Supporting Information

Synthesis and Biological Activity of 2,22-Dimethylene Analogues of 19-Norcalcitriol and Related Compounds

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Contents:

Purity criteria for the synthesized vitamin D compounds

All vitamin D analogues synthesized by us gave single sharp peaks on HPLC and they were judged at least 99% pure. Two HPLC systems (straight- and reversed-phase) were employed as indicated in the Table 2. The purity and identity of the synthesized vitamins were additionally confirmed by inspection of their ^{1}H NMR and high-resolution mass spectra.

Table 2. Purity Criteria for Target Vitamin D Compounds

Compound	Compd. No.	HPLC Retention Volumes	
		Straight-phase[a] (hexane/2-propanol)	Reversed-phase[b] (methanol/water)
(20*R*)-1α-Hydroxy-2,22-dimethylene-19-norvitamin D_3	**8**	h/p (95:5) 35 mL	m/w (94:6) 37 mL
(20*R*)-1α,25-Dihydroxy-2,22-dimethylene-19-norvitamin D_3	**9**	h/p (95:5) 36 mL	m/w (87:13) 29 mL
(20*R*)-1α,25-Dihydroxy-2,22-dimethylene-24-homo-19-norvitamin D_3	**10**	h/p (92:8) 45 mL	m/w (85:15) 52 mL
(20*R*)-1α,25-Dihydroxy-2,22-dimethylene-26,27-dihomo-19-norvitamin D_3	**11**	h/p (95:5) 27 mL	m/w (90:10) 29 mL
(20*R*)-1α,25-Dihydroxy-2,22-dimethylene-24,26,27-trihomo-19-norvitamin D_3	**12**	h/p (92:8) 25 mL	m/w (90:10) 38 mL
(20*R*)-1α-Hydroxy-2,22-dimethylene-24-(1'-hydroxycyclopentyl)-19,25,26,27-tetranorvitamin D_3	**13**	h/p (90:10) 40 mL	m/w (85:15) 50 mL

[a]Zorbax-Sil; 9.4 mm × 25 cm column; [b]Zorbax Eclipse XDB-C18; 9.4 mm × 25 cm column.

Conformational analysis of model 8-methylene *des*-A,B-compounds BM-FM

Structural analysis of model 8-methylene *des*-A,B-compounds **BM-FM**, performed according to a protocol described by us previously in detail,[1] in all cases gave similar results. They are exemplified by Figure 1S showing the "dot map" of model compound **BM** characterized by the presence of the side chain identical as in the planned 2MD analog **9**. Thus, in the low-energy side chain conformers of the examined compound, falling into the 1 kcal/mol energy window, the 25-oxygen resides in the spatial regions EG and EA; the former rear region (EG) is somewhat less active but the latter front region EA is considered as the most active one.[2]

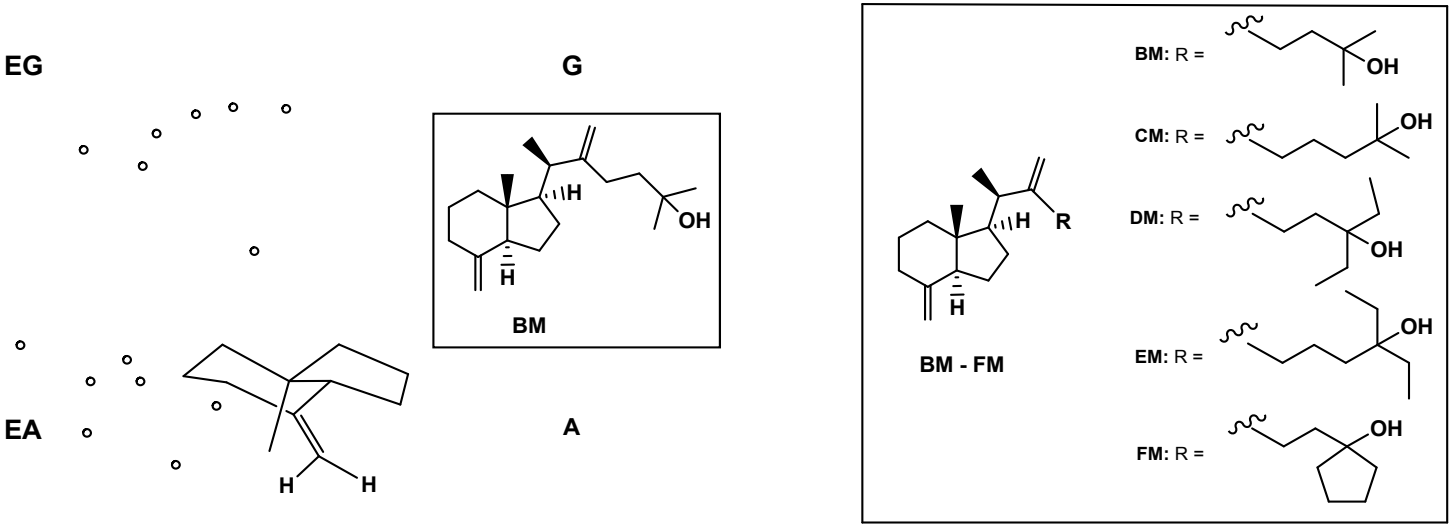
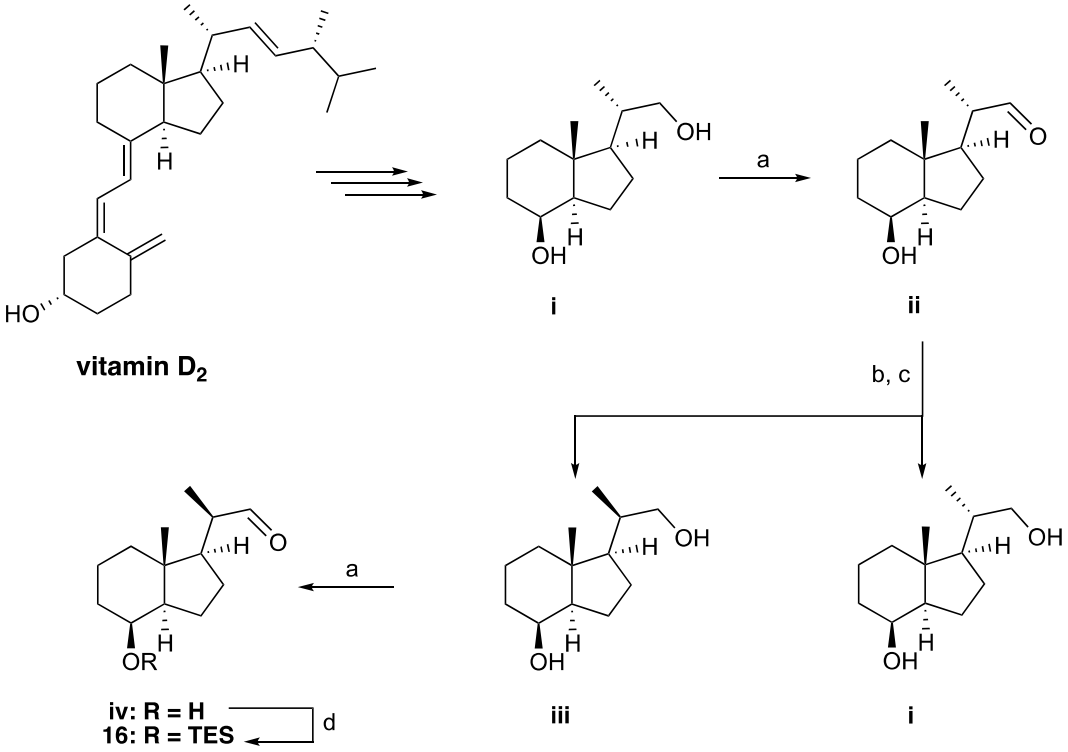


Figure 1S. The structures of the model 8-methylene compounds **BM-FM** possessing side chains identical to those of the synthesized analogs **9-13**. Stereo-view illustrating side-chain conformational analysis of compound **BM**. Energy-minimized conformations of this compound (energy window 1 kcal/mol) were overlaid; the circles show the location of 25-oxygen atoms in the corresponding conformers. Side-chain carbons and hydrogen atoms are omitted for clarity. There are also indicated spatial regions EG, G, EA, and A as defined by Yamada.[2]

Preparation of (20*R*)-8β-[(triethylsilyl)oxy]-*des*-A,B-23,24-dinorcholan-22-al (16)

For the preparation of **16** we used slightly modified literature procedures.[3,4] Thus, the Inhoffen-Lythgoe diol **i**, easily obtained from vitamin D_2,[5] was oxidized with TEMPO/NCS system[6] and the formed hydroxy aldehyde **ii** was subjected to equilibration with piperidine.[7] The resulting crude mixture of epimeric aldehydes (**iv** and unchanged **ii** at the ratio of 3:1) was directly reduced with $NaBH_4$ and the formed epimeric alcohols were separated by column chromatography. The minor isomer **i** was recycled and the main 20-epi-Ihoffen-Lythgoe diol (**iii**) oxidized to the hydroxy aldehyde **iv** which after silylation provided the desired synthon **16**.



Scheme 1S. (a) TEMPO, TBACl, NSC, $NaHCO_3/K_2CO_3$ buffer pH 8.6, CH_2Cl_2, (**ii**: 93%, **iv**: 88%); (b) piperidine, benzene, 93%; (c) $NaBH_4$, THF, EtOH, (**iii**: 75%); (d) TESOTf, 2,6-lutidine, CH_2Cl_2, 88%.

(20*S*)-8β-Hydroxy-*des*-A,B-23,24-dinorcholan-22-al (ii). To the vigorously stirred solution of Inhoffen-Lythgoe diol **i** [1.37 g, 6.48 mmol; prepared as described by Grzywacz et al.[5] in CH_2Cl_2 (50 mL) and $NaHCO_3/K_2CO_3$ buffer (pH 8.6; 50 mL), TEMPO (101 g, 647 μmol) was added followed by TBACl (180 mg, 1.64 mmol) and NCS (1.731 g, 12.9 mmol). The reaction mixture was stirred overnight at the room temperature, poured into water and extracted with CH_2Cl_2. The combined extracts were dried over Na_2SO_4 and concentrated. Purification by column chromatography on silica with hexane/ethyl acetate (7:3) yielded aldehyde **ii** (1.27 g, 93%) as a colorless oil which spectral data were identical with these reported in the literature.[4]

(20*R*)-*des*-A,B-23,24-Dinorcholano-8β,22-diol (iii). Solution of aldehyde **ii** (378 mg, 1.8 mmol) in anhydrous benzene (9 mL) and piperidine (213 μL, 2.15 mmol) was refluxed for 8 h using Bidwell-Sterling trap. The solvents were removed on the rotary evaporator. Spectral analysis (^{1}H NMR, 500 MHz, $CDCl_3$) of the crude products obtained in 93% yield confirmed a presence of two isomeric aldehydes: **iv** [9.542 ppm (1H, d, J = 5.1 Hz, 22-H)][4] and unchanged **ii** [9.575 ppm (1H, d, J = 3.1 Hz, 22-H)][4] at the ratio of 3:1. This crude mixture of aldehydes was then dissolved in dry THF (4 mL), treated with $NaBH_4$ (123 mg) and EtOH (4 mL). After 2 h reaction was quenched with water and materials were extracted with ethyl acetate. Combined organics were dried over Na_2SO_4 and concentrated. 20-*Epi*-Inhoffen- Lythgoe diol **iii** (261 mg, 75%) was isolated by column chromatography on silica with hexane/ethyl acetate (7:3) as white crystals which spectral data were identical with these reported in the literature.[4]

(20*R*)-8β-[(Triethylsilyl)oxy]-*des*-A,B-23,24-dinorcholan-22-al (16). Diol **iii** (273 mg, 1.28 mmol) was then subjected to selective oxidation of the primary alcohol according to the procedure described above for the alcohol **i**. The obtained hydroxy aldehyde **iv** (242 mg, 88%), which spectral data were identical with these reported in the literature,[4] was dissolved in anhydrous CH_2Cl_2 (30

mL) containing 2,6-lutidine (395 μL, 3.41 mmol). To this solution triethylsilyl trifluoromethanesulfonate (715 μL, 3.16 mmol) was added dropwise at -40 °C and the mixture was stirred for 30 min. After addition of saturated $NaHCO_3$, the mixture was allowed to warm up to room temperature, and then it was extracted with CH_2Cl_2, dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography on silica with hexane/ethyl acetate (95:5) to give silylated compound **16** (329 mg, 88%) as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 0.536 (6[H, q, *J* = 7.8 Hz, 3 × $SiCH_2$), 0.909 (3H, s, 18-H_3), 0.926 (9H, t, *J* = 7.8 Hz, 3 × $SiCH_2C\underline{H}_3$), 0.997 (3H, d, *J* = 6.8 Hz, 21-H_3), 4.03 (1H, narr m, 8α-H), 9.53 (1H, d, *J* = 10 Hz, C<u>H</u>O); ^{13}C NMR (100 MHz, $CDCl_3$) δ 5.2, 7.1, 13.6, 14.8, 17.6, 22.6, 25.8, 34.7, 39.7, 41.9, 48.5, 52.8, 52.9, 69.1, 206.1; HRMS (ESI) exact mass calculated for $C_{19}H_{37}O_2Si$ (M^+ + H) 325.2562, found 325.2574.

Preparation of bromides (A-G) and the corresponding Grignard reagents

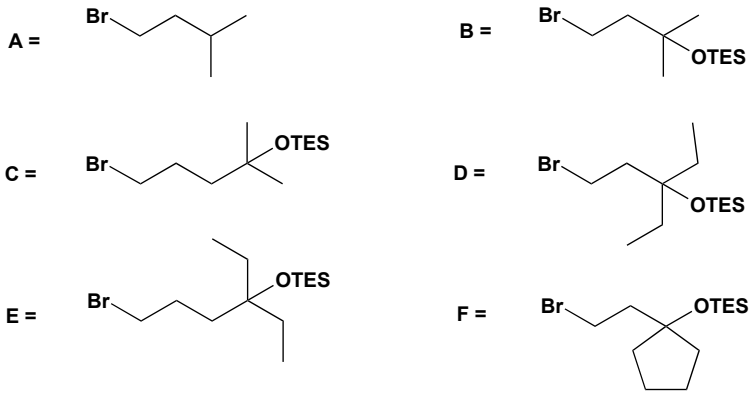


Figure 2S

1-Bromo-3-methyl-3-[(triethylsilyl)oxy]butane (B) and 1-bromo-3-ethyl-3-[(triethylsilyl)oxy]pentane (D).[8] Commercially available ethyl-3-bromopropionate was treated with methyl magnesium bromide or ethyl magnesium bromide, respectively, according to the standard procedures described in the literature.[9] The resultant tertiary alcohols were next silylated by treatment with TESOTf and 2,6-lutidine.[9]

1-Bromo-4-methyl-4-[(triethylsilyl)oxy]pentane (C) and 1-bromo-4-ethyl-4-[(triethylsilyl)oxy]hexane (E). Analogously, as described above for **B** and **D**, these bromides were obtained from commercially available ethyl-4-bromobutyrate in the reaction with the corresponding Grignard reagents followed by protection of the formed tertiary alcohols with TESOTf and 2,6-lutidine.

1-(2'-Bromoethyl)-1-[(triethylsilyl)oxy]cyclopentane (F) was obtained from commercially available β-propiolactone by the four-step procedure involving: (a) the reaction with the Grignard reagent derived from 1,4-dibromobutane,[9,10] (b) tosylation of the primary alcohol in the formed product using *p*-toluenesulfonyl chloride in pyridine, (c) conversion of the obtained tosylate to the bromide[11] (Adv. Synth. Catal. 2012, 354, 1519-28), and (d) protection of tertiary

alcohol with TESOTf and 2,6-lutidine.

(a) 1,4-Dibromobutane (3.82 mL, 32 mmol) was slowly added to the flask containing magnesium turnings (1.75 g, 73 mmol) in anhydrous THF (72 mL) at 0 ^{o}C under argon. After addition was complete, the mixture was stirred at room temperature for 30 min. Freshly generated Grignard reagent was then cooled to -40 ^{o}C and β-propiolactone (1.81 mL, 28.78 mmol) was slowly added with stirring. Cooling bath was removed after 20 min, the mixture was allowed to reach room temperature and the stirring was continued overnight. Reaction was quenched by addition of sat. aq. NH_4Cl solution, then it was extracted with ethyl acetate, dried over Na_2SO_4 and concentrated. Purification by column chromatography on silica (0→100% ethyl acetate/hexane) afforded 1-(2-hydroxyethyl)cyclopentanol (2.34 g, 64%).

(b) *p*-Toluenesulphonyl chloride (2.21 g, 11.6 mmol) was added to a stirred solution of the prepared diol (1.171 g, 9 mmol) and DMAP (76 mg, 0.62 mmol) in anh. CH_2Cl_2 (70 mL) containing TEA (4.7 mL) at 0 ^{o}C under argon. Reaction mixture was allowed to reach room temperature and stirring was continued for 2 h. Reaction was quenched by addition of water followed by 2% HCl aq. sol.. The mixture was extracted with CH_2Cl_2, dried over Na_2SO_4, and concentrated. Purification on silica column yielded 2-(1-hydroxycyclopentyl)ethyl 4-methylbenzenesulfonate (2.36 g, 92%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.353 (1H, s), 1.97 (2H, t, J = 6.8 Hz), 2.45 (3H, s), 4.24 (2H, t, J = 6.8 Hz), 7.34 and 7.79 (2H and 2H, each d, each J = 8.2 Hz, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 23.4, 39.6, 40.0, 68.0, 80.5, 127.9, 129.8, 132.9, 144.8; HRMS (ESI) exact mass calculated for $C_{14}H_{21}O_4S$ (M^+ + H) 285.1160, found 285.1171.

(c) Mixture of the tosylate (50 mg, 0.176 mmol) and anhydrous LiBr in anhydrous THF (1 mL)

was refluxed for 1 h. Oil bath was removed, mixture was allowed to reach room temperature and water was added. Materials were extracted with ethyl acetate, dried over Na_2SO_4, and concentrated. Purification on silica column yielded 1-(2-bromoethyl)cyclopentanol (32 mg, 95%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.64 (6H, m), 1.71 (2H, m), 2.20 (2H, t, *J* = 8.1 Hz), 3.54 (2H, t, *J* = 8.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.4, 28.9, 39.7, 44.7, 82.3; HRMS (ESI) exact mass calculated for $C_7H_{13}BrONa$ (M^+ + Na) 215.0047, found 215.0053.

(d) 2,6-Lutidine (940 µL, 8.135 mmol) was added to the solution of the prepared cyclopentanol (1.41 g, 7.33 mmol) in dry CH_2Cl_2 (20 mL) at -40 ^{o}C under argon followed by TESOTf (1.86 mL, 8.24 mmol). Reaction was stirred for 4 h, brine was added and the mixture was extracted with CH_2Cl_2. The combined organics were washed with 2% HCl aq. sol., dried over Na_2SO_4, and concentrated. Purification by column chromatography on silica eluted with hexane/ethyl acetate (95:5) afforded 1-(2'-bromoethyl)-1-[(triethylsilyl)oxy]cyclopentane (**F**; 2.19 g, 97%) as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.590 (6H, q, *J* = 6.7 Hz), 0.949 (9H, t, *J* = 6.7 Hz), 1.56 (6H, m), 1.72 (2H, m), 2.12 (2H, m), 3.48 (2H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 6.5, 7.1, 23.3, 29.3, 39.7, 45.8, 84.5; HRMS (ESI) exact mass calculated for $C_{11}H_{22}BrOSi$ (M^+ - Et) 277.0618, found 277.0628.

The ethereal solutions of the corresponding Grignard reagents were prepared from the bromides **A**-**F** and magnesium powder using standard procedure, analogous to that described below for the isopentyl magnesium bromide. Although this Grignard reagent was commercially available (Aldrich), it was freshly prepared by us from the bromide **A**.

1-Bromo-3-methylbutane (**A**; 393 µL, 3.28 mmol) was added dropwise to a stirred mixture of magnesium powder (78 mg, 3.21 mmol) in anhydrous THF (6 mL) under argon at room

temperature. After addition was complete the mixture was stirred at room temperature for 1 h. Then the prepared solution of isopentyl magnesium bromide was added to the solution of the aldehyde **16** as described in Experimental Section.

References

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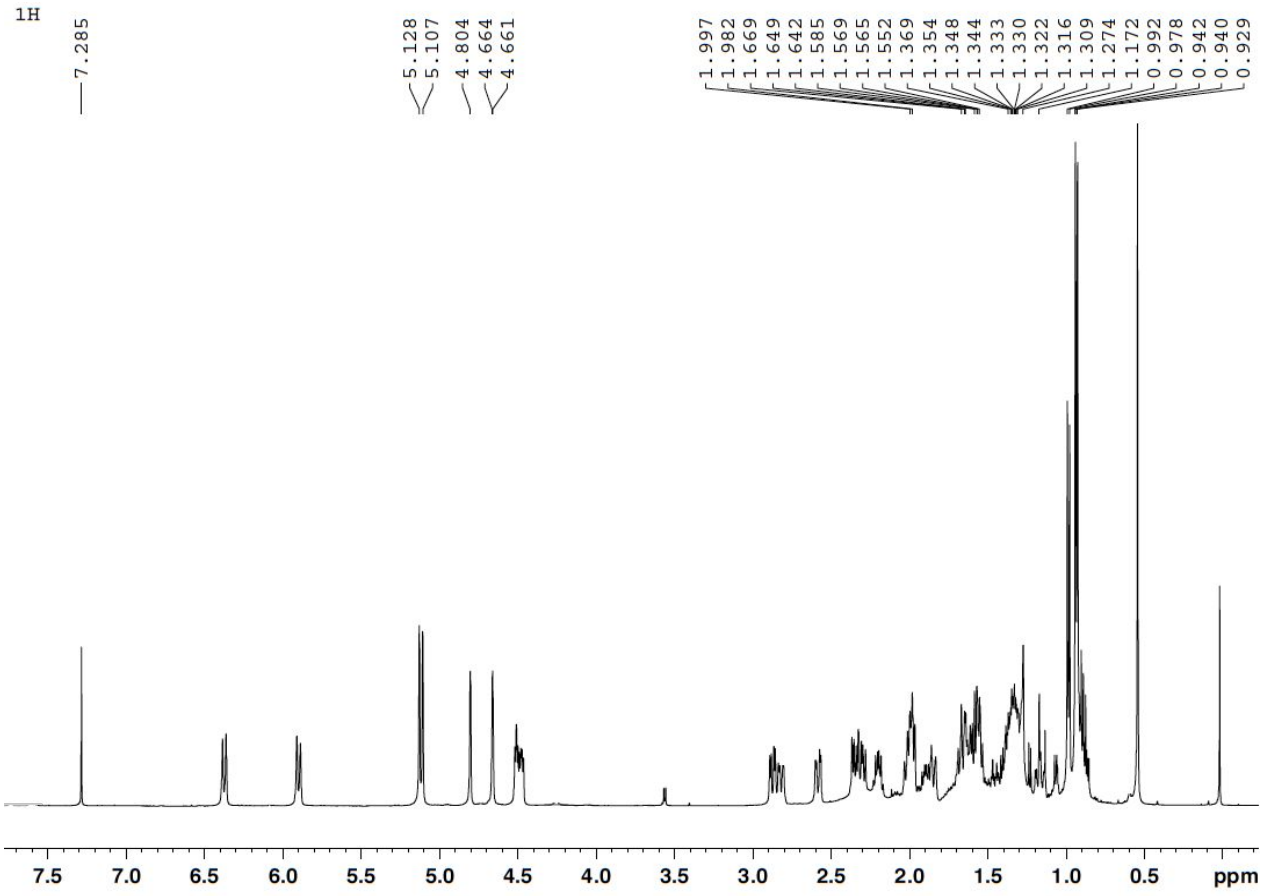


Figure 3S. ^{1}H NMR spectrum of the vitamin D analogue **8**

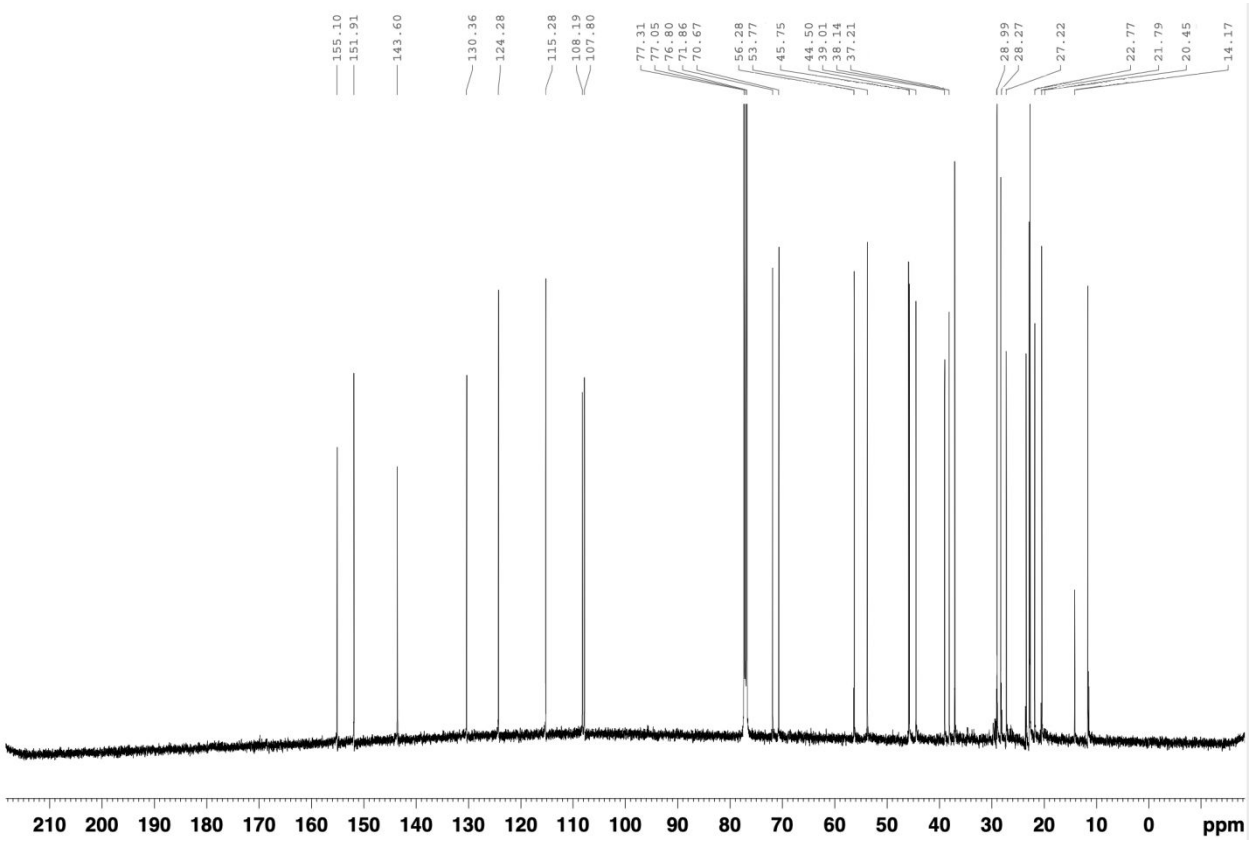


Figure 4S. ^{13}C NMR spectrum of the vitamin D analogue **8**

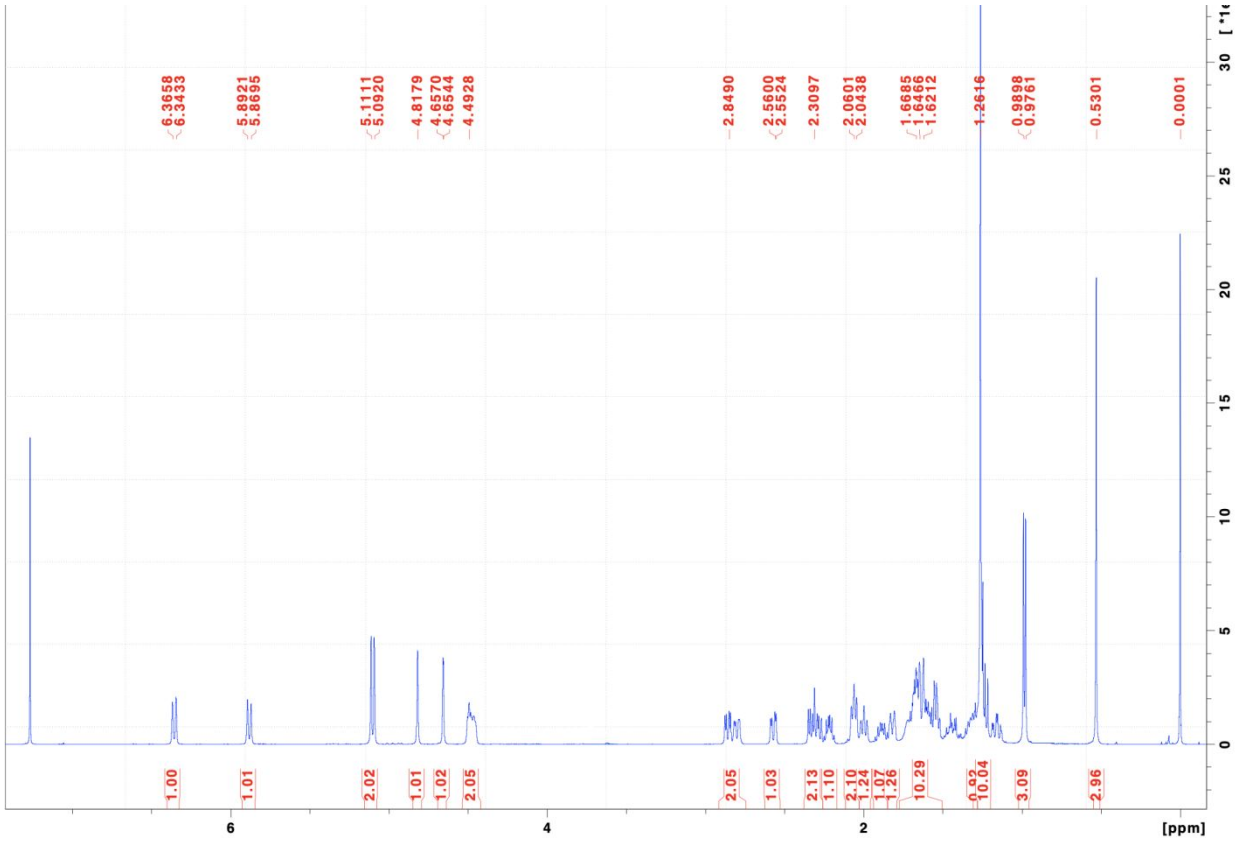
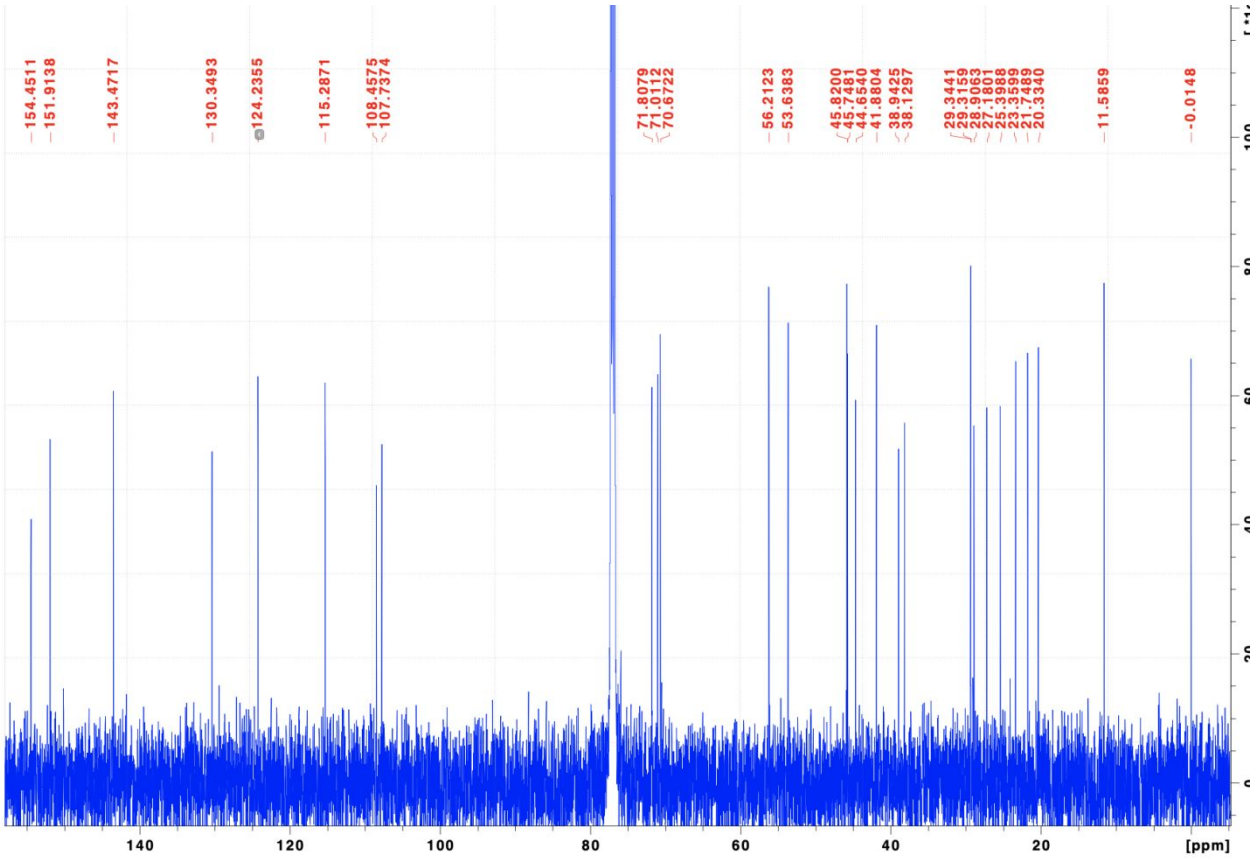


Figure 5S. ^{1}H NMR spectrum of the vitamin D analogue **9**



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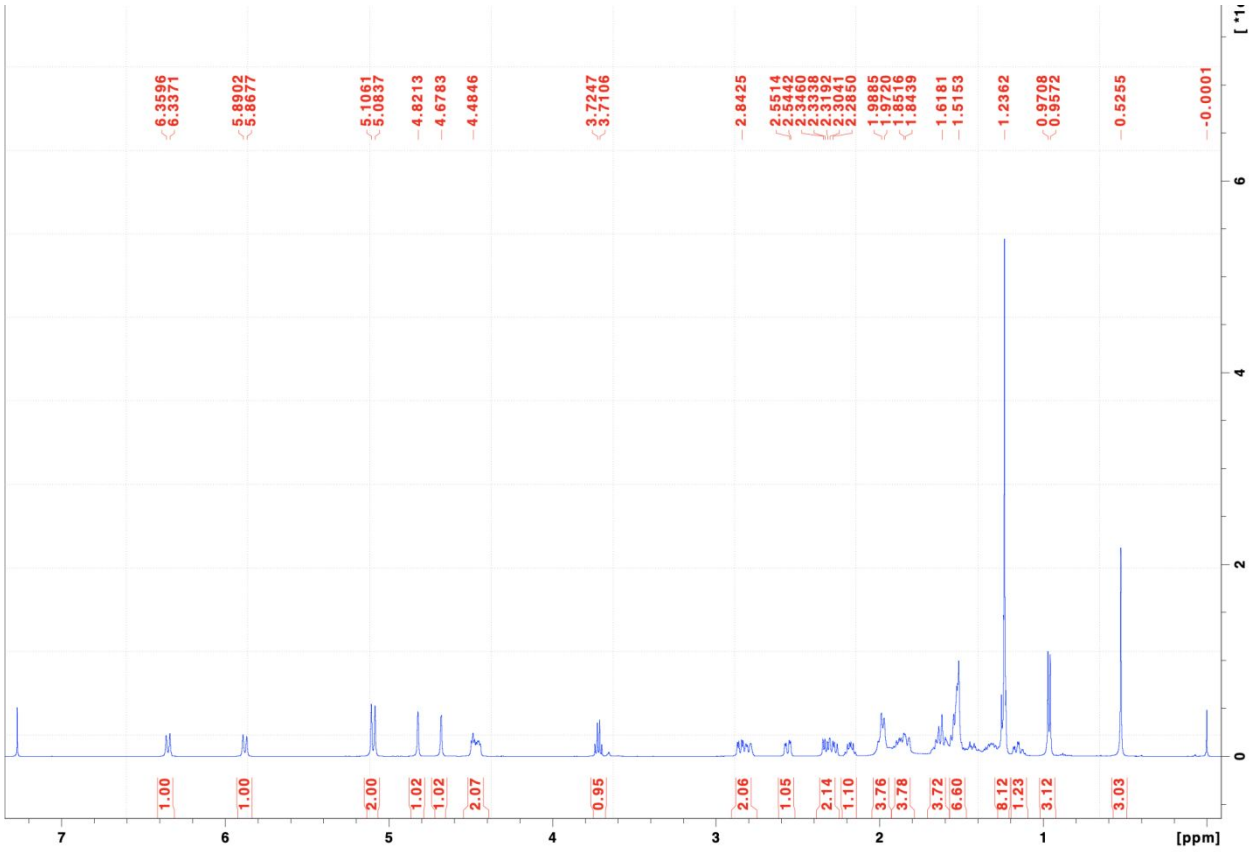
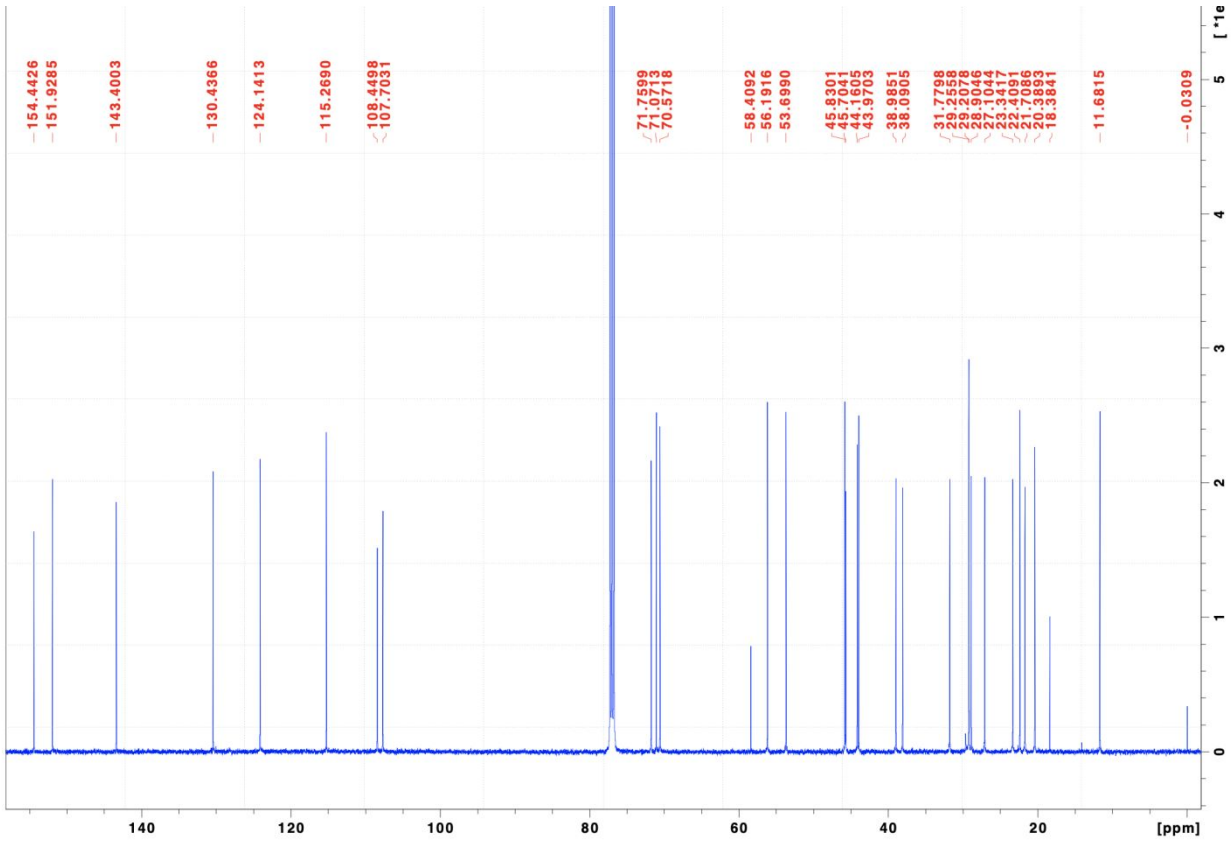


Figure 7S. ^{1}H NMR spectrum of the vitamin D analogue **10**



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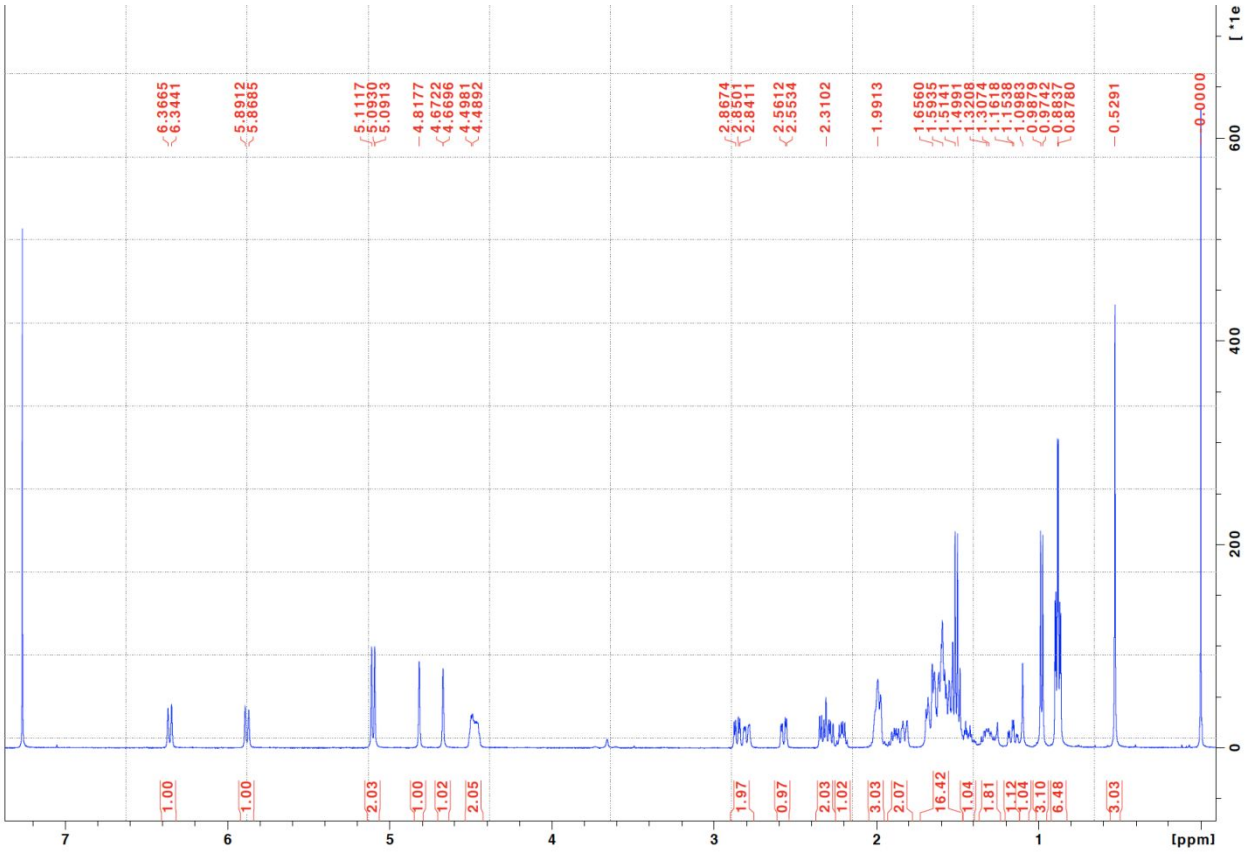


Figure 9S. ^{1}H NMR spectrum of the vitamin D analogue **11**

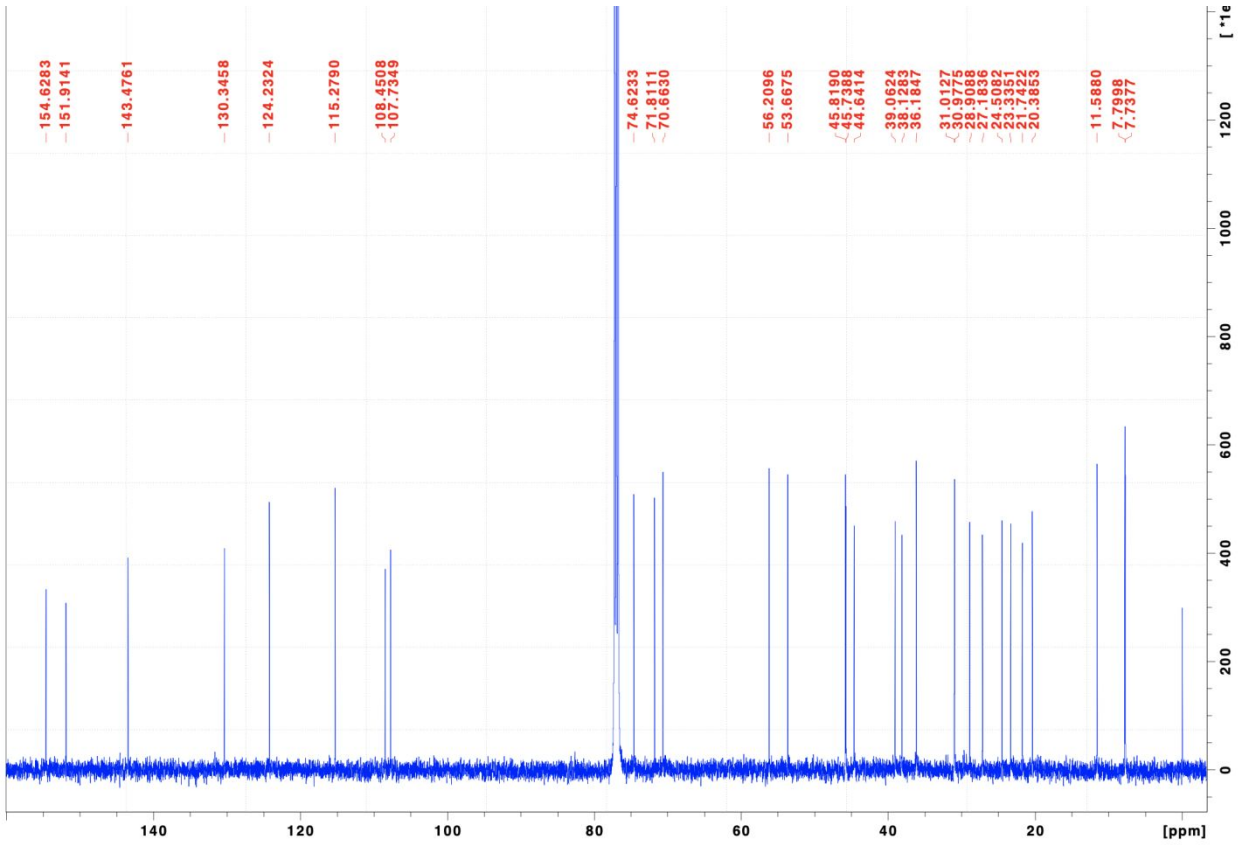


Figure 10S. ^{13}C NMR spectrum of the vitamin D analogue **11**

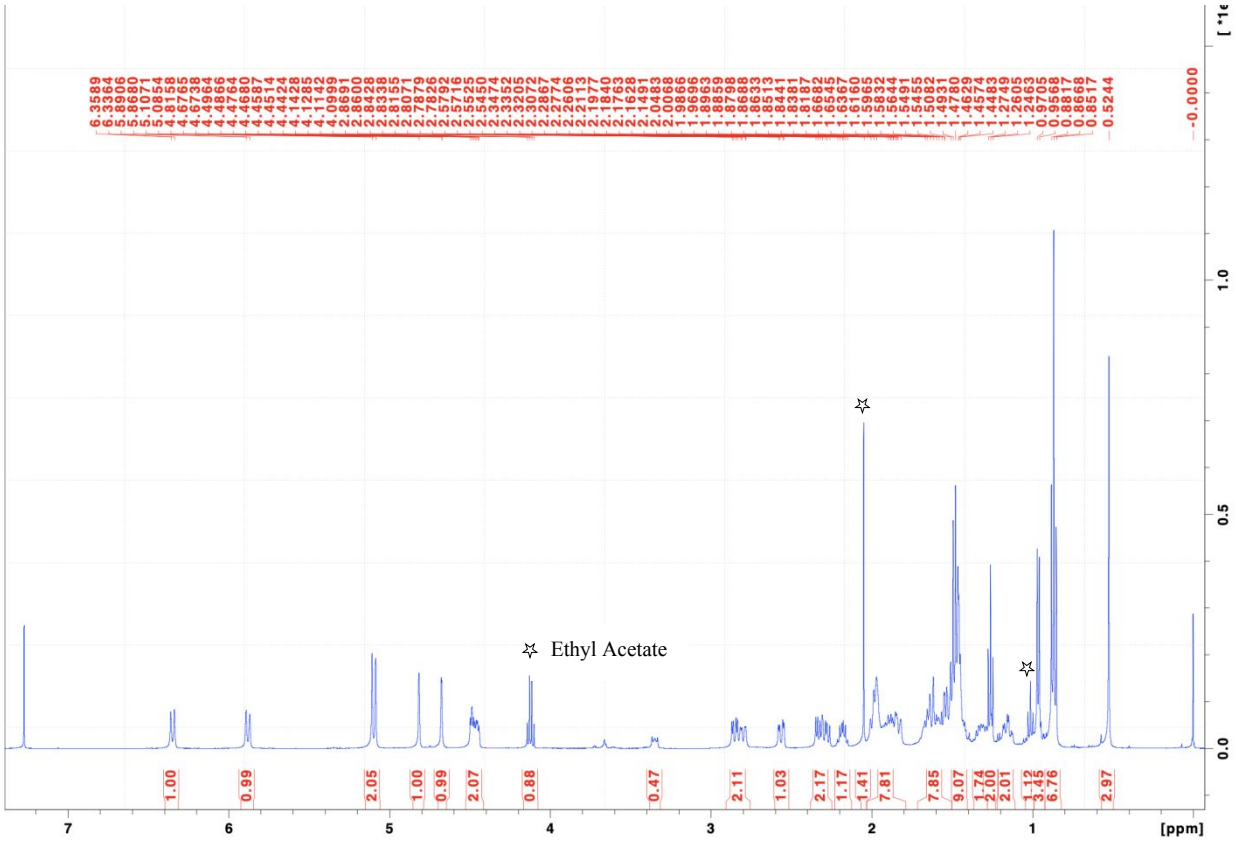
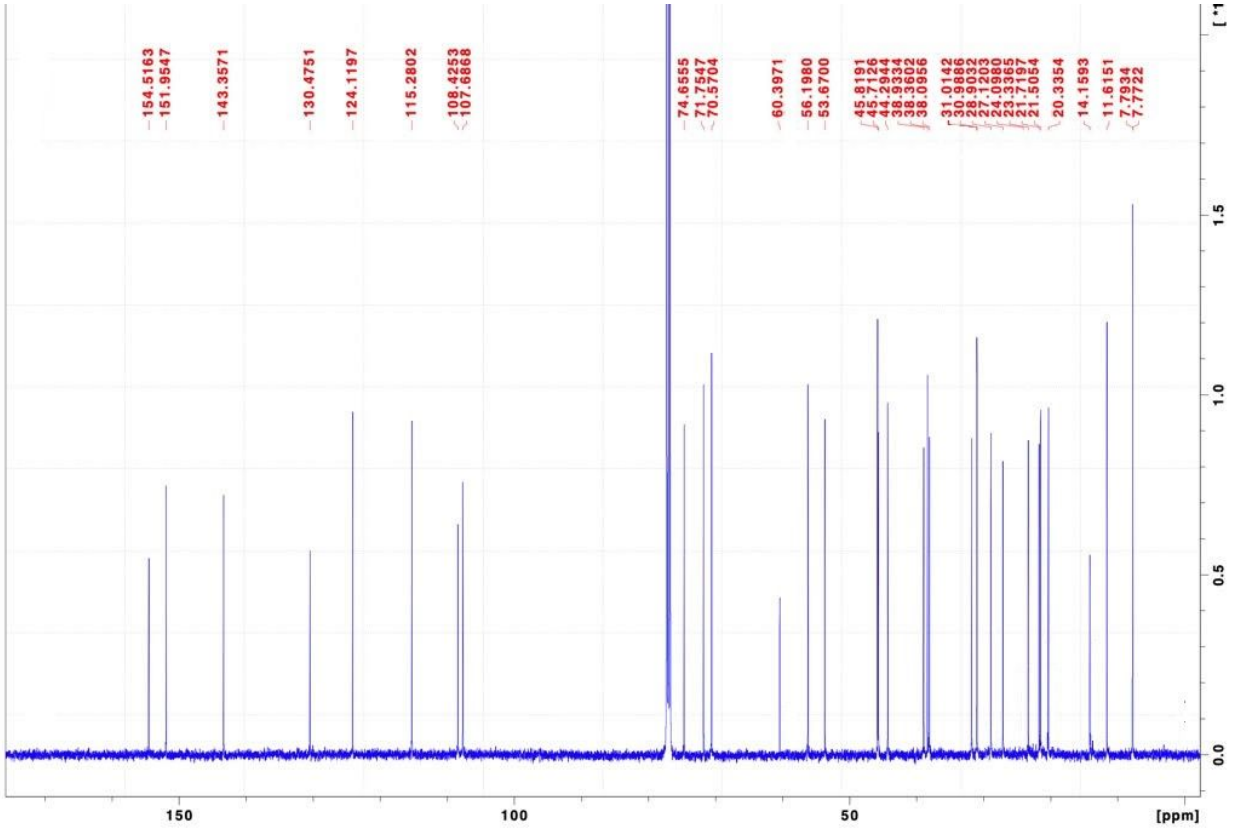


