Solid phase functionalization of peptides by an α-hydrazinoacetyl group

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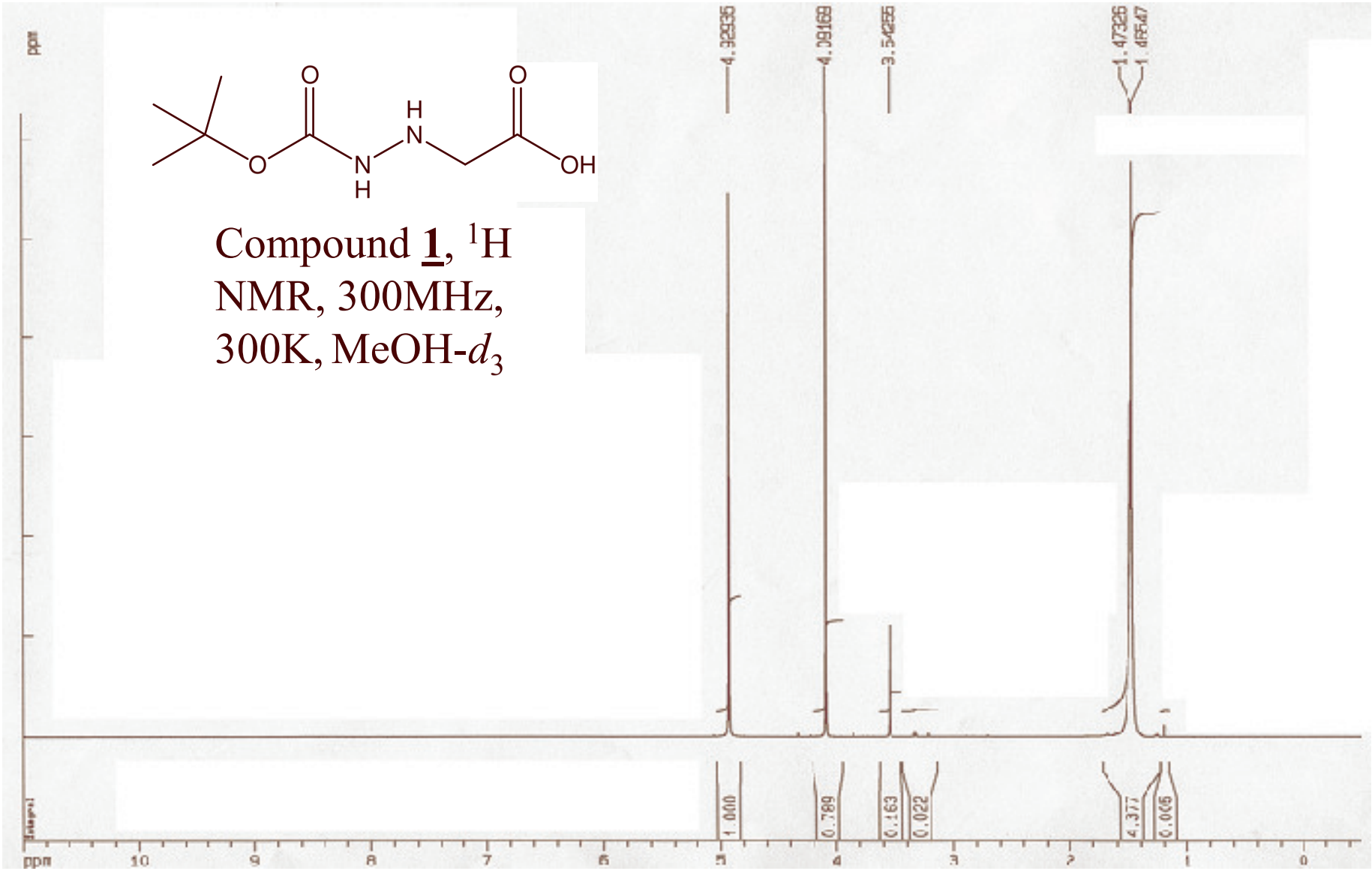
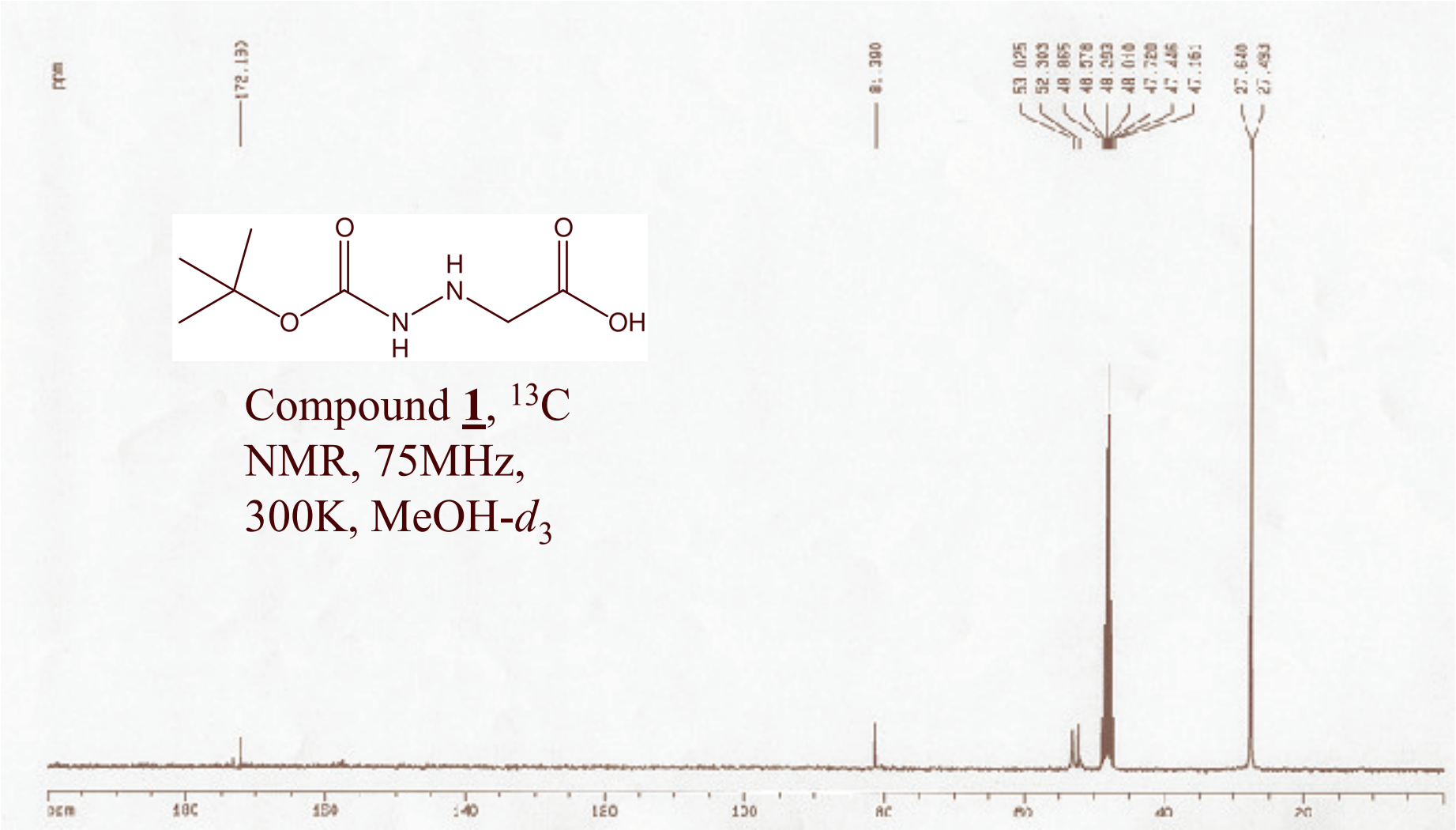
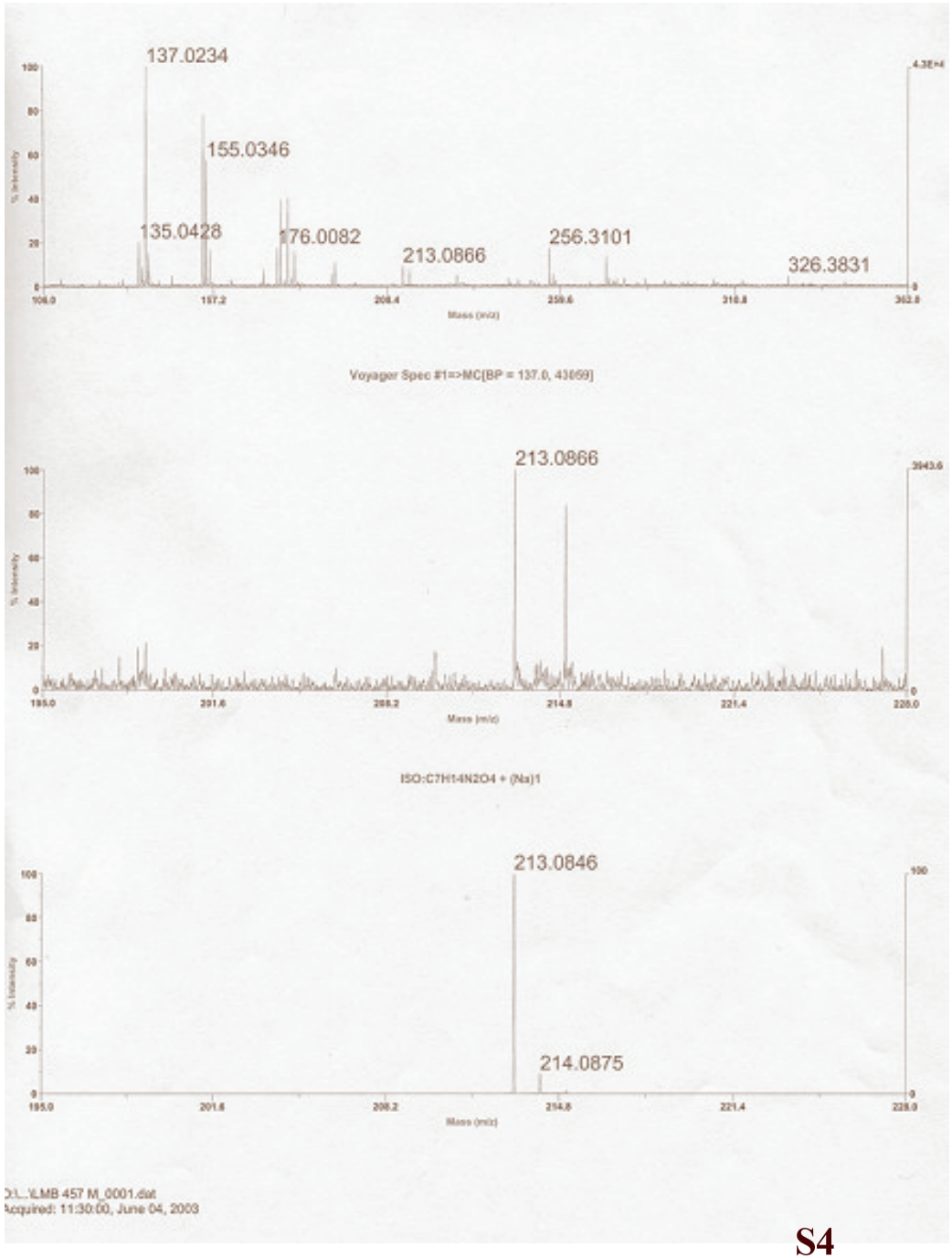
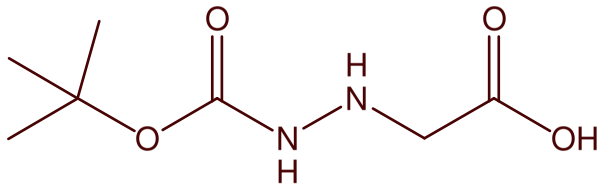

