Electronic Supplementary Information

Palladium-Catalyzed Air-based Oxidative Coupling of Arylboronic Acids with H-phosphine Oxides Leading to Aryl Phosphine Oxides

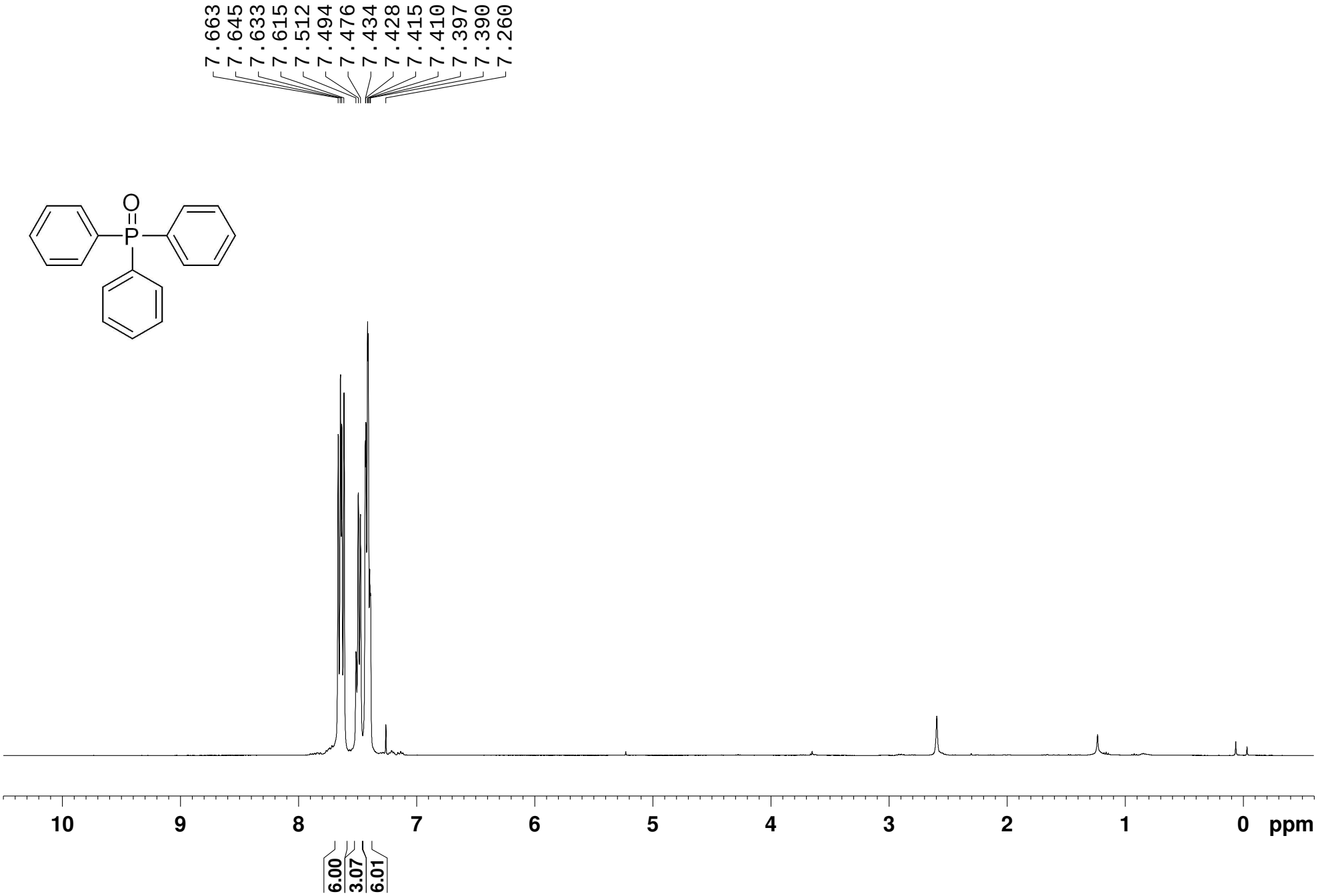
Tingting Fu,[b] Hongwei Qiao,[a] Zhimin Peng,[a] Gaobo Hu,[a] Xueji Wu,[b] Yuxing Gao,*[, a] Yufen Zhao[a,b]

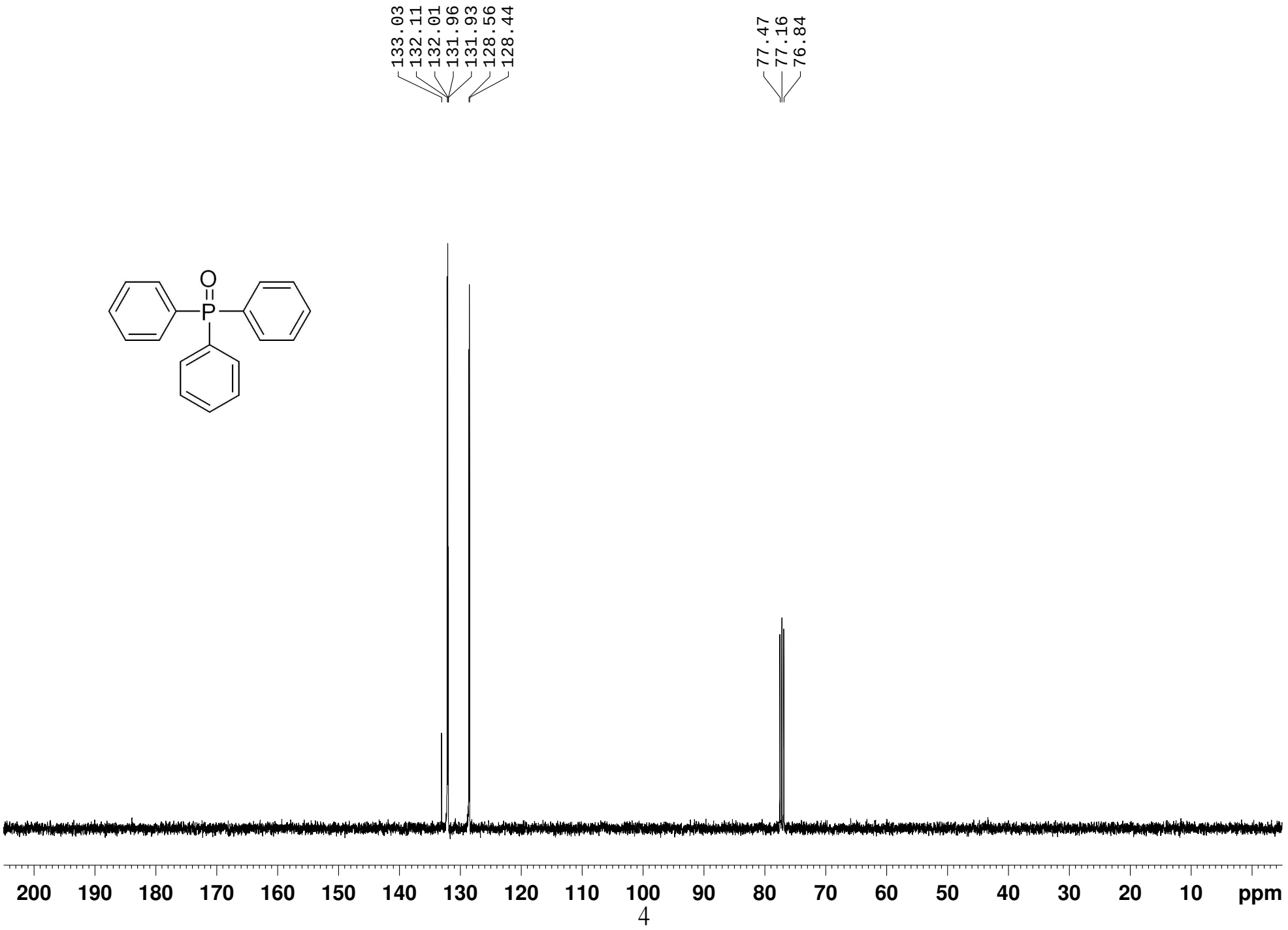
[a] Department of Chemistry, College of Chemistry and Chemical Engineering, and the Key Laboratory for Chemical Biology of Fujian Province, Xiamen University, Xiamen, Fujian 361005, China, Fax: (86)592-2185780; E-mail: gaoxingchem@xmu.edu.cn

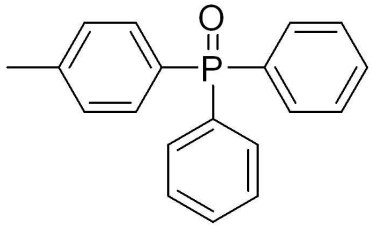
[b] Department of Chemical Biology, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen, Fujian 361005, China

Copies of ^{31}P NMR, ^{1}H NMR and ^{13}C NMR spectra of coupling products

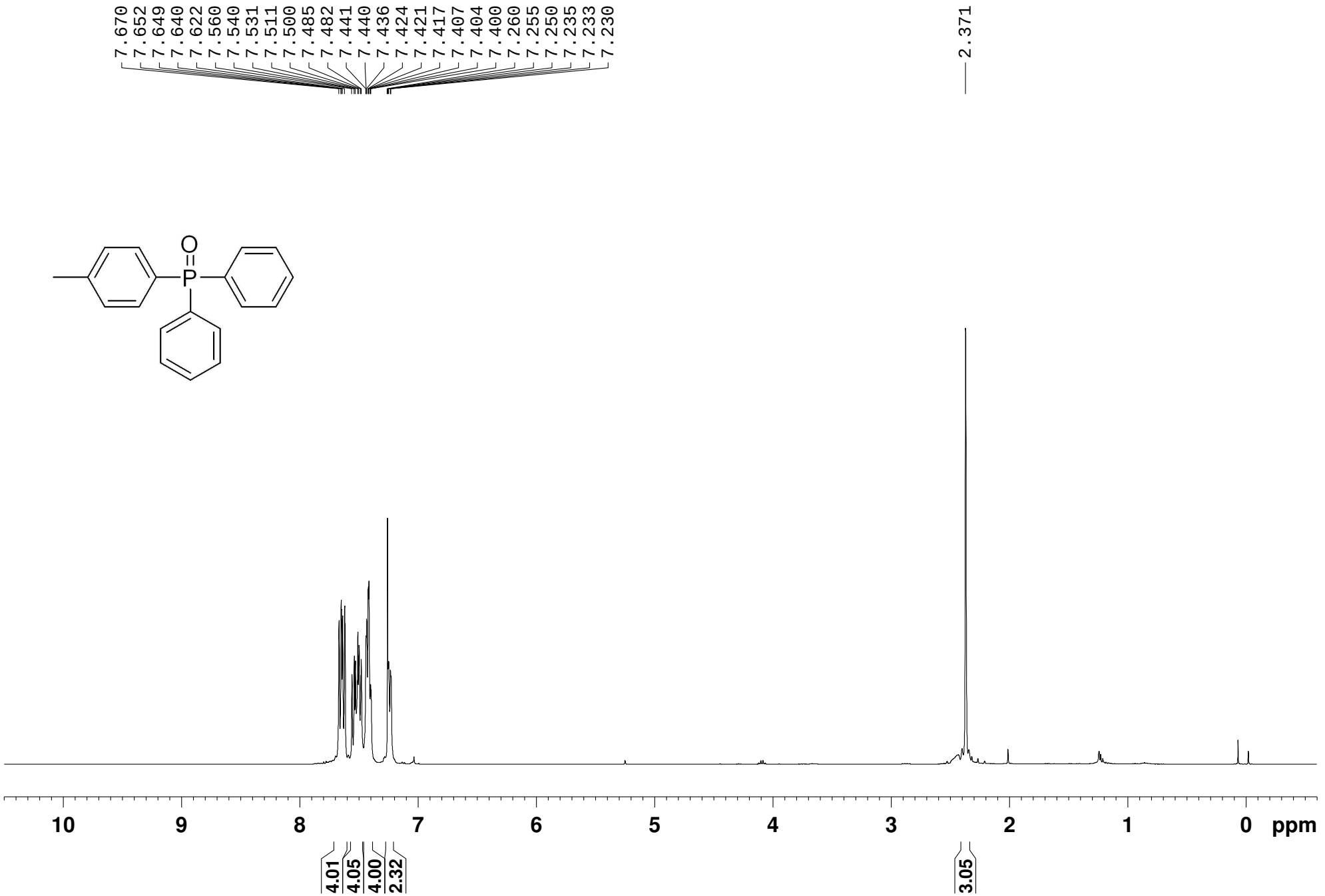

29.10
O
P
3a
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
−40
−50
ppm

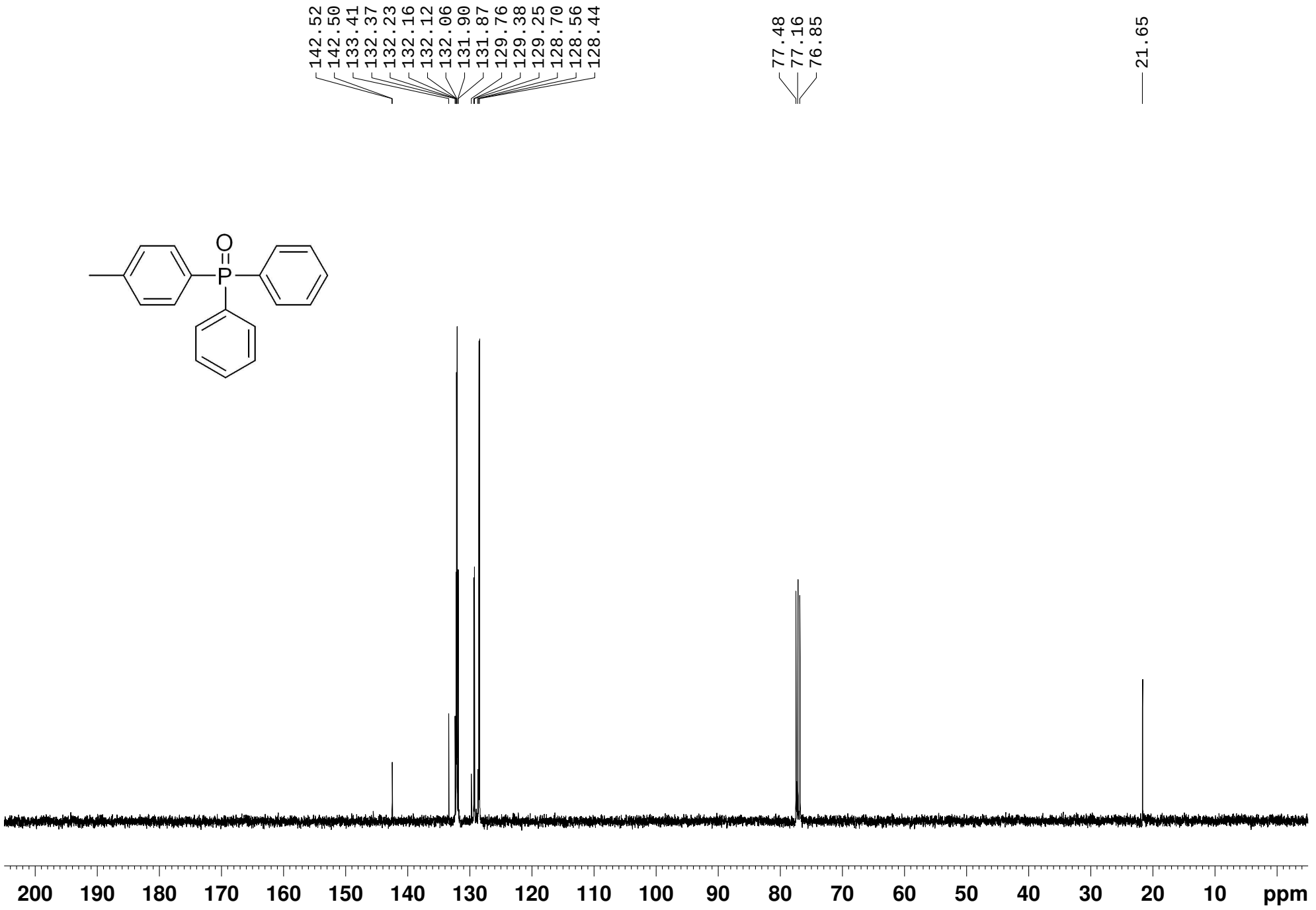

7.663
7.645
7.633
7.615
7.512
7.494
7.476
7.434
7.428
7.415
7.410
7.397
7.390
7.260
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
6.00
3.07
6.01

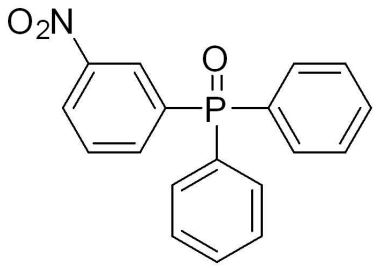

133.03
132.11
132.01
131.96
131.93
128.56
128.44
77.47
77.16
76.84
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



3b


7.670
7.652
7.649
7.640
7.622
7.560
7.540
7.531
7.511
7.500
7.485
7.482
7.441
7.440
7.436
7.424
7.421
7.417
7.407
7.404
7.400
7.260
7.255
7.250
7.235
7.233
7.230
2.371
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
4.01
4.05
4.00
2.32
3.05

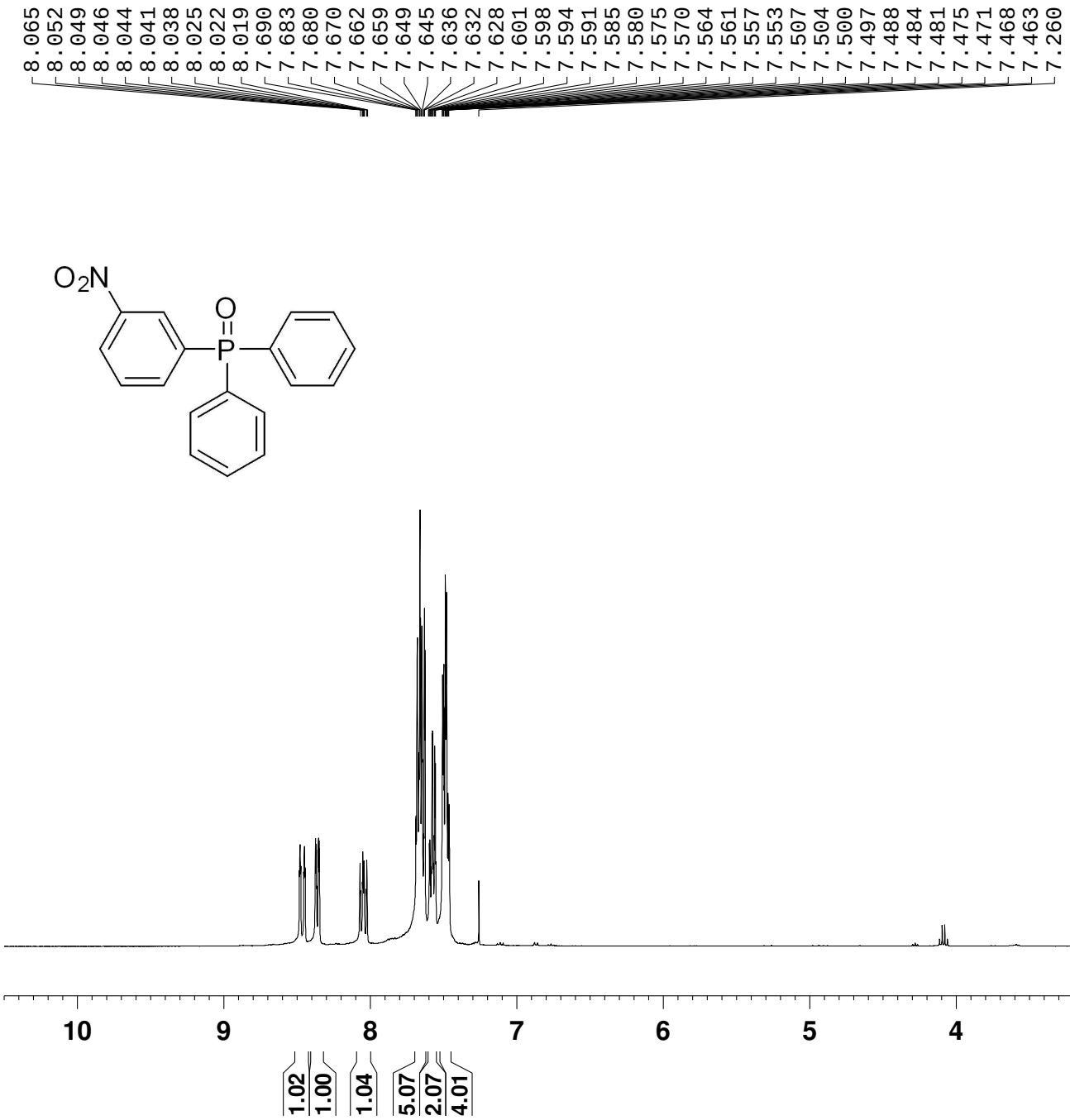

142.52
142.50
133.41
132.37
132.23
132.16
132.12
132.06
131.90
131.87
129.76
129.38
129.25
128.70
128.56
128.44
77.48
77.16
76.85
21.65
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

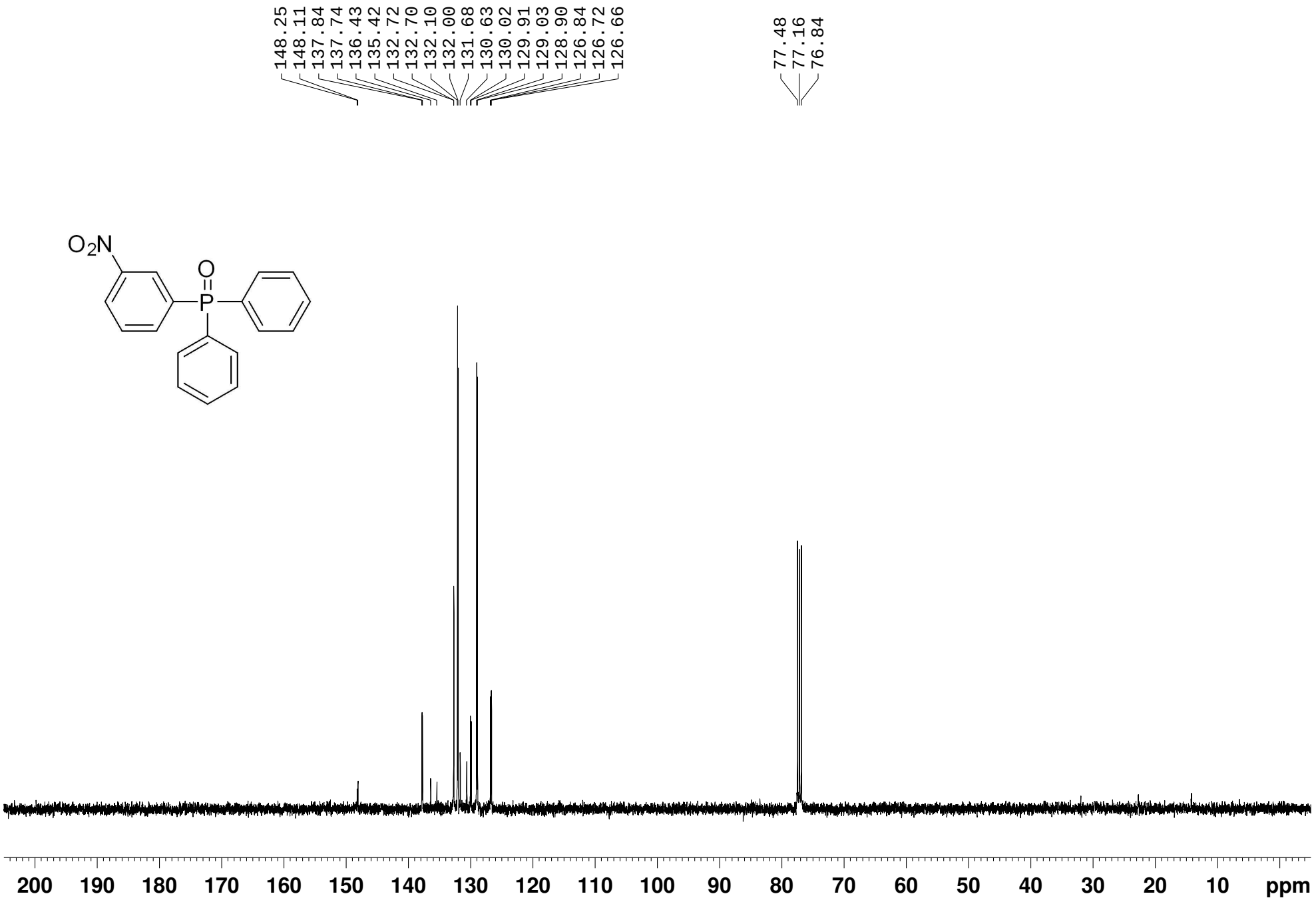


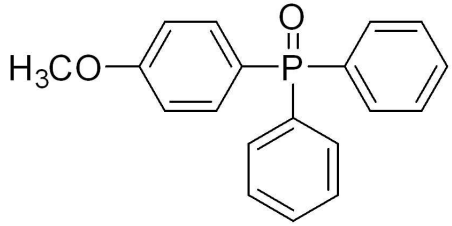
3c

27.40

100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm


8.065
8.052
8.049
8.046
8.044
8.041
8.038
8.025
8.022
8.019
7.690
7.683
7.680
7.670
7.662
7.659
7.649
7.645
7.636
7.632
7.628
7.601
7.598
7.594
7.591
7.585
7.580
7.575
7.570
7.564
7.561
7.557
7.553
7.507
7.504
7.500
7.497
7.488
7.484
7.481
7.475
7.471
7.468
7.463
7.260
O2N
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
1.02
1.00
1.04
5.07
2.07
4.01

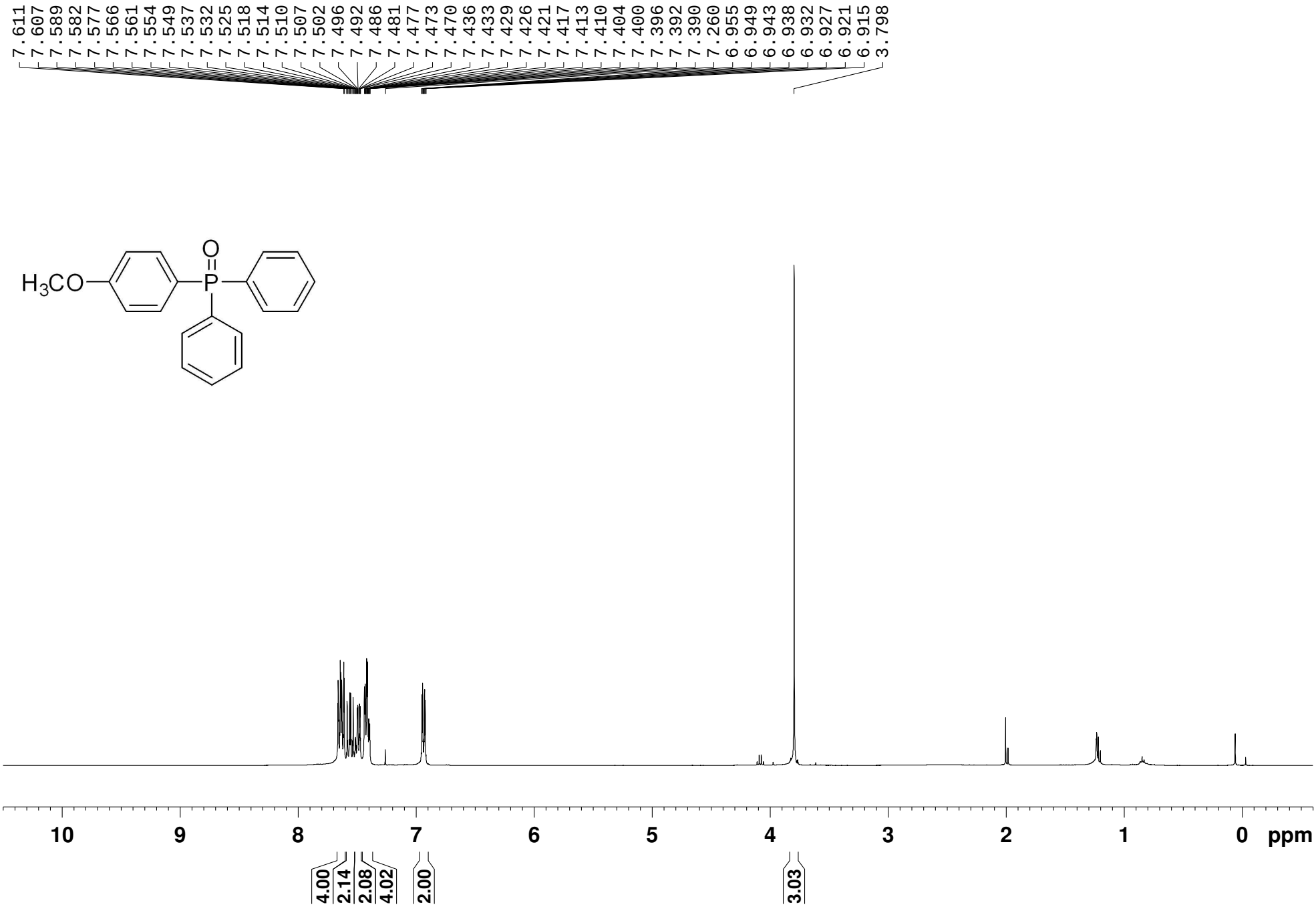

148.25
148.11
137.84
137.74
136.43
135.42
132.72
132.70
132.10
132.00
131.68
130.63
130.02
129.91
129.03
128.90
126.84
126.72
126.66
77.48
77.16
76.84
O2N
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

