Synthesis of Phidianidines A and B

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Interpretation of Data from NCI 60 Cell Screen of Phidianidine A (1a), Phidianidine B (1b), Amine 12a, and Amine 12b. Compounds submitted to the NCI 60 Cell screen are tested initially at a single high dose (10^{-5} M) in the full NCI 60 cell panel. The one-dose data are reported as a mean graph of the percent growth of treated cells. The number reported for the one-dose assay is growth relative to the no-drug control, and relative to the time zero number of cells. This allows detection of both growth inhibition (values between 0 and 100) and lethality (values less than 0). For example, a value of 100 means no growth inhibition. A value of 40 means 60% growth inhibition. A value of 0 means no net growth over the course of the experiment. A value of -40 would mean 40% lethality. A value of -100 means all cells are dead

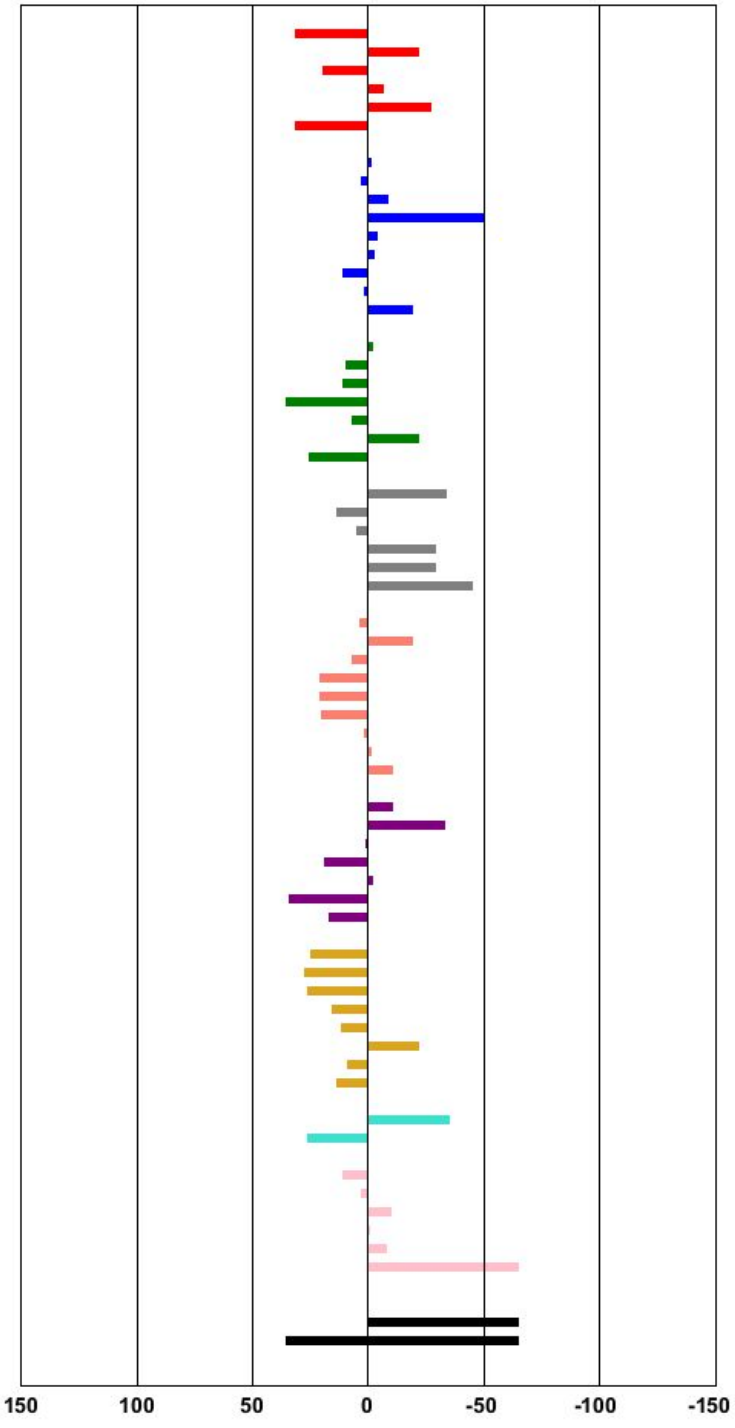
Results for phidianidine A (**1a**) in 60 cell line screen at 10^{-5} M:

Developmental Therapeutics Program

One Dose Mean Graph

NSC: 764069 / 1	Conc: 1.00E-5 Molar	Test Date: Mar 12, 2012
Experiment ID: 1203OS35		Report Date: Apr 18, 2012

Panel/Cell Line	Growth Percent
Leukemia	
CCRF-CEM	97.81
HL-60(TB)	44.86
K-562	86.42
MOLT-4	60.23
RPMI-8226	39.64
SR	98.27
Non-Small Cell Lung Cancer	
A549/ATCC	65.26
EKVX	69.56
HOP-62	58.32
HOP-92	16.18
NCI-H226	63.19
NCI-H23	64.02
NCI-H322M	77.30
NCI-H460	67.97
NCI-H522	47.79
Colon Cancer	
COLO 205	64.75
HCC-2998	76.05
HCT-116	77.19
HCT-15	101.80
HT29	73.73
KM12	45.13
SW-620	92.23
CNS Cancer	
SF-268	33.32
SF-295	80.25
SF-539	71.81
SNB-19	37.55
SNB-75	37.59
U251	21.57
Melanoma	
LOX IMVI	70.45
MALME-3M	47.79
M14	73.62
MDA-MB-435	87.73
SK-MEL-2	87.73
SK-MEL-28	86.88
SK-MEL-5	68.13
UACC-257	65.65
UACC-62	56.12
Ovarian Cancer	
IGROV1	56.34
OVCAR-3	33.39
OVCAR-4	67.52
OVCAR-5	85.35
OVCAR-8	64.62
NCI/ADR-RES	100.53
SK-OV-3	83.70
Renal Cancer	
786-0	91.71
A498	93.99
ACHN	92.64
CAKI-1	82.35
RXF 393	78.27
SN12C	44.69
TK-10	75.36
UO-31	80.23
Prostate Cancer	
PC-3	31.76
DU-145	92.84
Breast Cancer	
MCF7	77.16
MDA-MB-231/ATCC	69.43
HS 578T	56.92
BT-549	66.50
T-47D	59.19
MDA-MB-468	1.76
Mean	66.67
Delta	64.91
Range	100.04



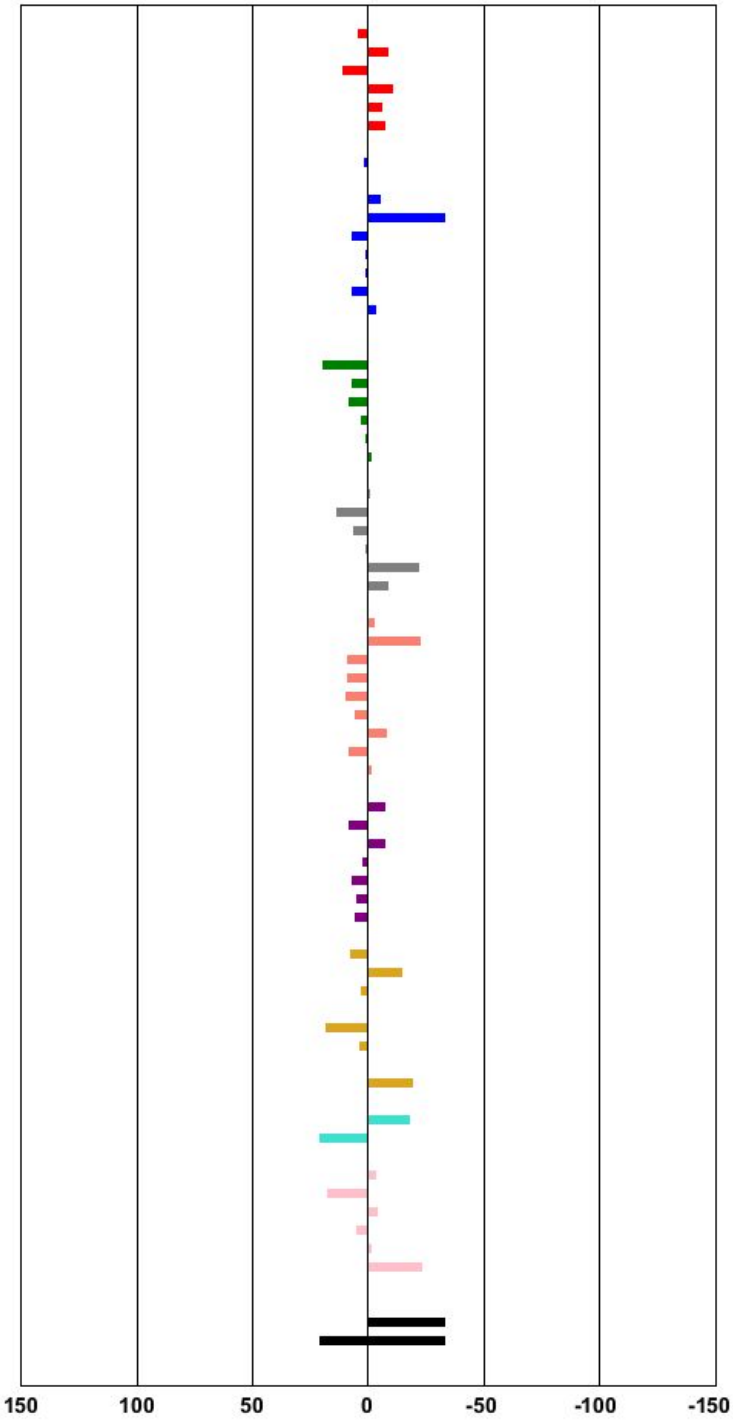
Results for phidianidine B (**1b**) in 60 cell line screen at 10^{-5} M:

Developmental Therapeutics Program

One Dose Mean Graph

NSC: 764070 / 1	Conc: 1.00E-5 Molar	Test Date: Mar 12, 2012
Experiment ID: 1203OS35		Report Date: Apr 18, 2012

Panel/Cell Line	Growth Percent
Leukemia	
CCRF-CEM	100.07
HL-60(TB)	87.05
K-562	106.81
MOLT-4	85.17
RPMI-8226	89.79
SR	88.99
Non-Small Cell Lung Cancer	
A549/ATCC	97.40
EKVX	95.73
HOP-62	90.59
HOP-92	62.79
NCI-H226	102.70
NCI-H23	96.58
NCI-H322M	96.84
NCI-H460	102.40
NCI-H522	92.94
Colon Cancer	
COLO 205	95.82
HCC-2998	114.91
HCT-116	102.34
HCT-15	104.04
HT29	98.73
KM12	96.97
SW-620	94.64
CNS Cancer	
SF-268	95.26
SF-295	109.44
SF-539	101.80
SNB-19	96.57
SNB-75	74.02
U251	87.66
Melanoma	
LOX IMVI	93.05
MALME-3M	73.32
M14	104.80
MDA-MB-435	104.73
SK-MEL-2	105.12
SK-MEL-28	101.49
SK-MEL-5	87.92
UACC-257	103.91
UACC-62	94.51
Ovarian Cancer	
IGROV1	89.00
OVCAR-3	104.07
OVCAR-4	89.02
OVCAR-5	97.69
OVCAR-8	102.35
NCI/ADR-RES	100.80
SK-OV-3	101.22
Renal Cancer	
786-0	103.28
A498	81.71
ACHN	98.92
CAKI-1	95.96
RXF 393	113.63
SN12C	99.39
TK-10	96.31
UO-31	76.93
Prostate Cancer	
PC-3	78.17
DU-145	116.74
Breast Cancer	
MCF7	92.73
MDA-MB-231/ATCC	113.18
HS 578T	91.77
BT-549	100.61
T-47D	94.99
MDA-MB-468	72.46
Mean	95.80
Delta	33.01
Range	53.95



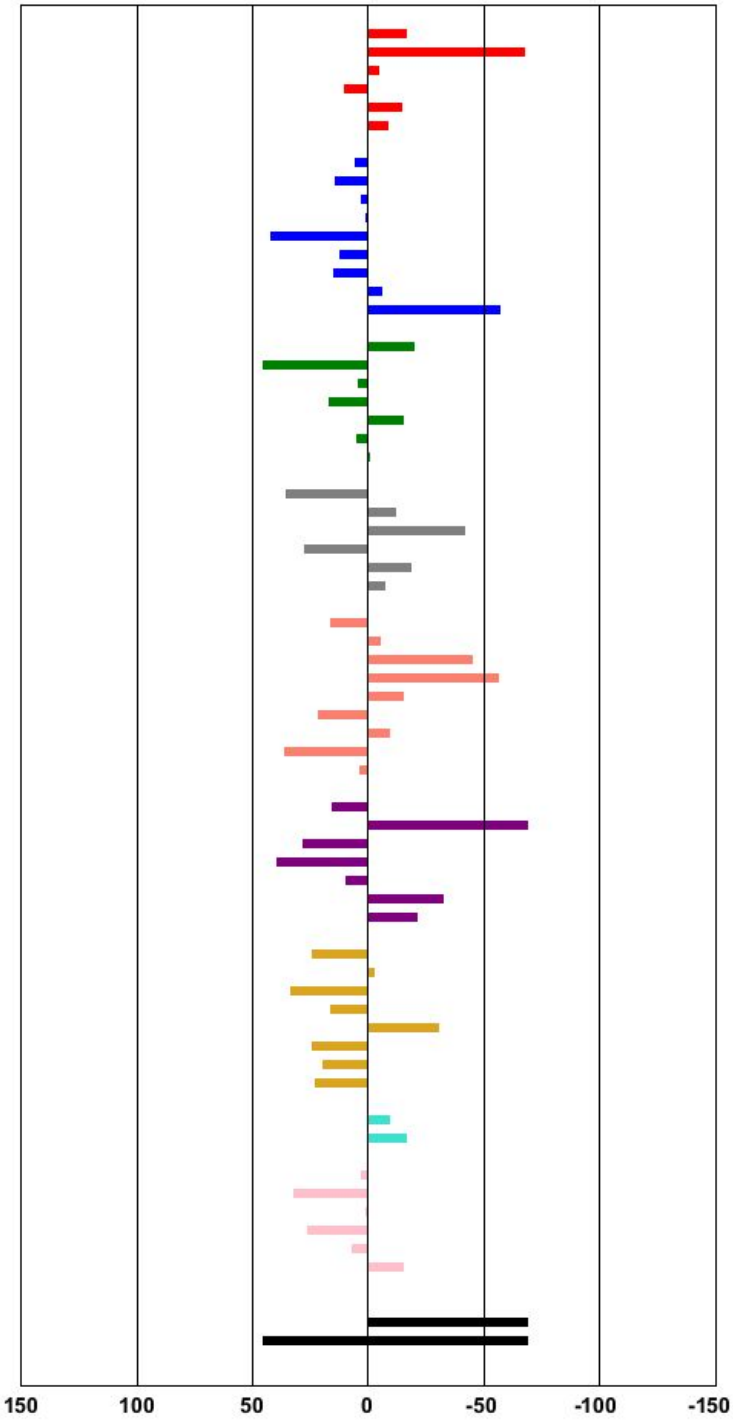
Results for phidianidine A amine precursor **12a** in 60 cell line screen at 10^{-5} M:

