Electronic Supporting Information

Use of the Dehydrophos Biosynthetic Enzymes to Prepare Antimicrobial Analogs of Alaphosphin

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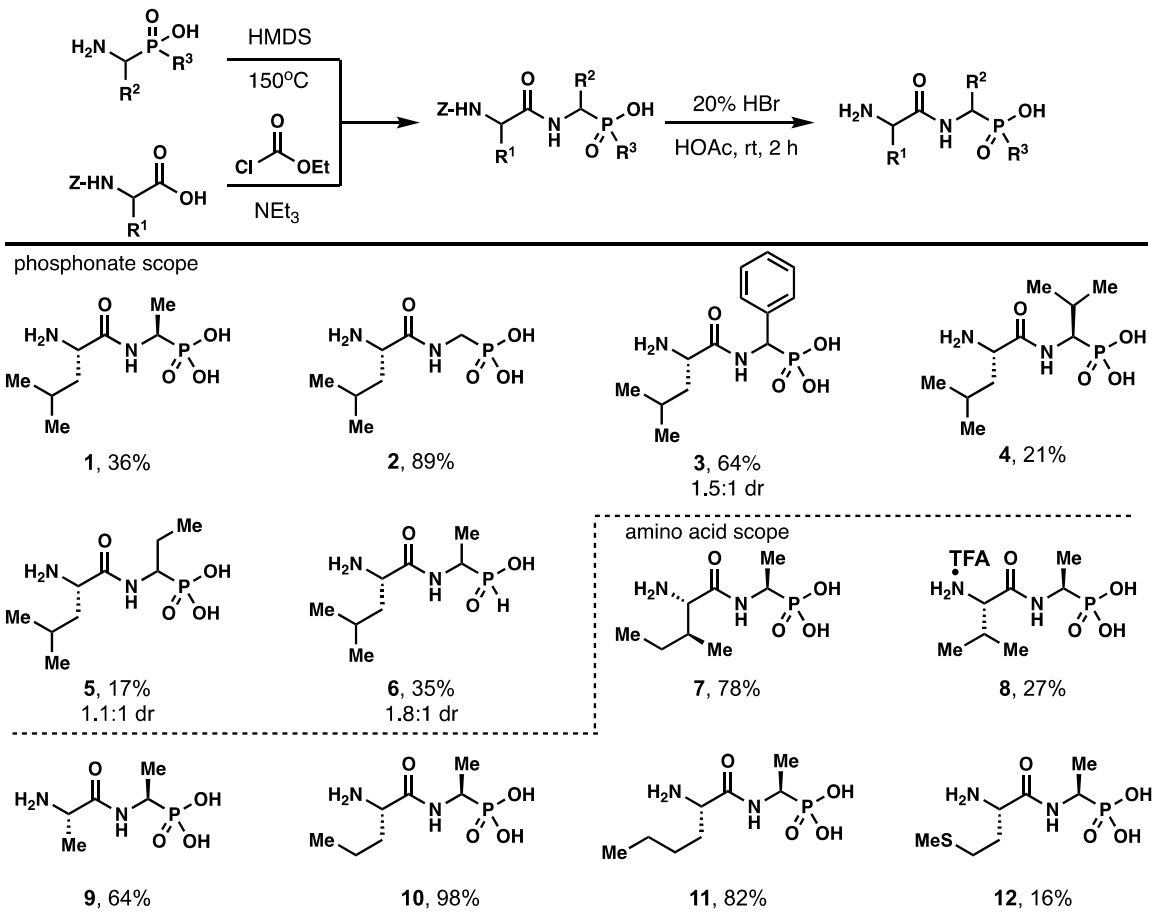


Figure S1. General procedure[1] and overall yields for the preparation of phosphonate dipeptides as authentic standards.

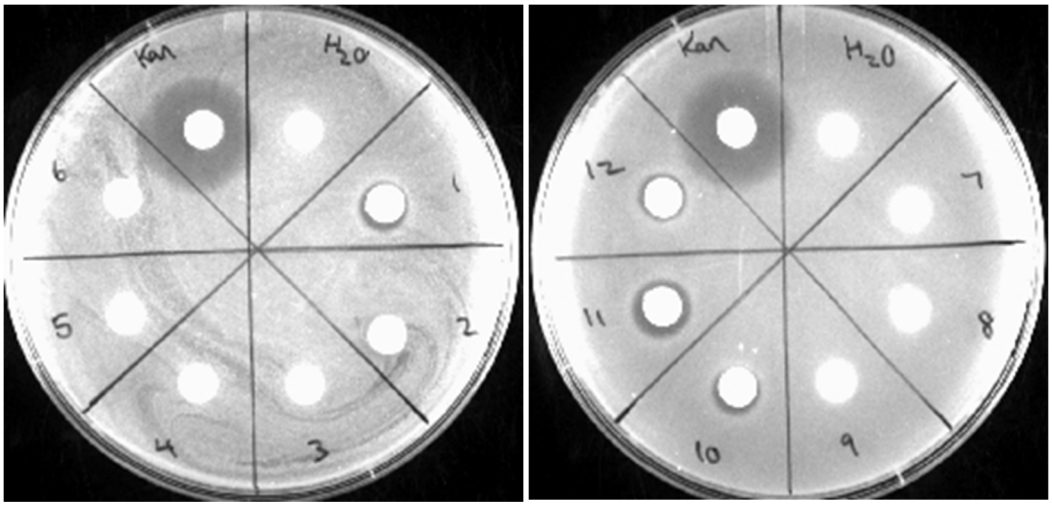


Figure S2. Agar diffusion growth inhibition assay for phosphonodipeptides (**1-12**, 30 μg each) against the indicator strain *E. coli* ATCC 25922. Kanamycin (Kan, 30 μg) and water (H_2O) were used as positive and negative controls respectively. The experiment was performed once.

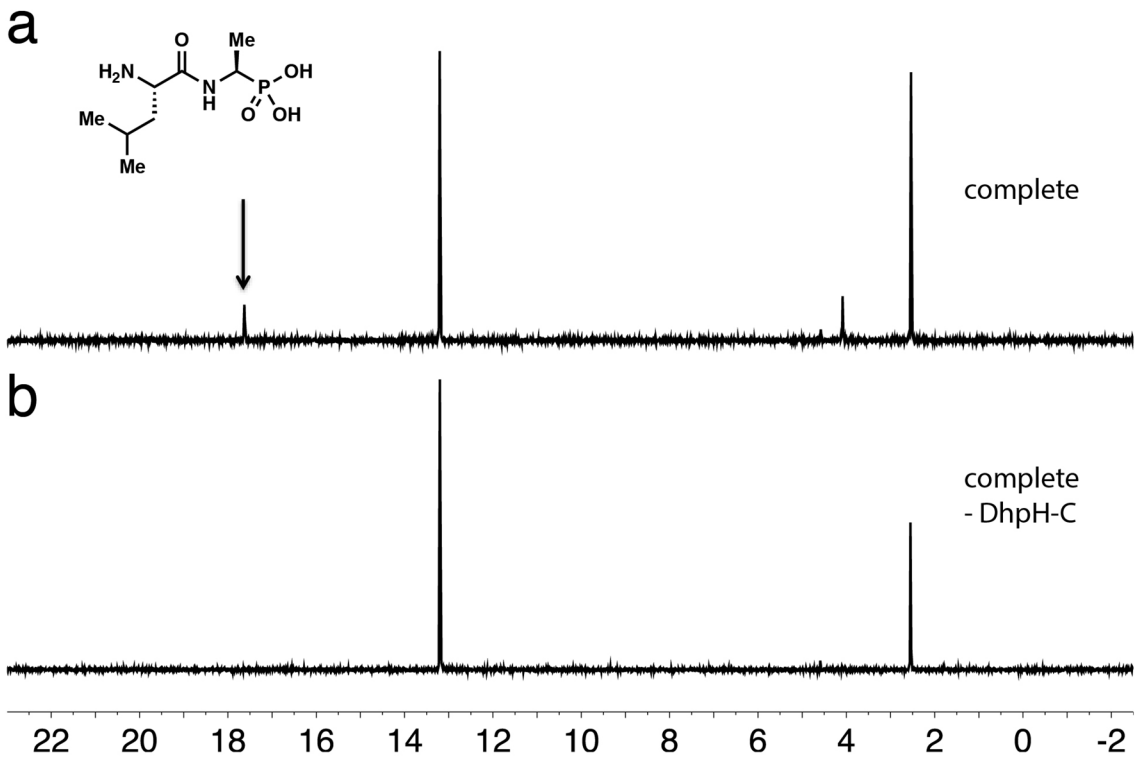
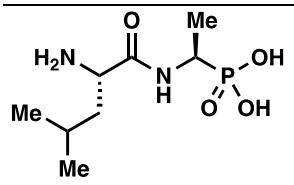
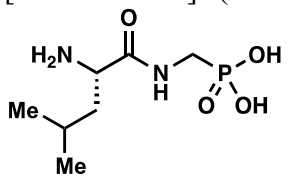


Figure S3. ^{31}P NMR analysis of DhpH-C activity with L-Leu and L-Ala(P). **a**. Complete reaction containing 5 mM Ala(P), 6 mM Leu, 6 mM ATP, 1.5 mg total tRNA from *E. coli*, 6 μM LeuRS (triple mutant), 10 U TIPP, and DhpH-C (50 μM) in 100 mM Na-HEPES, 10 mM KCl, 20 mM $MgCl_2$, pH 7.5. Arrow indicates the expected product. **b.** Control reaction without DhpH-C shows no product formation. Similarly, all reactions in Figures 7 and 8 required DhpH-C.

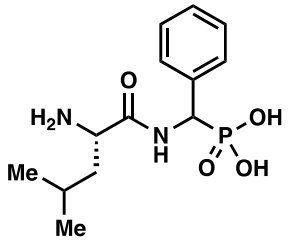
Characterization of synthetic compounds



Following the general procedure, L-Ala(P) (288 mg, 2.3 mmol, 1 equiv) and Z-L-Leu (610 mg, 2.3 mmol, 1 equiv) afforded 191 mg (35%) of compound **1** as a white solid. ^{1}H NMR (600 MHz, D_2O) δ 4.18 (dq, *J* = 14.8, 7.2 Hz, 1H), 4.00 (t, *J* = 6.9 Hz, 1H), 1.83 – 1.75 (m, 1H), 1.76 – 1.68 (m, 2H), 1.36 (dd, *J* = 15.9, 7.4 Hz, 3H), 0.97 (dd, *J* = 10.4, 6.3 Hz, 6H); ^{13}C NMR (150 MHz, D_2O) δ 169.5, 52.0, 43.4 (d, *J* = 153.4 Hz), 39.7, 23.6, 21.6, 20.9, 14.7; ^{31}P NMR (243 MHz, D_2O) δ 19.4; HRMS (ESI) calcd for $[C_8H_{20}N_2O_4P_1]^+$ $(M+H)^+$: m/z 239.1161, found 239.1157.

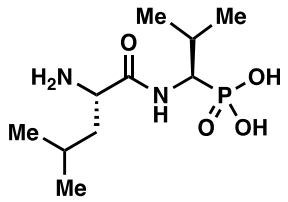


Following the general procedure, aminomethylphosphonate (58 mg, 0.52 mmol, 1 equiv) and Z-L-Leu (133 mg, 0.5 mmol, 1 equiv) afforded 100 mg (89%) of compound **2** as a clear oil. ^{1}H NMR (600 MHz, D_2O) δ 4.02 (t, J = 7.2 Hz, 1H), 3.58 (t, J = 14.1 Hz, 1H), 3.23 (t, J = 13.4 Hz, 1H), 1.78 (d, J = 7.2 Hz, 1H), 1.75 – 1.65 (m, 2H), 0.97 (dd, J = 10.1, 6.5 Hz, 6H); ^{13}C NMR (150 MHz, D_2O) δ 169.8, 52.2, 39.6, 38.1 (d, J = 141.7 Hz), 23.8, 21.6, 21.0; ^{31}P NMR (243 MHz, D_2O) δ 14.7; HRMS (ESI) calcd for $[C_7H_{18}N_2O_4P_1]^+$ $(M+H)^+$: m/z 225.1004, found 225.1001.

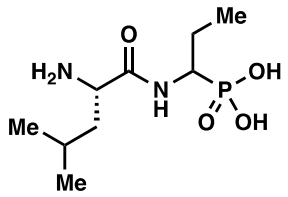


Following the general procedure, aminophenylmethylphosphonate (434 mg, 2.4 mmol, 1 equiv) and Z-L-Leu (610 mg, 2.3 mmol, 1 equiv) afforded 440 mg (65%) of compound **3** as a white solid as a 1.5:1 mixture of diastereomers. major isomer: ^{1}H NMR (600 MHz, D_2O) δ 7.29 – 7.12 (m, 5H), 5.06 (d, *J* = 23.4, 1H), 3.93 (dd, *J* = 8.3, 6.0 Hz, 1H), 1.68 – 1.36 (m, 2H), 1.32 – 1.20 (m, 1H), 0.67 (d, *J* = 6.7 Hz, 3H), 0.62 (d, *J* = 6.7 Hz, 3H); ^{31}P NMR (243 MHz, D_2O) δ 15.6. minor isomer: ^{1}H NMR (600 MHz, D_2O) δ 7.29 – 7.12 (m, 5H), 5.02 (d, *J* =,20.4 Hz, 1H), 3.93 (dd, *J* = 8.3, 6.0 Hz, 1H), 1.68 – 1.36 (m, 2H), 1.08 (td, *J* = 7.2, 2.4 Hz, 1H), 0.79 (dd, *J* = 14.0, 6.6, 6H); ^{31}P NMR (243 MHz, D_2O) δ 16.0. both isomers:^{13}C NMR (150 MHz, D_2O) δ 169.9, 169.9, 137.1, 136.9, 128.6, 128.4, 127.6, 127.6, 127.4, 127.4, 127.4, 127.2, 127.2, 54.1 (d, *J* = 142.2 Hz), 53.5 (d, *J* = 141.6

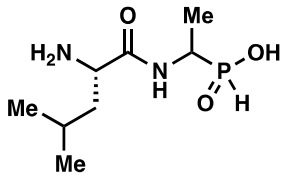
Hz), 52.0, 51.9, 39.8, 39.6, 23.8, 23.7, 21.8, 21.5, 21.1, 20.9; HRMS (ESI) calcd for $[C_{13}H_{22}N_2O_4P_1]^+$ $(M+H)^+$: m/z 301.1317, found 301.1316.



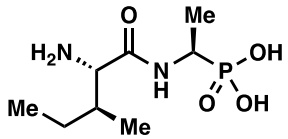
Following the general procedure, L-Val(P) (61 mg, 0.4 mmol, 1 equiv) and Z-L-Leu (106 mg, 0.4 mmol, 1 equiv) afforded 22 mg (21%) of compound **4** as a white solid.
^{1}H NMR (600 MHz, D_2O) δ 4.11 (t, J = 7.0 Hz, 1H), 3.92 (dd, J = 17.1, 5.5 Hz, 1H), 2.22 – 2.12 (m, 1H), 1.86 – 1.78 (m, 1H), 1.78 – 1.70 (m, 2H), 1.02 – 0.96 (m, 12H); ^{13}C NMR (150 MHz, D_2O) δ 170.1 (d, J = 5.8 Hz), 54.0 (d, J = 145.4 Hz), 52.0, 39.8, 28.8 (d, J = 2.4 Hz), 23.6, 21.8, 20.7, 20.3 (d, J = 9.9 Hz), 17.7 (d, J = 5.7 Hz); ^{31}P NMR (243 MHz, D_2O) δ 17.1; HRMS (ESI) calcd for $[C_{10}H_{24}N_2O_4P_1]^+$ $(M+H)^+$: m/z 267.1474, found 267.1476.



Following the general procedure, 1-aminopropylphosphonate (56 mg, 0.4 mmol, 1 equiv) and Z-L-Leu (106 mg, 0.4 mmol, 1 equiv) afforded 17 mg (17%) of compound **5** as a white solid as a 1.1:1 mixture of diastereomers.
^{1}H NMR (600 MHz, D_2O) δ 4.31 (q, J = 7.2 Hz, 1H), 4.17 – 4.09 (m, 1H), 4.02 (t, J = 7.5, 1H), 3.99 – 3.90 (t, J = 14.9, 1H), 1.95 – 1.84 (m, 2H), 1.83 – 1.63 (m, 4H), 1.61 – 1.54 (m, 2H), 1.16 (t, J = 7.2, 1H), 1.27 – 1.22 (m, 1H), 1.01 – 0.91 (m, 18H); ^{13}C NMR (150 MHz, D_2O) δ 170.0, 169.9, 52.3, 51.4, 50.5 (d, J = 147.1 Hz), 39.7, 38.7, 23.9, 22.6, 21.4, 21.2, 20.9, 13.8, 13.1, 10.6, 10.5; ^{31}P NMR (243 MHz, D_2O) δ 17.94; HRMS (ESI) calcd for $[C_9H_{22}N_2O_4P_1]^+$ $(M+H)^+$: m/z 253.1317, found 253.1319.

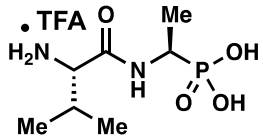


Following the general procedure, 1-aminoethylphosphinic acid (44 mg, 0.4 mmol, 1 equiv) and Z-L-Leu (106 mg, 0.4 mmol, 1 equiv) afforded 31 mg (35%) of compound **6** as a white solid as a 1.8:1 mixture of diastereomers.
^{1}H NMR (600 MHz, D_2O) δ 6.8 (d, J = 519.4 Hz, 1H), 4.1 – 3.9 (m, 2H), 1.8 – 1.6 (m, 3H), 1.4 – 1.2 (m, 3H), 1.0 – 0.9 (m, 6H); ^{13}C NMR (150 MHz, D_2O) δ 169.6 (d, J = 4.0 Hz), 169.2 (d, J = 5.3 Hz), 52.2, 52.1, 45.9 (d, J = 99.8 Hz), 44.4, 43.4, 39.7, 39.6, 23.9, 23.8, 21.5, 21.5, 21.2, 21.2, 15.1, 11.9; ^{31}P NMR (243 MHz, D_2O) δ 25.4, 18.7; HRMS (ESI) calcd for $[C_8H_{20}N_2O_3P_1]^+$ $(M+H)^+$: m/z 223.1212, found 223.1208.



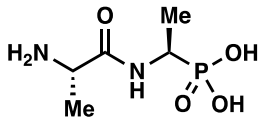
Following the general procedure, L-Ala(P) (25 mg, 0.2 mmol, 1 equiv) and Z-L-isoleucine (53 mg, 0.2 mmol, 1 equiv) afforded 35 mg (78%) of compound **7** as a clear oil.

^{1}H NMR (600 MHz, D_2O) δ 4.3 (dq, *J* = 14.7, 7.4 Hz, 1H), 3.8 (d, *J* = 5.7 Hz, 1H), 2.0 – 1.9 (m, 1H), 1.6 – 1.5 (m, 1H), 1.3 (dd, *J* = 16.6, 7.4 Hz, 3H), 1.3 – 1.2 (m, 1H), 1.0 (d, *J* = 6.9 Hz, 3H), 0.9 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (150 MHz, D_2O) δ 168.3, 57.8, 42.7 (d, *J* = 154.3 Hz), 36.4, 24.0, 14.2, 13.8, 10.4; ^{31}P NMR (243 MHz, D_2O) δ 22.3; HRMS (ESI) calcd for $[C_8H_{20}N_2O_4P_1]^+$ $(M+H)^+$: m/z 239.1161, found 239.1158.



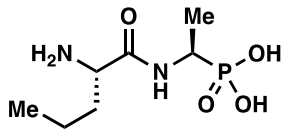
Following the general procedure, L-Ala(P) (25 mg, 0.2 mmol, 1 equiv) and Z-L-valine (50 mg, 0.2 mmol, 1 equiv) afforded 18 mg (40%) of compound **8** as a clear oil. For the purification 0.1% trifluoroacetic acid in H_2O was used as a mobile phase for C18 reversed-phase chromatography.

^{1}H NMR (600 MHz, D_2O) δ 4.26 – 4.19 (m, 1H), 3.80 (d, *J* = 8.3 Hz, 1H), 2.28 – 2.20 (m, 1H), 1.37 (dd, *J* = 16.3, 7.1 Hz, 3H), 1.06 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (150 MHz, D_2O) δ 168.3 (d, *J* = 5.3 Hz), 162.9 (q, *J* = 34.8 Hz), 116.2 (q, *J* = 290.0 Hz), 58.7, 43.1 (d, *J* = 151.9 Hz), 30.0, 17.4, 16.8, 14.6; ^{31}P NMR (243 MHz, D_2O) δ 20.7; HRMS (ESI) calcd for $[C_7H_{18}N_2O_4P_1]^+$ $(M+H)^+$: m/z 225.1004, found 225.1003.



Following the general procedure, L-Ala(P) (25 mg, 0.2 mmol, 1 equiv) and Z-L-alanine (45 mg, 0.2 mmol, 1 equiv) afforded 25 mg (64%) of compound **9** as a clear oil.

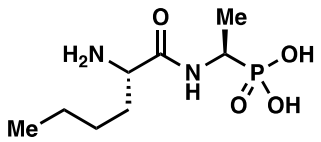
^{1}H NMR (600 MHz, D_2O) δ 4.20 – 3.98 (m, 2H), 1.55 (d, *J* = 6.9 Hz, 3H), 1.31 (dd, *J* = 14.7, 6.9 Hz, 3H); ^{13}C NMR (150 MHz, D_2O) δ 169.7, 49.3, 44.2 (d, J = 146.2 Hz), 16.4, 15.4; ^{31}P NMR (243 MHz, D_2O) δ 18.1; HRMS (ESI) calcd for $[C_5H_{14}N_2O_4P_1]^+$ $(M+H)^+$: m/z 197.0691, found 197.0689.



Following the general procedure, L-Ala(P) (25 mg, 0.2 mmol, 1 equiv) and Z-L-norvaline (50 mg, 0.2 mmol, 1 equiv) afforded 44 mg (98%) of compound **10** as a clear oil.

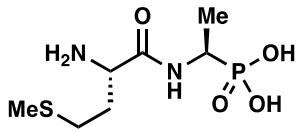
^{1}H NMR (600 MHz, D_2O) δ 4.25 (dq, *J* = 15.0, 7.5 Hz, 1H), 3.95 (t, *J* = 6.8 Hz, 1H), 1.86 – 1.71 (m, 2H), 1.42 – 1.29 (m, 5H), 0.90 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, D_2O)

δ 169.2, 53.1, 42.7 (d, J = 153.8 Hz), 32.9, 17.4, 14.2, 12.8; ^{31}P NMR (243 MHz, D_2O) δ 22.5; HRMS (ESI) calcd for $[C_7H_{18}N_2O_4P_1]^+$ $(M+H)^+$: m/z 225.1004, found 225.1000.



Following the general procedure, L-Ala(P) (25 mg, 0.2 mmol, 1 equiv) and Z-L-norleucine (53 mg, 0.2 mmol, 1 equiv) afforded 39 mg (82%) of compound **11** as a white solid.

^{1}H NMR (600 MHz, D_2O) δ 4.08 (dq, J = 14.8, 7.4 Hz, 1H), 3.97 (t, J = 6.8 Hz, 1H), 2.00 – 1.82 (m, 2H), 1.44 – 1.37 (m, 4H), 1.34 (dd, J = 14.9, 7.5 Hz, 3H), 0.91 (t, J = 3.3 Hz, 3H); ^{13}C NMR (150 MHz, D_2O) δ 169.1 (d, J = 6.2 Hz), 53.5, 43.9 (d, J = 148.3 Hz), 30.5, 26.1, 21.5, 15.2, 12.9; ^{31}P NMR (243 MHz, D_2O) δ 18.5; HRMS (ESI) calcd for $[C_8H_{20}N_2O_4P_1]^+$ $(M+H)^+$: m/z 239.1161, found 239.1158.

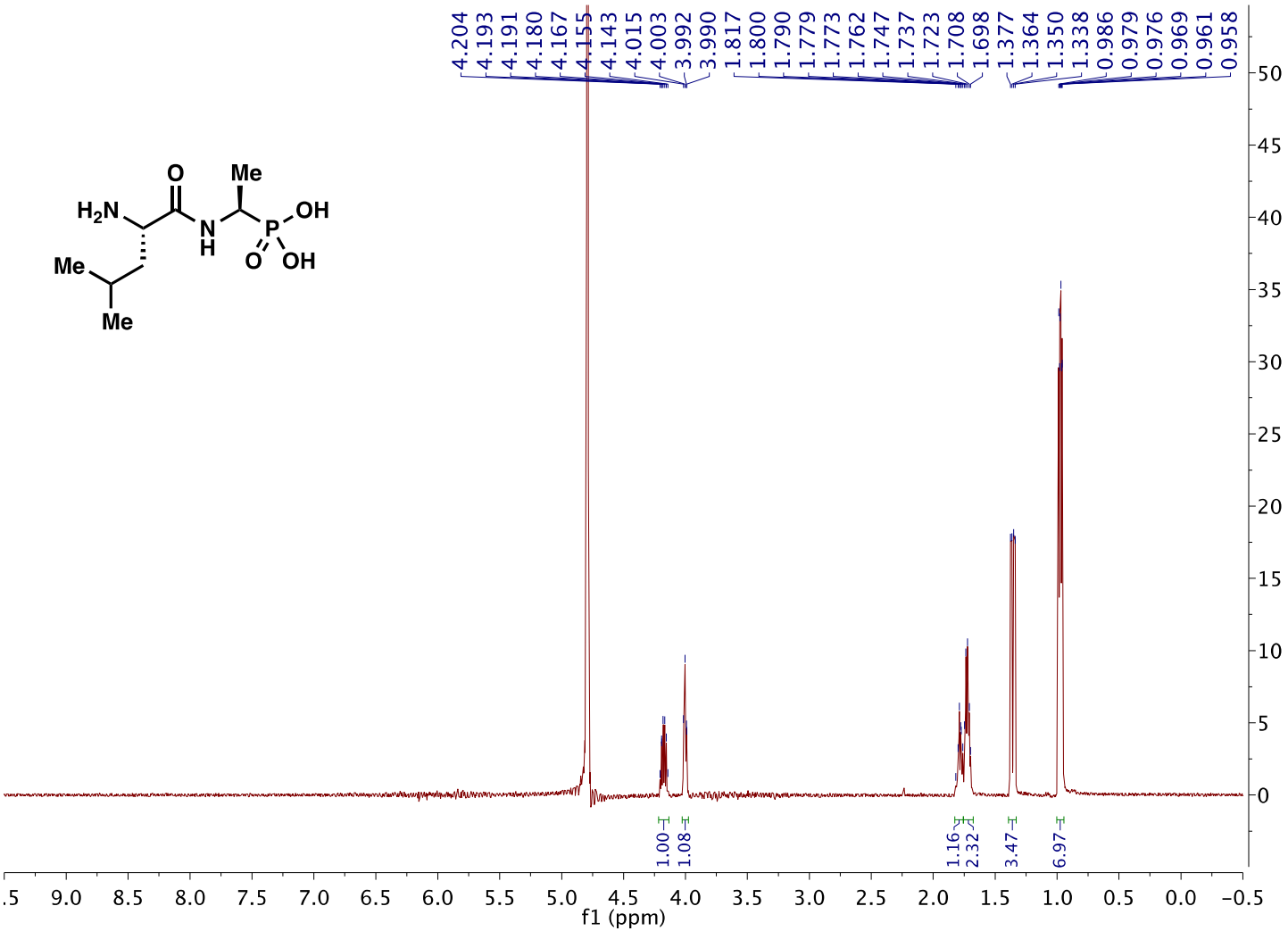


Following the general procedure, L-Ala(P) (25 mg, 0.2 mmol, 1 equiv) and Z-L-methionine (57 mg, 0.2 mmol, 1 equiv) afforded 8.0 mg (16%) of compound **12** as a clear oil.

^{1}H NMR (600 MHz, D_2O) δ 4.22 (dq, J = 14.0, 7.4, 6.7 Hz, 1H), 4.12 (t, J = 6.7 Hz, 1H), 2.72 – 2.58 (m, 2H), 2.27 – 2.15 (m, 2H), 2.13 (s, 3H), 1.36 (dd, J = 16.5, 7.4 Hz, 3H); ^{13}C NMR (150 MHz, D_2O) δ 168.3 (d, J = 5.4 Hz), 52.4, 43.3 (d, J = 151.5 Hz), 30.0, 28.0, 14.5, 13.9; ^{31}P NMR (243 MHz, D_2O) δ 20.7; HRMS (ESI) calcd for $[C_7H_{18}N_2O_4P_1S_1]^+$ $(M+H)^+$: m/z 257.0725, found 257.0723.