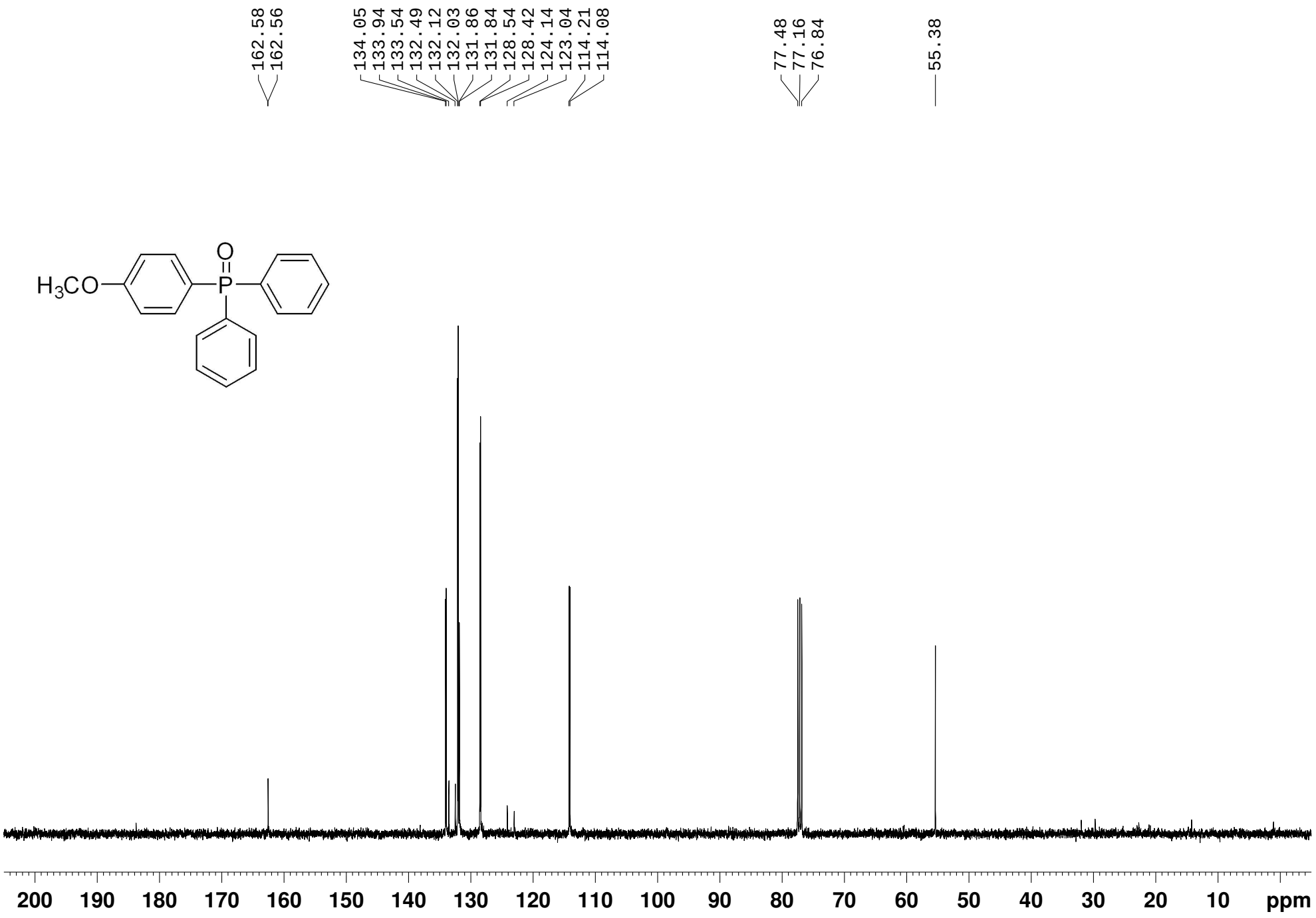


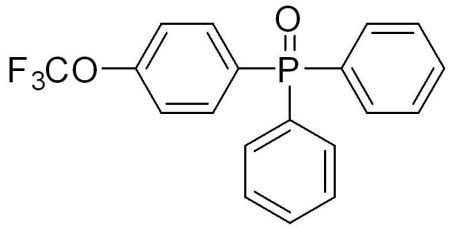
3d

29.09

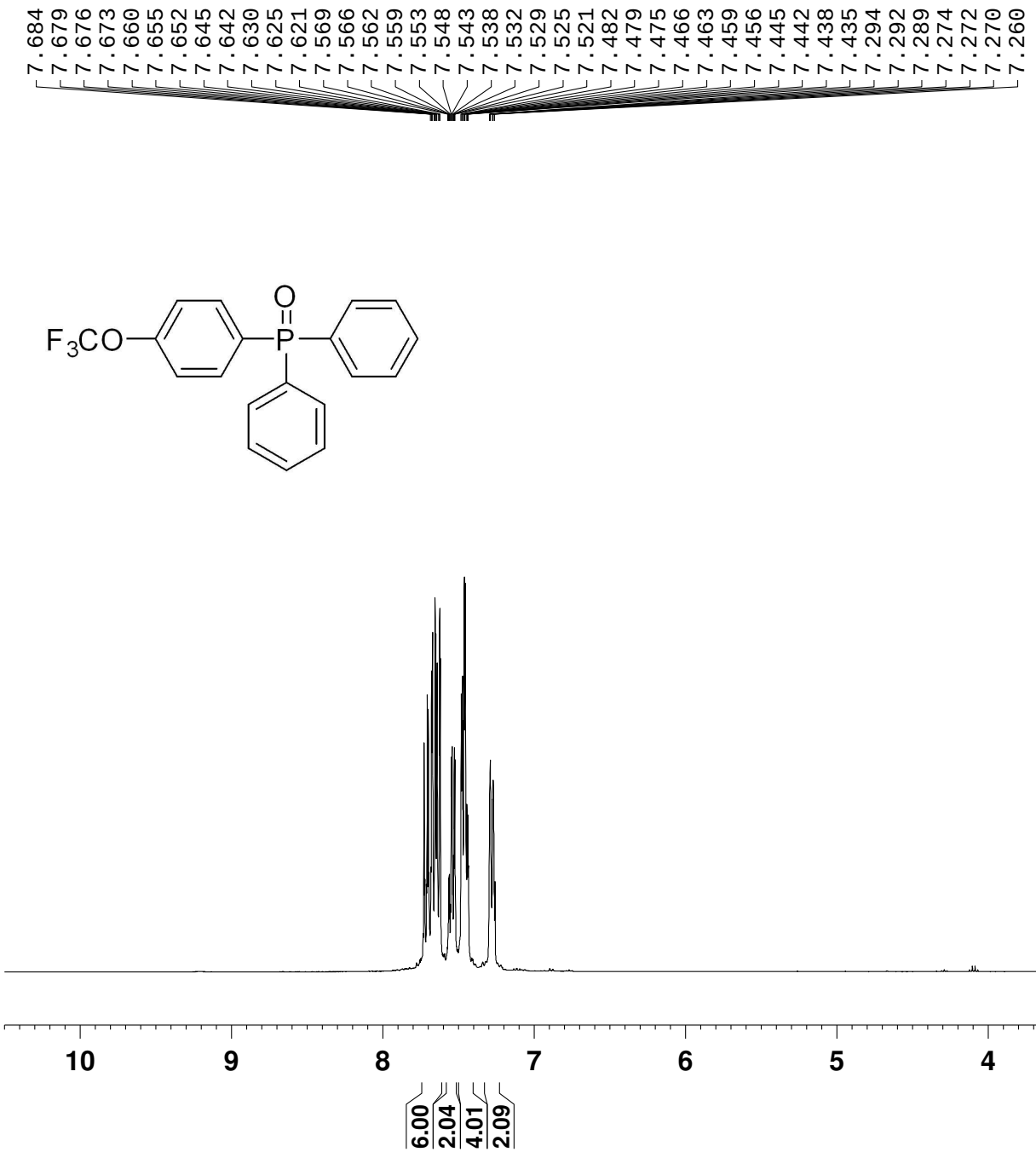
100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm

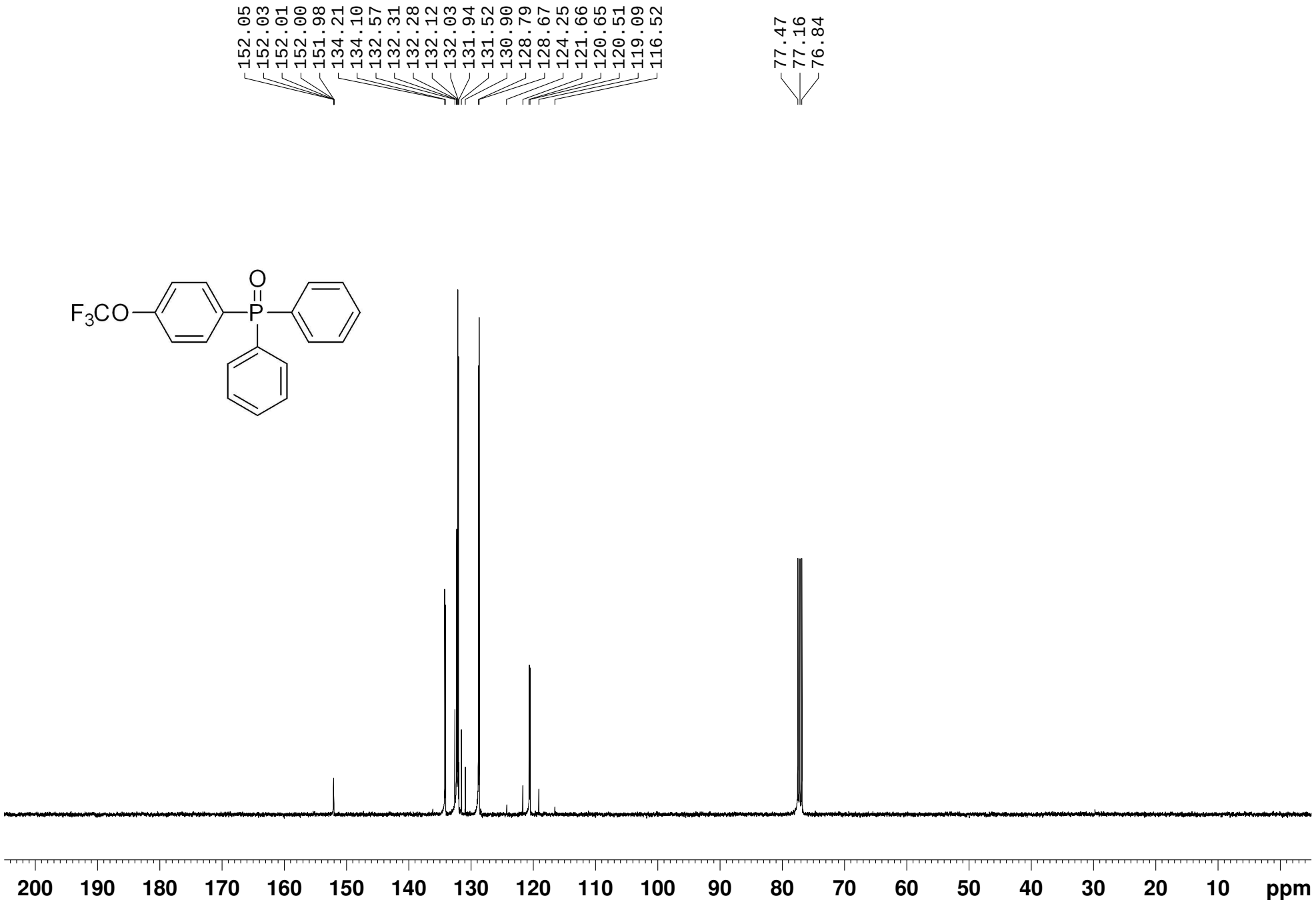

7.611
7.607
7.589
7.582
7.577
7.566
7.561
7.554
7.549
7.537
7.532
7.525
7.518
7.514
7.510
7.507
7.502
7.496
7.492
7.486
7.481
7.477
7.473
7.470
7.436
7.433
7.429
7.426
7.421
7.417
7.413
7.410
7.404
7.400
7.396
7.392
7.390
7.260
6.955
6.949
6.943
6.938
6.932
6.927
6.921
6.915
3.798
H3CO
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
4.00
2.14
2.08
4.02
2.00
3.03

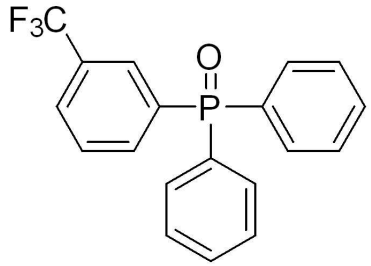

162.58
162.56
134.05
133.94
133.54
132.49
132.12
132.03
131.86
131.84
128.54
128.42
124.14
123.04
114.21
114.08
77.48
77.16
76.84
55.38
H3CO
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



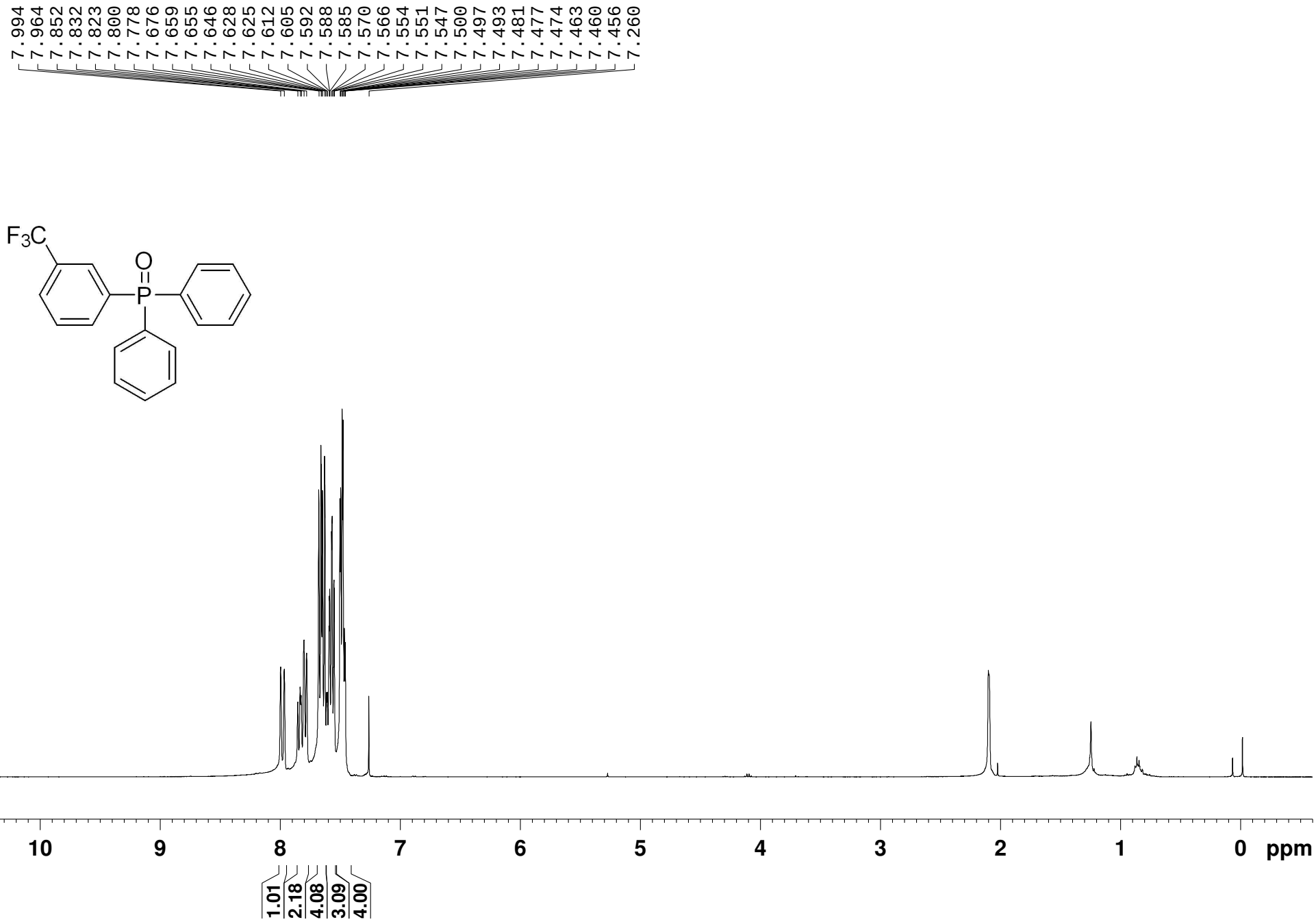
3e

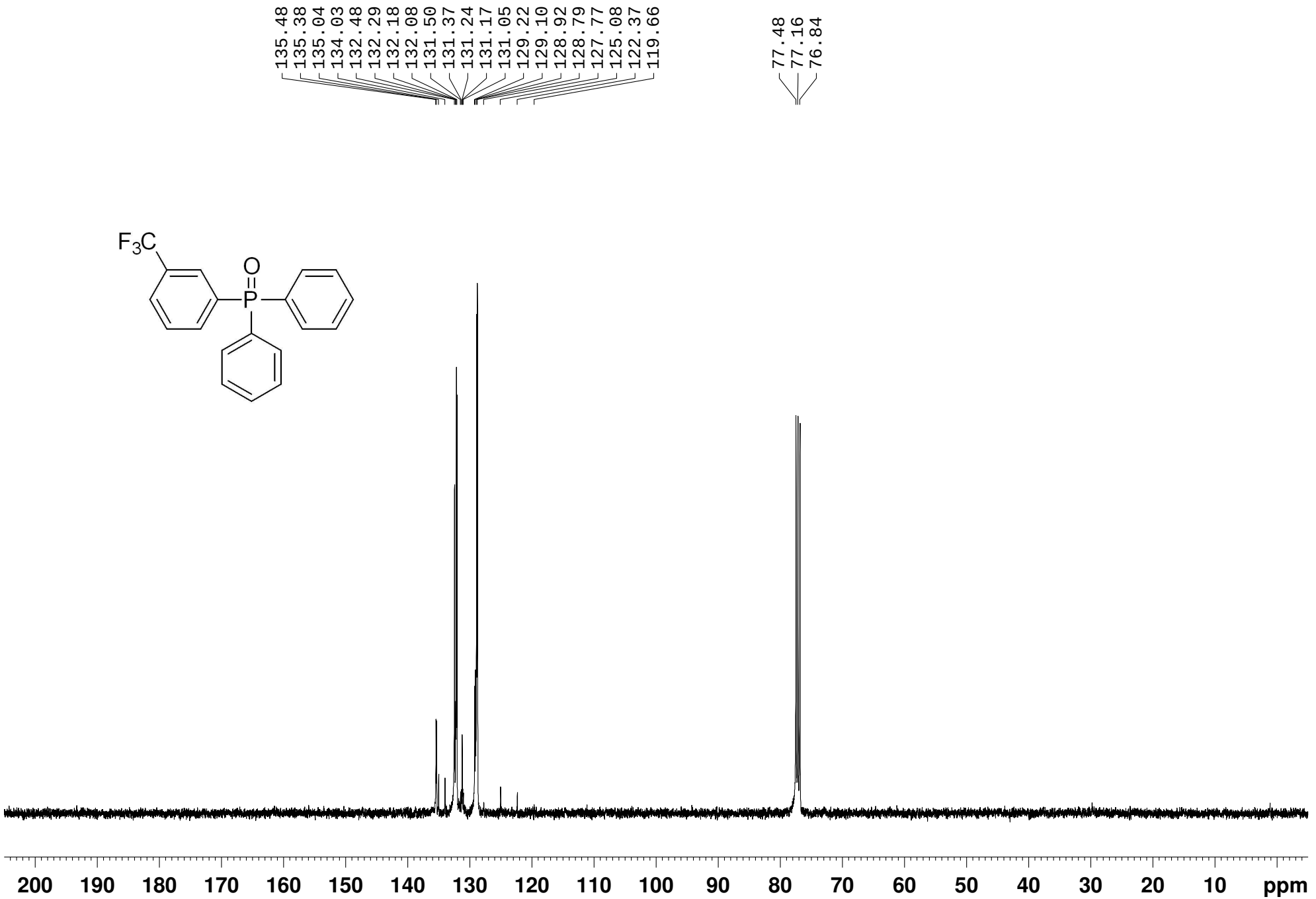

7.684
7.679
7.676
7.673
7.660
7.655
7.652
7.645
7.642
7.630
7.625
7.621
7.569
7.566
7.562
7.559
7.553
7.548
7.543
7.538
7.532
7.529
7.525
7.521
7.482
7.479
7.475
7.466
7.463
7.459
7.456
7.445
7.442
7.438
7.435
7.294
7.292
7.289
7.274
7.272
7.270
7.260
F3CO
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
6.00
2.04
4.01
2.09

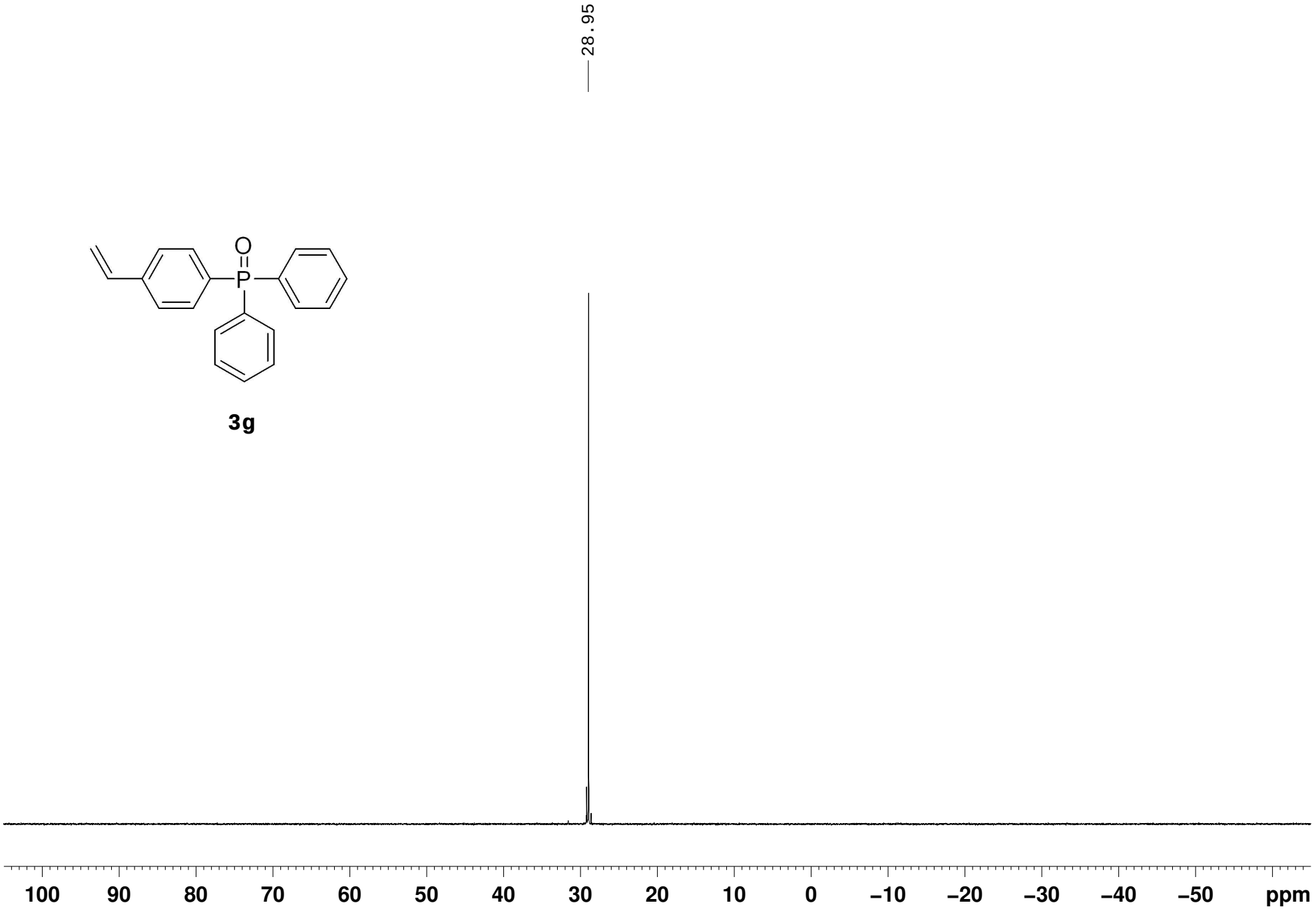

152.05
152.03
152.01
152.00
151.98
134.21
134.10
132.57
132.31
132.28
132.12
132.03
131.94
131.52
130.90
128.79
128.67
124.25
121.66
120.65
120.51
119.09
116.52
77.47
77.16
76.84
O
P
F3CO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



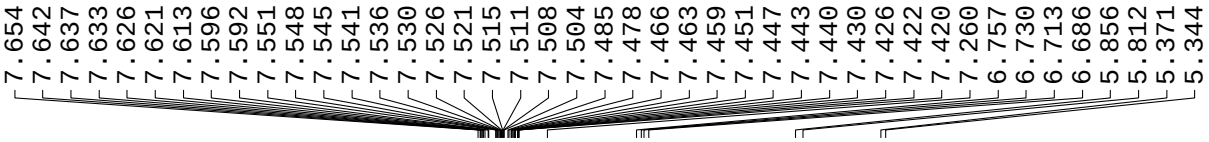
3f

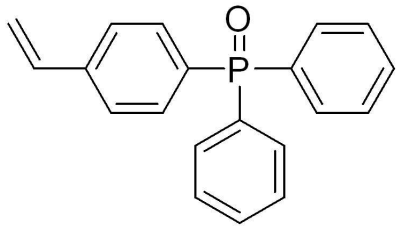

7.994
7.964
7.852
7.832
7.823
7.800
7.778
7.676
7.659
7.655
7.646
7.628
7.625
7.612
7.605
7.592
7.588
7.585
7.570
7.566
7.554
7.551
7.547
7.500
7.497
7.493
7.481
7.477
7.474
7.463
7.460
7.456
7.260
F3C
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.18
4.08
3.09
4.00