Developmental Therapeutics Program

One Dose Mean Graph

NSC: 764072 / 1	Conc: 1.00E-5 Molar	Test Date: Mar 12, 2012
Experiment ID: 1203OS35		Report Date: Apr 18, 2012

Panel/Cell Line	Growth Percent
Leukemia	
CCRF-CEM	2.39
HL-60(TB)	-48.83
K-562	14.29
MOLT-4	29.25
RPMI-8226	4.37
SR	10.55
Non-Small Cell Lung Cancer	
A549/ATCC	24.56
EKVX	32.98
HOP-62	21.58
HOP-92	19.68
NCI-H226	60.99
NCI-H23	30.93
NCI-H322M	33.43
NCI-H460	12.78
NCI-H522	-38.26
Colon Cancer	
COLO 205	-0.72
HCC-2998	64.01
HCT-116	23.05
HCT-15	35.71
HT29	3.86
KM12	23.92
SW-620	18.62
CNS Cancer	
SF-268	54.09
SF-295	7.26
SF-539	-23.00
SNB-19	46.17
SNB-75	0.27
U251	11.73
Melanoma	
LOX IMVI	34.87
MALME-3M	13.50
M14	-26.33
MDA-MB-435	-36.97
SK-MEL-2	4.12
SK-MEL-28	40.15
SK-MEL-5	9.74
UACC-257	54.83
UACC-62	22.60
Ovarian Cancer	
IGROV1	34.50
OVCAR-3	-50.18
OVCAR-4	46.81
OVCAR-5	58.07
OVCAR-8	28.38
NCI/ADR-RES	-13.58
SK-OV-3	-1.93
Renal Cancer	
786-0	43.25
A498	16.11
ACHN	52.18
CAKI-1	35.26
RXF 393	-11.49
SN12C	42.68
TK-10	38.61
UO-31	41.81
Prostate Cancer	
PC-3	9.96
DU-145	2.36
Breast Cancer	
MCF7	21.83
MDA-MB-231/ATCC	51.17
HS 578T	19.80
BT-549	44.89
T-47D	25.86
MDA-MB-468	4.11
Mean	18.88
Delta	69.06
Range	114.19



Results for phidianidine B amine precursor **12b** in 60 cell line screen at 10^{-5} M:

Developmental Therapeutics Program

One Dose Mean Graph

NSC: 764071 / 1	Conc: 1.00E-5 Molar	Test Date: Mar 12, 2012
Experiment ID: 1203OS35		Report Date: Apr 18, 2012

Panel/Cell Line	Growth Percent
Leukemia	
CCRF-CEM	95.38
HL-60(TB)	60.46
K-562	93.09
MOLT-4	90.60
RPMI-8226	86.16
SR	89.12
Non-Small Cell Lung Cancer	
A549/ATCC	100.67
EKVX	94.29
HOP-62	92.19
HOP-92	71.14
NCI-H226	103.95
NCI-H23	99.53
NCI-H322M	100.42
NCI-H460	115.10
NCI-H522	96.41
Colon Cancer	
COLO 205	95.69
HCC-2998	97.75
HCT-116	96.13
HCT-15	100.90
HT29	96.25
KM12	96.79
SW-620	97.31
CNS Cancer	
SF-268	100.76
SF-539	98.89
SNB-19	103.10
SNB-75	81.21
U251	91.76
Melanoma	
LOX IMVI	87.45
MALME-3M	73.38
M14	101.19
MDA-MB-435	66.92
SK-MEL-2	115.43
SK-MEL-28	101.79
SK-MEL-5	92.69
UACC-257	98.66
UACC-62	92.33
Ovarian Cancer	
IGROV1	98.32
OVCAR-3	103.48
OVCAR-4	93.26
OVCAR-5	98.46
OVCAR-8	98.68
NCI/ADR-RES	99.08
SK-OV-3	97.44
Renal Cancer	
786-0	110.10
A498	104.39
ACHN	99.16
CAKI-1	95.69
RXF 393	100.49
SN12C	92.39
TK-10	109.77
UO-31	69.90
Prostate Cancer	
PC-3	85.46
DU-145	109.19
Breast Cancer	
MCF7	87.91
MDA-MB-231/ATCC	84.23
HS 578T	91.33
BT-549	110.31
T-47D	96.04
MDA-MB-468	91.52
Mean	94.94
Delta	34.48
Range	54.97

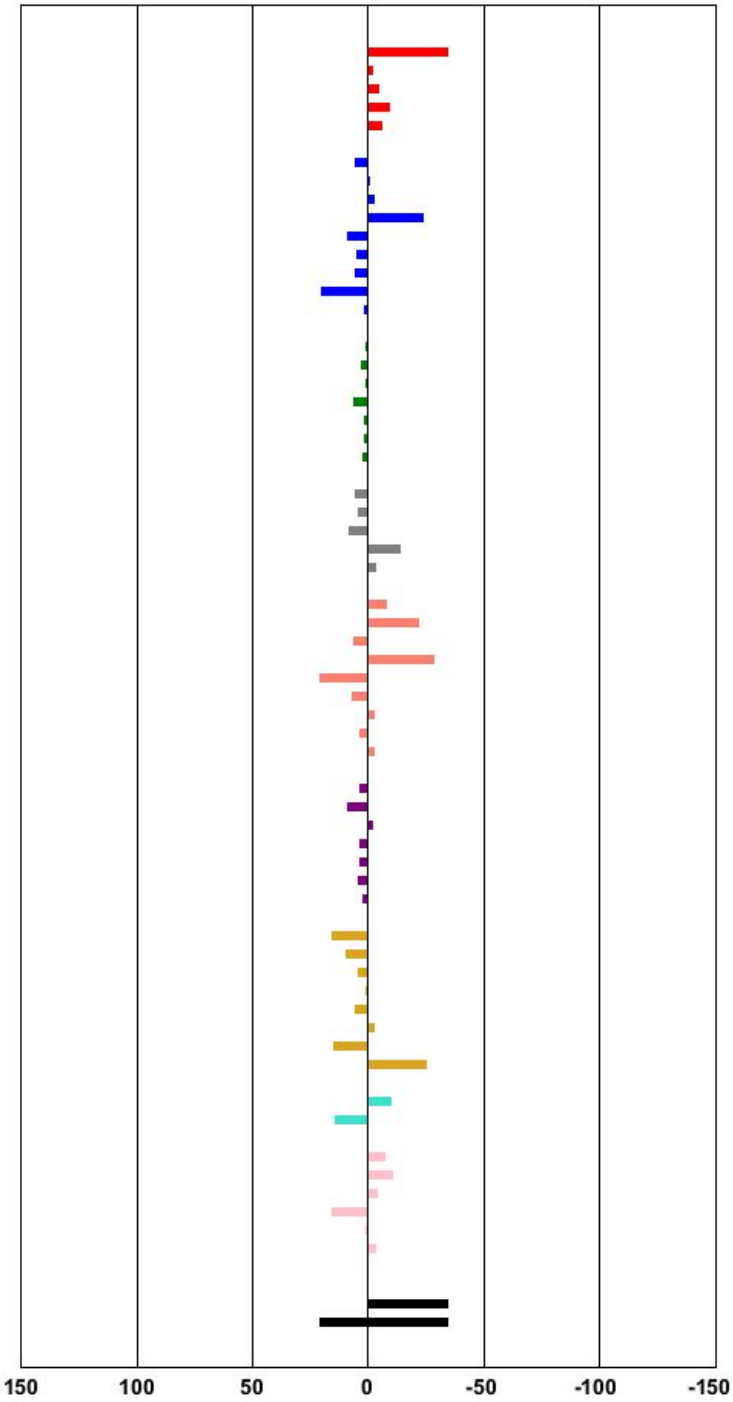
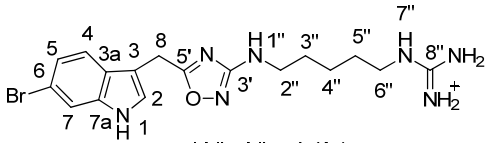


Table S1. Comparison of the Spectral Data of Natural and Synthetic Phidianidine A (**1a**). The Numbering Scheme is Shown Below.

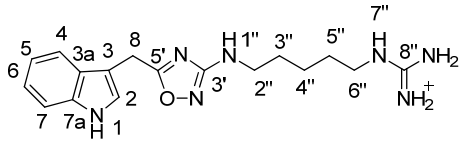


phidianidine A (**1a**)

Atom	Natural[a] DMSO-d_6 ^{13}C NMR	Synthetic DMSO-d_6 ^{13}C NMR	Natural[a] CD_3OD ^{13}C NMR	Synthetic CD_3OD ^{13}C NMR	Natural[a] DMSO-d_6 ^{1}H NMR	Synthetic DMSO-d_6 ^{1}H NMR	Natural[a] CD_3OD ^{1}H NMR	Synthetic CD_3OD ^{1}H NMR
1	---	---	---	---	11.15	11.18	---	---
2	125.0	125.3	124.4[b]	125.9	7.35	7.35	7.27	7.23
3	106.8[b]	107.4	108.4	108.8	---	---	---	---
3a	125.2	125.8	126.6	127.3	---	---	---	---
4	120.0	120.2	121.1	121.0	7.46 (d, 8)	7.47 (d, 8)	7.48 (d, 8)	7.45 (d, 8)
5	121.3	121.6	122.4[b]	123.4	7.13 (dd, 8, 1)	7.14 (dd, 8, 1)	7.16 (dd, 8, 1)	7.13 (dd, 8, 1)
6	113.5[c]	114.0	115.1	115.5	---	---	---	---
7	114.1	114.2	115.8	116.3	7.56 (d, 1)	7.56 (d, 1)	7.55 (d, 1)	7.52 (d, 1)
7a	137.0	137.0	138.6	139.1	---	---	---	---
8	22.5	22.5	23.8	24.1	4.20 (s)	4.20	4.23	4.20
3'	168.5	168.5	169.0[c]	170.3	---	---	---	---
5'	176.7	176.7	179.9[b]	179.1	---	---	---	---
1"	---	---	---	---	6.72	6.73	---	---
2"	40.5	40.7	43.0[b]	42.5	2.99	3.00	3.16	3.15
3"	28.1	28.1	29.5	29.7	1.36-1.56	1.42-1.56	1.62	1.66-1.54
4"	23.4	23.3	24.5	25.1	1.28	1.22-1.34	1.44	1.46-1.36
5"	28.1	28.1	29.7	29.9	1.36-1.56	1.42-1.56	1.62	1.66-1.54
6"	41.9[b]	42.3	43.5[b]	43.9	3.02	3.06	3.18	3.15
7"	---	---	---	---	7.41	7.49	---	---
8"	156.6	156.7	158.6	158.8	---	---	---	---

[a] Data taken from reference 1. [b] The reported data in reference 1 are not consistent with the spectrum provided in the supporting material. [c] This peak does not appear in the ^{13}C NMR spectrum and was determined less accurately from the HMBC spectrum.

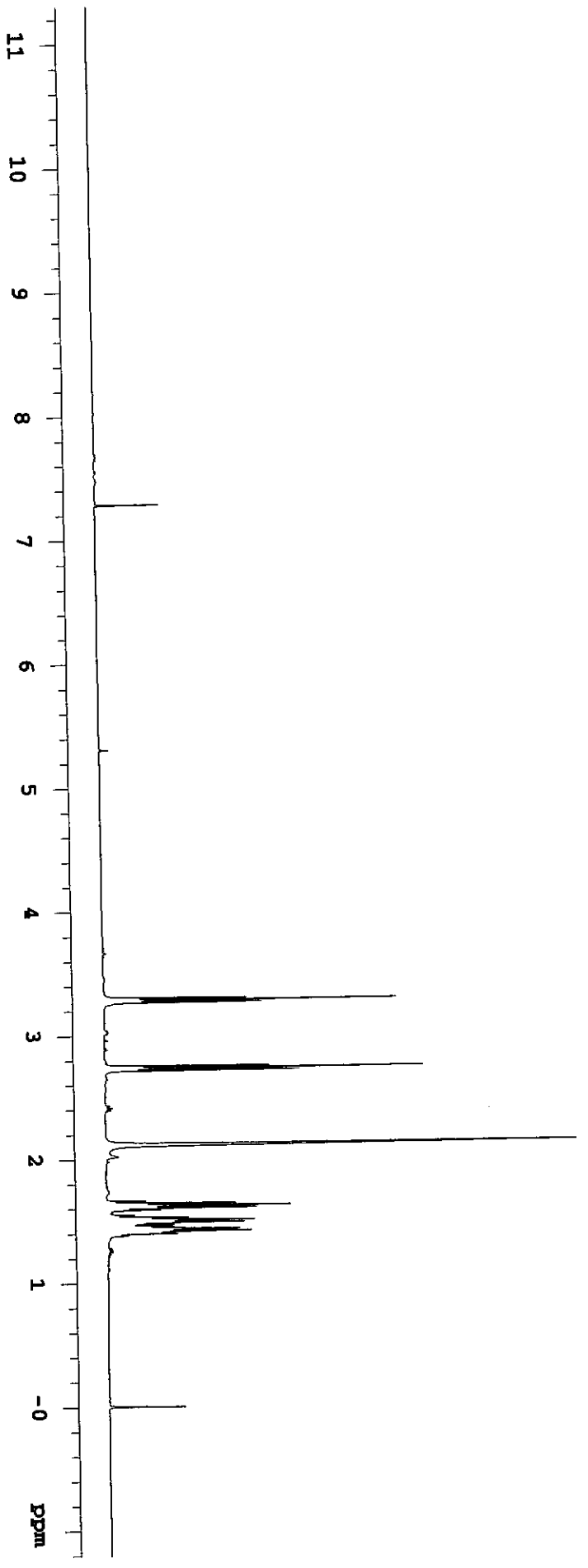
Table S2. Comparison of the Spectral Data of Natural and Synthetic Phidianidine B (**1b**). The Numbering Scheme is Shown Below.



phidianidine B (**1b**)