O
O
H
N
O
N
H
OH
Compound 1, 1H NMR, 300MHz, 300K, MeOH-d3
4.92935
4.89169
3.54255
1.47326
1.46547
1.000
0.789
0.163
0.022
4.377
0.005
ppm
10
9
8
7
6
5
4
3
2
1
0

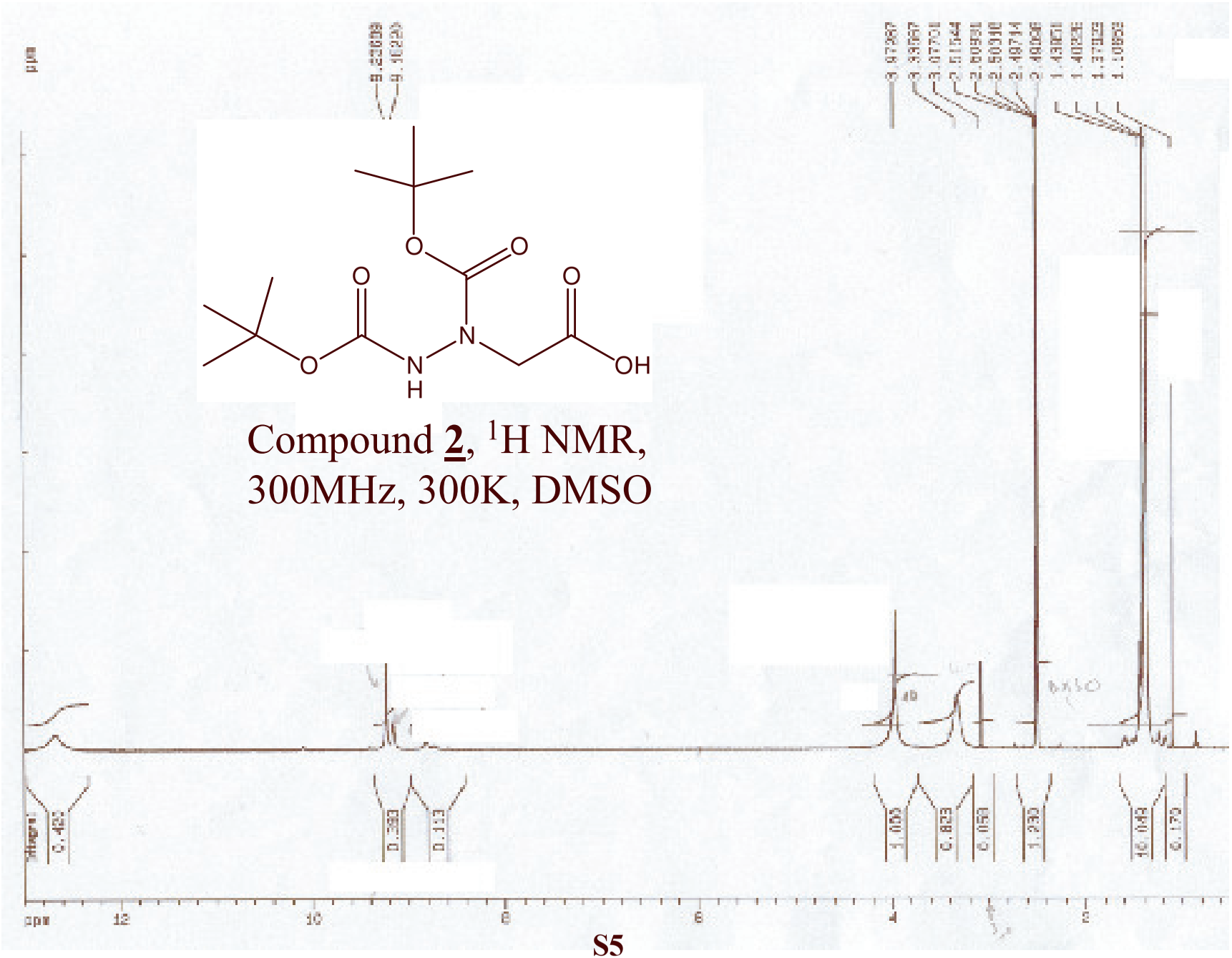


Compound **1**, ^{13}C NMR, 75MHz, 300K, MeOH-d_3

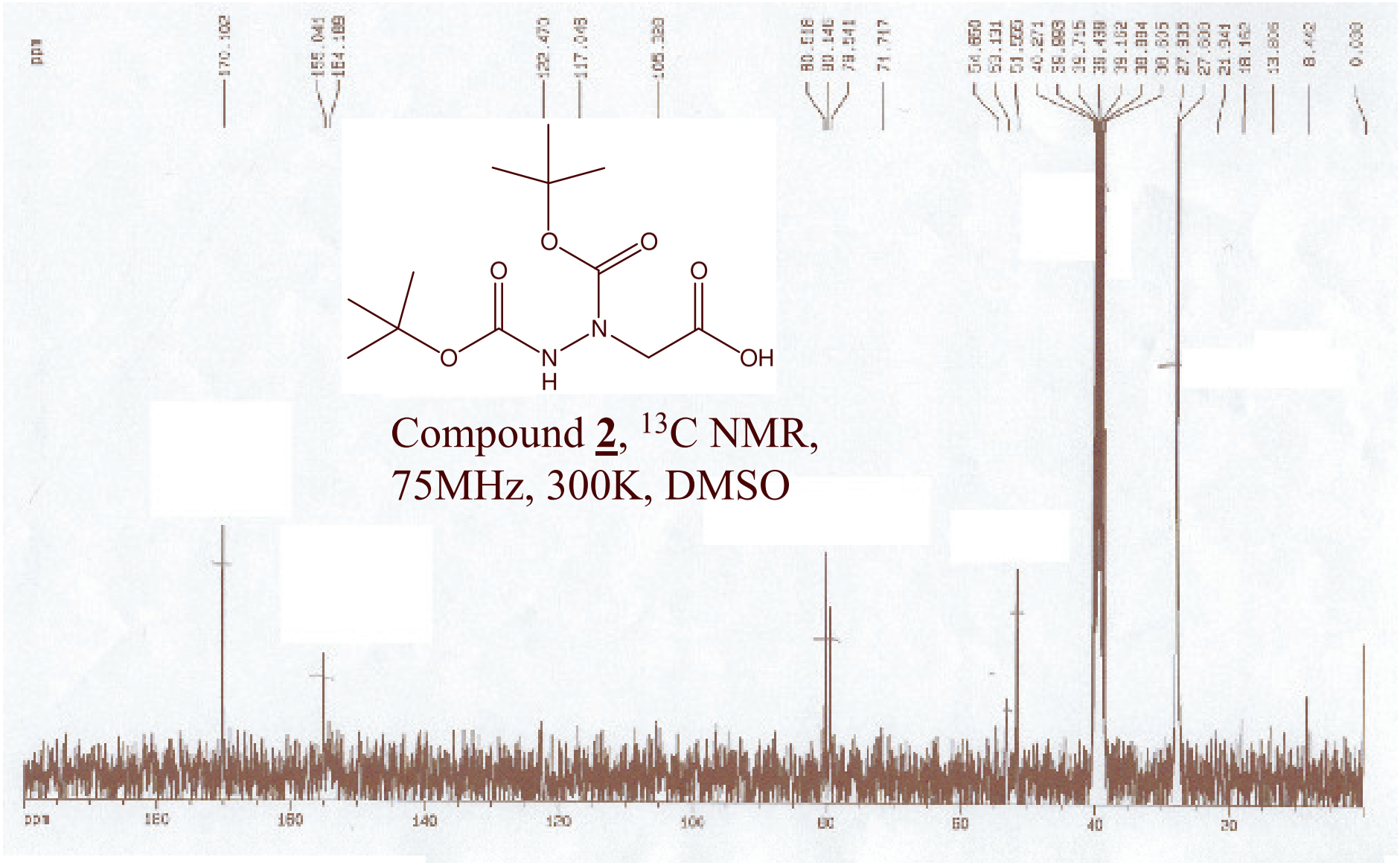




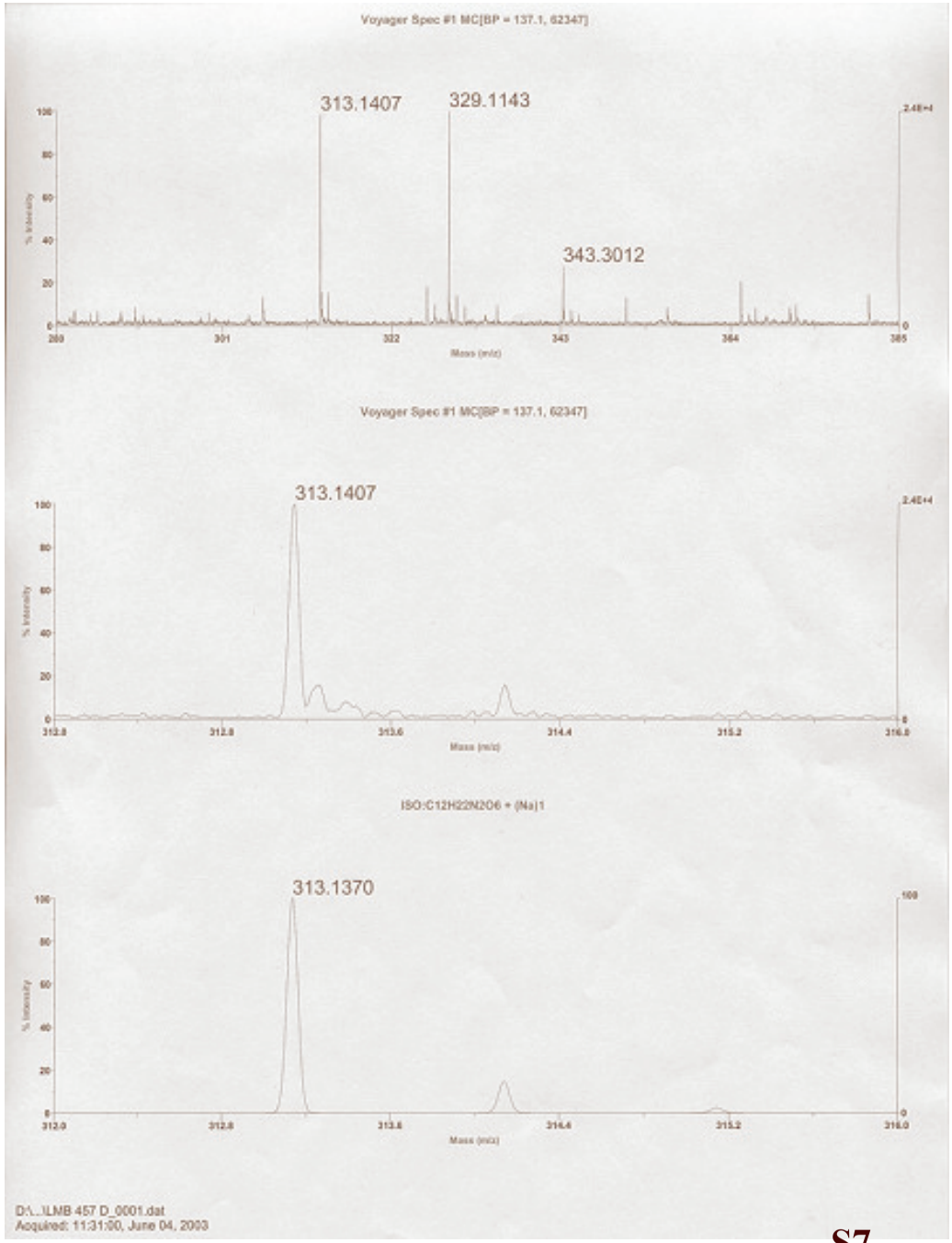
Compound **1**, Maldi-TOF, matrix: 2,5-dihydroxybenzoic acid