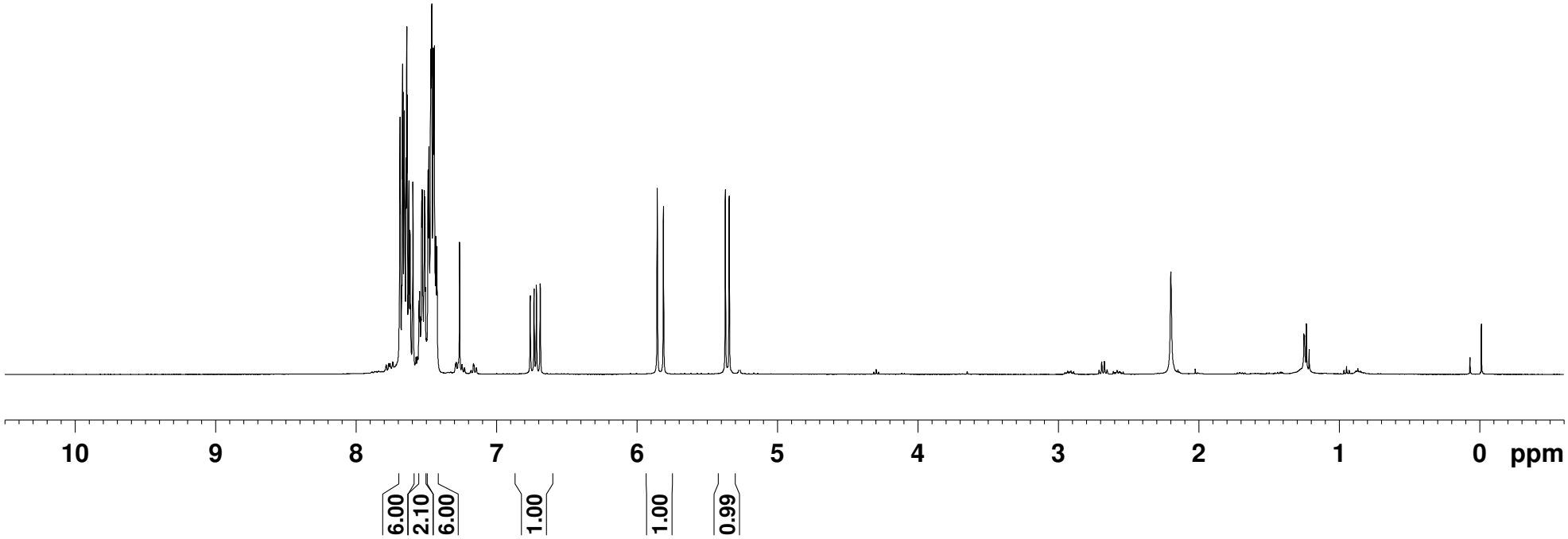

135.48
135.38
135.04
134.03
132.48
132.29
132.18
132.08
131.50
131.37
131.24
131.17
131.05
129.22
129.10
128.92
128.79
127.77
125.08
122.37
119.66
77.48
77.16
76.84
F3C
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

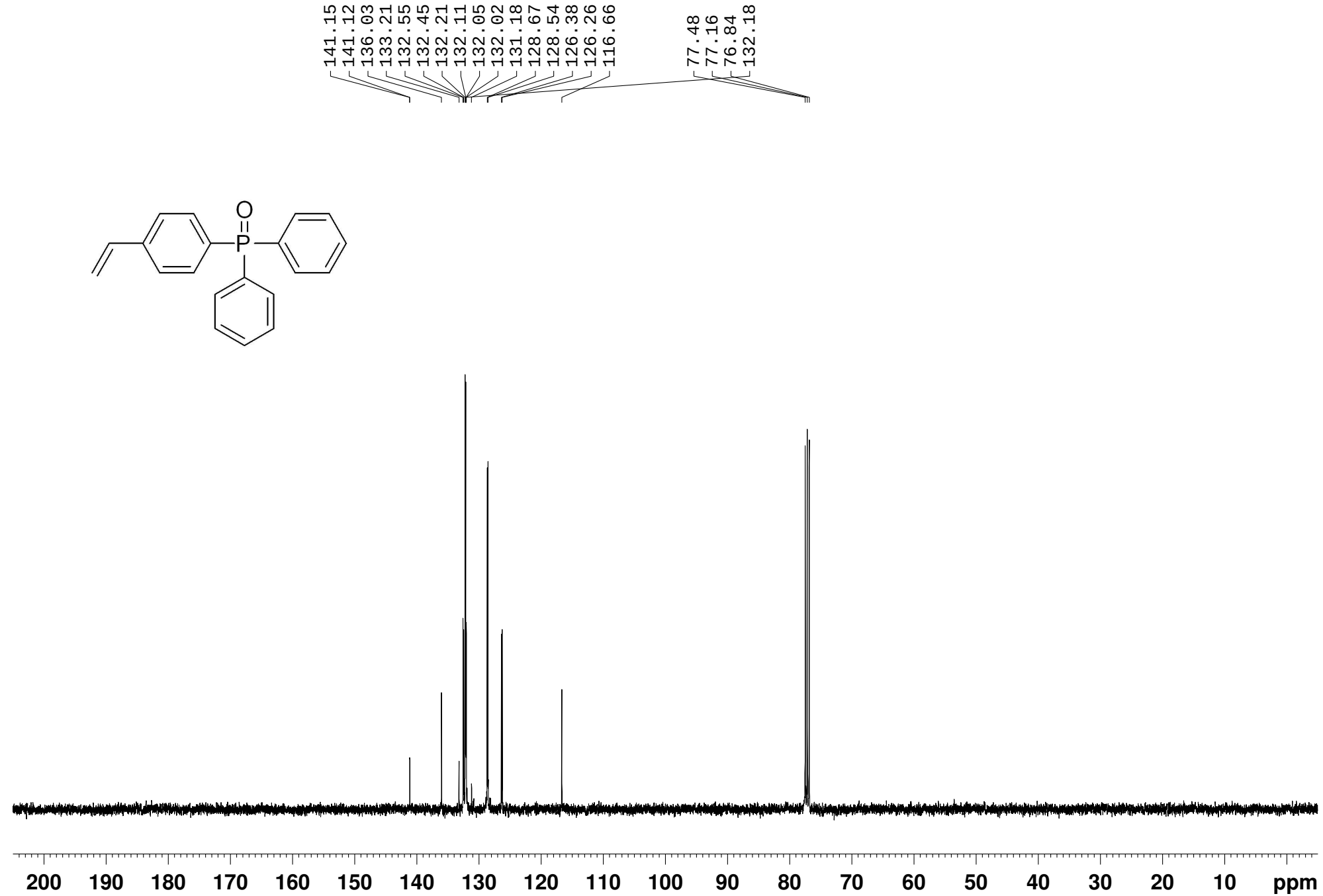


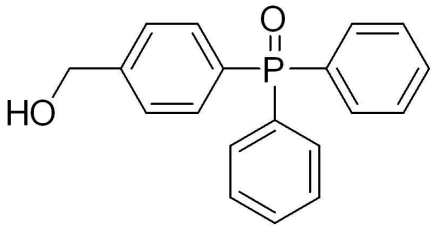
3g


7.654
7.642
7.637
7.633
7.626
7.621
7.613
7.596
7.592
7.551
7.548
7.545
7.541
7.536
7.530
7.526
7.521
7.515
7.511
7.508
7.504
7.485
7.478
7.466
7.463
7.459
7.451
7.447
7.443
7.440
7.430
7.426
7.422
7.420
7.260
6.757
6.730
6.713
6.686
5.856
5.812
5.371
5.344


O
P


10
9
8
7
6
5
4
3
2
1
0
ppm
6.00
2.10
6.00
1.00
1.00
0.99

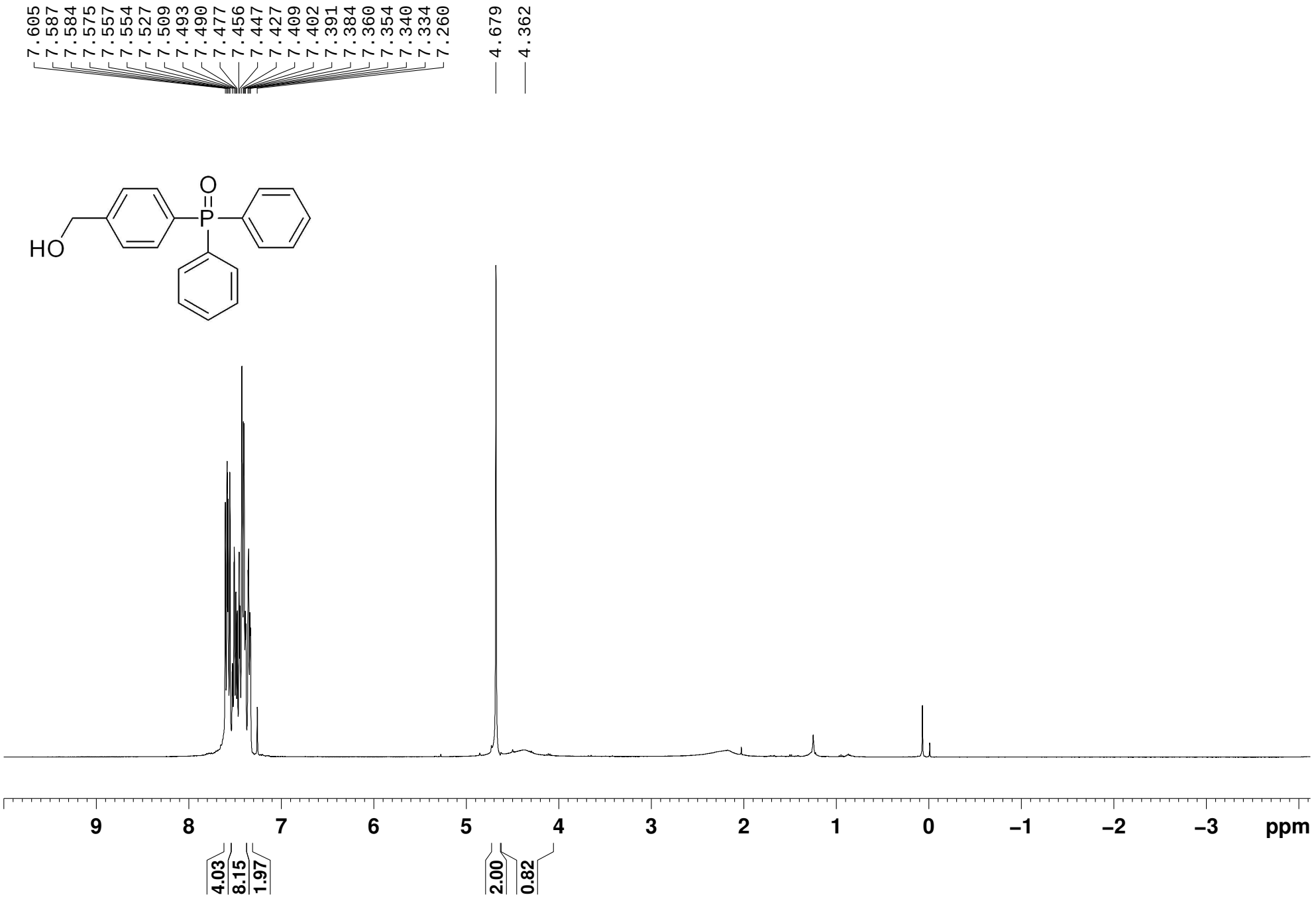

141.15
141.12
136.03
133.21
132.55
132.45
132.21
132.11
132.05
132.02
131.18
128.67
128.54
126.38
126.26
116.66
77.48
77.16
76.84
132.18
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

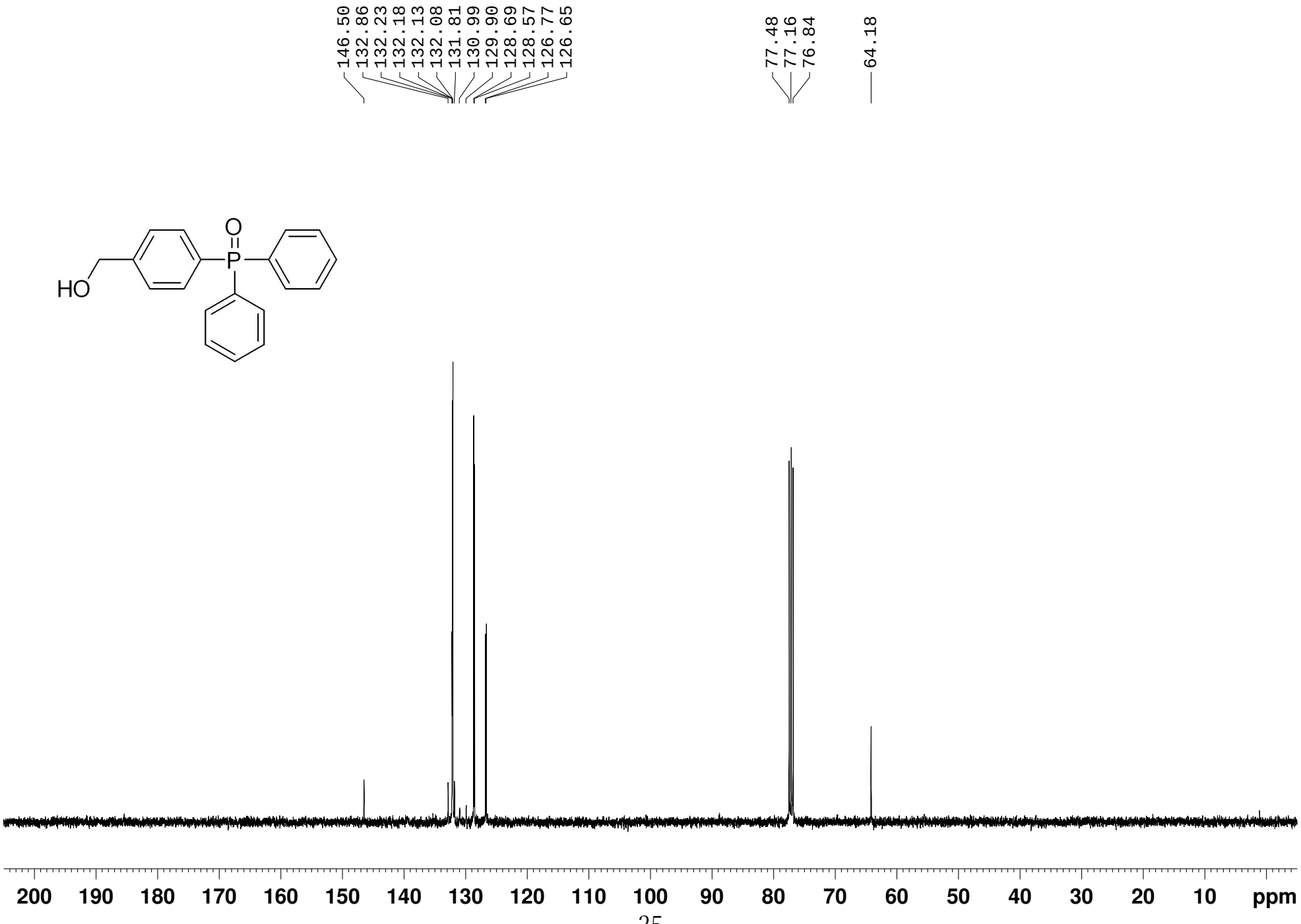


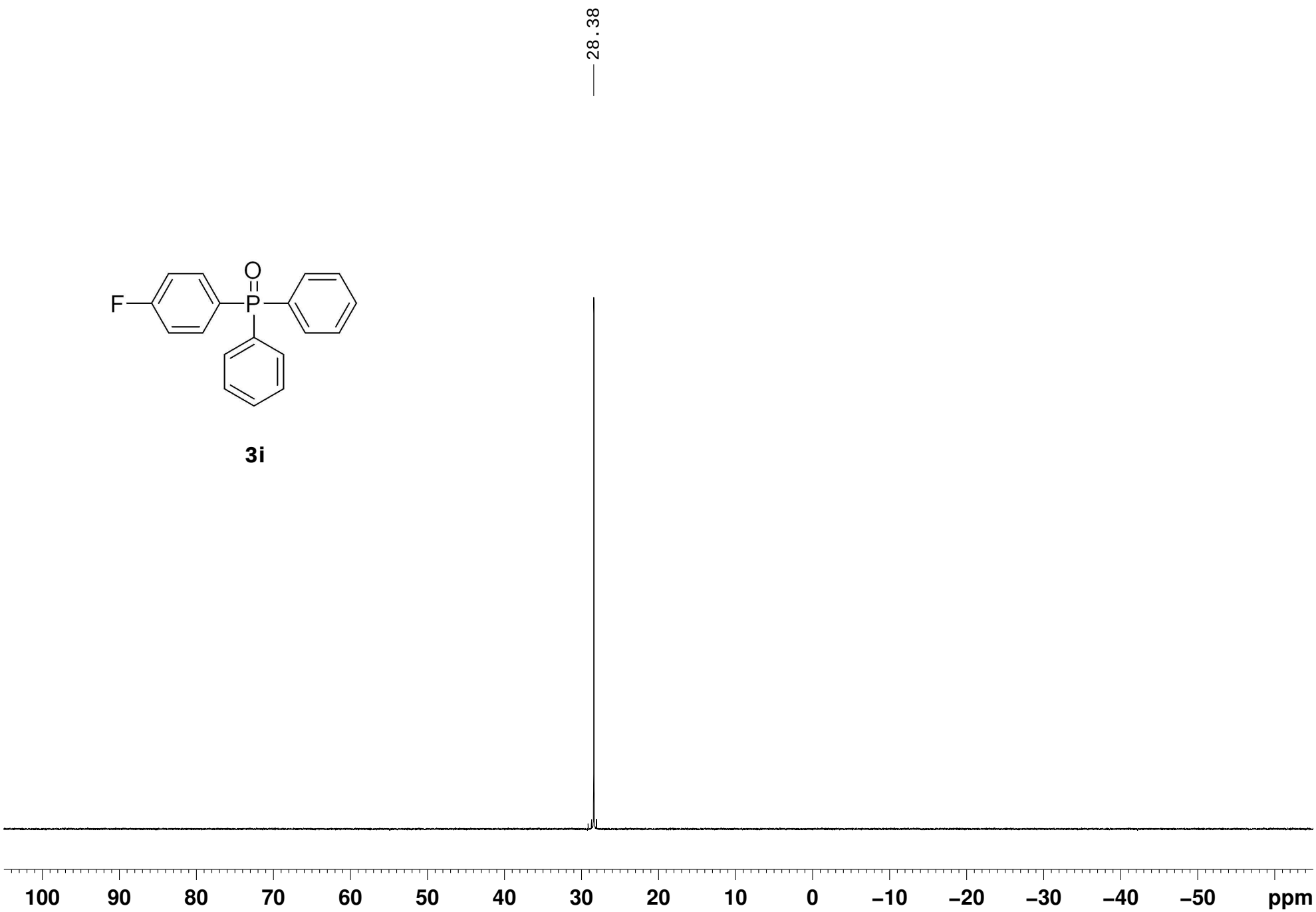
3h

30.13

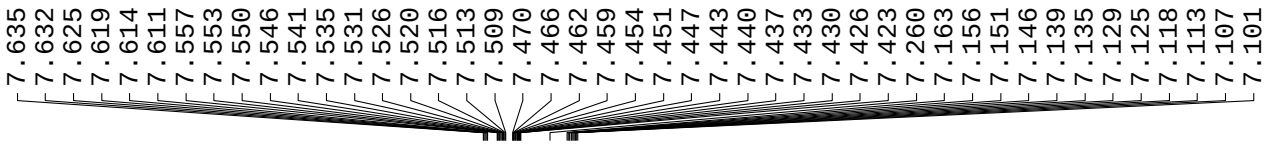
100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm

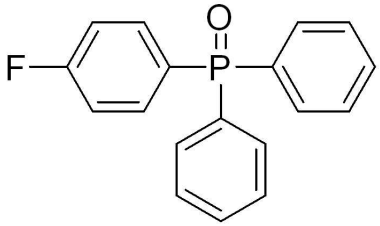

7.605
7.587
7.584
7.575
7.557
7.554
7.527
7.509
7.493
7.490
7.477
7.456
7.447
7.427
7.409
7.402
7.391
7.384
7.360
7.354
7.340
7.334
7.260
4.679
4.362
HO
O
P
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
4.03
8.15
1.97
2.00
0.82

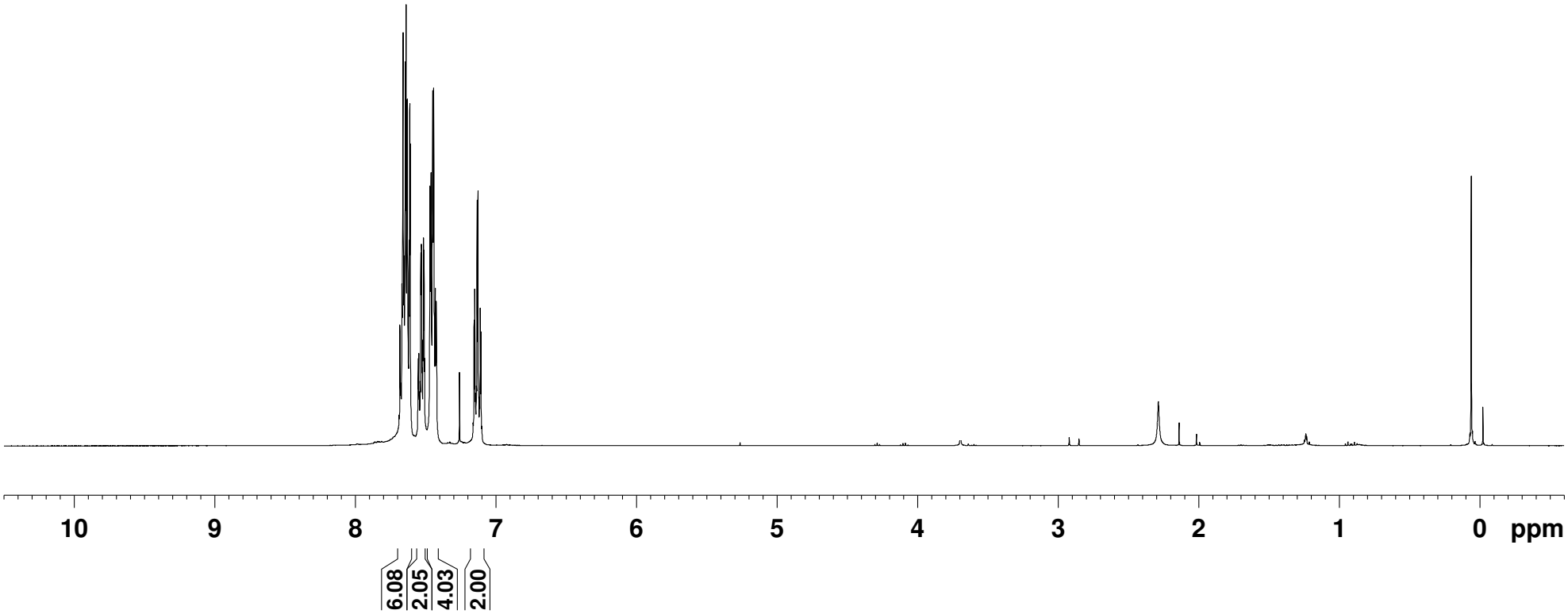

146.50
132.86
132.23
132.18
132.13
132.08
131.81
130.99
129.90
128.69
128.57
126.77
126.65
77.48
77.16
76.84
64.18
HO
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

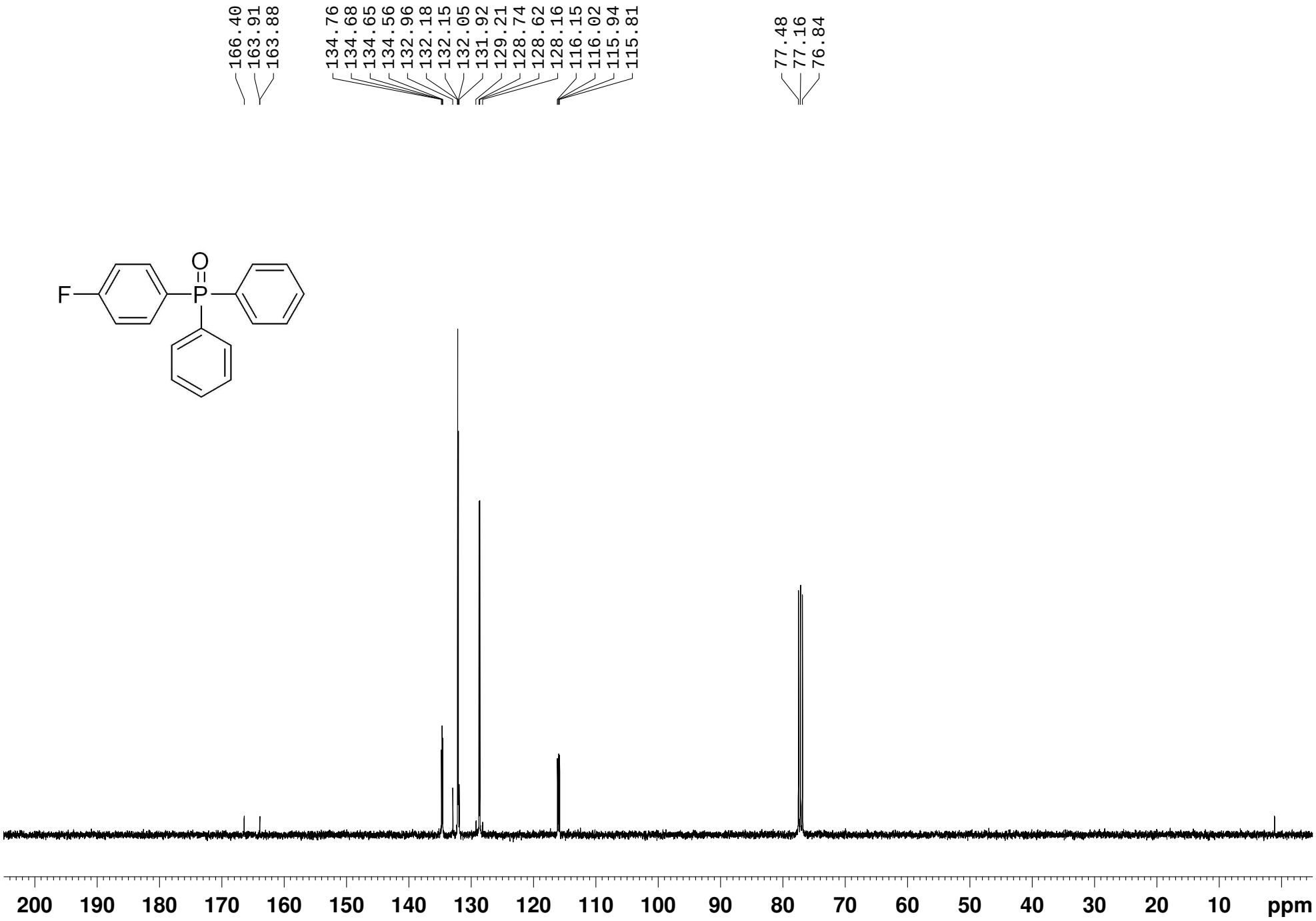


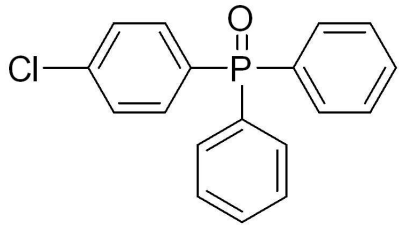
3i


7.635
7.632
7.625
7.619
7.614
7.611
7.557
7.553
7.550
7.546
7.541
7.535
7.531
7.526
7.520
7.516
7.513
7.509
7.470
7.466
7.462
7.459
7.454
7.451
7.447
7.443
7.440
7.437
7.433
7.430
7.426
7.423
7.260
7.163
7.156
7.151
7.146
7.139
7.135
7.129
7.125
7.118
7.113
7.107
7.101


F
O
P


10
9
8
7
6
5
4
3
2
1
0
ppm
6.08
2.05
4.03
2.00

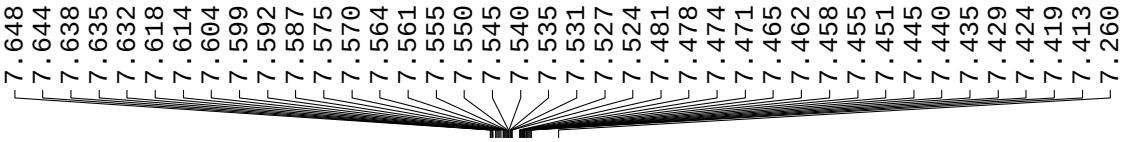

166.40
163.91
163.88
134.76
134.68
134.65
134.56
132.96
132.18
132.15
132.05
131.92
129.21
128.74
128.62
128.16
116.15
116.02
115.94
115.81
77.48
77.16
76.84
F
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