Atom	Natural[a] DMSO-d_6 ^{13}C NMR	Synthetic DMSO-d_6 ^{13}C NMR	Natural[a] CD_3OD ^{13}C NMR	Synthetic CD_3OD ^{13}C NMR	Natural[a] DMSO-d_6 ^{1}H NMR	Synthetic DMSO-d_6 ^{1}H NMR	Natural[a] CD_3OD ^{1}H NMR	Synthetic CD_3OD ^{1}H NMR
1	---	---	---	---	11.00	11.04	---	---
2	125.3[b]	124.2	124.4	124.8	7.31	7.31	7.28[b]	7.21
3	106.9	106.9	107.9	108.3	---	---	---	---
3a	126.7	126.7	127.9	128.3	---	---	---	---
4	118.3	118.4	118.9	119.4	7.50 (d, 8)	7.51 (d, 8)	7.38 (d, 8)	7.35 (d, 8)
5	118.6	118.7	119.6	120.2	6.99 (dd, 8, 8)	6.99 (dd, 8, 8)	7.04 (dd, 8, 8)	7.01 (dd, 8, 8)
6	120.2[b]	121.2	122.4	122.9	7.09 (dd, 8, 8)	7.09 (dd, 8, 8)	7.14 (dd, 8, 8)	7.11 (dd, 8, 8)
7	111.5	111.5	112.1	112.5	7.36 (d, 8)	7.37 (d, 8)	7.57 (d, 8)	7.52 (d, 8)
7a	136.1	136.2	137.6	138.2	---	---	---	---
8	22.7	22.7	23.8	24.2	4.20 (s)	4.20	4.23	4.22
3'	168.5	168.5	169.9	170.2	---	---	---	---
5'	176.9	176.9	179.0	179.5	---	---	---	---
1"	---	---	---	---	6.71 (t, 5)	6.72(t, 5)	---	---
2"	40.7	40.7	43.0[b]	42.5	3.00	3.01	3.16	3.14
3"	28.1	28.1	29.1[b]	29.7	1.36-1.56	1.40-.156	1.62	1.68-1.54
4"	23.4	23.4	24.8	25.0	1.28	1.25-1.35	1.44	1.46-1.36
5"	28.1	28.1	29.1[b]	29.9	1.36-1.56	1.42-1.56	1.62	1.68-1.54
6"	42.3	42.3	43.5[b]	43.9	3.05	3.06	3.18	3.15
7"	---	---	---	---	7.43	7.55	---	---
8"	156.6	156.7	158.6	158.8	---	---	---	---

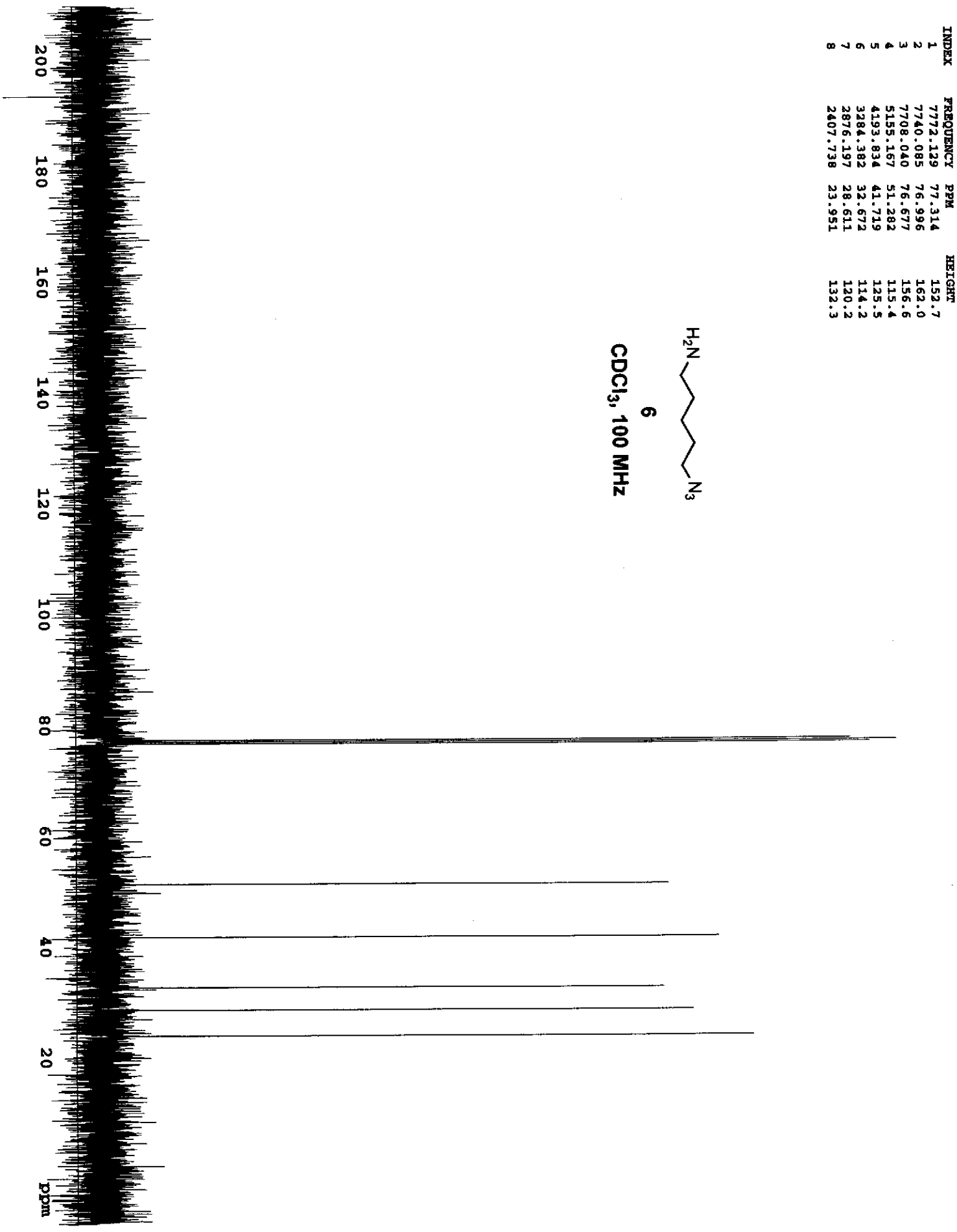
[(a)] Data taken from reference 1. [(b)] The reported data in reference 1 are not consistent with the spectrum provided in the supporting material.

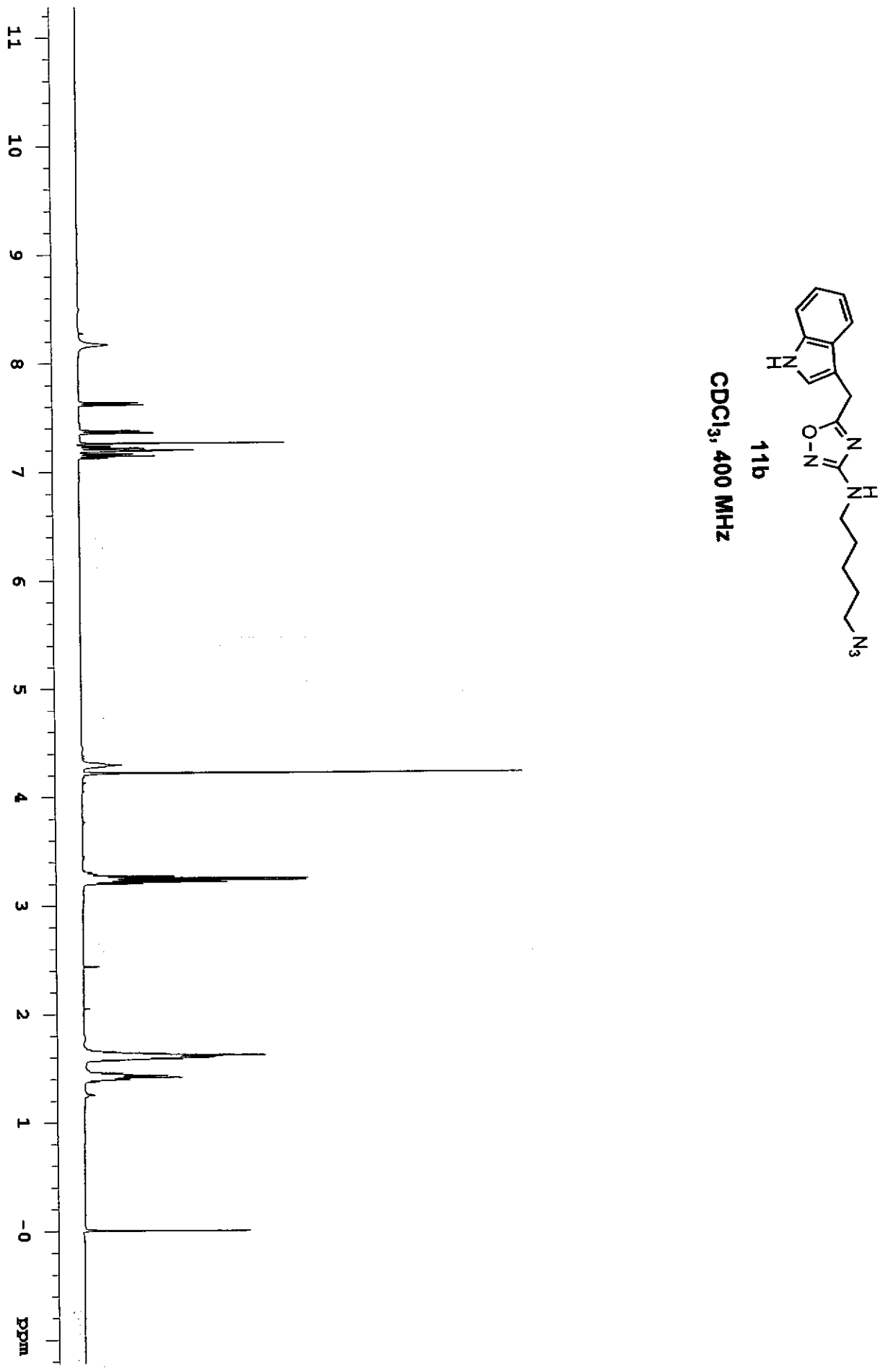


H_2N N_3

6

$CDCl_3$, 400 MHz

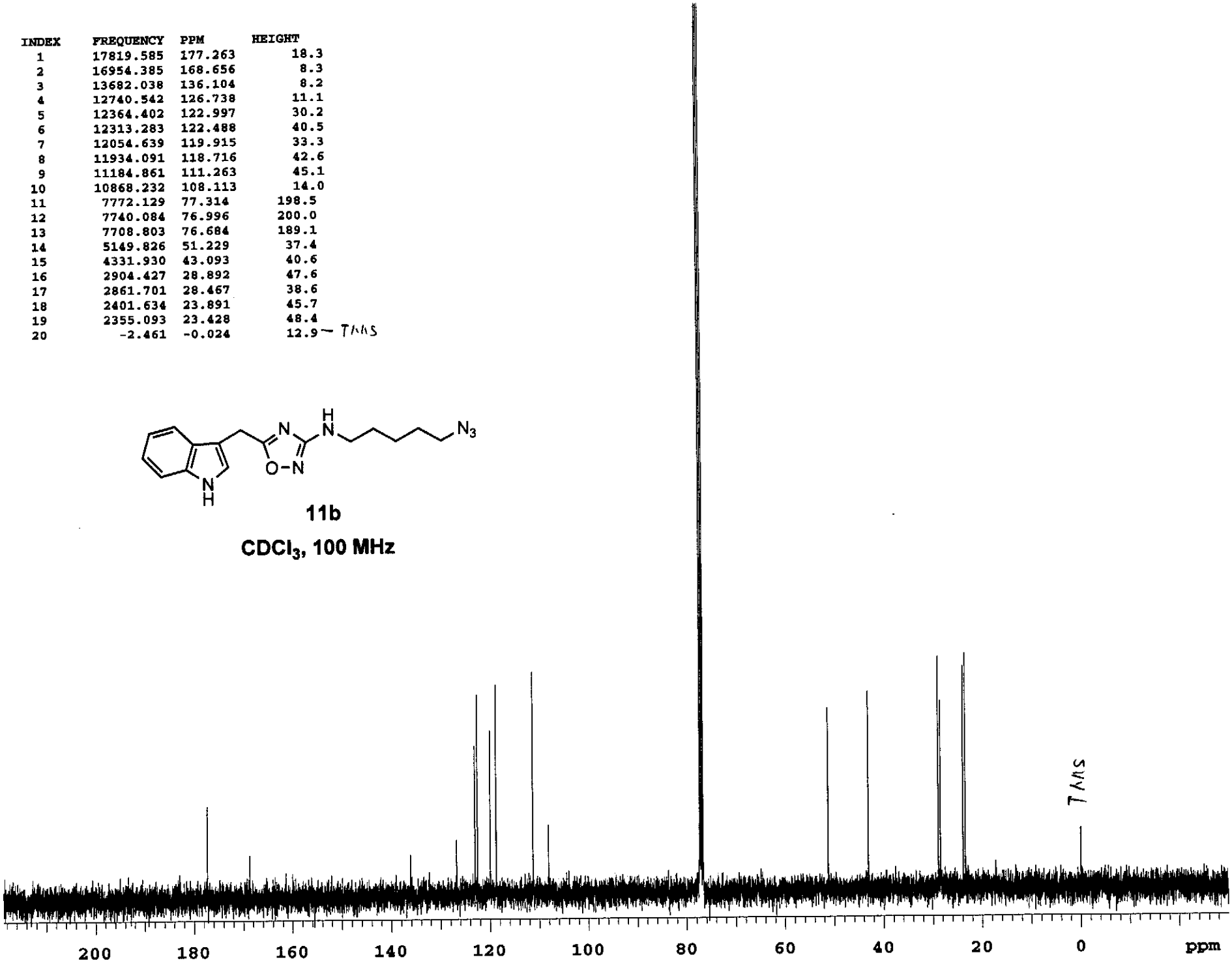

INDEX FREQUENCY PPM HEIGHT
1 7772.129 77.314 152.7
2 7740.085 76.996 162.0
3 7708.040 76.677 156.6
4 5155.167 51.282 115.4
5 4193.834 41.719 125.5
6 3284.382 32.672 114.2
7 2876.197 28.611 120.2
8 2407.738 23.951 132.3
H2N
N3
6
CDCl3, 100 MHz
200
180
160
140
120
100
80
60
40
20
ppm