Figure 11S. [1]H NMR spectrum of the vitamin D analogue **12**



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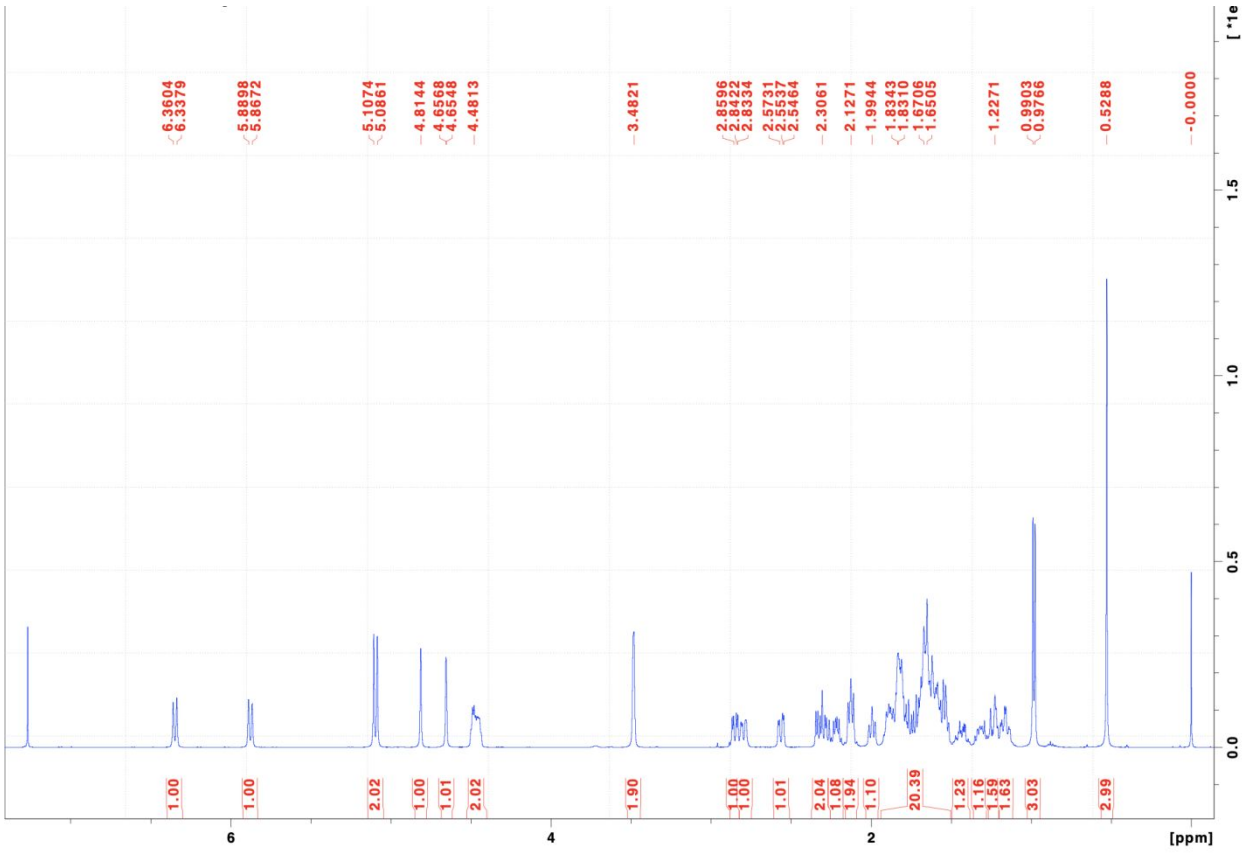


Figure 13S. ^{1}H NMR spectrum of the vitamin D analogue **13**

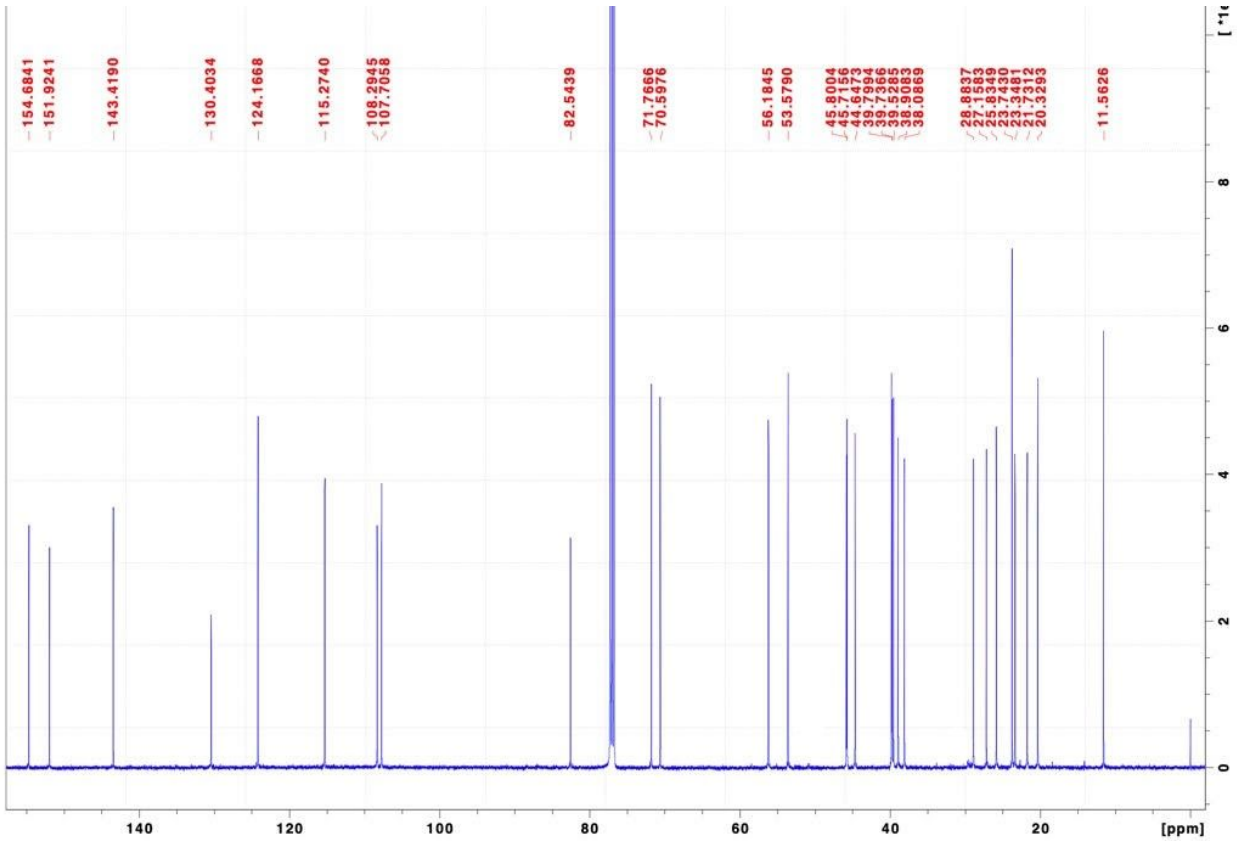
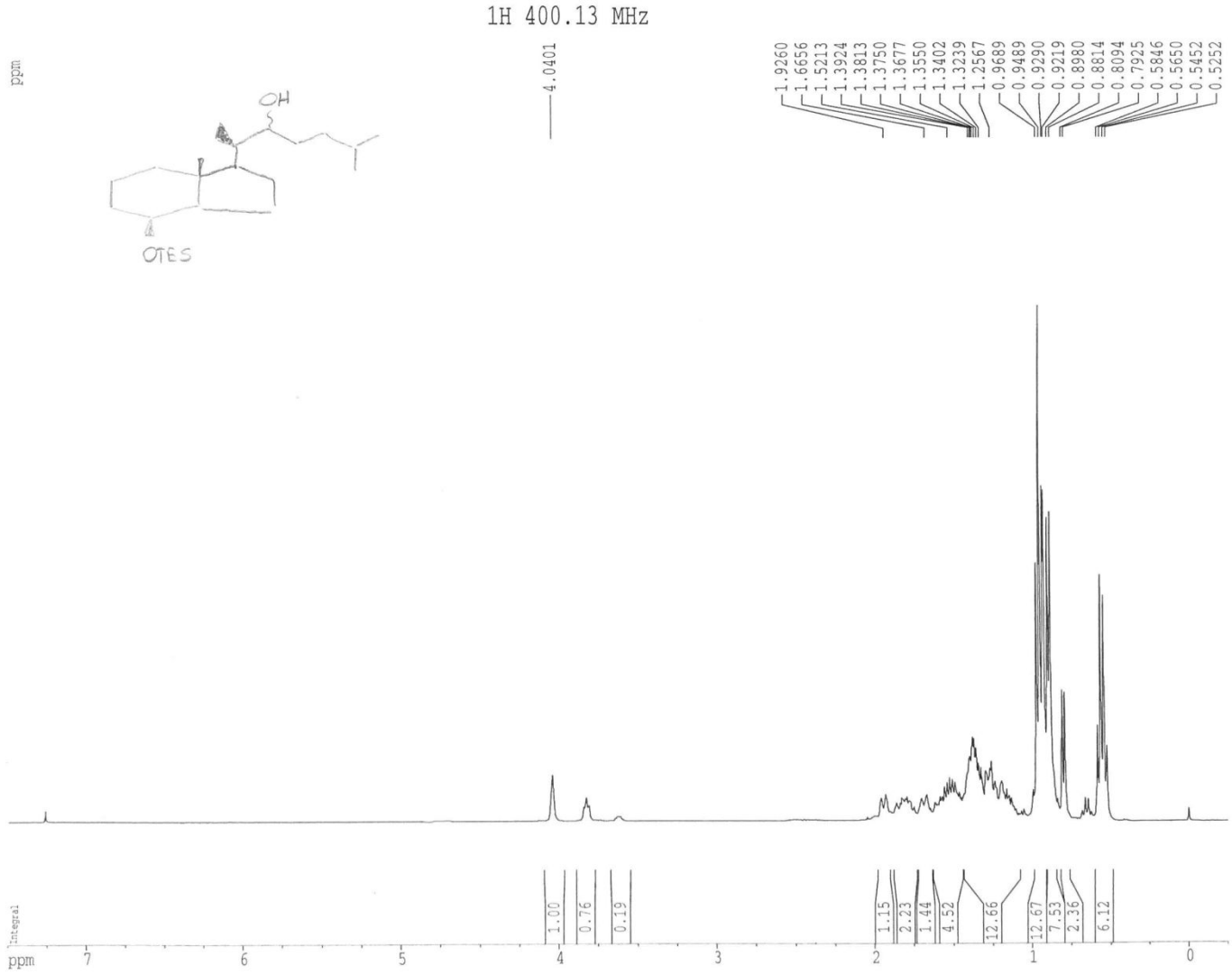


Figure 14S. ^{13}C NMR spectrum of the vitamin D analogue **13**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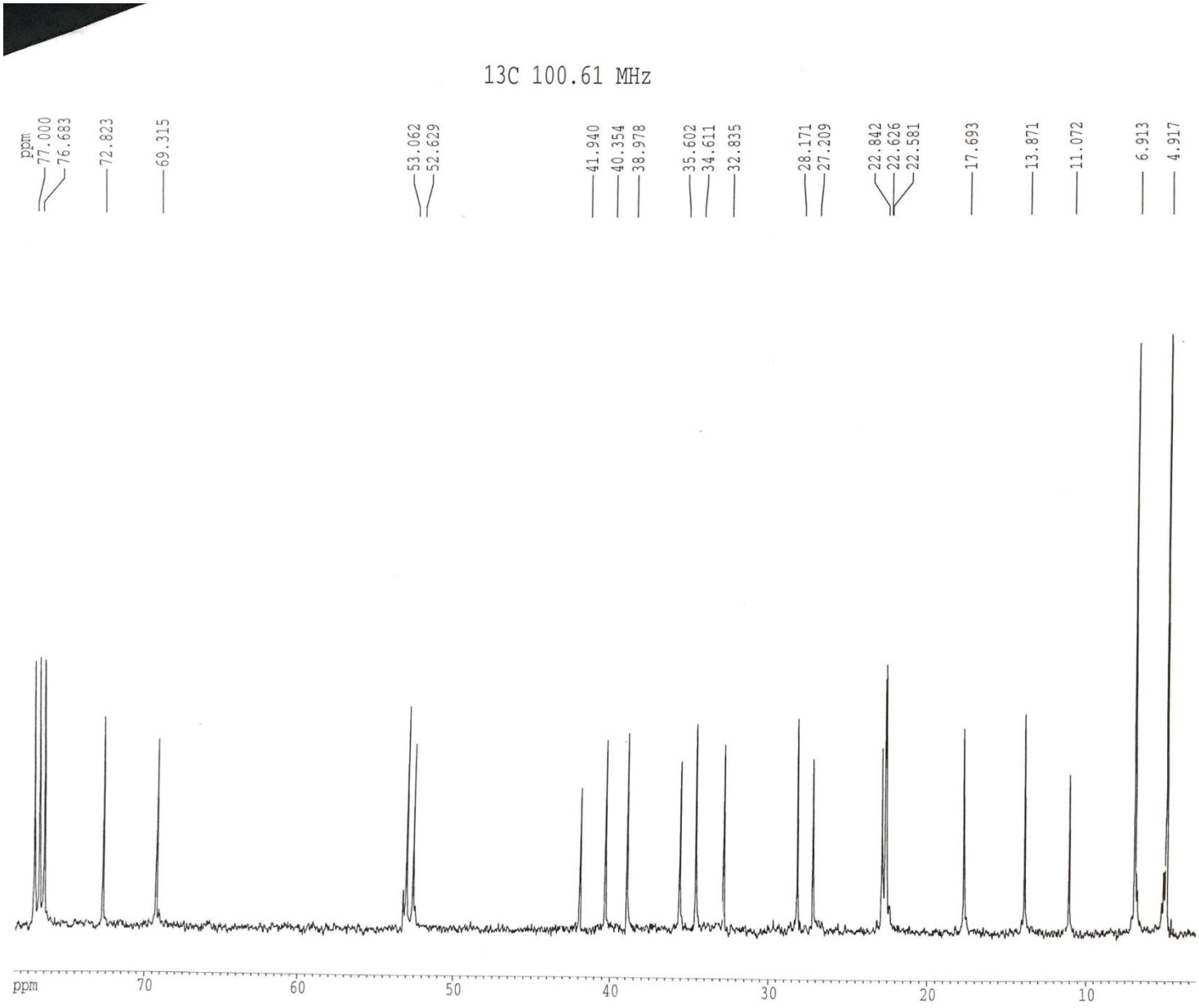
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20
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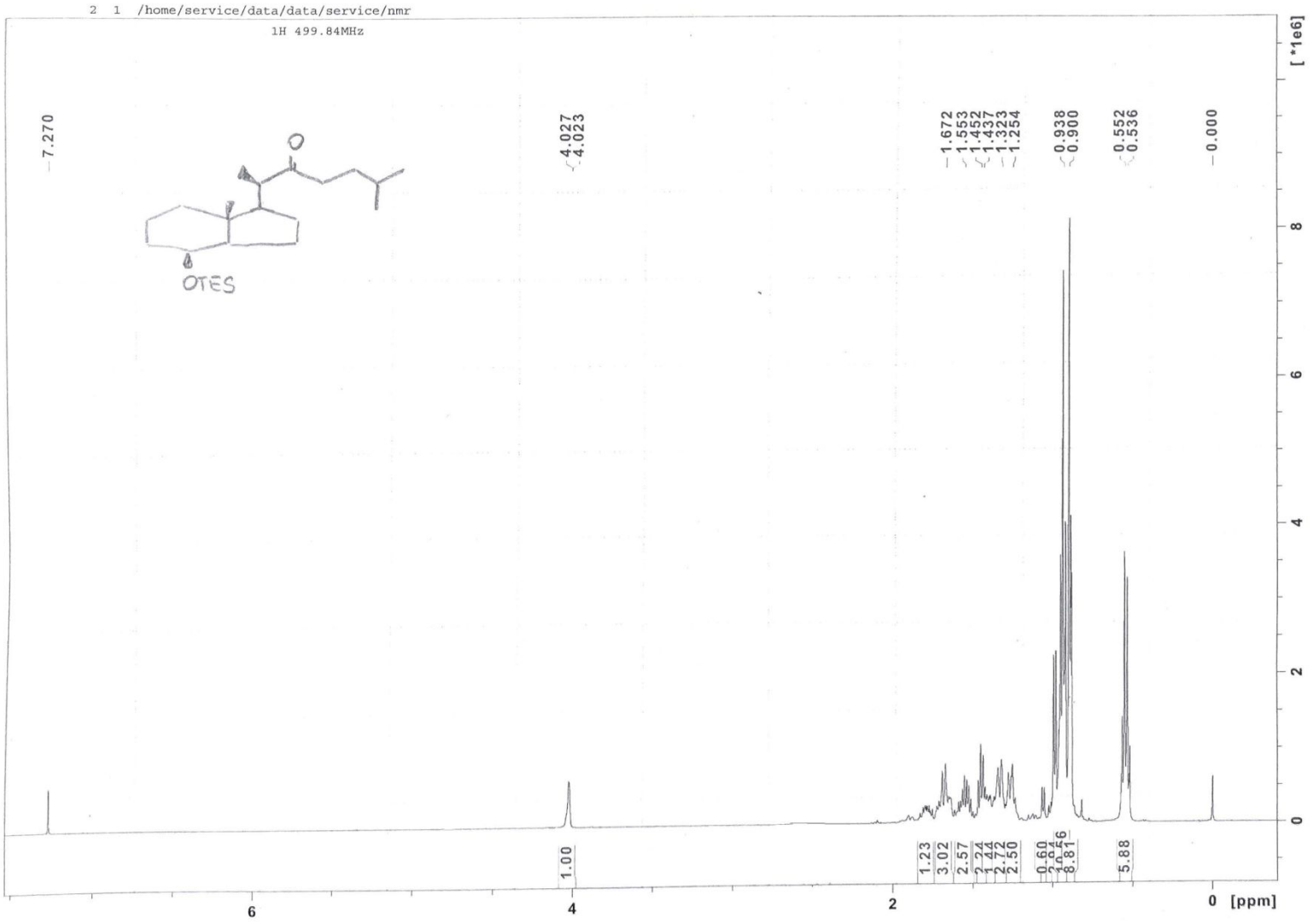
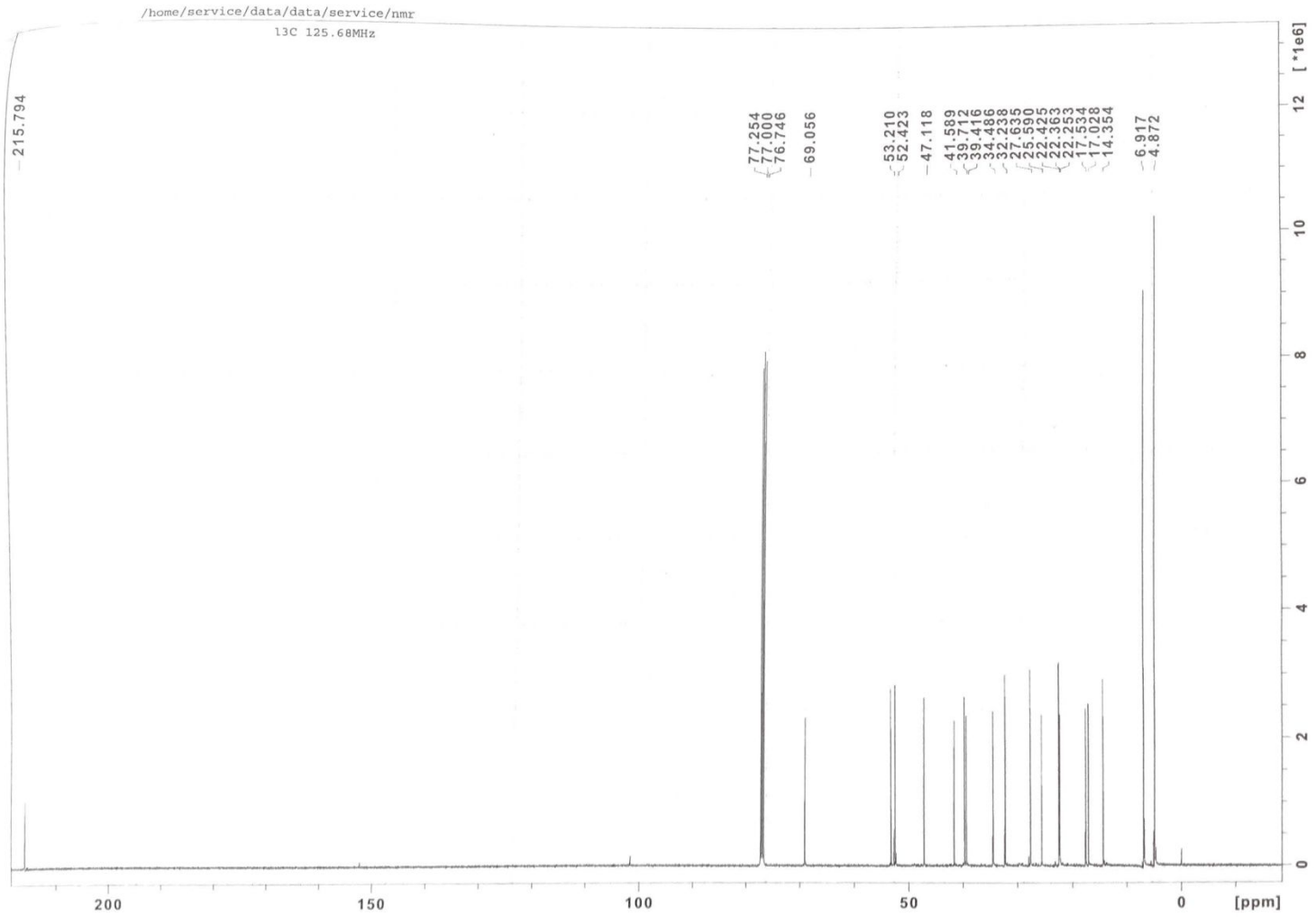
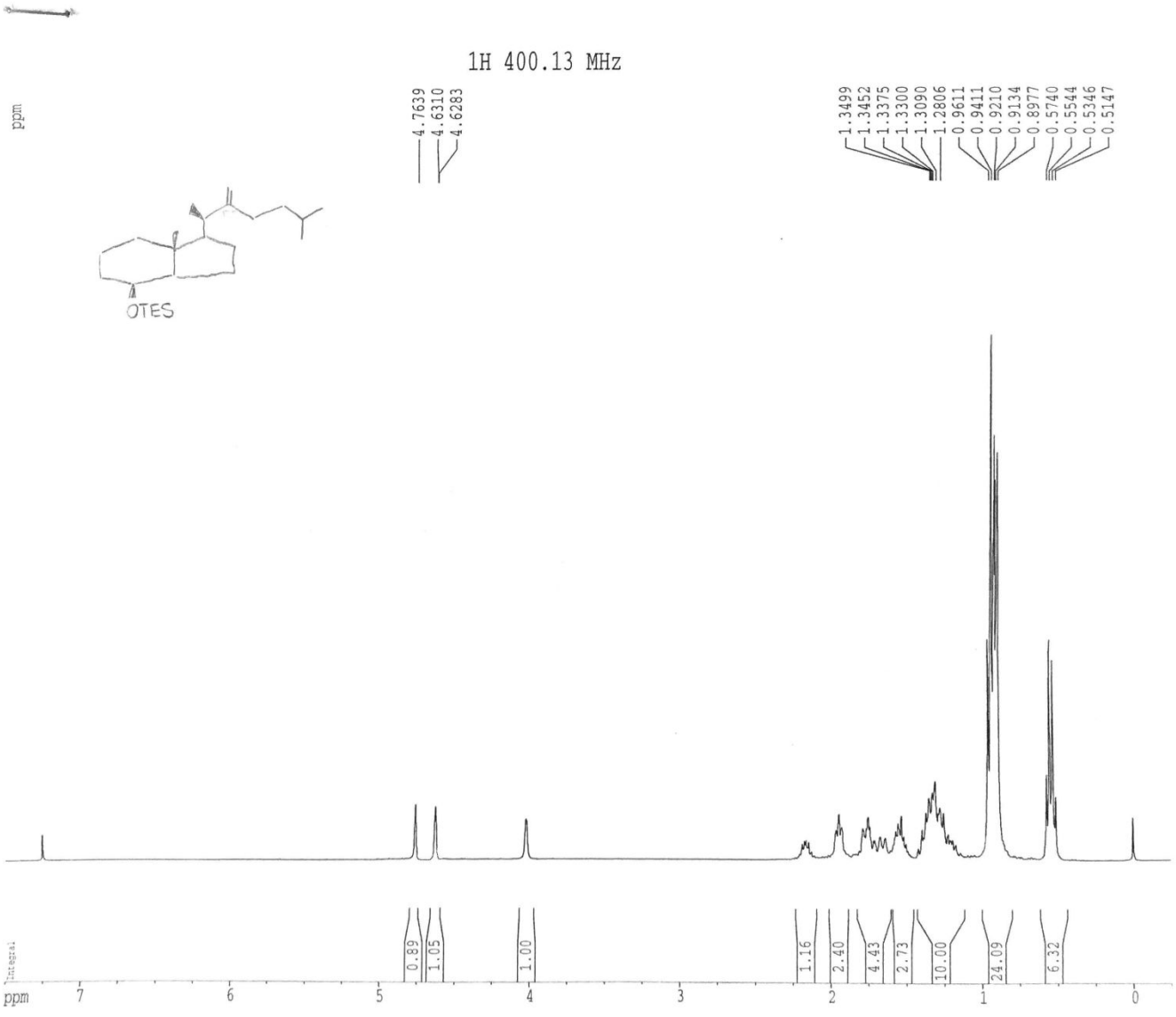
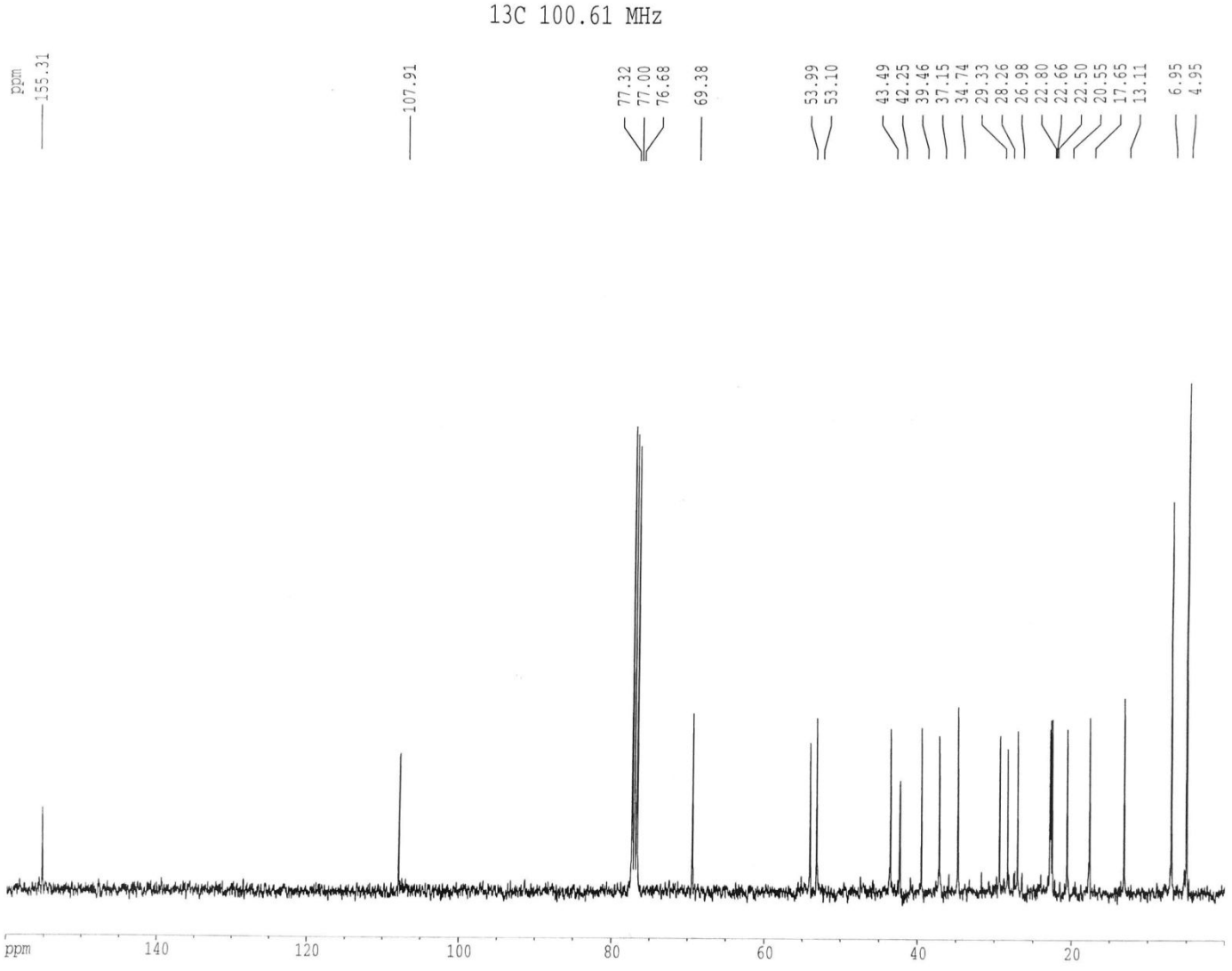
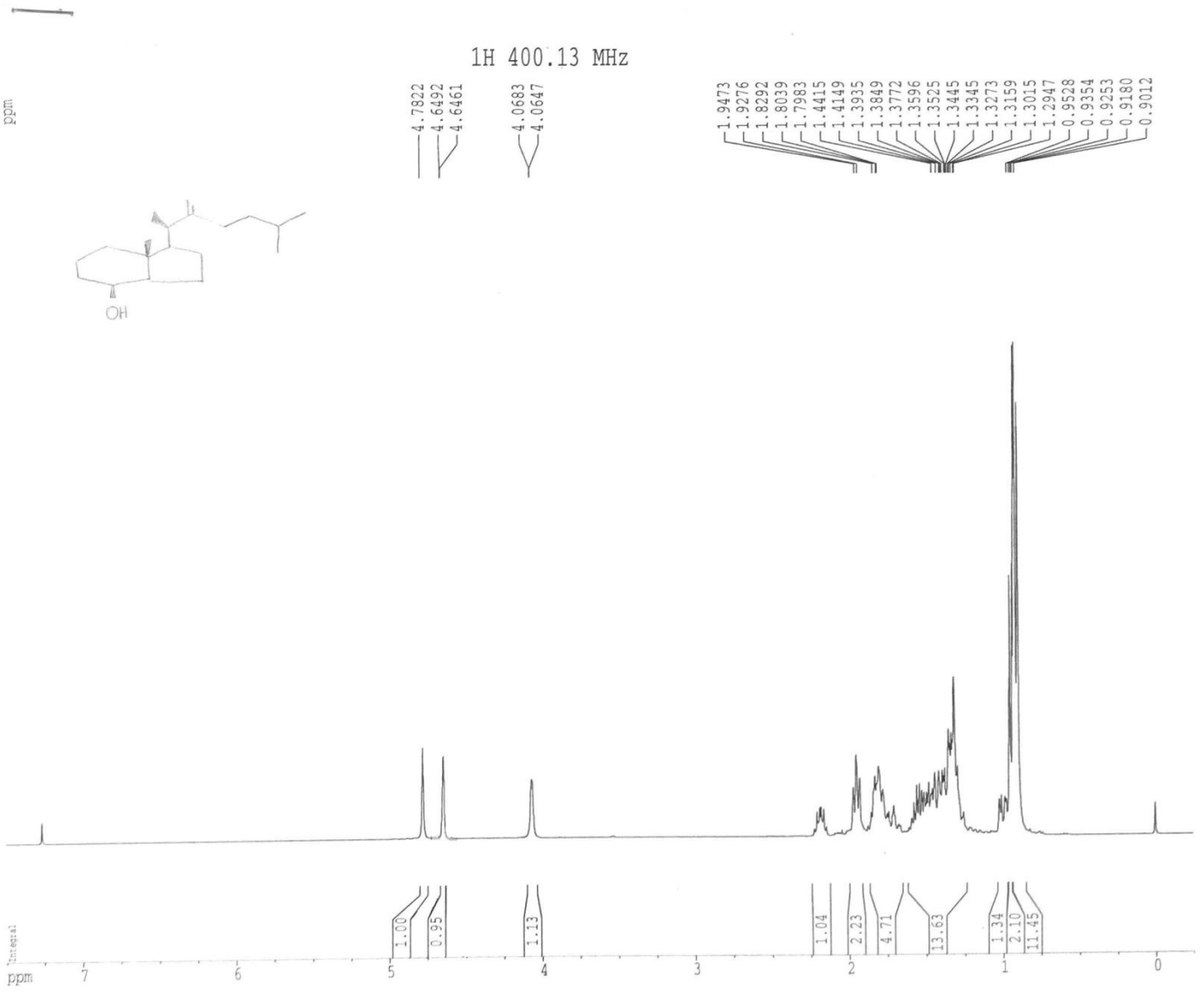
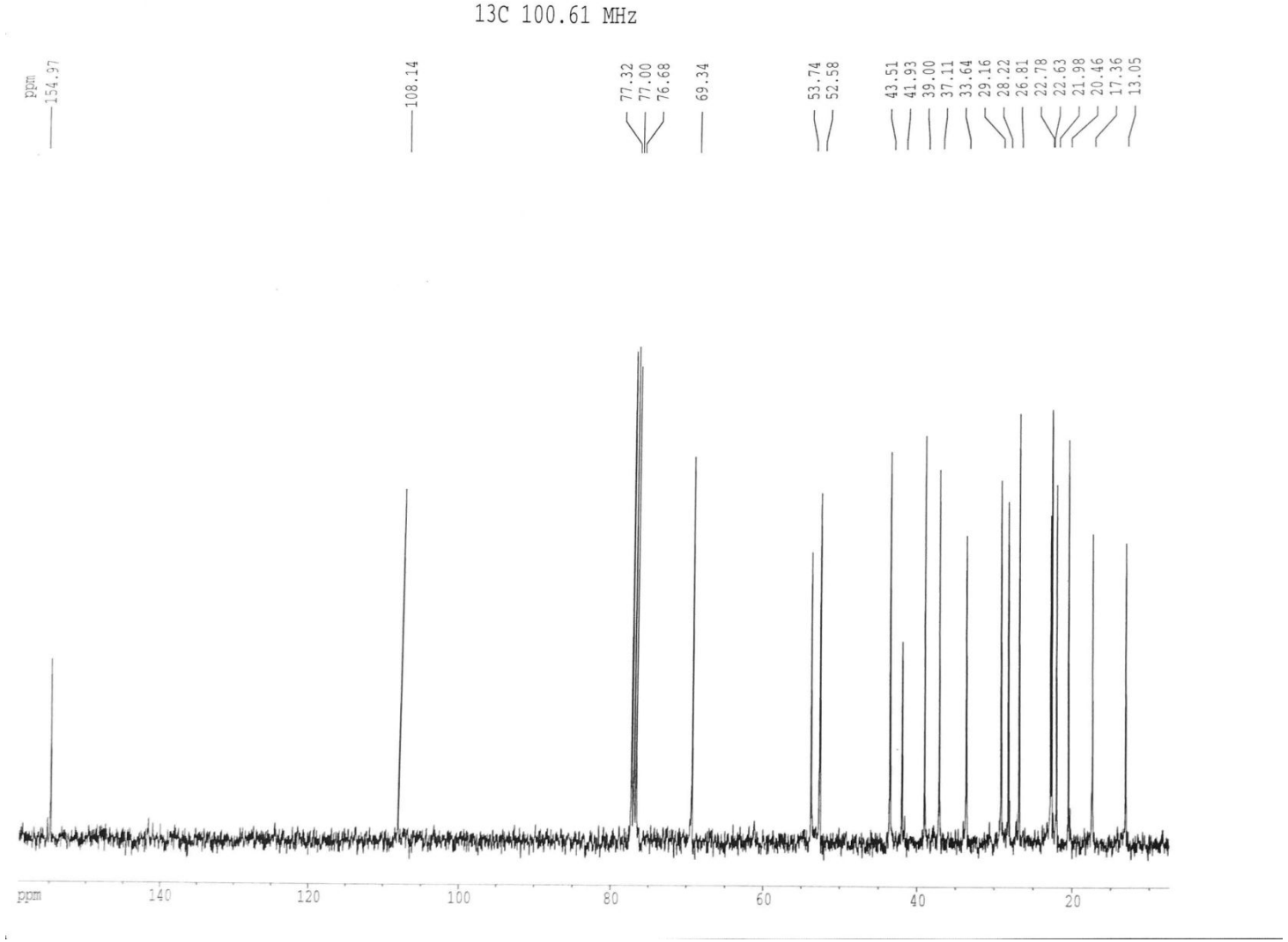

2 1 /home/service/data/data/service/nmr
1H 499.84MHz
7.270
4.027
4.023
1.672
1.553
1.452
1.437
1.323
1.254
0.938
0.900
0.552
0.536
0.000
OTES
1.00
1.23
3.02
2.57
2.24
1.44
2.72
2.50
0.60
5.88
[*1e6]
[ppm]

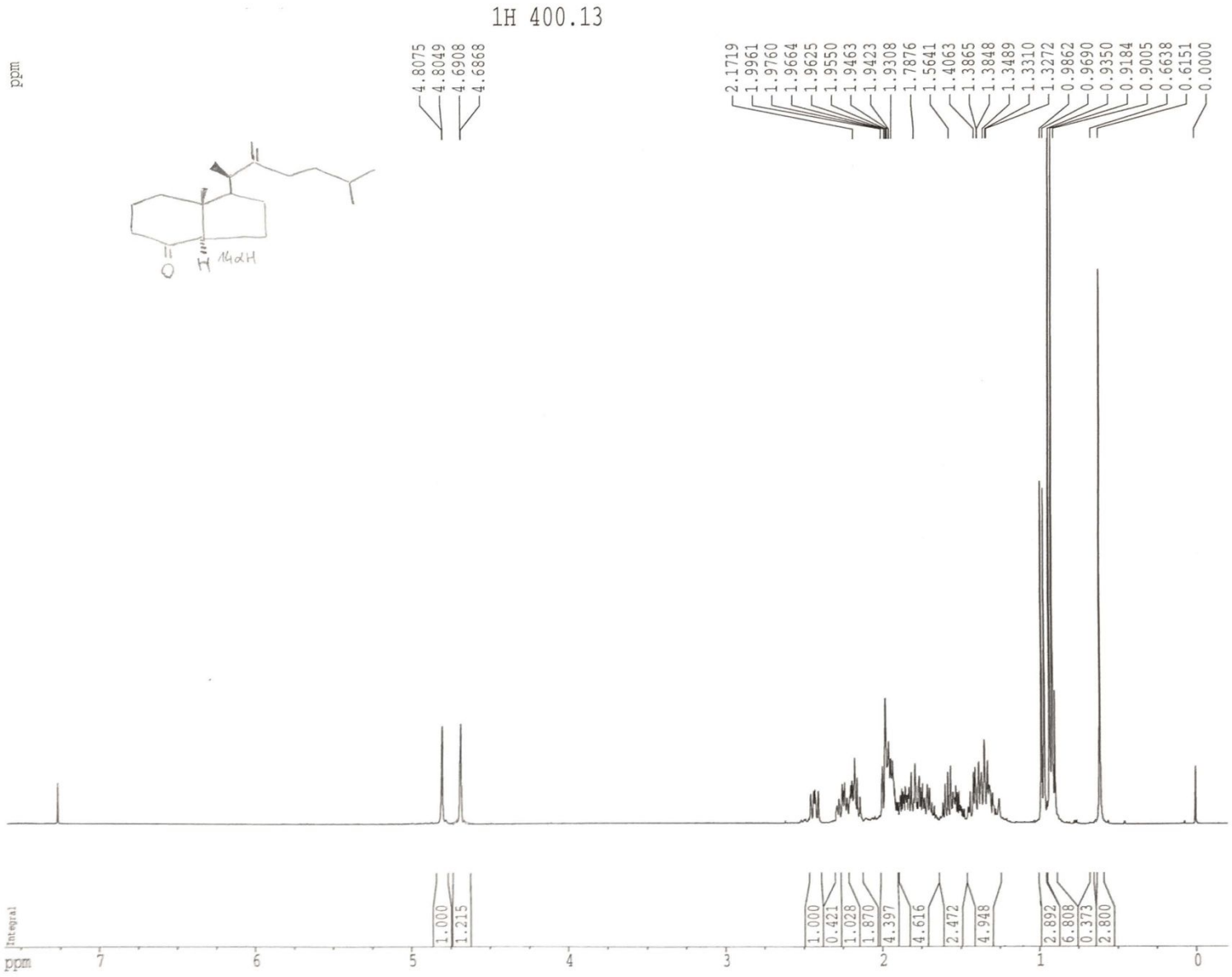

/home/service/data/data/service/nmr
13C 125.68MHz
215.794
77.254
77.000
76.746
69.056
53.210
52.423
47.118
41.589
39.712
39.416
34.486
32.238
27.635
25.590
22.425
22.363
22.253
17.534
17.028
14.354
6.917
4.872
[*1e6]
12
10
8
6
4
2
0
200
150
100
50
0
[ppm]

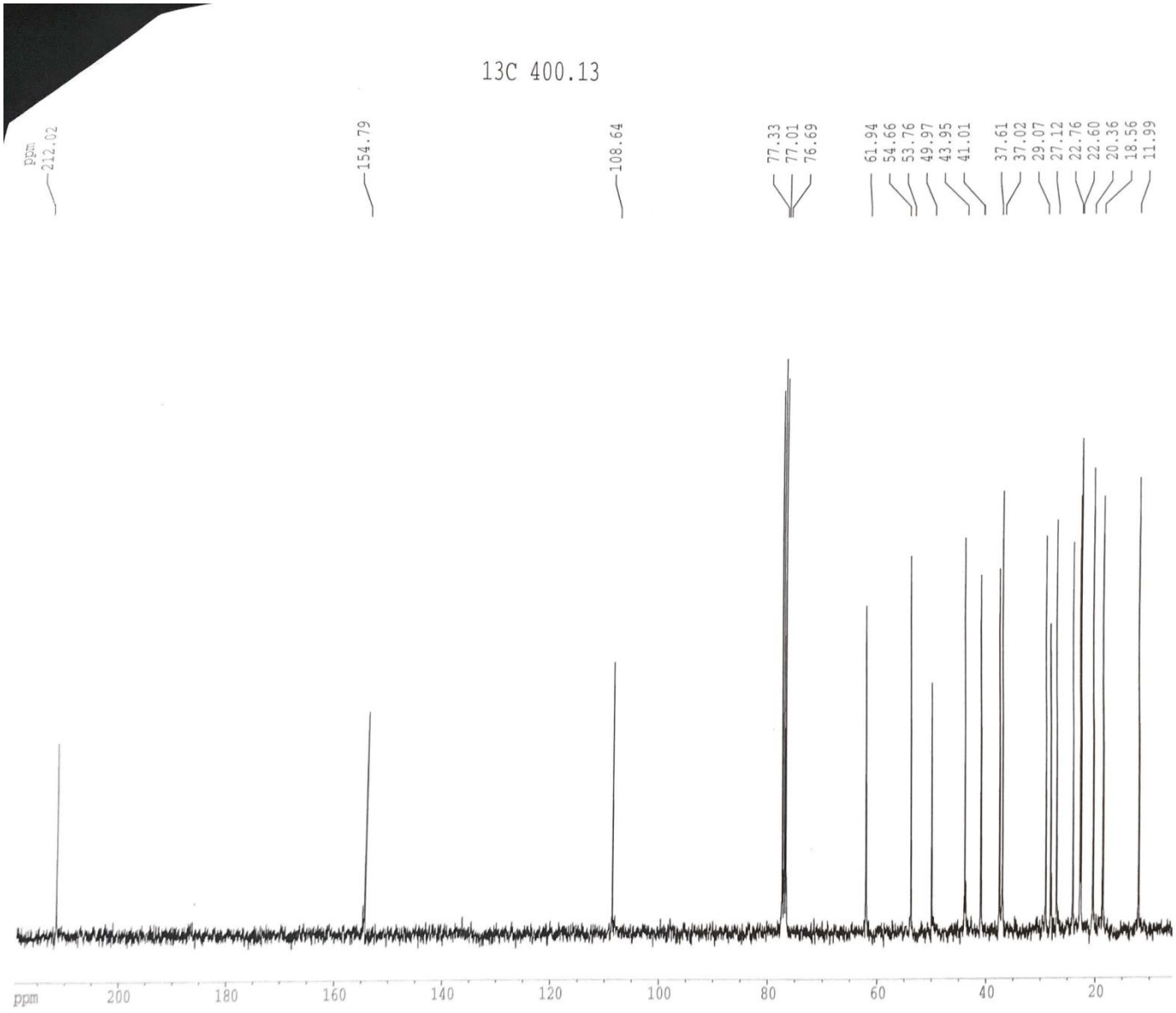

1H 400.13 MHz
ppm
4.7639
4.6310
4.6283
1.3499
1.3452
1.3375
1.3300
1.3090
1.2806
0.9611
0.9411
0.9210
0.9134
0.8977
0.5740
0.5544
0.5346
0.5147
OTES
Integral
0.89
1.05
1.00
1.16
2.40
4.43
2.73
10.00
24.09
6.32
ppm
7
6
5
4
3
2
1
0

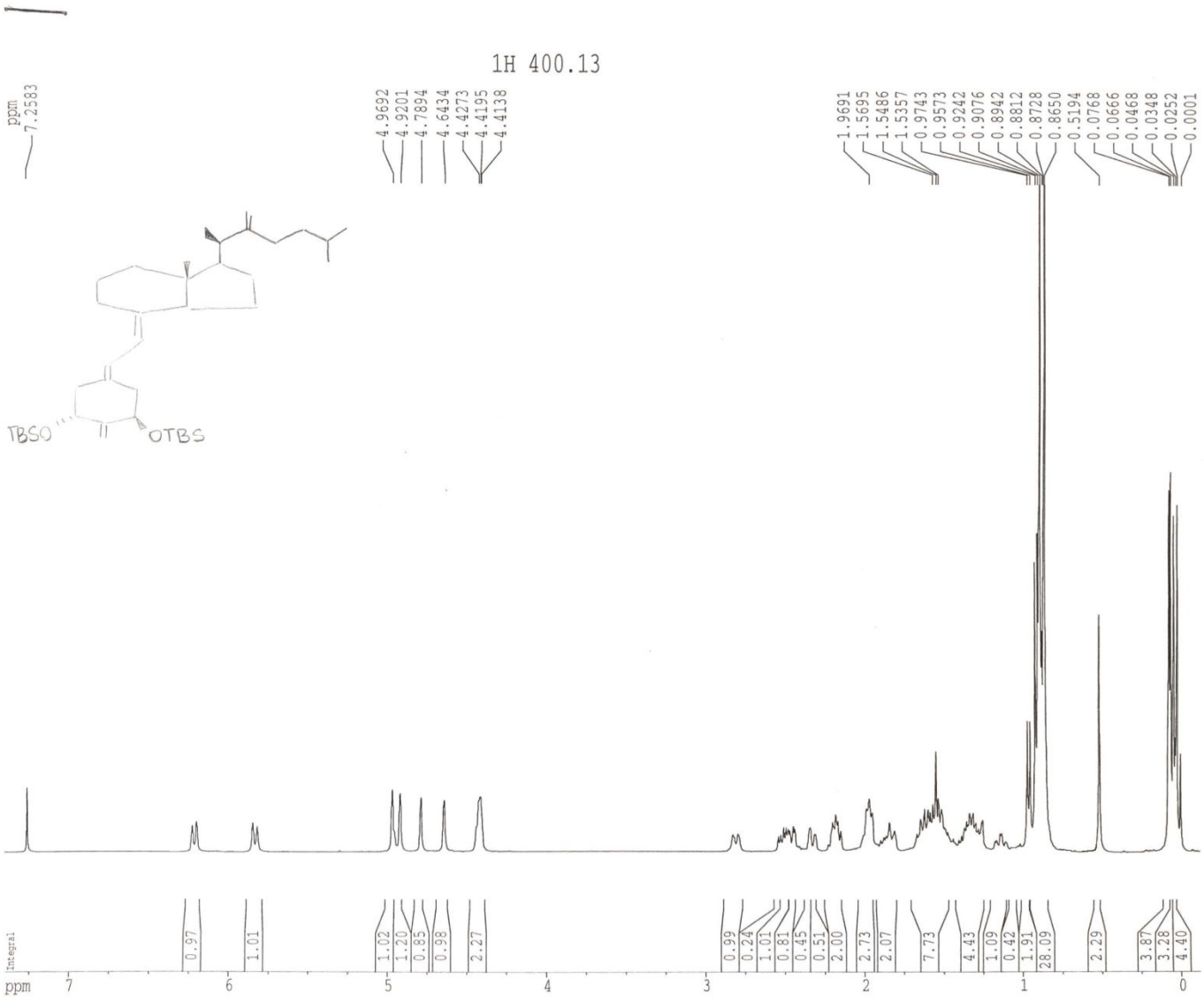

13C 100.61 MHz
ppm
155.31
107.91
77.32
77.00
76.68
69.38
53.99
53.10
43.49
42.25
39.46
37.15
34.74
29.33
28.26
26.98
22.80
22.66
22.50
20.55
17.65
13.11
6.95
4.95
ppm
140
120
100
80
60
40
20

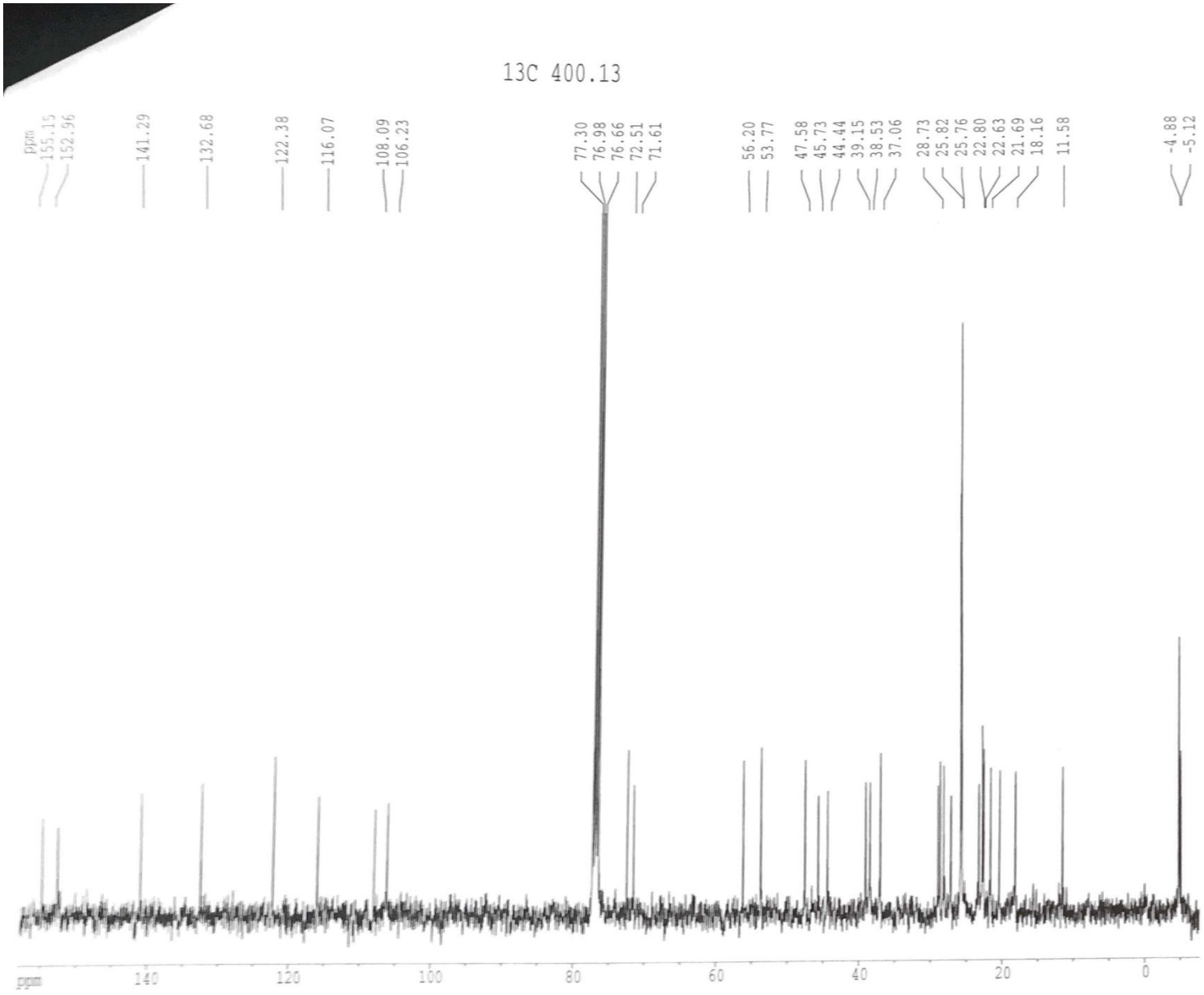

1H 400.13 MHz
ppm
4.7822
4.6492
4.6461
4.0683
4.0647
1.9473
1.9276
1.8292
1.8039
1.7983
1.4415
1.4149
1.3935
1.3849
1.3772
1.3596
1.3525
1.3445
1.3345
1.3273
1.3159
1.3015
1.2947
0.9528
0.9354
0.9253
0.9180
0.9012
OH
Integral
1.00
0.95
1.13
1.04
2.23
4.71
13.63
1.34
2.10
11.45
ppm
7
6
5
4
3
2
1
0