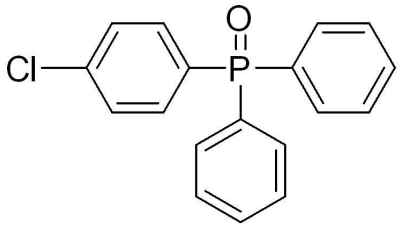


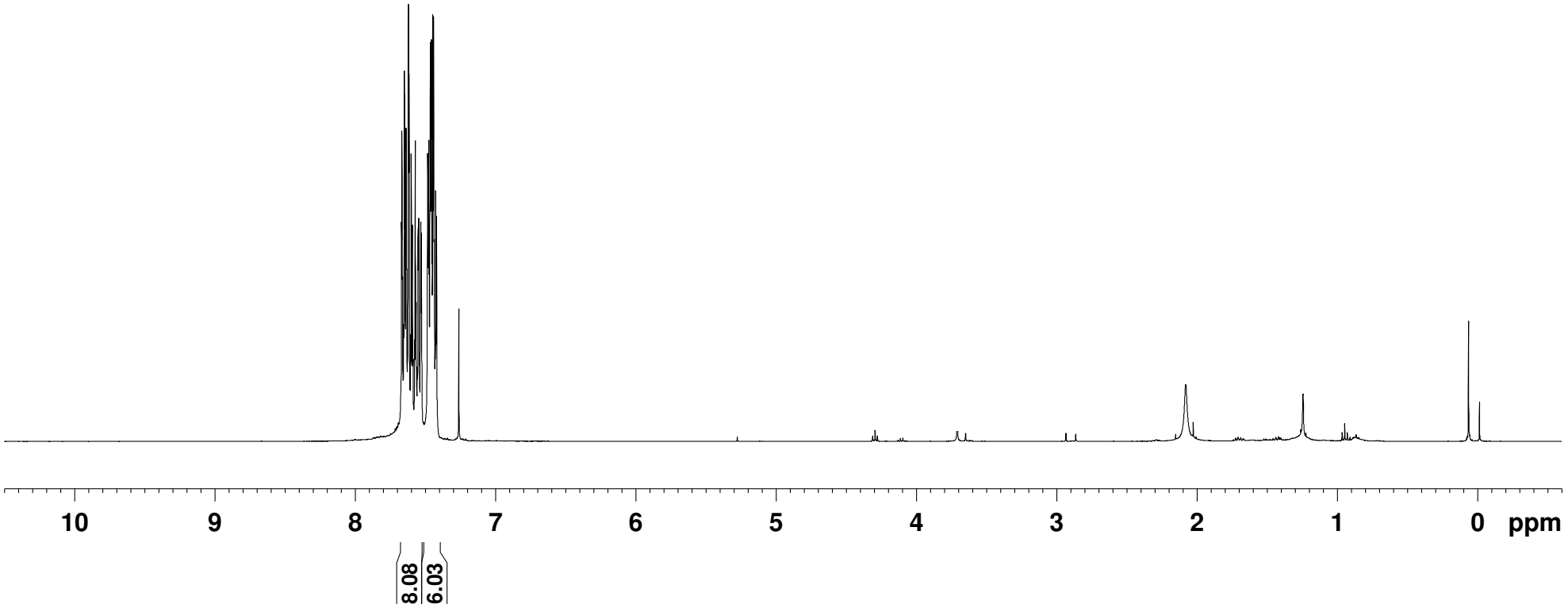
3j

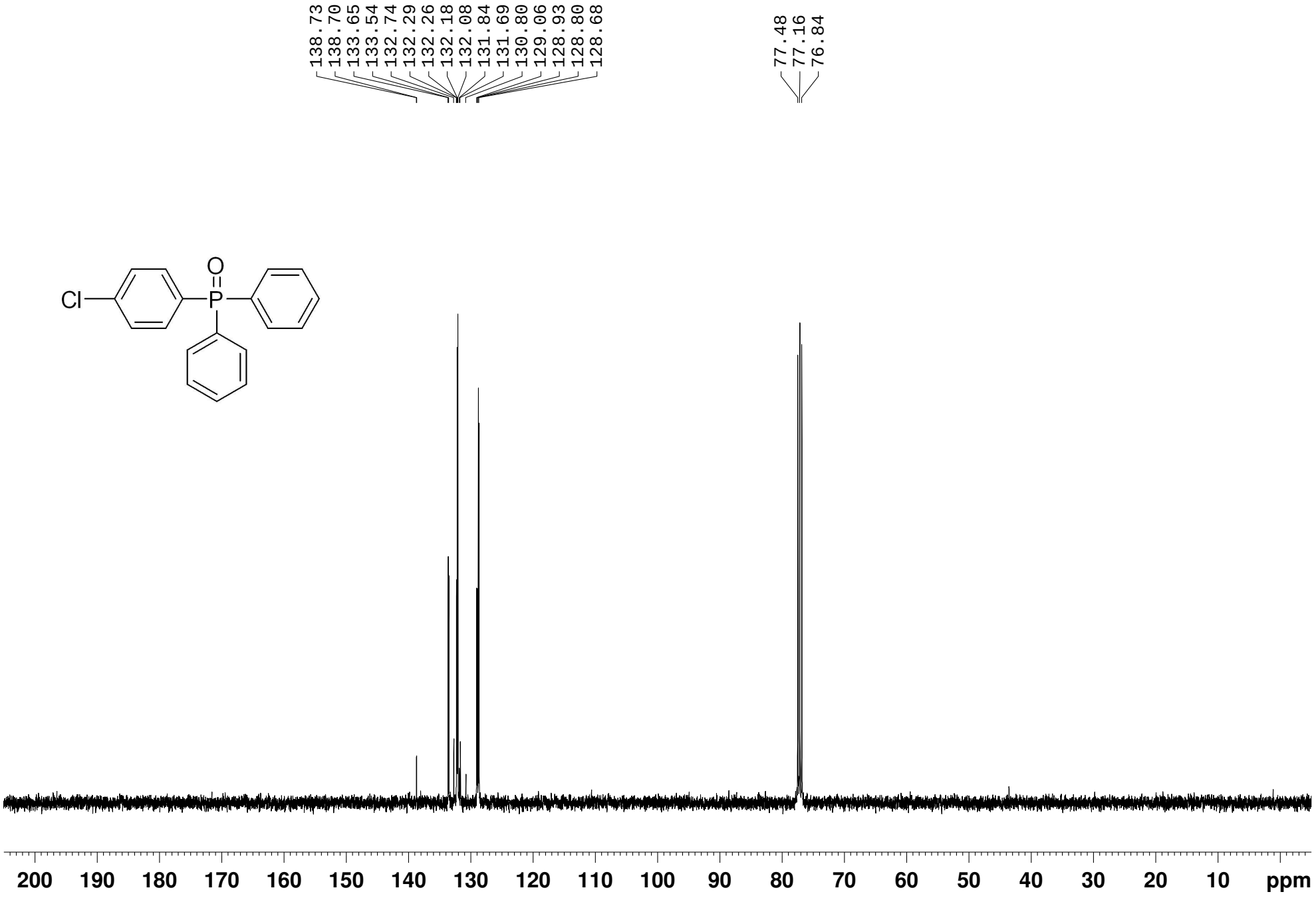
28.46

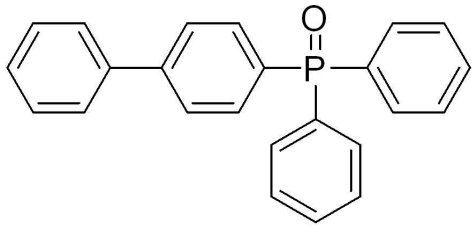
100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm


7.648
7.644
7.638
7.635
7.632
7.618
7.614
7.604
7.599
7.592
7.587
7.575
7.570
7.564
7.561
7.555
7.550
7.545
7.540
7.535
7.531
7.527
7.524
7.481
7.478
7.474
7.471
7.465
7.462
7.458
7.455
7.451
7.445
7.440
7.435
7.429
7.424
7.419
7.413
7.260

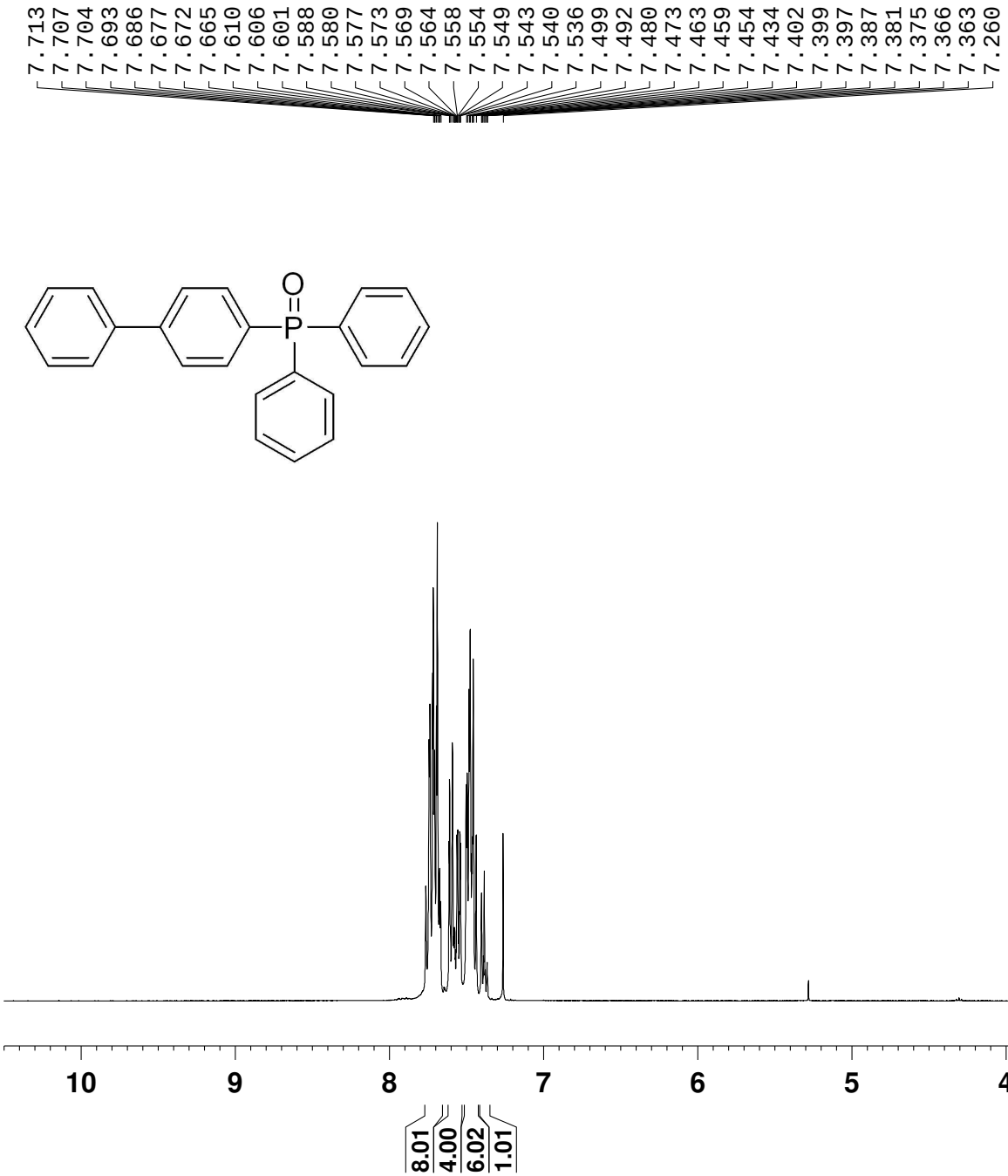

Cl
O
P

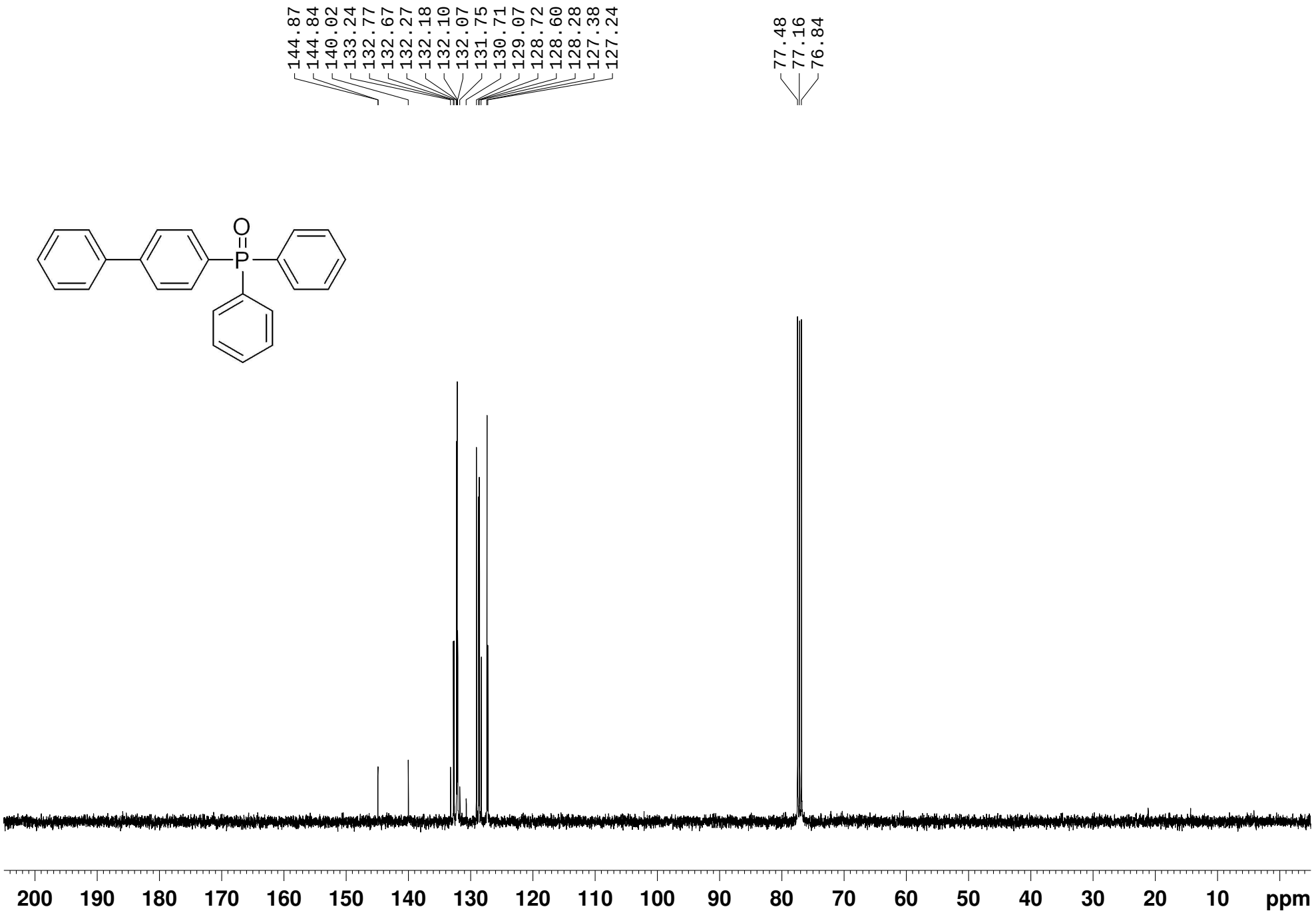

10
9
8
7
6
5
4
3
2
1
0
ppm
8.08
6.03

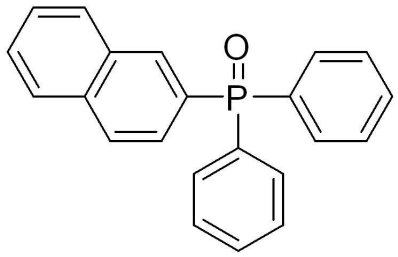

138.73
138.70
133.65
133.54
132.74
132.29
132.26
132.18
132.08
131.84
131.69
130.80
129.06
128.93
128.80
128.68
77.48
77.16
76.84
Cl
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



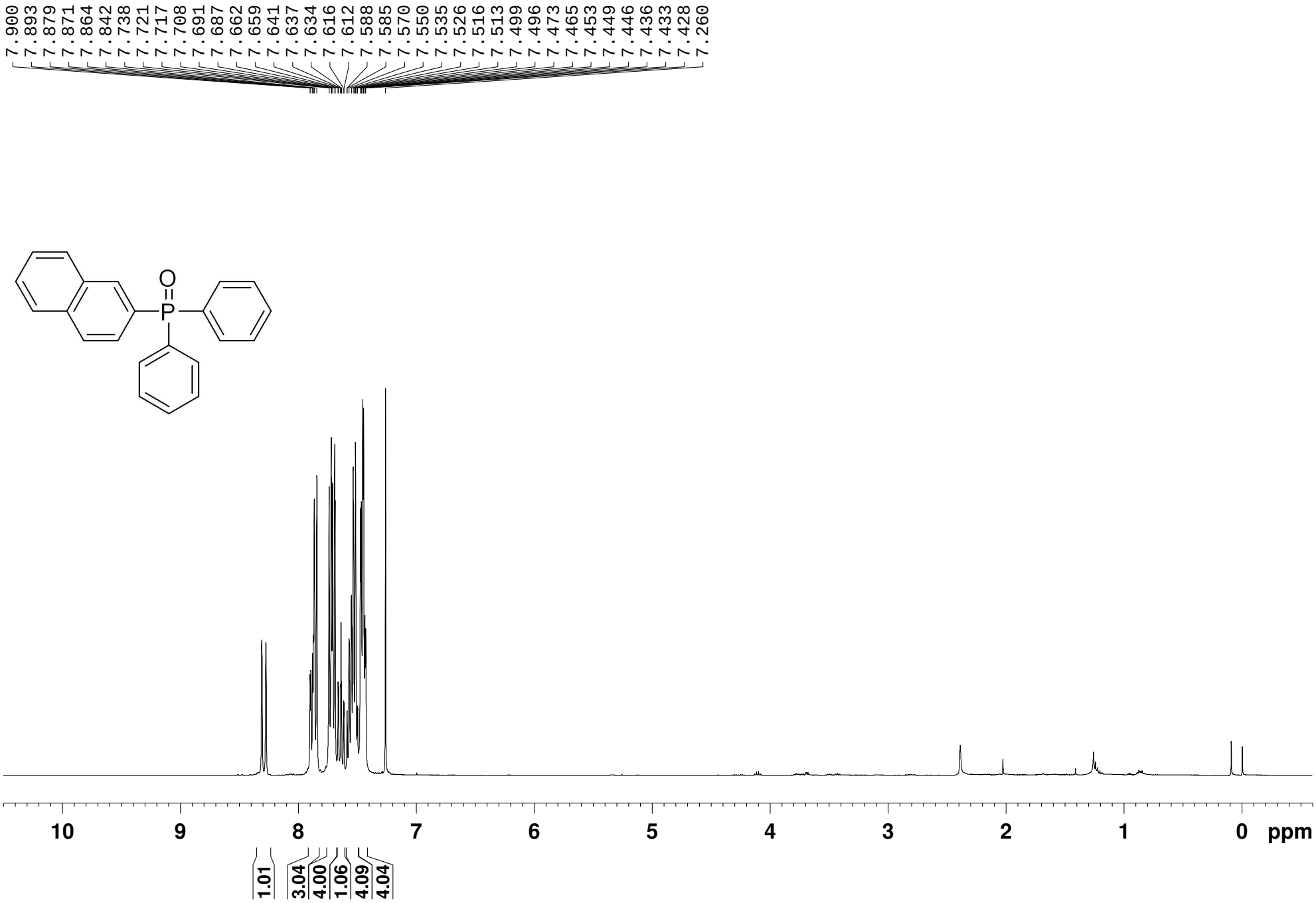
3k

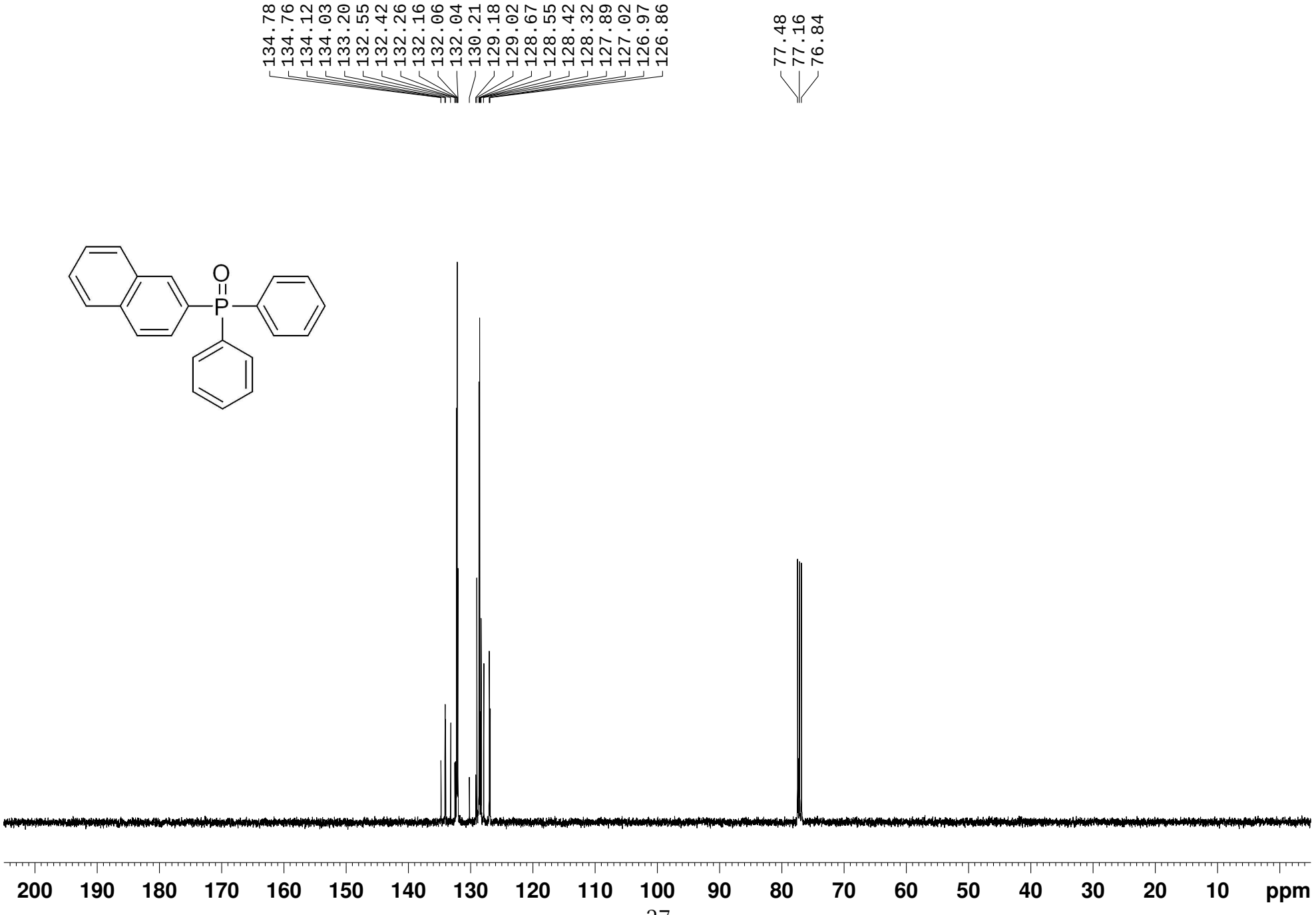

7.713
7.707
7.704
7.693
7.686
7.677
7.672
7.665
7.610
7.606
7.601
7.588
7.580
7.577
7.573
7.569
7.564
7.558
7.554
7.549
7.543
7.540
7.536
7.499
7.492
7.480
7.473
7.463
7.459
7.454
7.434
7.402
7.399
7.397
7.387
7.381
7.375
7.366
7.363
7.260
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
8.01
4.00
6.02
1.01

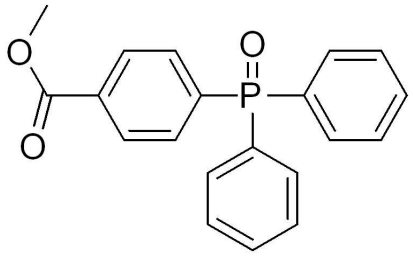

144.87
144.84
140.02
133.24
132.77
132.67
132.27
132.18
132.10
132.07
131.75
130.71
129.07
128.72
128.60
128.28
127.38
127.24
77.48
77.16
76.84
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



3l


7.900
7.893
7.879
7.871
7.864
7.842
7.738
7.721
7.717
7.708
7.691
7.687
7.662
7.659
7.641
7.637
7.634
7.616
7.612
7.588
7.585
7.570
7.550
7.535
7.526
7.516
7.513
7.499
7.496
7.473
7.465
7.453
7.449
7.446
7.436
7.433
7.428
7.260
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
3.04
4.00
1.06
4.09
4.04

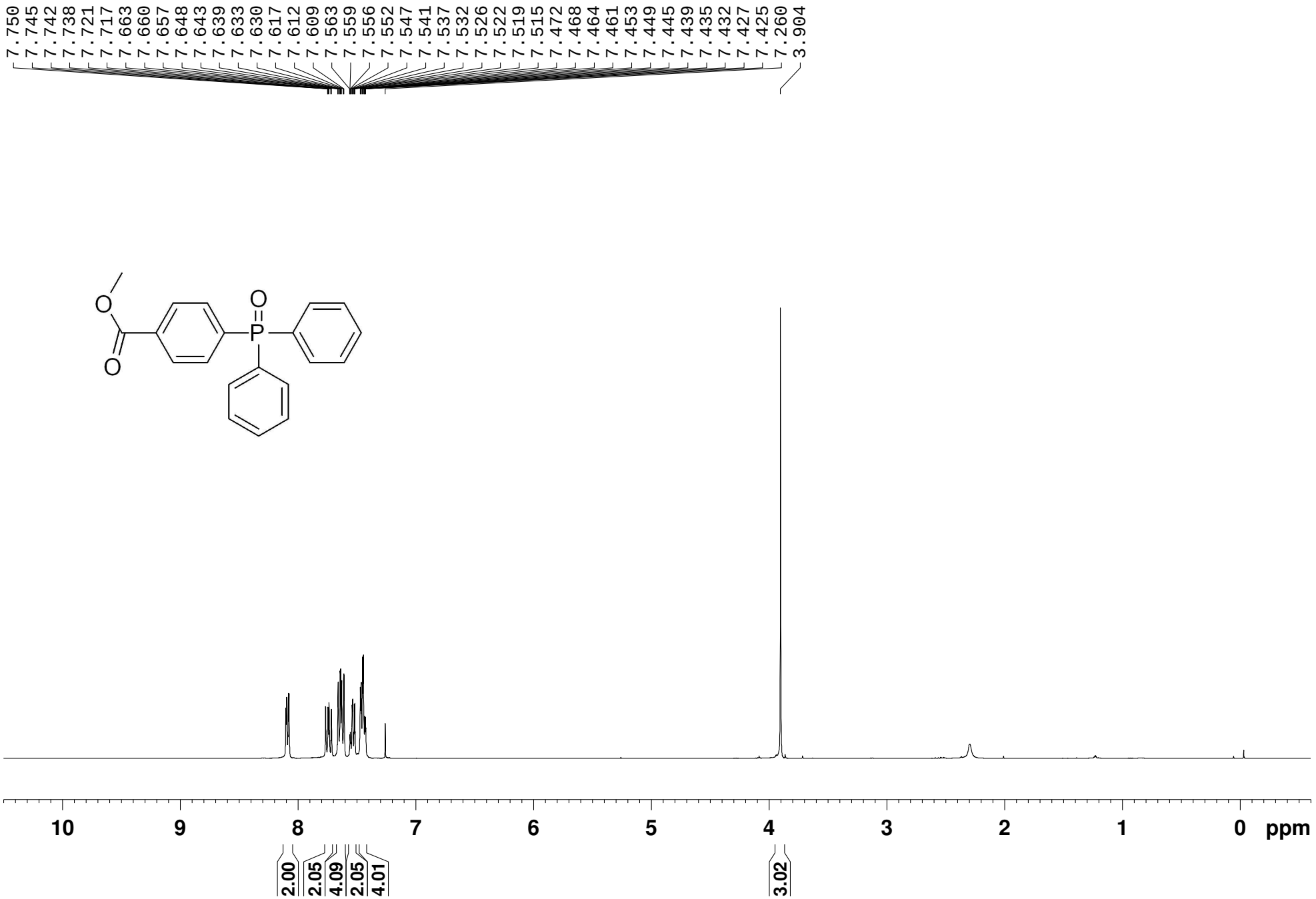

134.78
134.76
134.12
134.03
133.20
132.55
132.42
132.26
132.16
132.06
132.04
130.21
129.18
129.02
128.67
128.55
128.42
128.32
127.89
127.02
126.97
126.86
77.48
77.16
76.84
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