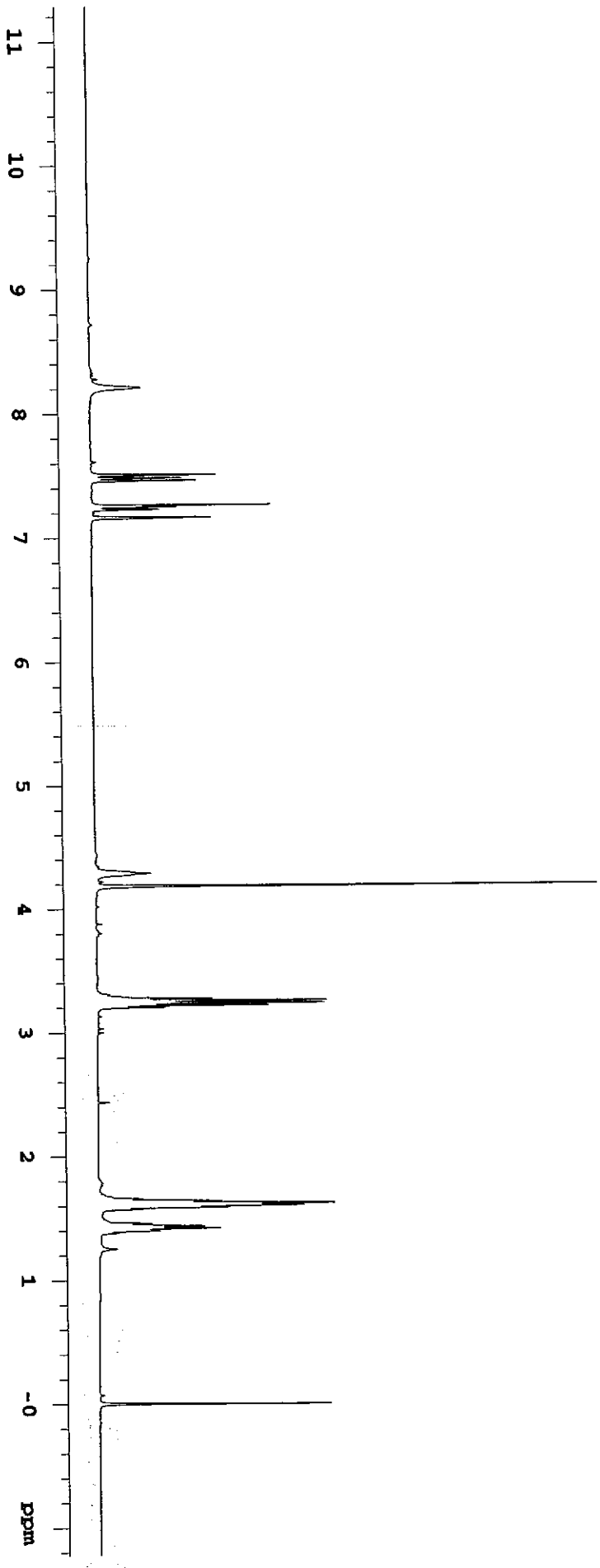

11
10
9
8
7
6
5
4
3
2
1
0
ppm
11b
CDCl3, 400 MHz

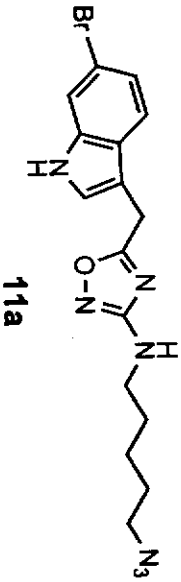
INDEX	FREQUENCY	PPM	HEIGHT
1	17819.585	177.263	18.3
2	16954.385	168.656	8.3
3	13682.038	136.104	8.2
4	12740.542	126.738	11.1
5	12364.402	122.997	30.2
6	12313.283	122.488	40.5
7	12054.639	119.915	33.3
8	11934.091	118.716	42.6
9	11184.861	111.263	45.1
10	10868.232	108.113	14.0
11	7772.129	77.314	198.5
12	7740.084	76.996	200.0
13	7708.803	76.684	189.1
14	5149.826	51.229	37.4
15	4331.930	43.093	40.6
16	2904.427	28.892	47.6
17	2861.701	28.467	38.6
18	2401.634	23.891	45.7
19	2355.093	23.428	48.4
20	-2.461	-0.024	12.9 — TMS

11b

$CDCl_3$, 100 MHz







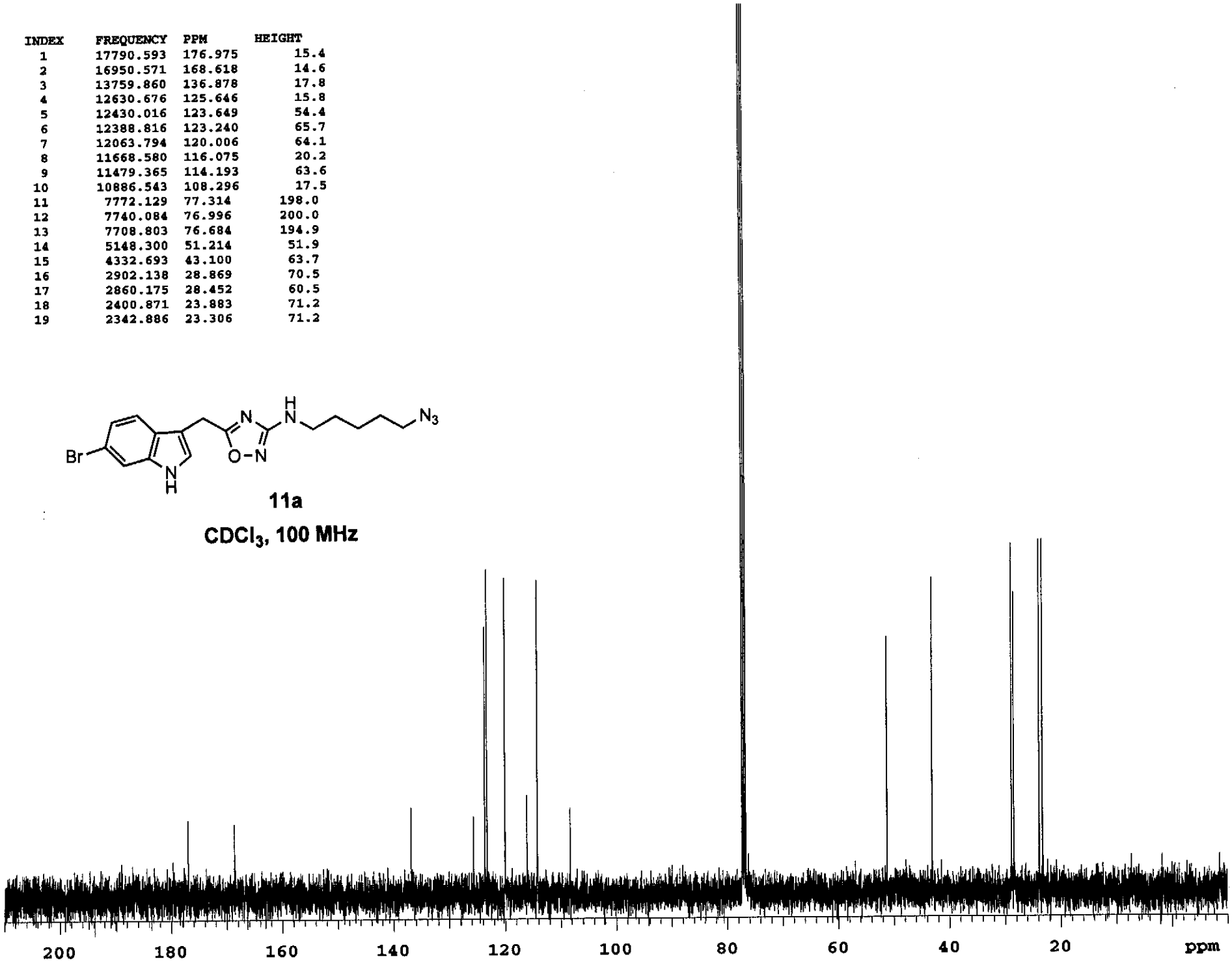
11a

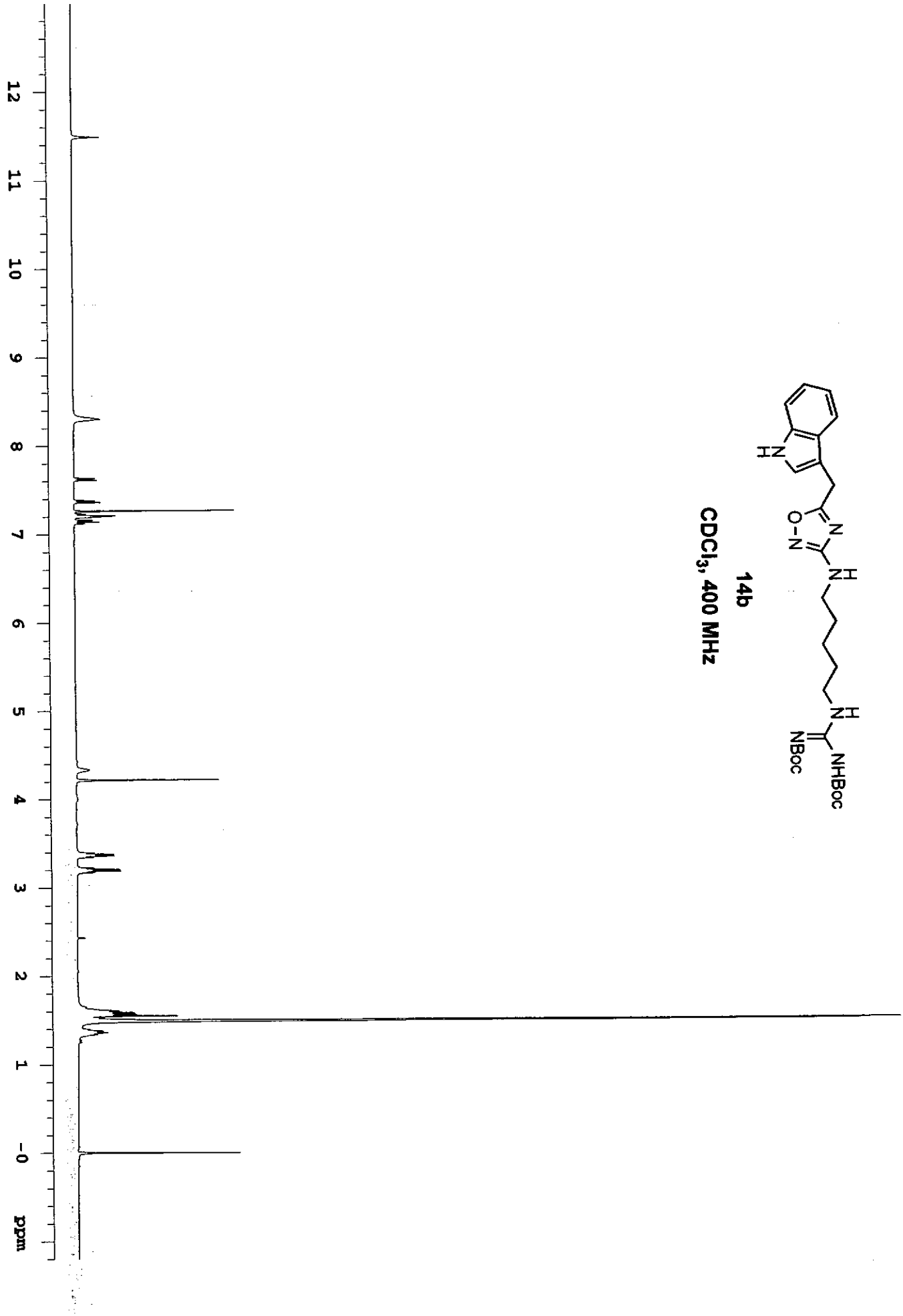
$CDCl_3$, 400 MHz

INDEX	FREQUENCY	PPM	HEIGHT
1	17790.593	176.975	15.4
2	16950.571	168.618	14.6
3	13759.860	136.878	17.8
4	12630.676	125.646	15.8
5	12430.016	123.649	54.4
6	12388.816	123.240	65.7
7	12063.794	120.006	64.1
8	11668.580	116.075	20.2
9	11479.365	114.193	63.6
10	10886.543	108.296	17.5
11	7772.129	77.314	198.0
12	7740.084	76.996	200.0
13	7708.803	76.684	194.9
14	5148.300	51.214	51.9
15	4332.693	43.100	63.7
16	2902.138	28.869	70.5
17	2860.175	28.452	60.5
18	2400.871	23.883	71.2
19	2342.886	23.306	71.2