References

1.) V. Solodenko, T. Kasheva and V. Kukhar, Preparation of N-Acylated Phosphonopeptides with Free Phosphonic Group, *Syn. Comm*., 1991, **21**, 1631-1641.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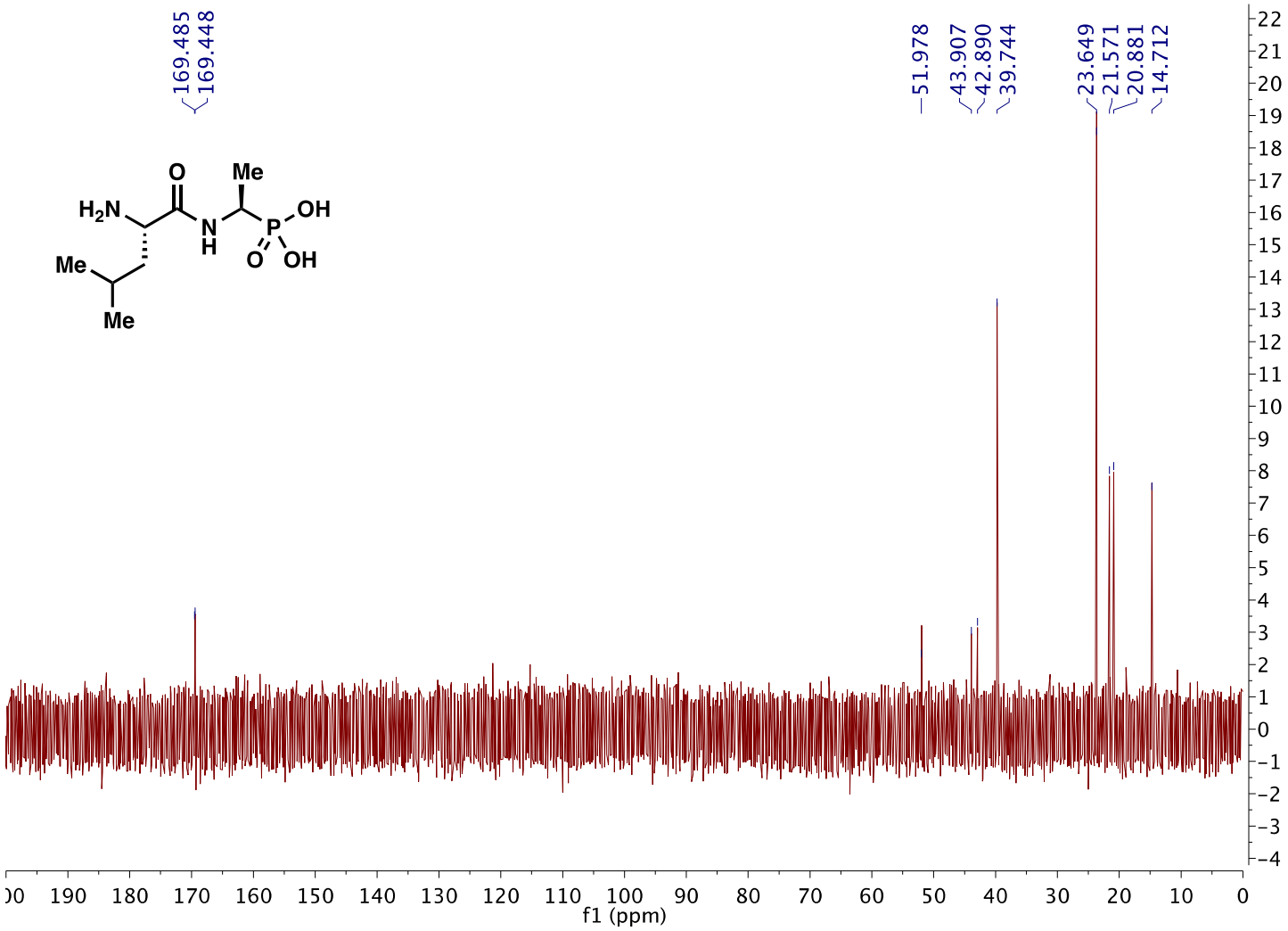
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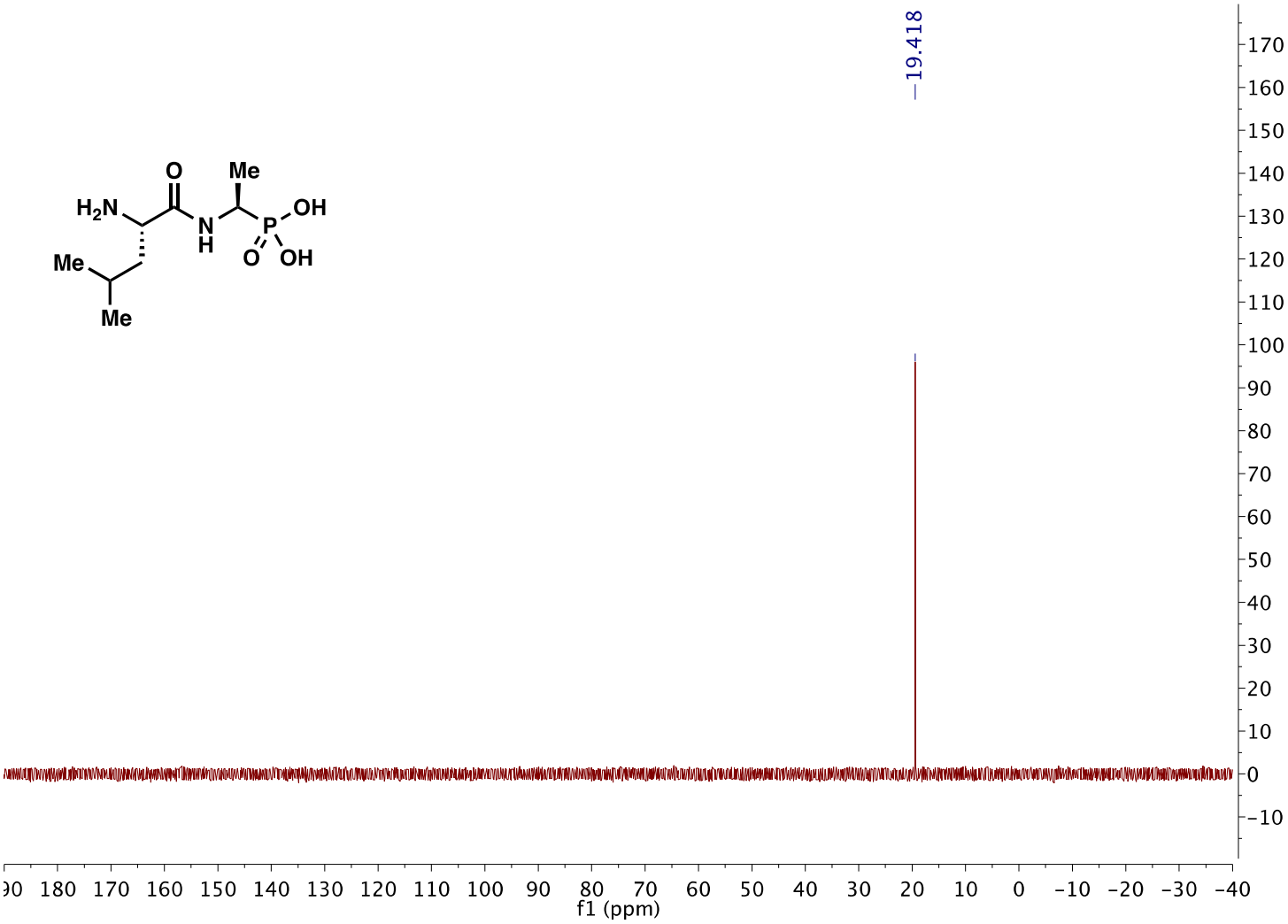
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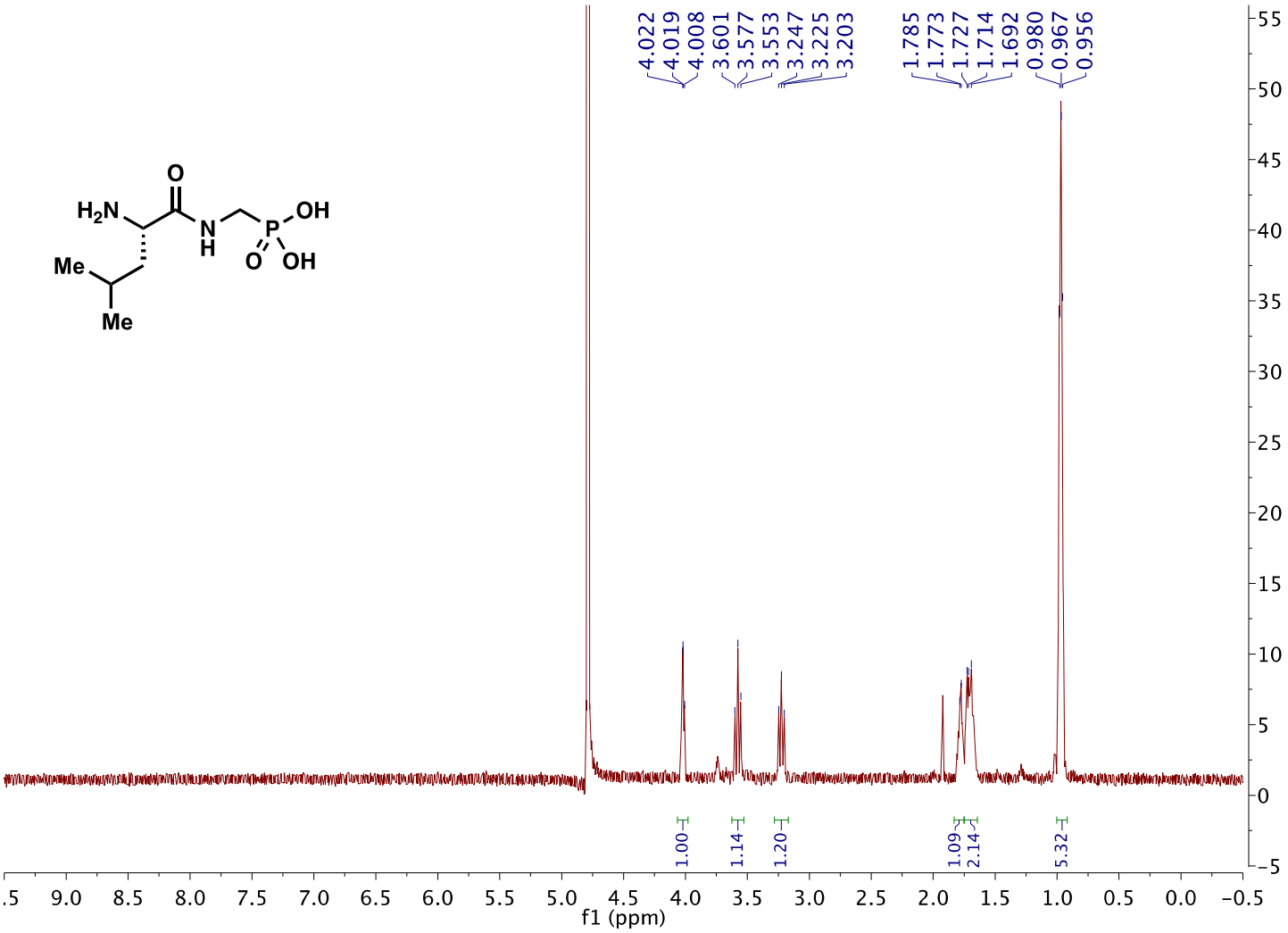
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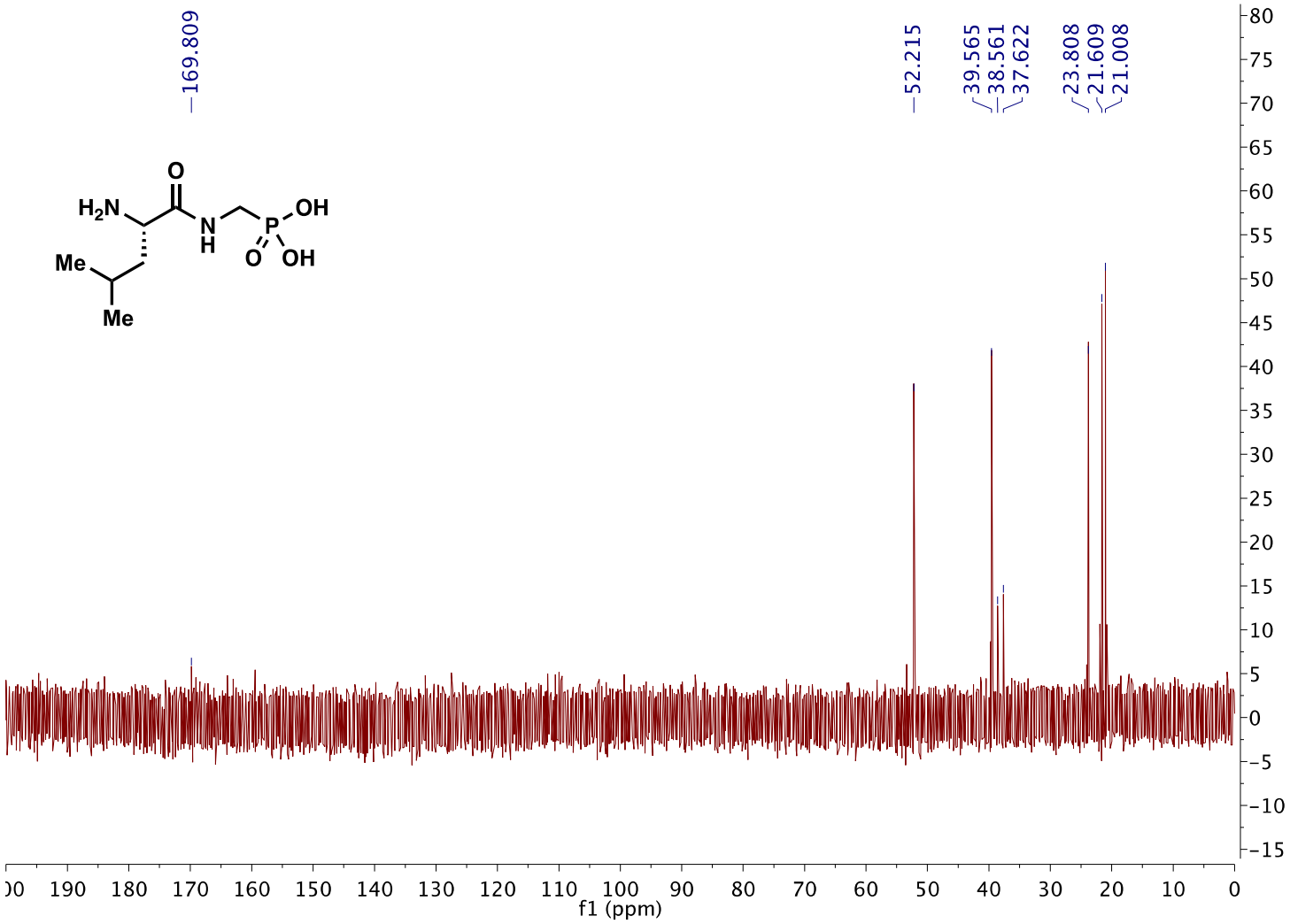
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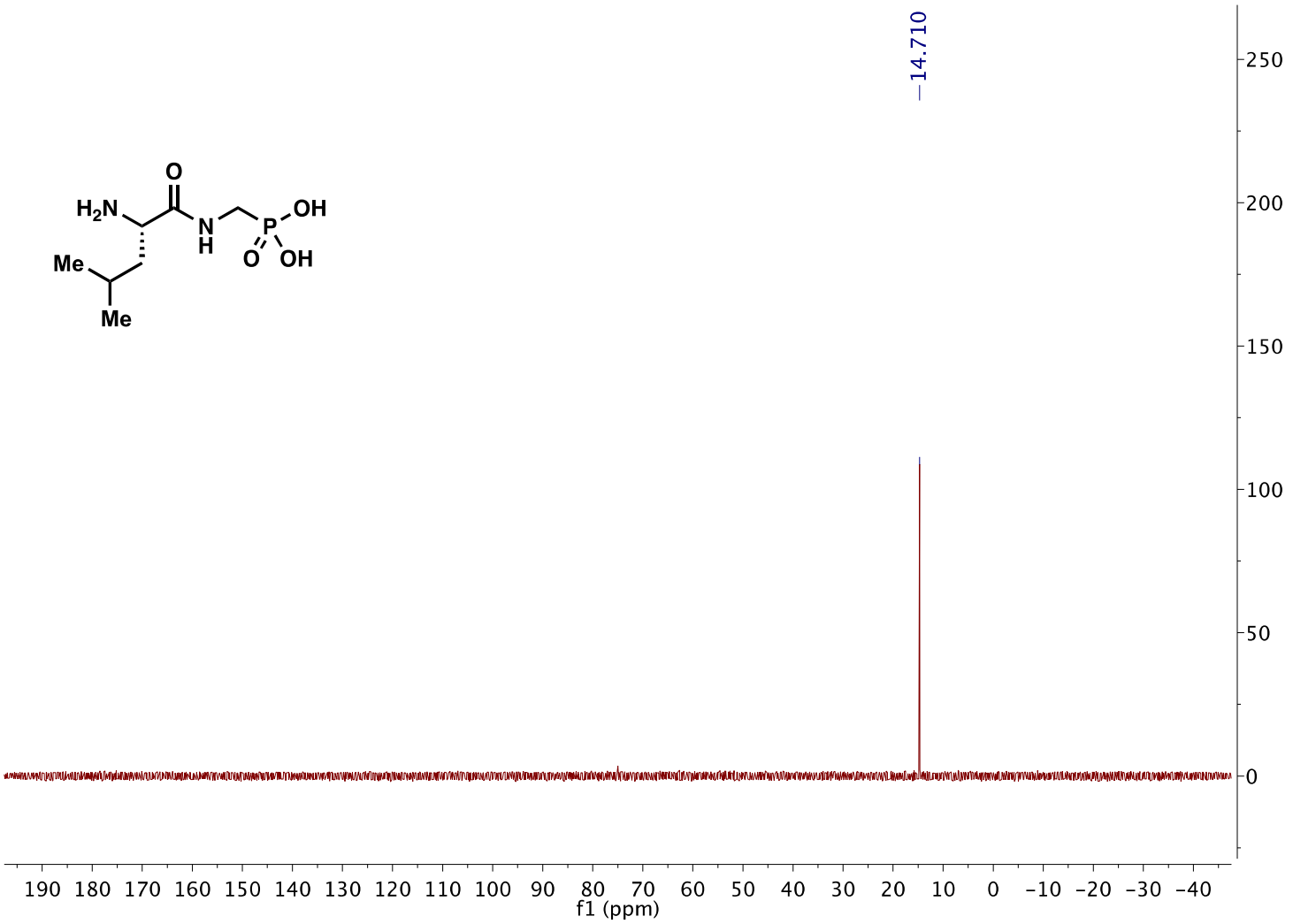
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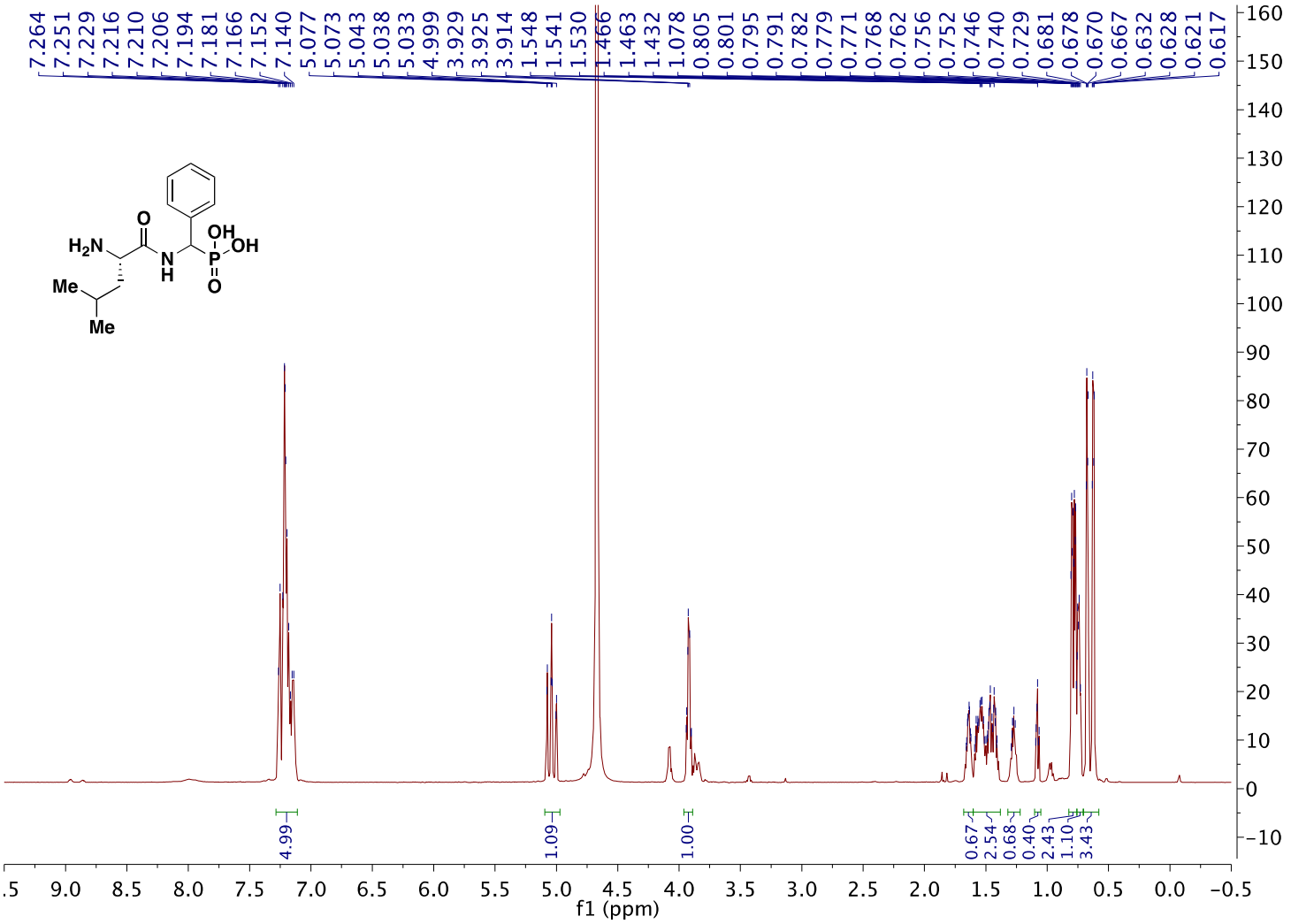
Me
Me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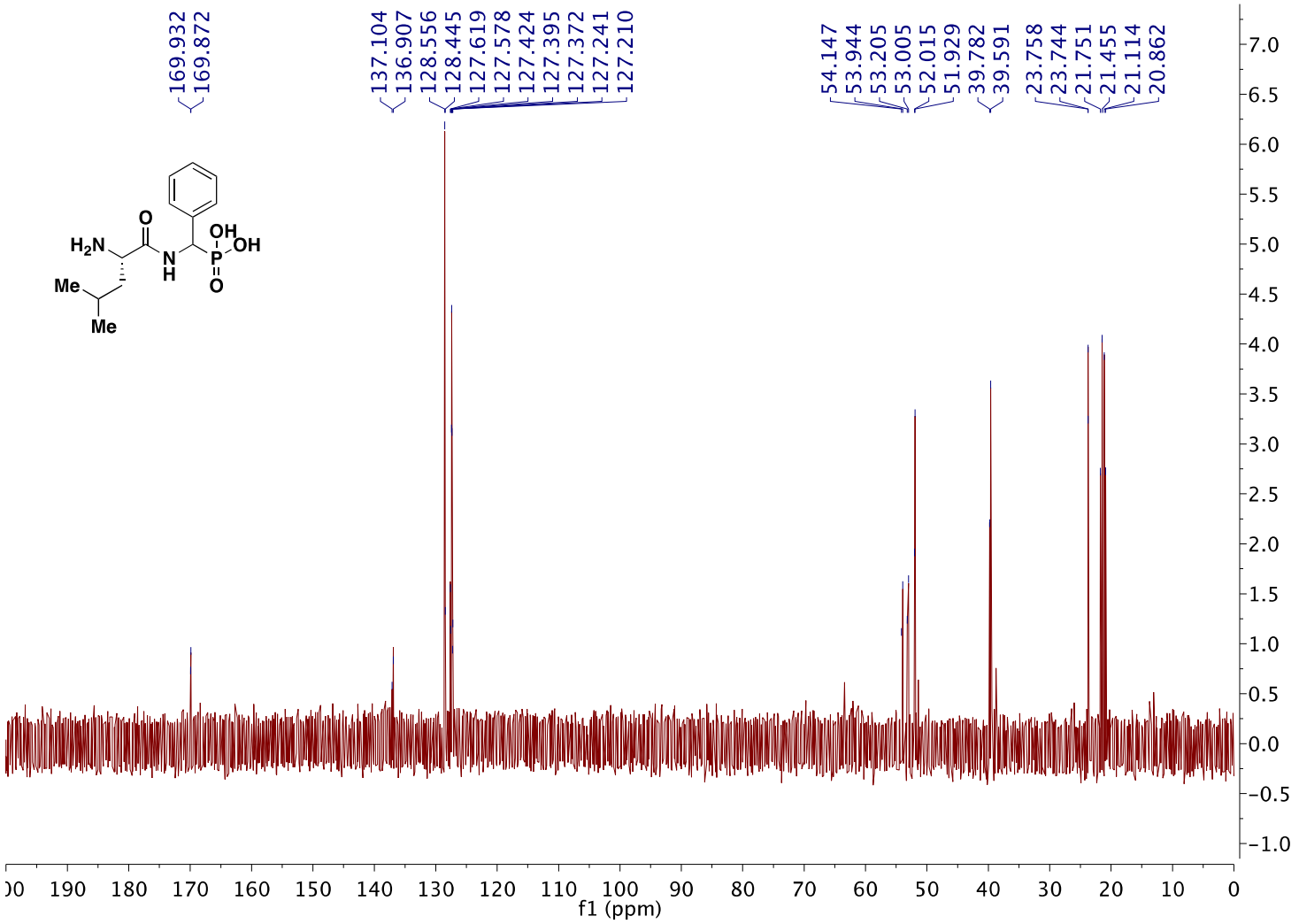
7.251
7.229
7.216
7.210
7.206
7.194
7.181
7.166
7.152
7.140
5.077
5.073
5.043
5.038
5.033
4.999
3.929
3.925
3.914
1.548
1.541
1.530
1.466
1.463
1.432
1.078
0.805
0.801
0.795
0.791
0.782
0.779
0.771
0.768
0.762
0.756
0.752
0.746
0.740
0.729
0.681
0.678
0.670
0.667
0.632
0.628
0.621
0.617
H2N
O
N
H
OH
OH
P
O
Me
Me
4.99
1.09
1.00
0.67
2.54
0.68
0.40
2.43
1.10
3.43
f1 (ppm)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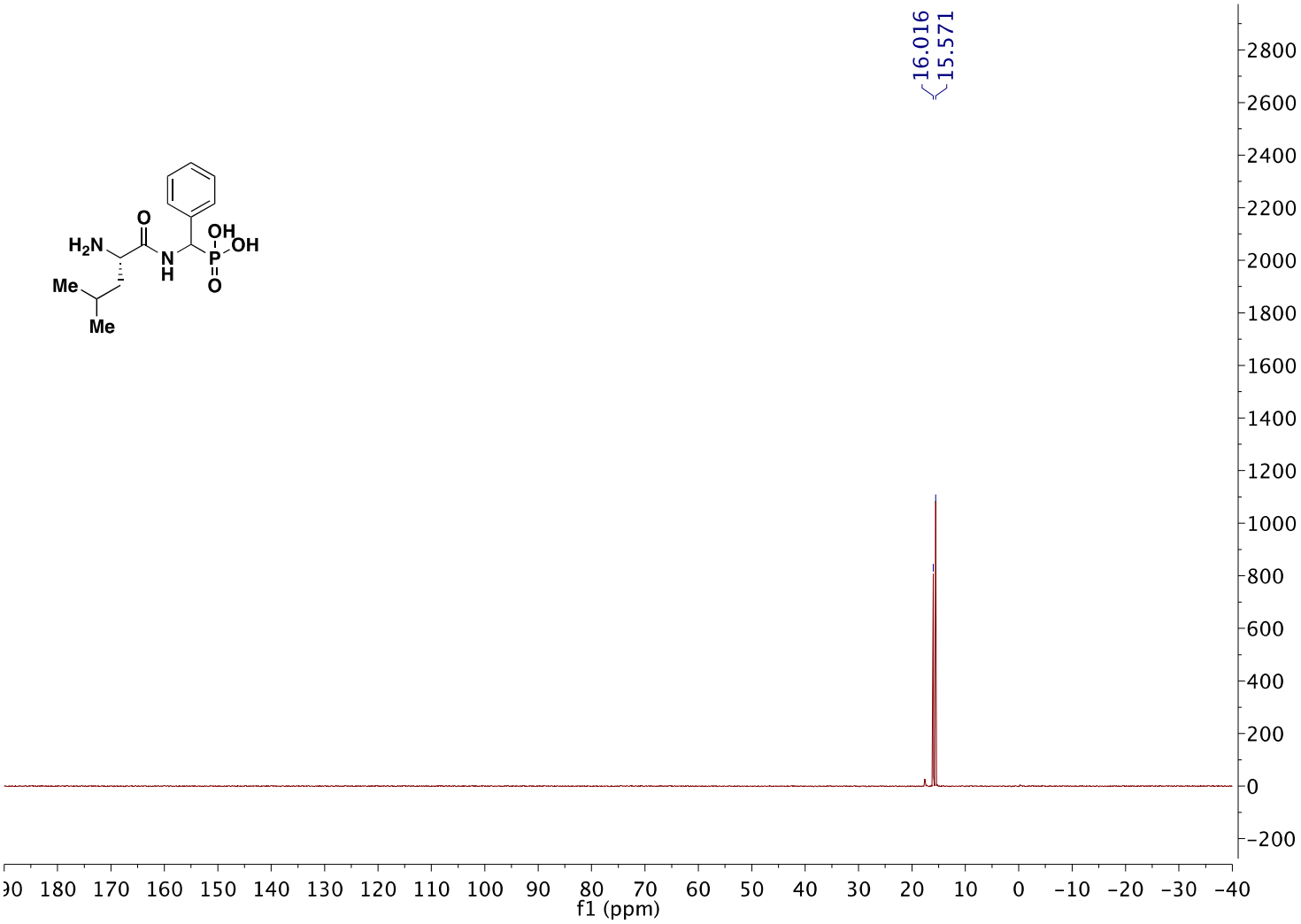