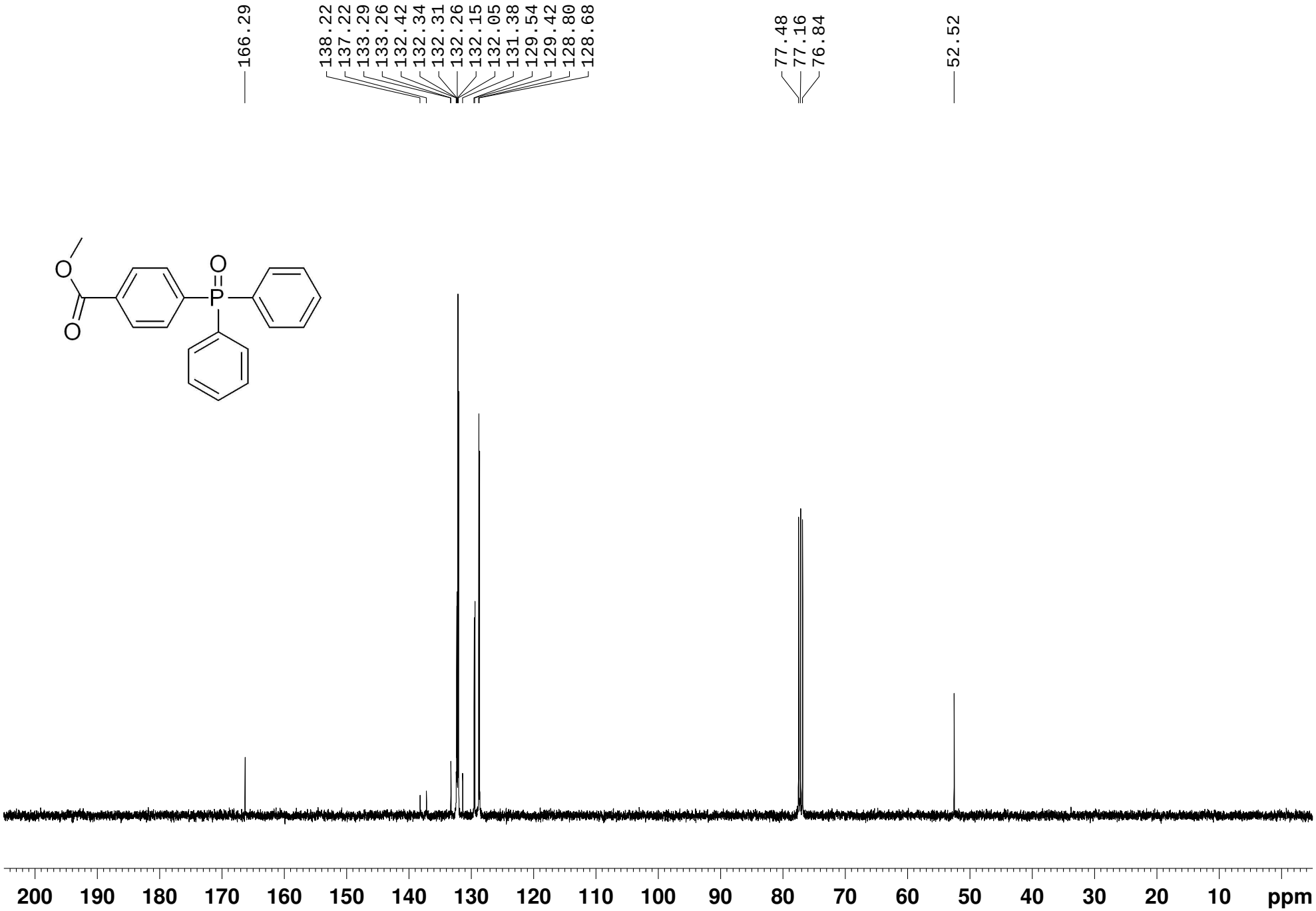


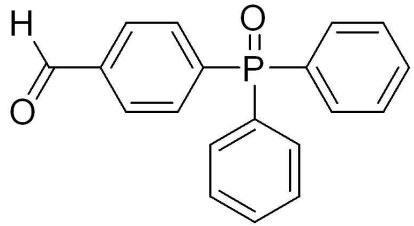
3m

28.50

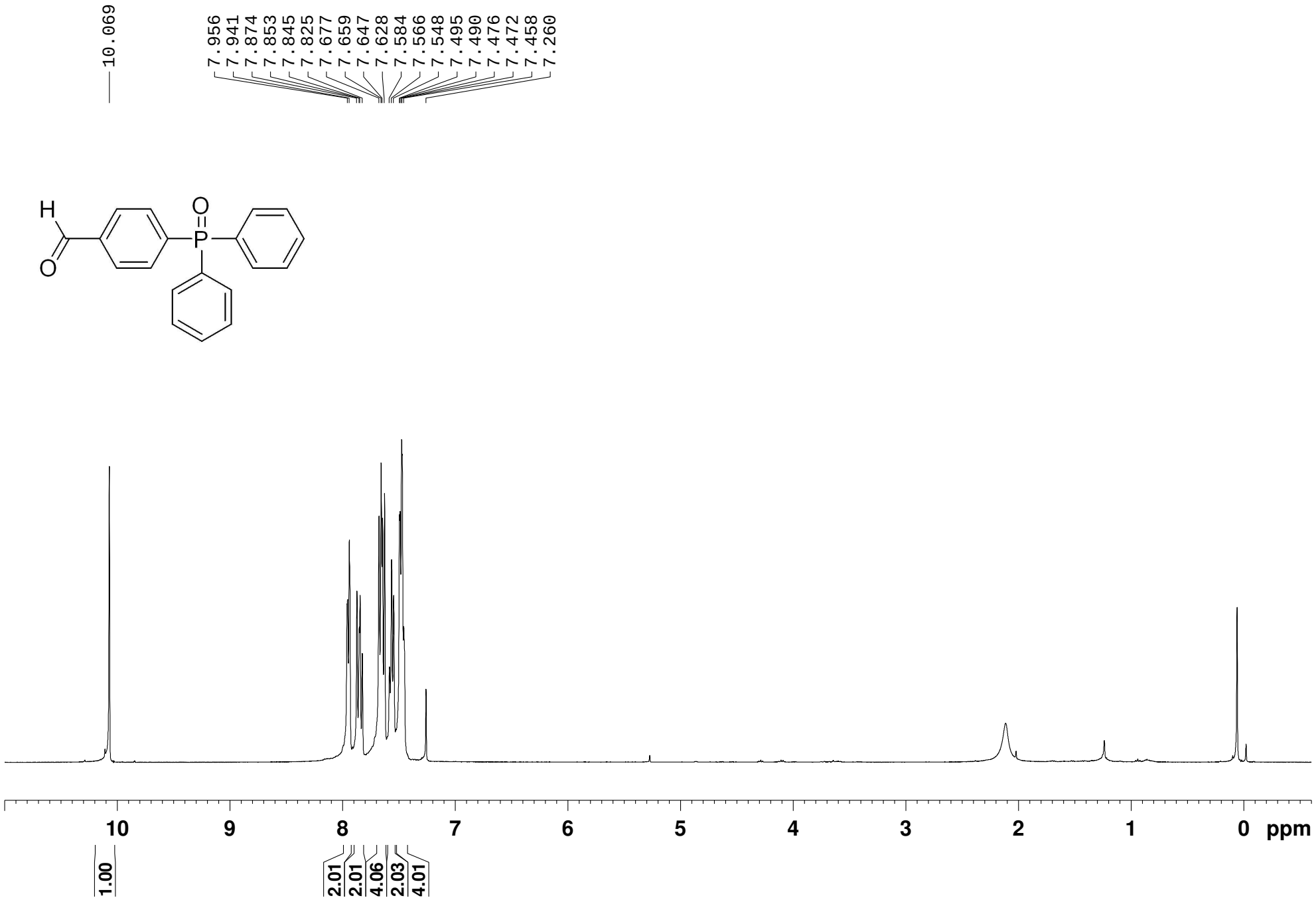
100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm

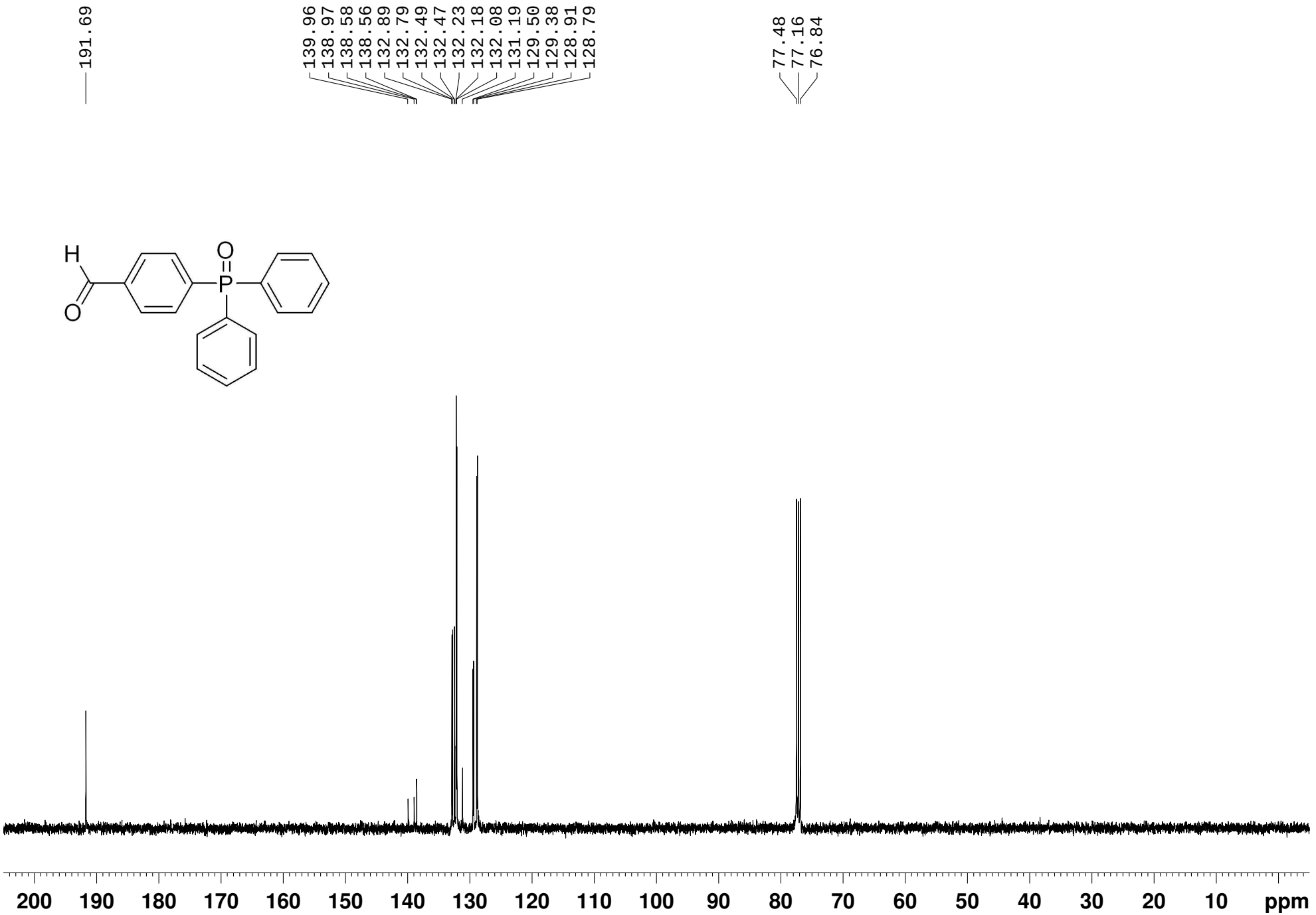

7.750
7.745
7.742
7.738
7.721
7.717
7.663
7.660
7.657
7.648
7.643
7.639
7.633
7.630
7.617
7.612
7.609
7.563
7.559
7.556
7.552
7.547
7.541
7.537
7.532
7.526
7.522
7.519
7.515
7.472
7.468
7.464
7.461
7.453
7.449
7.445
7.439
7.435
7.432
7.427
7.425
7.260
3.904
O
O
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.05
4.09
2.05
4.01
3.02

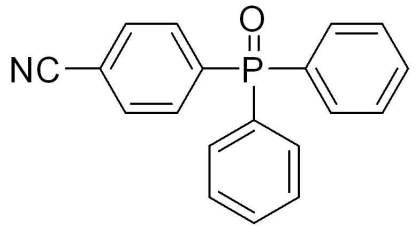

166.29
138.22
137.22
133.29
133.26
132.42
132.34
132.31
132.26
132.15
132.05
131.38
129.54
129.42
128.80
128.68
77.48
77.16
76.84
52.52
O
O
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



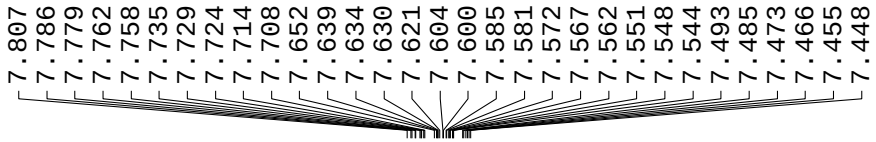
3n

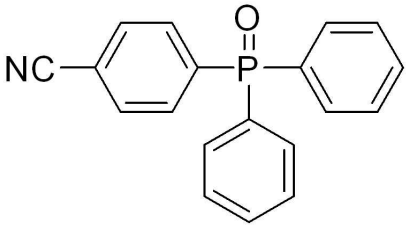

10.069
7.956
7.941
7.874
7.853
7.845
7.825
7.677
7.659
7.647
7.628
7.584
7.566
7.548
7.495
7.490
7.476
7.472
7.458
7.260
H
O
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.01
2.01
4.06
2.03
4.01

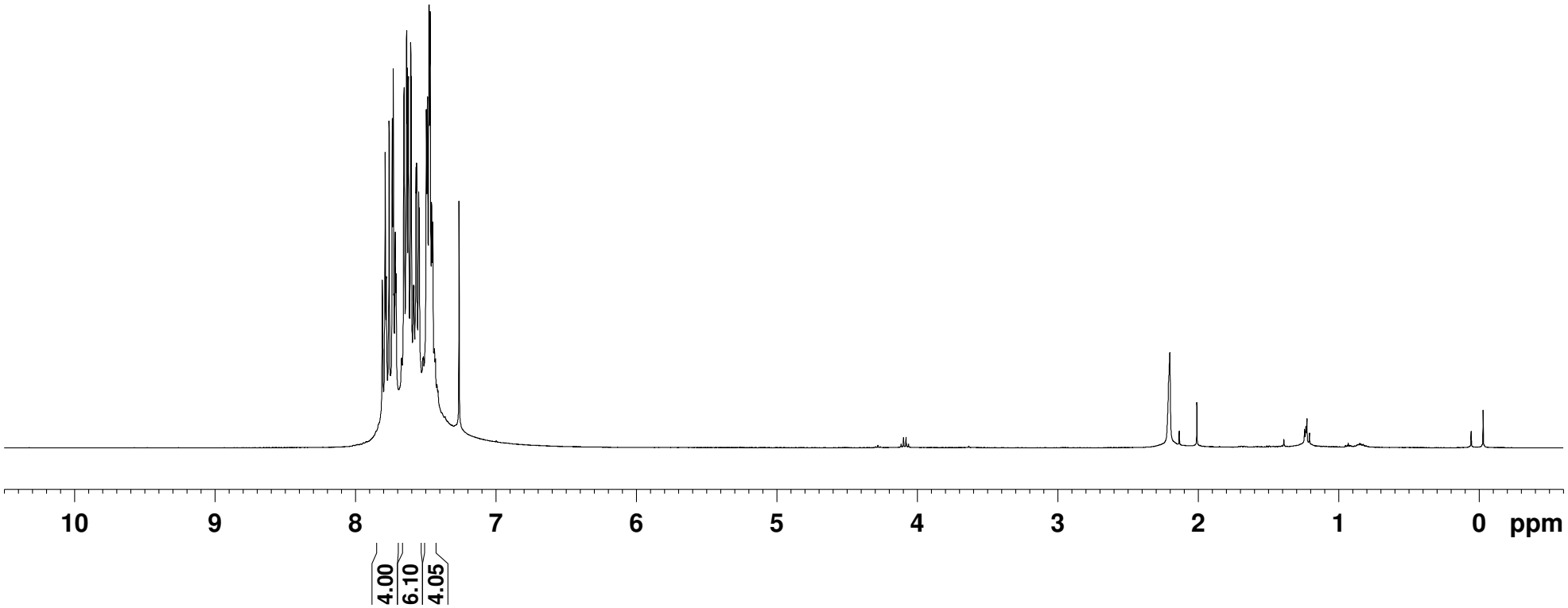

191.69
139.96
138.97
138.58
138.56
132.89
132.79
132.49
132.47
132.23
132.18
132.08
131.19
129.50
129.38
128.91
128.79
77.48
77.16
76.84
H
O
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

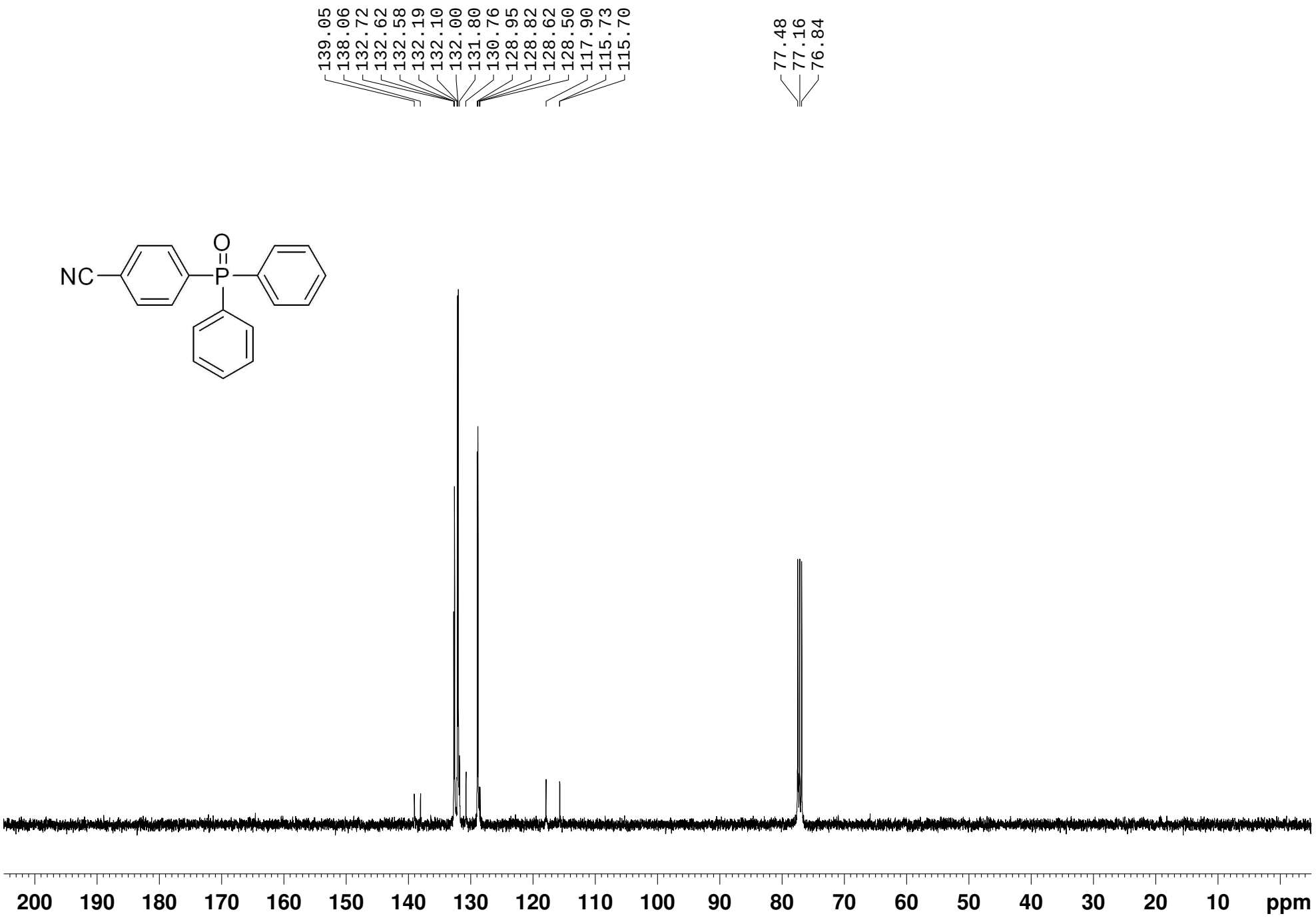


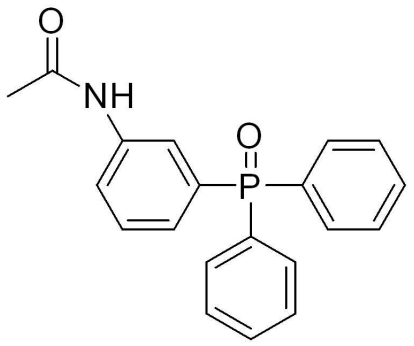
3o


7.807
7.786
7.779
7.762
7.758
7.735
7.729
7.724
7.714
7.708
7.652
7.639
7.634
7.630
7.621
7.604
7.600
7.585
7.581
7.572
7.567
7.562
7.551
7.548
7.544
7.493
7.485
7.473
7.466
7.455
7.448