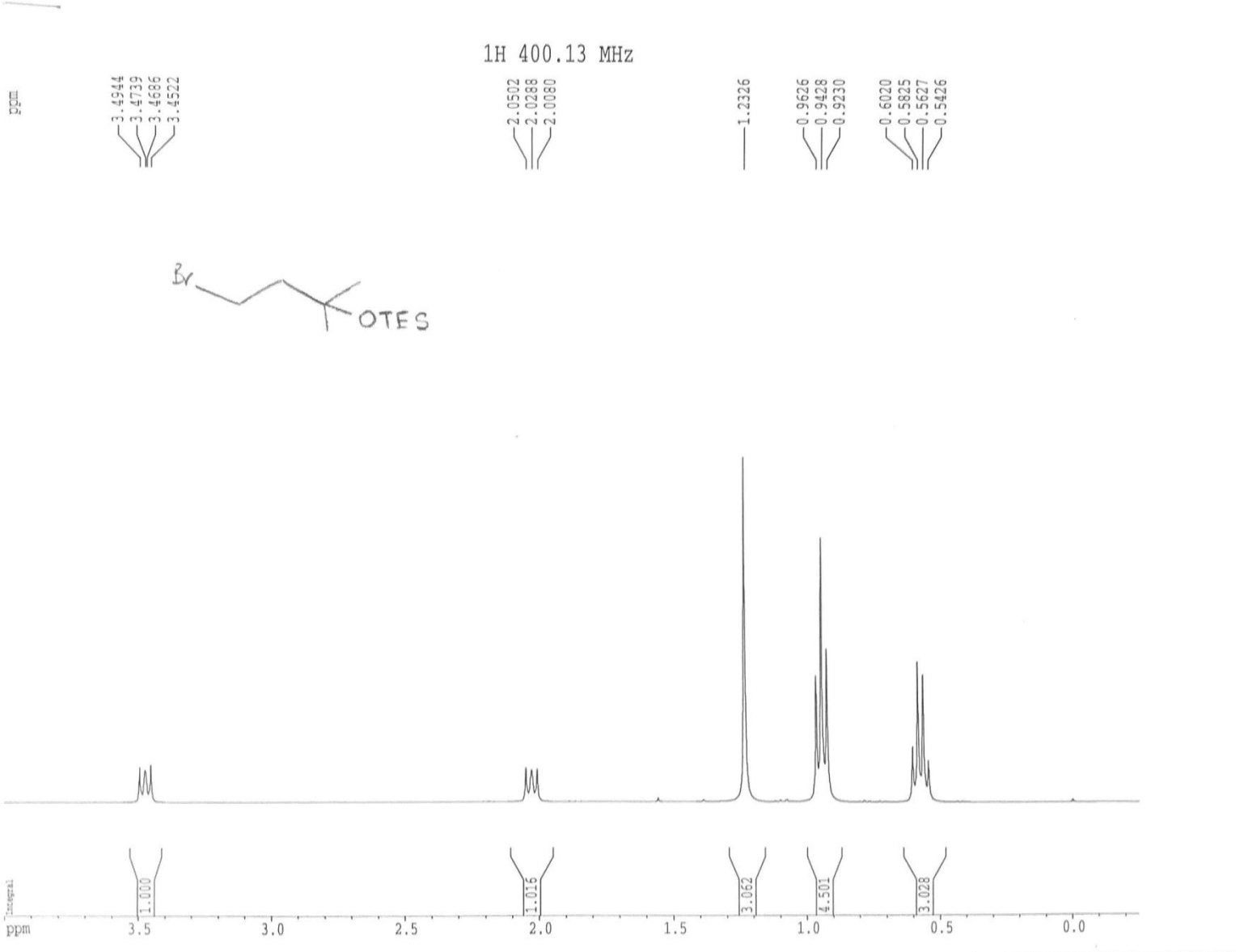

13C 100.61 MHz
ppm
154.97
108.14
77.32
77.00
76.68
69.34
53.74
52.58
43.51
41.93
39.00
37.11
33.64
29.16
28.22
26.81
22.78
22.63
21.98
20.46
17.36
13.05
ppm
140
120
100
80
60
40
20

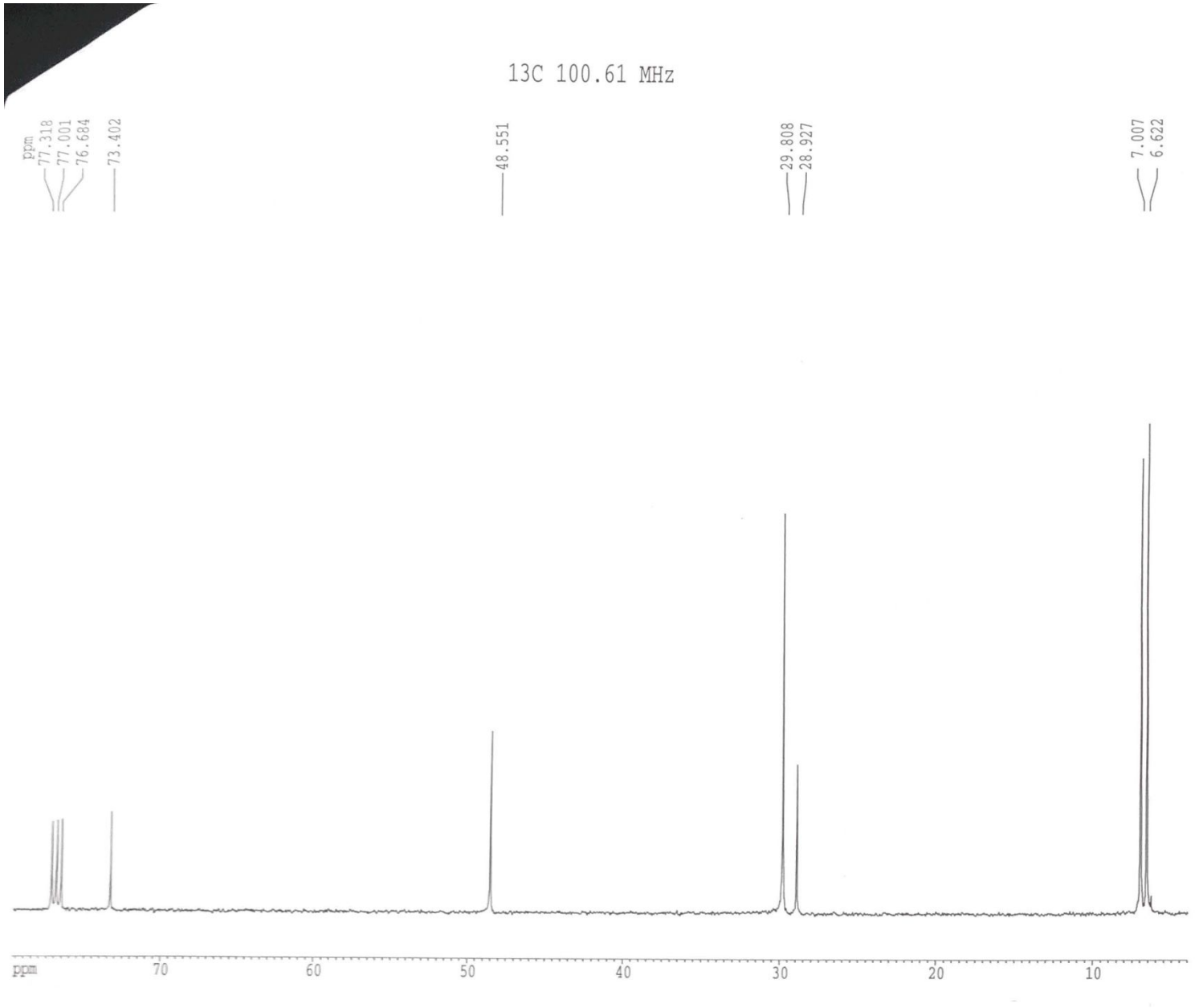

1H 400.13
ppm
4.8075
4.8049
4.6908
4.6868
2.1719
1.9961
1.9760
1.9664
1.9625
1.9550
1.9463
1.9423
1.9308
1.7876
1.5641
1.4063
1.3865
1.3848
1.3489
1.3310
1.3272
0.9862
0.9690
0.9350
0.9184
0.9005
0.6638
0.6151
0.0000
Integral
1.000
1.215
1.000
0.421
1.028
1.870
4.397
4.616
2.472
4.948
2.892
6.808
0.373
2.800
ppm
7
6
5
4
3
2
1
0

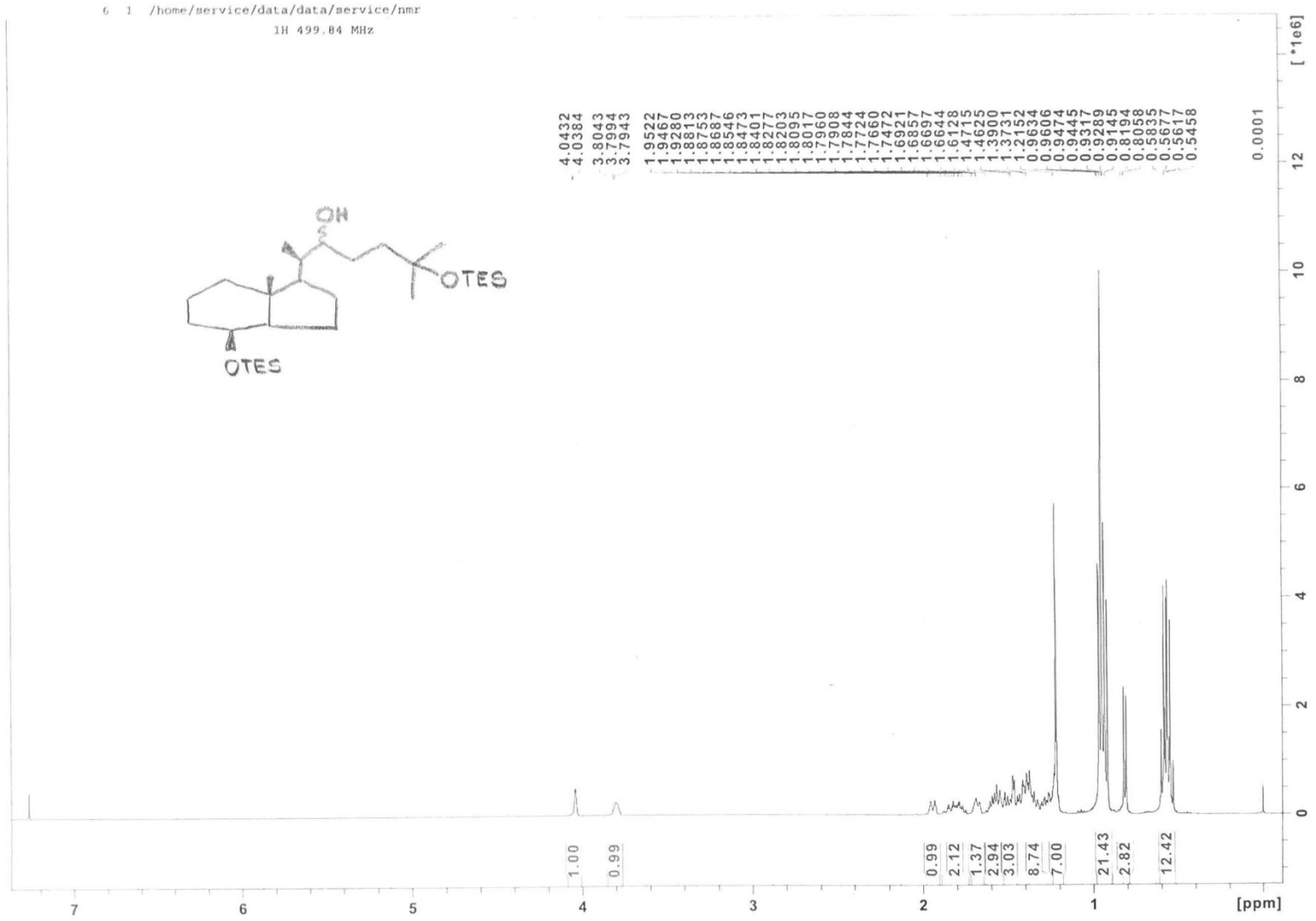

13C 400.13
ppm
212.02
154.79
108.64
77.33
77.01
76.69
61.94
54.66
53.76
49.97
43.95
41.01
37.61
37.02
29.07
27.12
22.76
22.60
20.36
18.56
11.99
ppm
200
180
160
140
120
100
80
60
40
20

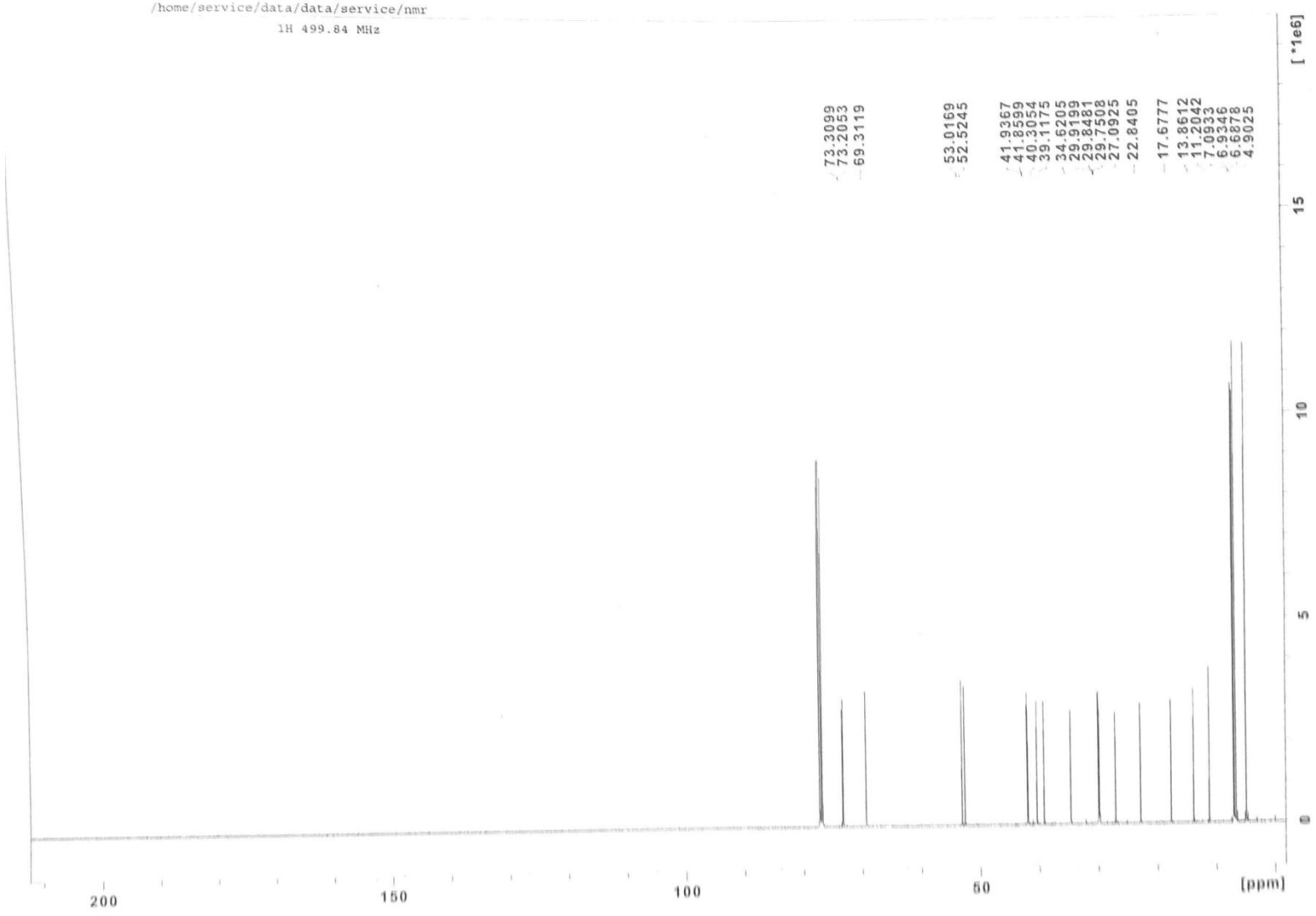

1H 400.13
ppm
7.2583
4.9692
4.9201
4.7894
4.6434
4.4273
4.4195
4.4138
1.9691
1.5695
1.5486
1.5357
0.9743
0.9573
0.9242
0.9076
0.8942
0.8812
0.8728
0.8650
0.5194
0.0768
0.0666
0.0468
0.0348
0.0252
0.0001
TBSO
OTBS
Integral
0.97
1.01
1.02
1.20
0.85
0.98
2.27
0.99
0.24
1.01
0.81
0.45
0.51
2.00
2.73
2.07
7.73
4.43
1.09
0.42
1.91
28.09
2.29
3.87
3.28
4.40
ppm
7
6
5
4
3
2
1
0

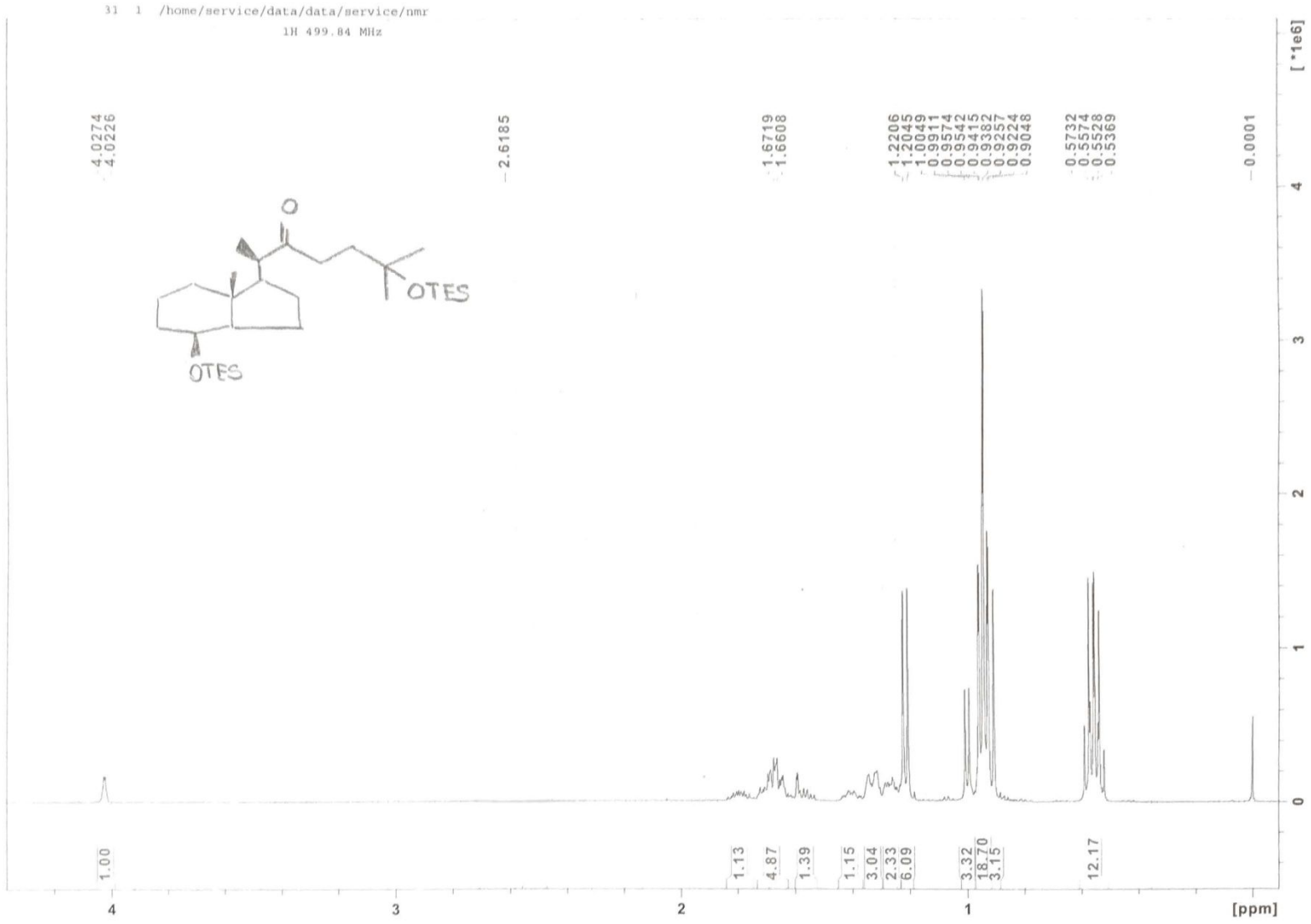

13C 400.13
ppm
155.15
152.96
141.29
132.68
122.38
116.07
108.09
106.23
77.30
76.98
76.66
72.51
71.61
56.20
53.77
47.58
45.73
44.44
39.15
38.53
37.06
28.73
25.82
25.76
22.80
22.63
21.69
18.16
11.58
-4.88
-5.12
ppm
140
120
100
80
60
40
20
0

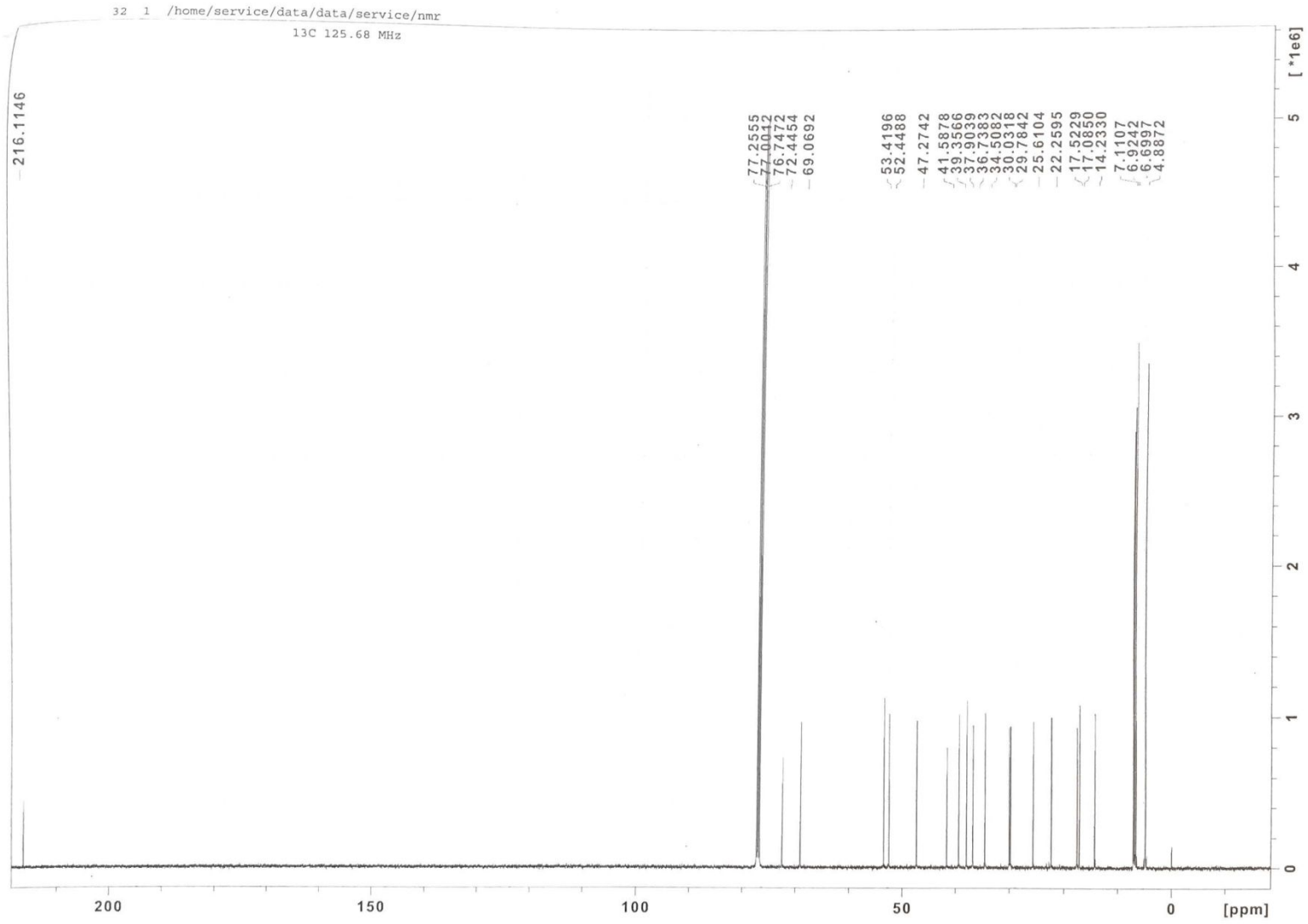

1H 400.13 MHz
ppm
3.4944
3.4739
3.4686
3.4522
2.0502
2.0288
2.0080
1.2326
0.9626
0.9428
0.9230
0.6020
0.5825
0.5627
0.5426
Br
OTES
Integral
1.000
1.016
3.062
4.501
3.028
ppm
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

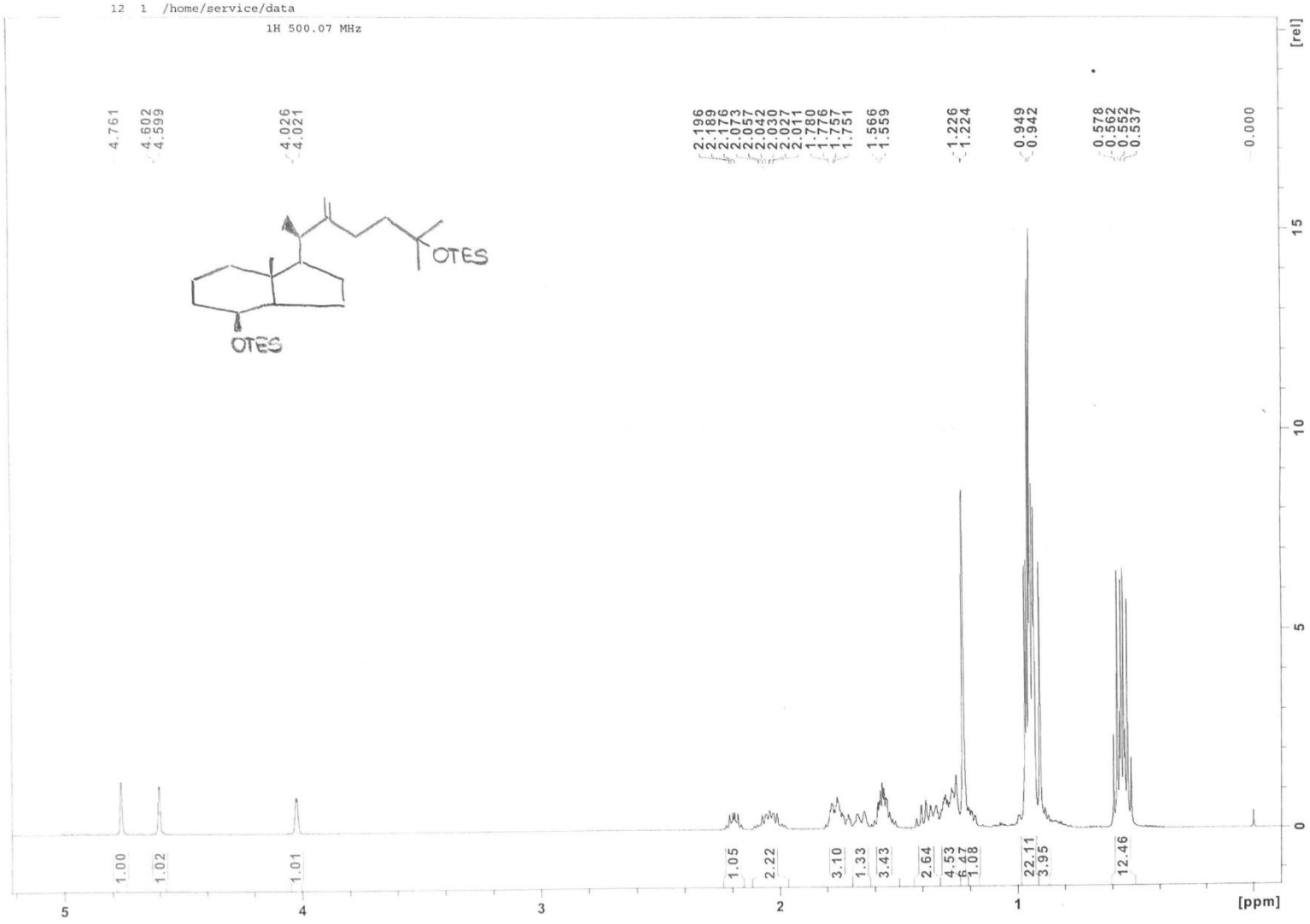

13C 100.61 MHz
ppm
77.318
77.001
76.684
73.402
48.551
29.808
28.927
7.007
6.622
ppm
70
60
50
40
30
20
10

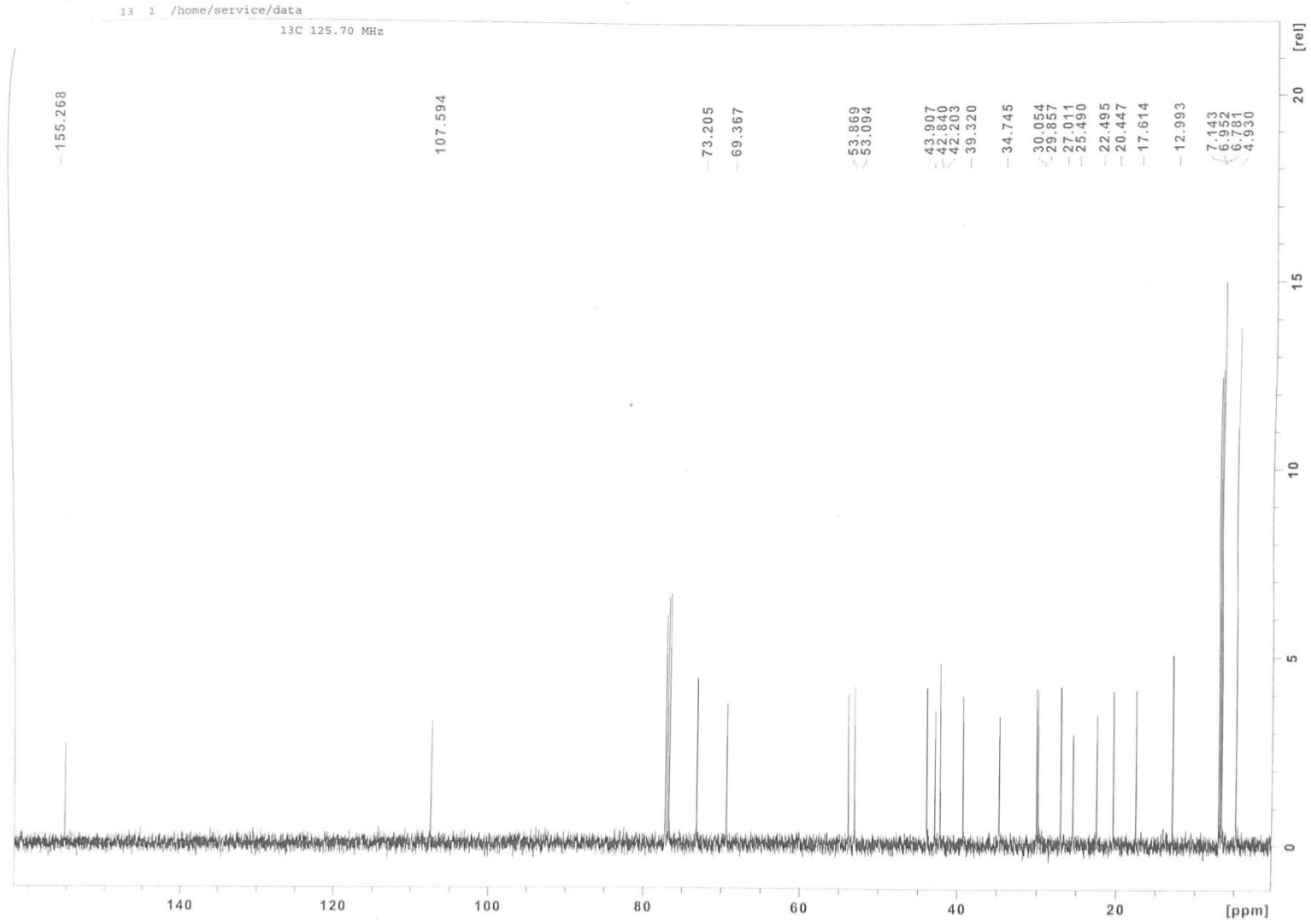

6 1 /home/service/data/data/service/nmr
1H 499.84 MHz
OH
OTES
OTES
4.0432
4.0384
3.8043
3.7994
3.7943
1.9522
1.9467
1.9280
1.8813
1.8753
1.8687
1.8546
1.8473
1.8401
1.8277
1.8203
1.8095
1.8017
1.7960
1.7908
1.7844
1.7724
1.7660
1.7472
1.6921
1.6857
1.6697
1.6644
1.6128
1.4715
1.4625
1.3900
1.3731
1.2152
0.9634
0.9606
0.9474
0.9445
0.9317
0.9289
0.9145
0.8194
0.8058
0.5835
0.5677
0.5617
0.5458
0.0001
[*1e6]
1.00
0.99
0.99
2.12
1.37
2.94
3.03
8.74
7.00
21.43
2.82
12.42
[ppm]

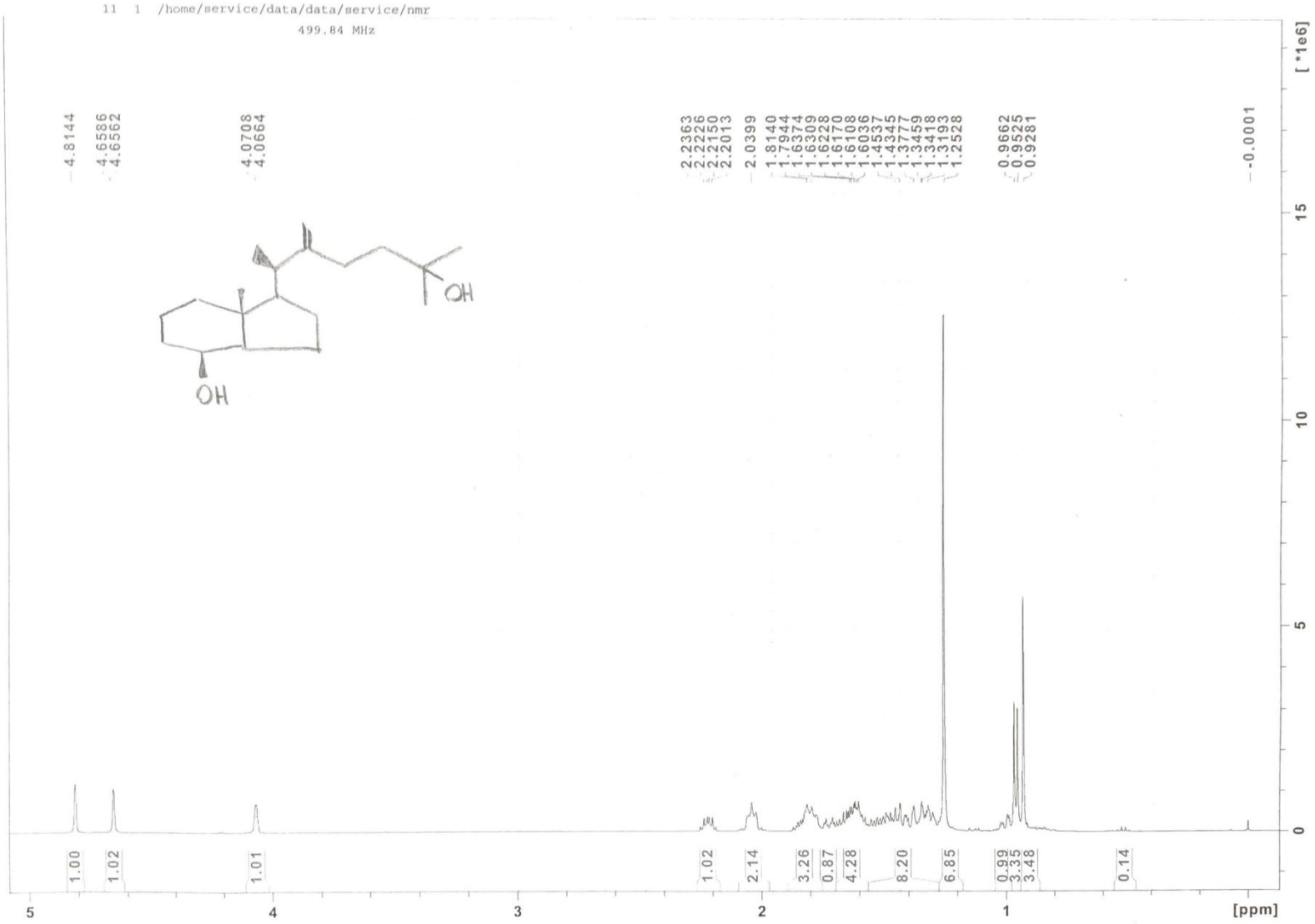

/home/service/data/data/service/nmr
1H 499.84 MHz
73.3099
73.2053
69.3119
53.0169
52.5245
41.9367
41.8599
40.3054
39.1175
34.6205
29.9199
29.8481
29.7508
27.0925
22.8405
17.6777
13.8612
11.2042
7.0933
6.9346
6.6878
4.9025
[*1e6]
15
10
5
0
200
150
100
50
[ppm]

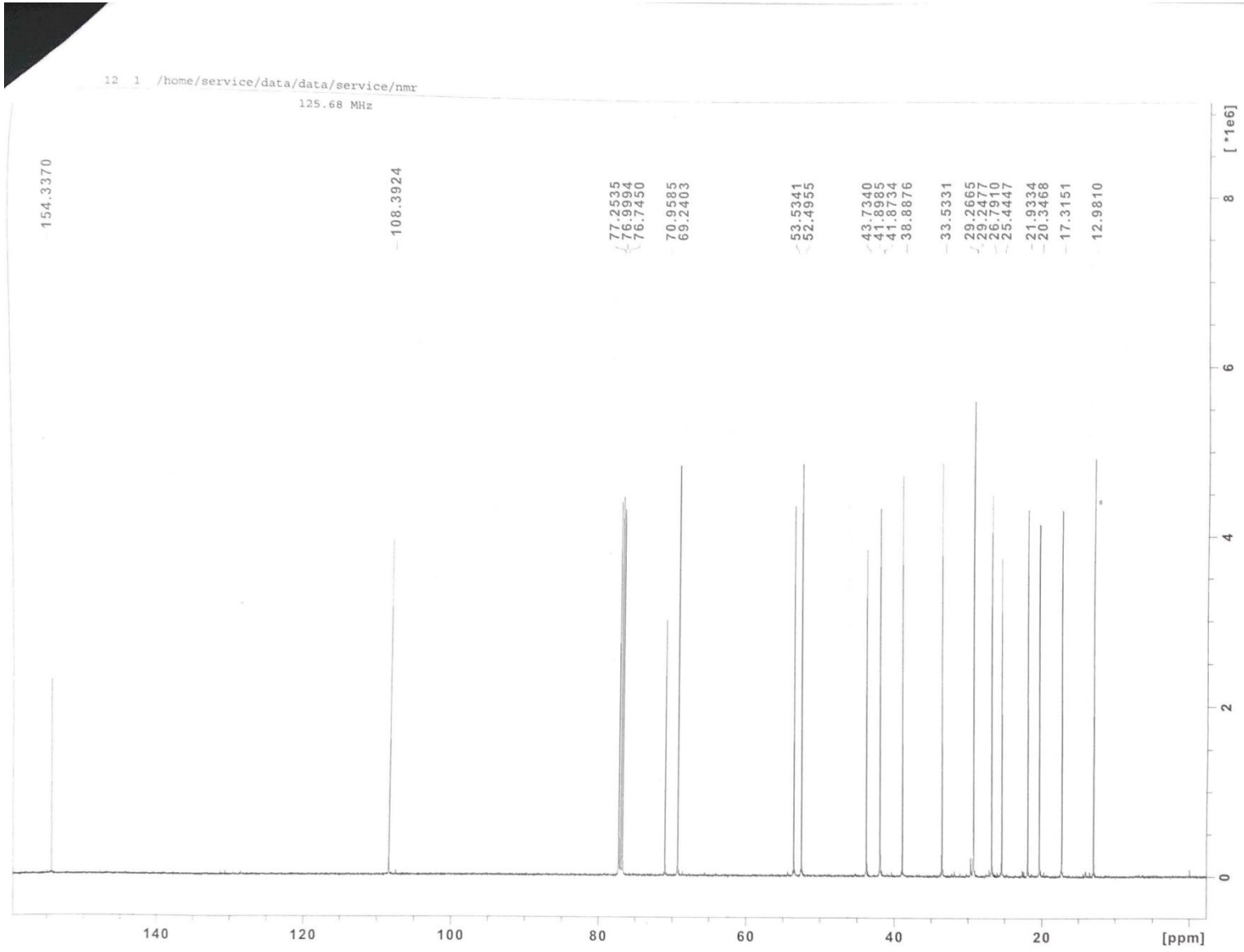

31 1 /home/service/data/data/service/nmr
1H 499.84 MHz
4.0274
4.0226
2.6185
1.6719
1.6608
1.2206
1.2045
1.0049
0.9911
0.9574
0.9542
0.9415
0.9382
0.9257
0.9224
0.9048
0.5732
0.5574
0.5528
0.5369
0.0001
[*1e6]
OTES
OTES
1.00
1.13
4.87
1.39
1.15
3.04
2.33
6.09
3.32
18.70
3.15
12.17
4
3
2
1
[ppm]

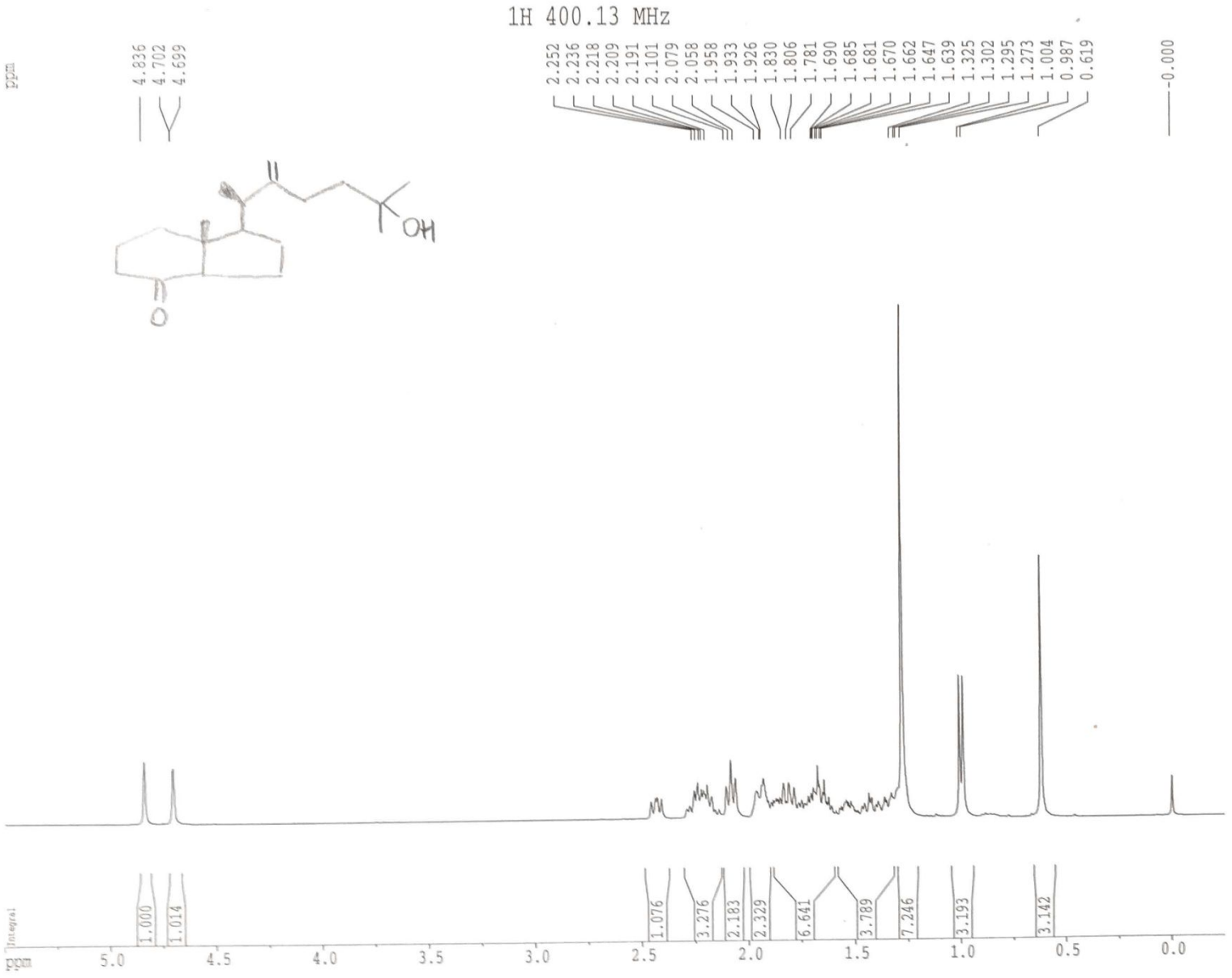

32 1 /home/service/data/data/service/nmr
13C 125.68 MHz
216.1146
77.2555
77.0042
76.7472
72.4454
69.0692
53.4196
52.4488
47.2742
41.5878
39.3566
37.9039
36.7383
34.5082
30.0318
29.7842
25.6104
22.2595
17.5229
17.0850
14.2330
7.1107
6.9242
6.6997
4.8872
[*1e6]
0
1
2
3
4
5
200
150
100
50
0
[ppm]

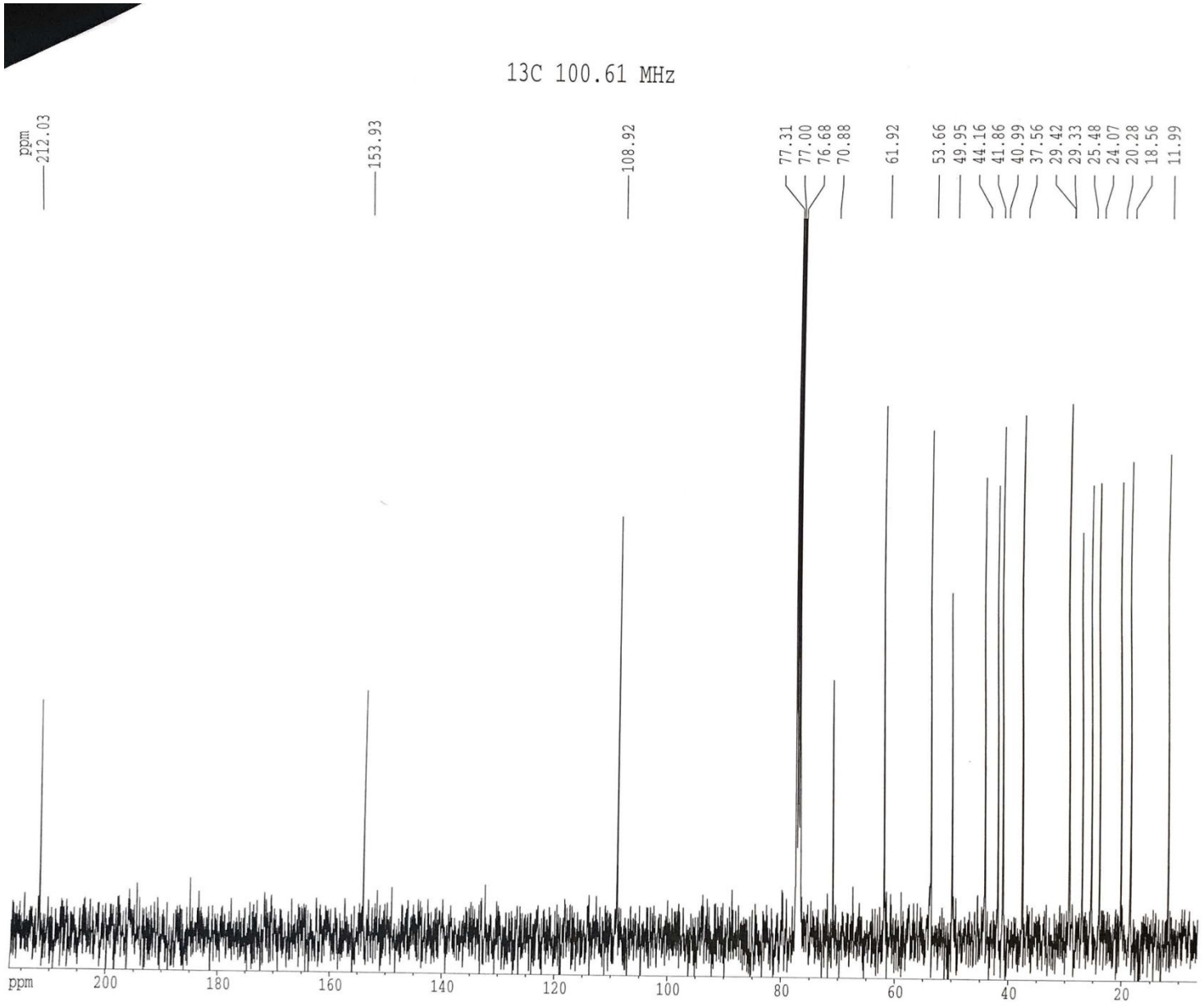

12 1 /home/service/data
1H 500.07 MHz
4.761
4.602
4.599
4.026
4.021
2.196
2.189
2.176
2.073
2.057
2.042
2.030
2.027
2.011
1.780
1.776
1.757
1.751
1.566
1.559
1.226
1.224
0.949
0.942
0.578
0.562
0.552
0.537
0.000
OTES
OTES
1.00
1.02
1.01
1.05
2.22
3.10
1.33
3.43
2.64
4.53
6.47
1.08
22.11
3.95
12.46
5
4
3
2
1
[ppm]
[rel]
15
10
5
0

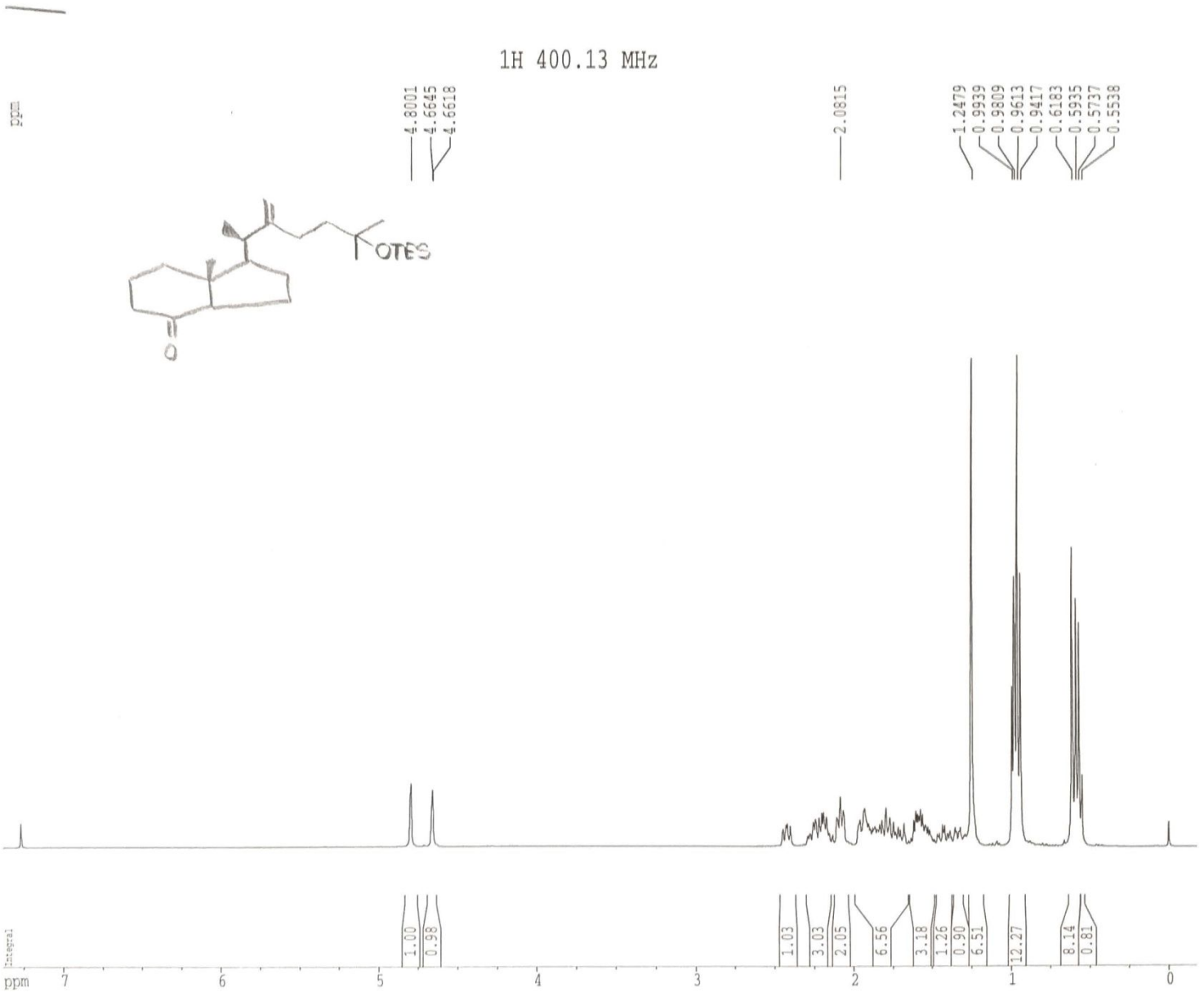

13 1 /home/service/data
13C 125.70 MHz
155.268
107.594
73.205
69.367
53.869
53.094
43.907
42.840
42.203
39.320
34.745
30.054
29.857
27.011
25.490
22.495
20.447
17.614
12.993
7.143
6.952
6.781
4.930
[rel]
20
15
10
5
0
140
120
100
80
60
40
20
[ppm]