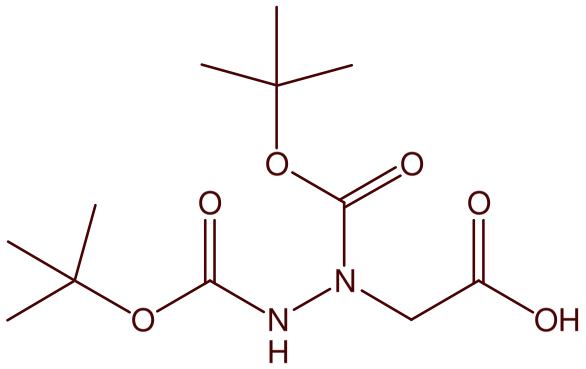


Compound **2**, ^{1}H NMR,
300MHz, 300K, DMSO

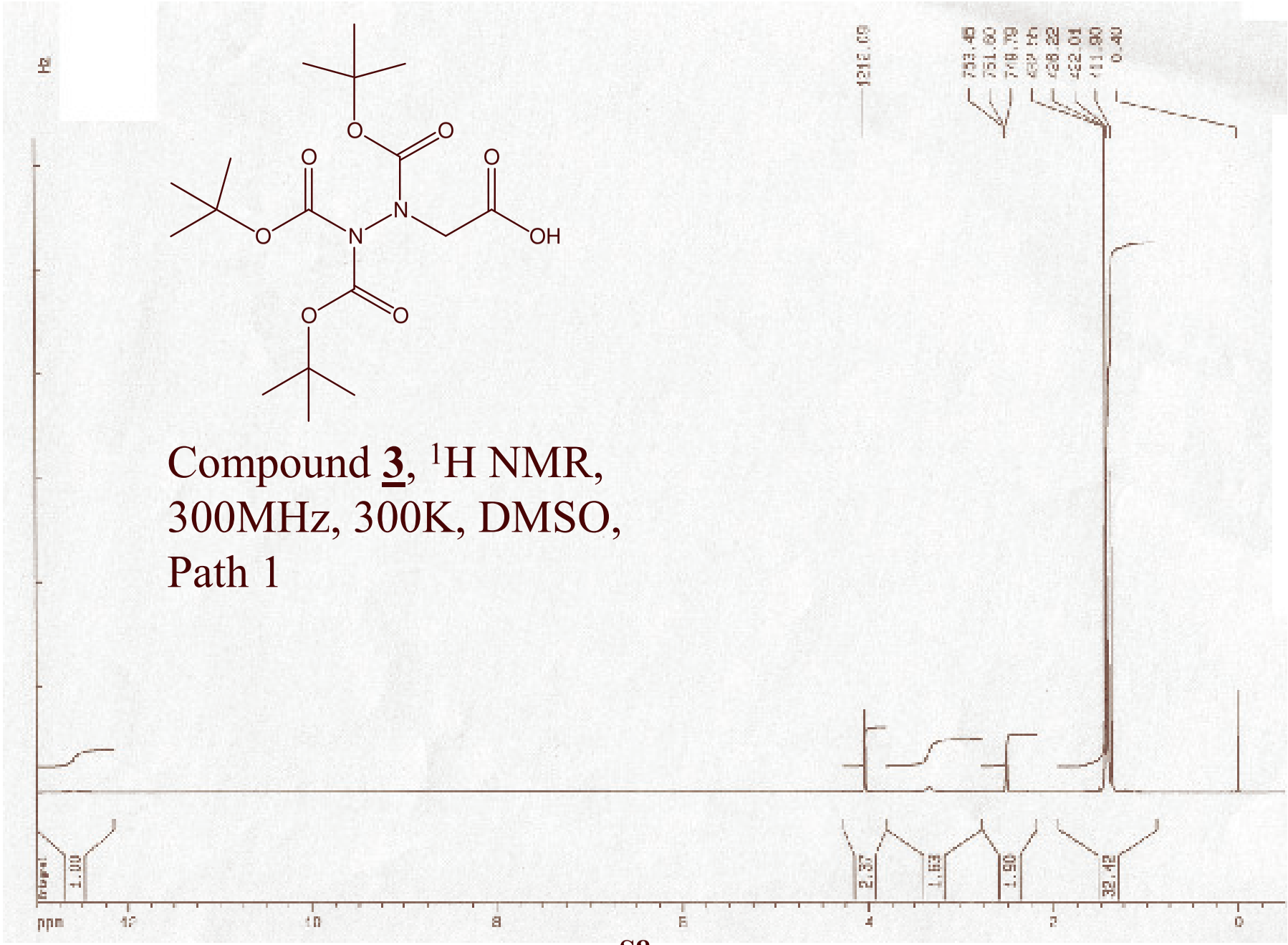


Compound **2**, ^{13}C NMR,
75MHz, 300K, DMSO

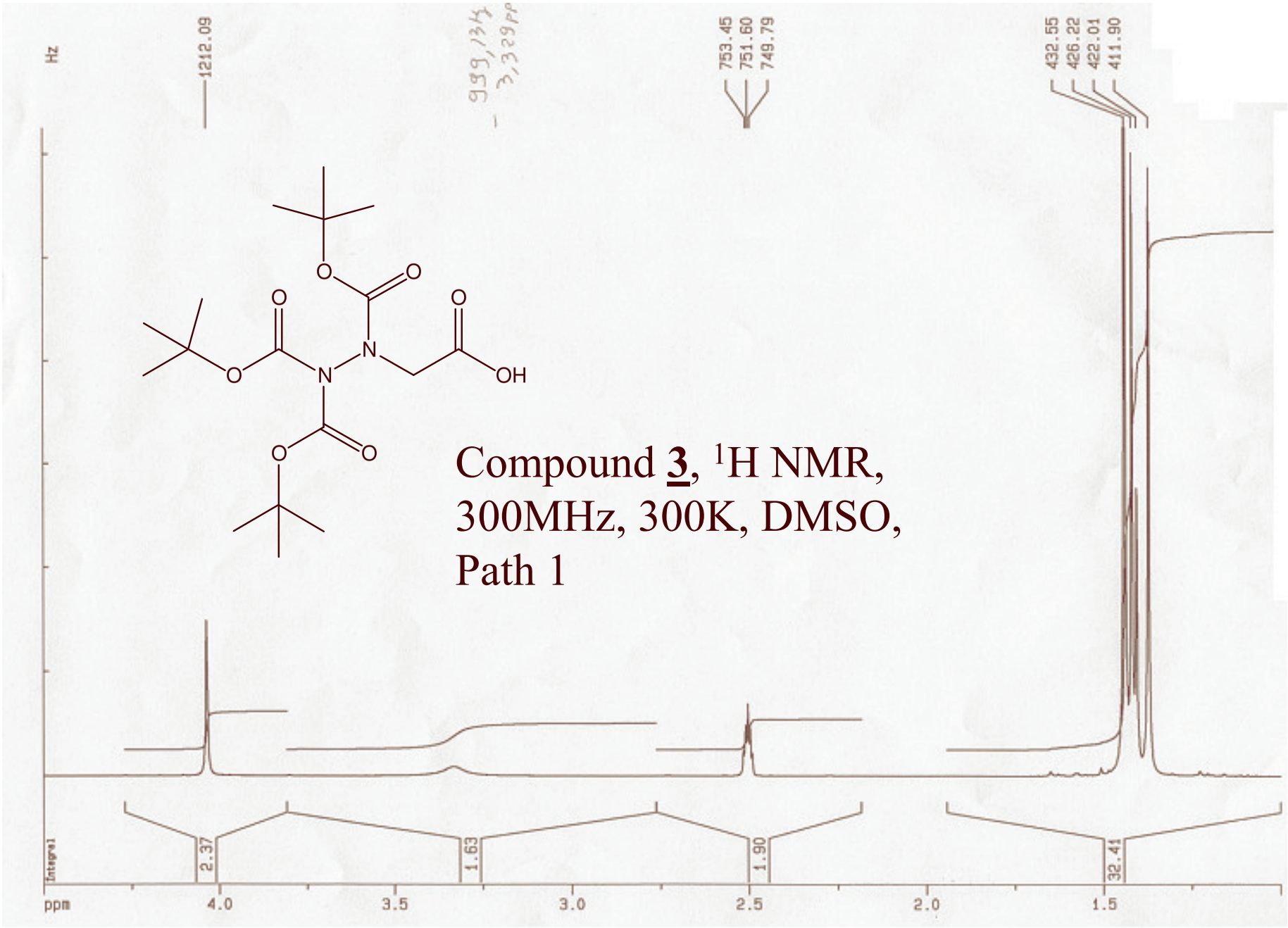


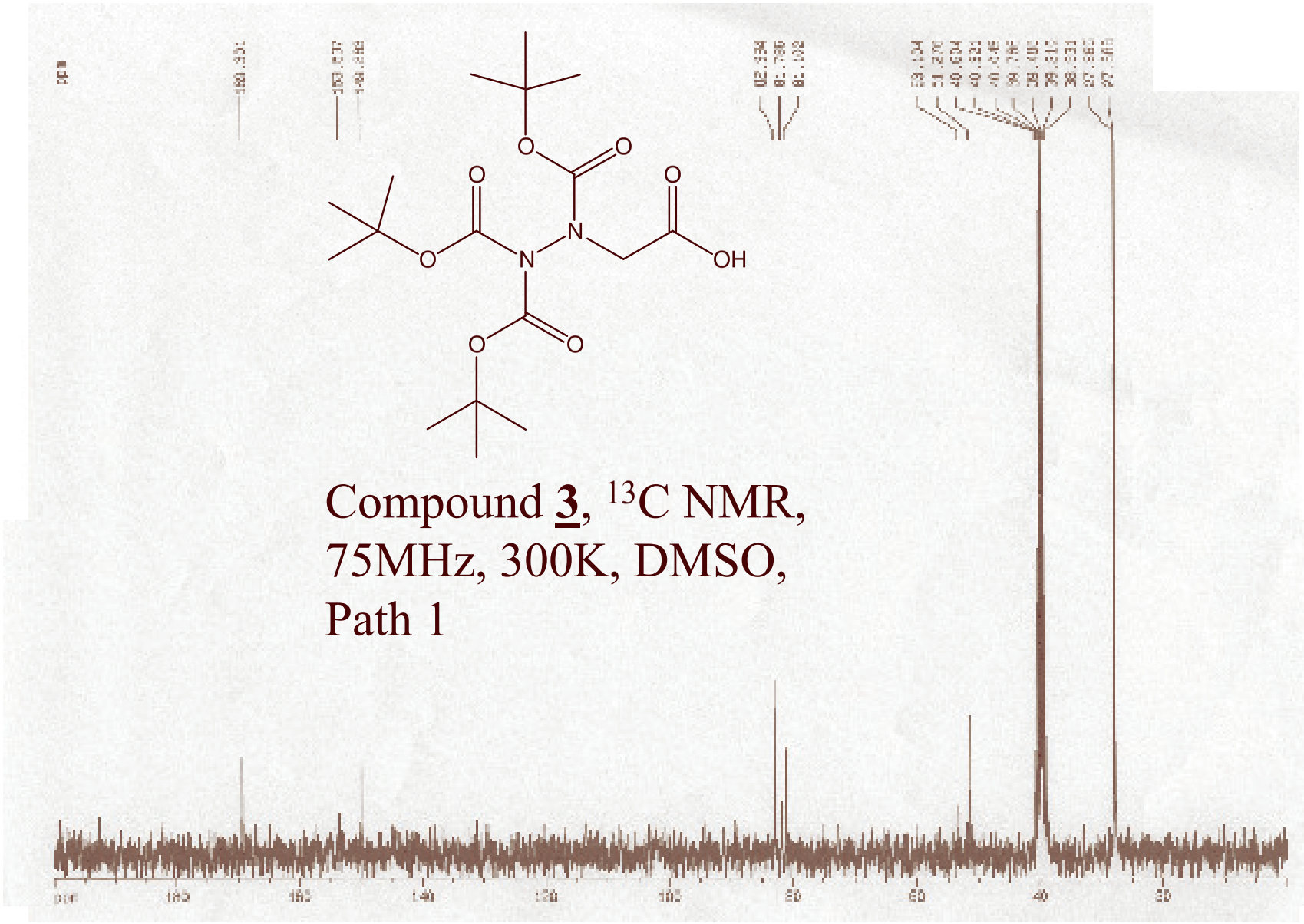


Compound **2**, Maldi-TOF, matrix: 2,5-dihydroxybenzoic acid

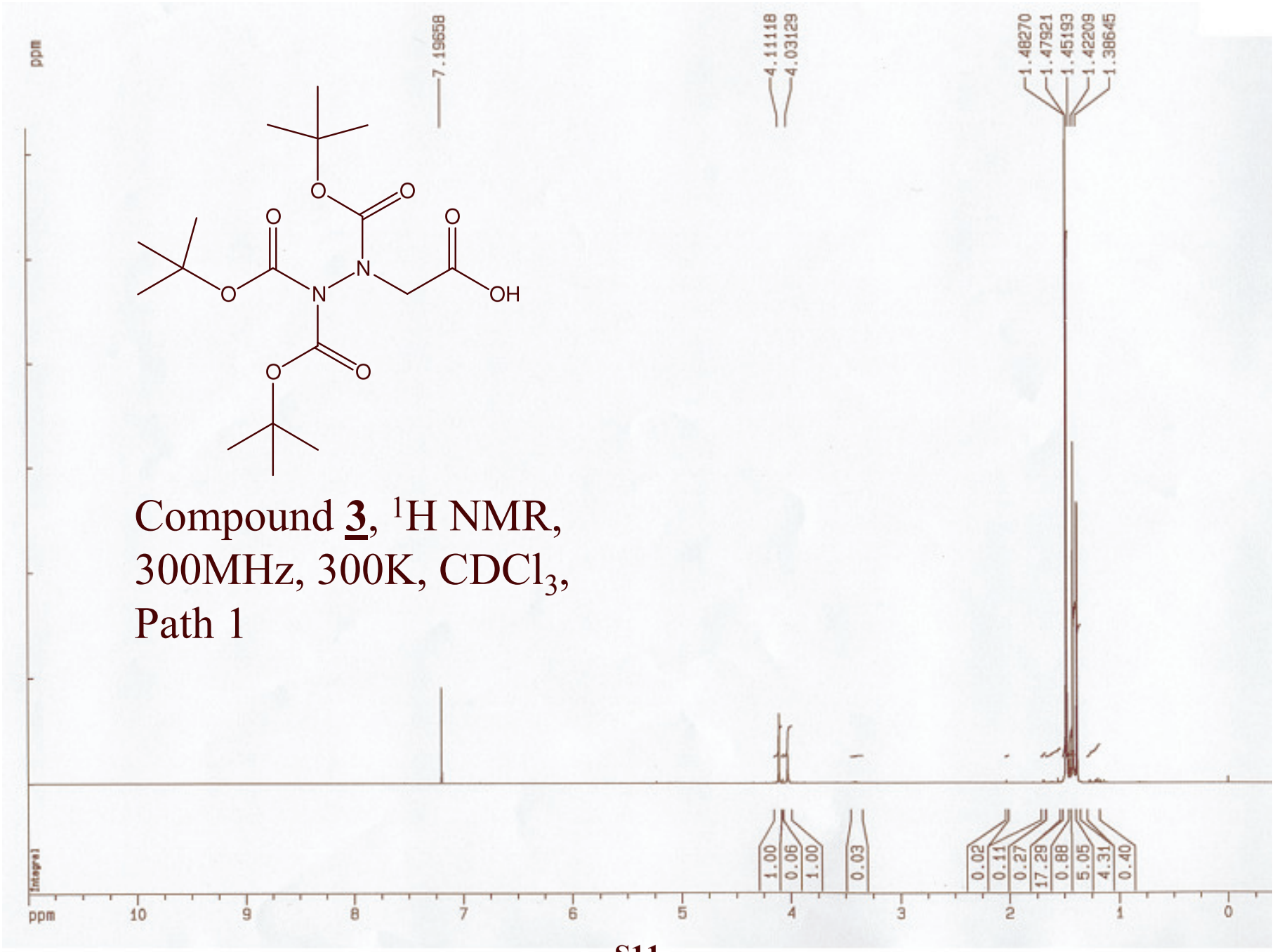