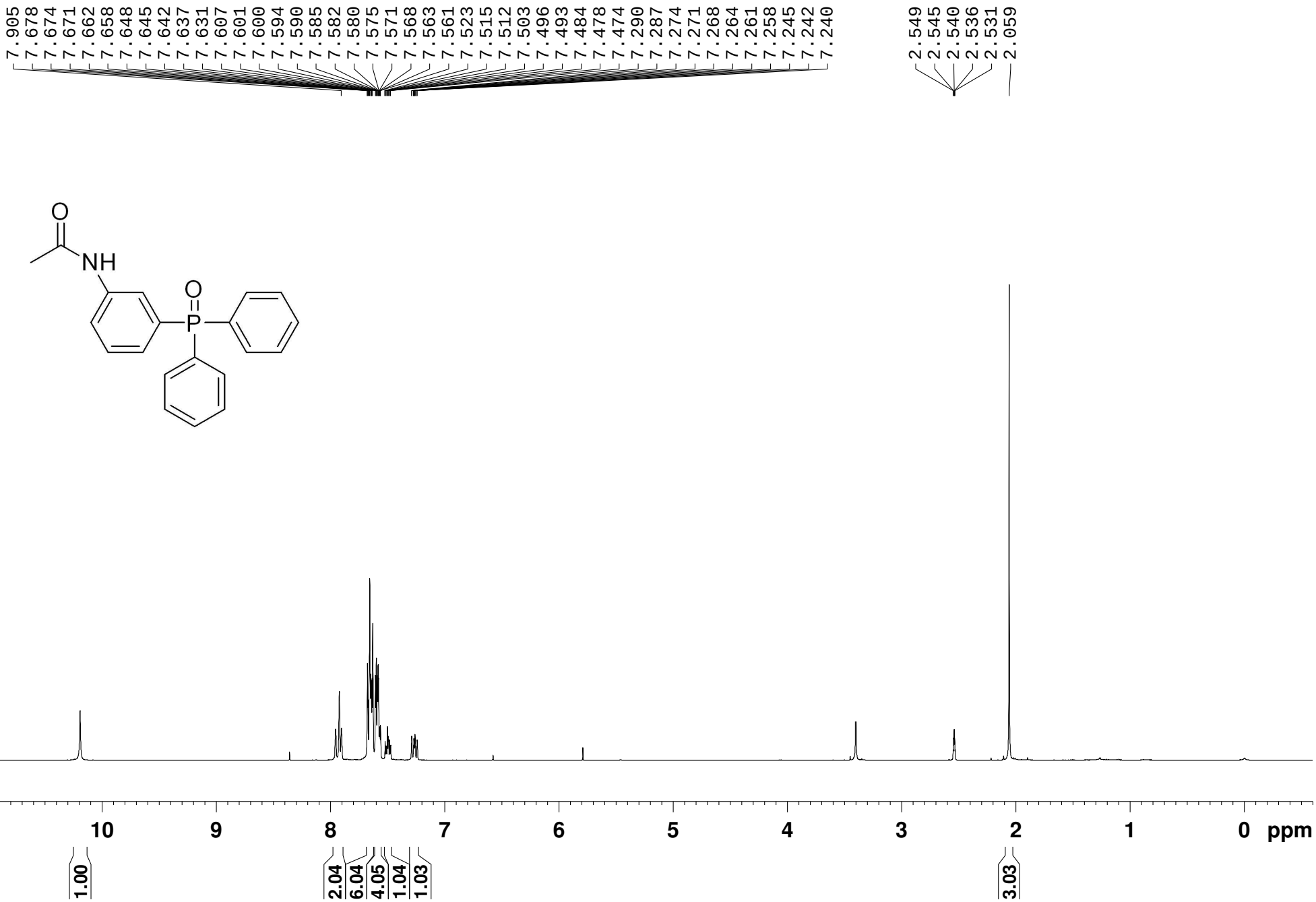

NC
O
P

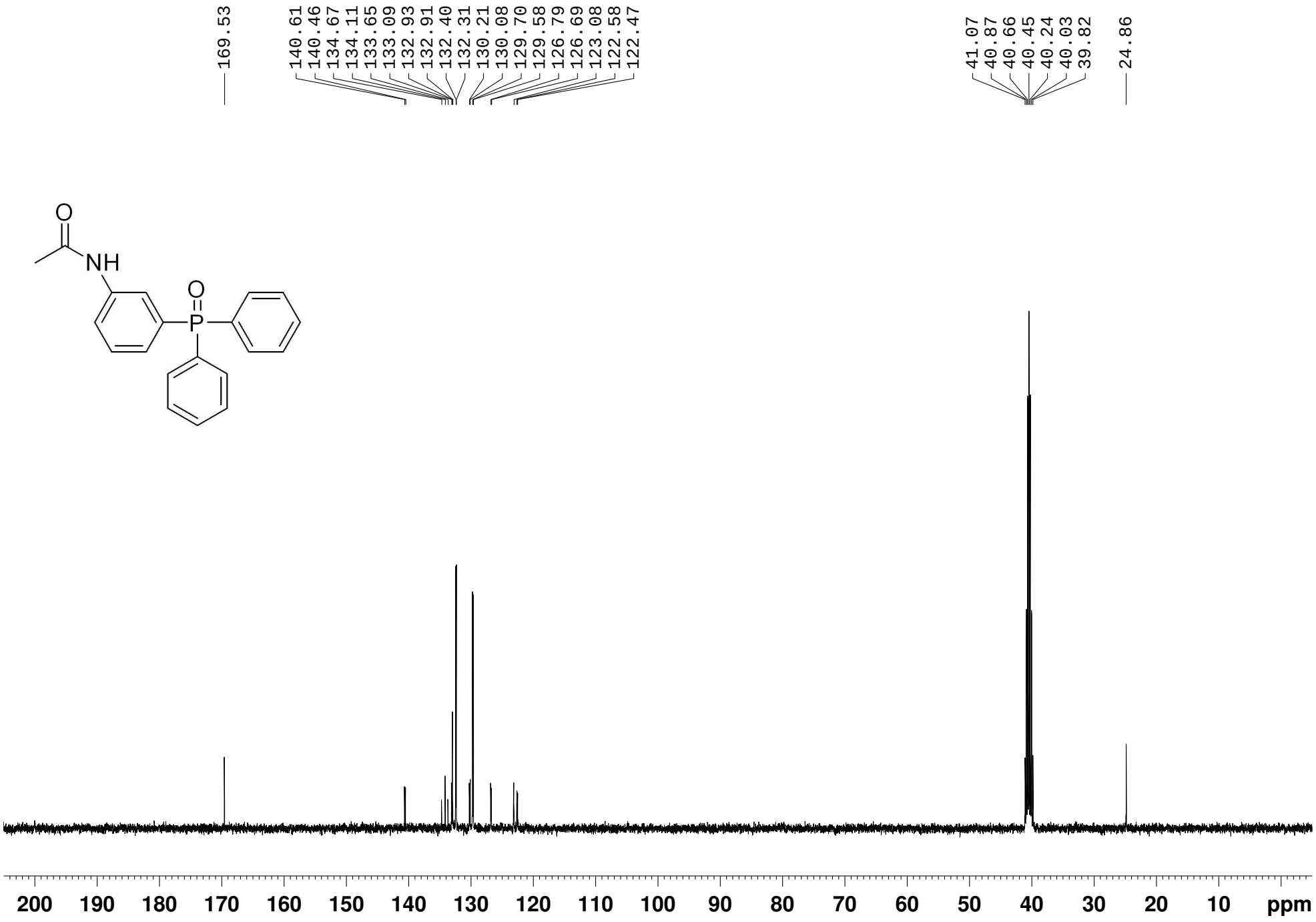

10
9
8
7
6
5
4
3
2
1
0
ppm
4.00
6.10
4.05

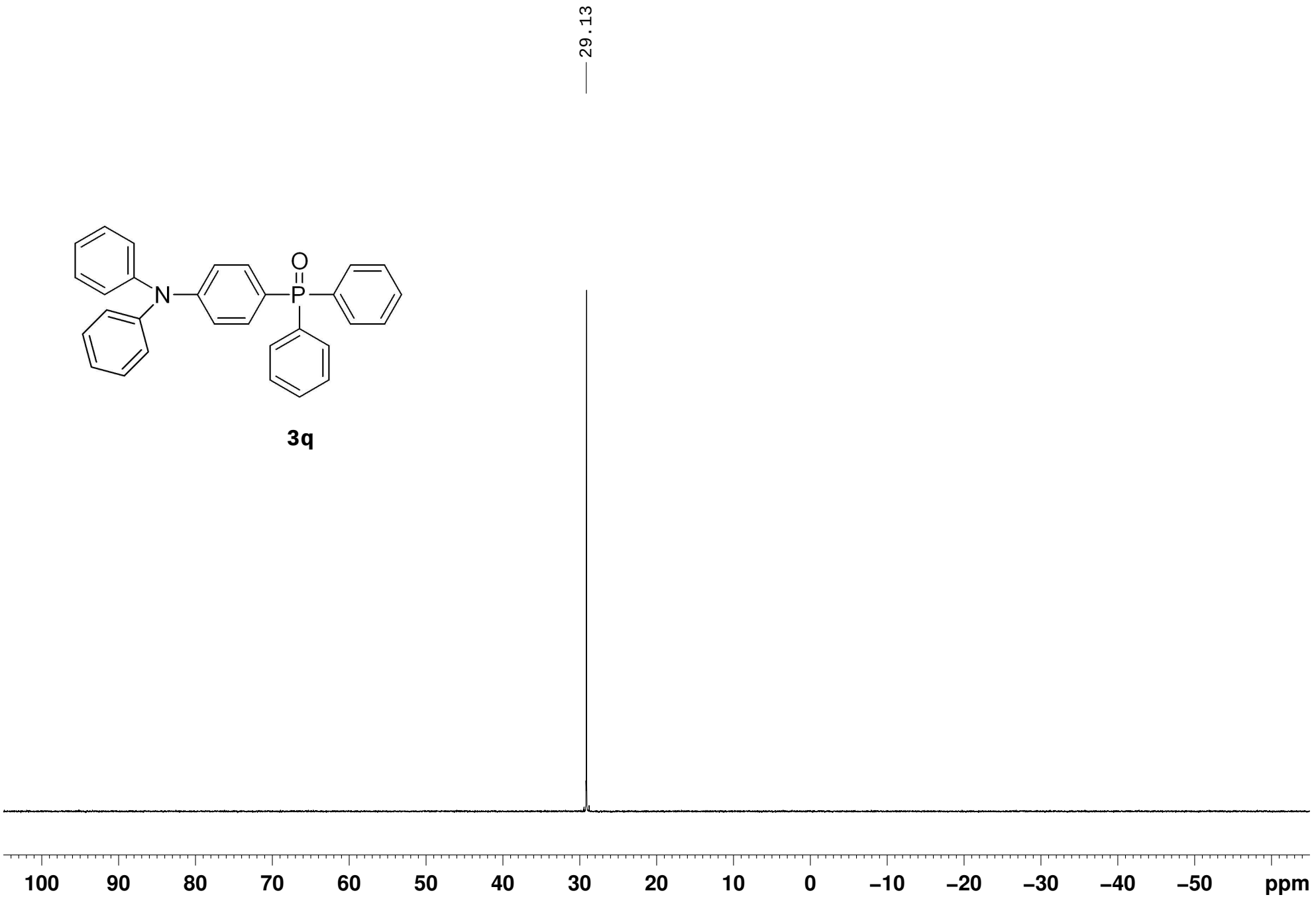

139.05
138.06
132.72
132.62
132.58
132.19
132.10
132.00
131.80
130.76
128.95
128.82
128.62
128.50
117.90
115.73
115.70
77.48
77.16
76.84
NC
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

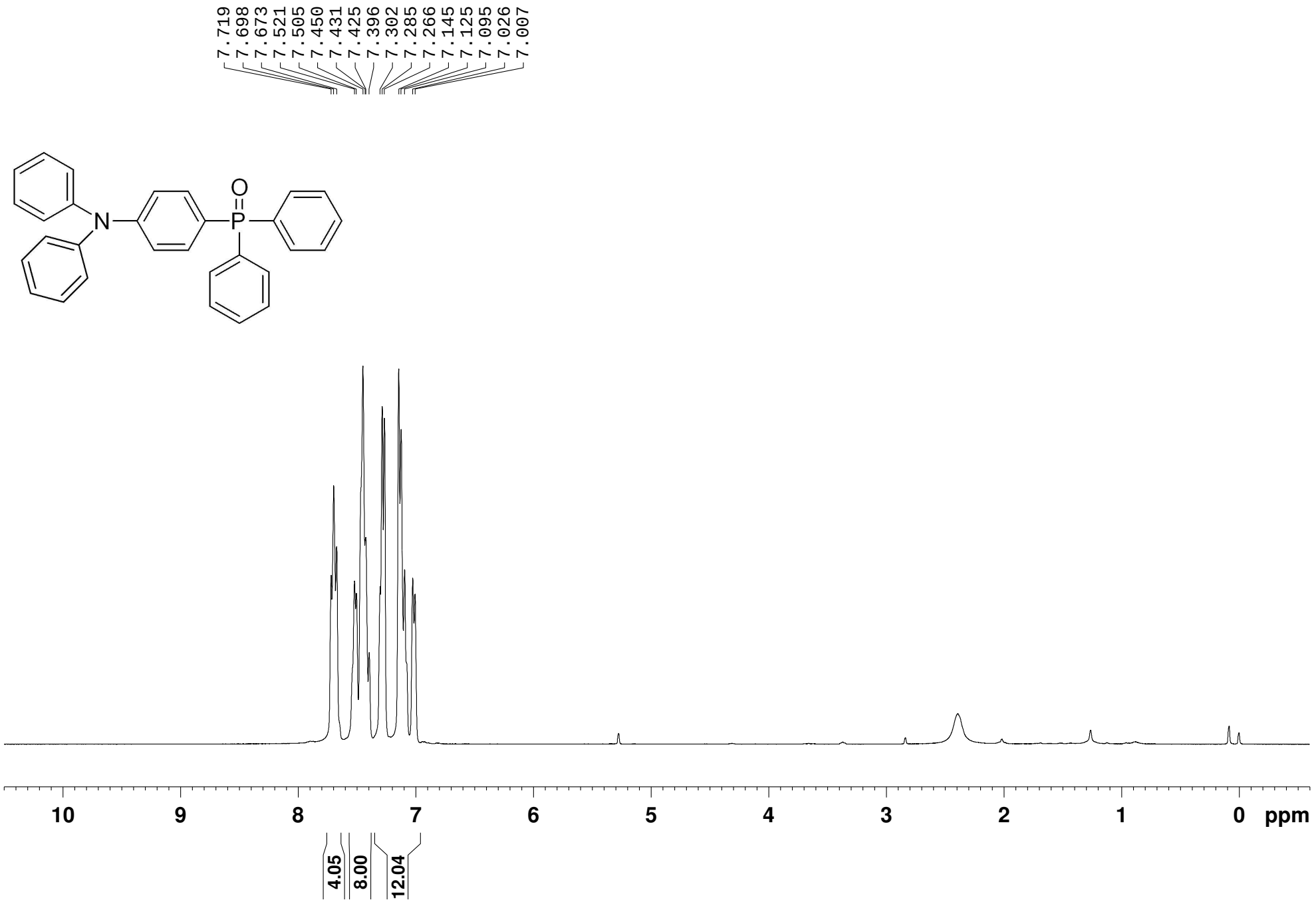


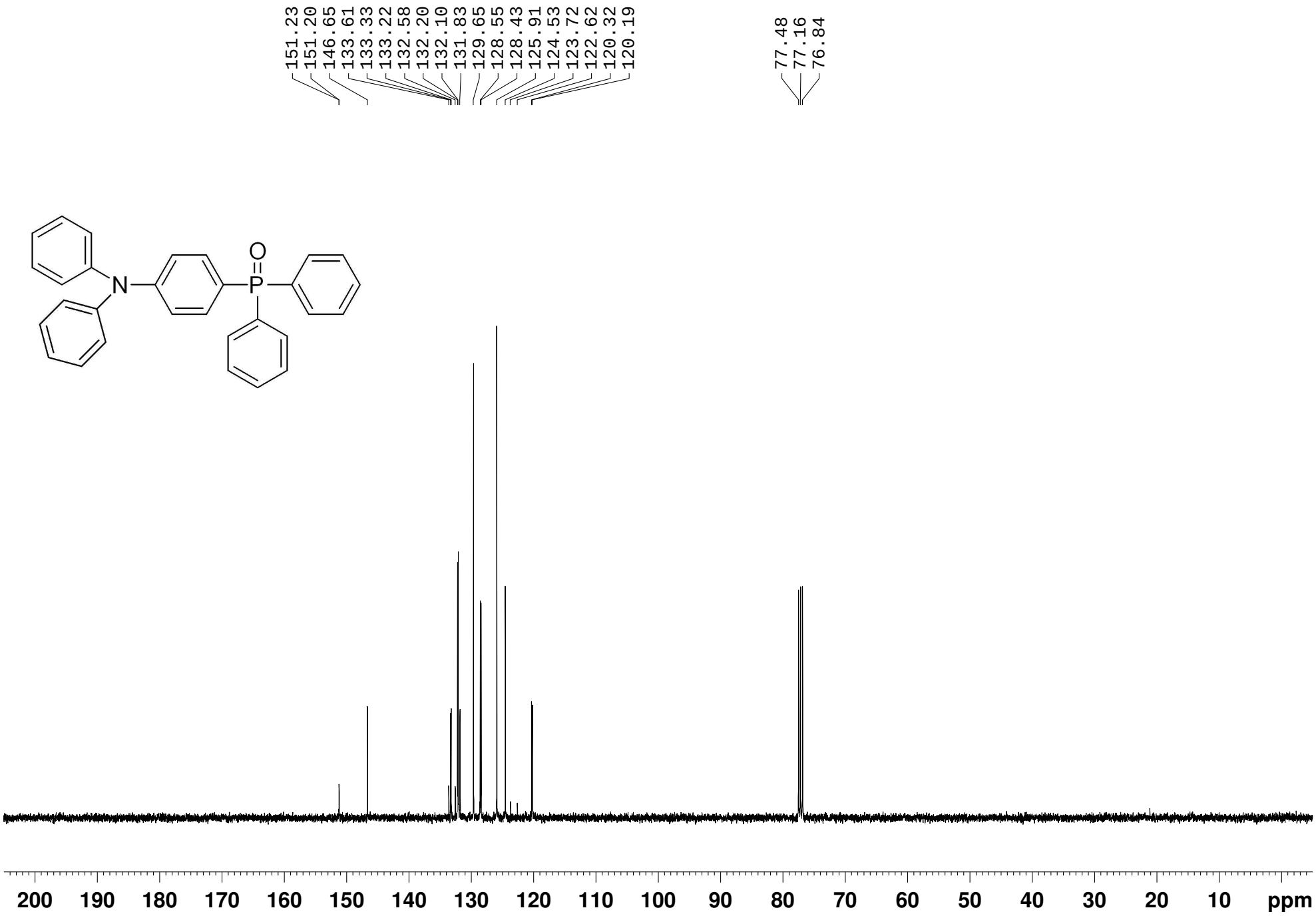
3p

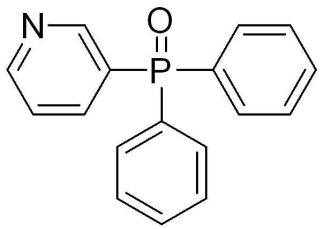

7.905
7.678
7.674
7.671
7.662
7.658
7.648
7.645
7.642
7.637
7.631
7.607
7.601
7.600
7.594
7.590
7.585
7.582
7.580
7.575
7.571
7.568
7.563
7.561
7.523
7.515
7.512
7.503
7.496
7.493
7.484
7.478
7.474
7.290
7.287
7.274
7.271
7.268
7.264
7.261
7.258
7.245
7.242
7.240
2.549
2.545
2.540
2.536
2.531
2.059
O
NH
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.04
6.04
4.05
1.04
1.03
3.03


169.53
140.61
140.46
134.67
134.11
133.65
133.09
132.93
132.91
132.40
132.31
130.21
130.08
129.70
129.58
126.79
126.69
123.08
122.58
122.47
41.07
40.87
40.66
40.45
40.24
40.03
39.82
24.86
O
NH
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

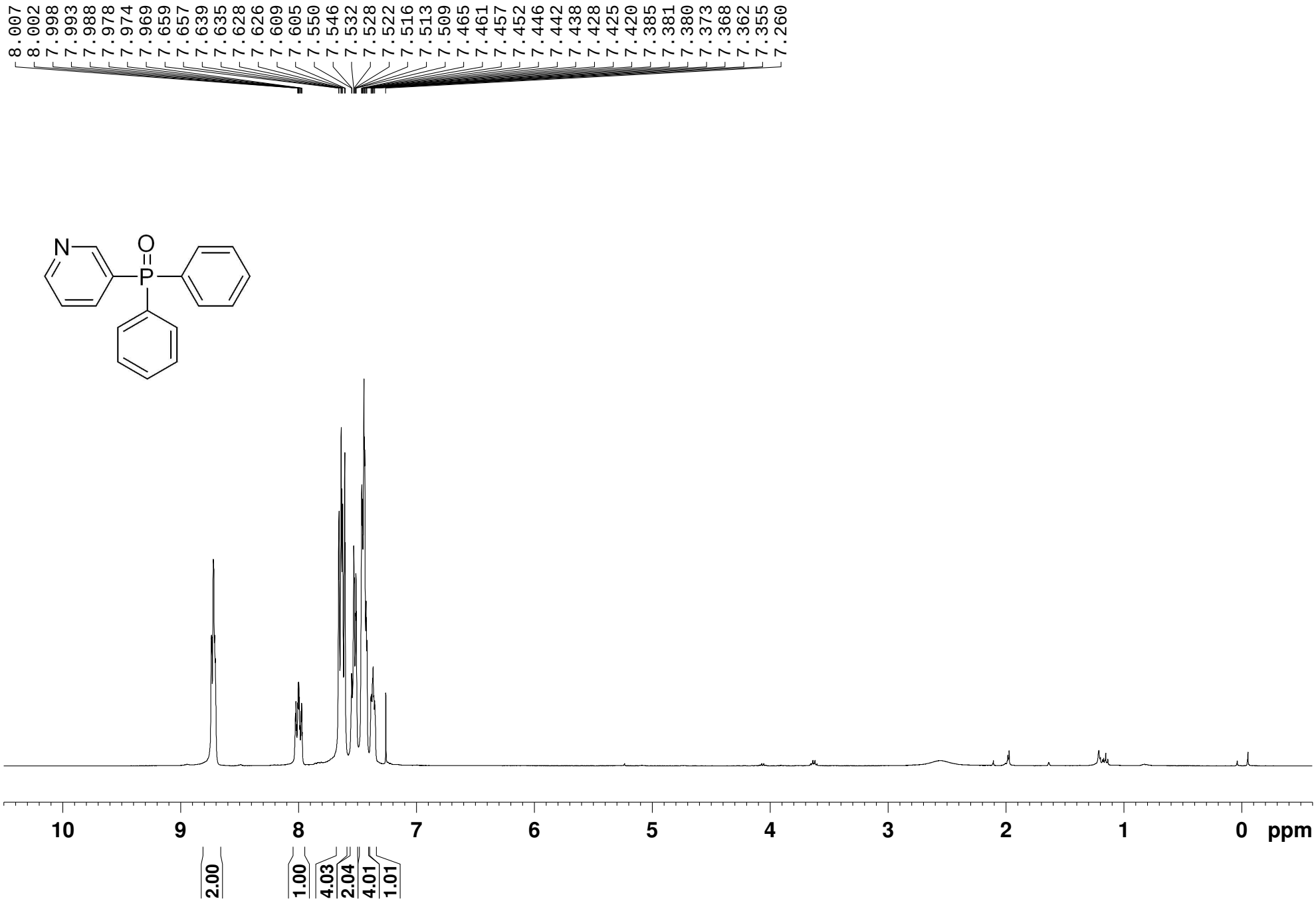

29.13
O
N
P
3q
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
−40
−50
ppm

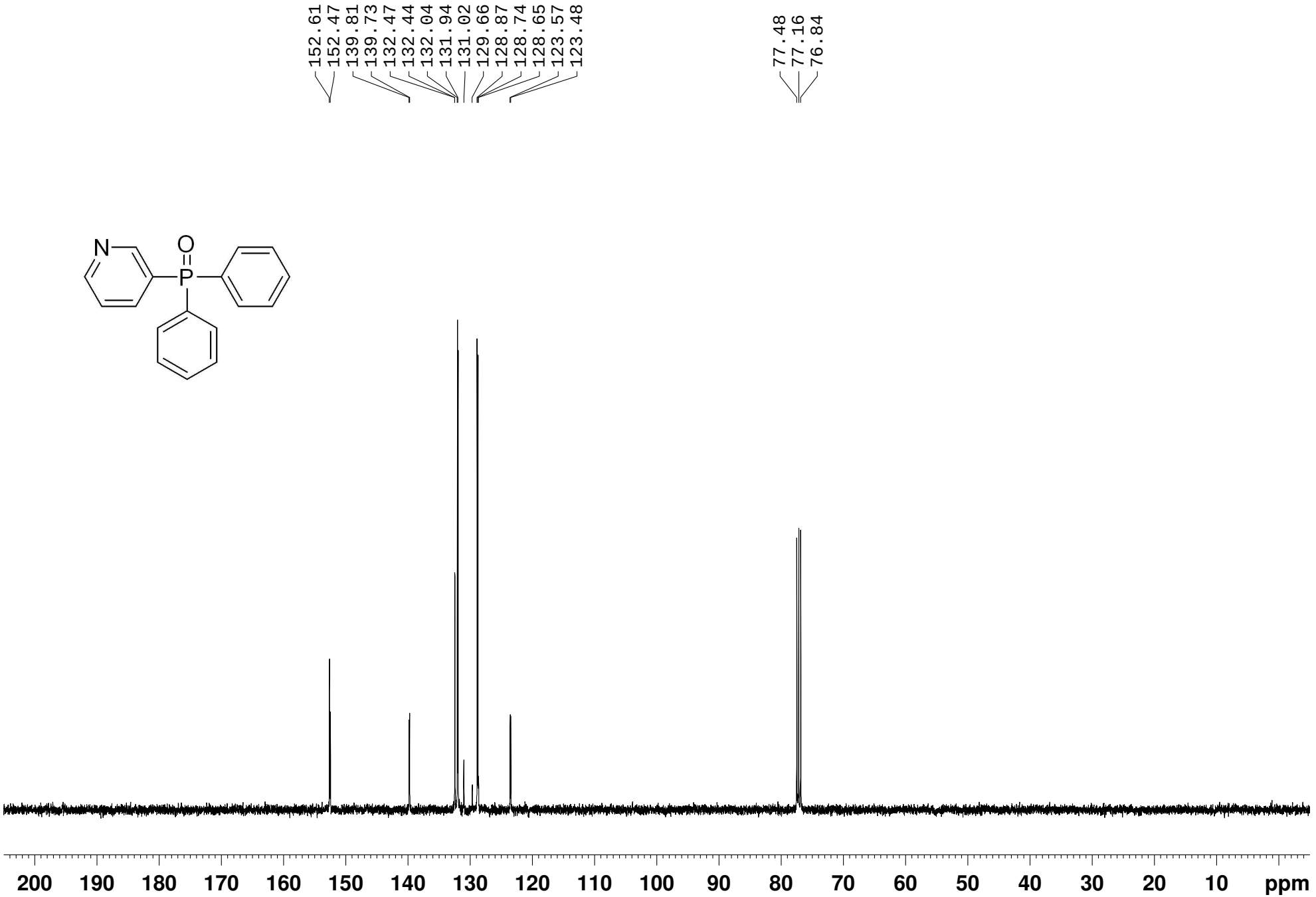

7.719
7.698
7.673
7.521
7.505
7.450
7.431
7.425
7.396
7.302
7.285
7.266
7.145
7.125
7.095
7.026
7.007
O
P
N
10
9
8
7
6
5
4
3
2
1
0
ppm
4.05
8.00
12.04

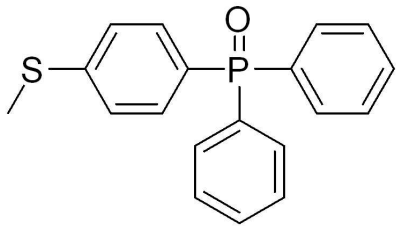

151.23
151.20
146.65
133.61
133.33
133.22
132.58
132.20
132.10
131.83
129.65
128.55
128.43
125.91
124.53
123.72
122.62
120.32
120.19
77.48
77.16
76.84
O
P
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



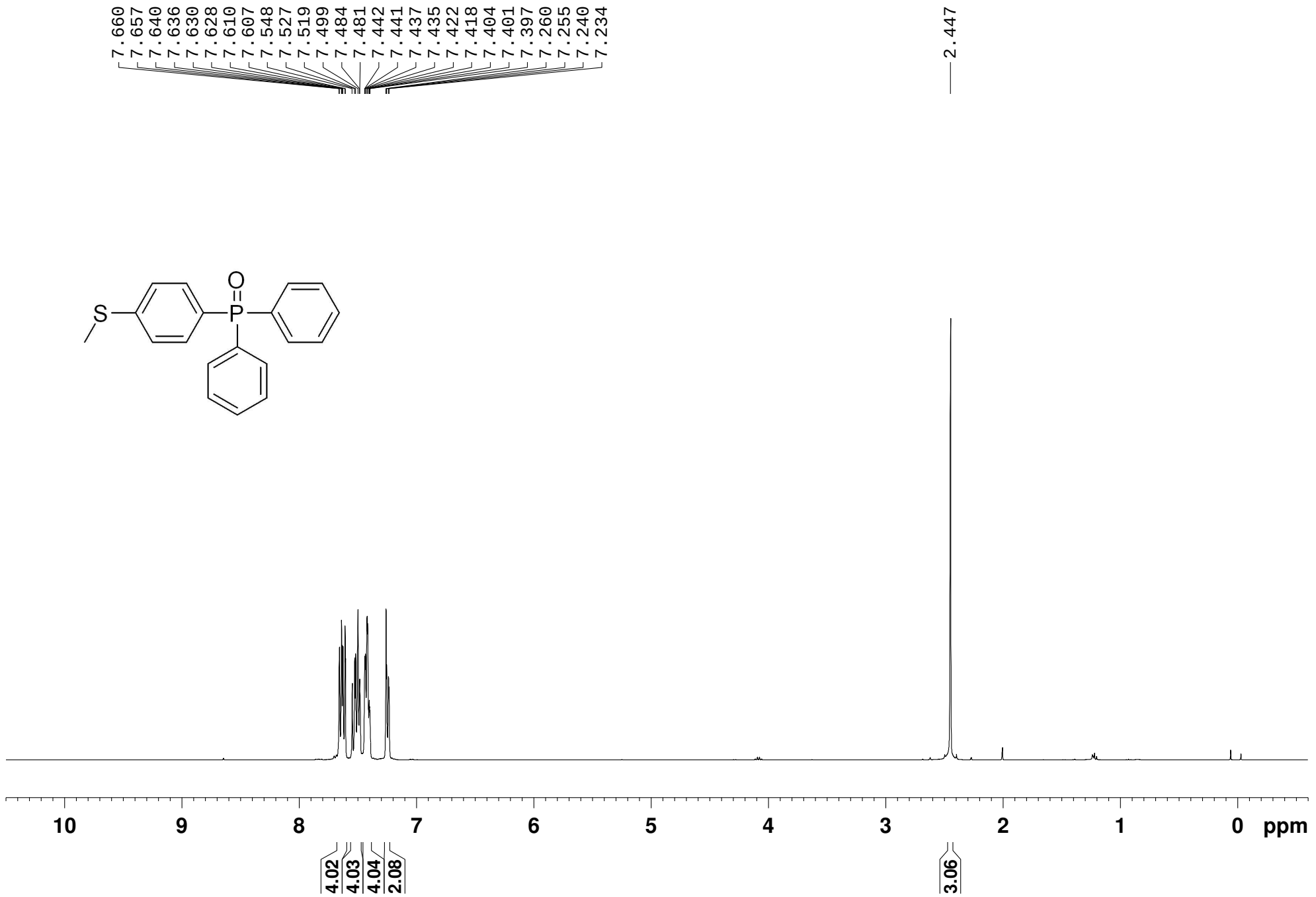
3r

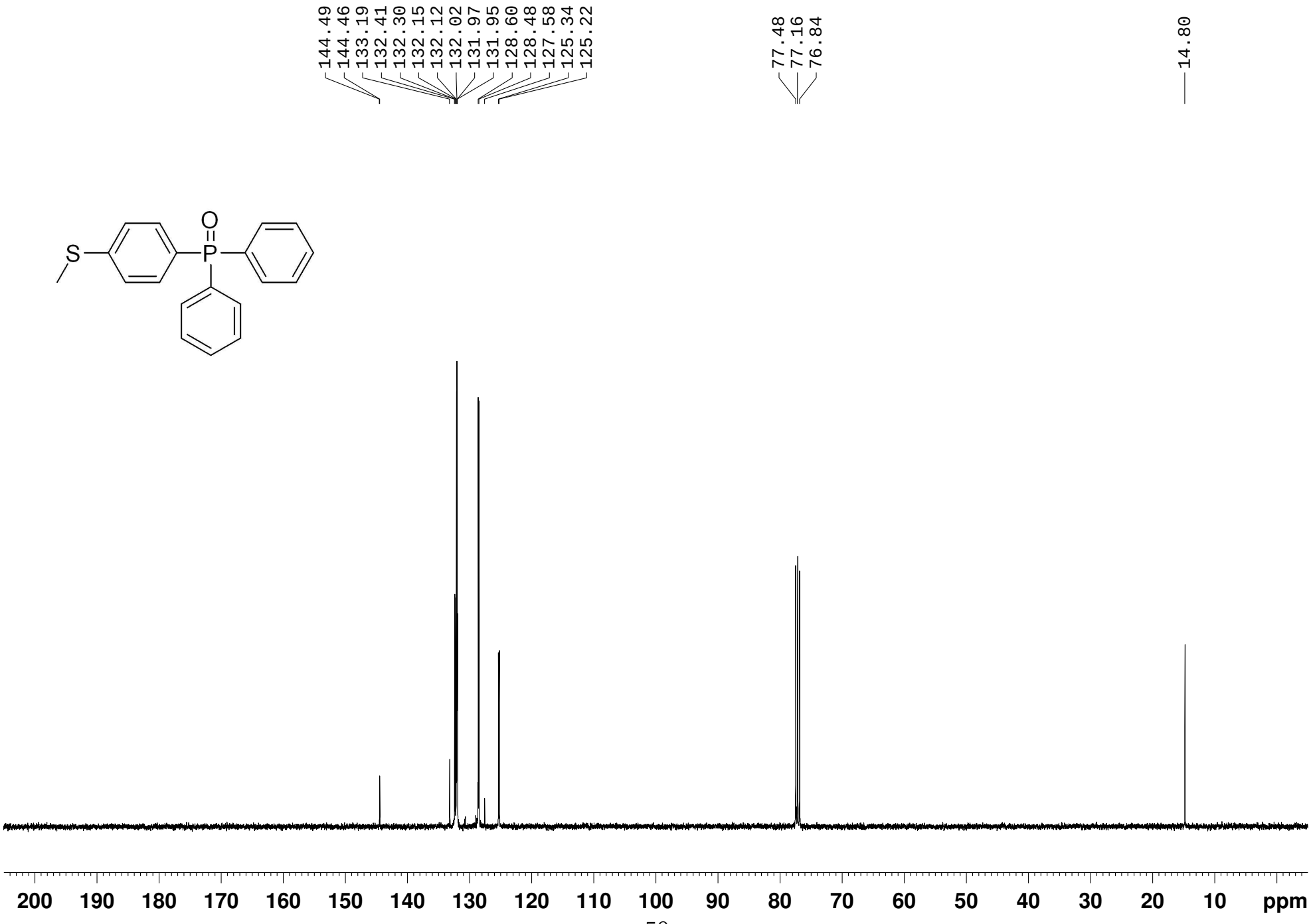

8.007
8.002
7.998
7.993
7.988
7.978
7.974
7.969
7.659
7.657
7.639
7.635
7.628
7.626
7.609
7.605
7.550
7.546
7.532
7.528
7.522
7.516
7.513
7.509
7.465
7.461
7.457
7.452
7.446
7.442
7.438
7.428
7.425
7.420
7.385
7.381
7.380
7.373
7.368
7.362
7.355
7.260
N
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
1.00
4.03
2.04
4.01
1.01