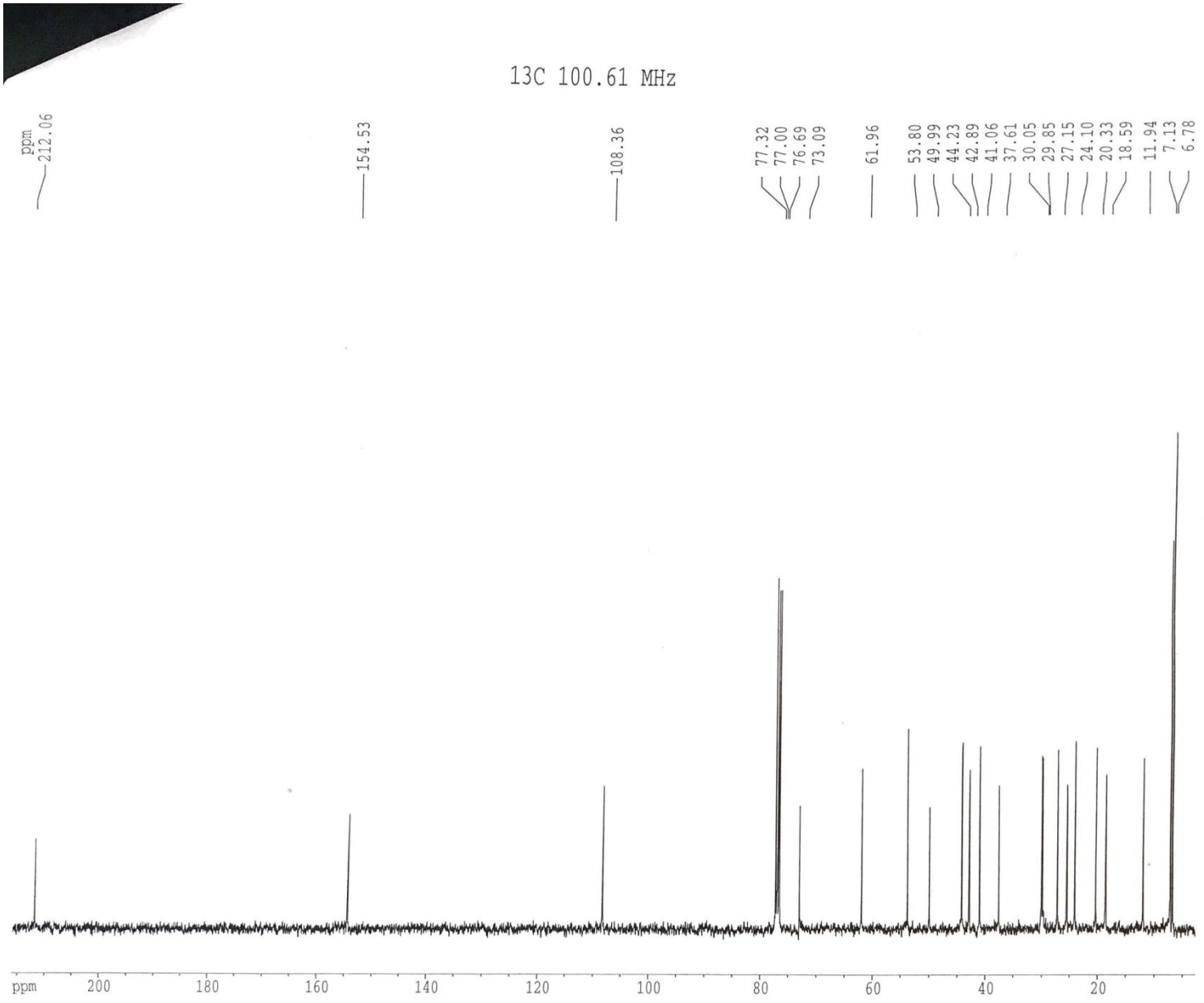

11 1 /home/service/data/data/service/nmr
499.84 MHz
4.8144
4.6586
4.6562
4.0708
4.0664
2.2363
2.2226
2.2150
2.2013
2.0399
1.8140
1.7944
1.6374
1.6309
1.6228
1.6170
1.6108
1.6036
1.4537
1.4345
1.3777
1.3459
1.3418
1.3193
1.2528
0.9662
0.9525
0.9281
-0.0001
OH
OH
[*1e6]
0
5
10
15
1.00
1.02
1.01
1.02
2.14
3.26
0.87
4.28
8.20
6.85
0.99
3.35
3.48
0.14
5
4
3
2
1
[ppm]

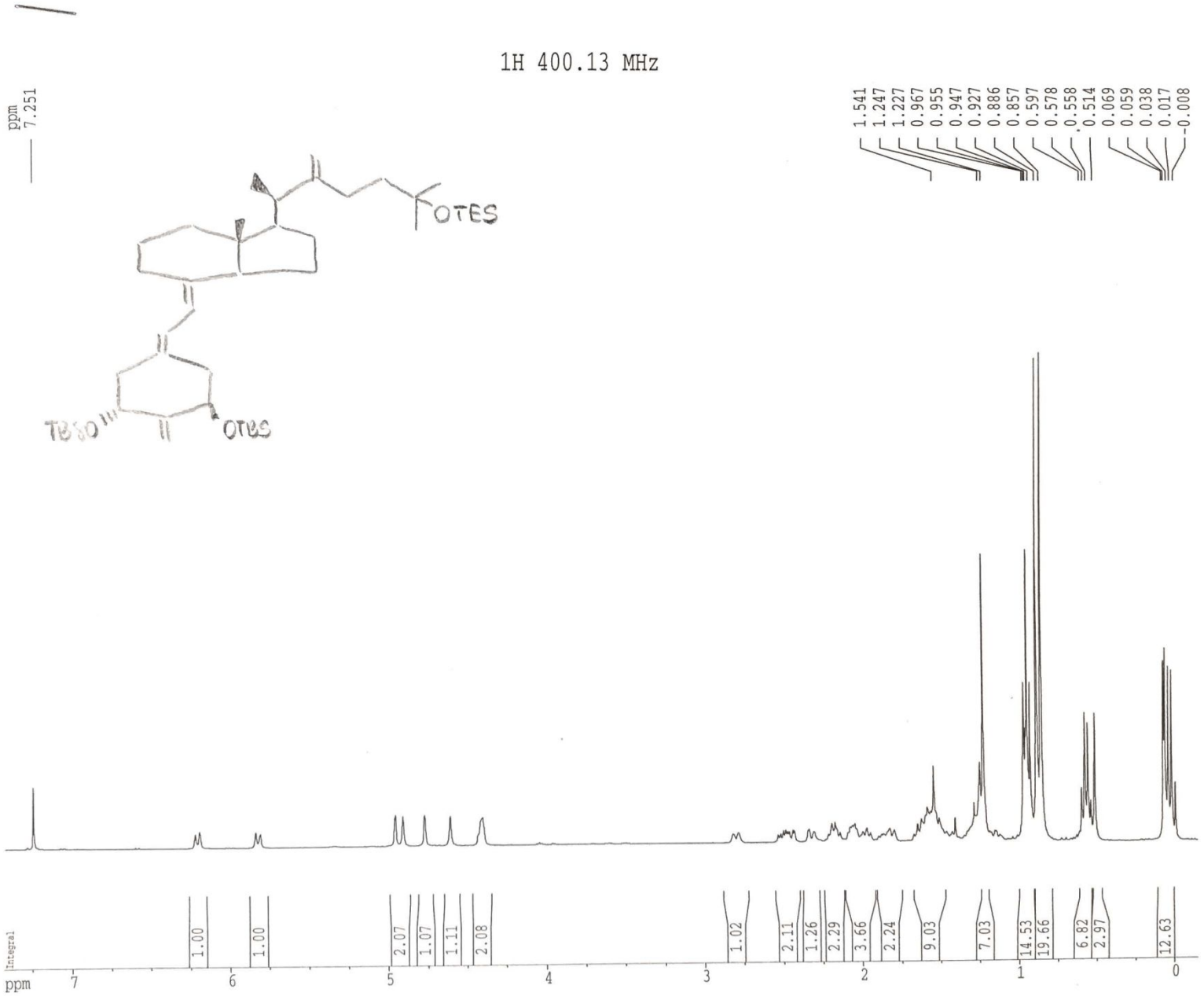

12 1 /home/service/data/data/service/nmr
125.68 MHz
154.3370
108.3924
77.2535
76.9994
76.7450
70.9585
69.2403
53.5341
52.4955
43.7340
41.6985
41.8734
38.8876
33.5331
29.2665
29.2477
26.7910
25.4447
21.9334
20.3468
17.3151
12.9810
[*1e6]
0
2
4
6
8
140
120
100
80
60
40
20
[ppm]

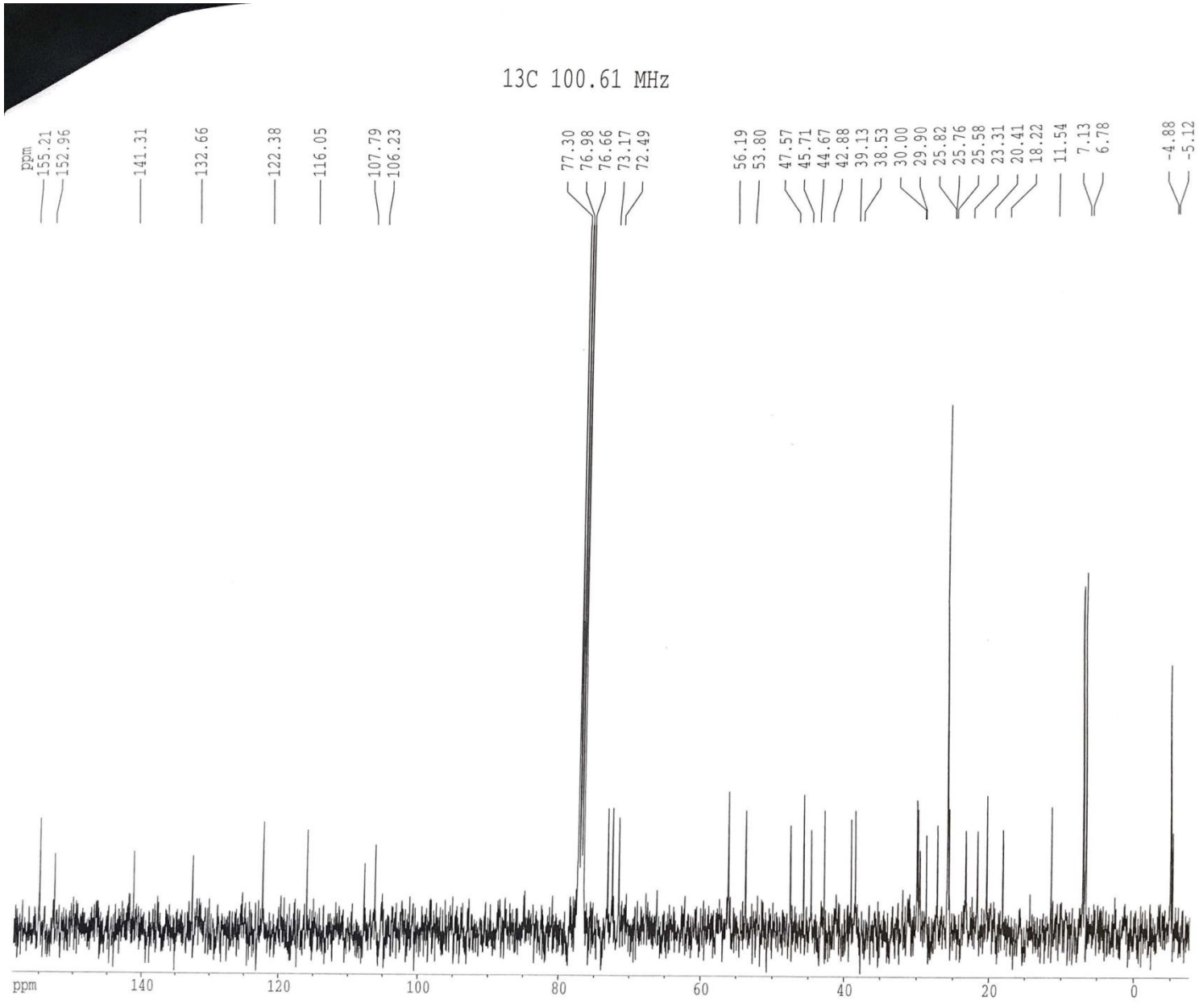

1H 400.13 MHz
ppm
4.836
4.702
4.699
2.252
2.236
2.218
2.209
2.191
2.101
2.079
2.058
1.958
1.933
1.926
1.830
1.806
1.781
1.690
1.685
1.681
1.670
1.662
1.647
1.639
1.325
1.302
1.295
1.273
1.004
0.987
0.619
-0.000
OH
O
Integral
1.000
1.014
1.076
3.276
2.183
2.329
6.641
3.789
7.246
3.193
3.142
ppm
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

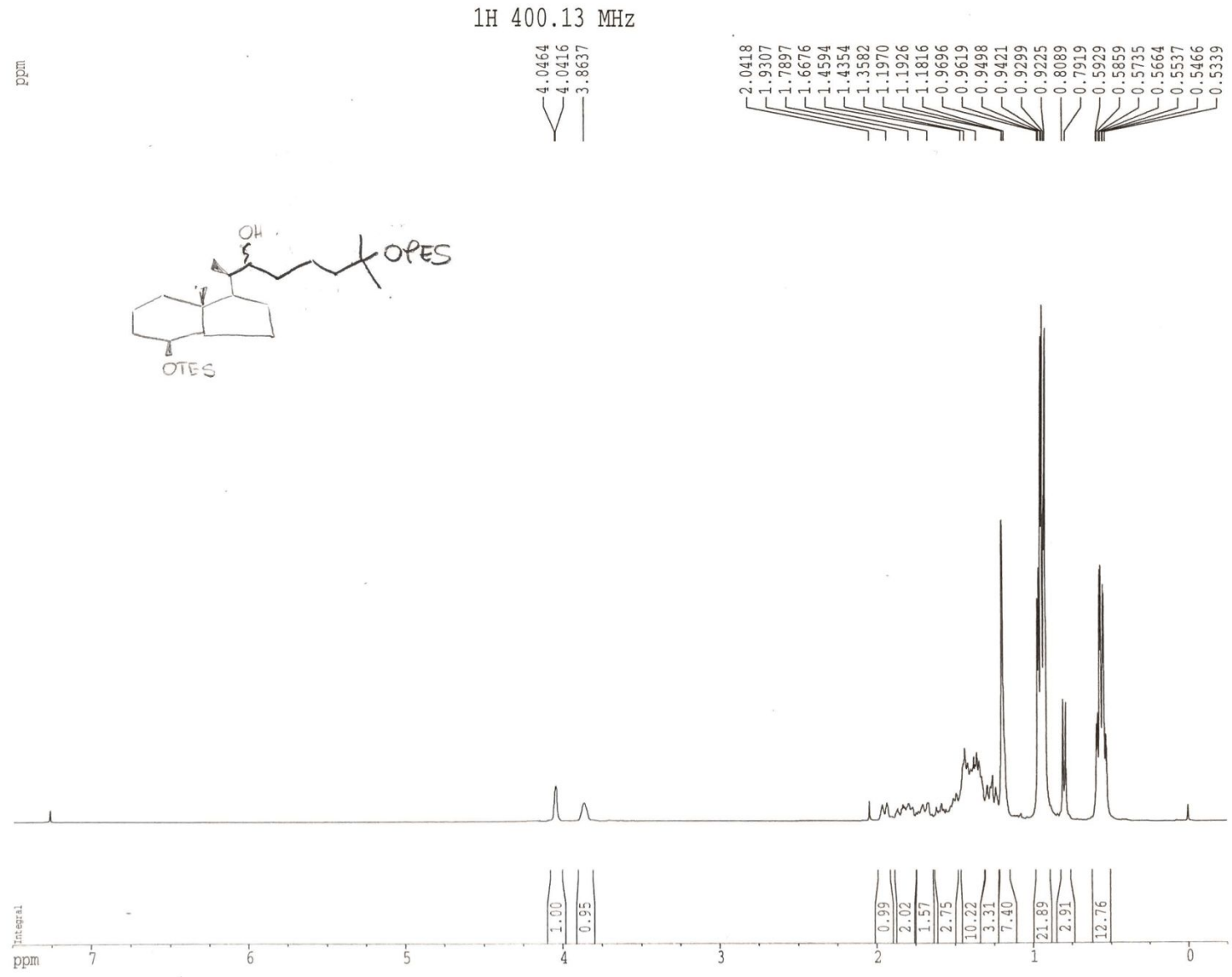

13C 100.61 MHz
ppm
212.03
153.93
108.92
77.31
77.00
76.68
70.88
61.92
53.66
49.95
44.16
41.86
40.99
37.56
29.42
29.33
25.48
24.07
20.28
18.56
11.99
ppm
200
180
160
140
120
100
80
60
40
20

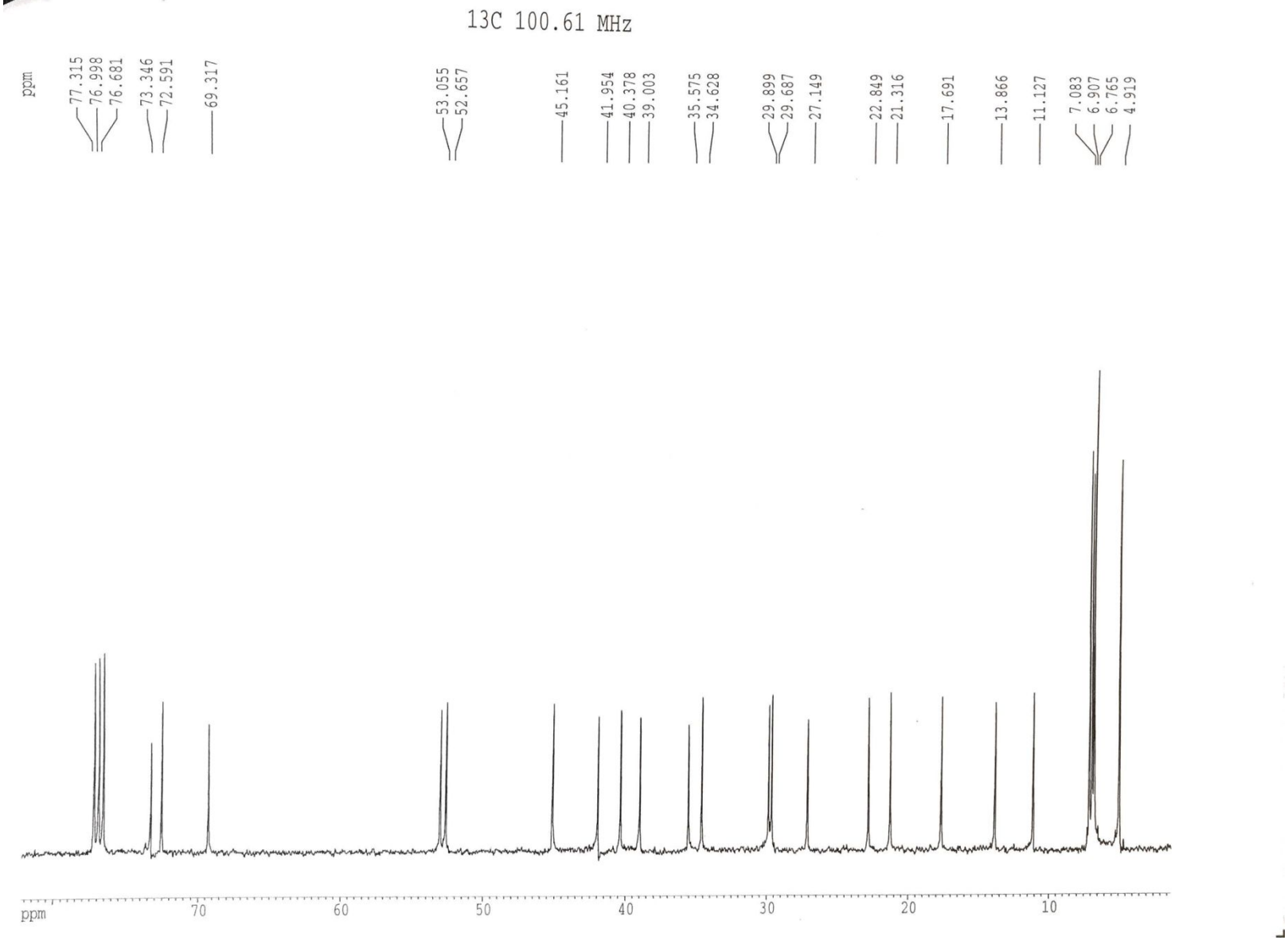

1H 400.13 MHz
ppm
4.8001
4.6645
4.6618
2.0815
1.2479
0.9939
0.9809
0.9613
0.9417
0.6183
0.5935
0.5737
0.5538
OTES
O
Integral
1.00
0.98
1.03
3.03
2.05
6.56
3.18
1.26
0.90
6.51
12.27
8.14
0.81
ppm
7
6
5
4
3
2
1
0


13C 100.61 MHz
ppm
212.06
154.53
108.36
77.32
77.00
76.69
73.09
61.96
53.80
49.99
44.23
42.89
41.06
37.61
30.05
29.85
27.15
24.10
20.33
18.59
11.94
7.13
6.78
ppm
200
180
160
140
120
100
80
60
40
20


1H 400.13 MHz
ppm
7.251
1.541
1.247
1.227
0.967
0.955
0.947
0.927
0.886
0.857
0.597
0.578
0.558
0.514
0.069
0.059
0.038
0.017
-0.008
OTES
TBSO
OTBS
Integral
1.00
1.00
2.07
1.07
1.11
2.08
1.02
2.11
1.26
2.29
3.66
2.24
9.03
7.03
14.53
19.66
6.82
2.97
12.63
ppm
7
6
5
4
3
2
1
0


13C 100.61 MHz
ppm
155.21
152.96
141.31
132.66
122.38
116.05
107.79
106.23
77.30
76.98
76.66
73.17
72.49
56.19
53.80
47.57
45.71
44.67
42.88
39.13
38.53
30.00
29.90
25.82
25.76
25.58
23.31
20.41
18.22
11.54
7.13
6.78
-4.88
-5.12
ppm
140
120
100
80
60
40
20
0


1H 400.13 MHz
ppm
4.0464
4.0416
3.8637
2.0418
1.9307
1.7897
1.6676
1.4594
1.4354
1.3582
1.1970
1.1926
1.1816
0.9696
0.9619
0.9498
0.9421
0.9299
0.9225
0.8089
0.7919
0.5929
0.5859
0.5735
0.5664
0.5537
0.5466
0.5339
OH
OPES
OTES
Integral
1.00
0.95
0.99
2.02
1.57
2.75
10.22
3.31
7.40
21.89
2.91
12.76
ppm
7
6
5
4
3
2
1
0


13C 100.61 MHz
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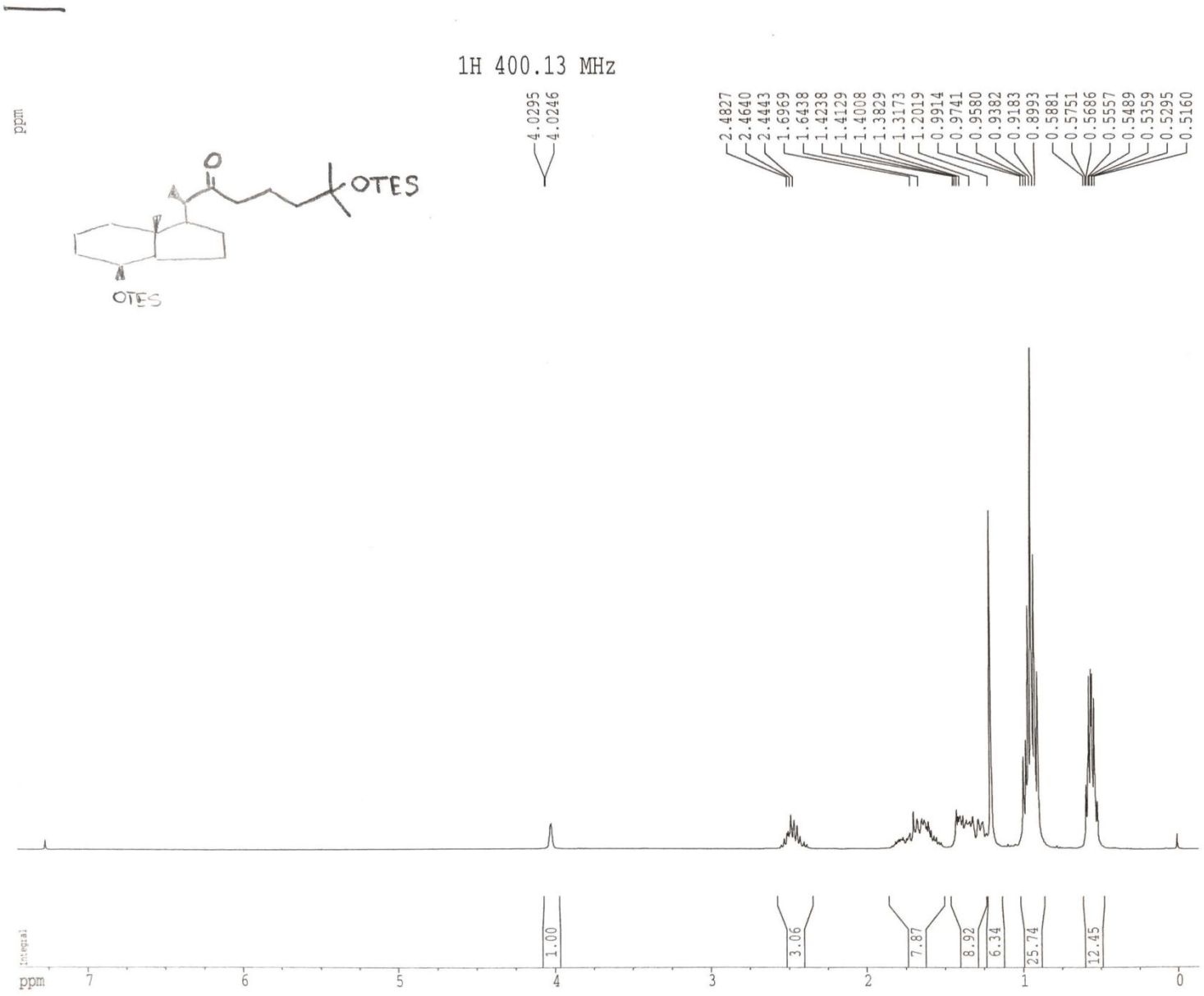
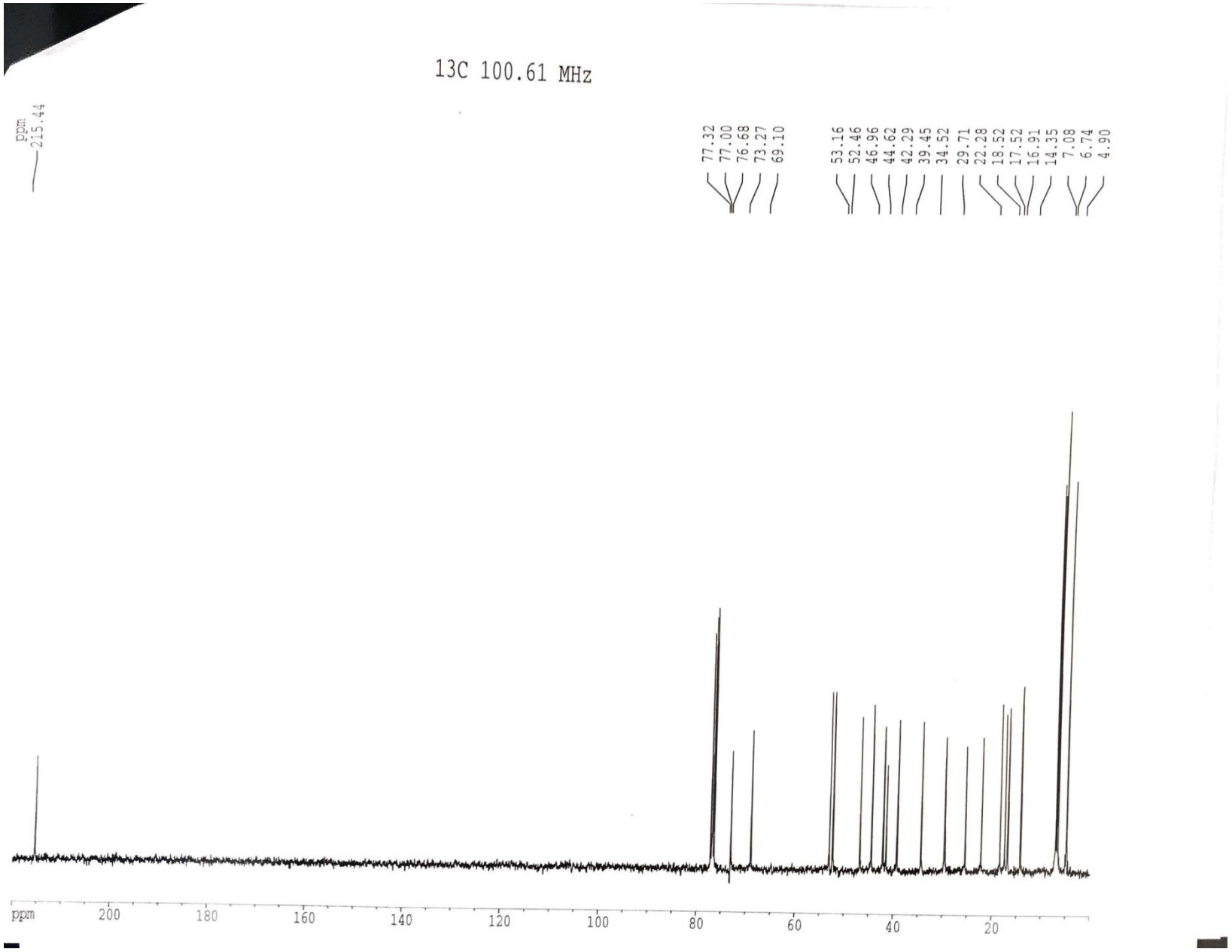
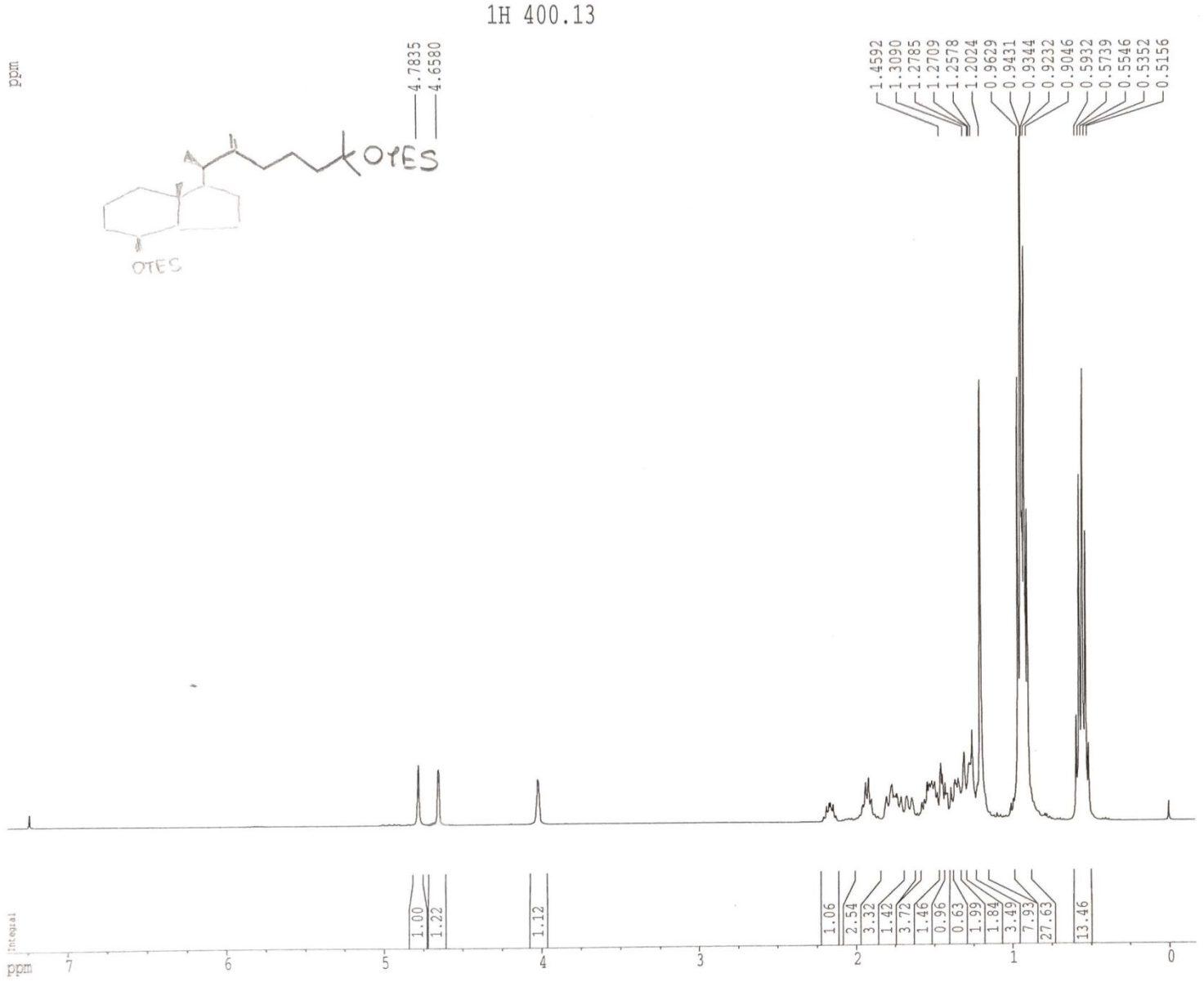
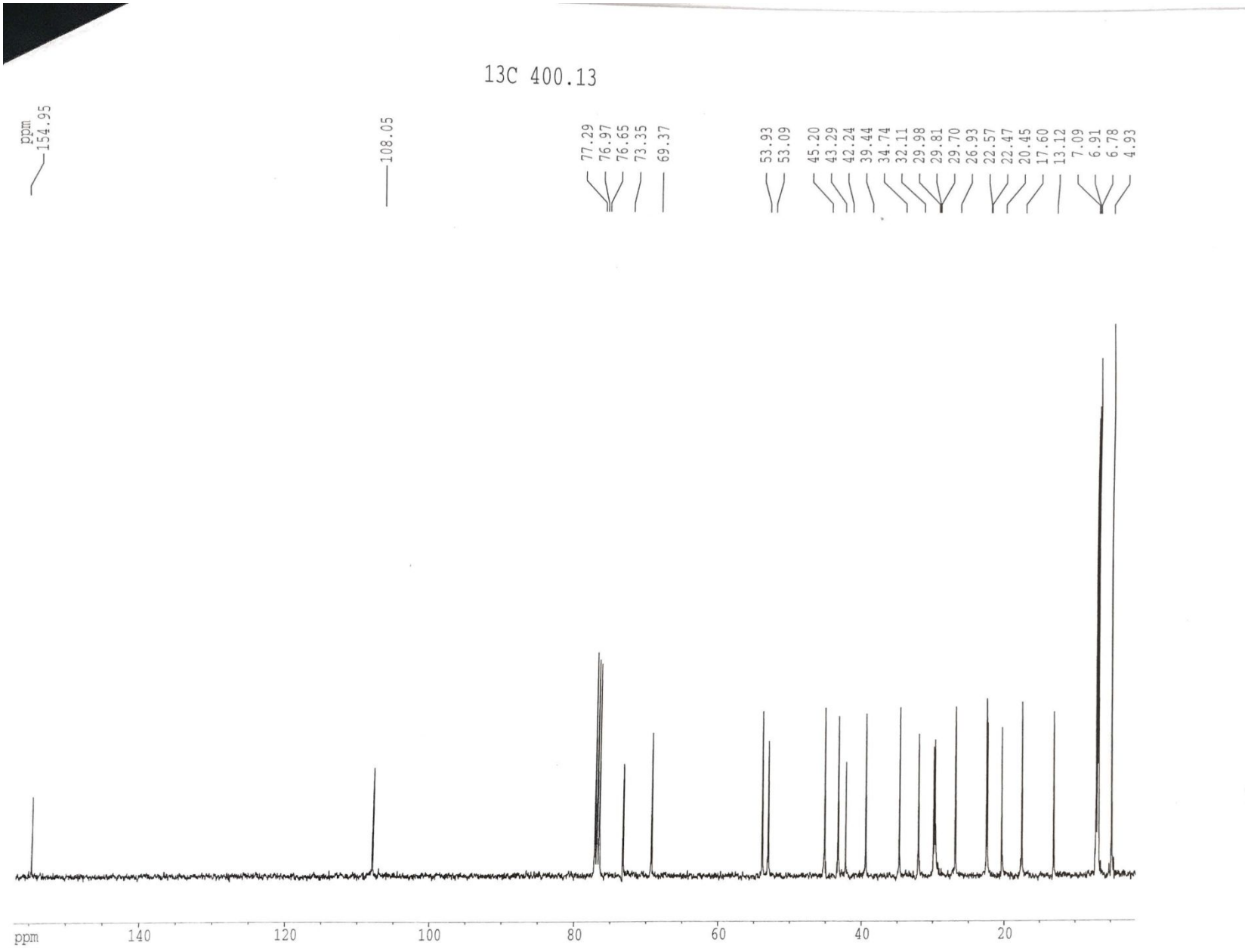
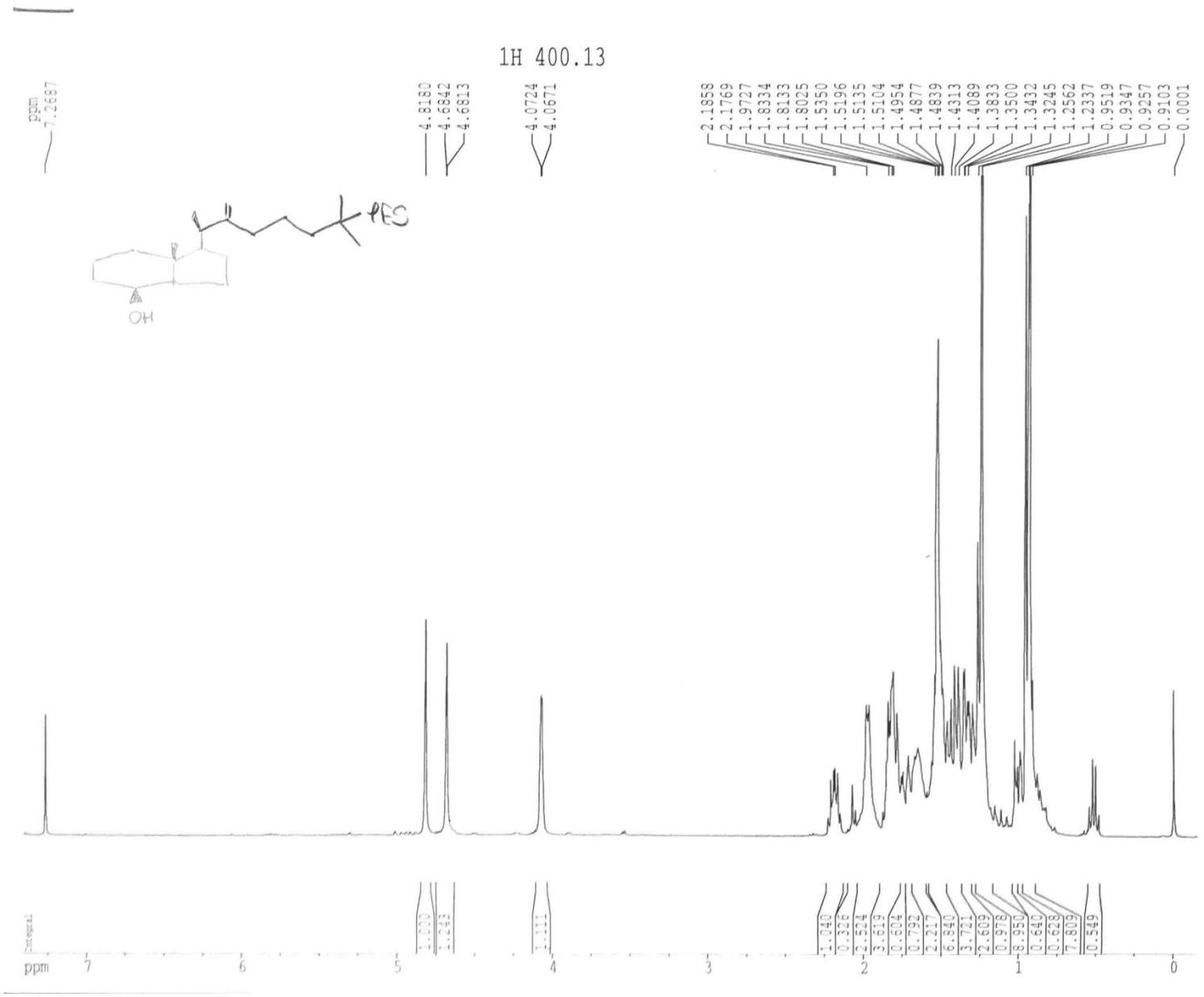
77.315
76.998
76.681
73.346
72.591
69.317
53.055
52.657
45.161
41.954
40.378
39.003
35.575
34.628
29.899
29.687
27.149
22.849
21.316
17.691
13.866
11.127
7.083
6.907
6.765
4.919
ppm
70
60
50
40
30
20
10

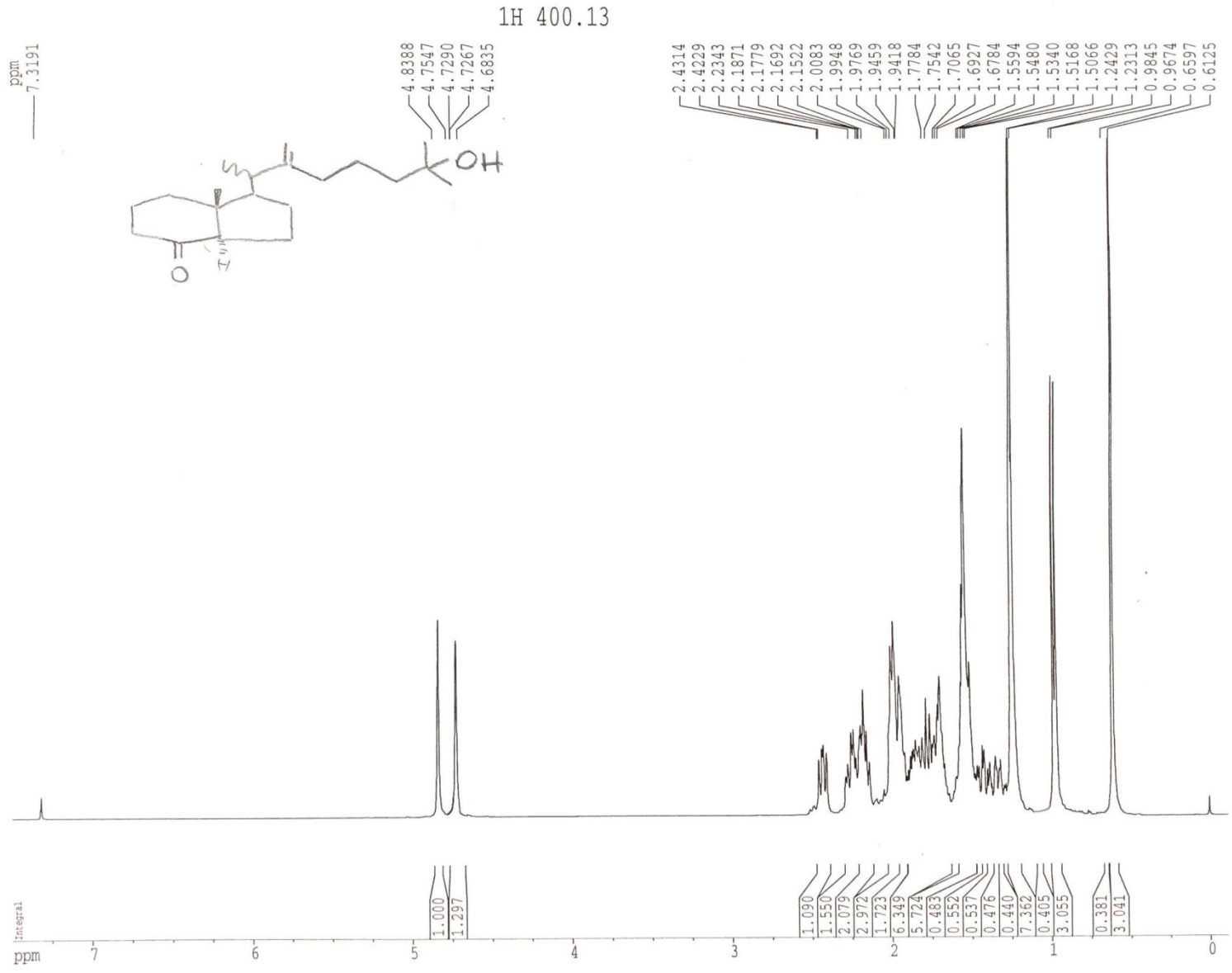

1H 400.13 MHz
ppm
4.0295
4.0246
2.4827
2.4640
2.4443
1.6969
1.6438
1.4238
1.4129
1.4008
1.3829
1.3173
1.2019
0.9914
0.9741
0.9580
0.9382
0.9183
0.8993
0.5881
0.5751
0.5686
0.5557
0.5489
0.5359
0.5295
0.5160
OTES
OTES
integral
1.00
3.06
7.87
8.92
6.34
25.74
12.45
ppm
7
6
5
4
3
2
1
0

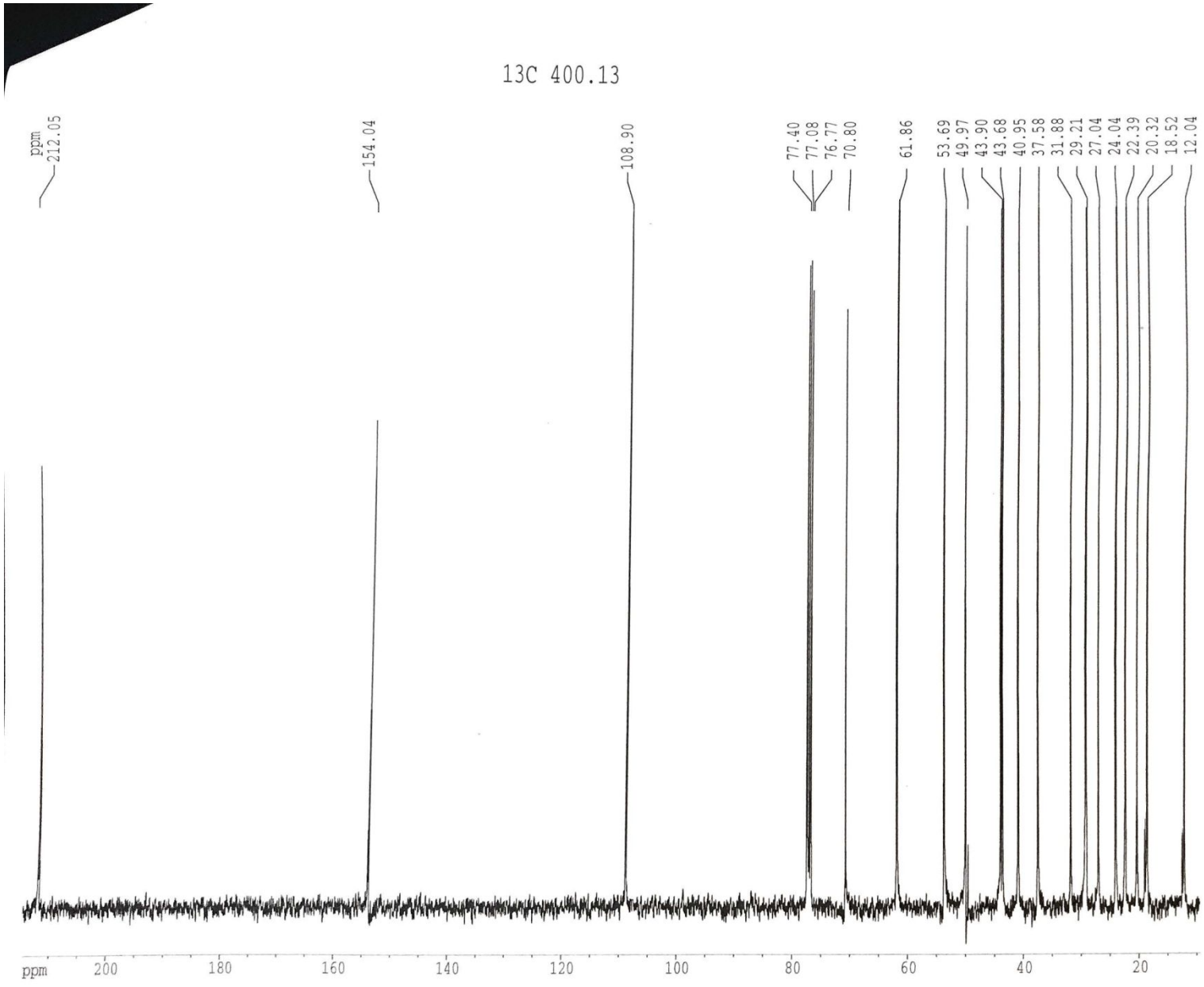

13C 100.61 MHz
ppm
215.44
77.32
77.00
76.68
73.27
69.10
53.16
52.46
46.96
44.62
42.29
39.45
34.52
29.71
22.28
18.52
17.52
16.91
14.35
7.08
6.74
4.90
ppm
200
180
160
140
120
100
80
60
40
20

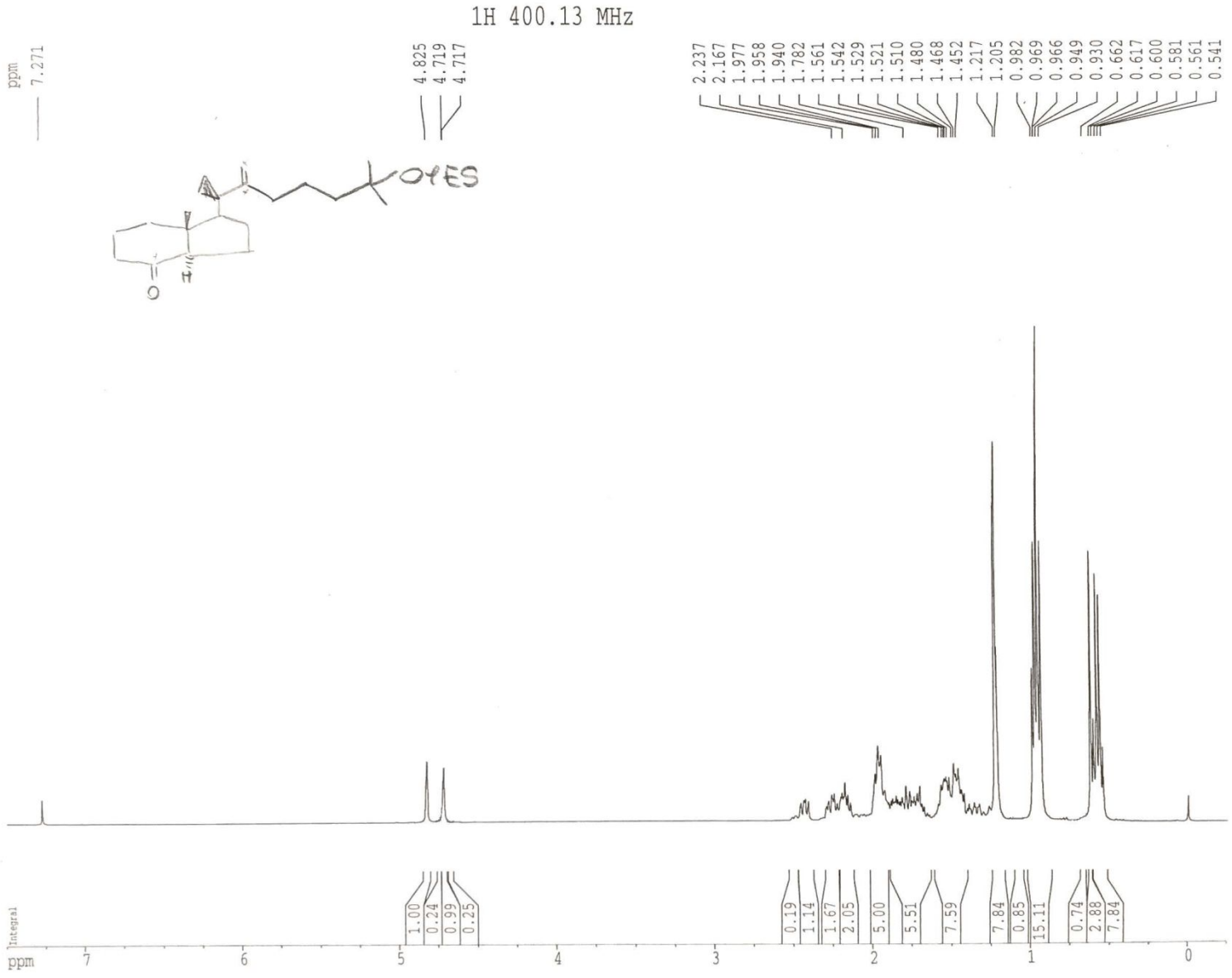

1H 400.13
ppm
4.7835
4.6580
OTES
OTES
1.4592
1.3090
1.2785
1.2709
1.2578
1.2024
0.9629
0.9431
0.9344
0.9232
0.9046
0.5932
0.5739
0.5546
0.5352
0.5156
Integral
1.00
1.22
1.12
1.06
2.54
3.32
1.42
3.72
1.46
0.96
0.63
1.99
1.84
3.49
7.93
27.63
13.46
ppm
7
6
5
4
3
2
1
0

