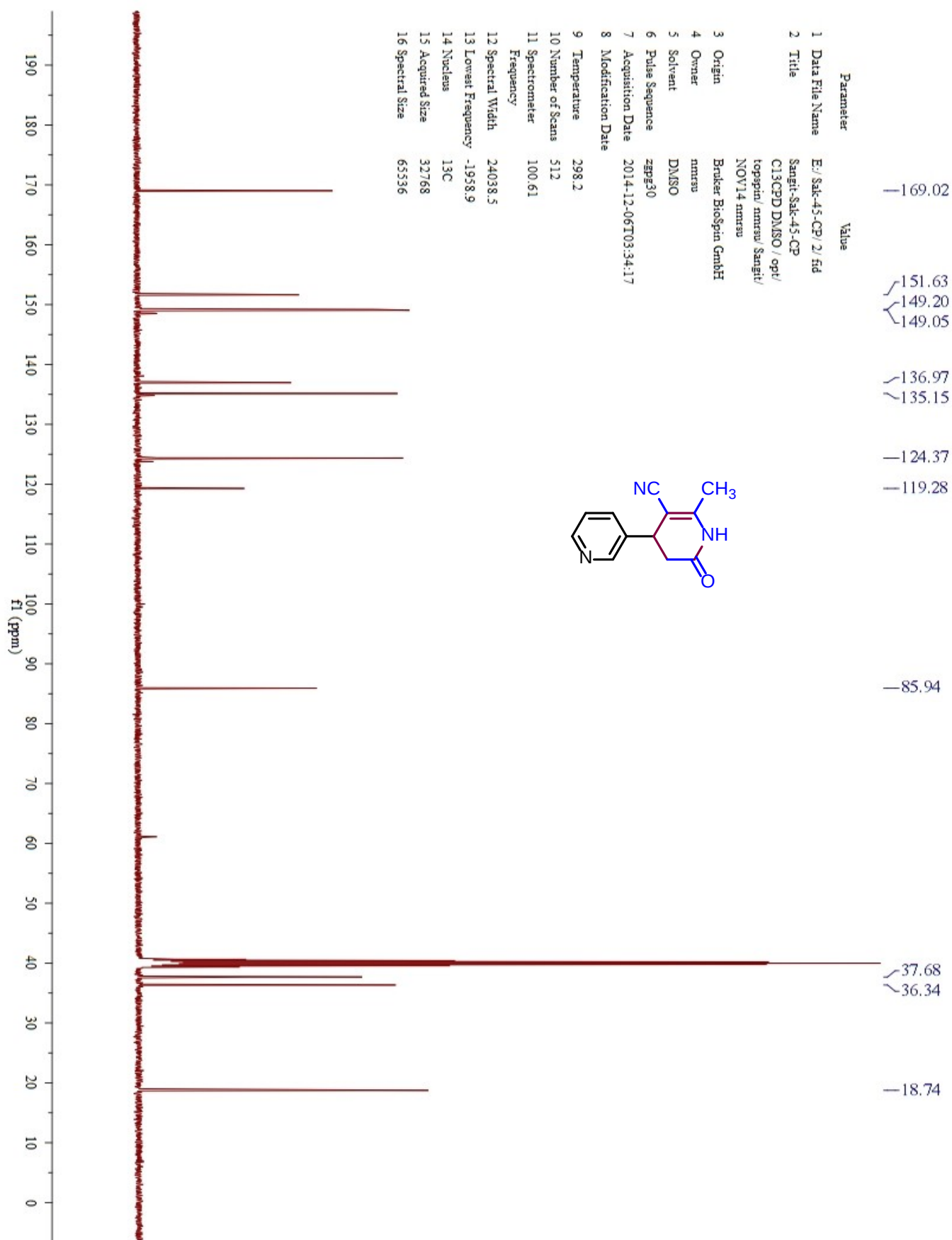


Figure S91 Mass spectra of 30

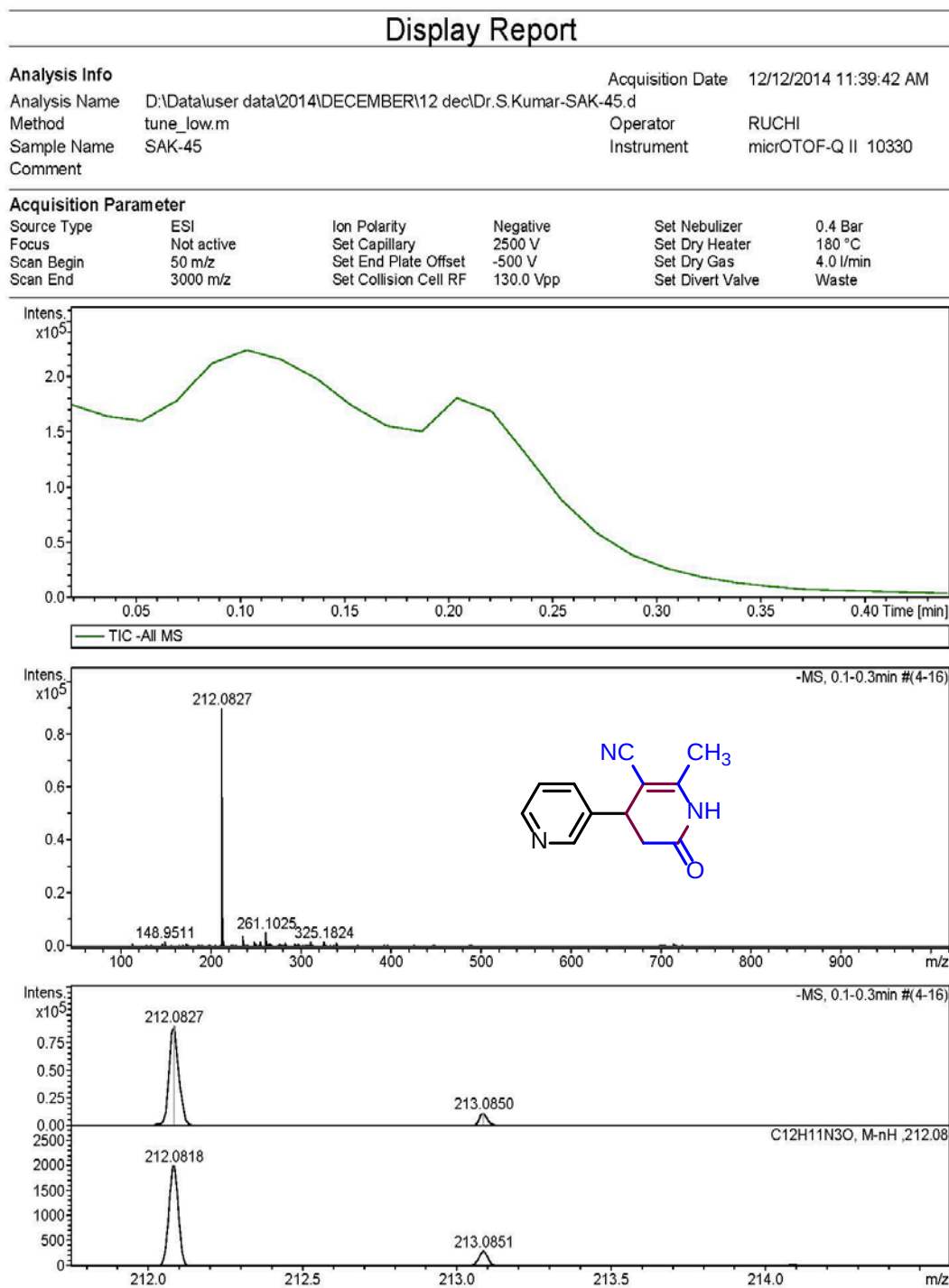


Figure S92 ^1H NMR spectra of **31**

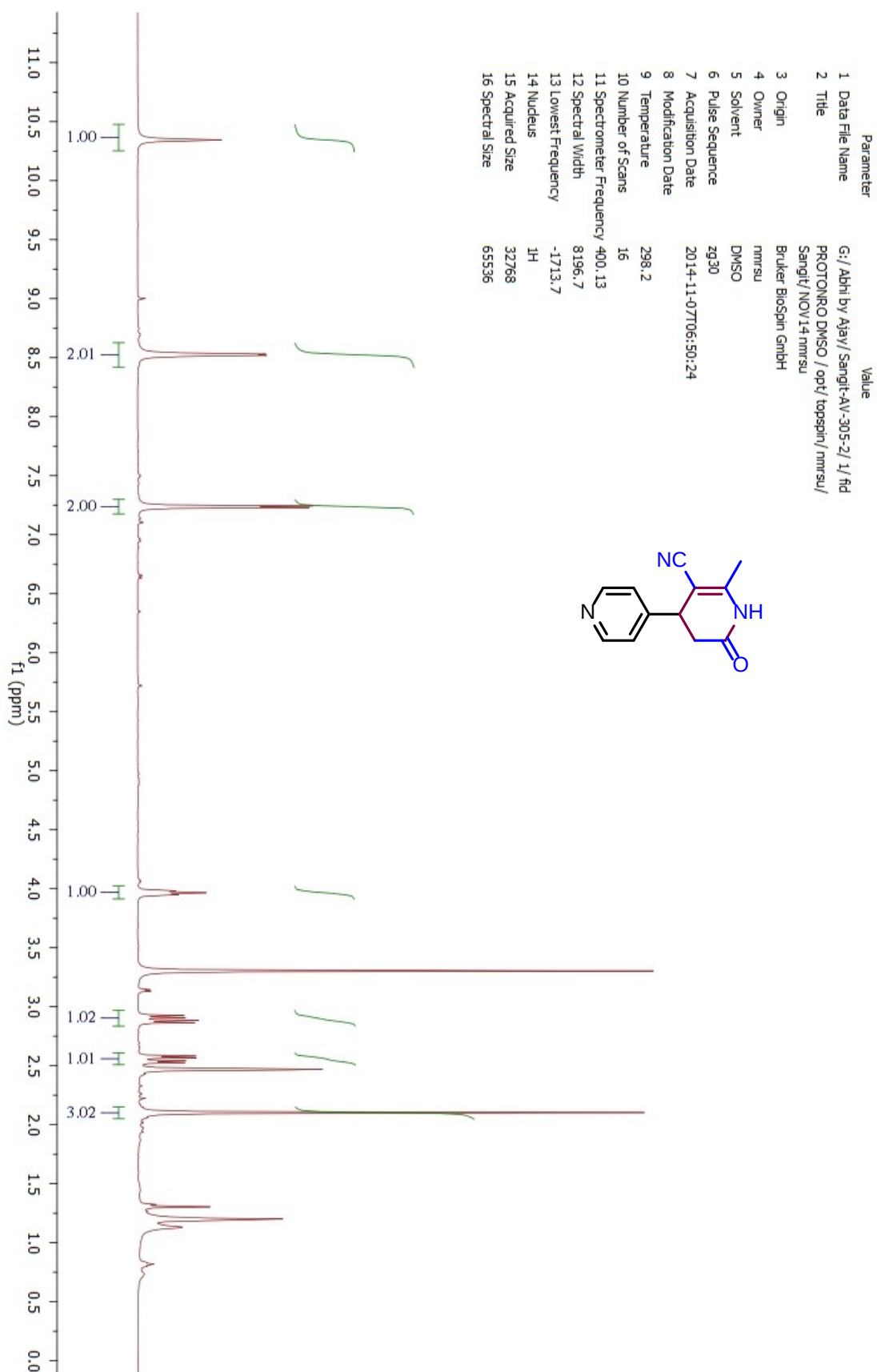


Figure S93 ^{13}C NMR spectra of **31**

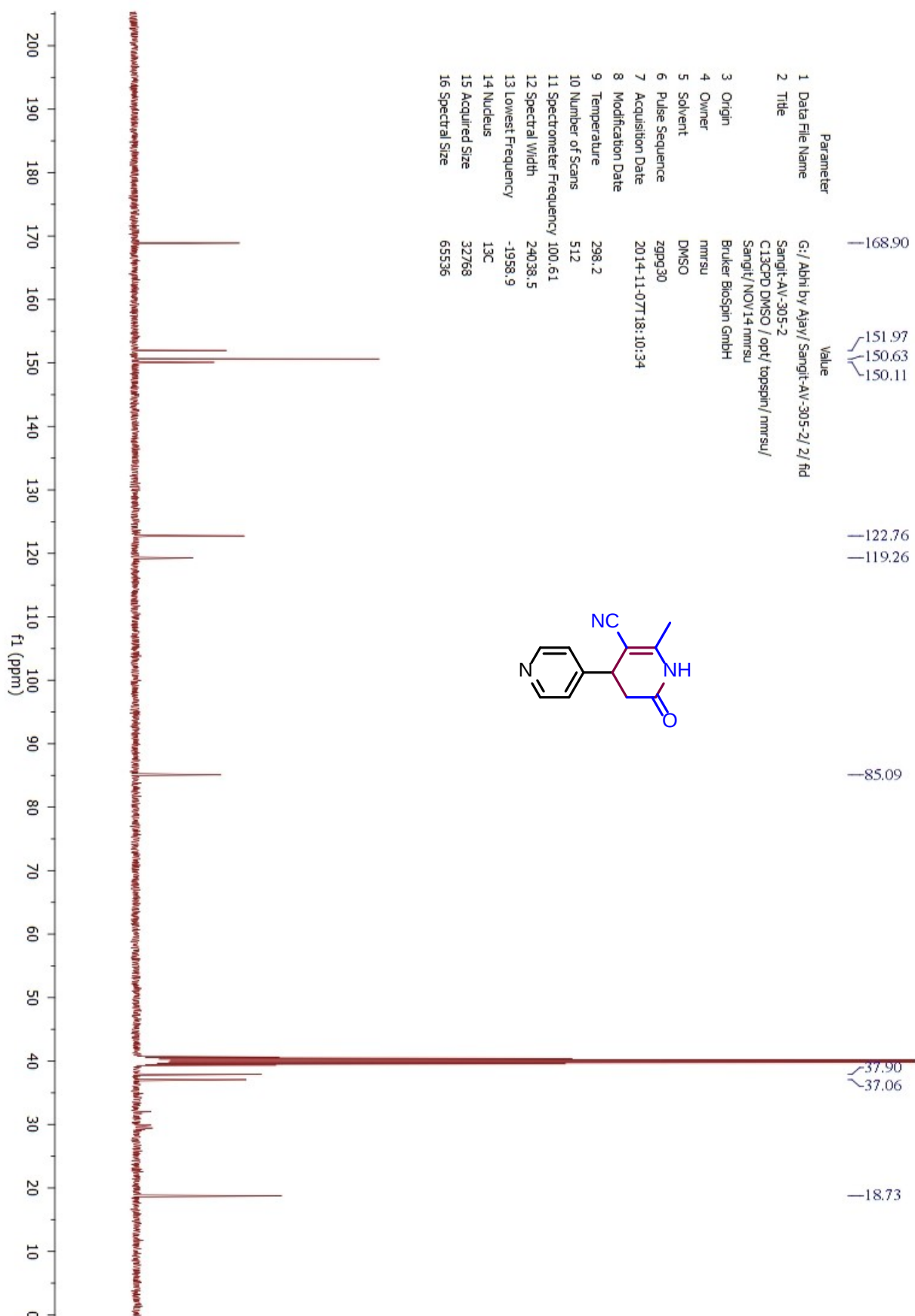


Figure S94 HRMS spectra of **31**

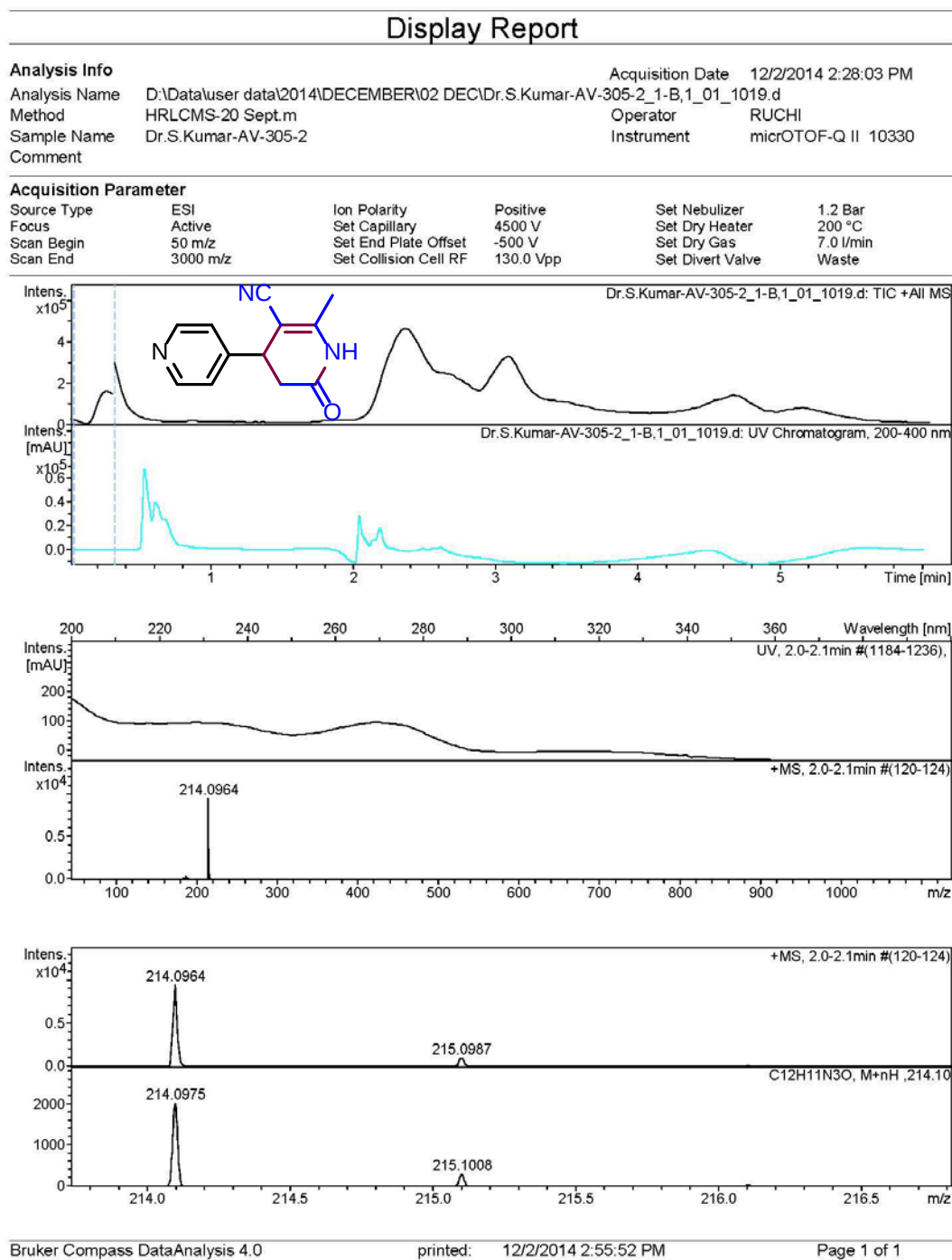


Figure S95 ¹H NMR spectra of **32**

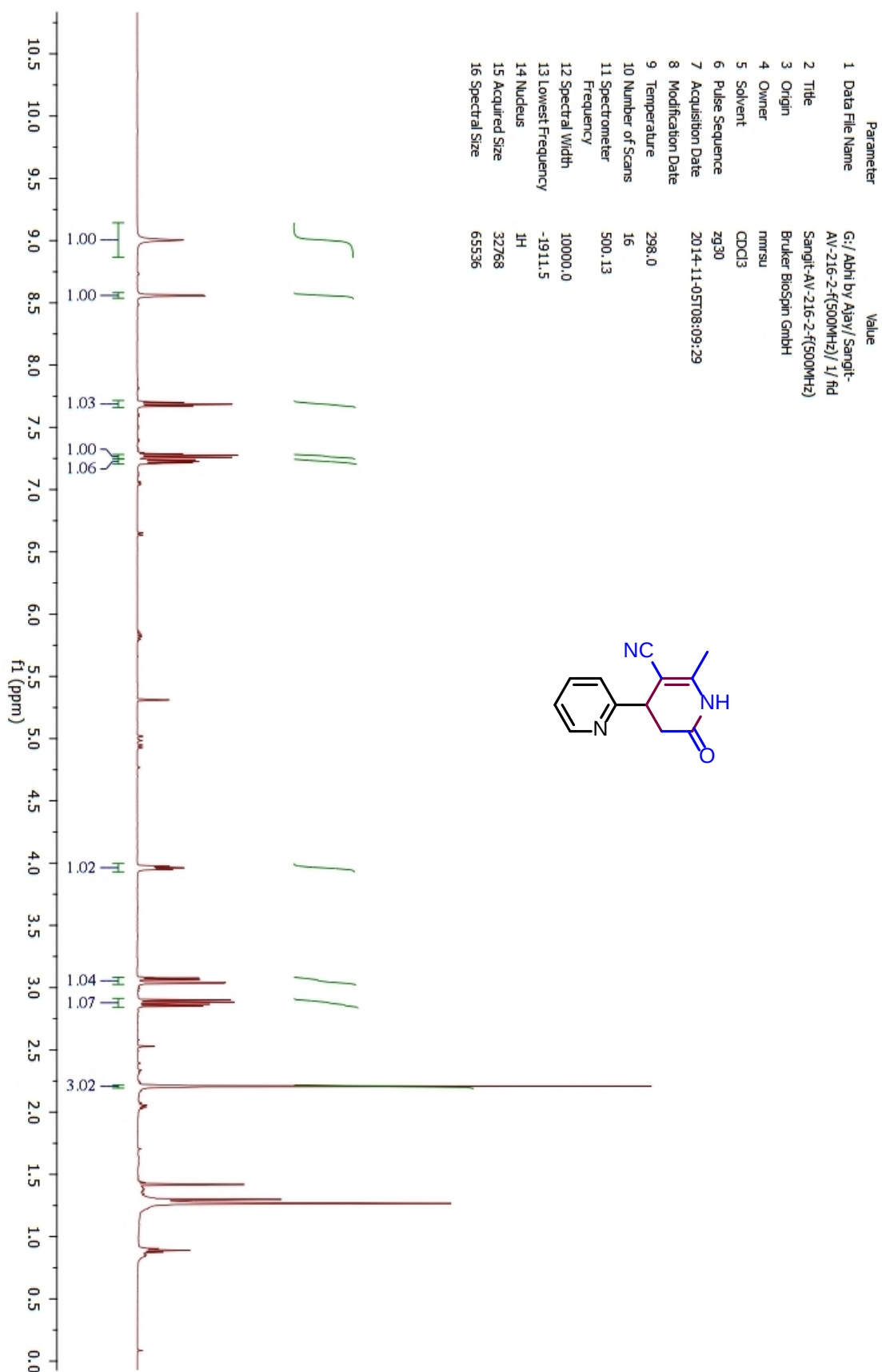


Figure S96 ^{13}C NMR spectra of **32**

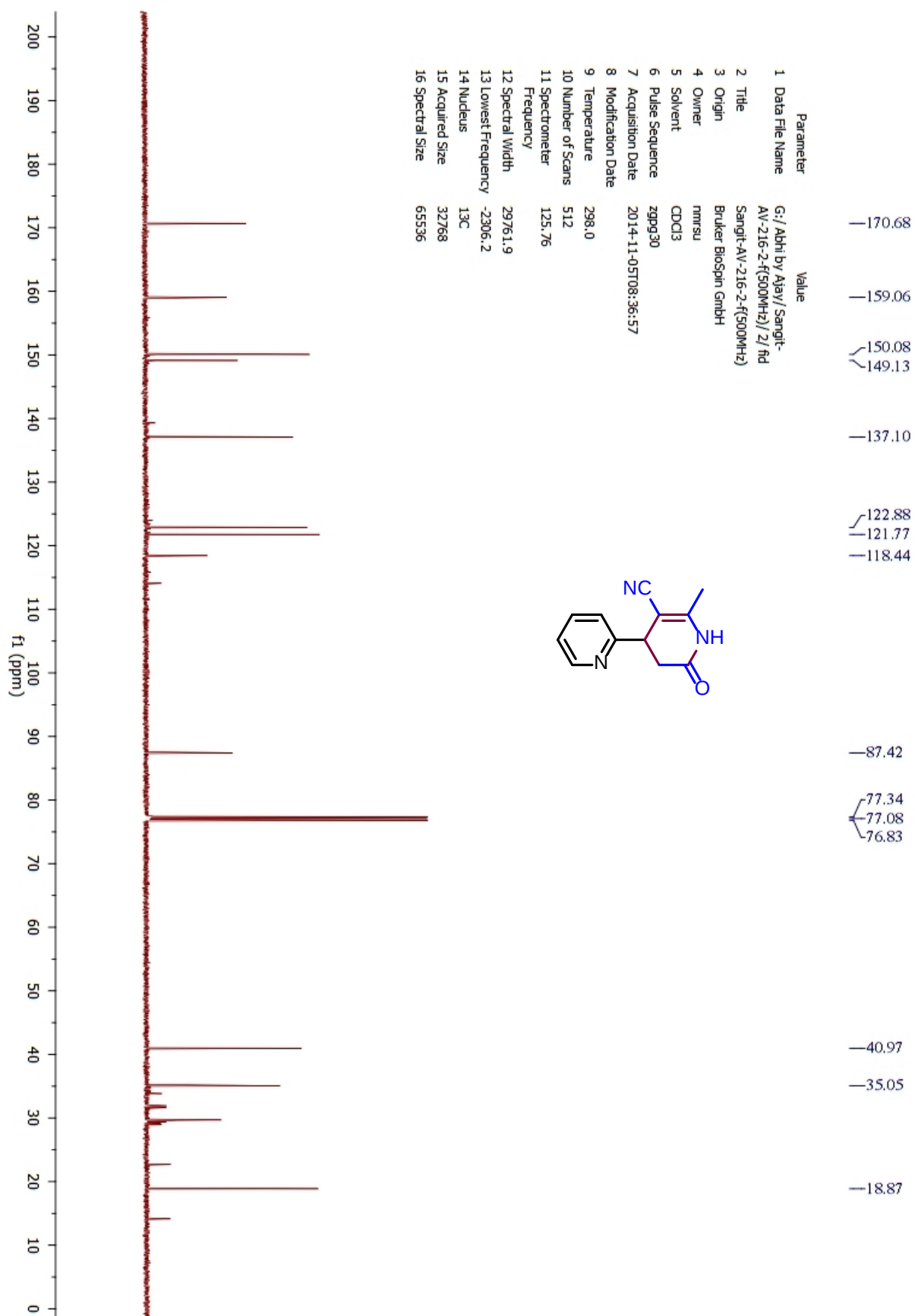


Figure S97 HRMS spectra of **32**

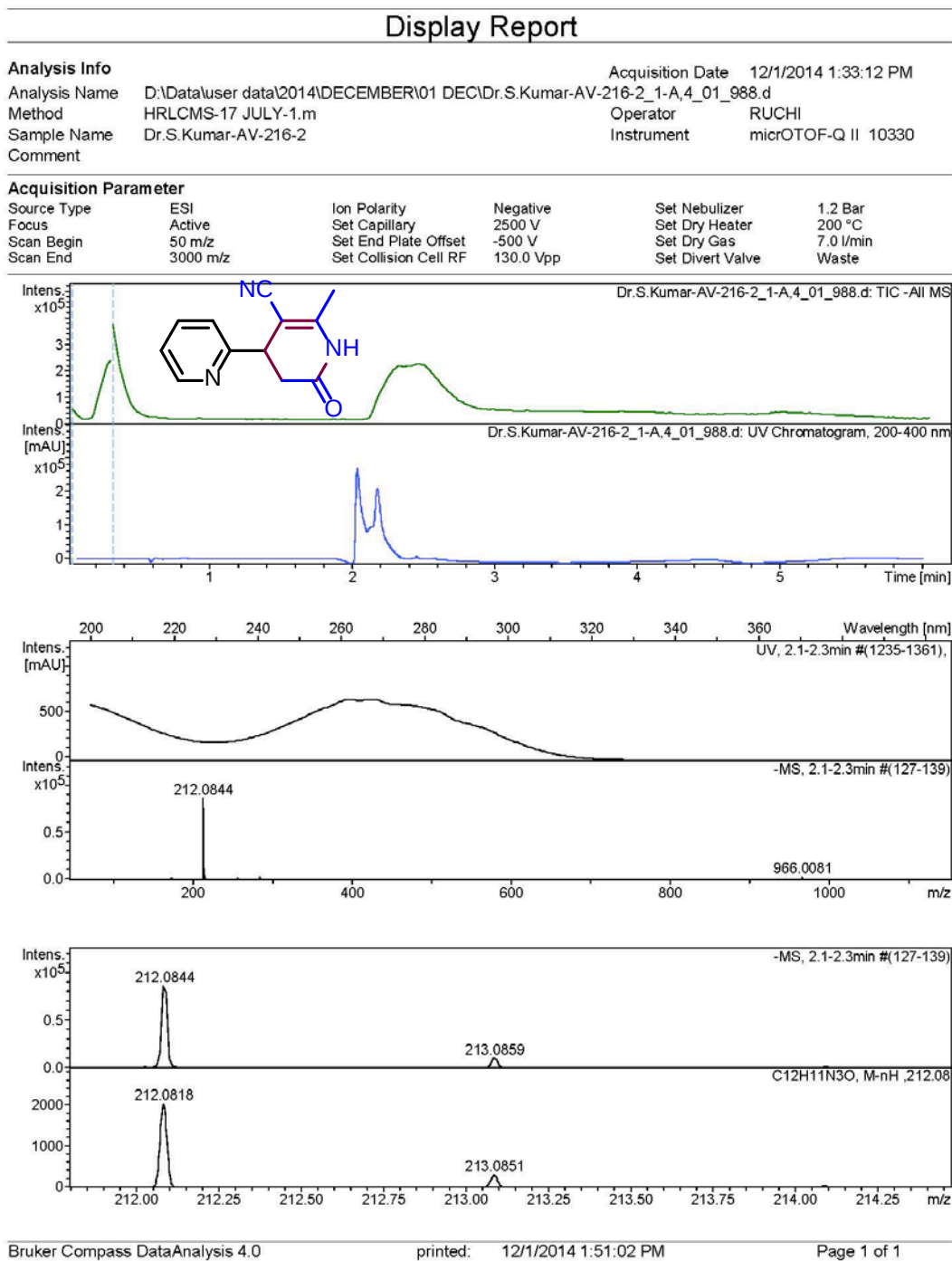


Figure S98 ¹H NMR spectra of 33

