Supplementary Material

for

Microwave-Assisted Kabachnik–Fields Reaction with Amino Alcohols as the Amine Component

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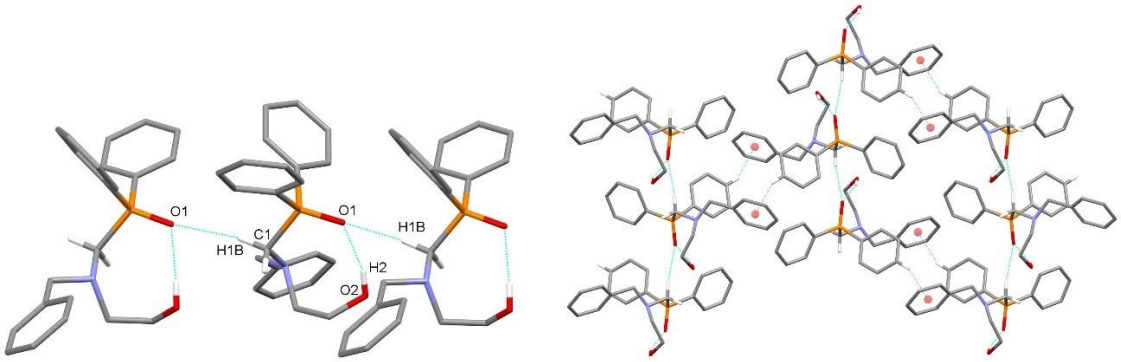
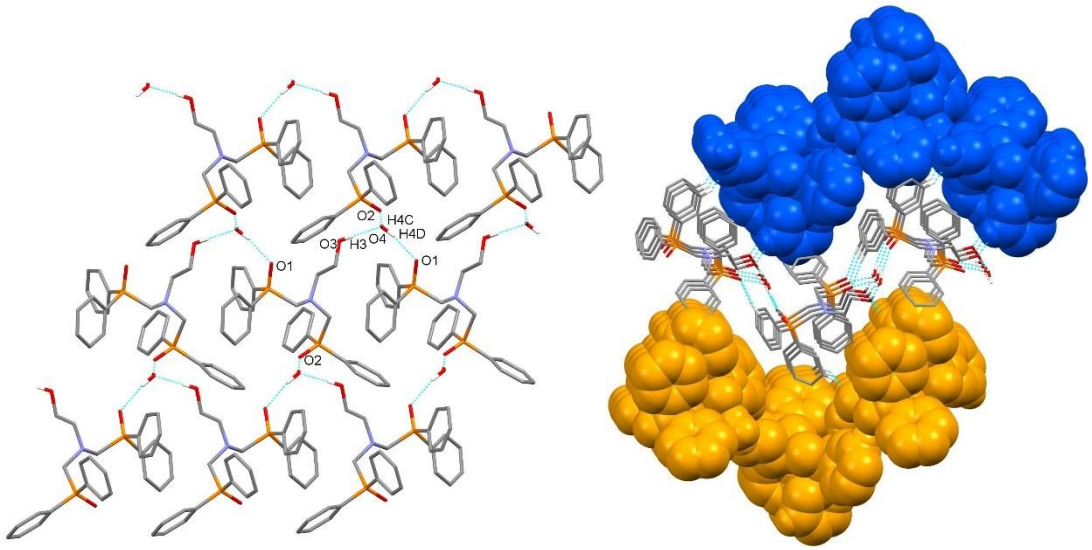
Single crystal X-ray diffraction measurements



(**a**) (**b**)

Figure S1. (**a**) Chain formation *via* C–H···O hydrogen bonding in **11a** (blue dashed lines). (**b**) Layer formation *via* C–H···π interactions. Hydrogen atoms not involved in the motif shown have been omitted for clarity.



(**a**) (**b**)

Figure S2. (**a**) Layer formation *via* O–H···O hydrogen bonding in **12a**·H_2O along *ab*-plane (blue dashed lines). (**b**) Packing of layers along c-axis. Hydrogen atoms not involved in the motif shown have been omitted for clarity.

Table S1. Hydrogen bond geometry for **11a** and **12a**·H_2O.

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)	Symmetry code
11a					
O2–H2···O1	0.82	2.04	2.824(2)	159	*x*, *y*, *z*
C1–H1B···O1	0.97	2.33	3.280(2)	168	–½ + *x*, 1½ – *y*, –½ + *z*
C20–H20···*Cg*1	0.93	2.74	3.603(2)	155	1 – *x*, 1 – *y*, –*z*
12a·H_2O					
O3–H3···O4	0.82	1.90	2.718(3)	179	*x*, *y*, *z*
O4–H4C···O2	0.83(2)	1.92(2)	2.737(3)	169(3)	1 – *x*, ½ + *y*, ½ – *z*
O4–H4D···O1	0.80(3)	1.98(3)	2.775(3)	171(3)	–1 + *x*, *y*, *z*
C19–H19···O1	0.93	2.46	3.371(4)	167	1 – *x*, –½ + *y*, ½ – *z*
C25–H25···O3	0.93	2.55	3.398(4)	152	1– *x*, 1 – *y*, 1 – *z*

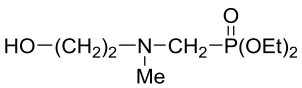
*Cg*1 is a C5–C10 ring centroid.