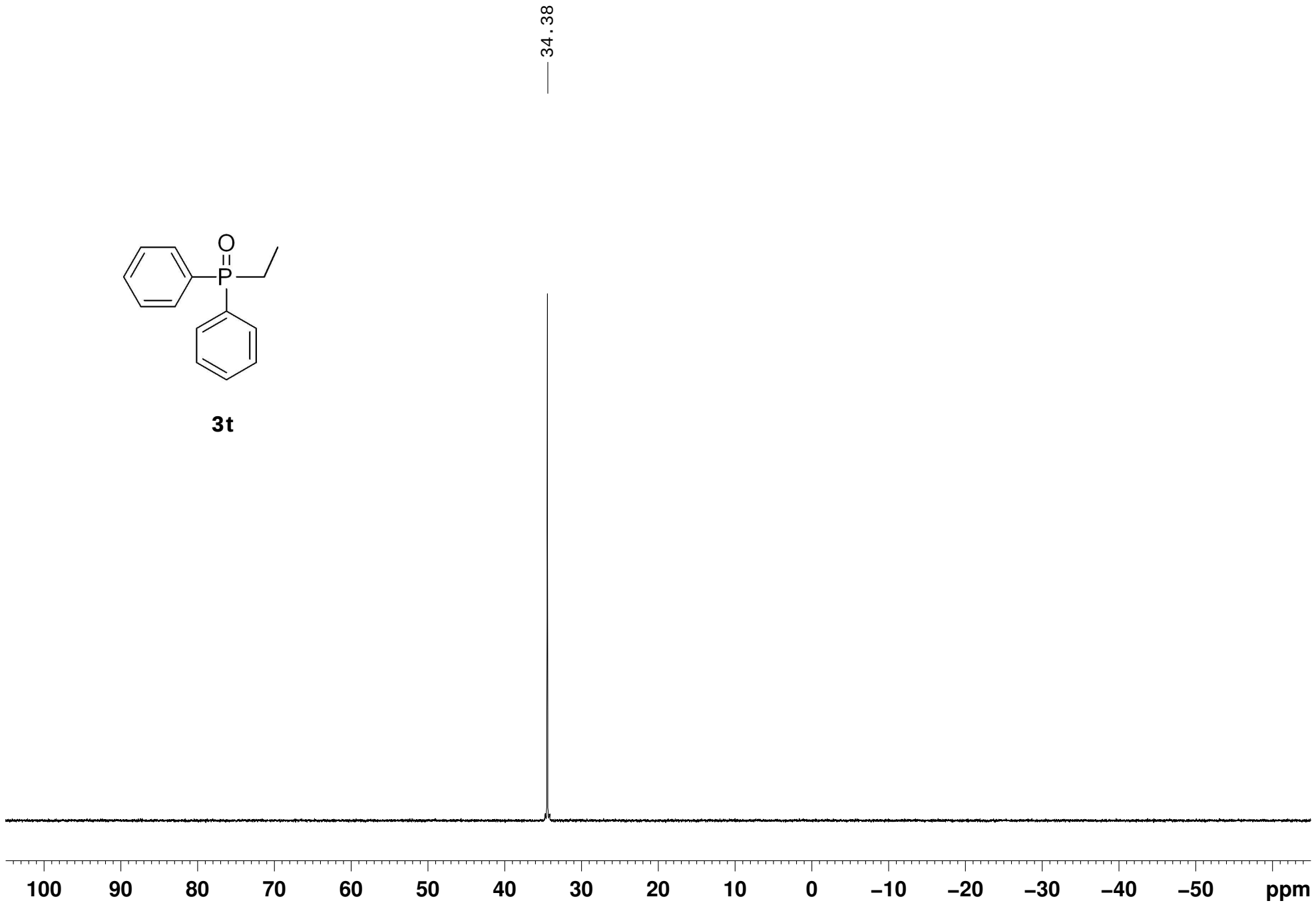

152.61
152.47
139.81
139.73
132.47
132.44
132.04
131.94
131.02
129.66
128.87
128.74
128.65
123.57
123.48
77.48
77.16
76.84
N
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



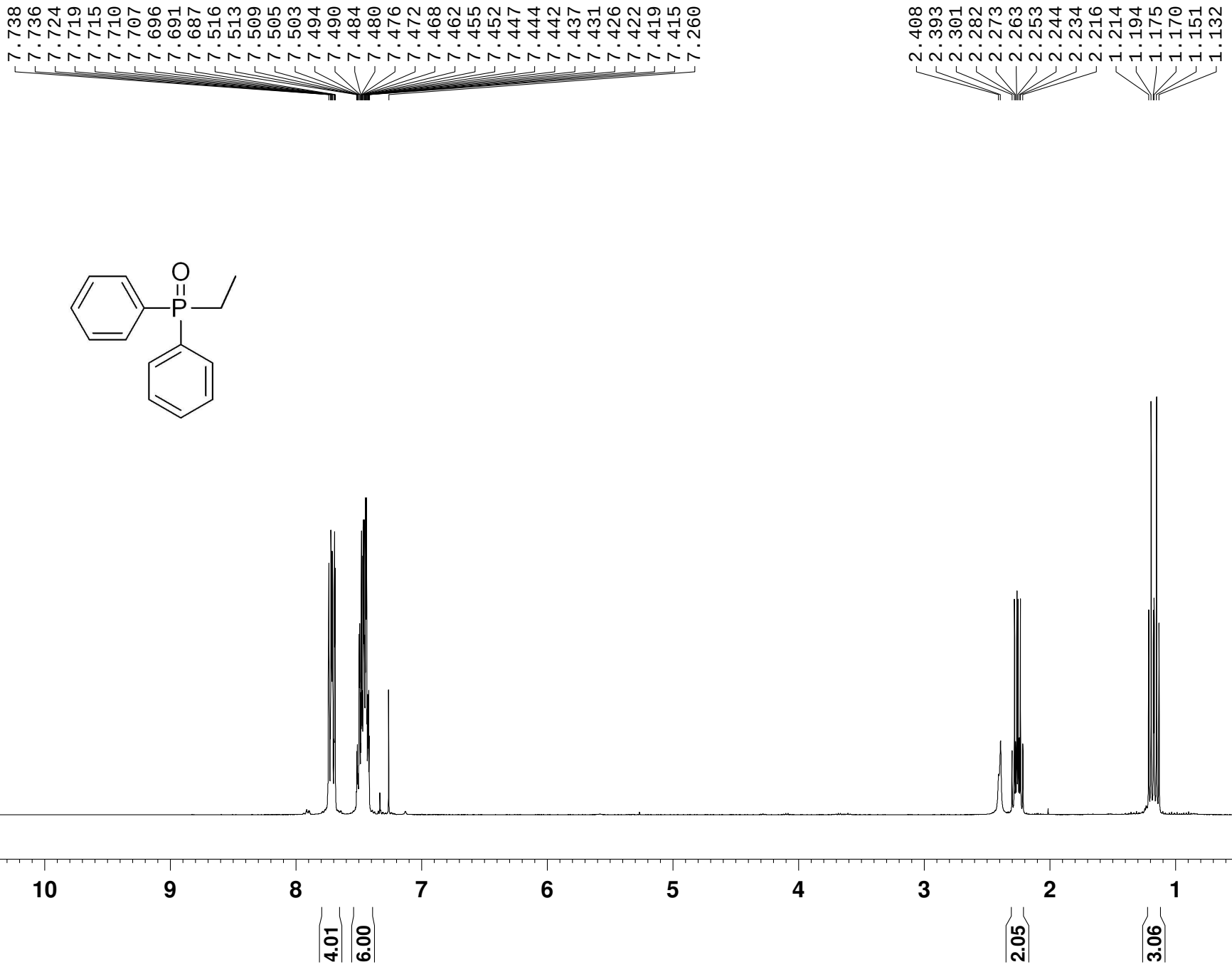
3s

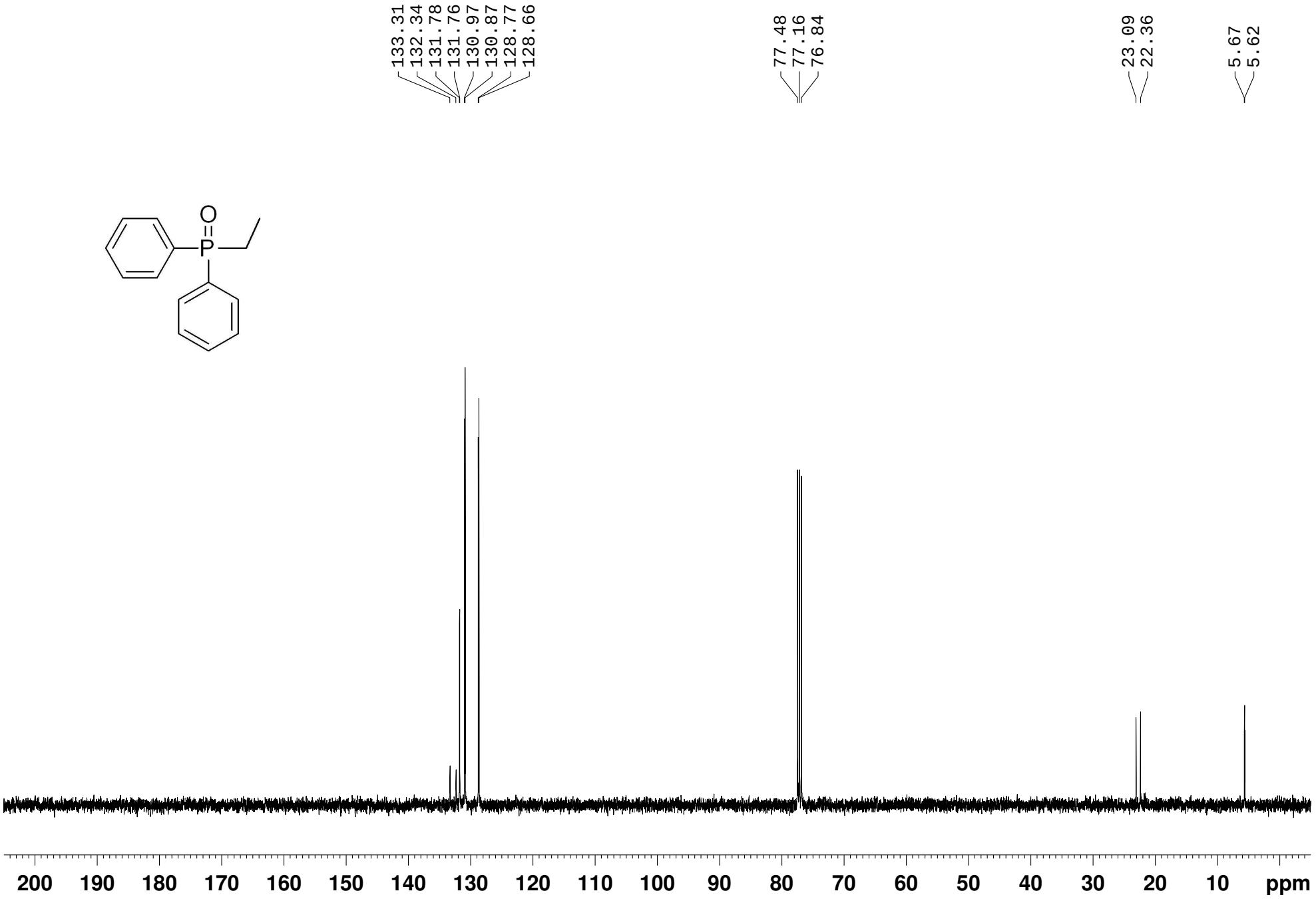

7.660
7.657
7.640
7.636
7.630
7.628
7.610
7.607
7.548
7.527
7.519
7.499
7.484
7.481
7.442
7.441
7.437
7.435
7.422
7.418
7.404
7.401
7.397
7.260
7.255
7.240
7.234
2.447
S
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
4.02
4.03
4.04
2.08
3.06

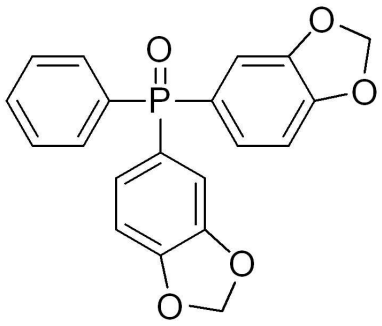

144.49
144.46
133.19
132.41
132.30
132.15
132.12
132.02
131.97
131.95
128.60
128.48
127.58
125.34
125.22
77.48
77.16
76.84
14.80
O
P
S
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



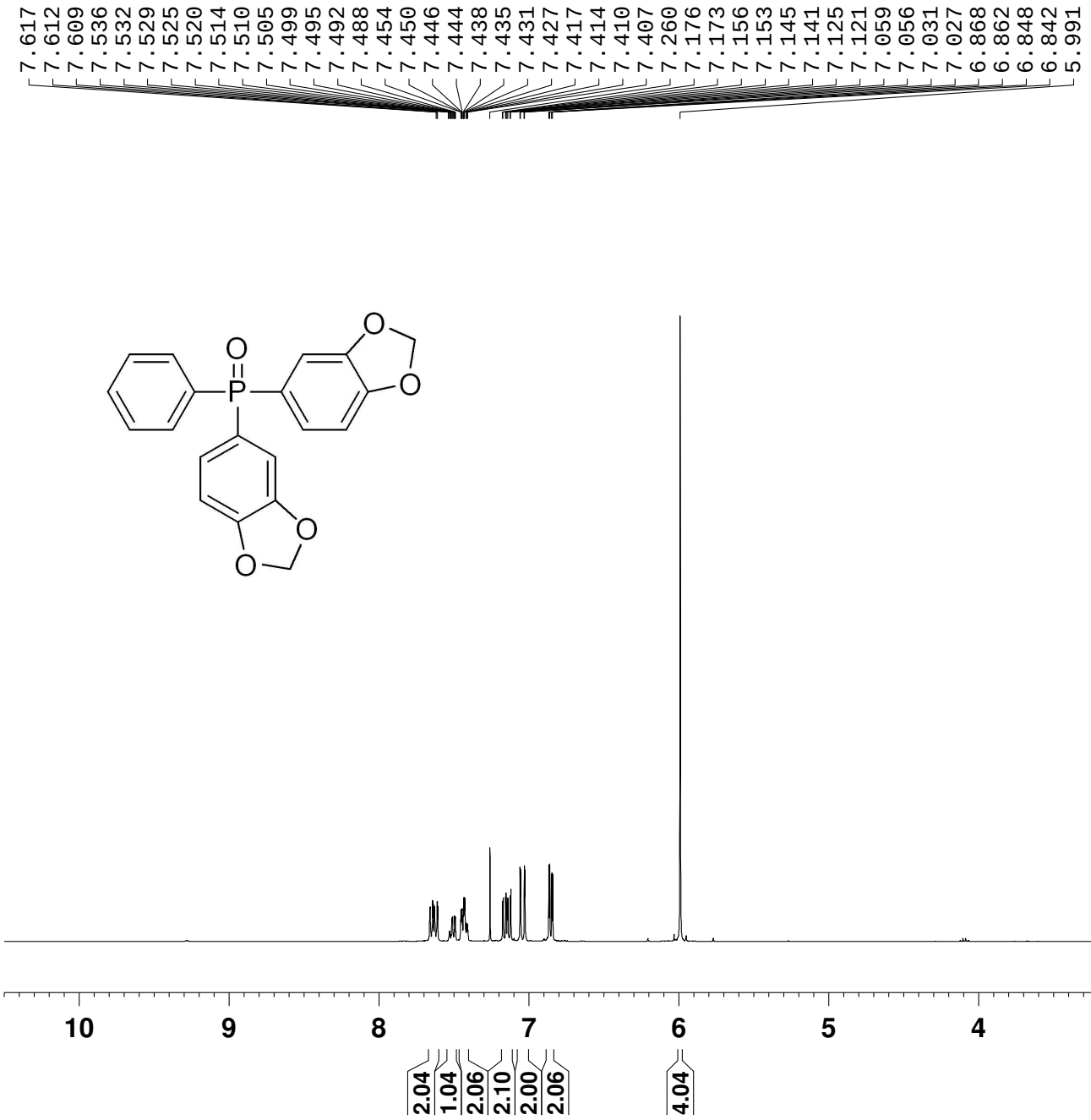
3t

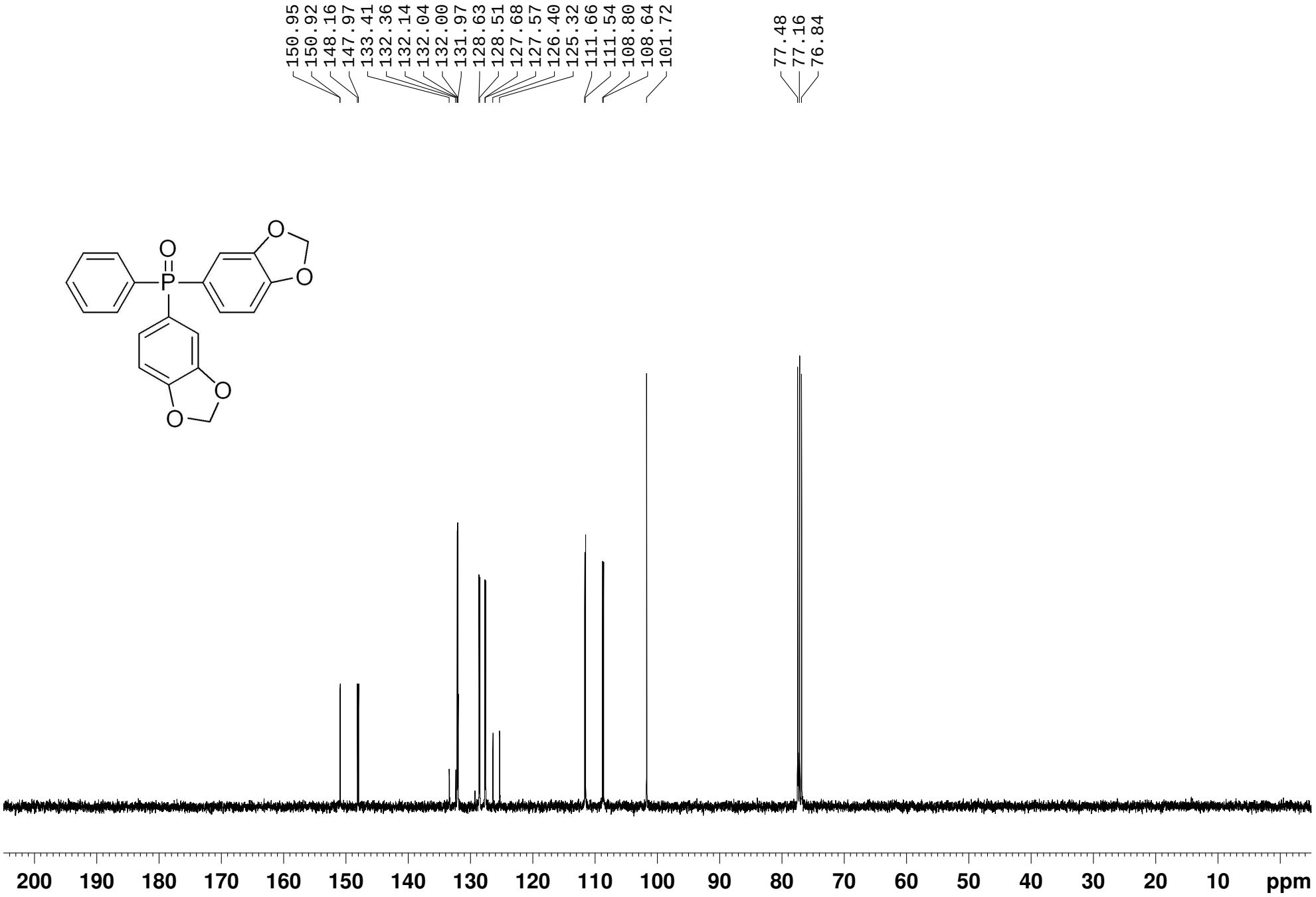

7.738
7.736
7.724
7.719
7.715
7.710
7.707
7.696
7.691
7.687
7.516
7.513
7.509
7.505
7.503
7.494
7.490
7.484
7.480
7.476
7.472
7.468
7.462
7.455
7.452
7.447
7.444
7.442
7.437
7.431
7.426
7.422
7.419
7.415
7.260
2.408
2.393
2.301
2.282
2.273
2.263
2.253
2.244
2.234
2.216
1.214
1.194
1.175
1.170
1.151
1.132
O
P
10
9
8
7
6
5
4
3
2
1
0
ppm
4.01
6.00
2.05
3.06

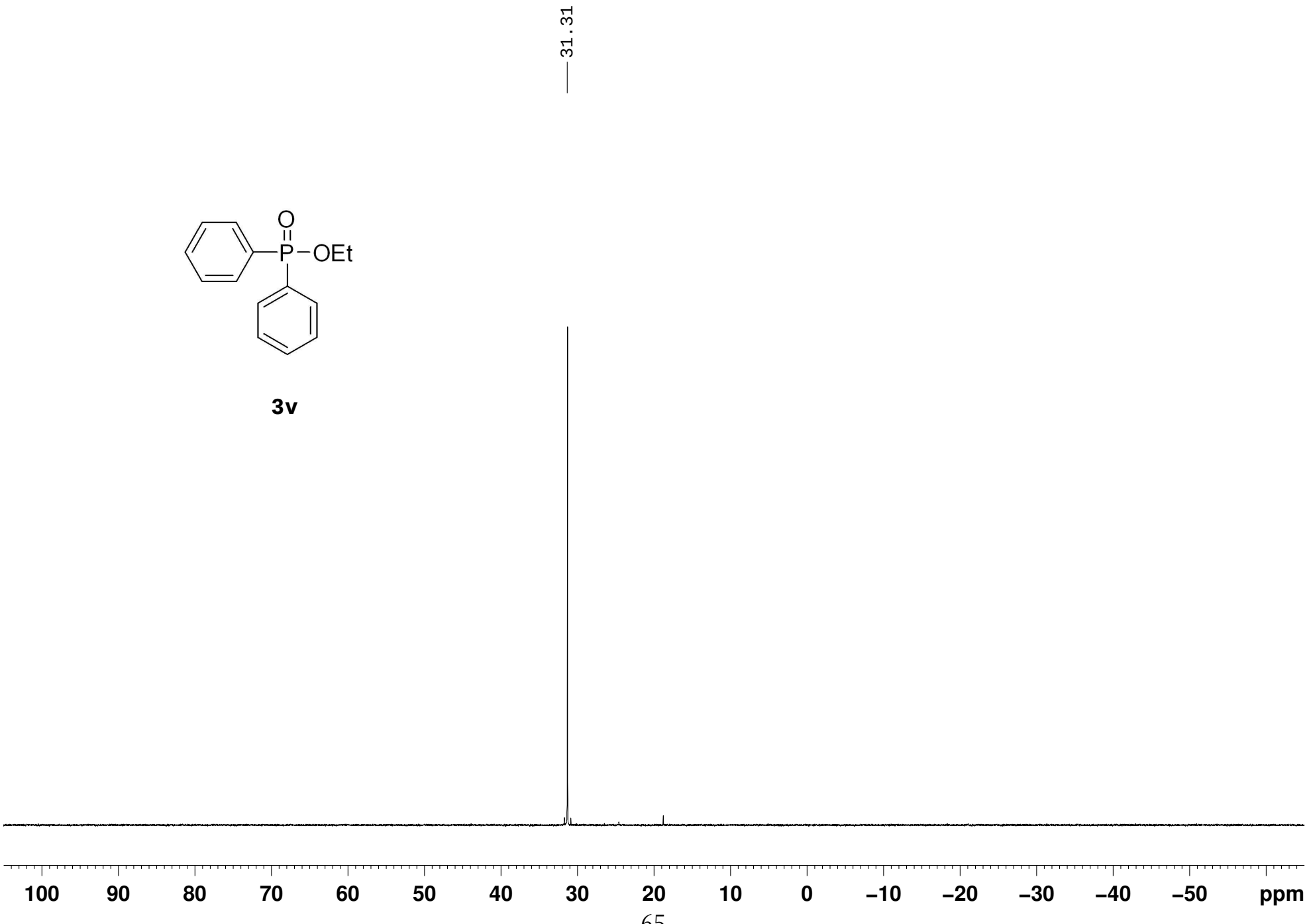

133.31
132.34
131.78
131.76
130.97
130.87
128.77
128.66
77.48
77.16
76.84
23.09
22.36
5.67
5.62
O
P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