11a

$CDCl_3$, 100 MHz

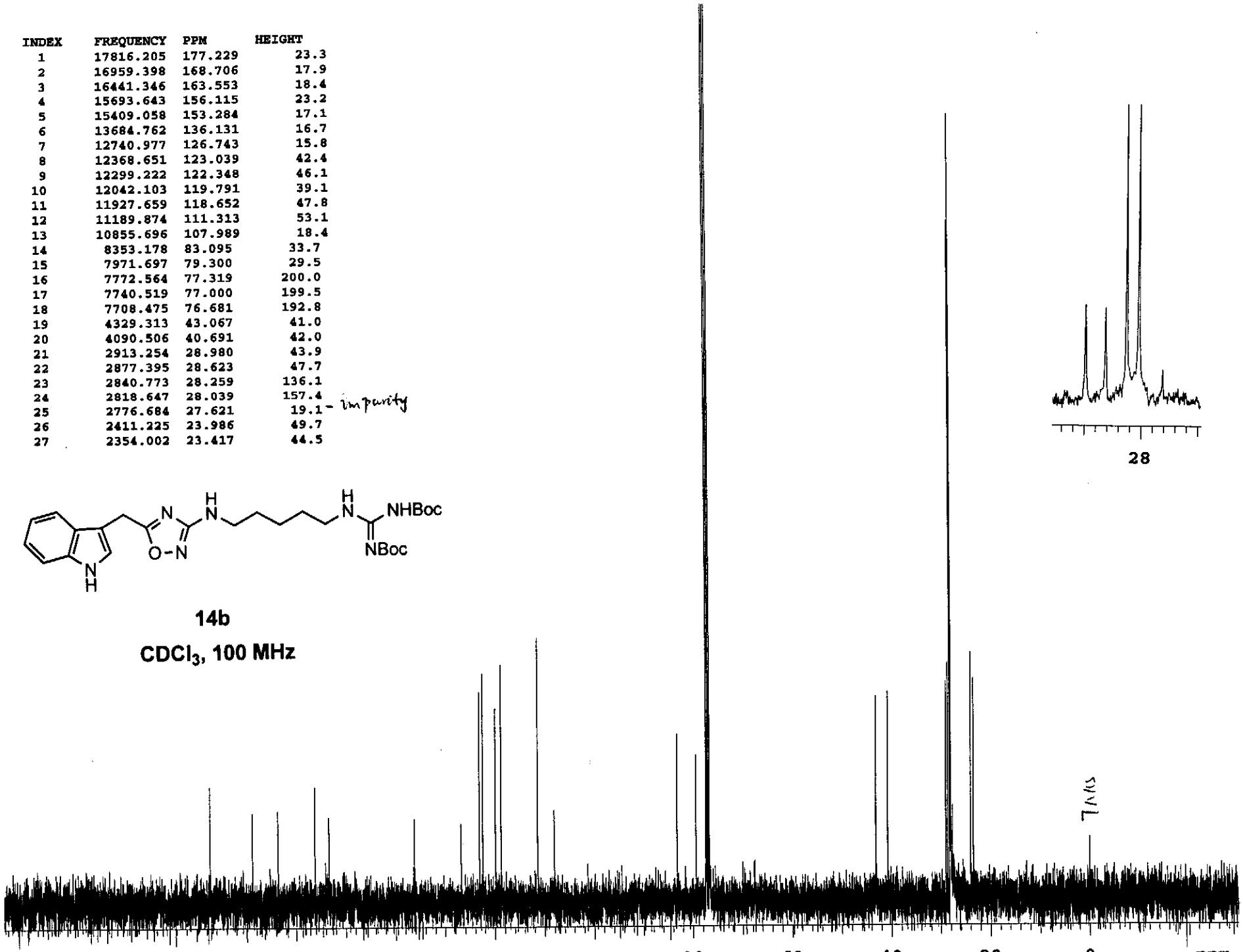


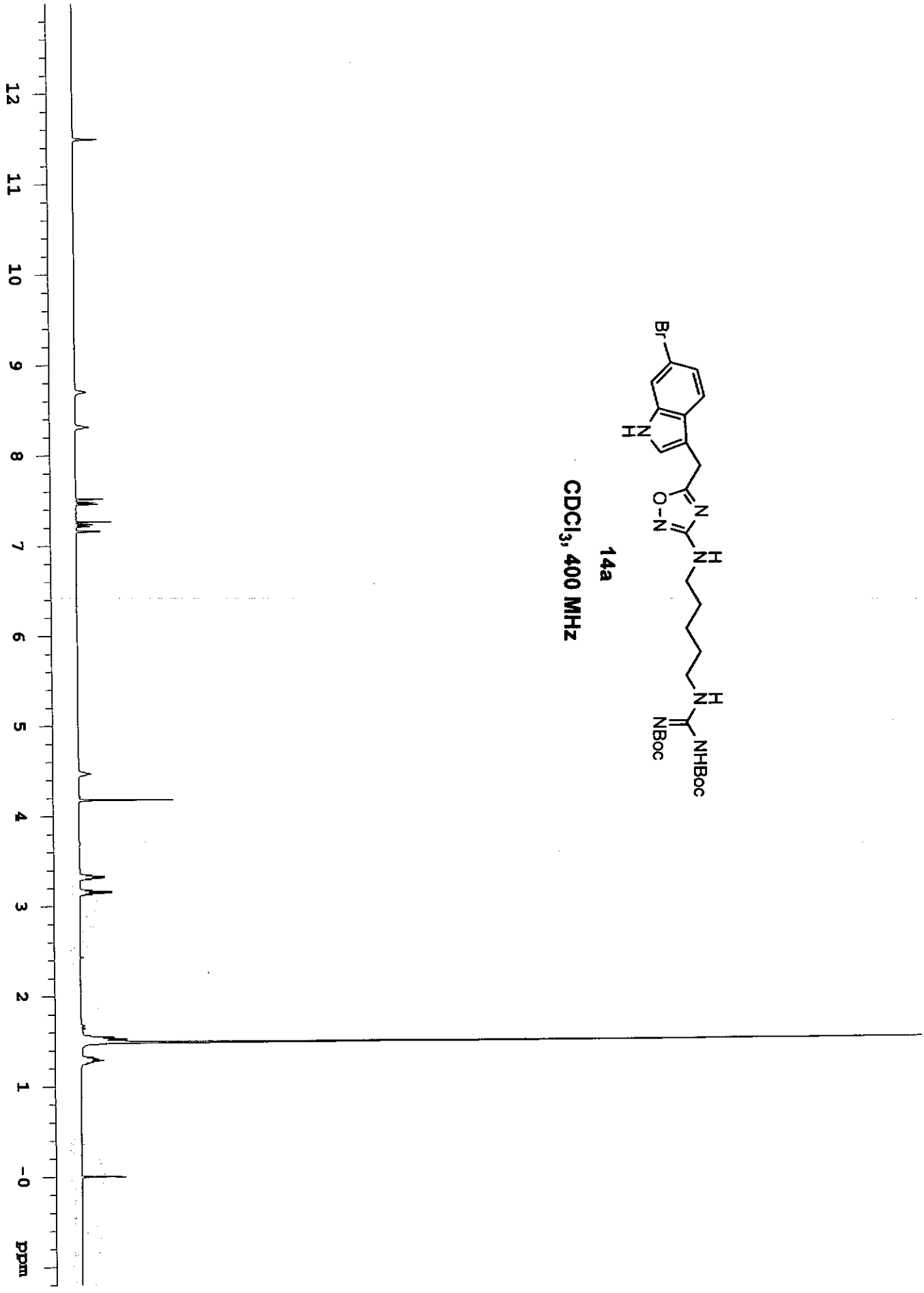

12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
14b
CDCl3, 400 MHz
NHBoc
NBoc

INDEX	FREQUENCY	PPM	HEIGHT
1	17816.205	177.229	23.3
2	16959.398	168.706	17.9
3	16441.346	163.553	18.4
4	15693.643	156.115	23.2
5	15409.058	153.284	17.1
6	13684.762	136.131	16.7
7	12740.977	126.743	15.8
8	12368.651	123.039	42.4
9	12299.222	122.348	46.1
10	12042.103	119.791	39.1
11	11927.659	118.652	47.8
12	11189.874	111.313	53.1
13	10855.696	107.989	18.4
14	8353.178	83.095	33.7
15	7971.697	79.300	29.5
16	7772.564	77.319	200.0
17	7740.519	77.000	199.5
18	7708.475	76.681	192.8
19	4329.313	43.067	41.0
20	4090.506	40.691	42.0
21	2913.254	28.980	43.9
22	2877.395	28.623	47.7
23	2840.773	28.259	136.1
24	2818.647	28.039	157.4
25	2776.684	27.621	19.1 - impurity
26	2411.225	23.986	49.7
27	2354.002	23.417	44.5

14b

$CDCl_3$, 100 MHz





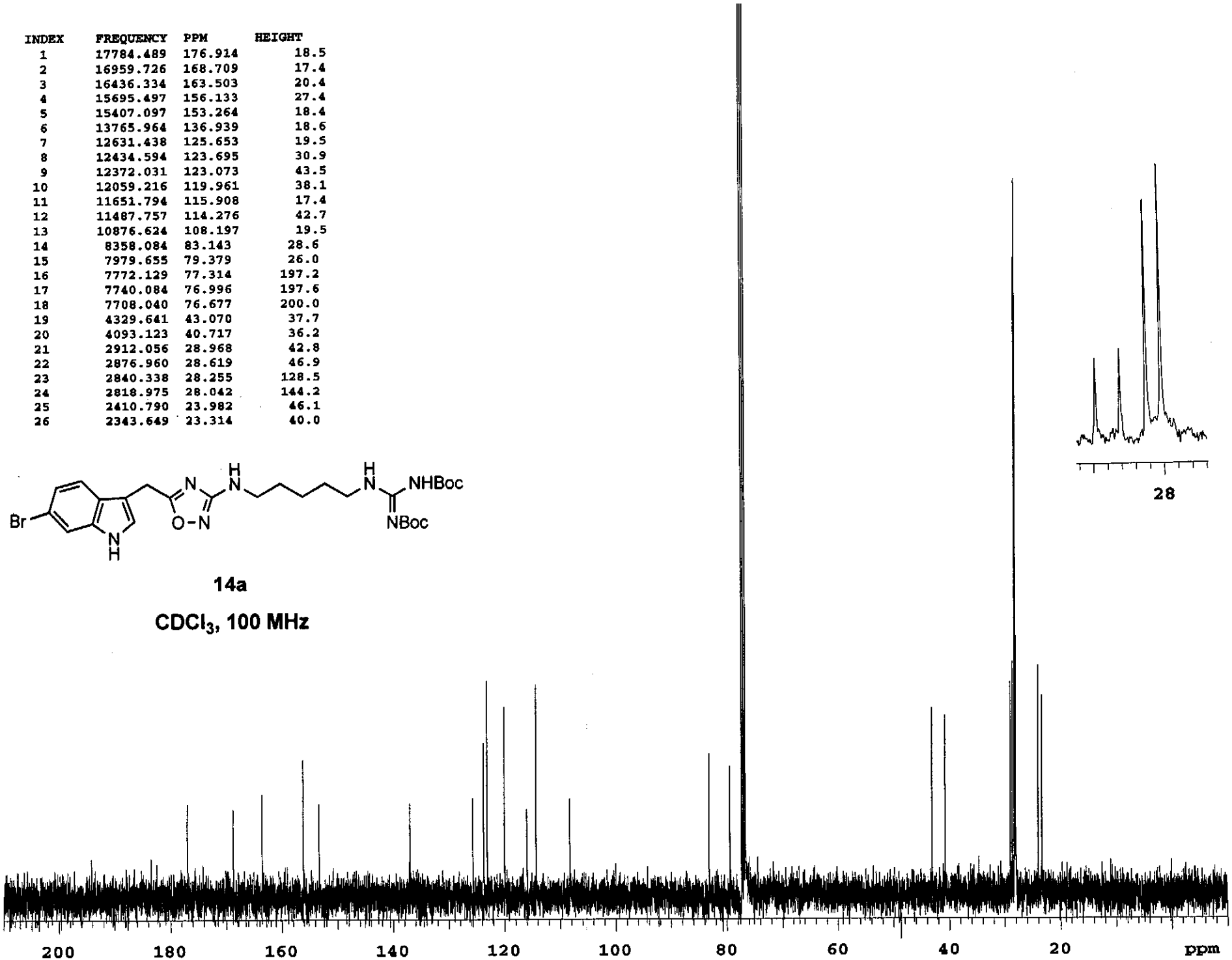
14a

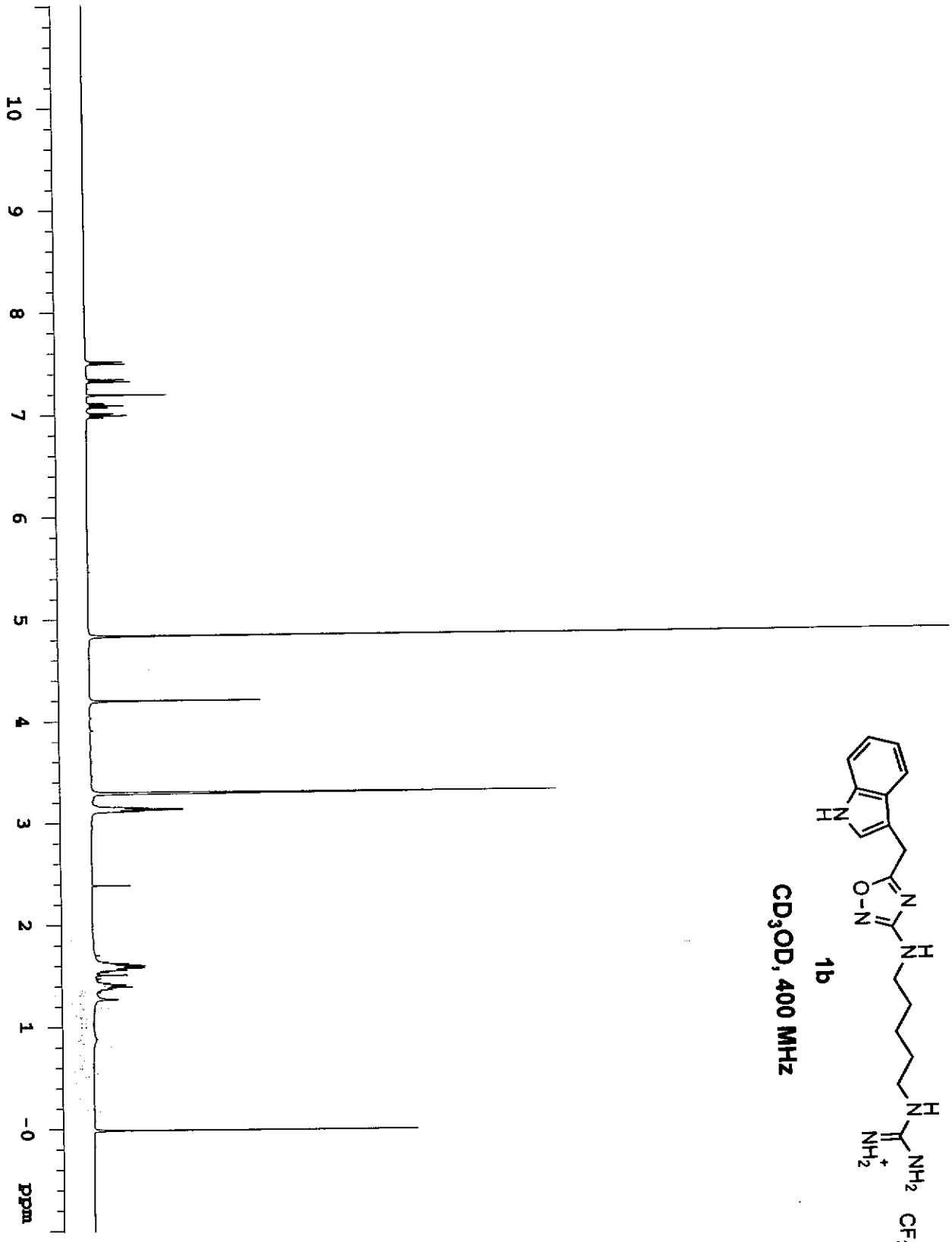
$CDCl_3$, 400 MHz

INDEX	FREQUENCY	PPM	HEIGHT
1	17784.489	176.914	18.5
2	16959.726	168.709	17.4
3	16436.334	163.503	20.4
4	15695.497	156.133	27.4
5	15407.097	153.264	18.4
6	13765.964	136.939	18.6
7	12631.438	125.653	19.5
8	12434.594	123.695	30.9
9	12372.031	123.073	43.5
10	12059.216	119.961	38.1
11	11651.794	115.908	17.4
12	11487.757	114.276	42.7
13	10876.624	108.197	19.5
14	8358.084	83.143	28.6
15	7979.655	79.379	26.0
16	7772.129	77.314	197.2
17	7740.084	76.996	197.6
18	7708.040	76.677	200.0
19	4329.641	43.070	37.7
20	4093.123	40.717	36.2
21	2912.056	28.968	42.8
22	2876.960	28.619	46.9
23	2840.338	28.255	128.5
24	2818.975	28.042	144.2
25	2410.790	23.982	46.1
26	2343.649	23.314	40.0