Table S2. Essential crystallographic data of the **11a** and **12a**·H_2O single-crystal diffraction experiments and model refinements

	11a	**12a**·H_2O
Formula	$C_{22}H_{24}NO_2P$	$C_{28}H_{31}NO_4P_2$
F. W.	365.39	507.48
Space grp.	$P2_1/n$ (no. 14)	$P2_1/c$ (no. 14)
***a, b, c* [Å]**	9.9927(7), 20.5357(10), 10.4303(7)	8.1148(4), 16.1422(10), 20.2089(9)
***β* [º]**	111.642(8)	91.644(4)
***V* [Å³]**	1989.5(2)	2646.1(2)
Z	4	4
D_{calc} **[g/cm³]**	1.220	1.274
***μ* [(MoKα)/mm]**	0.153	0.198
Cryst. Size [mm]	0.30 x 0.20 x 0.10	0.25 x 0.15 x 0.15
Temp. (K)	293(2)	293(2)
Radiation λ [Å]	MoKa 0.71073	MoKa 0.71073
***θ* Min-Max [º]**	5.218, 54.954	4.758, 54.966
Tot., Uniq. Data, R_{int}**,** R_{sigma}	10276, 4550, 0.0243, 0.0333	12825, 6081, 0.0275, 0.0391
Obsd data [*I* > 2.0 *σ*(*I*)]	3531	4408
Nref, Npar	4550, 236	6081, 323
R_1**,** wR^2	0.0419, 0.0974	0.0521, 0.1473
Max./Av. Shift/Error	0.00, 0.00	0.00, 0.00
Min/Max Res.Den. [e/Å³]	-0.27, 0.27	-0.26, 0.45

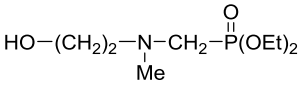
^{31}P NMR, ^{13}C NMR and ^{1}H NMR spectra of compounds

^{31}P NMR ($CDCl_3$)



4a

^{13}C NMR ($CDCl_3$)



4a

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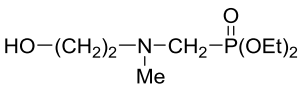
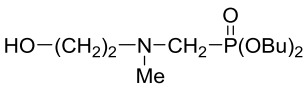


4a

^{31}P NMR ($CDCl_3$)



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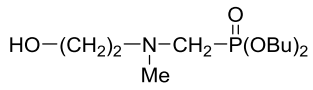
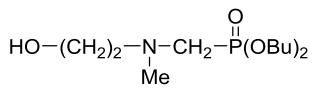
^{13}C NMR ($CDCl_3$)



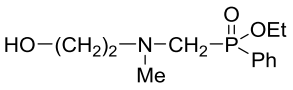
4b

^{1}H NMR ($CDCl_3$)



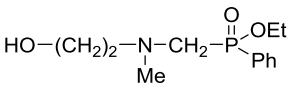
4b

^{31}P NMR ($CDCl_3$)



4c

^{13}C NMR ($CDCl_3$)