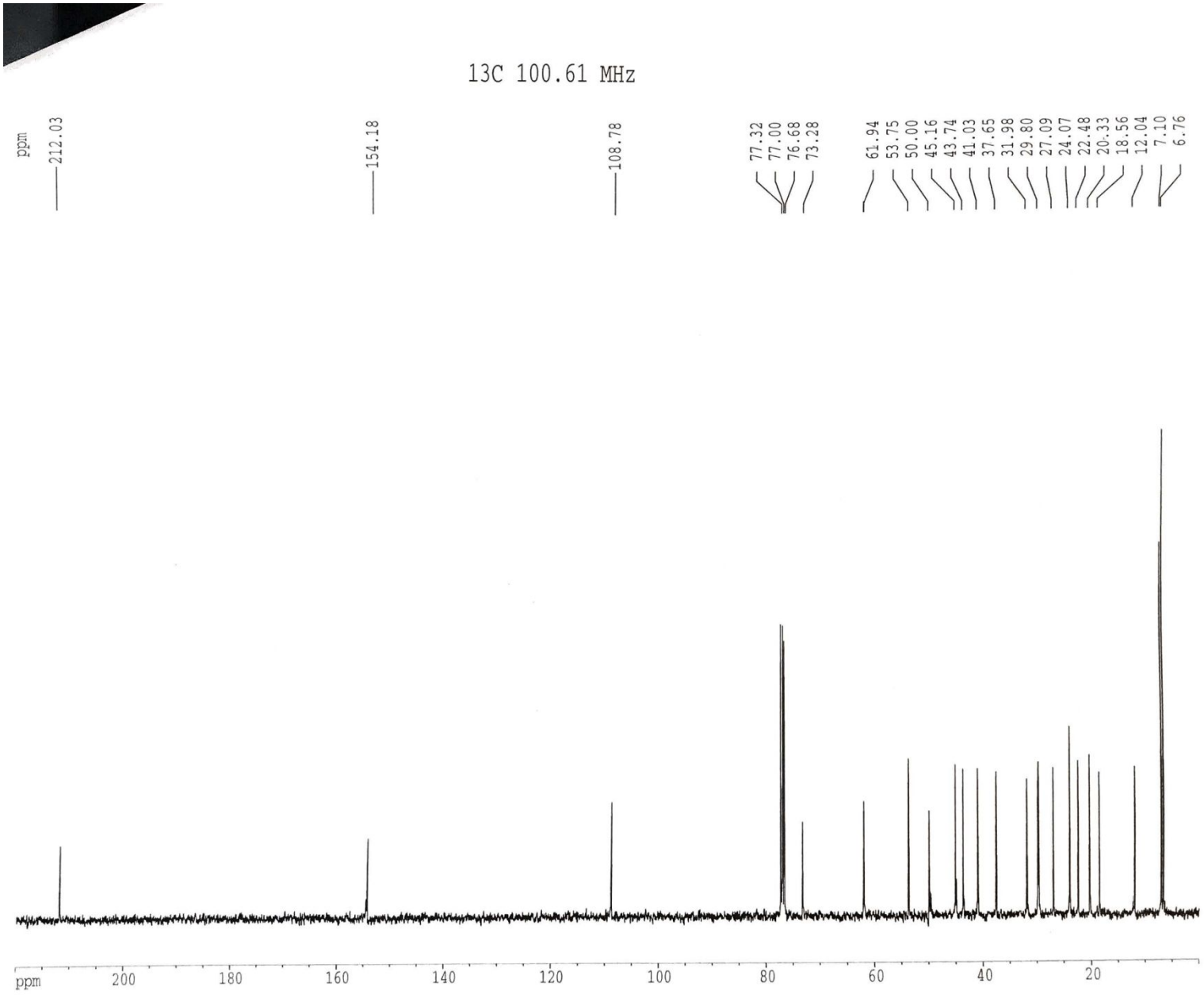

13C 400.13
ppm
154.95
108.05
77.29
76.97
76.65
73.35
69.37
53.93
53.09
45.20
43.29
42.24
39.44
34.74
32.11
29.98
29.81
29.70
26.93
22.57
22.47
20.45
17.60
13.12
7.09
6.91
6.78
4.93
ppm
140
120
100
80
60
40
20

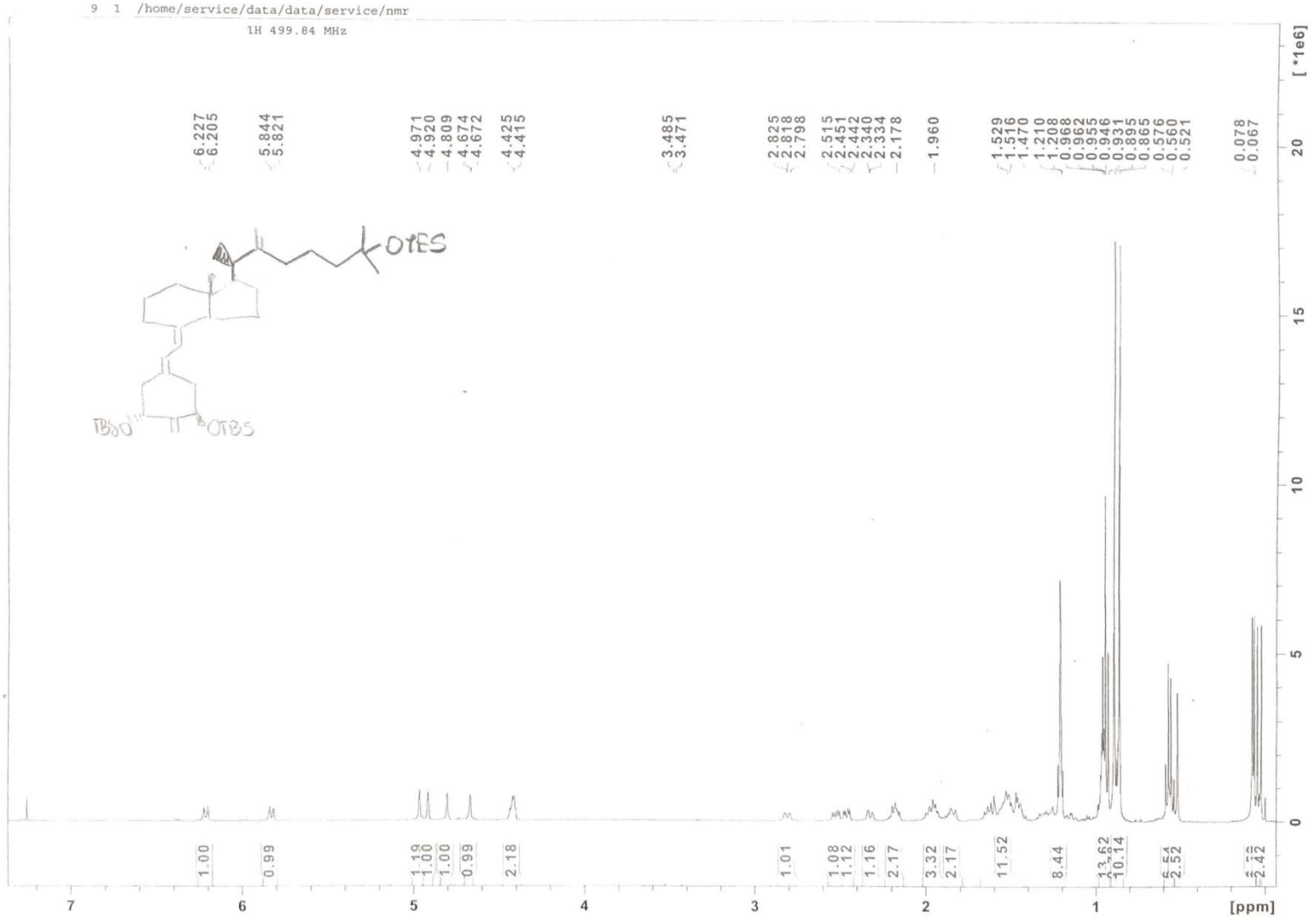

1H 400.13
ppm
7.2687
4.8180
4.6842
4.6813
4.0724
4.0671
2.1858
2.1769
2.1727
1.8334
1.8133
1.8025
1.5350
1.5196
1.5135
1.5104
1.4954
1.4877
1.4839
1.4313
1.4089
1.3833
1.3500
1.3432
1.3245
1.2562
1.2337
0.9519
0.9347
0.9257
0.9103
0.0001
PES
OH
Integral
1.000
1.243
1.111
1.040
0.326
2.524
3.619
0.604
0.792
2.211
6.840
3.721
12.609
0.978
8.950
0.640
0.628
7.809
0.549
ppm
7
6
5
4
3
2
1
0

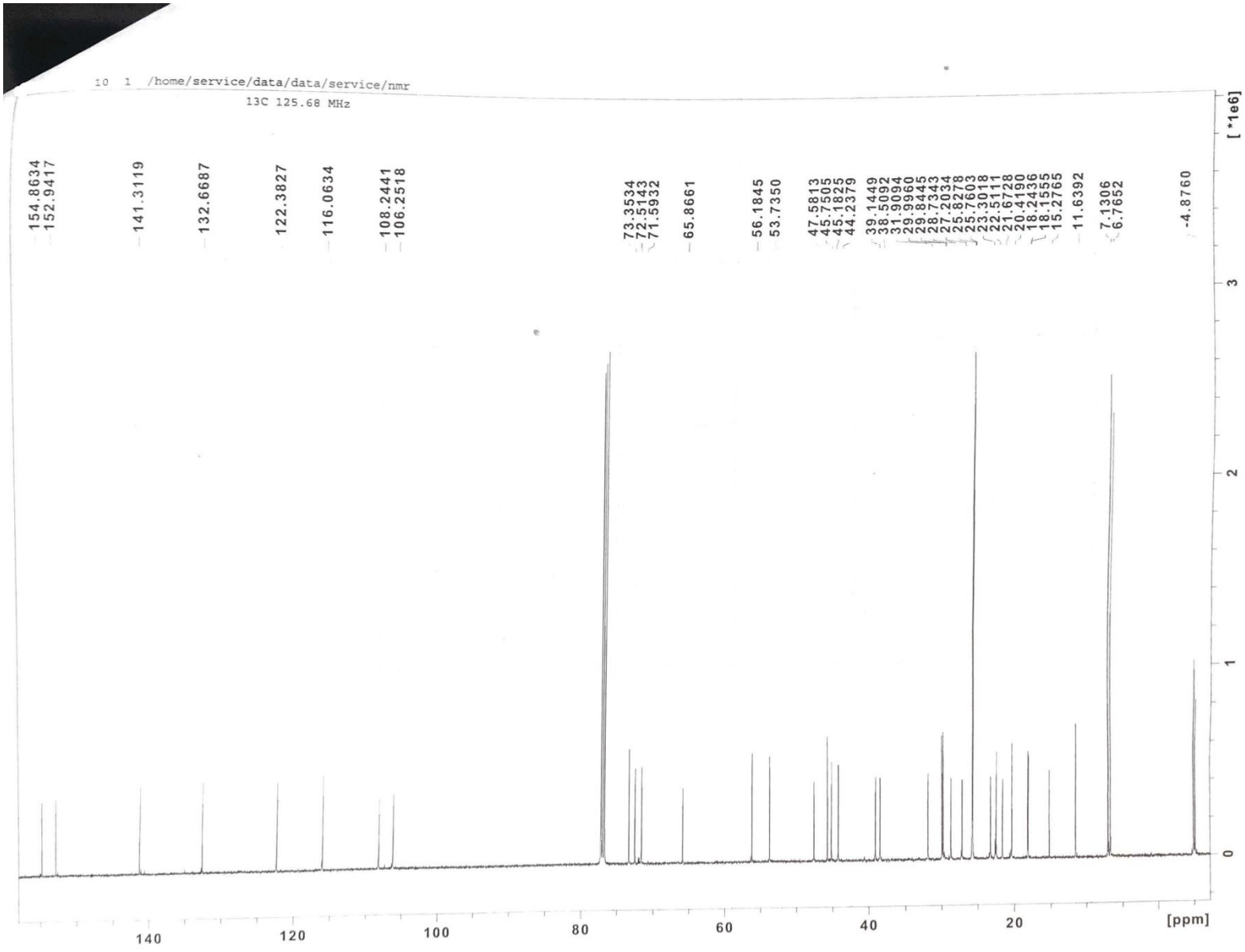

1H 400.13
ppm
7.3191
4.8388
4.7547
4.7290
4.7267
4.6835
OH
O
2.4314
2.4229
2.2343
2.1871
2.1779
2.1692
2.1522
2.0083
1.9948
1.9769
1.9459
1.9418
1.7784
1.7542
1.7065
1.6927
1.6784
1.5594
1.5480
1.5340
1.5168
1.5066
1.2429
1.2313
0.9845
0.9674
0.6597
0.6125
Integral
1.000
1.297
1.090
1.550
2.079
2.972
1.723
6.349
5.724
0.483
0.552
0.537
0.476
0.440
7.362
0.405
3.055
0.381
3.041
ppm
7
6
5
4
3
2
1
0

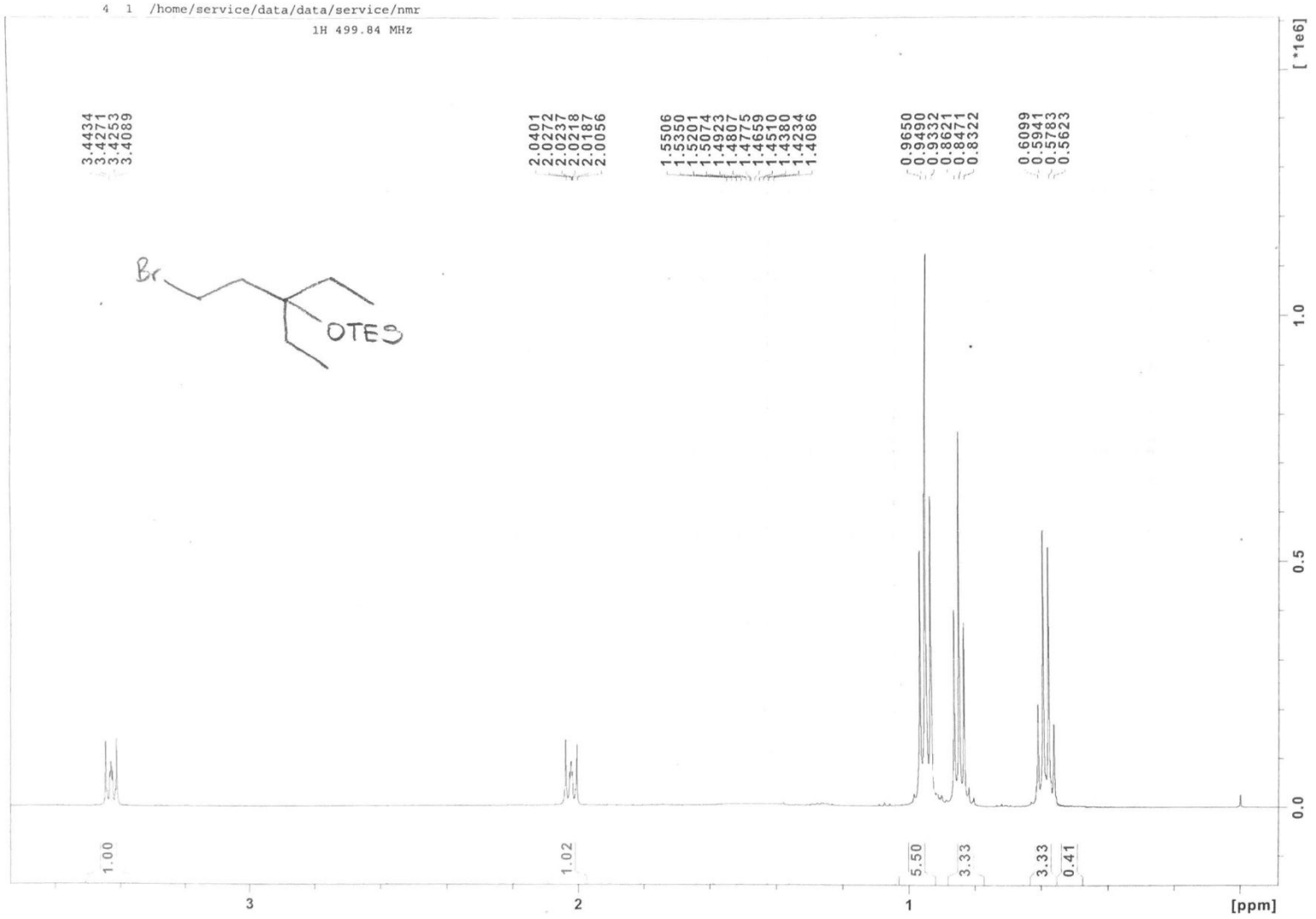

13C 400.13
ppm
212.05
154.04
108.90
77.40
77.08
76.77
70.80
61.86
53.69
49.97
43.90
43.68
40.95
37.58
31.88
29.21
27.04
24.04
22.39
20.32
18.52
12.04
ppm
200
180
160
140
120
100
80
60
40
20

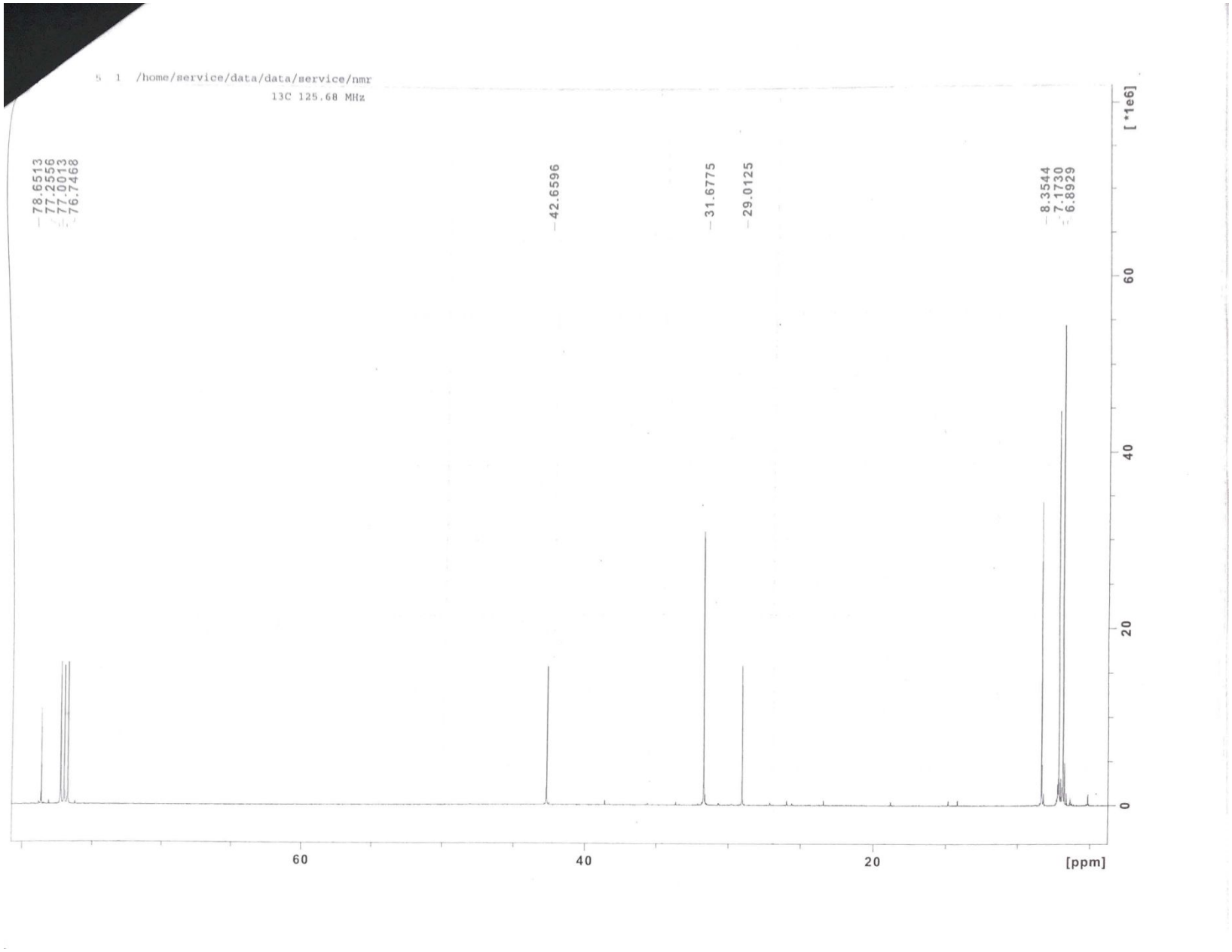

1H 400.13 MHz
ppm
7.271
4.825
4.719
4.717
2.237
2.167
1.977
1.958
1.940
1.782
1.561
1.542
1.529
1.521
1.510
1.480
1.468
1.452
1.217
1.205
0.982
0.969
0.966
0.949
0.930
0.662
0.617
0.600
0.581
0.561
0.541
OYES
O
H
Integral
1.00
0.24
0.99
0.25
0.19
1.14
1.67
2.05
5.00
5.51
7.59
7.84
0.85
15.11
0.74
2.88
7.84
ppm
7
6
5
4
3
2
1
0

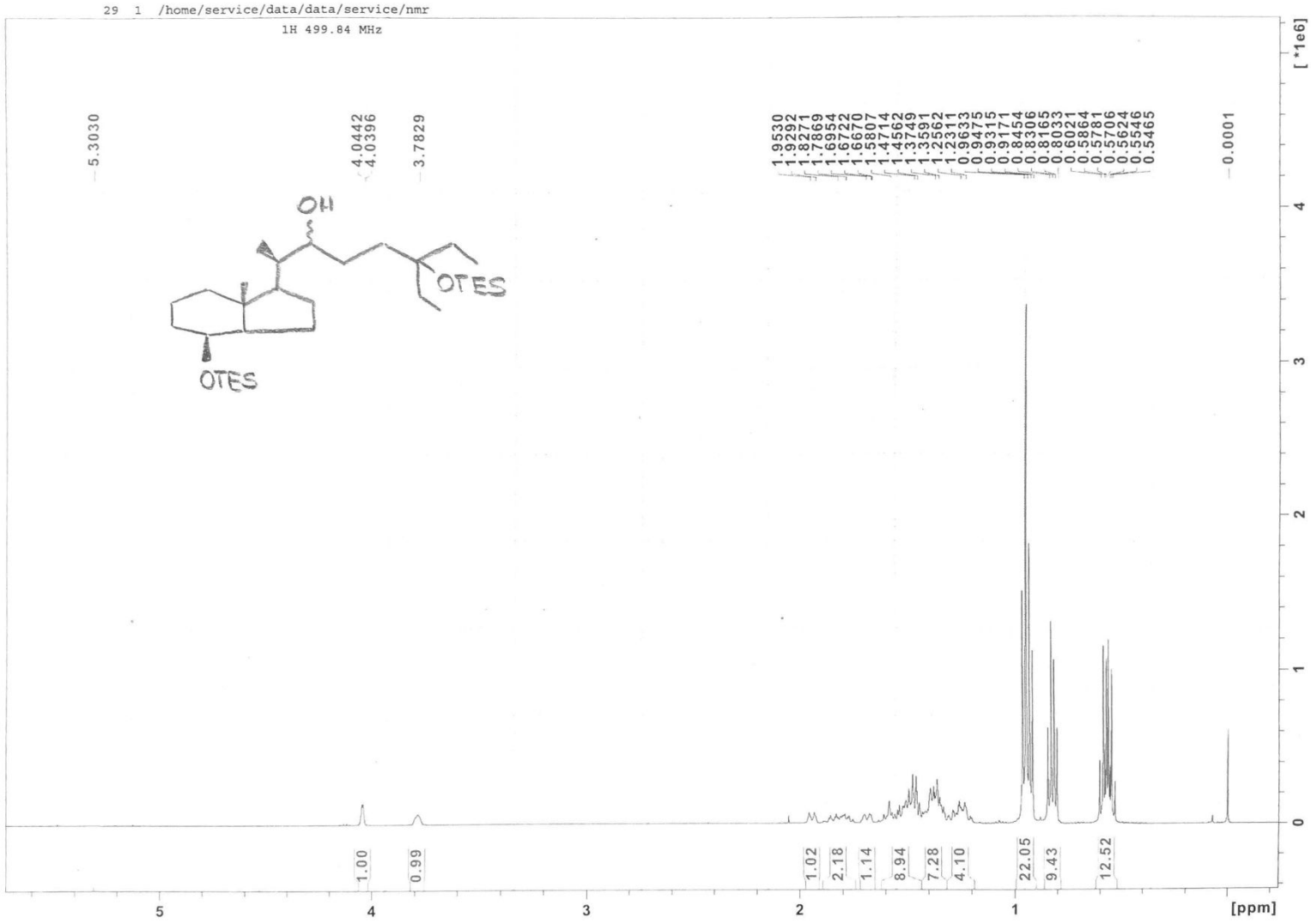

13C 100.61 MHz
ppm
212.03
154.18
108.78
77.32
77.00
76.68
73.28
61.94
53.75
50.00
45.16
43.74
41.03
37.65
31.98
29.80
27.09
24.07
22.48
20.33
18.56
12.04
7.10
6.76
ppm
200
180
160
140
120
100
80
60
40
20

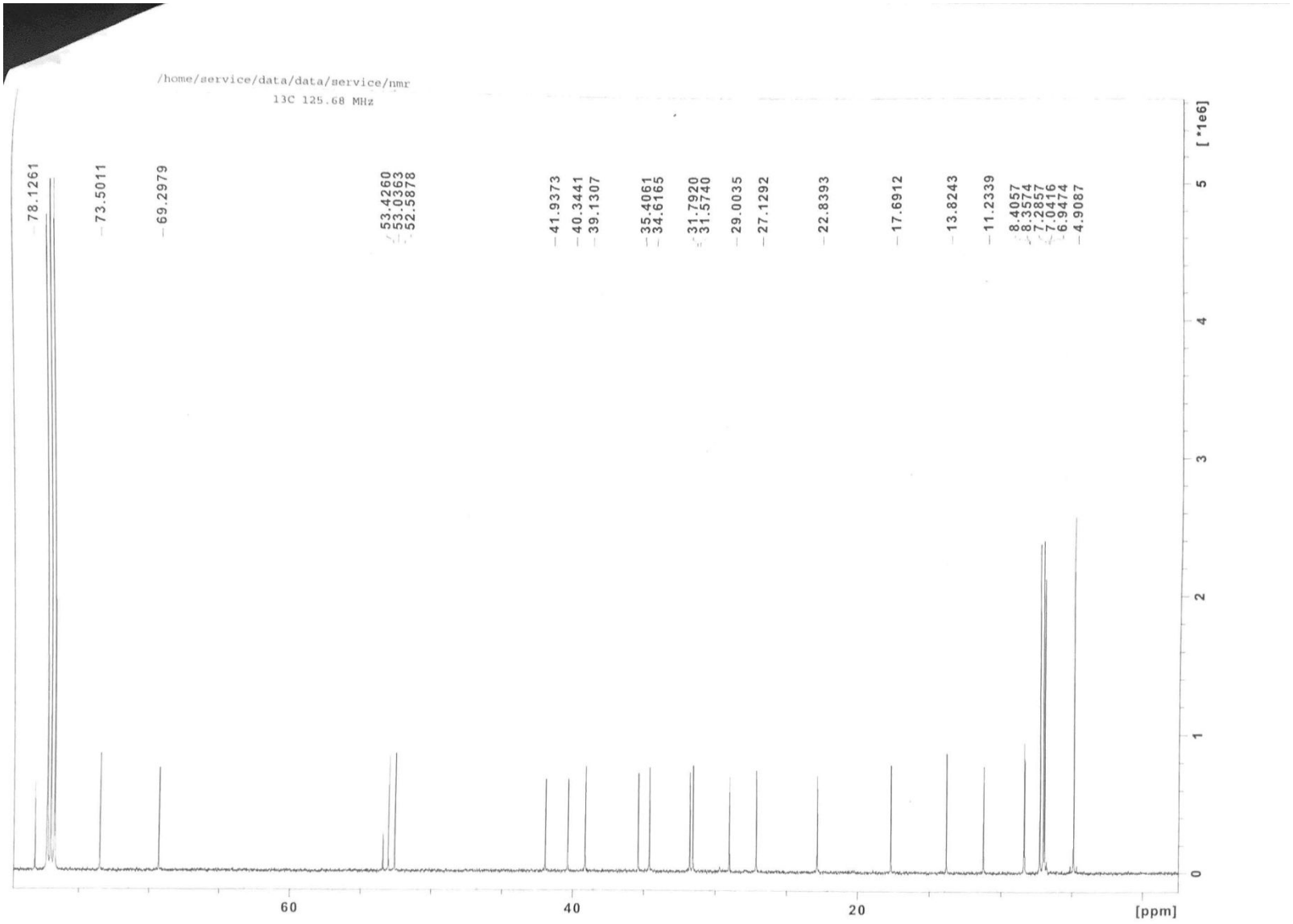

9 1 /home/service/data/data/service/nmr
1H 499.84 MHz
6.227
6.205
5.844
5.821
4.971
4.920
4.809
4.674
4.672
4.425
4.415
3.485
3.471
2.825
2.818
2.798
2.515
2.451
2.442
2.340
2.334
2.178
1.960
1.529
1.516
1.470
1.210
1.208
0.968
0.962
0.955
0.946
0.931
0.895
0.865
0.576
0.560
0.521
0.078
0.067
OTES
TBSO
OTES
[*1e6]
[ppm]

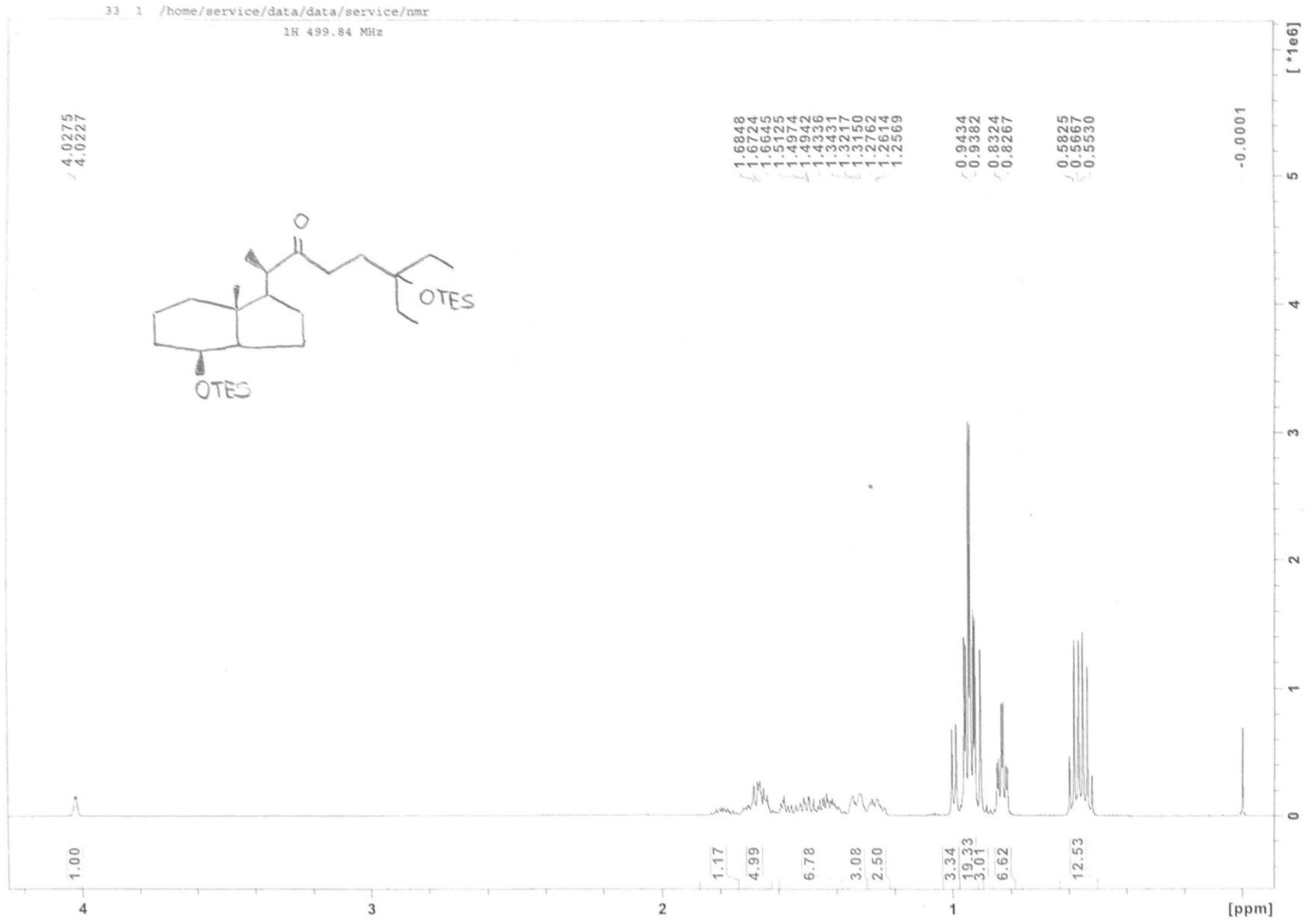

10 1 /home/service/data/data/service/nmr
13C 125.68 MHz
154.8634
152.9417
141.3119
132.6687
122.3827
116.0634
108.2441
106.2518
73.3534
72.5143
71.5932
65.8661
56.1845
53.7350
47.5813
45.7505
45.1825
44.2379
39.1449
38.5092
31.9094
29.9960
29.8445
28.7343
27.2034
25.8278
25.7603
23.3018
22.5111
21.6728
20.4190
18.2436
18.1555
15.2765
11.6392
7.1306
6.7652
-4.8760
[*1e6]
0
1
2
3
140
120
100
80
60
40
20
[ppm]

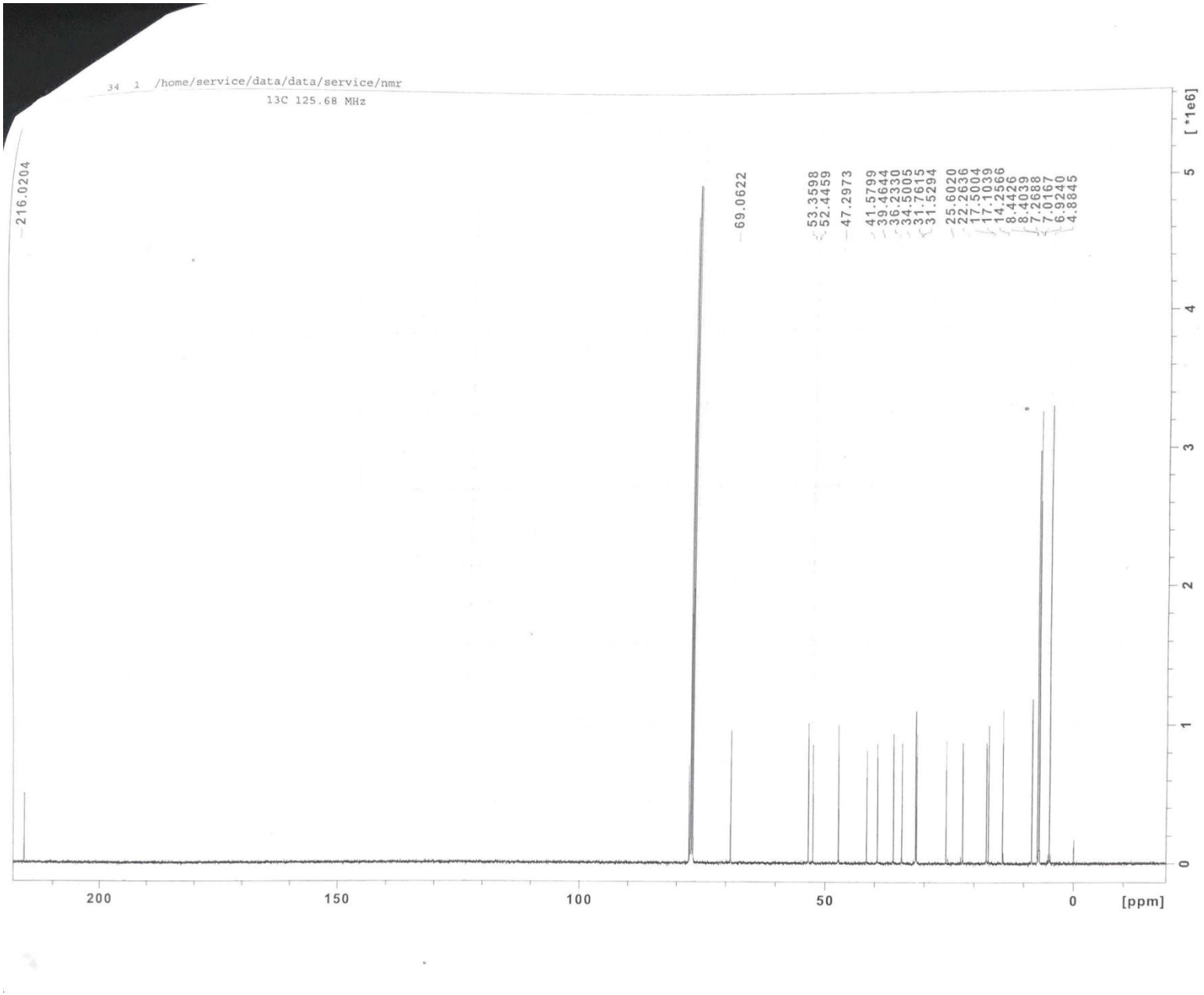

4 1 /home/service/data/data/service/nmr
1H 499.84 MHz
3.4434
3.4271
3.4253
3.4089
2.0401
2.0272
2.0237
2.0218
2.0187
2.0056
1.5506
1.5350
1.5201
1.5074
1.4923
1.4807
1.4775
1.4659
1.4510
1.4380
1.4234
1.4086
0.9650
0.9490
0.9332
0.8621
0.8471
0.8322
0.6099
0.5941
0.5783
0.5623
Br
OTES
[*1e6]
1.0
0.5
0.0
1.00
1.02
5.50
3.33
3.33
0.41
3
2
1
[ppm]

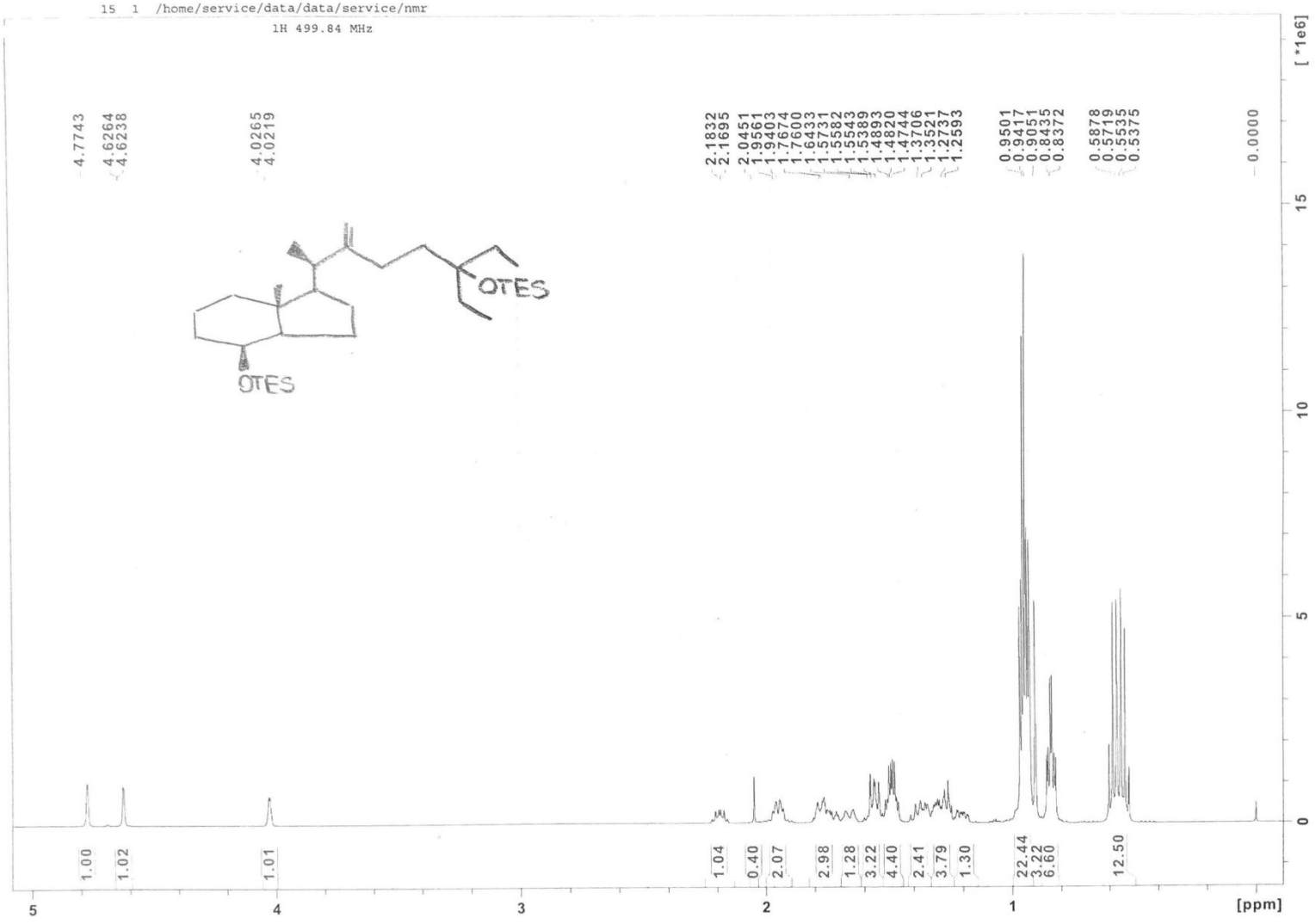

5 1 /home/service/data/data/service/nmr
13C 125.68 MHz
78.6513
77.2556
77.0013
76.7468
42.6596
31.6775
29.0125
8.3544
7.1730
6.8929
[*1e6]
60
40
20
0
60
40
20
[ppm]

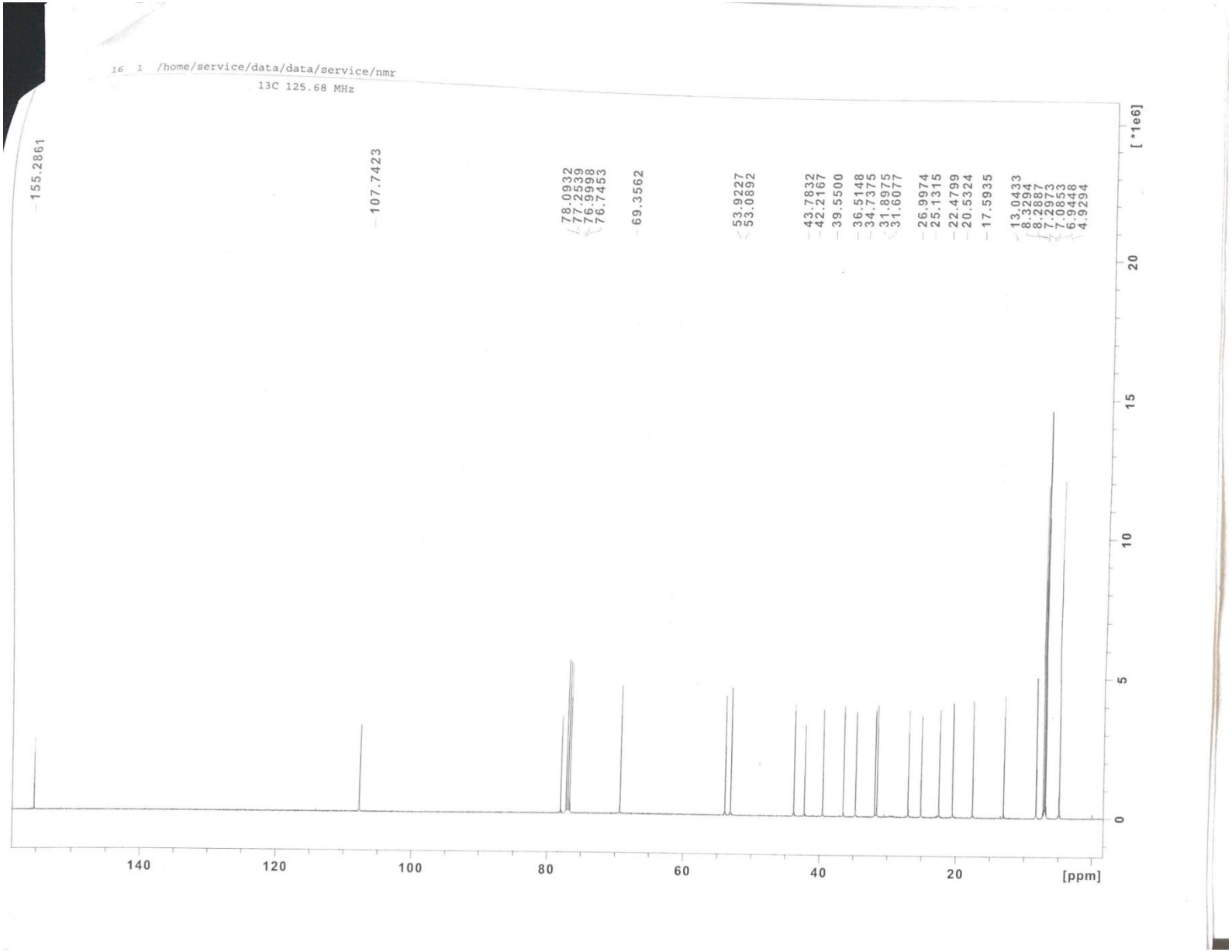

29 1 /home/service/data/data/service/nmr
1H 499.84 MHz
5.3030
4.0442
4.0396
3.7829
1.9530
1.9292
1.8271
1.7869
1.6954
1.6722
1.6670
1.5807
1.4714
1.4562
1.3749
1.3591
1.2562
1.2311
0.9633
0.9475
0.9315
0.9171
0.8454
0.8306
0.8165
0.8033
0.6021
0.5864
0.5781
0.5706
0.5624
0.5546
0.5465
0.0001
OH
OTES
OTES
1.00
0.99
1.02
2.18
1.14
8.94
7.28
4.10
22.05
9.43
12.52
5
4
3
2
1
[ppm]
[*1e6]

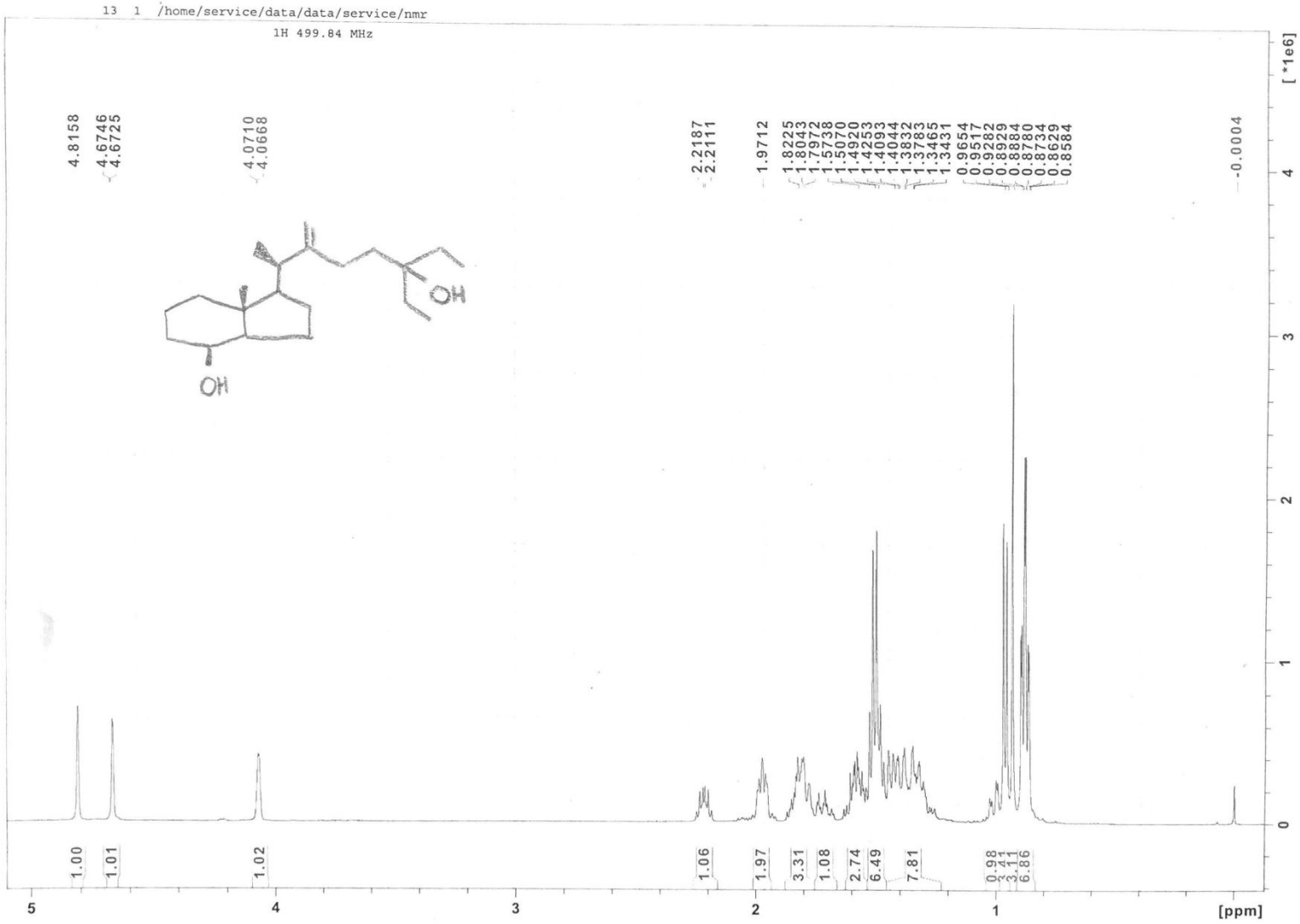

/home/service/data/data/service/nmr
13C 125.68 MHz
78.1261
73.5011
69.2979
53.4260
53.0363
52.5878
41.9373
40.3441
39.1307
35.4061
34.6165
31.7920
31.5740
29.0035
27.1292
22.8393
17.6912
13.8243
11.2339
8.4057
8.3574
7.2857
7.0416
6.9474
4.9087
[*1e6]
0
1
2
3
4
5
60
40
20
[ppm]

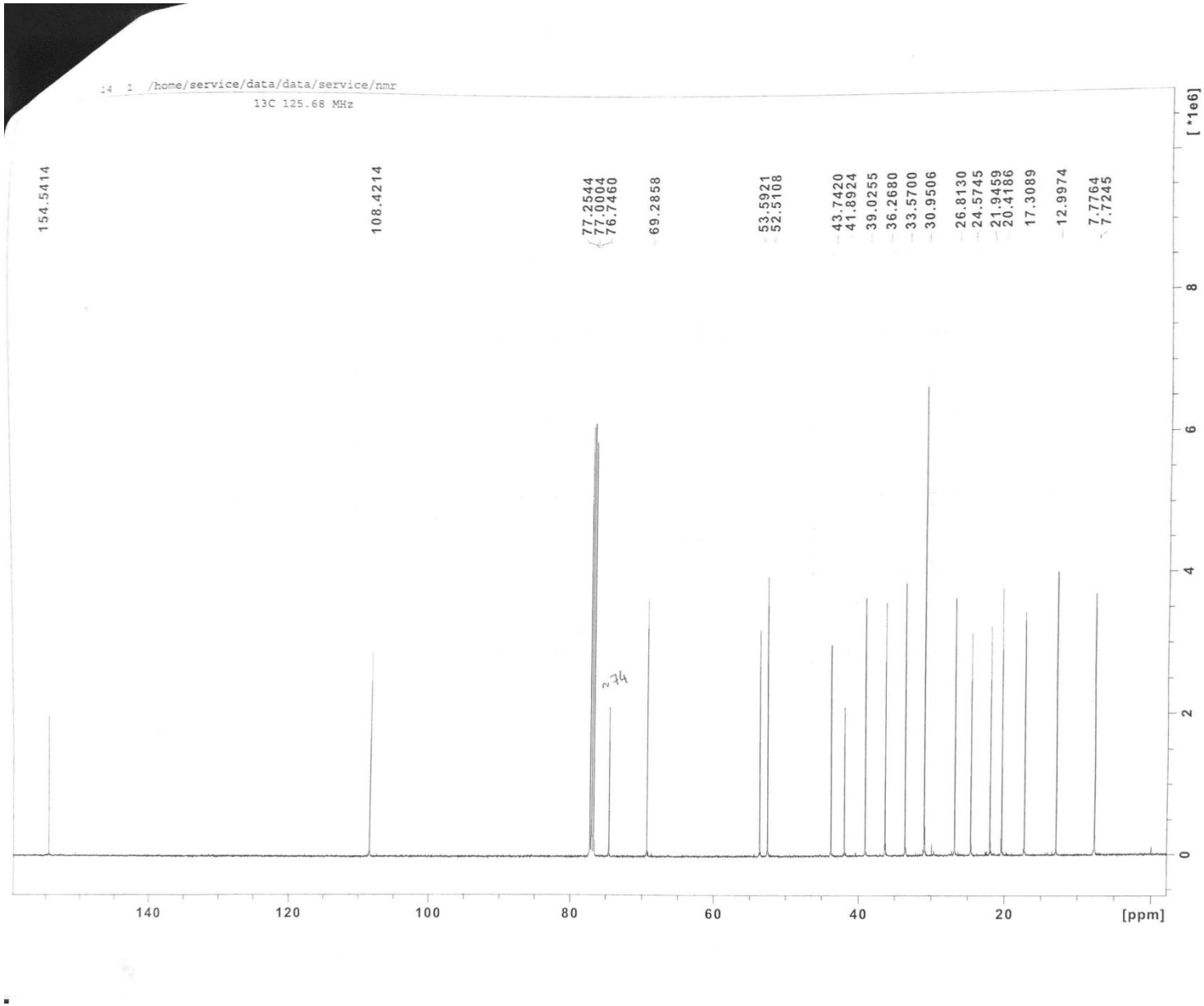

33 1 /home/service/data/data/service/nmr
1H 499.84 MHz
4.0275
4.0227
1.6848
1.6724
1.6645
1.5125
1.4974
1.4942
1.4336
1.3431
1.3217
1.3150
1.2762
1.2614
1.2569
0.9434
0.9382
0.8324
0.8267
0.5825
0.5667
0.5530
-0.0001
OTES
OTES
[*1e6]
0
1
2
3
4
5
1.00
1.17
4.99
6.78
3.08
2.50
3.34
19.33
3.01
6.62
12.53
4
3
2
1
[ppm]

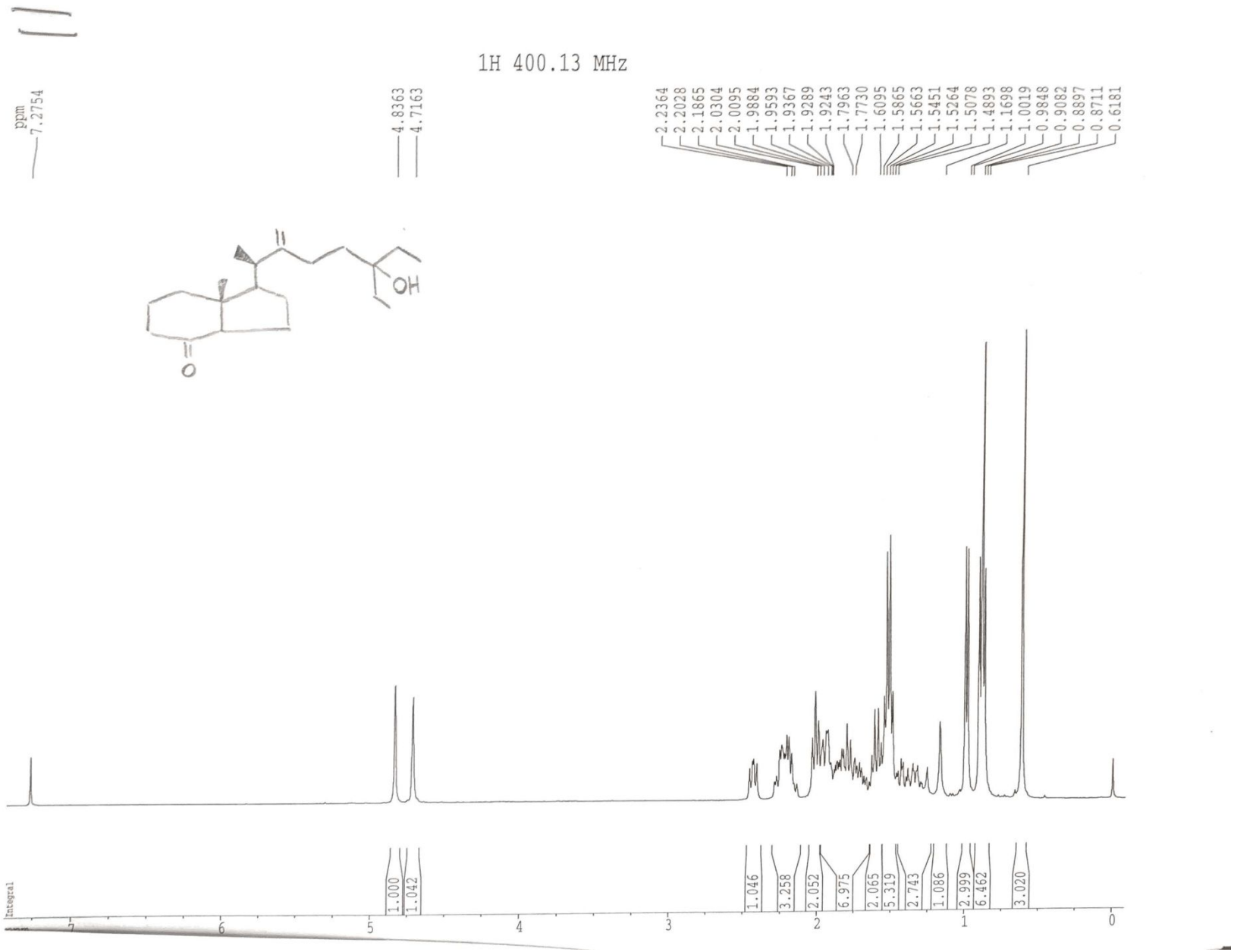

34 1 /home/service/data/data/service/nmr
13C 125.68 MHz
216.0204
69.0622
53.3598
52.4459
47.2973
41.5799
39.4644
36.2330
34.5005
31.7615
31.5294
25.6020
22.2636
17.5004
17.1039
14.2566
8.4426
8.4039
7.2688
7.0167
6.9240
4.8845
[*1e6]
0
1
2
3
4
5
200
150
100
50
0
[ppm]

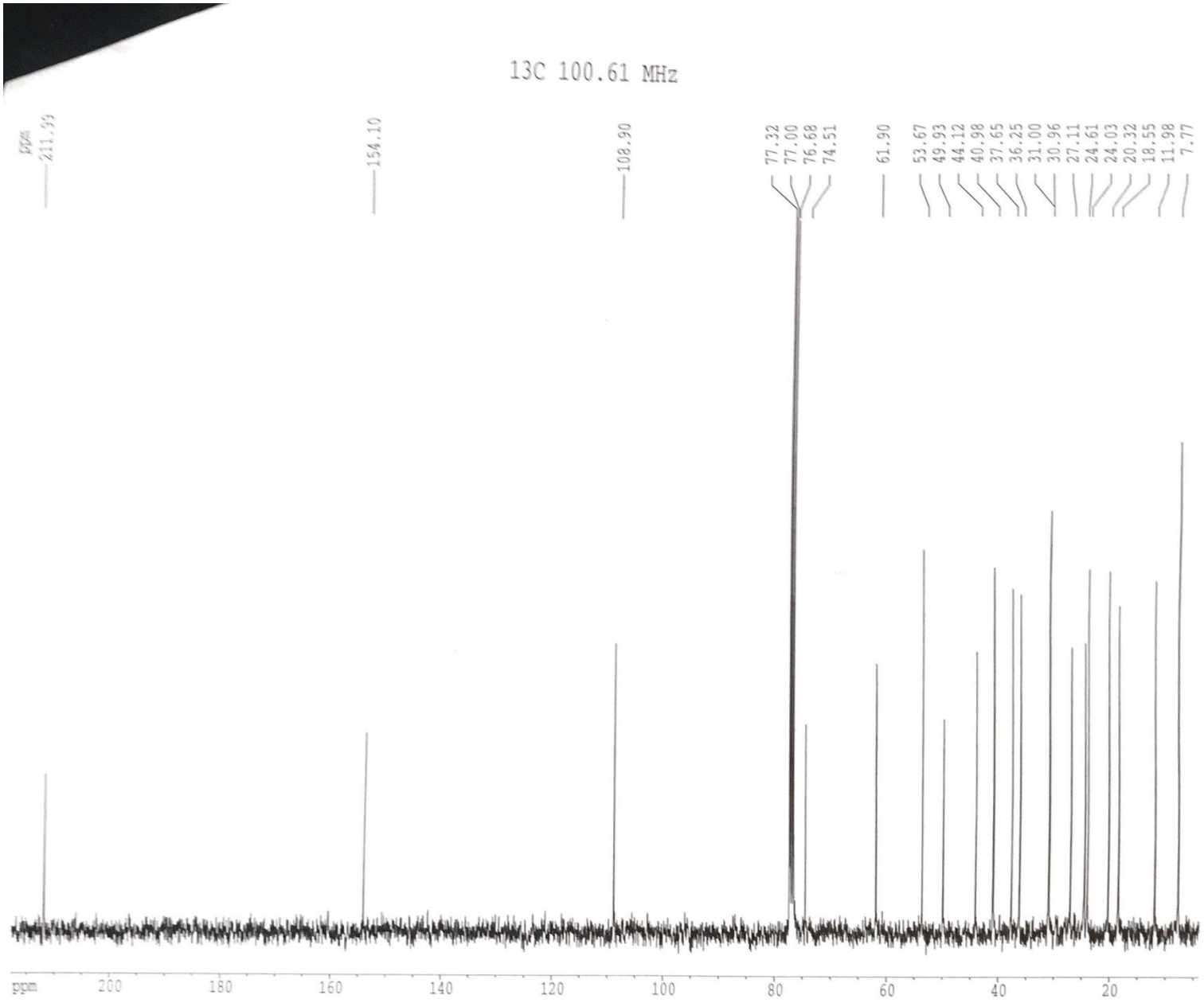

15 1 /home/service/data/data/service/nmr
1H 499.84 MHz
4.7743
4.6264
4.6238
4.0265
4.0219
2.1832
2.1695
2.0451
1.9561
1.9403
1.7674
1.7600
1.6433
1.5731
1.5582
1.5543
1.5389
1.4893
1.4820
1.4744
1.3706
1.3521
1.2737
1.2593
0.9501
0.9417
0.9051
0.8435
0.8372
0.5878
0.5719
0.5535
0.5375
0.0000
OTES
OTES
[*1e6]
15
10
5
0
1.00
1.02
1.01
1.04
0.40
2.07
2.98
1.28
3.22
4.40
2.41
3.79
1.30
22.44
3.22
6.60
12.50
5
4
3
2
1
[ppm]

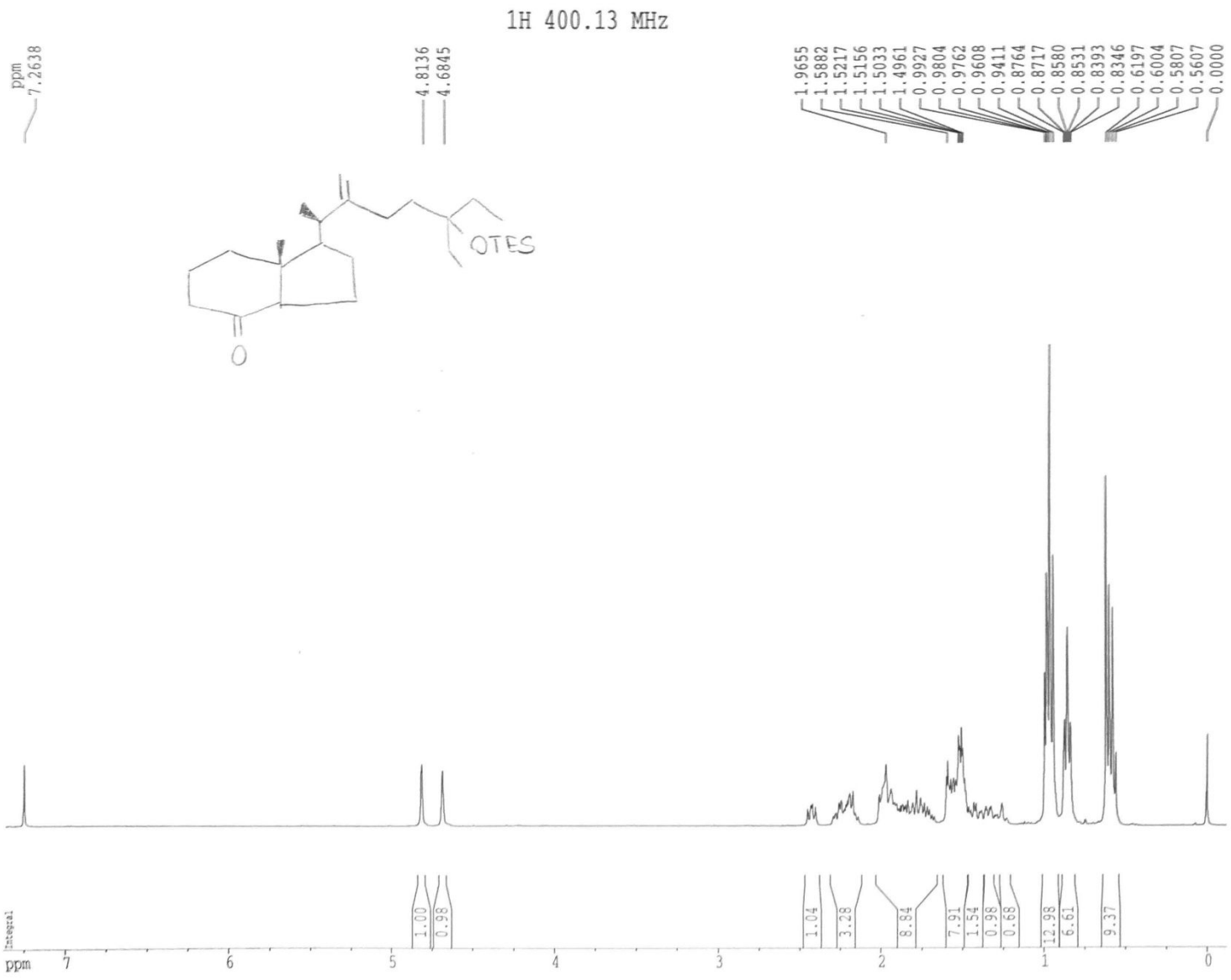

16 1 /home/service/data/data/service/nmr
13C 125.68 MHz
155.2861
107.7423
78.0932
77.2533
76.6998
76.7453
69.3562
53.9227
53.0892
43.7832
42.2167
39.5500
36.5148
34.7375
31.8975
31.6077
26.9974
25.1315
22.4799
20.5324
17.5935
13.0433
8.3294
8.2887
7.2973
7.0853
6.9448
4.9294
[*1e6]
0
5
10
15
20
140
120
100
80
60
40
20
[ppm]

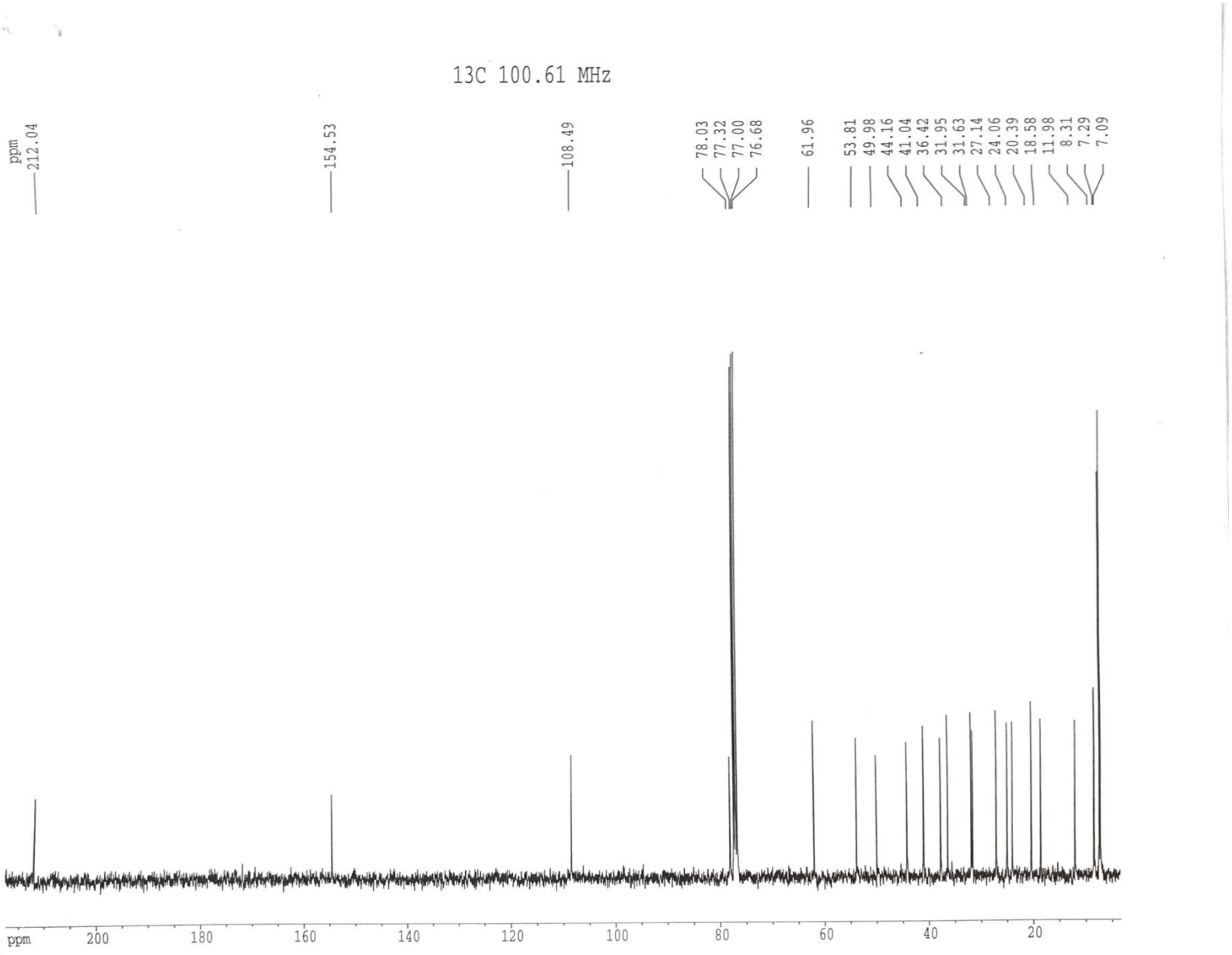

13 1 /home/service/data/data/service/nmr
1H 499.84 MHz
4.8158
4.6746
4.6725
4.0710
4.0668
2.2187
2.2111
1.9712
1.8225
1.8043
1.7972
1.5738
1.5070
1.4920
1.4253
1.4093
1.4044
1.3832
1.3783
1.3465
1.3431
0.9654
0.9517
0.9282
0.8929
0.8884
0.8780
0.8734
0.8629
0.8584
-0.0004
OH
OH
1.00
1.01
1.02
1.06
1.97
3.31
1.08
2.74
6.49
7.81
0.98
2.41
3.11
6.86
5
4
3
2
1
[ppm]
[*1e6]
0
1
2
3
4

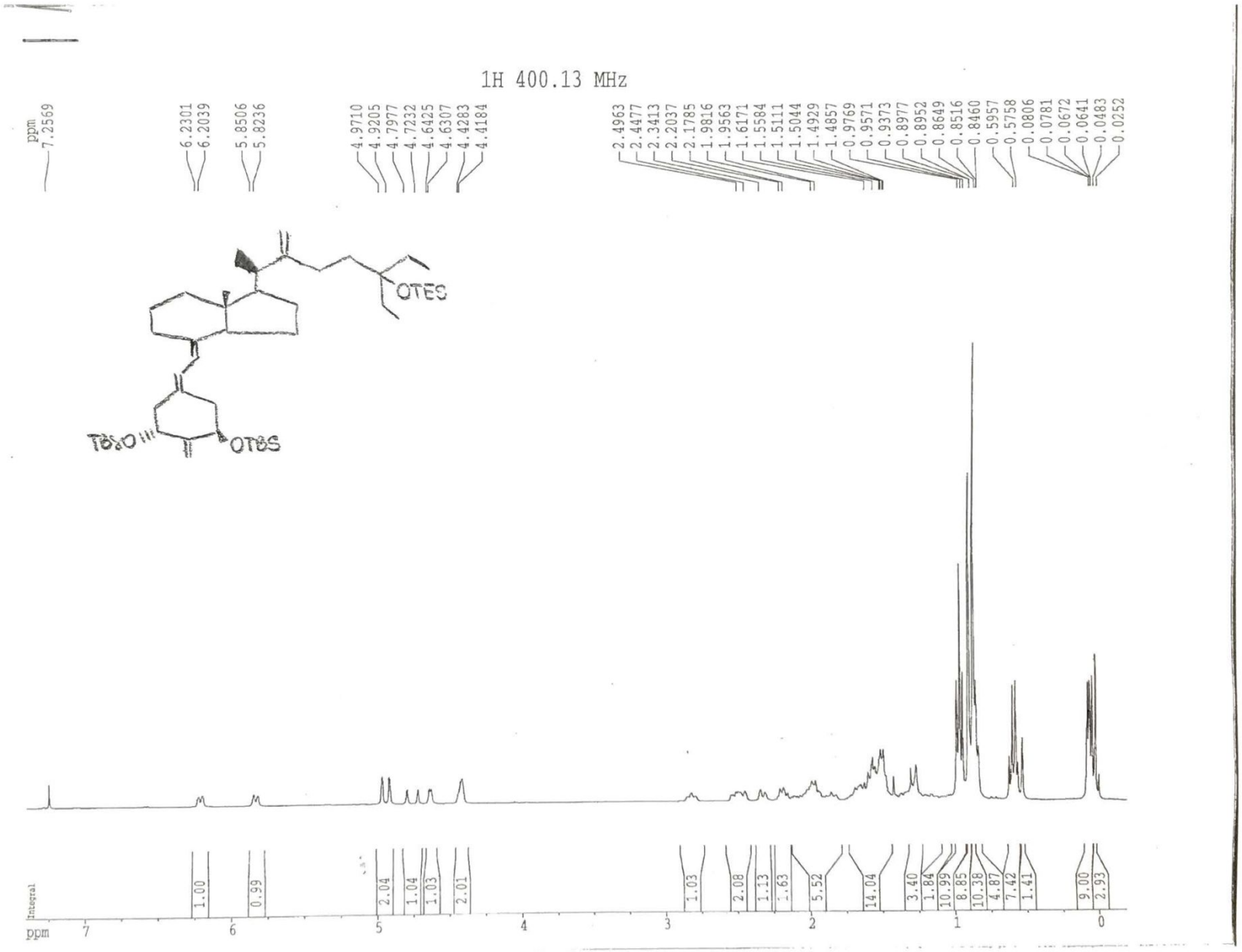

14 1 /home/service/data/data/service/nmr
13C 125.68 MHz
154.5414
108.4214
77.2544
77.0004
76.7460
69.2858
53.5921
52.5108
43.7420
41.8924
39.0255
36.2680
33.5700
30.9506
26.8130
24.5745
21.9459
20.4186
17.3089
12.9974
7.7764
7.7245
~74
[*1e6]
0
2
4
6
8
140
120
100
80
60
40
20
[ppm]

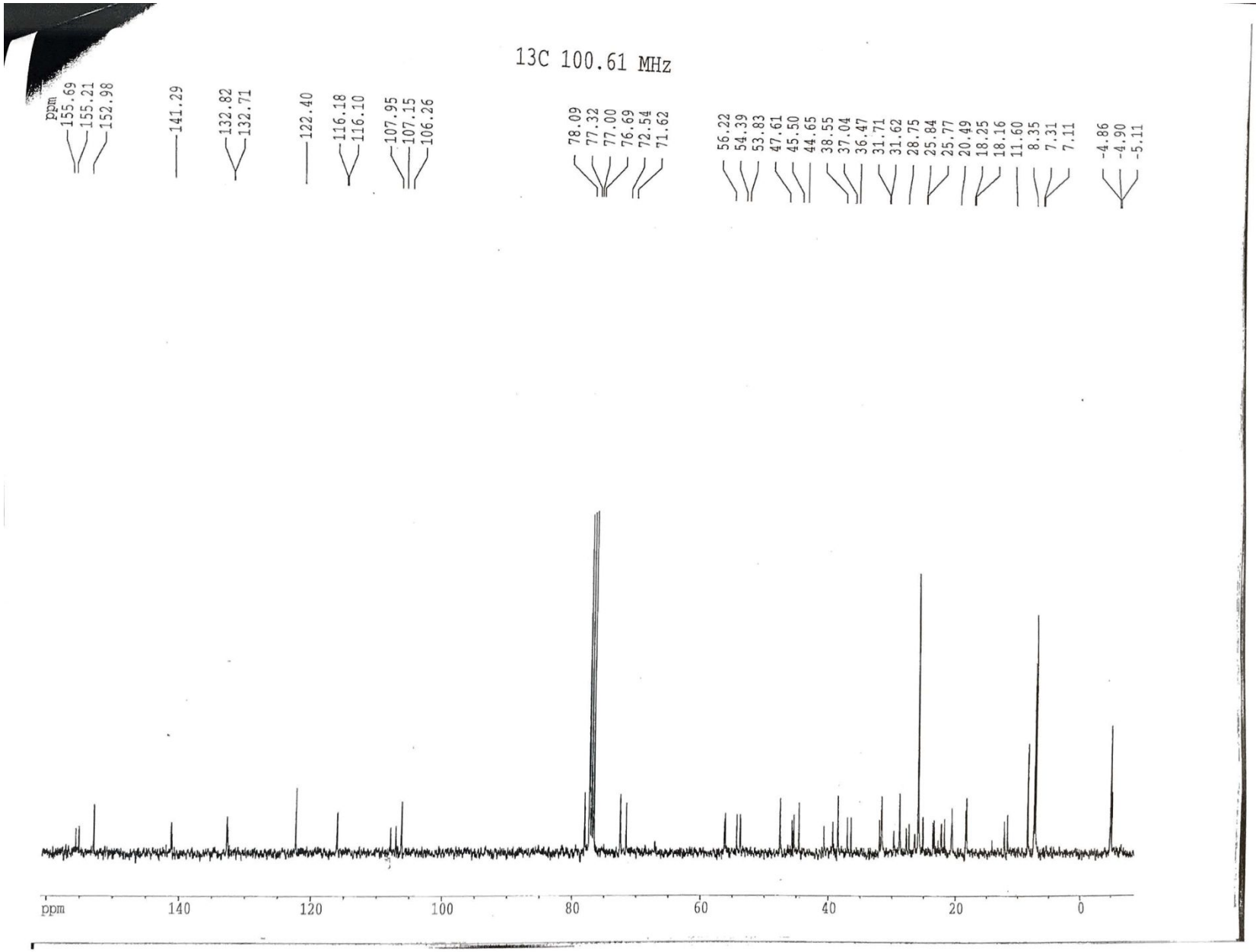

1H 400.13 MHz
ppm
7.2754
4.8363
4.7163
2.2364
2.2028
2.1865
2.0304
2.0095
1.9884
1.9593
1.9367
1.9289
1.9243
1.7963
1.7730
1.6095
1.5865
1.5663
1.5451
1.5264
1.5078
1.4893
1.1698
1.0019
0.9848
0.9082
0.8897
0.8711
0.6181
OH
O
Integral
1.000
1.042
1.046
3.258
2.052
6.975
2.065
5.319
2.743
1.086
2.999
6.462
3.020
7
6
5
4
3
2
1
0

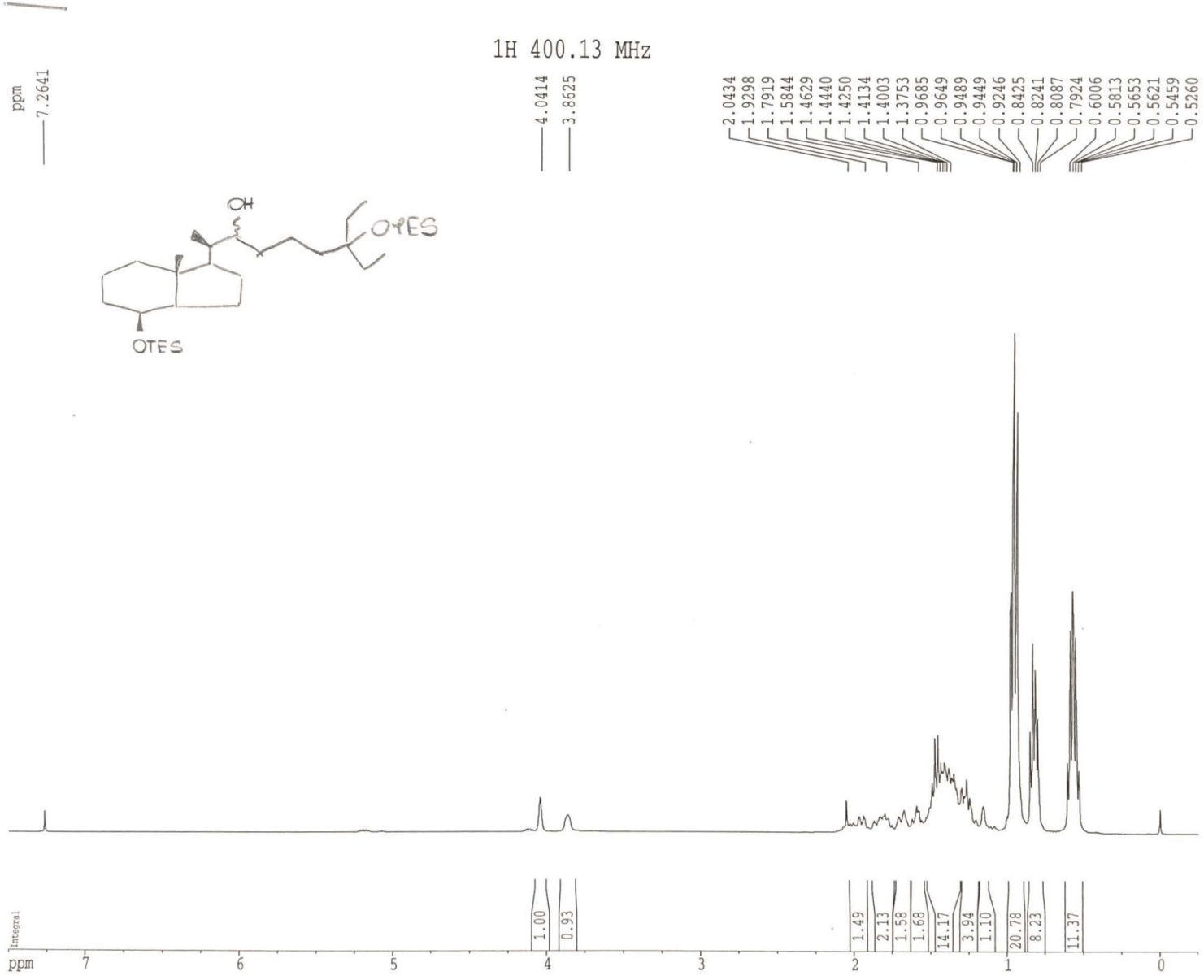

13C 100.61 MHz
ppm
211.33
154.10
108.90
77.32
77.00
76.68
74.51
61.90
53.67
49.93
44.12
40.98
37.65
36.25
31.00
30.96
27.11
24.61
24.03
20.32
18.55
11.98
7.77
ppm
200
180
160
140
120
100
80
60
40
20

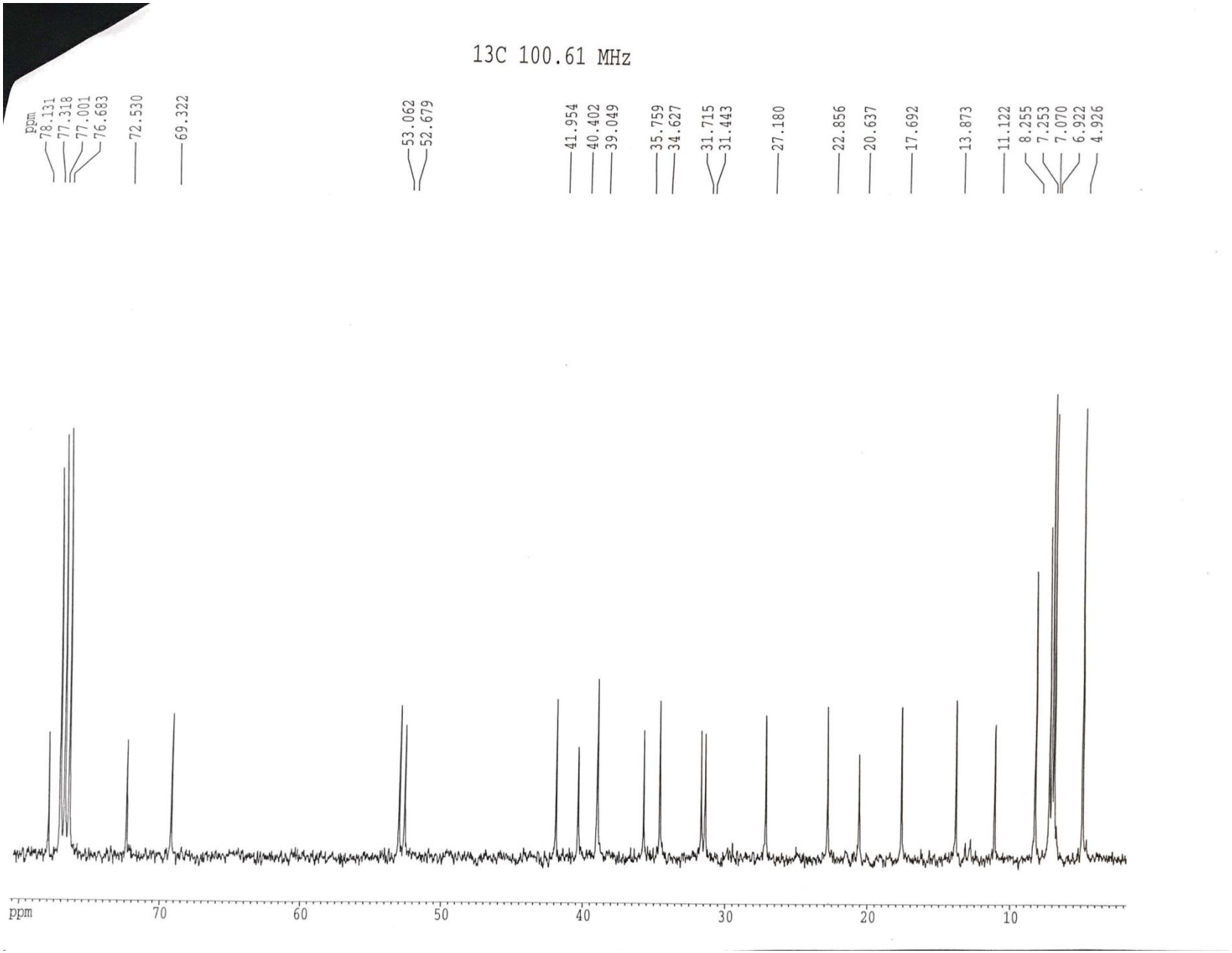

1H 400.13 MHz
ppm
7.2638
4.8136
4.6845
1.9655
1.5882
1.5217
1.5156
1.5033
1.4961
0.9927
0.9804
0.9762
0.9608
0.9411
0.8764
0.8717
0.8580
0.8531
0.8393
0.8346
0.6197
0.6004
0.5807
0.5607
0.0000
OTES
O
Integral
1.00
0.98
1.04
3.28
8.84
7.91
1.54
0.98
0.68
12.98
6.61
9.37
ppm
7
6
5
4
3
2
1
0

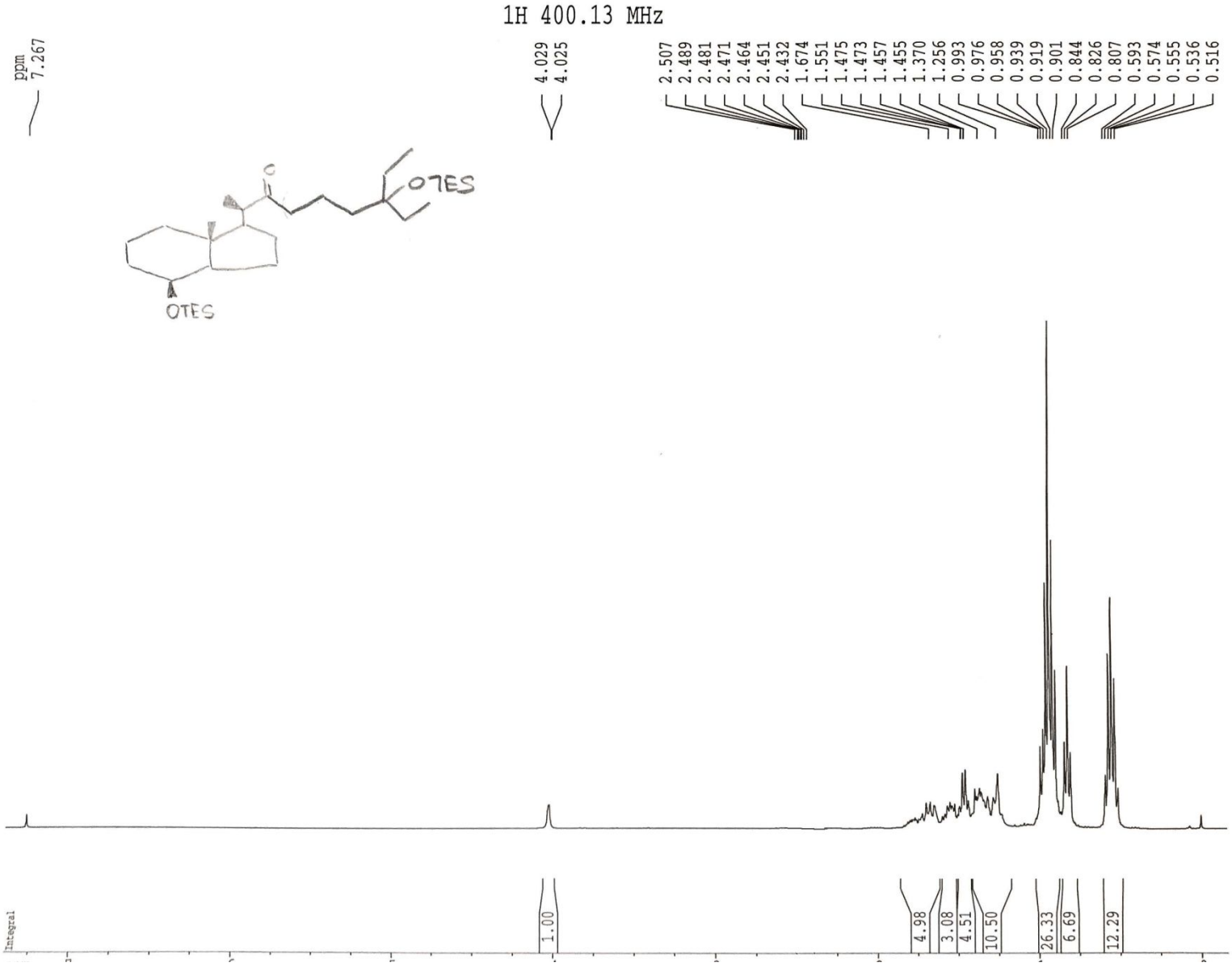

13C 100.61 MHz
ppm
212.04
154.53
108.49
78.03
77.32
77.00
76.68
61.96
53.81
49.98
44.16
41.04
36.42
31.95
31.63
27.14
24.06
20.39
18.58
11.98
8.31
7.29
7.09
ppm
200
180
160
140
120
100
80
60
40
20

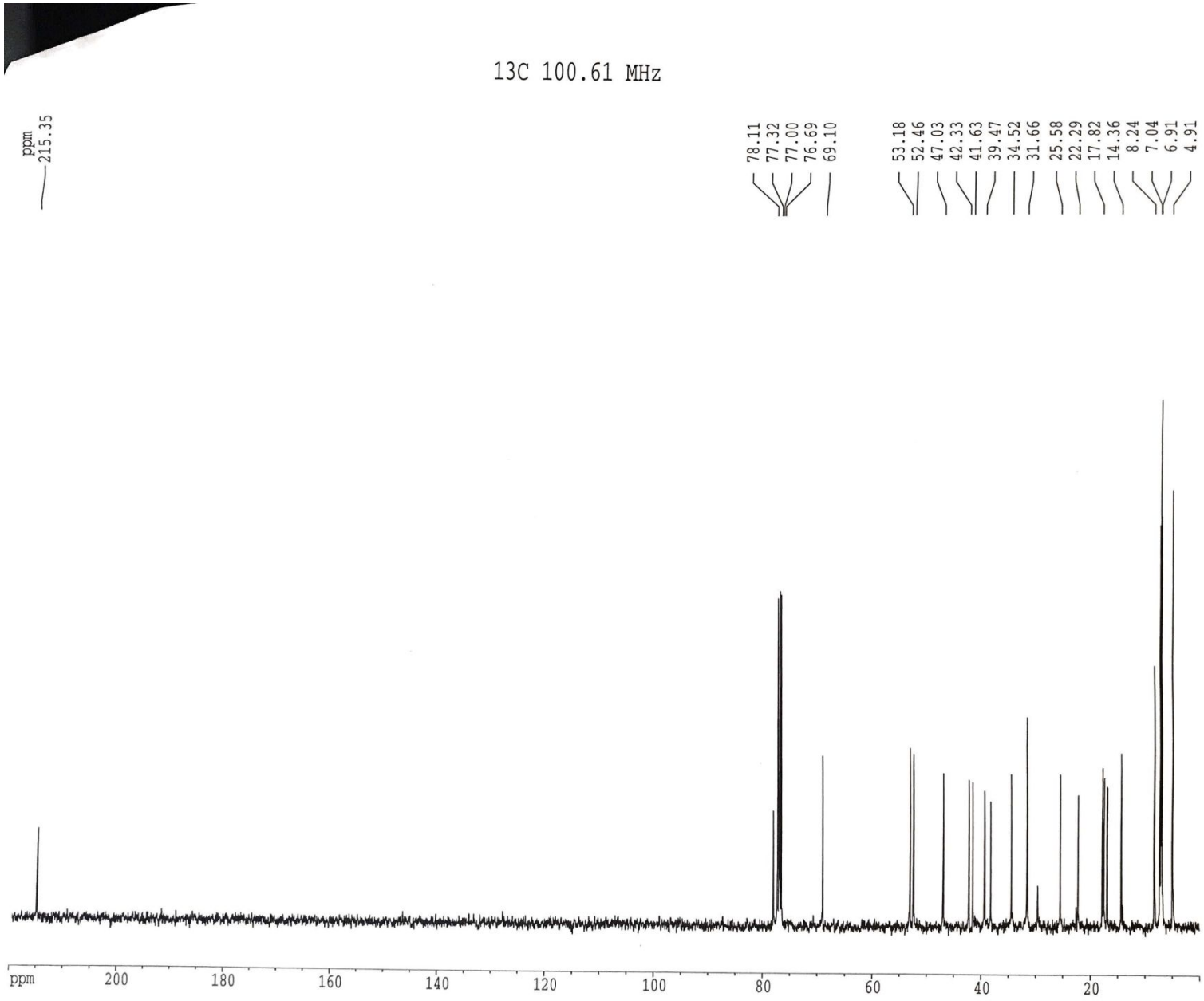

1H 400.13 MHz
ppm
7.2569
6.2301
6.2039
5.8506
5.8236
4.9710
4.9205
4.7977
4.7232
4.6425
4.6307
4.4283
4.4184
2.4963
2.4477
2.3413
2.2037
2.1785
1.9816
1.9563
1.6171
1.5584
1.5111
1.5044
1.4929
1.4857
0.9769
0.9571
0.9373
0.8977
0.8952
0.8649
0.8516
0.8460
0.5957
0.5758
0.0806
0.0781
0.0672
0.0641
0.0483
0.0252
OTES
TBSO
OTBS
Integral
1.00
0.99
2.04
1.04
1.03
2.01
1.03
2.08
1.13
.63
5.52
14.04
3.40
1.84
10.99
8.85
10.38
4.87
7.42
1.41
9.00
2.93
ppm
7
6
5
4
3
2
1
0

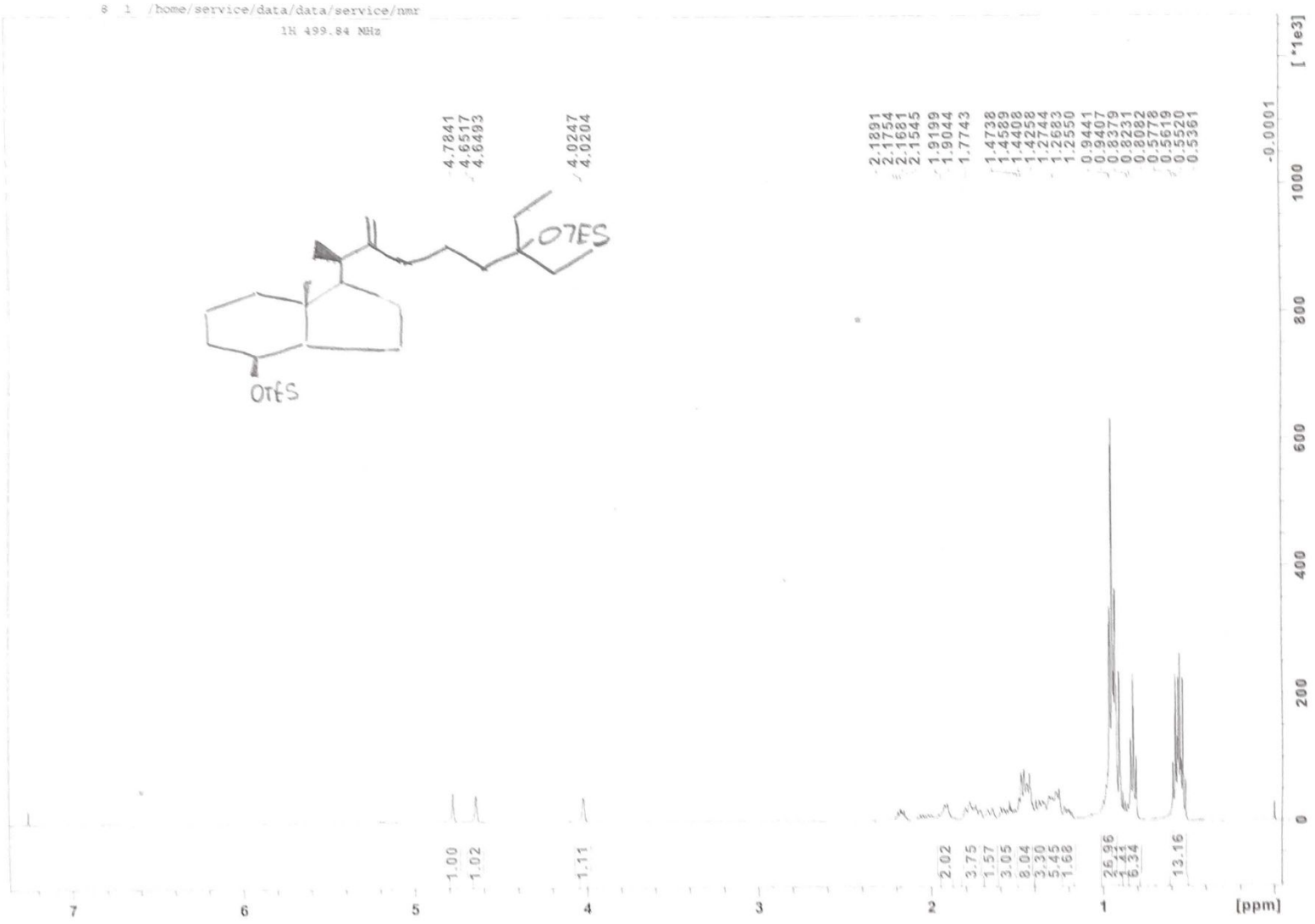

13C 100.61 MHz
ppm
155.69
155.21
152.98
141.29
132.82
132.71
122.40
116.18
116.10
107.95
107.15
106.26
78.09
77.32
77.00
76.69
72.54
71.62
56.22
54.39
53.83
47.61
45.50
44.65
38.55
37.04
36.47
31.71
31.62
28.75
25.84
25.77
20.49
18.25
18.16
11.60
8.35
7.31
7.11
-4.86
-4.90
-5.11
ppm
140
120
100
80
60
40
20
0

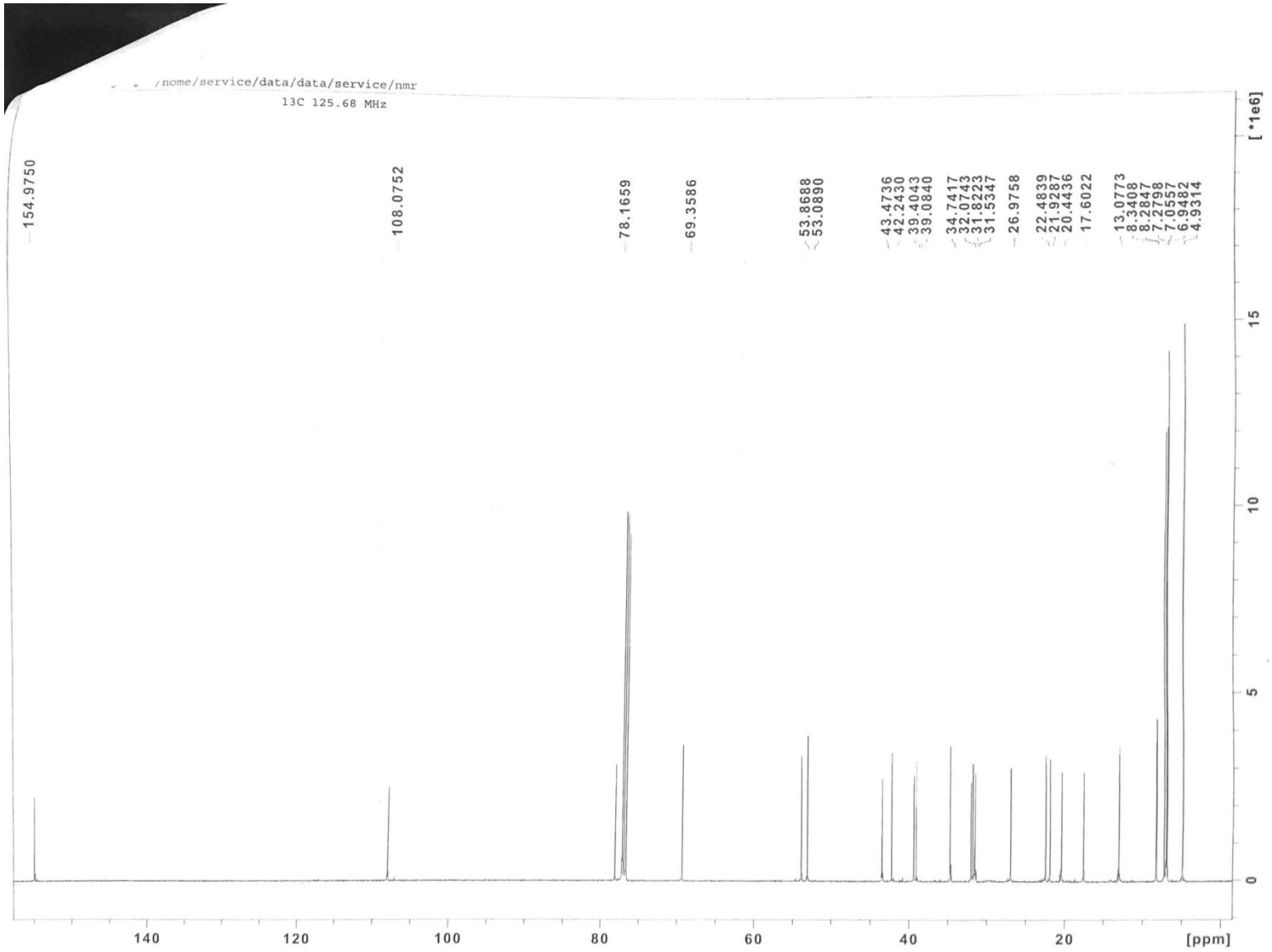

1H 400.13 MHz
ppm
7.2641
4.0414
3.8625
2.0434
1.9298
1.7919
1.5844
1.4629
1.4440
1.4250
1.4134
1.4003
1.3753
0.9685
0.9649
0.9489
0.9449
0.9246
0.8425
0.8241
0.8087
0.7924
0.6006
0.5813
0.5653
0.5621
0.5459
0.5260
OH
OTES
OTES
Integral
1.00
0.93
1.49
2.13
1.58
1.68
14.17
3.94
1.10
20.78
8.23
11.37
ppm
7
6
5
4
3
2
1
0

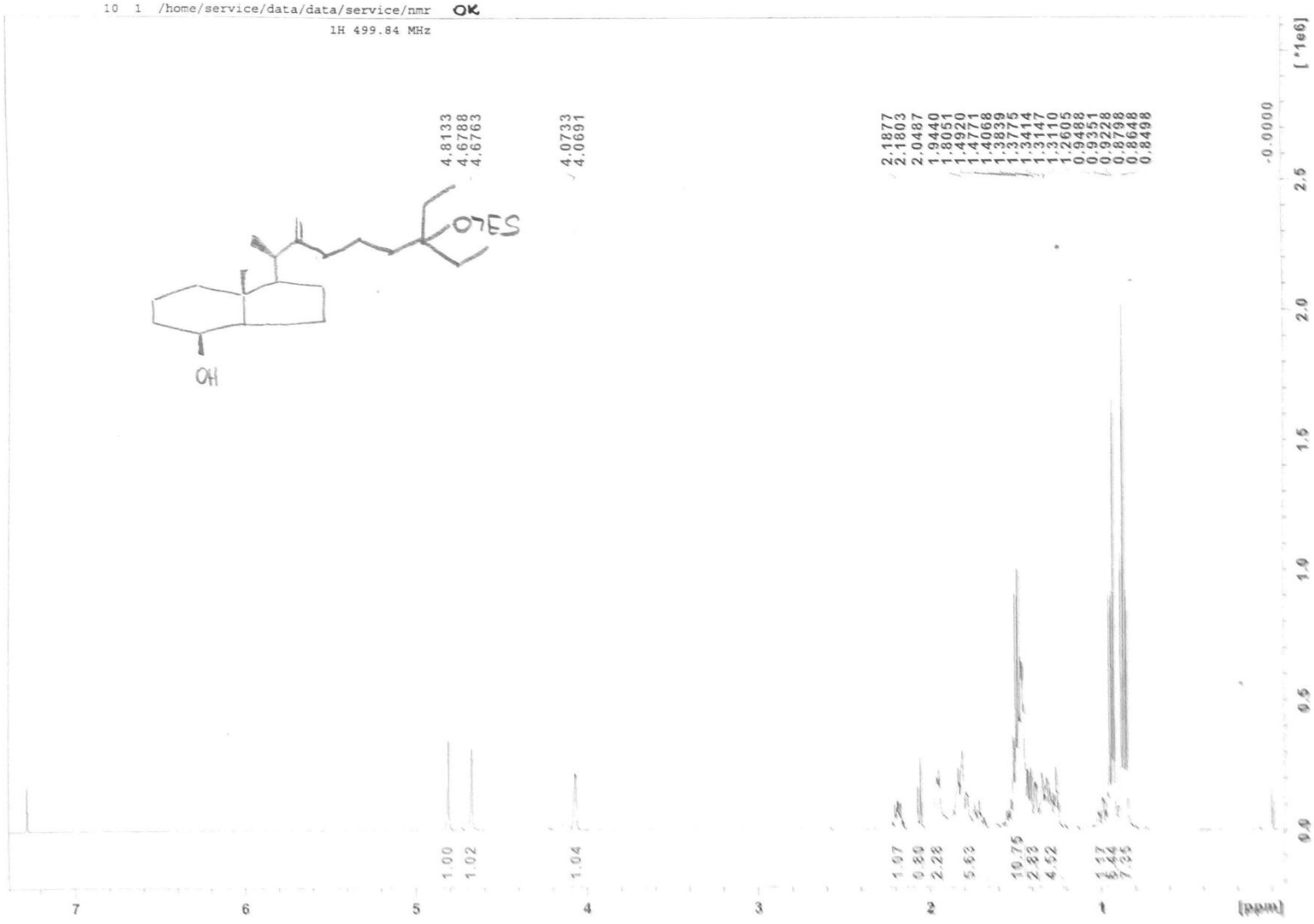

13C 100.61 MHz
ppm
78.131
77.318
77.001
76.683
72.530
69.322
53.062
52.679
41.954
40.402
39.049
35.759
34.627
31.715
31.443
27.180
22.856
20.637
17.692
13.873
11.122
8.255
7.253
7.070
6.922
4.926
ppm
70
60
50
40
30
20
10