14a

$CDCl_3$, 100 MHz

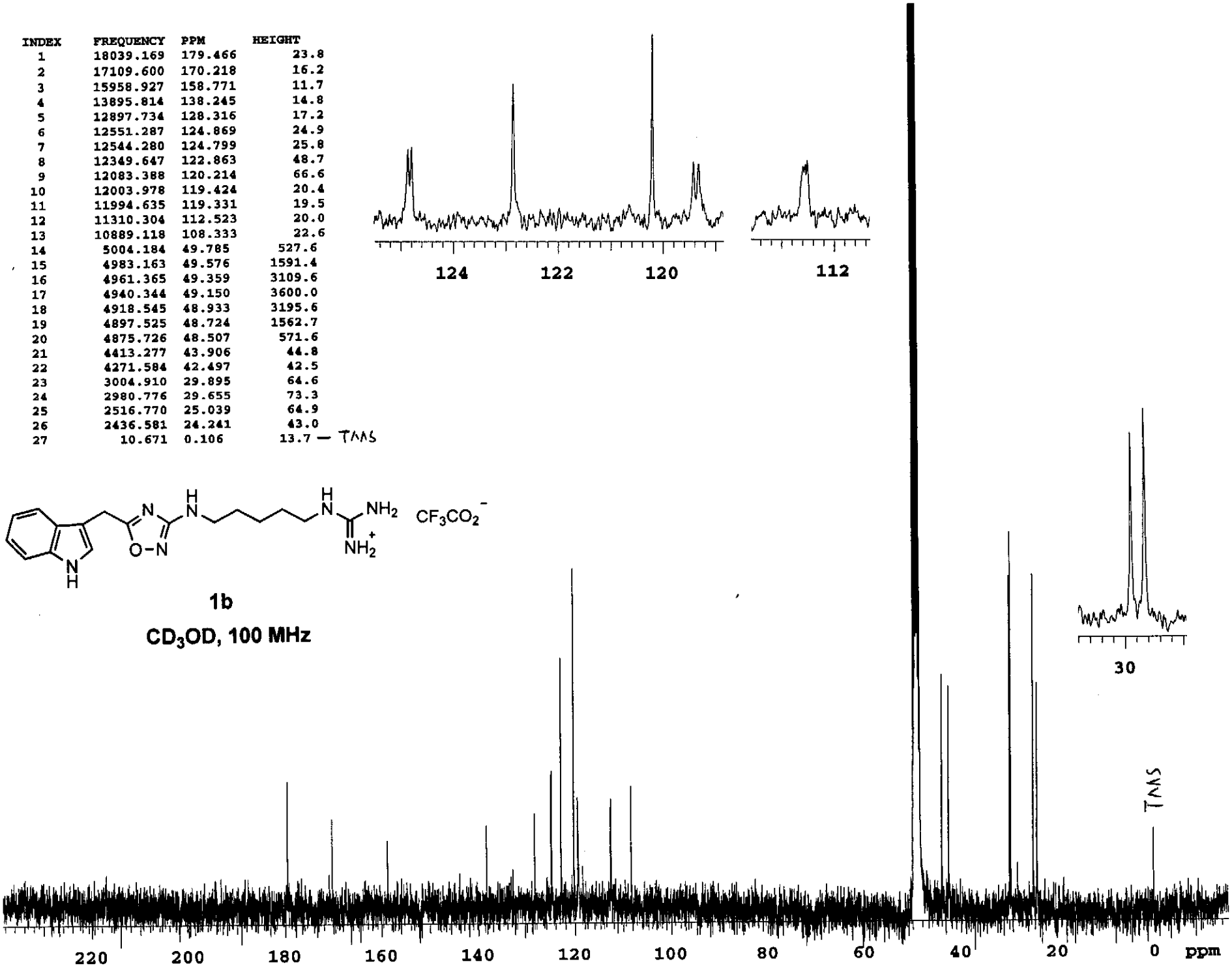


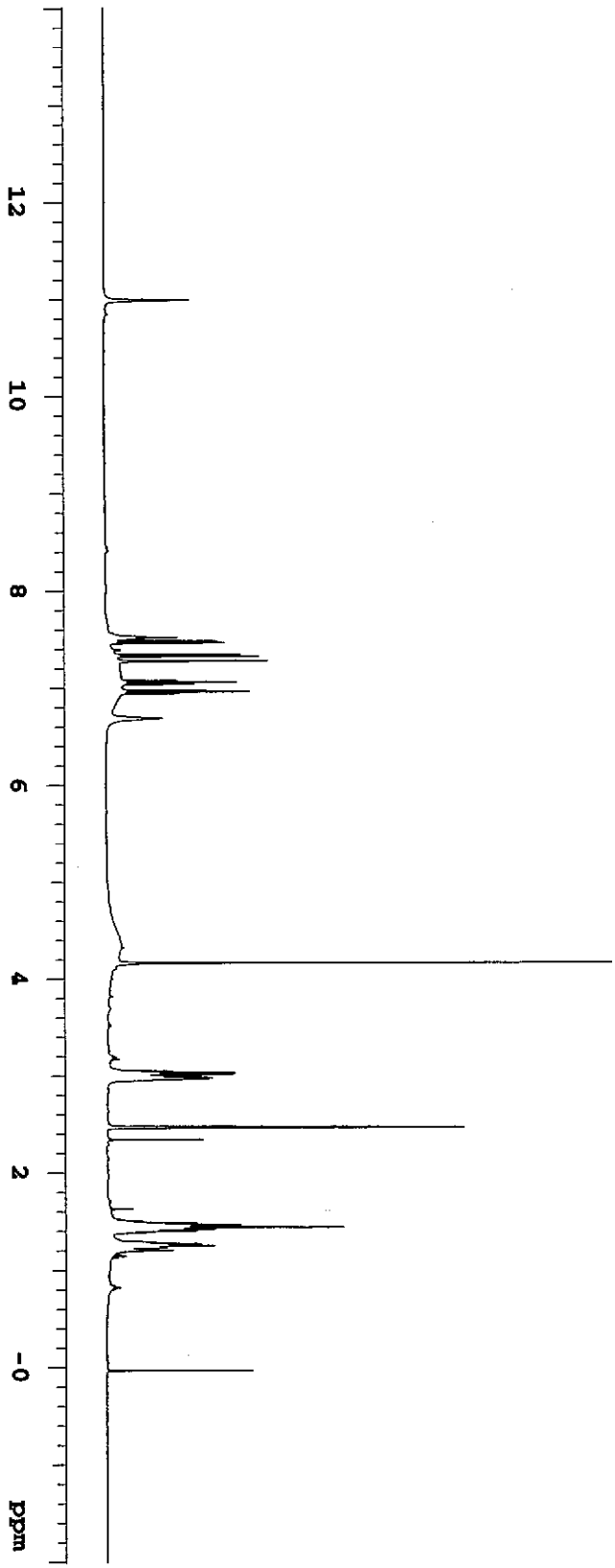

10
9
8
7
6
5
4
3
2
1
-0
ppm
1b
CD3OD, 400 MHz
CF3CO2-

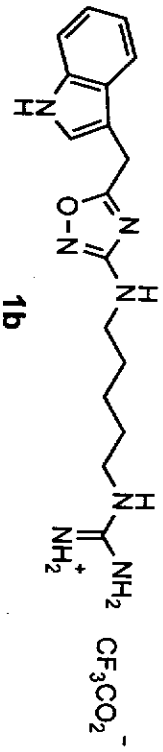
INDEX	FREQUENCY	PPM	HEIGHT
1	18039.169	179.466	23.8
2	17109.600	170.218	16.2
3	15958.927	158.771	11.7
4	13895.814	138.245	14.8
5	12897.734	128.316	17.2
6	12551.287	124.869	24.9
7	12544.280	124.799	25.8
8	12349.647	122.863	48.7
9	12083.388	120.214	66.6
10	12003.978	119.424	20.4
11	11994.635	119.331	19.5
12	11310.304	112.523	20.0
13	10889.118	108.333	22.6
14	5004.184	49.785	527.6
15	4983.163	49.576	1591.4
16	4961.365	49.359	3109.6
17	4940.344	49.150	3600.0
18	4918.545	48.933	3195.6
19	4897.525	48.724	1562.7
20	4875.726	48.507	571.6
21	4413.277	43.906	44.8
22	4271.584	42.497	42.5
23	3004.910	29.895	64.6
24	2980.776	29.655	73.3
25	2516.770	25.039	64.9
26	2436.581	24.241	43.0
27	10.671	0.106	13.7 — TMS

1b

CD_3OD, 100 MHz







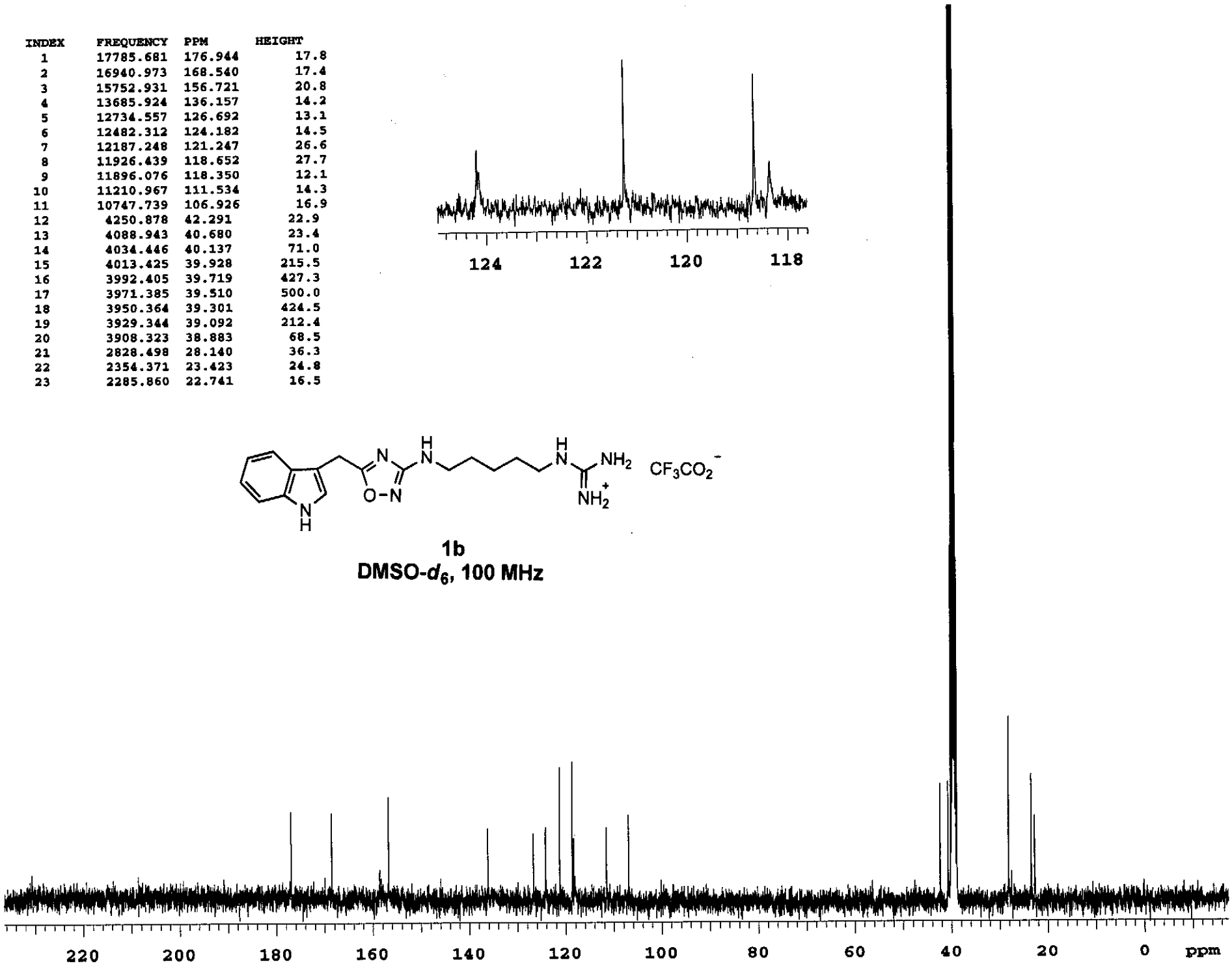
1b

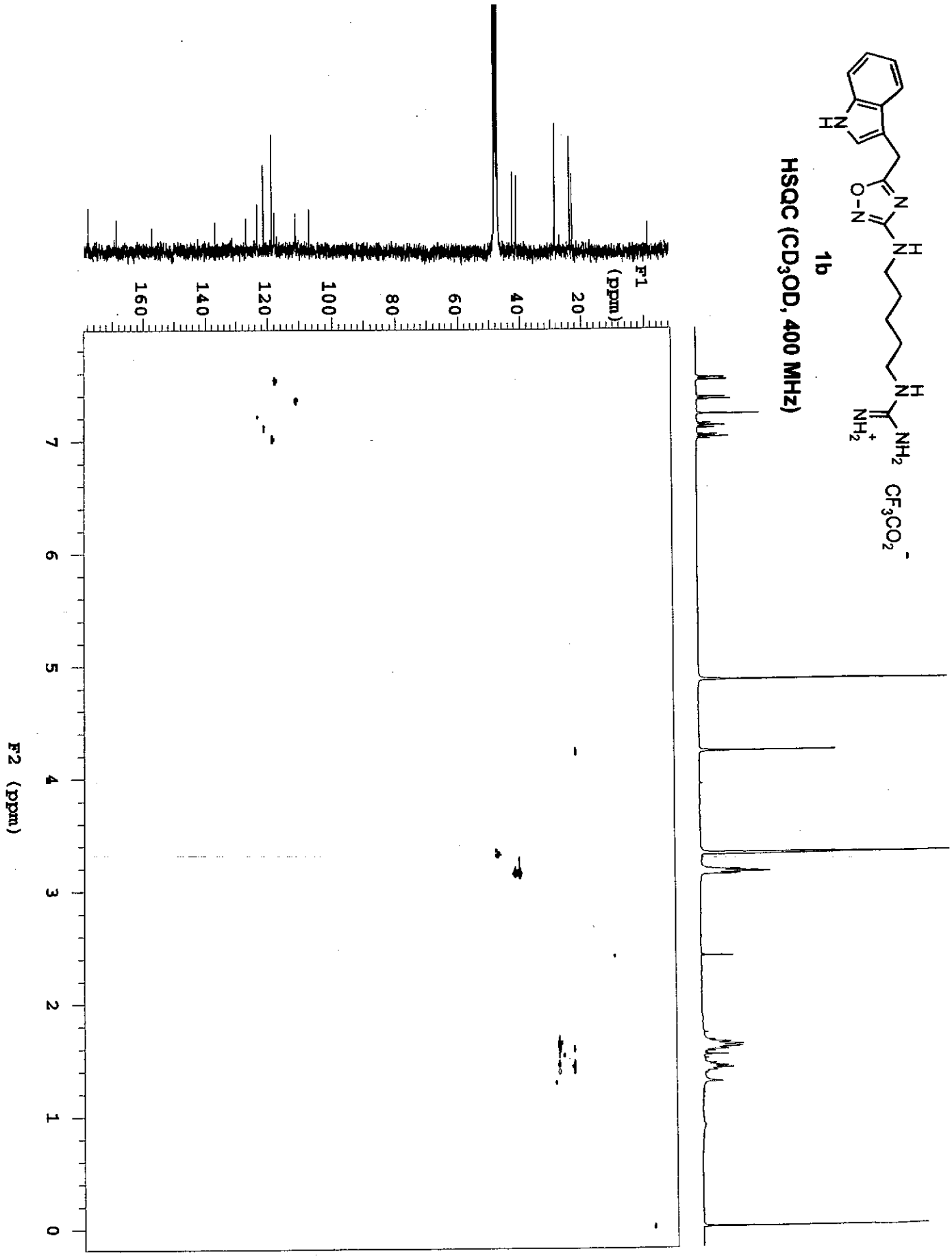
DMSO-d_6, 400 MHz

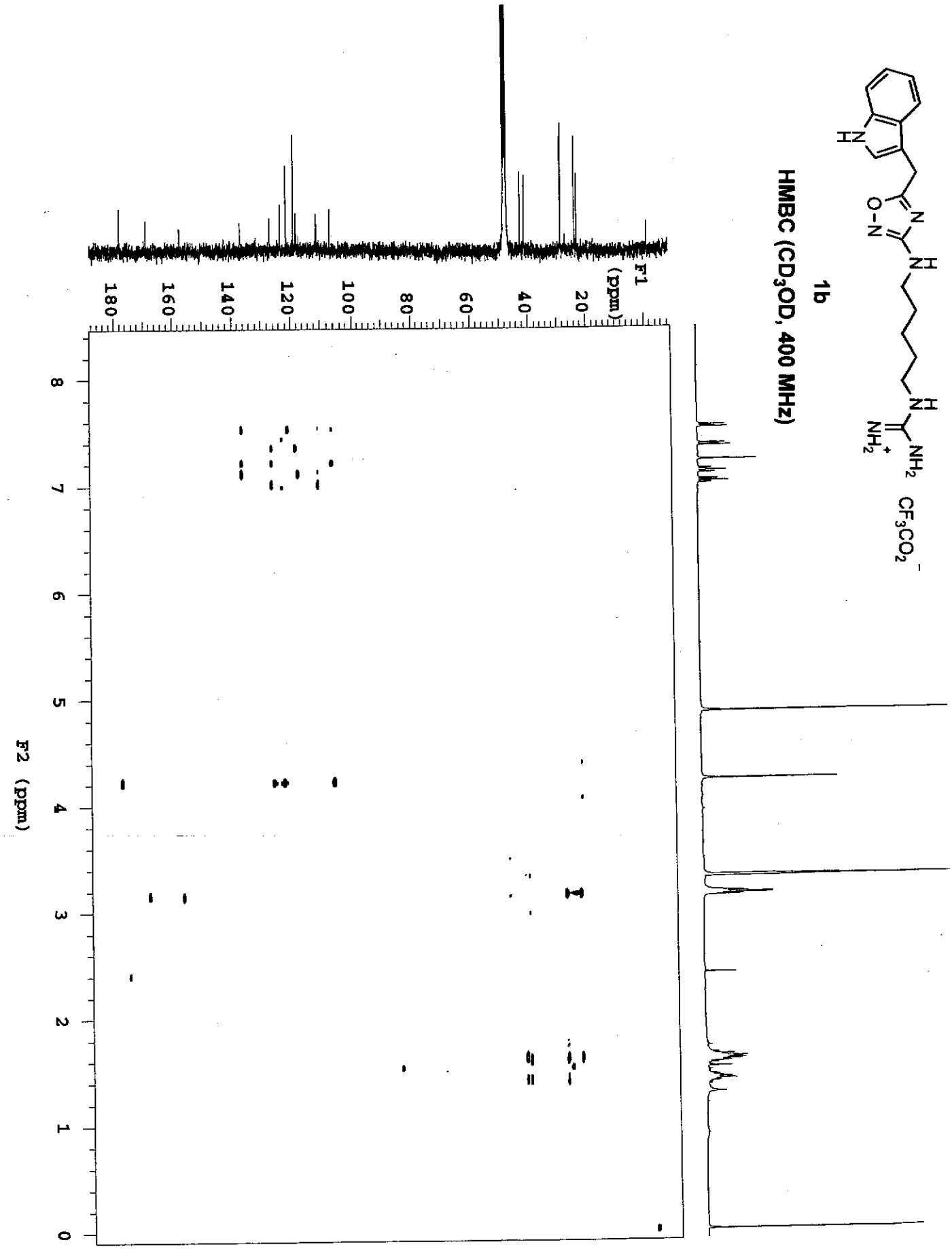
INDEX	FREQUENCY	PPM	HEIGHT
1	17785.681	176.944	17.8
2	16940.973	168.540	17.4
3	15752.931	156.721	20.8
4	13685.924	136.157	14.2
5	12734.557	126.692	13.1
6	12482.312	124.182	14.5
7	12187.248	121.247	26.6
8	11926.439	118.652	27.7
9	11896.076	118.350	12.1
10	11210.967	111.534	14.3
11	10747.739	106.926	16.9
12	4250.878	42.291	22.9
13	4088.943	40.680	23.4
14	4034.446	40.137	71.0
15	4013.425	39.928	215.5
16	3992.405	39.719	427.3
17	3971.385	39.510	500.0
18	3950.364	39.301	424.5
19	3929.344	39.092	212.4
20	3908.323	38.883	68.5
21	2828.498	28.140	36.3
22	2354.371	23.423	24.8
23	2285.860	22.741	16.5

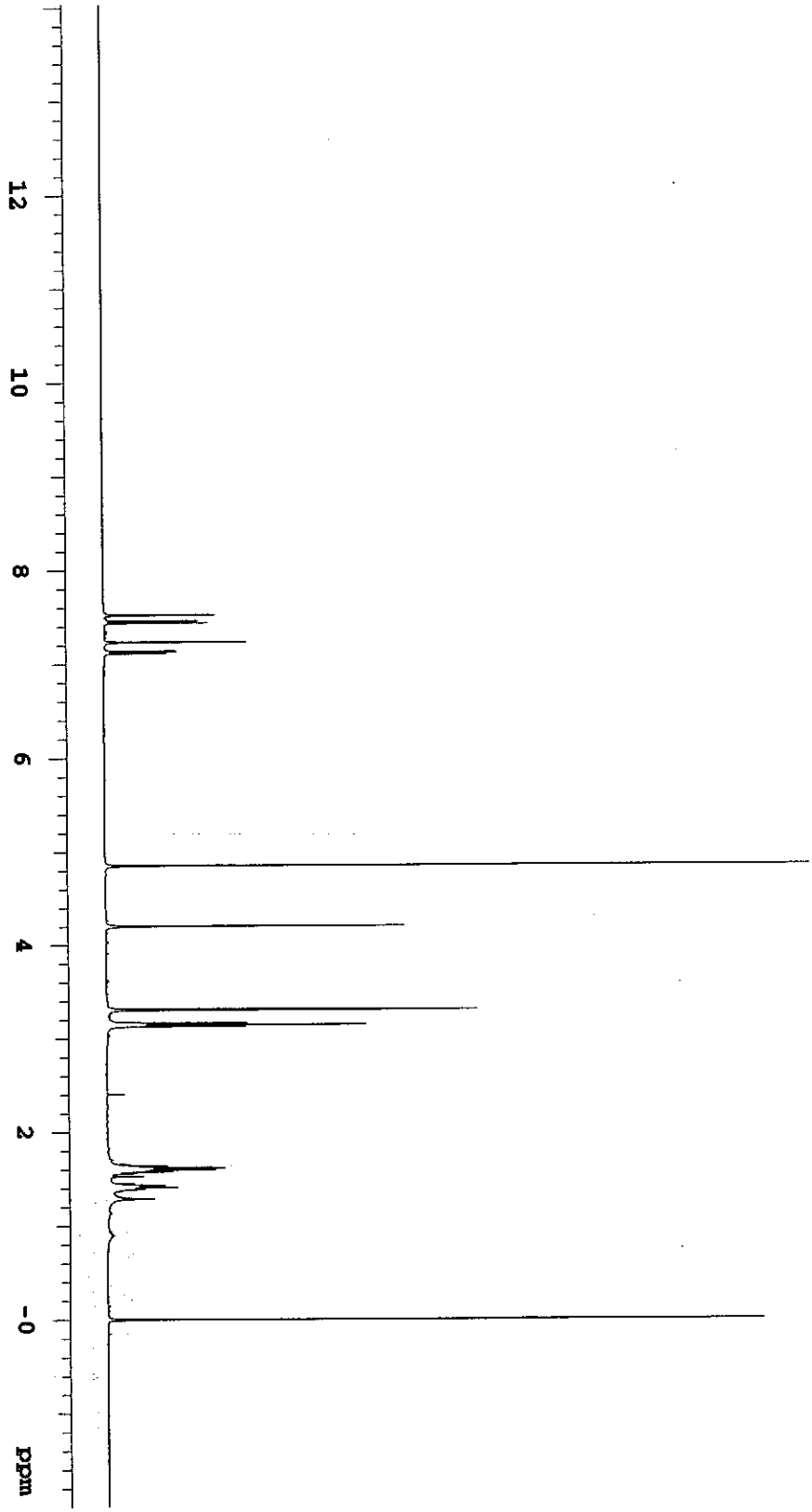
1b

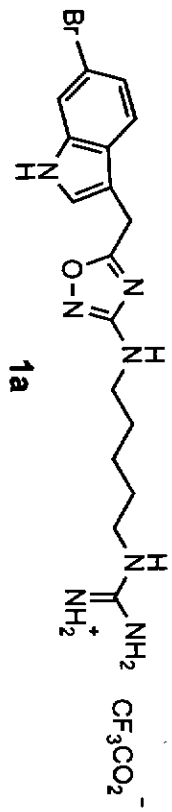
DMSO-d_6, 100 MHz




HSQC (CD3OD, 400 MHz)
1b
CF3CO2-
F1 (ppm)
F2 (ppm)