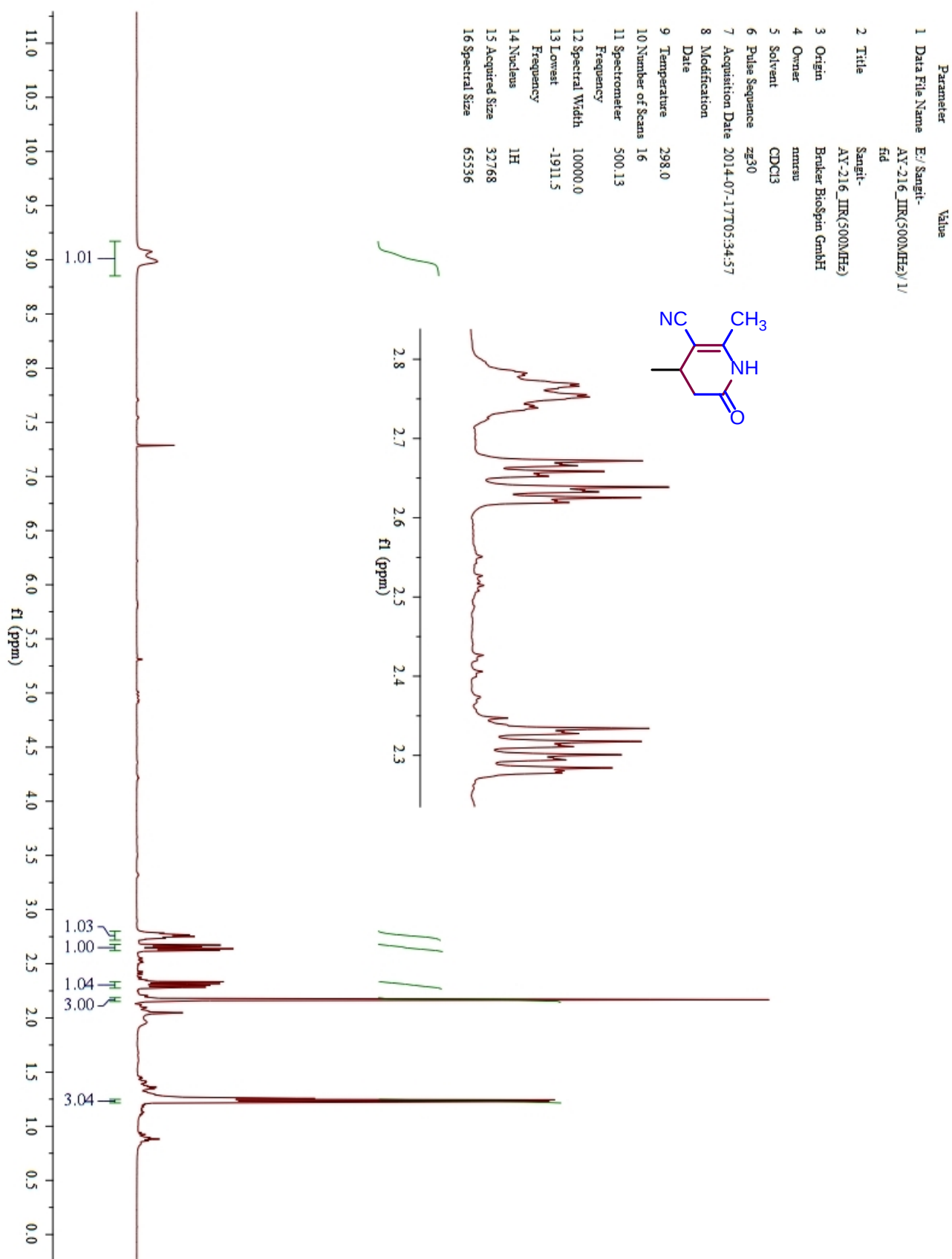


Figure S99 ¹³C NMR spectra of **33**

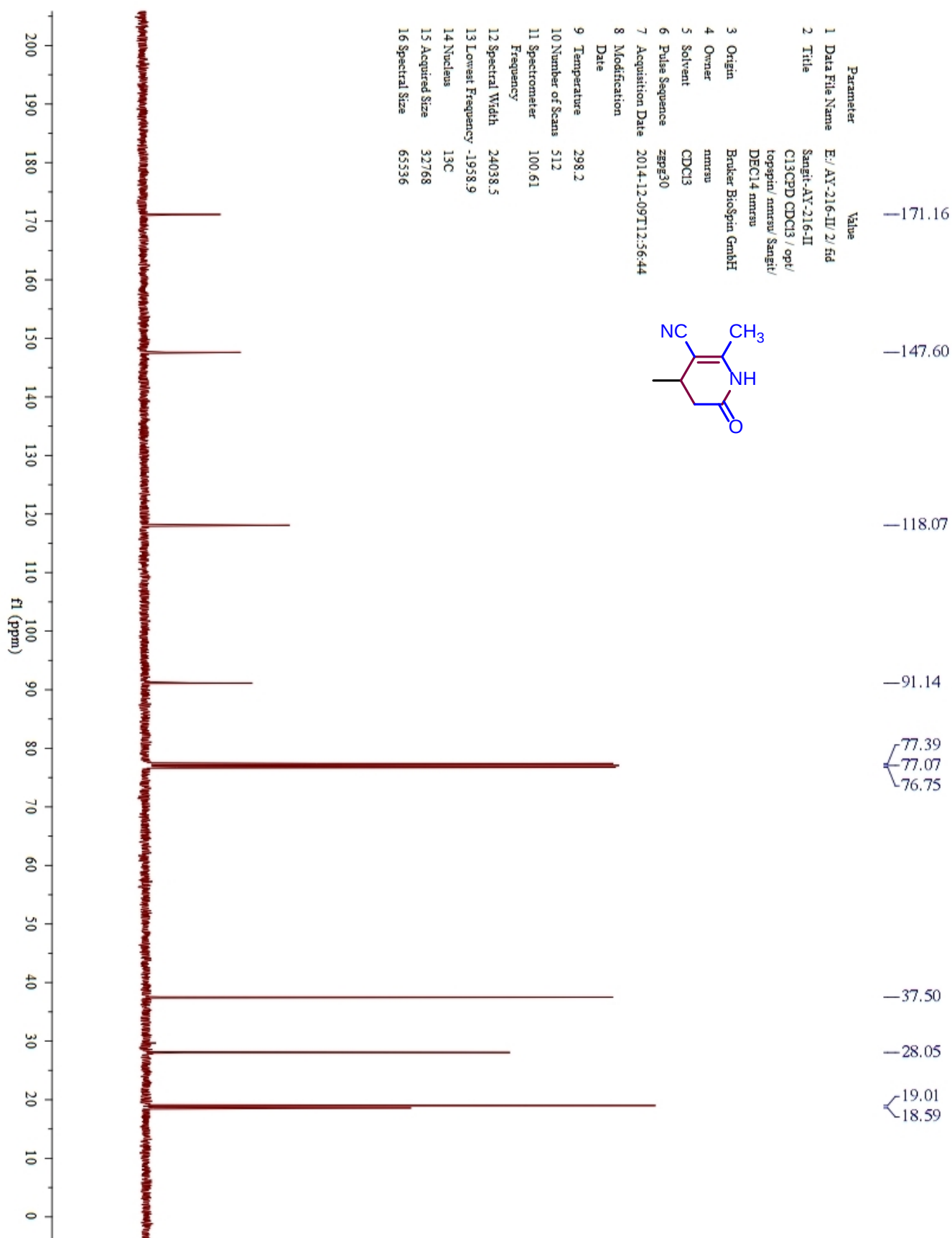
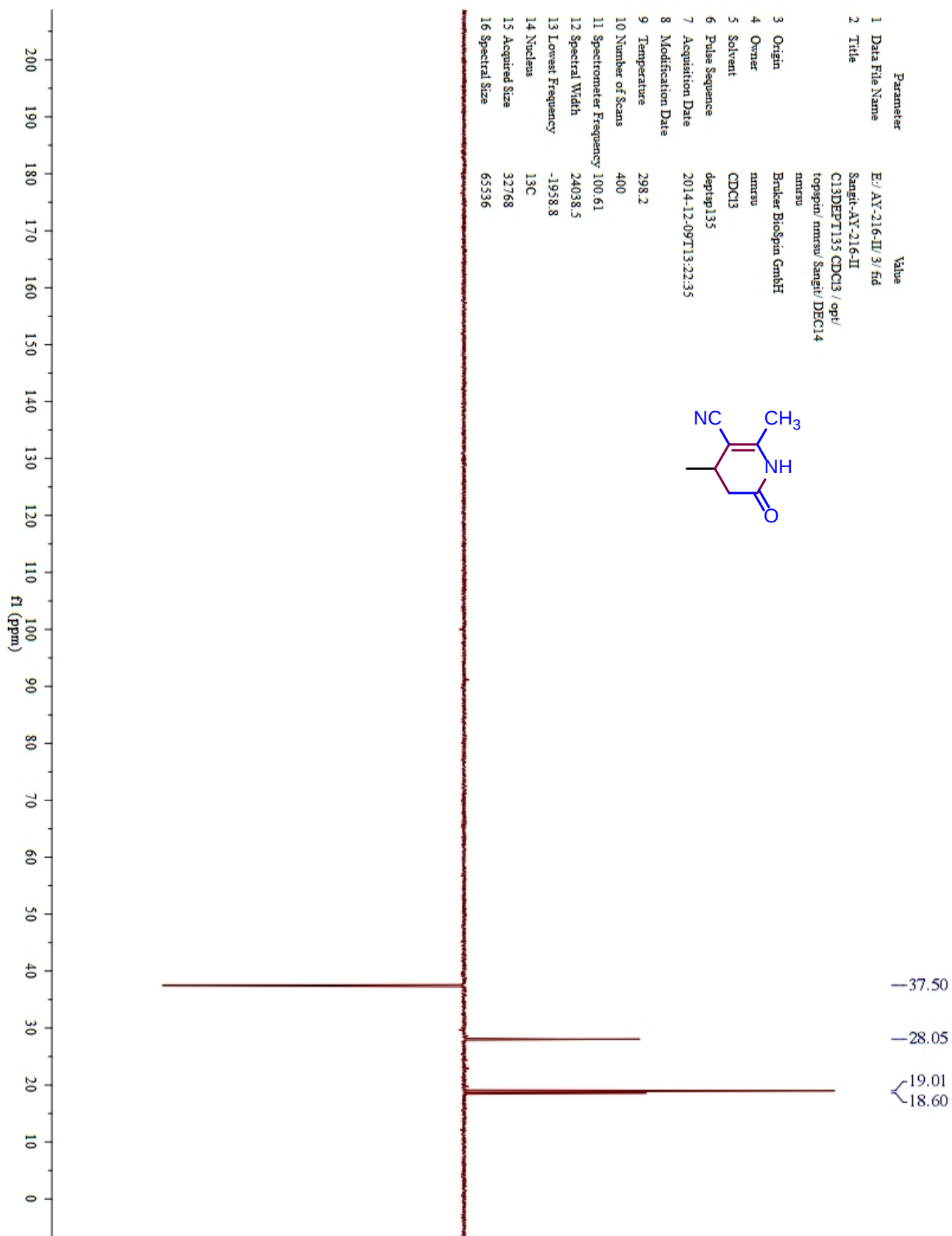


Figure S100 135-DEPT NMR spectra of **33**



S102

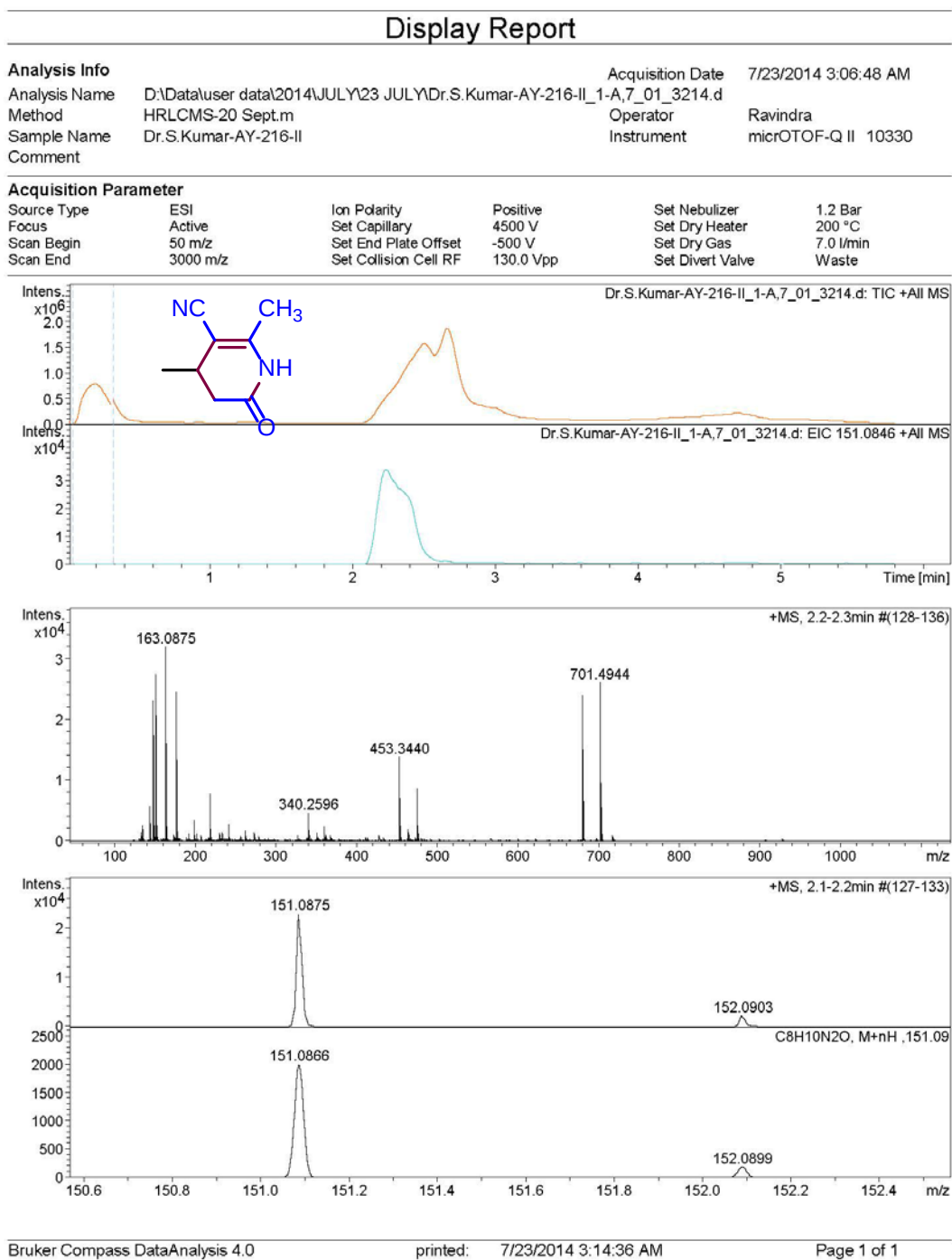


Figure S102 ^1H NMR spectra of **34**

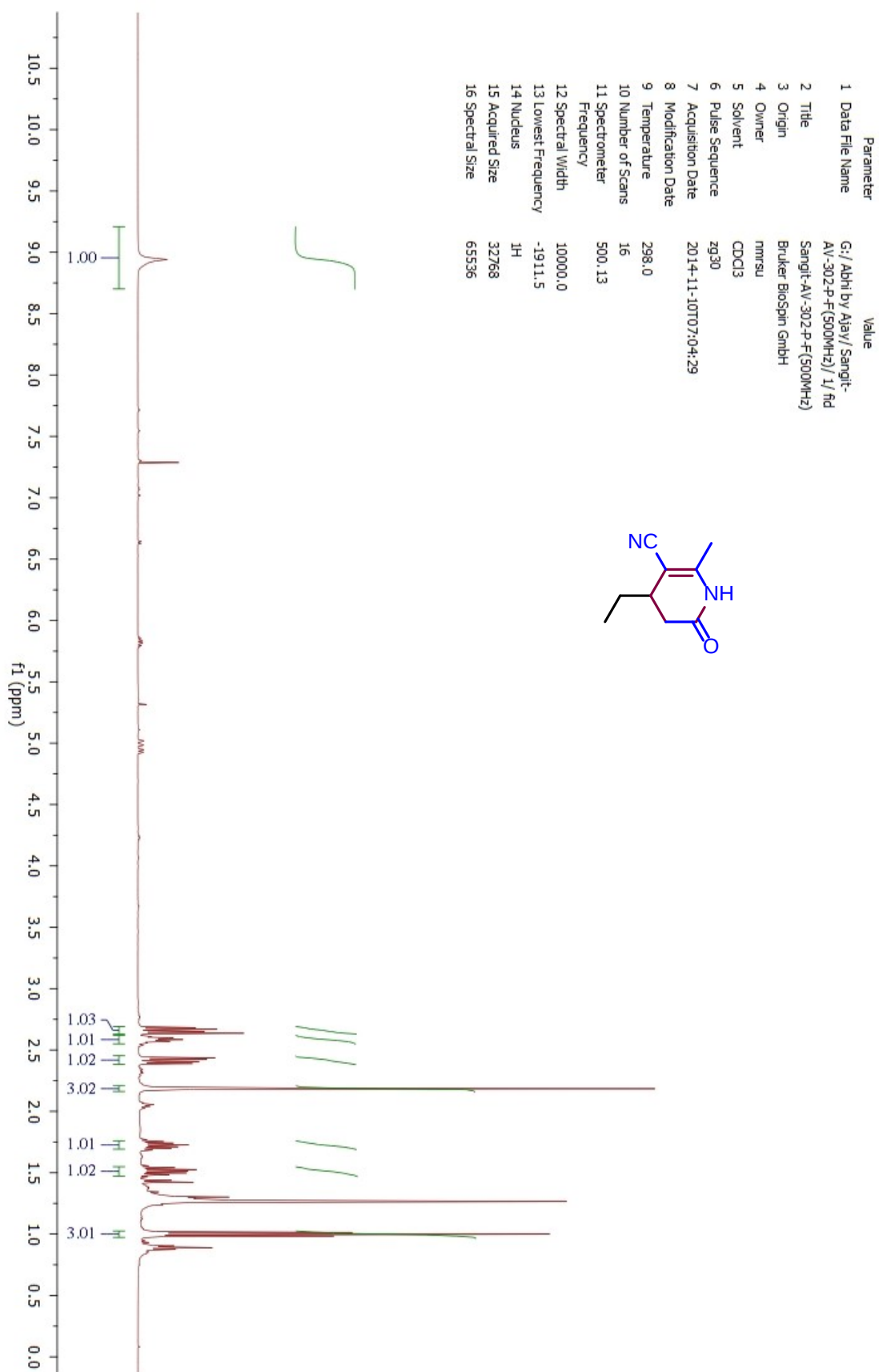


Figure S103 ^{13}C NMR spectra of **34**

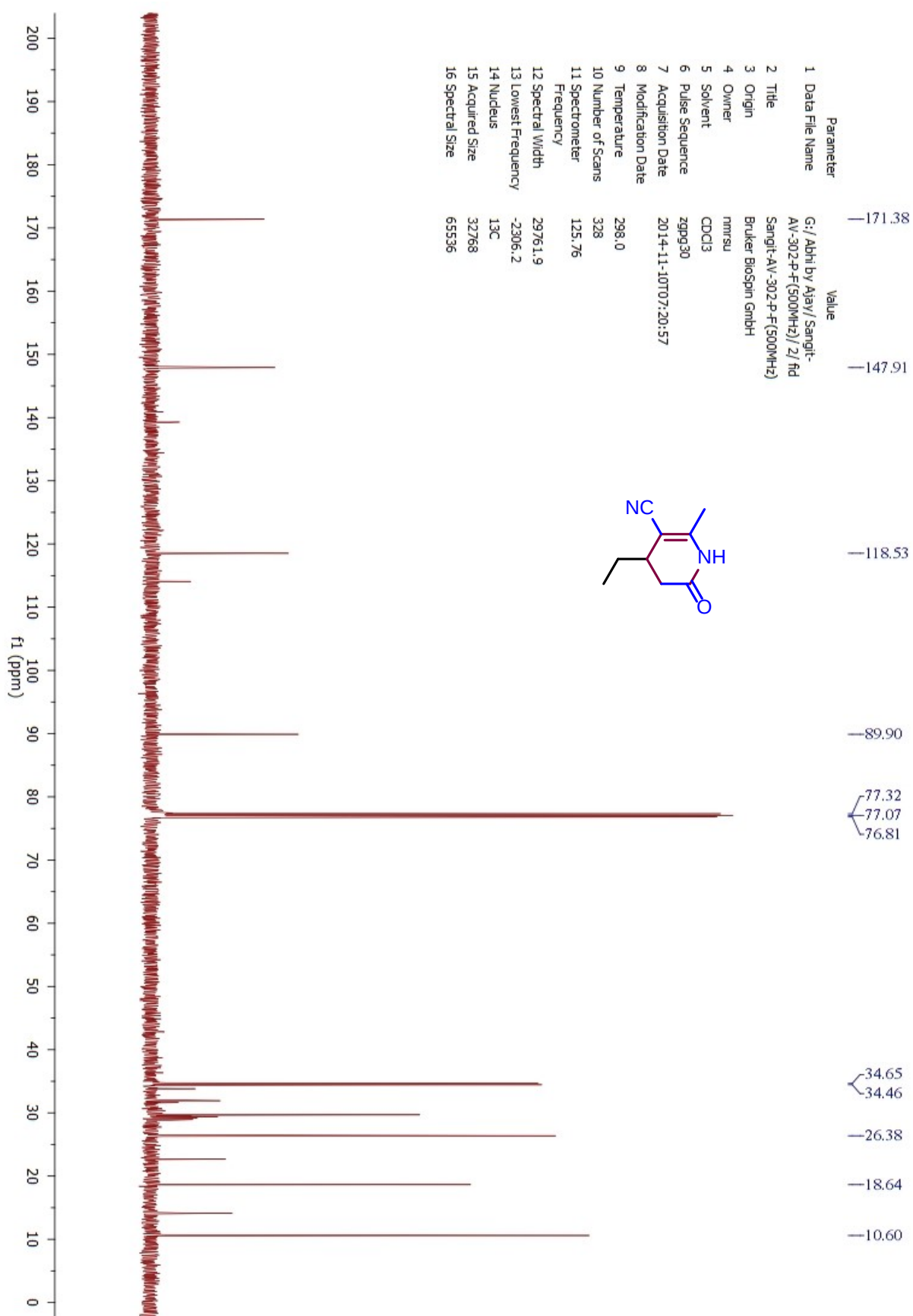


Figure S104 HRMS spectra of **34**

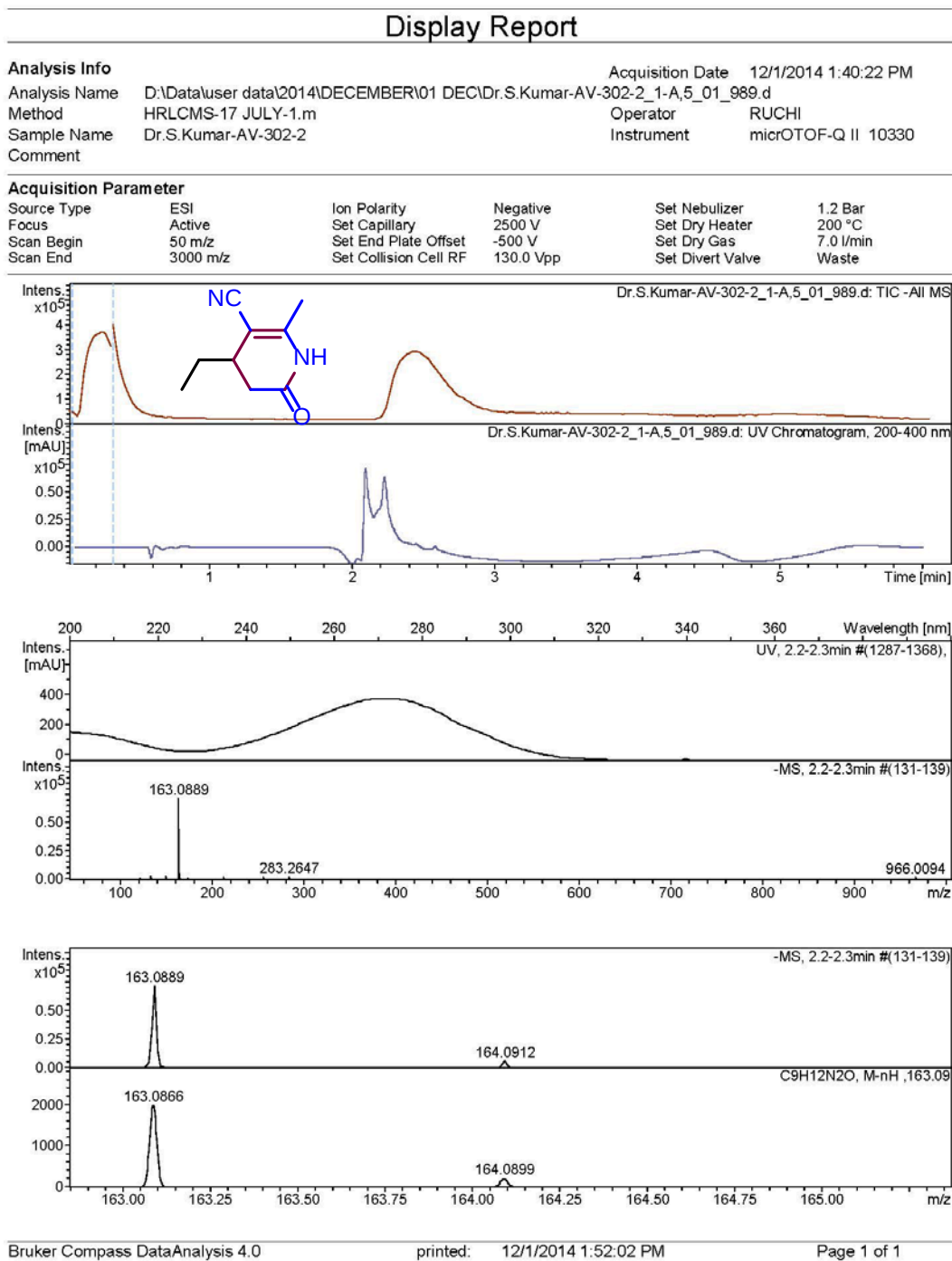


Figure S105 ¹H NMR spectra of 35

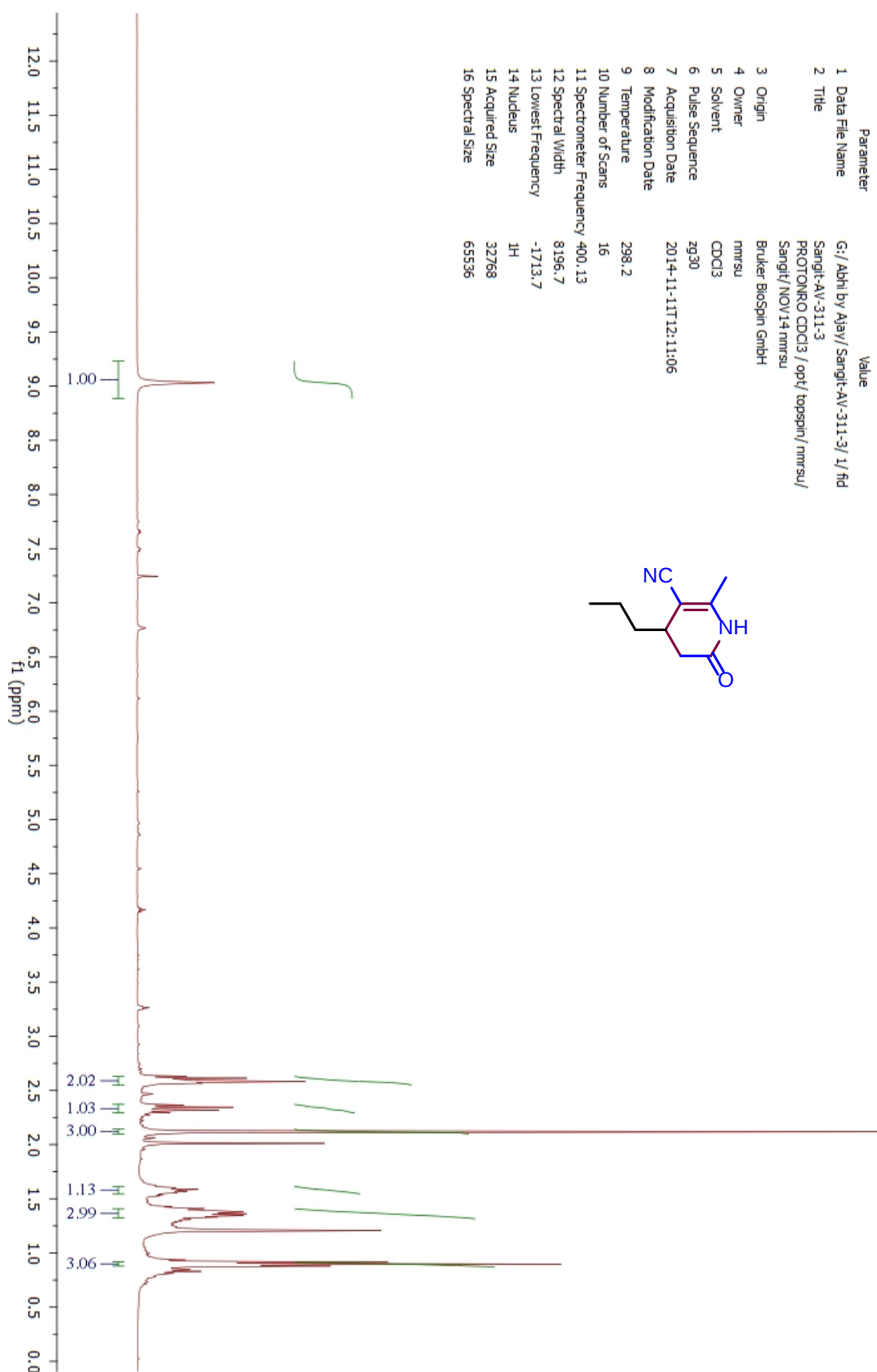
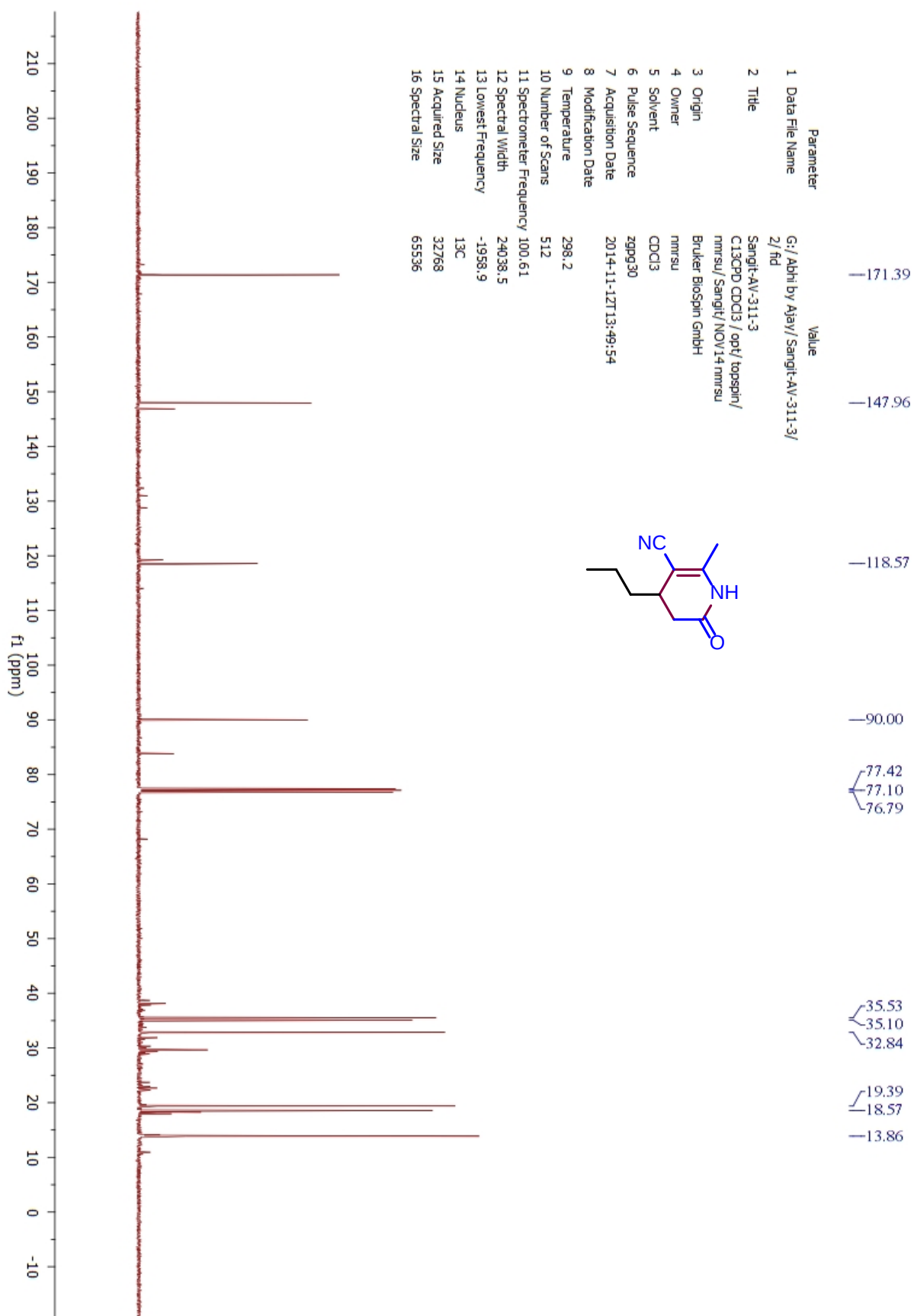