137.104
136.907
128.556
128.445
127.619
127.578
127.424
127.395
127.372
127.241
127.210
54.147
53.944
53.205
53.005
52.015
51.929
39.782
39.591
23.758
23.744
21.751
21.455
21.114
20.862
O
H2N
N
H
OH
P
OH
O
Me
Me
f1 (ppm)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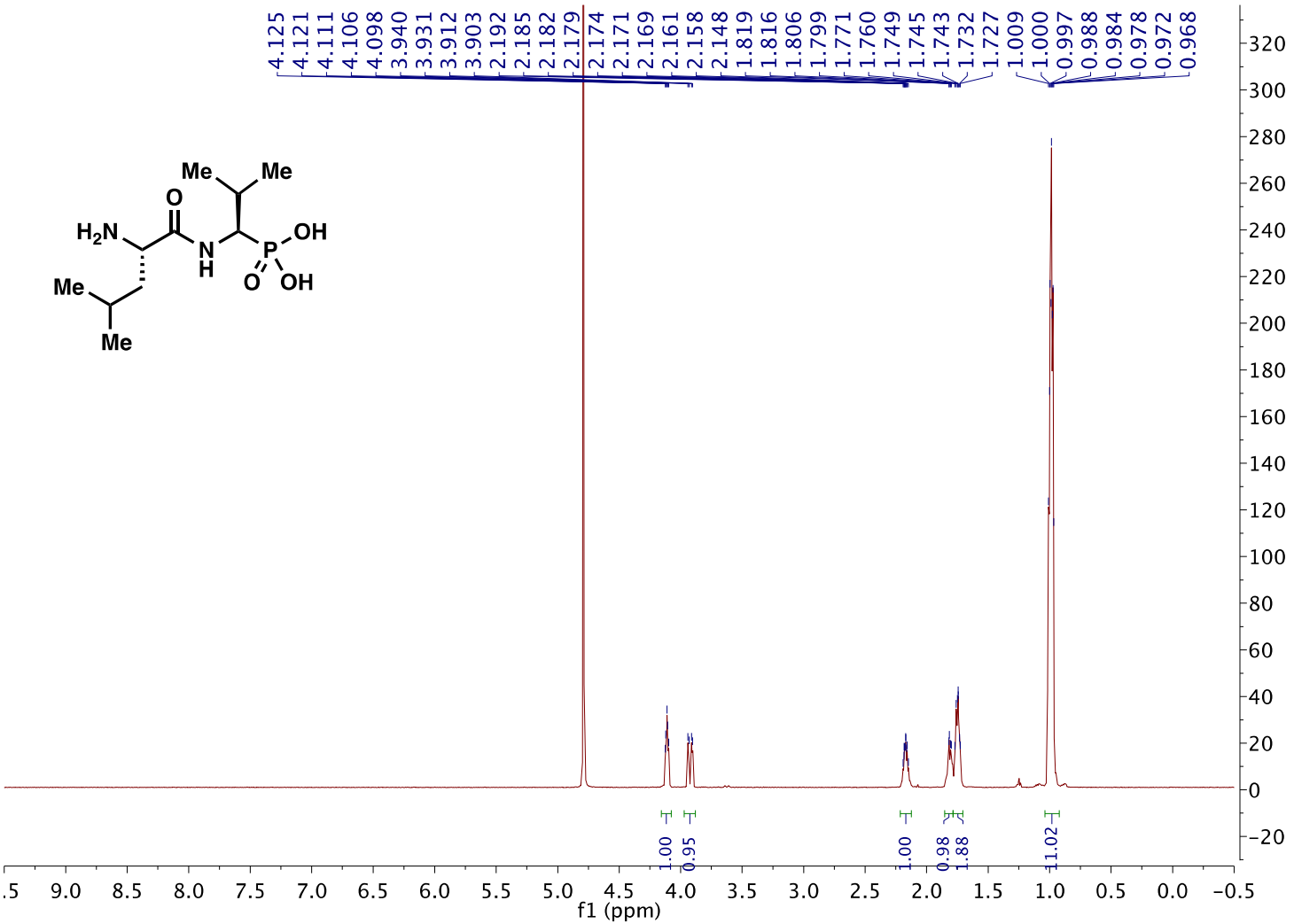
15.571
H2N
O
N
H
OH
OH
P
O
Me
Me
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)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4.121
4.111
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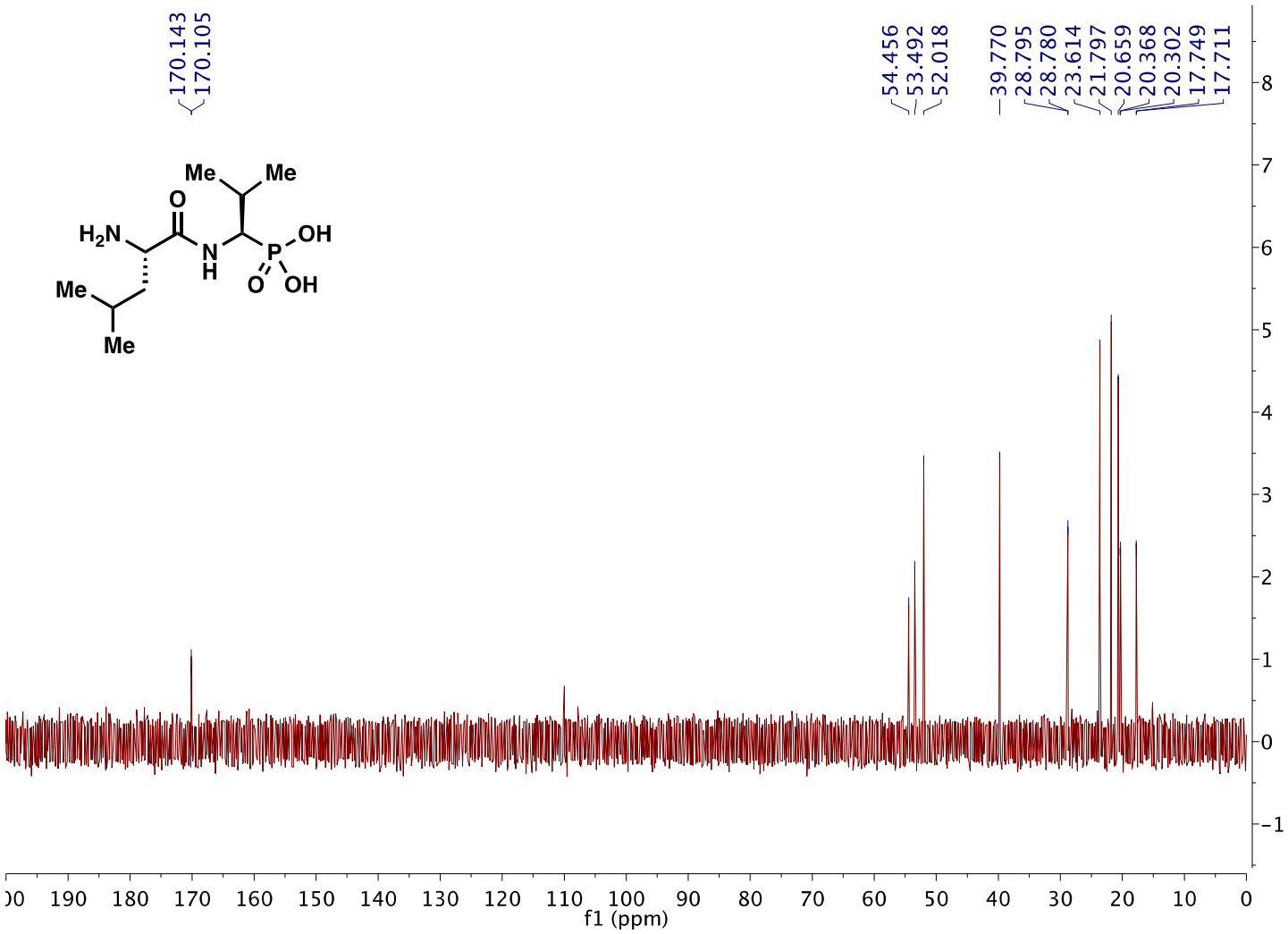
4.098
3.940
3.931
3.912
3.903
2.192
2.185
2.182
2.179
2.174
2.171
2.169
2.161
2.158
2.148
1.819
1.816
1.806
1.799
1.771
1.760
1.749
1.745
1.743
1.732
1.727
1.009
1.000
0.997
0.988
0.984
0.978
0.972
0.968
Me
Me
O
H2N
N
H
P
OH
O
OH
Me
Me
1.00
0.95
1.00
0.98
1.88
11.02
f1 (ppm)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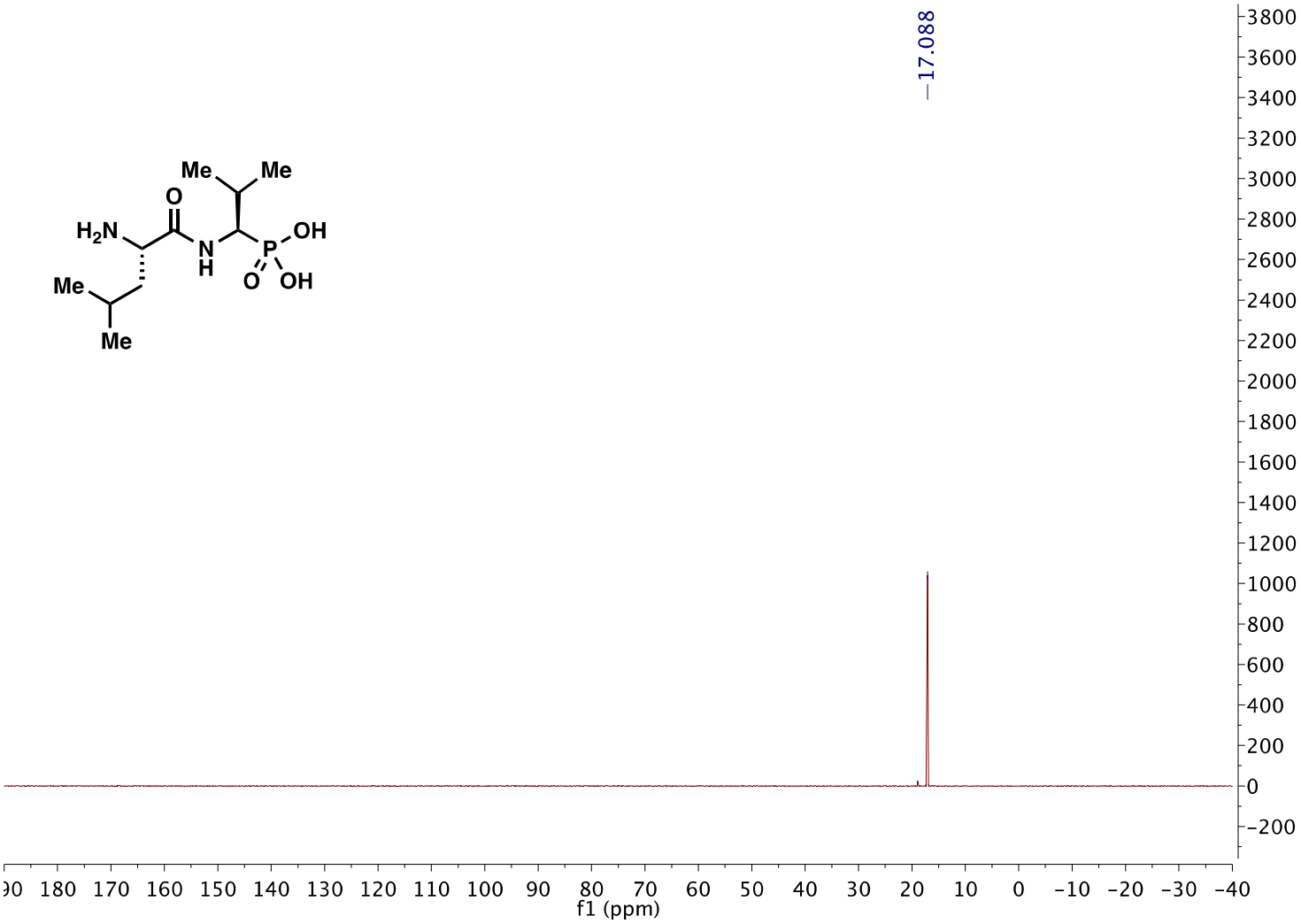
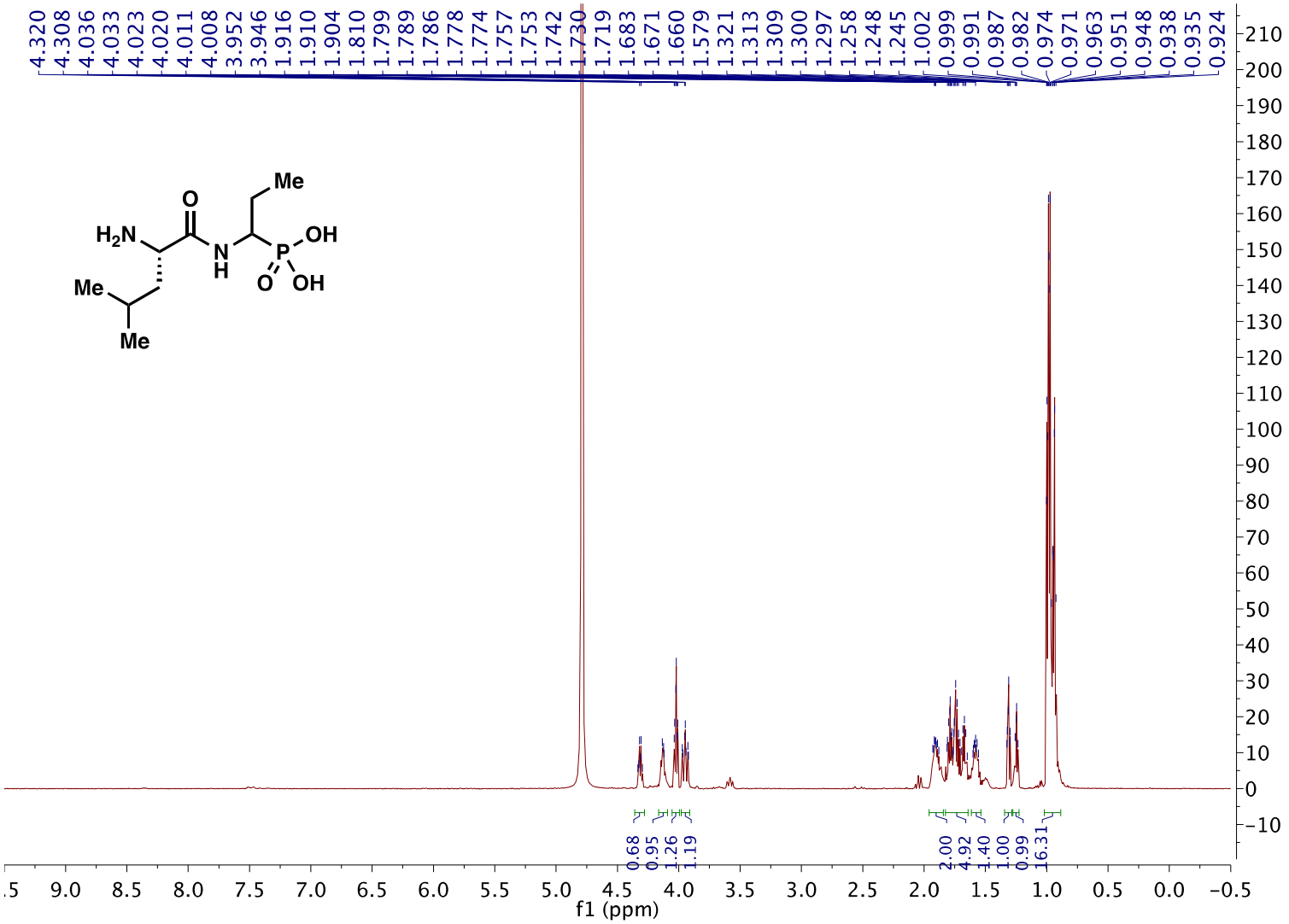
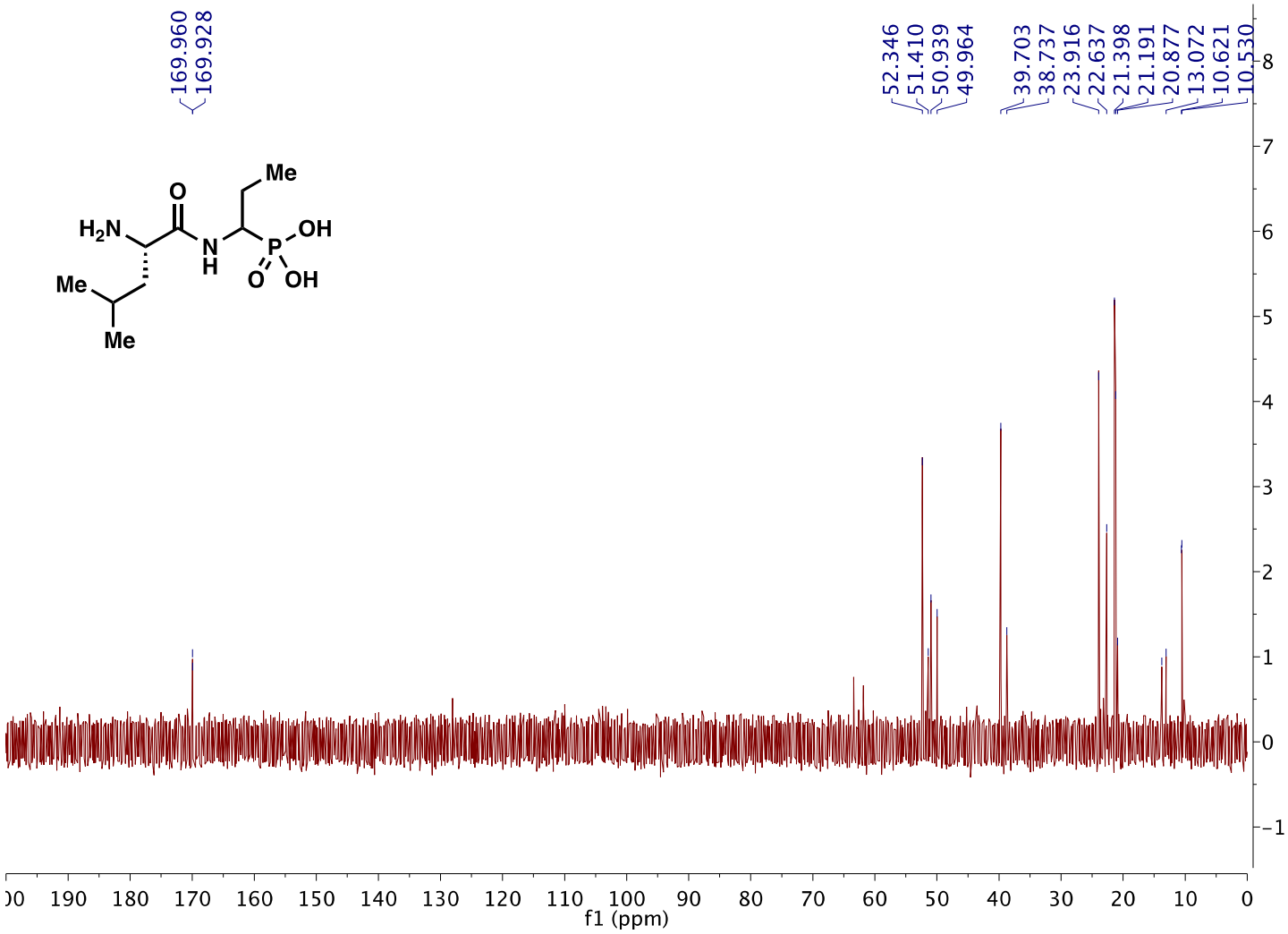
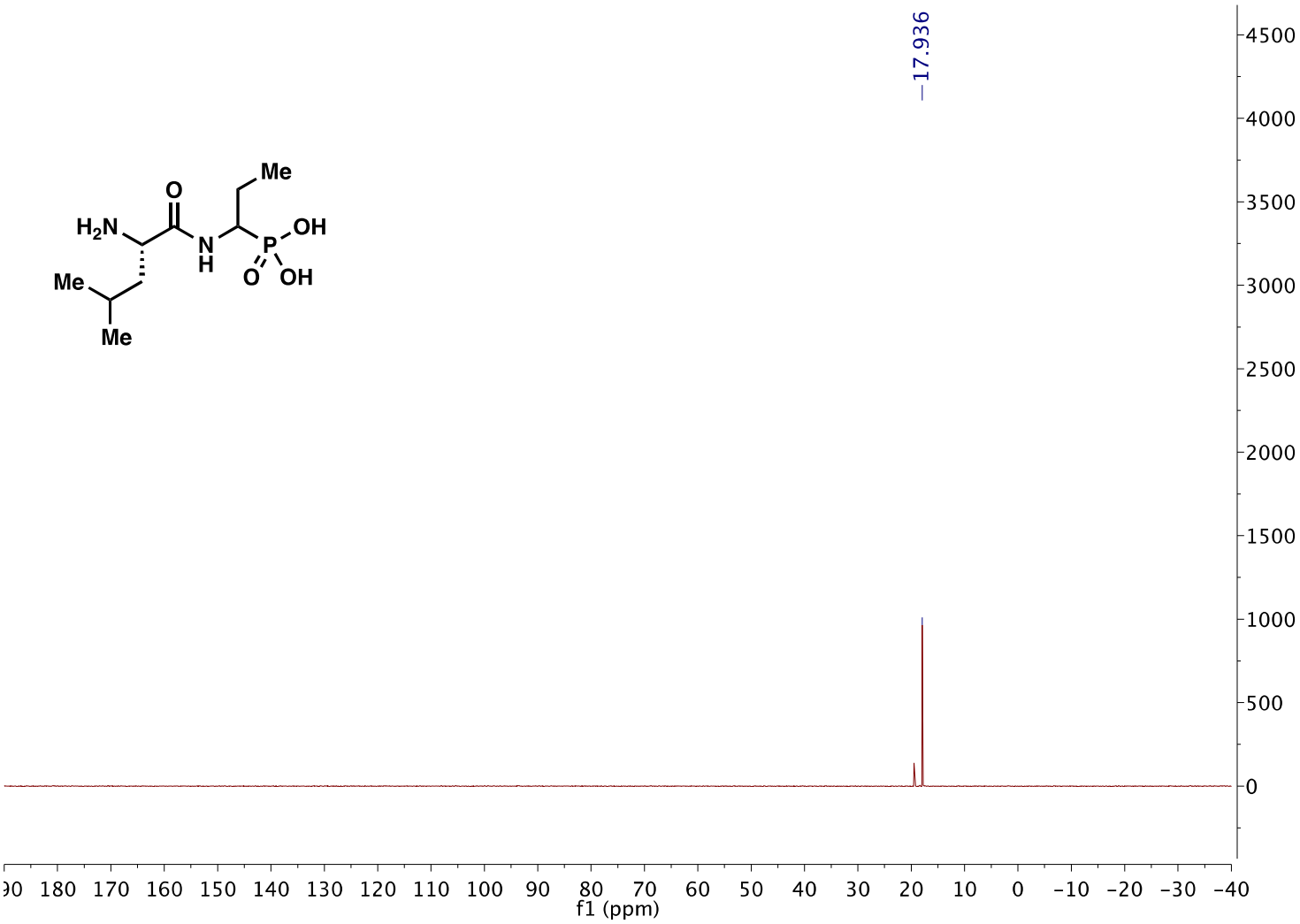
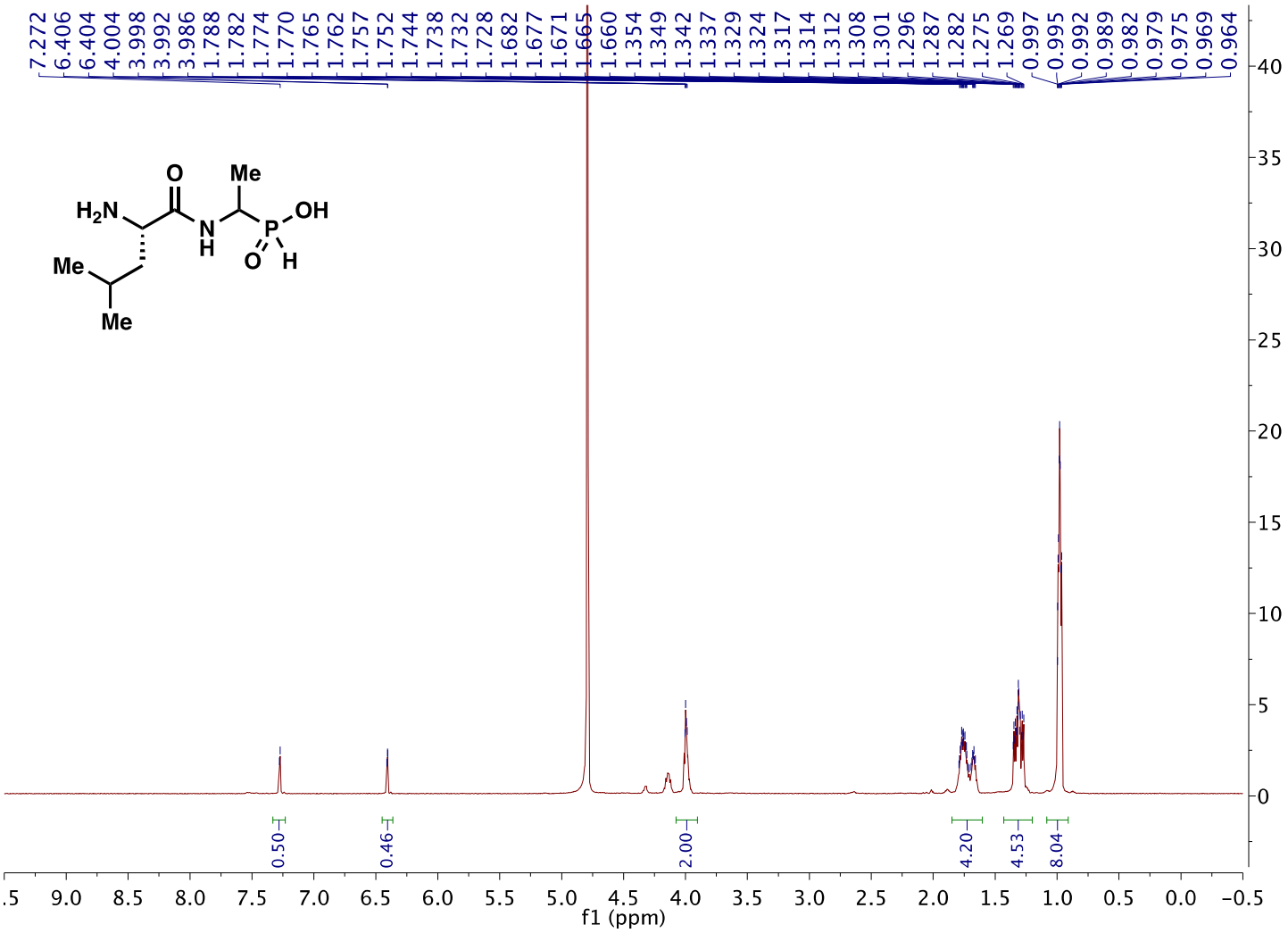
170.105
54.456
53.492
52.018
39.770
28.795
28.780
23.614
21.797
20.659
20.368
20.302
17.749
17.711
Me
Me
O
H2N
N
H
P
OH
O
OH
Me
Me
f1 (ppm)

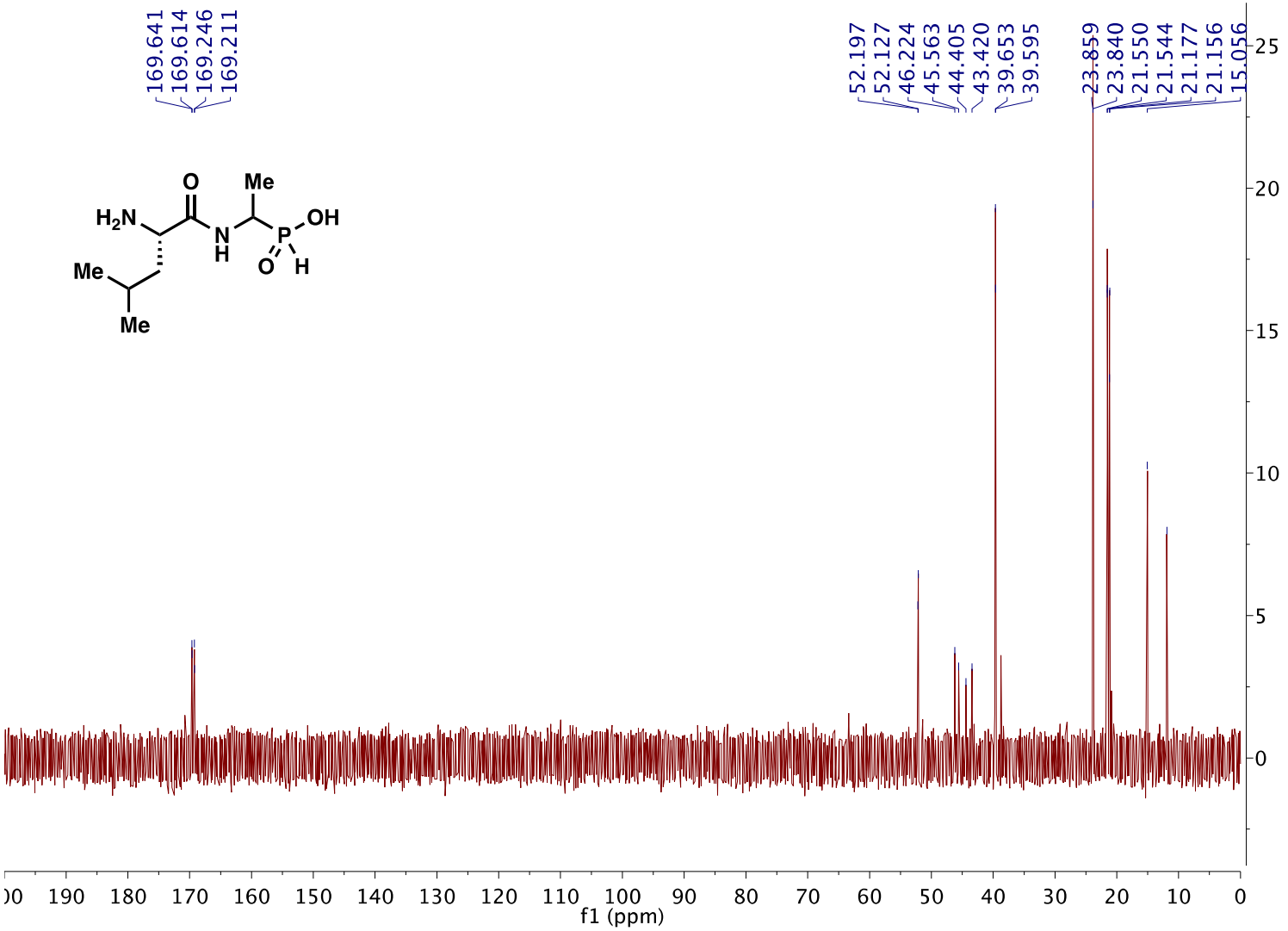

17.088
Me
Me
O
H2N
N
H
P
OH
O
OH
Me
Me
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)

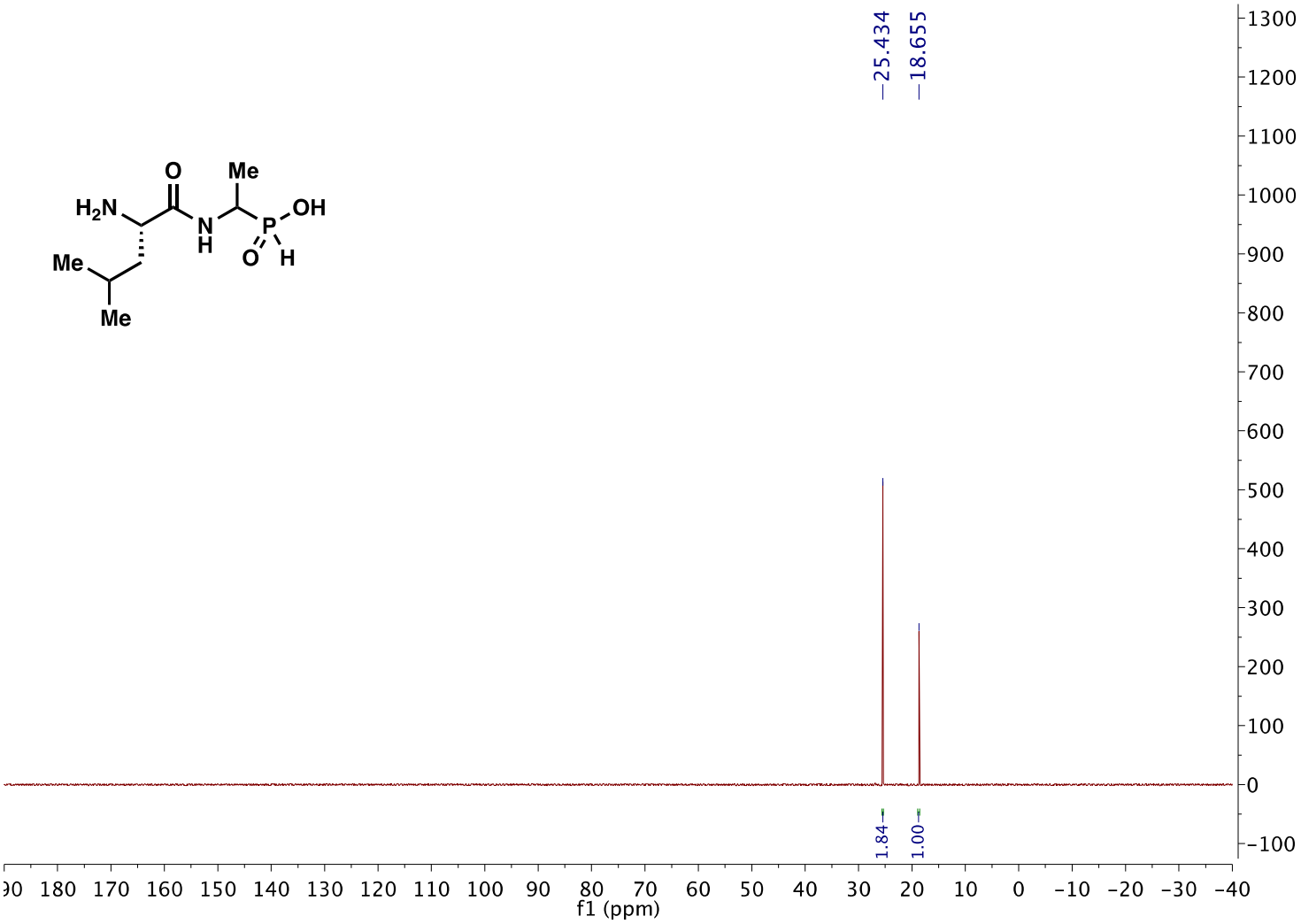

4.320
4.308
4.036
4.033
4.023
4.020
4.011
4.008
3.952
3.946
1.916
1.910
1.904
1.810
1.799
1.789
1.786
1.778
1.774
1.757
1.753
1.742
1.730
1.719
1.683
1.671
1.660
1.579
1.321
1.313
1.309
1.300
1.297
1.258
1.248
1.245
1.002
0.999
0.991
0.987
0.982
0.974
0.971
0.963
0.951
0.948
0.938
0.935
0.924
Me
O
H2N
N
H
P
OH
O
OH
Me
Me
0.68
0.95
1.26
1.19
2.00
4.92
1.40
1.00
0.99
16.31
f1 (ppm)

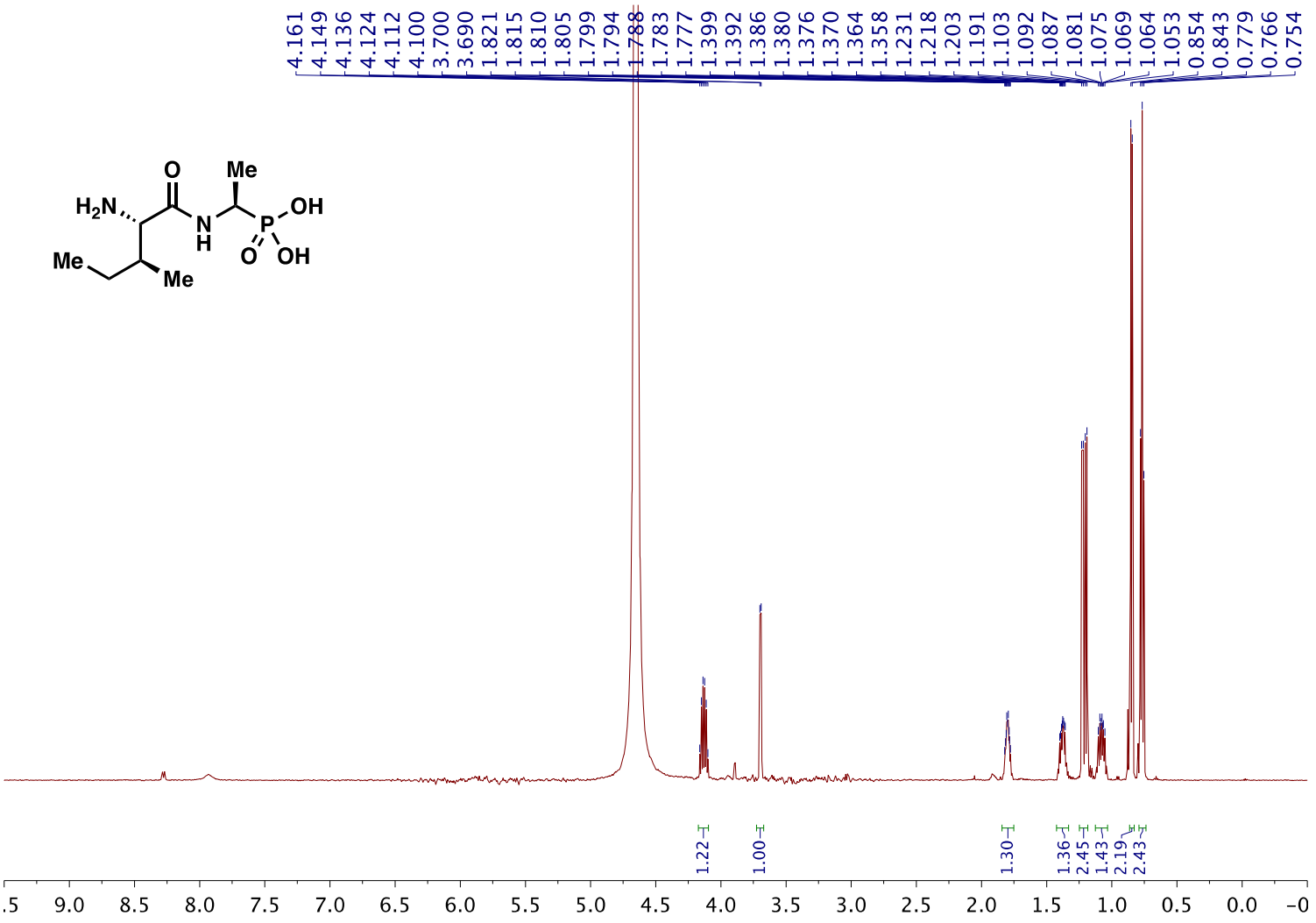

169.960
169.928
52.346
51.410
50.939
49.964
39.703
38.737
23.916
22.637
21.398
21.191
20.877
13.072
10.621
10.530
O
H2N
N
H
Me
P
OH
O
OH
Me
Me
f1 (ppm)

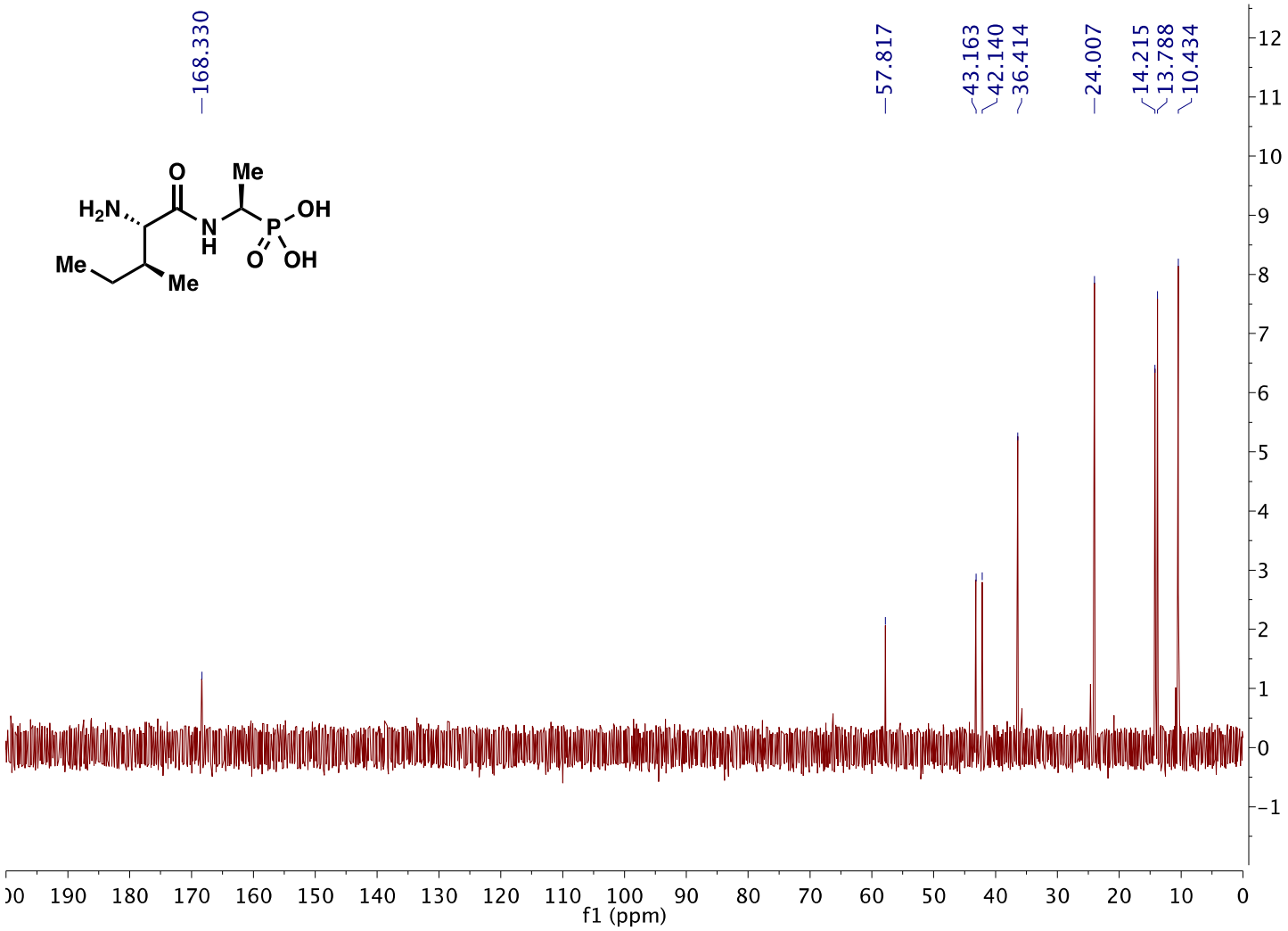

17.936
4500
4000
3500
3000
2500
2000
1500
1000
500
0
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)
Me
O
H2N
N
H
P
OH
O
OH
Me
Me

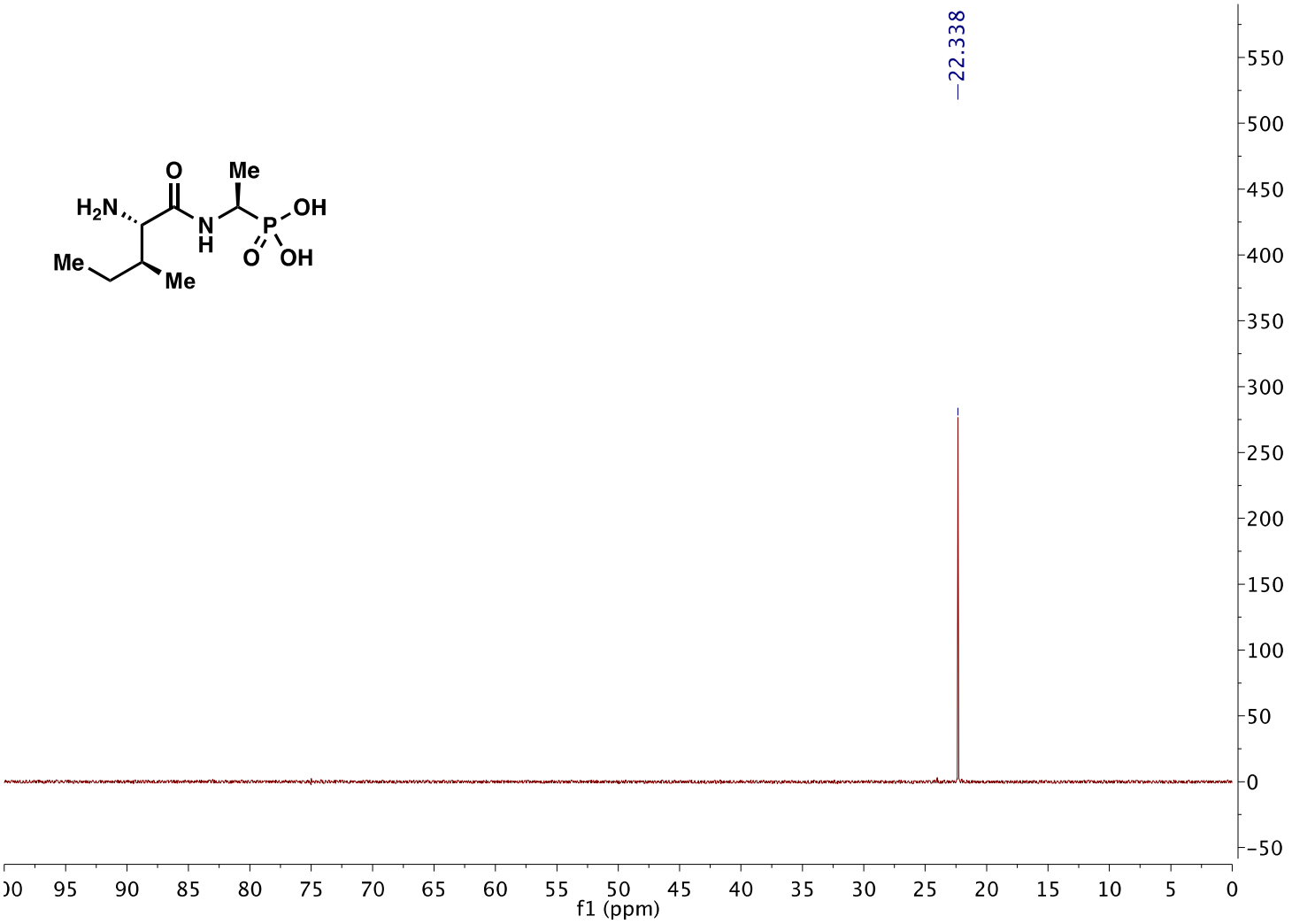

7.272
6.406
6.404
4.004
3.998
3.992
3.986
1.788
1.782
1.774
1.770
1.765
1.762
1.757
1.752
1.744
1.738
1.732
1.728
1.682
1.677
1.671
1.665
1.660
1.354
1.349
1.342
1.337
1.329
1.324
1.317
1.314
1.312
1.308
1.301
1.296
1.287
1.282
1.275
1.269
0.997
0.995
0.992
0.989
0.982
0.979
0.975
0.969
0.964
O
Me
H2N
OH
N
H
P
O
H
Me
Me
0.50
0.46
2.00
4.20
4.53
8.04
f1 (ppm)

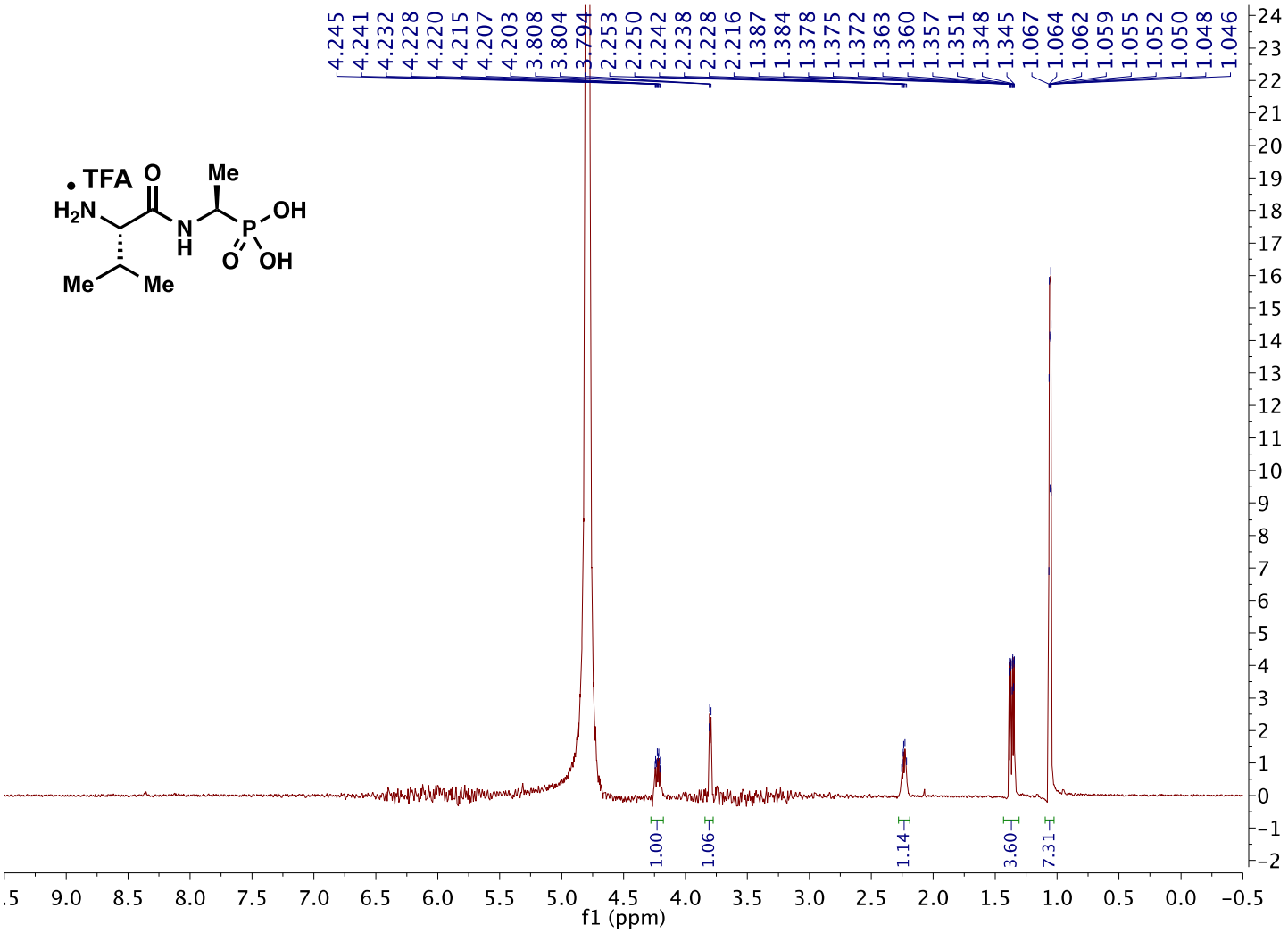

169.641
169.614
169.246
169.211
52.197
52.127
46.224
45.563
44.405
43.420
39.653
39.595
23.859
23.840
21.550
21.544
21.177
21.156
15.056
O
Me
H2N
OH
N
H
P
O
H
Me
Me
25
20
15
10
5
0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

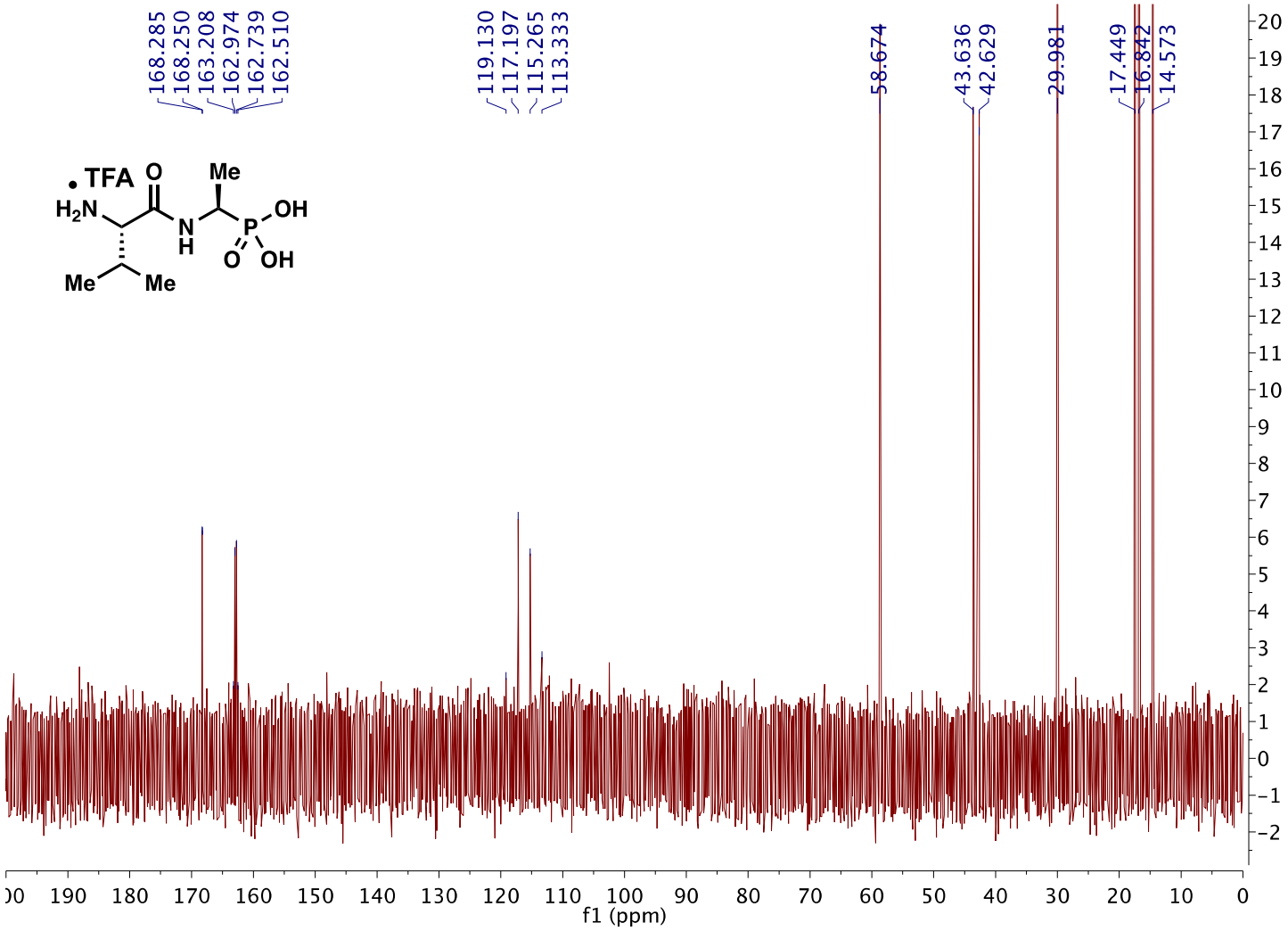

25.434
18.655
1.84
1.00
f1 (ppm)

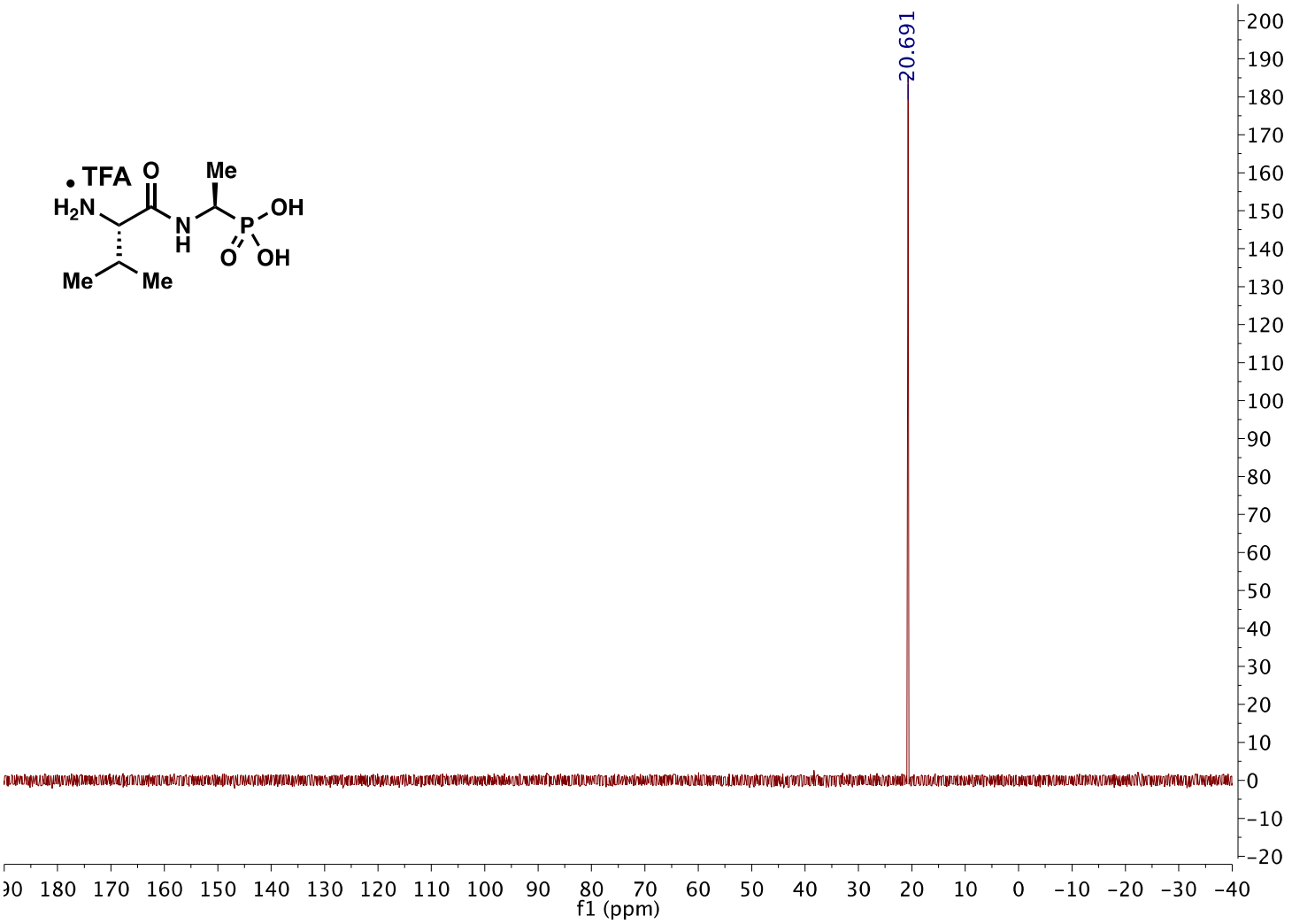

4.161
4.149
4.136
4.124
4.112
4.100
3.700
3.690
1.821
1.815
1.810
1.805
1.799
1.794
1.788
1.783
1.777
1.399
1.392
1.386
1.380
1.376
1.370
1.364
1.358
1.231
1.218
1.203
1.191
1.103
1.092
1.087
1.081
1.075
1.069
1.064
1.053
0.854
0.843
0.779
0.766
0.754
O
Me
H2N
N
H
P
OH
O
OH
Me
Me
1.22
1.00
1.30
1.36
2.45
1.43
2.19
2.43
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

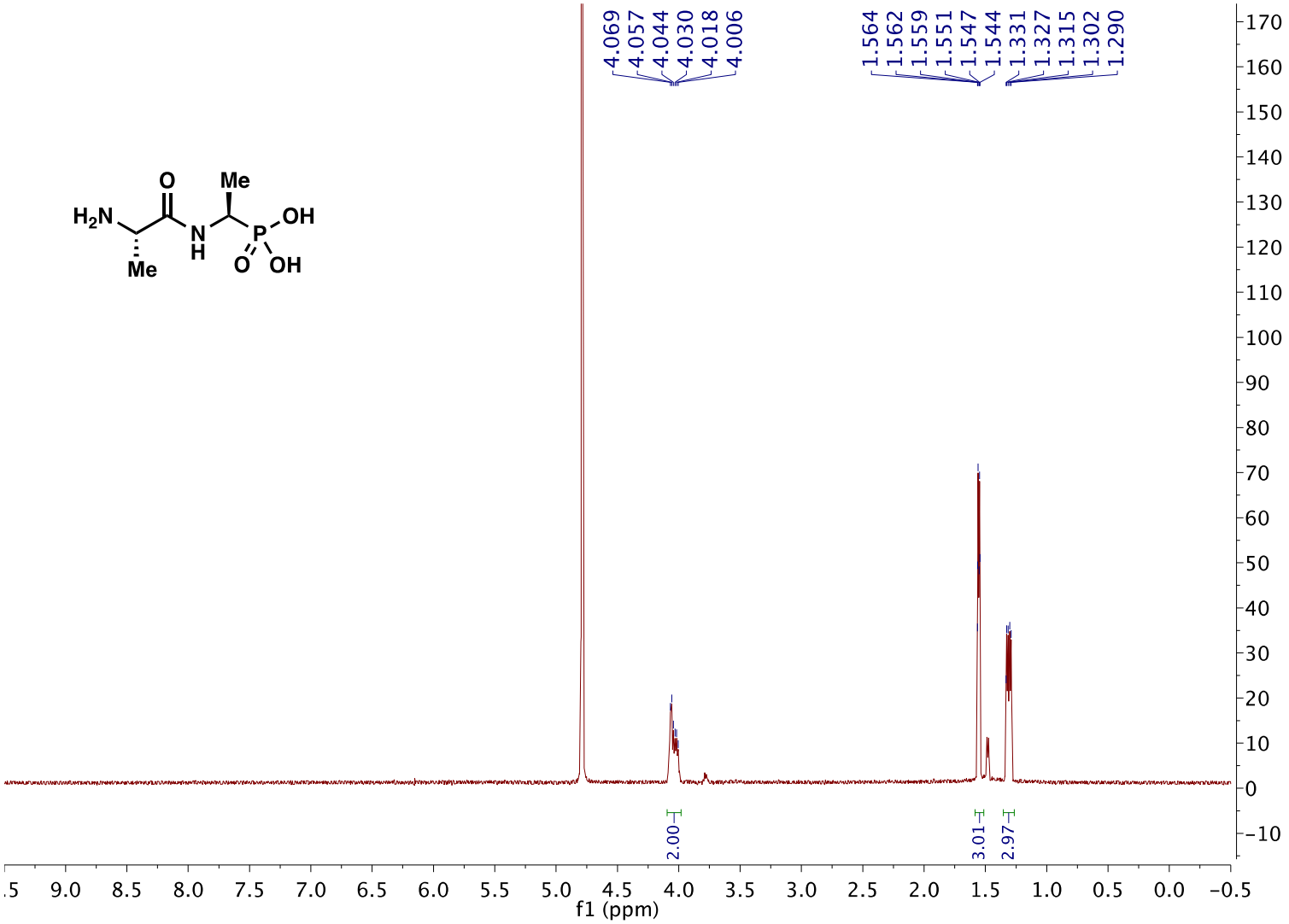

168.330
57.817
43.163
42.140
36.414
24.007
14.215
13.788
10.434
O
Me
H2N
N
H
P
OH
O
OH
Me
Me
f1 (ppm)

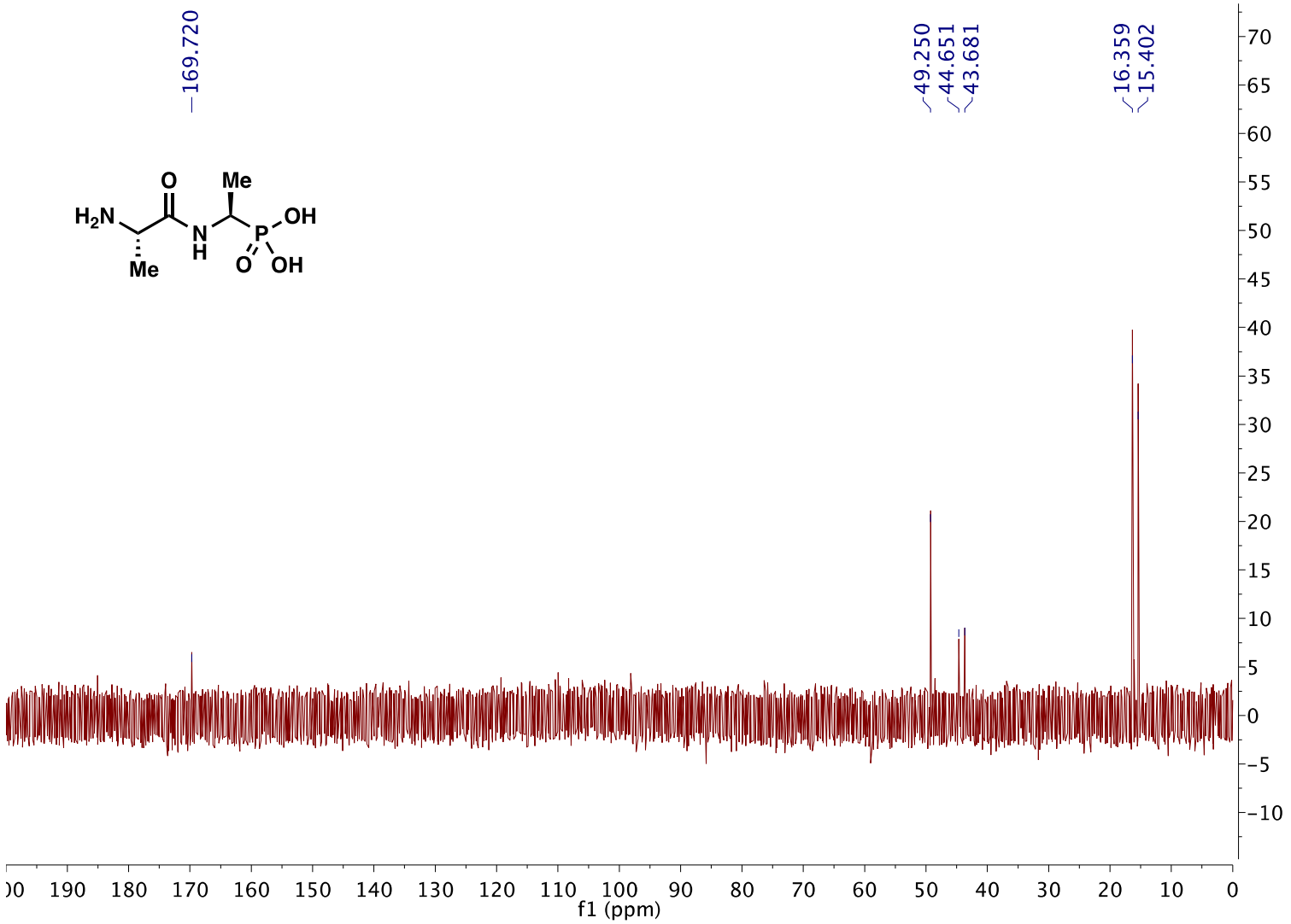

22.338
550
500
450
400
350
300
250
200
150
100
50
0
−50
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
f1 (ppm)


4.245
4.241
4.232
4.228
4.220
4.215
4.207
4.203
3.808
3.804
3.794
2.253
2.250
2.242
2.238
2.228
2.216
1.387
1.384
1.378
1.375
1.372
1.363
1.360
1.357
1.351
1.348
1.345
1.067
1.064
1.062
1.059
1.055
1.052
1.050
1.048
1.046
• TFA
O
Me
H2N
N
H
P
OH
O
OH
Me
Me
1.00
1.06
1.14
3.60
7.31
f1 (ppm)


168.285
168.250
163.208
162.974
162.739
162.510
119.130
117.197
115.265
113.333
58.674
43.636
42.629
29.981
17.449
16.842
14.573
• TFA
O
Me
H2N
N
H
P
OH
O
OH
Me
Me
f1 (ppm)


• TFA
O
Me
H2N
N
H
P
OH
O
OH
Me
Me
20.691
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
−20
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
f1 (ppm)


4.069
4.057
4.044
4.030
4.018
4.006
1.564
1.562
1.559
1.551
1.547
1.544
1.331
1.327
1.315
1.302
1.290
2.00
3.01
2.97
f1 (ppm)


169.720
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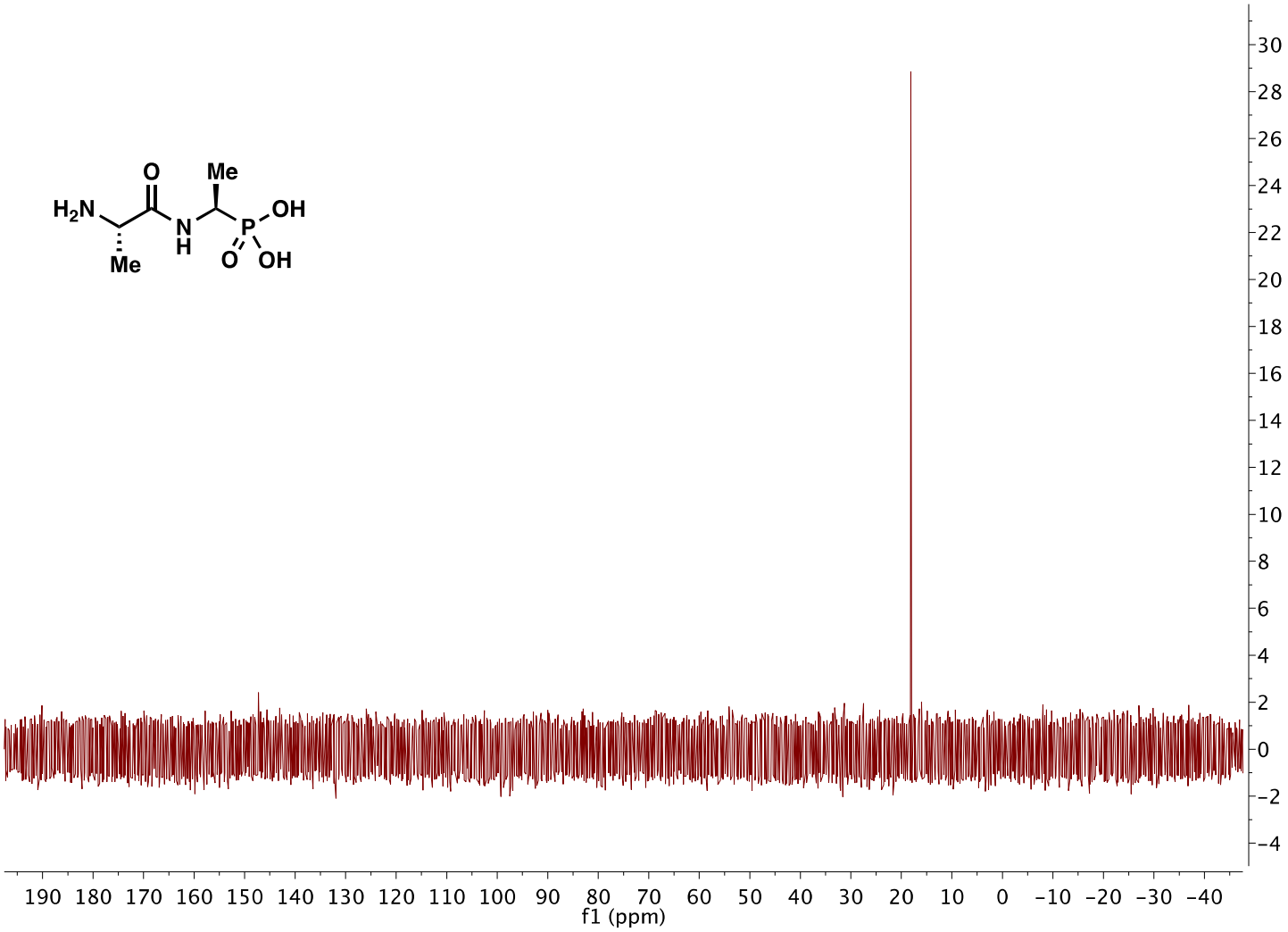
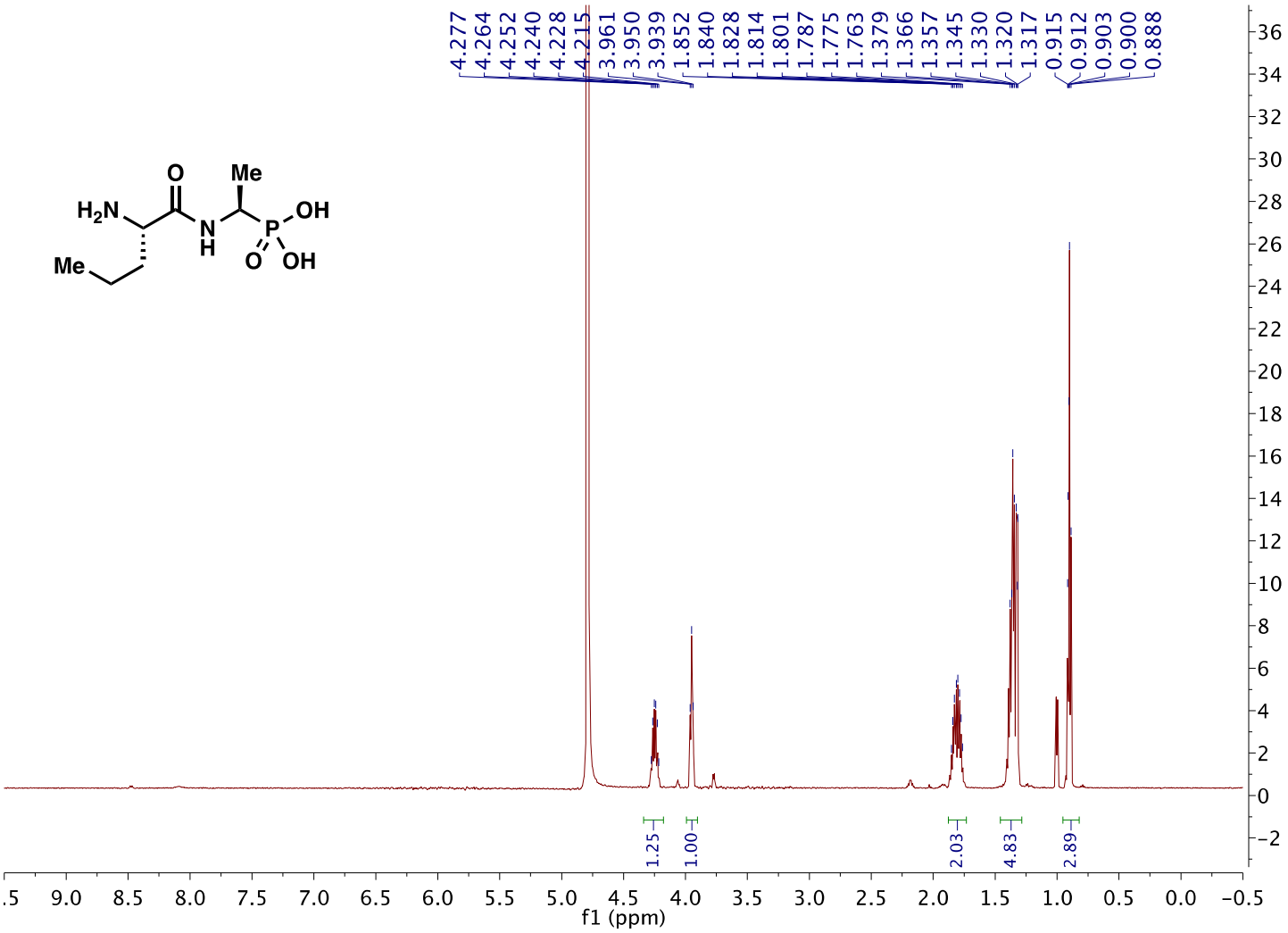
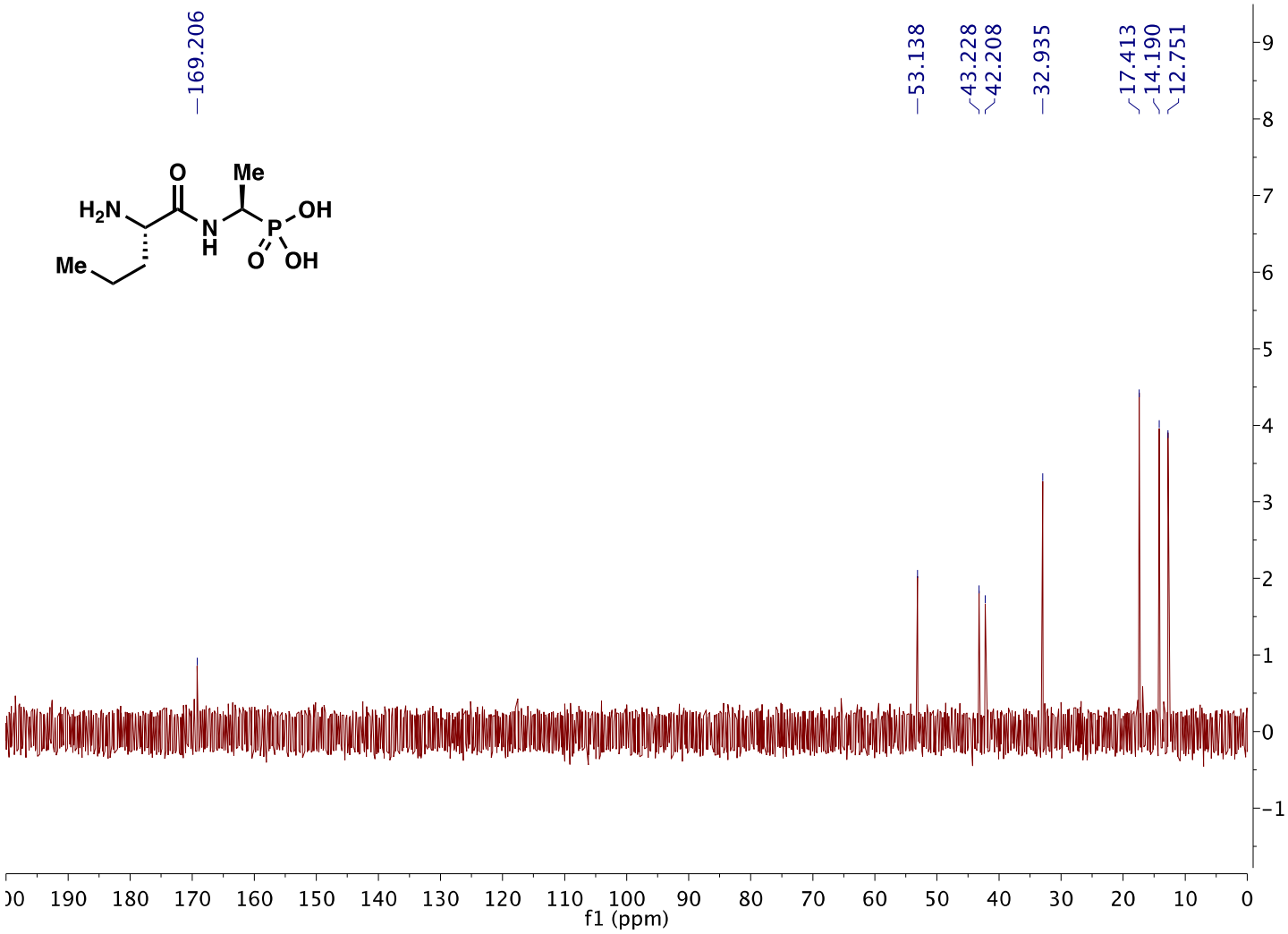
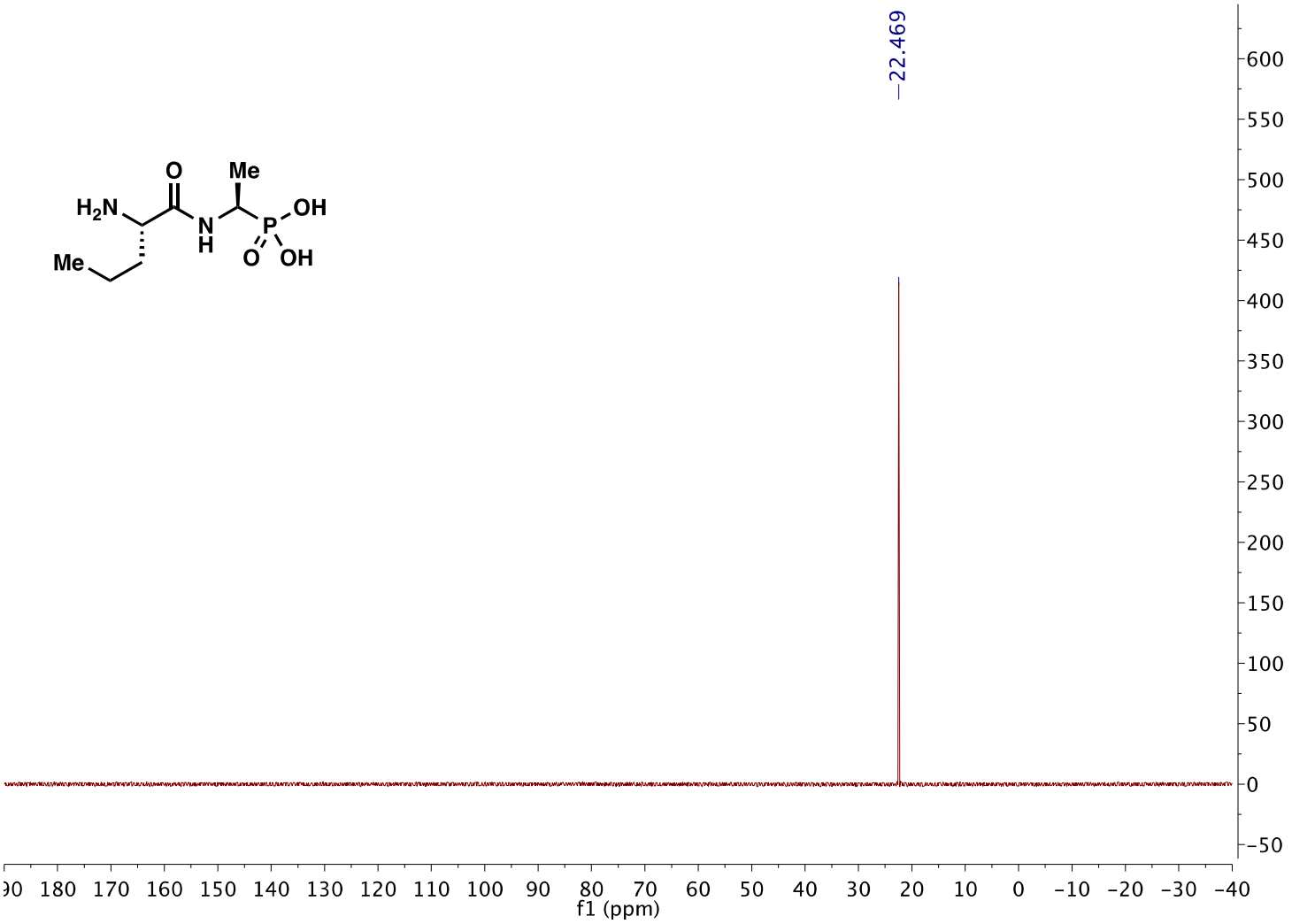
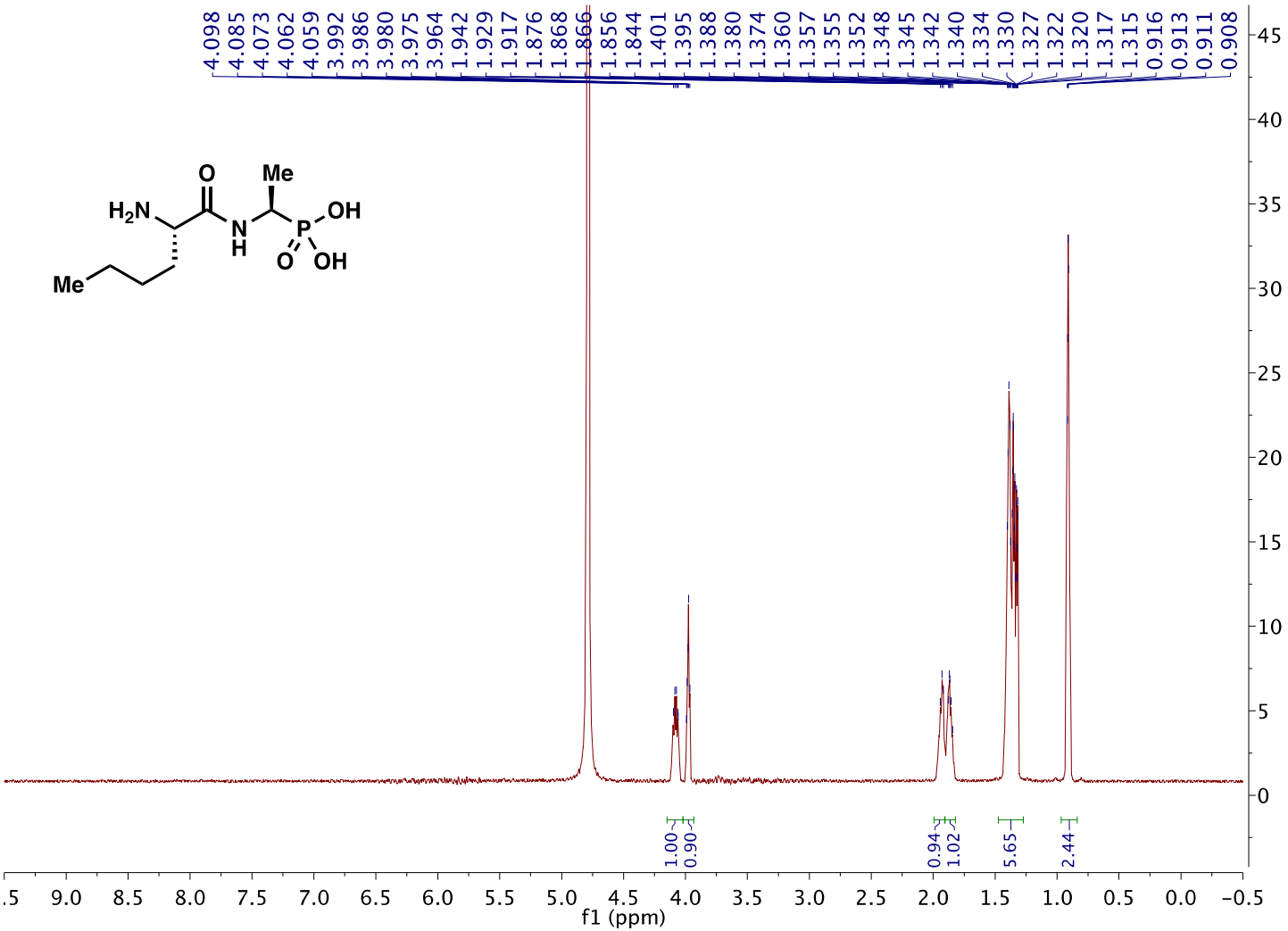
44.651
43.681
16.359
15.402
O
Me
H2N
OH
N
H
P
O
OH
Me
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
−10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

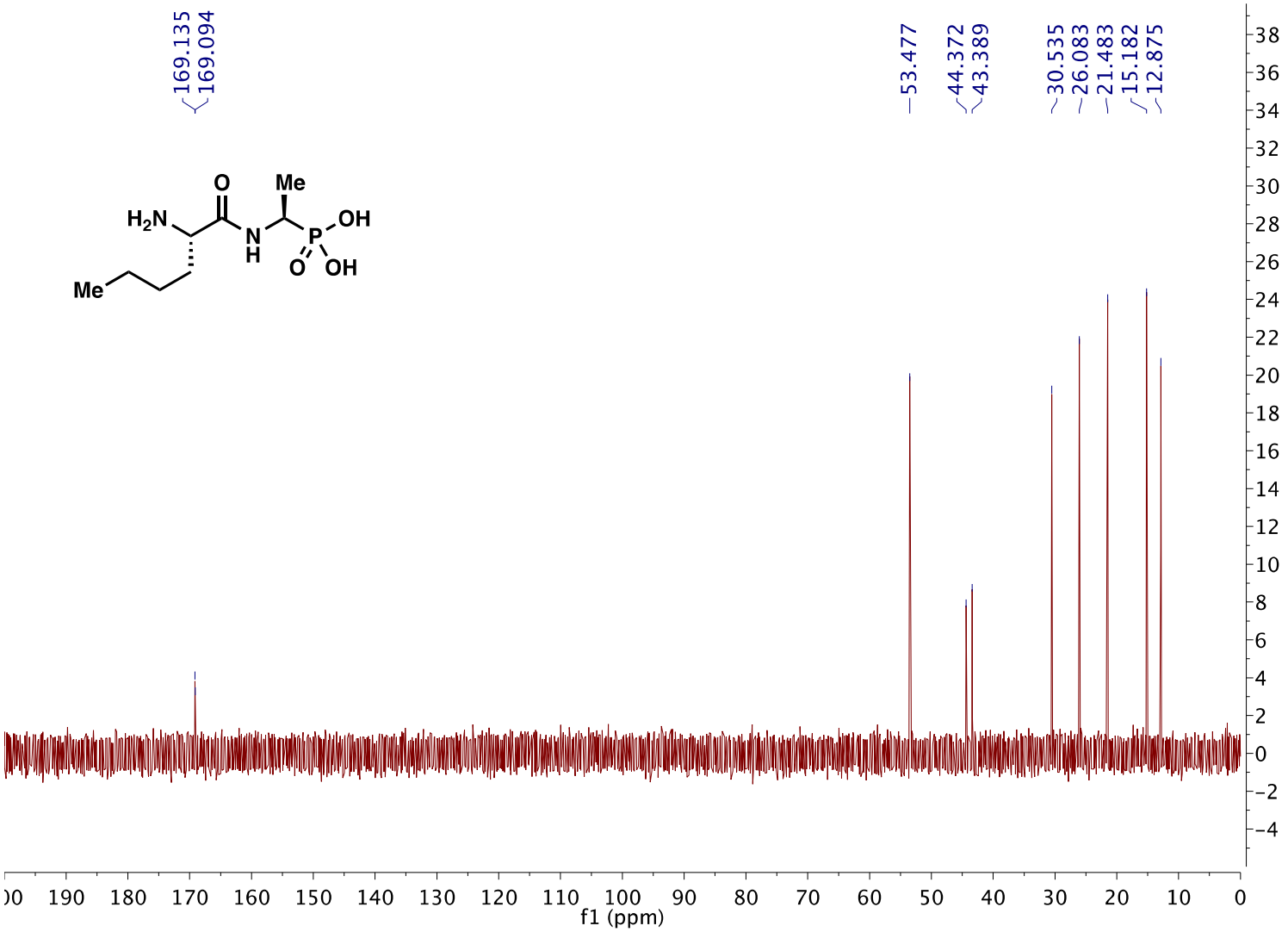

O
Me
H2N
OH
N
H
P
O
OH
Me
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
−2
−4
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
f1 (ppm)

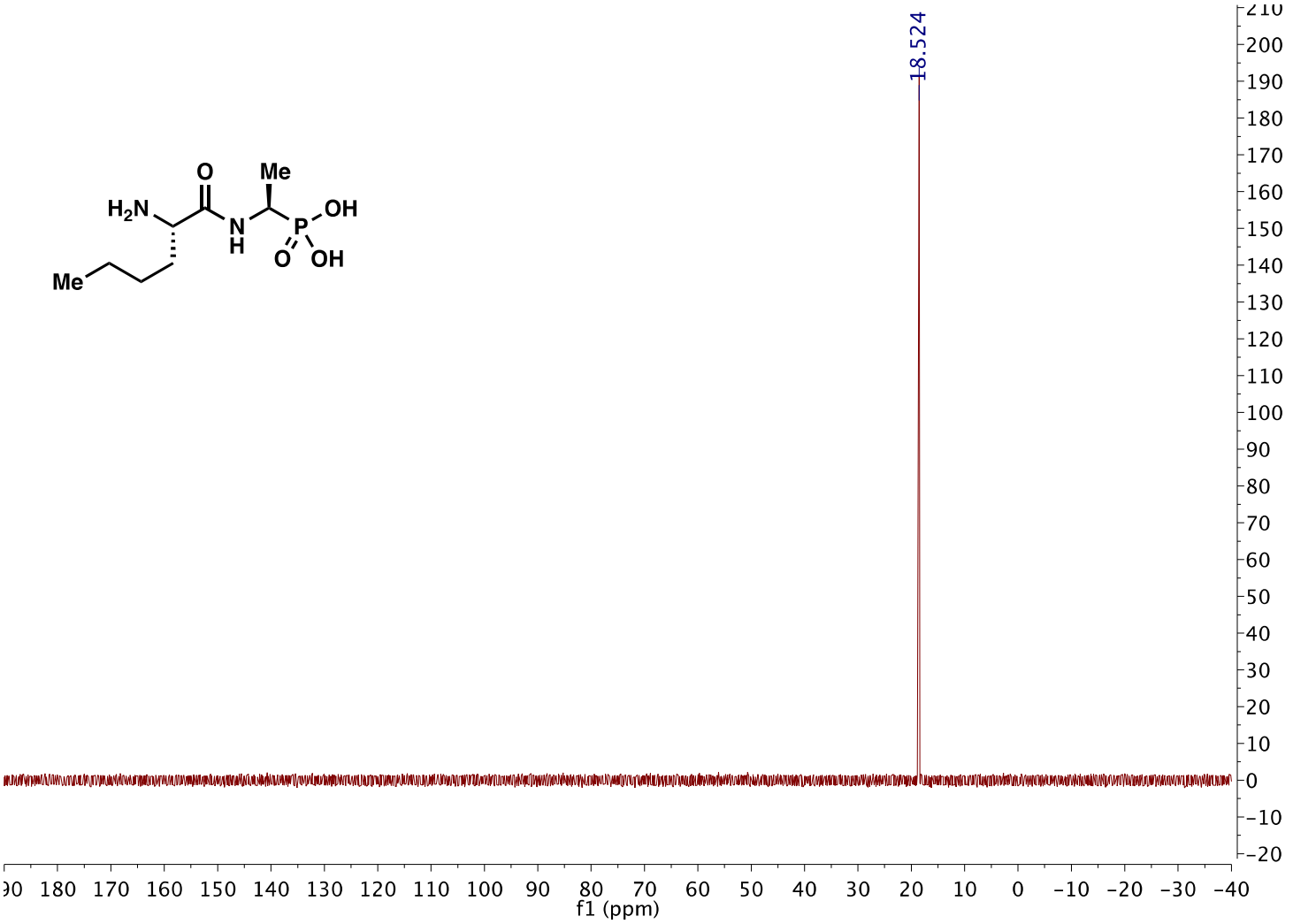

4.277
4.264
4.252
4.240
4.228
4.215
3.961
3.950
3.939
1.852
1.840
1.828
1.814
1.801
1.787
1.775
1.763
1.379
1.366
1.357
1.345
1.330
1.320
1.317
0.915
0.912
0.903
0.900
0.888
O
Me
H2N
N
H
P
OH
O
OH
Me
1.25
1.00
2.03
4.83
2.89
f1 (ppm)

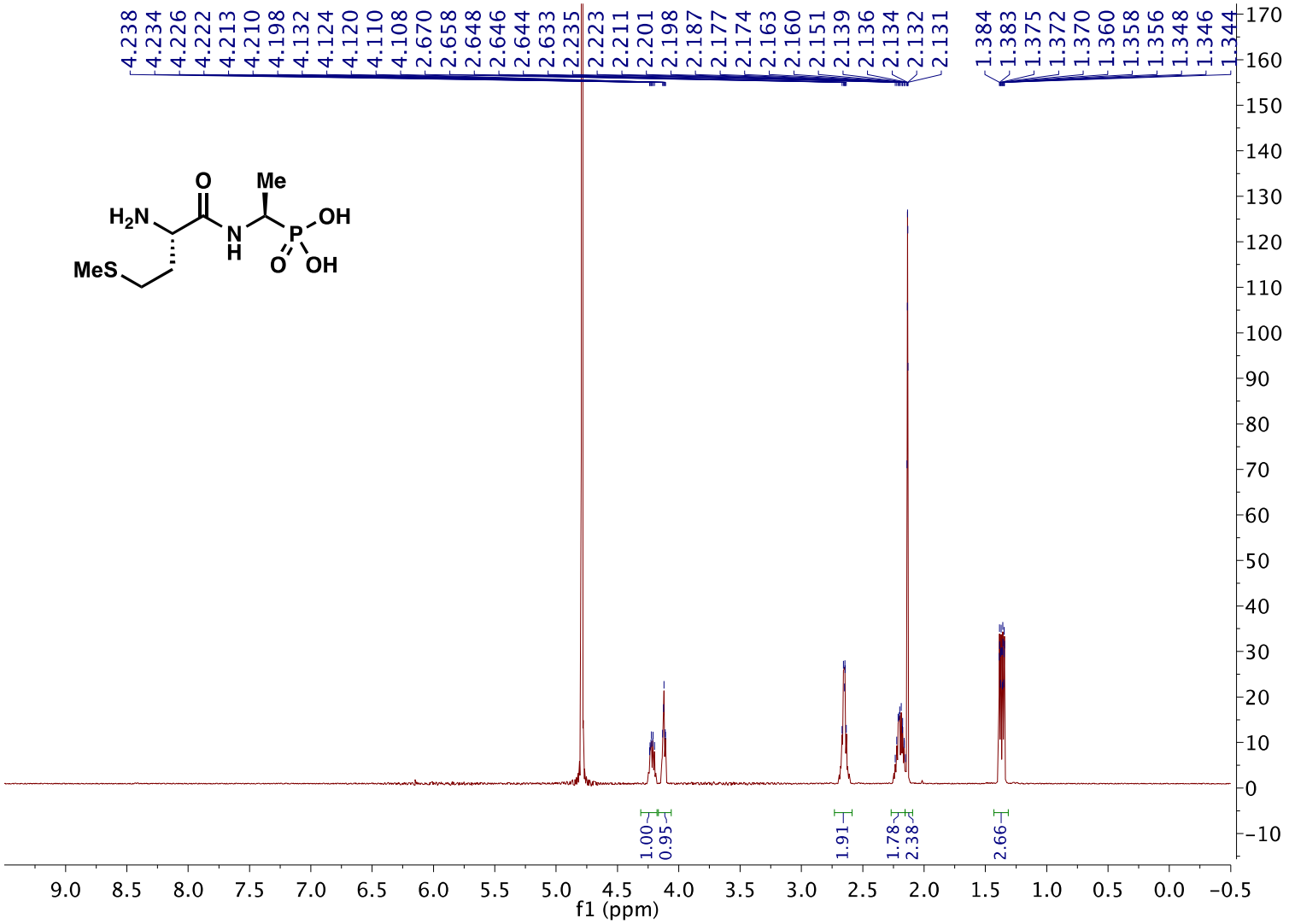

169.206
53.138
43.228
42.208
32.935
17.413
14.190
12.751
O
Me
H2N
N
H
P
OH
O
OH
Me
f1 (ppm)

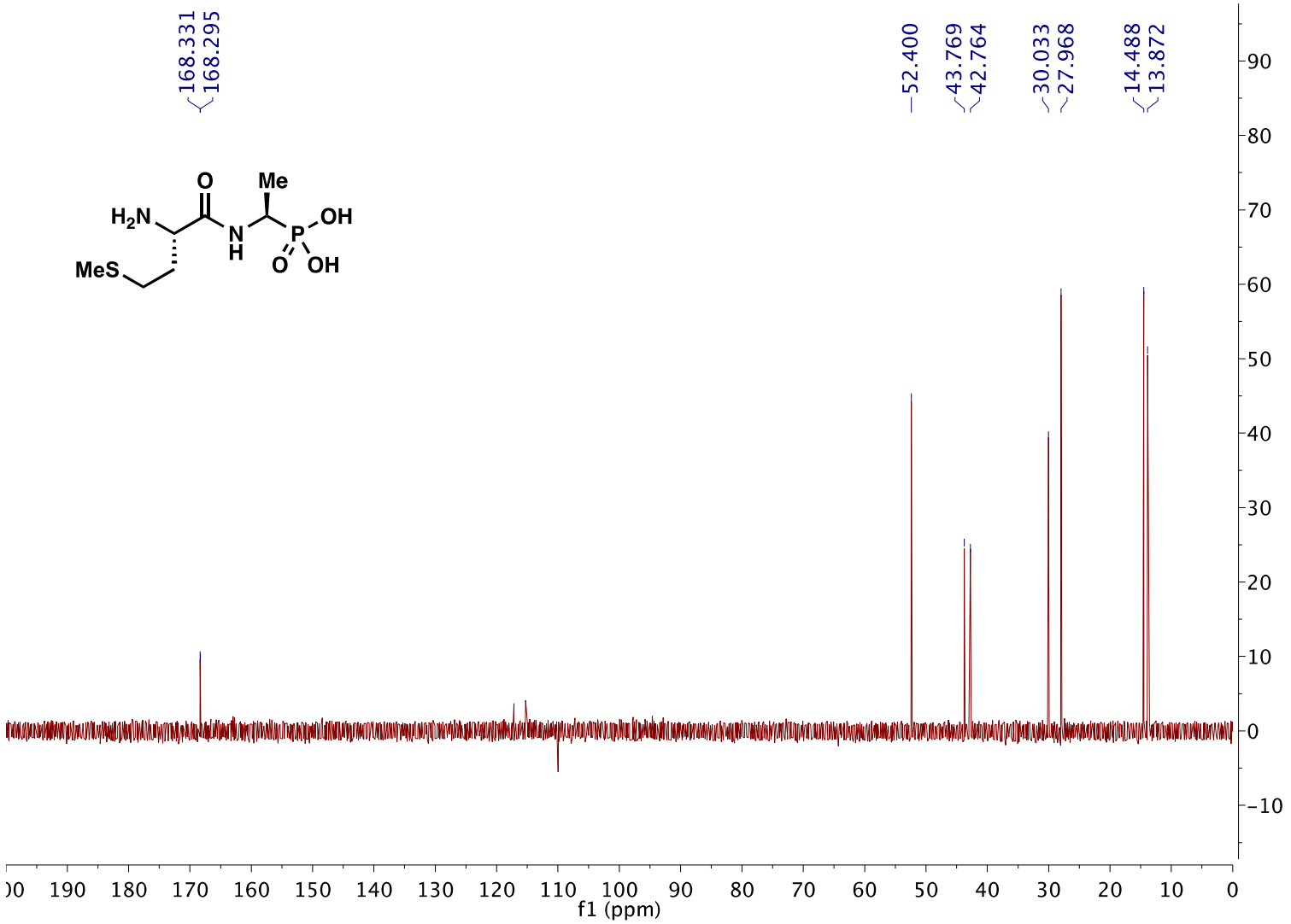

22.469
600
550
500
450
400
350
300
250
200
150
100
50
0
−50
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
f1 (ppm)

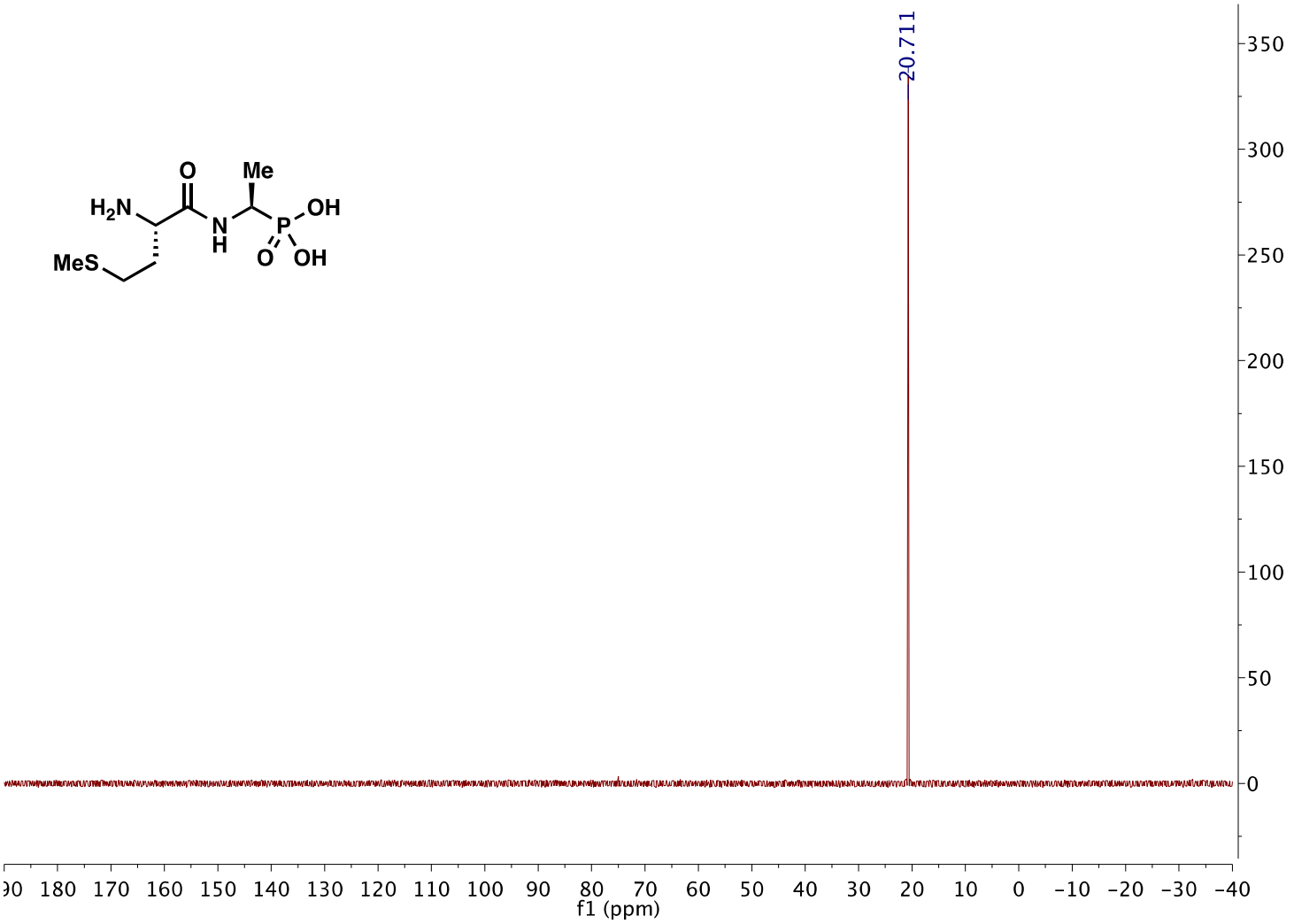

4.098
4.085
4.073
4.062
4.059
3.992
3.986
3.980
3.975
3.964
1.942
1.929
1.917
1.876
1.868
1.866
1.856
1.844
1.401
1.395
1.388
1.380
1.374
1.360
1.357
1.355
1.352
1.348
1.345
1.342
1.340
1.334
1.330
1.327
1.322
1.320
1.317
1.315
0.916
0.913
0.911
0.908
O
Me
H2N
N
H
P
OH
O
OH
Me
1.00
0.90
0.94
1.02
5.65
2.44
f1 (ppm)


169.135
169.094
53.477
44.372
43.389
30.535
26.083
21.483
15.182
12.875
O
Me
H2N
N
H
P
OH
O
OH
Me
f1 (ppm)


18.524
O
Me
H2N
N
H
P
OH
O
OH
Me
f1 (ppm)


4.238
4.234
4.226
4.222
4.213
4.210
4.198
4.132
4.124
4.120
4.110
4.108
2.670
2.658
2.648
2.646
2.644
2.633
2.235
2.223
2.211
2.201
2.198
2.187
2.177
2.174
2.163
2.160
2.151
2.139
2.136
2.134
2.132
2.131
1.384
1.383
1.375
1.372
1.370
1.360
1.358
1.356
1.348
1.346
1.344
O
Me
H2N
OH
N
H
P
O
OH
MeS
1.00
0.95
1.91
1.78
2.38
2.66
f1 (ppm)


168.331
168.295
52.400
43.769
42.764
30.033
27.968
14.488
13.872
O
Me
H2N
OH
N
H
P
O
OH
MeS
90
80
70
60
50
40
30
20
10
0
−10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


20.711
O
Me
H2N
OH
N
H
P
O
OH
MeS
350
300
250
200
150
100
50
0
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)