Compound **3**, ^{1}H NMR, 300MHz, 300K, DMSO, Path 1

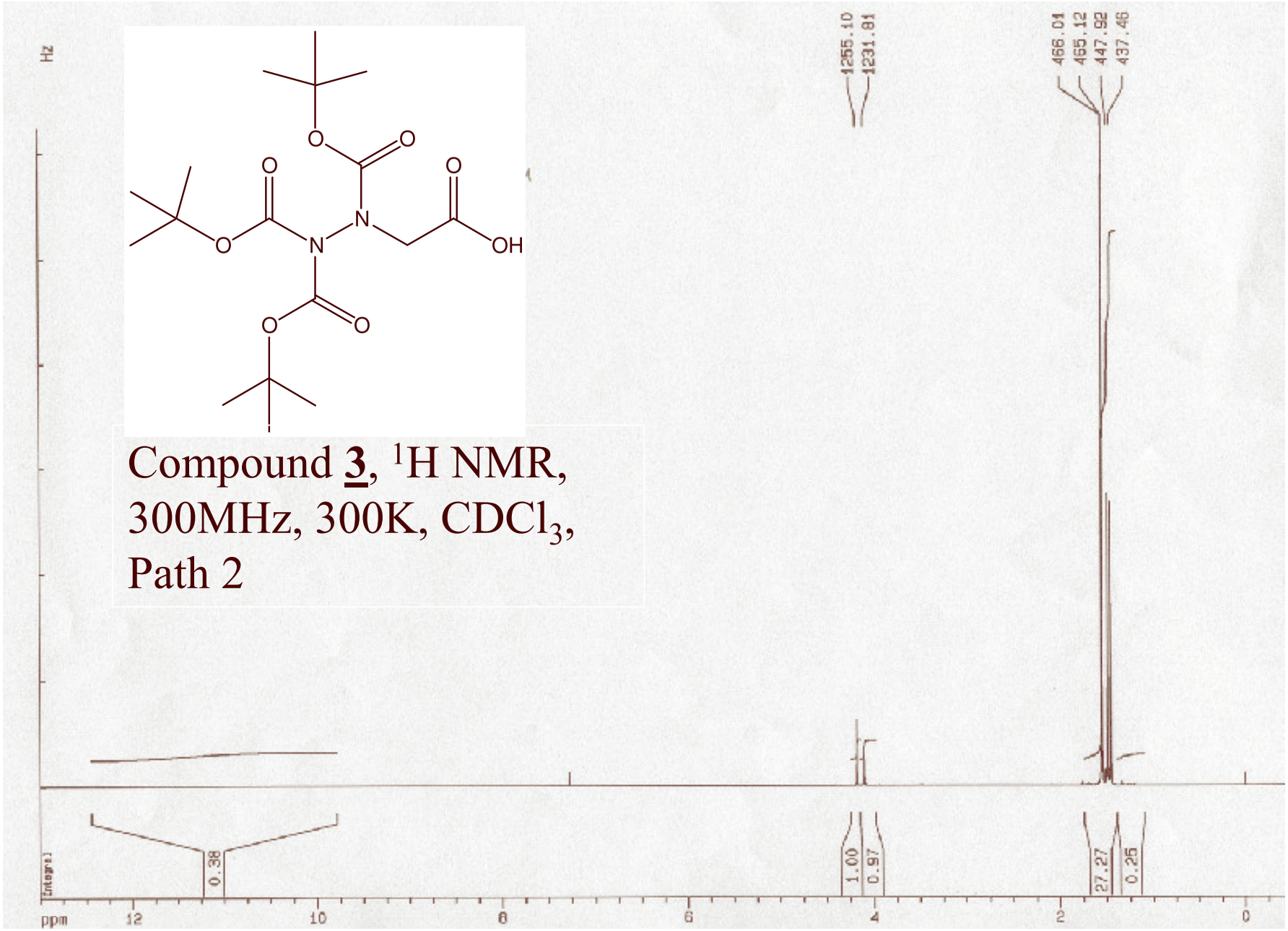

Hz
1212.09
753.45
751.60
749.79
432.55
426.22
422.01
411.90
OH
Compound 3, 1H NMR,
300MHz, 300K, DMSO,
Path 1
Integral
2.37
1.63
1.90
32.41
ppm
4.0
3.5
3.0
2.5
2.0
1.5



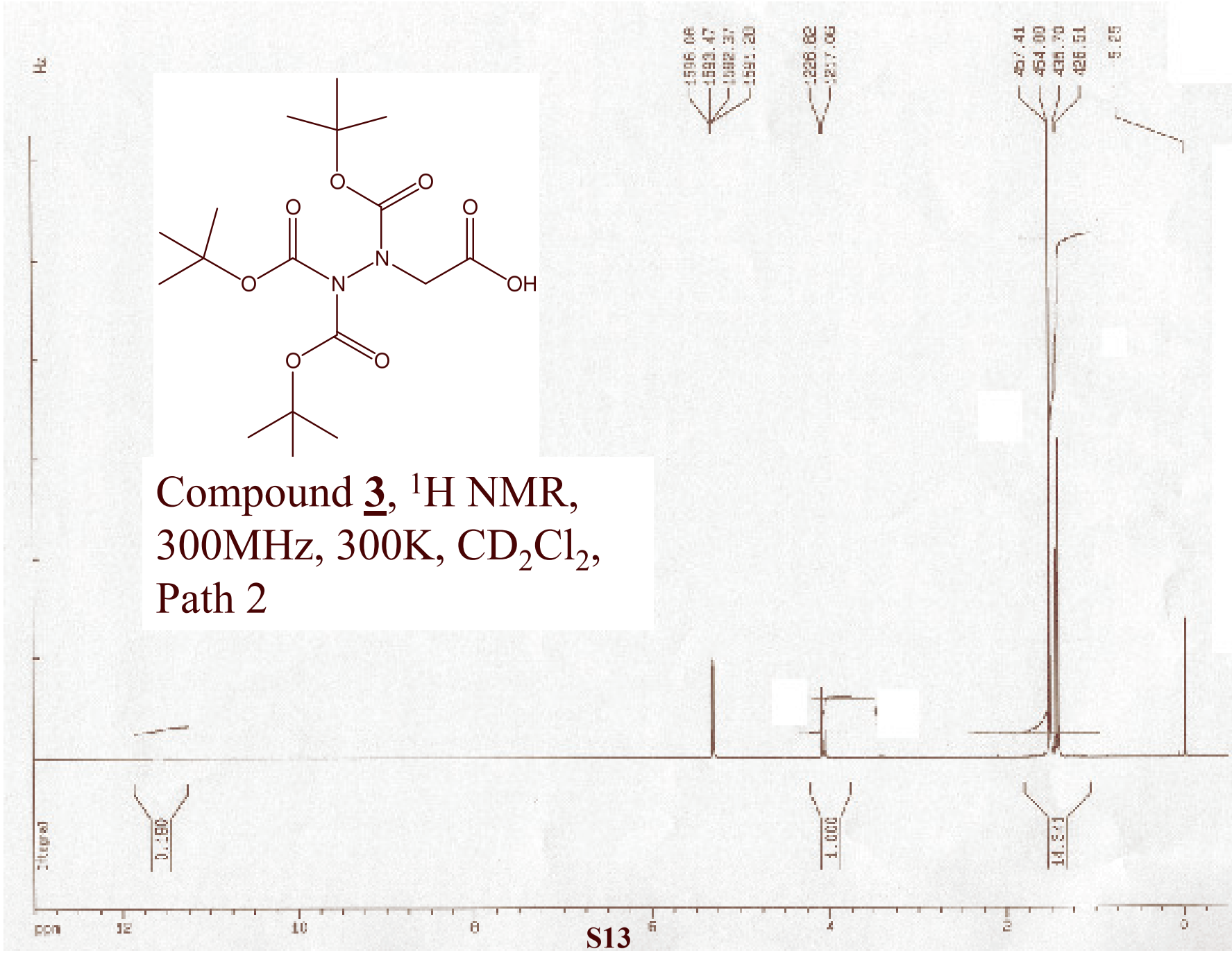
Compound **3**, ^{13}C NMR, 75MHz, 300K, DMSO, Path 1