Figure S106 ^{13}C NMR spectra of **35**



S108

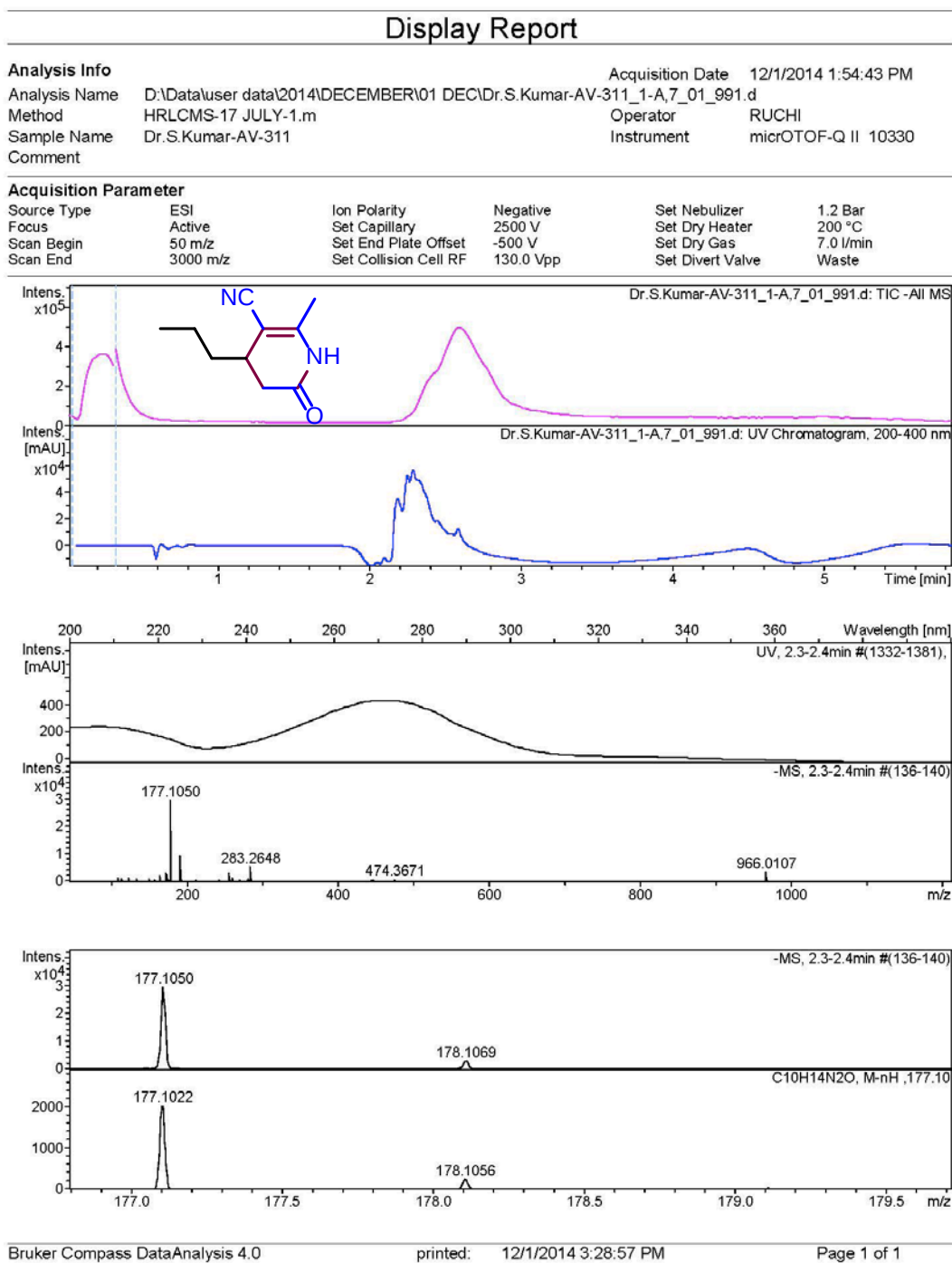


Figure S108 ¹H NMR spectra of **36**

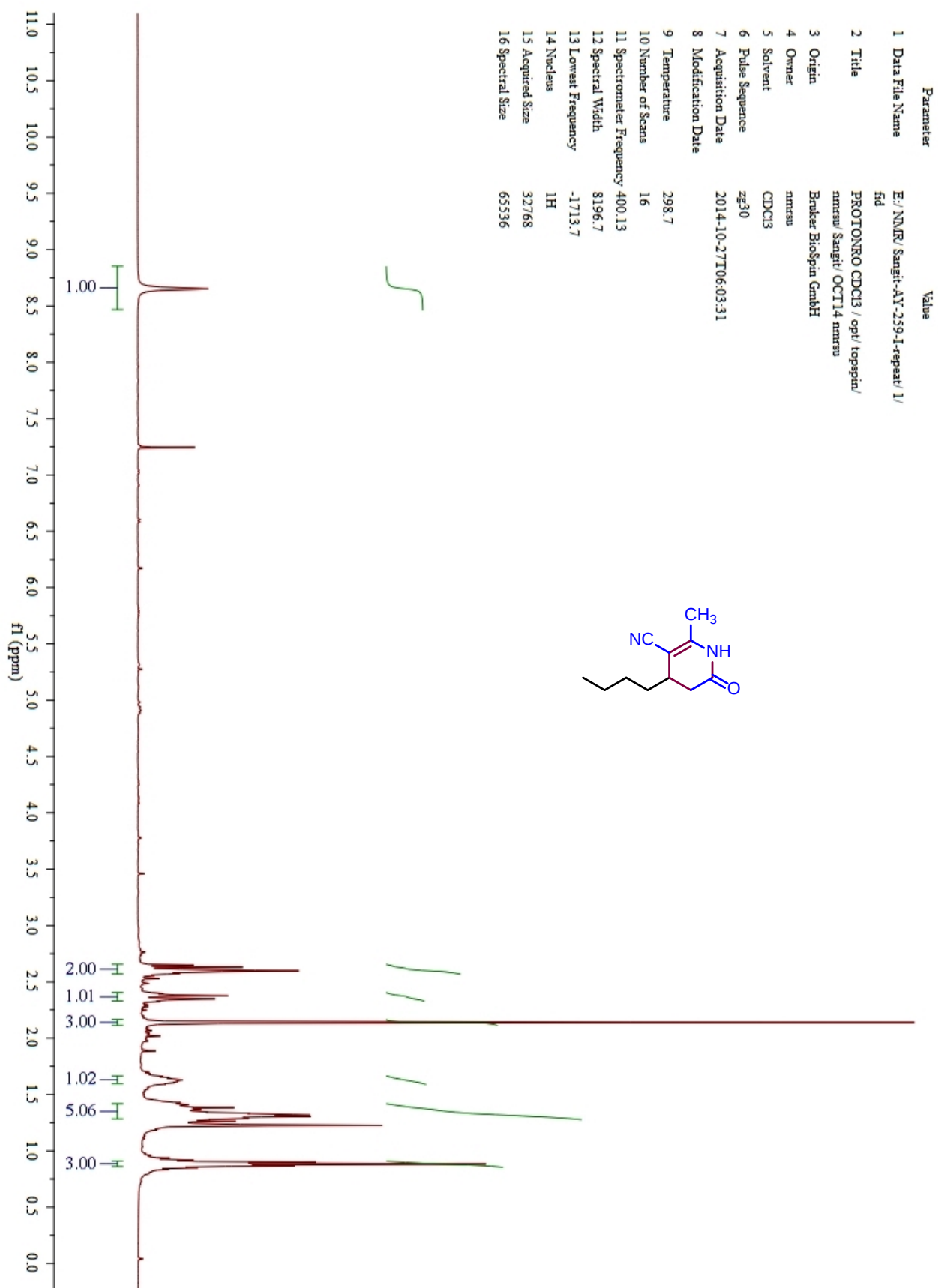


Figure S109 ^{13}C NMR spectra of **36**

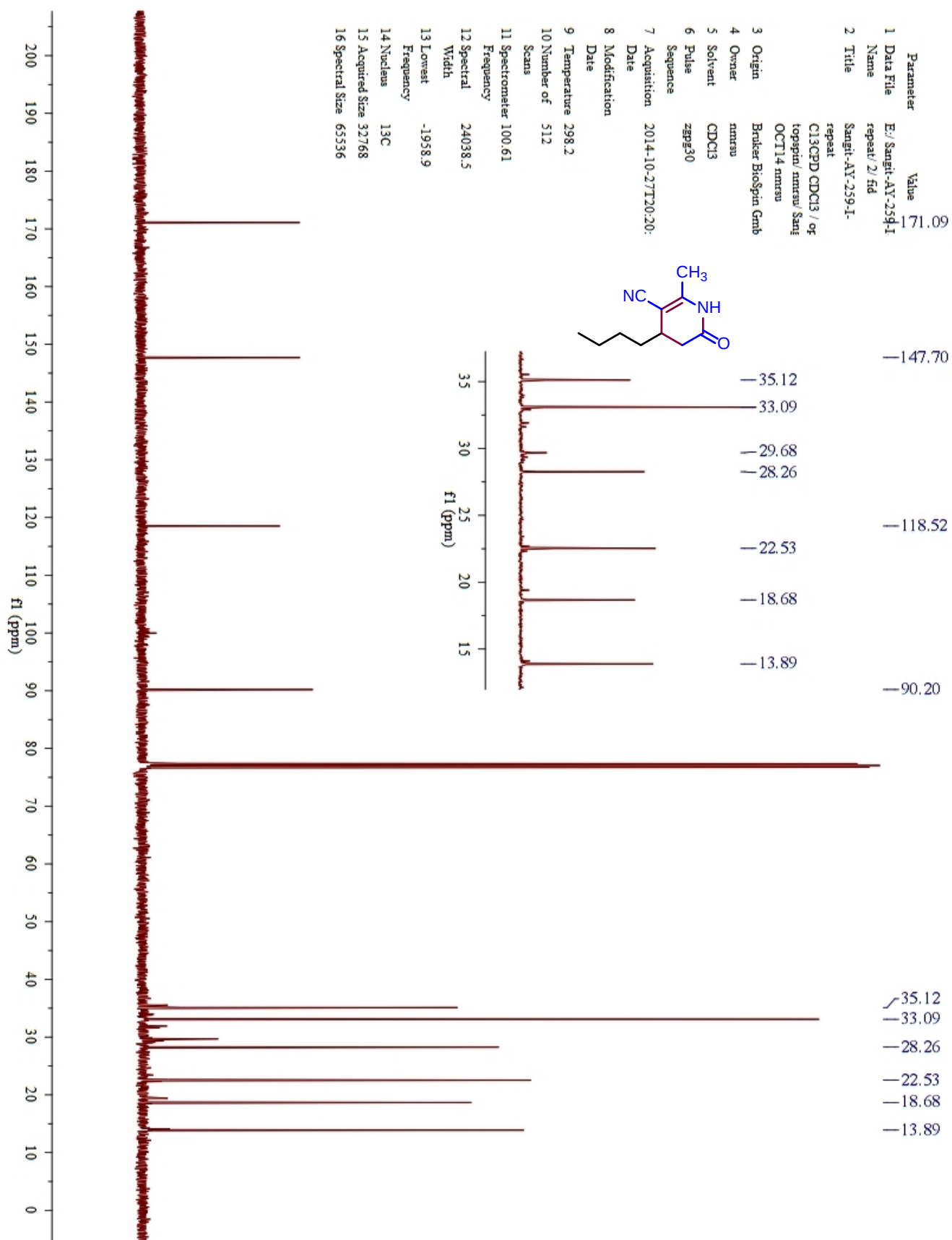


Figure S110 HRMS spectra of **36**

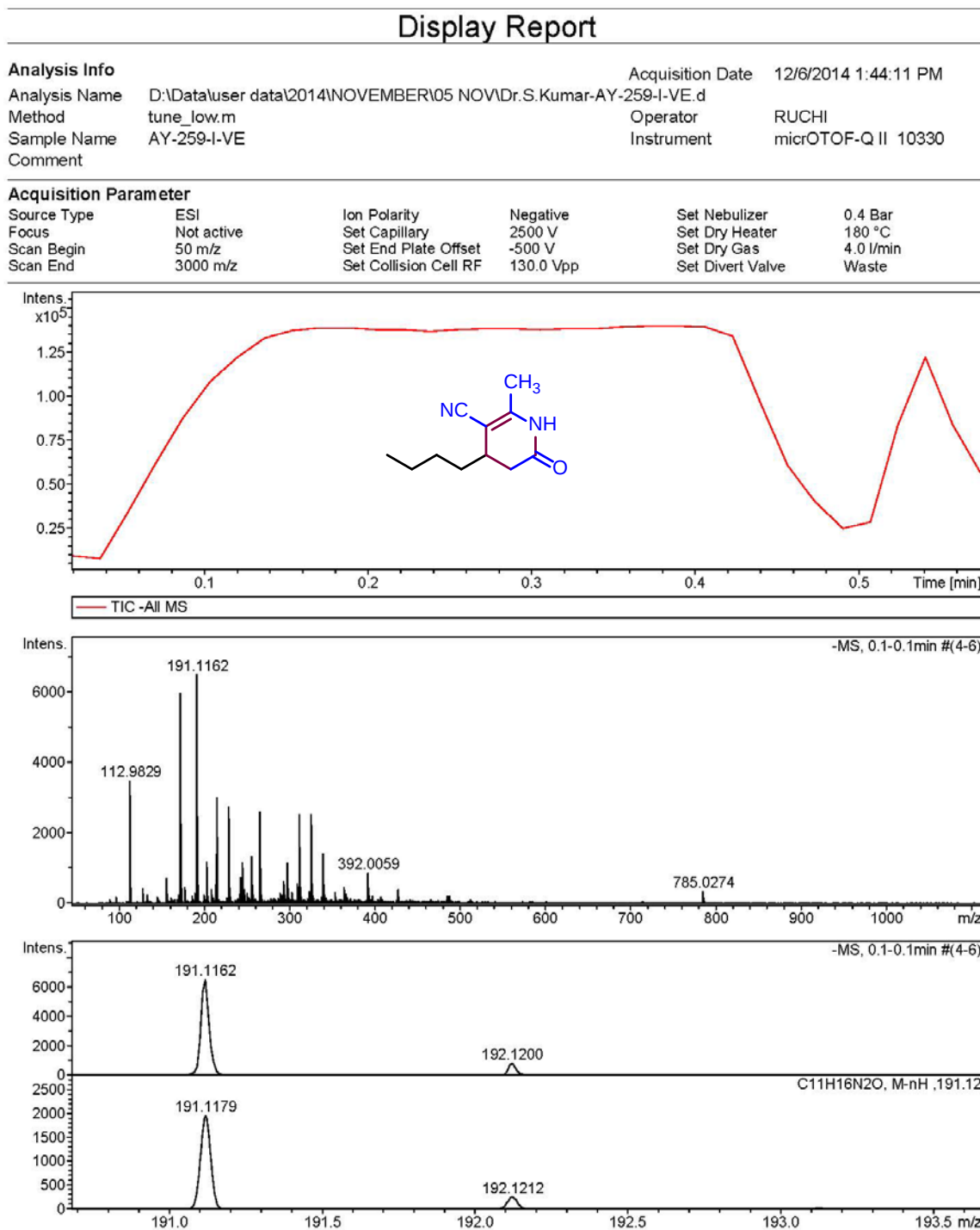


Figure S111 ¹H NMR spectra of **37**

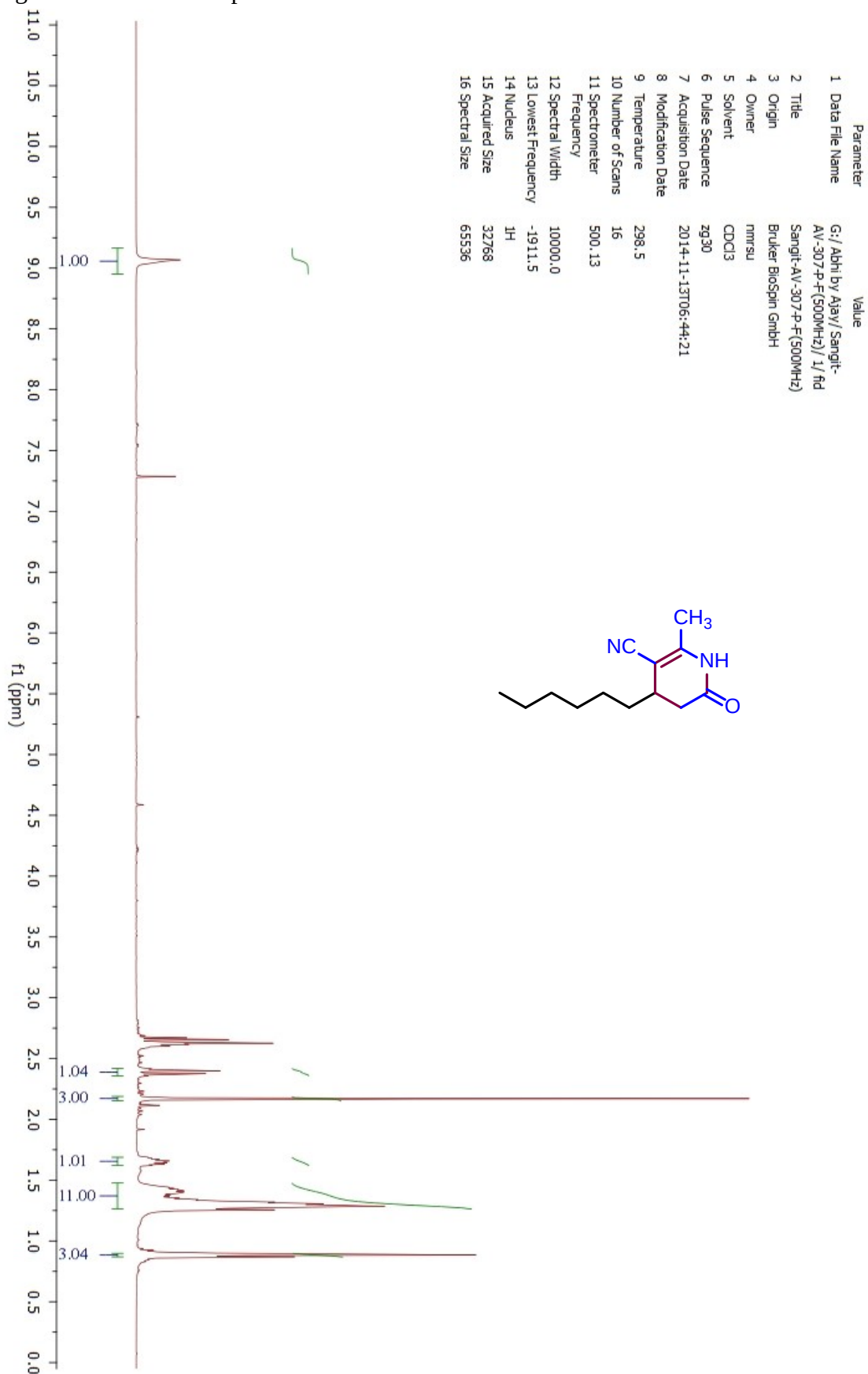
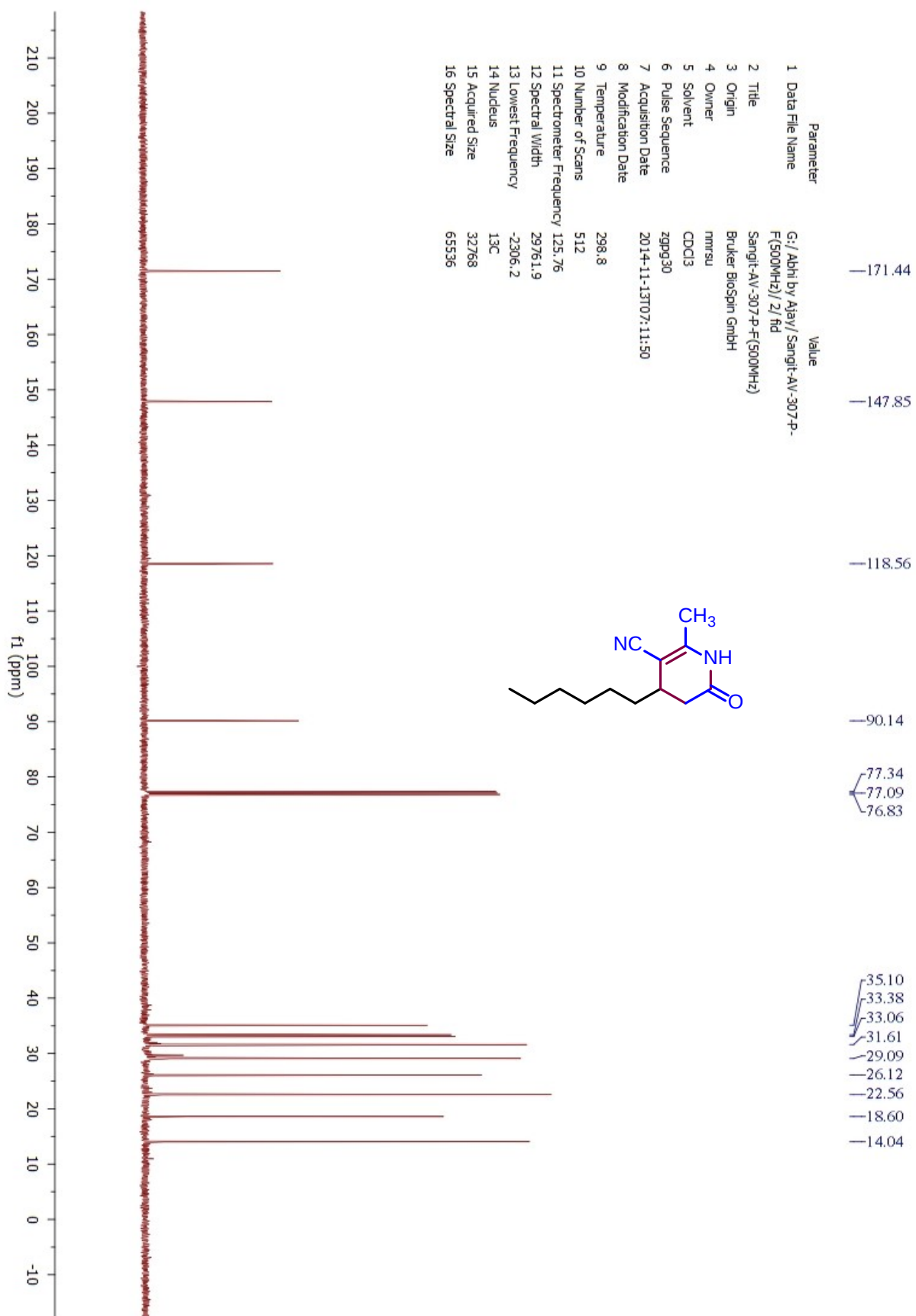


Figure S112 ^{13}C NMR spectra of **37**



S114

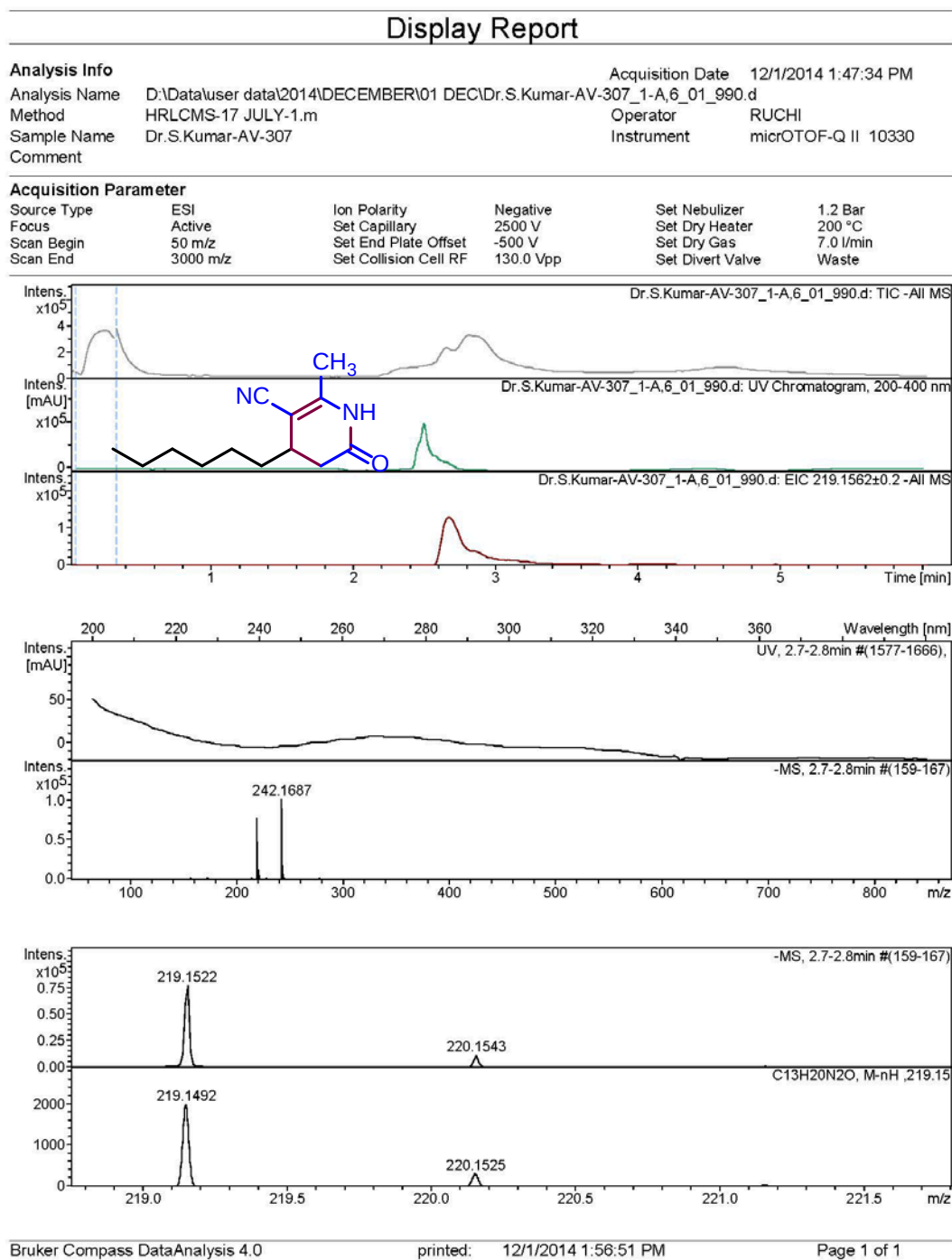


Figure S114 ^1H NMR spectra of **38**

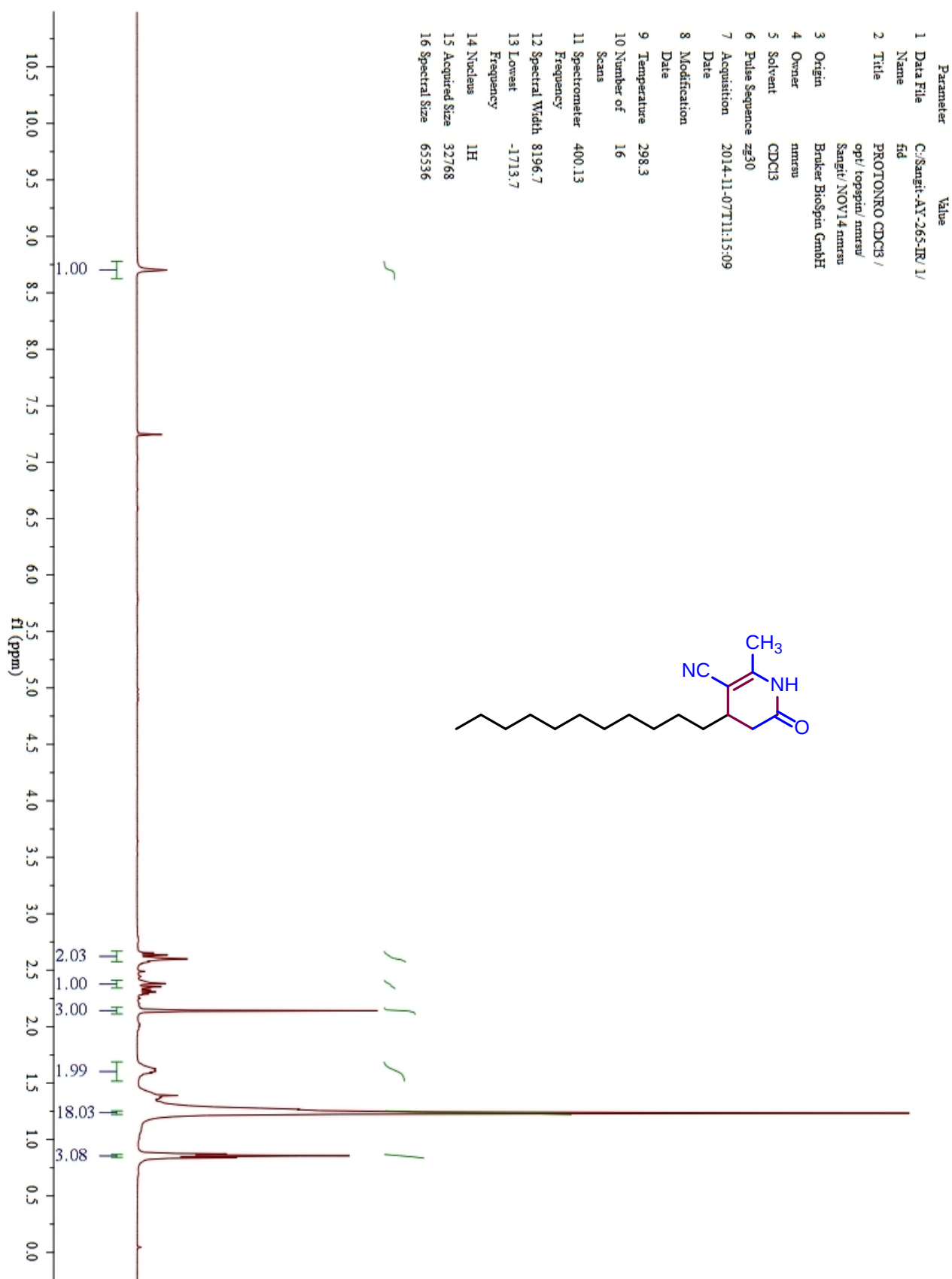


Figure S115 ^{13}C NMR spectra of **38**

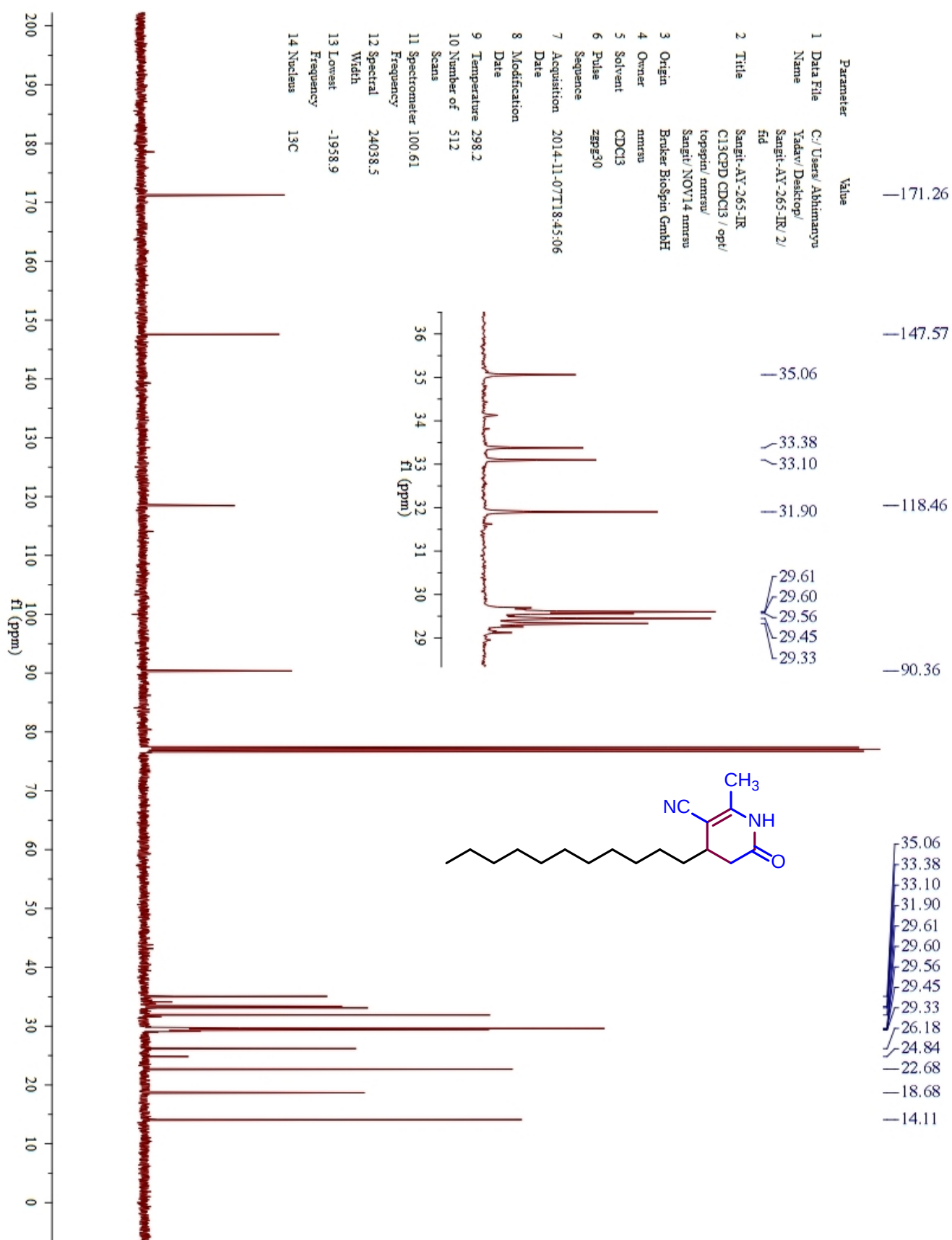


Figure S116 Mass spectra of **38**

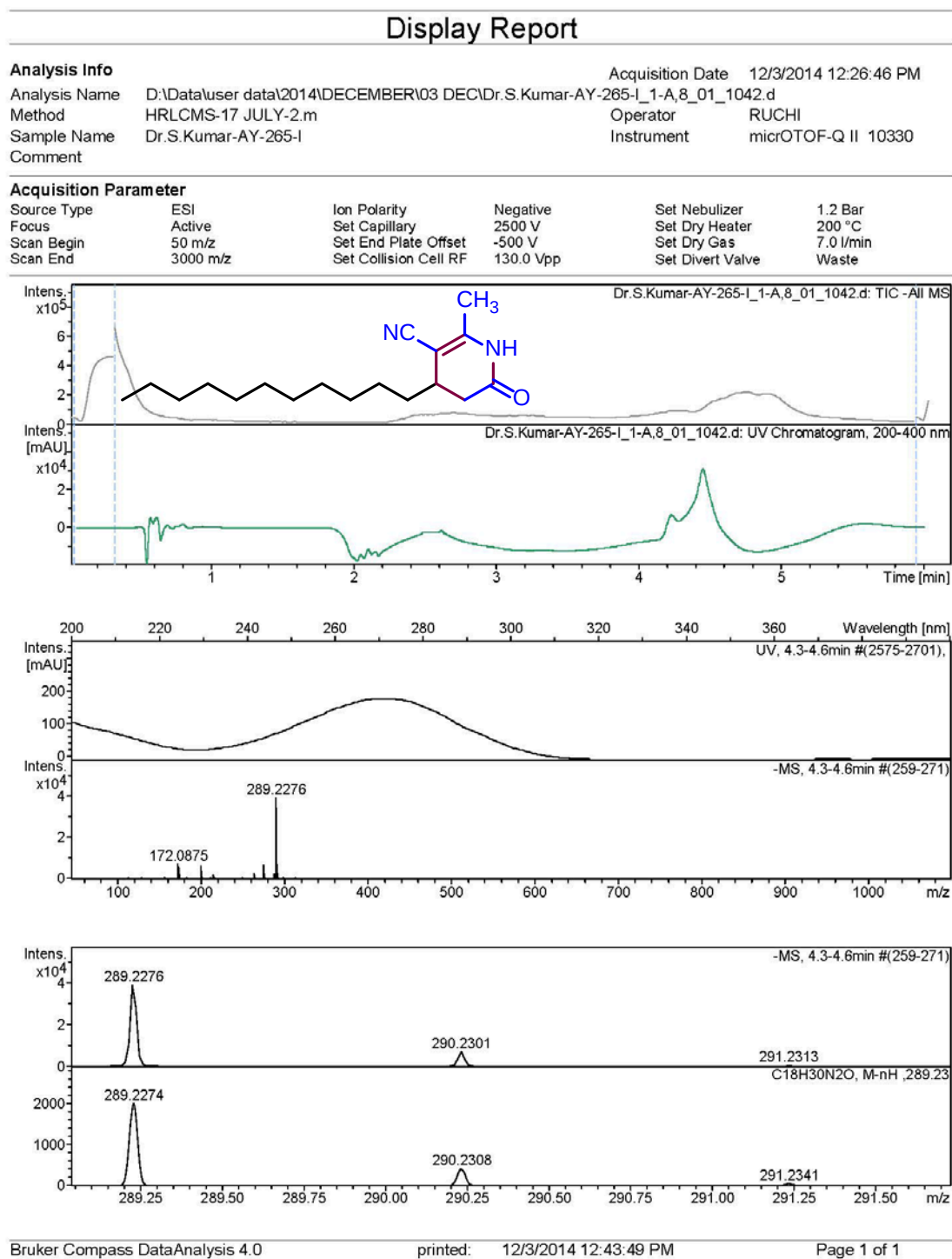


Figure S117 ^1H NMR spectra of **39**

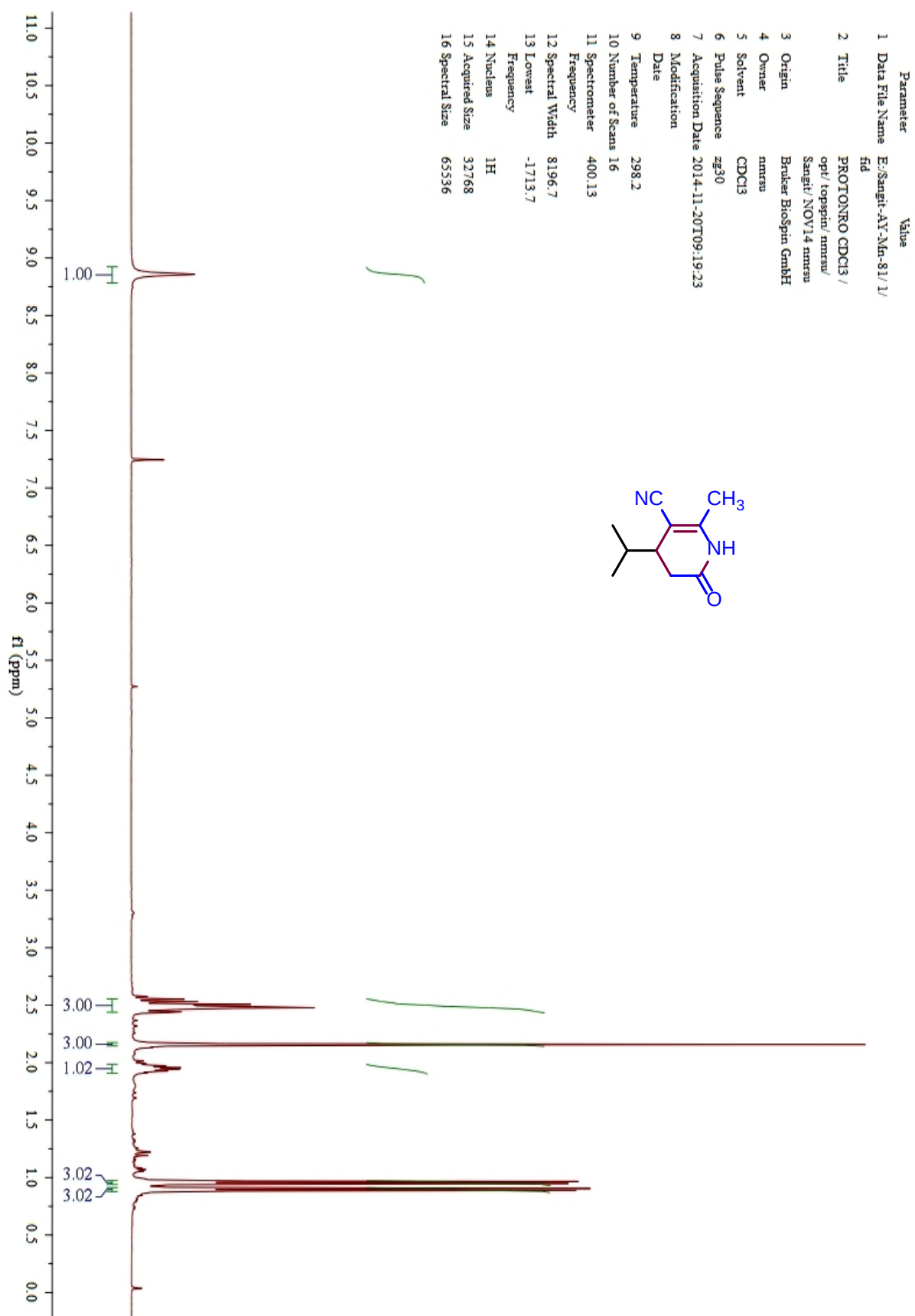


Figure S118 ¹³C NMR spectra of **39**

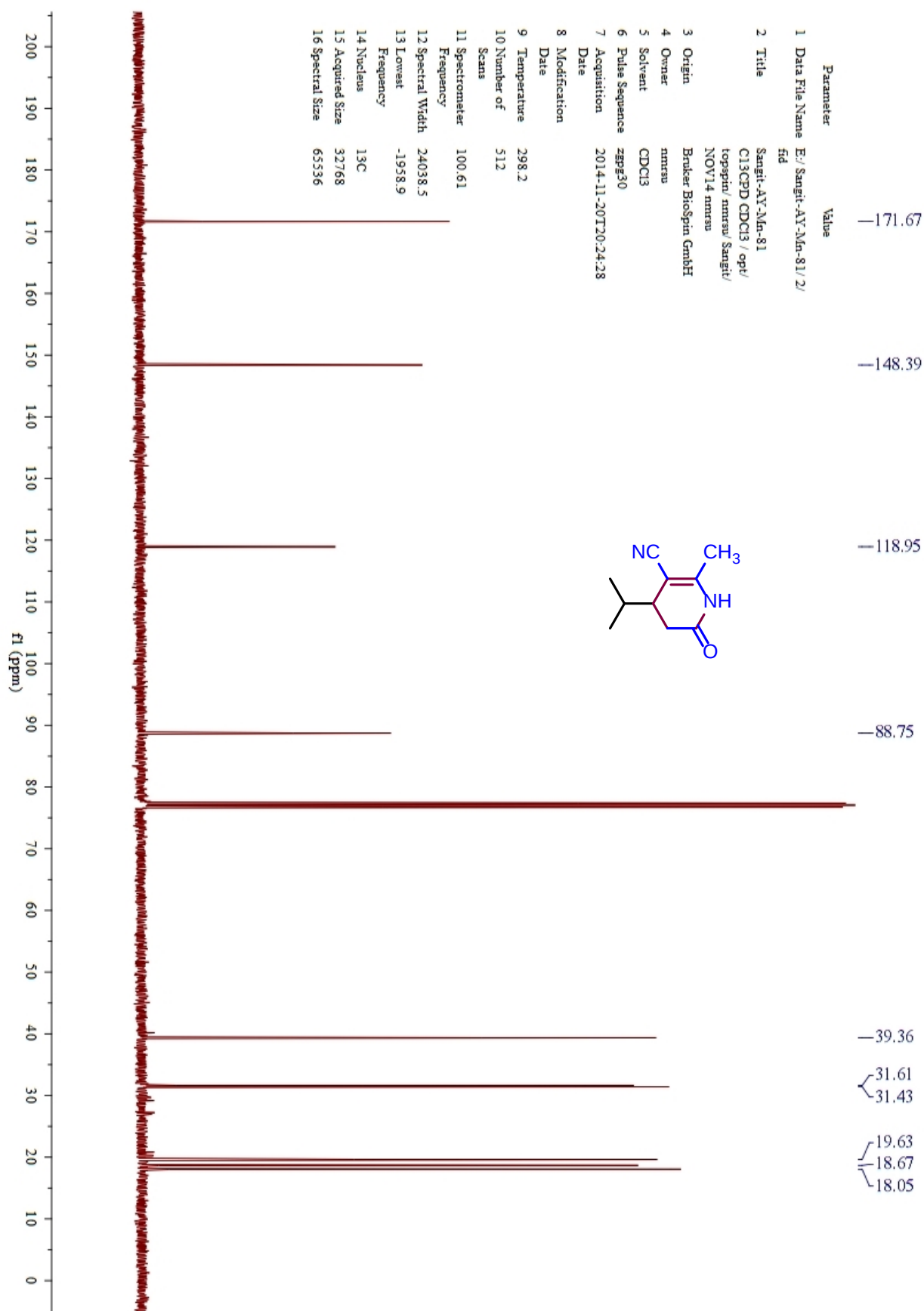


Figure S119 Mass spectra of 39

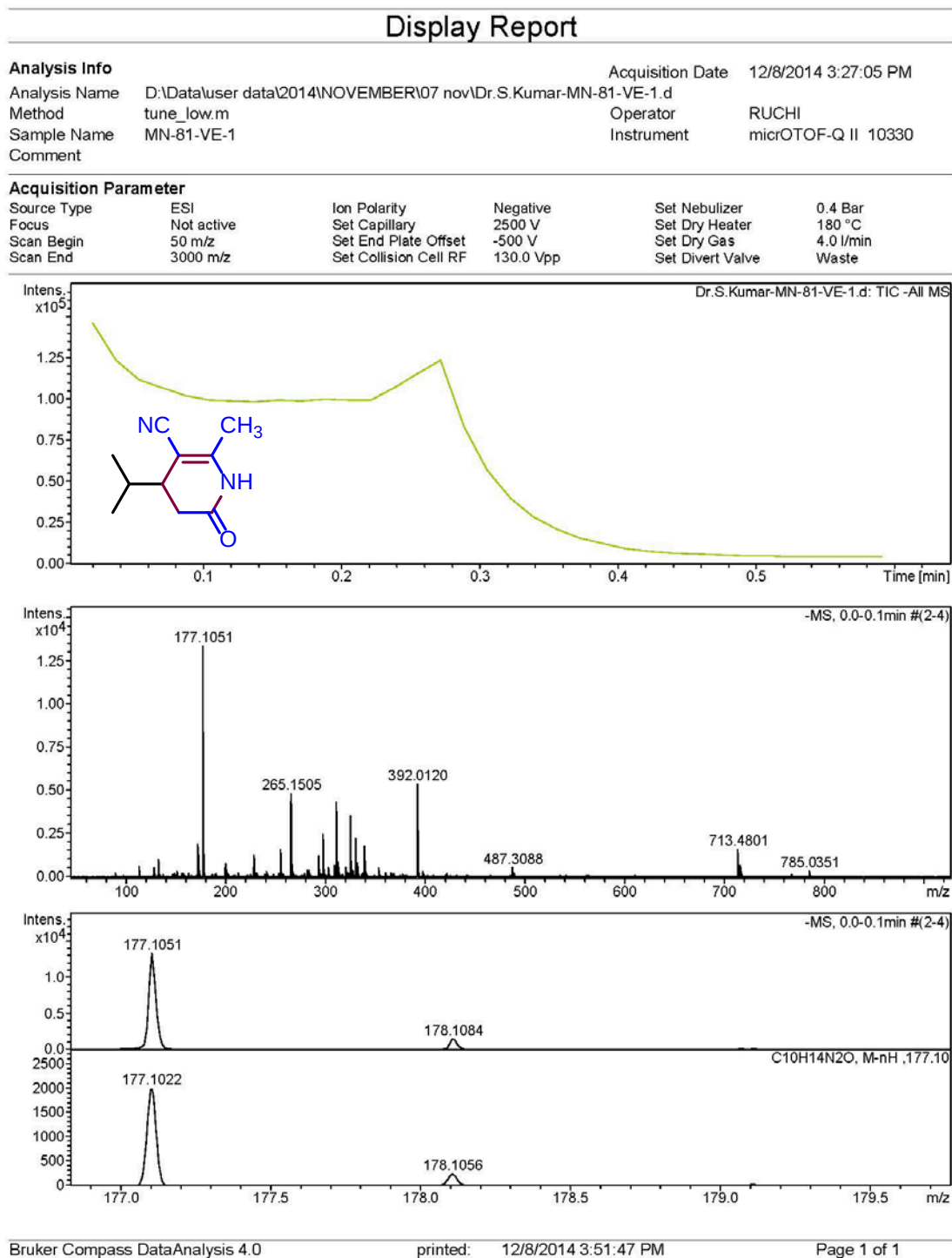


Figure S120 ^1H NMR spectra of **40**

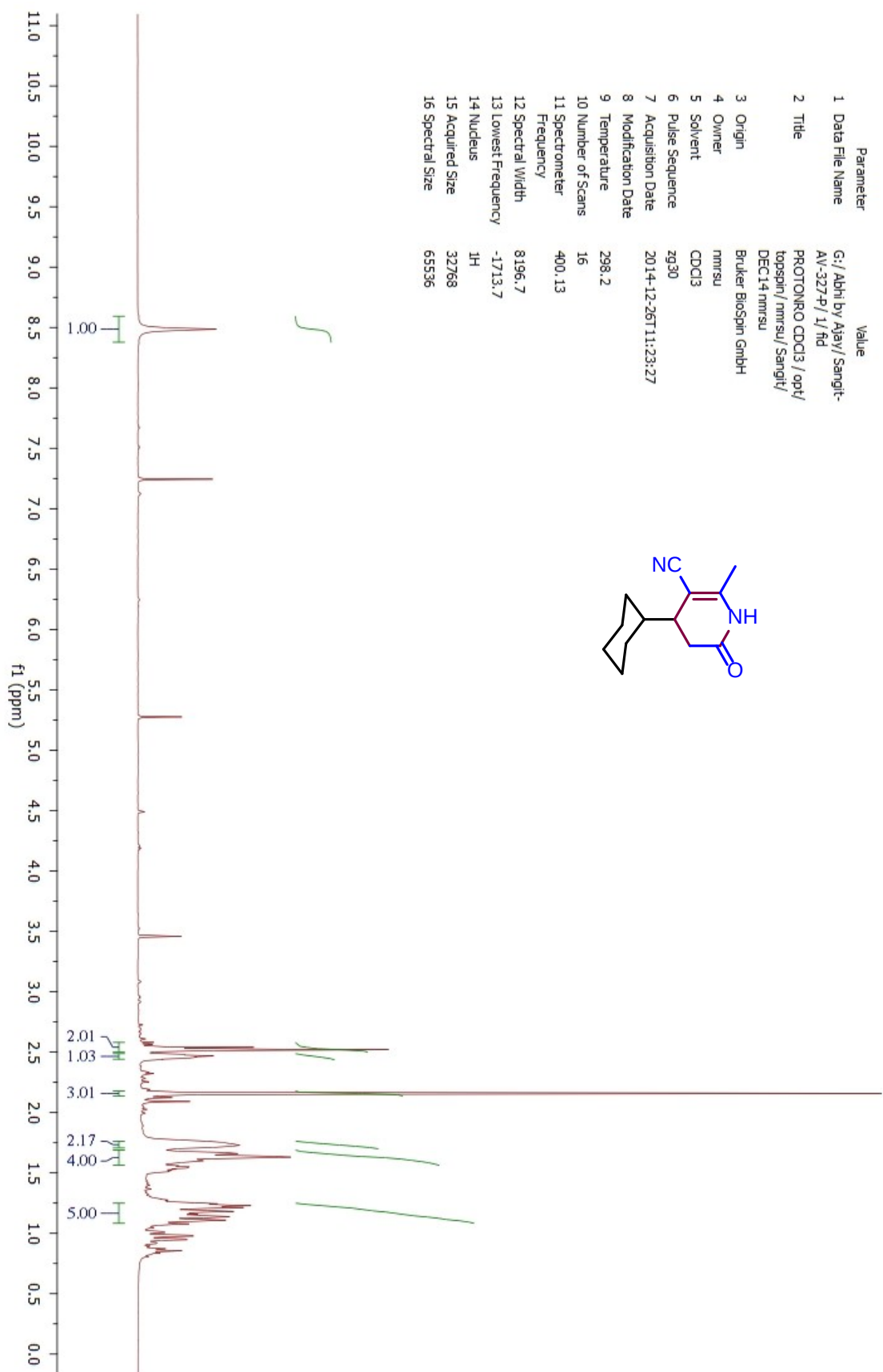


Figure S121 ^{13}C NMR spectra of **40**

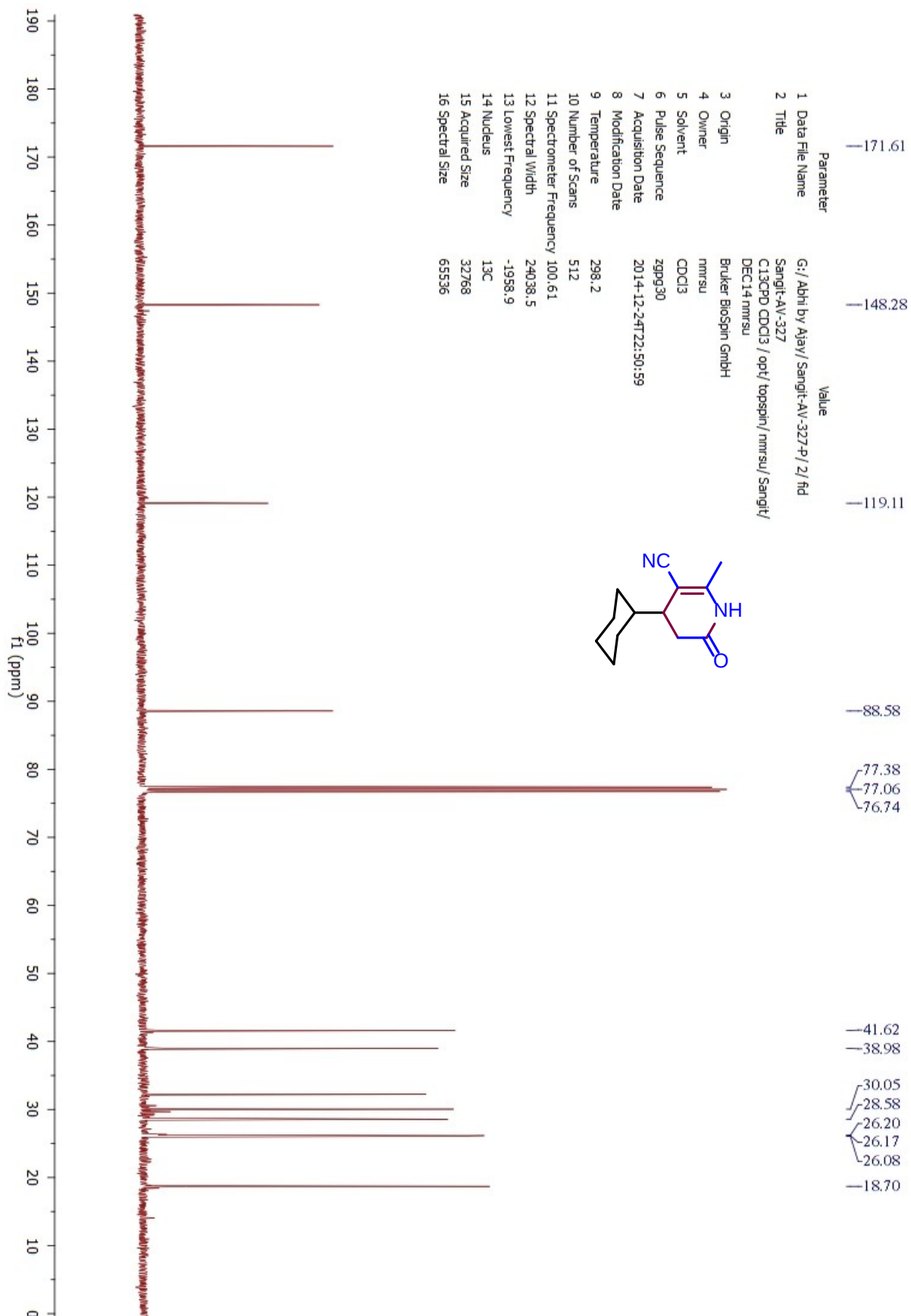


Figure S122 HRMS spectra of **40**

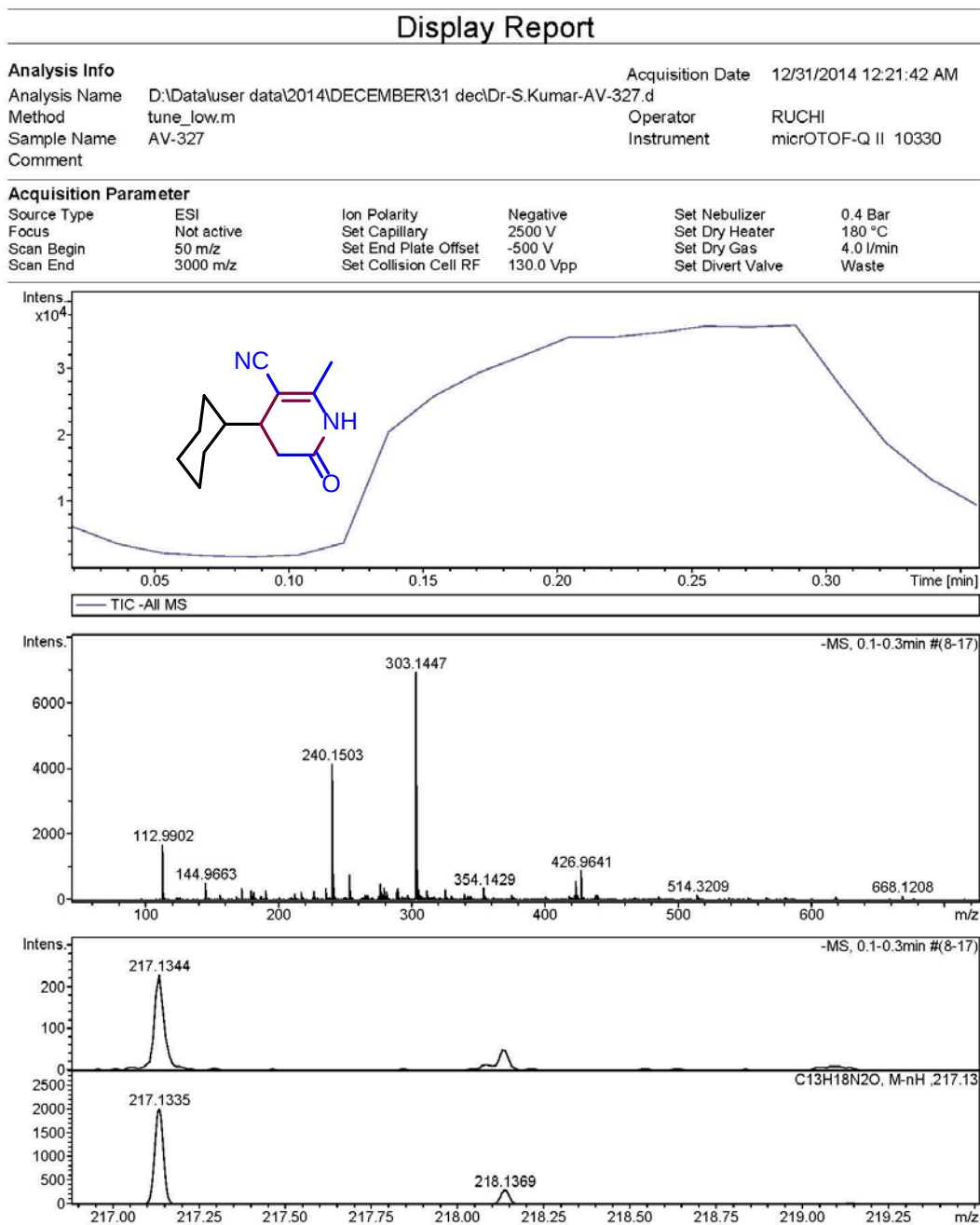


Figure S123 ^1H NMR spectra of **41**

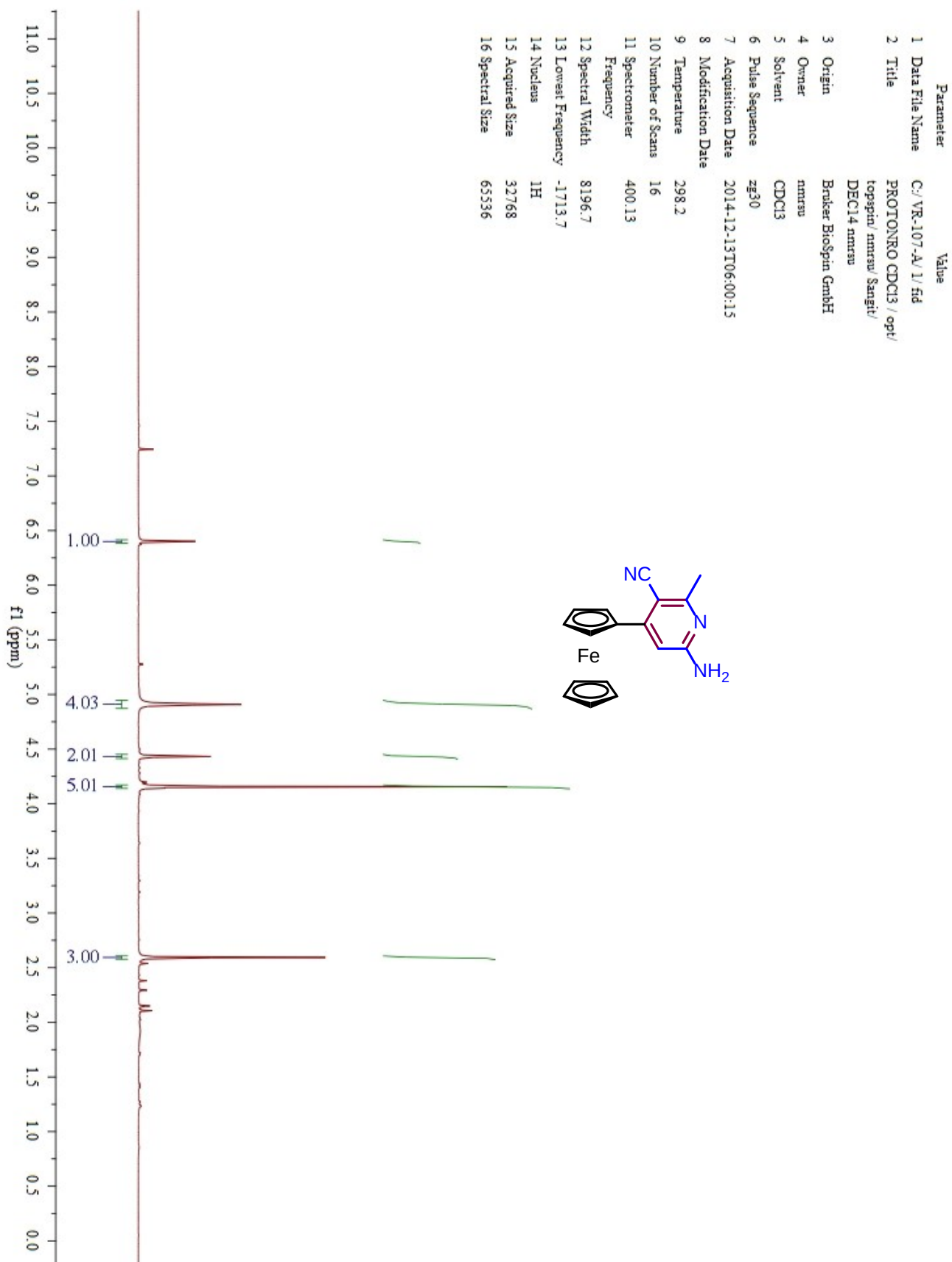


Figure S124 ¹³C NMR spectra of **41**

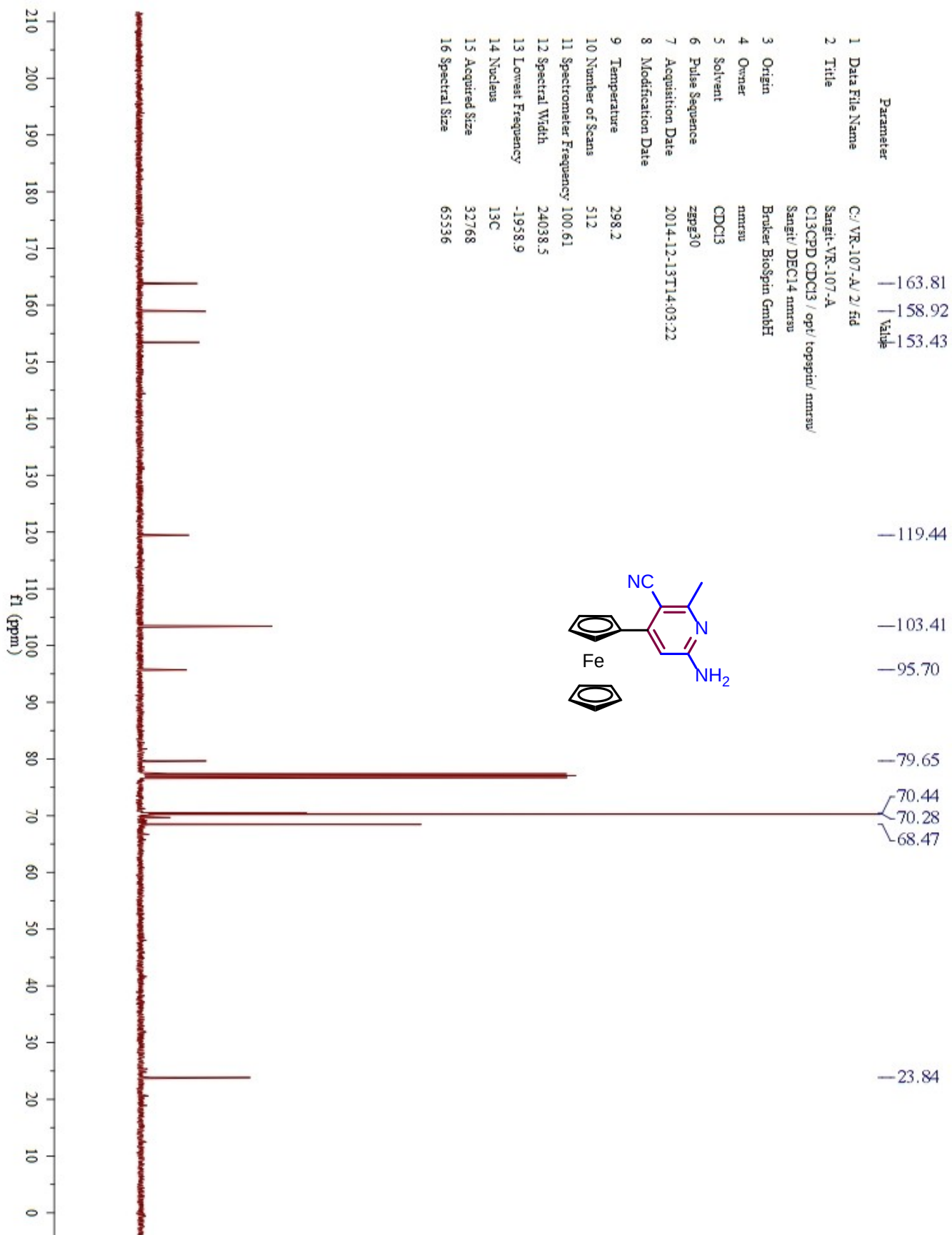


Figure S125 DEPT spectra of **41**

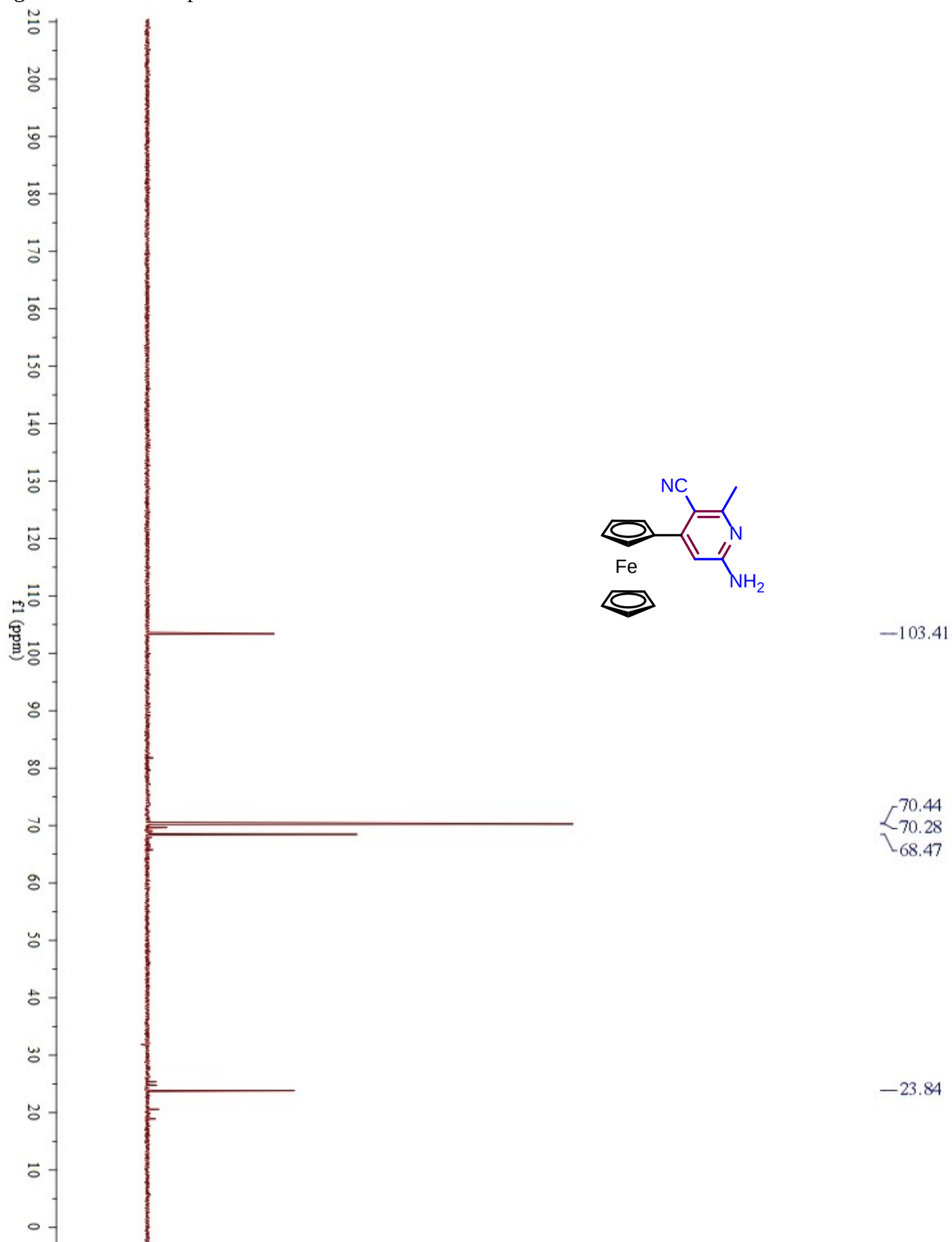


Figure S126 Mass spectra of **41**

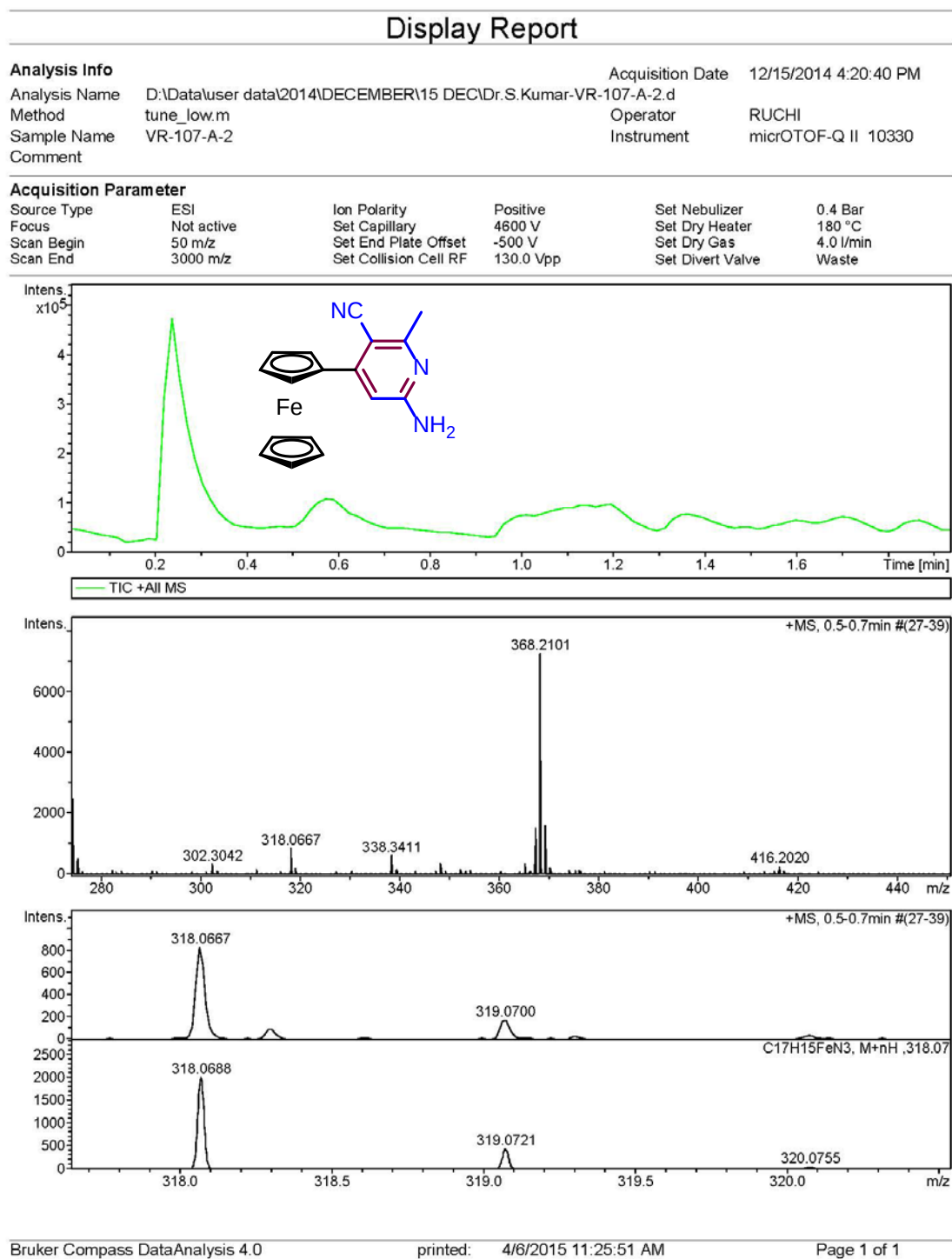


Figure S127 ¹H NMR spectra of **42**

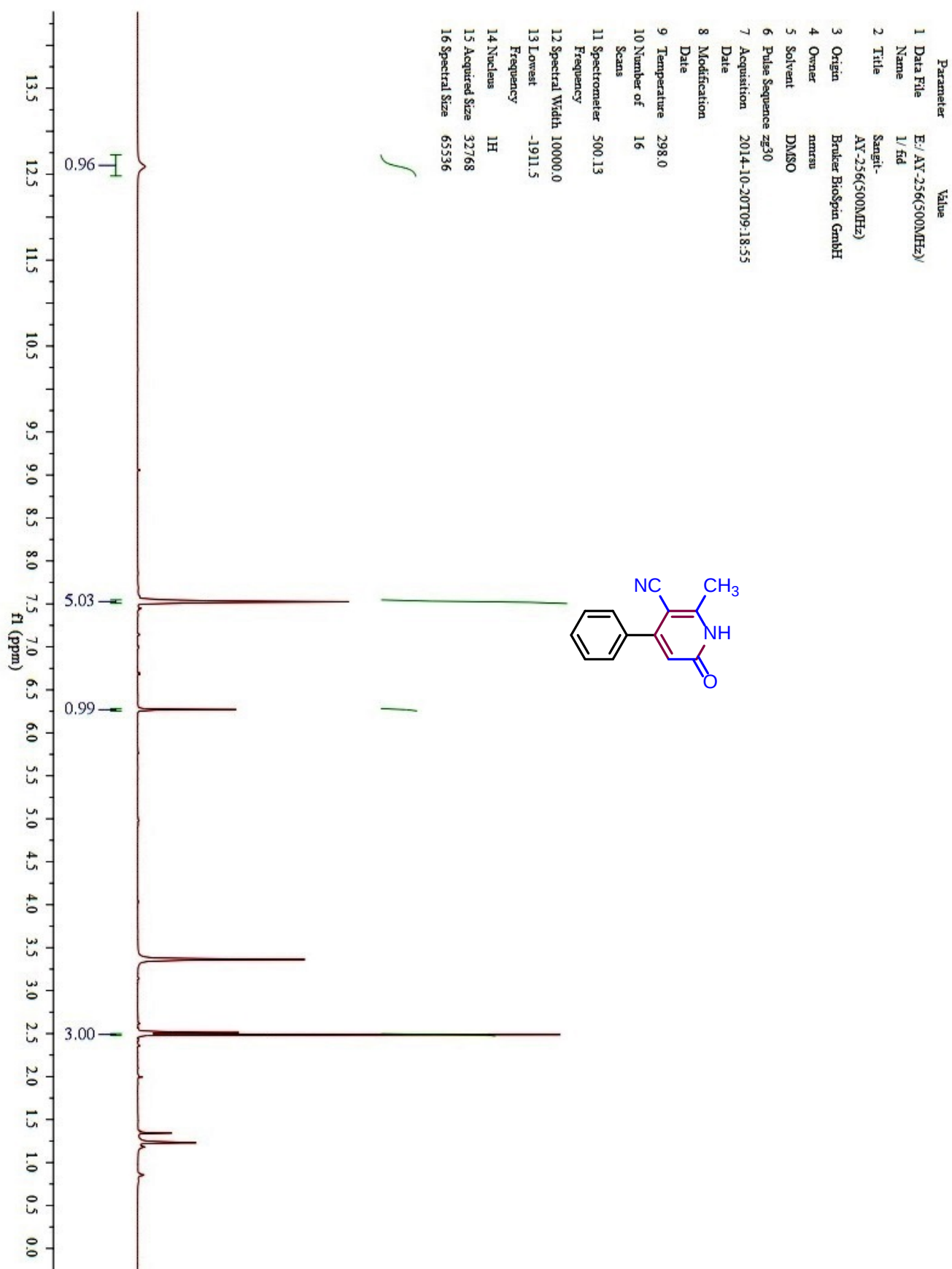


Figure S128 ^{13}C NMR spectra of **42**

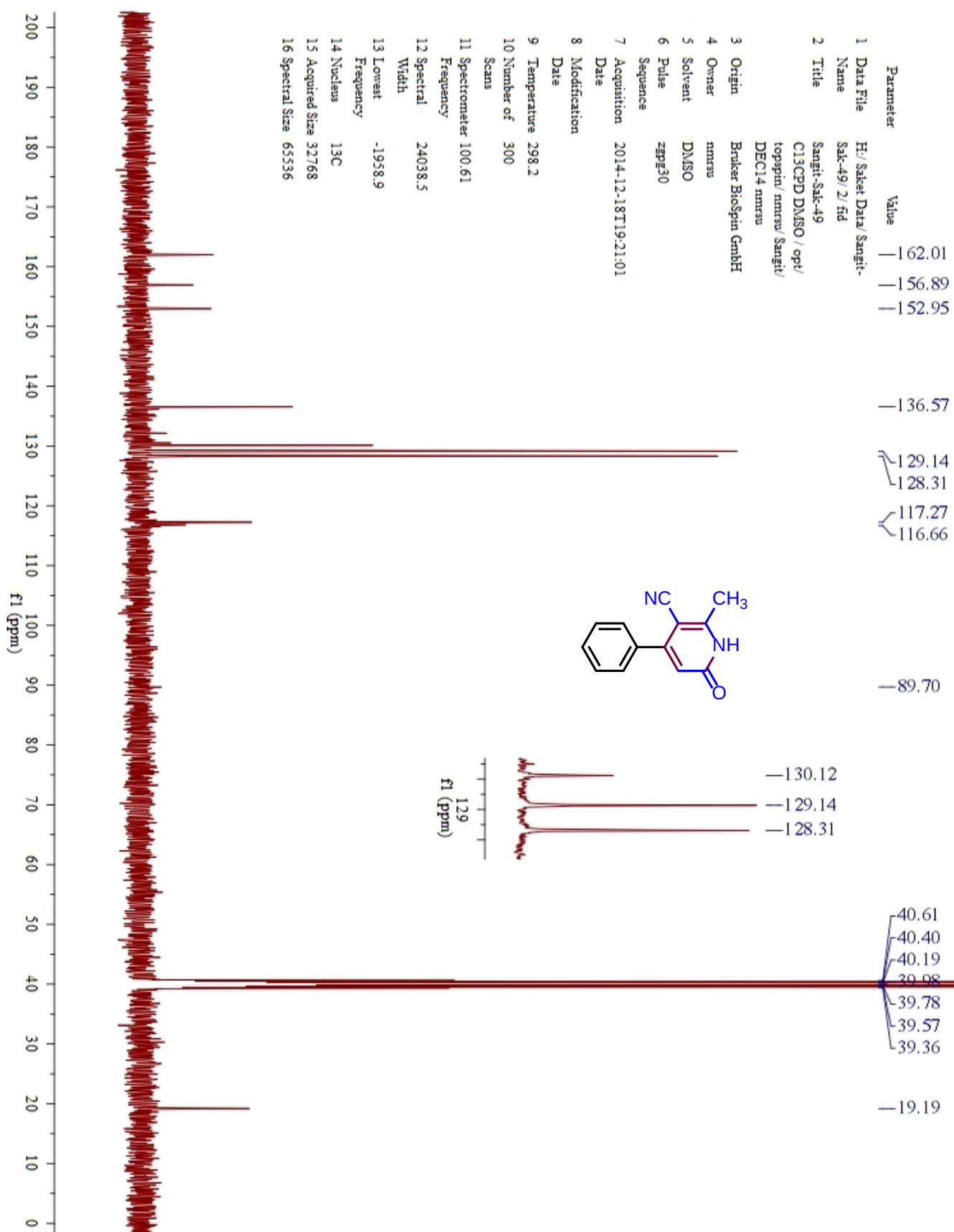


Figure S129 135-DEPT spectra of **42**