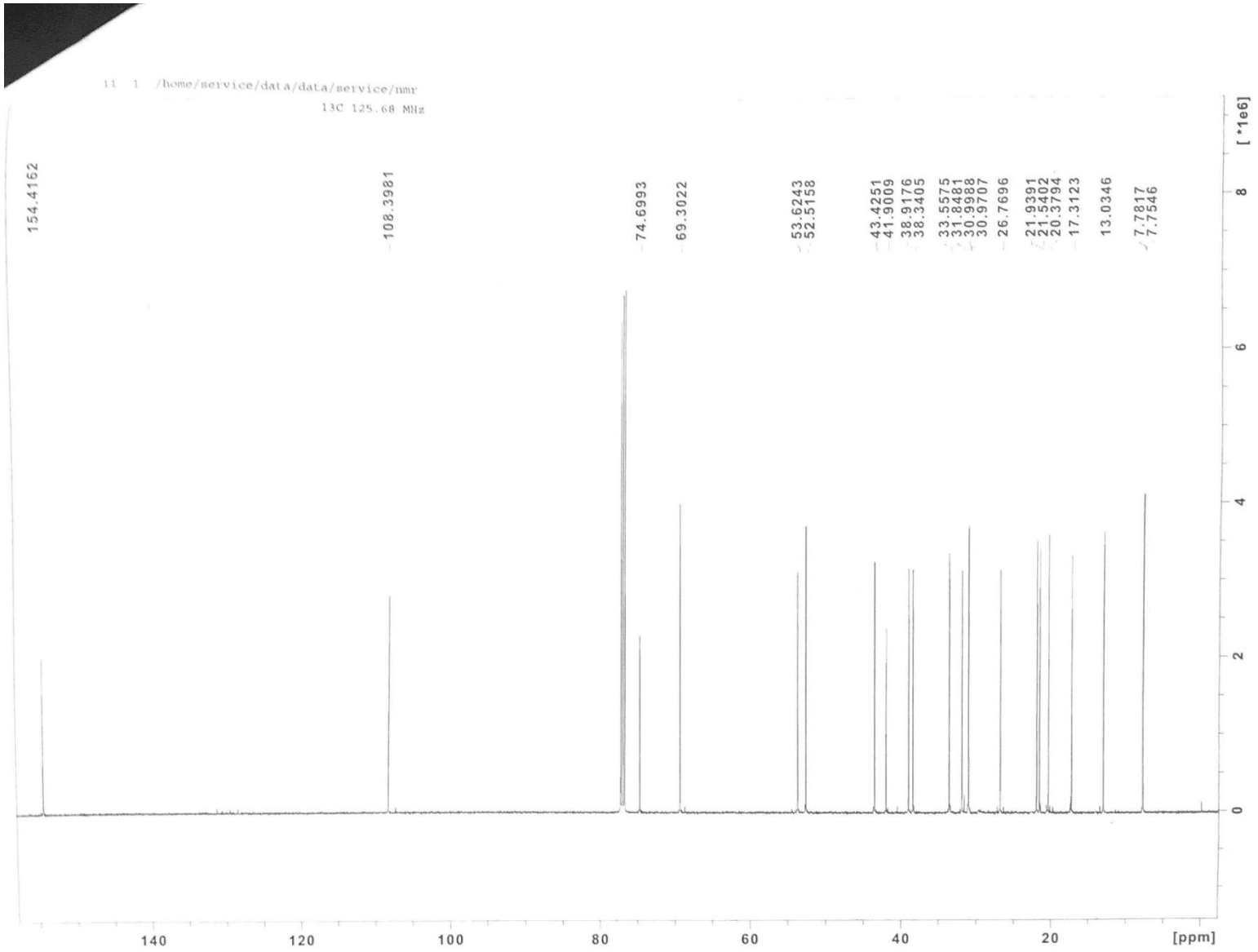

1H 400.13 MHz
ppm
7.267
4.029
4.025
2.507
2.489
2.481
2.471
2.464
2.451
2.432
1.674
1.551
1.475
1.473
1.457
1.455
1.370
1.256
0.993
0.976
0.958
0.939
0.919
0.901
0.844
0.826
0.807
0.593
0.574
0.555
0.536
0.516
OTES
OTES
Integral
1.00
4.98
3.08
4.51
10.50
26.33
6.69
12.29

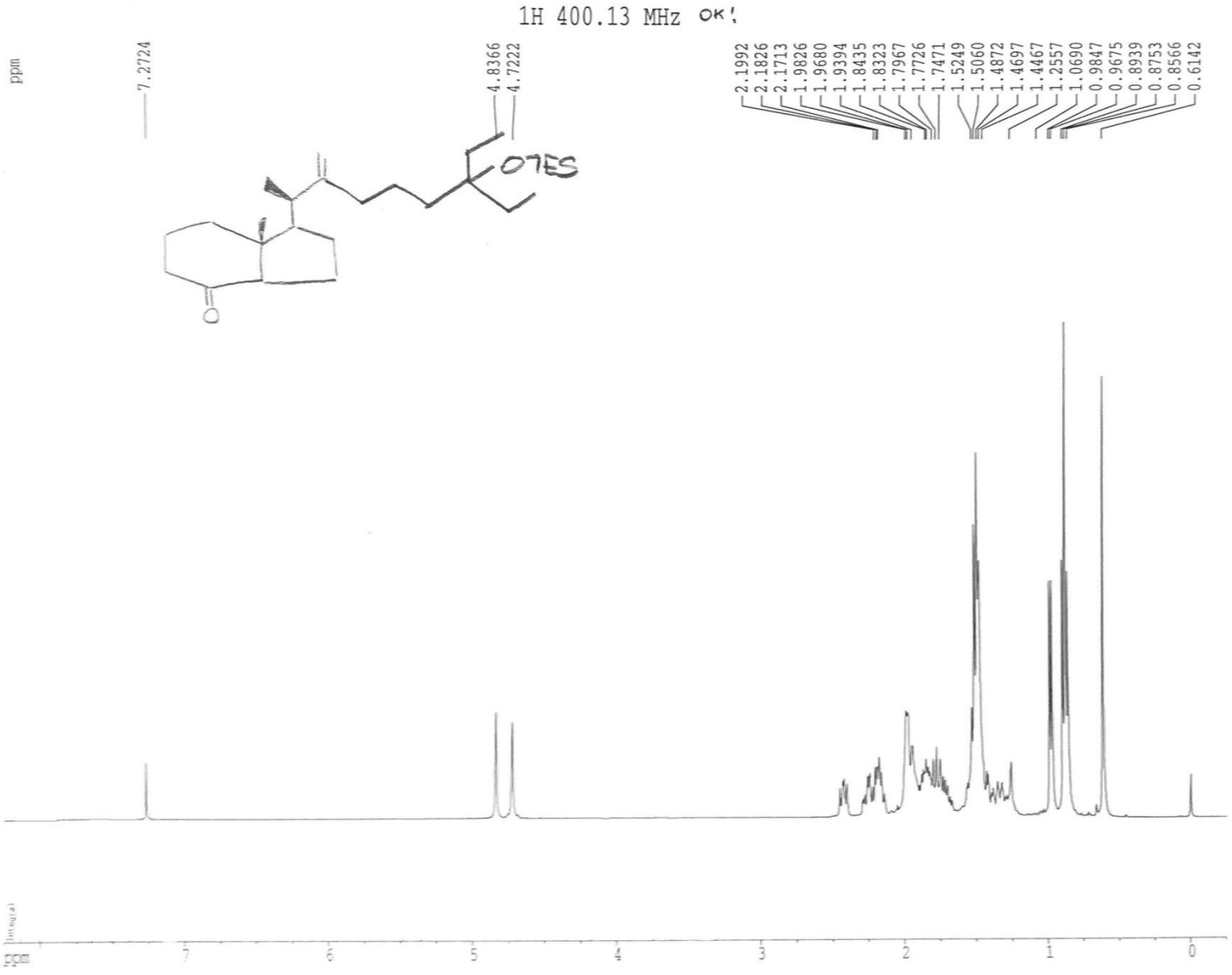

13C 100.61 MHz
ppm
215.35
78.11
77.32
77.00
76.69
69.10
53.18
52.46
47.03
42.33
41.63
39.47
34.52
31.66
25.58
22.29
17.82
14.36
8.24
7.04
6.91
4.91
ppm
200
180
160
140
120
100
80
60
40
20

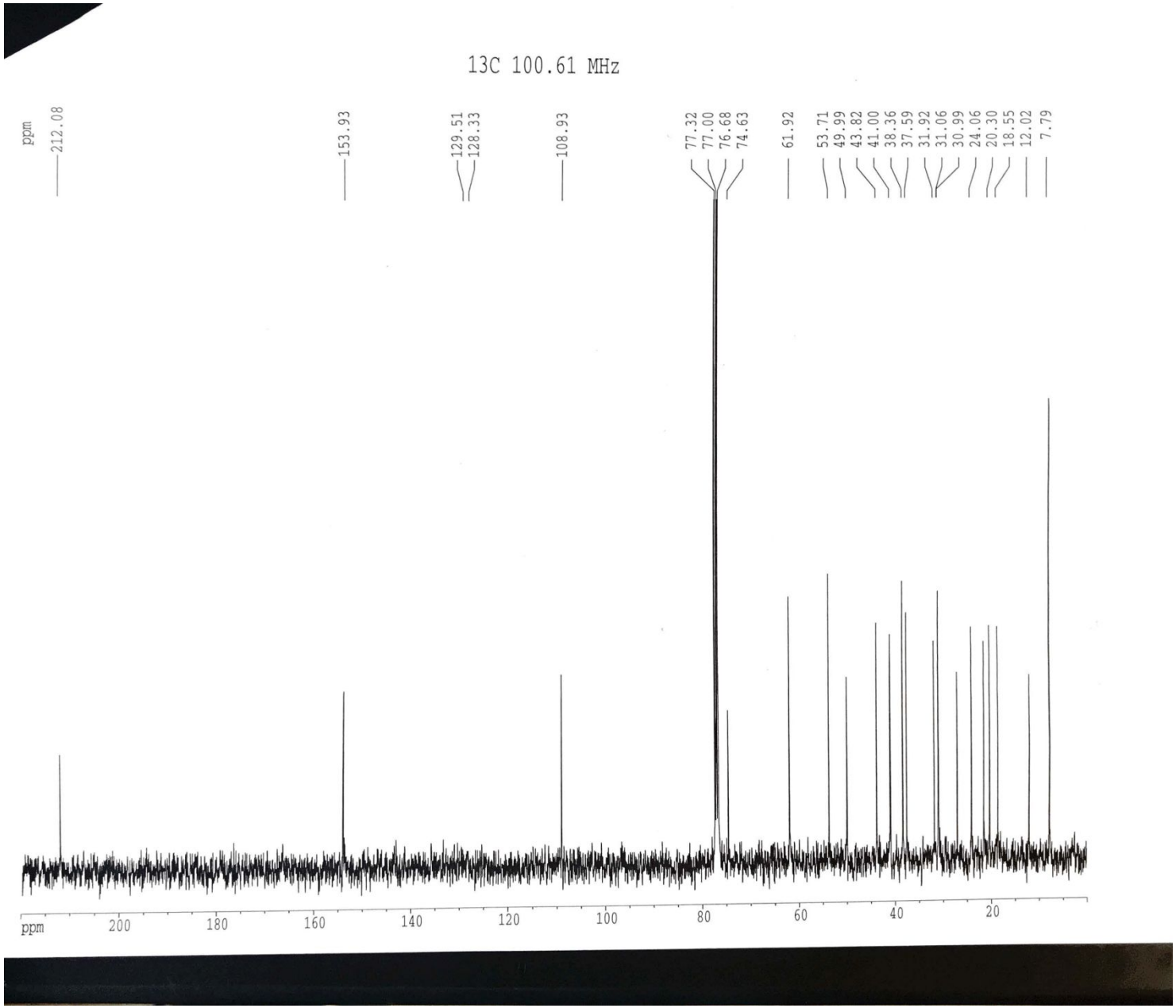

8 1 /home/service/data/data/service/nmr
1H 499.84 MHz
4.7841
4.6517
4.6493
4.0247
4.0204
2.1891
2.1754
2.1681
2.1545
1.9199
1.9044
1.7743
1.4738
1.4589
1.4408
1.4258
1.2744
1.2683
1.2550
0.9441
0.9407
0.8379
0.8231
0.8082
0.5778
0.5619
0.5520
0.5361
OTES
OTES
1.00
1.02
1.11
2.02
3.75
1.57
3.05
8.04
3.30
5.45
1.68
26.96
1.44
6.34
13.16
[ppm]
[*1e3]

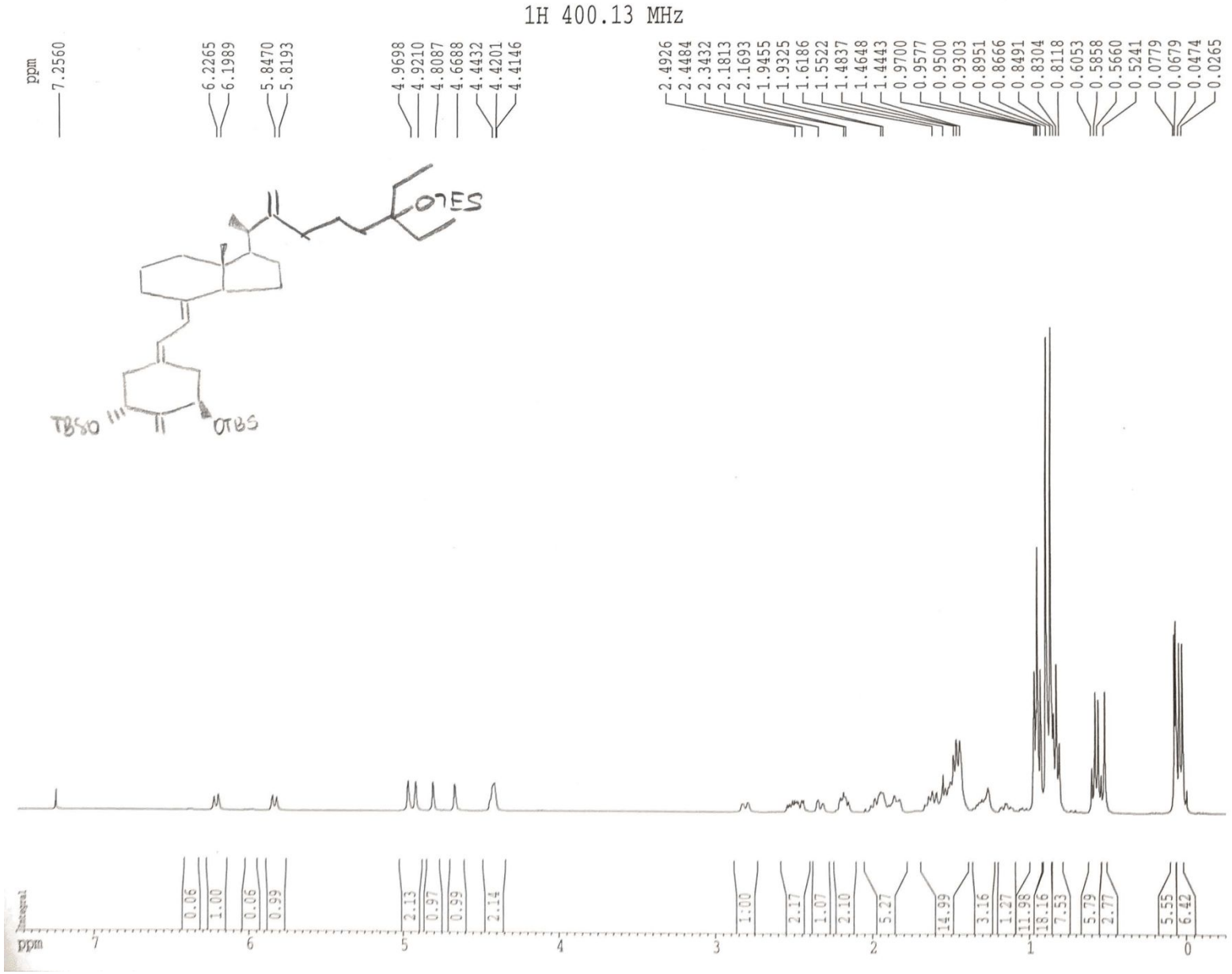

/nome/service/data/data/service/nmr
13C 125.68 MHz
154.9750
108.0752
78.1659
69.3586
53.8688
53.0890
43.4736
42.2430
39.4043
39.0840
34.7417
32.0743
31.8223
31.5347
26.9758
22.4839
21.9287
20.4436
17.6022
13.0773
8.3408
8.3047
7.2798
7.0557
6.9482
4.9314
[*1e6]
0
5
10
15
140
120
100
80
60
40
20
[ppm]

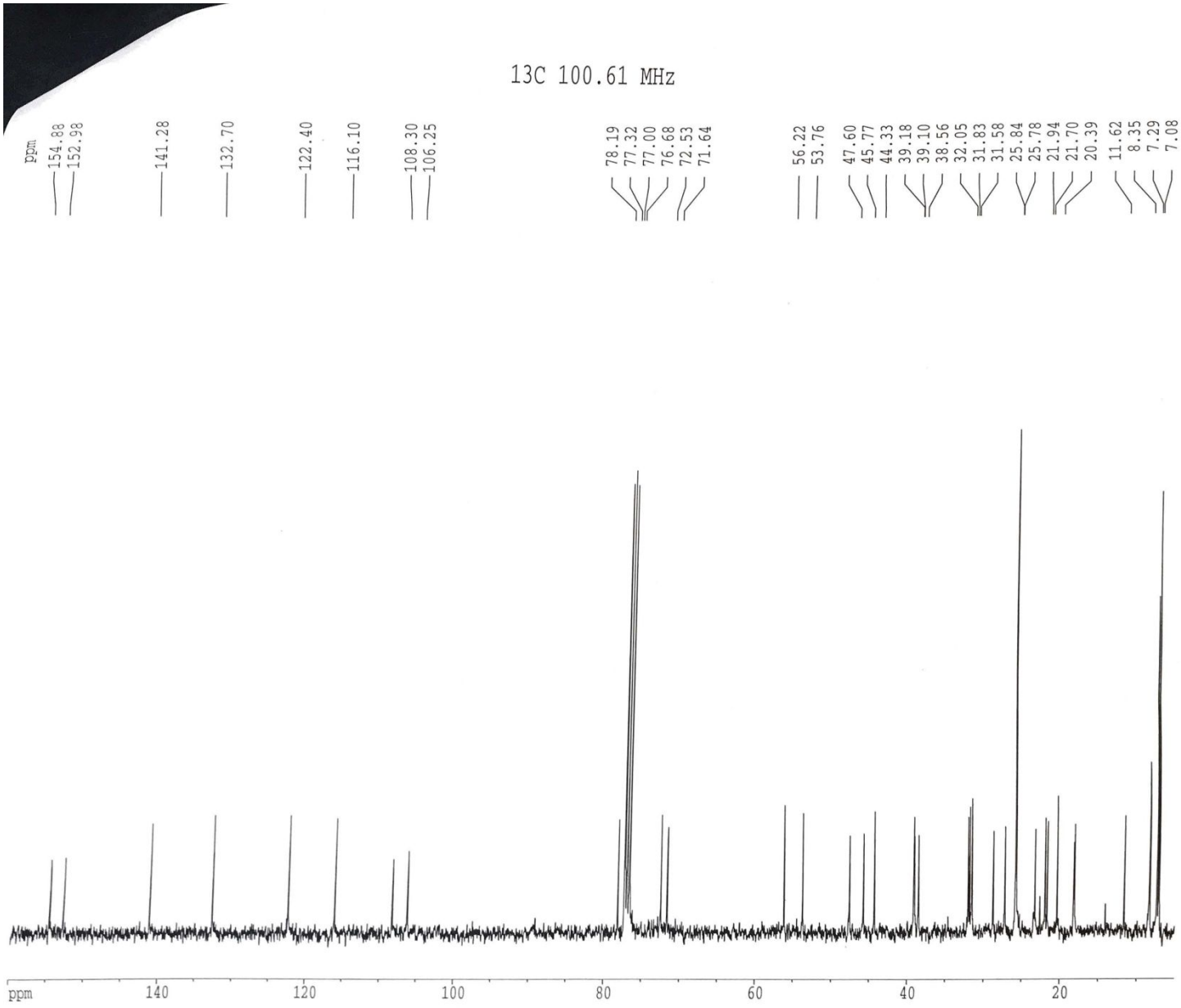

10 1 /home/service/data/data/service/nmr OK
1H 499.84 MHz
4.8133
4.6788
4.6763
4.0733
4.0691
2.1877
2.1803
2.0487
1.9440
1.8051
1.4920
1.4771
1.4068
1.3839
1.3775
1.3414
1.3147
1.3110
1.2605
0.9488
0.9351
0.9228
0.8798
0.8648
0.8498
OTES
OH
-0.0000
[*1e6]
2.5
2.0
1.5
1.0
0.5
0.0
1.00
1.02
1.04
1.97
0.89
2.28
5.63
10.75
2.83
4.52
1.17
1.44
7.35
7
6
5
4
3
2
1
[ppm]

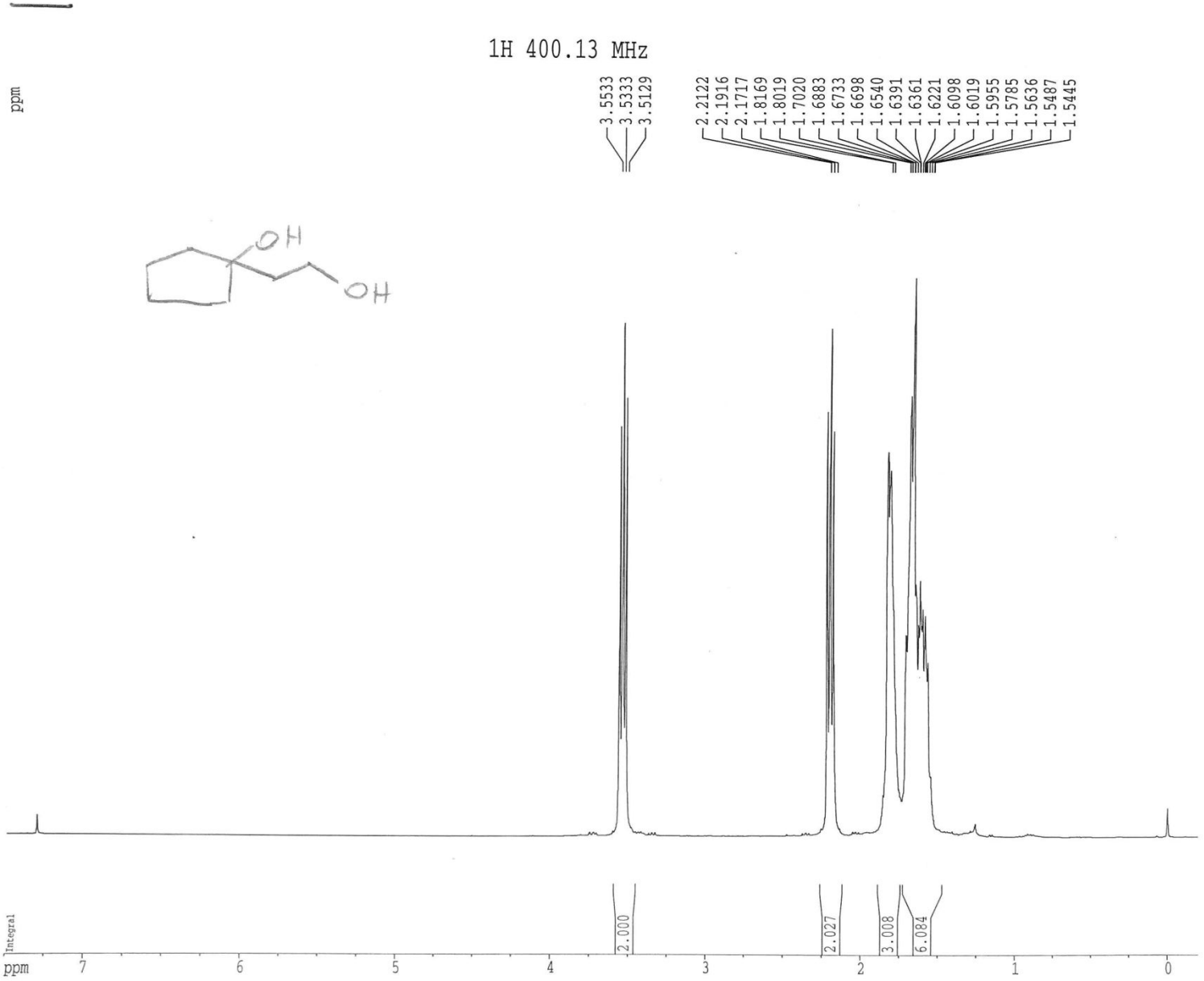

11 1 /home/service/data/data/service/nmr
13C 125.68 MHz
154.4162
108.3981
74.6993
69.3022
53.6243
52.5158
43.4251
41.9009
38.9176
38.3405
33.5575
31.8481
30.9988
30.9707
26.7696
21.9391
21.5402
20.3794
17.3123
13.0346
7.7817
7.7546
[*1e6]
0
2
4
6
8
140
120
100
80
60
40
20
[ppm]

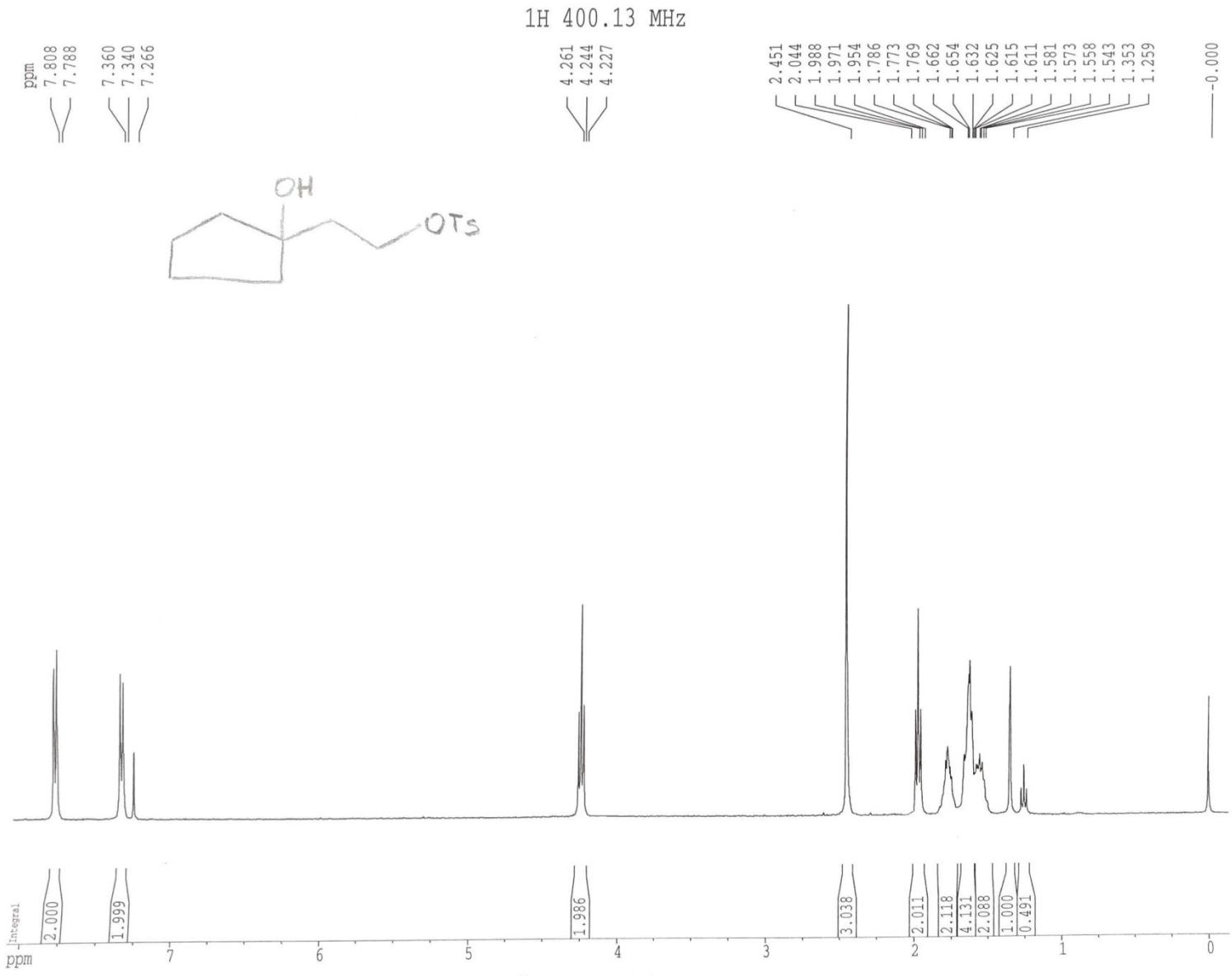

1H 400.13 MHz OK!
ppm
7.2724
4.8366
4.7222
OTES
2.1992
2.1826
2.1713
1.9826
1.9680
1.9394
1.8435
1.8323
1.7967
1.7726
1.7471
1.5249
1.5060
1.4872
1.4697
1.4467
1.2557
1.0690
0.9847
0.9675
0.8939
0.8753
0.8566
0.6142
Integral
ppm
7
6
5
4
3
2
1
0

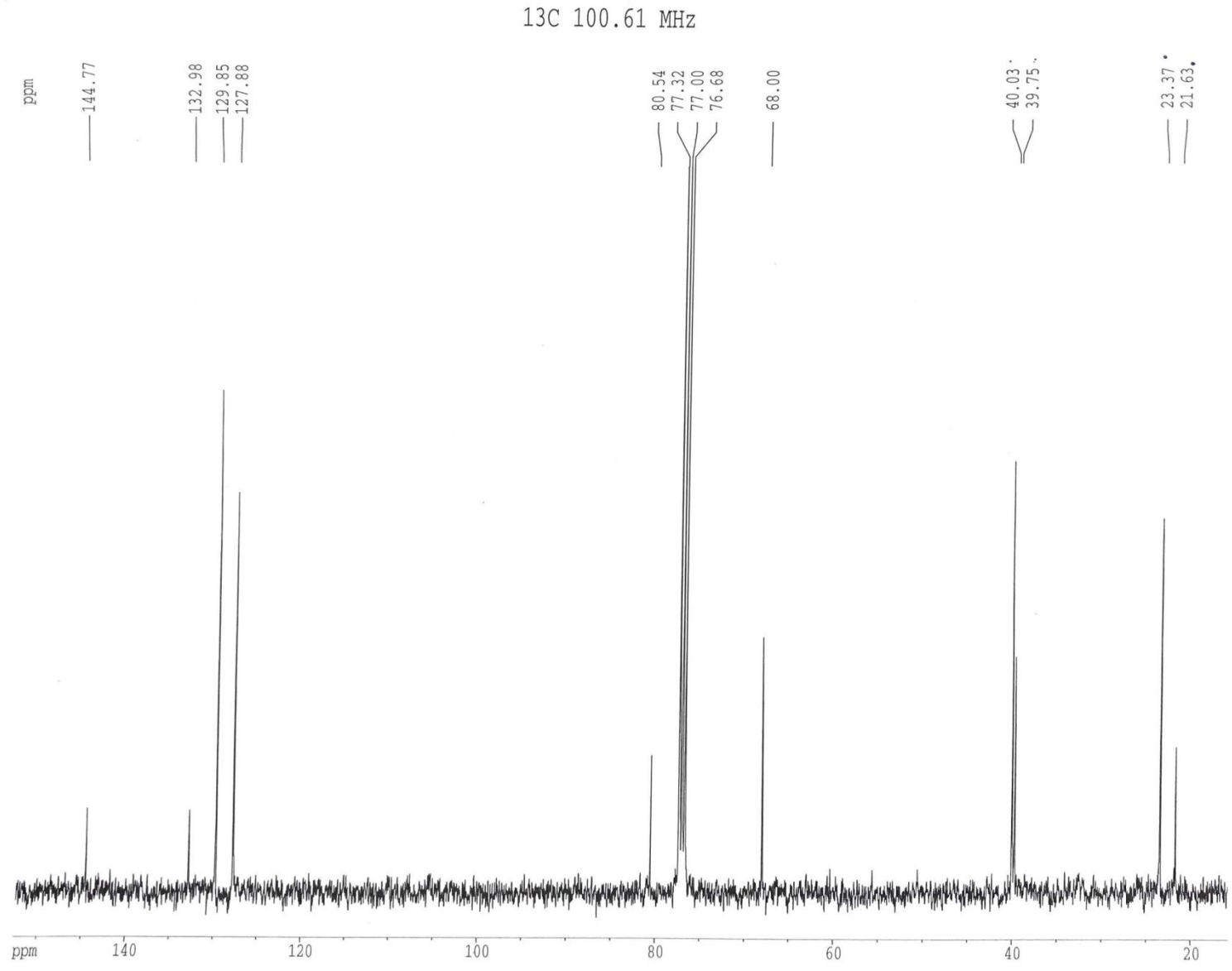

13C 100.61 MHz
ppm
212.08
153.93
129.51
128.33
108.93
77.32
77.00
76.68
74.63
61.92
53.71
49.99
43.82
41.00
38.36
37.59
31.92
31.06
30.99
24.06
20.30
18.55
12.02
7.79
ppm
200
180
160
140
120
100
80
60
40
20

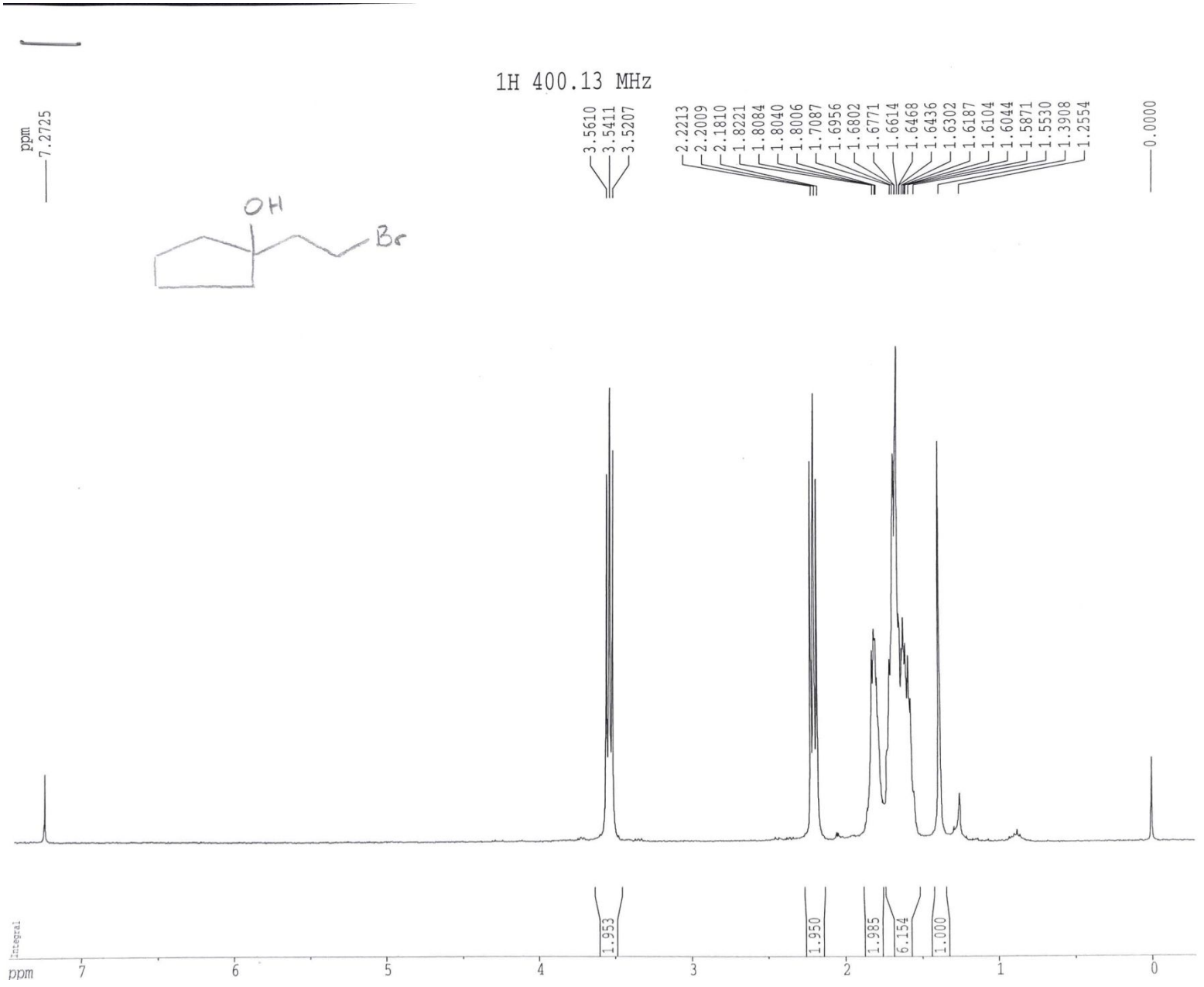

1H 400.13 MHz
ppm
7.2560
6.2265
6.1989
5.8470
5.8193
4.9698
4.9210
4.8087
4.6688
4.4432
4.4201
4.4146
2.4926
2.4484
2.3432
2.1813
2.1693
1.9455
1.9325
1.6186
1.5522
1.4837
1.4648
1.4443
0.9700
0.9577
0.9500
0.9303
0.8951
0.8666
0.8491
0.8304
0.8118
0.6053
0.5858
0.5660
0.5241
0.0779
0.0679
0.0474
0.0265
OTES
TBSO
OTBS
Integral
0.06
1.00
0.06
0.99
2.13
0.97
0.99
2.14
1.00
2.17
1.07
2.10
5.27
14.99
3.16
1.27
11.98
18.16
7.53
5.79
2.77
5.55
6.42
ppm
7
6
5
4
3
2
1
0

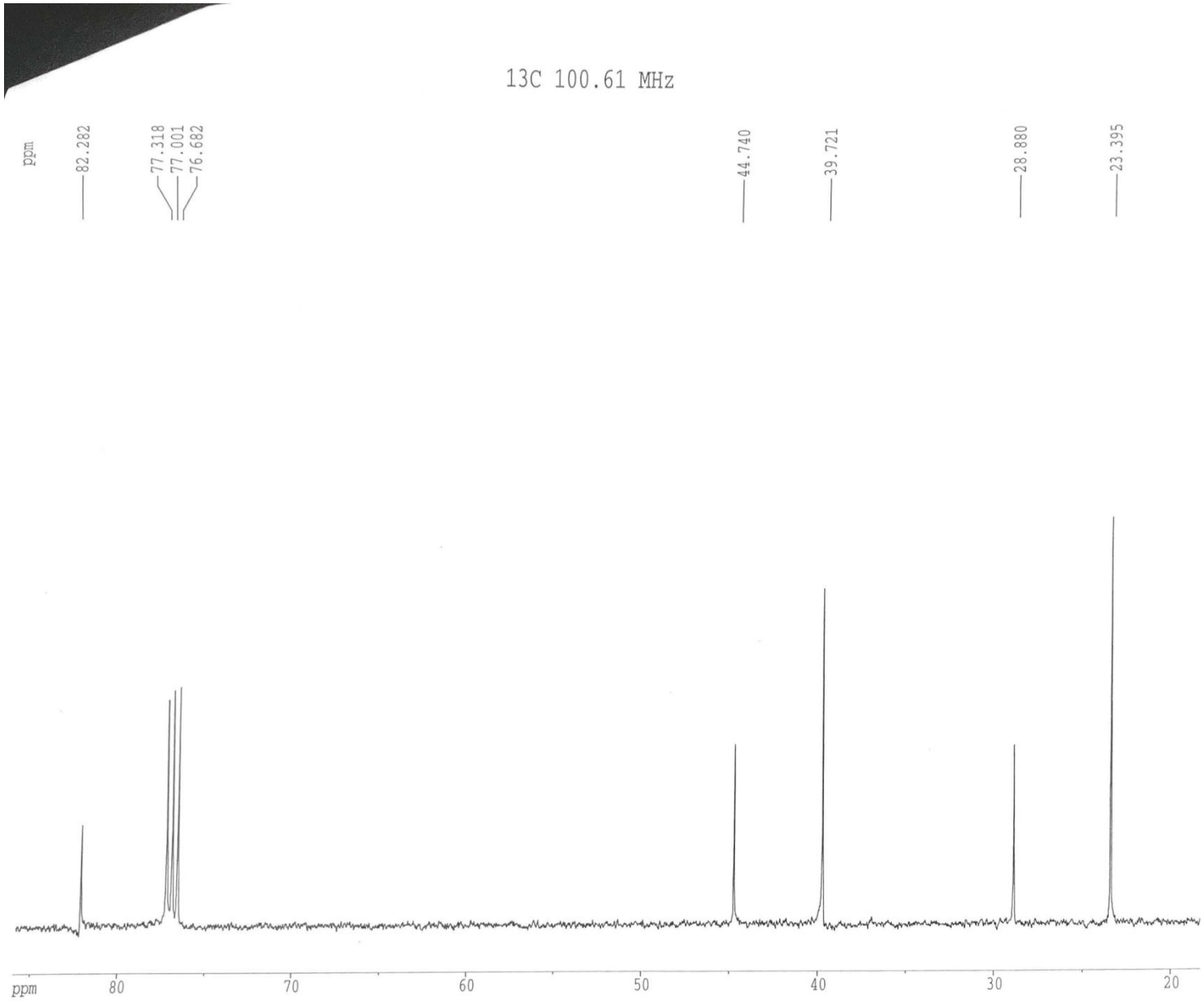

13C 100.61 MHz
ppm
154.88
152.98
141.28
132.70
122.40
116.10
108.30
106.25
78.19
77.32
77.00
76.68
72.53
71.64
56.22
53.76
47.60
45.77
44.33
39.18
39.10
38.56
32.05
31.83
31.58
25.84
25.78
21.94
21.70
20.39
11.62
8.35
7.29
7.08
ppm
140
120
100
80
60
40
20

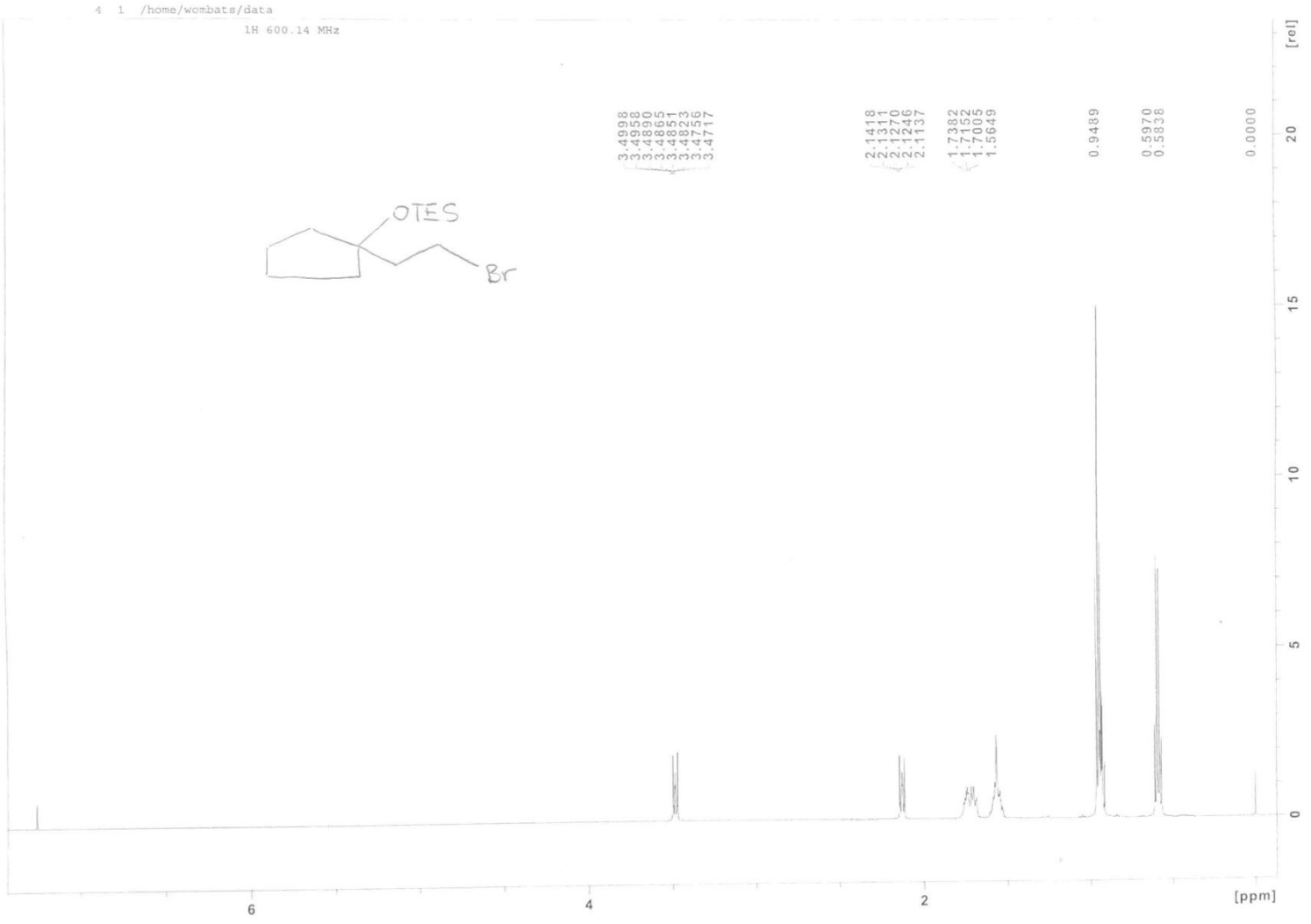

1H 400.13 MHz
ppm
3.5533
3.5333
3.5129
2.2122
2.1916
2.1717
1.8169
1.8019
1.7020
1.6883
1.6733
1.6698
1.6540
1.6391
1.6361
1.6221
1.6098
1.6019
1.5955
1.5785
1.5636
1.5487
1.5445
OH
OH
Integral
2.000
2.027
3.008
6.084
ppm
7
6
5
4
3
2
1
0

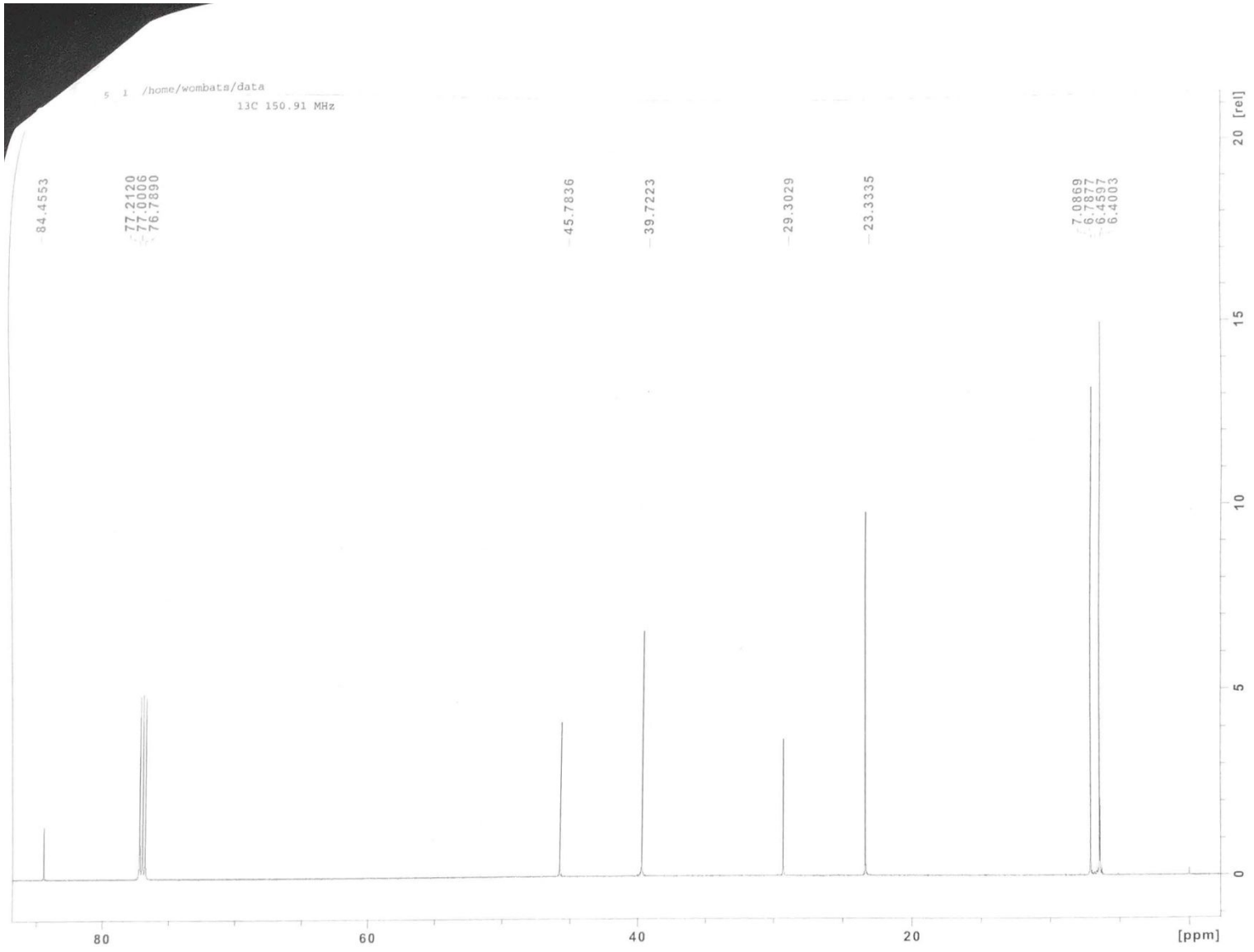

1H 400.13 MHz
ppm
7.808
7.788
7.360
7.340
7.266
4.261
4.244
4.227
2.451
2.044
1.988
1.971
1.954
1.786
1.773
1.769
1.662
1.654
1.632
1.625
1.615
1.611
1.581
1.573
1.558
1.543
1.353
1.259
0.000
OH
OTs
Integral
2.000
1.999
1.986
3.038
2.011
2.118
4.131
2.088
1.000
0.491
ppm
7
6
5
4
3
2
1
0

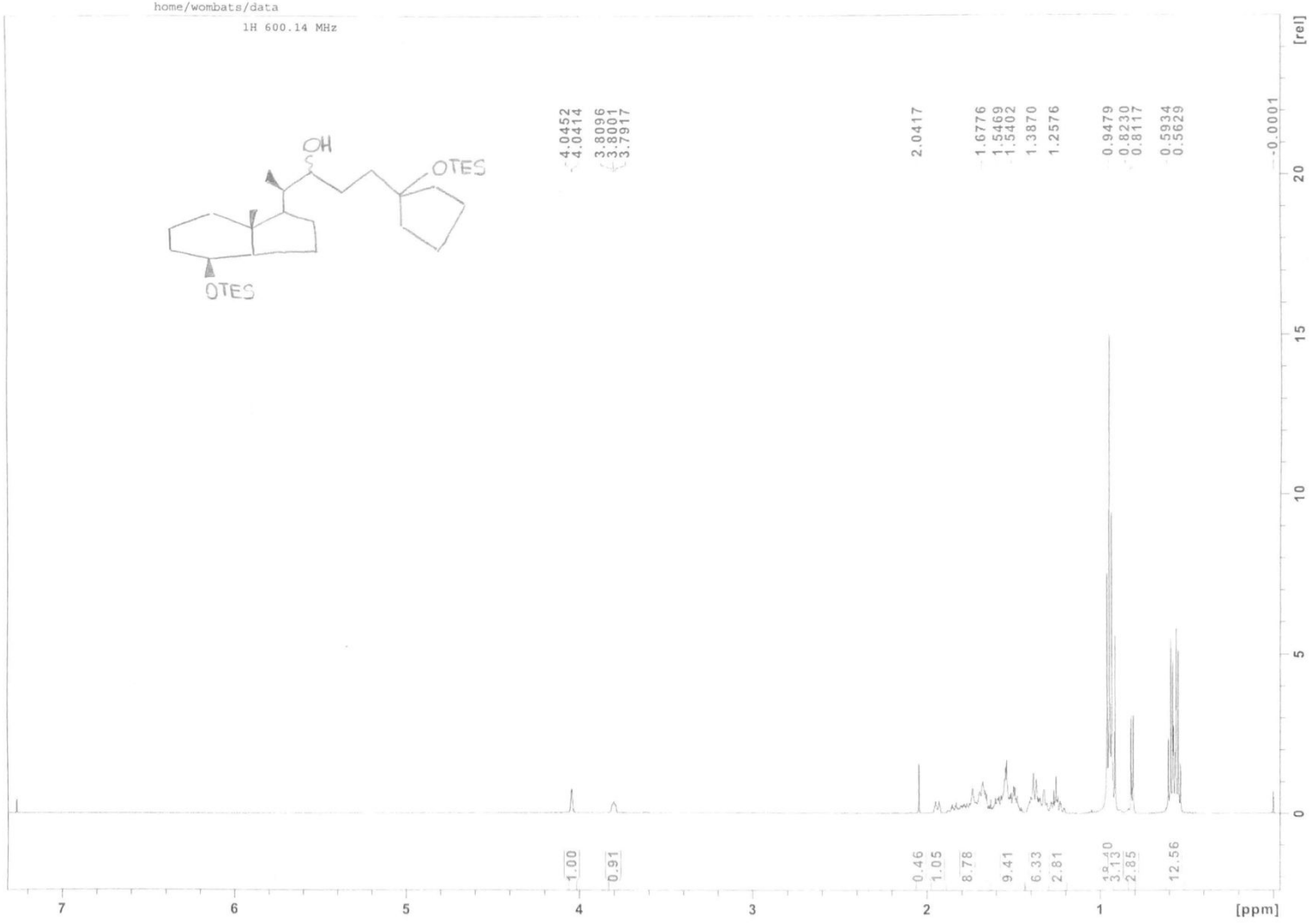

13C 100.61 MHz
ppm
144.77
132.98
129.85
127.88
80.54
77.32
77.00
76.68
68.00
40.03
39.75
23.37
21.63
ppm
140
120
100
80
60
40
20

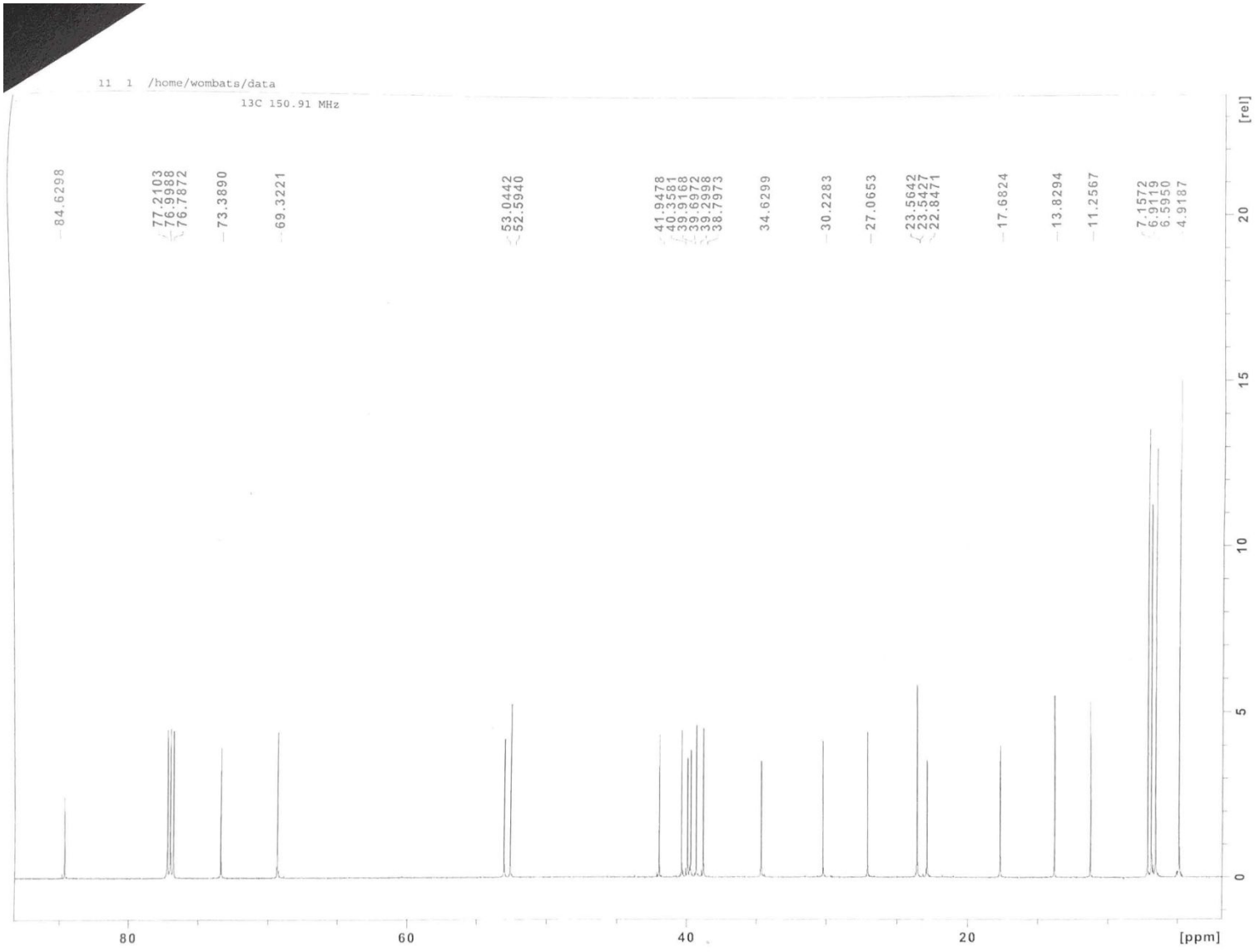

1H 400.13 MHz
ppm
7.2725
3.5610
3.5411
3.5207
2.2213
2.2009
2.1810
1.8221
1.8084
1.8040
1.8006
1.7087
1.6956
1.6802
1.6771
1.6614
1.6468
1.6436
1.6302
1.6187
1.6104
1.6044
1.5871
1.5530
1.3908
1.2554
0.0000
OH
Br
Integral
1.953
1.950
1.985
6.154
1.000
ppm
7
6
5
4
3
2
1
0

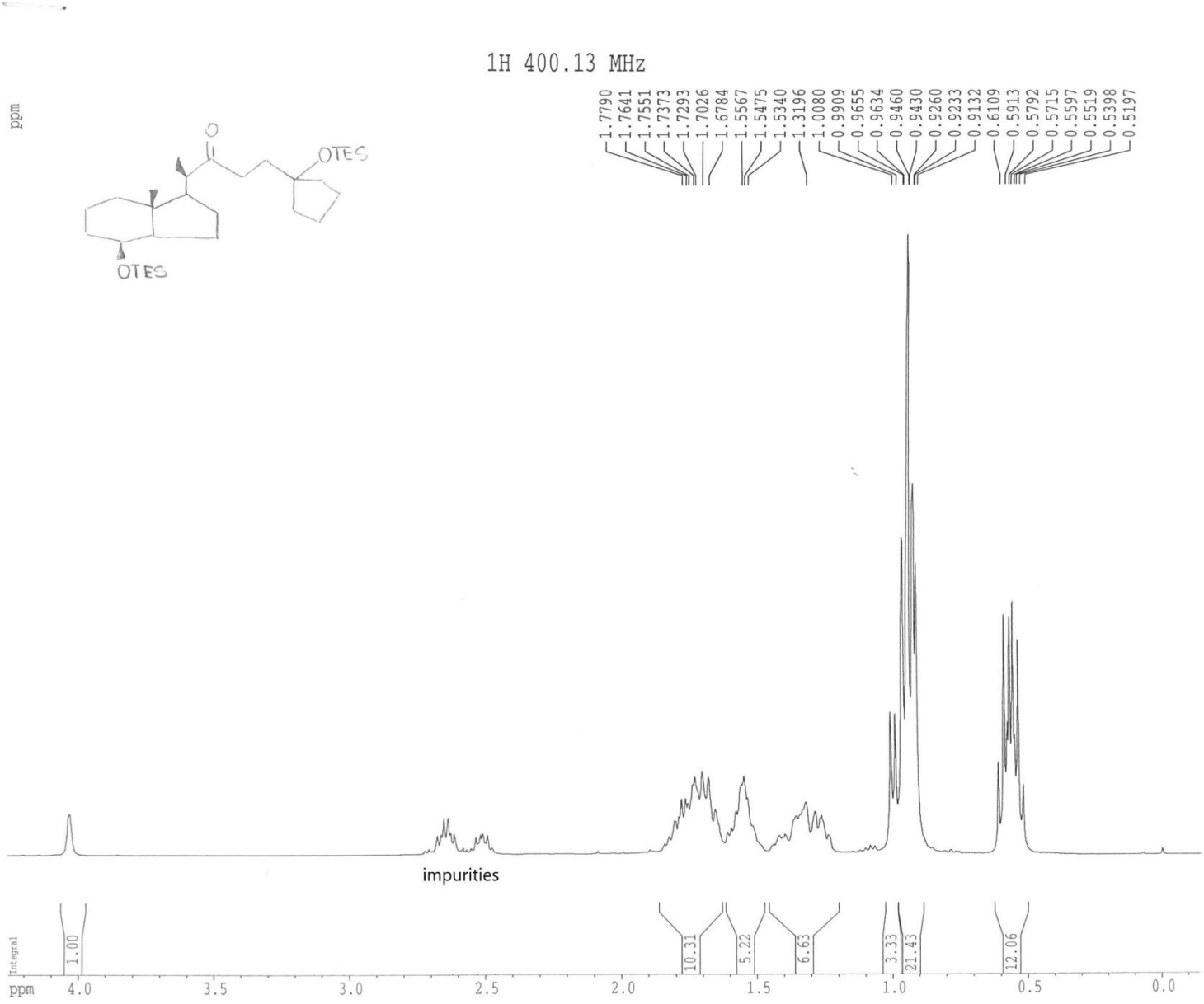

13C 100.61 MHz
ppm
82.282
77.318
77.001
76.682
44.740
39.721
28.880
23.395
ppm
80
70
60
50
40
30
20

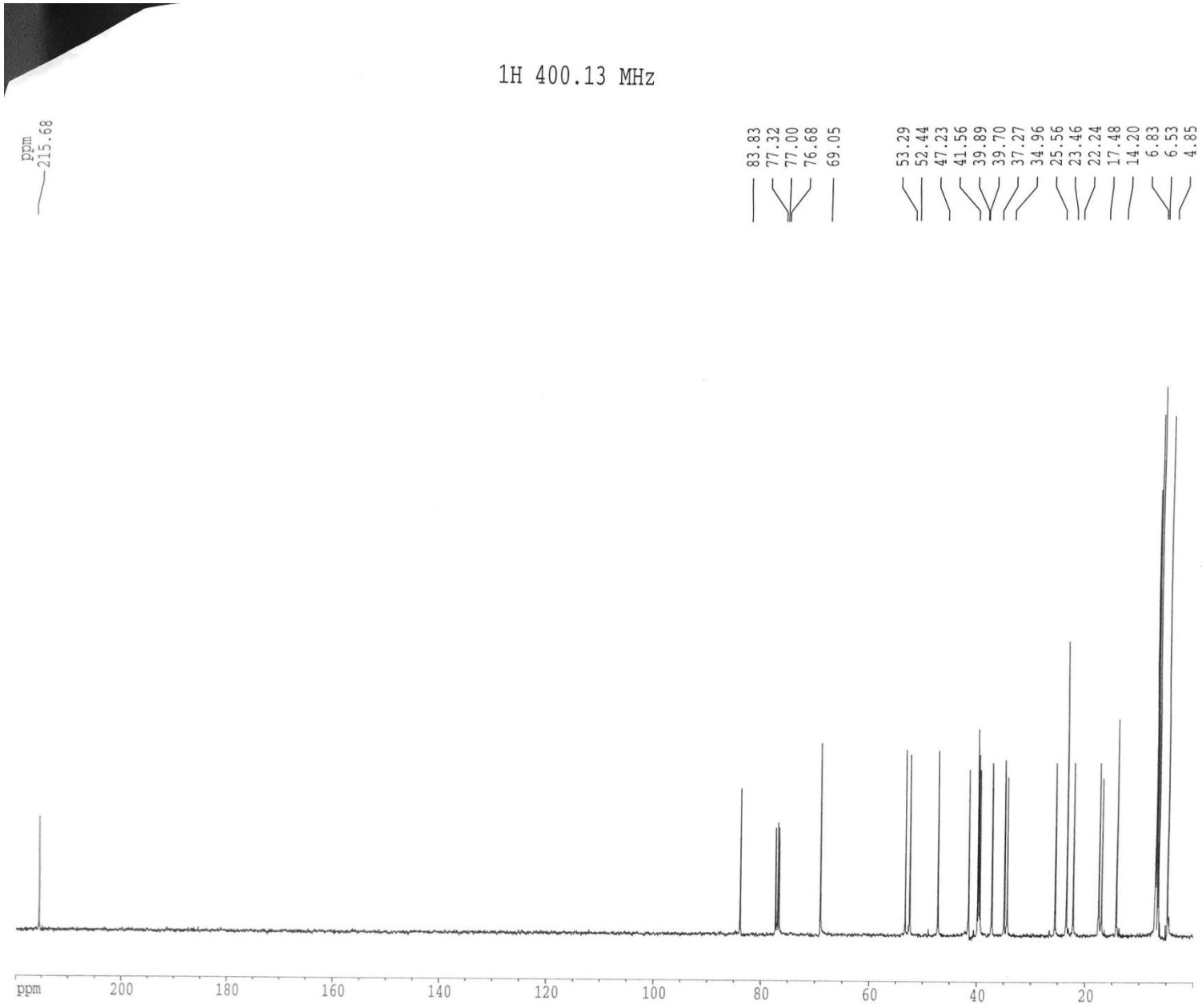

4 1 /home/wombats/data
1H 600.14 MHz
3.4998
3.4990
3.4880
3.4865
3.4851
3.4823
3.4755
3.4717
2.1418
2.1311
2.1270
2.1246
2.1137
1.7382
1.7152
1.7005
1.5649
0.9489
0.5970
0.5838
0.0000
OTES
Br
[rel]
20
15
10
5
0
6
4
2
[ppm]

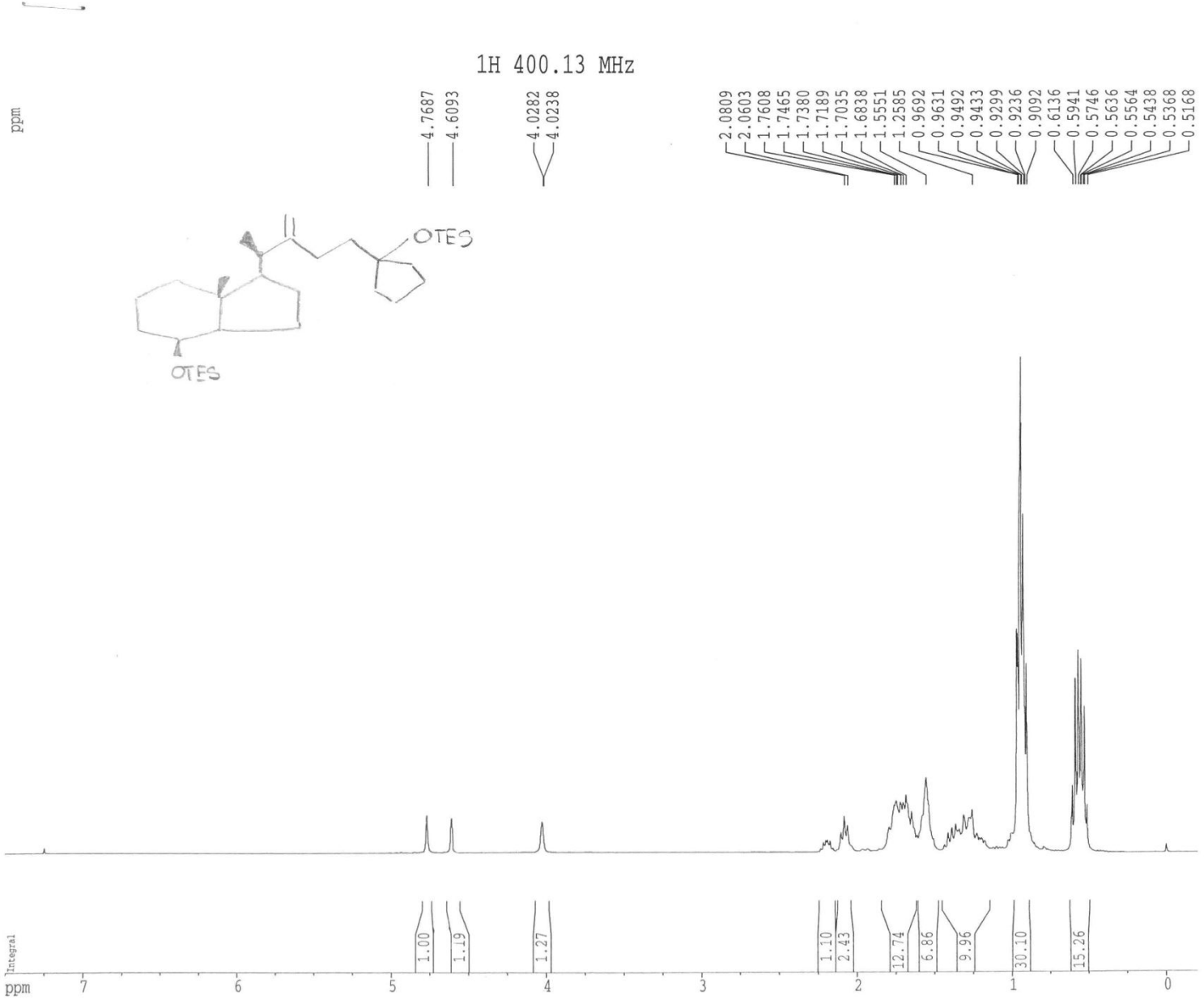

5 1 /home/wombats/data
13C 150.91 MHz
84.4553
77.2120
77.0006
76.7890
45.7836
39.7223
29.3029
23.3335
7.0869
6.7877
6.4597
6.4003
20 [rel]
15
10
5
0
80
60
40
20
[ppm]

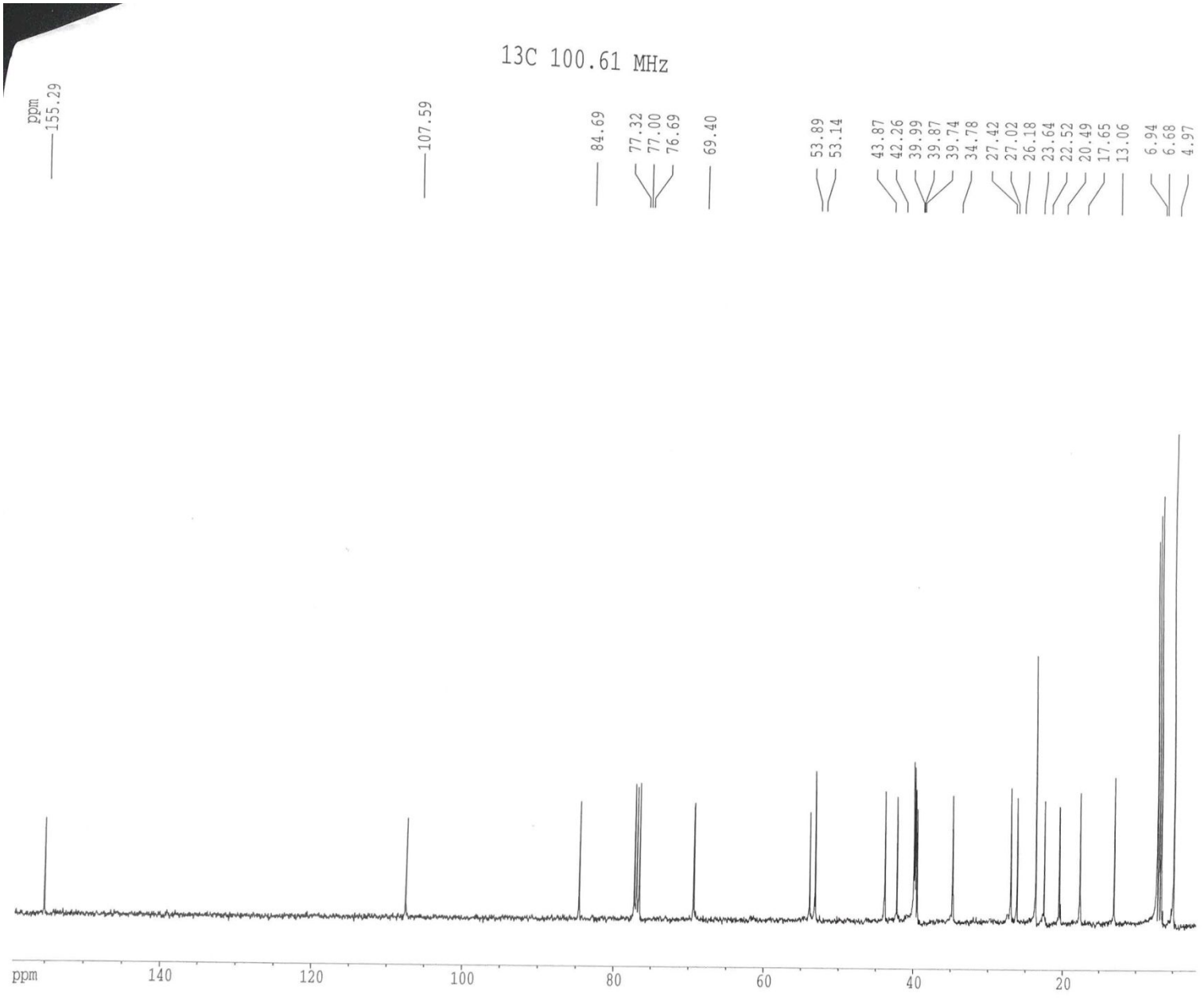

home/wombats/data
1H 600.14 MHz
OH
OTES
OTES
4.0452
4.0414
3.8096
3.8001
3.7917
2.0417
1.6776
1.5469
1.5402
1.3870
1.2576
0.9479
0.8230
0.8117
0.5934
0.5629
-0.0001
[rel]
0
5
10
15
20
1.00
0.91
0.46
1.05
8.78
9.41
6.33
2.81
18.40
3.13
2.85
12.56
7
6
5
4
3
2
1
[ppm]

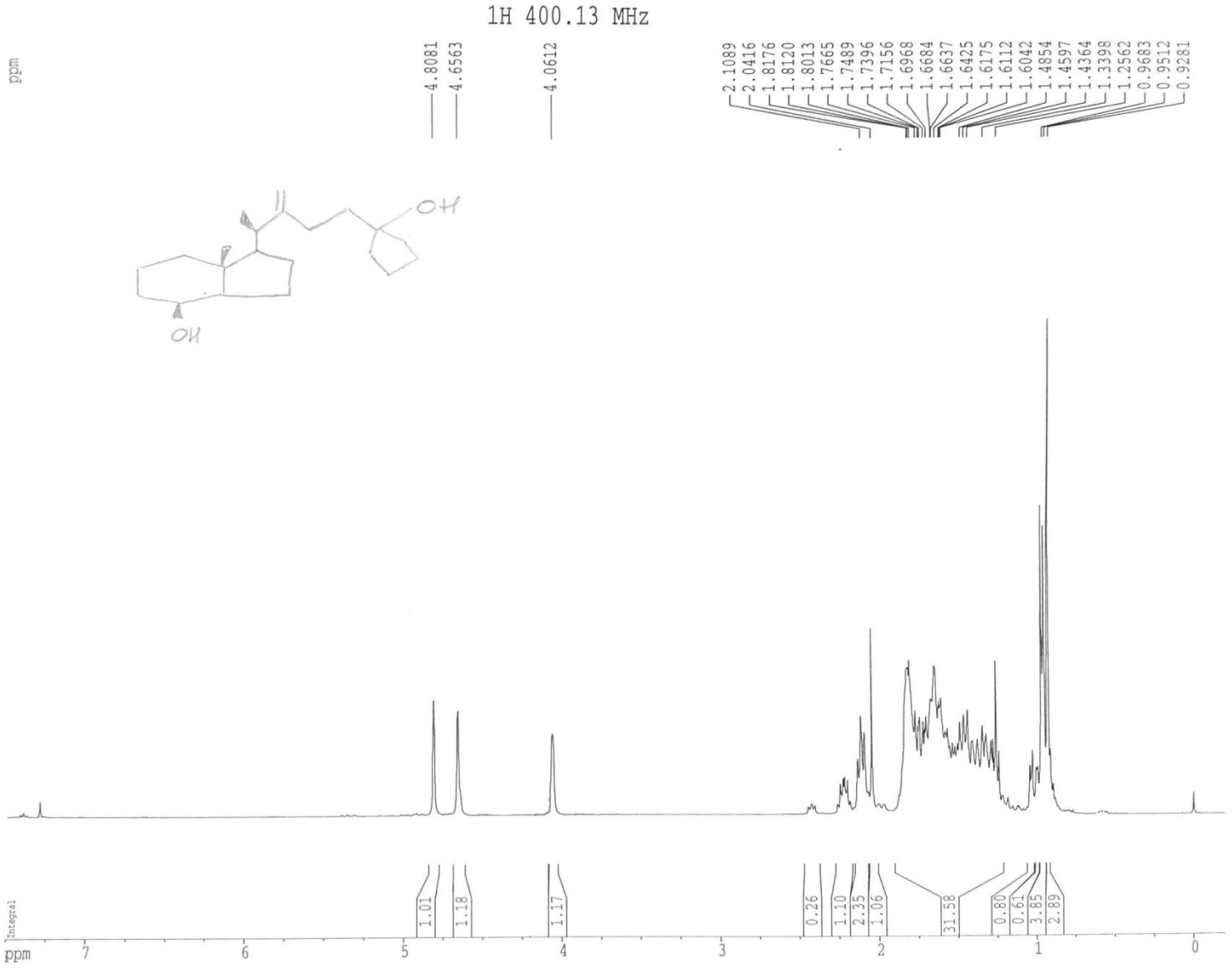

11 1 /home/wombats/data
13C 150.91 MHz
84.6298
77.2103
76.9988
76.7872
73.3890
69.3221
53.0442
52.5940
41.9478
40.3581
39.9168
39.6972
39.2998
38.7973
34.6299
30.2283
27.0653
23.5642
23.5427
22.8471
17.6824
13.8294
11.2567
7.1572
6.9119
6.5950
4.9187
[rel]
0
5
10
15
20
80
60
40
20
[ppm]

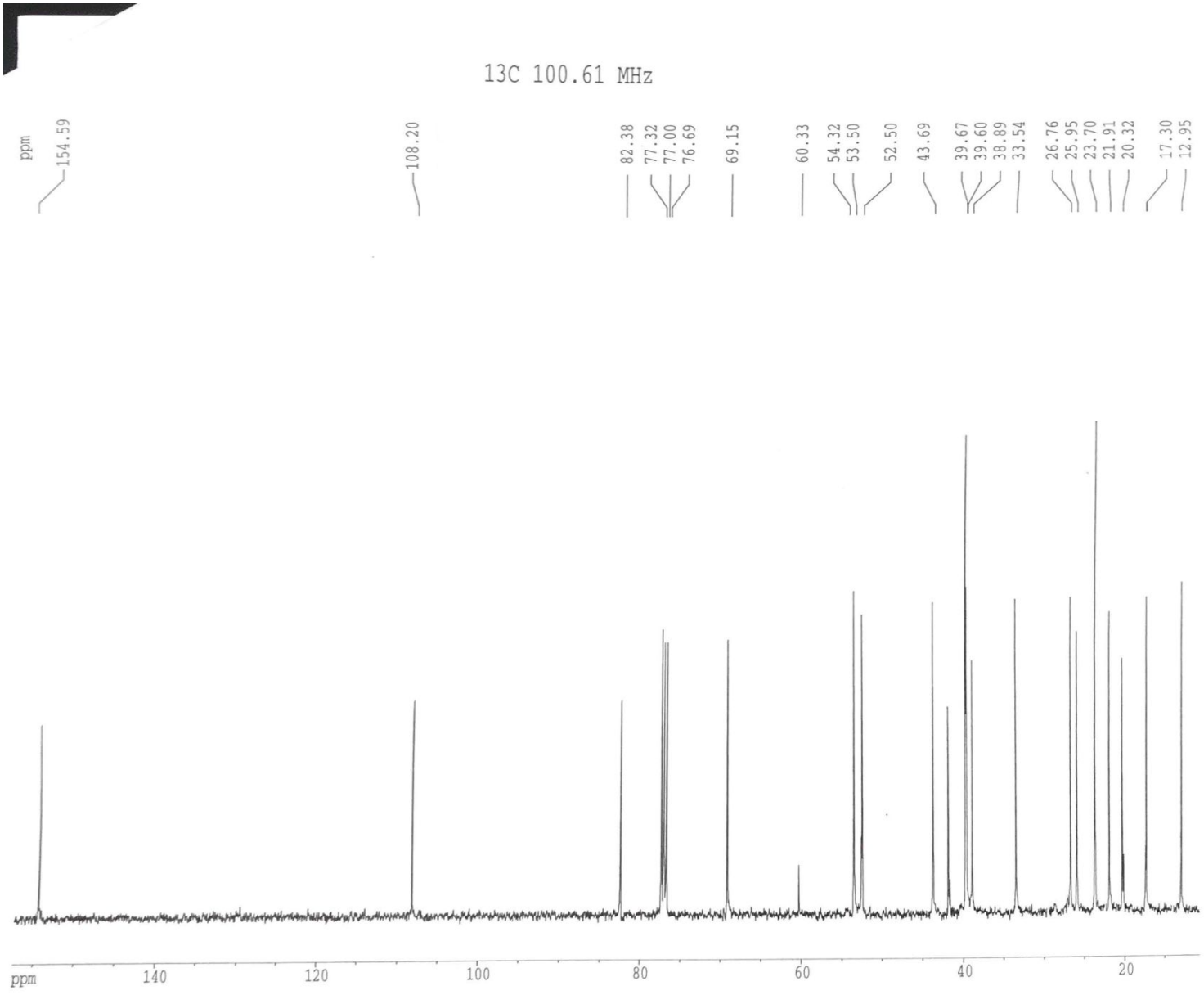

1H 400.13 MHz
ppm
1.7790
1.7641
1.7551
1.7373
1.7293
1.7026
1.6784
1.5567
1.5475
1.5340
1.3196
1.0080
0.9909
0.9655
0.9634
0.9460
0.9430
0.9260
0.9233
0.9132
0.6109
0.5913
0.5792
0.5715
0.5597
0.5519
0.5398
0.5197
O
OTES
OTES
impurities
Integral
1.00
10.31
5.22
6.63
3.33
21.43
12.06
ppm
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

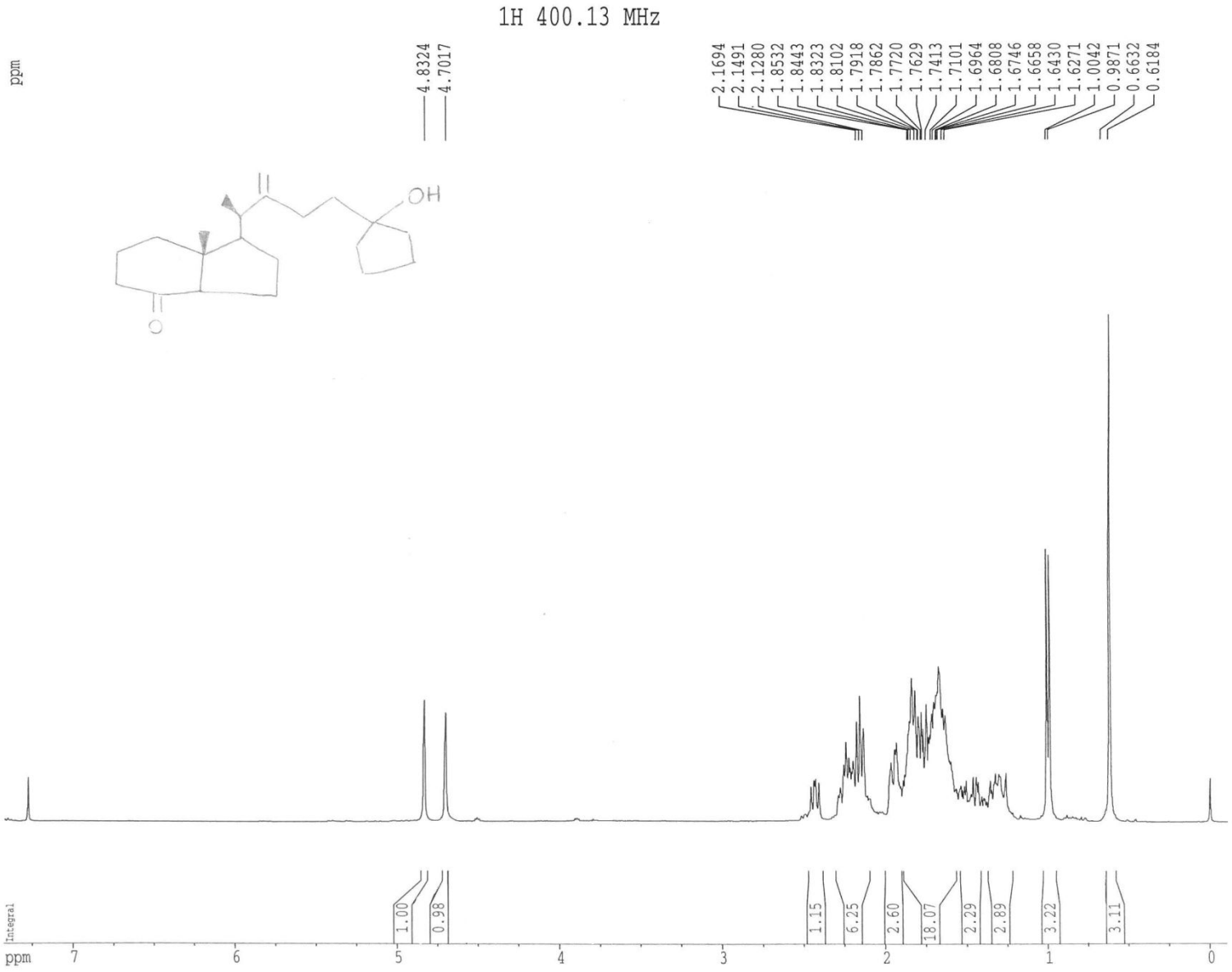

1H 400.13 MHz
ppm
215.68
83.83
77.32
77.00
76.68
69.05
53.29
52.44
47.23
41.56
39.89
39.70
37.27
34.96
25.56
23.46
22.24
17.48
14.20
6.83
6.53
4.85
ppm
200
180
160
140
120
100
80
60
40
20

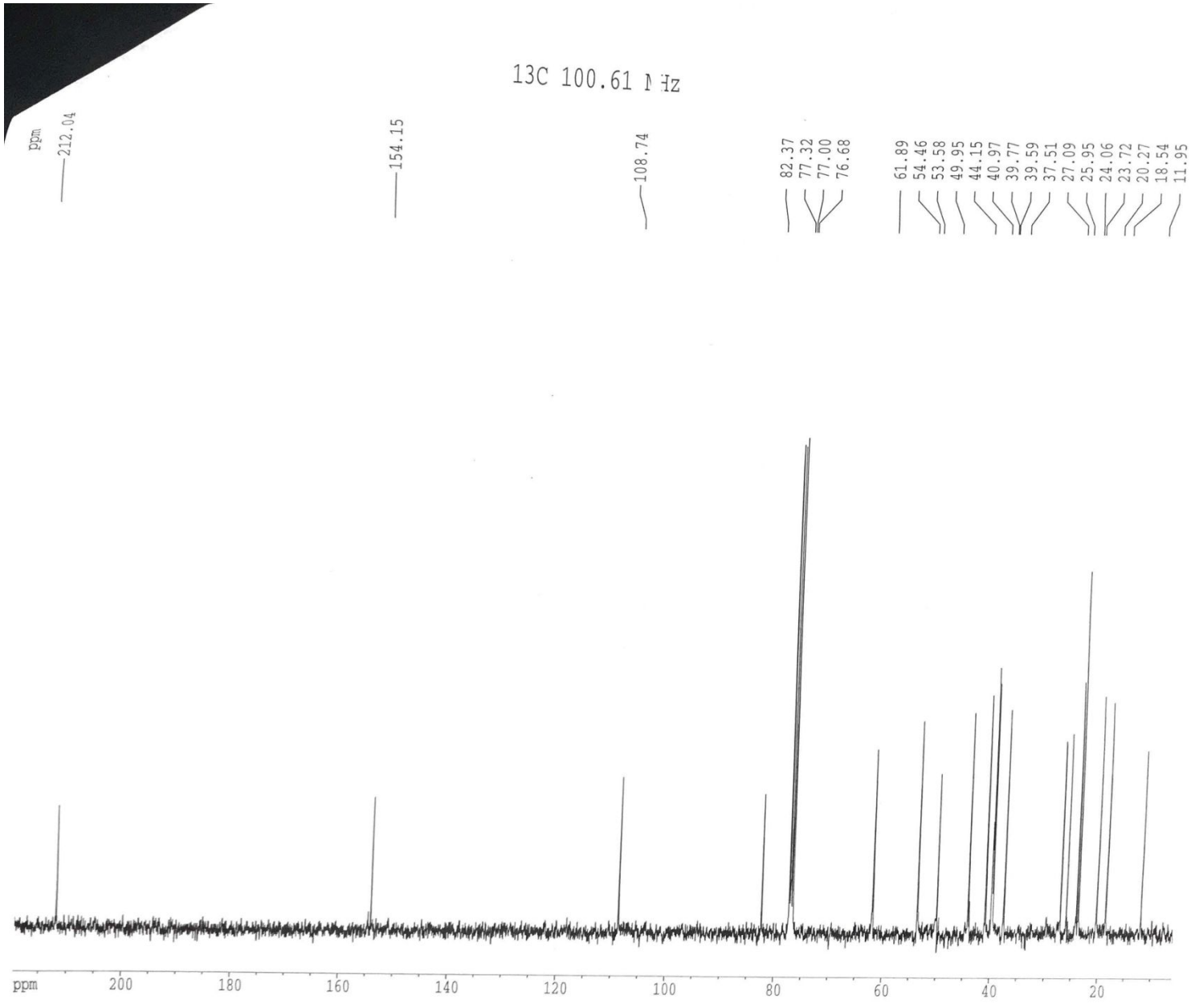

1H 400.13 MHz
ppm
4.7687
4.6093
4.0282
4.0238
2.0809
2.0603
1.7608
1.7465
1.7380
1.7189
1.7035
1.6838
1.5551
1.2585
0.9692
0.9631
0.9492
0.9433
0.9299
0.9236
0.9092
0.6136
0.5941
0.5746
0.5636
0.5564
0.5438
0.5368
0.5168
OTES
OTES
Integral
1.00
1.19
1.27
1.10
2.43
12.74
6.86
9.96
30.10
15.26
ppm
7
6
5
4
3
2
1
0

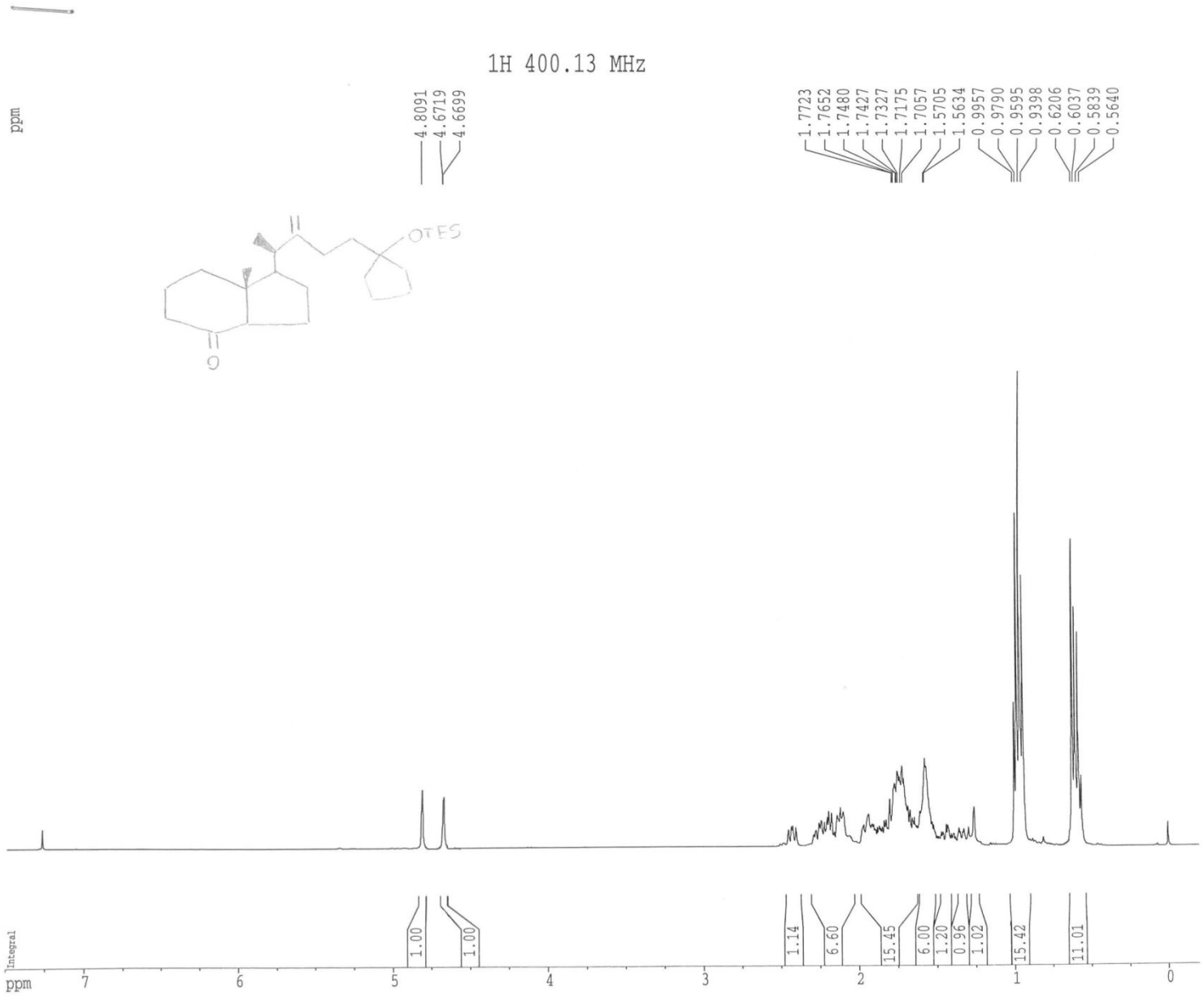

13C 100.61 MHz
ppm
155.29
107.59
84.69
77.32
77.00
76.69
69.40
53.89
53.14
43.87
42.26
39.99
39.87
39.74
34.78
27.42
27.02
26.18
23.64
22.52
20.49
17.65
13.06
6.94
6.68
4.97
ppm
140
120
100
80
60
40
20

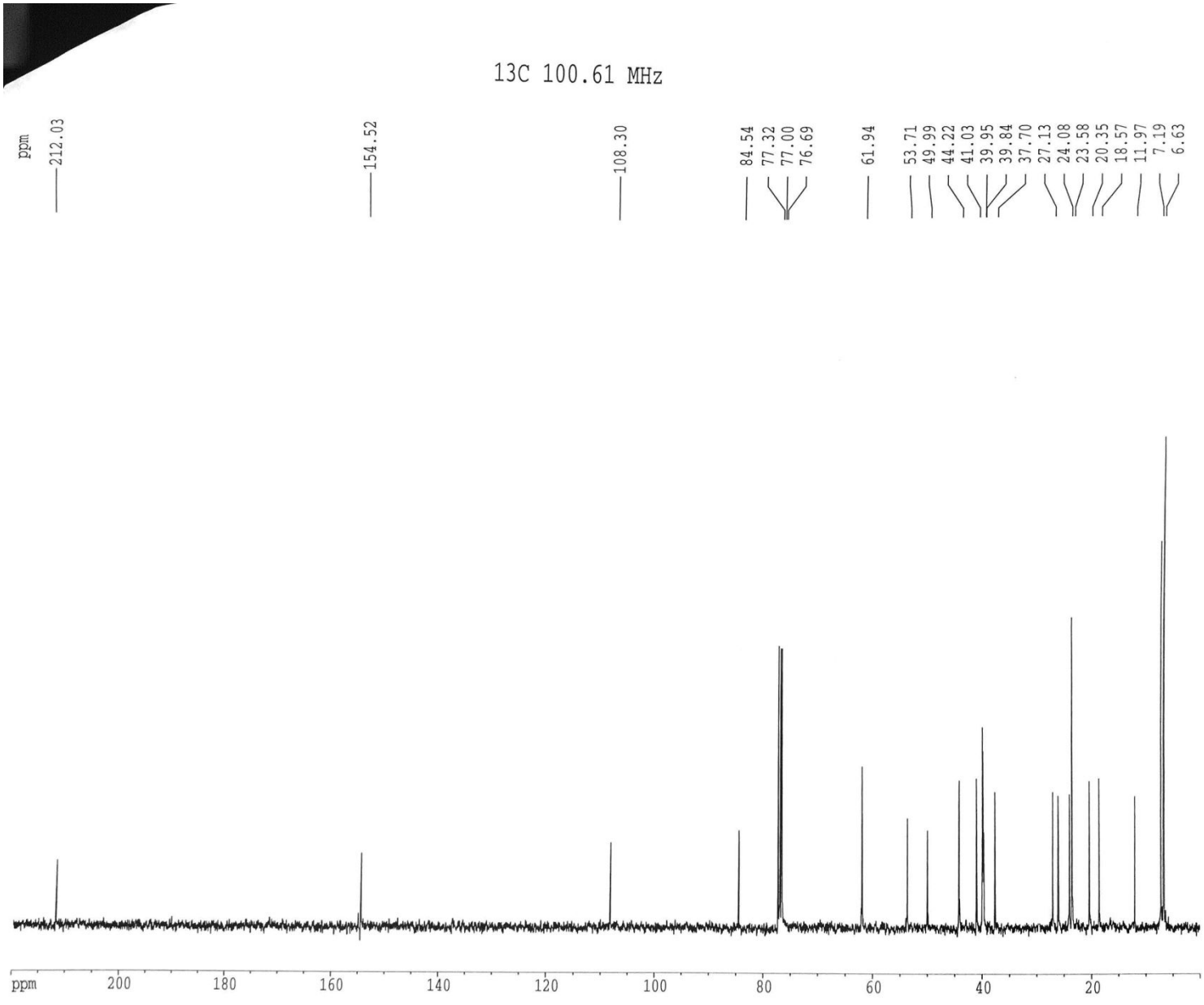

1H 400.13 MHz
ppm
4.8081
4.6563
4.0612
2.1089
2.0416
1.8176
1.8120
1.8013
1.7665
1.7489
1.7396
1.7156
1.6968
1.6684
1.6637
1.6425
1.6175
1.6112
1.6042
1.4854
1.4597
1.4364
1.3398
1.2562
0.9683
0.9512
0.9281
OH
OH
Integral
1.01
1.18
1.17
0.26
1.10
2.35
1.06
31.58
0.80
0.61
3.85
2.89
ppm
7
6
5
4
3
2
1
0


13C 100.61 MHz
ppm
154.59
108.20
82.38
77.32
77.00
76.69
69.15
60.33
54.32
53.50
52.50
43.69
39.67
39.60
38.89
33.54
26.76
25.95
23.70
21.91
20.32
17.30
12.95
ppm
140
120
100
80
60
40
20

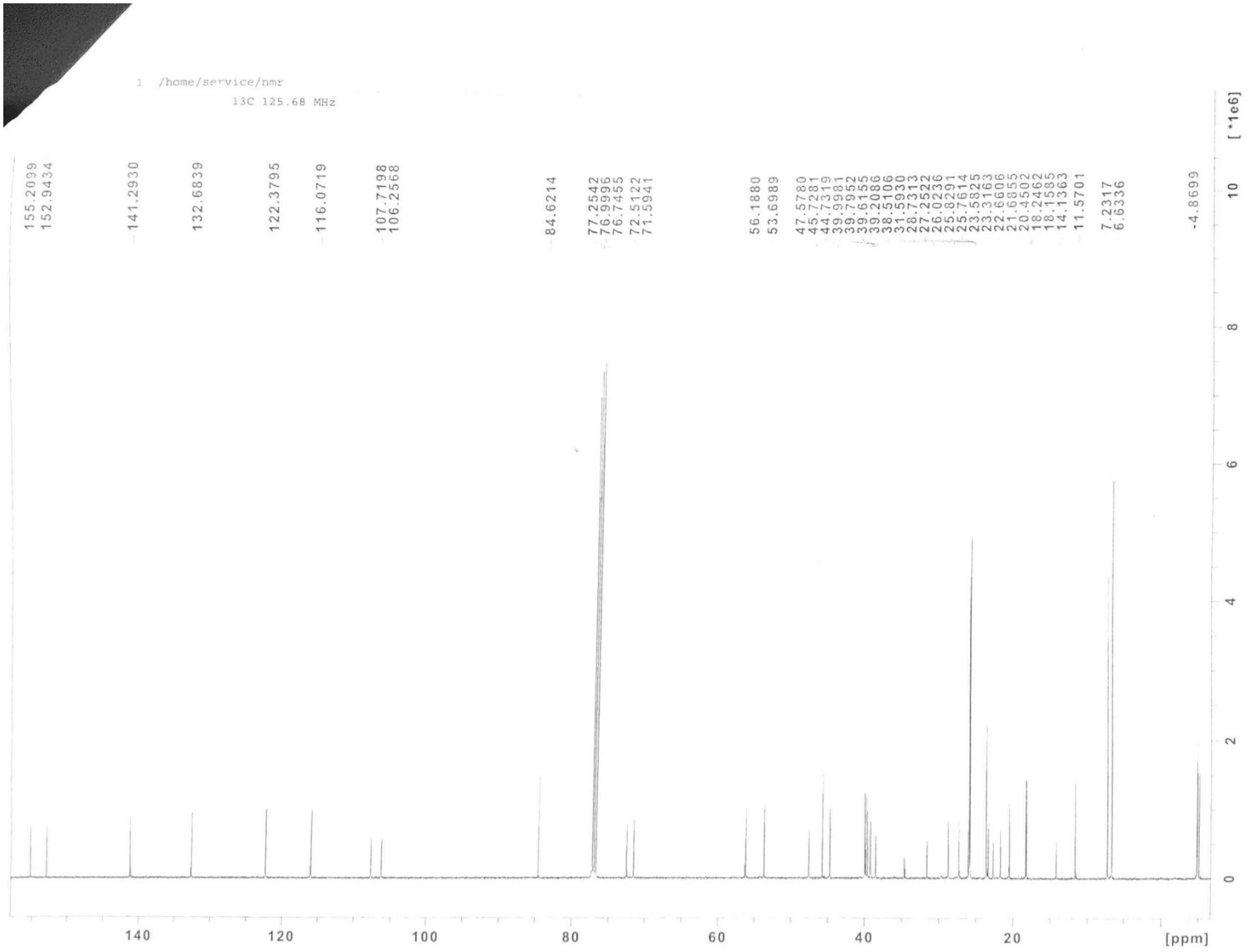

1H 400.13 MHz
ppm
4.8324
4.7017
2.1694
2.1491
2.1280
1.8532
1.8443
1.8323
1.8102
1.7918
1.7862
1.7720
1.7629
1.7413
1.7101
1.6964
1.6808
1.6746
1.6658
1.6430
1.6271
1.0042
0.9871
0.6632
0.6184
OH
O
Integral
1.00
0.98
1.15
6.25
2.60
18.07
2.29
2.89
3.22
3.11
ppm
7
6
5
4
3
2
1
0


13C 100.61 MHz
ppm
212.04
154.15
108.74
82.37
77.32
77.00
76.68
61.89
54.46
53.58
49.95
44.15
40.97
39.77
39.59
37.51
27.09
25.95
24.06
23.72
20.27
18.54
11.95
ppm
200
180
160
140
120
100
80
60
40
20


1H 400.13 MHz
ppm
4.8091
4.6719
4.6699
1.7723
1.7652
1.7480
1.7427
1.7327
1.7175
1.7057
1.5705
1.5634
0.9957
0.9790
0.9595
0.9398
0.6206
0.6037
0.5839
0.5640
OTES
O
Integral
1.00
1.00
1.14
6.60
15.45
6.00
1.20
0.96
1.02
15.42
11.01
ppm
7
6
5
4
3
2
1
0


13C 100.61 MHz
ppm
212.03
154.52
108.30
84.54
77.32
77.00
76.69
61.94
53.71
49.99
44.22
41.03
39.95
39.84
37.70
27.13
24.08
23.58
20.35
18.57
11.97
7.19
6.63
ppm
200
180
160
140
120
100
80
60
40
20


14 1 /home/service/nmr
1H 499.84 MHz
6.2268
6.2046
5.8449
5.8225
4.9706
4.9208
4.7921
4.6252
4.4392
4.4250
4.4152
2.8243
2.8191
2.7935
2.5142
2.4513
2.3411
2.3355
2.1966
2.1058
1.9811
1.8391
1.7080
1.5690
1.2647
1.2551
0.9534
0.8944
0.8644
0.5962
0.5803
0.5240
0.0776
0.0674
OTES
TBSO
OTBS
1.00
0.99
2.02
0.88
0.99
2.04
1.02
2.10
1.12
5.59
2.05
5.71
3.32
9.56
4.05
1.23
12.99
20.70
6.74
2.59
12.55
7
6
5
4
3
2
1
[ppm]
0
100
200
300
400
500
[*1e3]


1 /home/service/nmr
13C 125.68 MHz
155.2099
152.9434
141.2930
132.6839
122.3795
116.0719
107.7198
106.2568
84.6214
77.2542
76.9996
76.7455
72.5122
71.5941
56.1880
53.6989
47.5780
45.7281
44.7319
39.9981
39.7952
39.6155
39.2086
38.5106
31.5930
28.7313
27.2522
26.0236
25.8291
25.7614
23.5825
23.3163
22.6606
21.6555
20.4502
18.2462
18.1585
14.1363
11.5701
7.2317
6.6336
-4.8699
[*1e6]
10
8
6
4
2
0
140
120
100
80
60
40
20
[ppm]