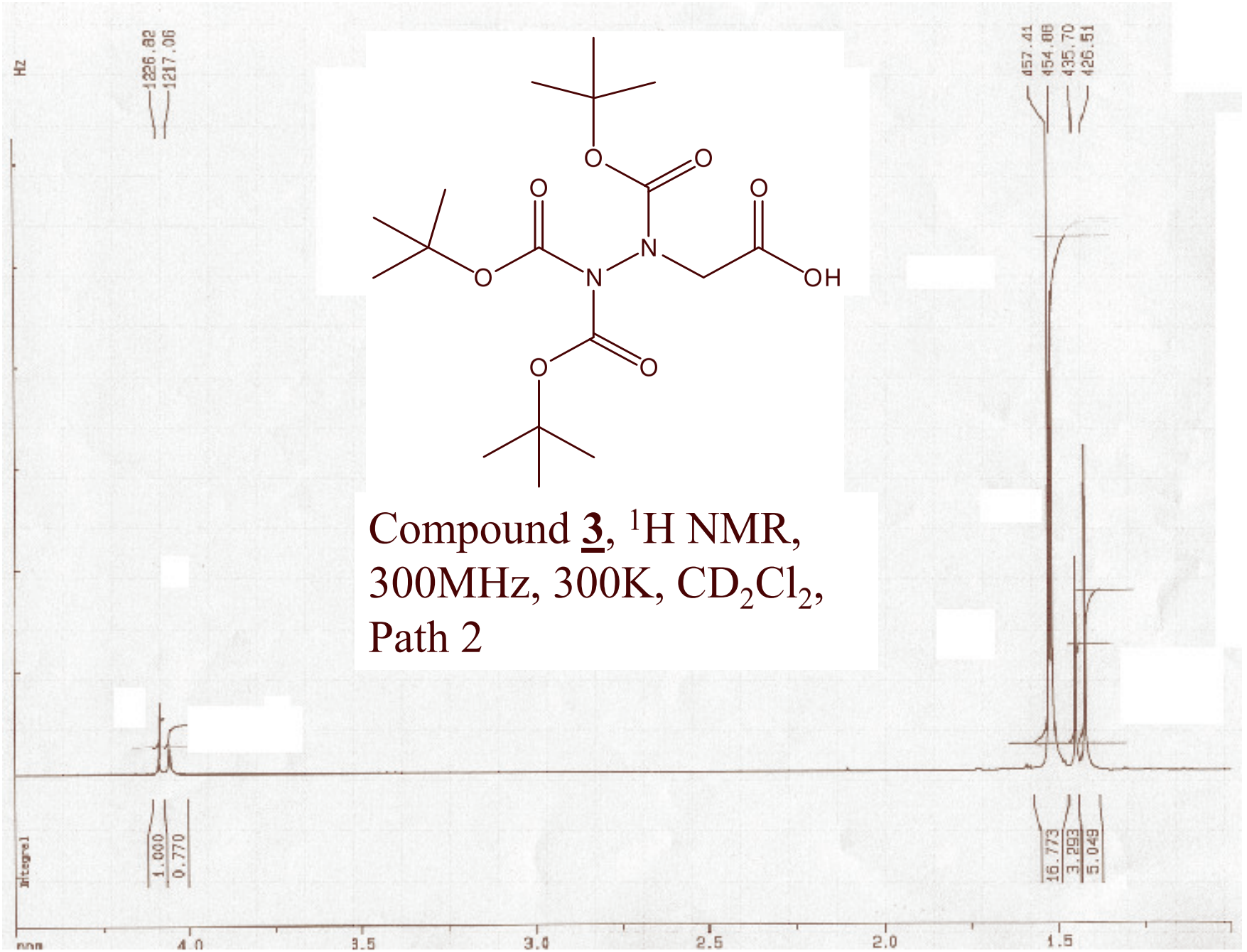
Compound **3**, ^{1}H NMR, 300MHz, 300K, $CDCl_3$, Path 1



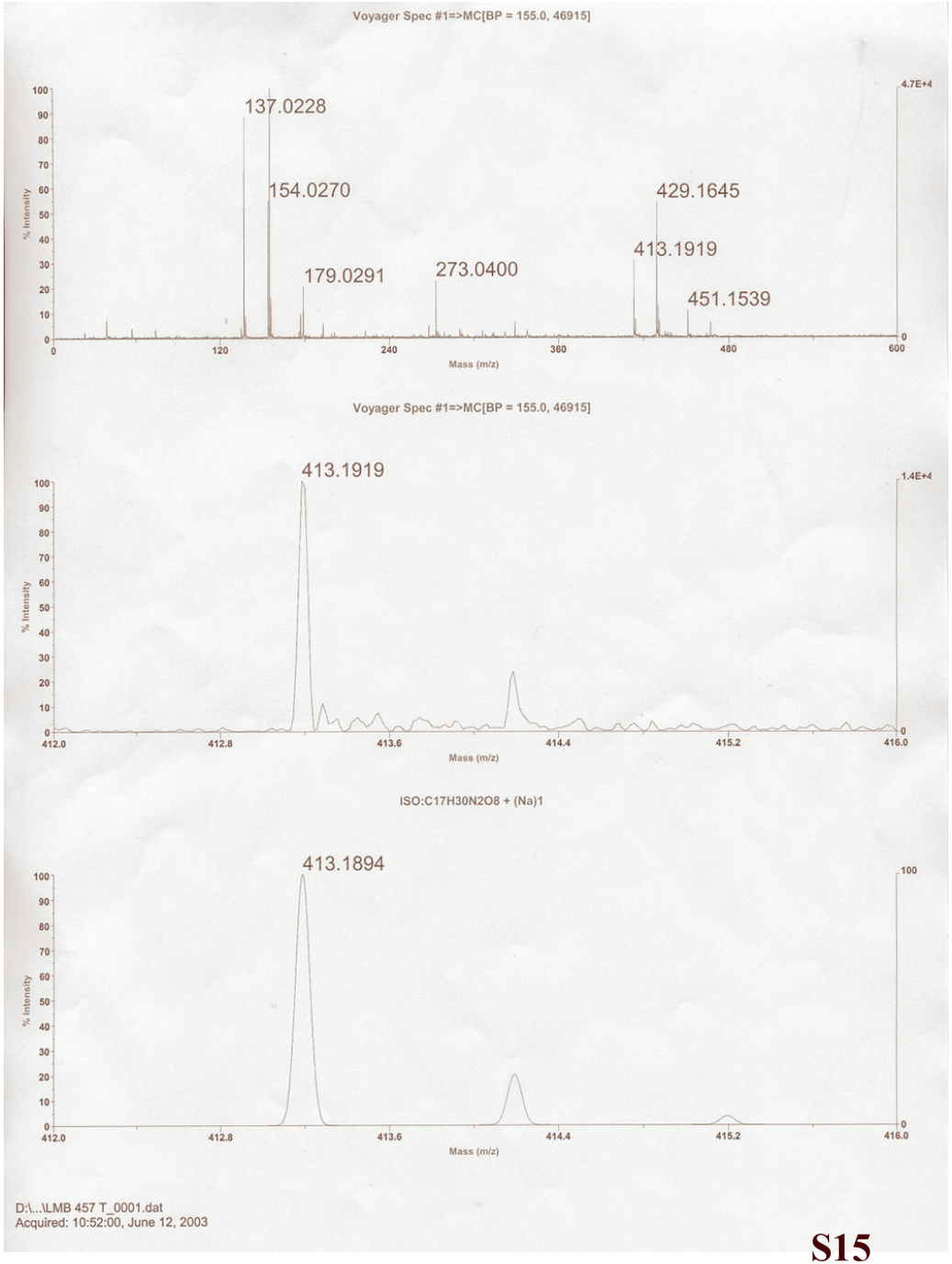
Compound **3**, ^{1}H NMR, 300MHz, 300K, $CDCl_3$, Path 2

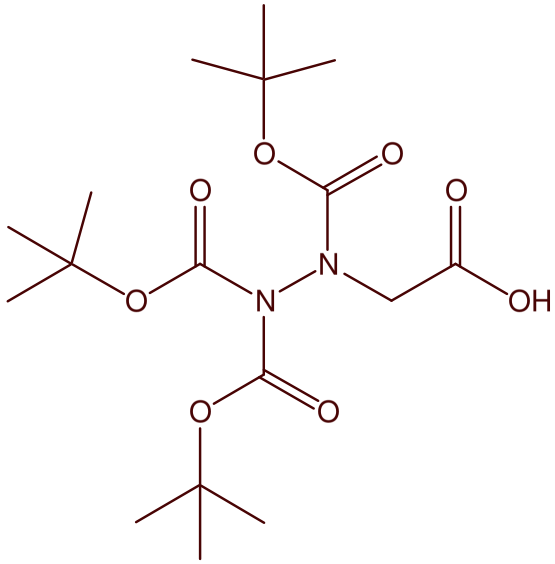


Compound **3**, ^{1}H NMR, 300MHz, 300K, CD_2Cl_2, Path 2

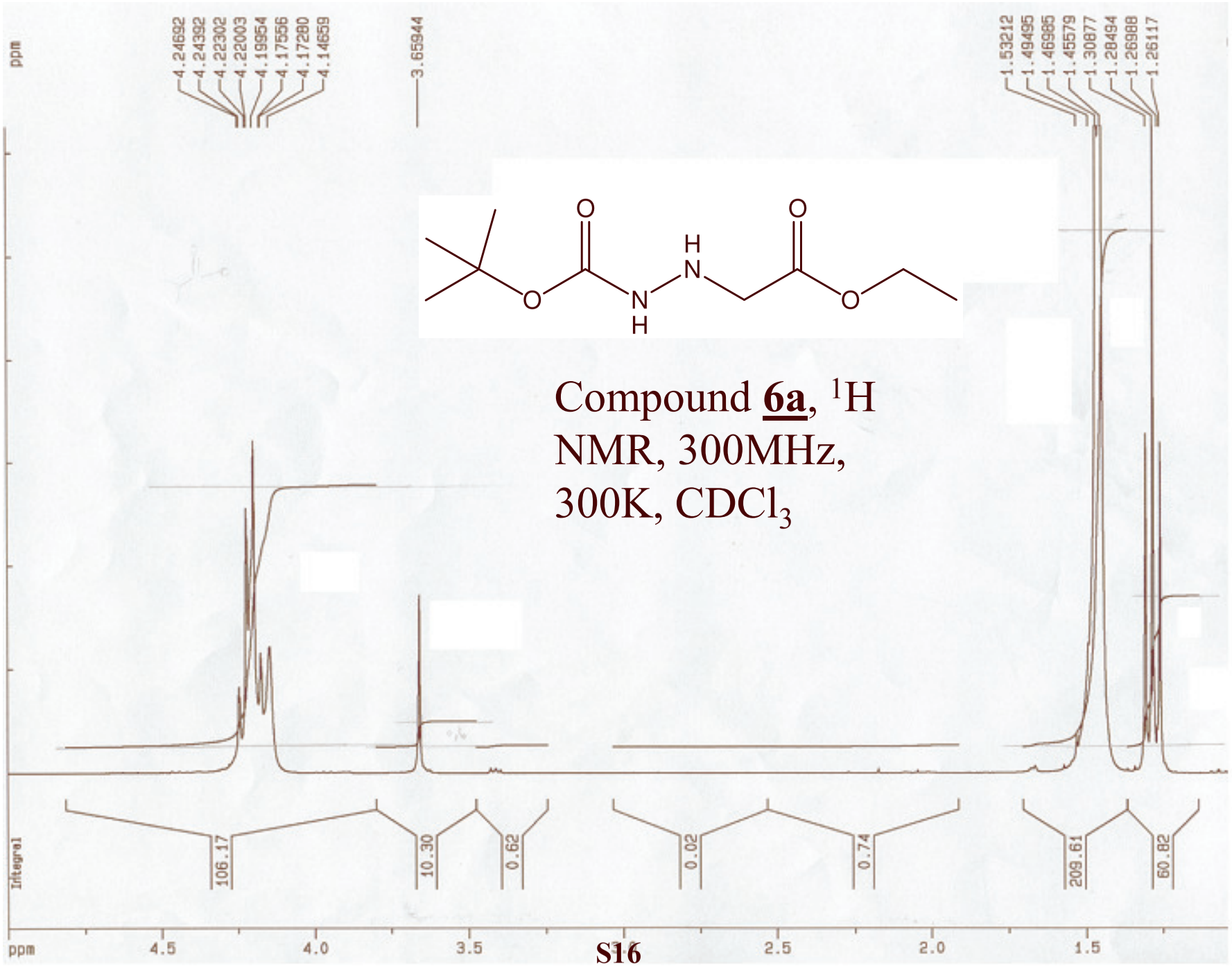


Compound **3**, ^{1}H NMR, 300MHz, 300K, CD_2Cl_2, Path 2

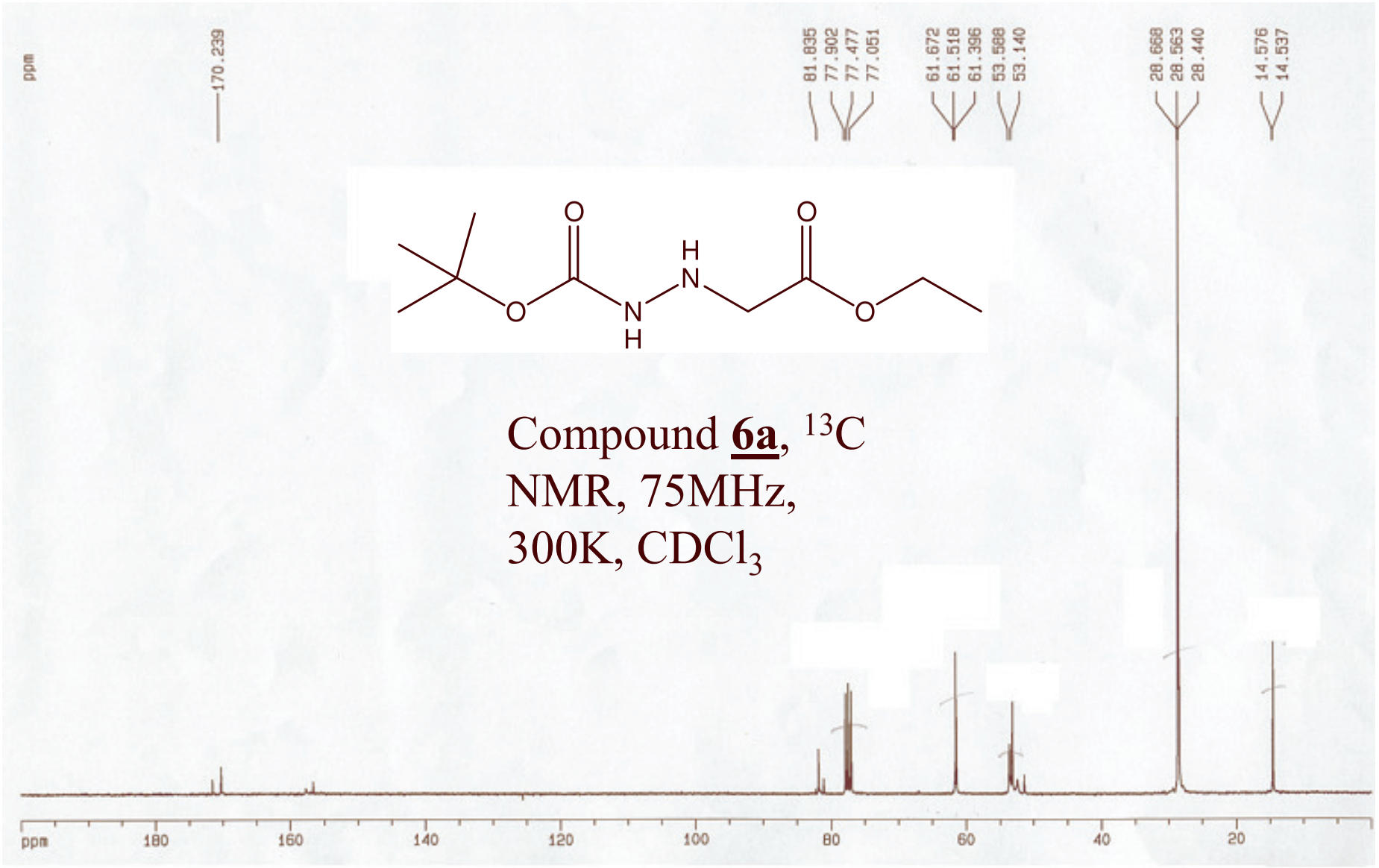


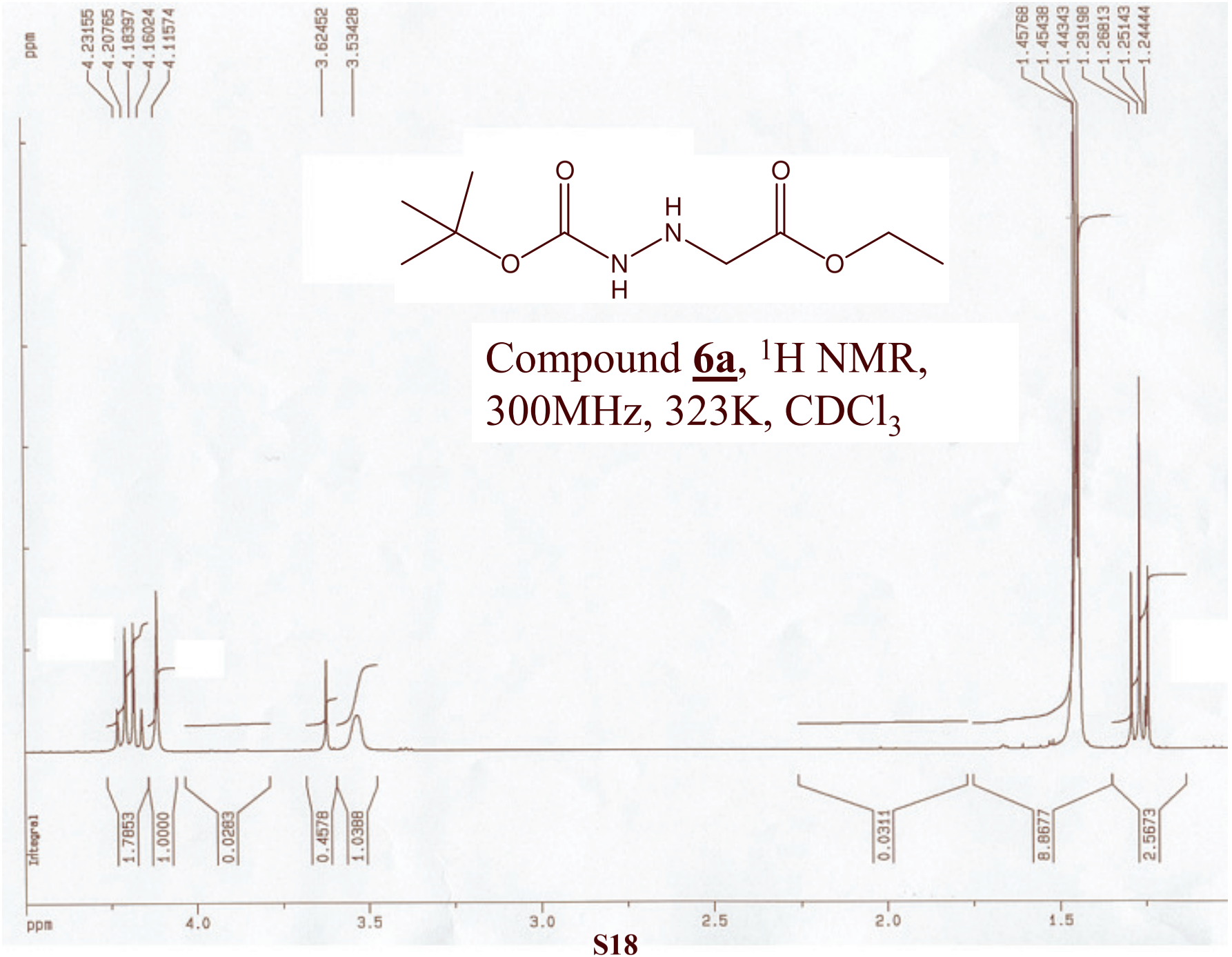


Compound **3**, Maldi-TOF, Matrix 2,5-dihydroxybenzoic acid, Path 1

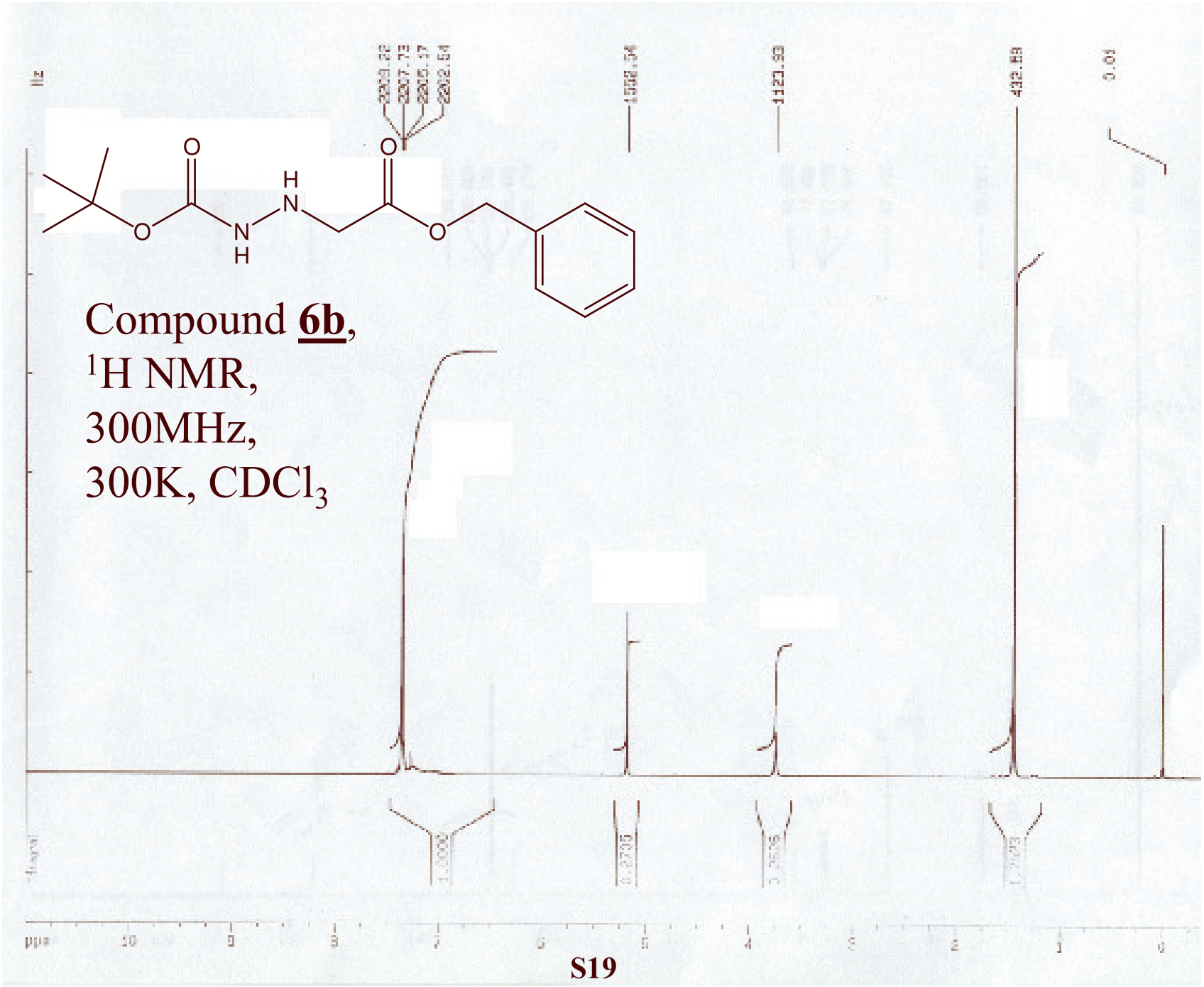


Compound **6a**, ^{1}H NMR, 300MHz, 300K, $CDCl_3$

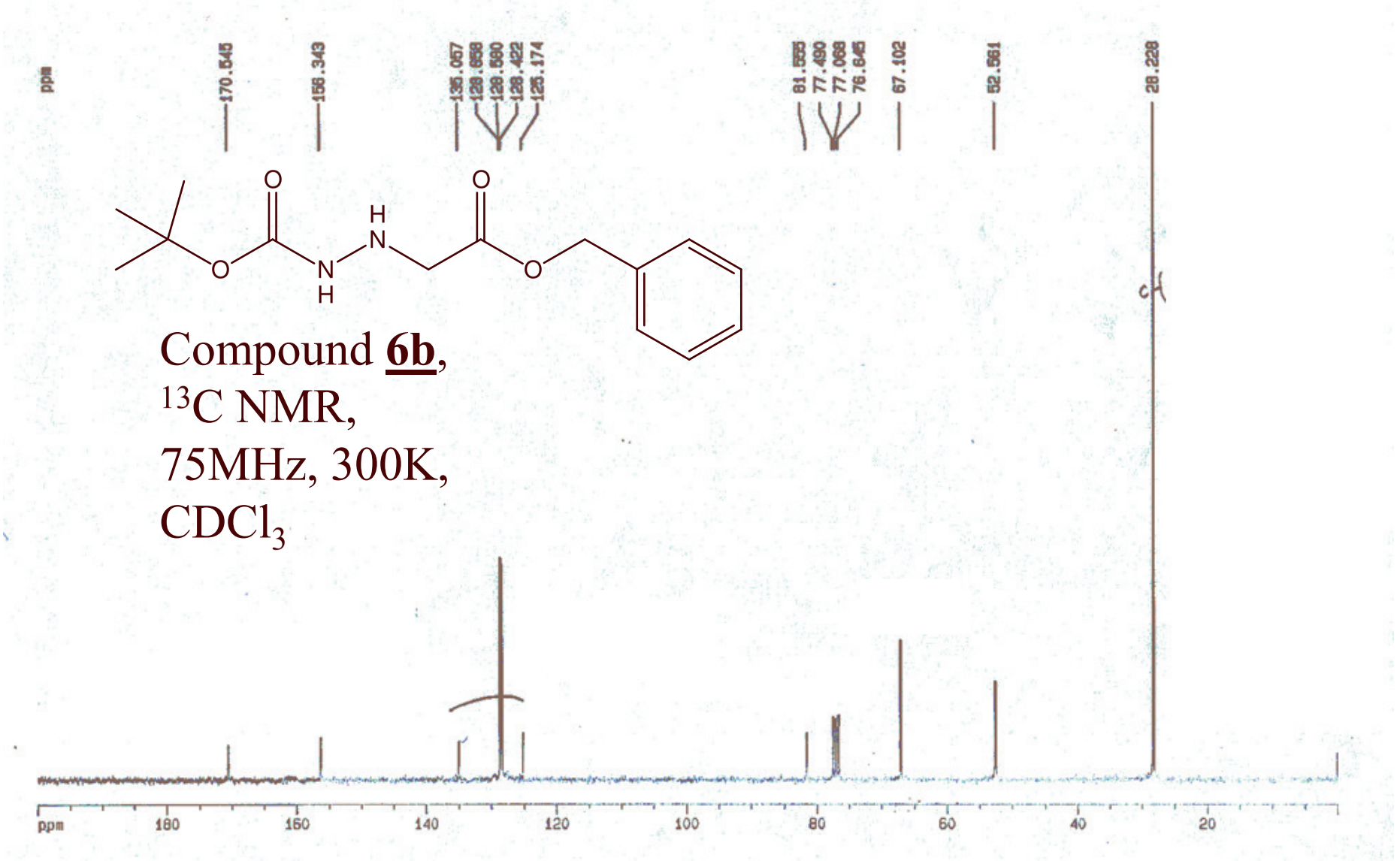

ppm
170.239
81.835
77.902
77.477
77.051
61.672
61.518
61.396
53.588
53.140
28.668
28.563
28.440
14.576
14.537
O
O
H
N
N
H
O
O
Compound 6a, 13C
NMR, 75MHz,
300K, CDCl3
ppm
180
160
140
120
100
80
60
40
20



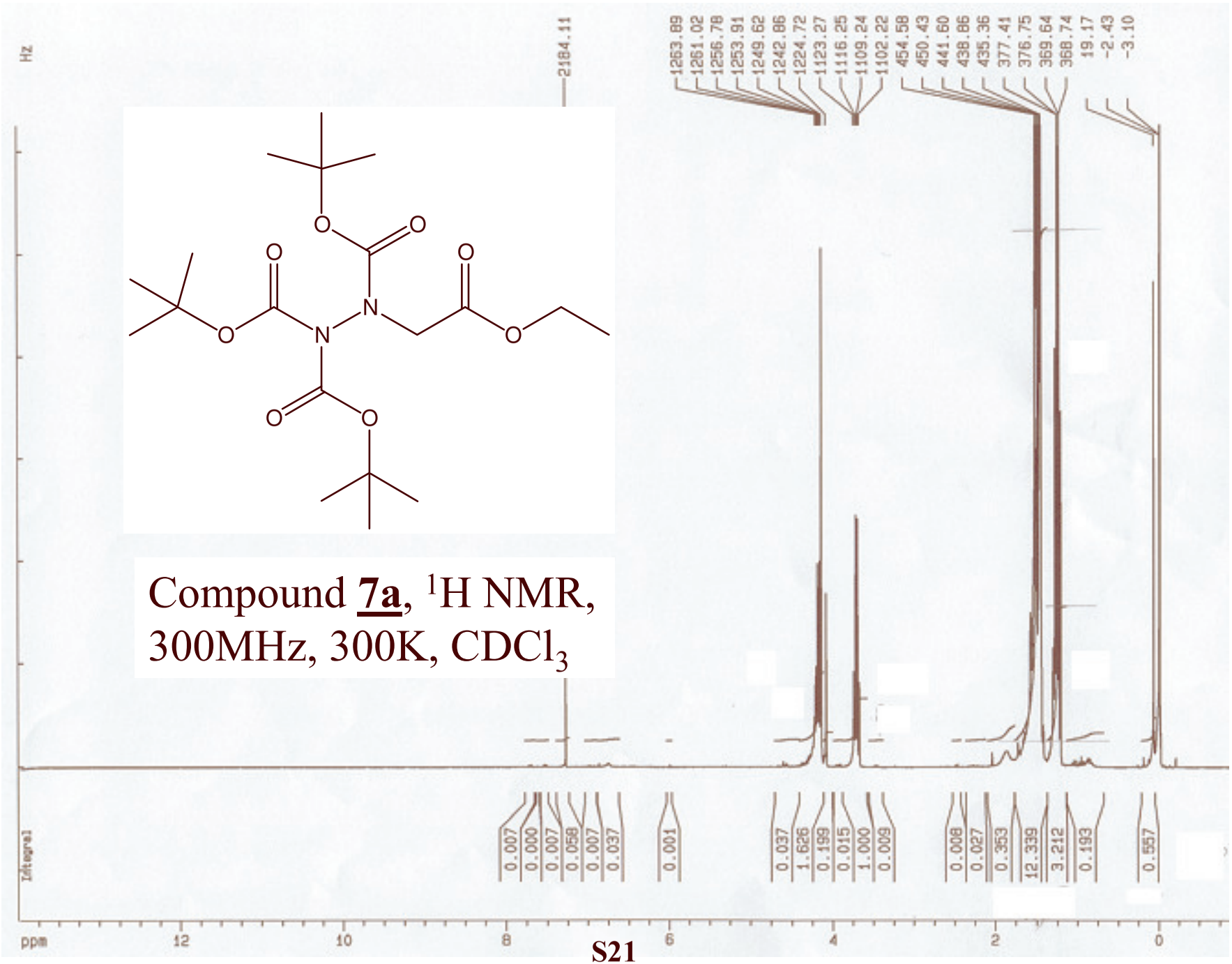
Compound **6a**, ^{1}H NMR, 300MHz, 323K, $CDCl_3$