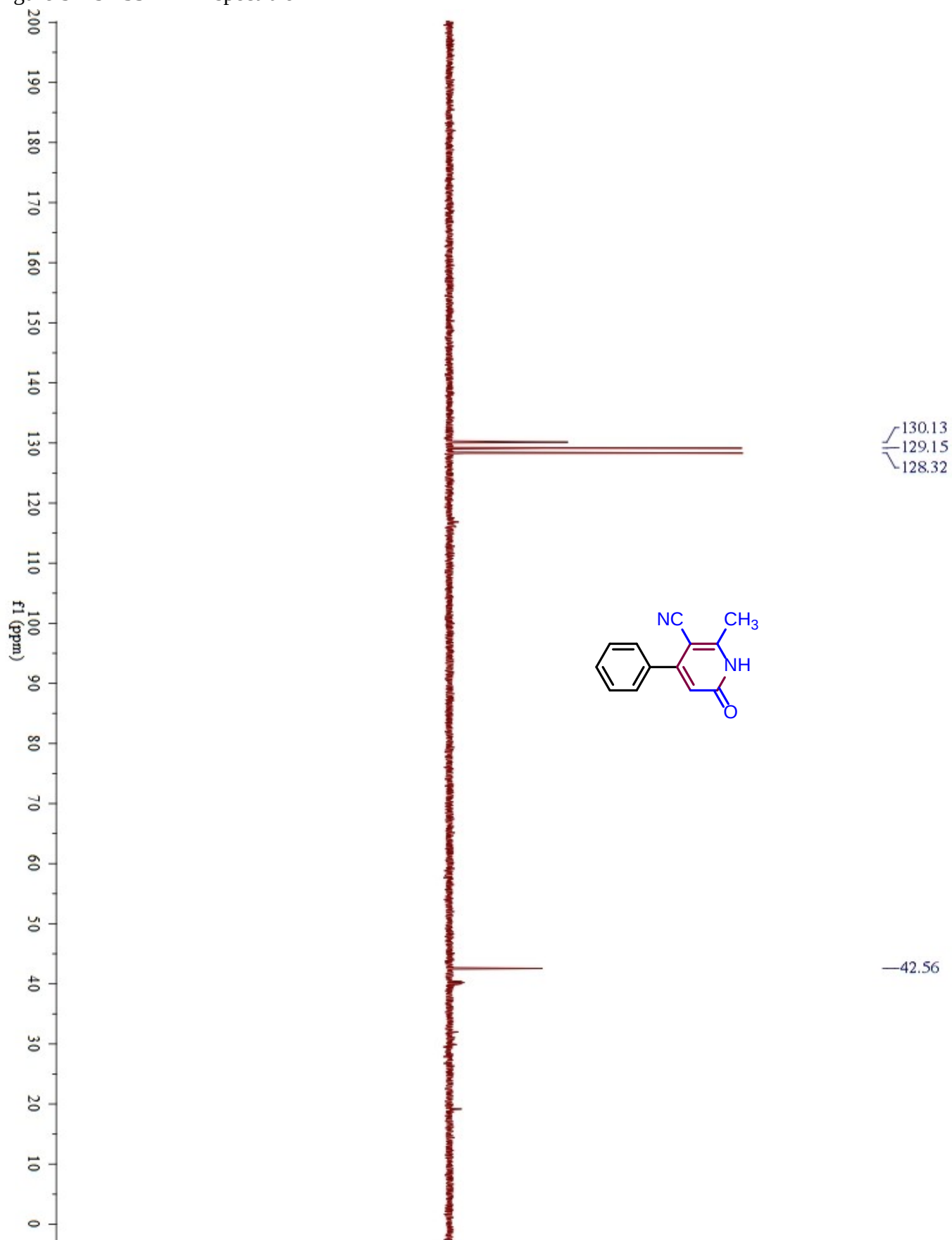


Figure S130 Mass spectra of 42

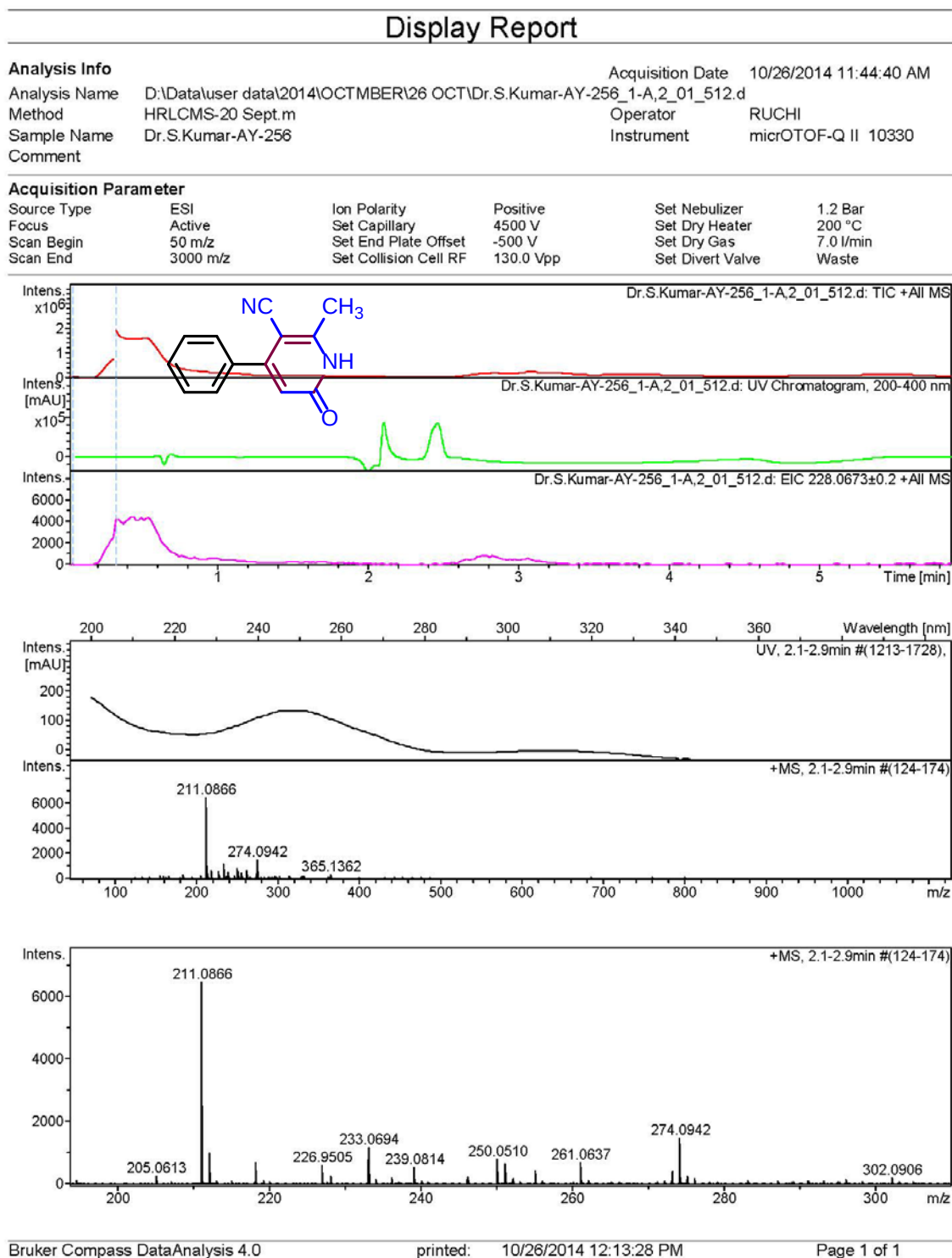


Figure S131 ^1H NMR spectra of **43**

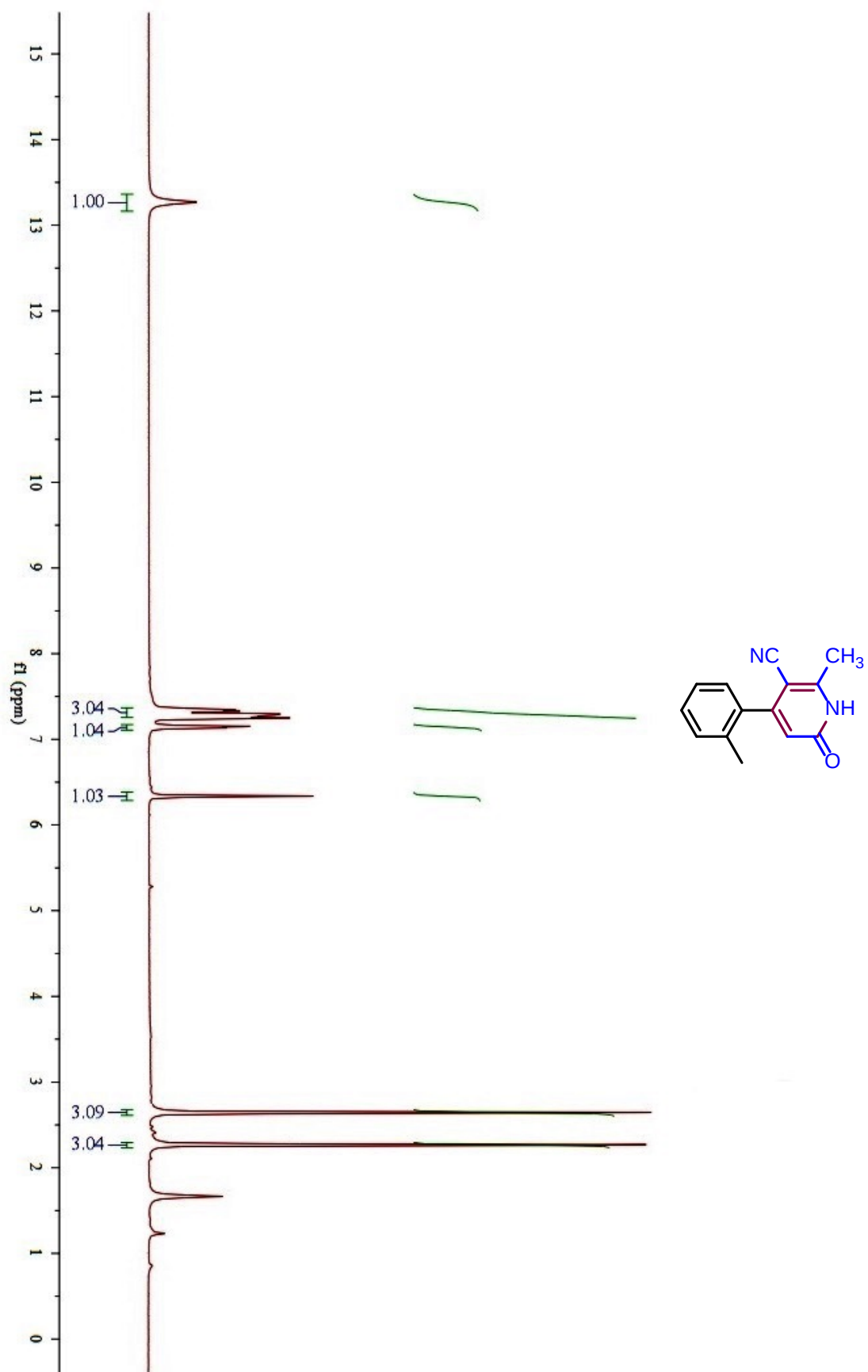


Figure S132 ^{13}C NMR spectra of **43**

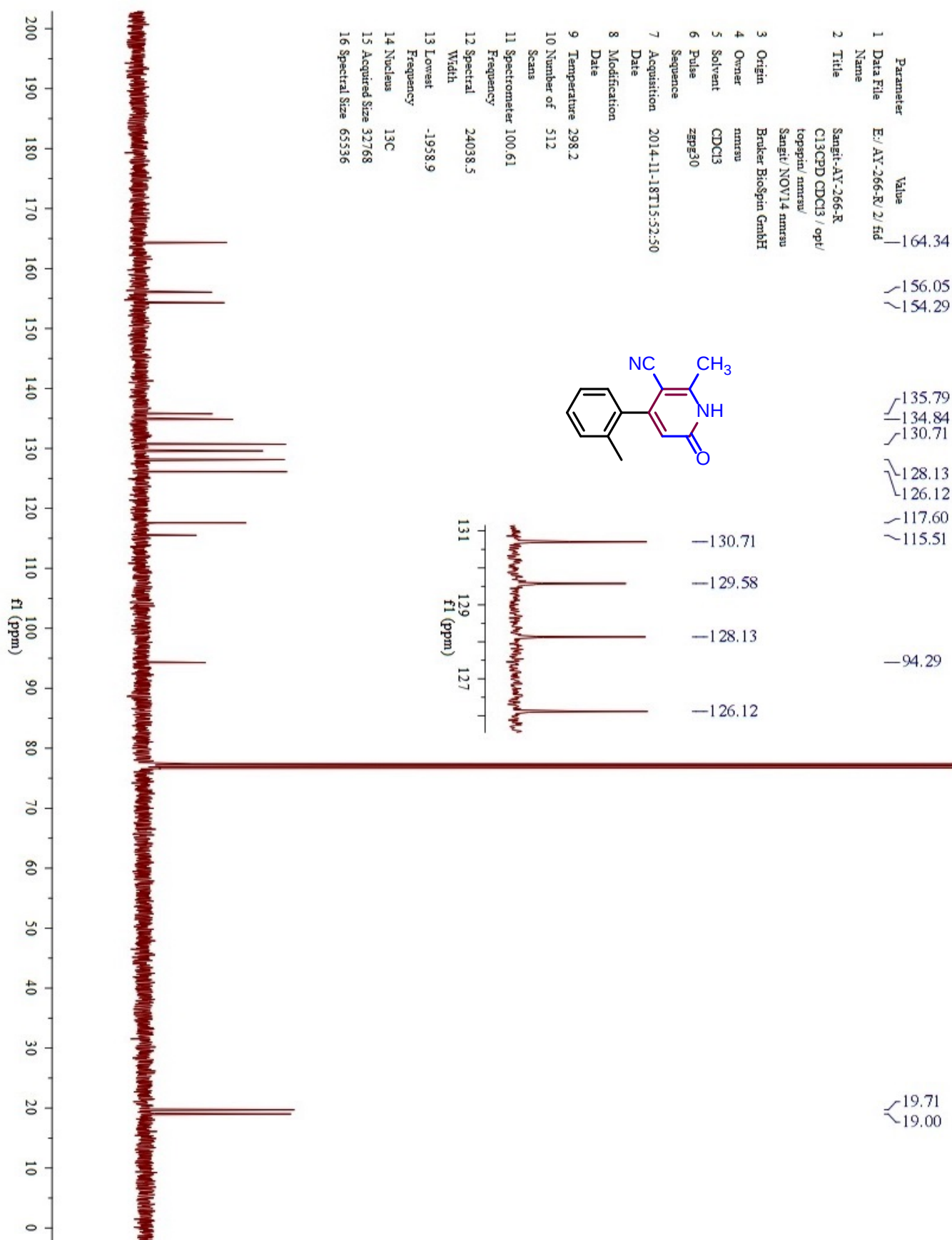


Figure S133 Mass spectra of 43

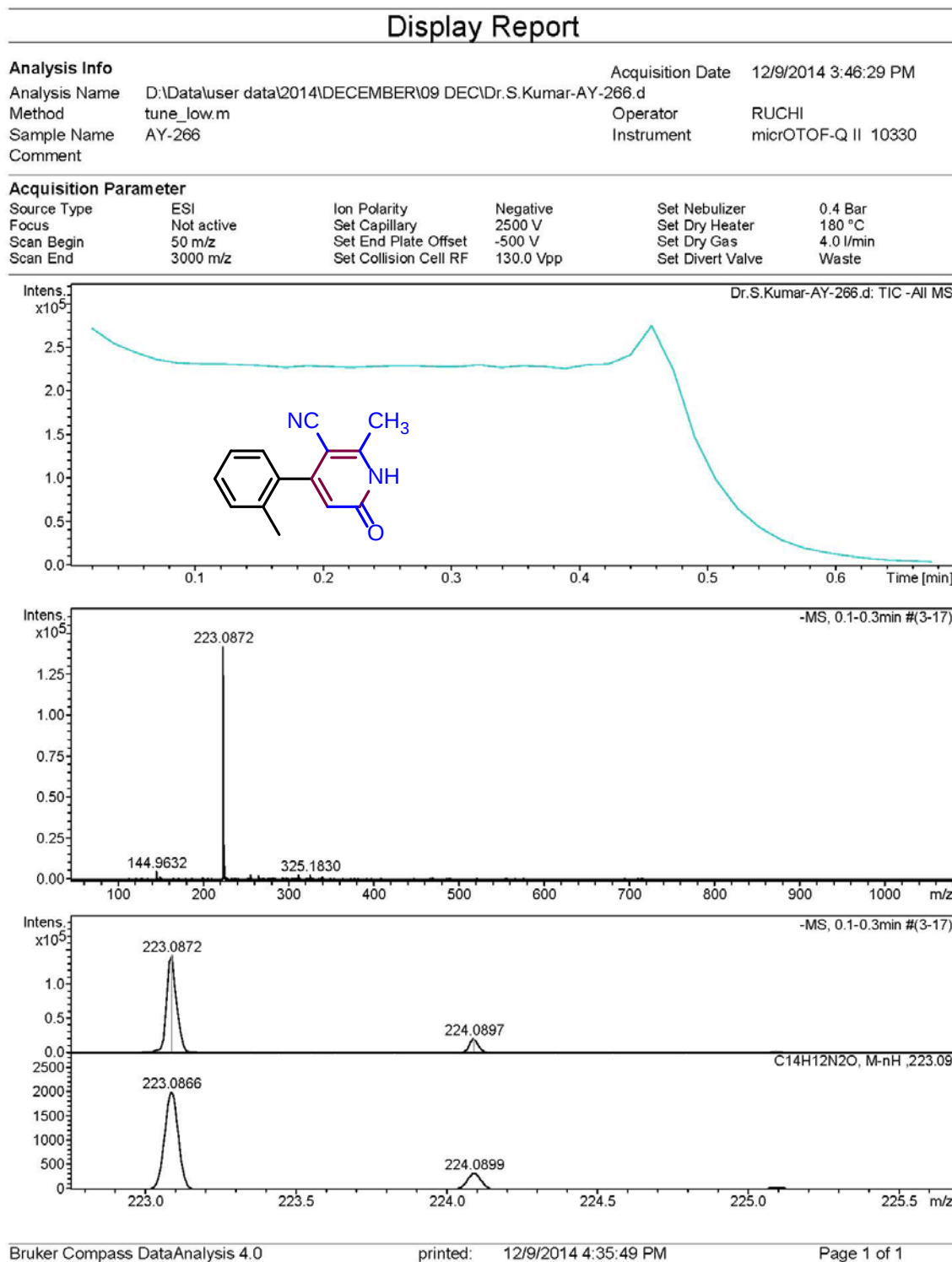


Figure S134 ¹H NMR spectra of **44**

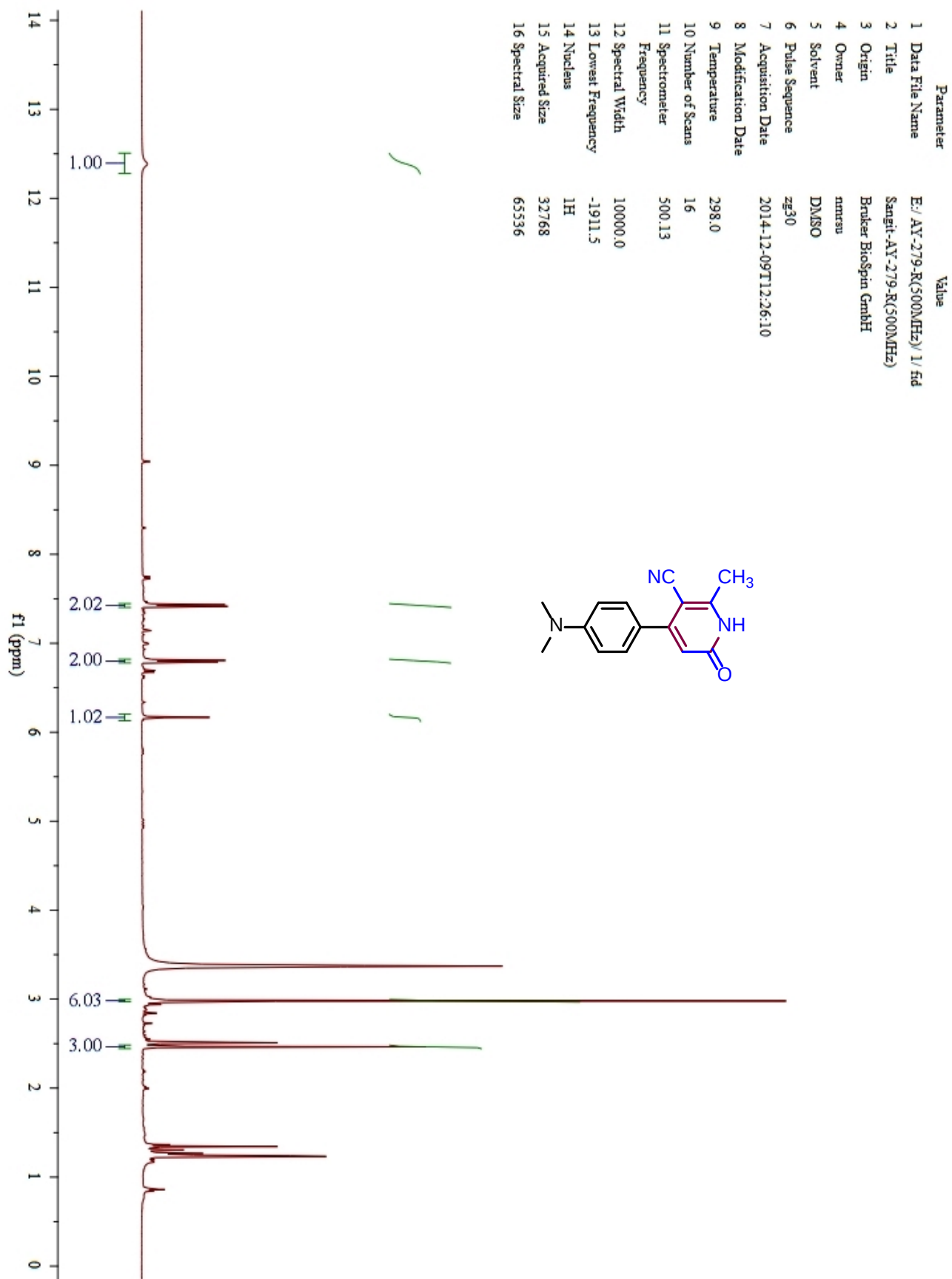


Figure S135 ^{13}C NMR spectra of **44**

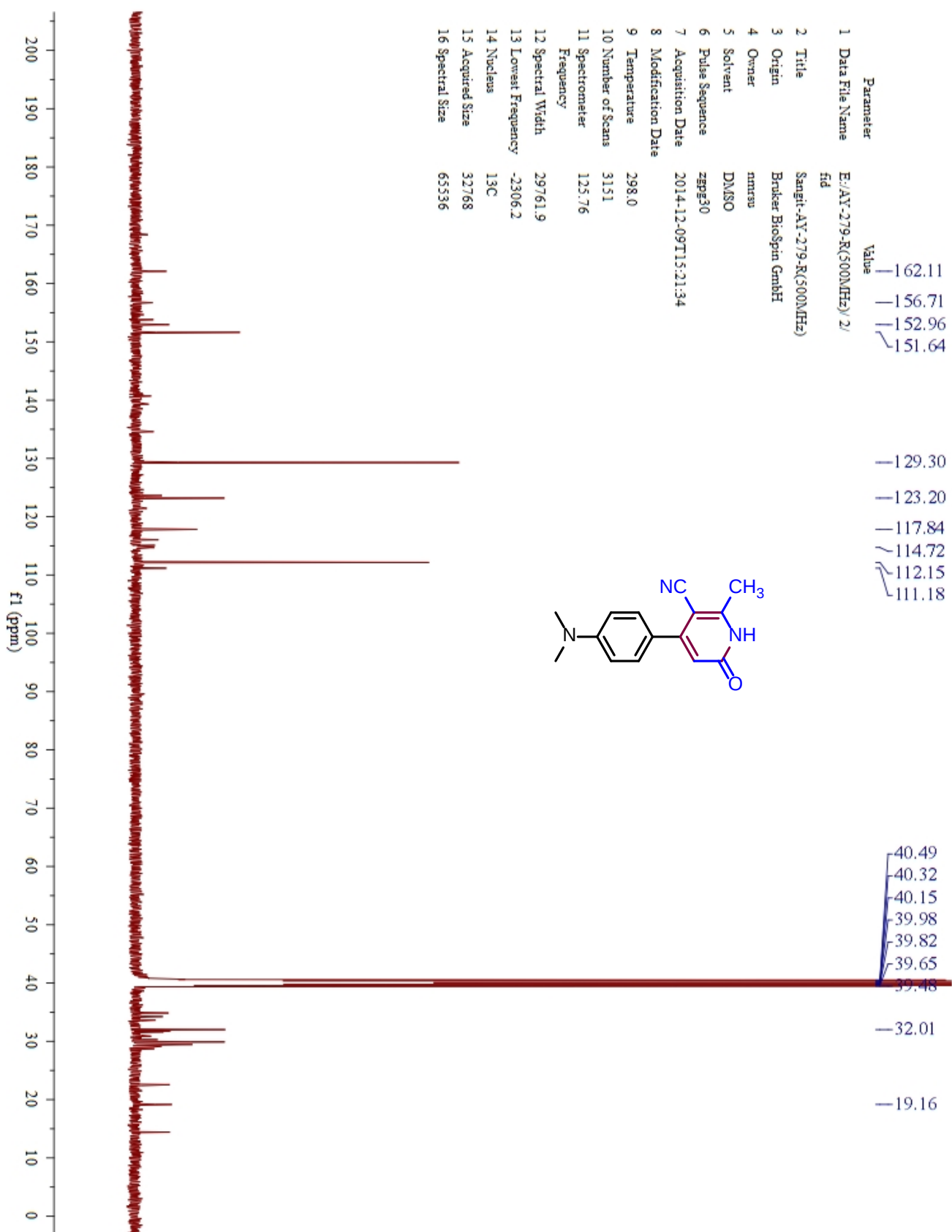
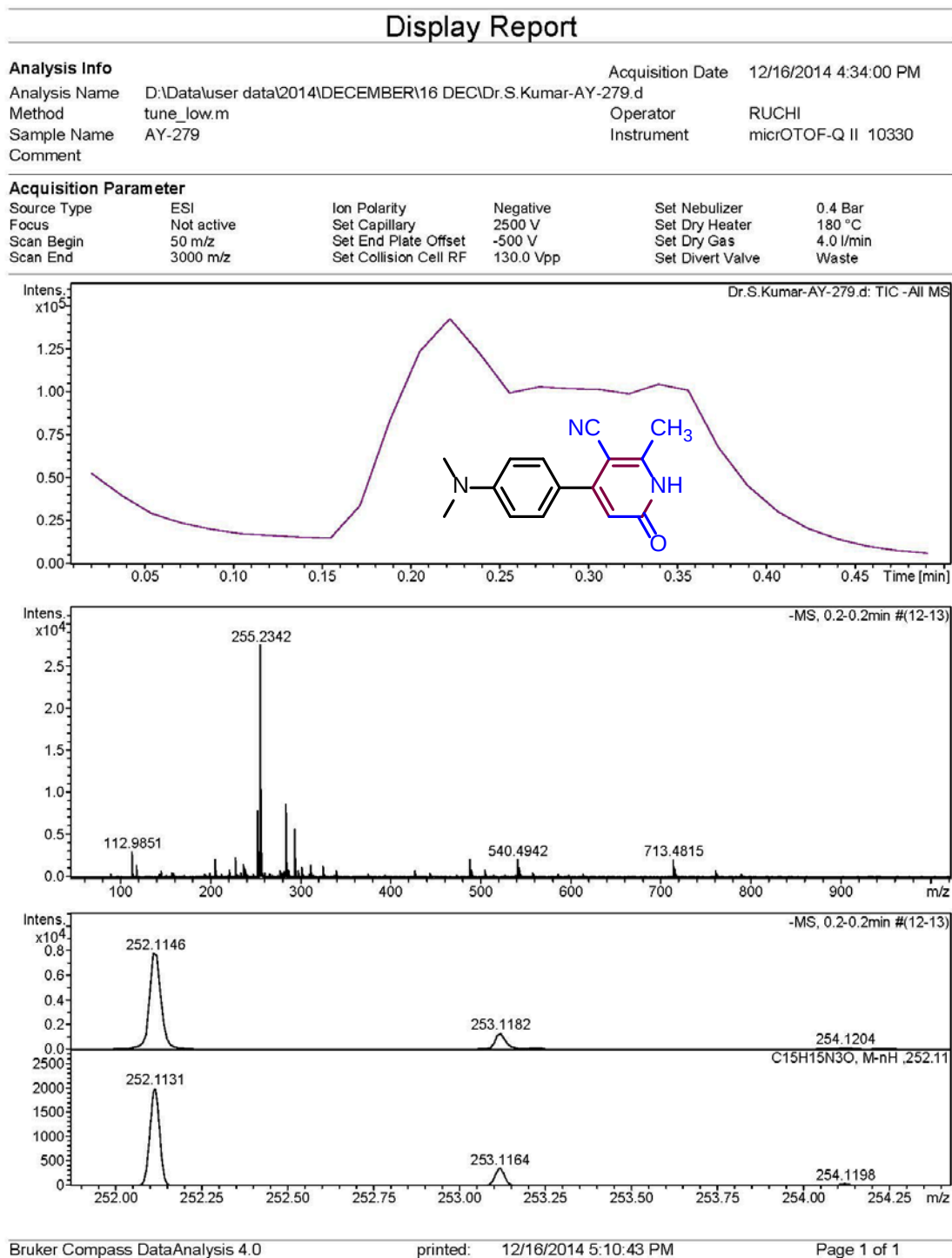


Figure S136 Mass spectra of **44**



Parameter	Value
1 Data File Name	E:/AY-296/1/ f1d
2 Title	PROTONRO DMSO / opt/ topspin/ nmrvu/ Sangiv/ DEC14
3 Origin	nmrvu
4 Owner	Broker BioSpin GmbH
5 Solvent	DMSO
6 Pulse Sequence	zg30
7 Acquisition Date	2014-12-23T09:21:37
8 Modification Date	
9 Temperature	298.2
10 Number of Scans	16
11 Spectrometer Frequency	400.13
12 Spectral Width	8196.7
13 Lowest Frequency	-1713.7
14 Nucleus	¹ H
15 Acquired Size	32768
16 Spectral Size	65536

Cc1nc(C#N)c(C(=O)N)cc1-c1ccc(C)cc1

Chemical structure of 4-(4-methylphenyl)-2-methyl-6-oxo-1,2,3,4-tetrahydropyridine-3-carbonitrile.

¹H NMR spectrum (DMSO-d₆) showing peaks at 1.00 (s, 3H), 2.04 (s, 3H), 2.05 (s, 3H), 3.00 (s, 3H), 3.01 (s, 3H), and 7.04 (d, 2H). Integration values are 1.00, 2.04, 2.05, 1.01, 3.00, and 3.01 respectively.

Figure S138 ^{13}C NMR spectra of **45**

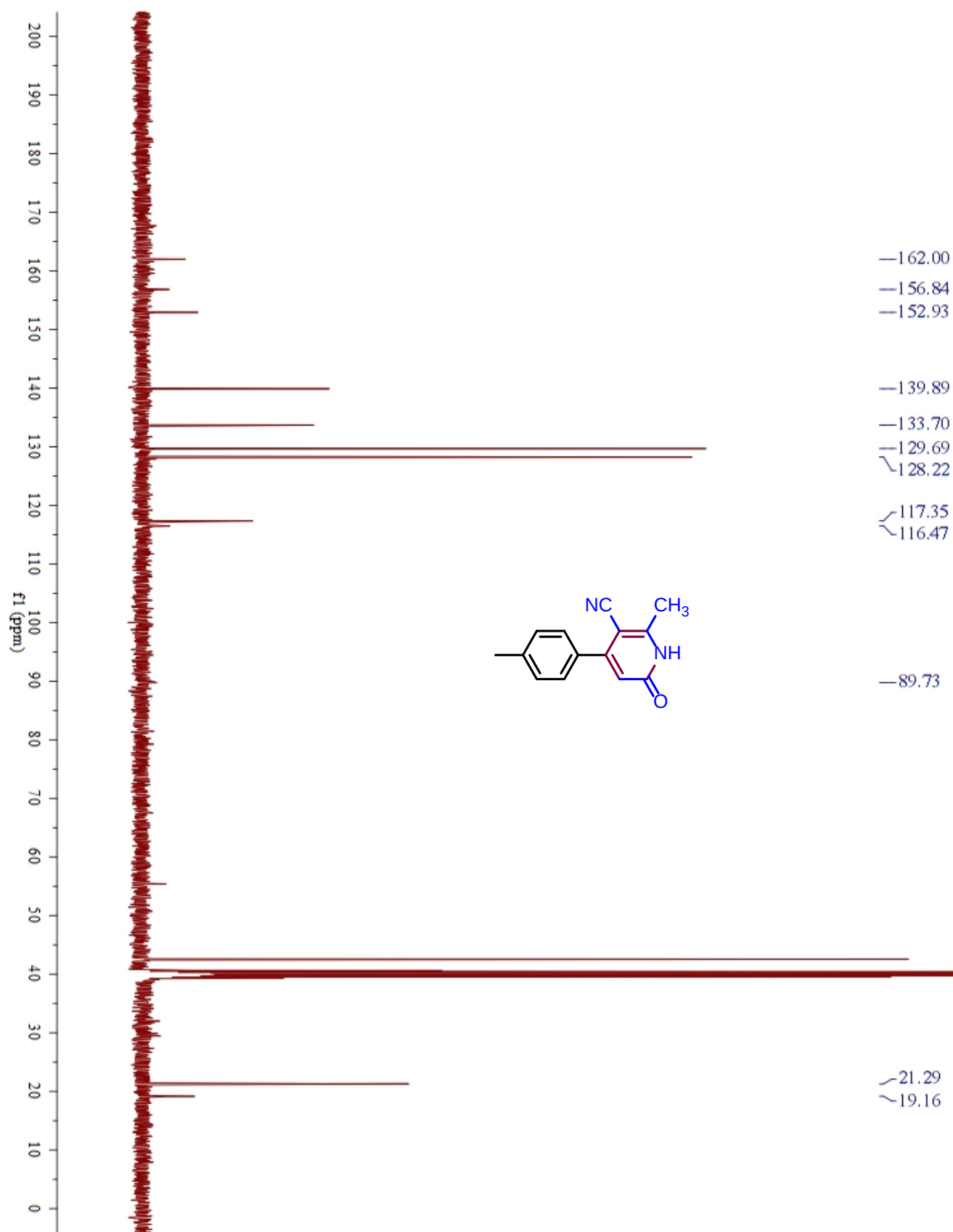


Figure S139 Mass spectra of 45

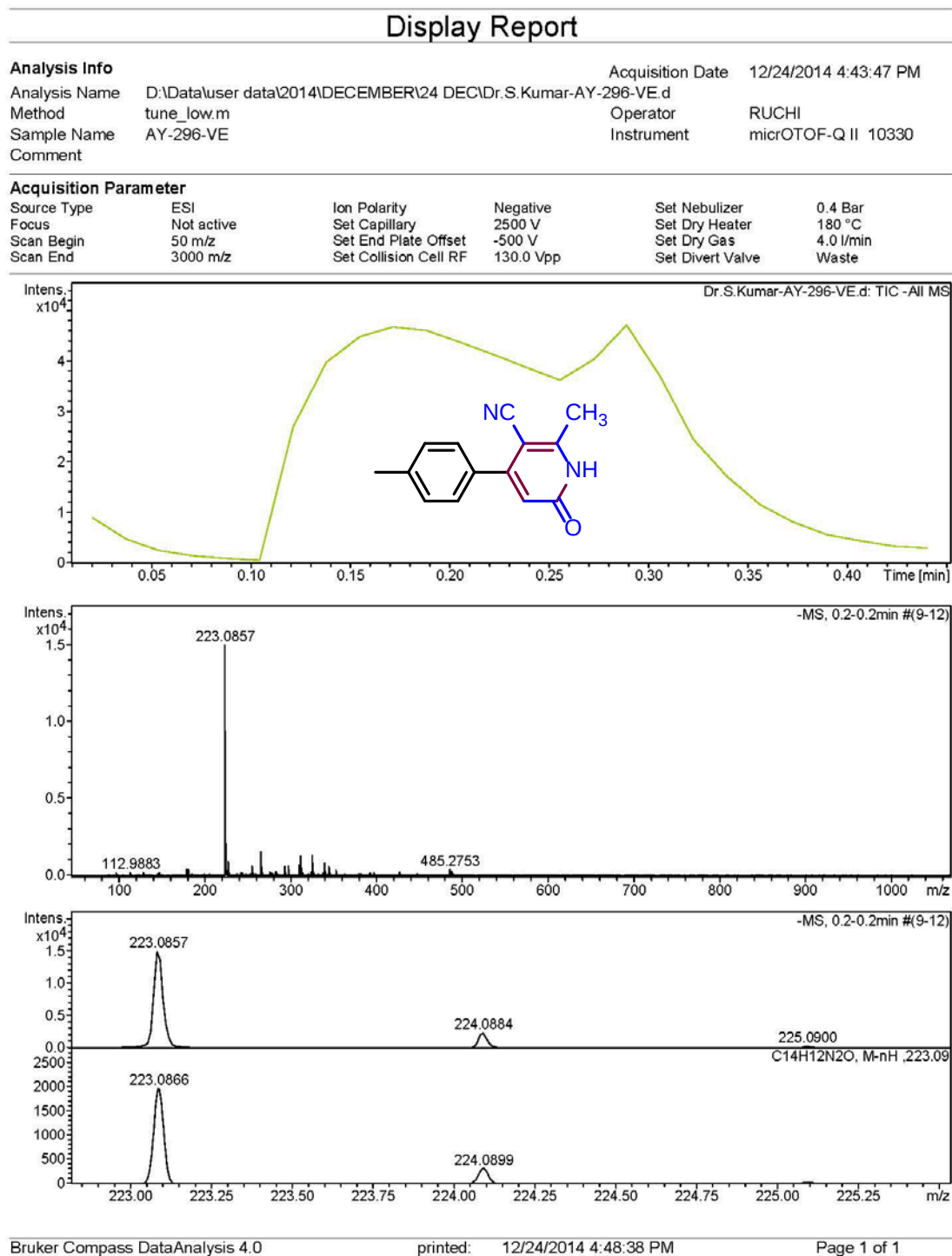


Figure S140 ¹H NMR spectra of **46**

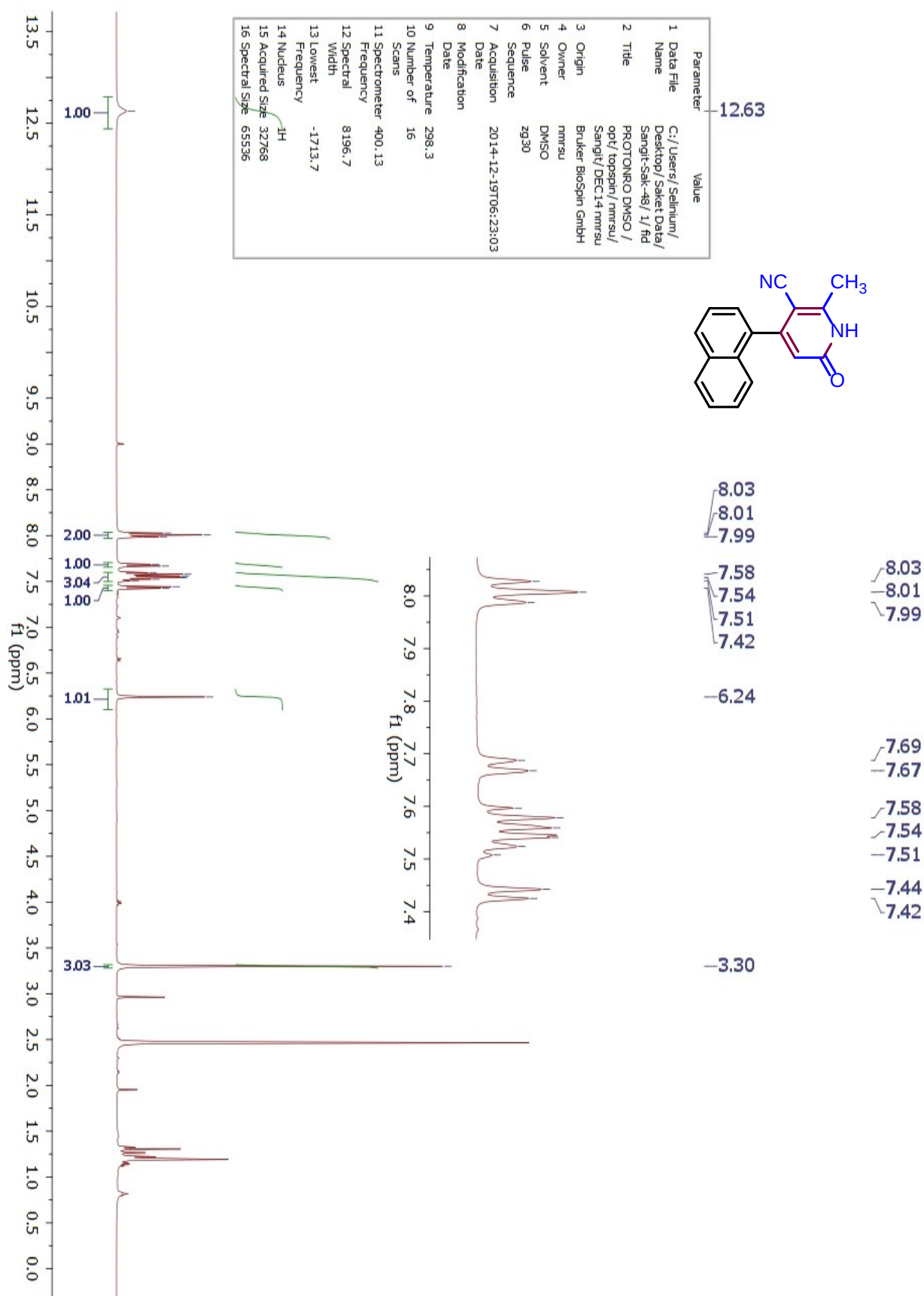


Figure S141 ^{13}C NMR spectra of **46**

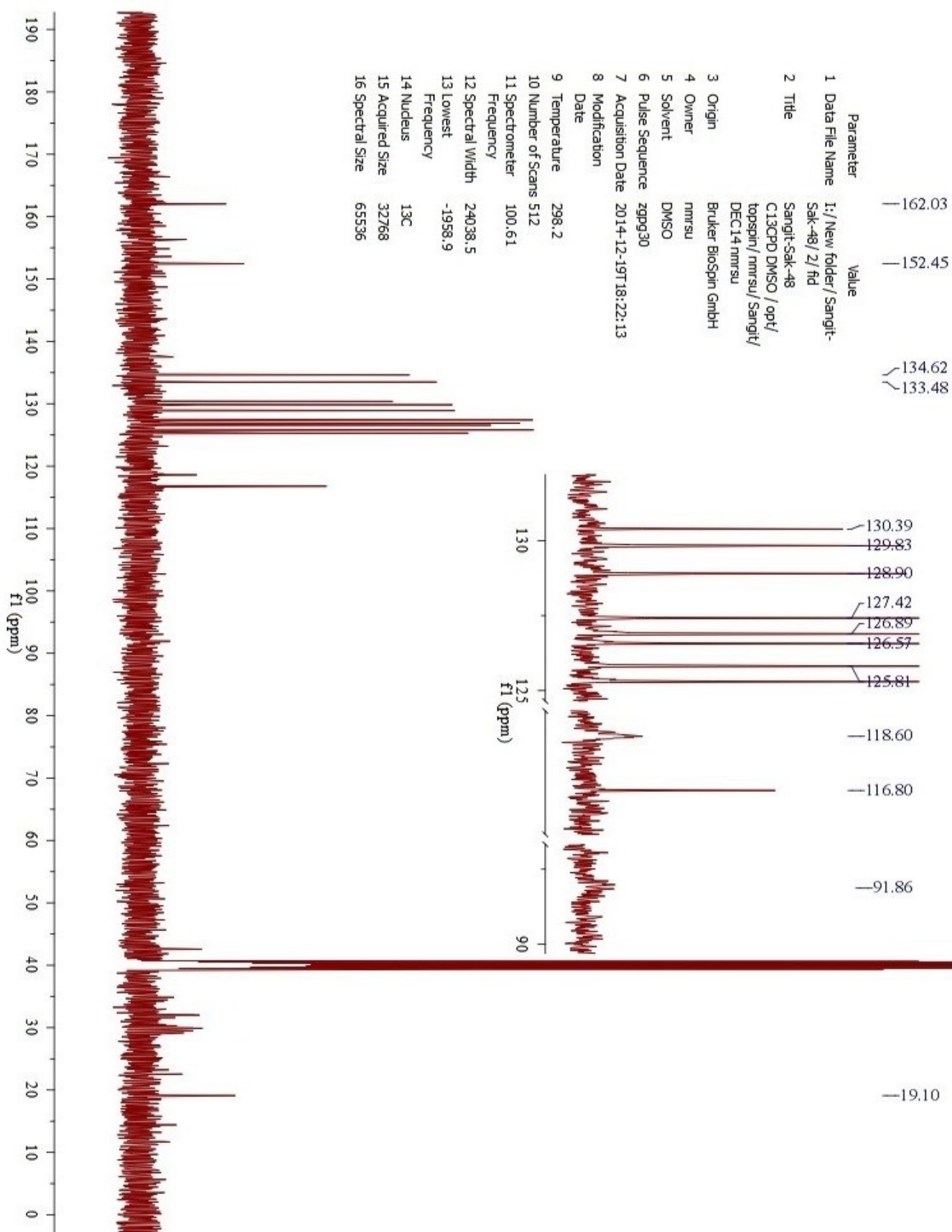


Figure S142 Mass spectra of **46**

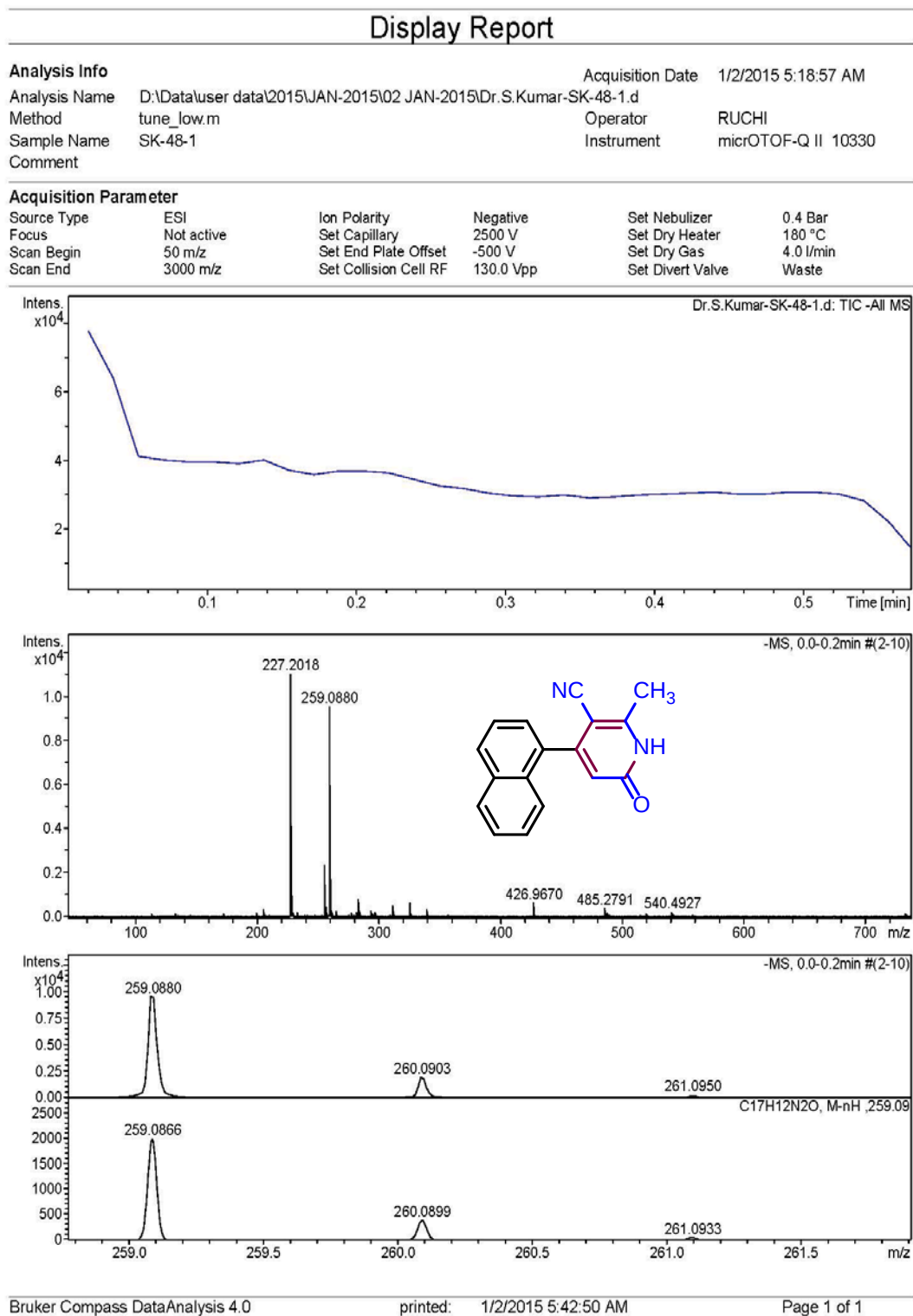


Figure S143 ^1H NMR spectra of **47**

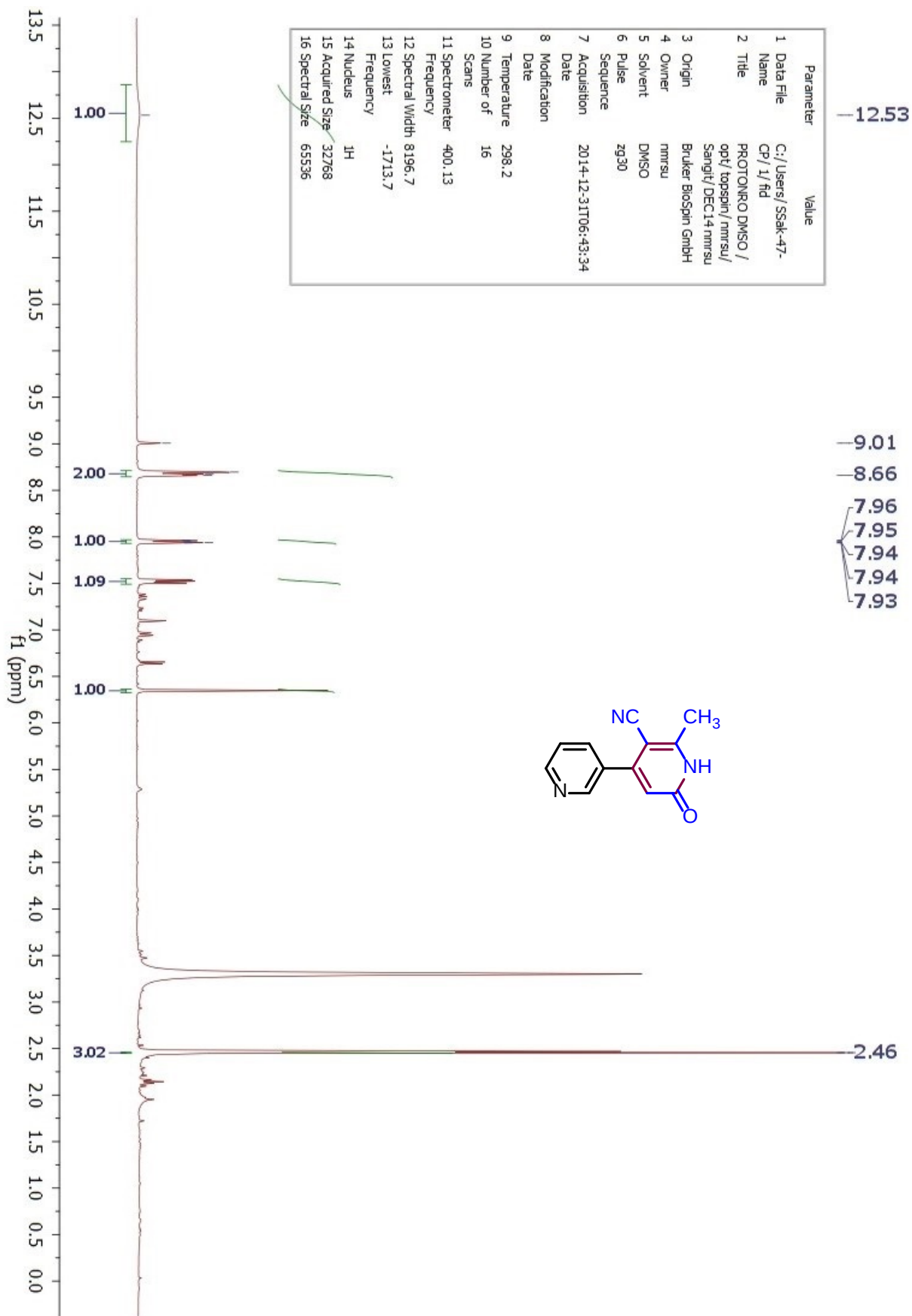


Figure S144 ^{13}C NMR spectra of 47

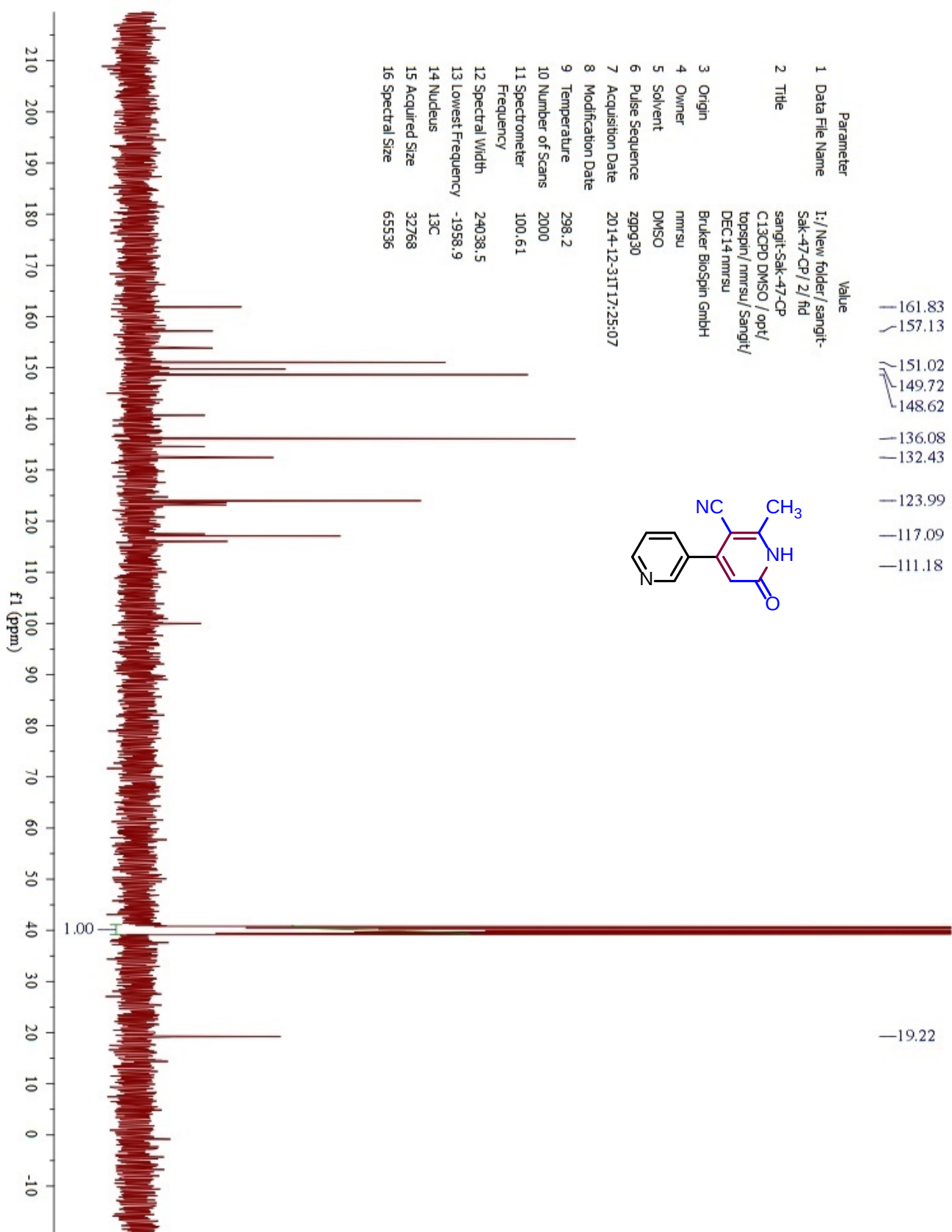


Figure S145 Mass spectra of **47**

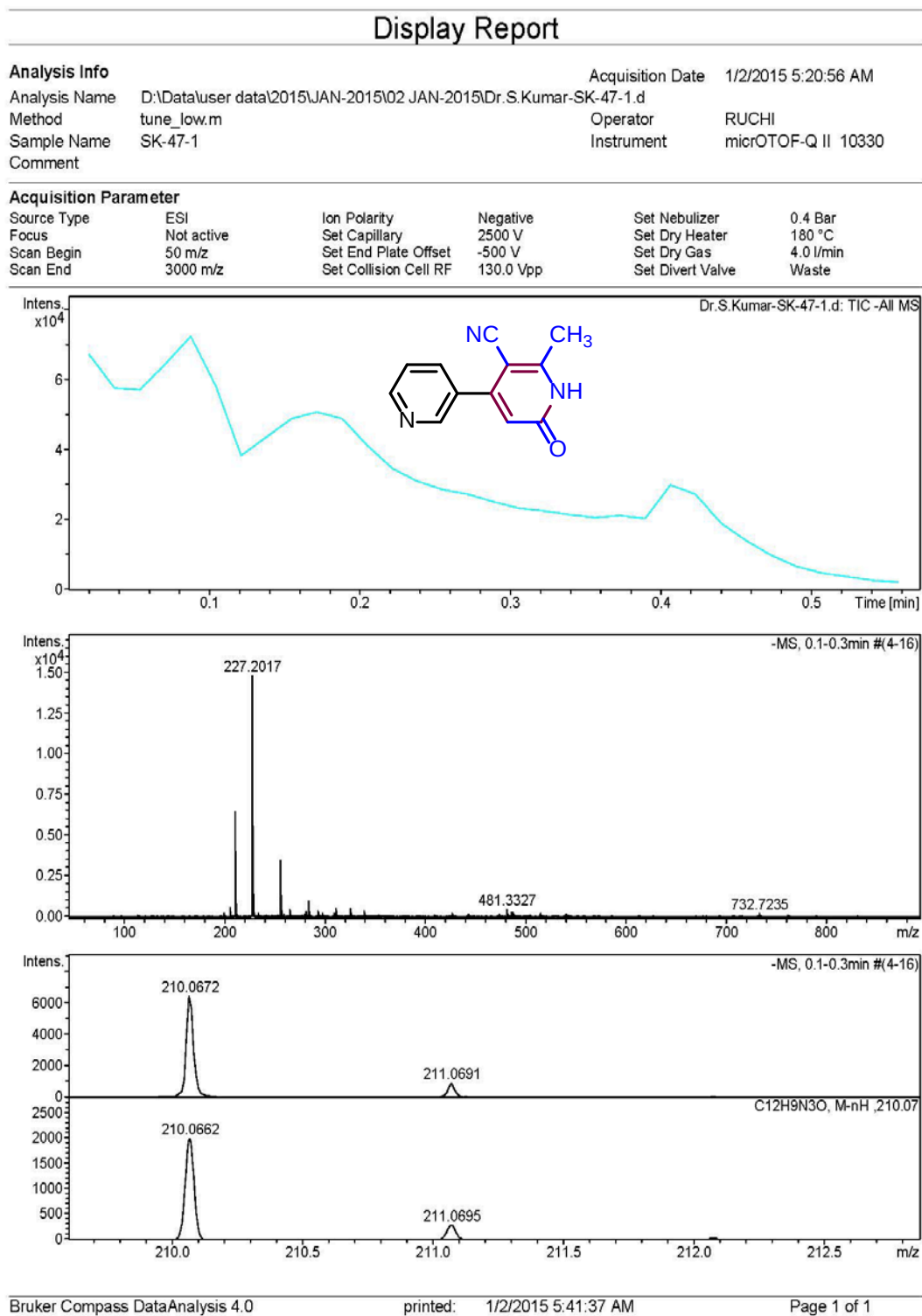


Figure S146 ^1H NMR spectra of **48**

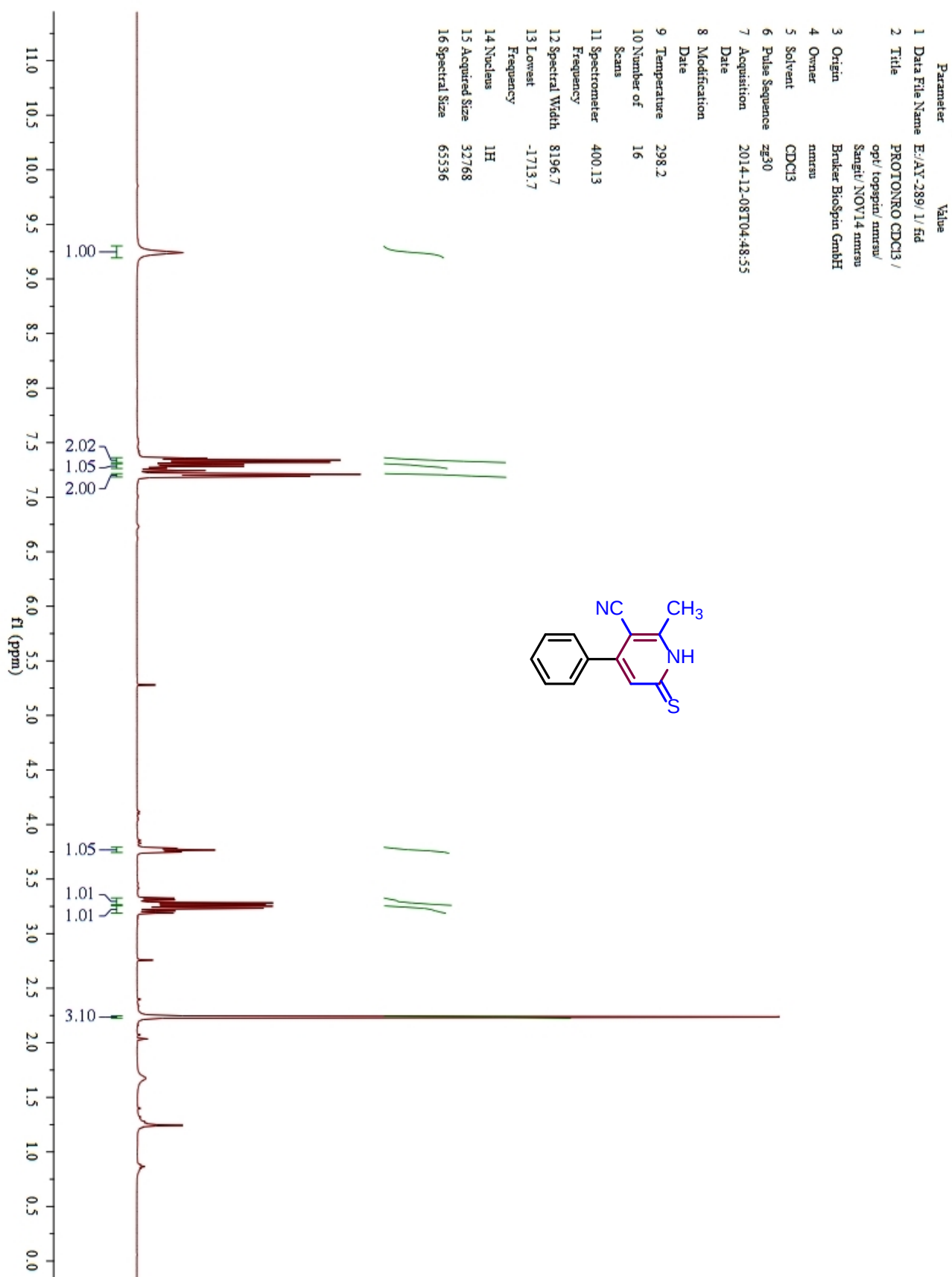


Figure S147 ^{13}C NMR spectra of **48**

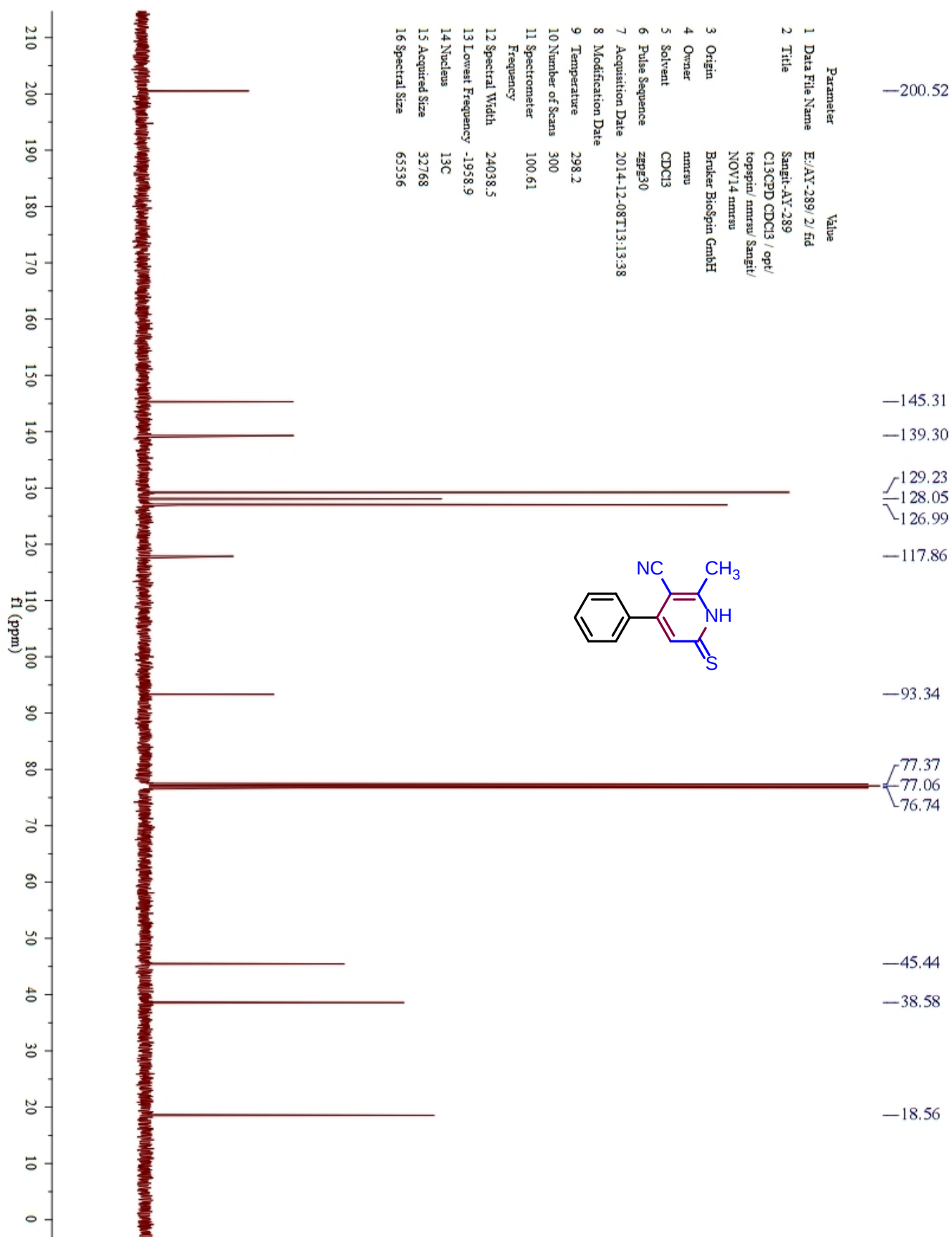


Figure S148 135-DEPT spectra of **48**

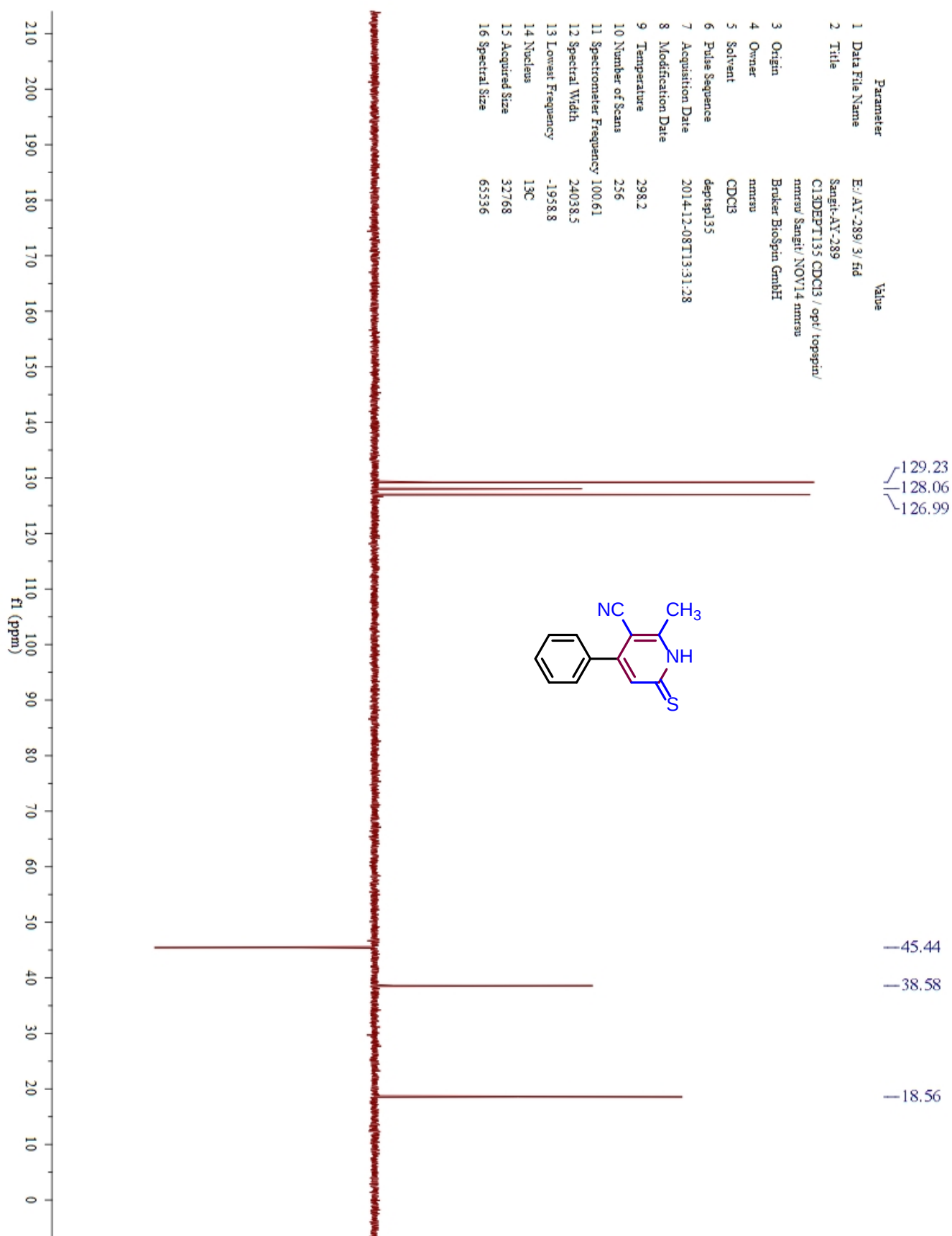


Figure S149 Mass spectra of **48**

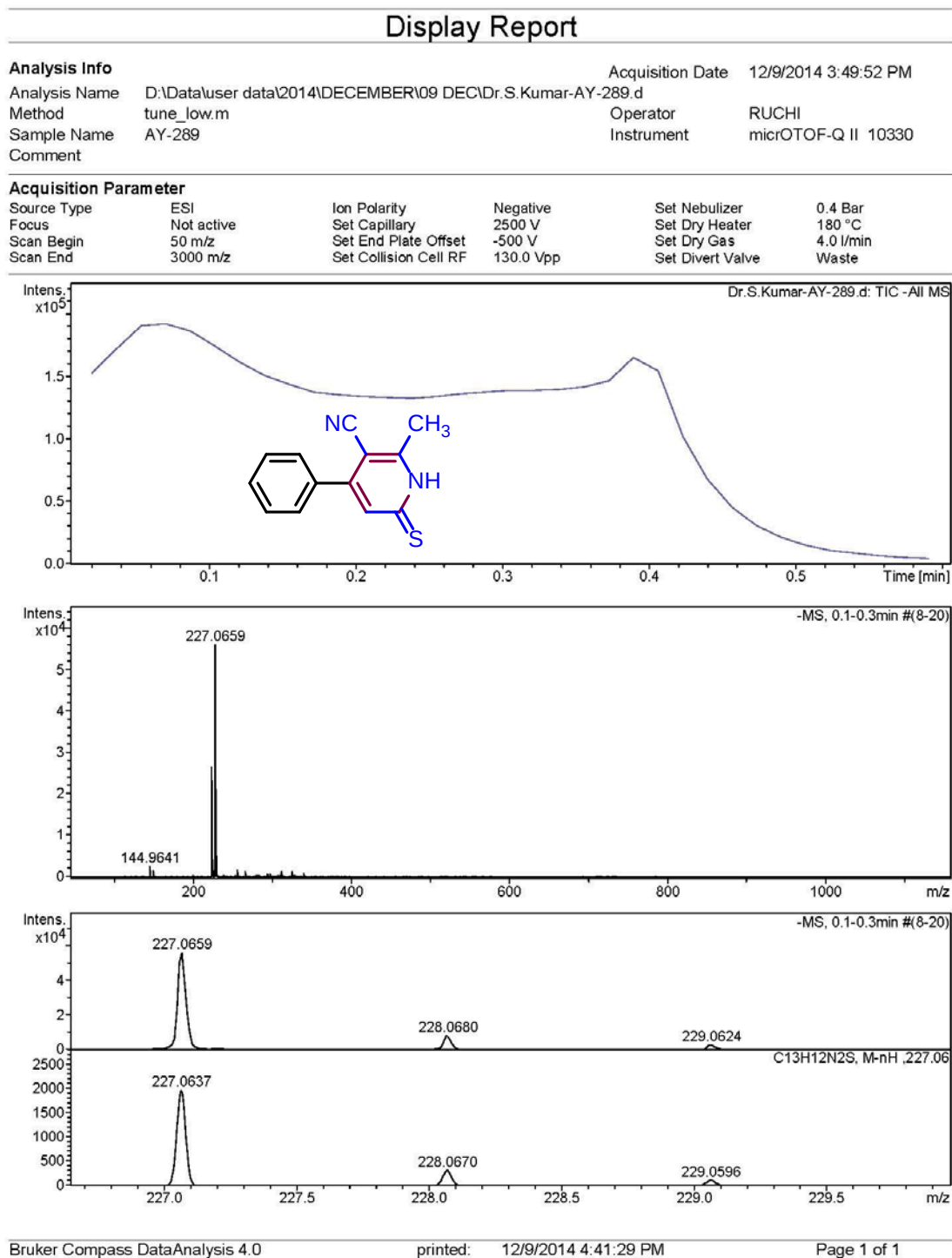


Figure S150 ^1H NMR spectra of **49**

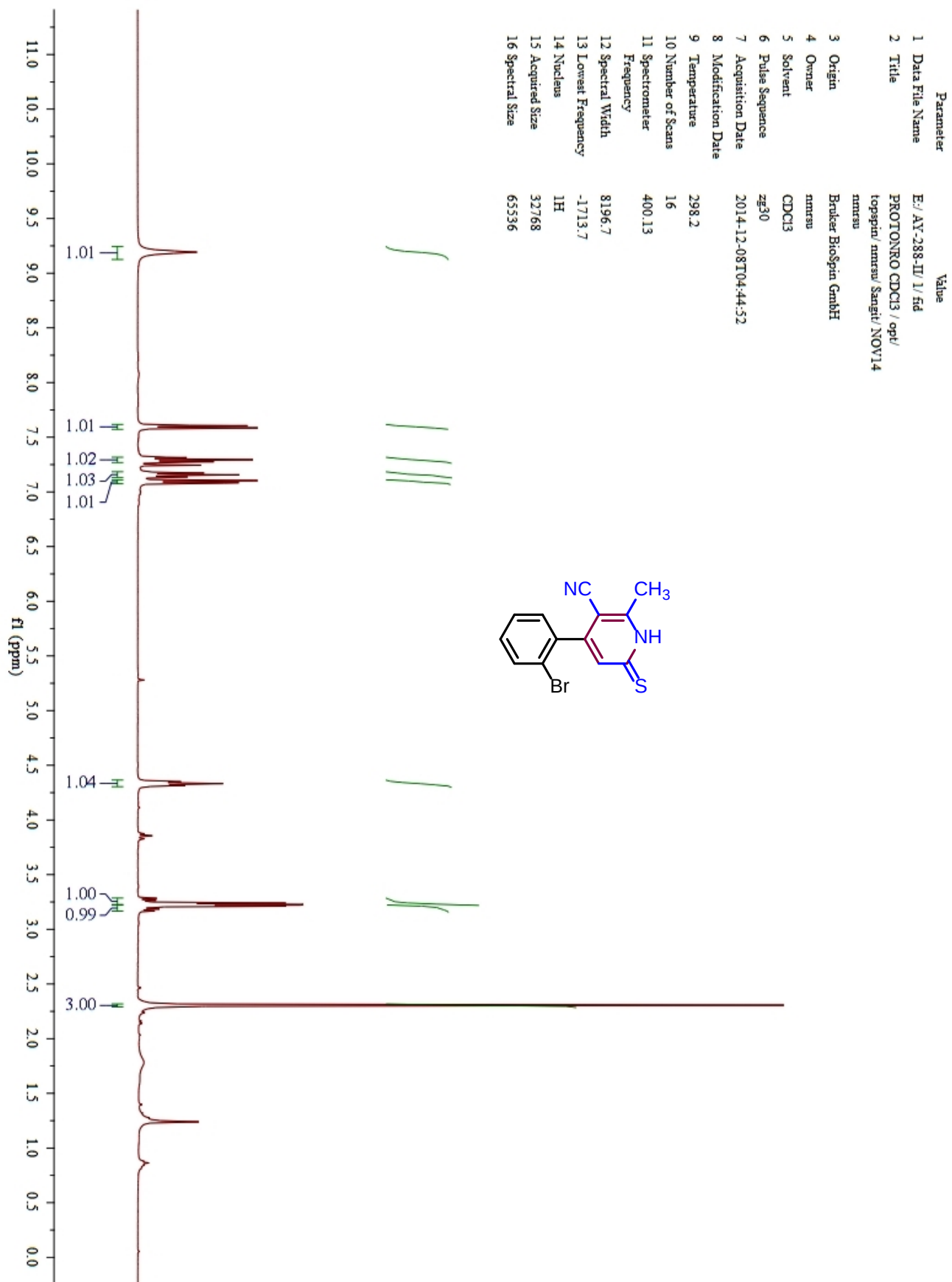


Figure S151 ^{13}C NMR spectra of **49**

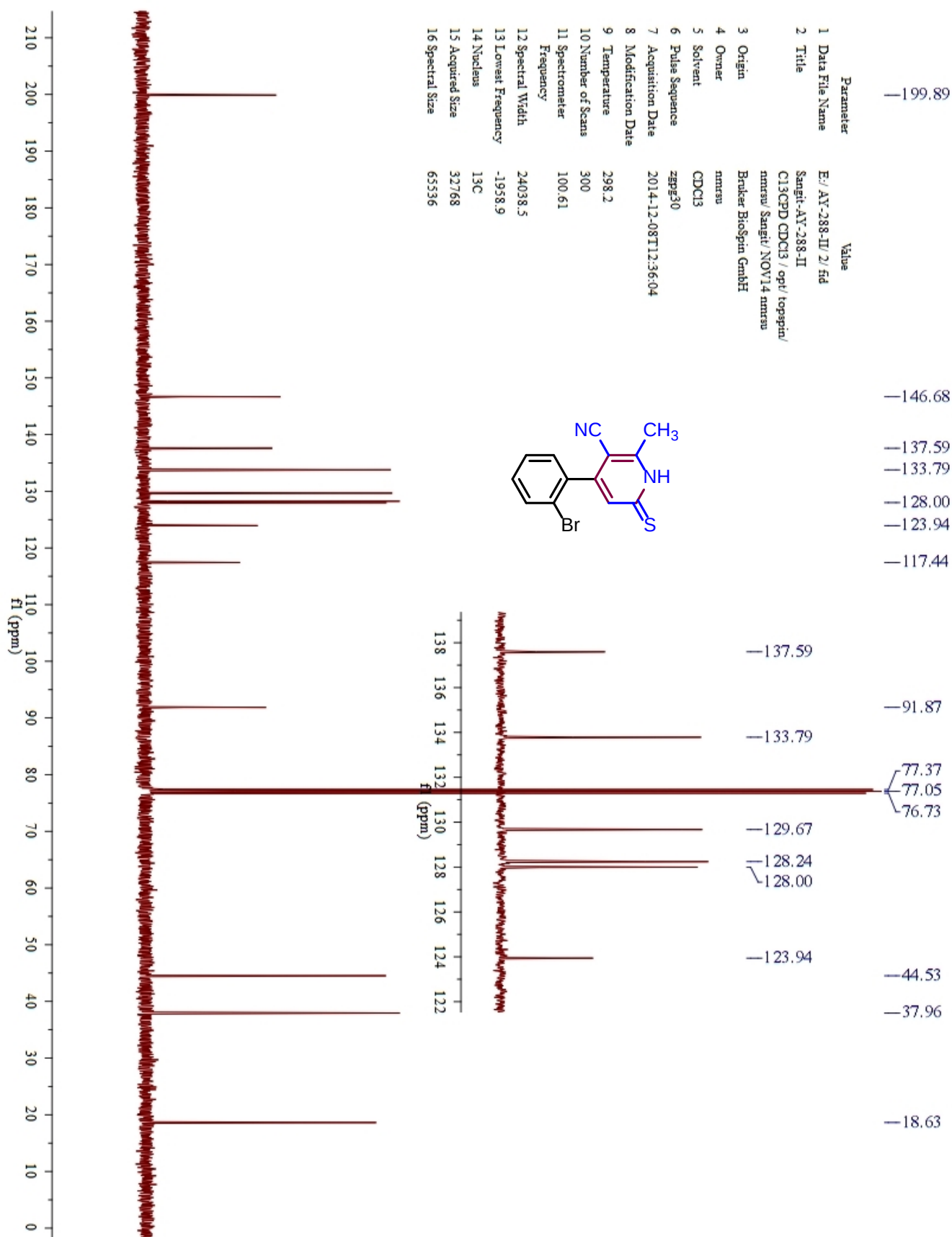


Figure S152 135-DEPT spectra of **49**

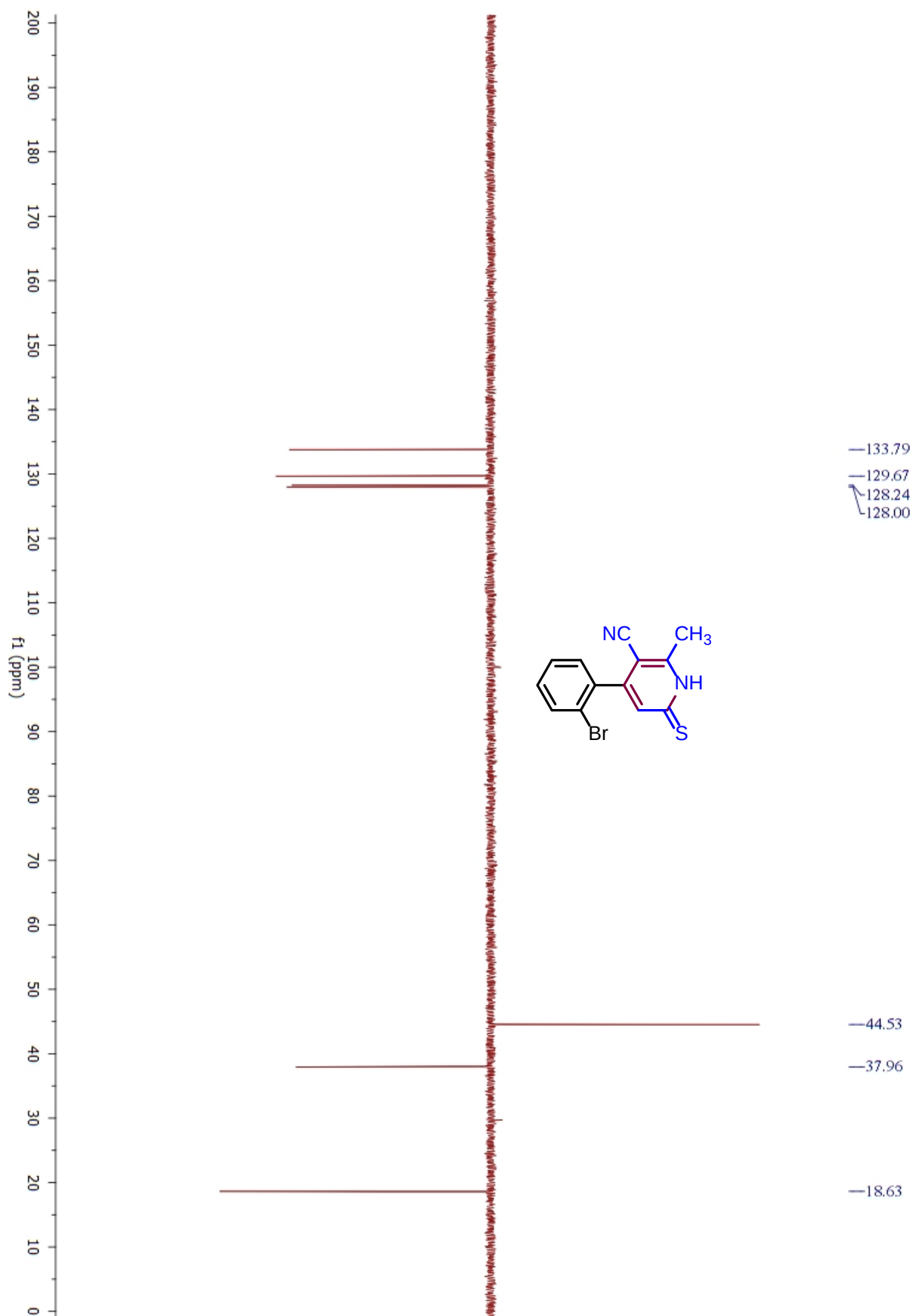


Figure S153 Mass spectra of 49

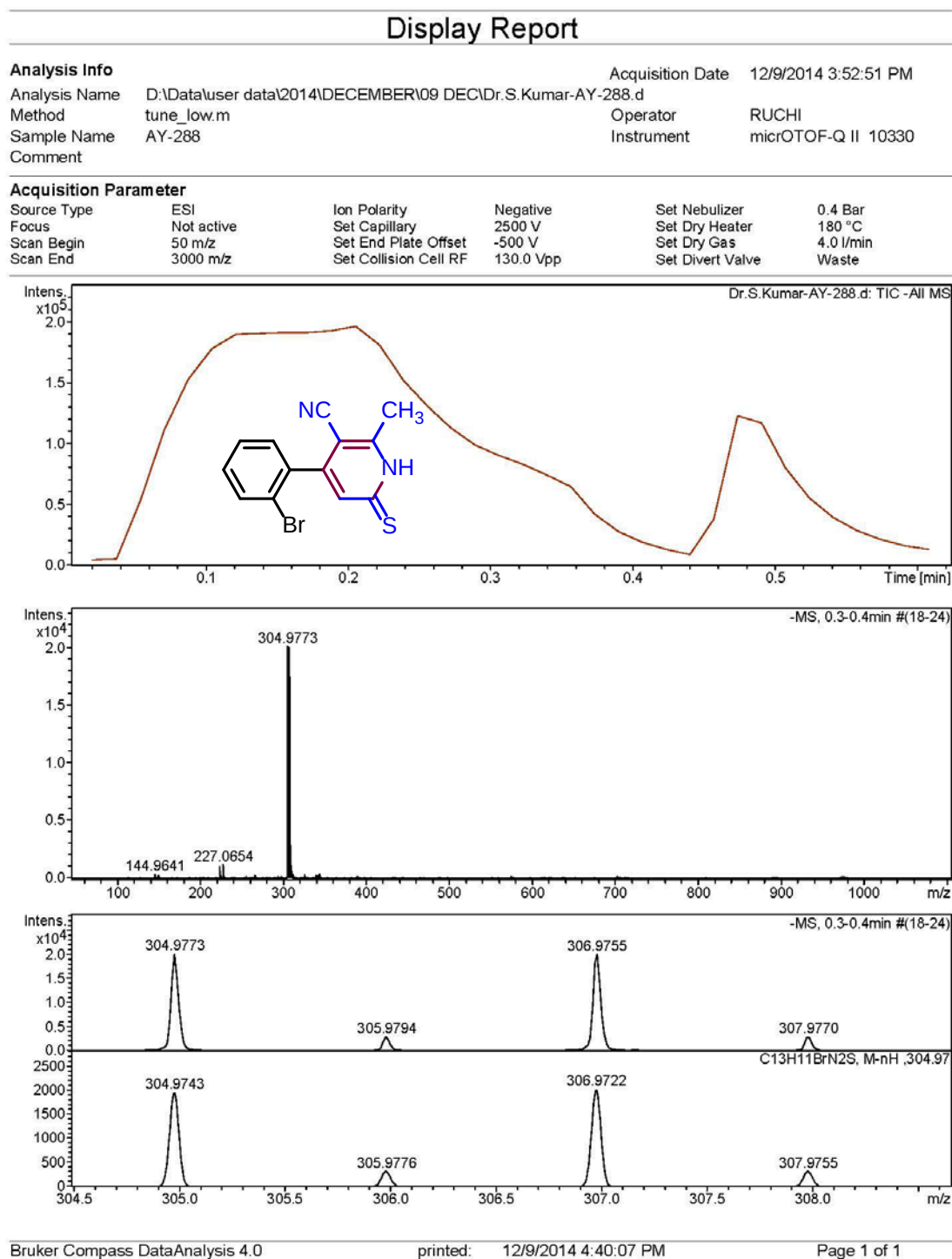


Figure S154 ^1H NMR spectra of **50**

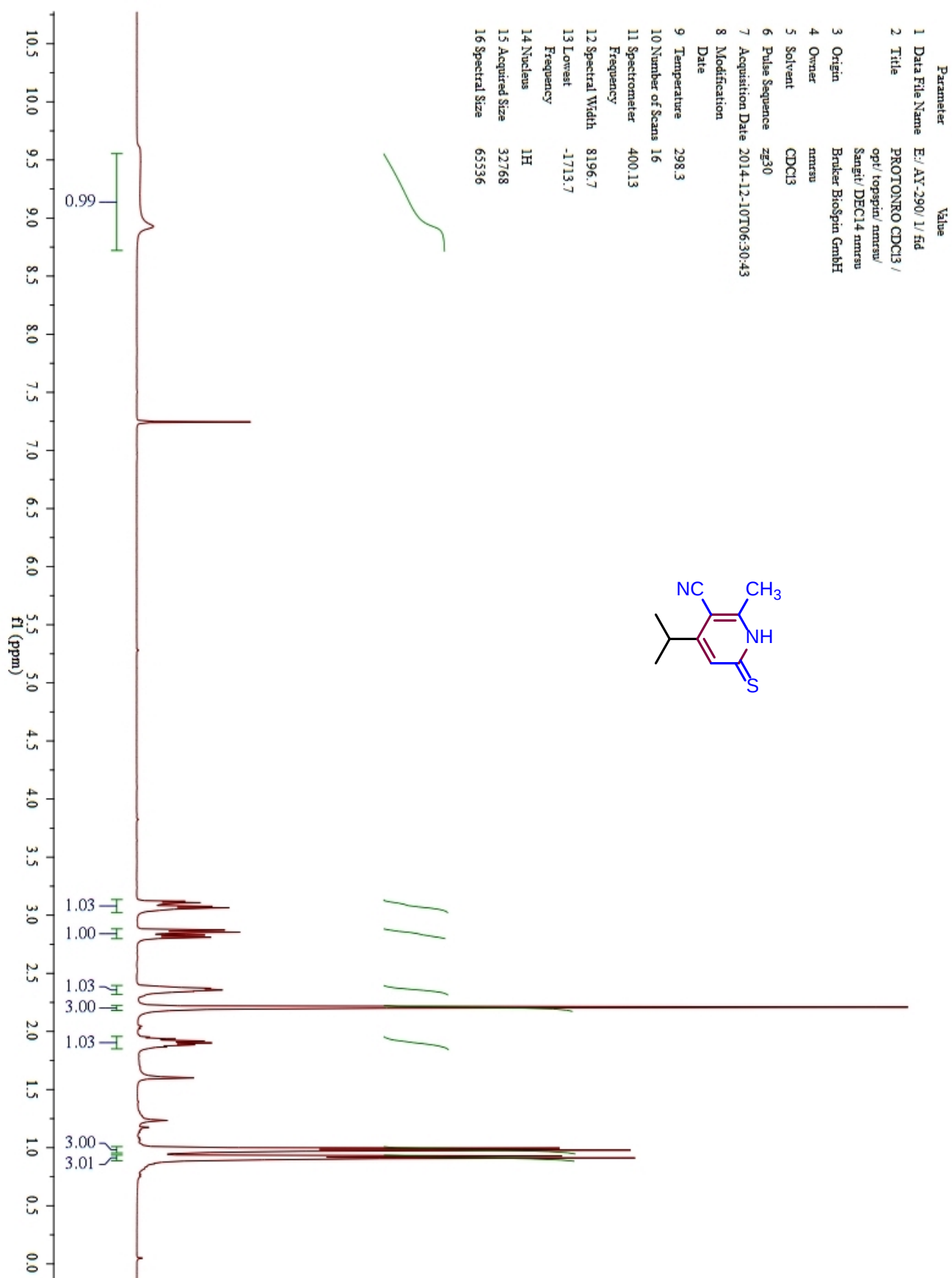
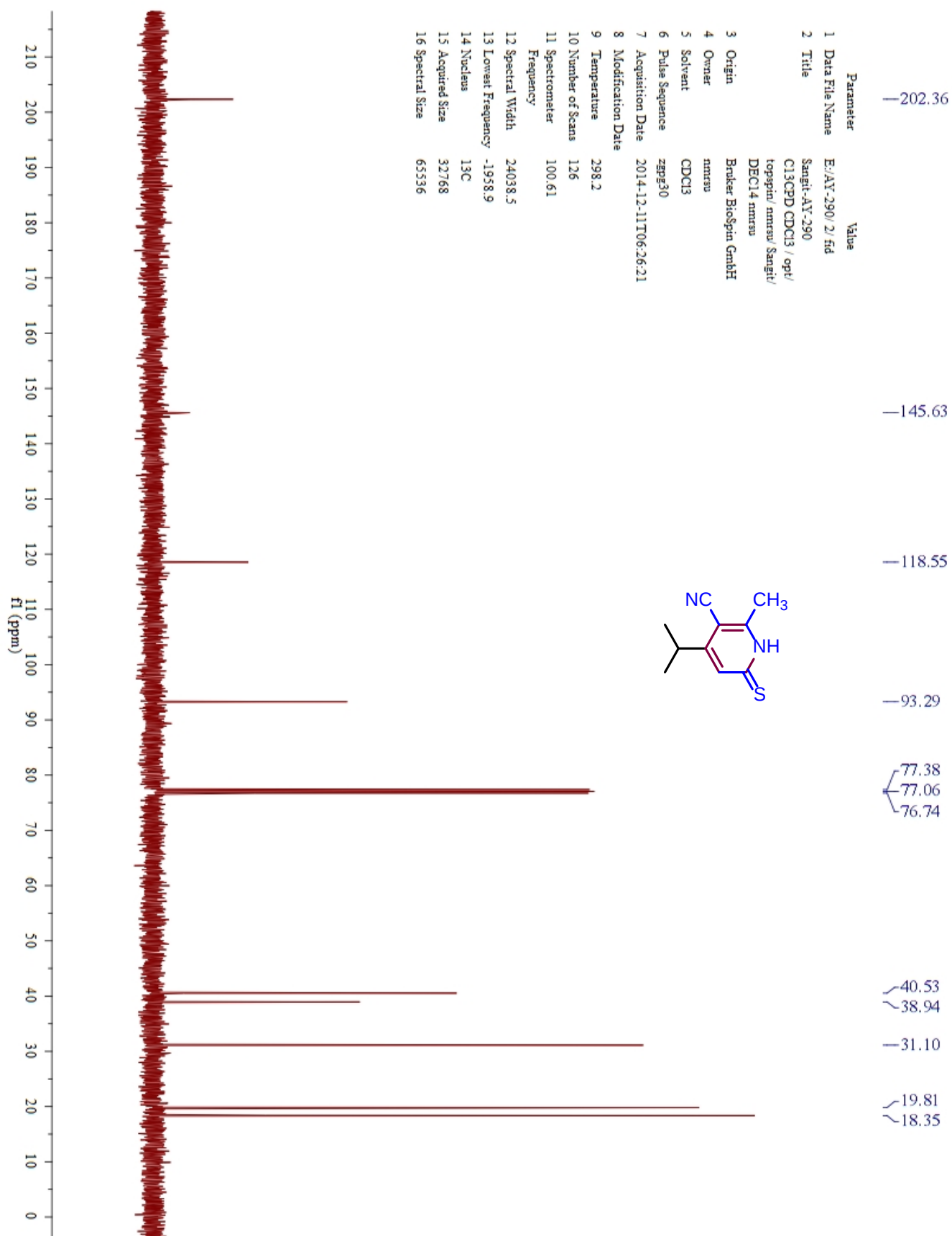


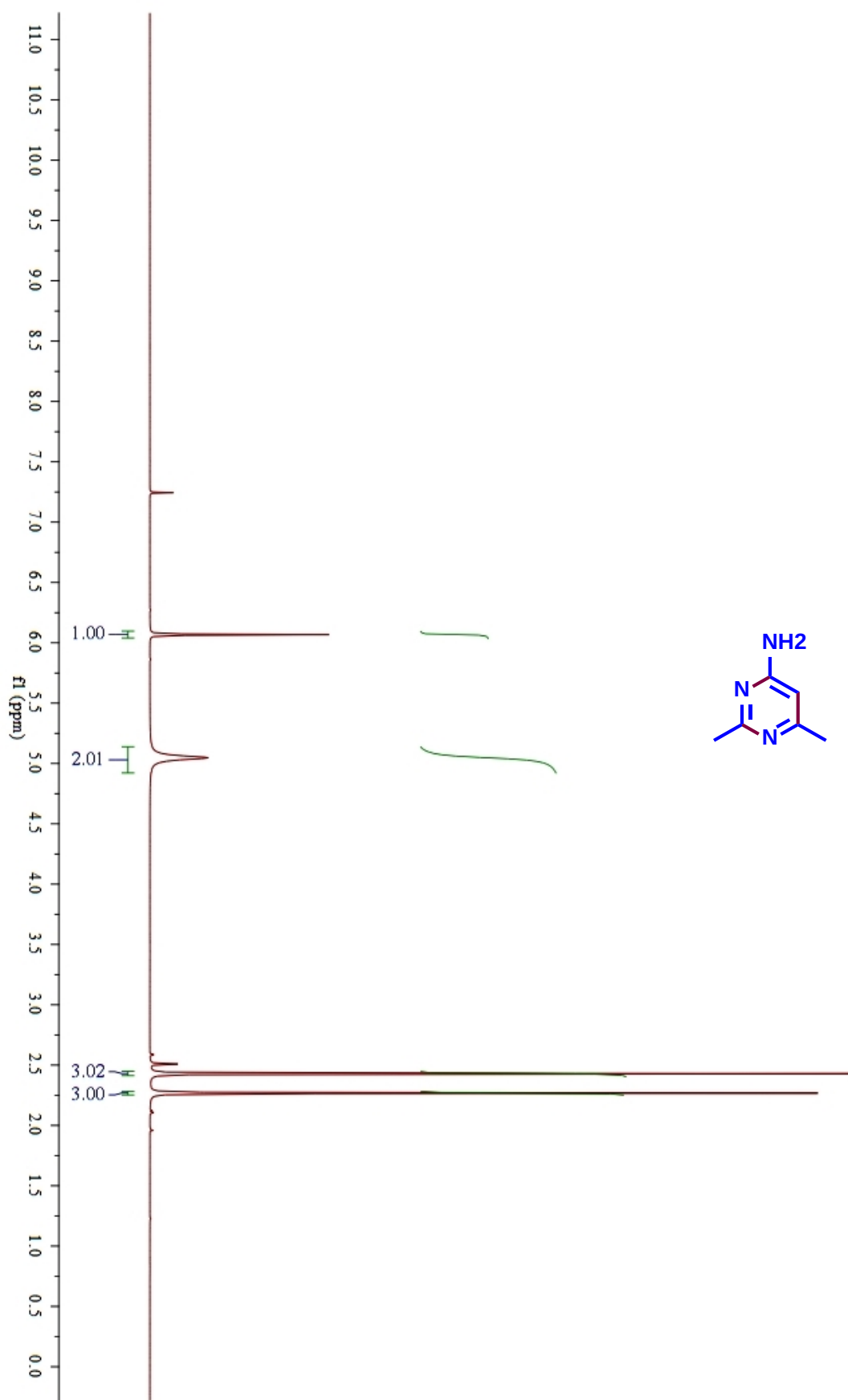
Figure S155 ^{13}C NMR spectra of **50**



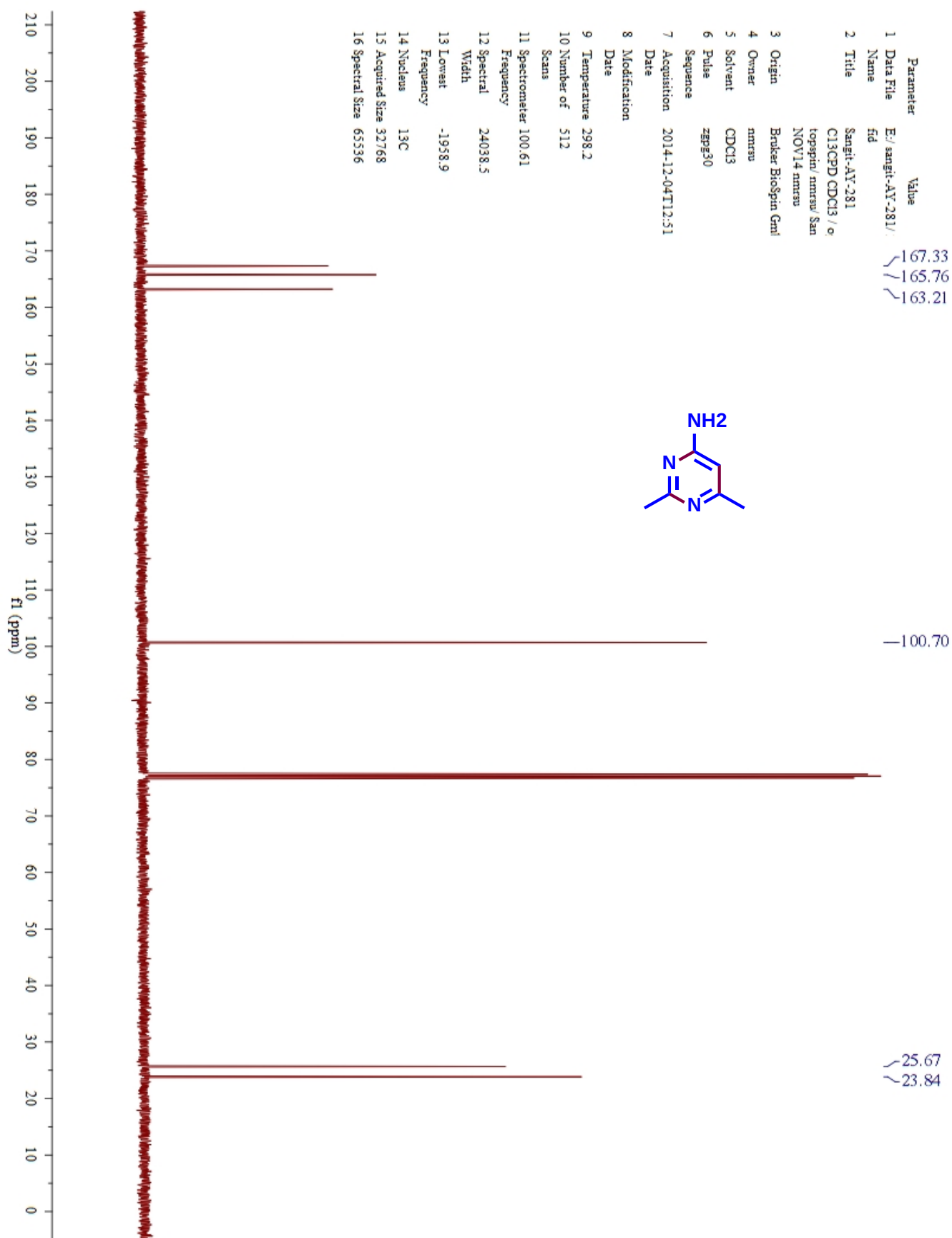
S157



¹H NMR spectra of 2,6-Dimethylpyrimidin-4-amine



¹³C NMR spectra of 2,6-Dimethylpyrimidin-4-amine



GCMS spectra of 2,6-Dimethylpyrimidin-4-amine



Sample ID: SS-52-2

Instrument: Agilent 7890A GC
with 5975C MS system

Supervisor: Dr. S. Kumar

Column: HP-5

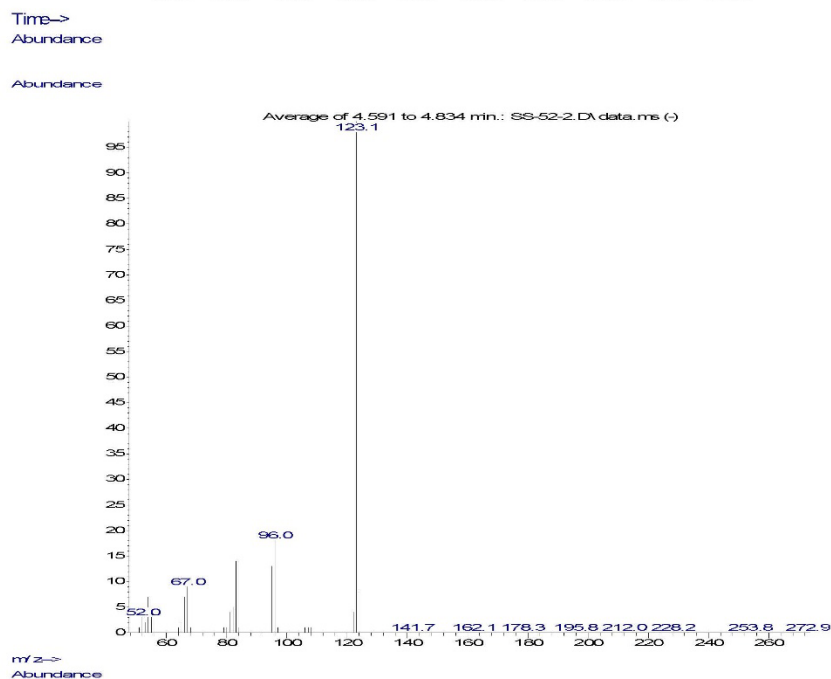
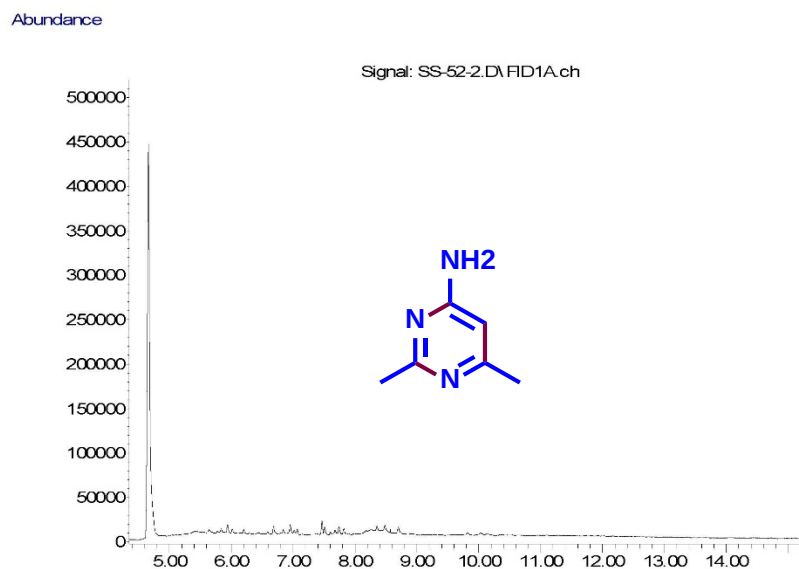
Method: General_1_HP5_80_DEG.M

Acquisition date: 26/02/14

Operator: IISERB-CIF-Mass Facility

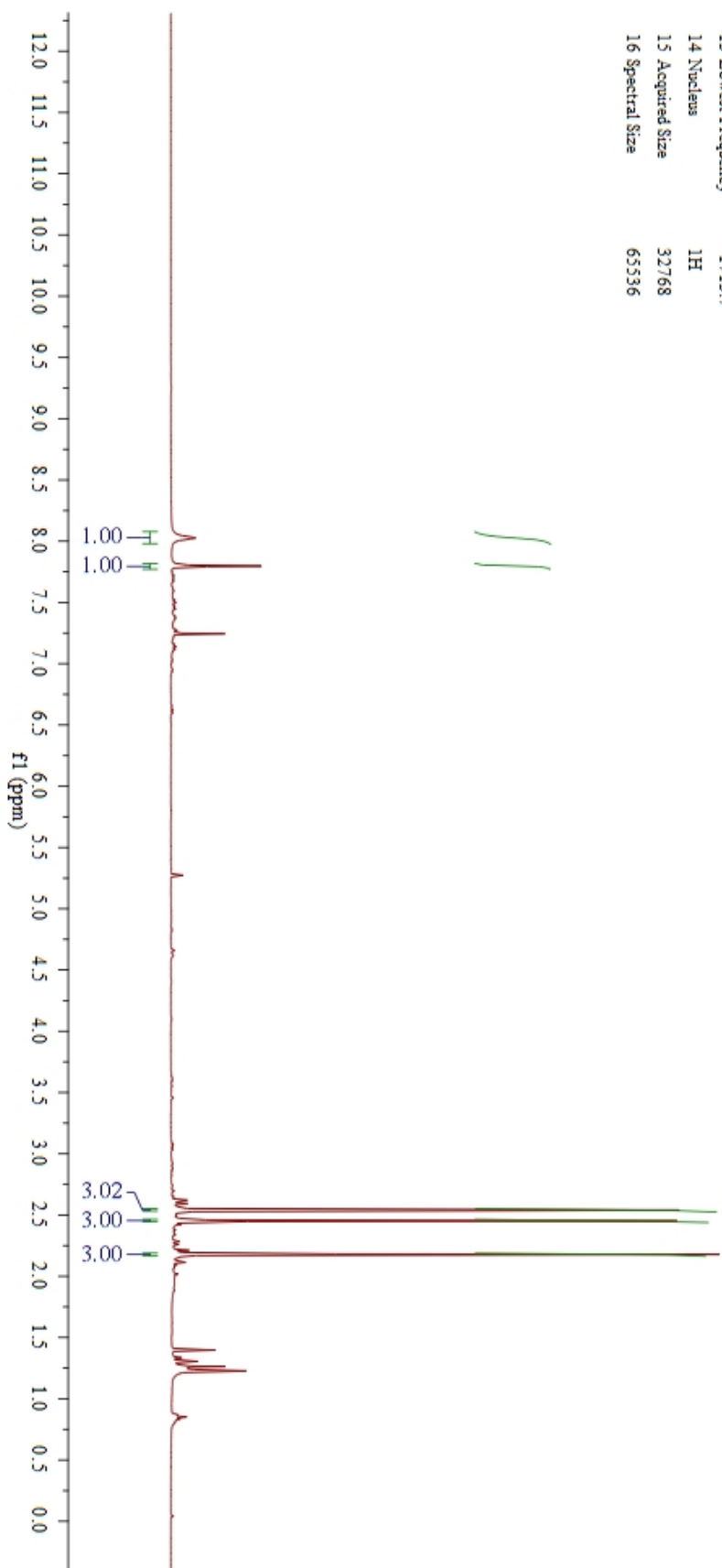
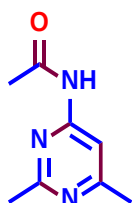
Ionization: EI (70 eV)

MSD: Single Quad.

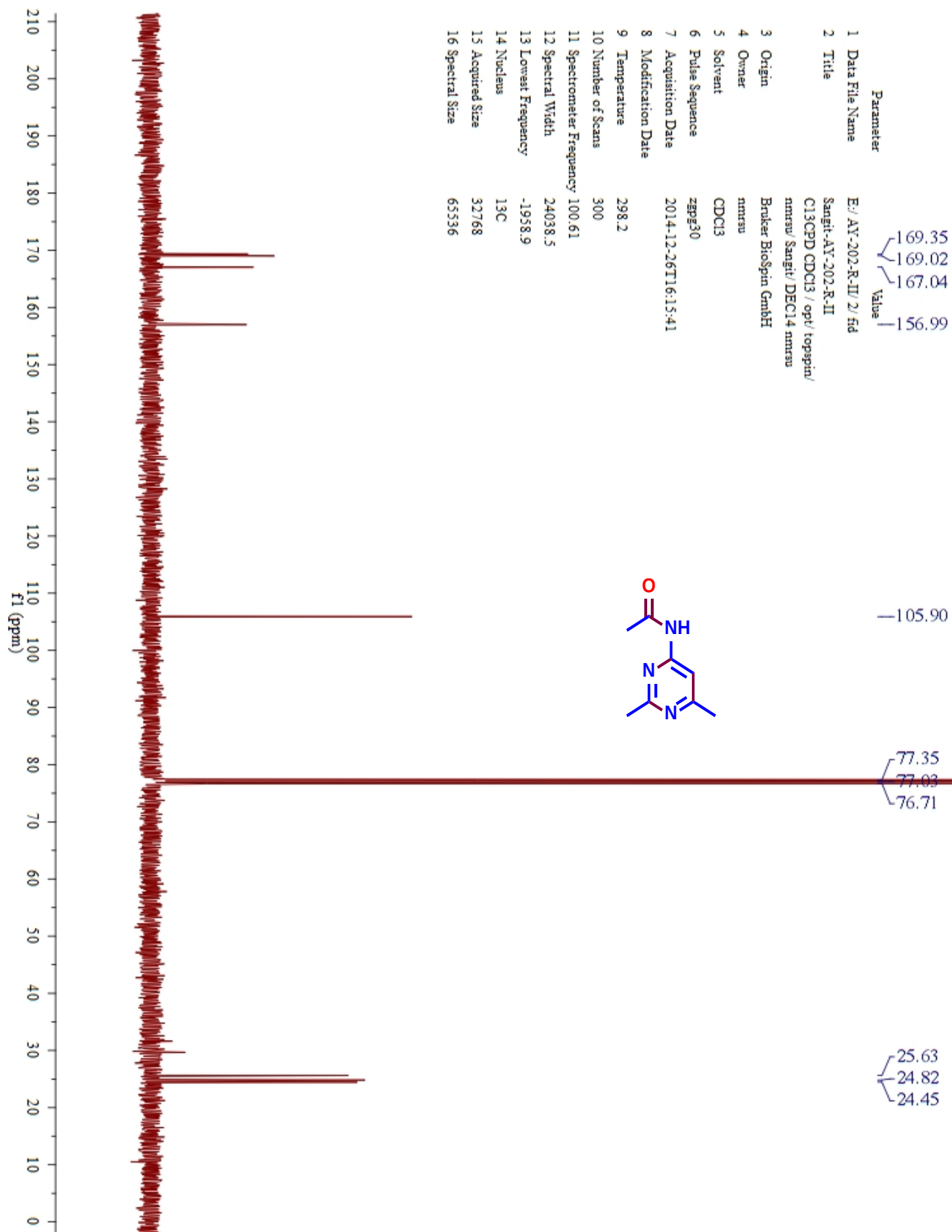


¹H NMR spectra of *N*-(2,6-Dimethylpyrimidin-4-yl)acetamide

Parameter	Value
1 Data File Name	E:/AY-202-R-II/ 1/ fid
2 Title	PROTONRO CDCl3 / opt/ topspin/ nmruv/ Sangit/ DECI4 nmruv
3 Origin	Broker Biospin GmbH
4 Owner	nmruv
5 Solvent	CDCl3
6 Pulse Sequence	zg30
7 Acquisition Date	2014-12-26T11:55:57
8 Modification Date	
9 Temperature	298.1
10 Number of Scans	16
11 Spectrometer Frequency	400.13
12 Spectral Width	8196.7
13 Lowest Frequency	-1713.7
14 Nucleus	¹ H
15 Acquired Size	32768
16 Spectral Size	65536



¹³C NMR spectra of *N*-(2,6-Dimethylpyrimidin-4-yl)acetamide



LRMS spectra of *N*-(2,6-Dimethylpyrimidin-4-yl)acetamide

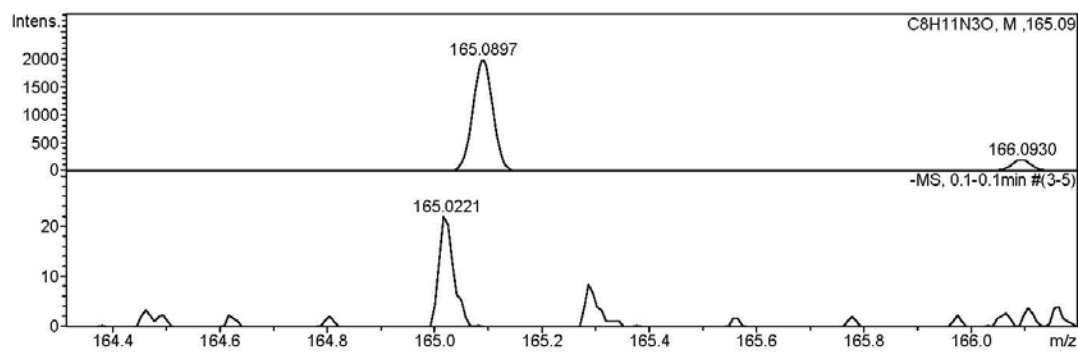
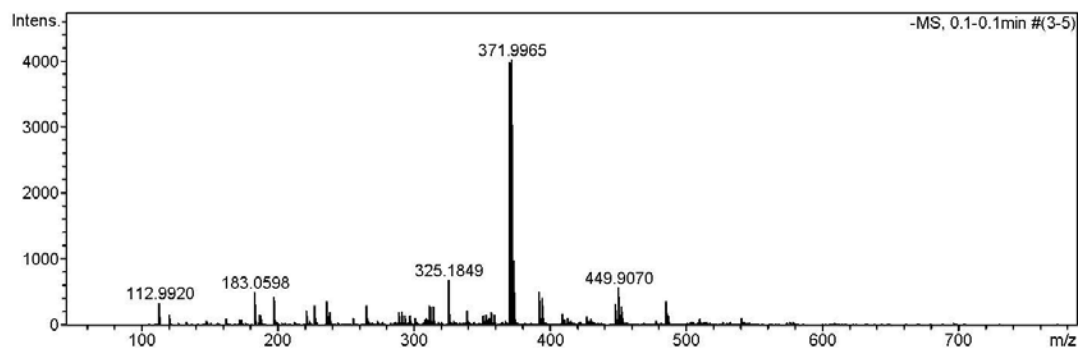
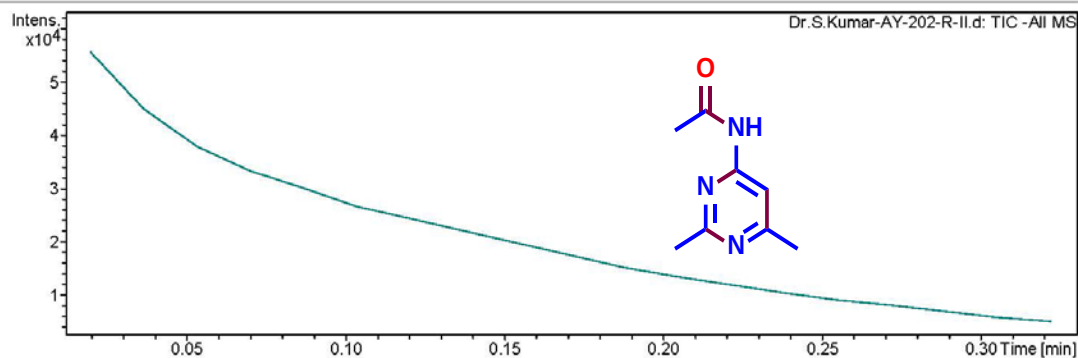
Display Report

Analysis Info

Analysis Name	D:\Data\user data\2015\JAN-2015\02 JAN-2015\Dr.S.Kumar-AY-202-R-II.d	Acquisition Date	1/2/2015 4:58:53 AM
Method	tune_low.m	Operator	RUCHI
Sample Name	AY-202-R-II	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Mass spectra for optimization of reaction with benzaldehyde, KOH in acetonitrile (see optimization table)



Sample ID: AV-381

Supervisor: Dr. S. Kumar

Acquisition date: 27/03/15

Instrument: Agilent 7890A GC
with 5975C MS system

Column: HP-5

Operator: IISERB-CIF-Mass Facility

Method: General_1_HP5_80_DEG.M

Ionization: EI (70 eV)

MSD: Single Quad