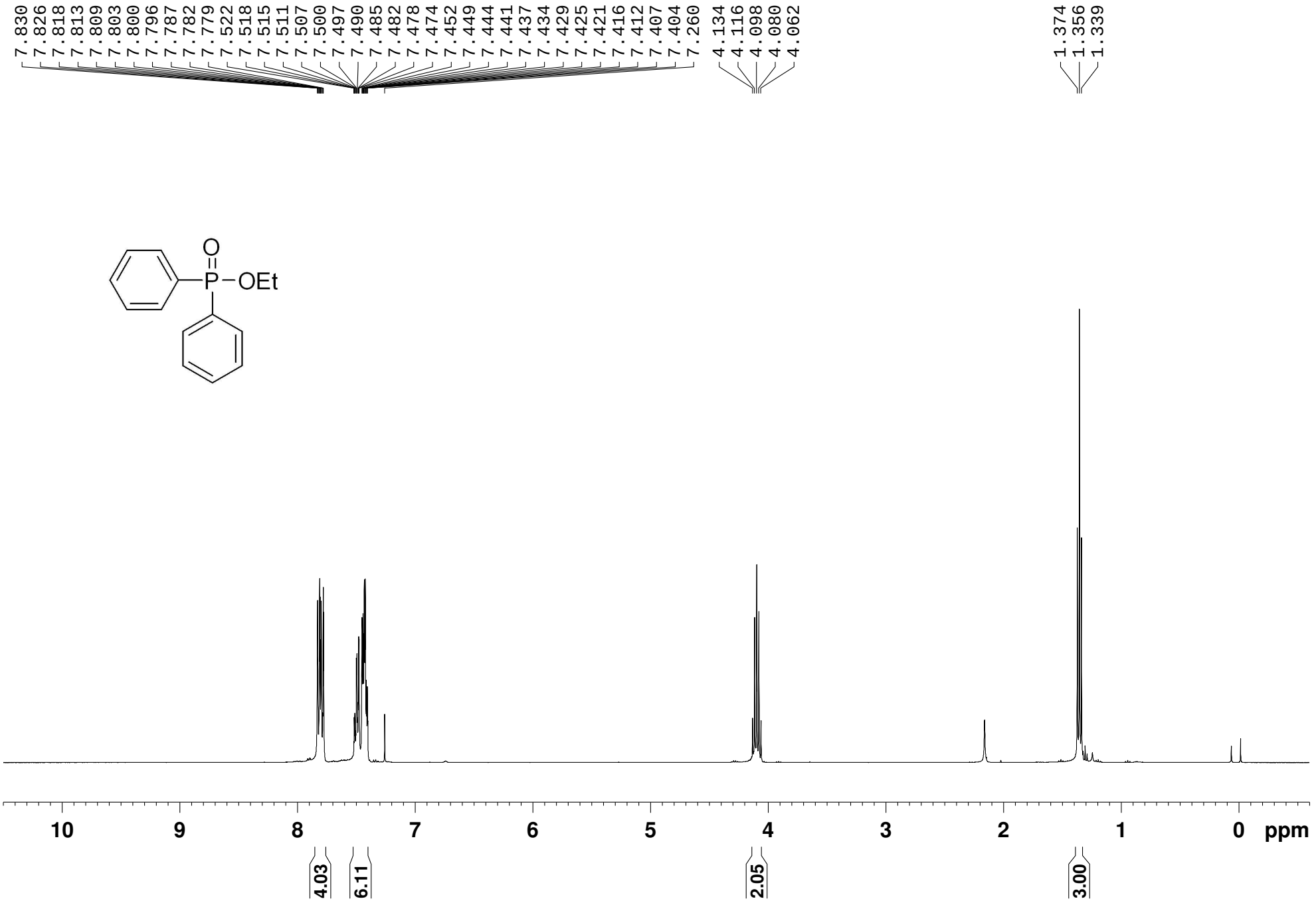


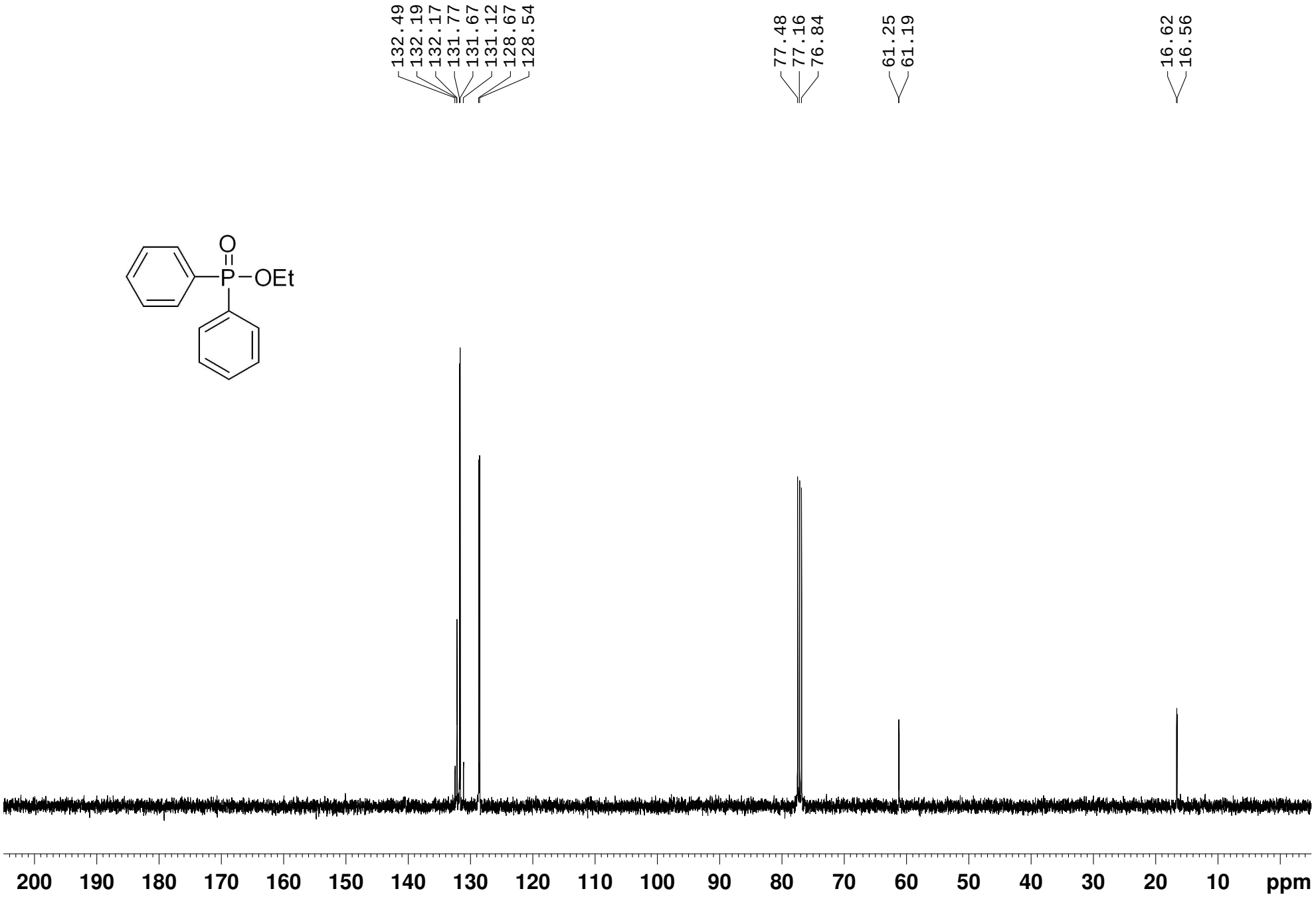
3u

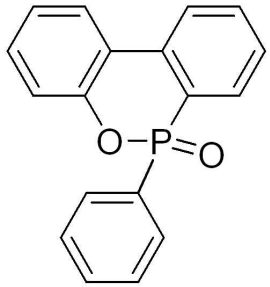

7.617
7.612
7.609
7.536
7.532
7.529
7.525
7.520
7.514
7.510
7.505
7.499
7.495
7.492
7.488
7.454
7.450
7.446
7.444
7.438
7.435
7.431
7.427
7.417
7.414
7.410
7.407
7.260
7.176
7.173
7.156
7.153
7.145
7.141
7.125
7.121
7.059
7.056
7.031
7.027
6.868
6.862
6.848
6.842
5.991
O
P
O
O
O
O
10
9
8
7
6
5
4
3
2
1
0
ppm
2.04
1.04
2.06
2.10
2.00
2.06
4.04


150.95
150.92
148.16
147.97
133.41
132.36
132.14
132.04
132.00
131.97
128.63
128.51
127.68
127.57
126.40
125.32
111.66
111.54
108.80
108.64
101.72
77.48
77.16
76.84
O
P
O
O
O
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


31.31
O
P
OEt
3v
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
−40
−50
ppm


7.830
7.826
7.818
7.813
7.809
7.803
7.800
7.796
7.787
7.782
7.779
7.522
7.518
7.515
7.511
7.507
7.500
7.497
7.490
7.485
7.482
7.478
7.474
7.452
7.449
7.444
7.441
7.437
7.434
7.429
7.425
7.421
7.416
7.412
7.407
7.404
7.260
4.134
4.116
4.098
4.080
4.062
1.374
1.356
1.339
O
P
OEt
10
9
8
7
6
5
4
3
2
1
0
ppm
4.03
6.11
2.05
3.00

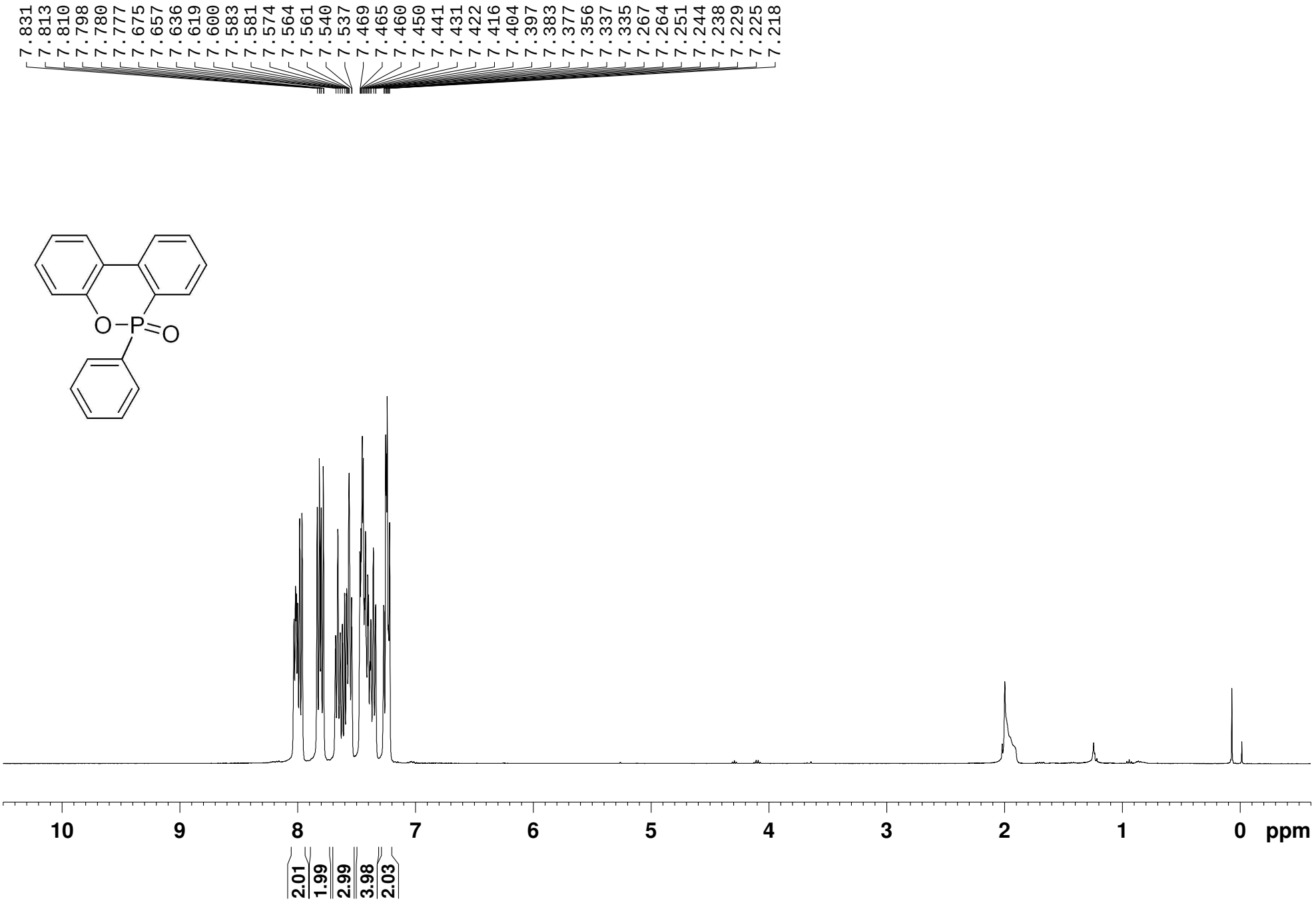

132.49
132.19
132.17
131.77
131.67
131.12
128.67
128.54
77.48
77.16
76.84
61.25
61.19
16.62
16.56
O
P
OEt
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

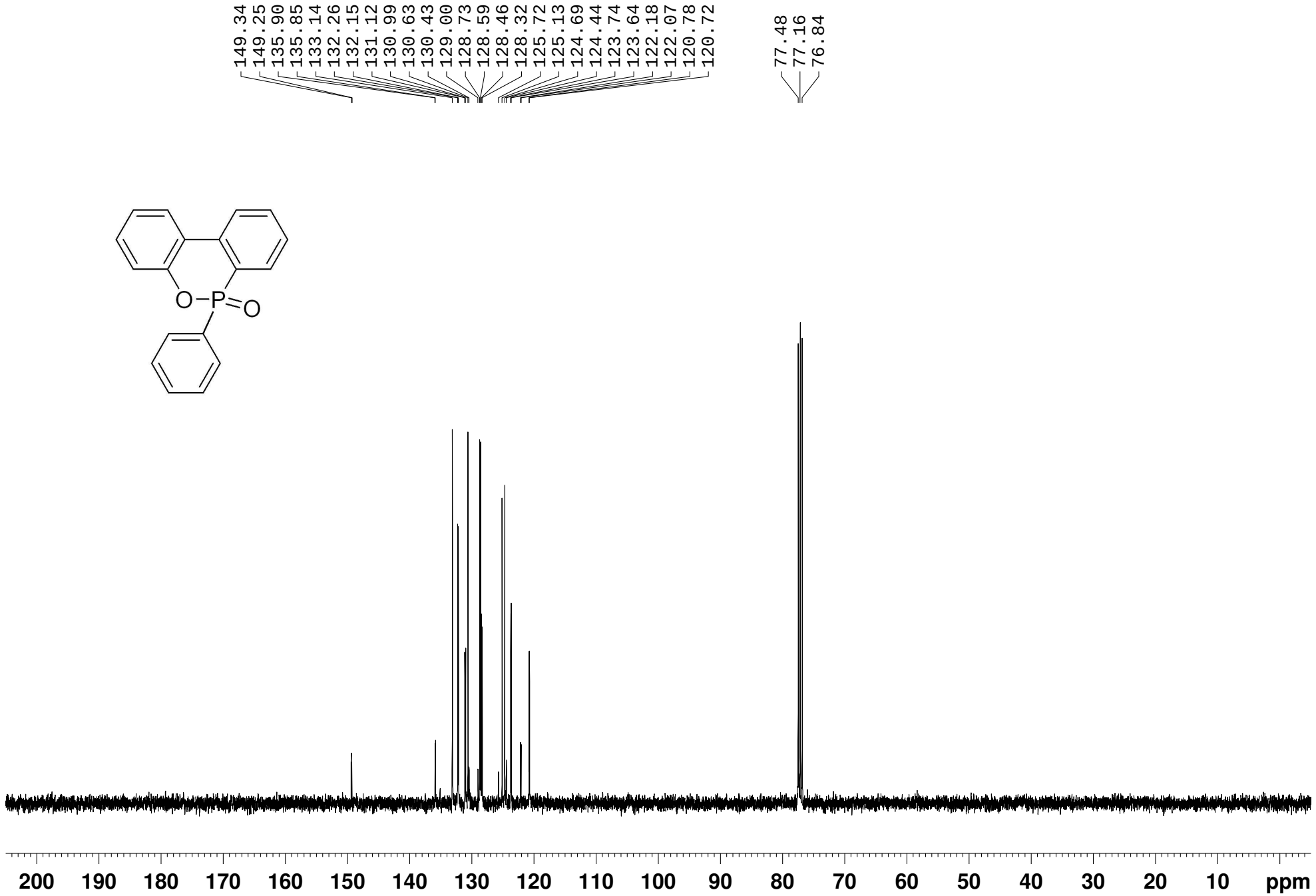


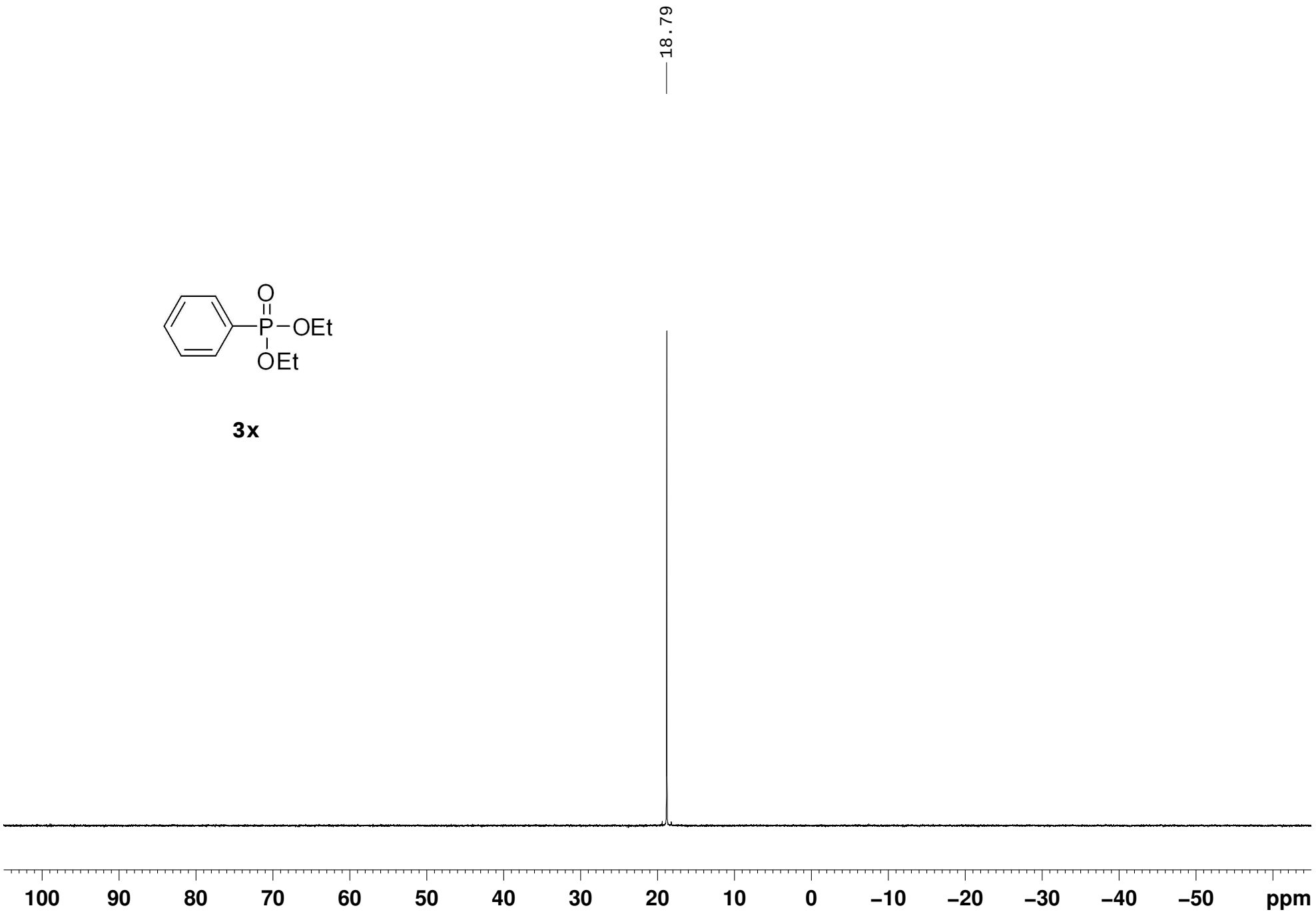
3w

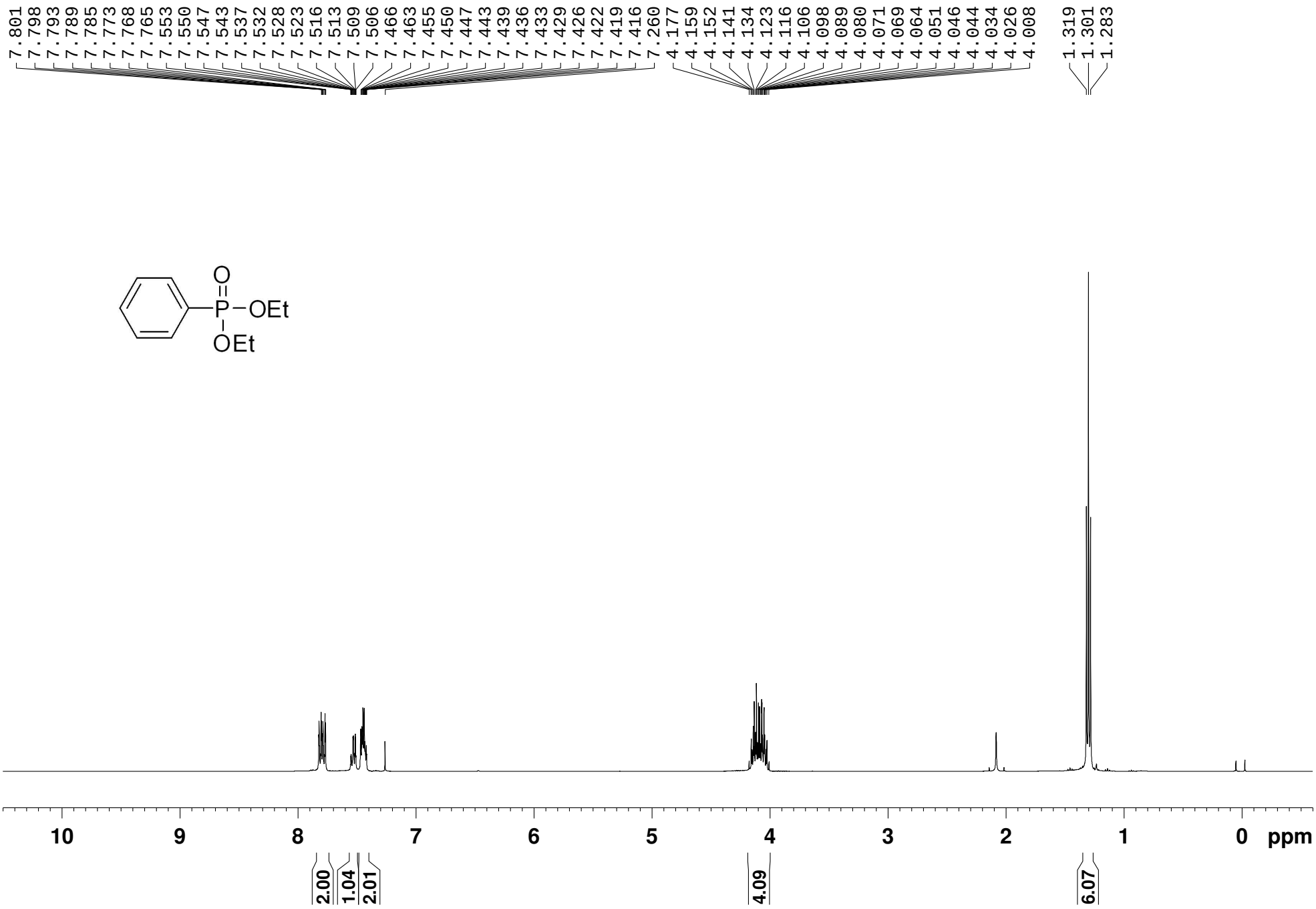
24.50

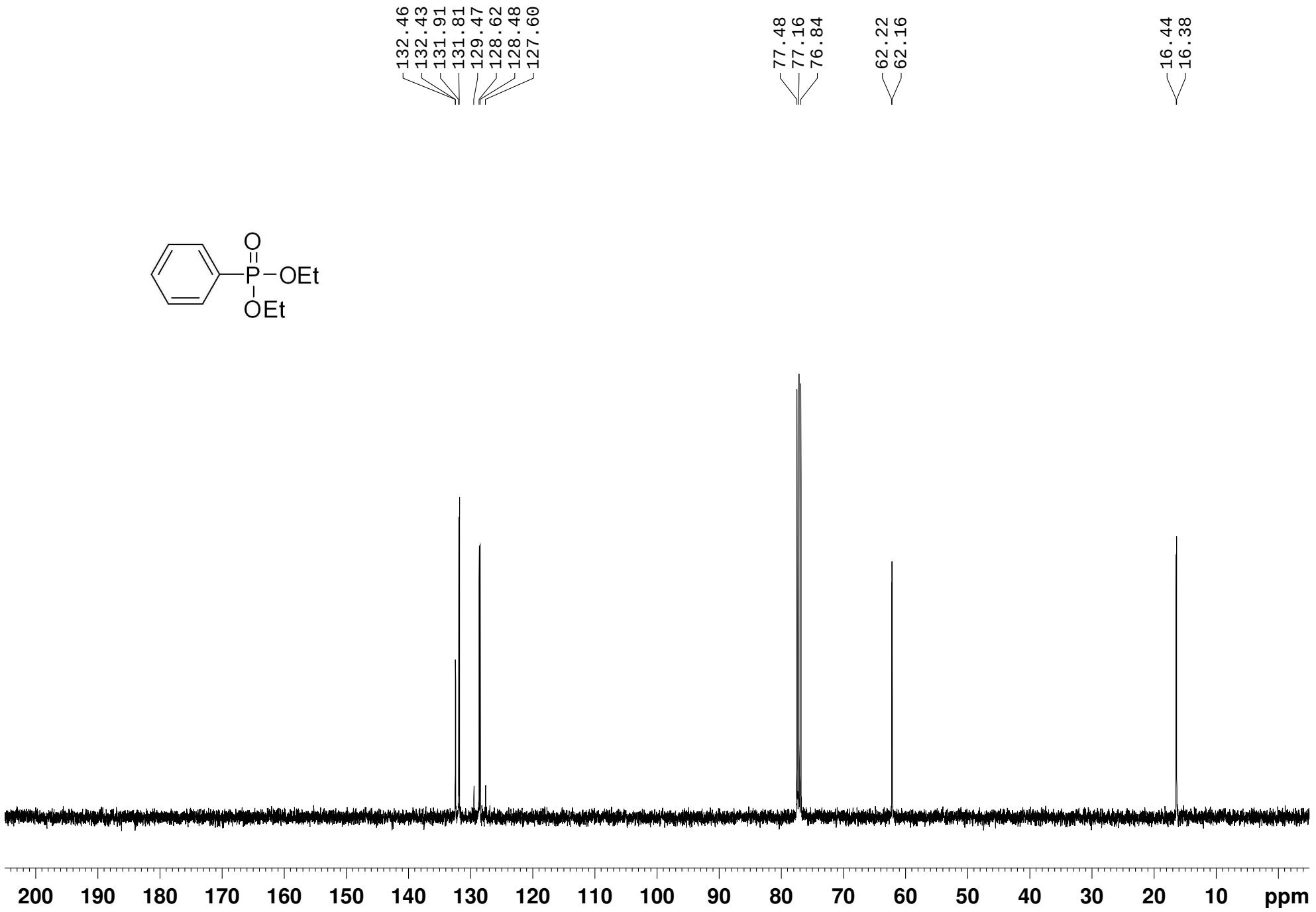
100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm


7.831
7.813
7.810
7.798
7.780
7.777
7.675
7.657
7.636
7.619
7.600
7.583
7.581
7.574
7.564
7.561
7.540
7.537
7.469
7.465
7.460
7.450
7.441
7.431
7.422
7.416
7.404
7.397
7.383
7.377
7.356
7.337
7.335
7.267
7.264
7.251
7.244
7.238
7.229
7.225
7.218
O
P
O
10
9
8
7
6
5
4
3
2
1
0
ppm
2.01
1.99
2.99
3.98
2.03


149.34
149.25
135.90
135.85
133.14
132.26
132.15
131.12
130.99
130.63
130.43
129.00
128.73
128.59
128.46
128.32
125.72
125.13
124.69
124.44
123.74
123.64
122.18
122.07
120.78
120.72
77.48
77.16
76.84
O
P
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


18.79
O
P
OEt
OEt
3x
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
−40
−50
ppm


7.801
7.798
7.793
7.789
7.785
7.773
7.768
7.765
7.553
7.550
7.547
7.543
7.537
7.532
7.528
7.523
7.516
7.513
7.509
7.506
7.466
7.463
7.455
7.450
7.447
7.443
7.439
7.436
7.433
7.429
7.426
7.422
7.419
7.416
7.260
4.177
4.159
4.152
4.141
4.134
4.123
4.116
4.106
4.098
4.089
4.080
4.071
4.069
4.064
4.051
4.046
4.044
4.034
4.026
4.008
1.319
1.301
1.283
O
P
OEt
OEt
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
1.04
2.01
4.09
6.07


132.46
132.43
131.91
131.81
129.47
128.62
128.48
127.60
77.48
77.16
76.84
62.22
62.16
16.44
16.38
O
P
OEt
OEt
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm