Synthesis of aminobisphosphinates through a cascade reaction between hypophosphorous acid and bis(trimethylsilyl)imidates mediated by ZnI_2

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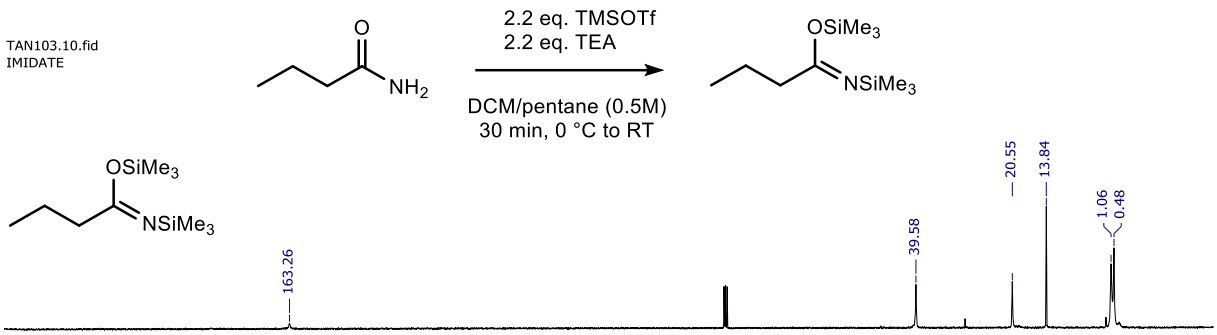
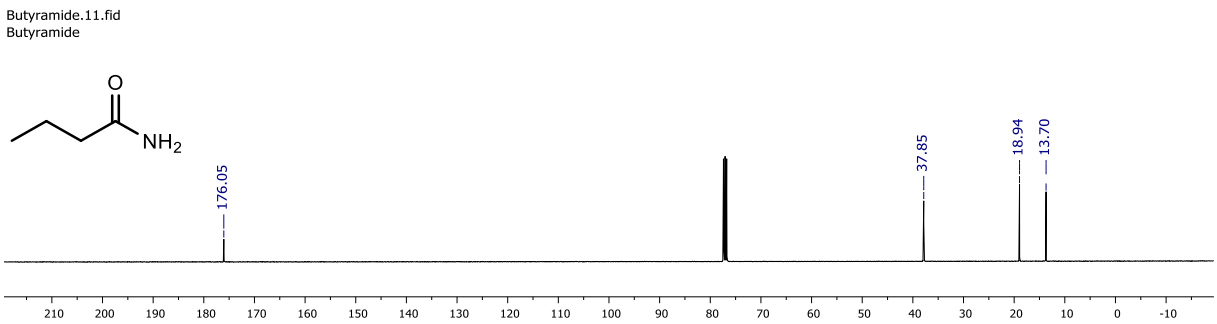
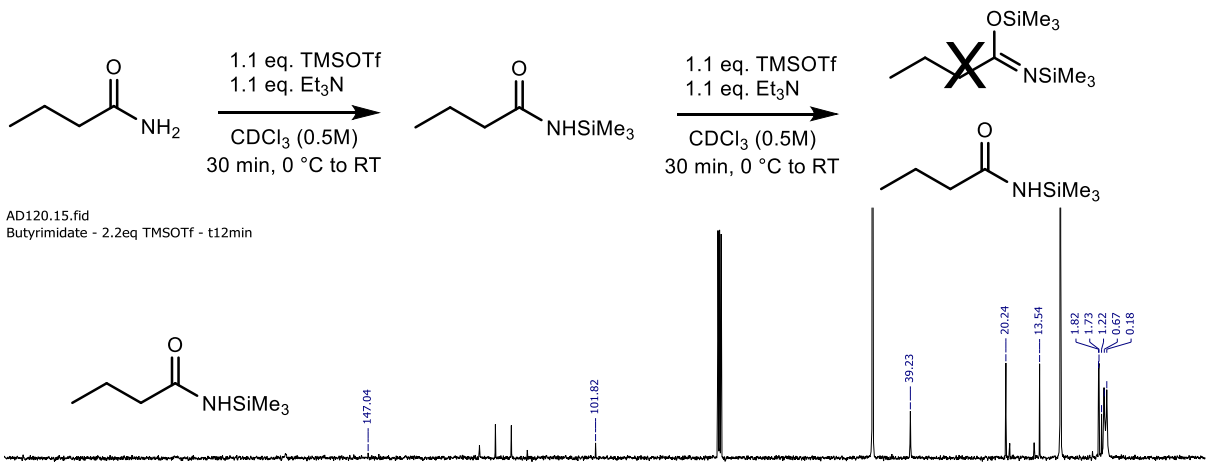
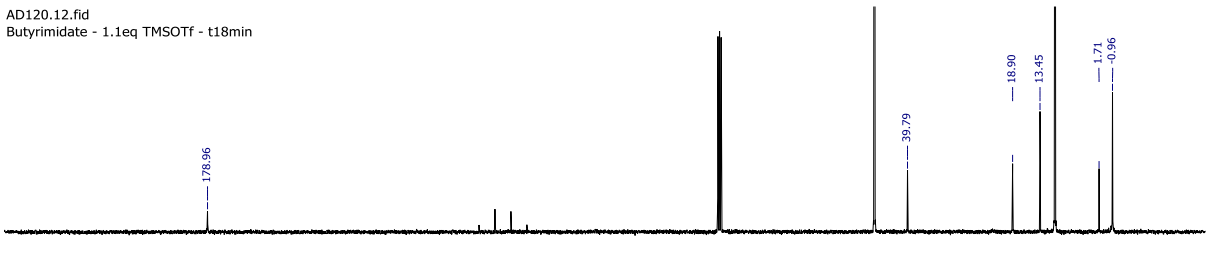
.Supporting Information

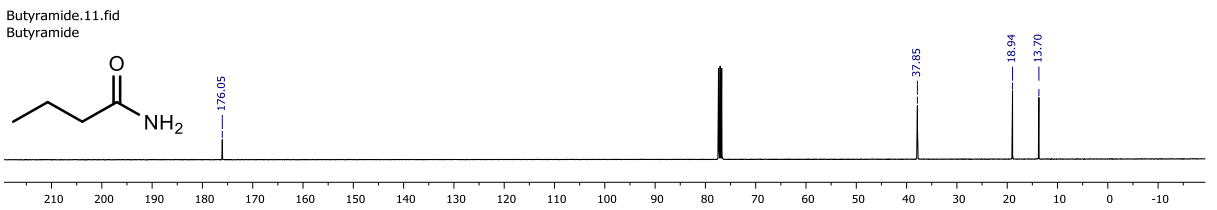
1) NMR monitoring of imidate formation 2b



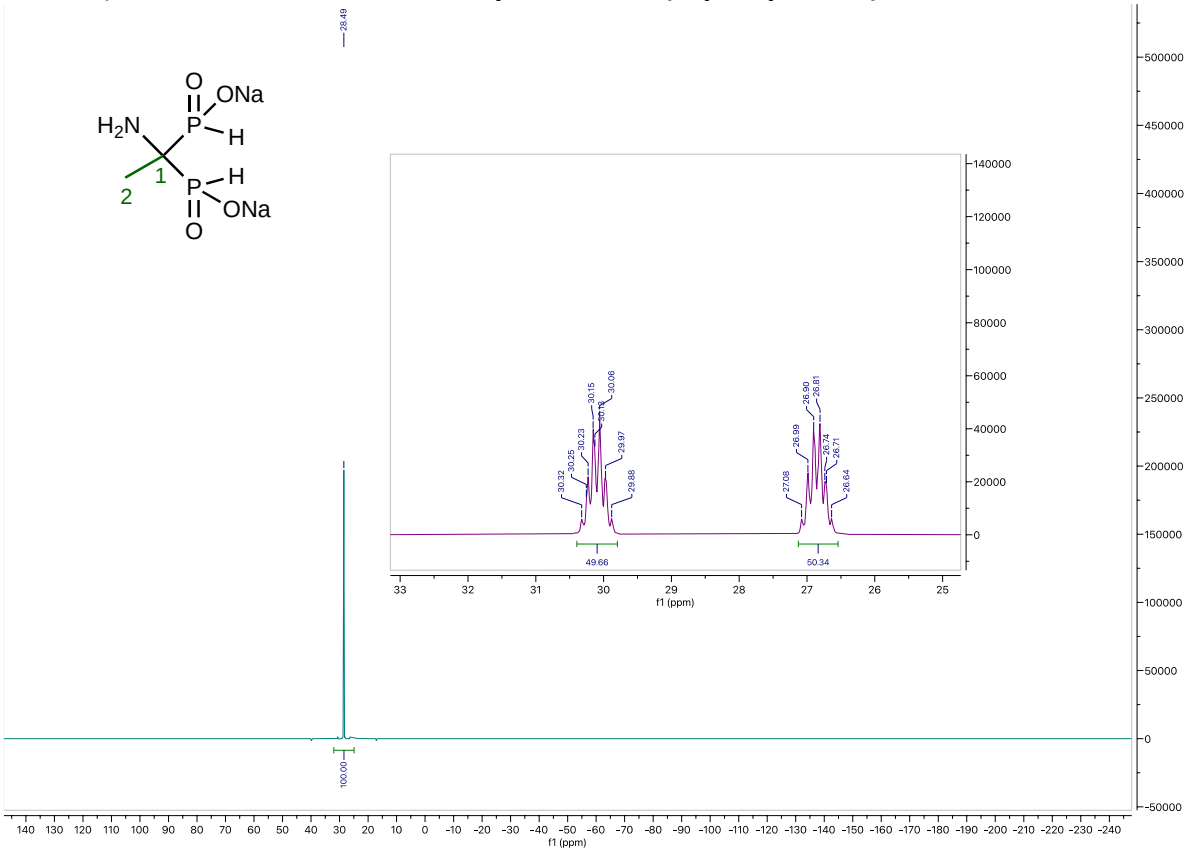


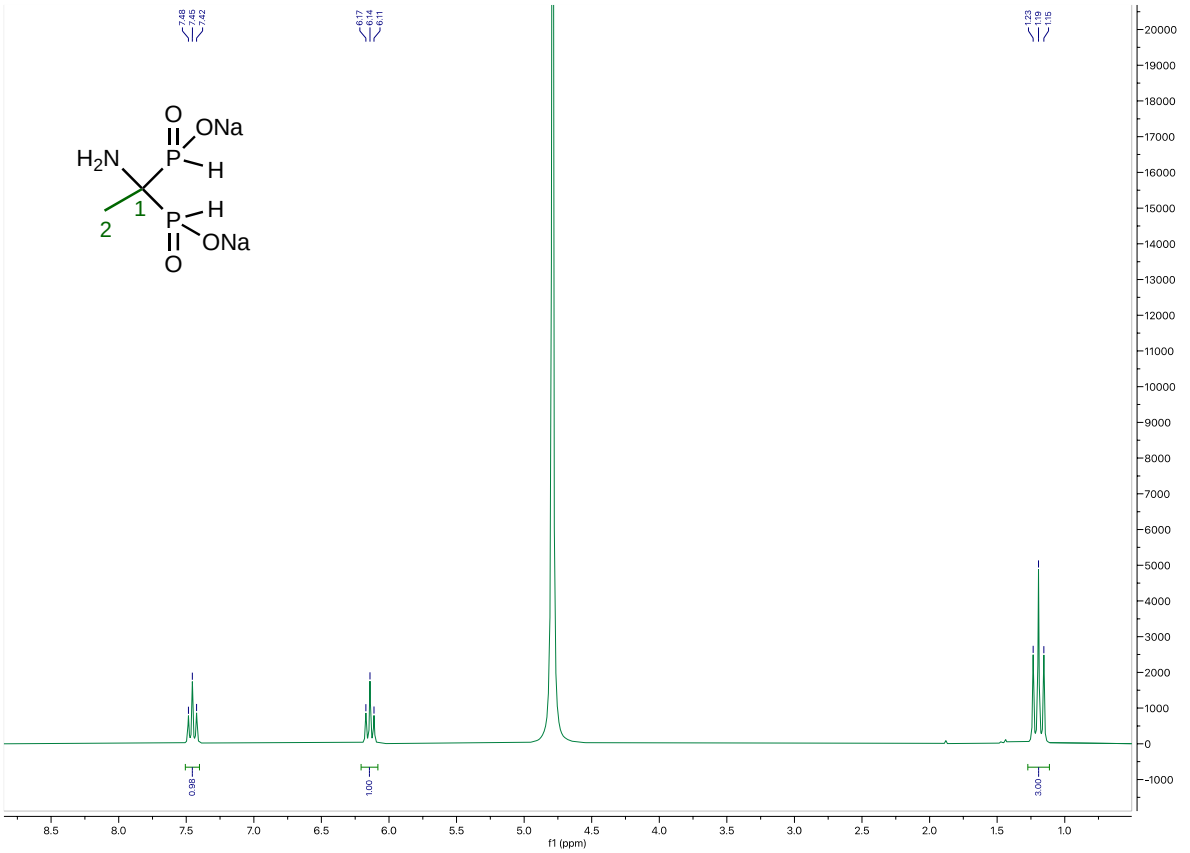






2) **NMR data of 1-aminomethylene-1,1-bis(H-phosphinate) disodium salts 3a-l**





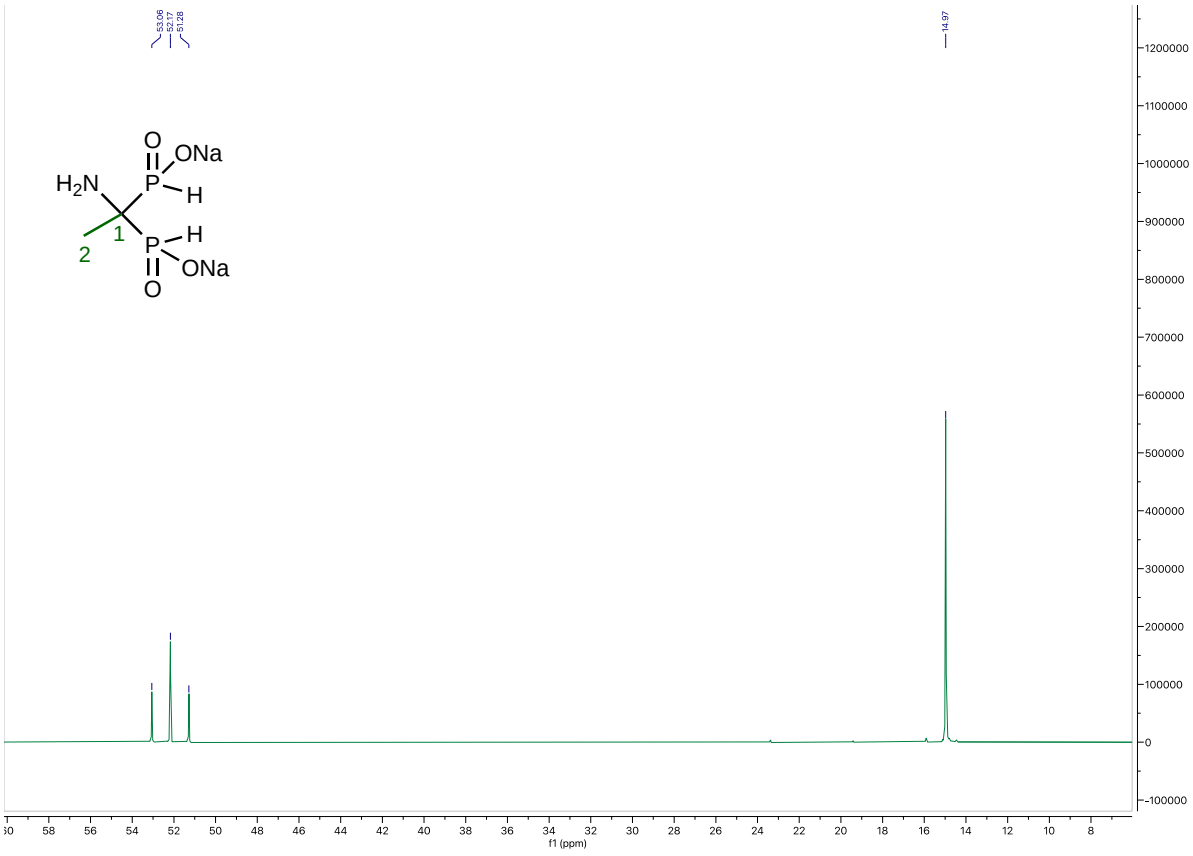
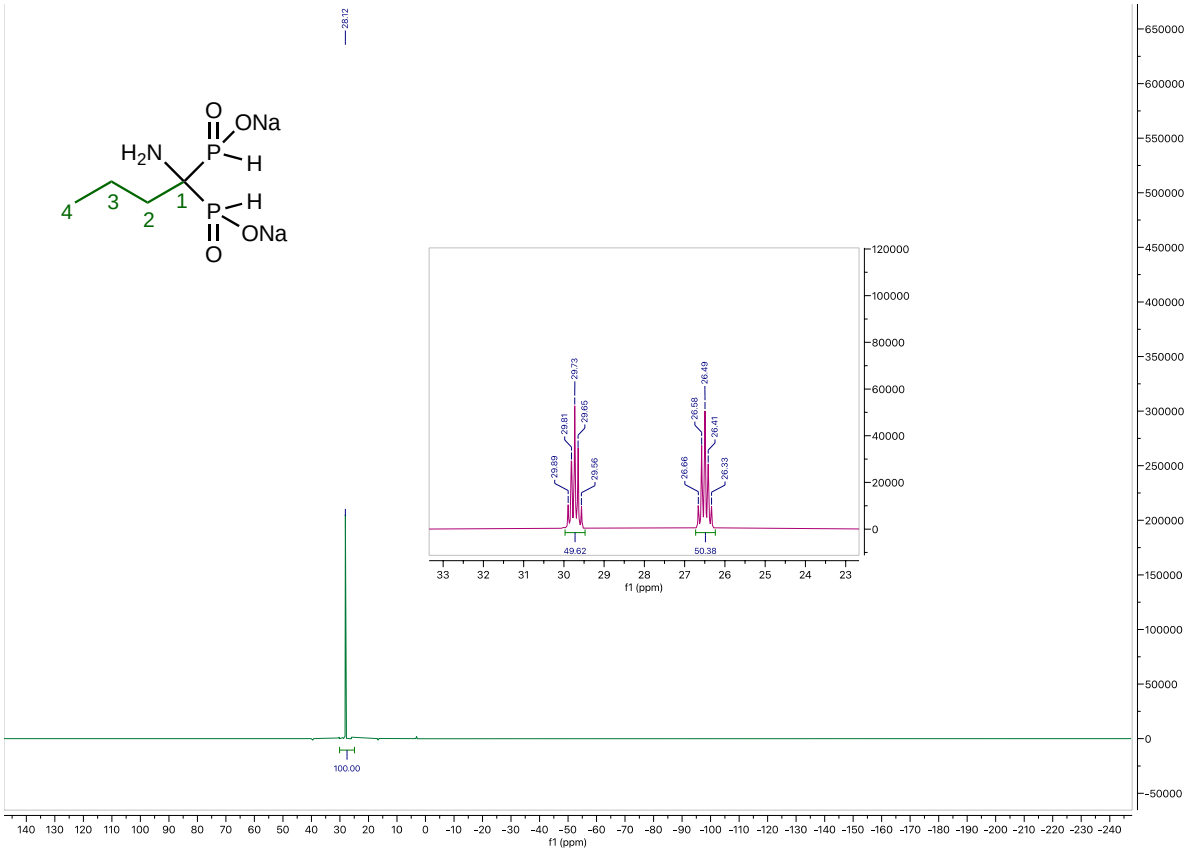


Figure 1. ^{31}P NMR spectrum (162 MHz, D_2O), ^{1}H NMR spectrum (400MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-aminoethane-1,1-bis(H-phosphinate) disodium salts **3a**.



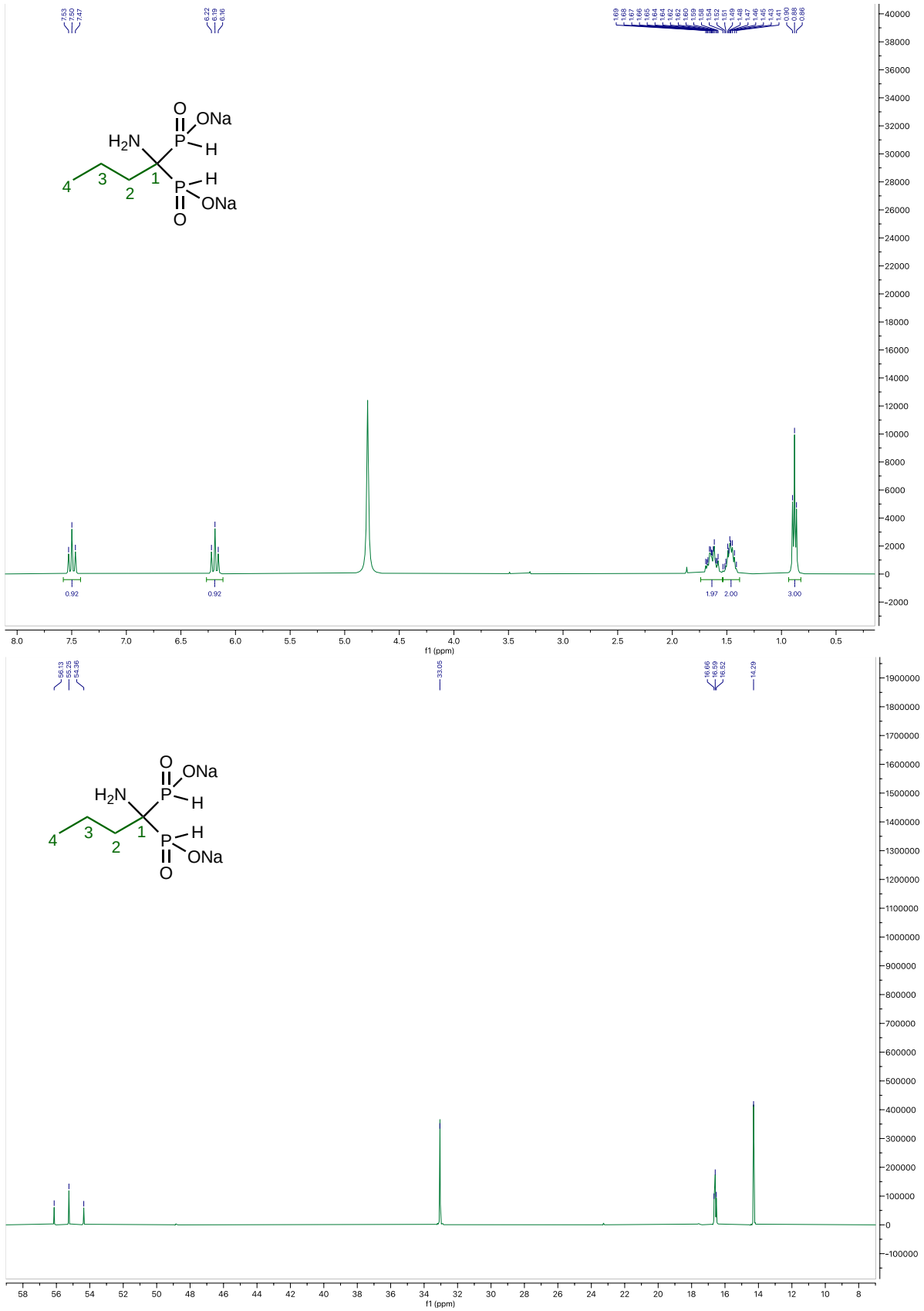
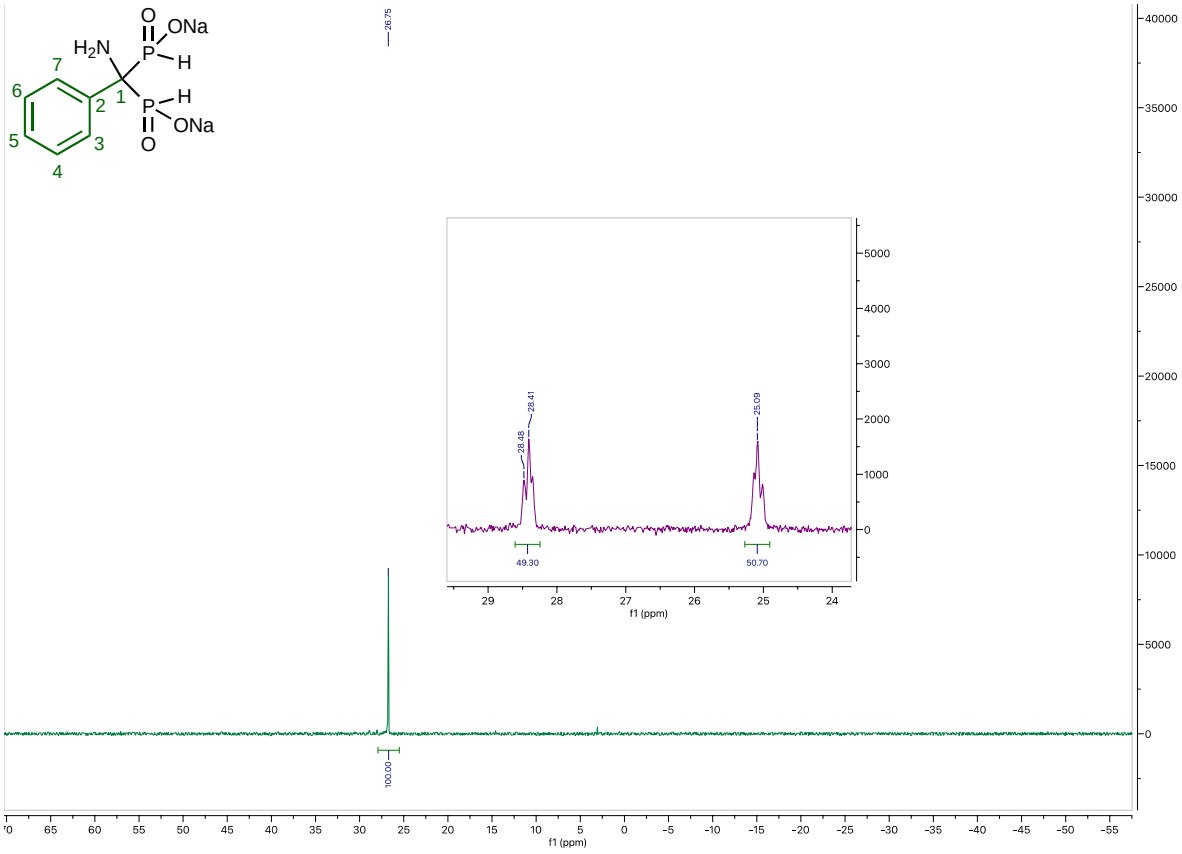
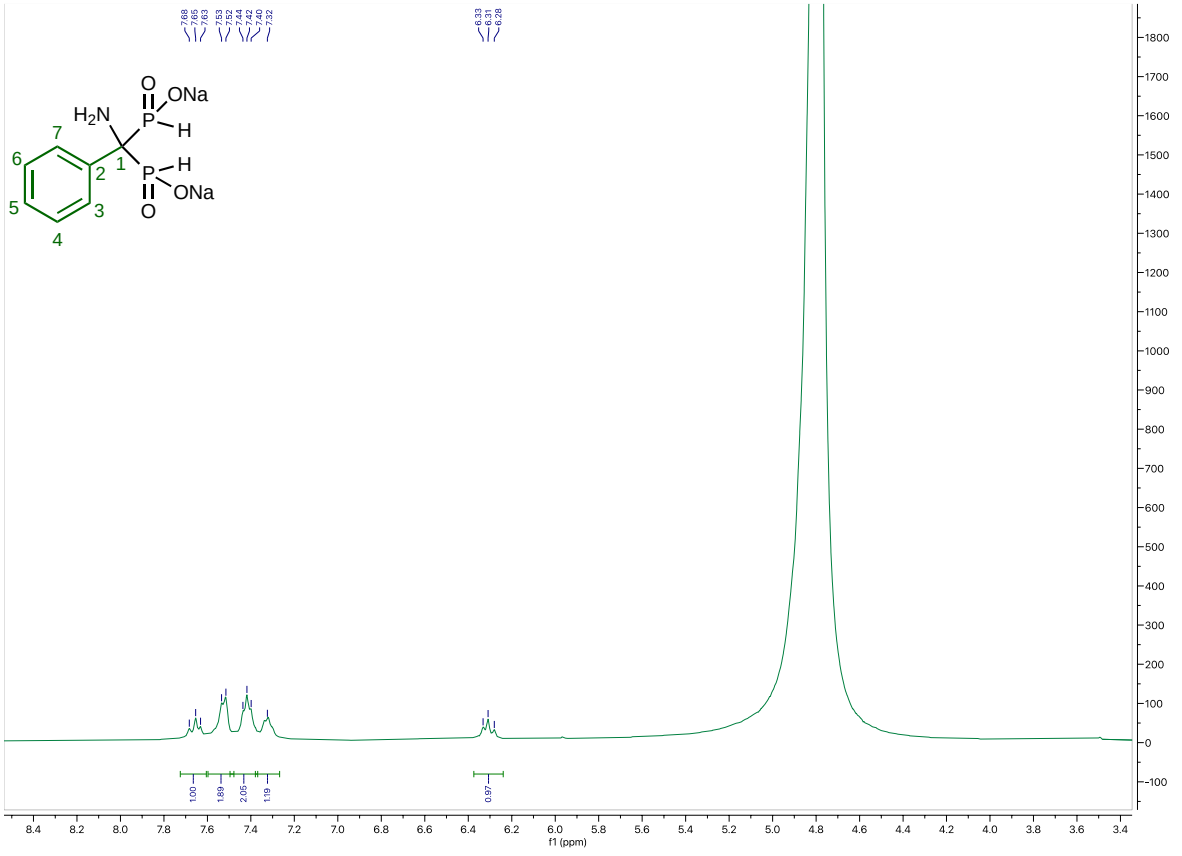


Figure 2. ^{31}P NMR spectrum (162 MHz, D_2O), ^{1}H NMR spectrum (400 MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-amino-1-propylmethane-1,1-bis(H-phosphinate) disodium salts **3b**.


O
ONa
H2N
P
H
7
6
1
2
P
H
ONa
O
5
3
4
26.75
28.41
28.48
25.09
49.30
50.70
100.00
f1 (ppm)


7.68
7.66
7.65
7.63
7.53
7.52
7.44
7.42
7.40
7.32
6.33
6.31
6.28
O
ONa
H2N
P
H
7
6
1
2
P
H
ONa
O
5
3
4
1.00
1.89
2.05
1.19
0.97
f1 (ppm)

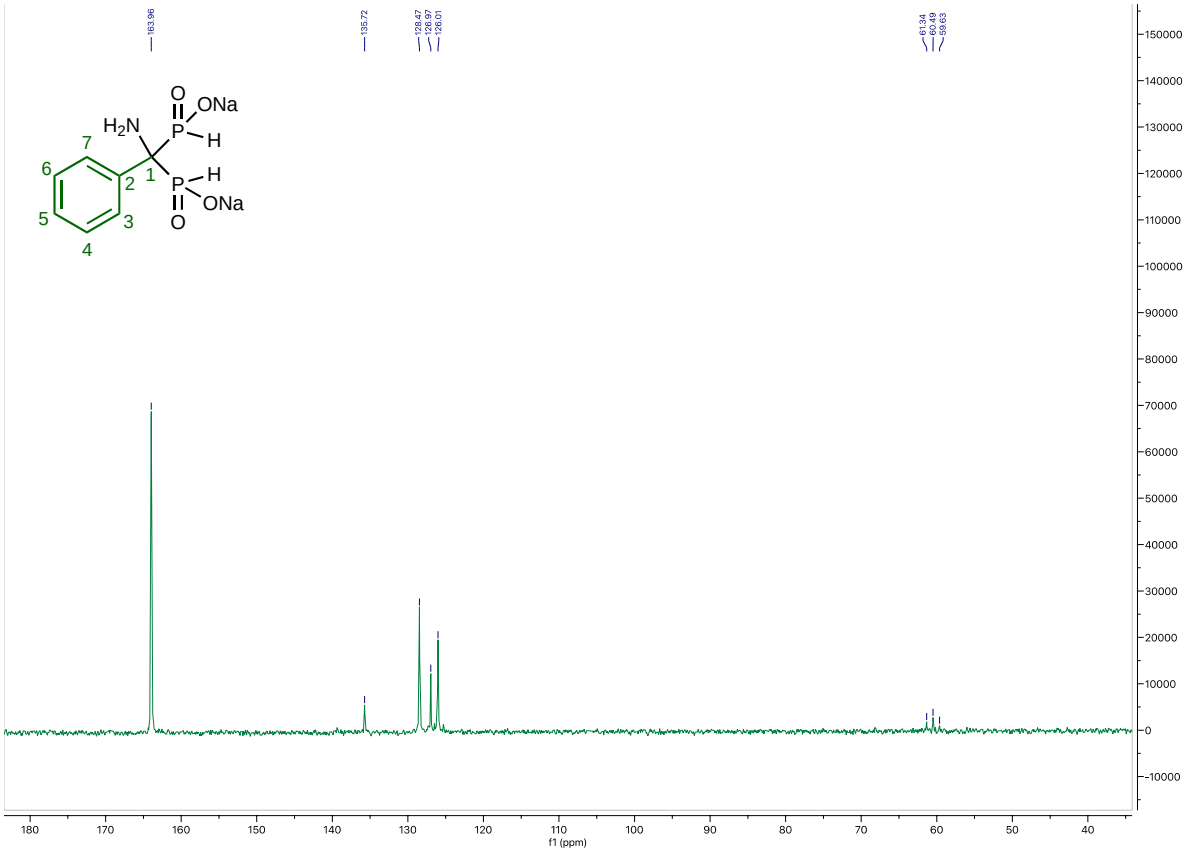
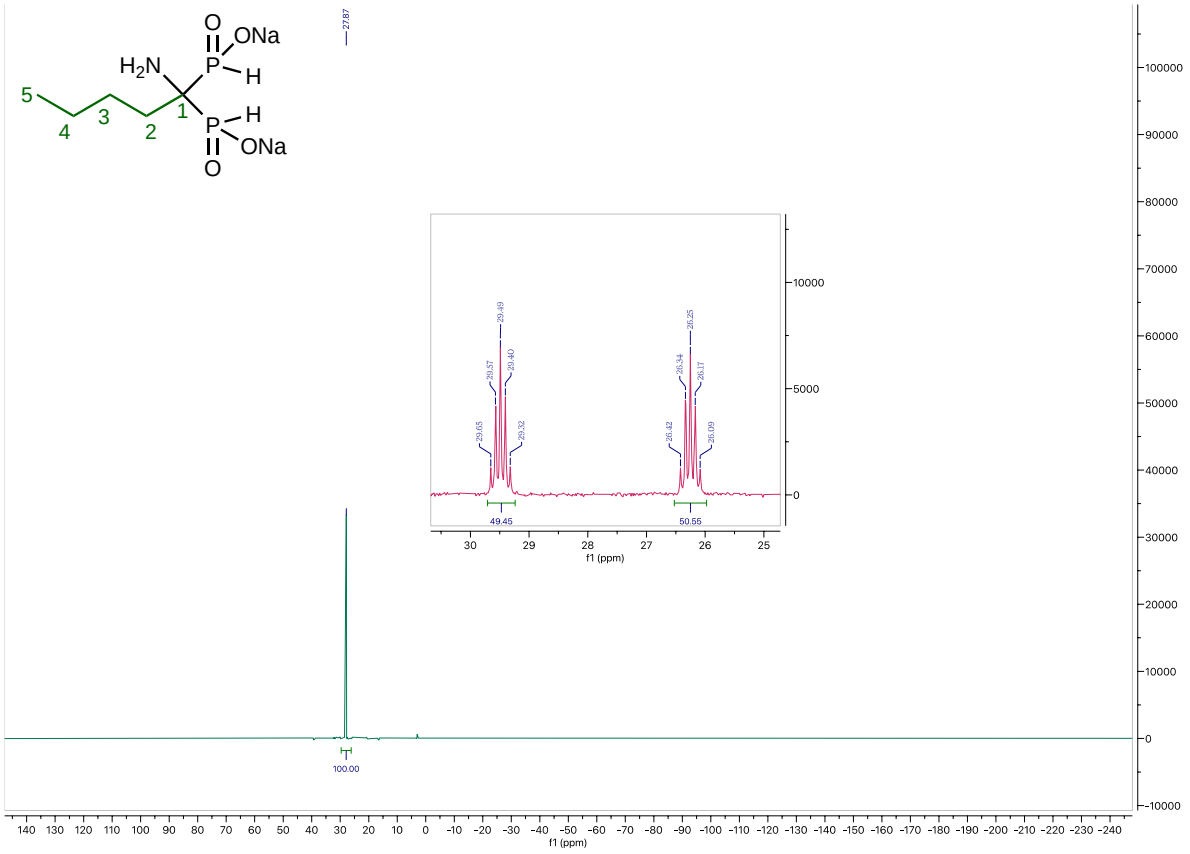
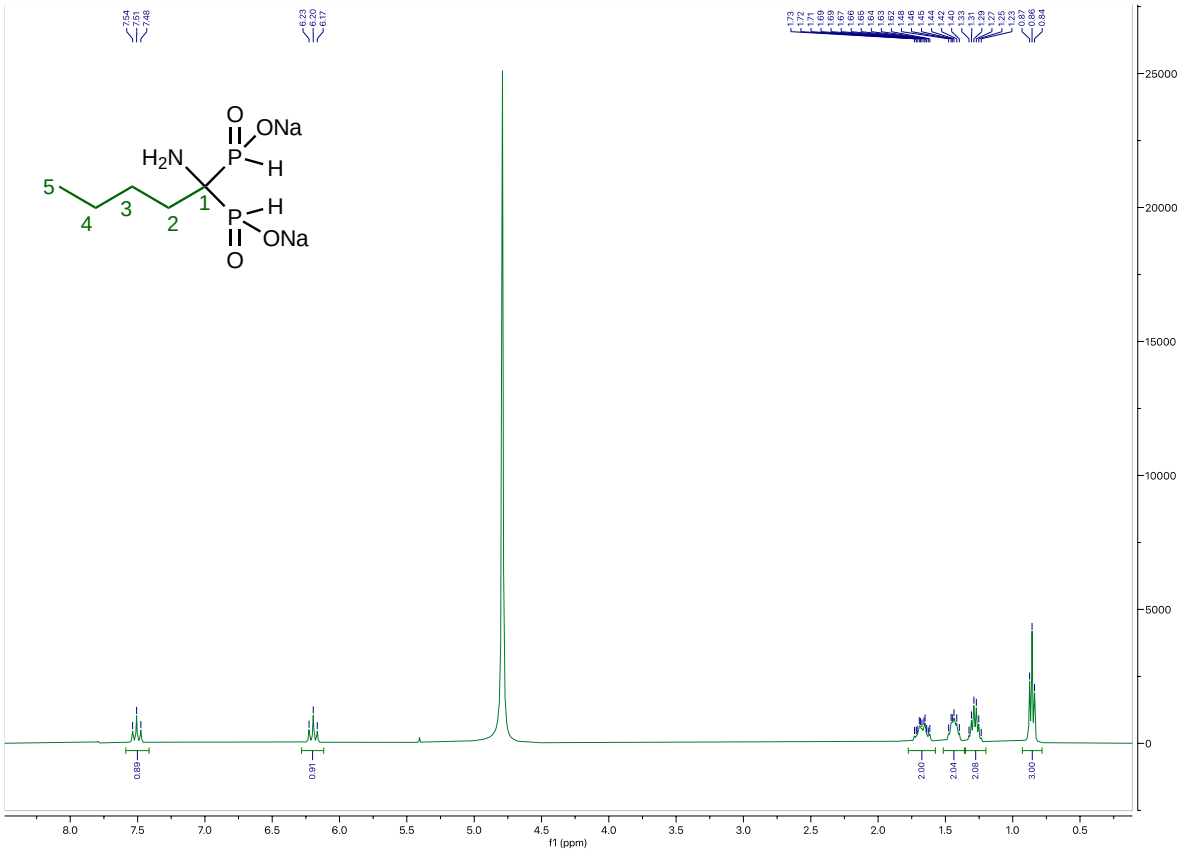


Figure 3. ^{31}P NMR spectrum (162 MHz, D_2O), ^{1}H NMR spectrum (400 MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-amino-1-phenylmethane-1,1-bis(H-phosphinate) disodium salts **3c**.


O
ONa
H2N
P
H
5
4
3
2
1
P
H
ONa
O
27.87
29.65
29.57
29.49
29.40
29.32
26.42
26.34
26.25
26.17
26.09
49.45
50.55
f1 (ppm)
100.00
f1 (ppm)


7.54
7.53
7.48
6.23
6.20
6.17
O
ONa
H2N
P
H
5
4
3
2
1
P
H
ONa
O
0.89
0.91
2.00
2.04
2.08
3.00
f1 (ppm)

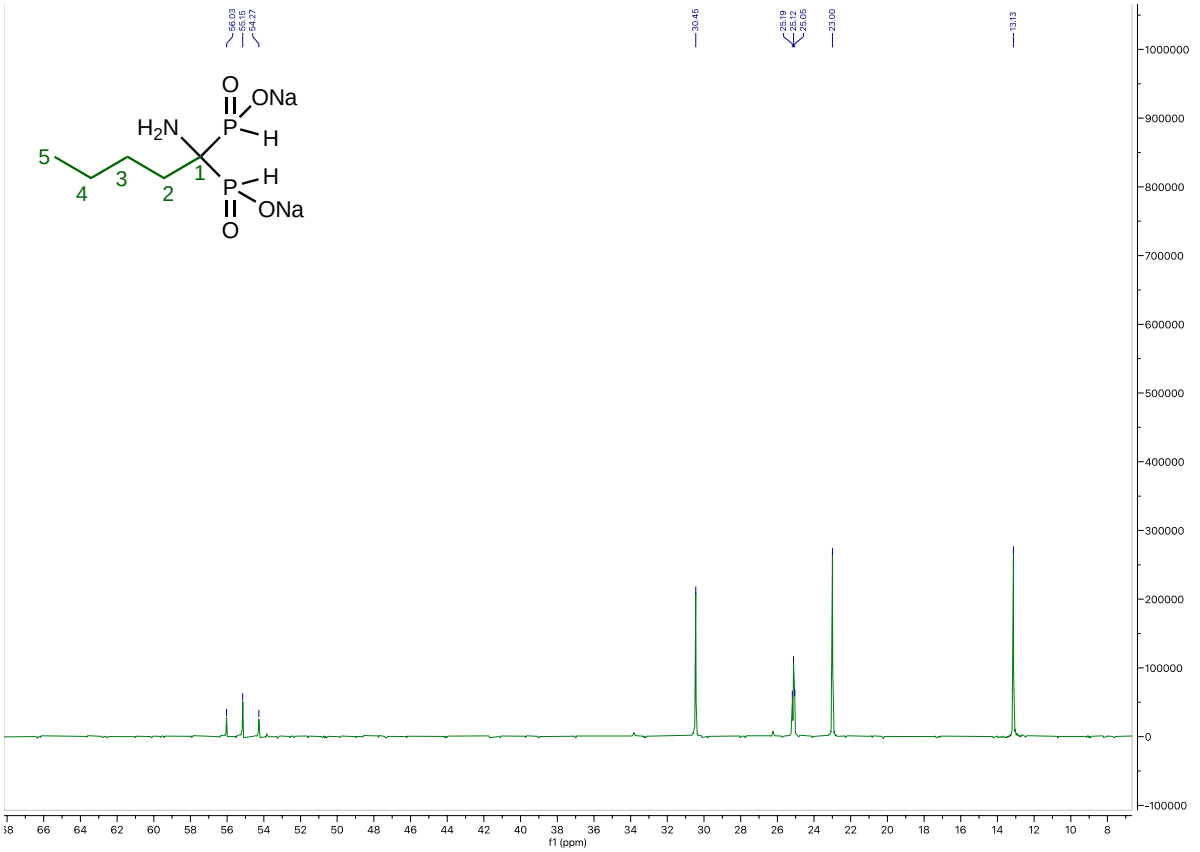
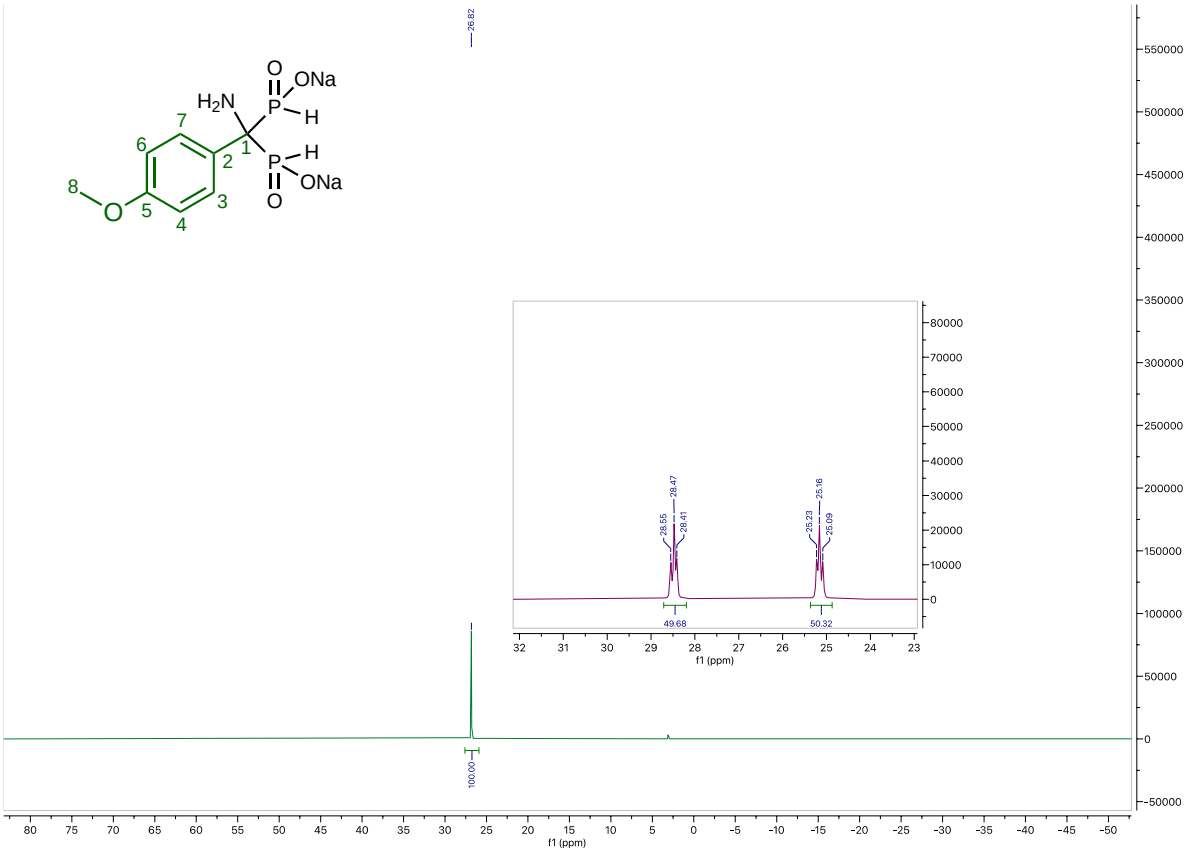


Figure 4. ^{31}P NMR spectrum (162 MHz, D_2O), ^{1}H NMR spectrum (400 MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-amino-1-butylmethane-1,1-bis(H-phosphinate) disodium salts **3d**.



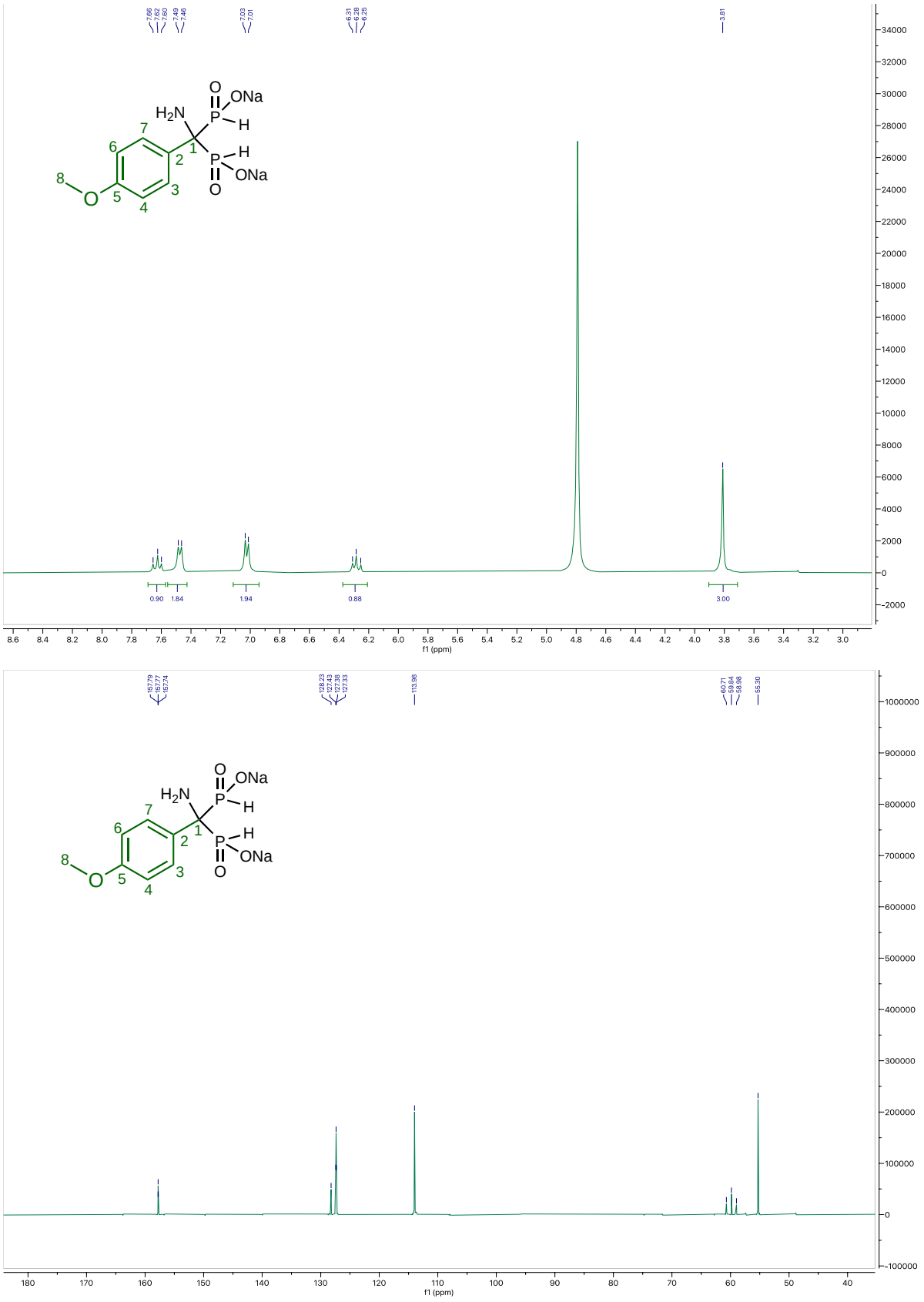
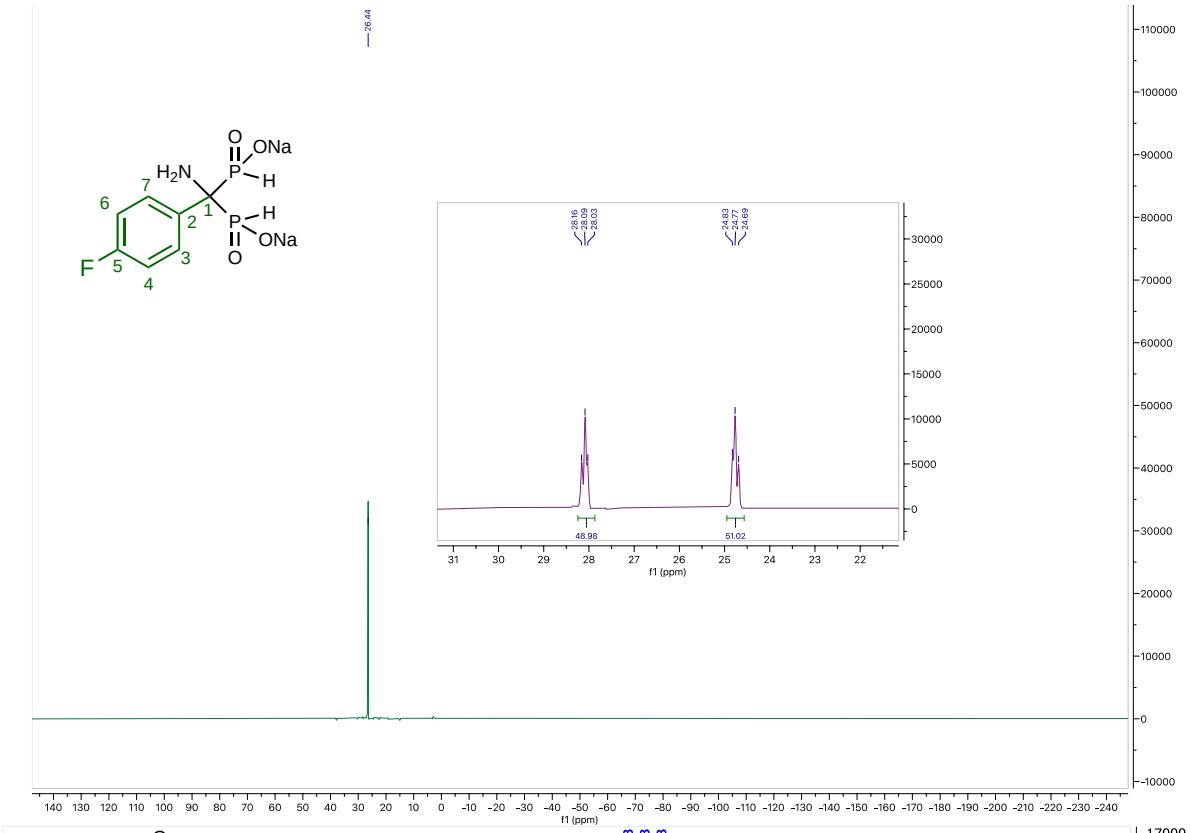
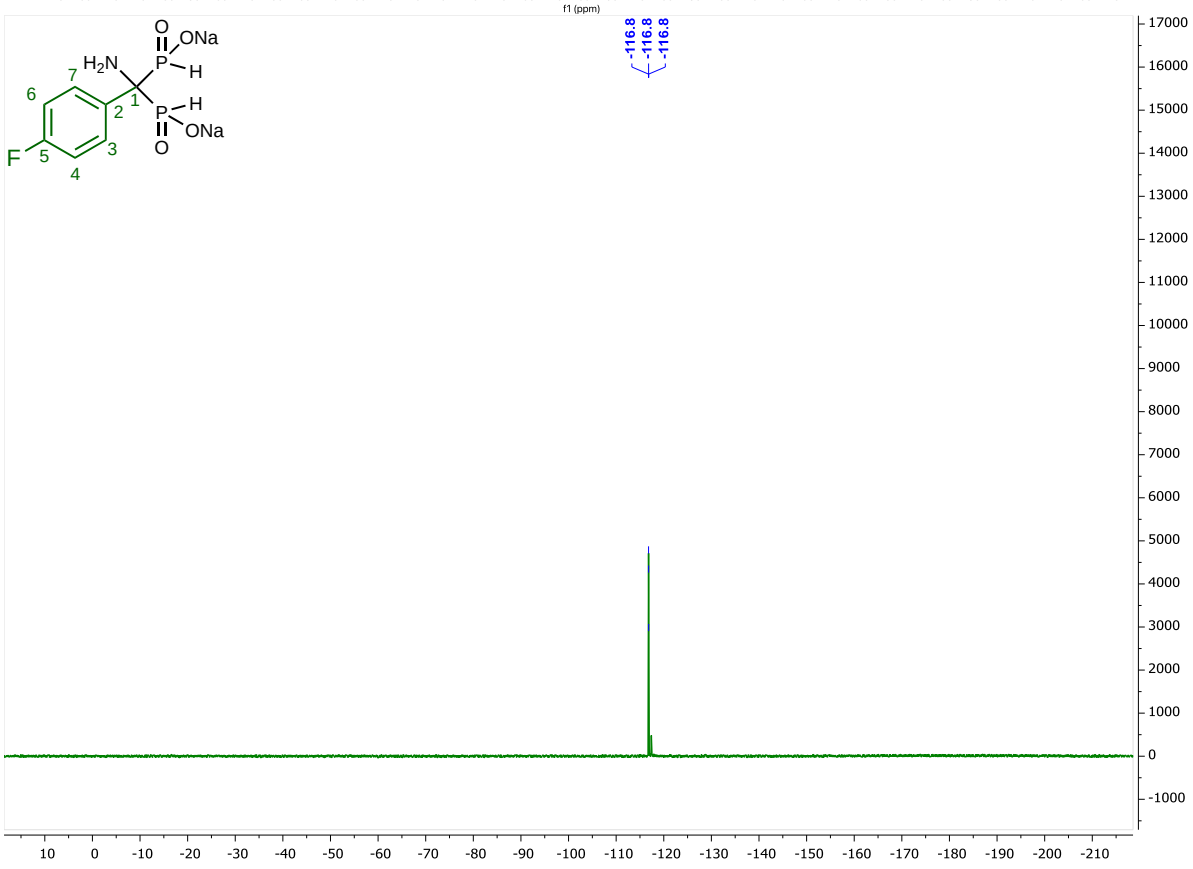


Figure 5. ^{31}P NMR spectrum (162 MHz, D_2O), ^{1}H NMR spectrum (400 MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-amino-1-(4-methoxyphenyl)methane-1,1-bis(H-phosphinate) disodium salts **3f**.


26.44
28.16
28.09
28.03
28.03
24.93
24.77
24.69
48.98
51.02
f1 (ppm)


-116.8
-116.8
-116.8

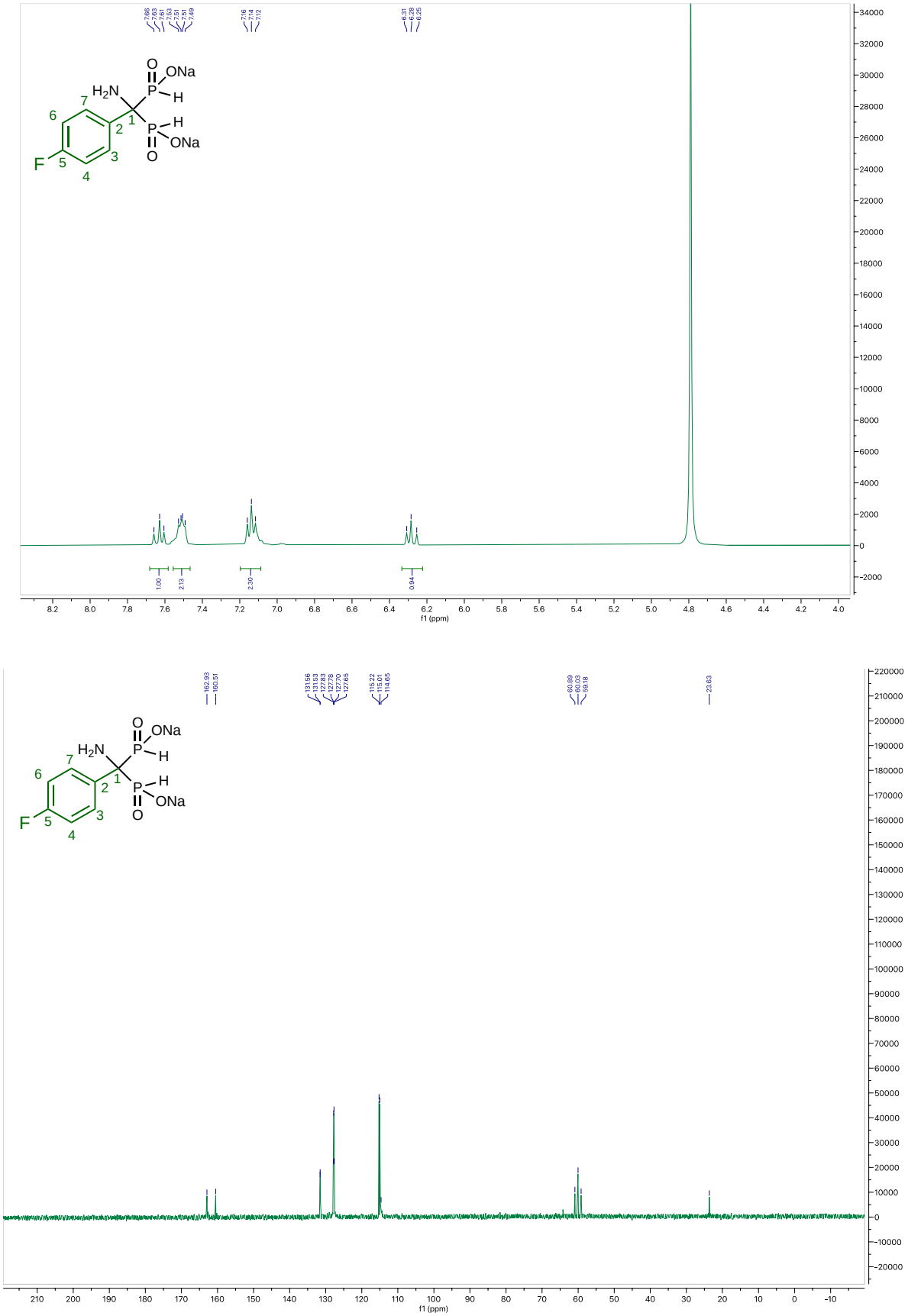
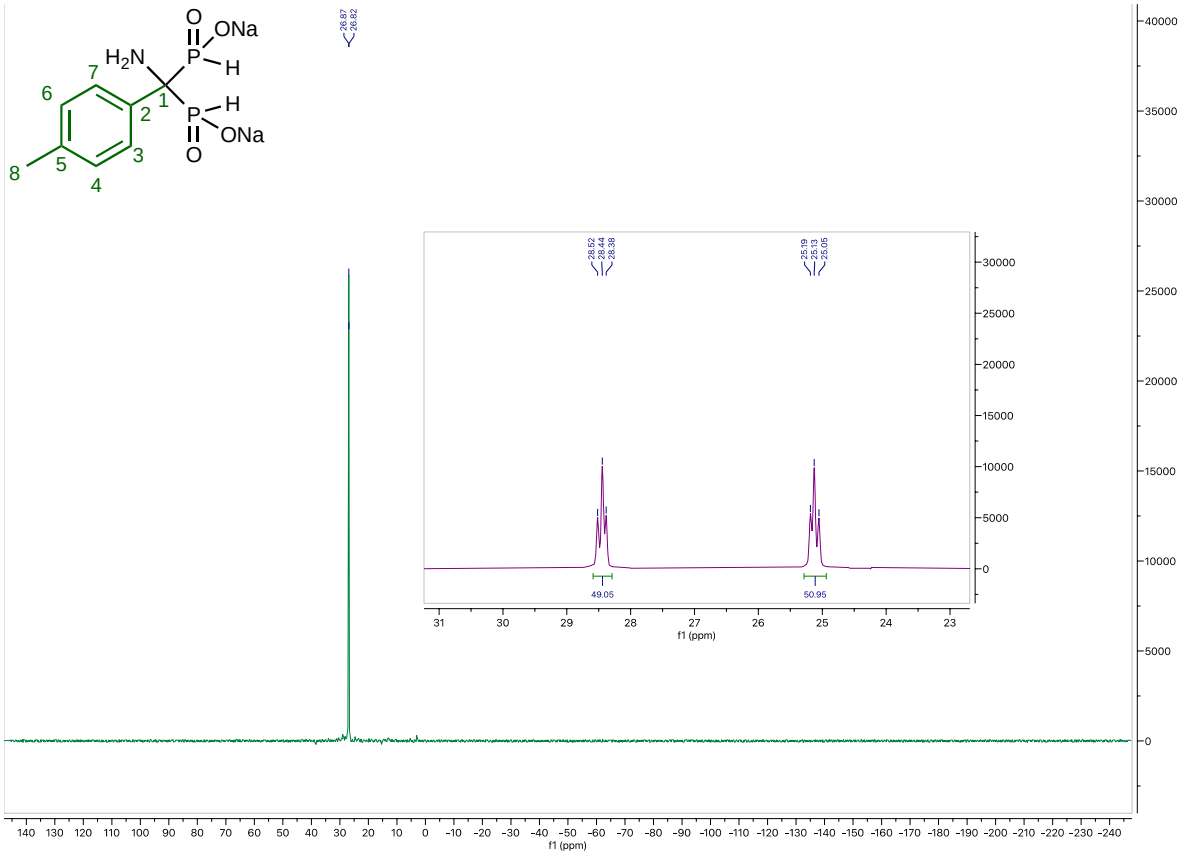
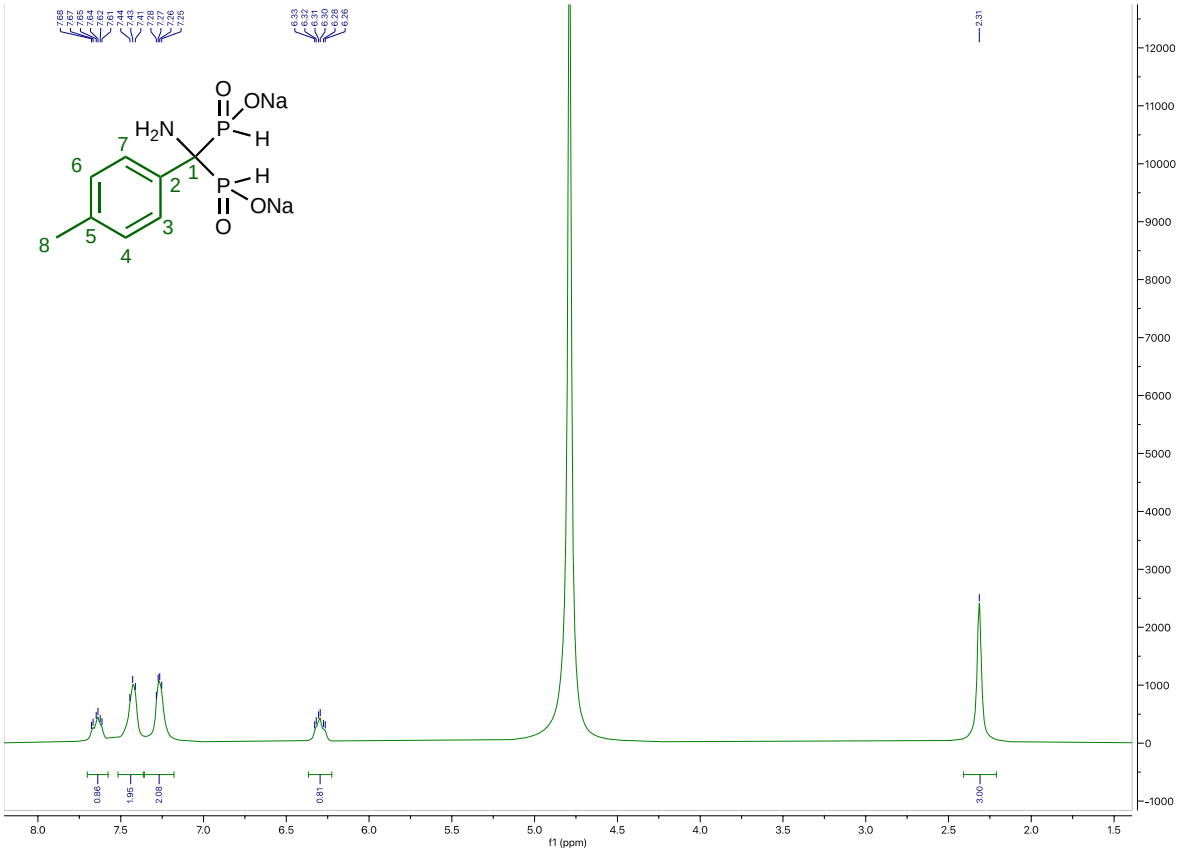


Figure 6. ^{31}P NMR spectrum (162 MHz, D_2O), ^{19}F NMR spectrum (377 MHz, D_2O), ^{1}H NMR spectrum (400MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-amino-1-(4-fluorophenyl)methane-1,1-bis(H-phosphinate) disodium salts **3g**


ONa
H2N
H
H
ONa
O
P
1
2
3
4
5
6
7
8
26.87
26.82
28.52
28.44
28.38
25.19
25.13
25.05
49.05
50.95
f1 (ppm)


ONa
H2N
H
H
ONa
O
P
1
2
3
4
5
6
7
8
7.68
7.67
7.65
7.64
7.62
7.61
7.44
7.43
7.41
7.39
7.27
7.26
7.25
6.33
6.32
6.31
6.30
6.28
6.26
2.31
0.86
1.95
2.08
0.81
3.00
f1 (ppm)

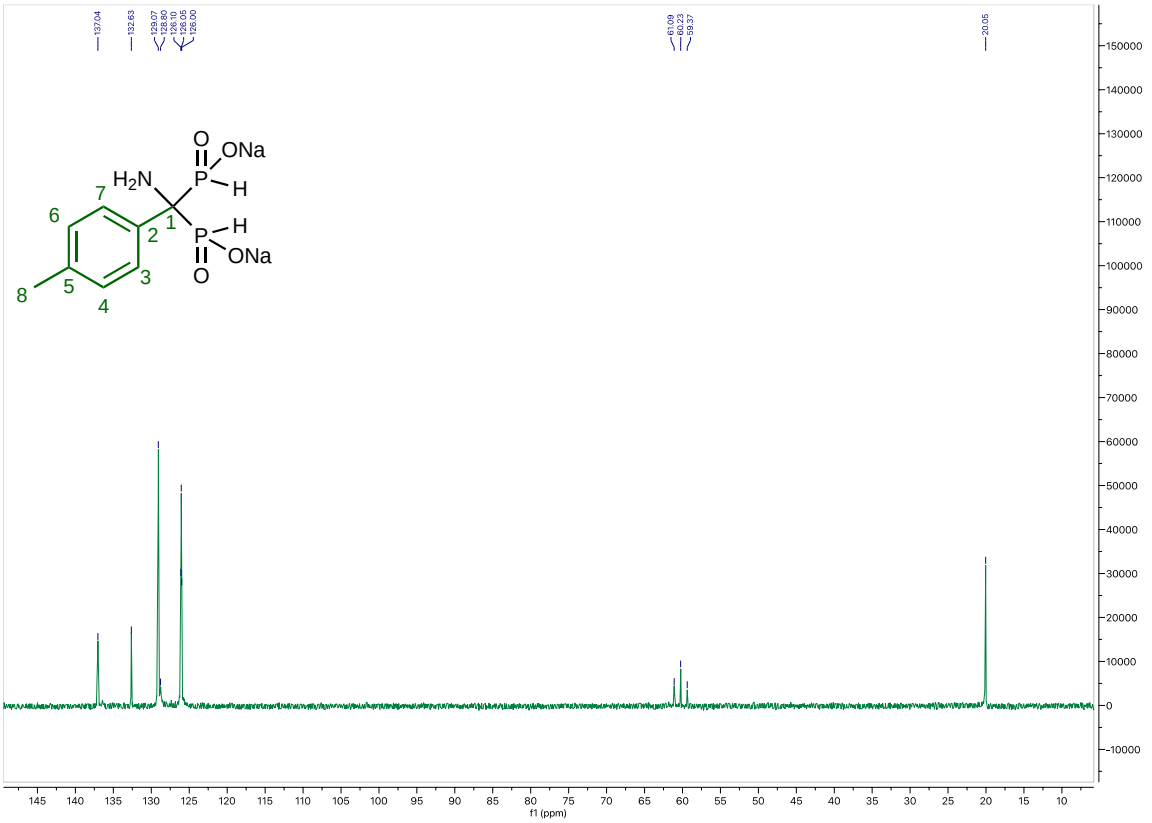
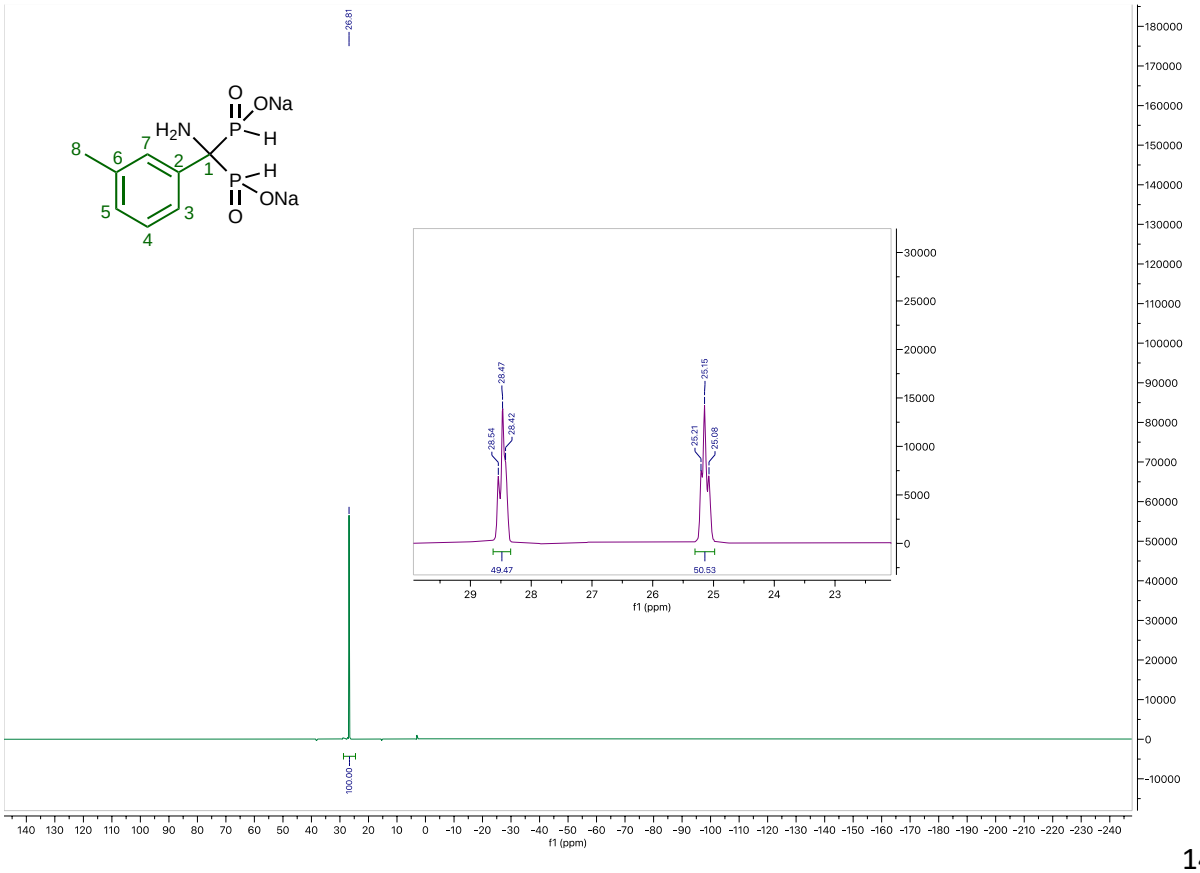


Figure 7. ^{31}P NMR spectrum (162 MHz, D_2O), ^{1}H NMR spectrum (400 MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-amino-1-(4-tolyl)methane-1,1-bis(H-phosphinate) disodium salts **3i**



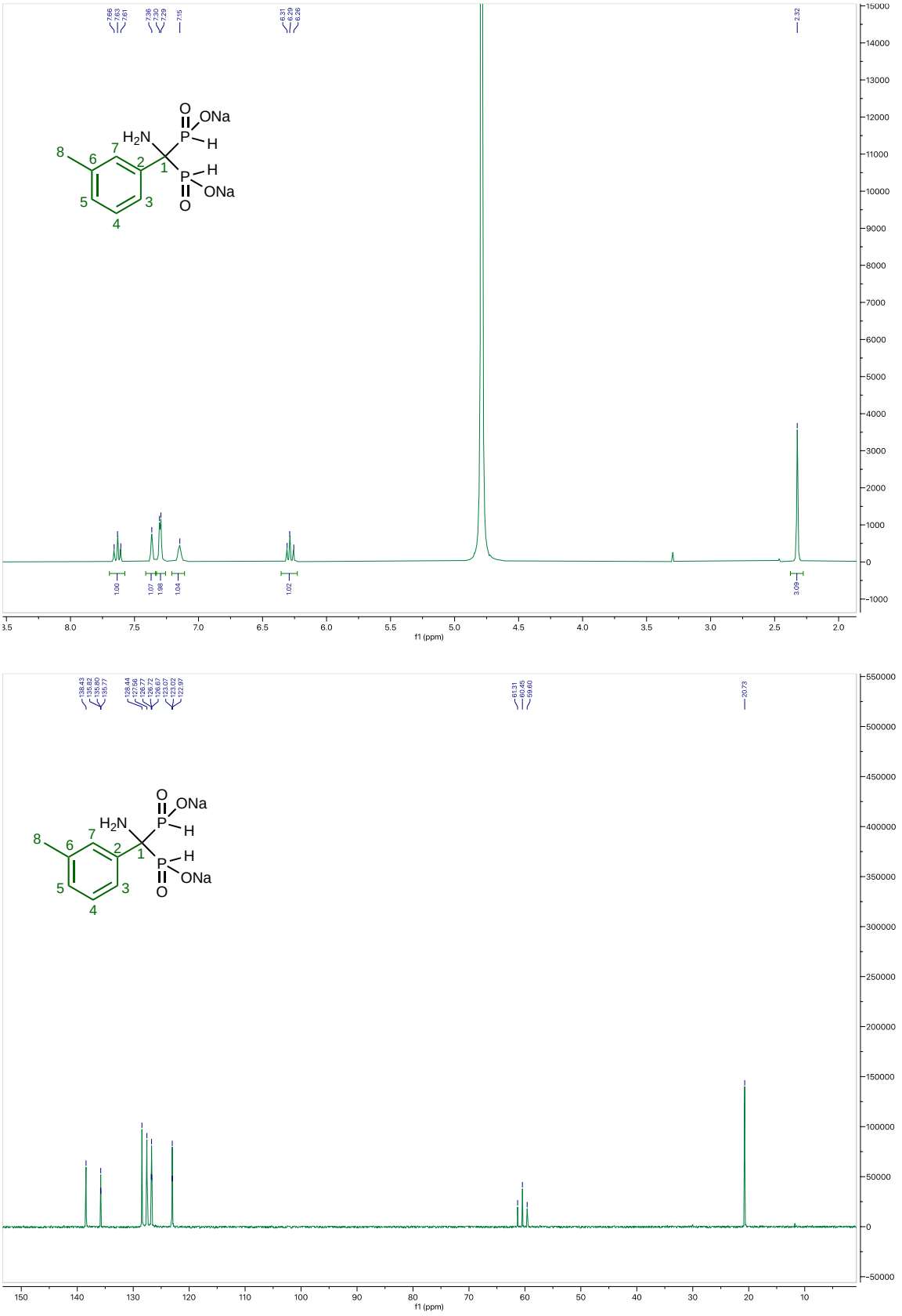
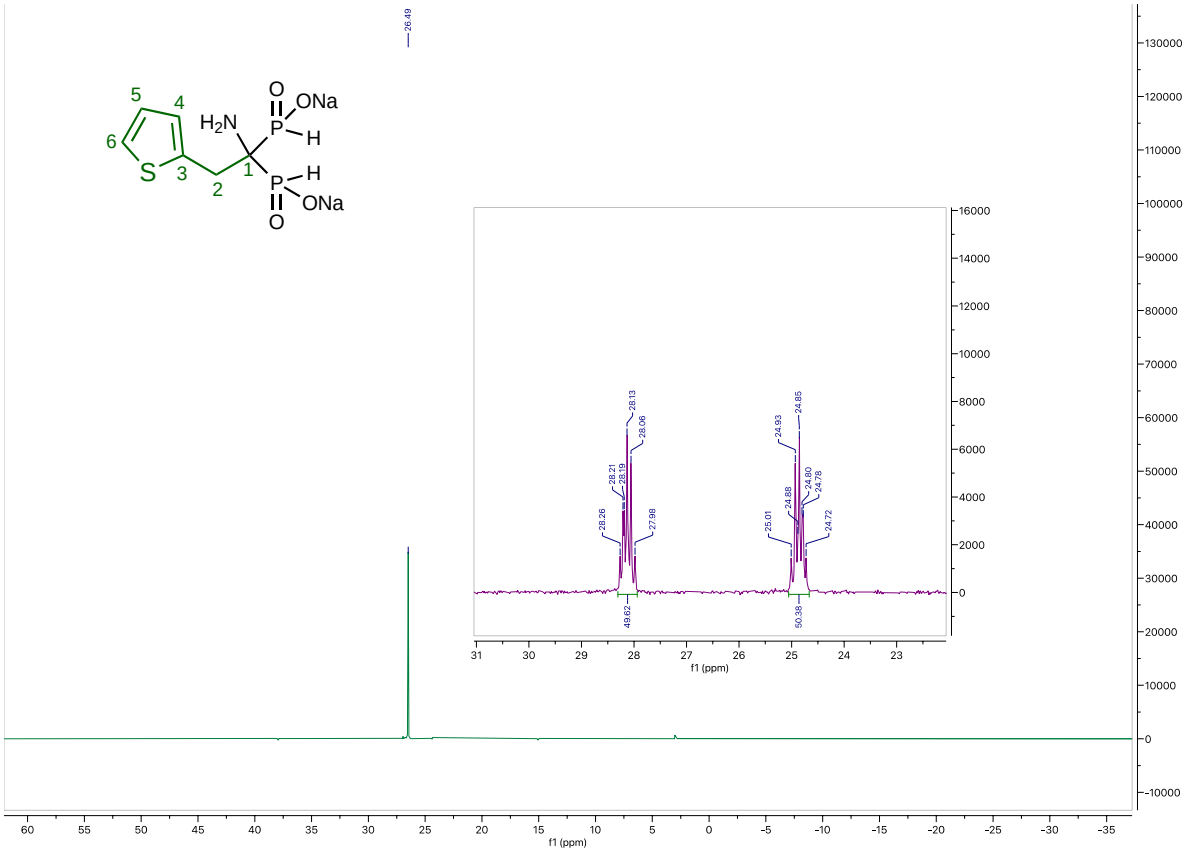
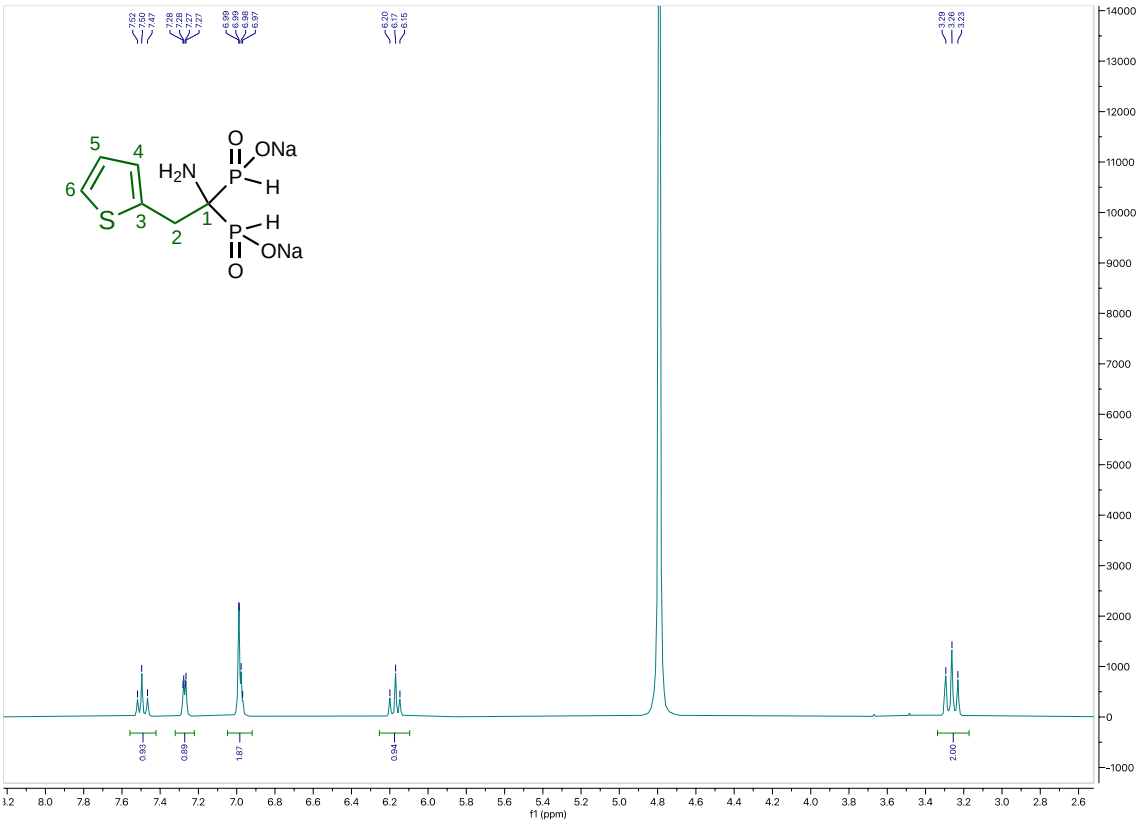


Figure 8. ^{31}P NMR spectrum (162 MHz, D_2O), ^{1}H NMR spectrum (400 MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-amino-1-(3-tolyl)phenyl)methane-1,1-bis(H-phosphinate) disodium salts **3j**


26.49
28.26
28.21
28.19
28.13
28.06
27.98
25.01
24.93
24.88
24.85
24.80
24.78
24.72
49.62
50.38
f1 (ppm)


7.62
7.60
7.47
7.28
7.28
7.27
7.27
6.99
6.99
6.98
6.97
6.20
6.17
6.15
3.29
3.26
3.23
0.93
0.89
1.87
0.94
2.00
f1 (ppm)

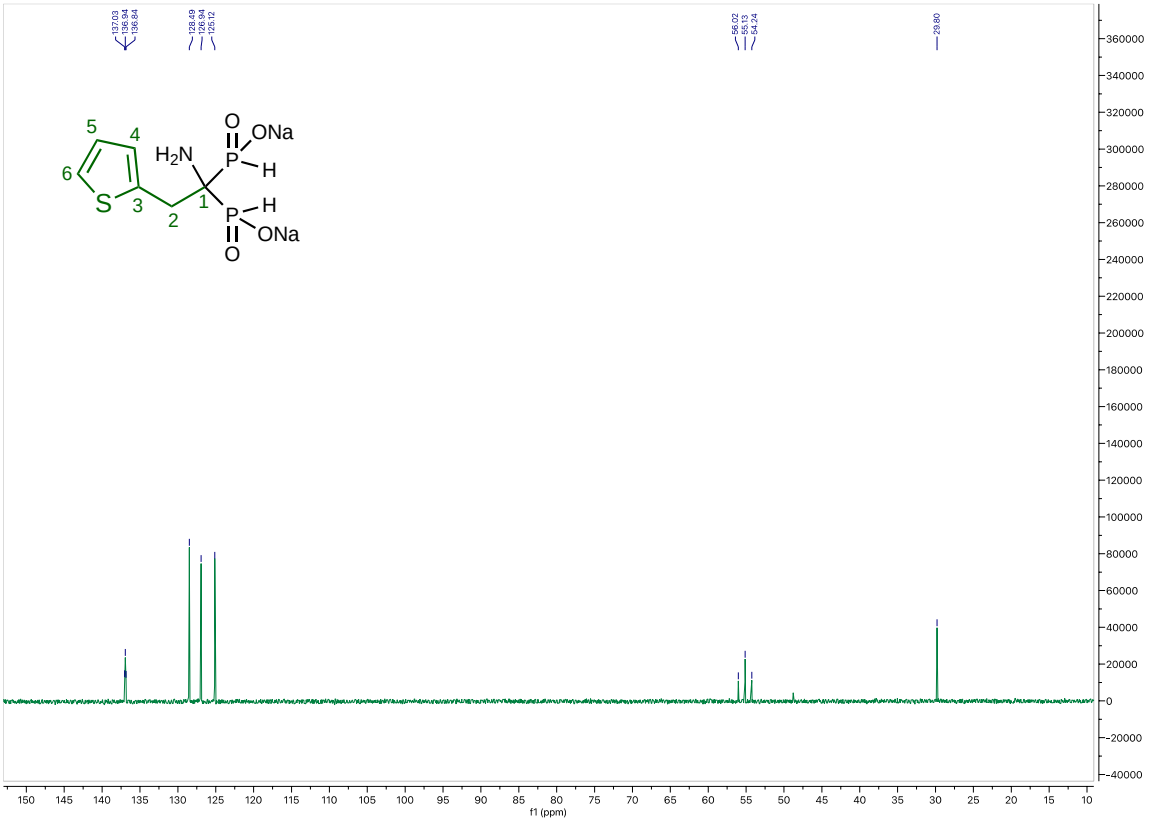


Figure 9. ^{31}P NMR spectrum (162 MHz, D_2O), ^{1}H NMR spectrum (400 MHz, D_2O), ^{13}C NMR (101 MHz, D_2O) of 1-amino-1-(2-thienyl)ethane-1,1-bis(H-phosphinate) disodium salts **3l**.