

Synthesis of 2-triazolyl-imidazo[1,2-*a*]pyridine through one-pot three-component reaction using nano copper oxide assisted click-catalyst

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Title page	1
Experimental section and Spectra data of Compounds 4 and 5	2-3
Crystal Data and Structure Refinement for Compound 4f and 8c	4
Copies of ¹ H NMR, ¹³ C NMR and HRMS spectra of all Compounds	5-94

I. General Information and Methods.

¹H and ¹³C NMR spectra were recorded on 400 MHz, 600 MHz and 100 MHz, 150 MHz spectrometer TMS as internal reference; chemical shifts (δ scale) are reported in parts per million (ppm). ¹H NMR Spectra are reported in the order: multiplicity, coupling constant (J value) in hertz (Hz) and no of protons; signals were characterized as s (singlet), d (doublet), t (triplet), q (quatret), m (multiplet) and bs (broad). IR spectra were recorded in KBr or neat. HRMS spectra were recorded using ESI (TOF) mode. The X-ray crystal structures were determined with a diffractometer. Complete crystallographic data of **4f** (CCDC no. 979591) and **8c** (CCDC no. 1019691) for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, Copies of this information may be obtained free of charge from the Director, Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44-1223-336033, e-mail: deposit@ccdc.cam.ac.uk or via: www.ccdc.cam.ac.uk). The copper oxide nanoparticle (particle size <50 nm, CAS Number 1317-38-0) was purchased from Sigma-Aldrich.

Spectra data of Compounds 4 and 5

(1-benzyl-1H-1,2,3-triazol-4-yl)methanol (4a): Yield 92%, white solid, mp 78-79 °C, ¹H NMR (400 MHz, CDCl₃): δ 7.51 (s, 1H), 7.32-7.31 (m, 3H), 7.23-7.21 (m, 2H), 5.44 (s, 2H), 4.69 (s, 2H), 4.23 (br s, 1H); ¹³C NMR (150 MHz, CDCl₃): δ 148.8, 134.6, 129.1, 128.8, 128.2, 122.4, 56.0, 54.2; IR (KBr) ν_{\max} 3265, 3139, 3086, 3030, 2935, 2882, 1605, 1551, 1456, 1371, 1325, 1222, 1132, 1014 cm⁻¹.

(1-(4-methylbenzyl)-1H-1,2,3-triazol-4-yl)methanol (4b): Yield 90%, white solid, mp 91-92 °C, ¹H NMR (400 MHz, CDCl₃): δ 7.43 (s, 1H), 7.12 (br s, 4H), 5.39 (s, 2H), 4.67 (s, 2H), 3.89 (br s, 1H), 2.29 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 148.3, 138.5, 131.5, 129.6, 128.1, 122.2, 55.7, 53.9, 21.1; IR (KBr) ν_{\max} 3252, 3140, 3086, 3050, 2945, 2917, 1612, 1553, 1515, 1446, 1375, 1330, 1223, 1134, 1013 cm⁻¹.

(1-(4-bromobenzyl)-1H-1,2,3-triazol-4-yl)methanol (4c): Yield 86%, white solid, mp 224-225 °C, ¹H NMR (400 MHz, CDCl₃): δ 7.47 (d, J = 8.4 Hz, 2H), 7.44 (s, 1H), 7.12 (d, J = 8.4 Hz, 2H), 5.44 (s, 2H), 4.74 (s, 2H), 2.33 (br s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 148.2, 133.6, 131.1, 129.1, 121.5, 121.5, 55.0, 52.2; IR (KBr) ν_{\max} 3259, 3115, 3063, 2957, 2866, 1591, 1543, 1489, 1465, 1407, 1357, 1336, 1230, 1126, 1064, 1012 cm⁻¹.

(1-(4-fluorobenzyl)-1H-1,2,3-triazol-4-yl)methanol (4d): Yield 90%, white solid, mp 68-69 °C, ¹H NMR (400 MHz, CDCl₃): δ 7.53 (s, 1H), 7.25-7.21 (m, 2H), 7.02-6.97 (m, 2H), 5.42 (s, 2H), 4.82 (br s, 1H), 4.68 (s, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 163.8, 161.3, 148.3, 130.6, 130.5, 129.9, 129.8, 122.1, 115.9, 115.7, 55.6, 53.1; IR (KBr) ν_{\max} 3291, 3144, 3000, 2949, 2877, 1606, 1546, 1511, 1460, 1420, 1366, 1229, 1157, 1127, 1006 cm⁻¹.

(1-allyl-1H-1,2,3-triazol-4-yl)methanol (4e): Yield 80%, white solid, mp 81-82 °C, ¹H NMR (400 MHz, CDCl₃): δ 7.59 (s, 1H), 6.04-5.95 (m, 1H), 5.35 (d, J = 10.4 Hz, 1H), 5.30 (d, J = 17.2 Hz, 1H), 4.96 (d, J = 6.4 Hz, 2H), 4.75 (s, 2H), 3.48 (br s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 148.2, 131.2, 122.1, 120.1, 55.8, 52.6; IR (KBr) ν_{\max} 3266, 3147, 3092, 2933, 2877, 1646, 1553, 1457, 1435, 1420, 1336, 1225, 1141, 1056, 1011 cm⁻¹.

Ethyl 2-(4-(hydroxymethyl)-1H-1,2,3-triazol-1-yl)acetate (4f): Yield 78%, white solid, mp 68-69 °C, ¹H NMR (400 MHz, CDCl₃): δ 7.65 (s, 1H), 5.09 (s, 2H), 4.64 (s, 2H), 4.17 (q, J = 7.2 Hz, 2H), 1.22 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 166.7, 148.3, 123.9, 62.4, 55.8, 50.9, 14.1; IR (KBr) ν_{\max} 3260, 3133, 3088, 2993, 2936, 2865, 1744, 1556, 1455, 1421, 1400, 1379, 1345, 1237, 1145, 1063, 1021 cm⁻¹.

1-benzyl-1H-1,2,3-triazole-4-carbaldehyde (5a): Yield 82%, white solid, mp 90-91 °C, ¹H NMR (400 MHz, CDCl₃): δ 10.12 (s, 1H), 7.98 (s, 1H), 7.41-7.39 (m, 3H), 7.31-7.29 (m, 2H), 5.58 (s, 2H); ¹³C NMR (150 MHz, CDCl₃): δ 185.2, 148.2, 133.5, 129.6, 129.5, 128.6, 125.3, 54.8; IR (KBr) ν_{\max} 3127, 3049, 2995, 2925, 2854, 2775, 1694, 1586, 1534, 1495, 1456, 1358, 1237, 1166, 1048 cm⁻¹; HRMS (ESI) Calcd For C₁₀H₁₀N₃O 188.0817 (M + H⁺); Found 188.0819.

1-(4-methylbenzyl)-1H-1,2,3-triazole-4-carbaldehyde (5b): Yield 78%, white solid, mp 77-78 °C, ¹H NMR (600 MHz, CDCl₃): δ 10.11 (s, 1H), 7.96 (s, 1H), 7.20 (s, 4H), 5.54 (s, 2H), 2.36 (s, 3H); ¹³C NMR (150 MHz, CDCl₃): δ 185.3, 148.1,

139.6, 130.5, 130.2, 128.6, 125.2, 54.6, 21.4; IR (KBr) ν_{\max} 3132, 3041, 2988, 2924, 2854, 2768, 1699, 1610, 1532, 1517, 1463, 1437, 1355, 1243, 1160, 1044 cm^{-1} ; HRMS (ESI) Calcd For $\text{C}_{11}\text{H}_{12}\text{N}_3\text{O}$ 202.0975 ($\text{M} + \text{H}^+$); Found 202.0975.

1-(4-bromobenzyl)-1H-1,2,3-triazole-4-carbaldehyde (5c): Yield 76%, white solid, mp 107-108 °C, ^1H NMR (600 MHz, CD_2Cl_2): δ 10.8 (s, 1H), 8.06 (s, 1H), 7.55 (d, $J = 8.4$ Hz, 2H), 7.20 (d, $J = 8.4$ Hz, 2H), 5.55 (s, 2H); ^{13}C NMR (150 MHz, CD_2Cl_2): δ 184.7, 148.1, 133.1, 133.0, 132.4, 130.1, 125.7, 125.6, 123.2, 53.2; IR (KBr) ν_{\max} 3120, 3090, 3039, 2925, 2843, 2767, 1696, 1593, 1532, 1490, 1438, 1356, 1241, 1166, 1046 cm^{-1} ; HRMS (ESI) Calcd For $\text{C}_{10}\text{H}_9\text{BrN}_3\text{O}$ 265.9924 ($\text{M} + \text{H}^+$); Found 265.9909.

1-(4-fluorobenzyl)-1H-1,2,3-triazole-4-carbaldehyde (5d): Yield 78%, white solid, mp 73-74 °C, ^1H NMR (400 MHz, CDCl_3): δ 10.08 (s, 1H), 7.97 (s, 1H), 7.27-7.29 (m, 2H), 7.03-7.08 (m, 2H), 5.53 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 185.1, 164.4, 161.9, 148.1, 130.5, 130.4, 129.5, 129.4, 125.3, 116.6, 116.4, 53.9; IR (KBr) ν_{\max} 3139, 3120, 3053, 2956, 2925, 2870, 1699, 1603, 1536, 1512, 1465, 1432, 1345, 1236, 1167, 1049 cm^{-1} ; HRMS (ESI) Calcd For $\text{C}_{10}\text{H}_9\text{FN}_3\text{O}$ 206.0724 ($\text{M} + \text{H}^+$); Found 206.0722.

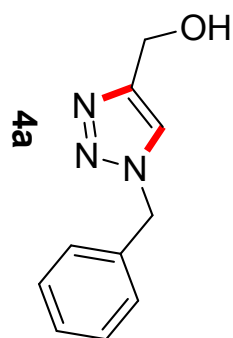
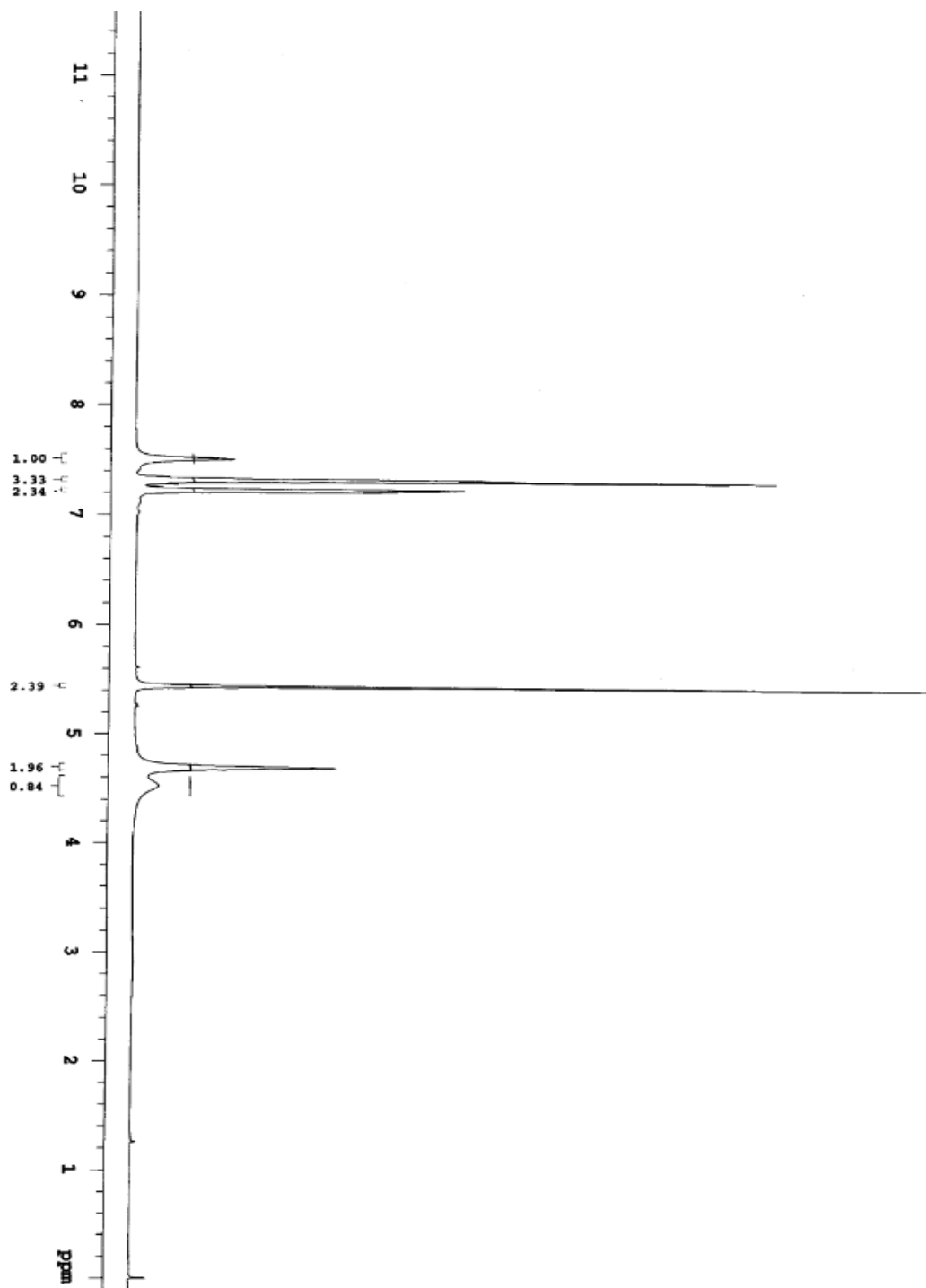
1-allyl-1H-1,2,3-triazole-4-carbaldehyde (5e): Yield 72%, white solid, mp 71-72 °C, ^1H NMR (600 MHz, CDCl_3): δ 10.08 (s, 1H), 8.13 (s, 1H), 6.01 (s, 1H), 5.39-5.32 (m, 2H), 5.03 (s, 2H); ^{13}C NMR (150 MHz, CDCl_3): δ 185.0, 147.9, 130.2, 125.4, 121.4, 53.1; IR (KBr) ν_{\max} 3132, 3029, 2924, 2871, 1699, 1615, 1532, 1517, 1462, 1436, 1355, 1243, 1160, 1044 cm^{-1} ; MS (ESI) Calcd For $\text{C}_6\text{H}_7\text{N}_3\text{ONa}$ 160.0481 ($\text{M} + \text{Na}^+$); Found 160.0816.

Ethyl 2-(4-formyl-1H-1,2,3-triazol-1-yl)acetate (5f): Yield 68%, white solid, mp 65-66 °C, ^1H NMR (400 MHz, CDCl_3): δ 10.15 (s, 1H), 8.28 (s, 1H), 5.24 (s, 2H), 4.29 (br s, 2H), 1.31 (br s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 185.0, 165.6, 148.2, 127.0, 63.1, 51.2, 14.2; IR (KBr) ν_{\max} 3132, 3062, 2997, 2959, 2849, 2781, 1745, 1703, 1540, 1478, 1416, 1397, 1379, 1249, 1170, 1049 cm^{-1} ; HRMS (ESI) Calcd For $\text{C}_7\text{H}_{10}\text{N}_3\text{O}_3$ 184.0717 ($\text{M} + \text{H}^+$); Found 184.0715.

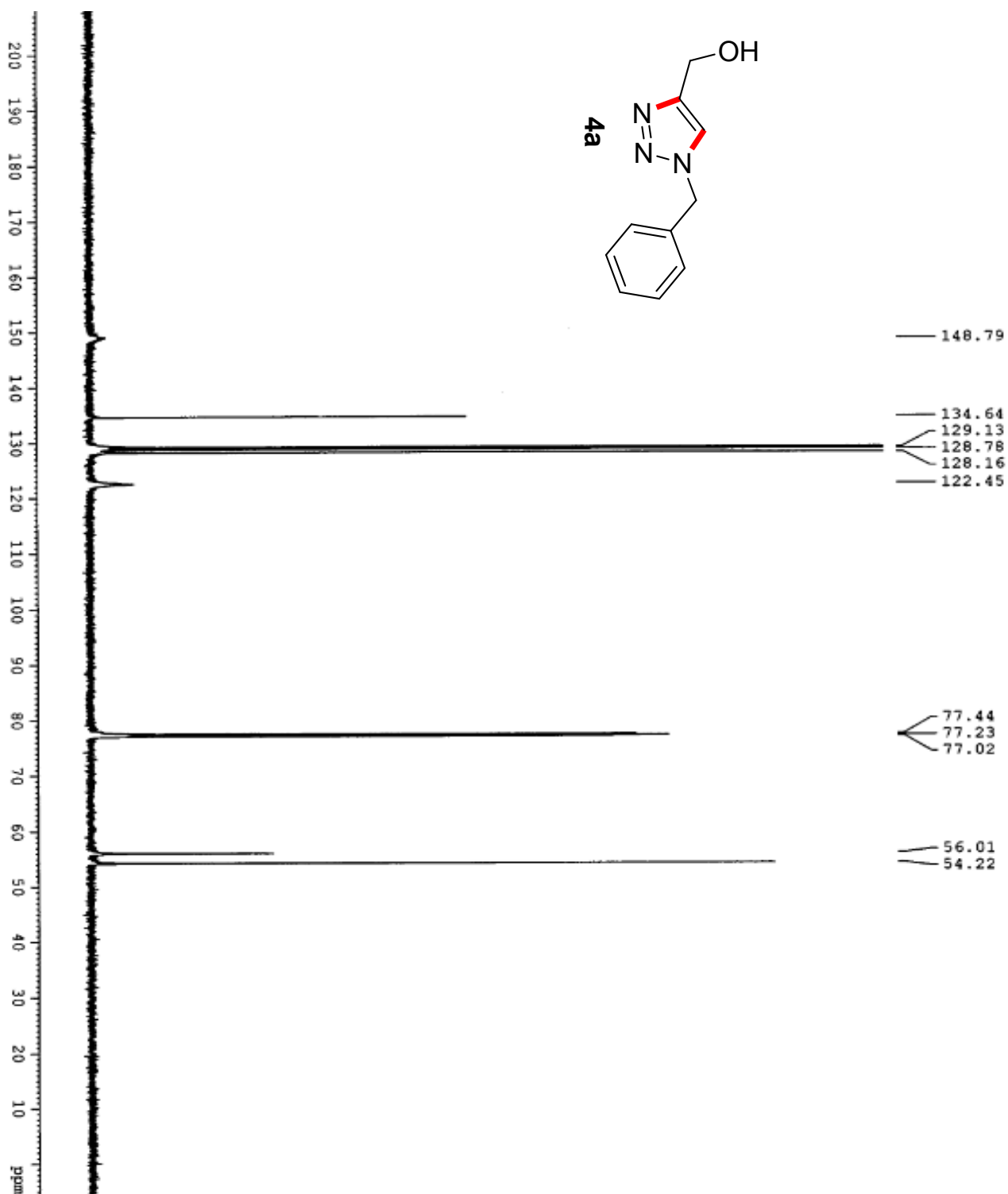
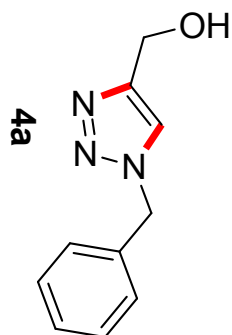
Table 6 Crystal Data and Structure Refinement for Compound **4f** and **8c**

Entry	Identification code	Compound 4f	Compound 8c
01	Empirical formula	C7 H11 N3 O3	C23 H18 Br N5
02	Formula weight	185.19	444.32
03	Temperature	296(2) K	296(2) K
04	Wavelength	0.71073	0.71073
05	Radiation type	Mo K α	Mo K α
06	Radiation source	Fine-focus sealed tube	Fine-focus sealed tube
07	Crystal system	monoclinic	orthorhombic
08	Space group	P 2 ₁ /n	P b c a
09	Cell length	a 7.9591(4) b 4.8417(3) c 23.7648(14)	a 10.5296(11) b 17.3251(18) c 22.405(2)
10	Cell Angle	α 90.0 β 94.296(5) δ 90.0	α 90.0 β 90.00 δ 90.0
11	Cell Volume	913.21(9)	4087.3(7)
12	Density	1.347	1.444
13	Completeness to theta	25.25° / 100%	26.61° / 98.4%
14	Absorption correction	multi-scan	multi-scan
15	Refinement method	Full-matrix least-squares on F ²	Full-matrix least-squares on F ²
16	Index ranges	-10 \leq h \leq 10, -6 \leq k \leq 3, - 28 \leq l \leq 29	-11 \leq h \leq 13, -21 \leq k \leq 21, - 26 \leq l \leq 27
17	Reflection number	1648	4219
18	Theta range	3.44 - 25.25	1.82-26.61
19	Cell formula units Z	4	8
20	CCDC no	979591	1019691

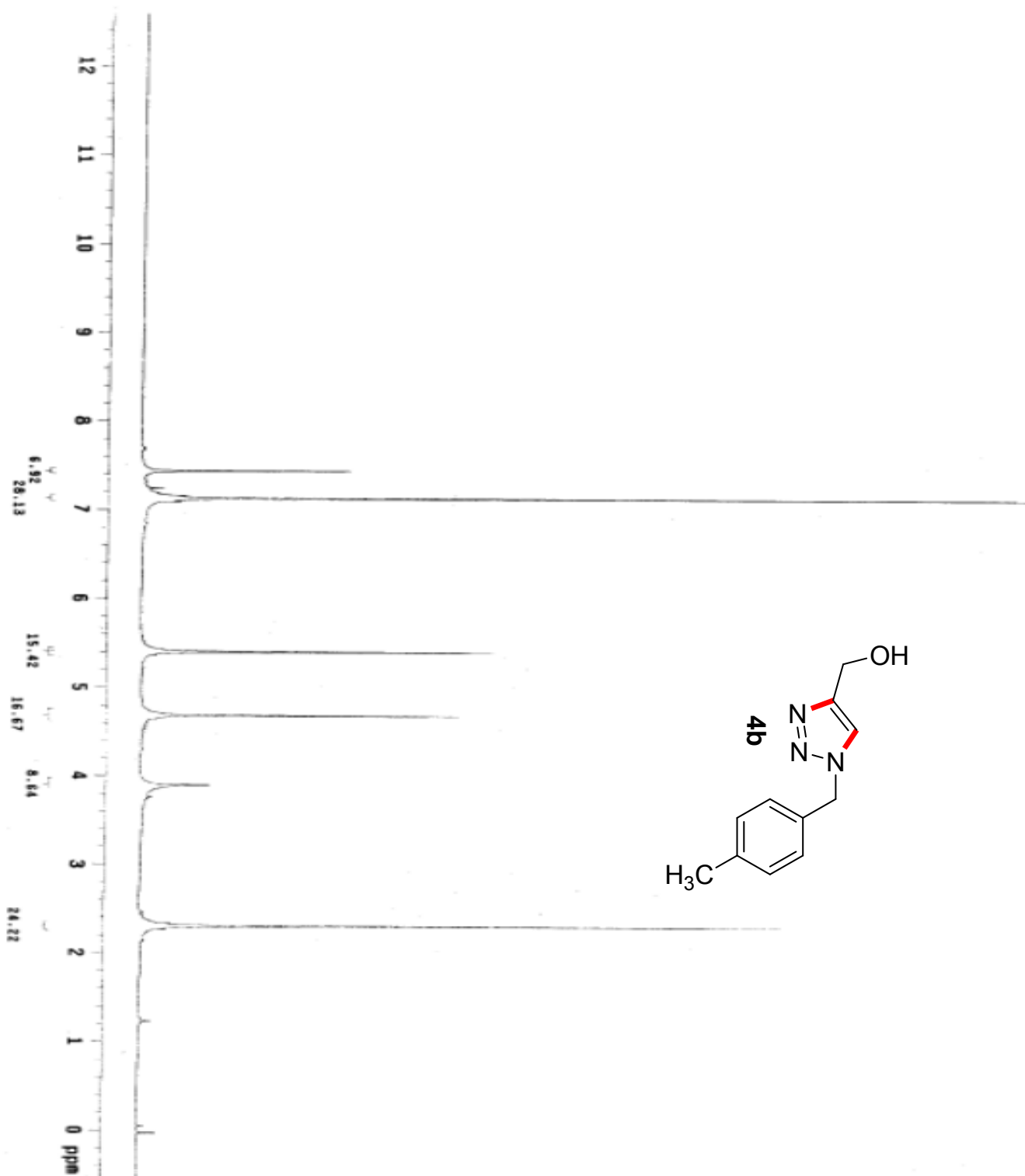
¹H NMR spectra of **4a**



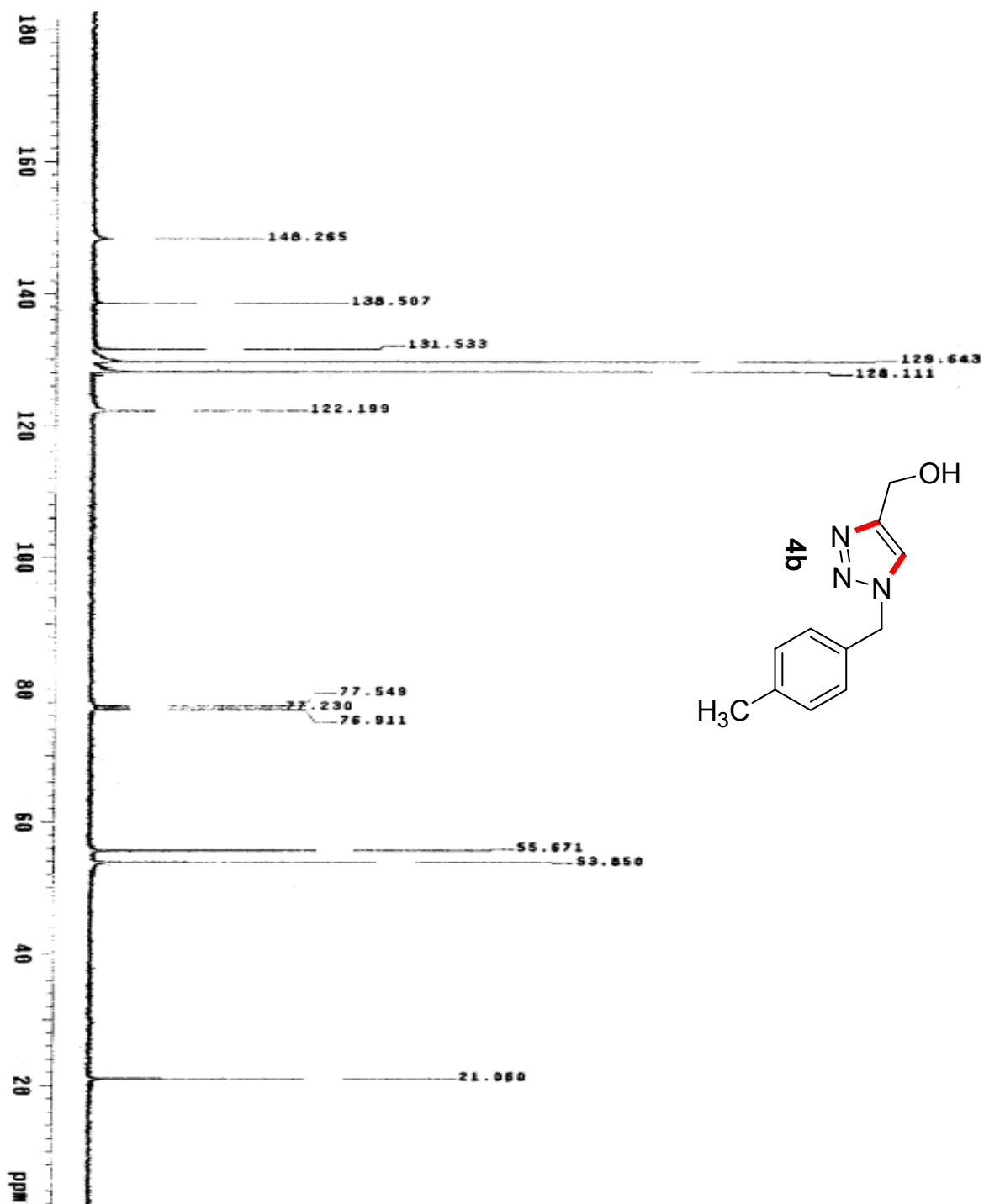
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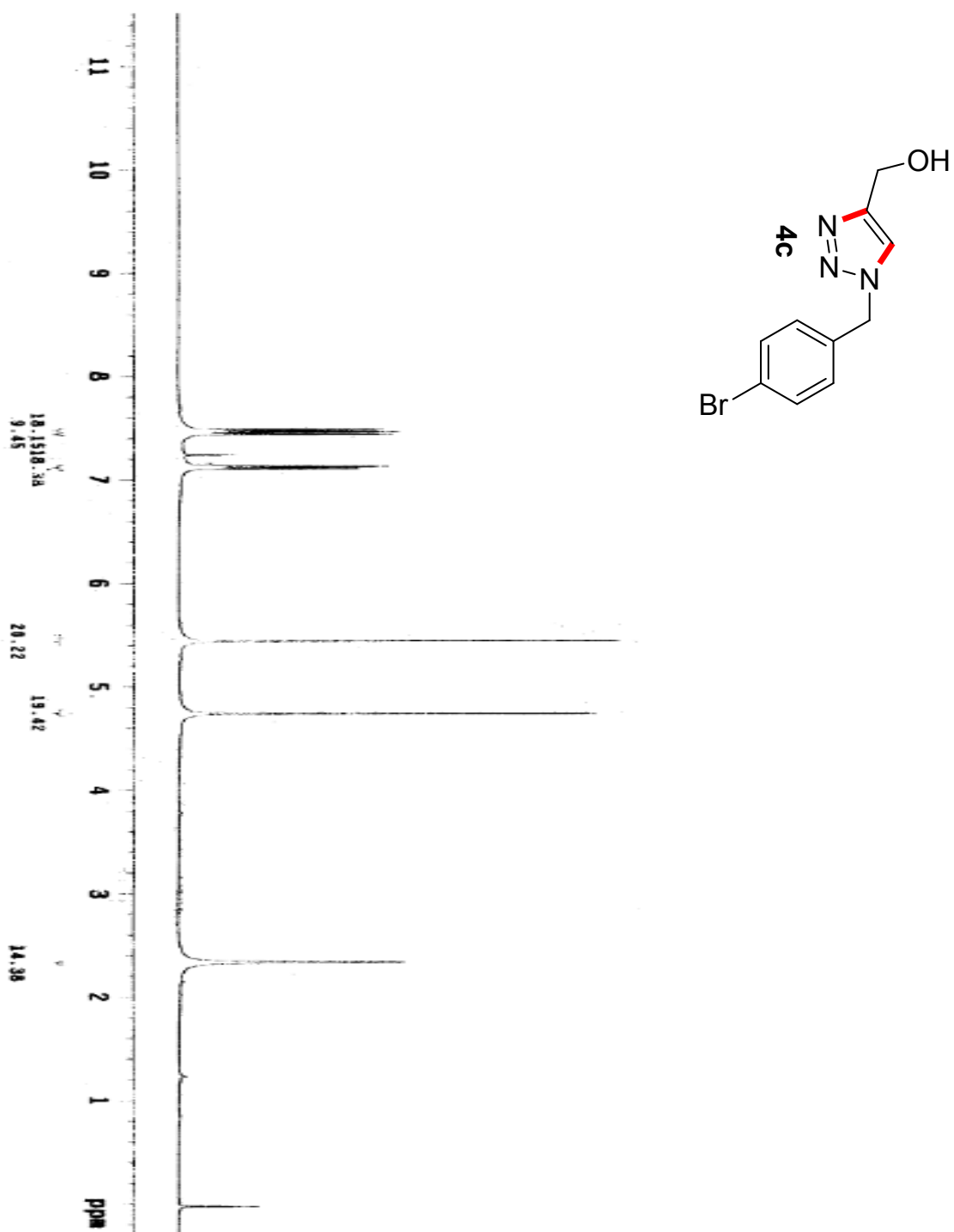
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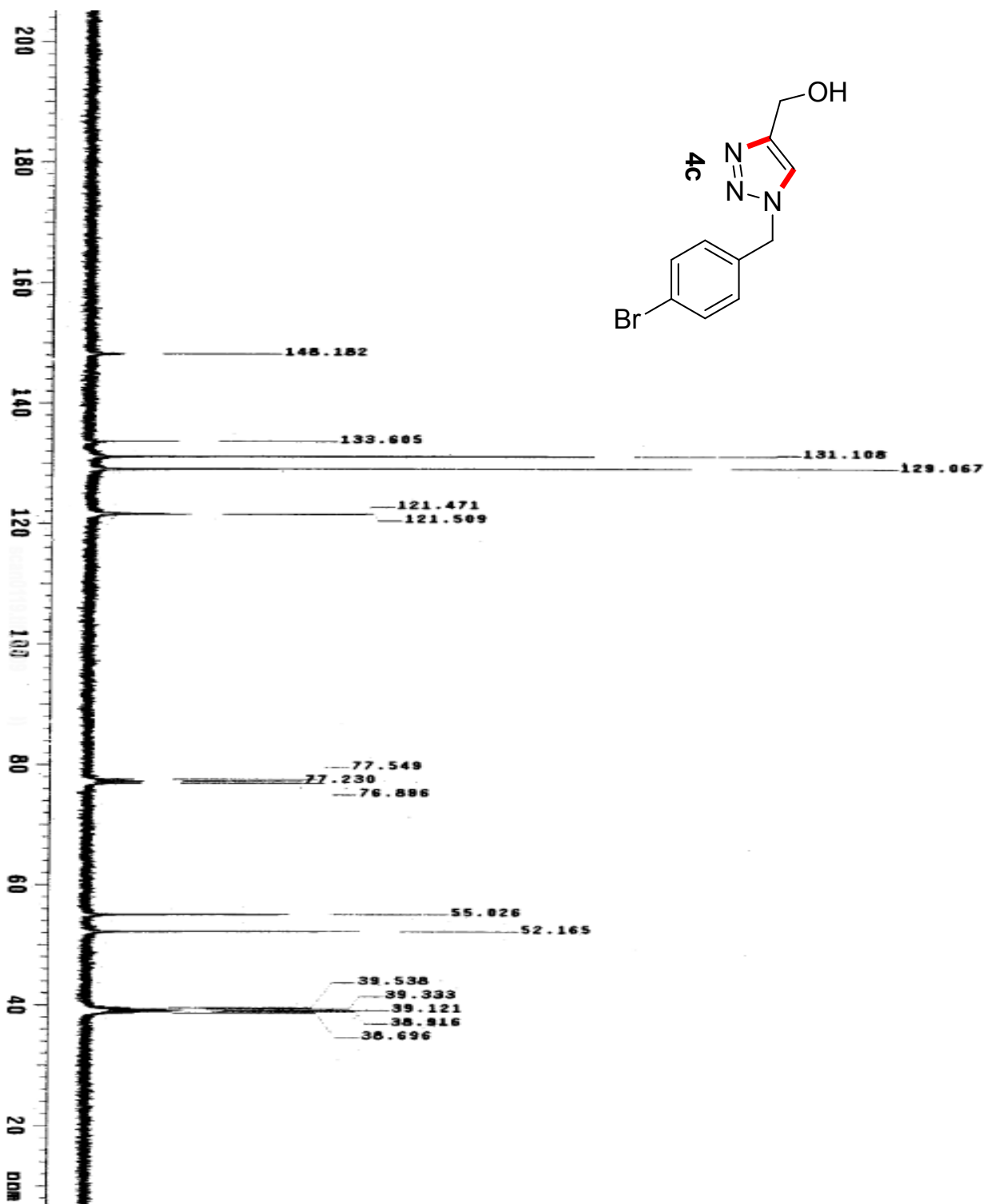
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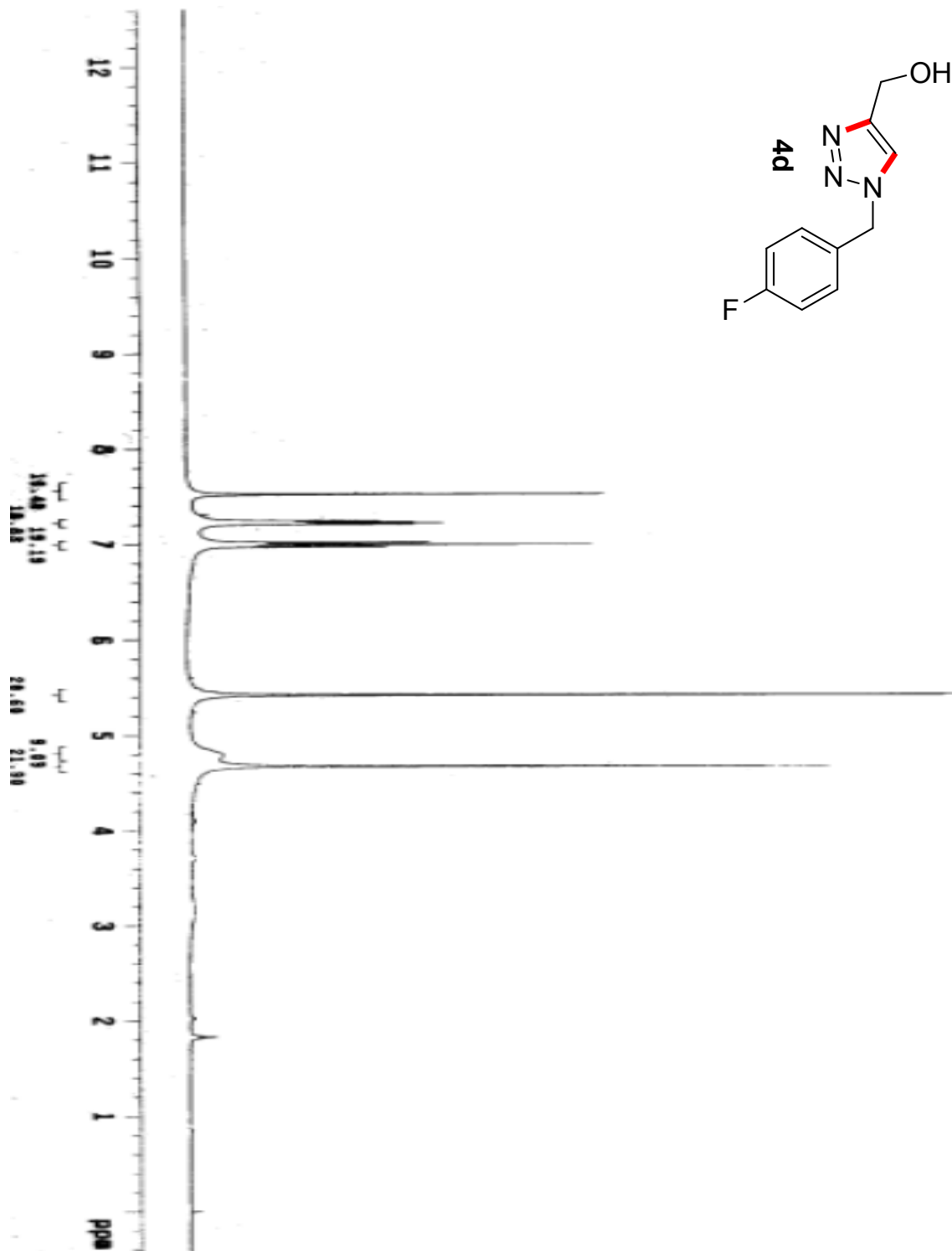
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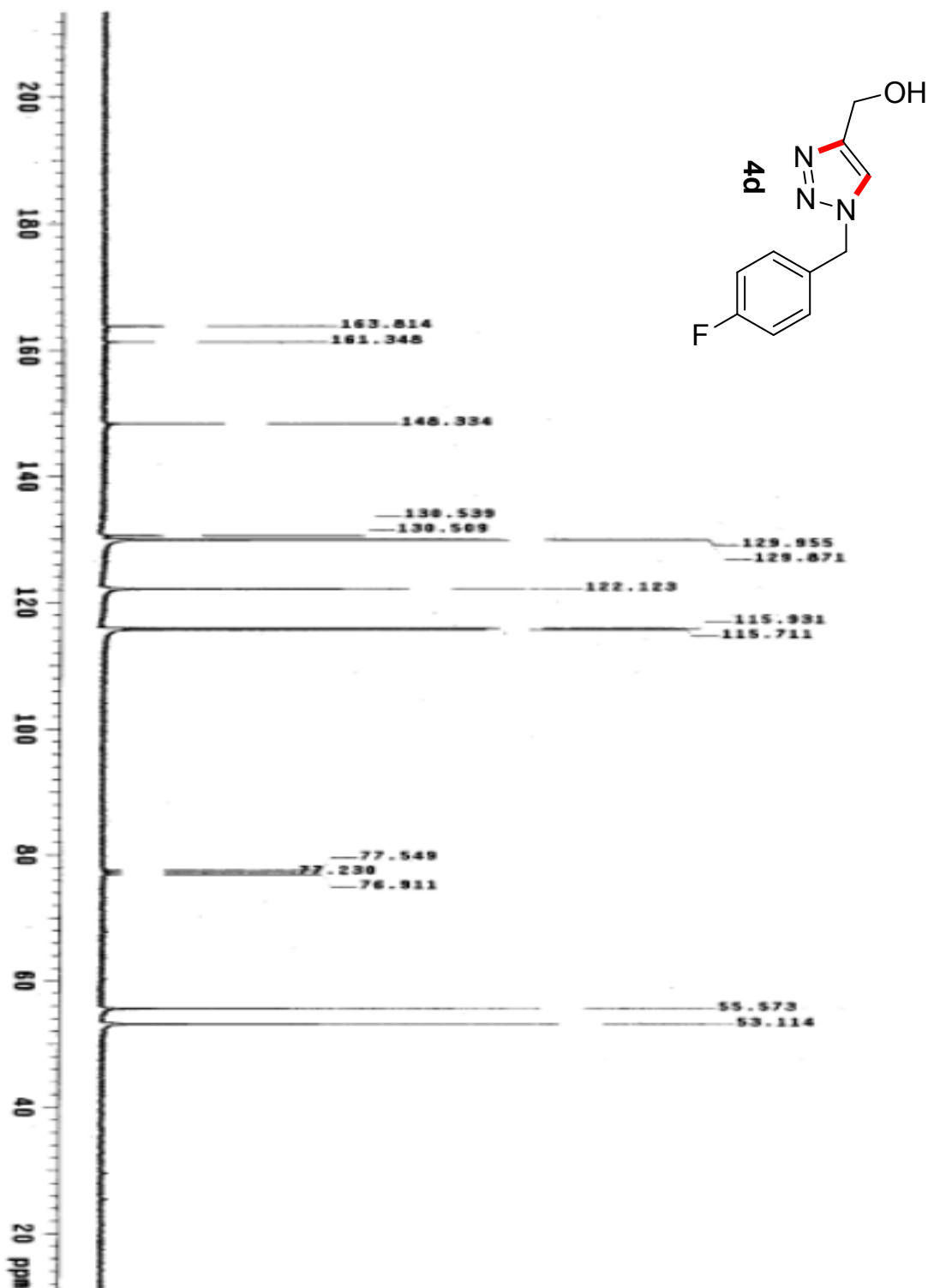
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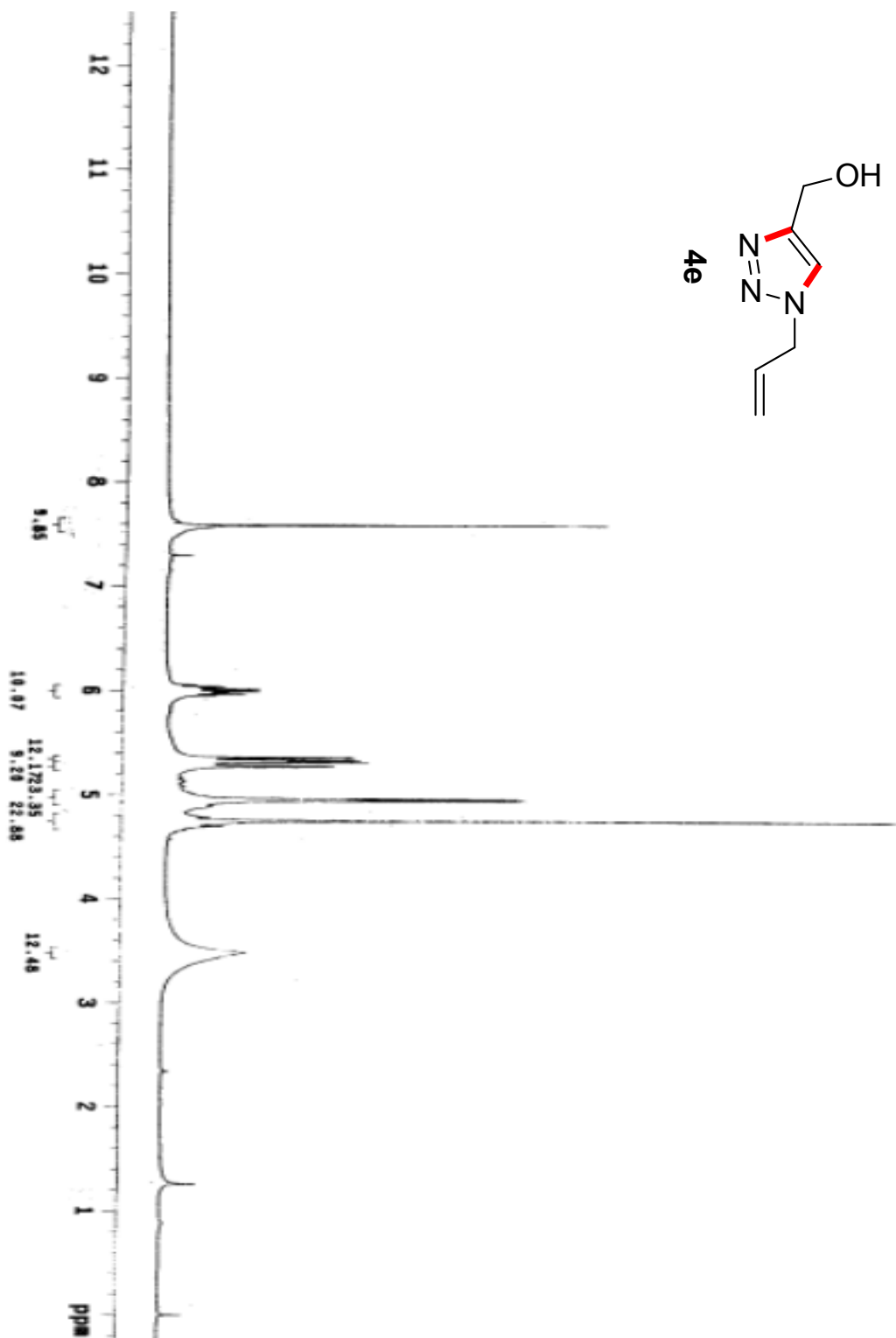
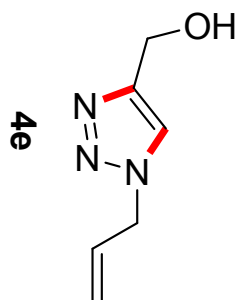
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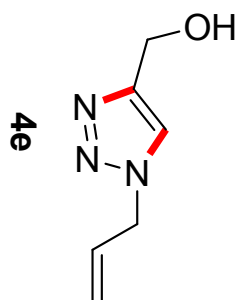
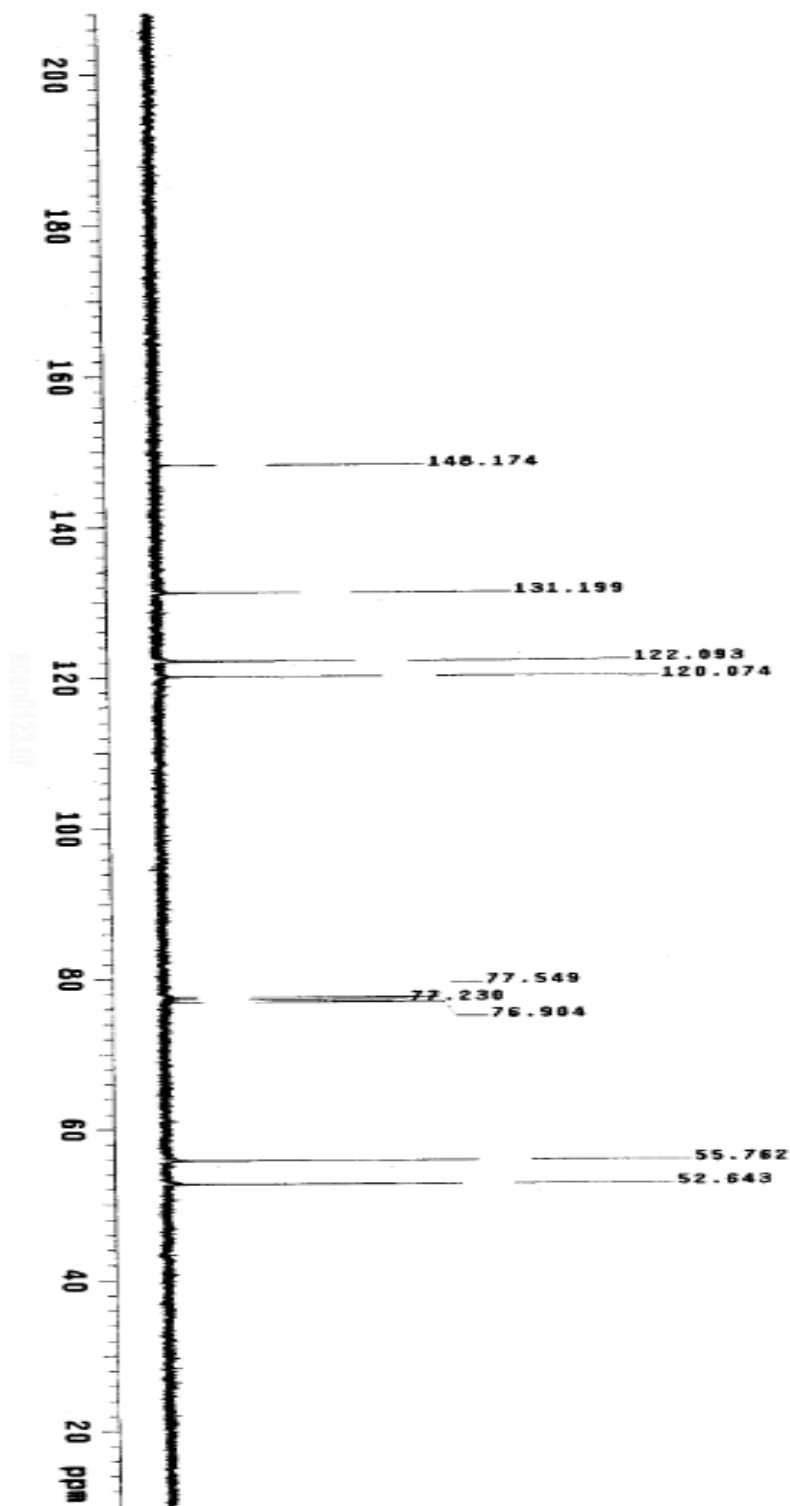
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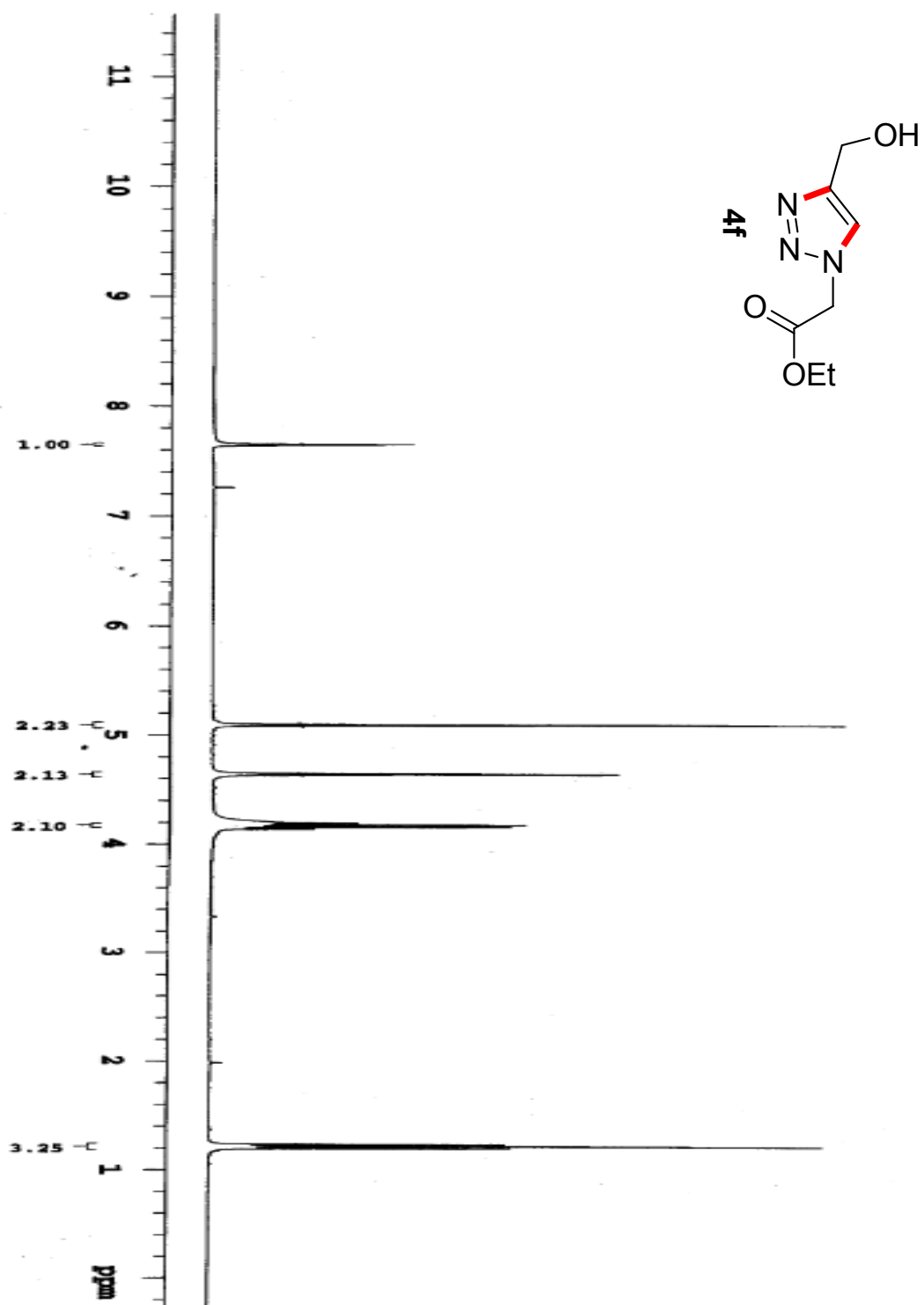
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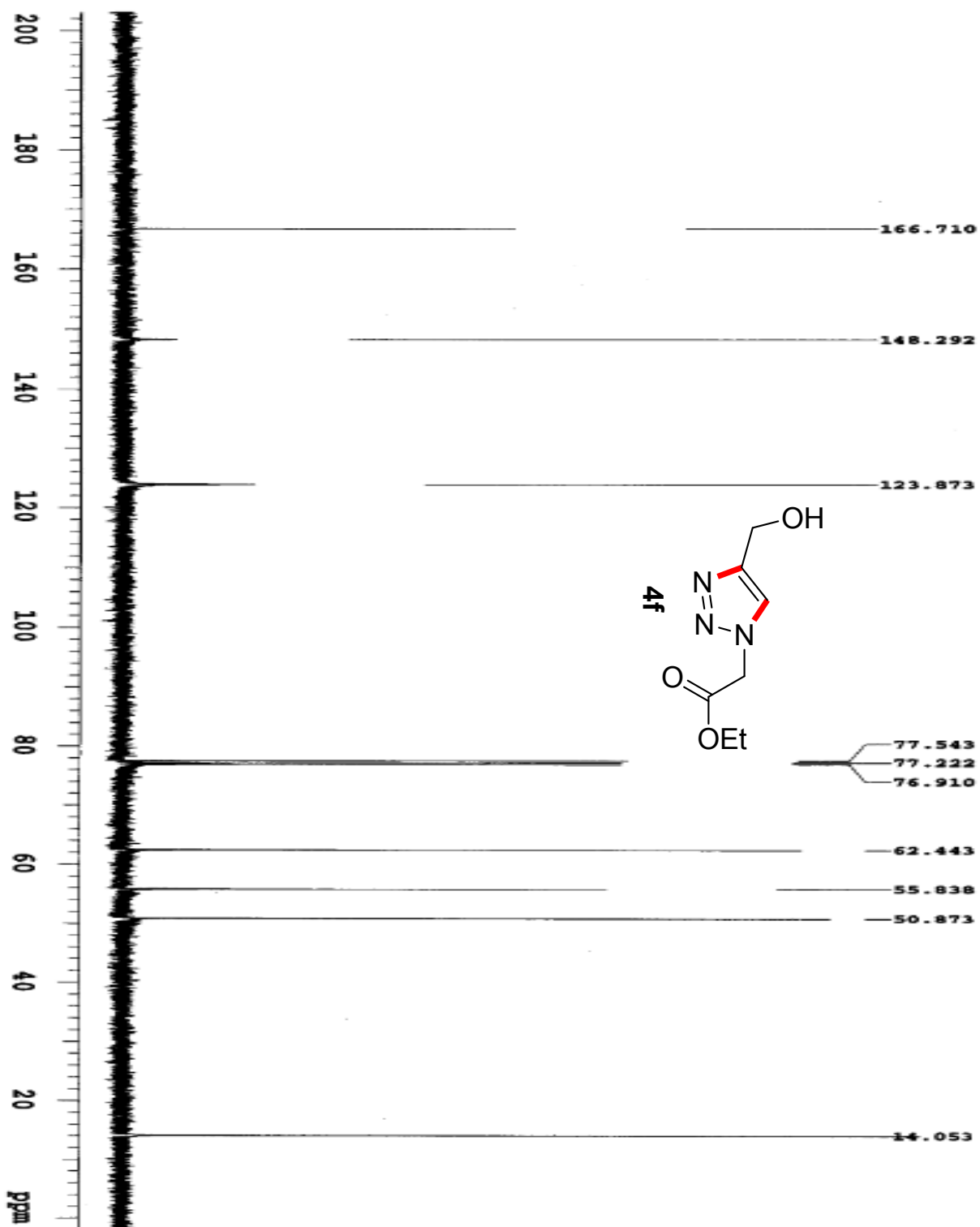
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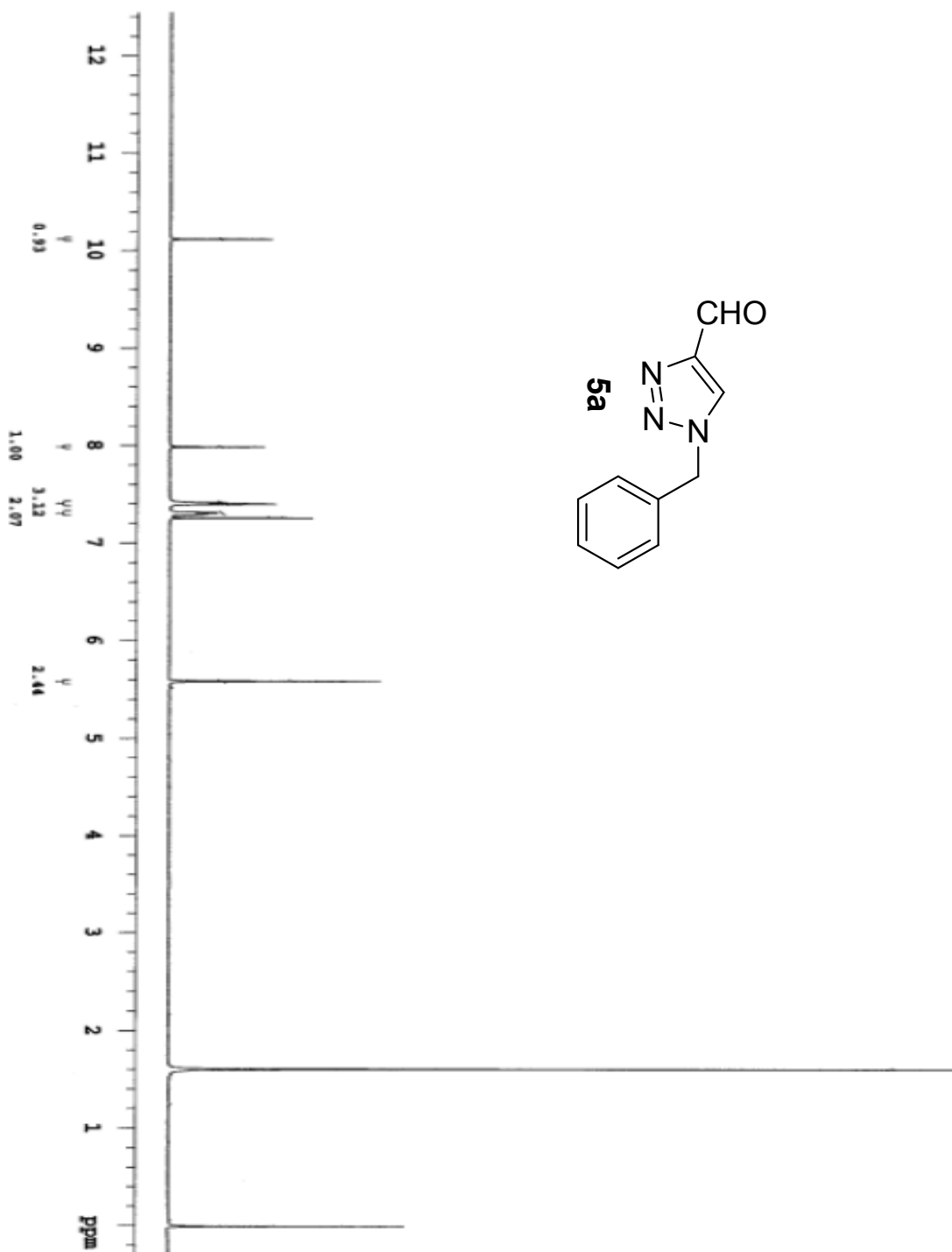
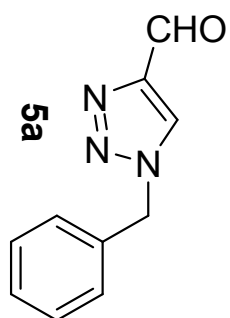
¹H NMR spectra of 4f



¹³C NMR spectra of 4f

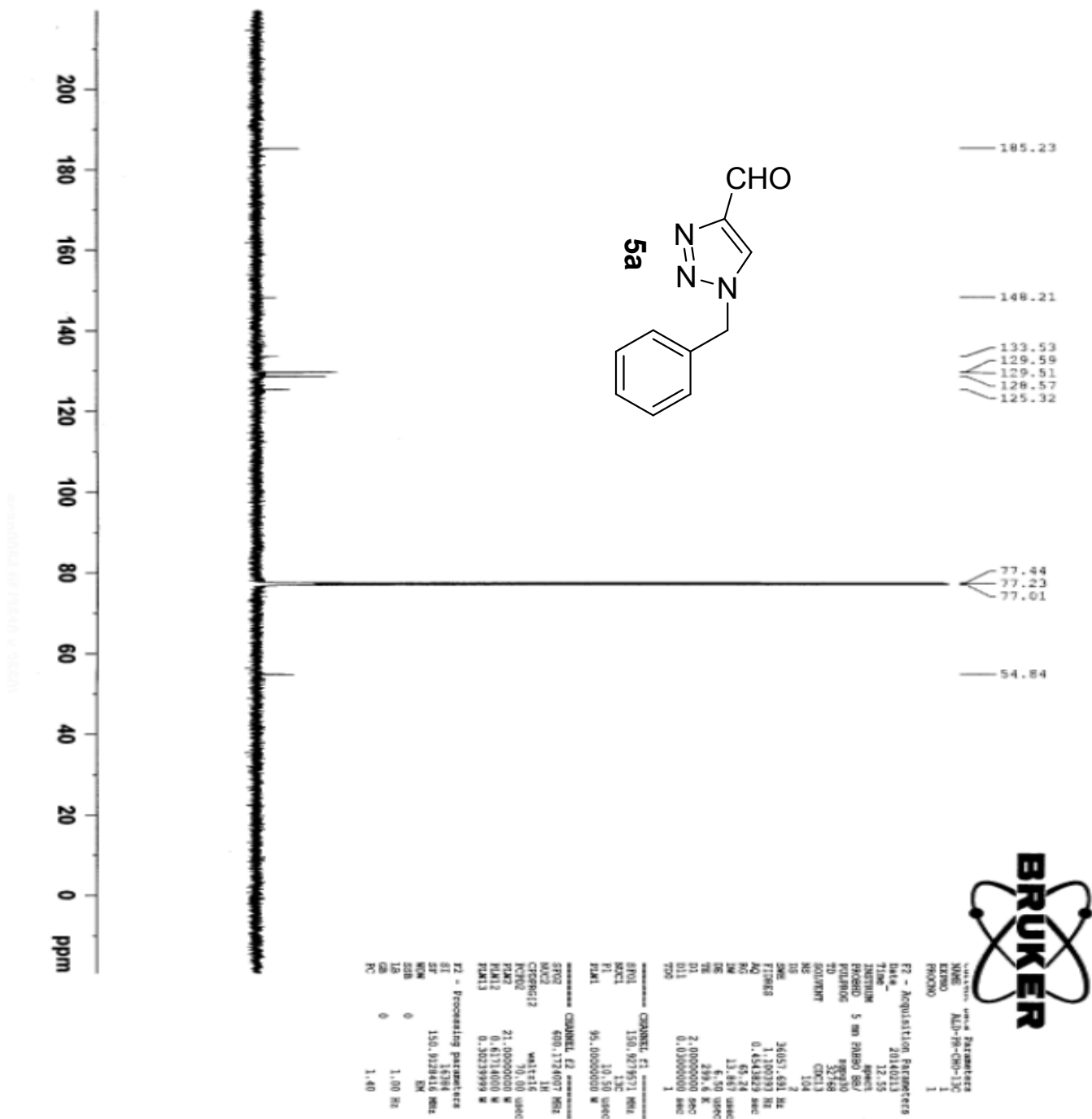


¹H NMR spectra of 5a

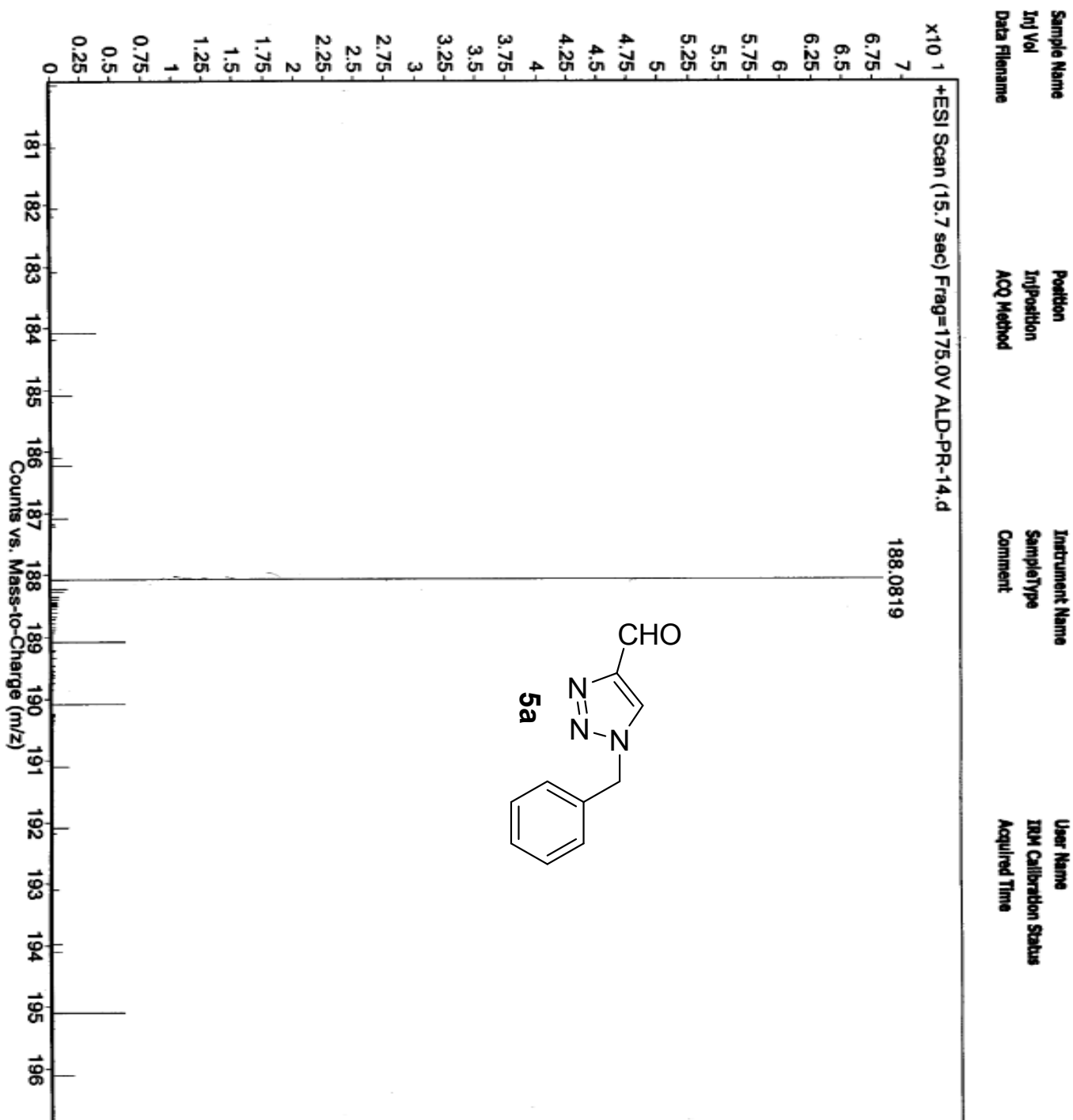


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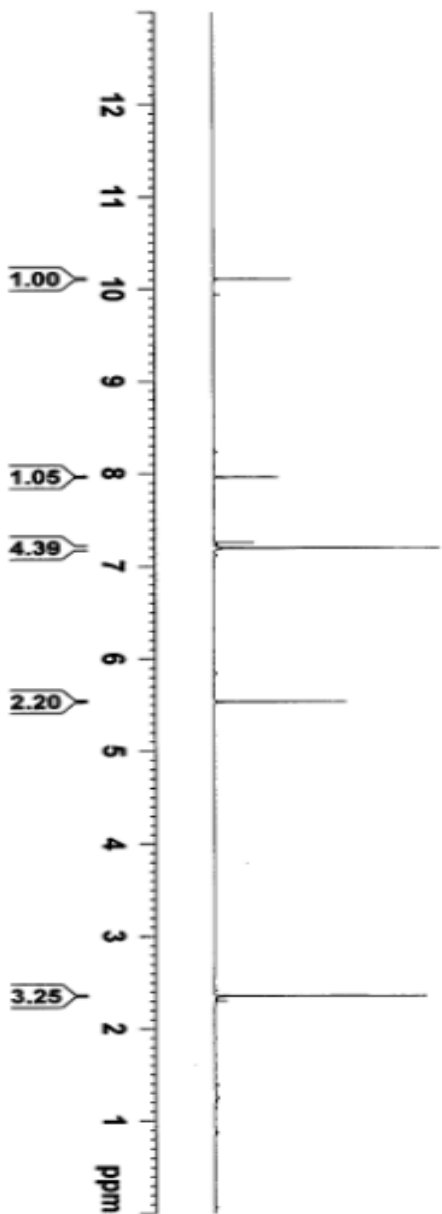
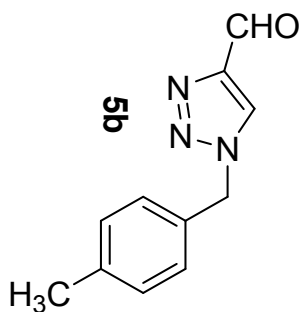
¹³C NMR spectra of 5a



HRMS spectra of 5a



¹H NMR spectra of **5b**



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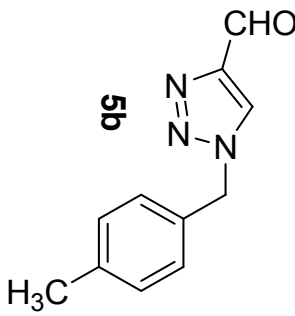
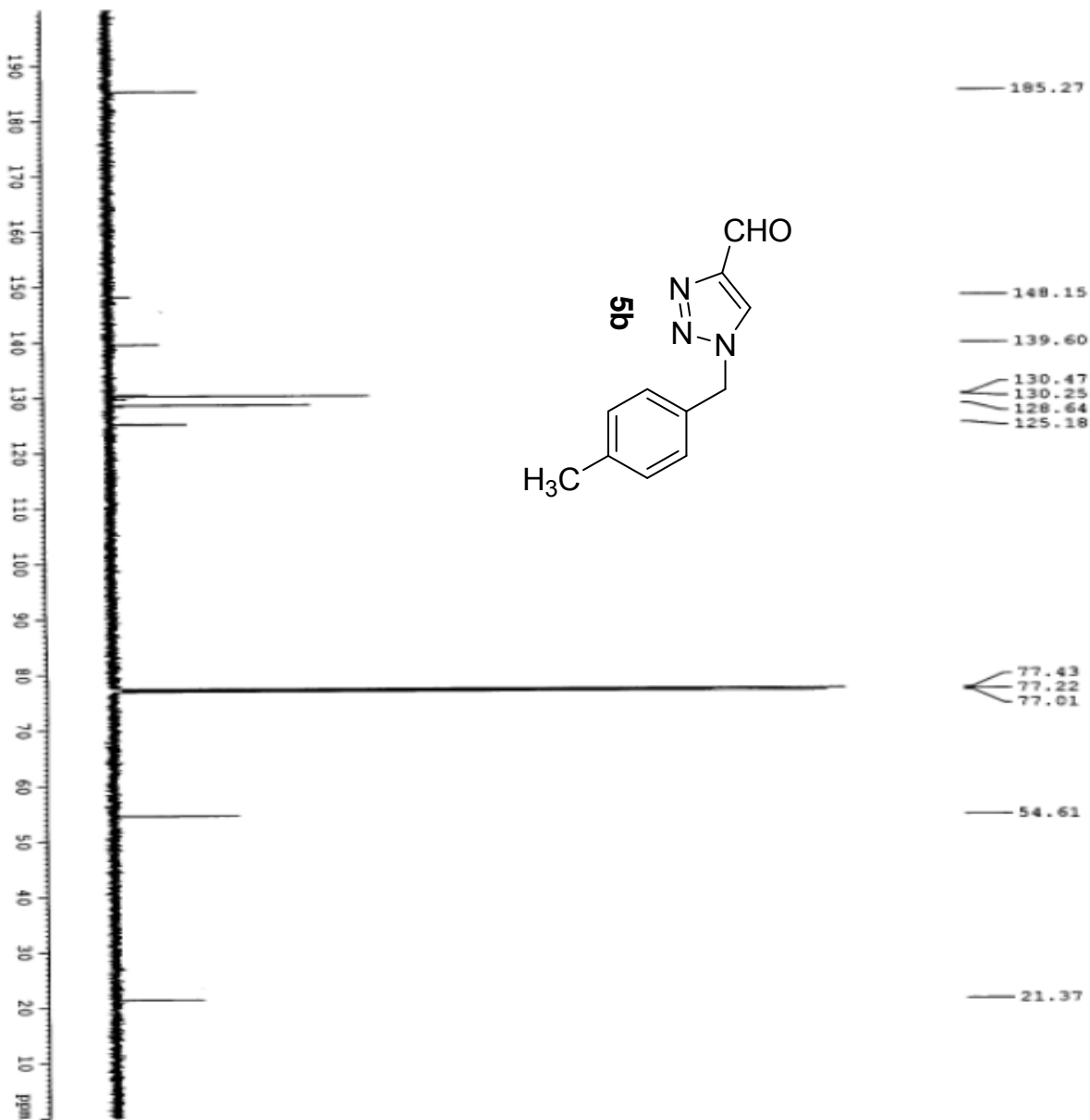
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¹³C NMR spectra of **5b**



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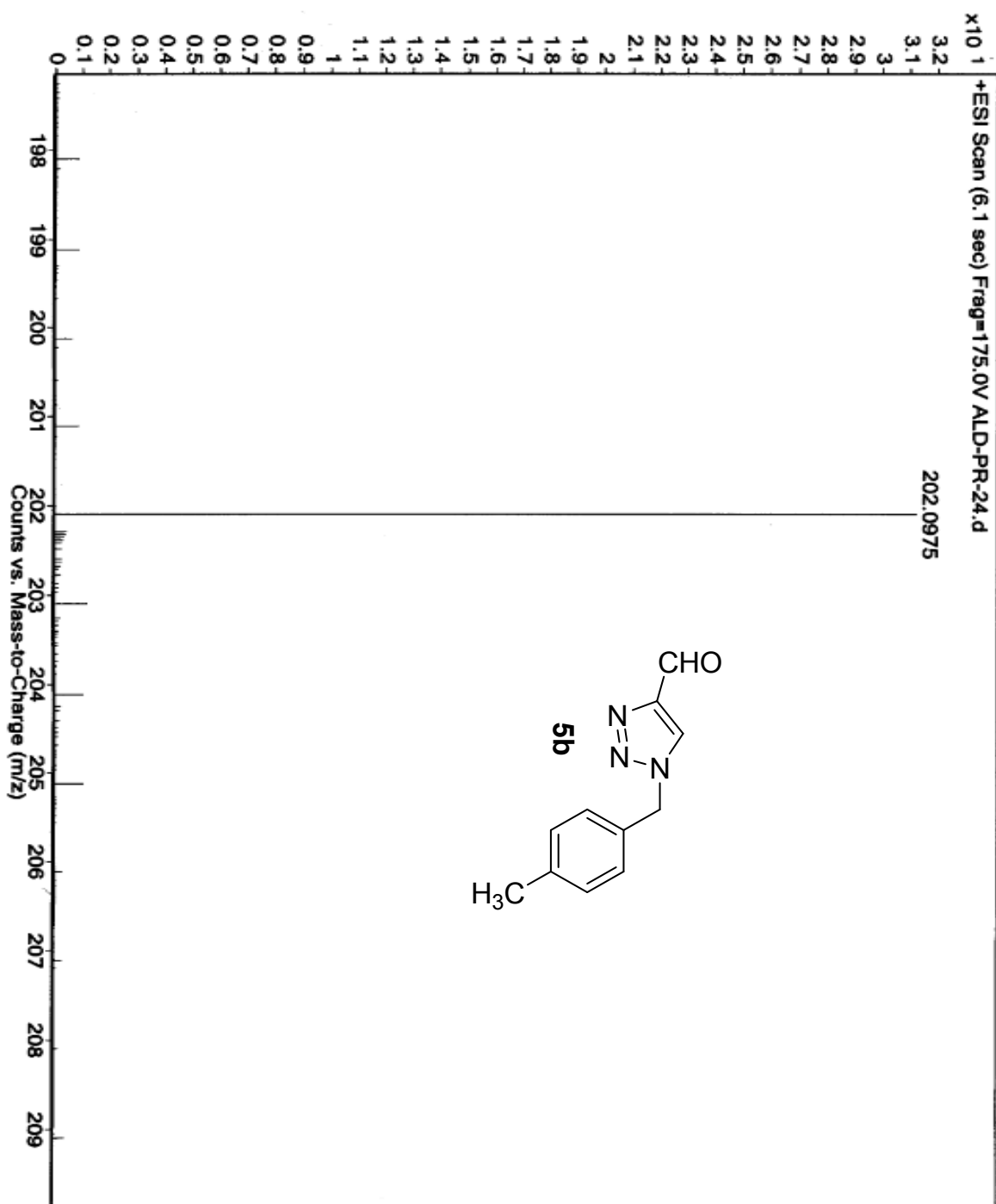
HRMS spectra of 5b

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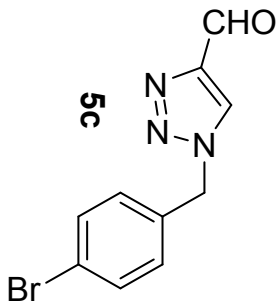
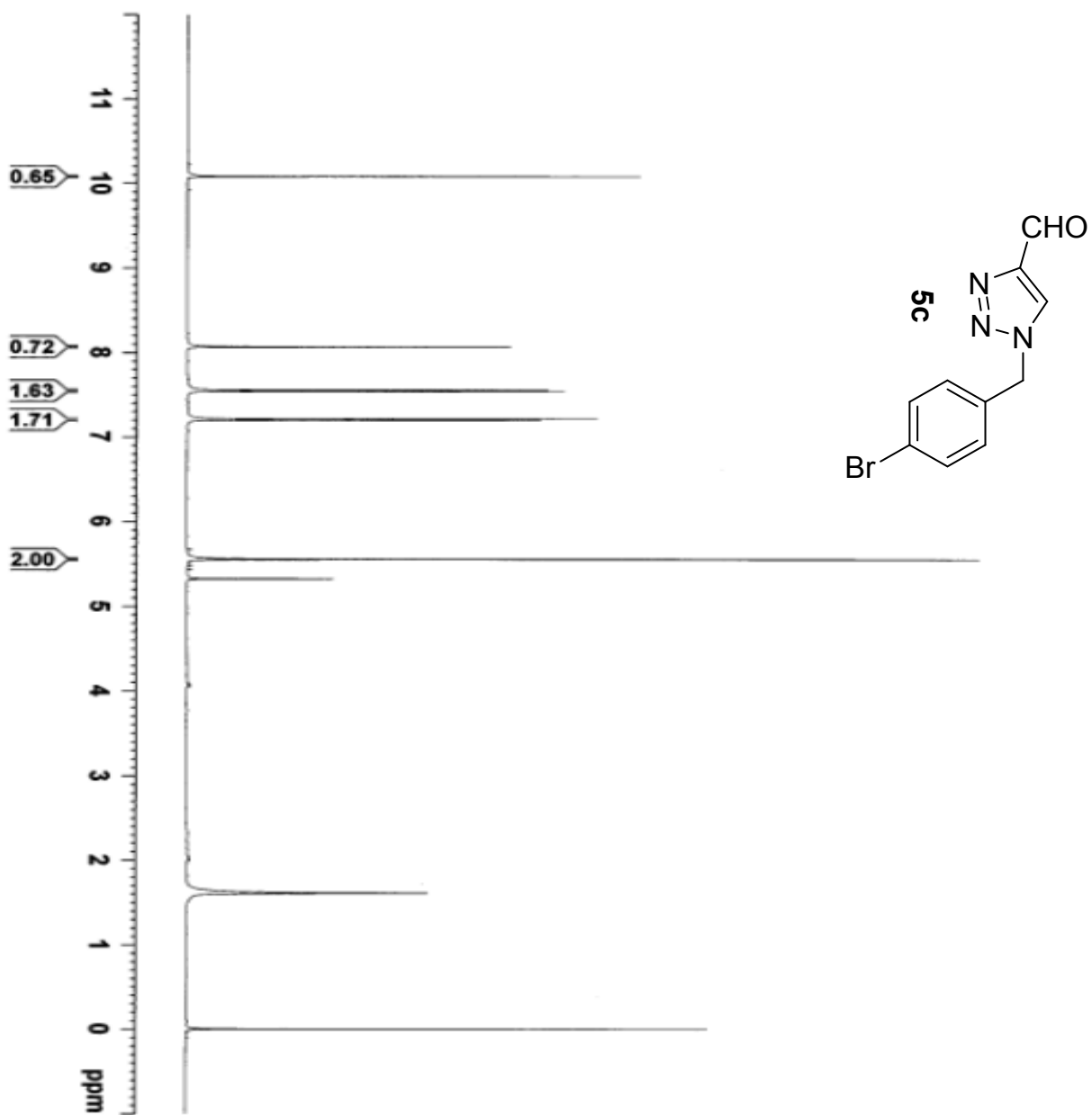
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¹H NMR spectra of 5c



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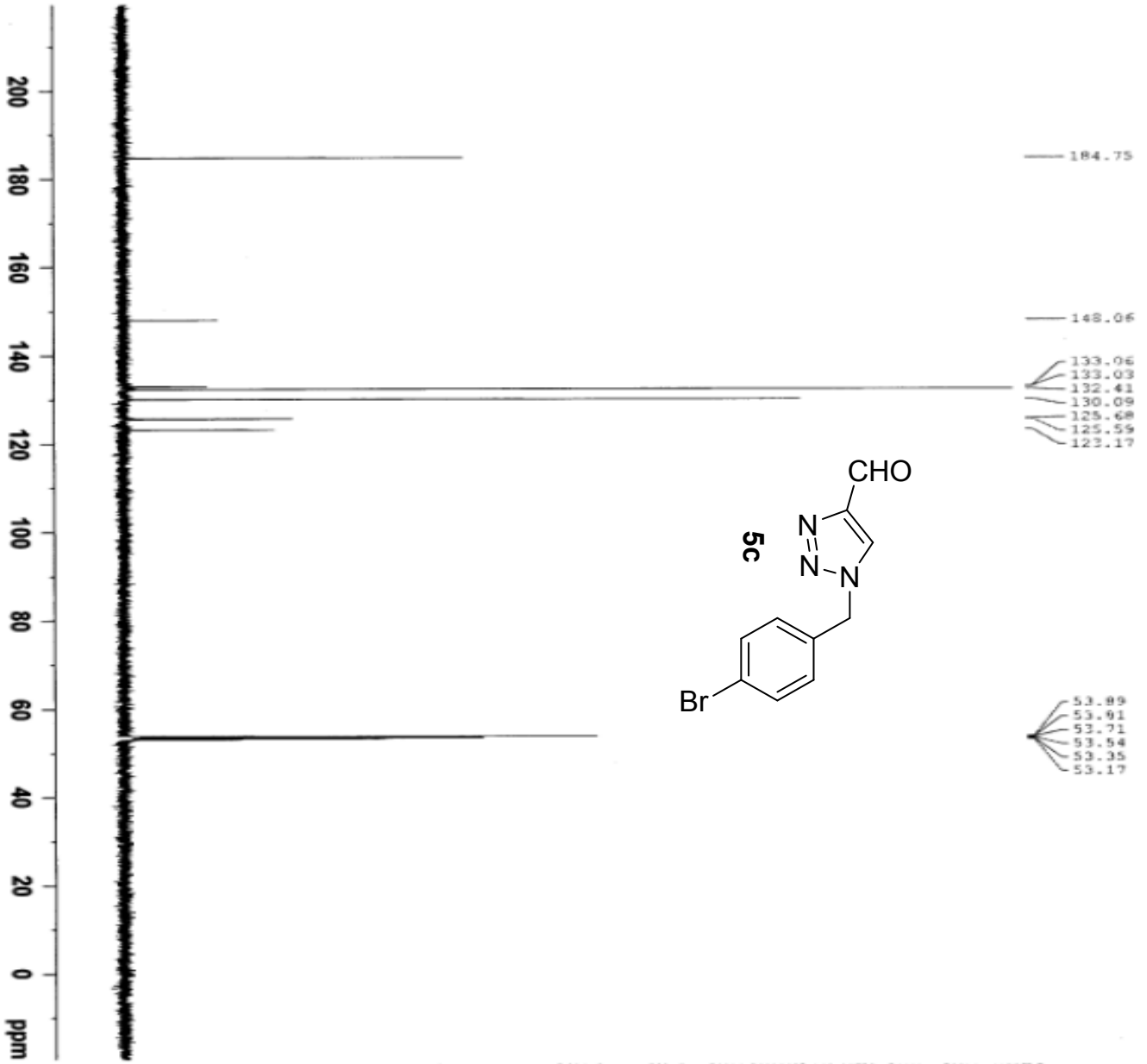
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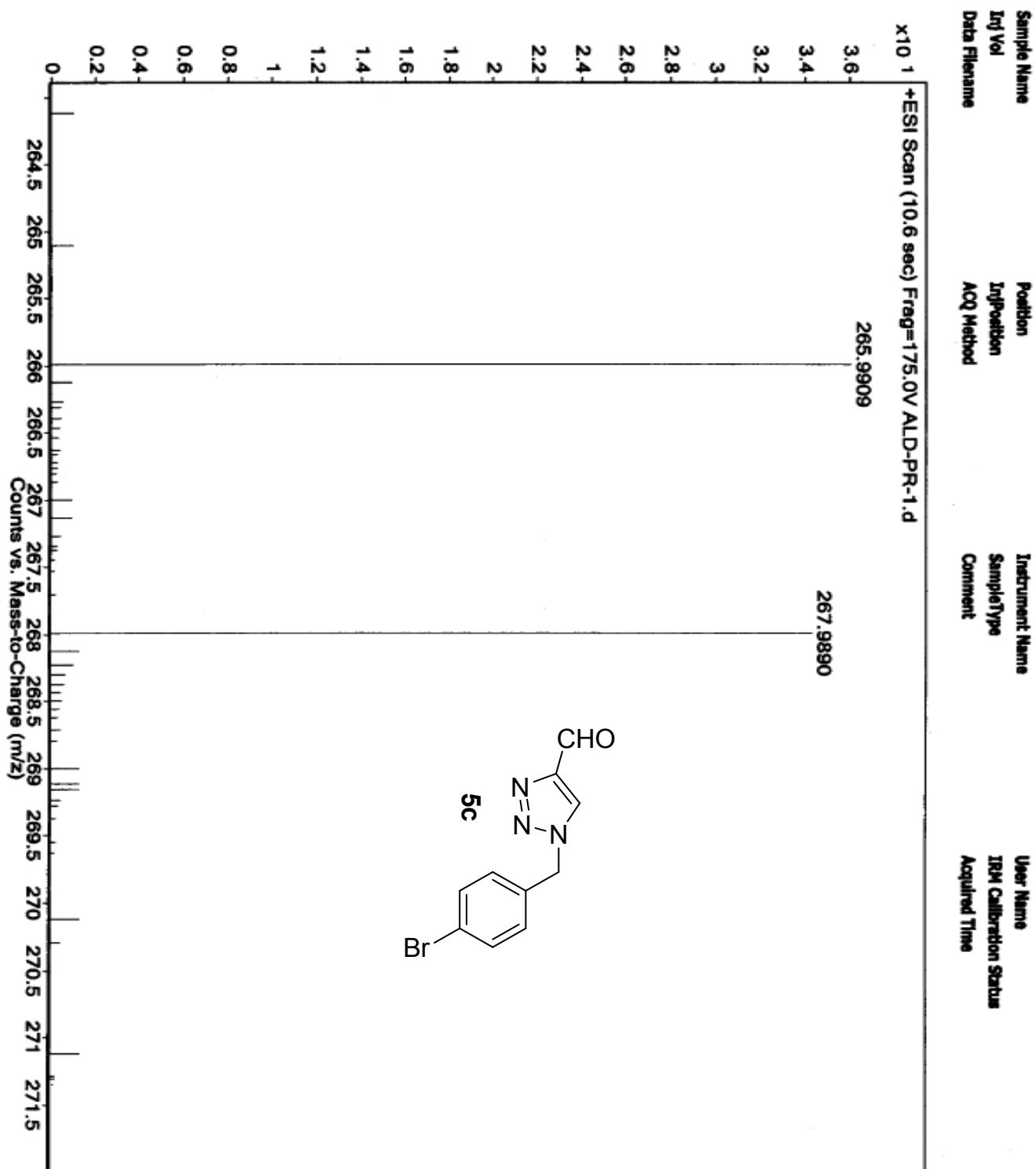
¹³C NMR spectra of 5c



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HRMS spectra of 5c



¹H NMR spectra of 5d

Sample Name:
ALD-4F-PR
Data Collected on:
ITG-NMR-mercury400
Archive directory:

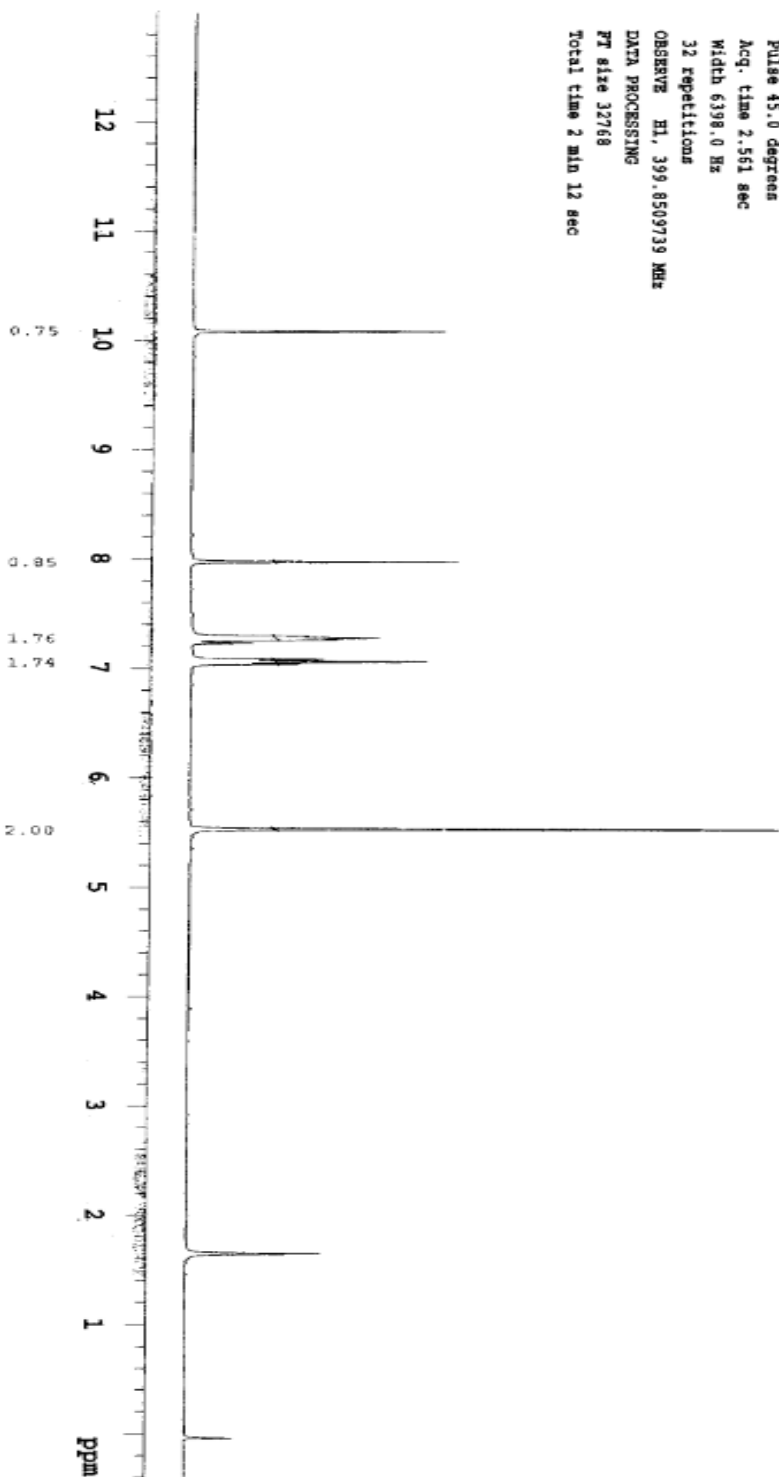
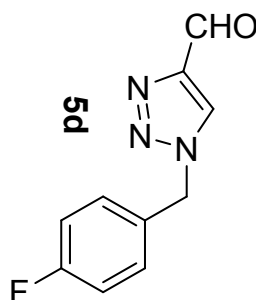
Sample directory:

Filefile: PROTON

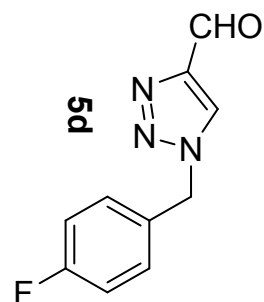
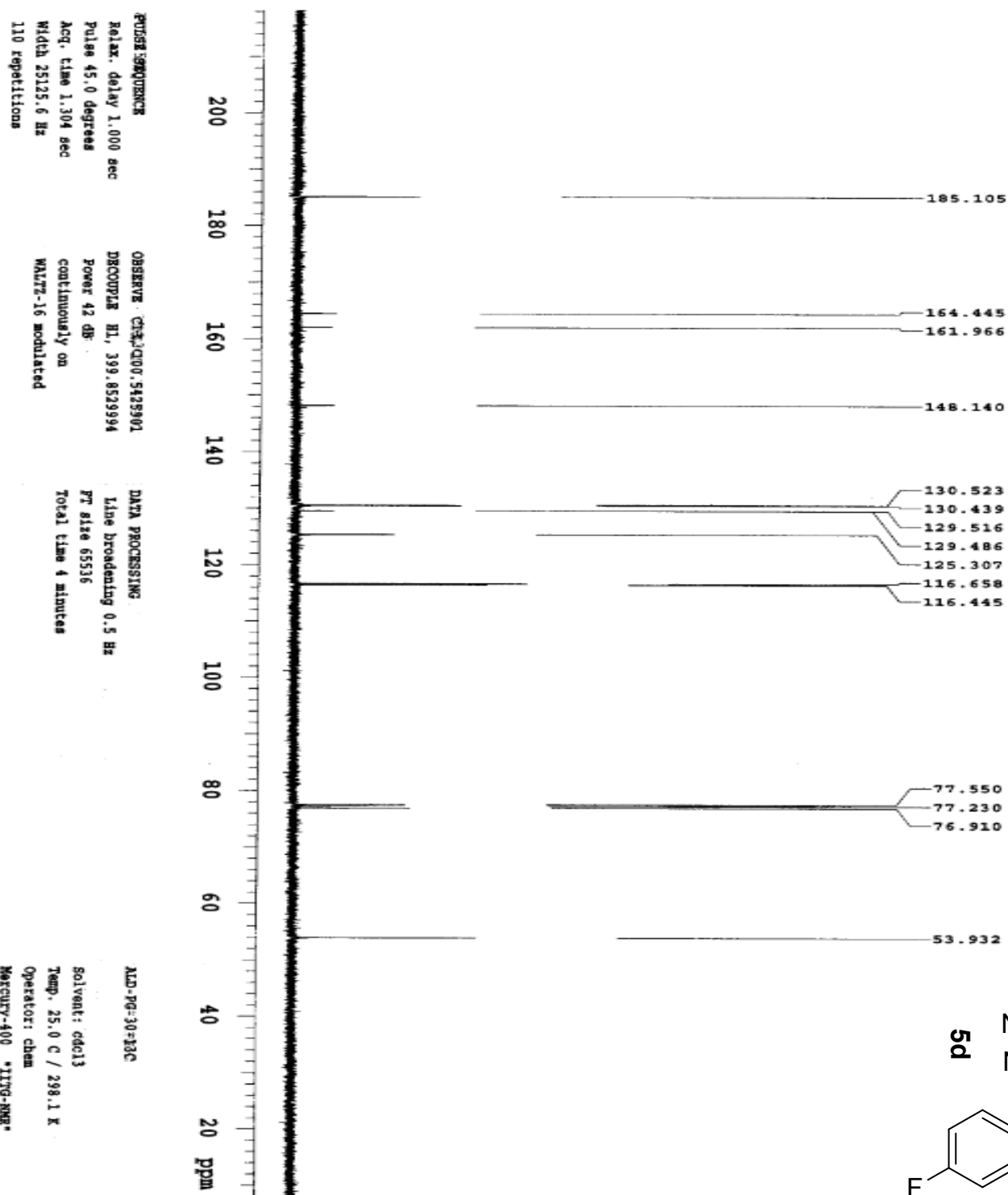
Pulse Sequence: PROTON (zgpg3)
Solvent: cdcl3
Data collected on: Mar 11 2014

Temp: 25.0 C / 298.1 K
Operator: chem

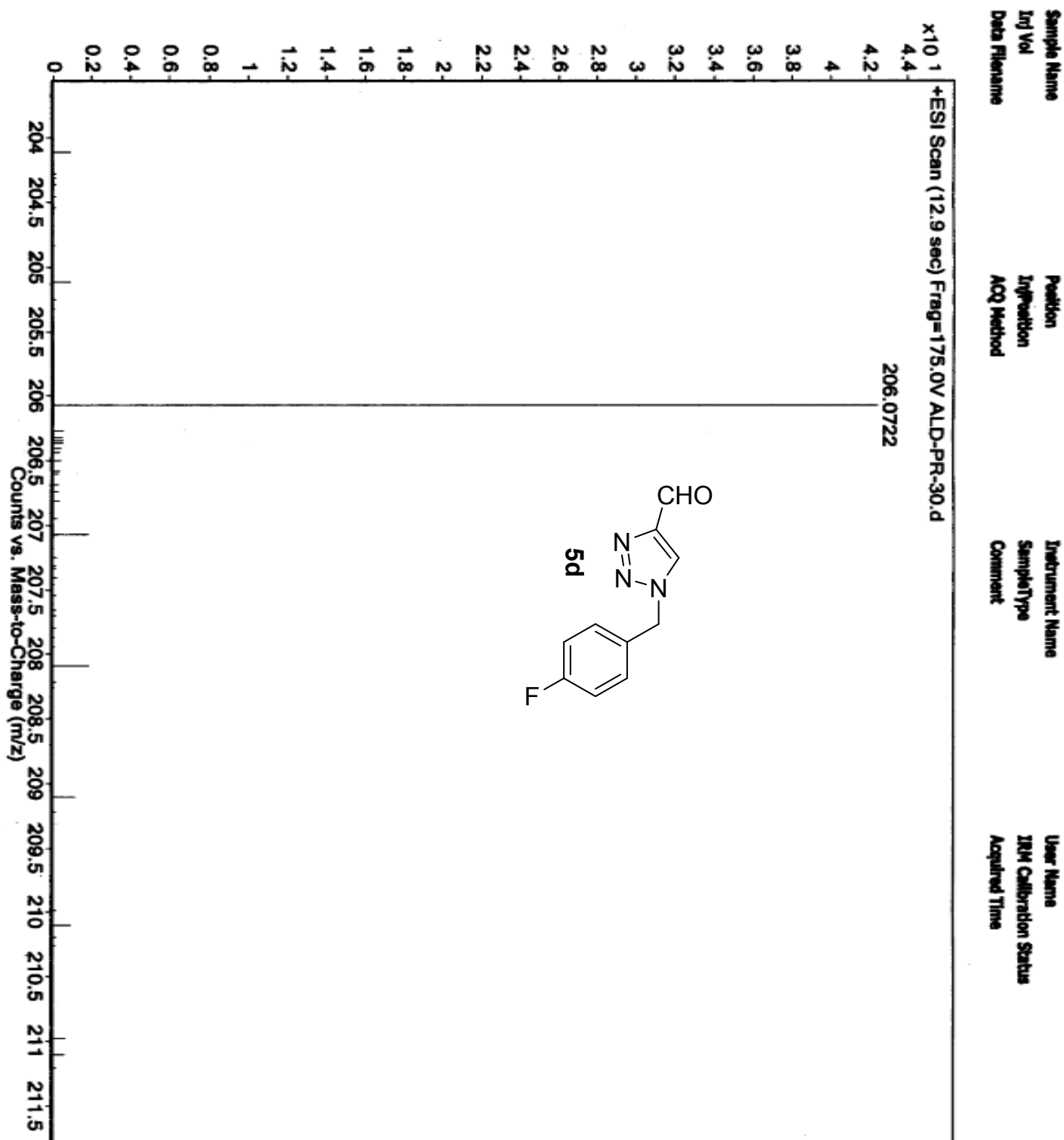
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.8509739 MHz
DATA PROCESSING
FT size 32768
Total time 2 min 12 sec



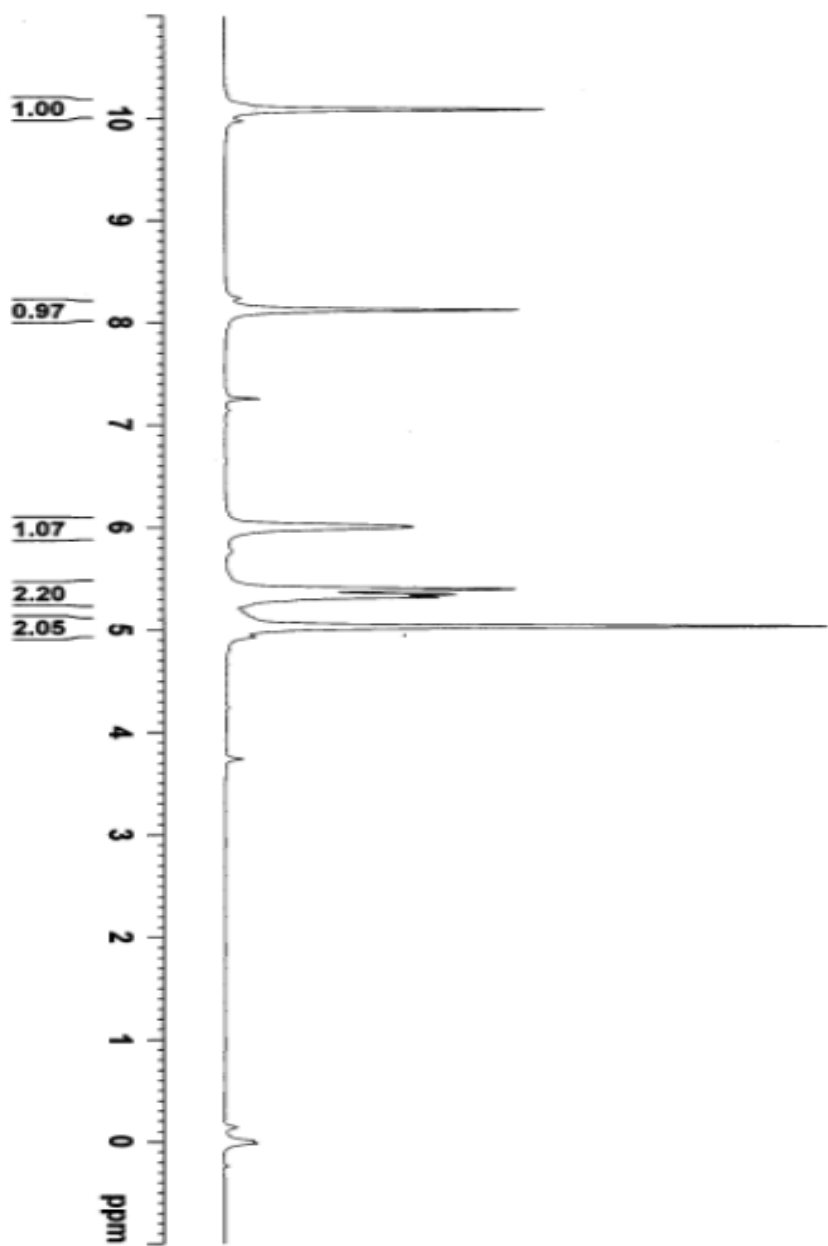
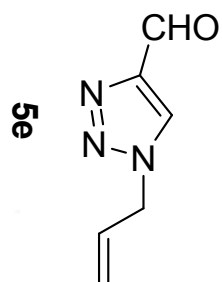
¹³C NMR spectra of 5d



HRMS spectra of 5d



¹H NMR spectra of 5e



Current Data Parameters
 NAME ALD-PR-12-11
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131219
 Time 12.24

INSTRUM spect
 PULPROG zgpg30
 F2FREQ 2930
 F1 32168
 SOLVENT CDCl3
 NS 16
 DS 2

SI 12019.230 Hz
 SF 0.366788 Hz
 AQ 1.1631488 sec
 RG 39.59

SW 41.000 usec
 SR 6.50 usec
 TR 296.6 K
 D1 1.00000000 sec
 T10 1

***** CHANNEL f1 *****
 SFO1 600.137063 MHz
 NUC1 13
 P1 12.00 usec
 PL1 21.00000000 W

F2 - Processing parameters
 SI 16384
 SF 600.137063 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR spectra of 5e



Current Data Parameters
 NAME ALD-PR-12-13C
 EXPRNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20131219
 Time 12.17
 INSTRUM spect
 PROBD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 56
 DS 2
 SWH 36057.691 Hz
 FIDRES 1.100393 Hz
 AQ 0.4543829 sec
 RG 65.24
 DW 13.867 usec
 DE 6.50 usec
 TE 296.7 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

CHANNEL F1

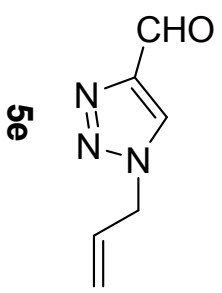
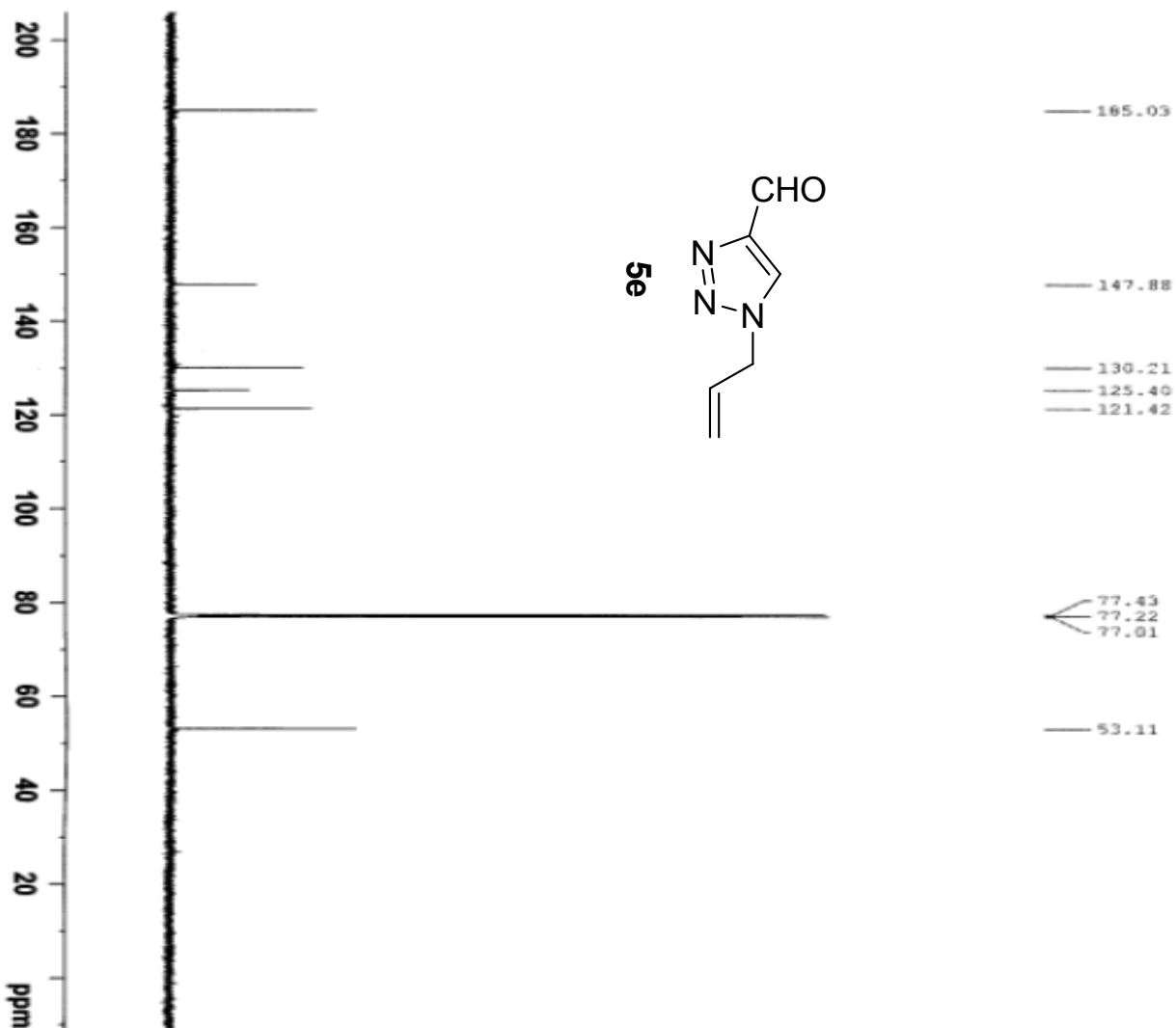
SFO1 150.9279571 MHz
 NUC1 13C
 P1 10.50 usec
 PLW1 95.00000000 W

CHANNEL F2

SFO2 600.1724007 MHz
 NUC2 1H
 CPOPRG(2) waltz16
 PCPD2 70.00 usec
 PLW2 21.00000000 W
 PLW12 0.61714000 W
 PLW13 0.30239999 W

F2 - Processing parameters

SI 16384
 SF 150.9128657 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



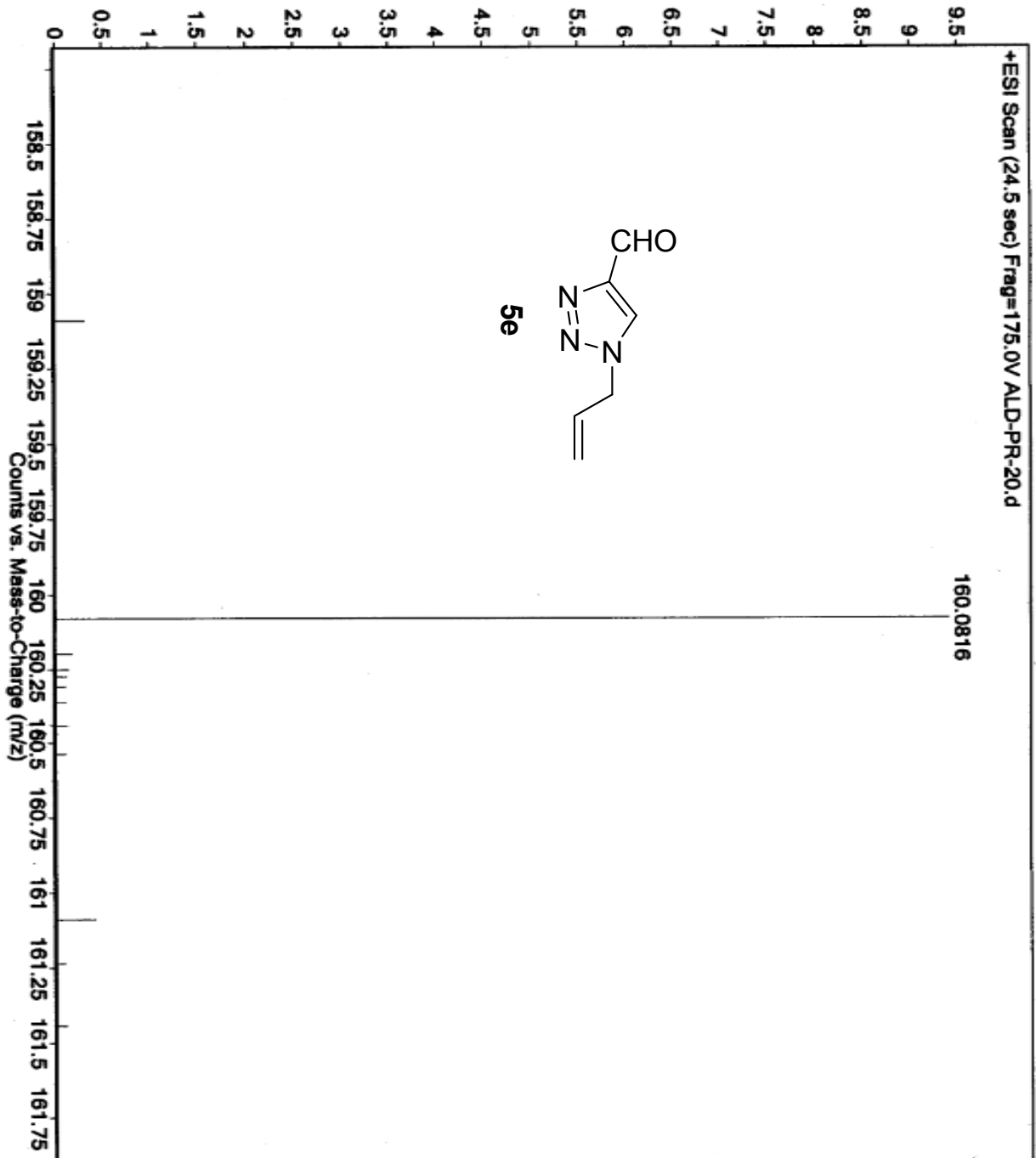
MS spectra of 5e

Sample Name
Inj Vol
Data Filename

Position
InjPosition
Acq Method

Instrument Name
SampleType
Comment

User Name
IRM Calibration Status
Acquired Time



ALD_PR-Rt-CHO

Sample Name:

ALD_PR-Rt-CHO

Data Collected on:

IITG-NMR-mercury400

Archive directory:

/export/home/chempack/vnmrpy/data

Sample directory:

FIDFile: PROTON

Pulse Sequence: PROTON (zgpg3)

Solvent: cdcl3

Data collected on: Mar 21 2014

Temp: 25.0 C / 298.1 K

Operator: chem

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.561 sec

Width 6398.0 Hz

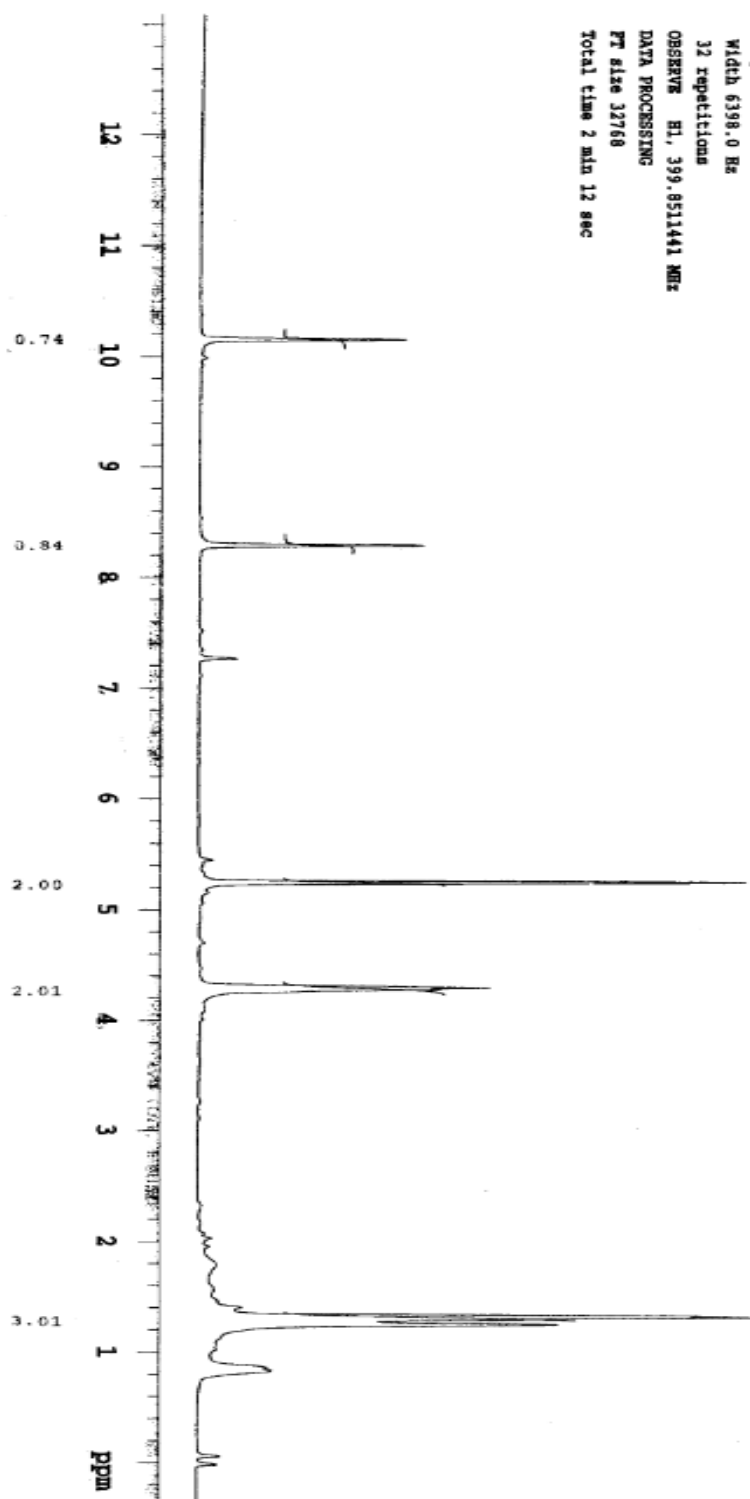
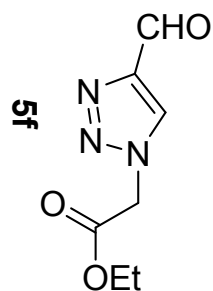
32 repetitions

OBSERVE H1, 399.851441 MHz

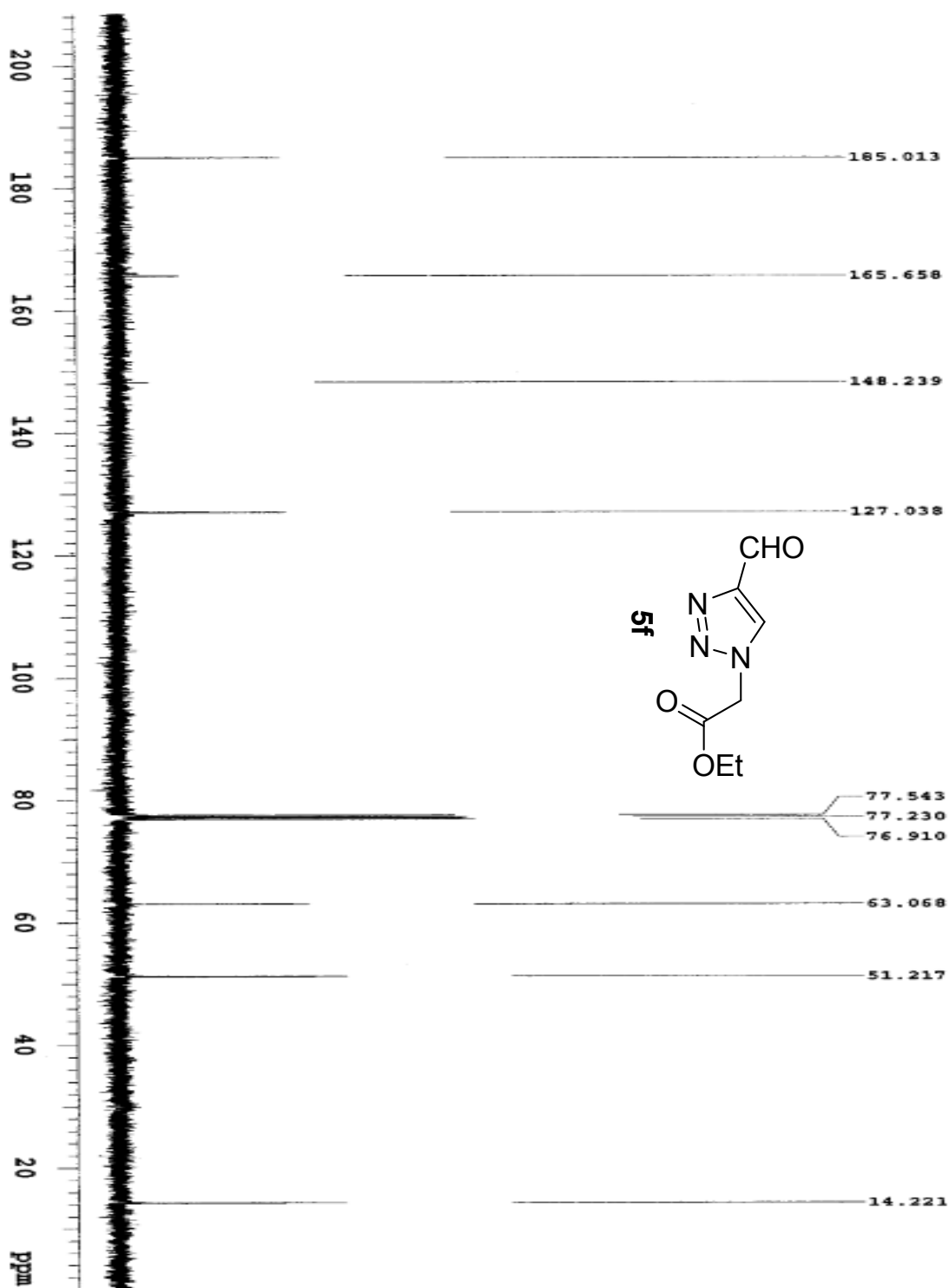
DATA PROCESSING

FT else 32768

Total time 2 min 12 sec



¹³C NMR spectra of 5f



PROB:SEQUENCE:
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.304 sec
Width 25125.6 Hz
550 repetitions

OBSERVE:CL3, 100, 5426269
DECUPLE HL, 39, 852994
Power 42 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING:
Line broadening 0.5 Hz
FT size 65536
Total time 21 minutes

ALD-PR-BE-CHO-13C
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: chem
Mercury-400 *11TG-NMR*

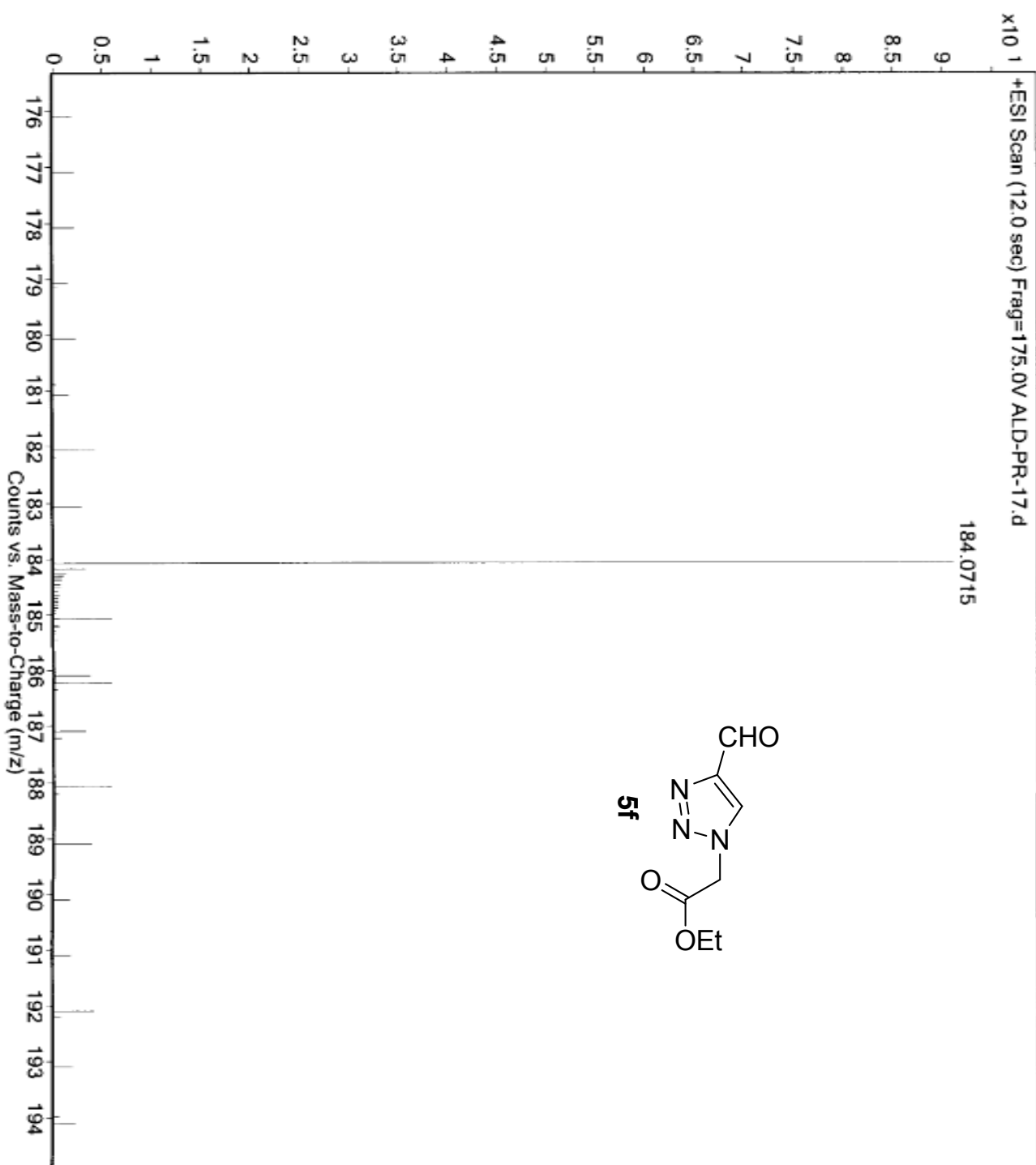
HRMS spectra of **5f**

Sample Name
Inj Vol
Data Filename

Position
InjPosition
ACQ Method

Instrument Name
SampleType
Comment

User Name
IRM Calibration Status
Acquired Time



¹H NMR spectra of 8a

Sample Name:
ALD_PPG_PP_2
Data Collected on:
IT70-NMR-mercury400
Archive directory:

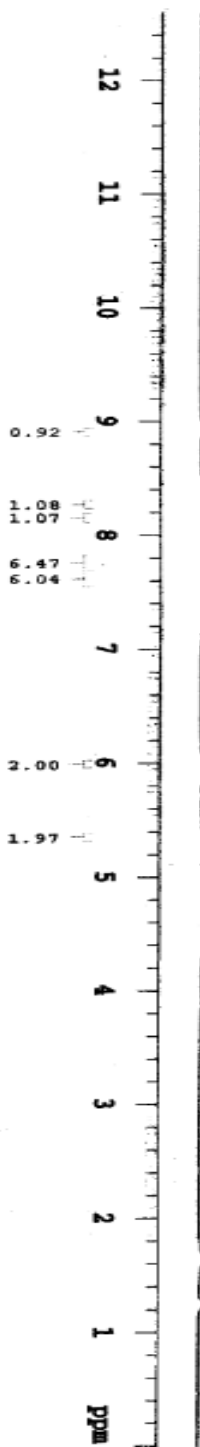
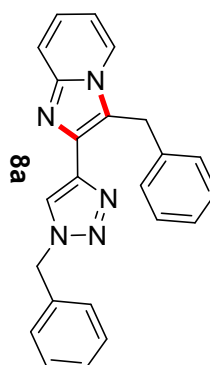
Sample directory:

FIDFile: PROTON

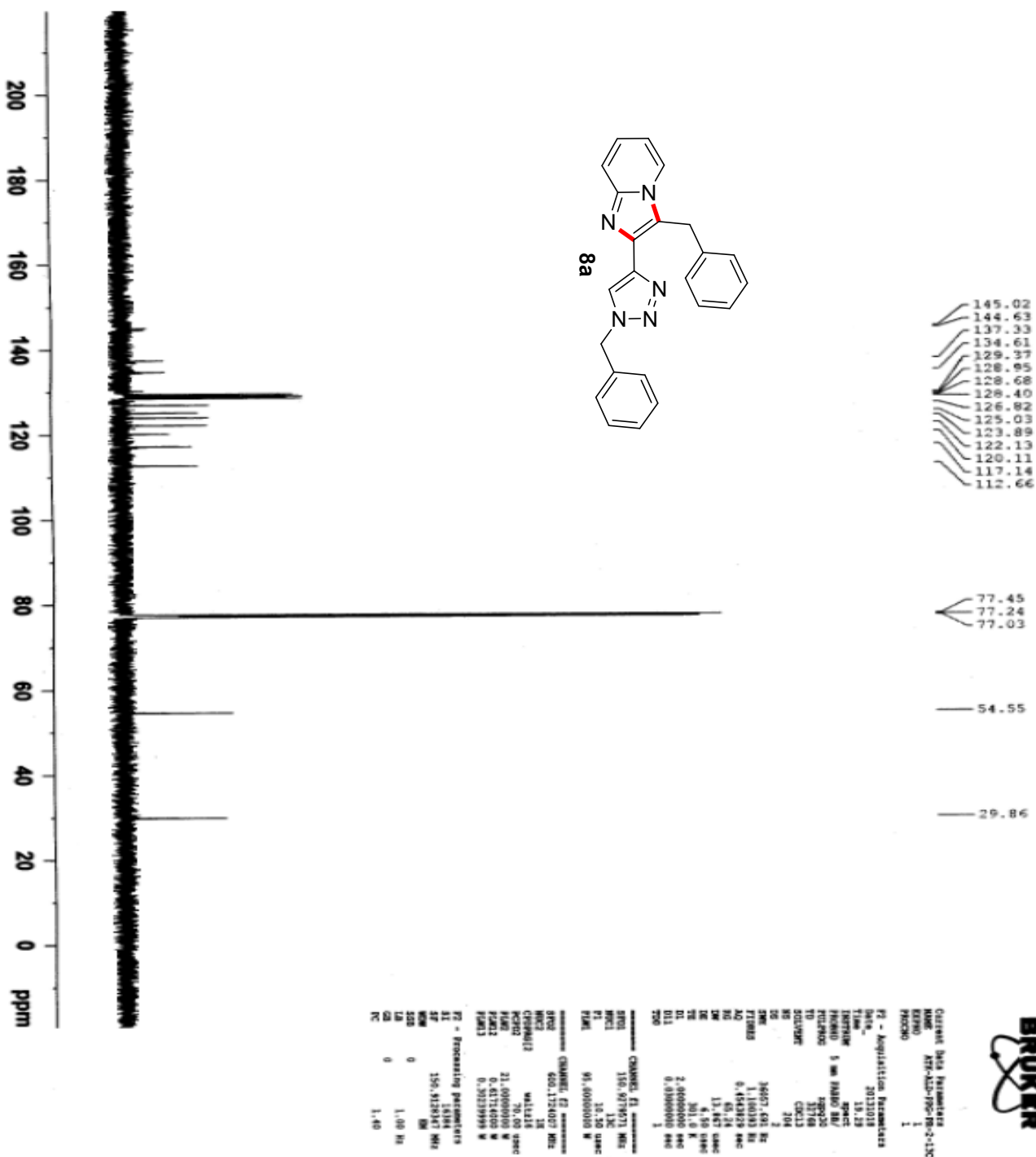
Pulse Sequence: PROTON (zgpg3)
Solvent: cdcl3
Data collected on: Mar 20 2014

Temp: 25.0 C / 298.1 K
Operator: cham

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
OBSERVE HI, 399.850383 MHz
DATA PROCESSING
PT size 32768
Total time 2 min 12 sec

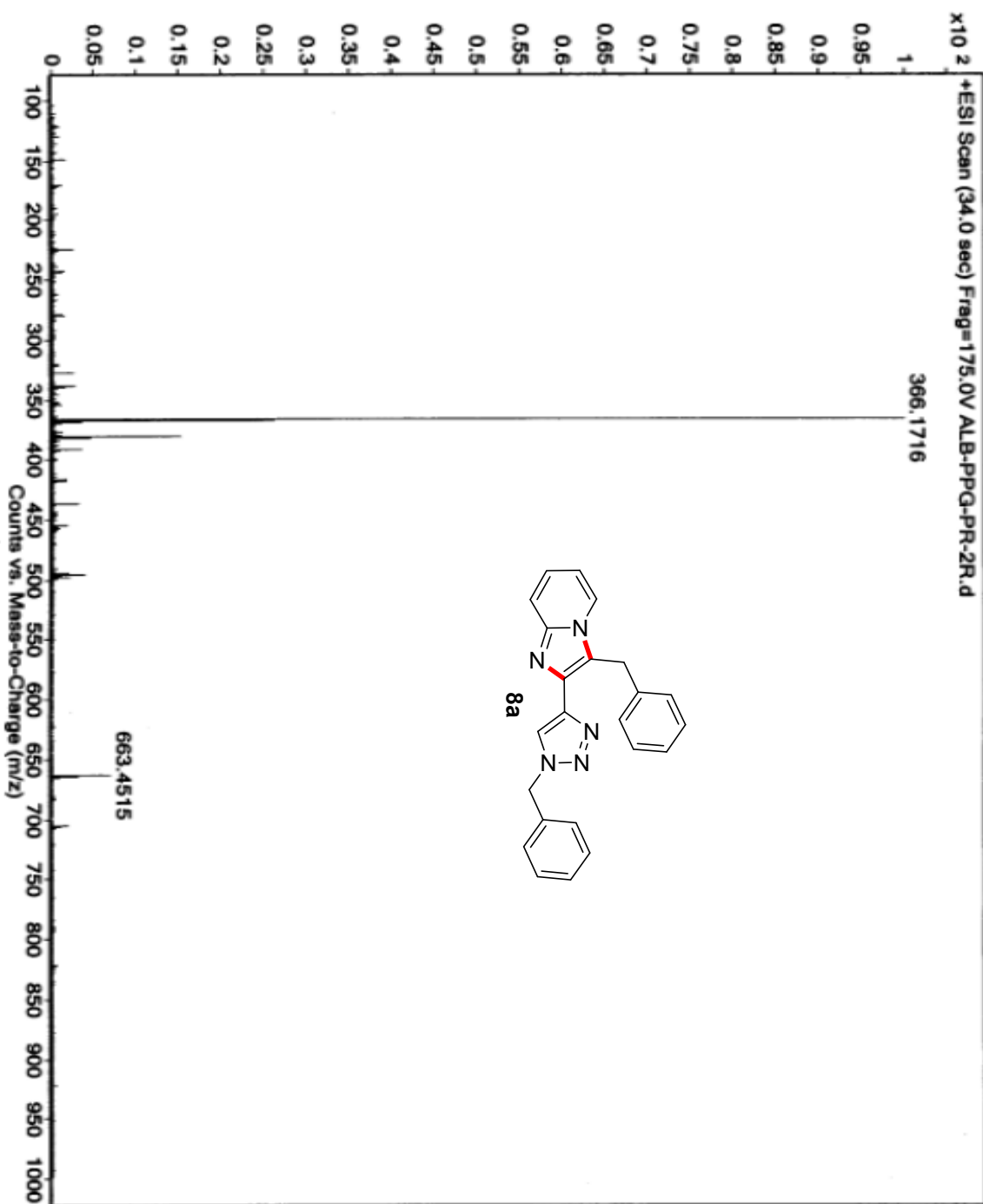


¹³C NMR spectra of **8a**

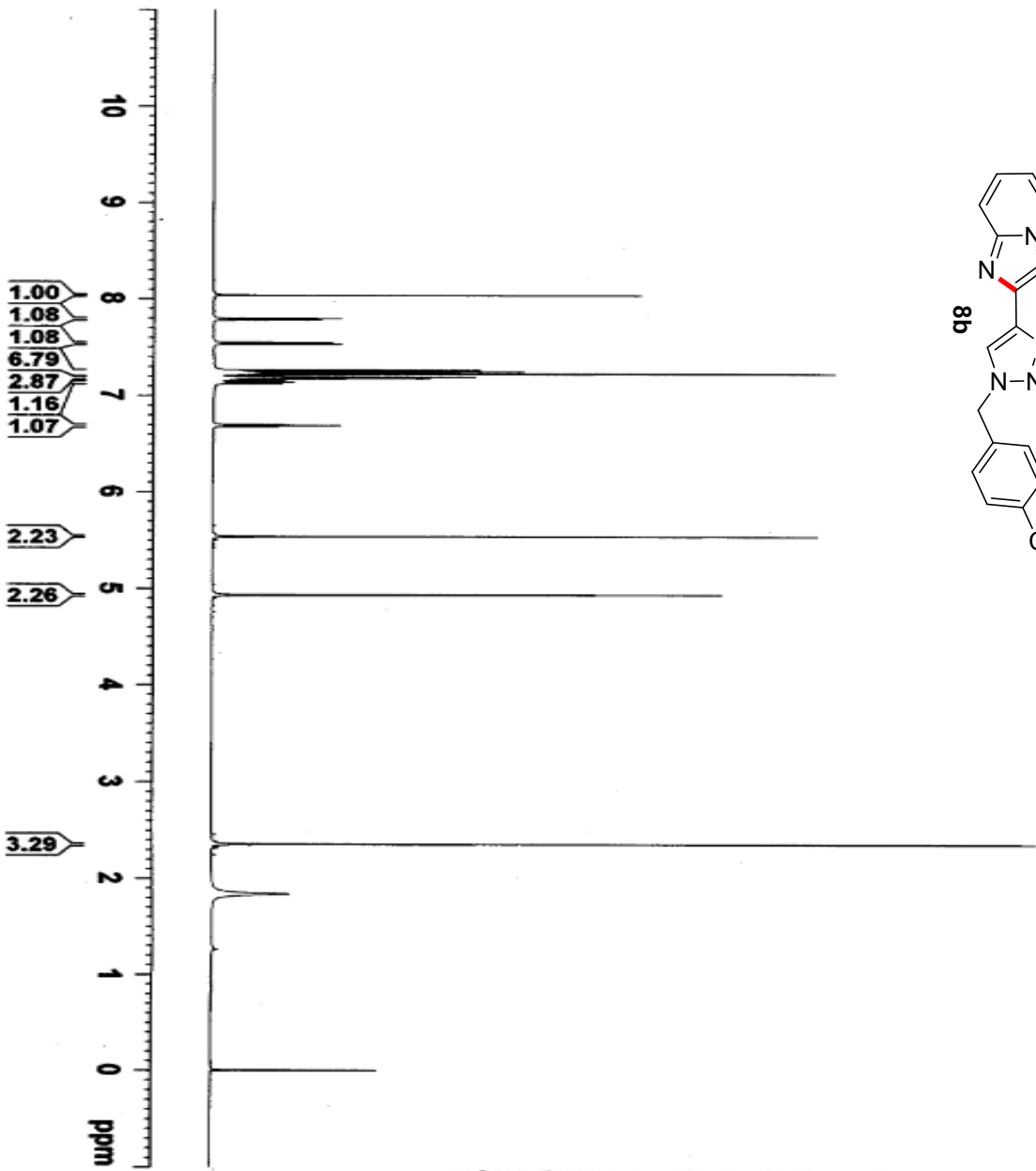
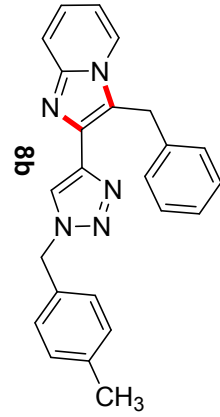


HRMS spectra of 8a

Sample Name ALB-PPG-PR-2R Position -1 Instrument Name Instrument 1 User Name
 Inj Vol -10 InjPosition ACQ Method Comment SampleType Sample Sample IRM Calibration Status Success
 Data Filename ALB-PPG-PR-2R.d



¹H NMR spectra of **8b**



Current Data Parameters
 NAME ALD-PPG-PN-17_1H
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140904
 Time 14.56
 INSTRUM spect
 PROBD 5 mm PABBO BB/
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 89.67
 DW 41.600 usec
 DE 6.50 usec
 TE 299.8 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 600.1737063 MHz
 NUC1 1H
 P1 12.00 usec
 PL1 21.00000000 W

F2 - Processing parameters
 SI 16384
 SF 600.1700141 MHz
 MDW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR spectra of 8b

Sample Name:
ALD-PRG-17-13C
Data Collected on:
11/19/2014
Archive directory:

Sample directory:

Yield: ALD-PRG-17-13C

Pulse Sequence: CARRON (4pul)

Solvent: cdcl3

Data collected on: Jan 29 2014

Temp: 25.0 C / 298.1 K

Operator: chm

Relax. delay 1.000 sec

Pulse 45.0 degree

Acq. time 1.304 sec

Width 25125.6 Hz

270 repetitions

OBSERVE C13, 100.6425909 MHz

DECOUPLE H1, 399.8529994 MHz

Power 42 dB

continuously on

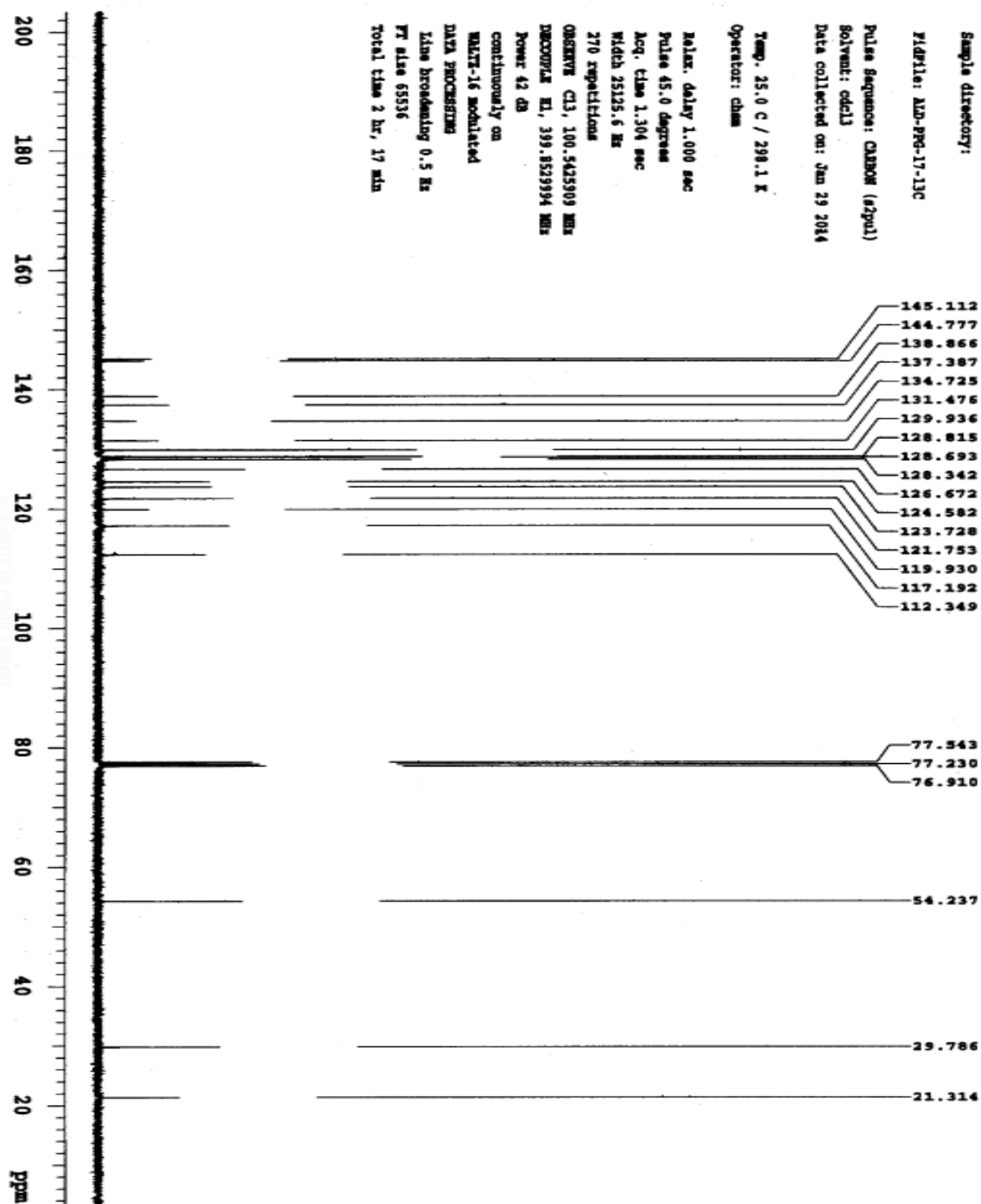
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

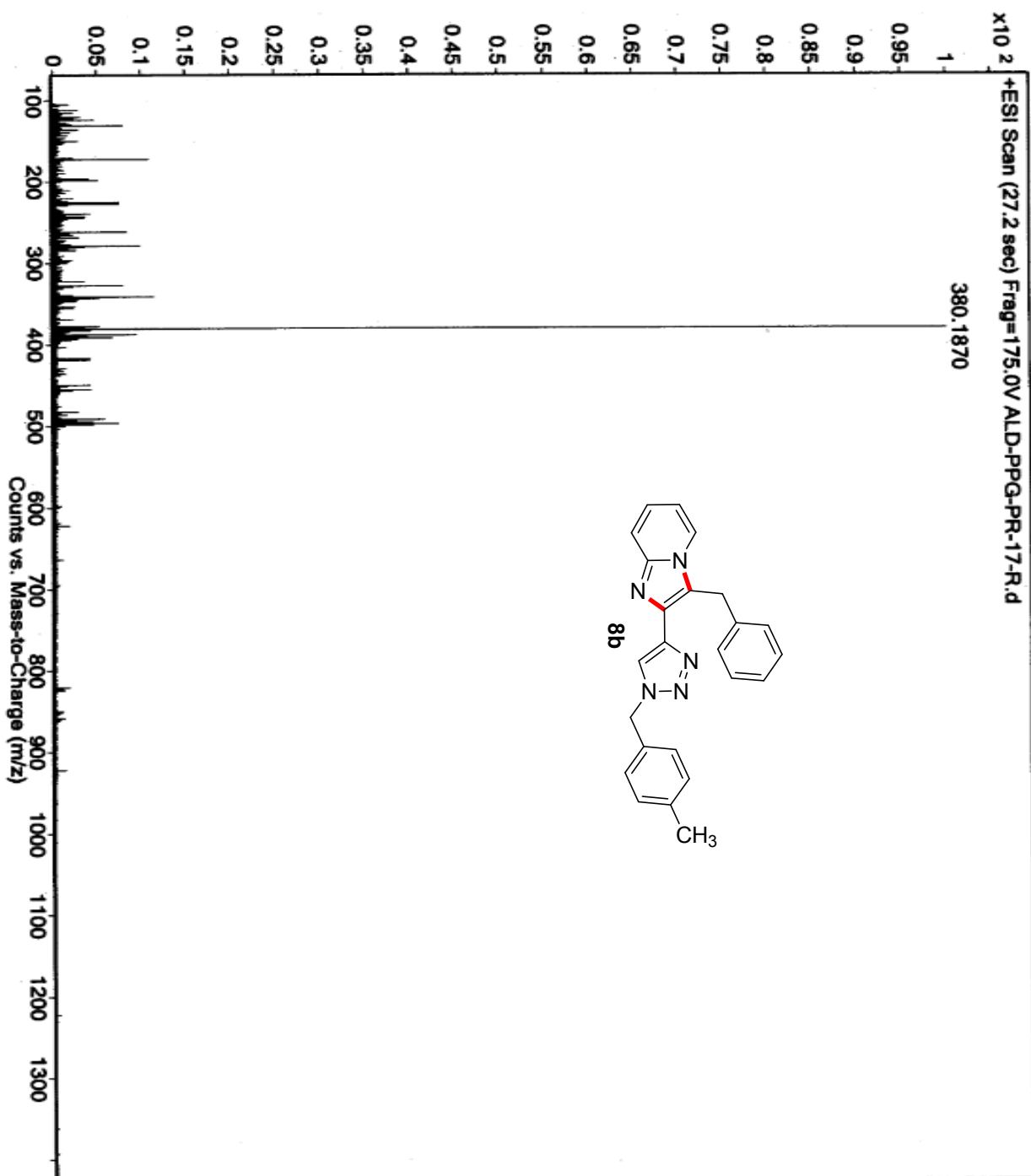
FT axis 65536

Total time 2 hr, 17 min



HRMS spectra of 8b

Sample Name	ALD-PPG-PR-17-R	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	Inj Position		Sample Type	Sample	IRM Calibration Status	Success
Data Filename	ALD-PPG-PR-17-R.d	Acq Method		Comment		Acquired Time	3/18/2014 2:35:40 PM

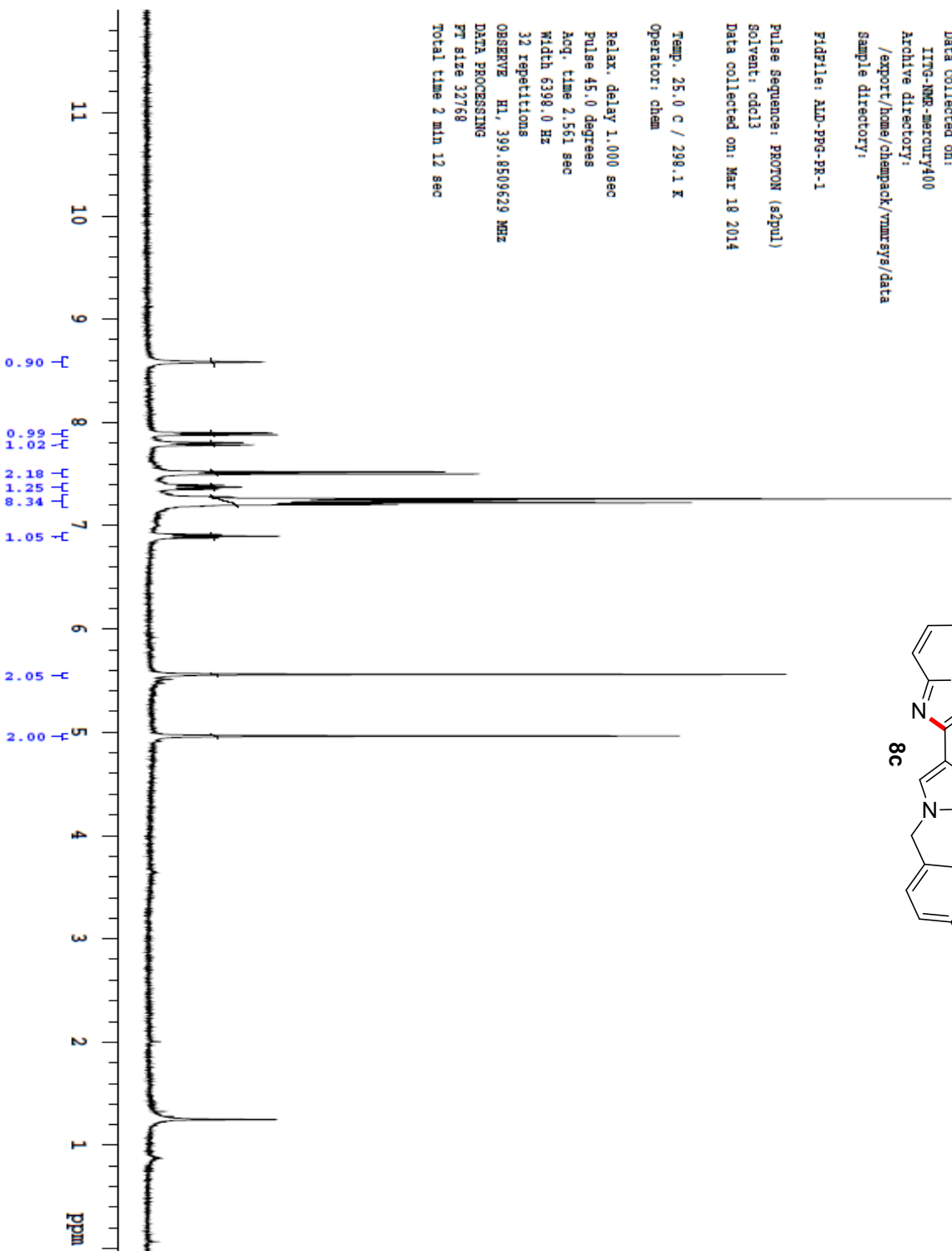
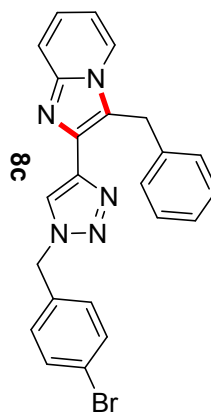


¹H NMR spectra of **8c**

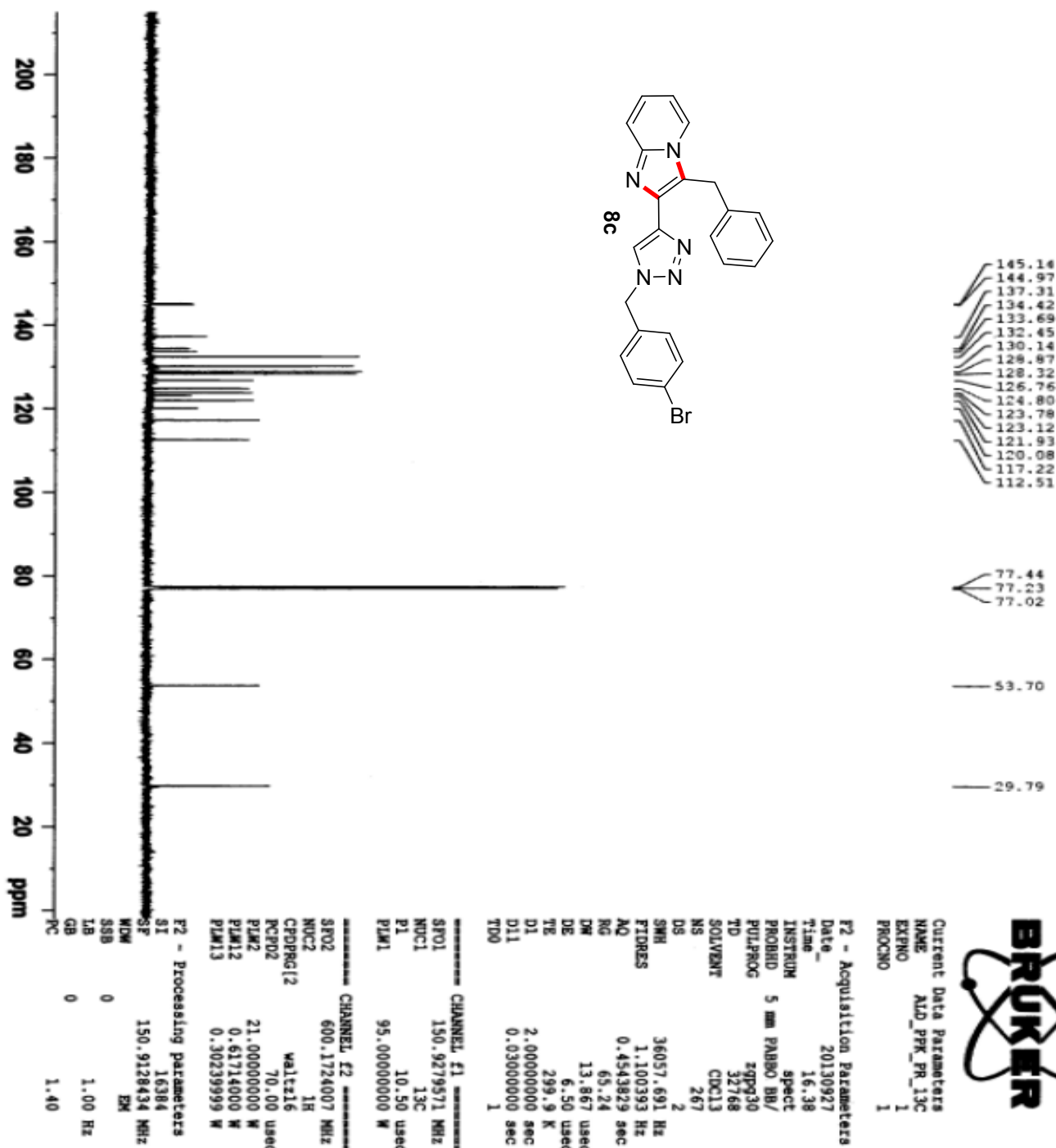
ALD-PPG-PR-1
Sample Name:
ALD-PPG-PR-1
Data Collected on:
ITG-NMR-mercury400
Archive directory:
/export/home/chempack/vmmrsvs/data
Sample directory:

FIDFile: ALD-PPG-PR-1
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 18 2014

Temp. 25.0 C / 298.1 K
Operator: chem
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.850929 MHz
DATA PROCESSING
PT size 32768
Total time 2 min 12 sec

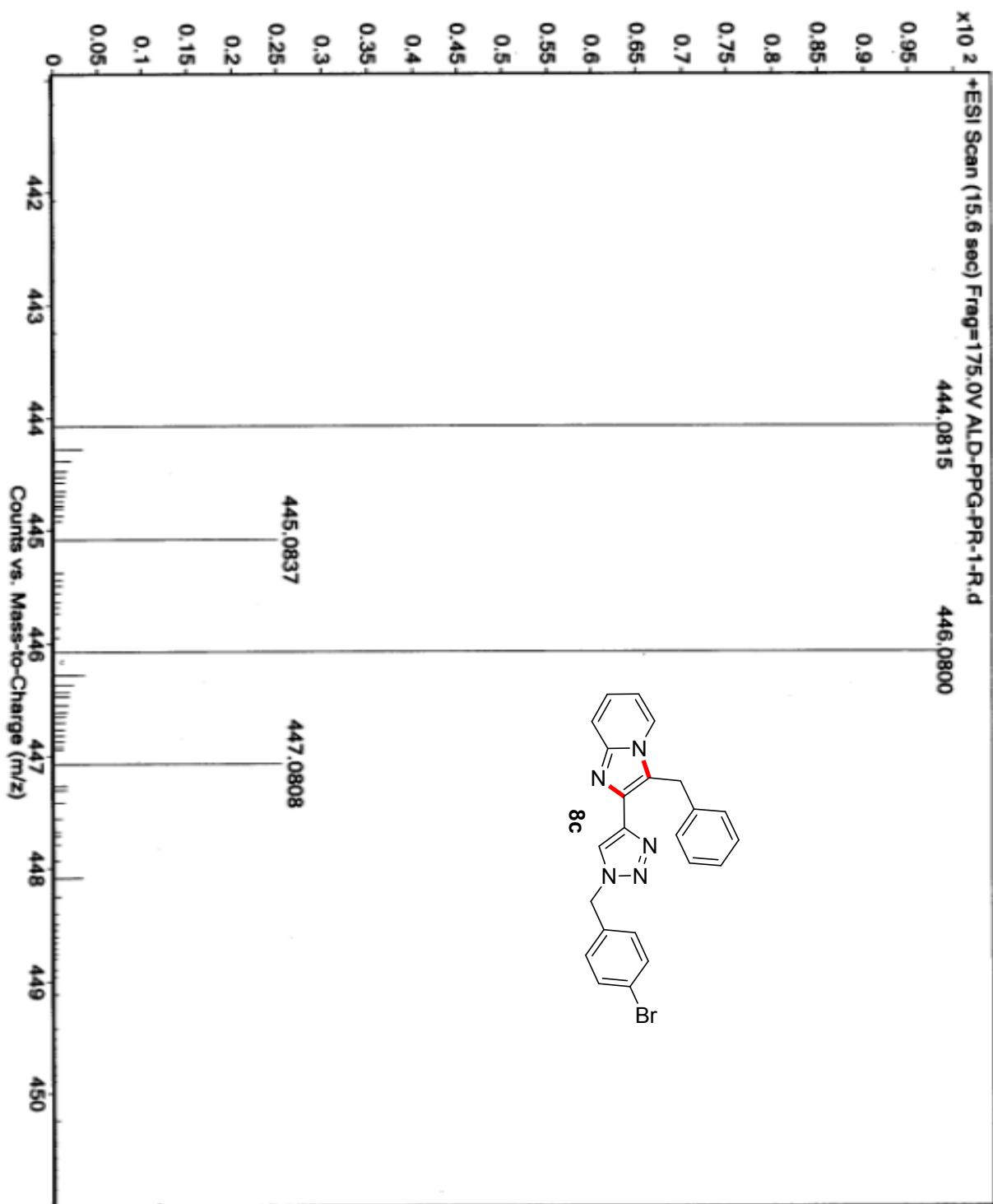


¹³C NMR spectra of **8c**

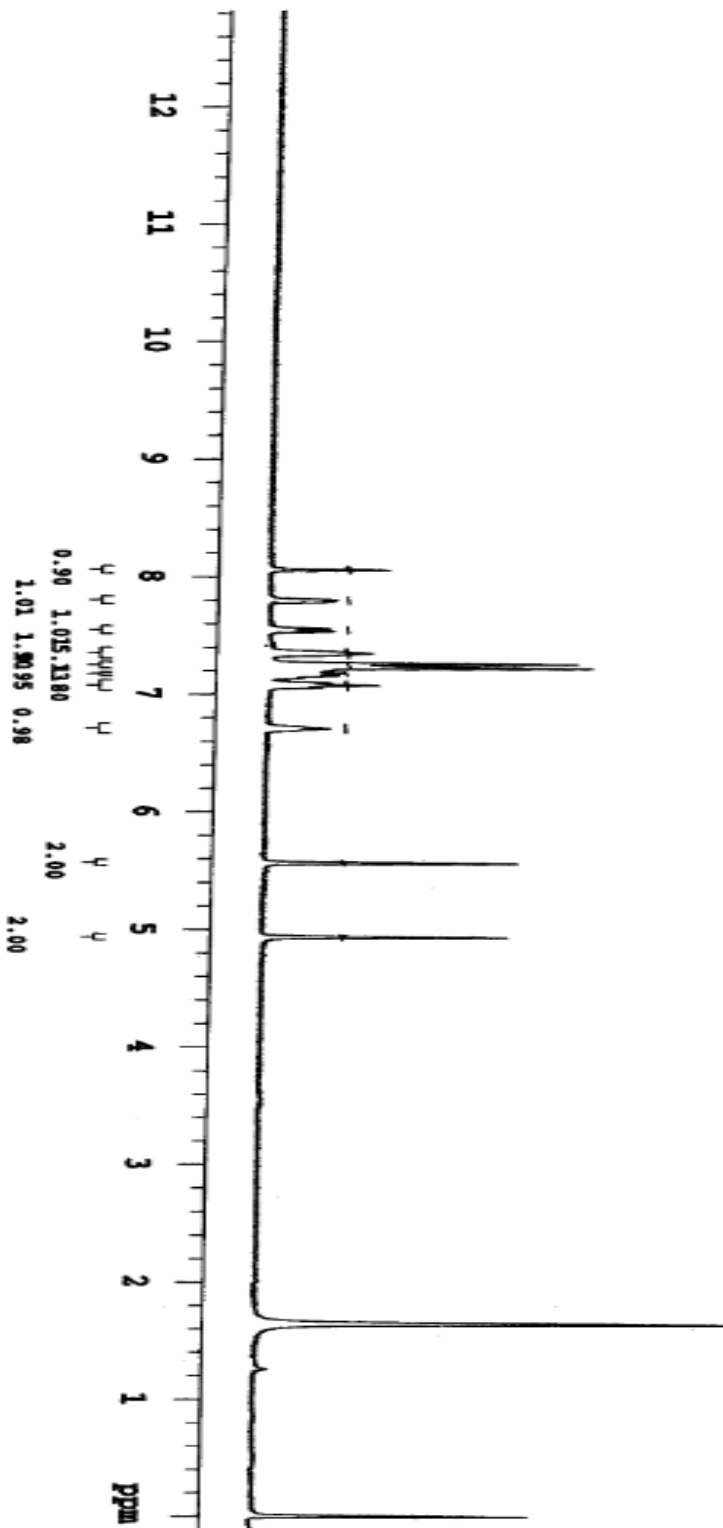
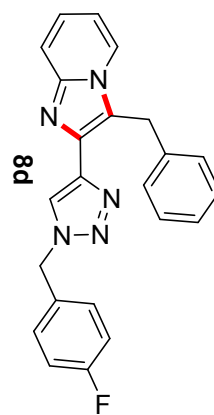


HRMS spectra of 8c

Sample Name	ALD-PPG-PR-1-R	Position	-1	Instrument Name	Instrument 1	User Name	Success
Inj Vol	-10	InjPosition		SampleType	Sample	ITM Calibration Status	
Data Filename	ALD-PPG-PR-1-R.d	Acq Method		Comment		Acquired Time	3/18/2014 2:43:23

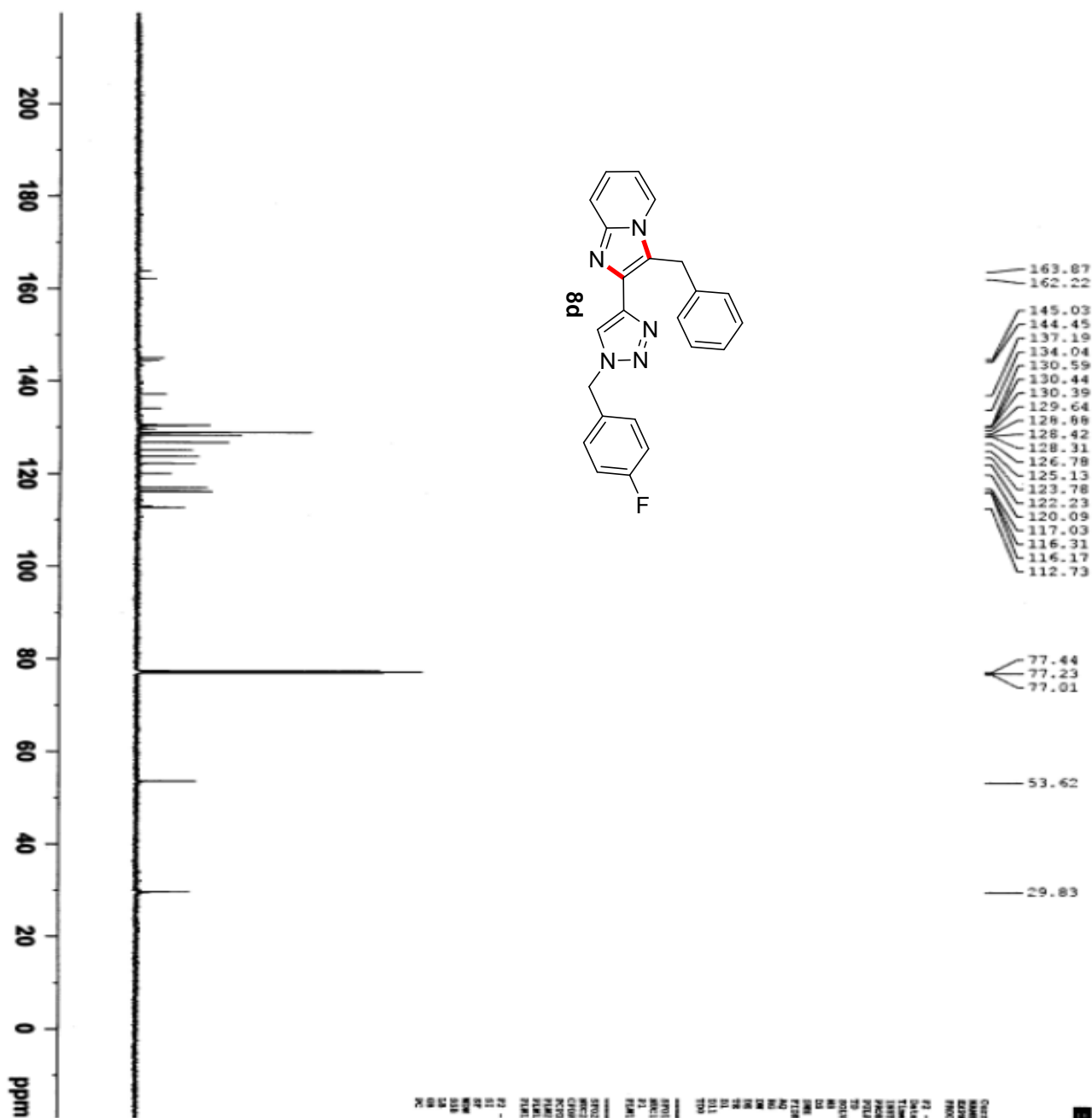


¹H NMR spectra of 8d



PULSE PROGRAM : 138P2 Relax. delay 1.000 sec Pulse 45.0 degree Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 319.8509629	DATA PROCESSING FT also 32768 Total time 1 minutes	ALD-PPQ-PR-5 Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chuan File: ALD-PPQ-PR-5_PROTON_01 Mercury-400 *ITV-NMR*
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¹³C NMR spectra of **8d**

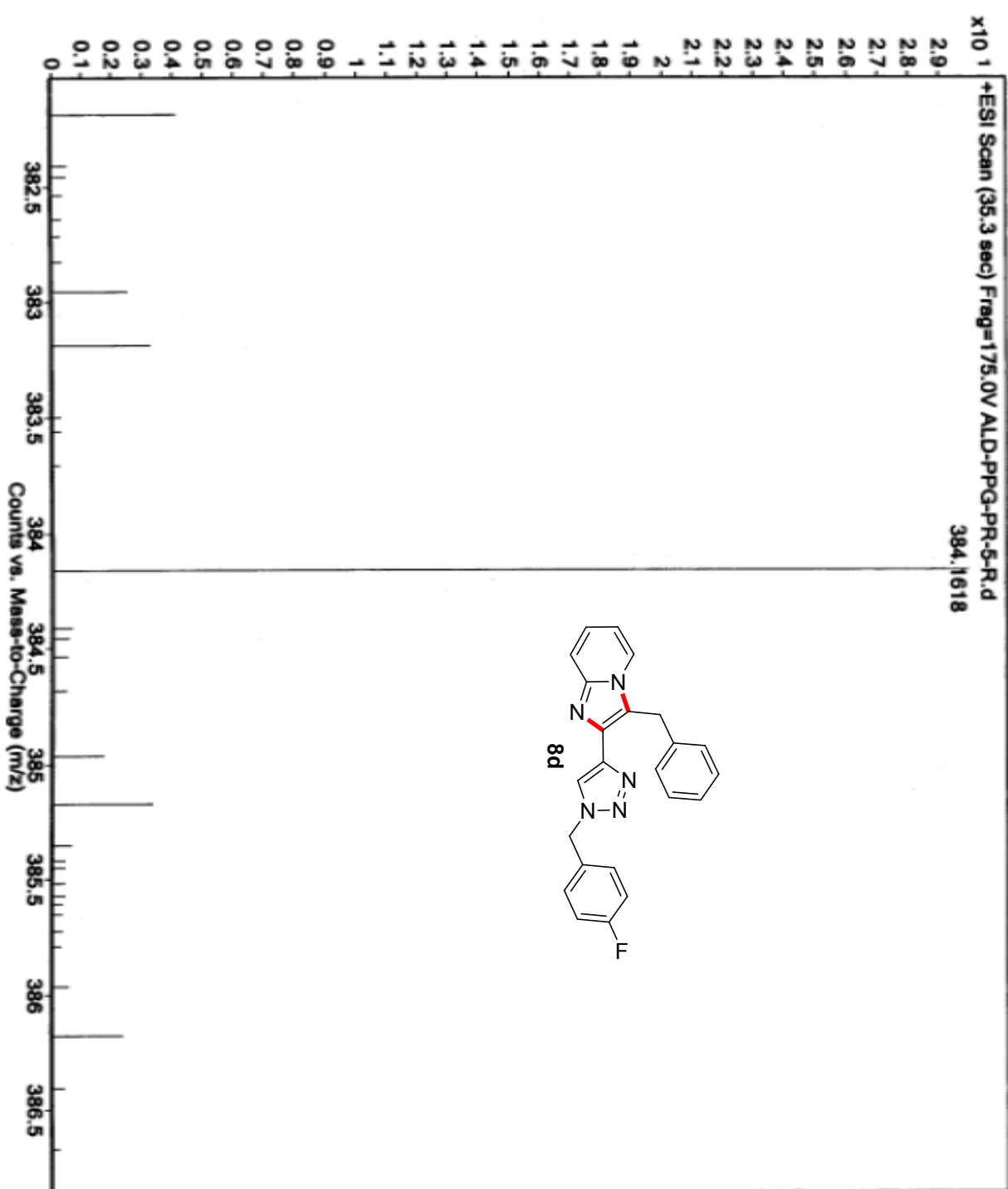


Current Data Parameters
 Name: 20131021
 Date_: 20131021
 Time: 20:57
 Acquisition: 5 mm HASTE
 Processing: 17548
 F2 - Acquisition Parameters
 Date_: 20131021
 Time: 20:57
 Acquisition: 5 mm HASTE
 Processing: 17548
 F2 - Processing parameters
 SI: 16384
 SF: 125.517425 MHz
 AQ: 0.50000000 sec
 FIDRES: 0.120229989 Hz
 AQRES: 0.120229989 Hz
 CHANNEL f1
 NUC1: 13C
 P1: 12.00 usec
 PL1: 0.00000000 W
 CHANNEL f2
 NUC2: 1H
 P2: 70.00 usec
 PL2: 0.00000000 W
 PL12: 0.43148000 W
 PL123: 0.20229989 W
 SI: 16384
 SF: 125.517425 MHz
 AQ: 0.50000000 sec
 FIDRES: 0.120229989 Hz
 AQRES: 0.120229989 Hz



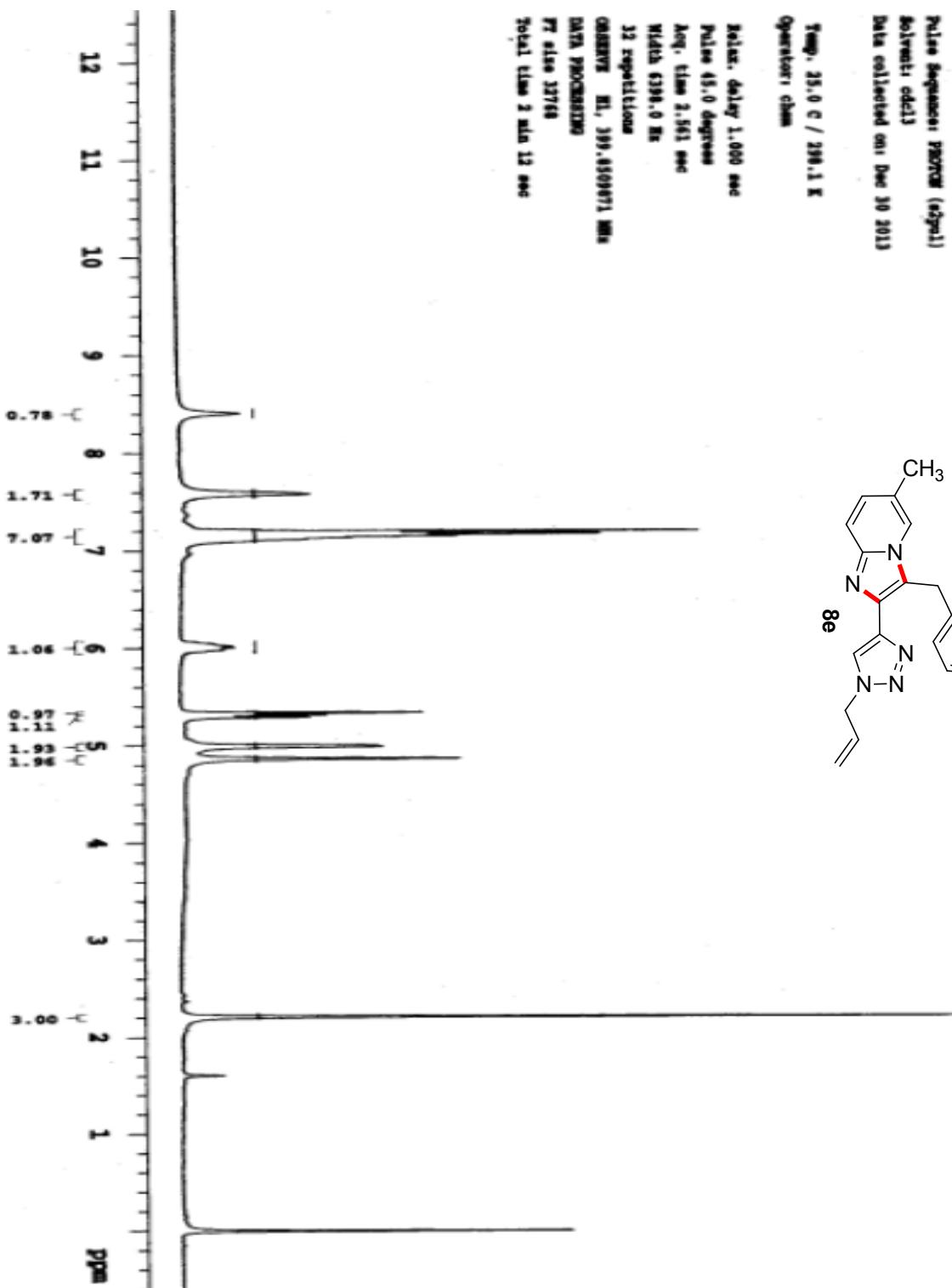
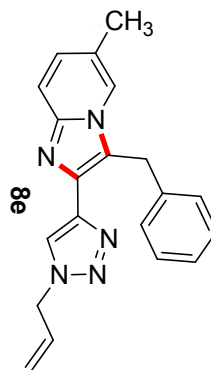
HRMS spectra of 8d

Sample Name	ALD-PPG-PR-5-R	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	RM Calibration Status	Success
Data Filename	ALD-PPG-PR-5-R.d	Acq Method		Comment		Acquired Time	3/18/2014 2:37:12 PM

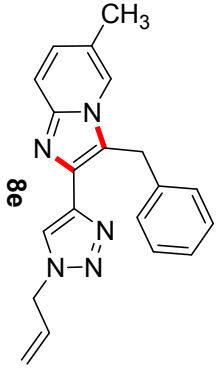
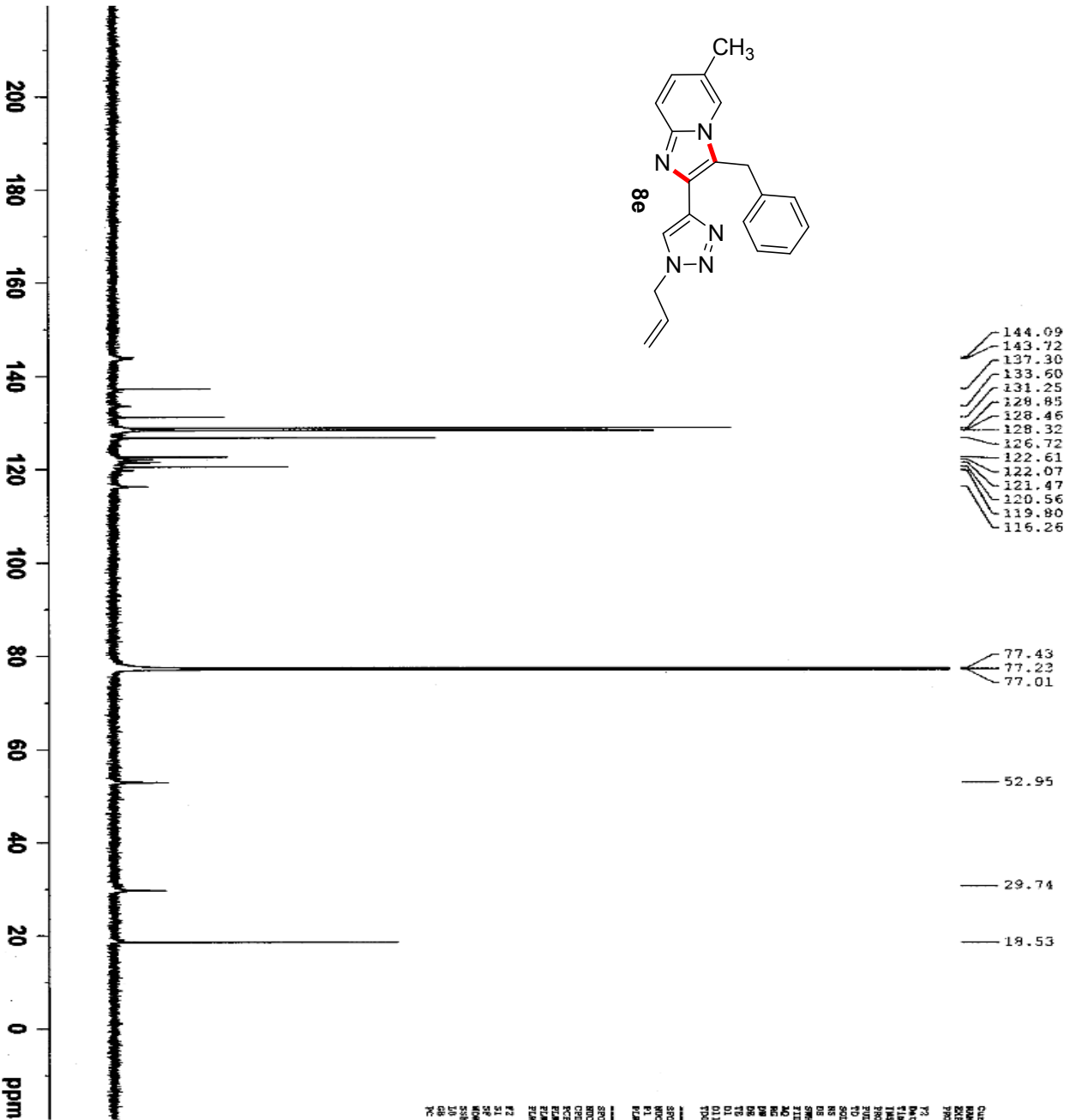


¹H NMR spectra of 8e

Sample Name: ALD-PP9-PP-14
Data Collected on: 11/9-2013-mercury400
Acquire directory:
Sample directory:
F1/DIR1: PP9PP
Pulse sequence: PP9PP (zgpg3)
Solvent: cdcl3
Data collected on: Dec 30 2013
Temp: 25.0 C / 298.1 K
Operator: chm
Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 2.561 sec
Width: 6398.0 Hz
32 repetitions
CONVERT HI, 399.4509871 MHz
DATA PROCESSING
PT file: 23768
Total time: 2 min 12 sec



¹³C NMR spectra of **8e**



- 144.09
- 143.72
- 137.30
- 133.60
- 131.25
- 129.85
- 128.46
- 128.32
- 126.72
- 122.61
- 122.07
- 121.47
- 120.56
- 119.80
- 116.26

- 77.43
- 77.23
- 77.01

- 52.95

- 29.74

- 18.53

Current Data Parameters
 Name ALI-PR-78-148_13C
 ExpNo 1
 ProcNo 1

% - Acquisition Parameters
 Date_ 20140113
 Time 13.44
 Instrument spect
 Processor 5 m PABBO 817
 F2 31148
 F1 31148
 TD 32768
 SFO 150
 SOLVENT CDCl3
 NS 1348
 DS 2
 SWH 36031.651 Hz
 FWHM 1342.323 Hz
 AQ 0.444132 sec
 RG 63.24
 PW 13.887 usec
 DE 6.50 usec
 TE 300.2 K
 D1 2.0000000 sec
 D11 0.3000000 sec
 TPO

===== CHANNEL f1 =====
 SFO1 150.9279571 MHz
 WC1 1K
 FID 16.26 usec
 FFL1 95.0000000 M

===== CHANNEL f2 =====
 SFO2 600.1724007 MHz
 WC2 1K
 FID 16.26 usec
 FFL2 95.0000000 M

===== CHANNEL f3 =====
 SFO3 600.1724007 MHz
 WC3 1K
 FID 16.26 usec
 FFL3 95.0000000 M

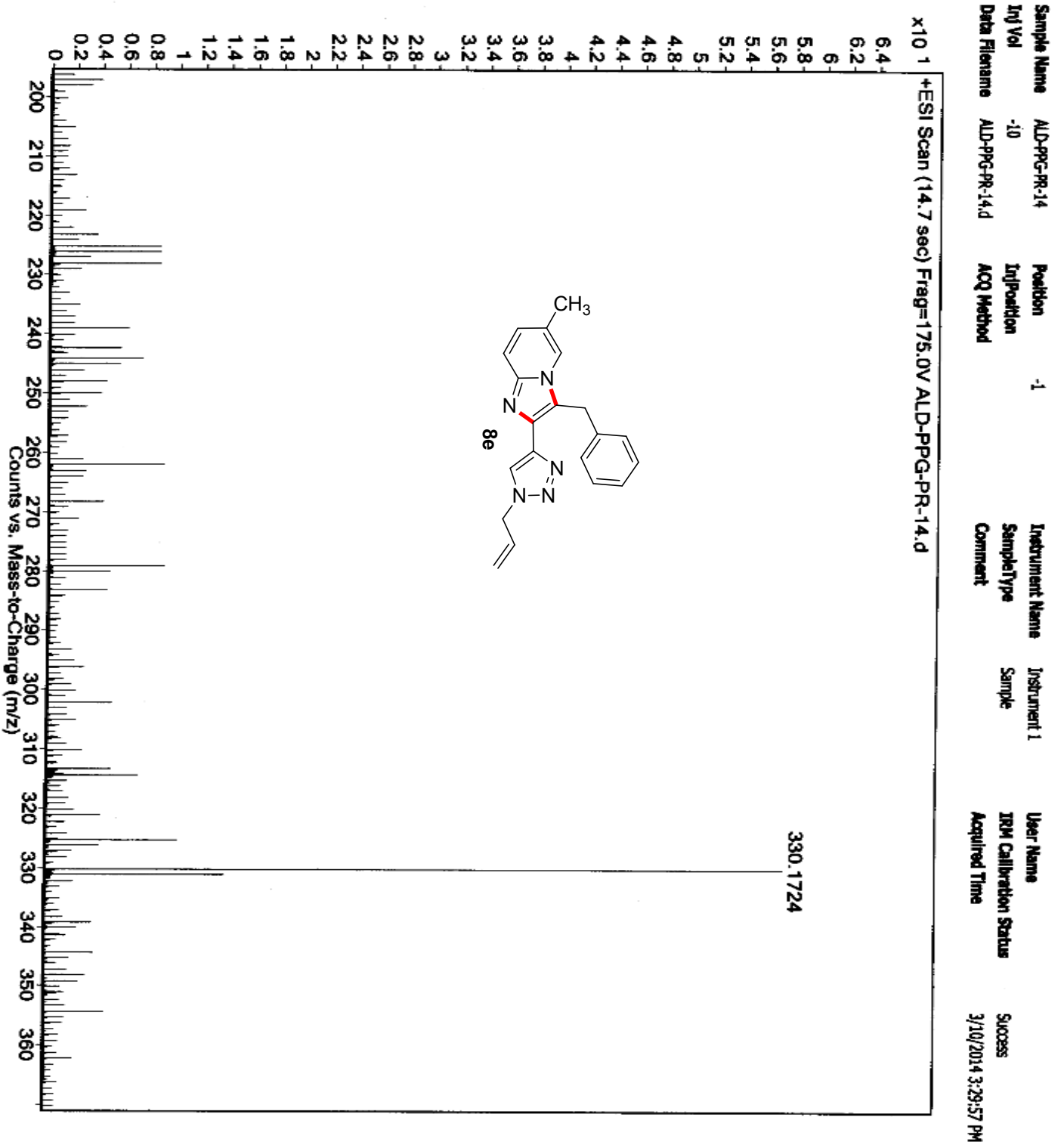
===== CHANNEL f4 =====
 SFO4 600.1724007 MHz
 WC4 1K
 FID 16.26 usec
 FFL4 95.0000000 M

===== CHANNEL f5 =====
 SFO5 600.1724007 MHz
 WC5 1K
 FID 16.26 usec
 FFL5 95.0000000 M

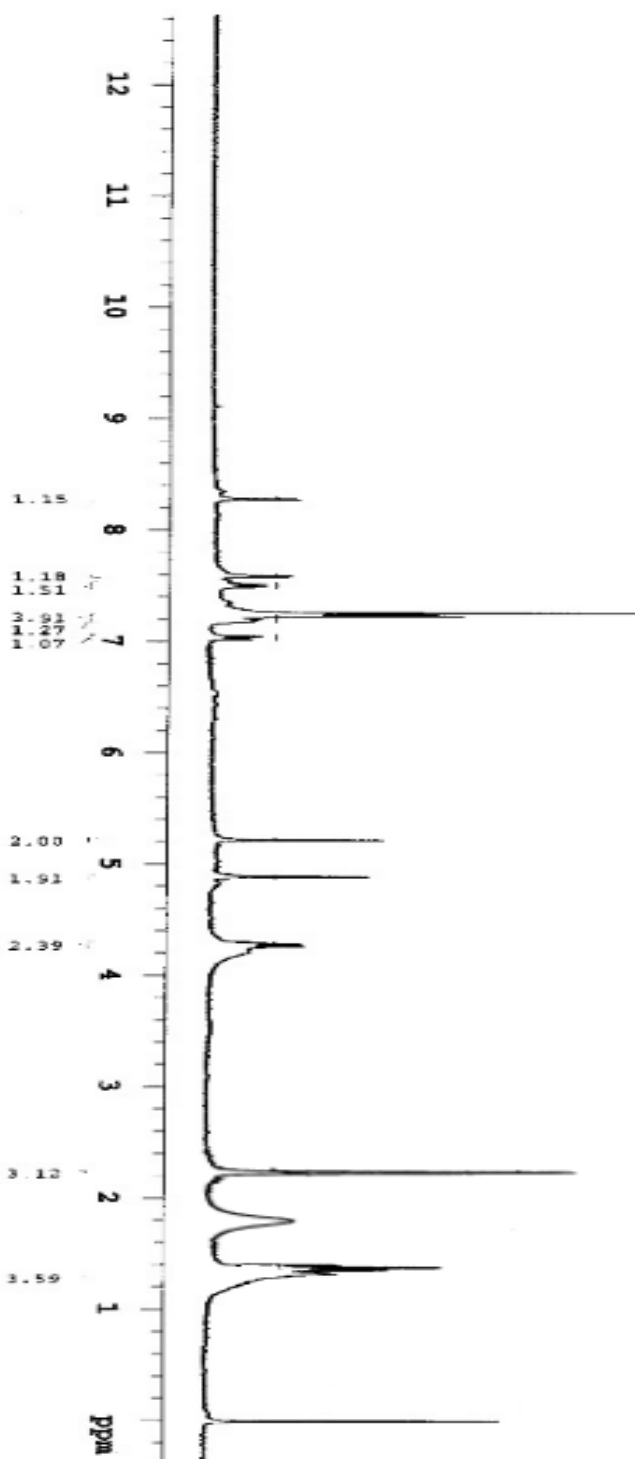
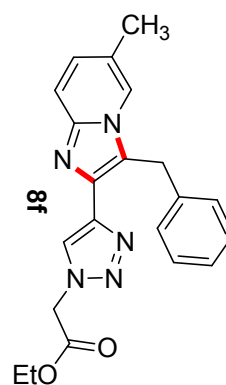
F2 - Processing parameters
 SI 150.9124477 MHz
 SF 600.1724007 MHz
 WDW EM
 SSB 0
 GB 0
 PC 1.40



HRMS spectra of 8e



¹H NMR spectra of **8f**

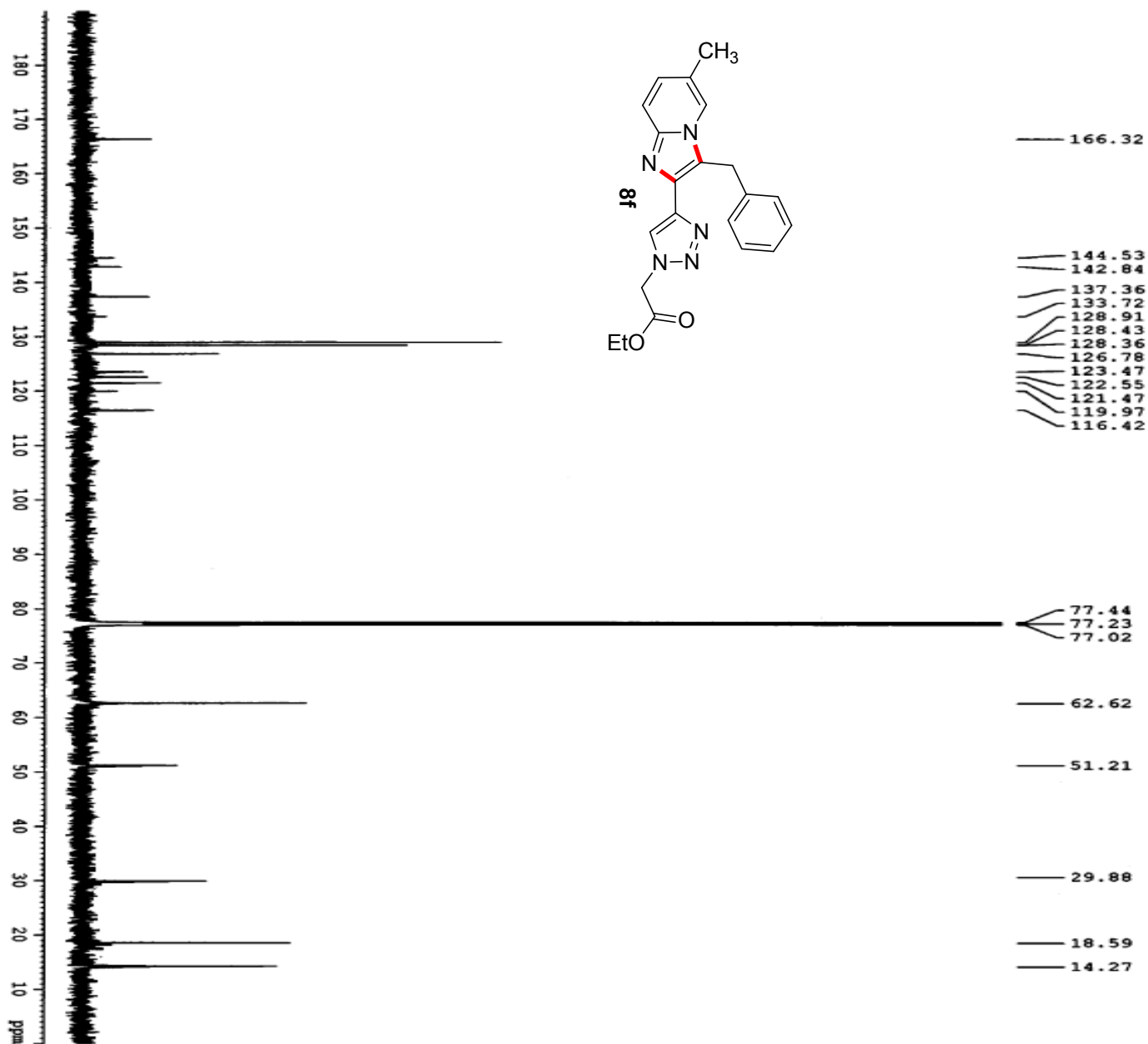


INSTRUMENT: spect
 Relax. delay 1.000 sec
 Pulse 45.0° degrees
 Acq. time 2.561 sec
 Width 6398.0 Hz
 32 repetitions

DATA PROCESSING
 FT size 32768
 Total time 1 minutes

ALD-PRO-25-26
 Solvent: cdcl3
 Temp: 25.0 C / 298.1 K
 Operator: chm
 File: ALD-PRO-25
 Mercury-400 1170-MHz

¹³C NMR spectra of **8f**



Current Data Parameters
 NAME ALD-PE-24_13C
 EXNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160213
 Time_ 13.04
 INSTRUM spect
 PROBO 5 mm BBBO DM/
 PULPROG zgpg30
 TD 32768
 SFO 400.146
 SOLVENT CDCl3
 NS 1001
 DS 2
 SWE 36051.691 Hz
 FIDRES 1.100393 Hz
 AQ 0.454829 sec
 RG 65.24
 DW 13.867 usec
 DE 6.50 usec
 TE 299.4 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

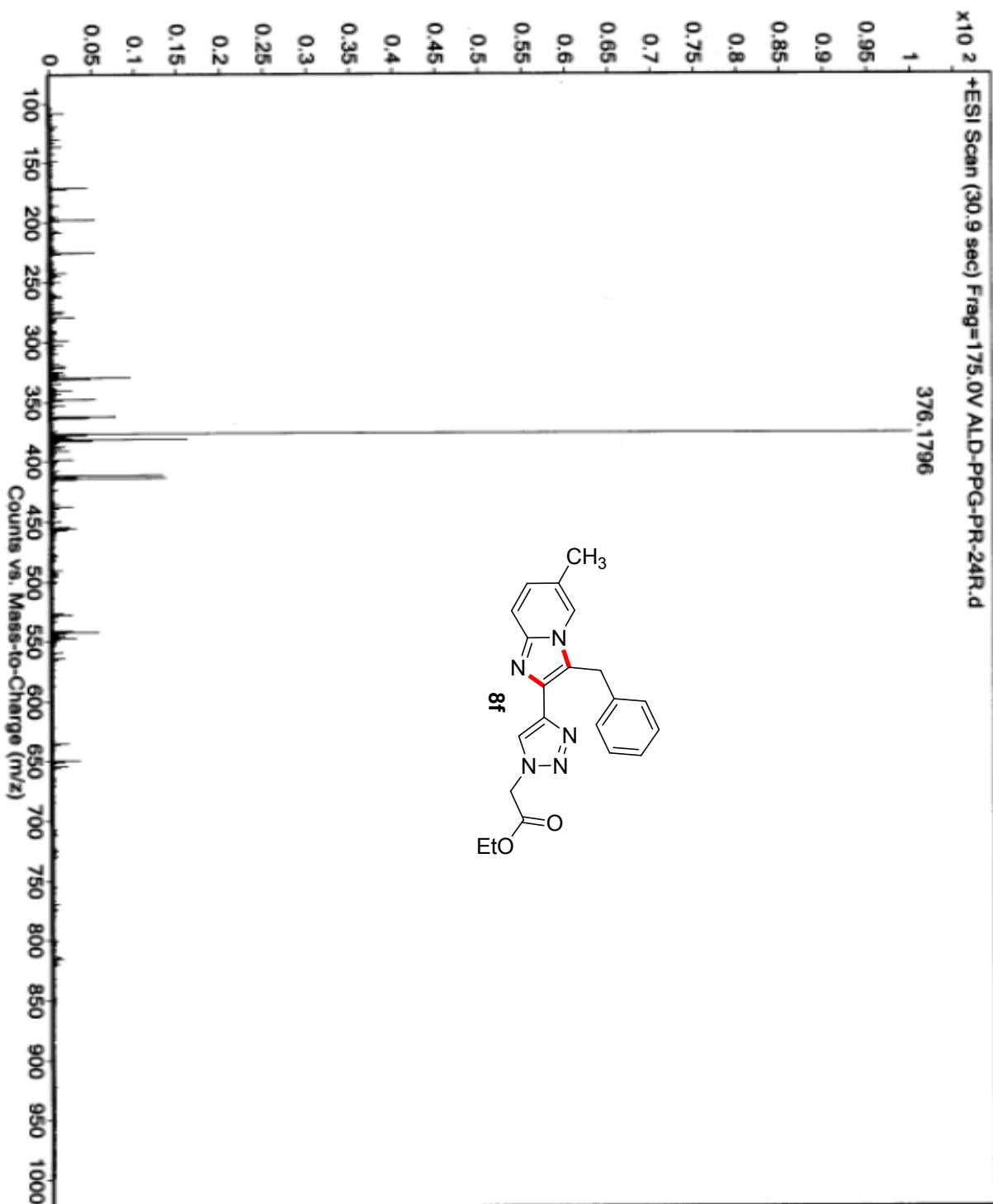
***** CHANNEL f1 *****
 SFO1 150.9279571 MHz
 NUC1 13C
 P1 10.50 usec
 PLM1 95.0000000 W

***** CHANNEL f2 *****
 SFO2 600.1724007 MHz
 NUC2 1H
 CPROG12 waltz16
 PCPD2 70.00 usec
 PLM2 21.0000000 W
 PLM12 0.61714000 W
 PLM13 0.30239999 W

F2 - Processing parameters
 SI 16384
 SF 150.9128373 MHz
 KW 64
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

HRMS spectra of 8f

Sample Name	ALD-PPG-PR-24R	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	ALD-PPG-PR-24R.d	Acq Method		Comment		Acquired Time	3/21/2014 11:25:17 AM



¹H NMR spectra of 8g

ALD-PPQ-PR-10

Sample Name:
ALD-PPQ-PR-10
Data Collected on:
11/29/2013
Acquire directory:
/export/home/olimpak/vmmyy/data
Sample directory:

FIDFile: PROTON

Pulse sequence: PROTON (zgpg3)

Solvent: cdcl3

Data collected on: Dec 4 2013

Temp: 25.0 C / 298.1 K

Operator: chem

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.561 sec

Width 6398.0 Hz

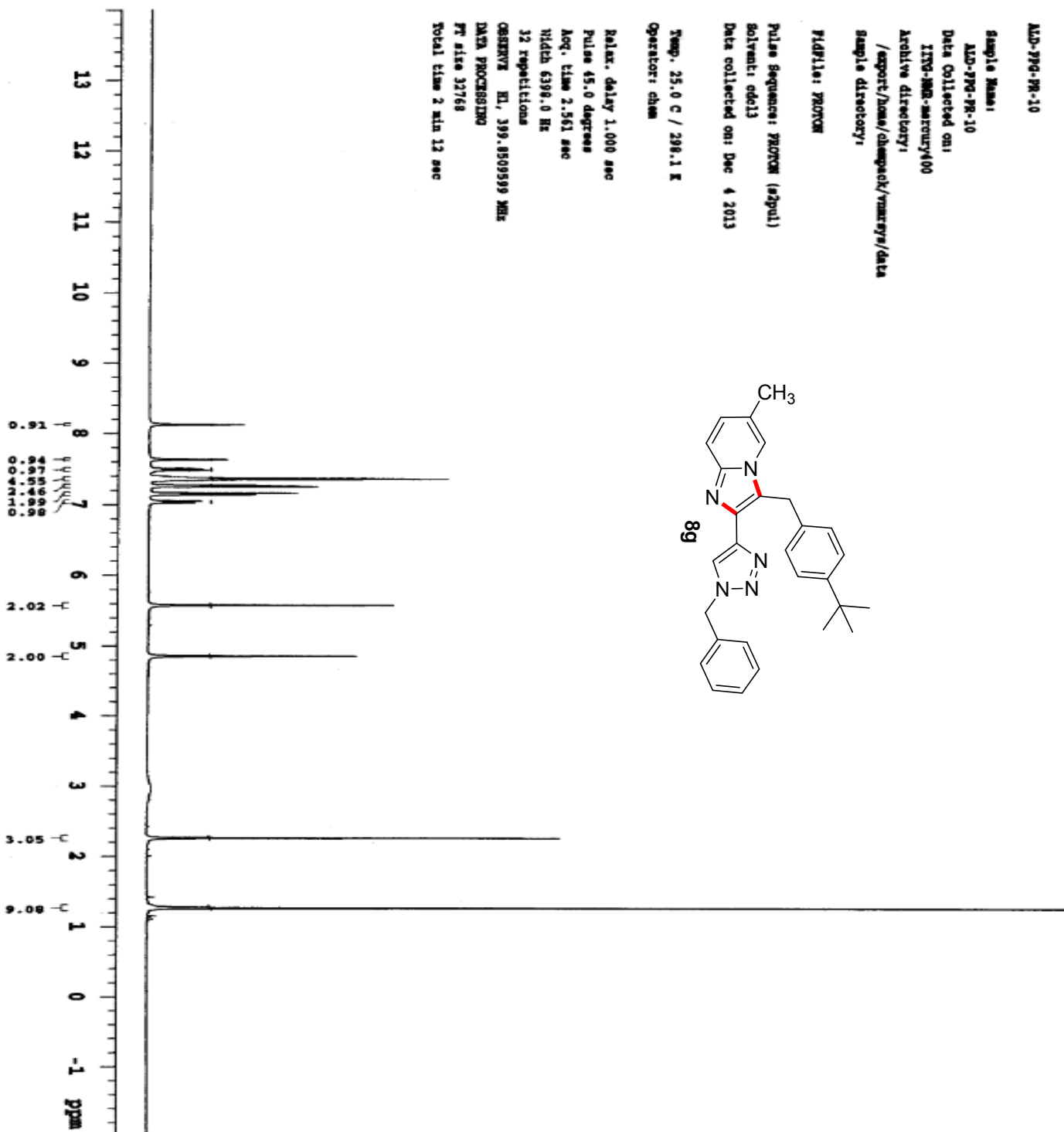
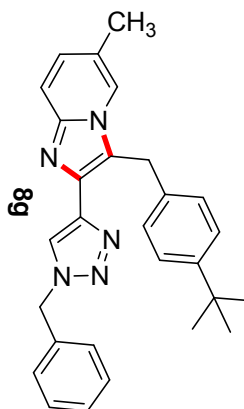
32 repetitions

OBSERVE H1, 399.9509399 MHz

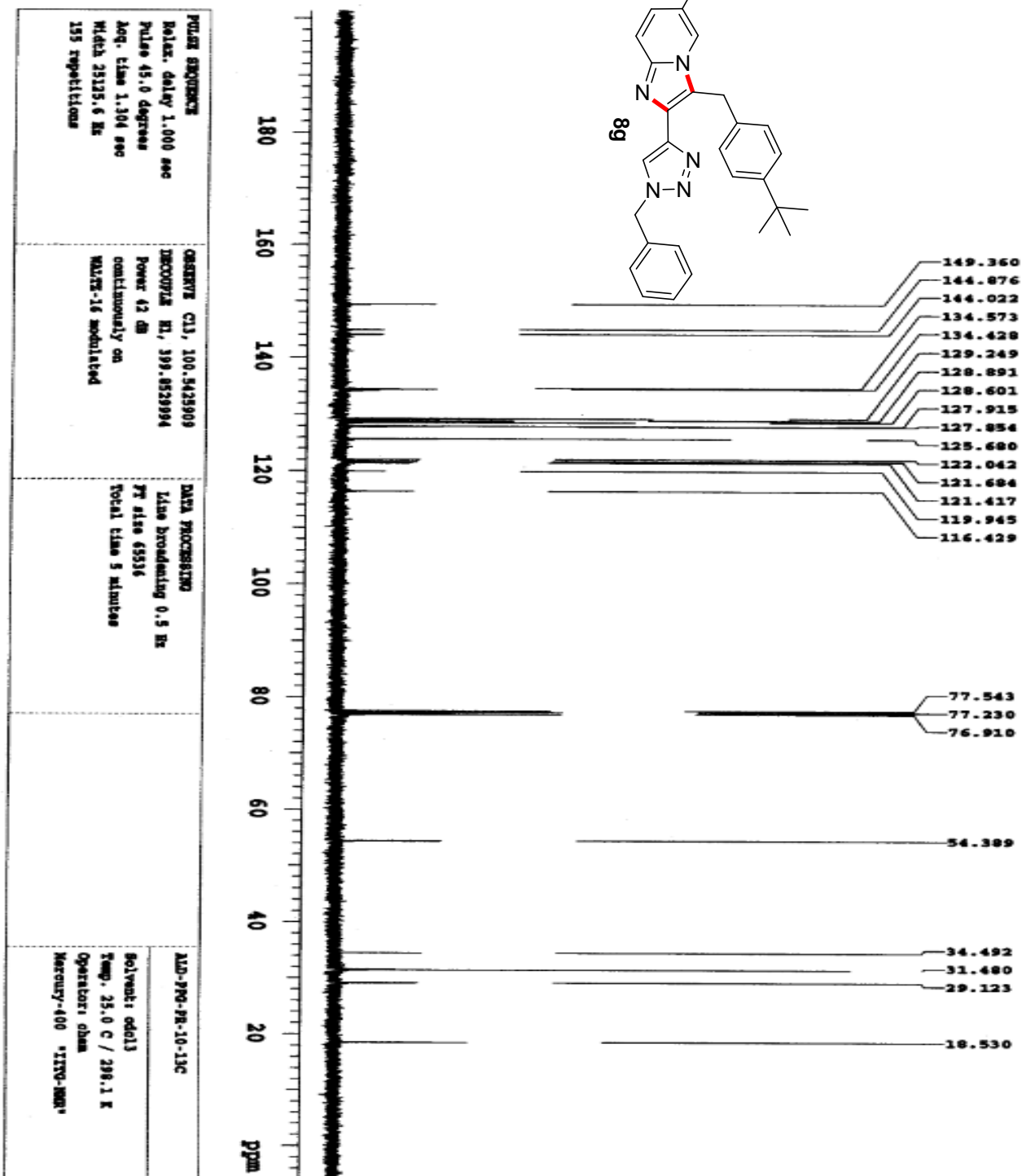
DATA PROCESSED

PR size 32768

Total time 2 min 12 sec

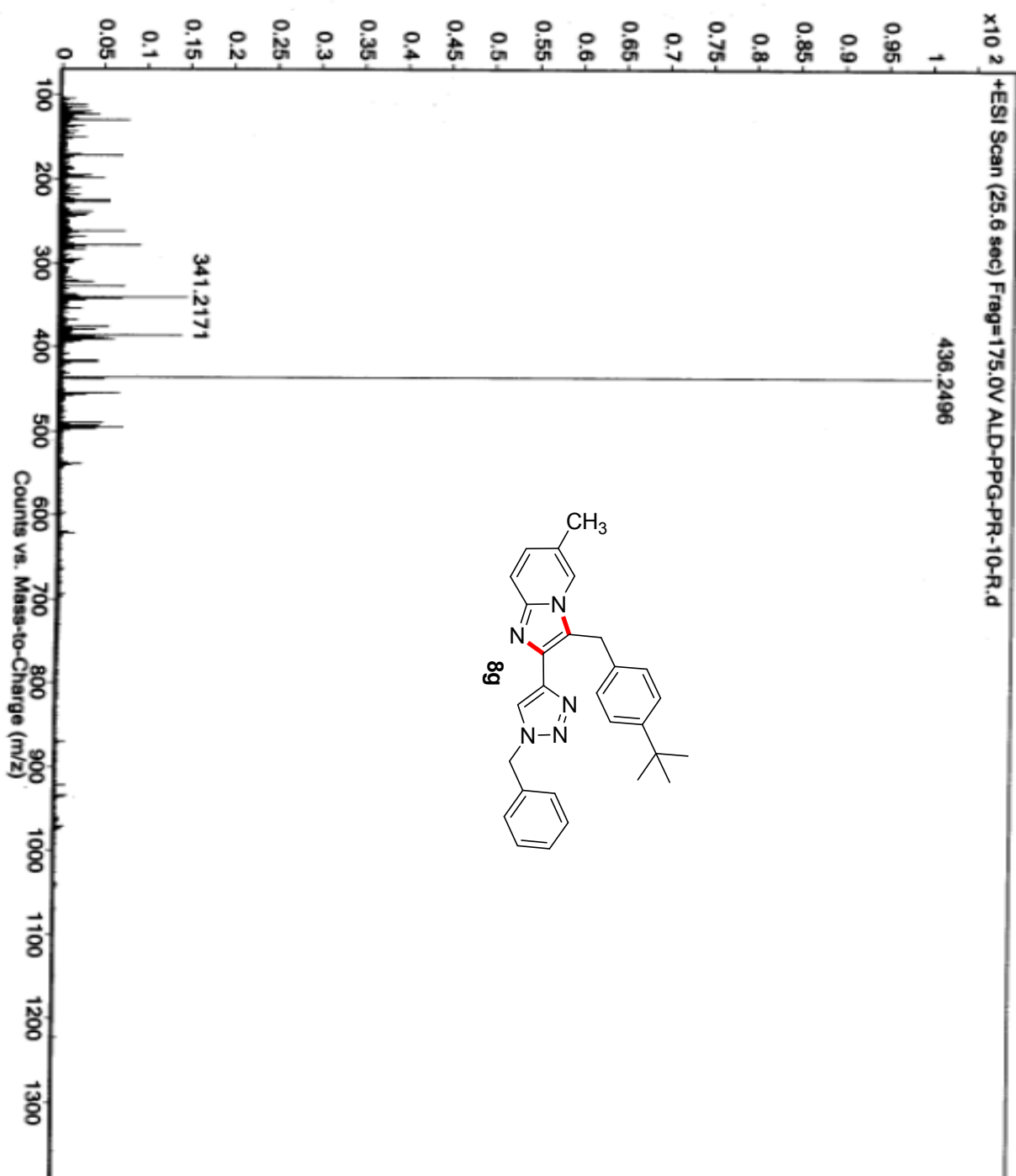


¹³C NMR spectra of 8g



HRMS spectra of 8g

Sample Name ALD-PPG-PR-10-R Position -1
Inj Vol -10 InjPosition
Data Filename ALD-PPG-PR-10-R.d ACQ Method
Instrument Name Instrument 1
SampleType Sample
Comment
User Name
IM Calibration Status
Acquired Time 3/18/2014 2:31:44 PM
Success

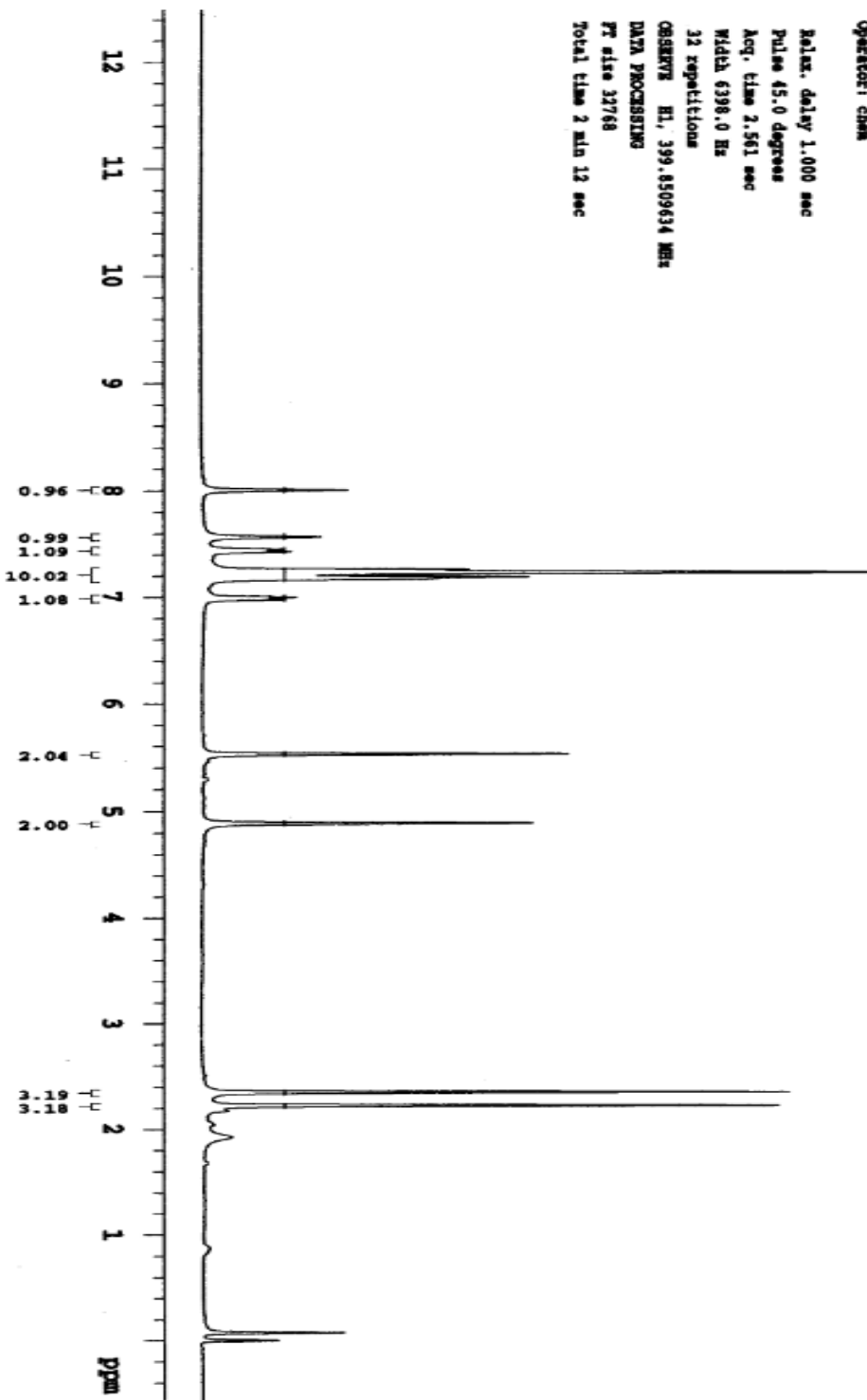
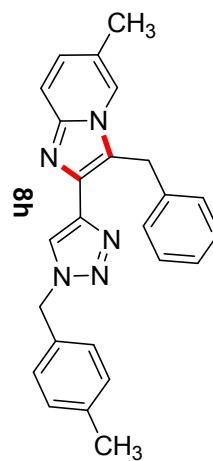


¹H NMR spectra of 8h

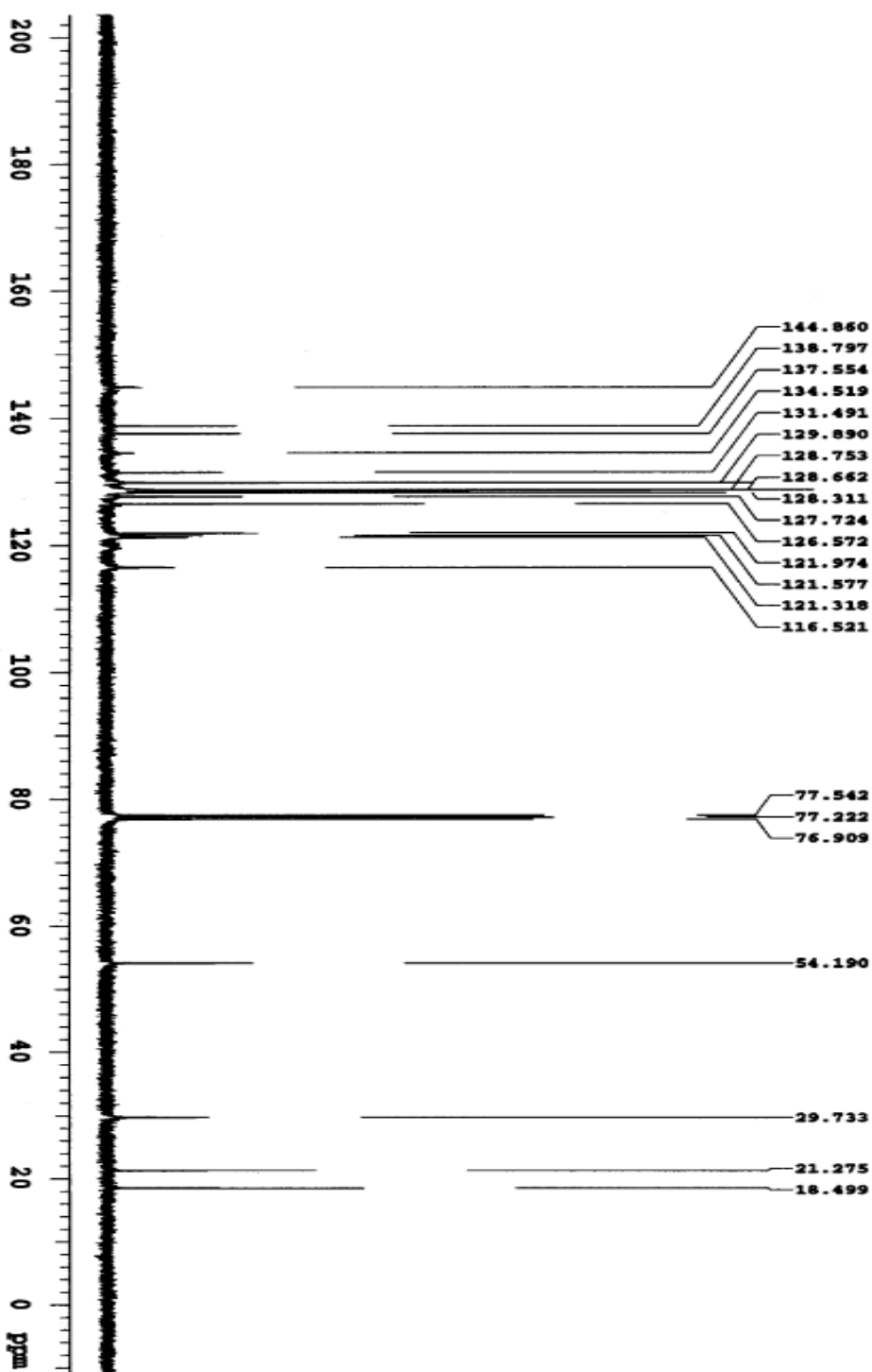
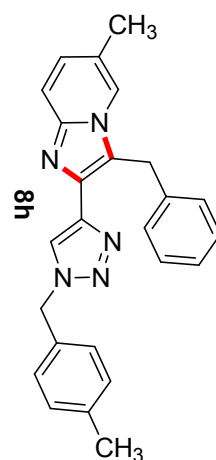
Sample Name:
ALD_P04_P0_15
Data Collected on:
1190-NMR-mercury400
Archive directory:
Sample directory:
FIDFile: PROTON
Pulse Sequence: PROTON (zgpg3)
Solvent: cdcl3
Data collected on: Jan 25 2014

Temp: 25.0 C / 298.1 K
Operator: chm

Relax. delay: 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.8509634 MHz
DATA PROCESSING
FT size 32768
Total time 2 min 12 sec



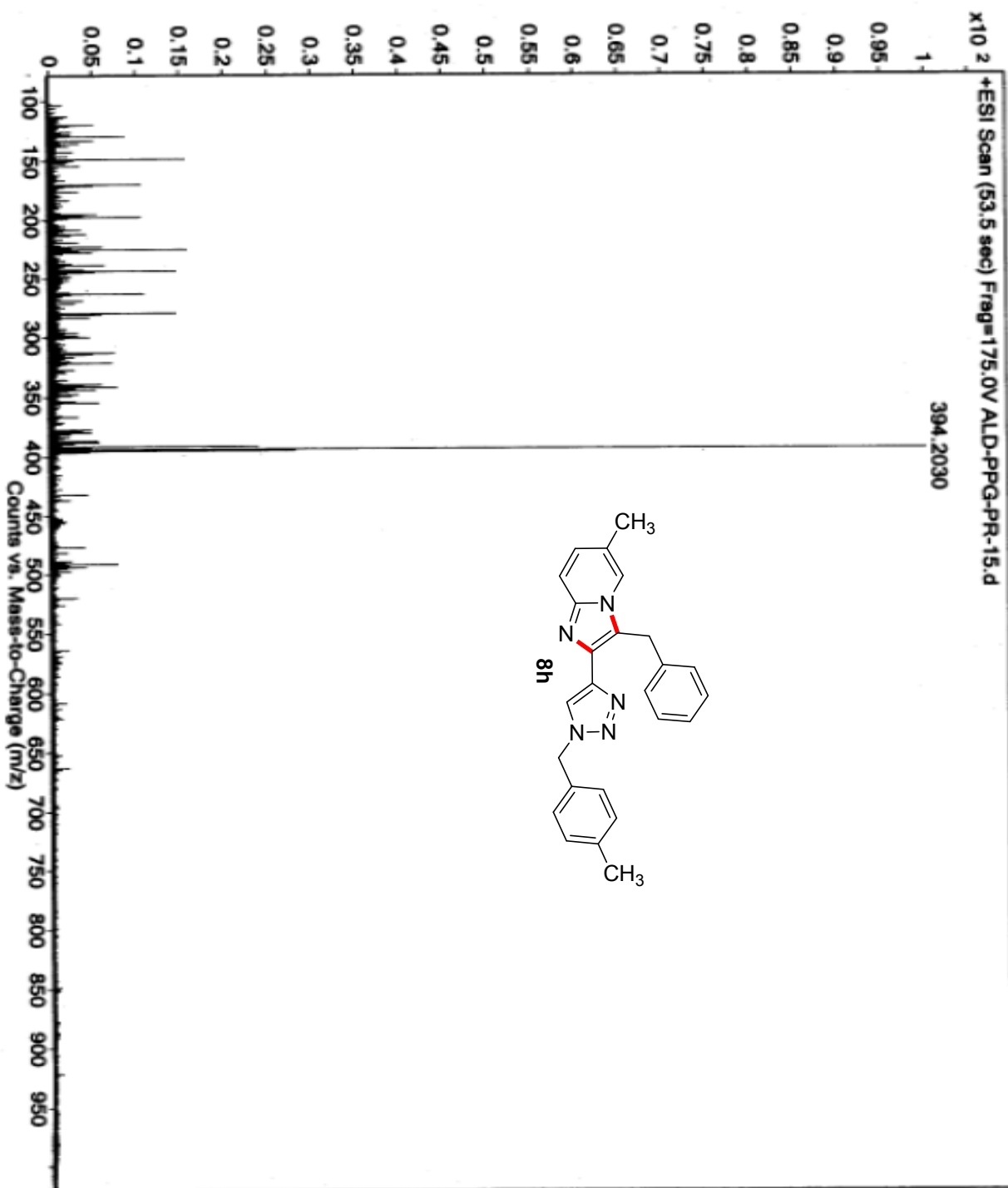
¹³C NMR spectra of **8h**



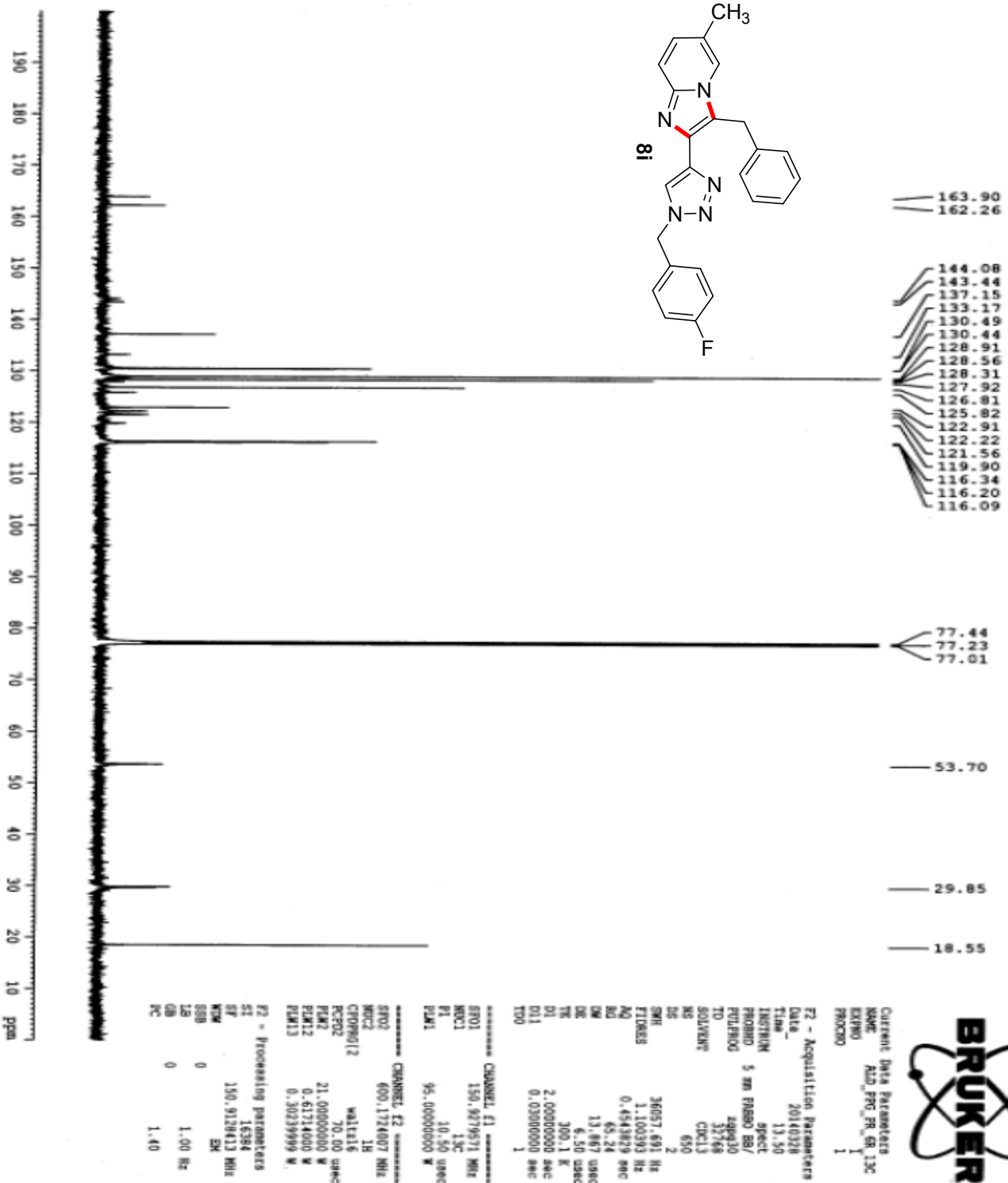
<p>PULSE SEQUENCE</p> <p>Relax. delay 1.000 sec</p> <p>Pulse 45.0 degree</p> <p>Acq. time 1.304 sec</p> <p>Width 25125.6 Hz</p> <p>1180 repetitions</p>	<p>OBSERVE C13, 100.5425932</p> <p>INSTRUM BL, 399.8529994</p> <p>Power 42 dB</p> <p>continuously on</p> <p>WALTZ-16 modulated</p>	<p>DATA PROCESSING</p> <p>line broadening 0.5 Hz</p> <p>FT also 65536</p> <p>Total time 45 minutes</p>	<p>ALD-PRD-28-15-13C</p> <p>Solvent: cdcl3</p> <p>Temp. 25.0 C / 298.1 K</p> <p>Operator: clem</p> <p>Mercury-400 13C-NMR</p>
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HRMS spectra of 8h

Sample Name	ALD-PPG-PR-15	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	ALD-PPG-PR-15.d	Acq Method		Comment		Acquired Time	3/11/2014 3:52:19 PM

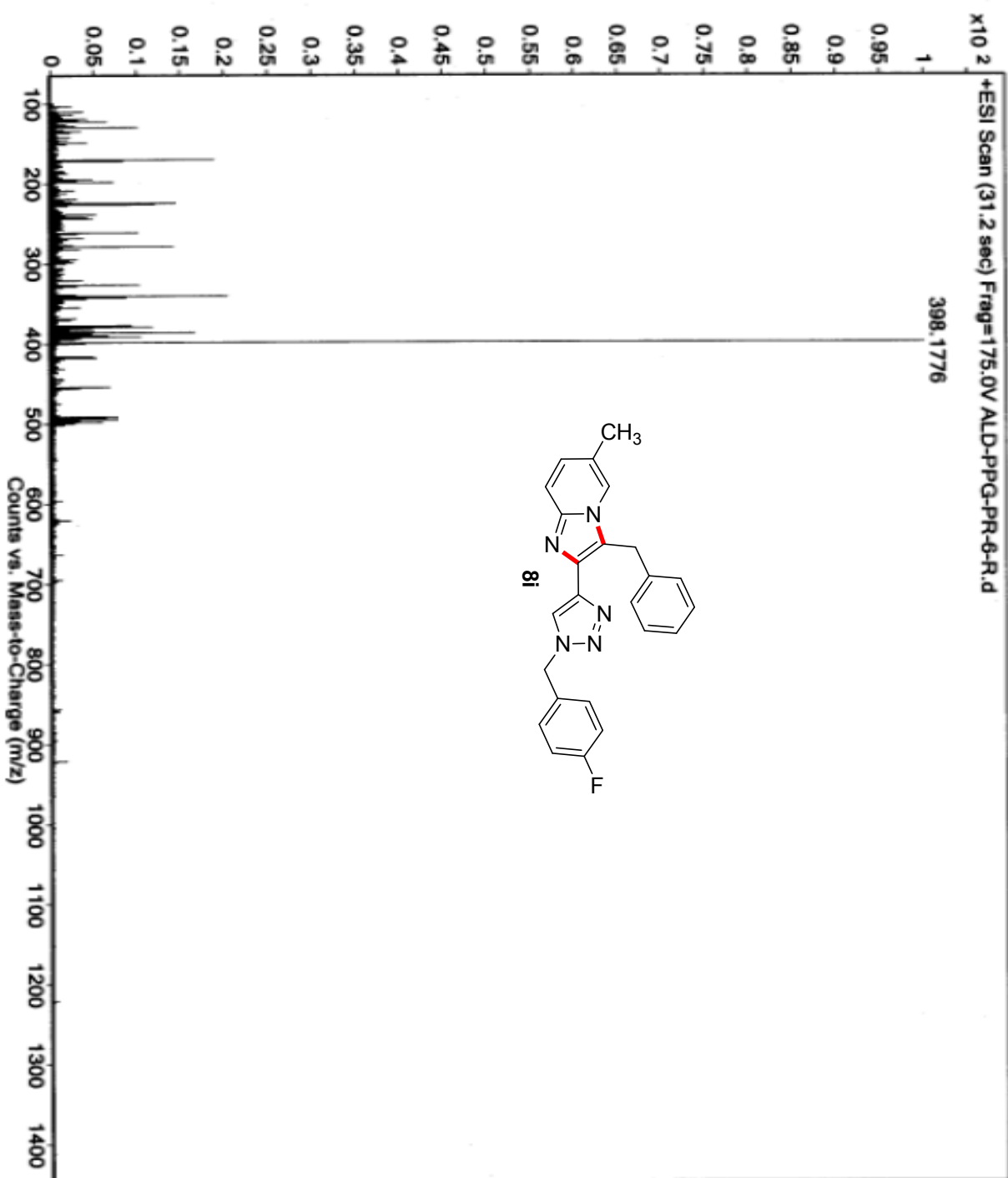


¹³C NMR spectra of **8i**



HRMS spectra of 8i

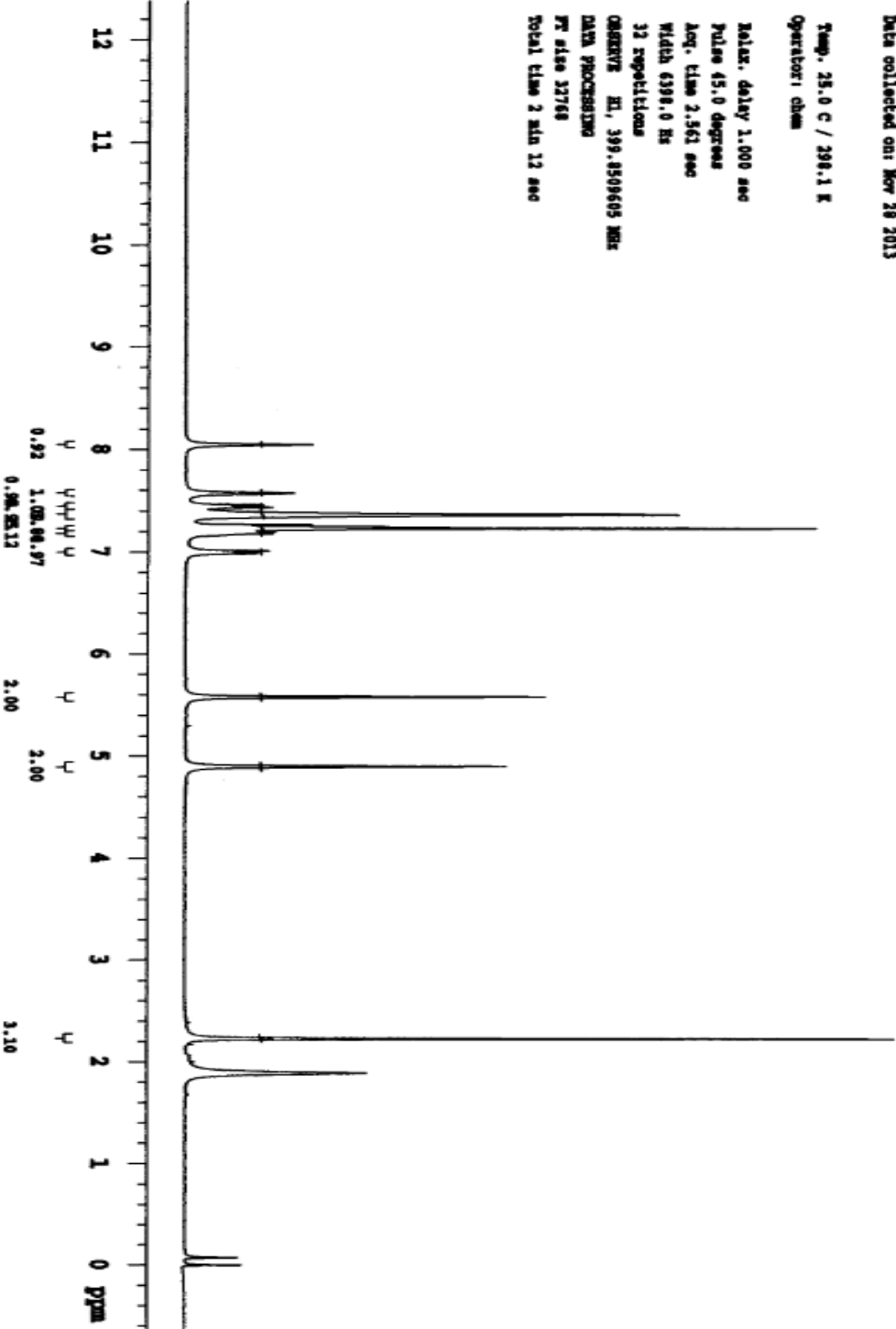
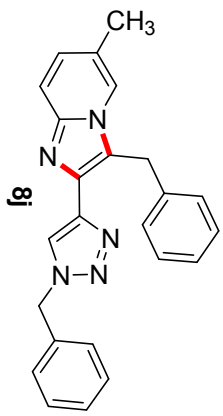
Sample Name	ALD-PPG-PR-6-R	Position	-1	Instrument Name	Instrument 1	User Name	Success
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	
Data Filename	ALD-PPG-PR-6-R.d	Acq Method		Comment		Acquired Time	3/18/2014 2:38:46 PM



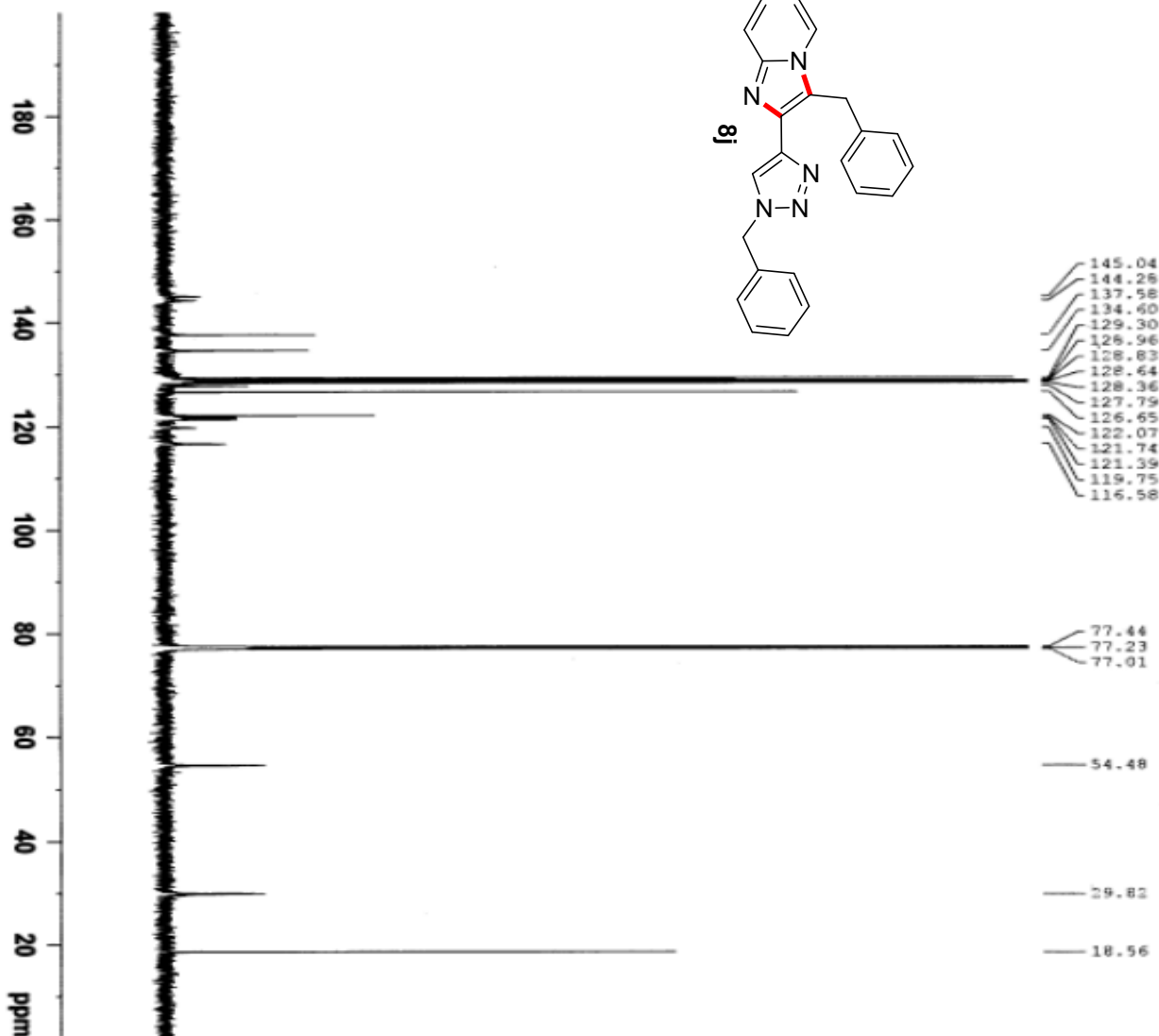
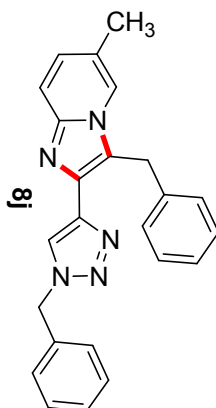
¹H NMR spectra of 8j

ALD-PPQ-PR-9
Sample Name:
ALD-PPQ-PR-9
Data Collected on:
11/20/2013
Acquire directory:
Sample directory:
FID/FLI: PROTON
Pulse Sequence: PROTON (zgpg30)
Solvent: cdcl3
Data collected on: Nov 20 2013

Temp: 25.0 C / 298.1 K
Operator: chm
Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 2.561 sec
Width: 6398.0 Hz
32 repetitions
OBSERVED: 401, 399, 850605 MHz
DATA PROCESSING
PI: 45.000000
Total time: 2 min 12 sec



¹³C NMR spectra of **8j**



Current Data Parameters
 NAME ALD-PRG-PR-9-2-13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131129
 Time_ 10.38
 INSTRUM spect
 PROBD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 36057.691 Hz
 FIDRES 1.100393 Hz
 AQ 0.4543829 sec
 RG 65.24
 DM 13.067 usec
 DE 6.50 usec
 TE 298.7 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

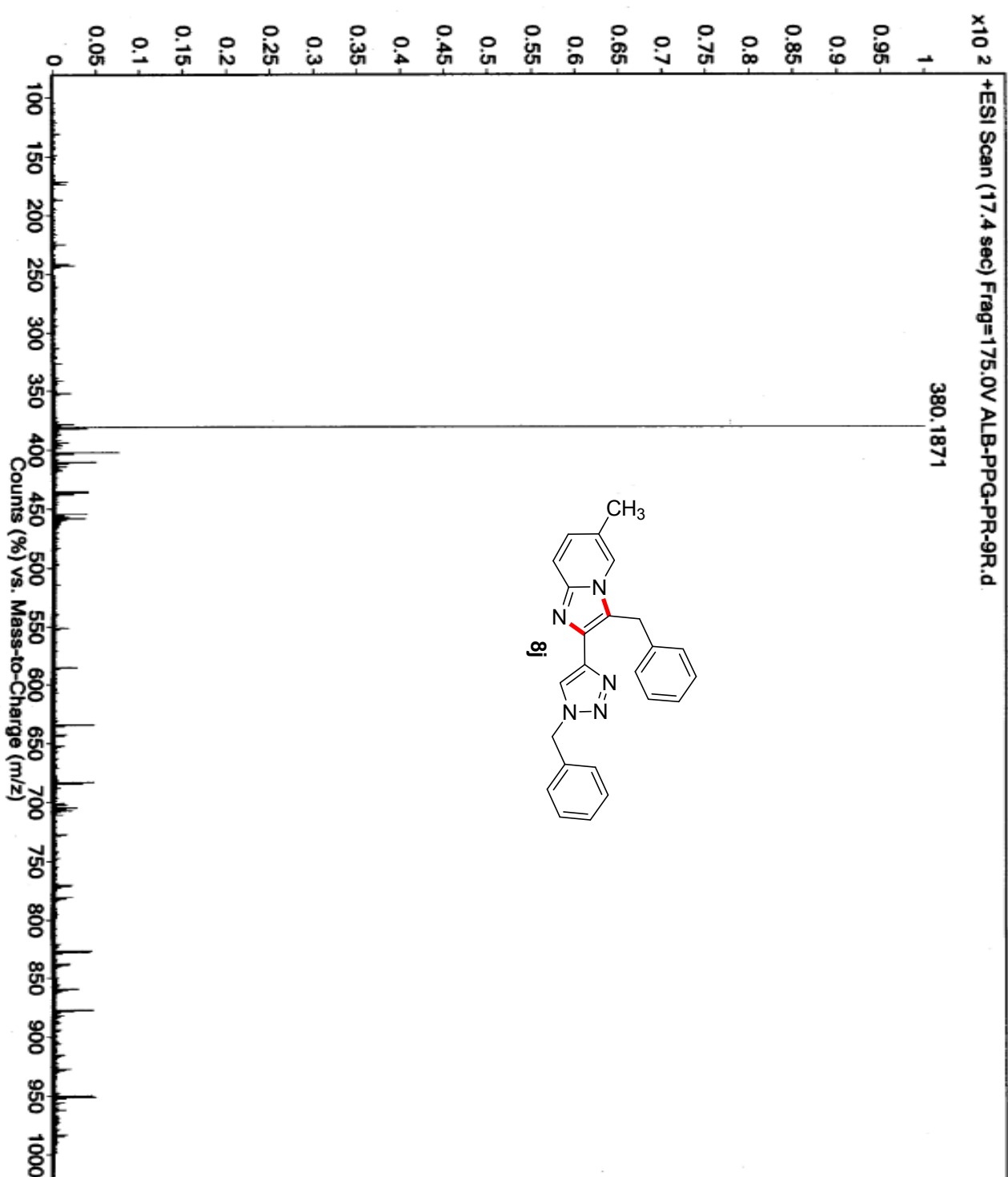
===== CHANNEL f1 =====
 SFO1 150.9279571 MHz
 NUC1 13C
 P1 10.50 usec
 PLM1 95.00000000 W

===== CHANNEL f2 =====
 SFO2 600.1724007 MHz
 NUC2 1H
 CPOPRG12 WALTZ16
 RCPD2 70.00 usec
 PLM2 21.00000000 W
 PLM12 0.61714000 W
 PLM13 0.30239999 W

F2 - Processing parameters
 SI 16384
 SF 150.9128413 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

HRMS spectra of 8j

Sample Name	ALB-PPG-PR-9R	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	ALB-PPG-PR-9R.d	Acq Method		Comment		Acquired Time	3/20/2014 9:47:11 AM



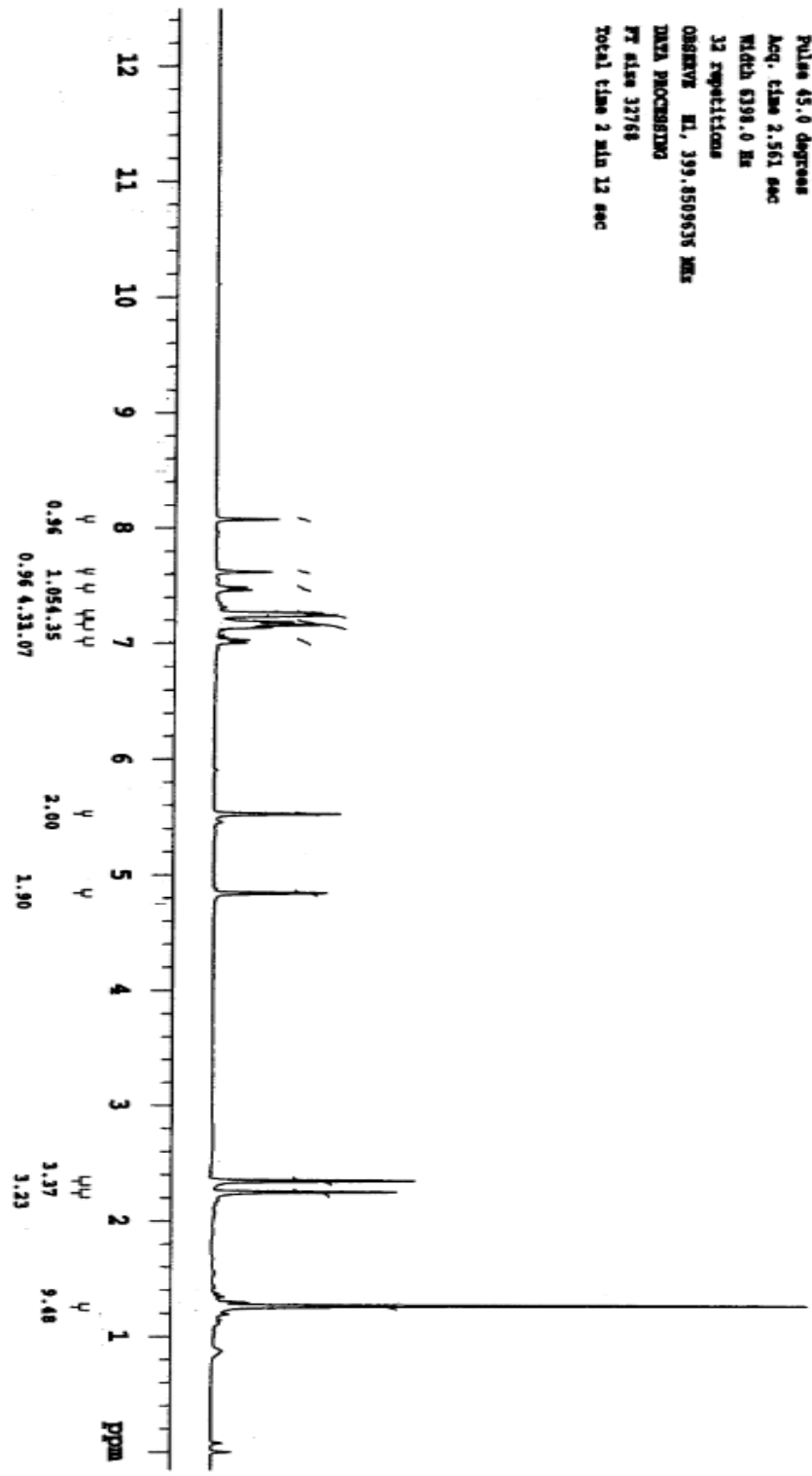
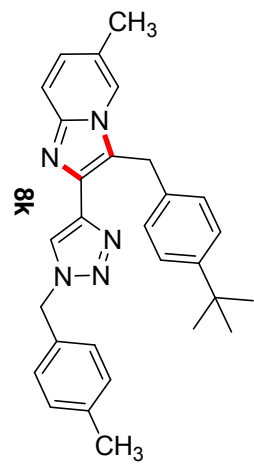
¹H NMR spectra of 8k

Sample Name:
ALD-PPQ-PR-16
Data Collected on:
ITW-NMR-marcury400
Archive directory:
/home/chem/data/study
Sample directory:
test-proton-01
P1/Title: ALD-PPQ-PR-16

Pulse Sequence: PROTON (zgpg3)
Solvent: cdcl3
Data collected on: Jan 28 2014

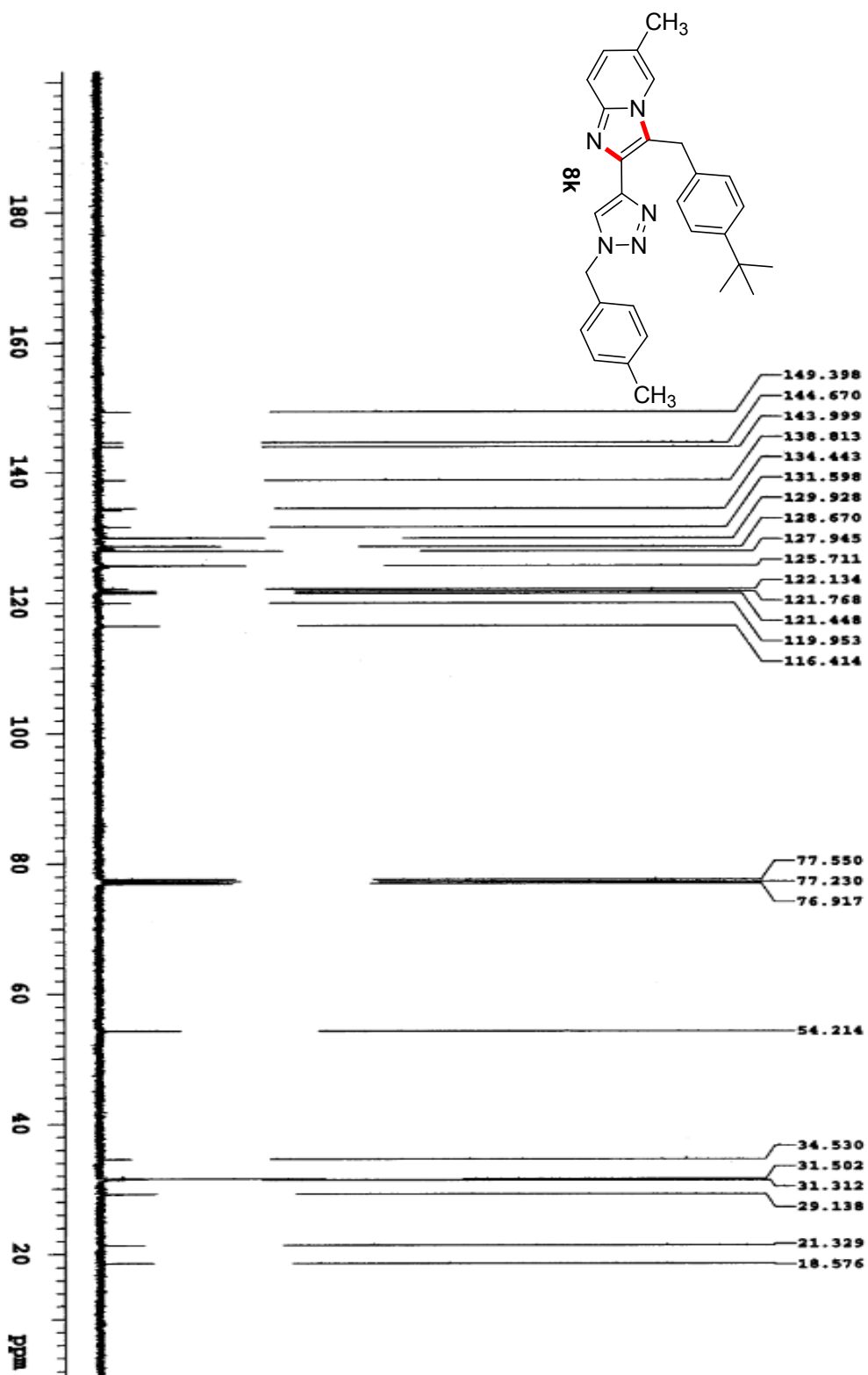
Temp. 25.0 C / 298.1 K
Operator: chem

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz
12 repetitions
OBSERVE H1, 399.8509636 MHz
DATA PROCESSING
F1 also 32768
Total time 2 min 12 sec

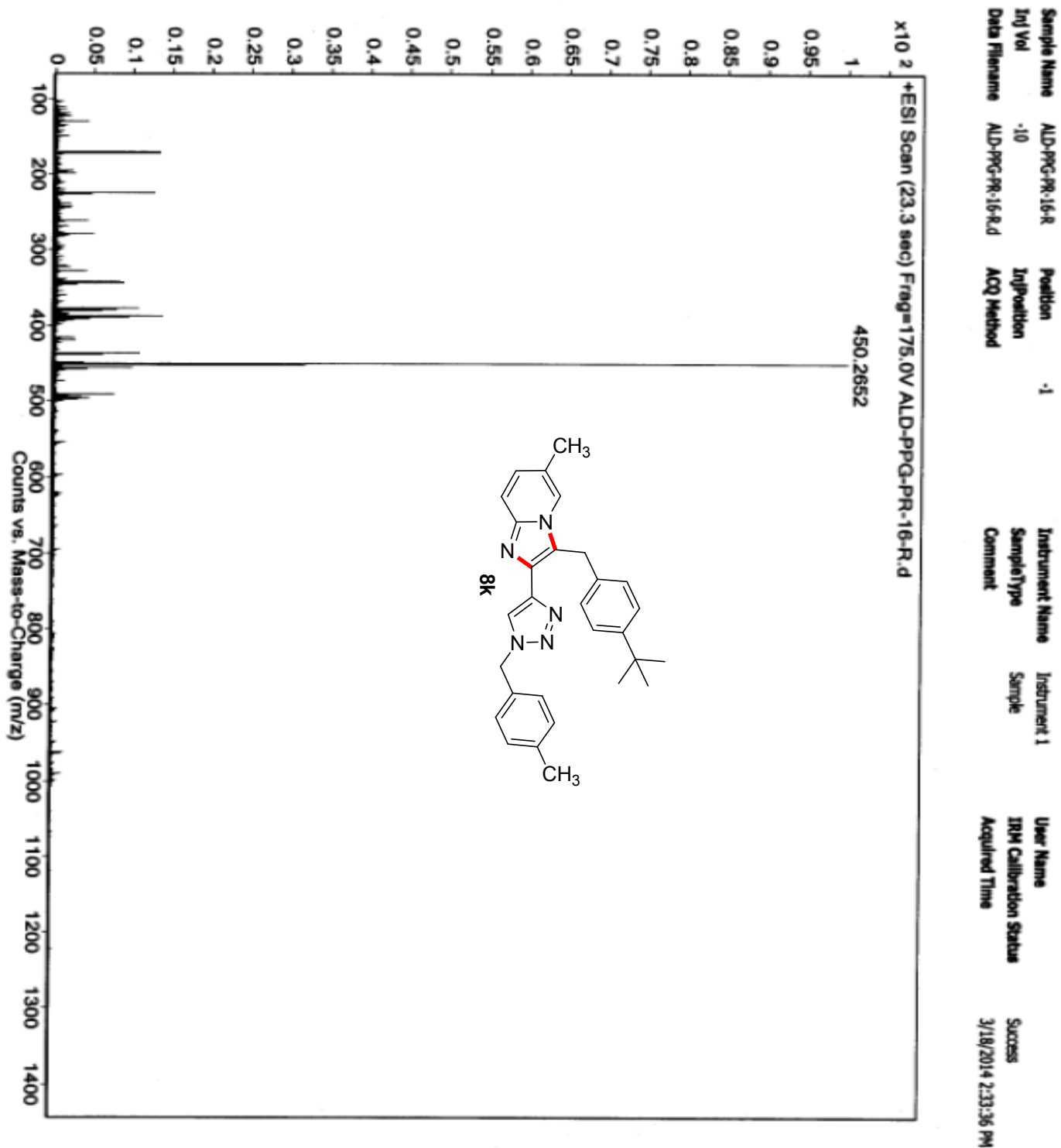


¹³C NMR spectra of **8k**

<p>PROB EXPERIMENT</p> <p>Pulse: delay 1.000 sec</p> <p>Pulse: 45.0 degrees</p> <p>Acq. time 1.304 sec</p> <p>Width 25125.6 Hz</p> <p>1400 repetitions</p>	<p>OSCILLATE C13, 400.5425878</p> <p>RECORDING HI, 299.8529994</p> <p>Power: 42 dB</p> <p>continuously on</p> <p>NAIIR-16 modulated</p>	<p>DATA PROCESSING</p> <p>Line broadening 0.5 Hz</p> <p>FT size 65536</p> <p>Total time 15 minutes</p>	<p>ALD_PP0_PP_16_03C</p> <p>Solvent: cdcl3</p> <p>Temp. 25.0 C / 298.1 K</p> <p>Operator: cbm</p> <p>Mercury-400 "1170-MHz"</p>
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HRMS spectra of 8k



¹H NMR spectra of 81

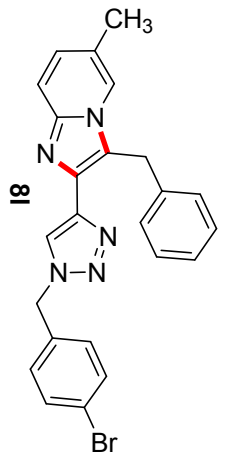
Sample Name: ALD-PTD-PT-13
Data Collected on: ITRV-NMR-Mercury400
Archive directory:
Sample directory:
P14P11:1: PROTON

Pulse Sequence: PROTON (zgpg3)
Solvent: cdcl3
Data collected on: Dec 10 2013

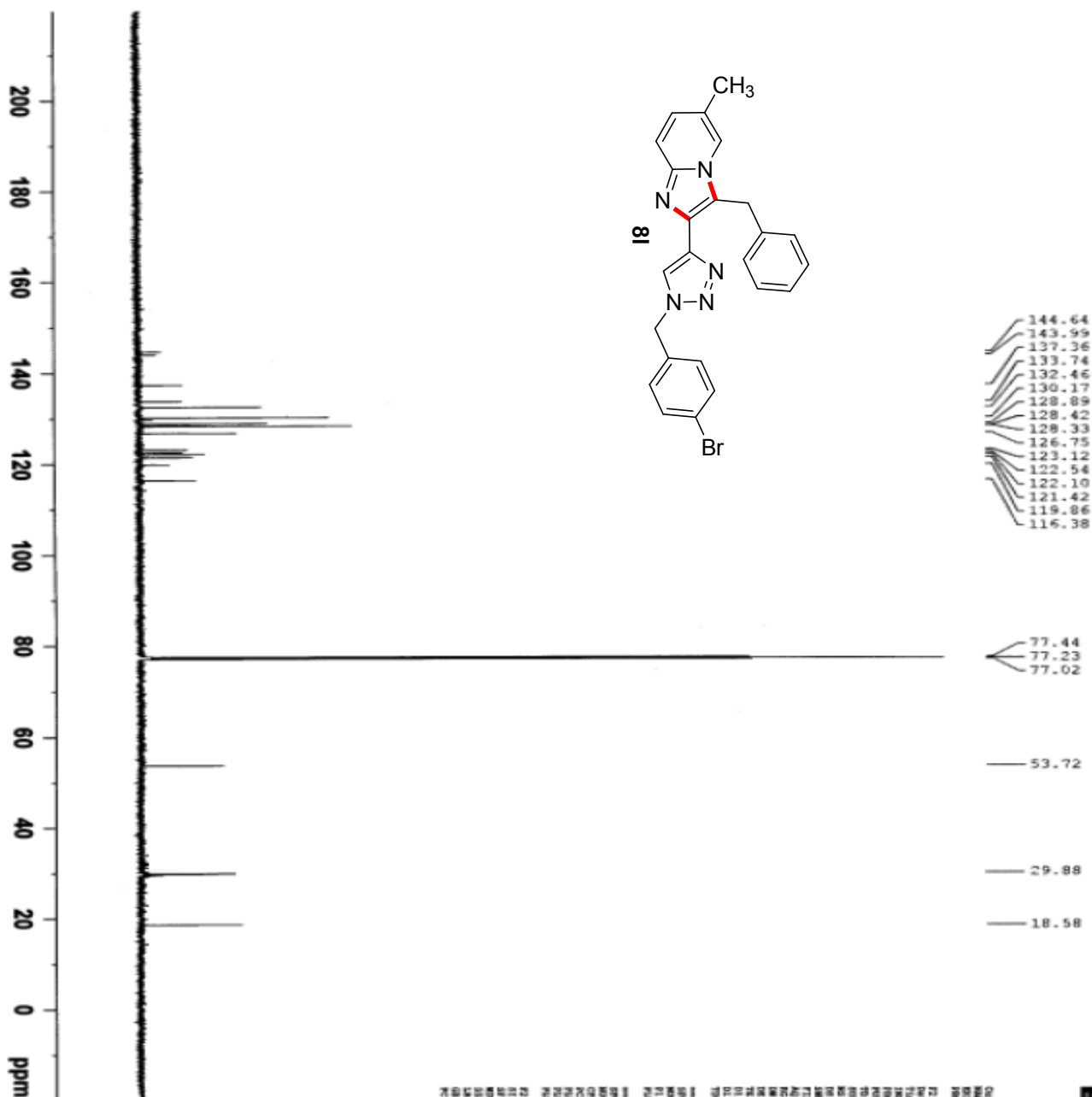
Temp. 25.0 C / 298.1 K
Operator: dbm

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz

32 repetitions
OBSERVE HI, 399.850890 MHz
DATA PROCESSING
PT also 32768
Total time 2 min 12 sec

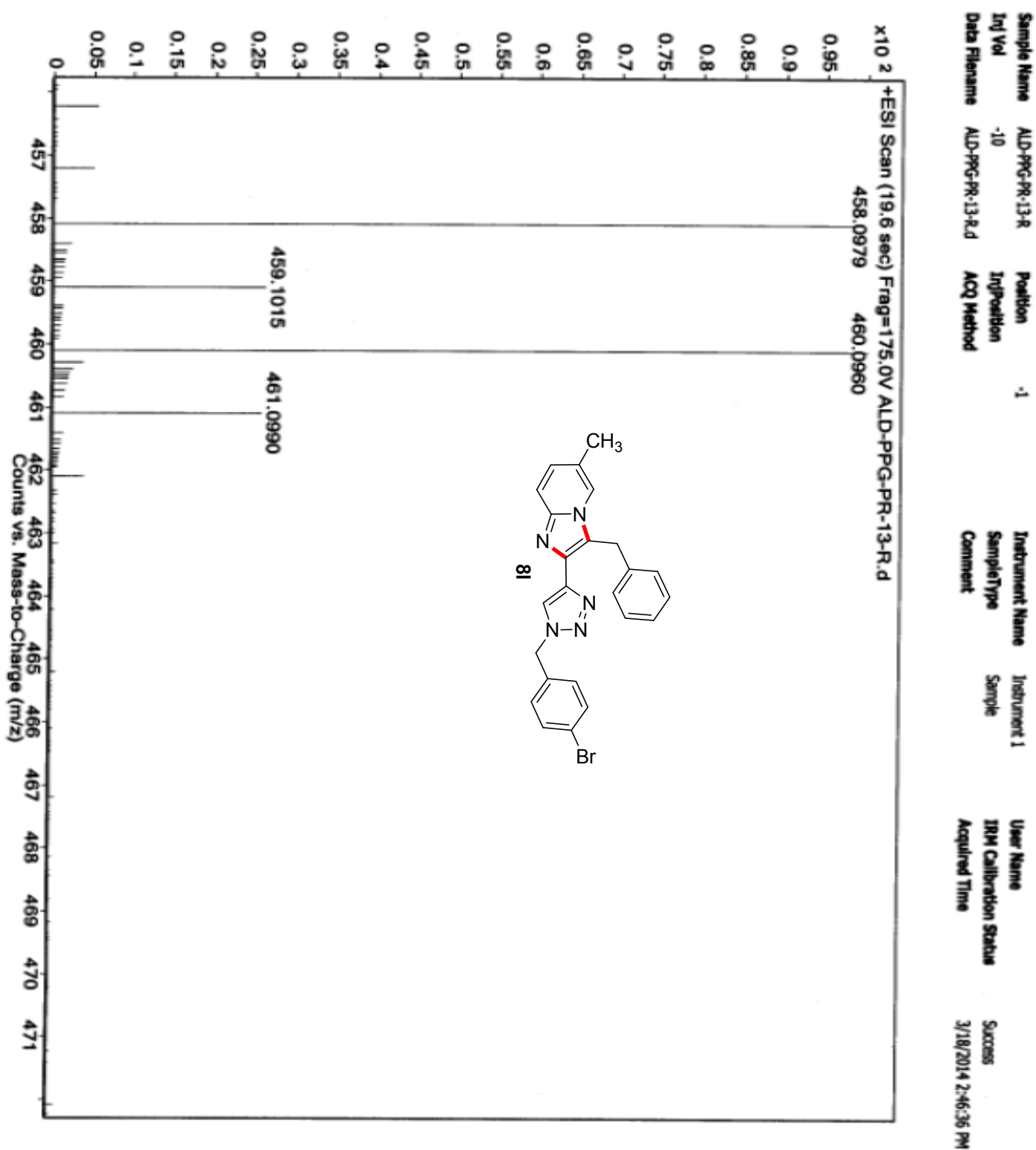


¹³C NMR spectra of 81

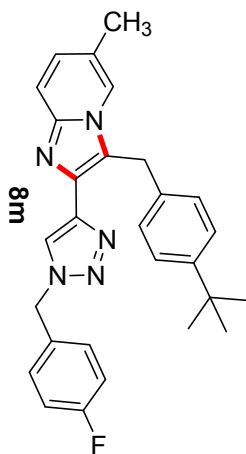
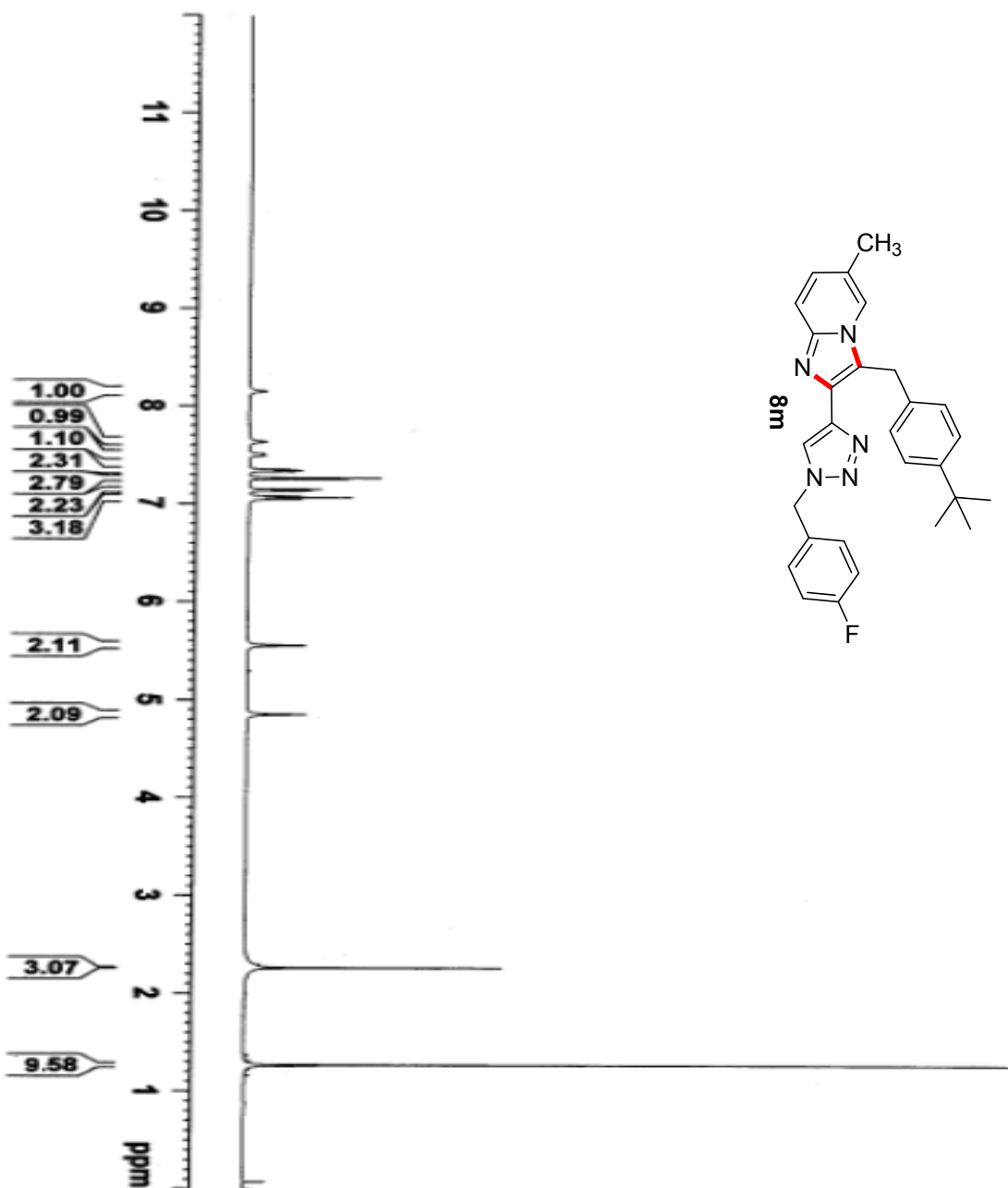


Current Data Parameters
 Name: 211212
 Date_Time: 11/16/16
 Instrument: spect
 PROBHD: 5 mm BBO-1H/1
 PULPROG: zgpg30
 TD: 65536
 SFO: 500.136199
 AQ: 1.000000
 F2 - Acquisition Parameters
 Name: 211212
 Date_Time: 11/16/16
 Instrument: spect
 PROBHD: 5 mm BBO-1H/1
 PULPROG: zgpg30
 TD: 65536
 SFO: 500.136199
 AQ: 1.000000
 F2 - Processing parameters
 SI: 32768
 SF: 125.013282 MHz
 DS: 4
 SSF: 31.253205 MHz
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 1.48

HRMS spectra of 81



¹H NMR spectra of **8m**



Current Data Parameters
 NAME ALD_FMG_FR_7.1R
 EXTNO 1
 PROCNO 1

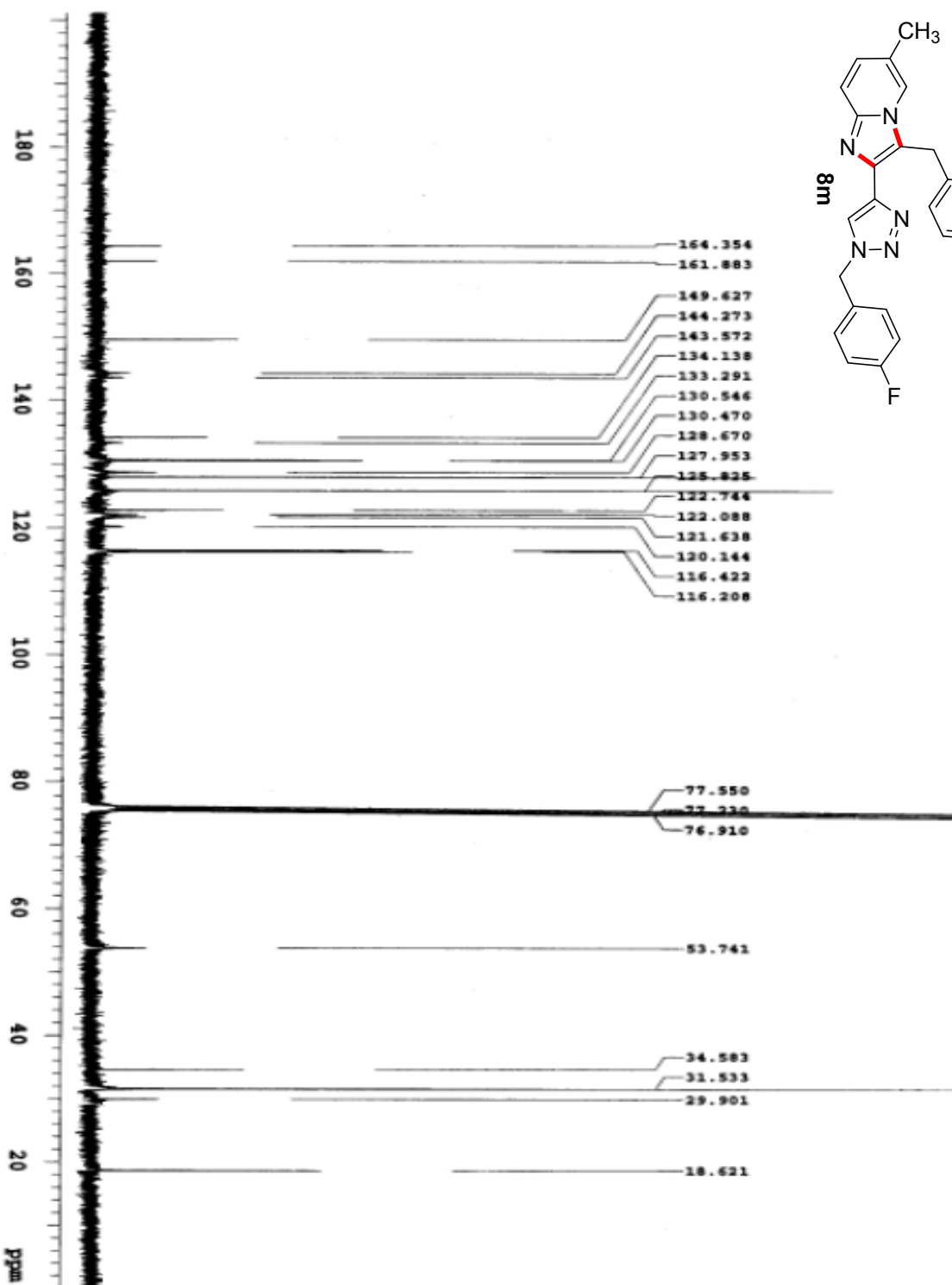
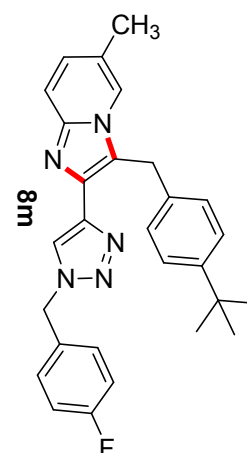
F2 - Acquisition Parameters
 Date_ 20131023
 Time 11.09
 INSTRUM spect
 PROBD 5 MM SBBO BB/
 PULPROG zgpg30
 TO 32768
 SOLVENT DMF-D₇
 NS 16
 DS 2
 SFO 12019.230 Hz
 FIDRES 0.360796 Hz
 Aq 1.3631488 sec
 RG 40.32
 SW 41.600 kHz
 FREQ 285.3 K
 TE 298.3 K
 D1 1.00000000 sec
 TSD 1

===== CHANNEL f1 =====
 SFO1 400.117063 MHz
 NUCL1 1H
 P1 12.00 usec
 PL1 21.00000000 W

F2 - Processing parameters
 SI 16384
 SF 400.1170130 MHz
 KW 2M
 SFO 0
 TA 0.30 Hz
 GM 0
 PC 1.00



¹³C NMR spectra of 8m

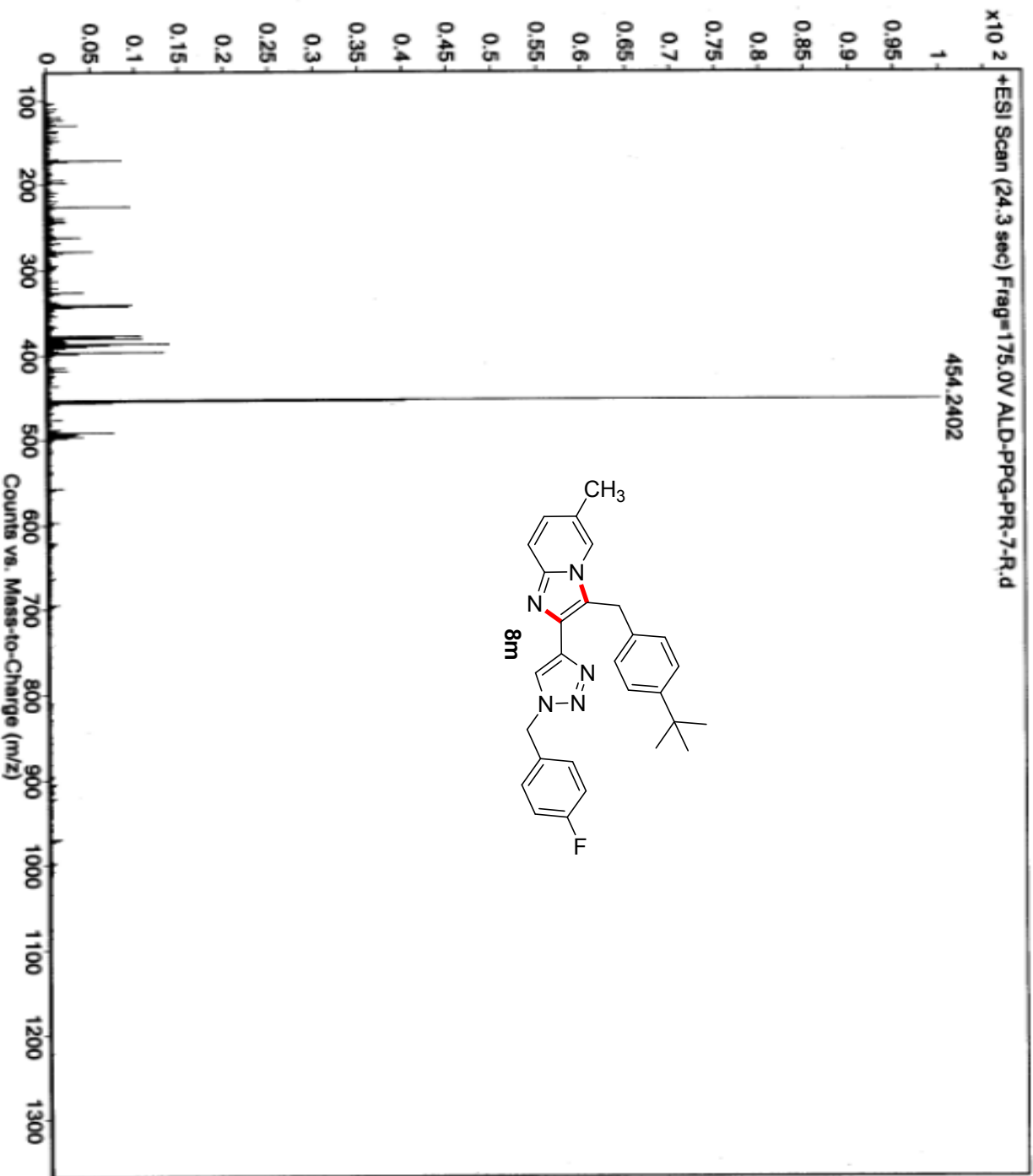


PULSE SEQUENCE: zgpg30
 INSTRUMENT: spect
 PULPROG: zgpg30
 Acq. time 1.304 sec
 WIDTH 25135.6 Hz
 15460 repetitions

DATA PROCESSING: waltz16, 100, 5425617
 DMSO-D6, 100, 399.8529994
 Power: 40 dB
 continuously on
 WALTZ-16 modulated
 Total time 9.9 hours

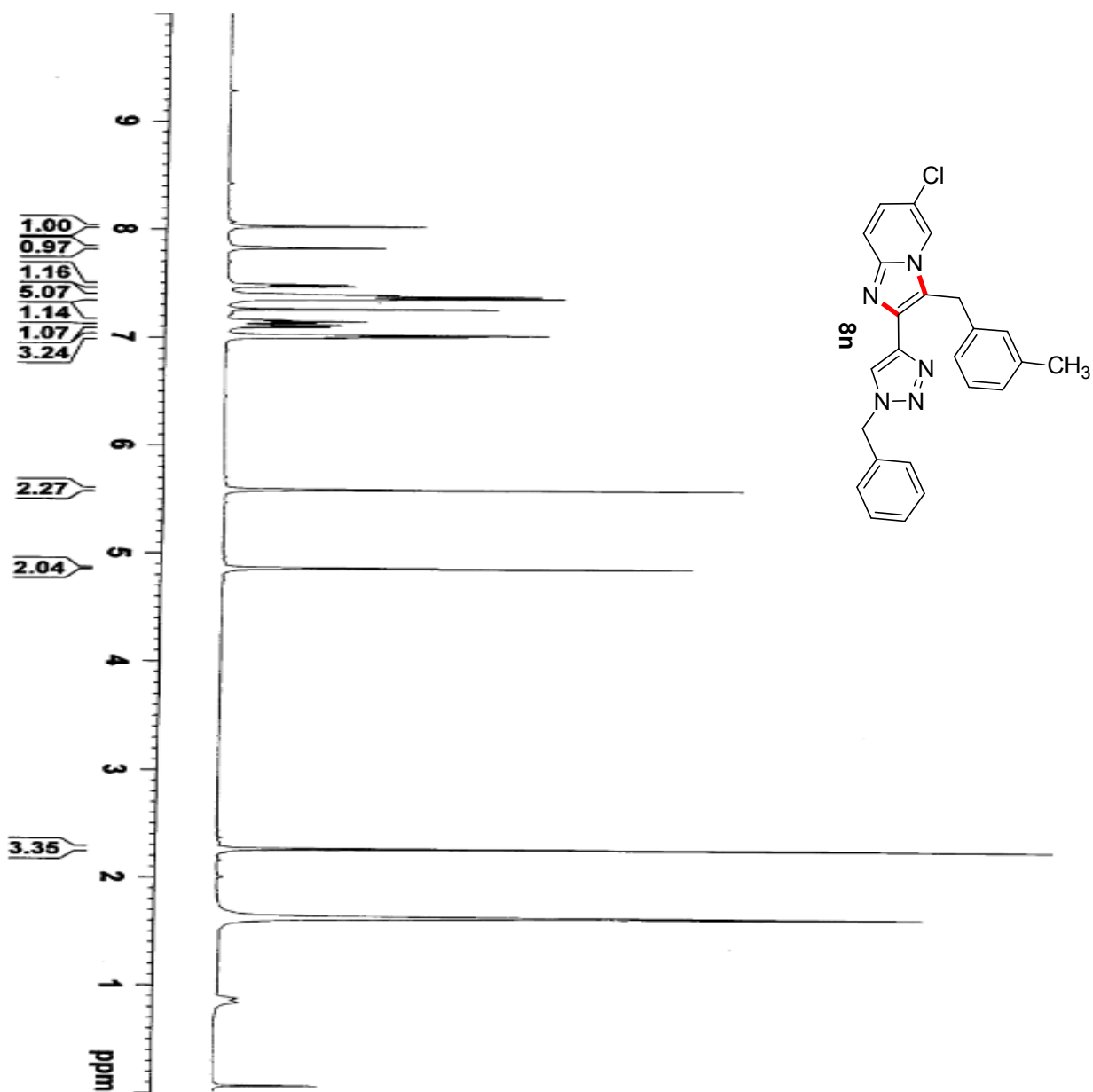
NAME: 8m-7R-13C
Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 Operator: cbm
FILE: ALD-8m-7R-13C
 Mercury-400 *ITV-8M8*

HRMS spectra of 8m



Sample Name	ALD-PPG-PR-7-R	Position	-1	Instrument Name	Instrument 1	User Name
Inj Vol	-10	InjPosition		SampleType	Sample	IBM Calibration Status
Data Filename	ALD-PPG-PR-7-R.d	Acq Method		Comment		Acquired Time
						Success
						3/18/2014 2:41:30 PM

¹H NMR spectra of **8n**



Current Data Parameters
 NAME Pz-3-Ph-Me-1H
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20130518
 Time 16:55
 INSTRUM spect
 PROSD 5 mm NMRB1 DE/
 PULPROG zgpg
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SFO 12019.230 Hz
 FIDRES 0.364799 Hz
 AQ 1.3531488 sec
 R0 140.33
 DN 41.600 umc
 DE 6.50 umc
 TE 296.1 K
 D1 1.0000000 sec
 D11 1
 210

===== CHANNEL f1 =====
 SFO1 600.137063 MHz
 NUC1 1H
 P1 12.00 umc
 PL1 21.0000000 W

F2 - Processing parameters
 S1 16384
 SF 600.137063 MHz
 WDM 1H
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

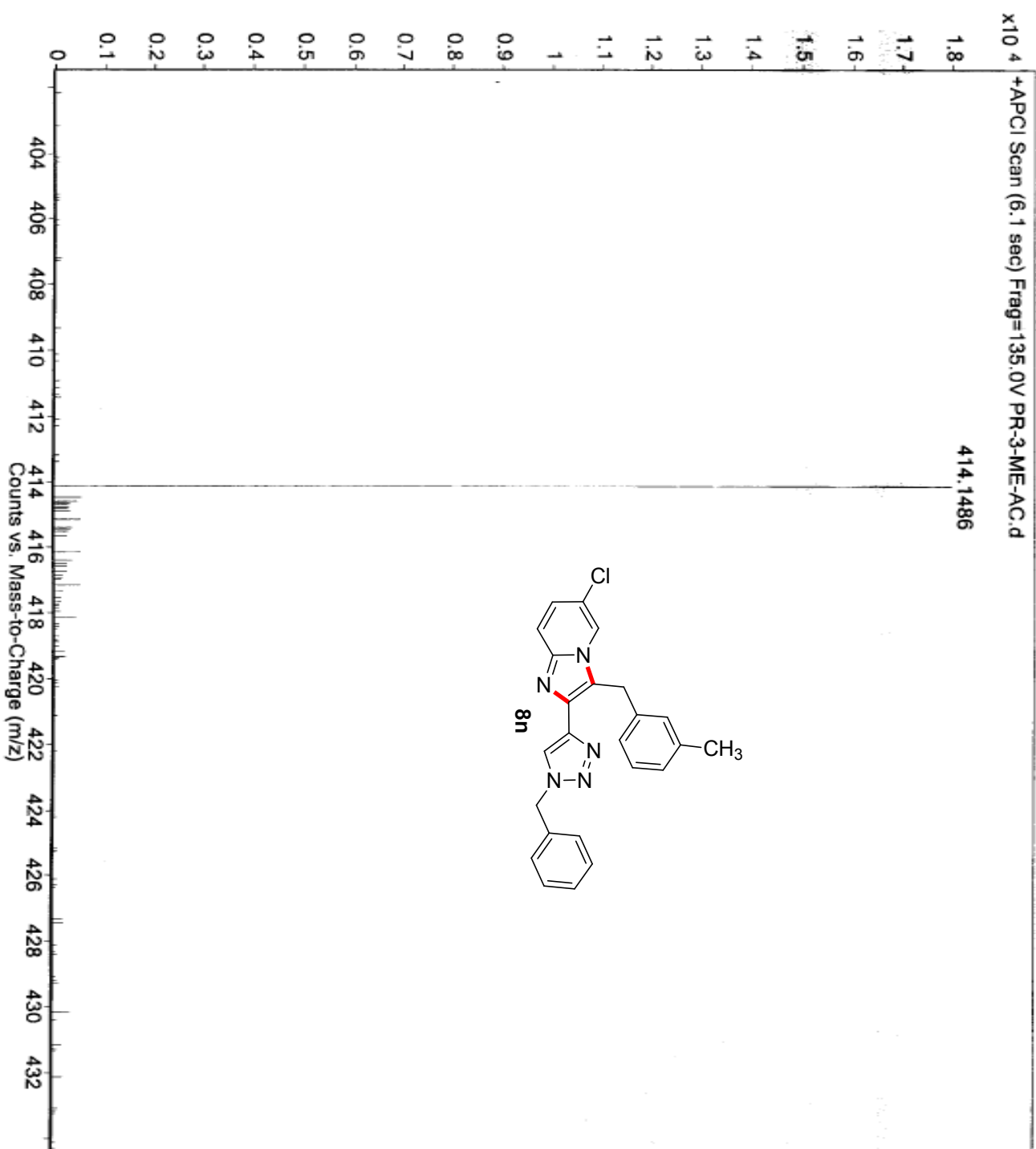
HRMS spectra of 8n

Sample Name
Inj Vol
Data Filename

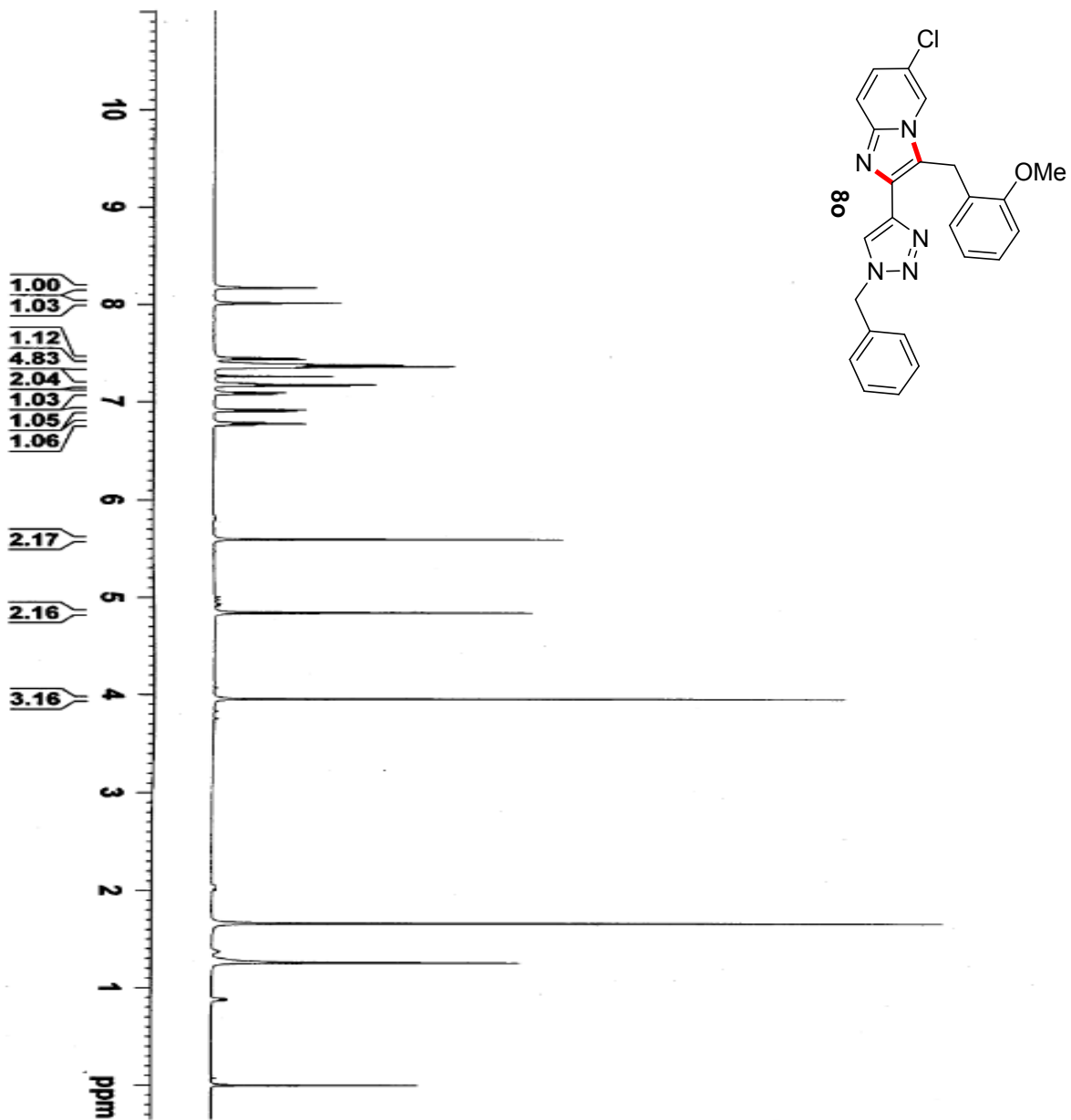
Position
InjPosition
ACQ Method

Instrument Name
SampleType
Comment

User Name
IRM Calibration Status
Acquired Time



¹H NMR spectra of **80**



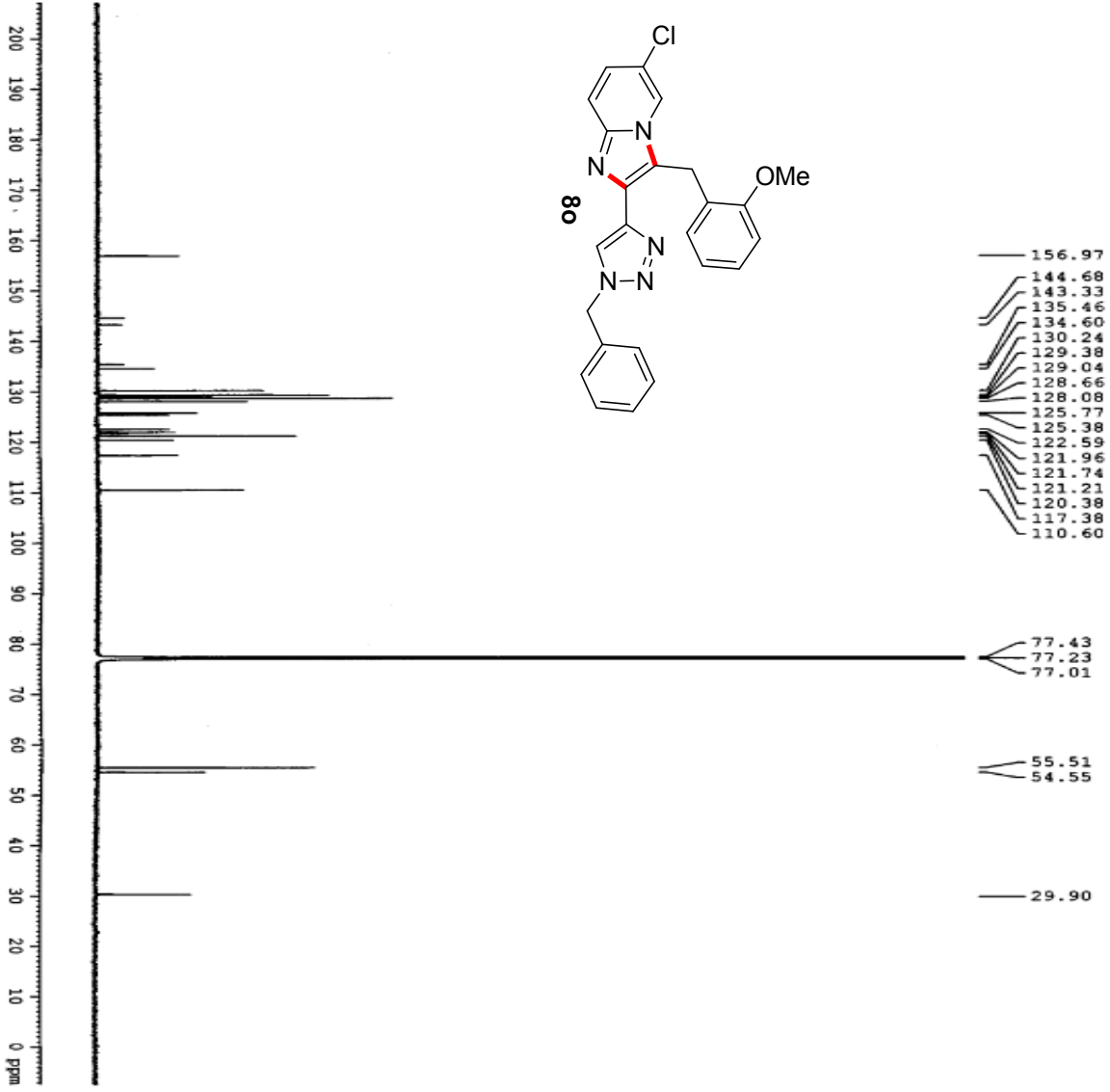
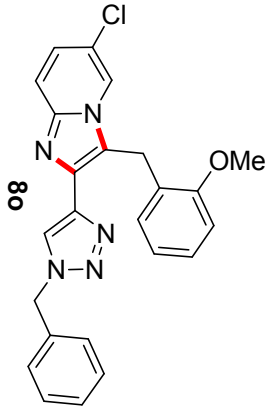
Current Data Parameters
 NAME R8-2-06-40-118
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 DATE_ 20150618
 TIME 21:14
 INSTRUM spect
 PULPROG zgpg30
 PROBRG 5 mm PABBO BB/
 PZPRG06 2930
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12019.230 Hz
 FWHM 0.366798 Hz
 AQ 1.3621488 sec
 RG 89.67
 DW 41.600 usec
 DE 6.50 usec
 TE 296.5 K
 D1 1.00000000 sec
 T10 1

===== CHANNEL f1 =====
 SF01 600.137063 MHz
 NUC1 1H
 P1 12.00 usec
 PL1 21.00000000 W

F2 - Processing parameters
 SI 16384
 SF 600.137063 MHz
 NUX 1H
 SFO 60
 DS 0
 LA 0 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR spectra of **80**



Current Data Parameters
 NAME PR-2-OMe-NC-13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150618
 Time 21.22

INSTRUM spect
 PROBRD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 14876
 DS 2

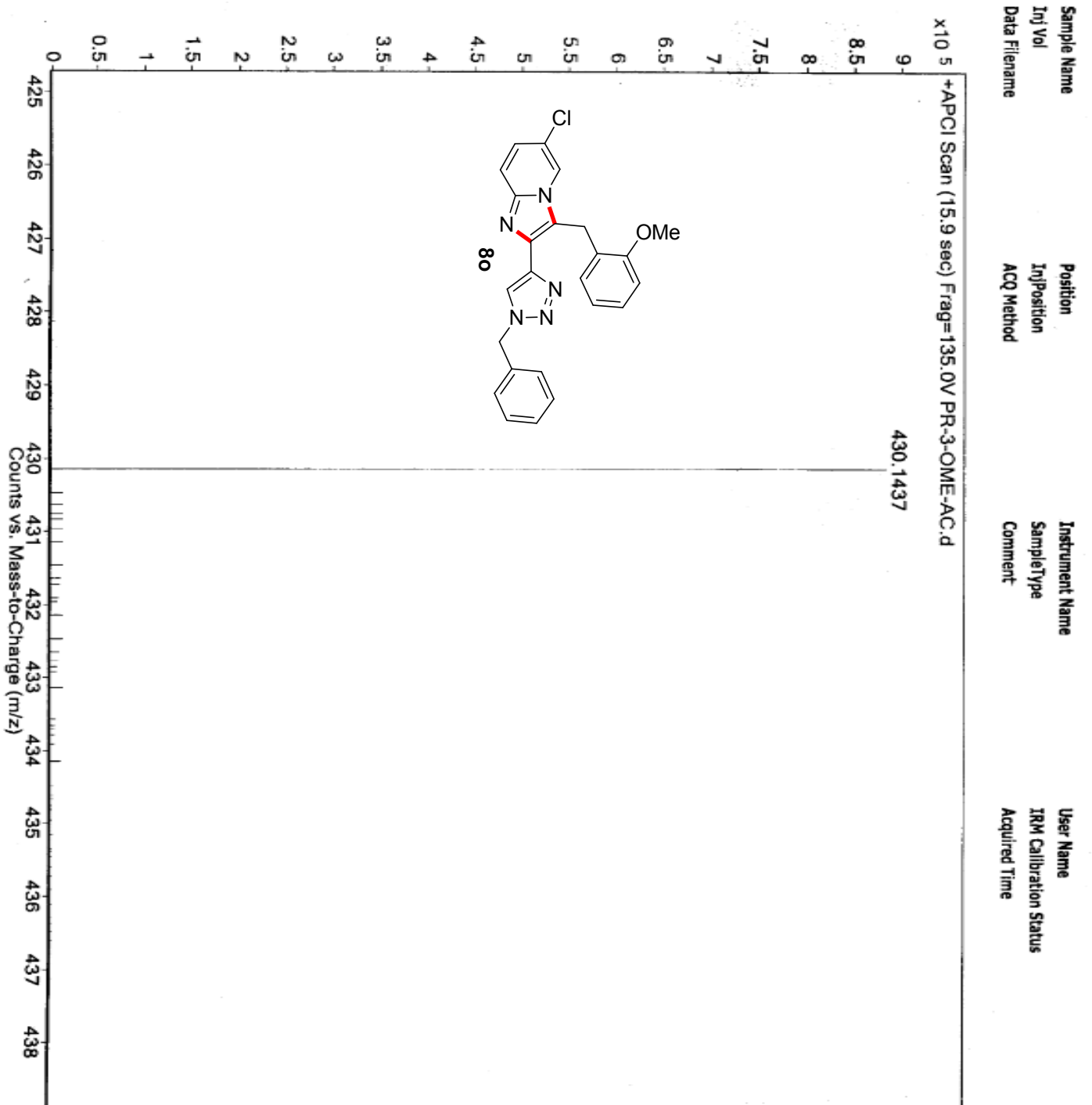
SWH 36057.691 Hz
 FIDRES 1.100393 Hz
 AQ 0.4543829 sec
 RG 65.24
 DW 13.867 usec
 DE 6.50 usec
 TE 297.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

CHANNEL f1
 SFO1 150.9279571 MHz
 NUCL1 13C
 P1 10.50 usec
 PLW1 95.00000000 W

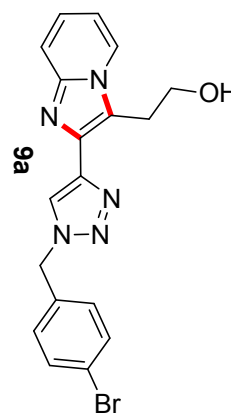
CHANNEL f2
 SFO2 600.1724007 MHz
 NUCL2 1H
 CPOPRG12 waltz16
 PCPDZ 70.00 usec
 PLM2 21.00000000 W
 PLM12 0.61714000 W
 PLM13 0.30239999 W

F2 - Processing parameters
 SI 16384
 SF 150.9128351 MHz
 WSM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

HRMS spectra of 80



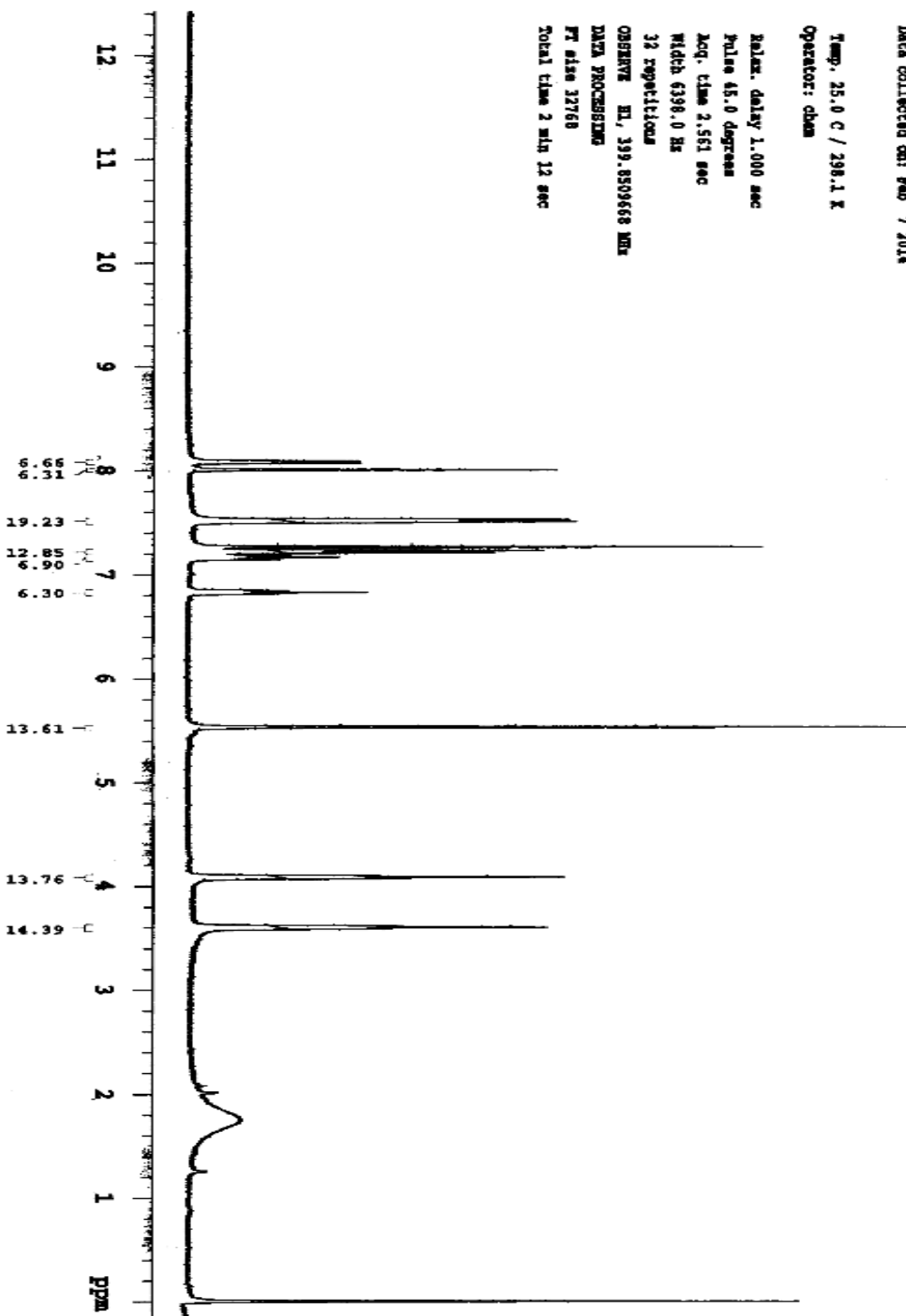
¹H NMR spectra of 9a



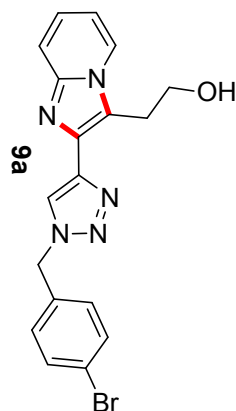
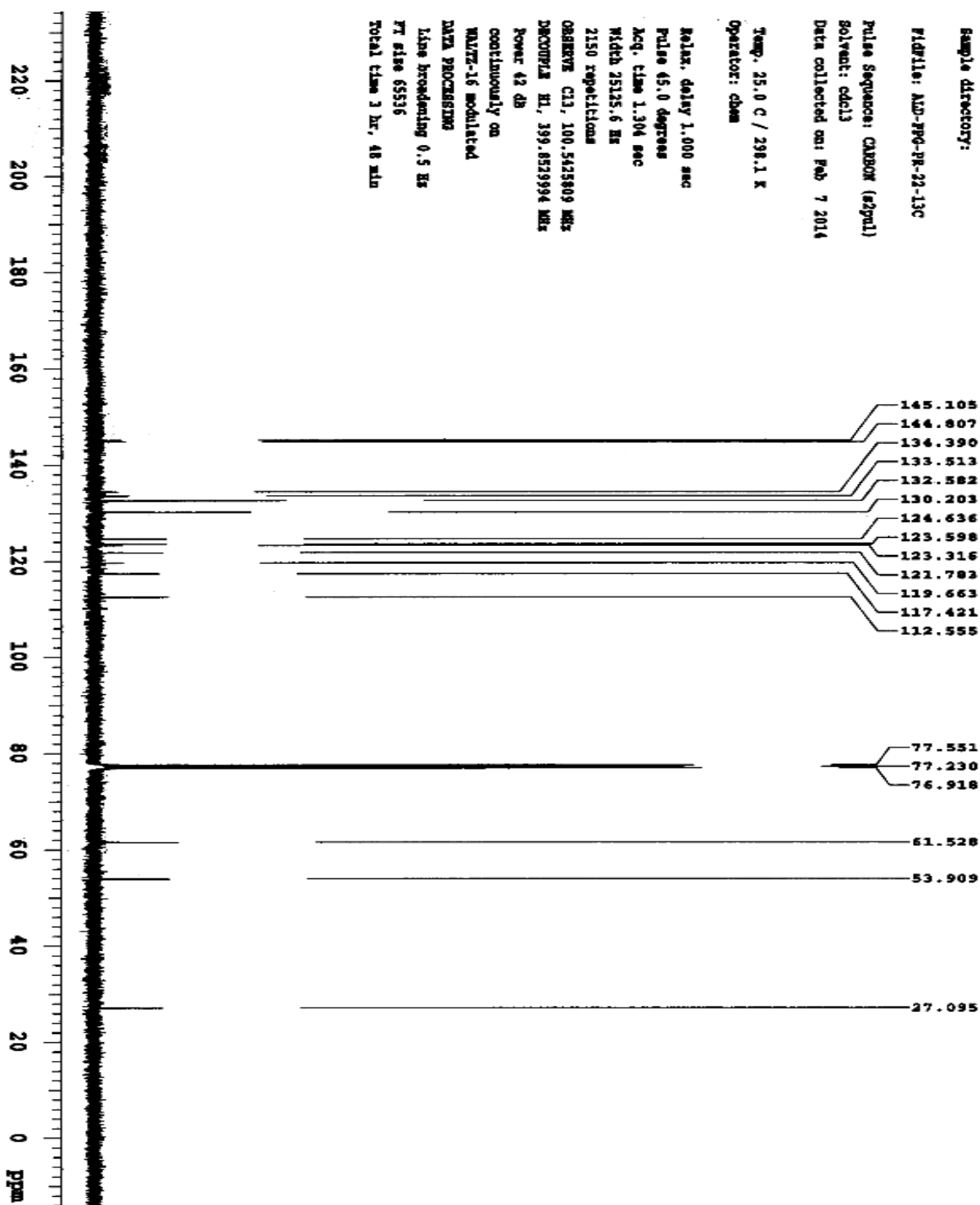
Sample Name: ALD-PTO-PR-22
Data Collected on: 1170-NMR-marcuy400
Acquire directory: /Doms/chem/data/study
Sample directory: P1(CHEM)-SR-tit-4-01
Yieldfile: ALD-PTO-PR-22
Pulse sequence: PROTON (zgpg1)
Solvent: cdcl3
Data collected on: Feb 7 2014

Temp: 25.0 C / 298.1 K
Operator: chm

Relax. delay 1.000 sec
Pulse 45.0 degree
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.850668 MHz
DATA PROCESSING
F1 file 32768
Total time 2 min 12 sec



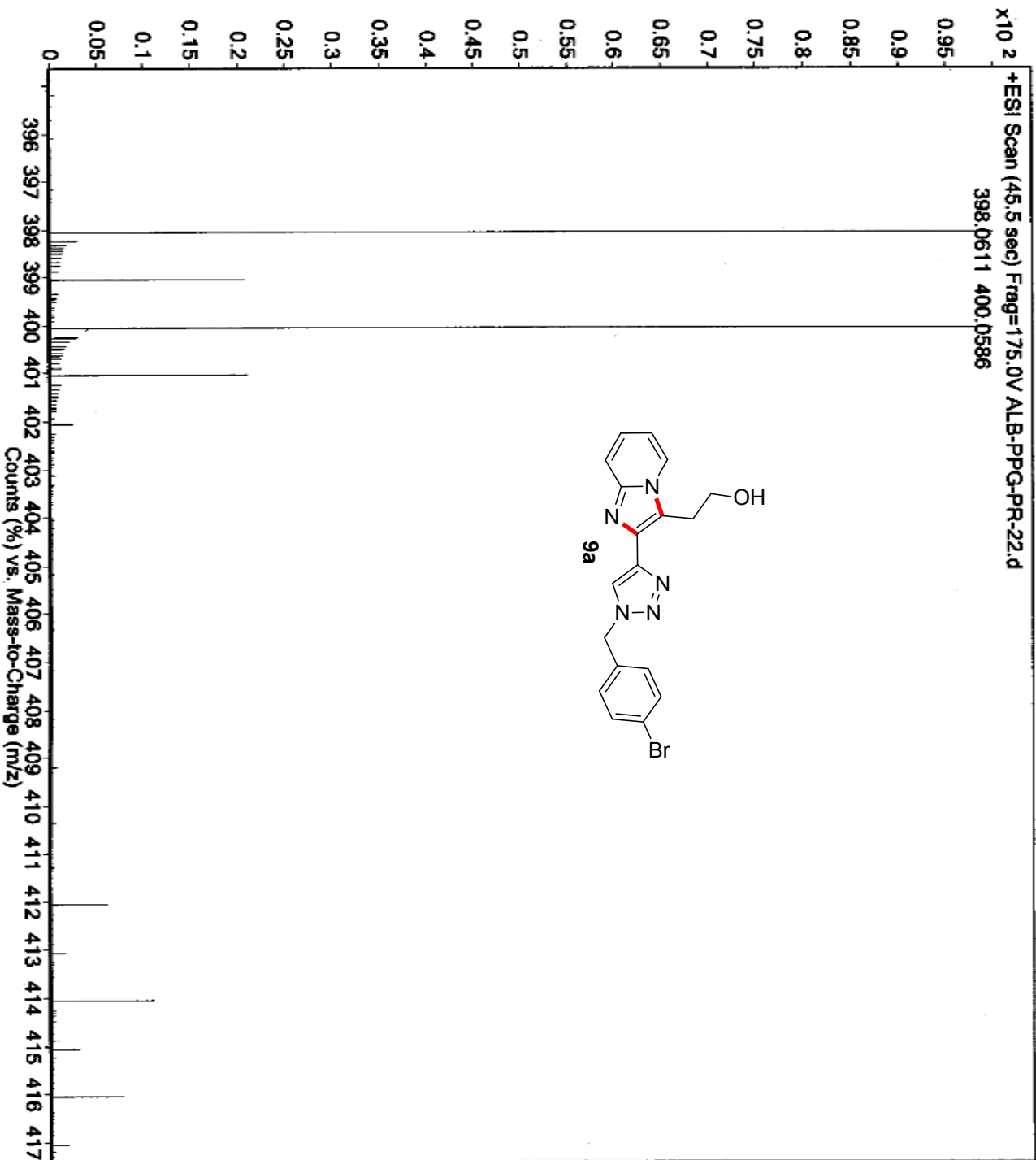
¹³C NMR spectra of 9a



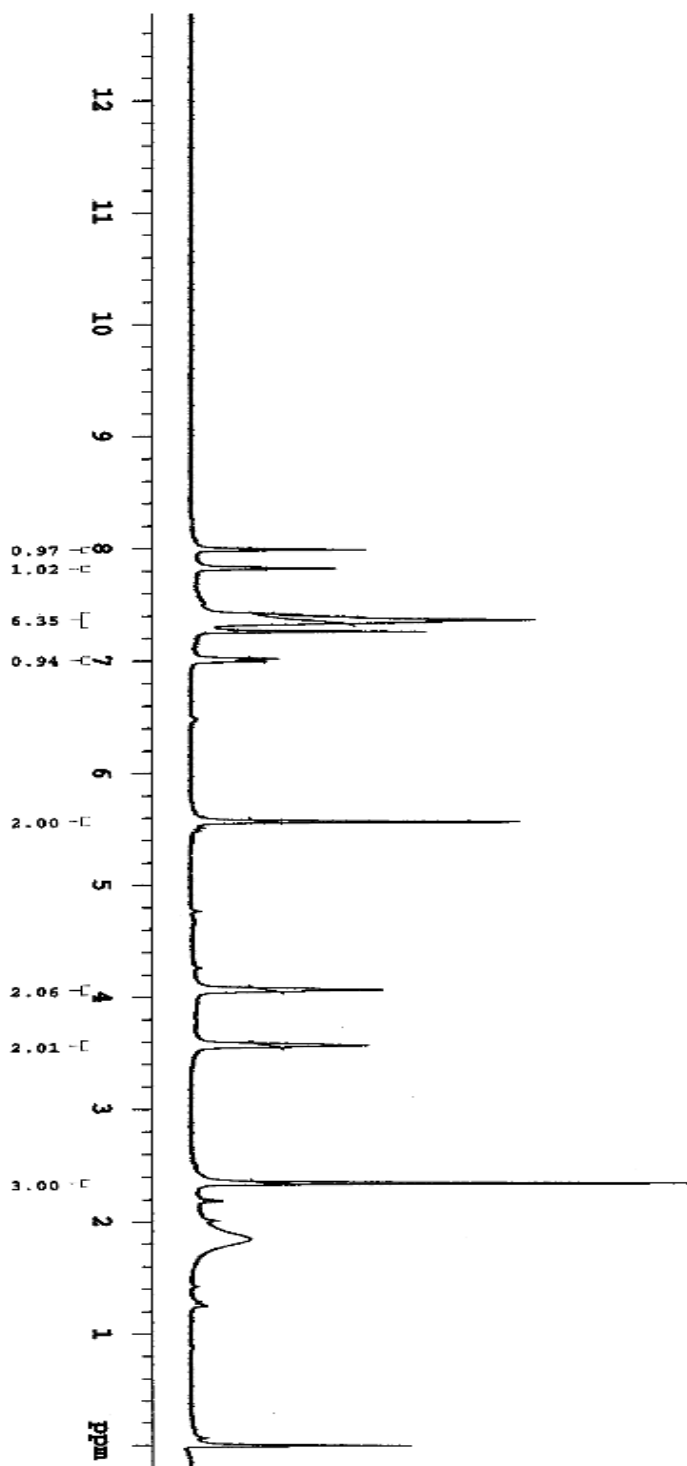
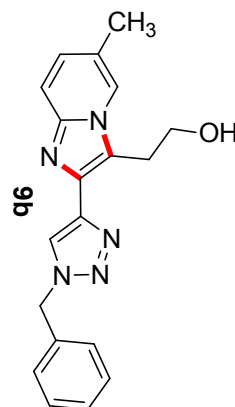
Sample Name: ALD-PP4-PP-22-13C
 Data Collected on: 1170-NMR-mercury400
 Archive directory: /export/home/chempack/mercury/data
 Sample directory:
 File(s): ALD-PP4-PP-22-13C
 Pulse Sequence: CARBON (zgpg3)
 Solvent: cdcl3
 Data collected on: Feb 7 2014
 Temp: 25.0 C / 298.1 K
 Operator: cbam
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.304 sec
 Width 25125.6 Hz
 2150 repetitions
 OBSERVE C13, 100.542809 MHz
 DECUPLE H1, 399.852994 MHz
 Power 42 db
 continuously on
 NALYZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 3 hr, 48 min

HRMS spectra of 9a

Sample Name	ALB-PPG-PR-22	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	
Data Filename	ALB-PPG-PR-22.d	ACQ Method		Comment		Acquired Time	3/20/2014 9:54:38 AM
							Success

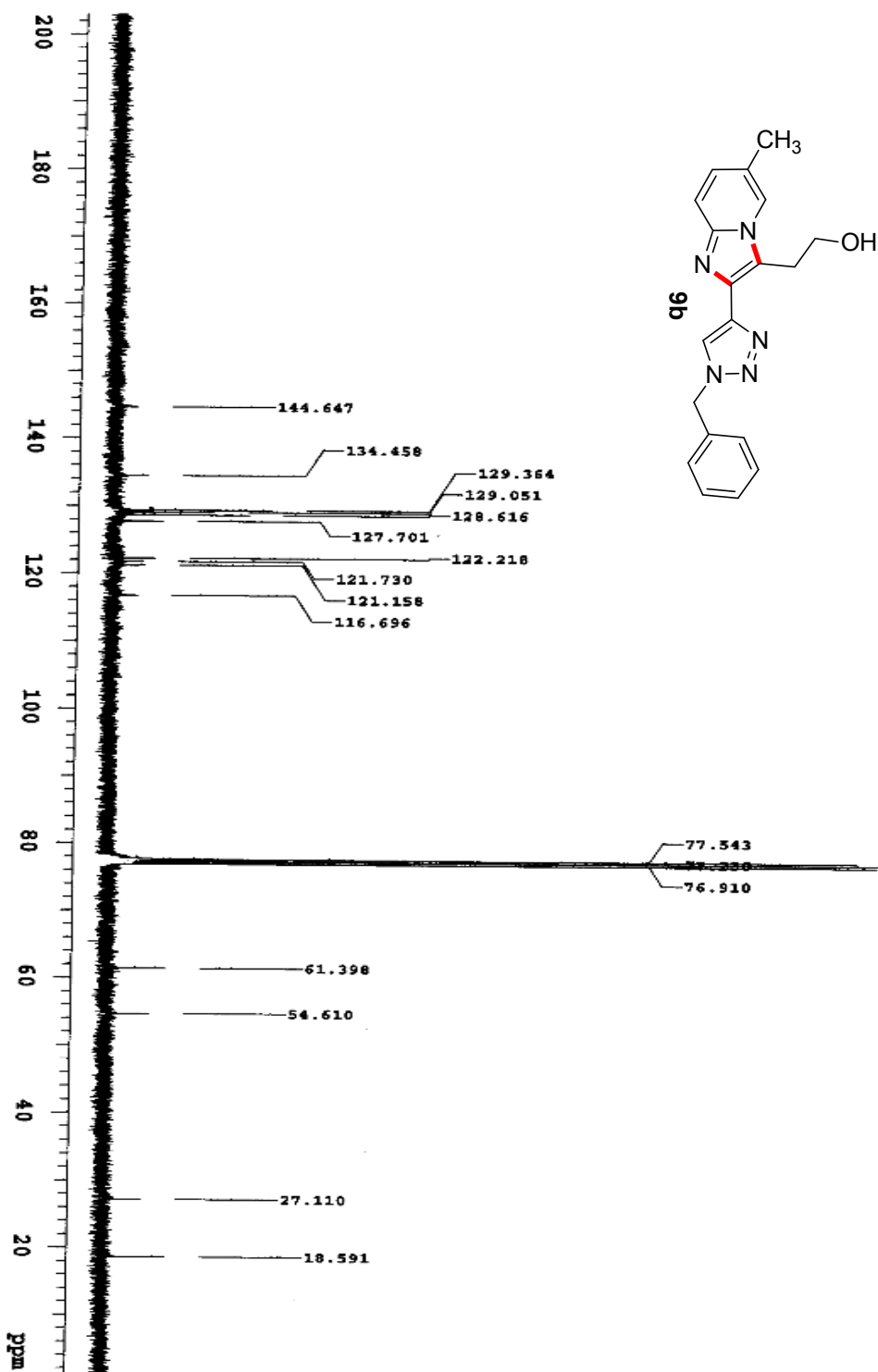
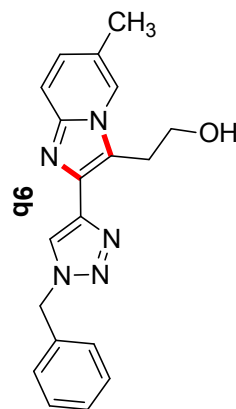


¹H NMR spectra of **9b**



<p>PROBHD SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 33 repetitions</p>	<p>OBSERVED F1, 399.8509534</p>	<p>DATA PROCESSING F1 size 32768 Total time 1 minutes</p>	<p>AXD-PPG-PR-23 Solvent: CDCl3 Temp: 25.0 C / 298.1 K Operator: chm File: AXD-PPG-PR-23R Mercury-400 1H-1MR</p>
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¹³C NMR spectra of 9b



PPHASE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.304 sec
Width 25125.6 Hz
2730 repetitions

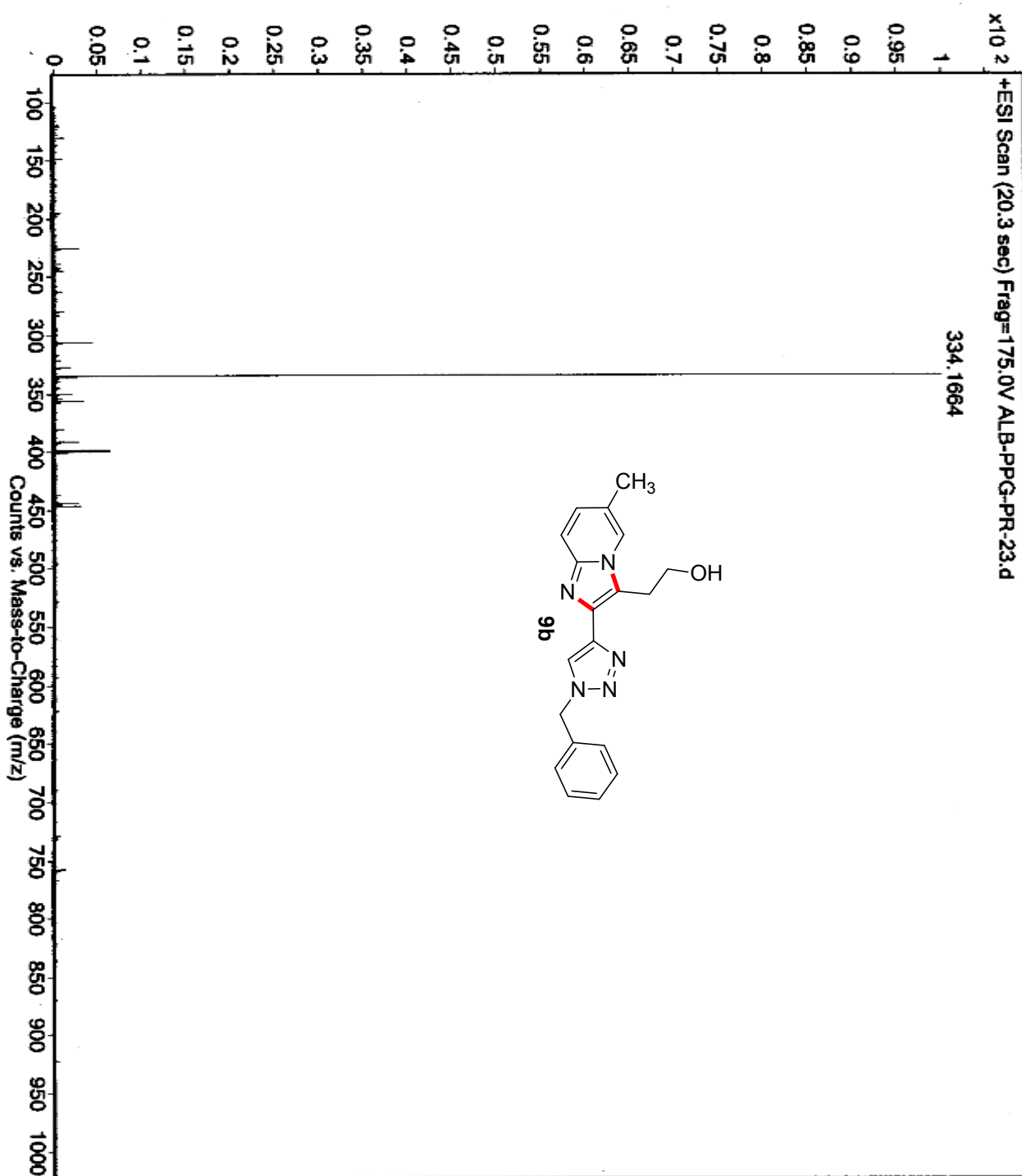
OBSERVE C13, 4005428832
DECOUPLE H1, 399.8529994
Power 42 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
line broadening 0.5 Hz
FT size 65536
Total time 104 minutes

ALD-PRQ-PR-23-45PC
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: chas
Mercury-400 "UNITY-NMR"

HRMS spectra of 9b

Sample Name	ALB-PPG-PR-23	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	Inj Position		Sample Type	Sample	IRM Calibration Status	Success
Data Filename	ALB-PPG-PR-23.d	Acq Method		Comment		Acquired Time	3/20/2014 9:56:36 AM



¹H NMR spectra of 9c

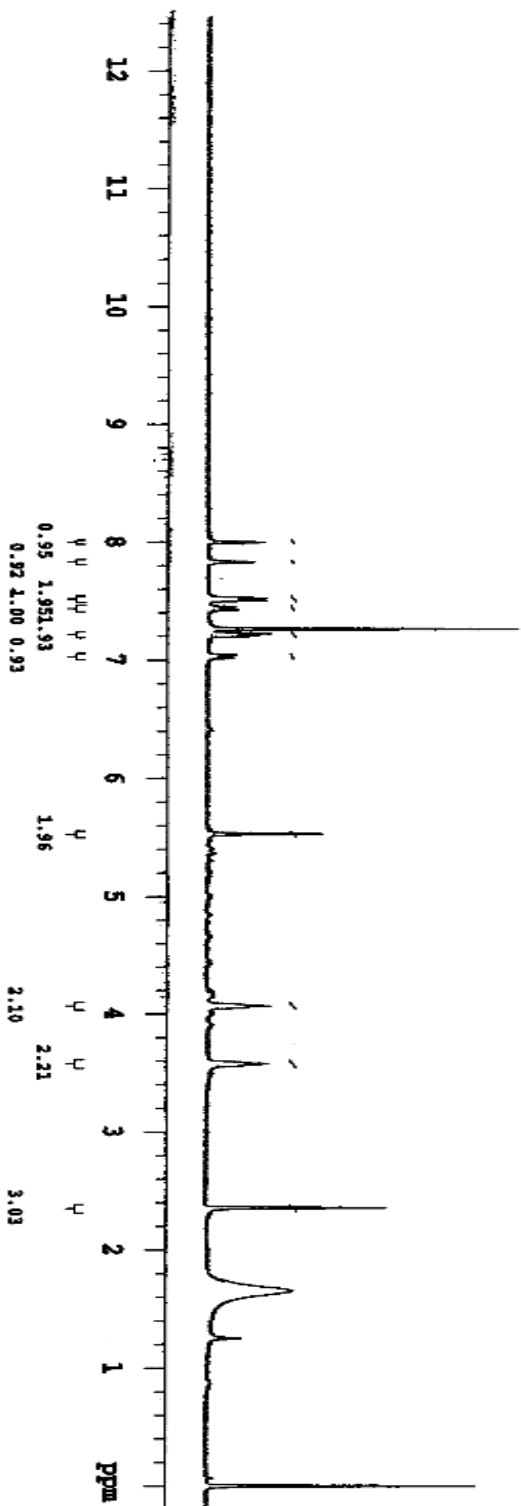
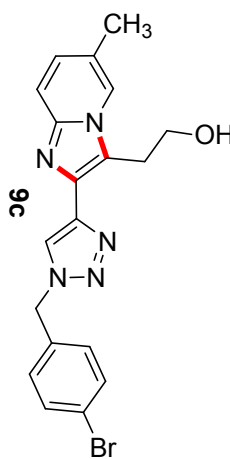
Sample Name:
MID-PPQ-PR-21
Date Collected on:
11/9-11/9-mercury400
Archive directory:
/export/home/chempack/vmurya/data
Sample directory:

Yield: MID-PPQ-PR-21A

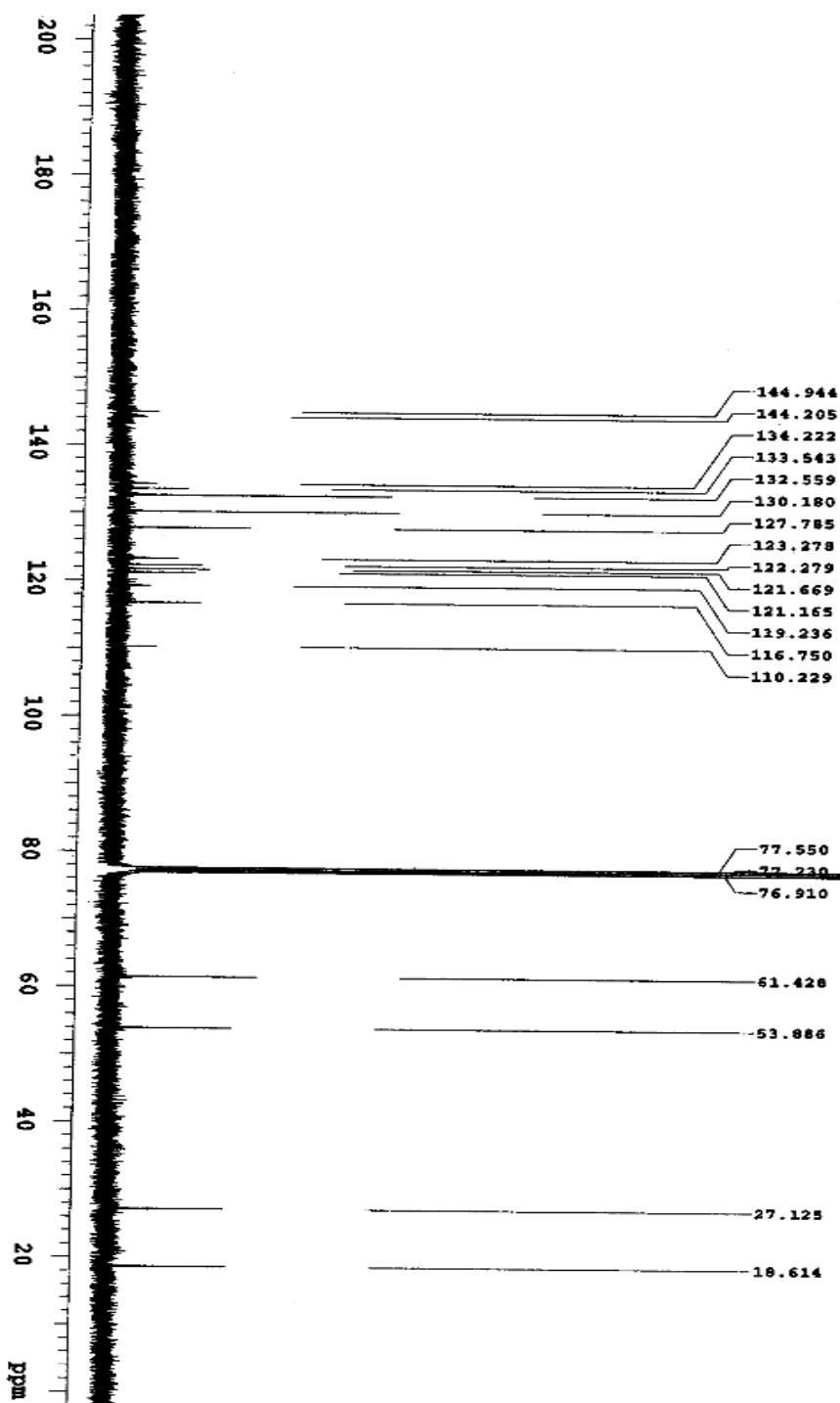
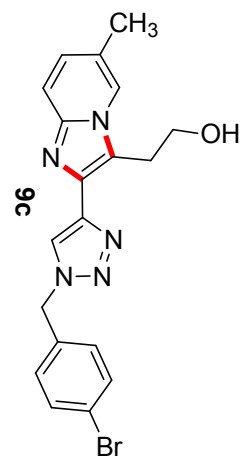
Pulse Sequence: zgpg30 (zgpg1)
Solvent: cdcl3
Data collected on: Feb 9 2014

Temp. 25.0 C / 298.1 K
Operator: cbm

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
OBSERVE: H1, 399.850637 MHz
DATA PROCESSING
PT: also 32768
Total time 2 min 12 sec



¹³C NMR spectra of 9c



EXPERIMENTAL
Pulse delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.304 sec
Width 25125.6 Hz
4000 repetitions

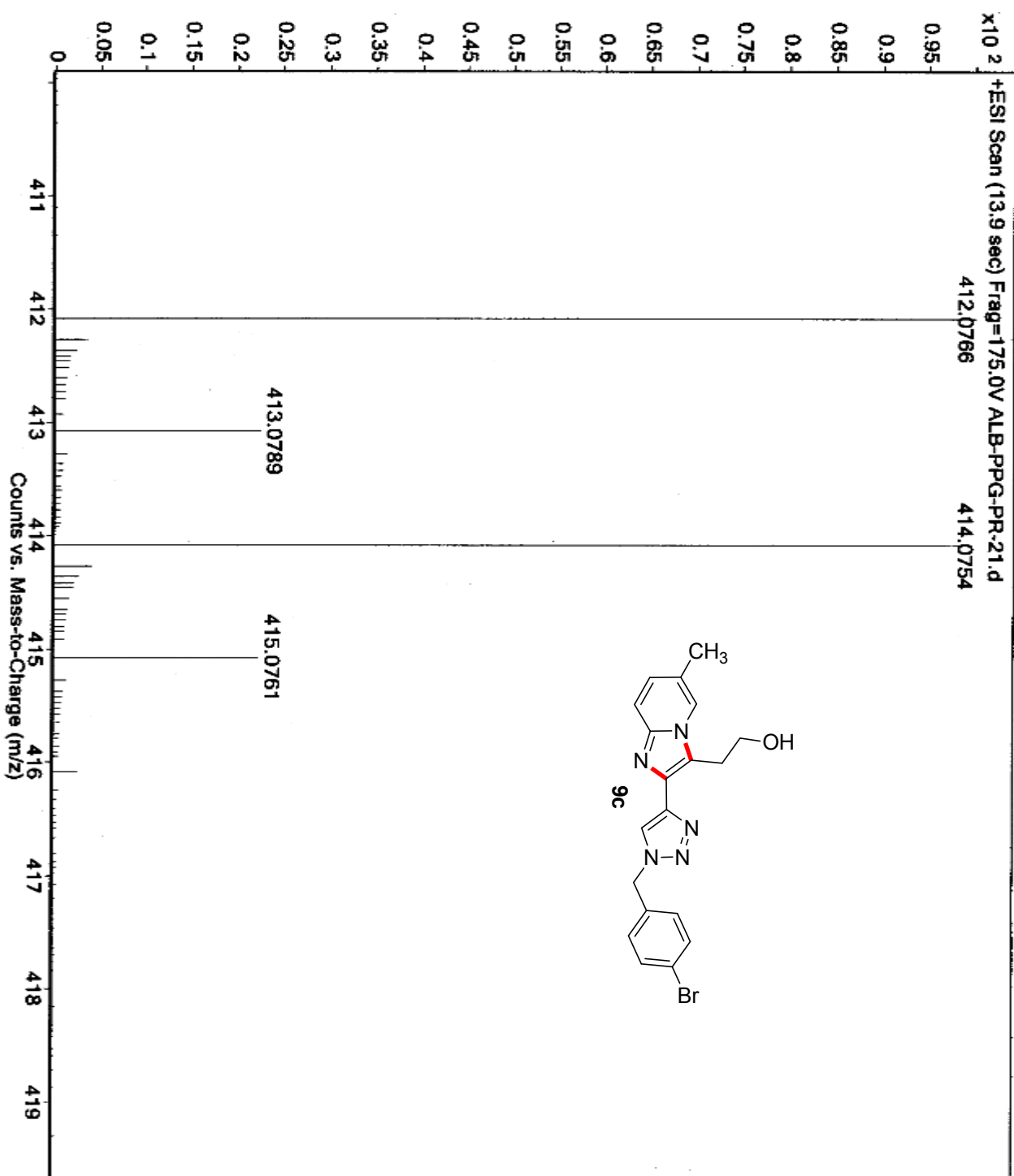
DATA ACQUISITION
DIRCONTROL: 399.8529994
Power: 48 dB
continuously on
MULTI-16 Modulated

DATA PROCESSING
Line broadening 0.5 Hz
PT also 6536
Total time 2.6 hours

NAME: 399.8529994-13C
Solvent: CDCl3
Temp: 25.0 C / 298.1 K
Operator: chm
File: 399.8529994-13C
Nucleus: 13C

HRMS spectra of 9c

Sample Name ALB-PPG-RR-21 **Position** -1
Inf Vol -10 **InfPosition**
Data Filename ALB-PPG-RR-21.d **ACQ Method**
Instrument Name Instrument 1 **User Name**
SampleType Sample **TRM Calibration Status**
Comment
Acquired Time 3/20/2014 9:50:44 AM **Success**



¹H NMR spectra of 9d

ALD-PPG-PR-32-1H

Sample Name:
ALD-PPG-PR-32-1H
Data Collected on:
ITG-NMR-mercury400
Archive directory:
/export/home/chempack/ymurys/data
Sample directory:

Filename: ALD-PPG-PR-32-1H

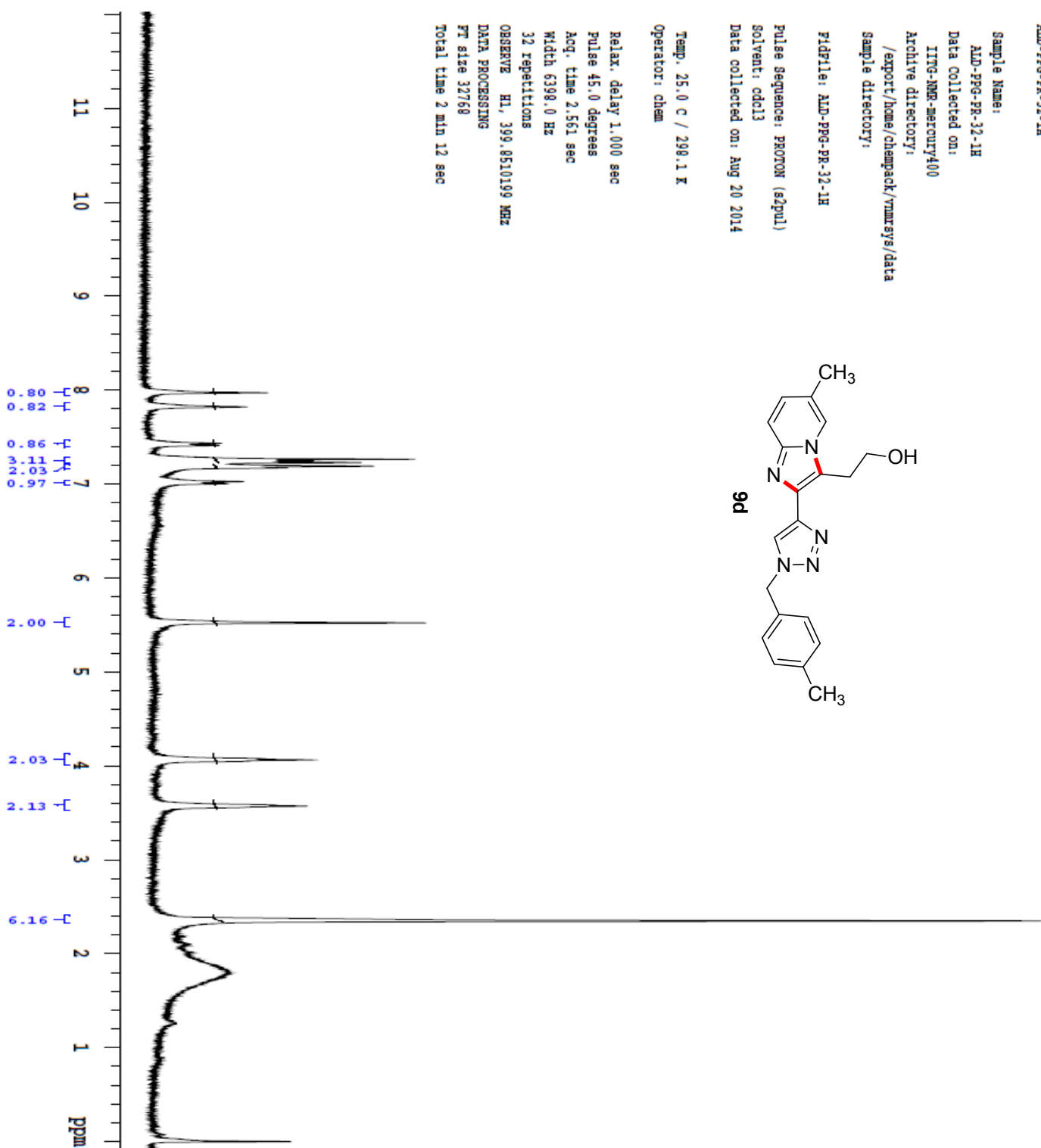
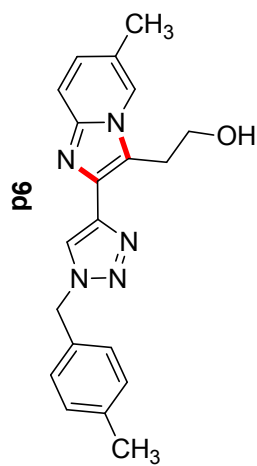
Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

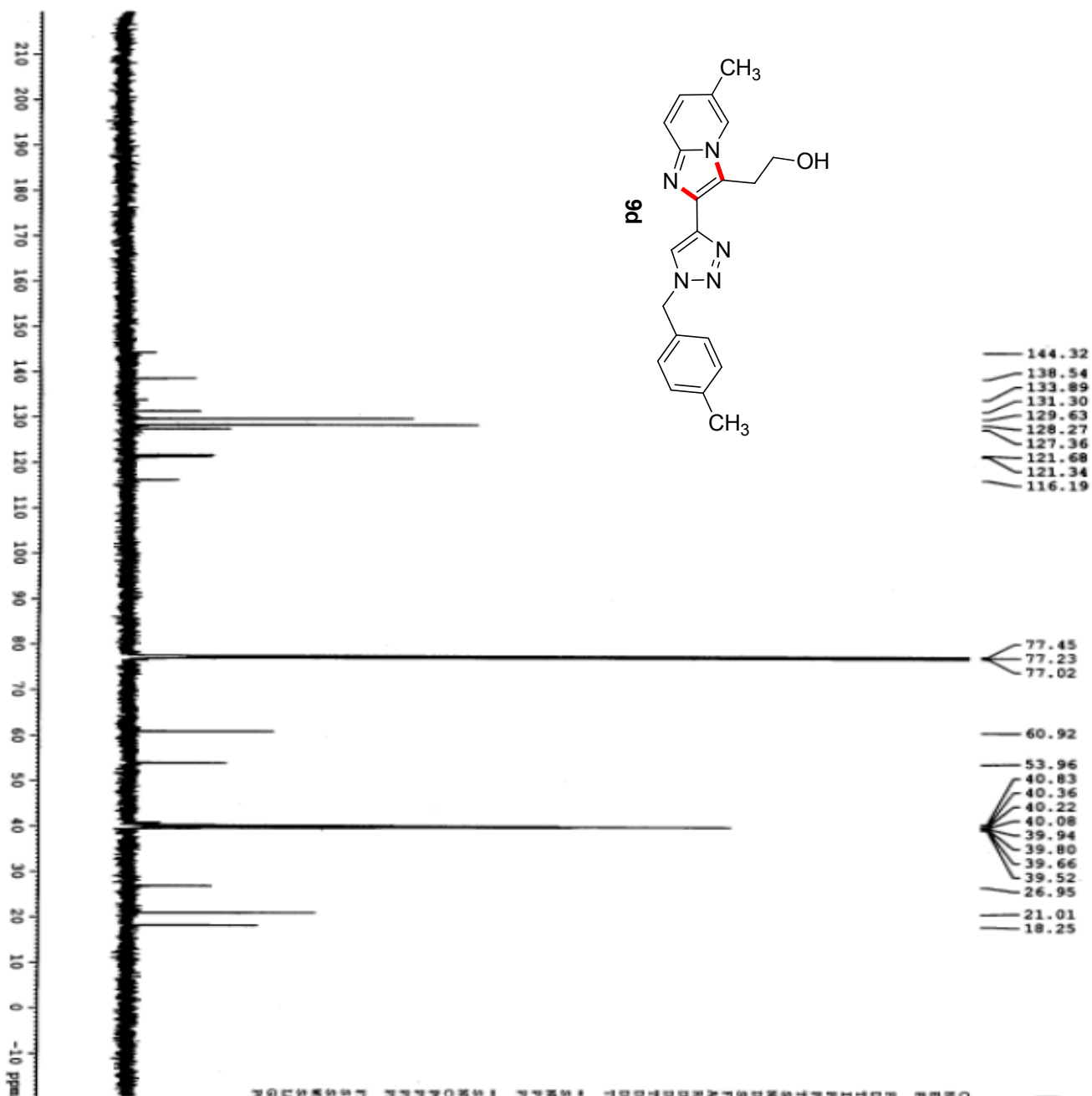
Data collected on: Aug 20 2014

Temp. 25.0 C / 298.1 K
Operator: chem

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.851019 MHz
DATA PROCESSING
FT size 32768
Total time 2 min 12 sec



¹³C NMR spectra of 9d



Current Data Parameters
 NAME ALD-990-1932_13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140821
 Time 13.46
 INSTRUM spect
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 815
 DS 2
 SWH 36057.491 MHz
 FIDRES 1.100393 MHz
 AQ 0.4543829 sec
 RG 65.34
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 T00 1

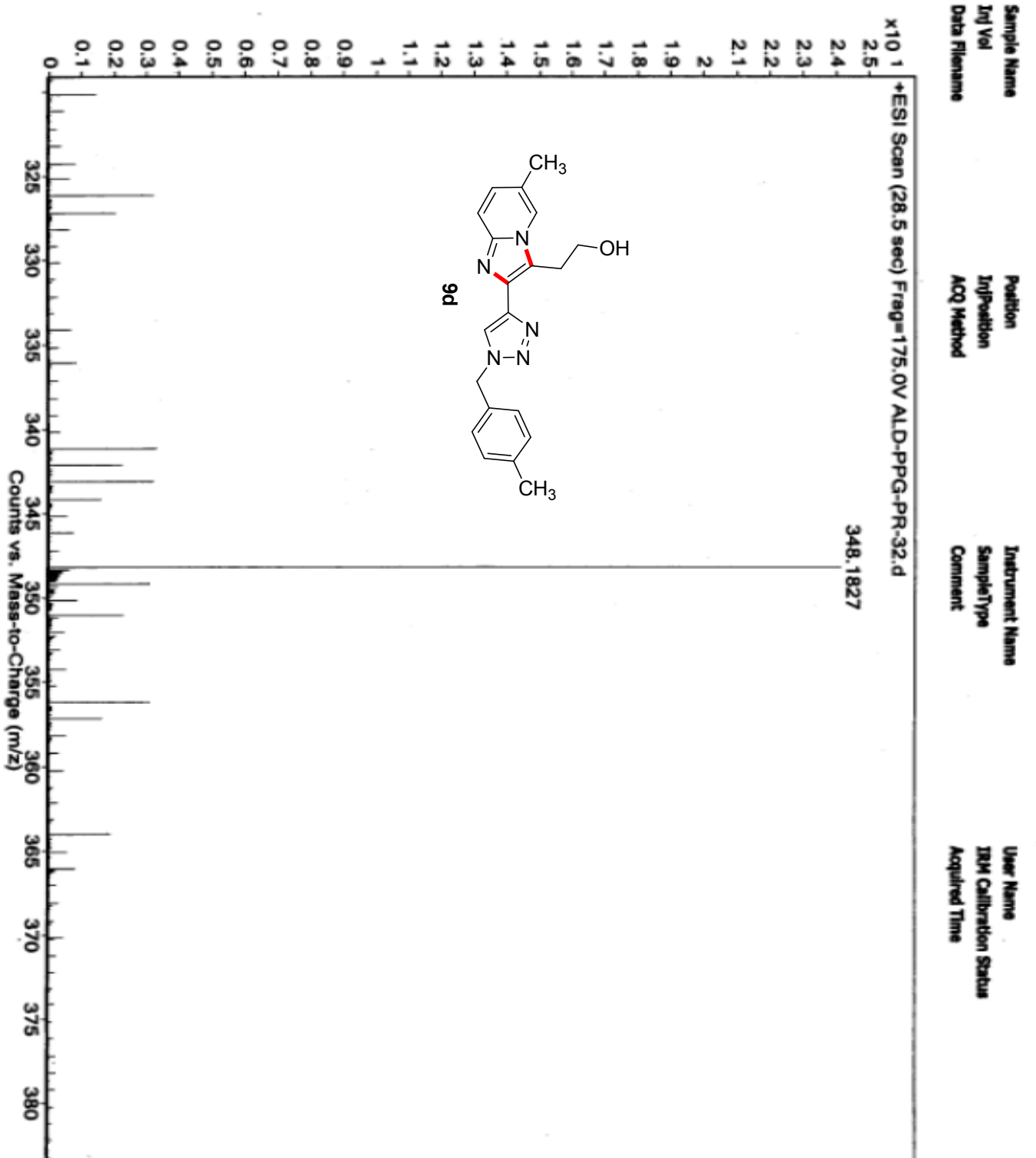
===== CHANNEL F1 =====
 SFO1 150.9579571 MHz
 NUC1 13C
 P1 10.50 usec
 PL1 95.00000000 W

===== CHANNEL F2 =====
 SFO2 600.1724007 MHz
 NUC2 1H
 CHPROG12 waltz16
 PCY2 70.00 usec
 PL2 21.00000000 W
 PL12 0.61714000 W
 PL13 0.30239999 W

F2 - Processing parameters
 SI 32768
 SF 150.9128704 MHz
 WIM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



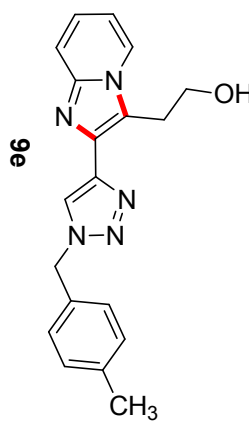
HRMS spectra of 9d



¹H NMR spectra of **9e**

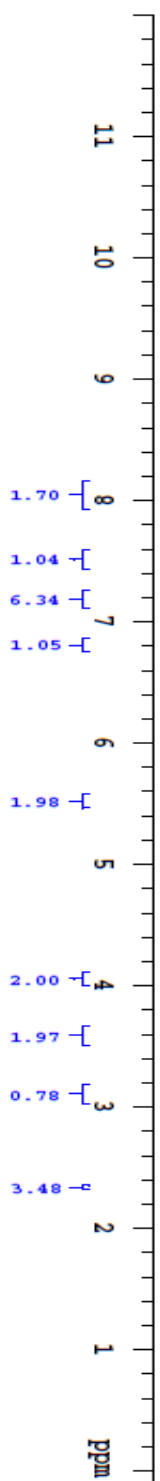
ALD-PPG-PR-34-1H

Sample Name:
ALD-PPG-PR-34-1H
Data Collected on:
IITG-NMR-mercury400
Archive directory:
/home/yumrl/yumrmys/data/fid11b
Sample directory:
Ethylandanone
FIDFile: ALD-PPG-PR-34-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 21 2014

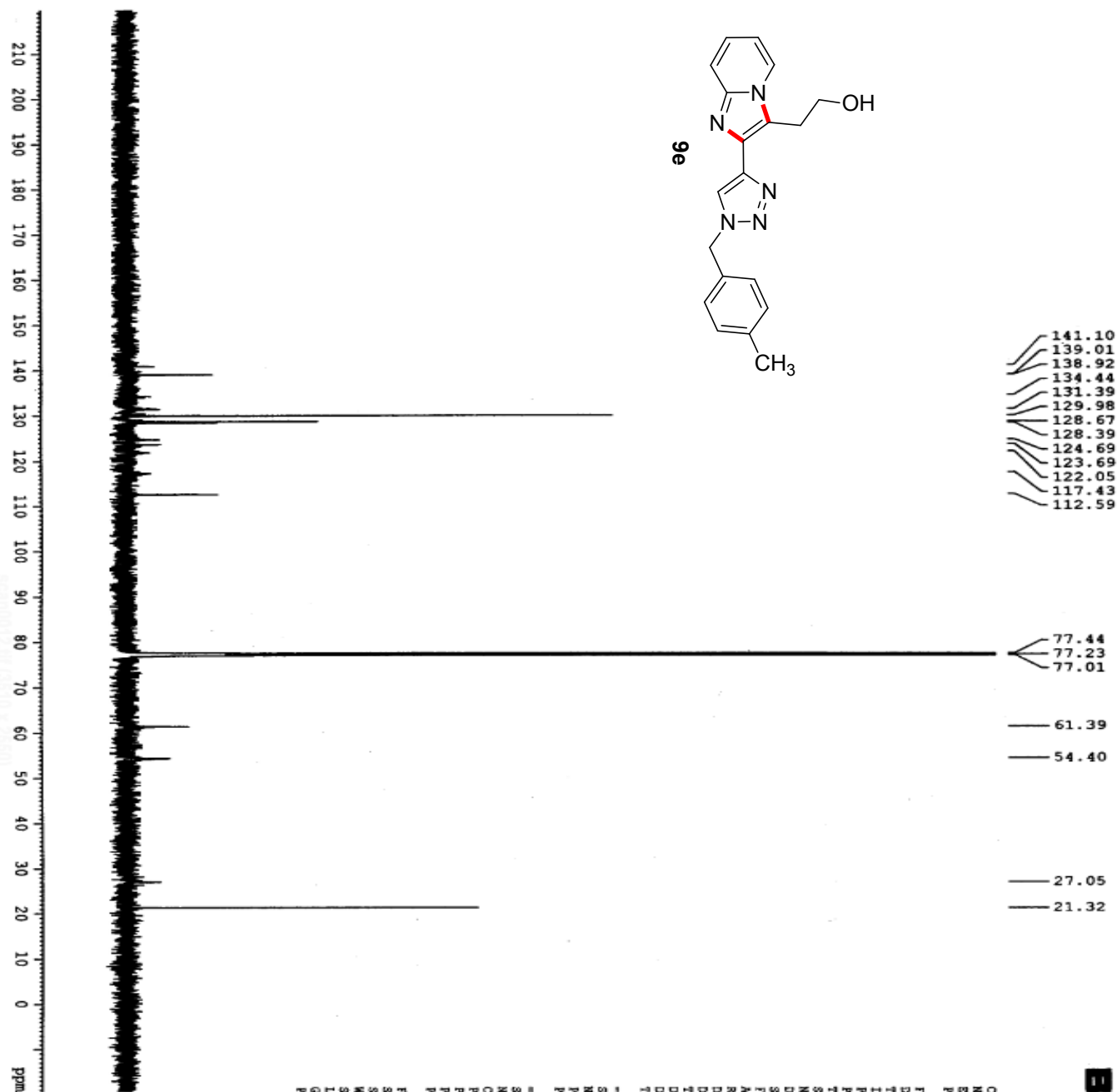
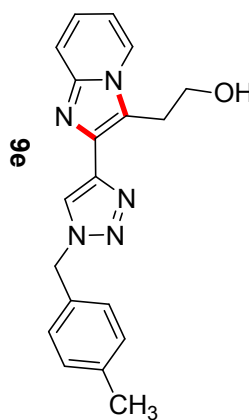


Temp. 25.0 C / 299.1 K
Operator: chem

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.8509535 MHz
DATA PROCESSING
PT. size 32768
Total time 2 min 12 sec



¹³C NMR spectra of 9e



Current Data Parameters
 NAME ALD-PFG-PR34_13C
 EXNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20160921
 Time 14:24

INSTRUM spect
 PROBRD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 32768
 SOLTVENT CDCl3
 NS 1094
 DS 2

SMH 36051.691 Hz
 FIDRES 1.100993 Hz
 AQ 0.1543829 sec
 RG 65.24
 DM 13.867 usec
 DE 6.50 usec
 TE 299.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

CHANNEL F1
 SFO1 150.9279571 MHz
 NUC1 13C
 P1 10.50 usec
 PL1 95.00000000 W

CHANNEL F2
 SFO2 600.1124007 MHz
 NUC2 1H
 CDPORG12 waltz16
 PCPD2 70.00 usec
 PAM2 21.00000000 W
 PAM12 0.61114000 W
 PAM13 0.30239999 W

F2 - Processing parameters
 SI 16384
 SF 150.9128393 MHz
 KCM KM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

HRMS spectra of 9e