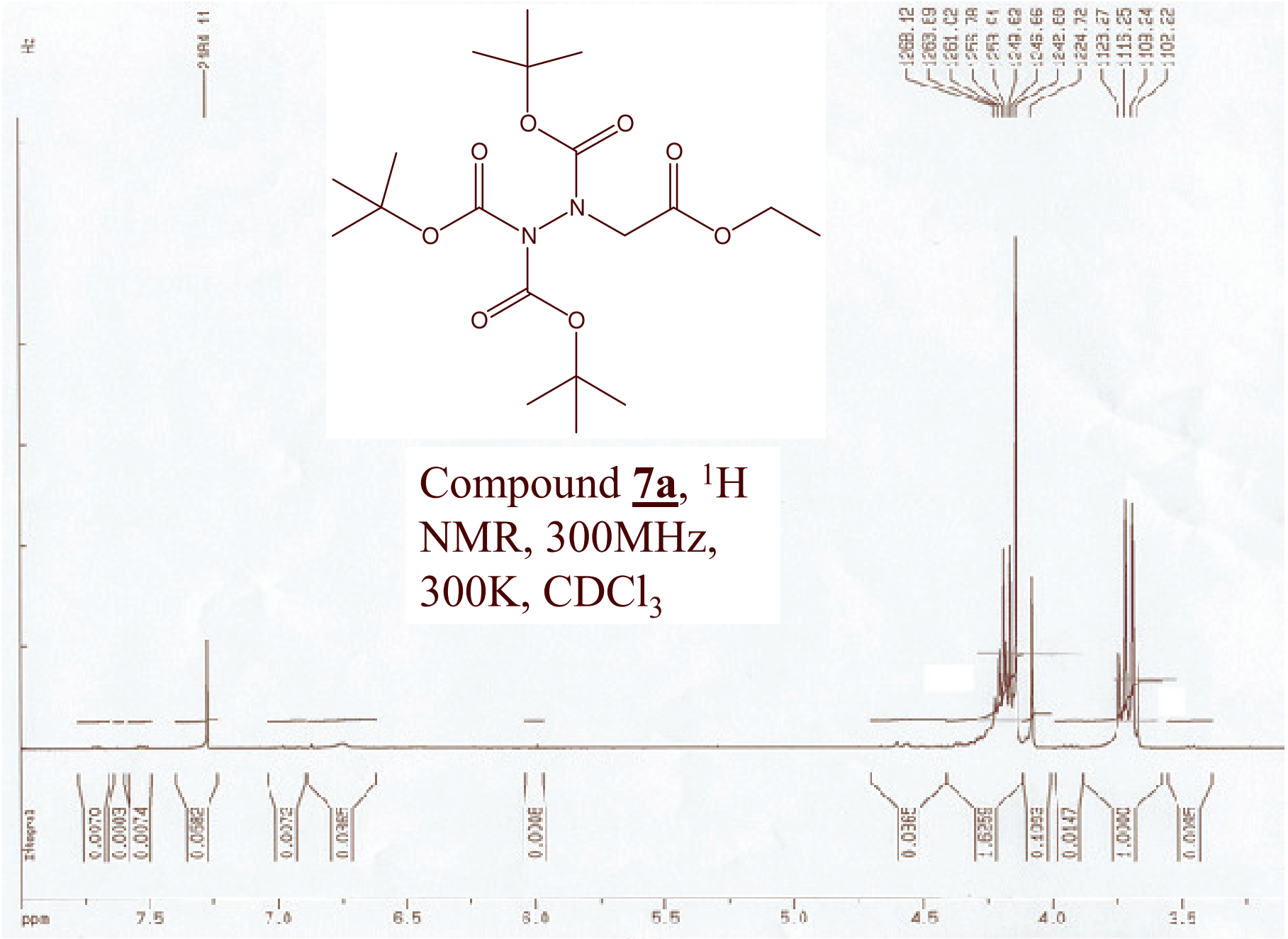
Compound **6b**,
^{1}H NMR,
300MHz,
300K, $CDCl_3$



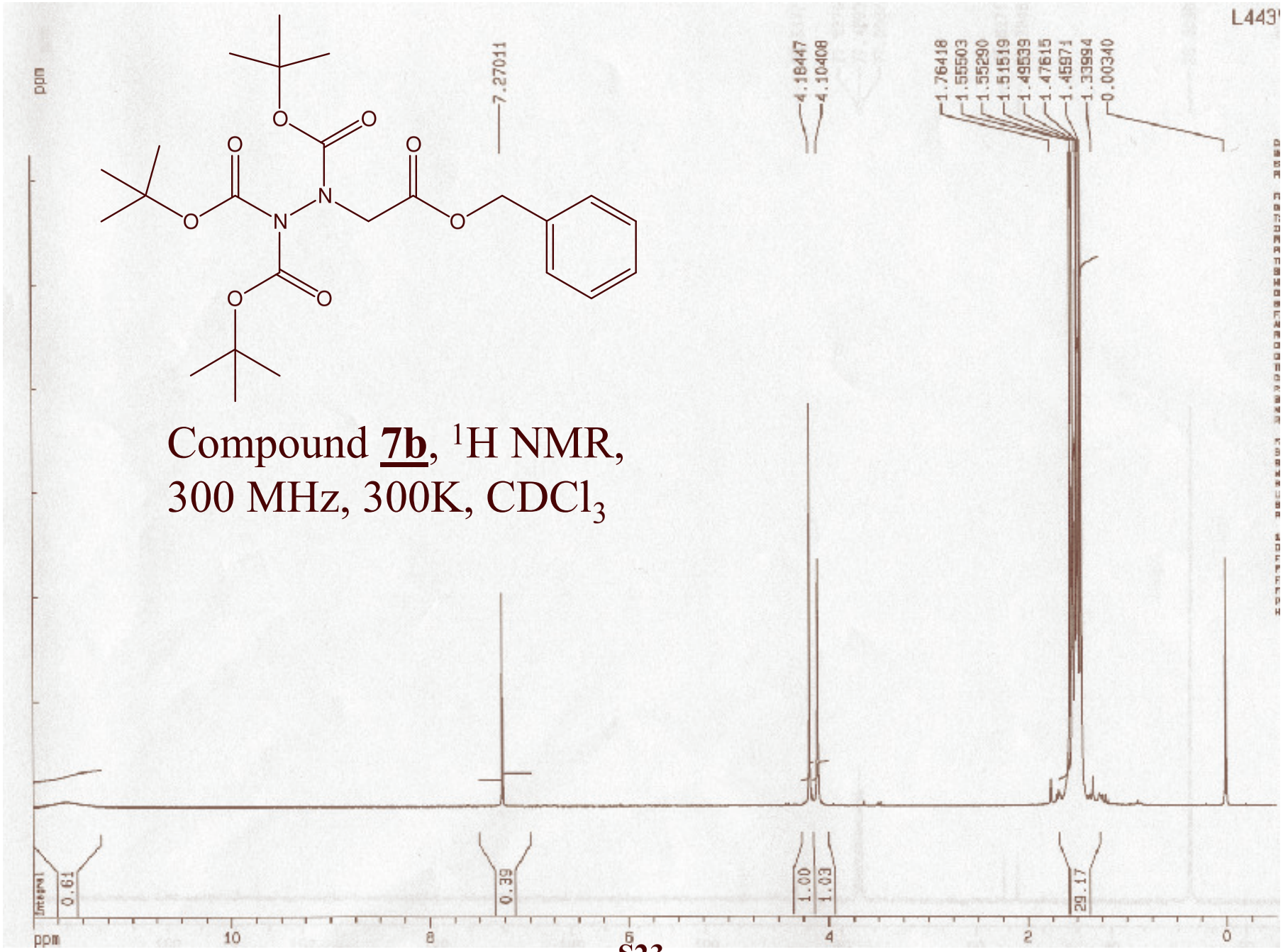
Compound **6b**,
^{13}C NMR,
75MHz, 300K,
$CDCl_3$



Compound **7a**, ^{1}H NMR, 300MHz, 300K, $CDCl_3$



Compound **7a**, ^{1}H NMR, 300MHz, 300K, $CDCl_3$



Compound **7b**, ^{1}H NMR,
300 MHz, 300K, $CDCl_3$

Peptide **13** : RP- HPLC before purification

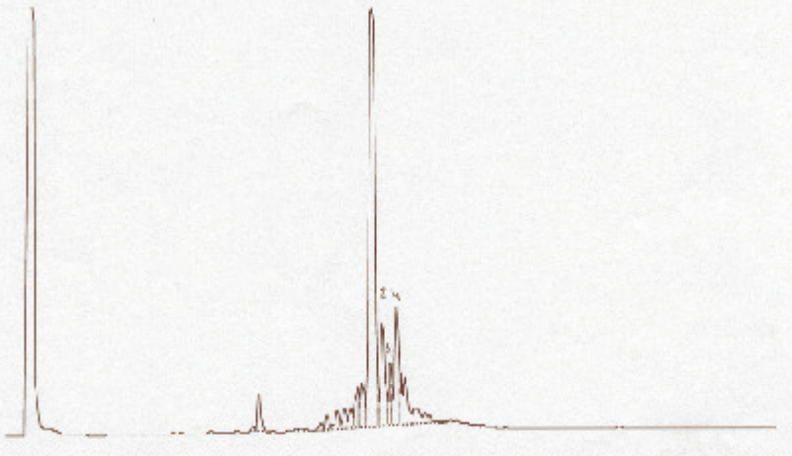
Date: Lun 11 Oct 1999 10:32
Data: Nef 201 triboc-001

Sample: Colonne C3 Zorbax 1ml/min 215 nm
0%---->100% de B en 60 min
T°: 50°C CH3CN 80%/H2O/TFA

nef 201 triboc Brut TFA

Method: MB 60 min
Inject Vol: 40
Sampling Int: 0.1 Seconds

Data:



0.0 59.9

Analysis: Channel A

Peak No.	Time	Type	Height(µV)	Area(µV-sec)	Area%
1	19.601	*N	52621	738159	2.234
2	26.735	*N1	28926	1719611	5.204
3	26.866	*N2-	15076	988922	2.993
4	27.463	*N3-	28581	1332893	4.034
5	28.248	*N4	625764	17059686	51.636
6	28.961	*N5	35517	2942657	8.906
7	29.616	*N6-	35489	1327372	4.017
8	30.251	*N7	176147	3325993	10.067
9	30.583	*N8	78682	3602846	10.905
Total Area				33038139	99.996

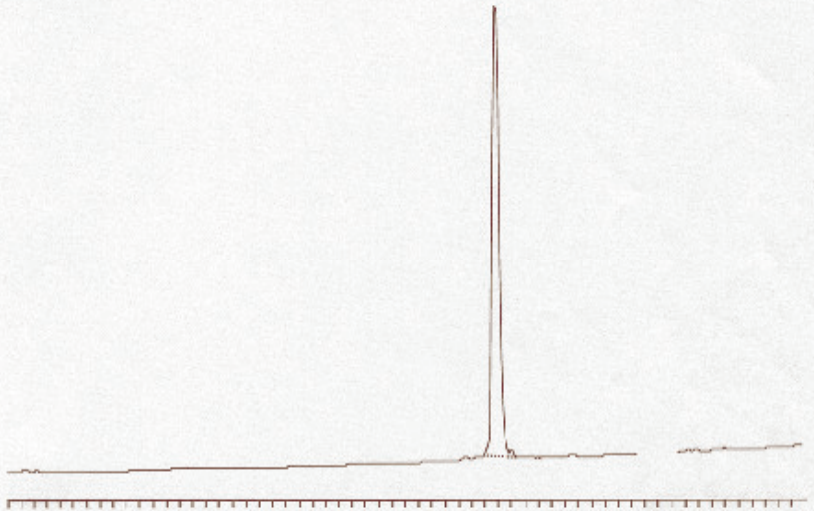
Peptide **13** : RP-HPLC after purification

Date: Ven 29 Oct 1999 9:35
Data: Réunion finale

Sample: Colonne C3 Zorbax 1ml/min 215 nm
0%---->100% de B en 30 min T°: 50°C
IprOH 40% / 0,05% TFA/H2O
Nef 201-225 Hdz
Réunion finale Prépa 1 à 6
Poids: 141 mg

Method: MB 60 min
Inject Vol: 10
Sampling Int: 0.1 Seconds

Data:



0.0 60.0

Analysis: Channel A

Peak No.	Time	Type	Height(µV)	Area(µV-sec)	Area%
1	36.680	N1	443990	12404490	99.245
2	37.926	N2	6688	94339	0.754
Total Area				12498829	99.999

Peptide **13**, ES-MS