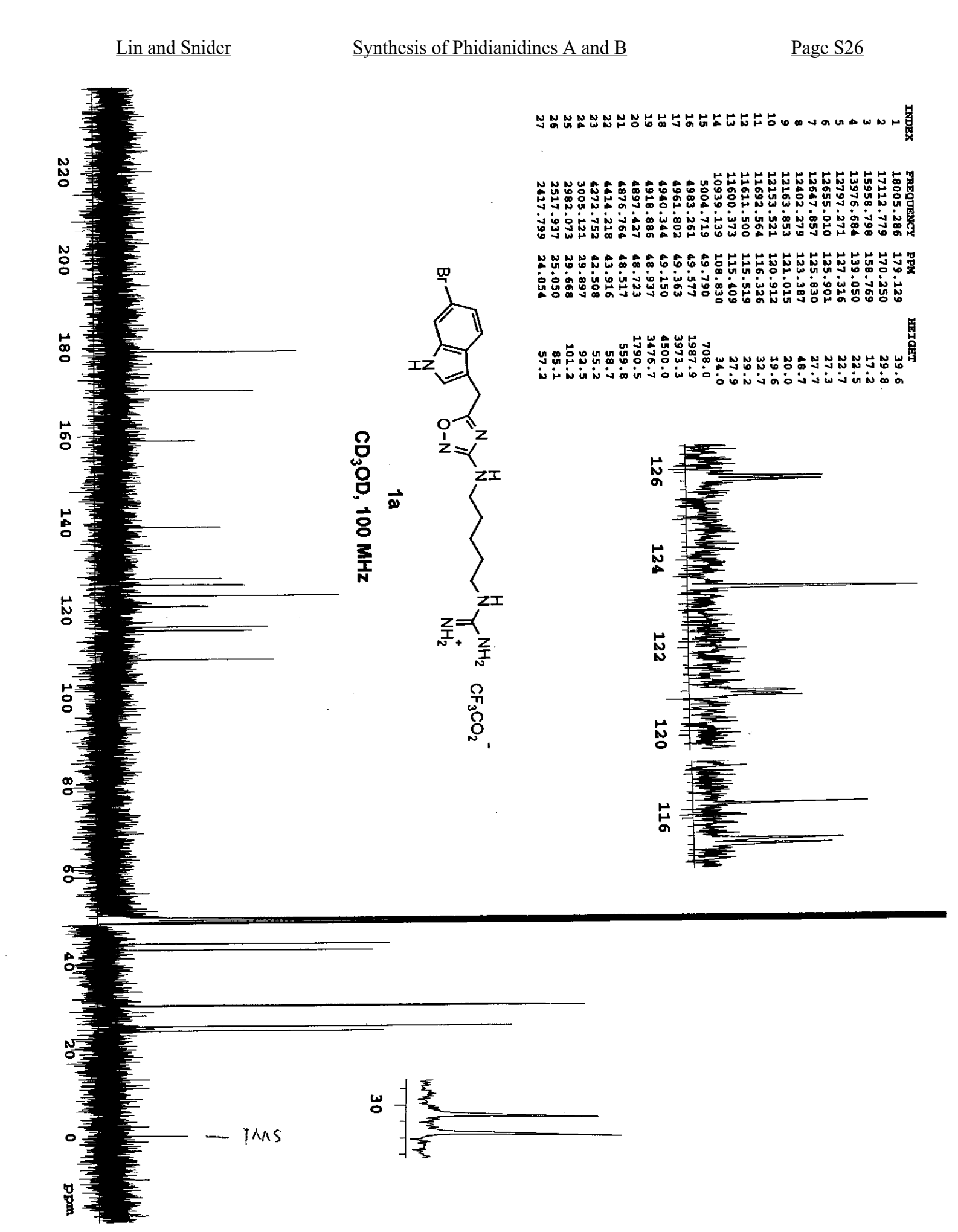

1b
HMBC (CD_3OD, 400 MHz)
F1 (ppm)
F2 (ppm)
$CF_3CO_2^-$

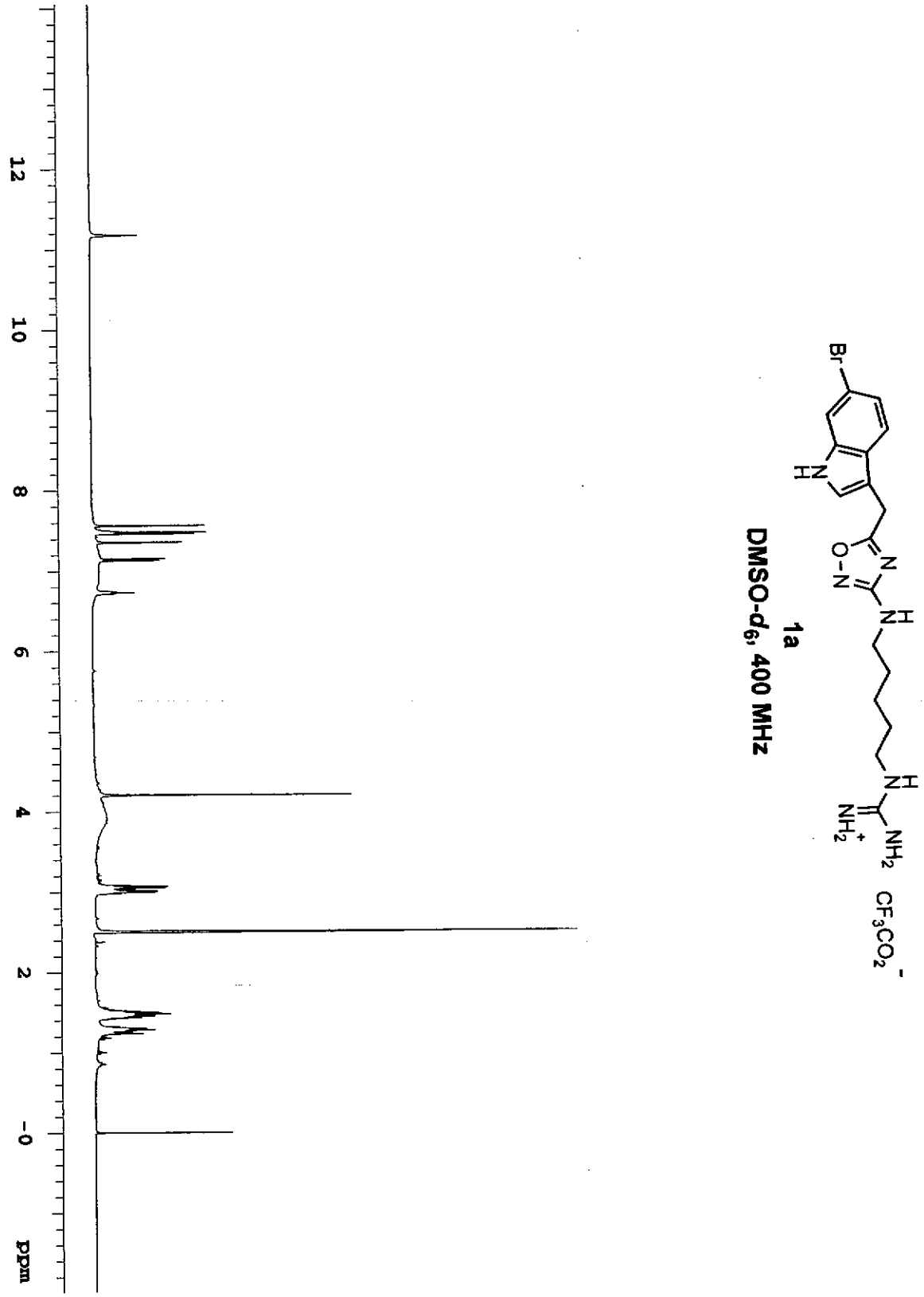




1a

CD_3OD, 400 MHz


INDEX FREQUENCY PPM HEIGHT
1 18005.286 179.129 39.6
2 17112.779 170.250 29.8
3 15958.798 158.769 17.2
4 13976.684 139.050 22.5
5 12797.271 127.316 22.7
6 12655.010 125.901 27.3
7 12647.857 125.830 27.7
8 12402.279 123.387 48.7
9 12163.853 121.015 20.0
10 12153.521 120.912 19.6
11 11692.564 116.326 32.7
12 11611.500 115.519 29.2
13 11600.373 115.409 27.9
14 10939.139 108.830 34.0
15 5004.719 49.790 708.0
16 4983.261 49.577 1987.9
17 4961.802 49.363 3973.3
18 4940.344 49.150 4500.0
19 4918.886 48.937 3476.7
20 4897.427 48.723 1790.5
21 4876.764 48.517 559.8
22 4414.218 43.916 58.7
23 4272.752 42.508 55.2
24 3005.121 29.897 92.5
25 2982.073 29.668 101.2
26 2517.937 25.050 85.1
27 2417.799 24.054 57.2
Br
N
H
O-N
N
H
N
H
NH2
NH2+
CF3CO2-
1a
CD3OD, 100 MHz
220
200
180
160
140
120
100
80
60
40
20
0
ppm
126
124
122
120
116
30
SW1



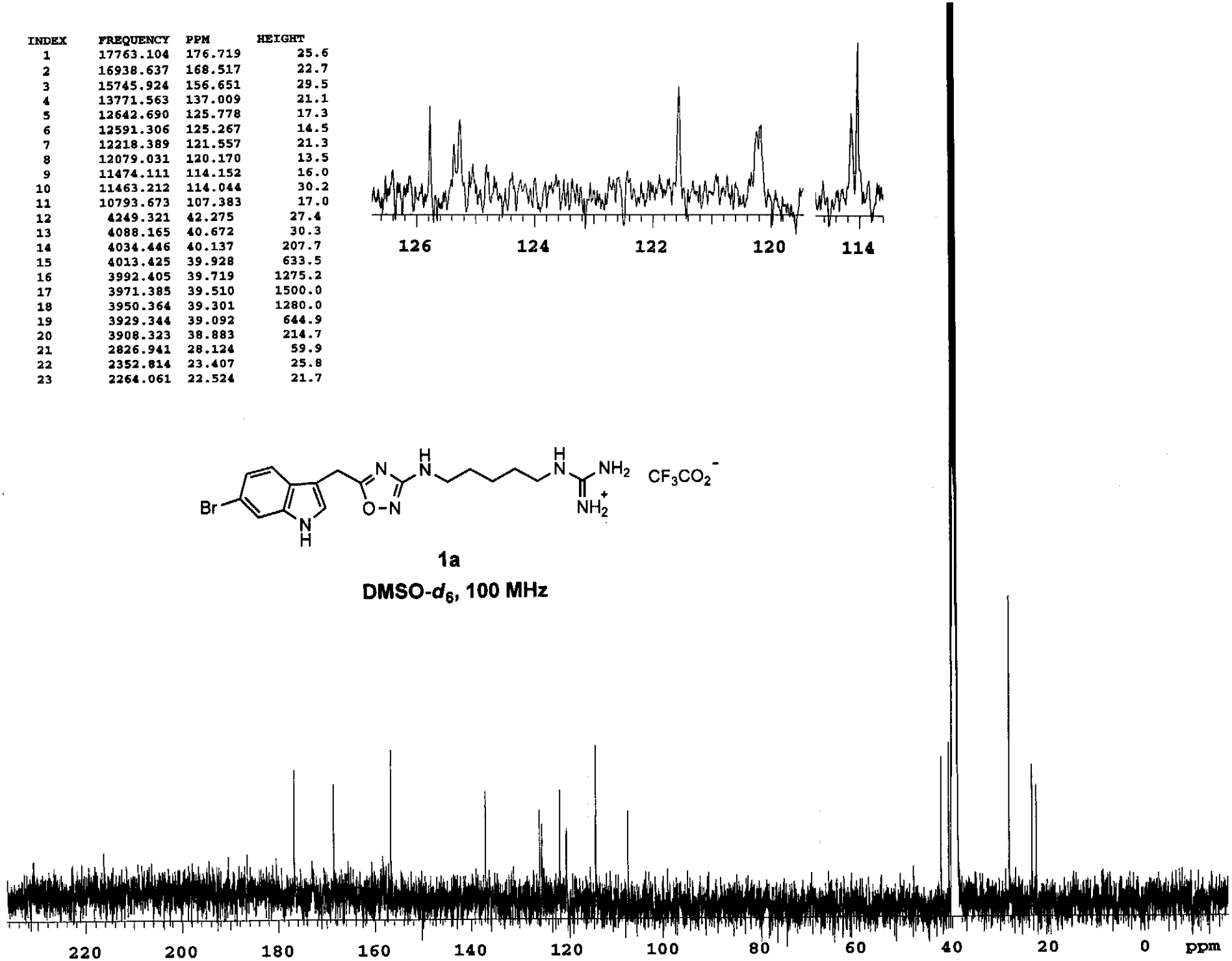
1a

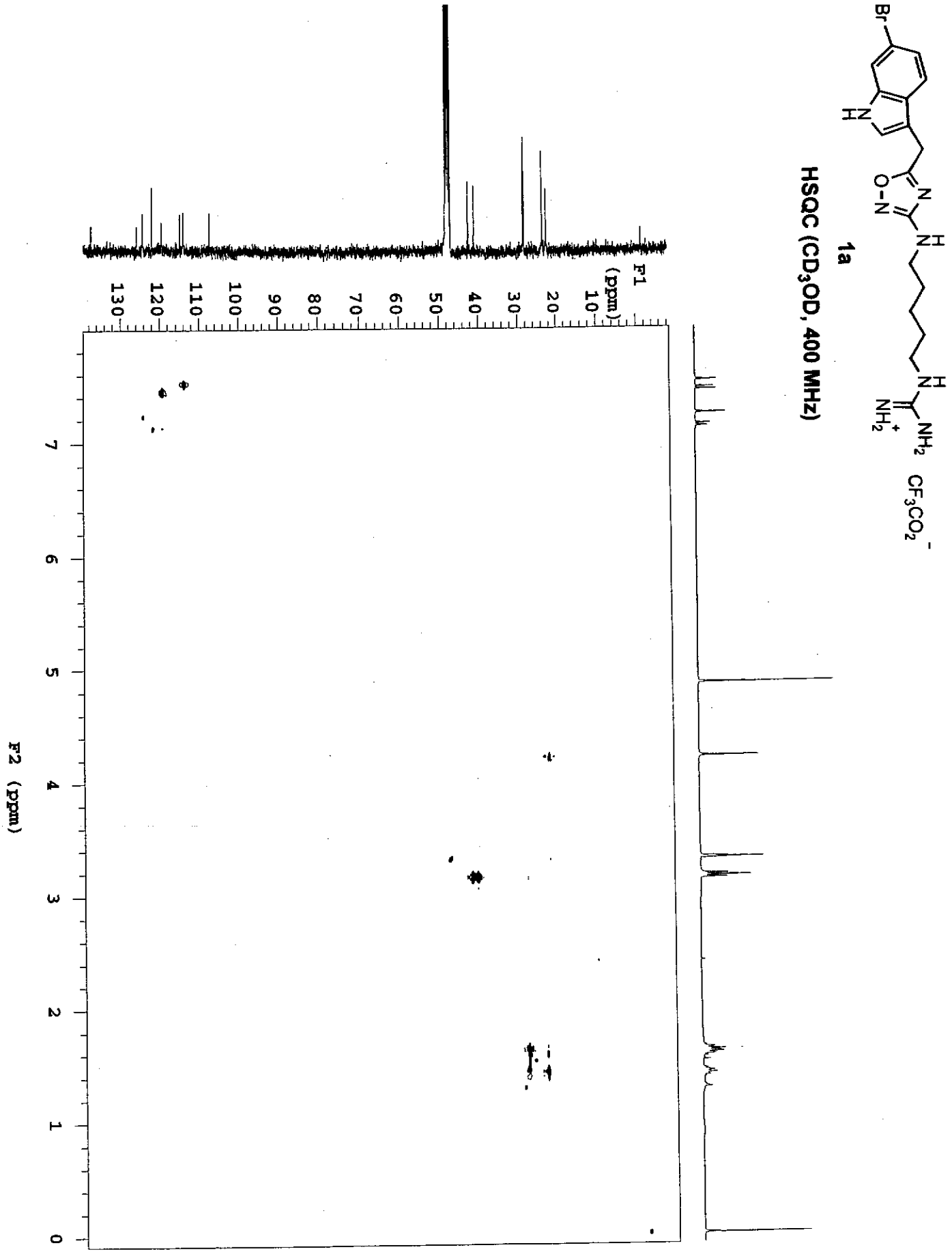
DMSO-d_6, 400 MHz

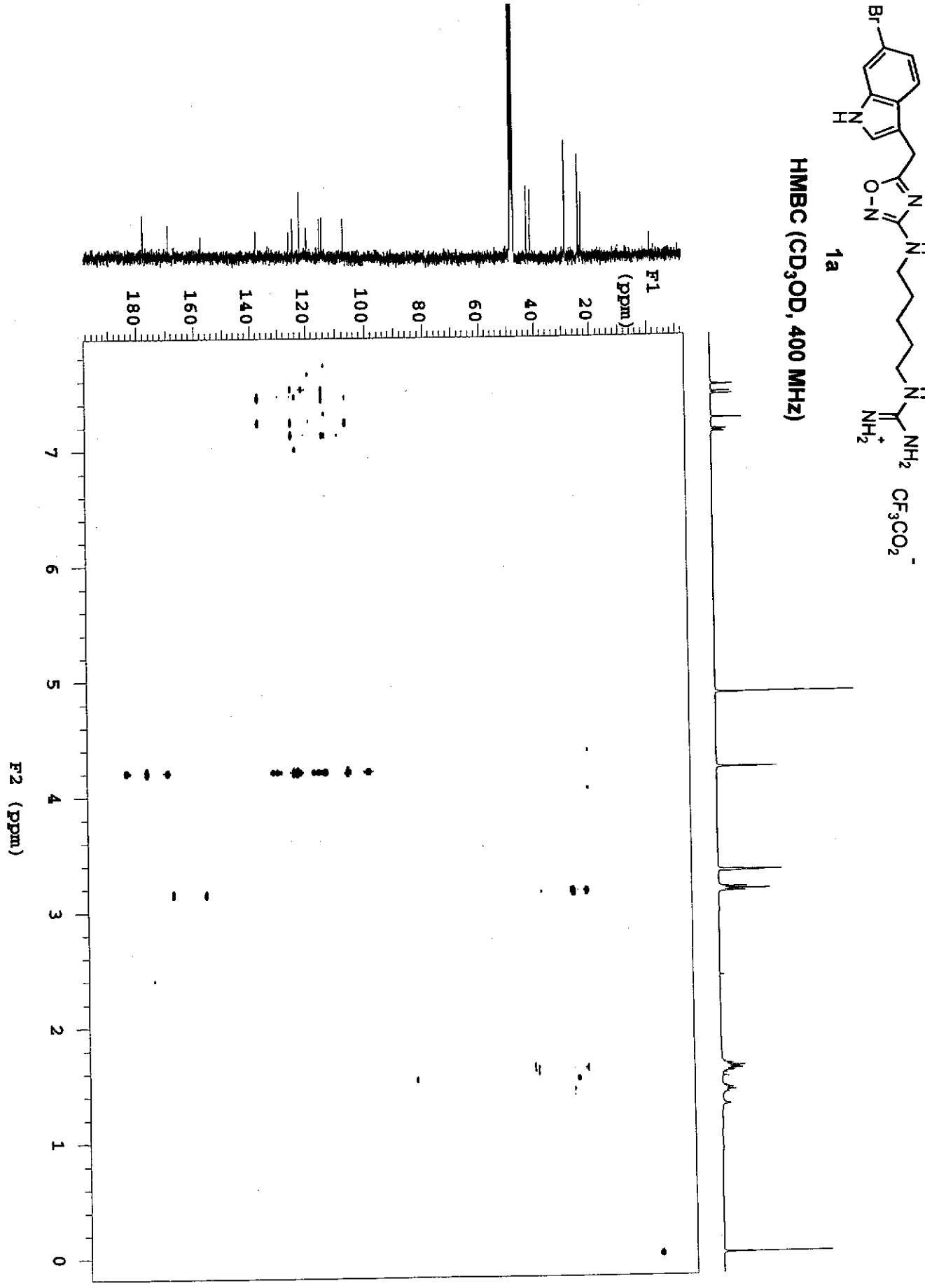
INDEX	FREQUENCY	PPM	HEIGHT
1	17763.104	176.719	25.6
2	16938.637	168.517	22.7
3	15745.924	156.651	29.5
4	13771.563	137.009	21.1
5	12642.690	125.778	17.3
6	12591.306	125.267	14.5
7	12218.389	121.557	21.3
8	12079.031	120.170	13.5
9	11474.111	114.152	16.0
10	11463.212	114.044	30.2
11	10793.673	107.383	17.0
12	4249.321	42.275	27.4
13	4088.165	40.672	30.3
14	4034.446	40.137	207.7
15	4013.425	39.928	633.5
16	3992.405	39.719	1275.2
17	3971.385	39.510	1500.0
18	3950.364	39.301	1280.0
19	3929.344	39.092	644.9
20	3908.323	38.883	214.7
21	2826.941	28.124	59.9
22	2352.814	23.407	25.8
23	2264.061	22.524	21.7

1a

DMSO-d_6, 100 MHz




Br
N
H
O-N
N
H
N
H
NH2
NH2+
CF3CO2-
1a
HSQC (CD3OD, 400 MHz)
F1 (ppm)
10
20
30
40
50
60
70
80
90
100
110
120
130
F2 (ppm)
0
1
2
3
4
5
6
7


HMBC (CD3OD, 400 MHz)
1a
F1 (ppm)
F2 (ppm)