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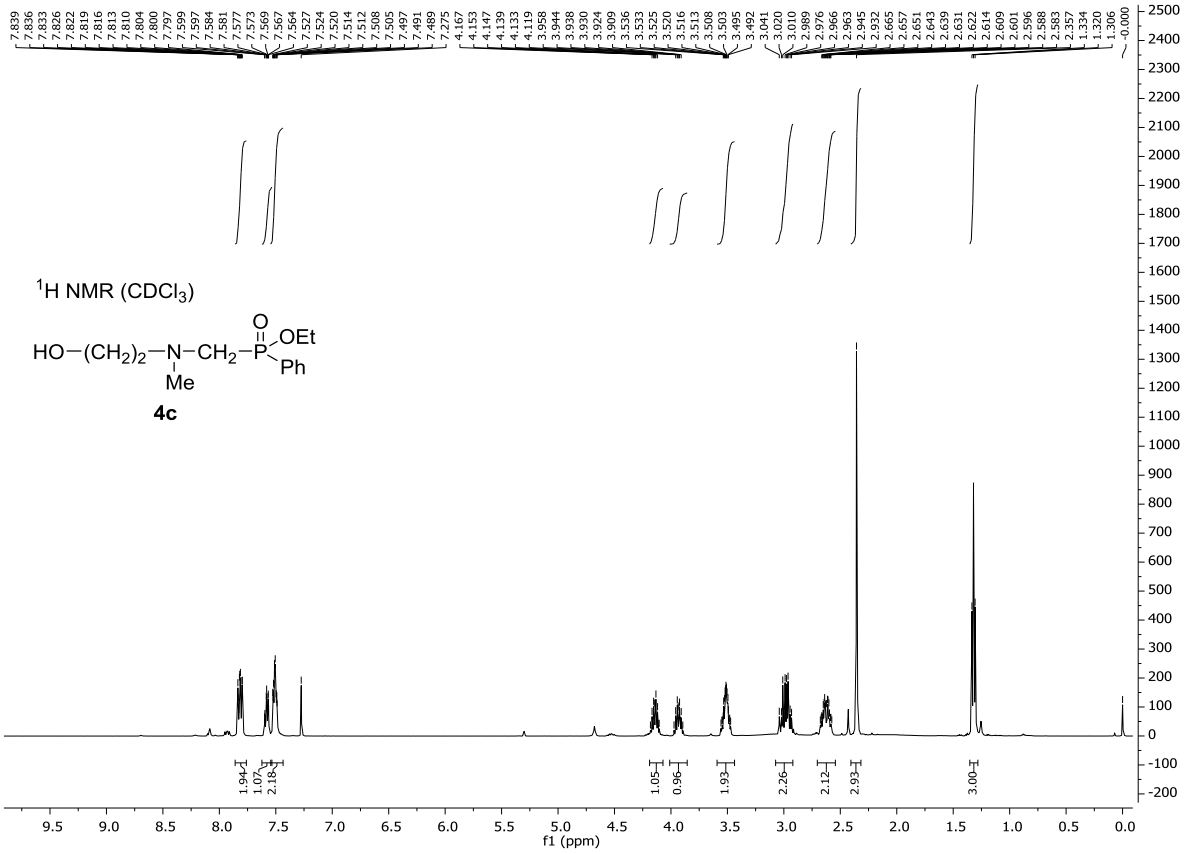
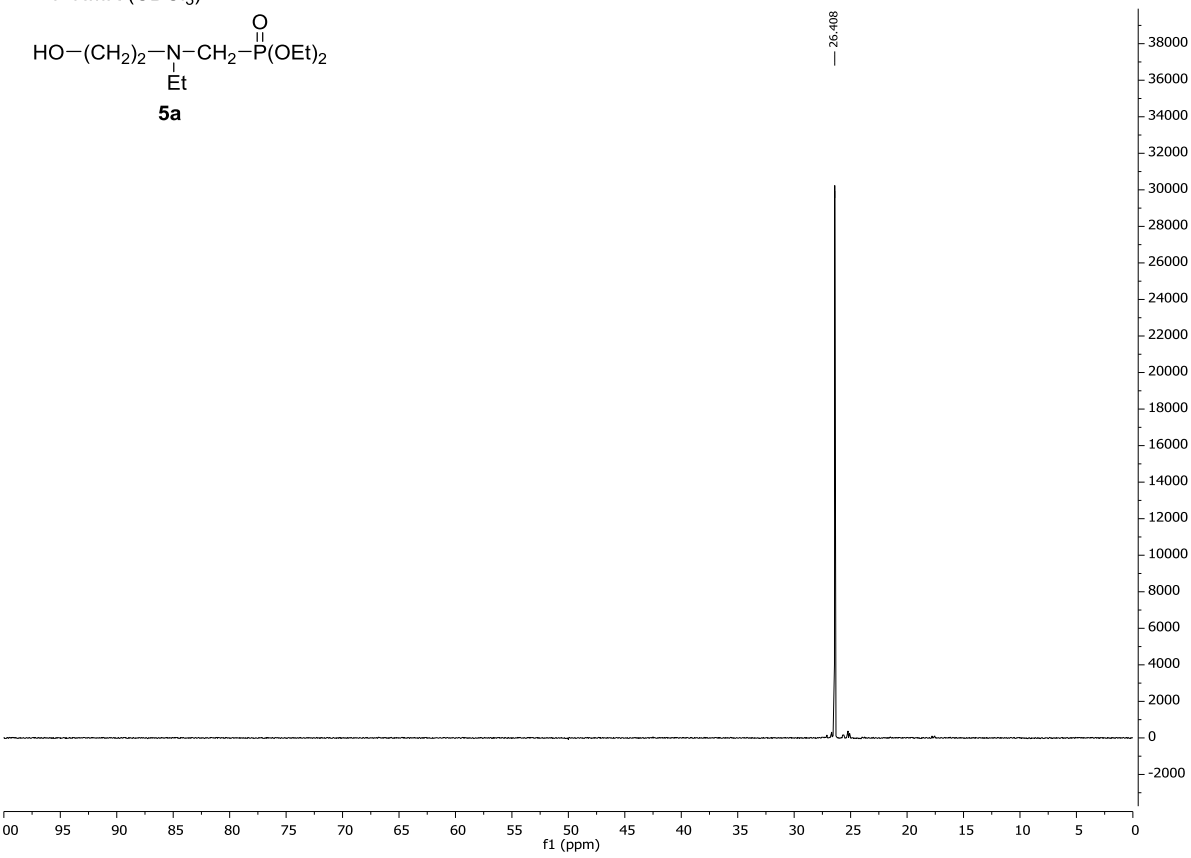
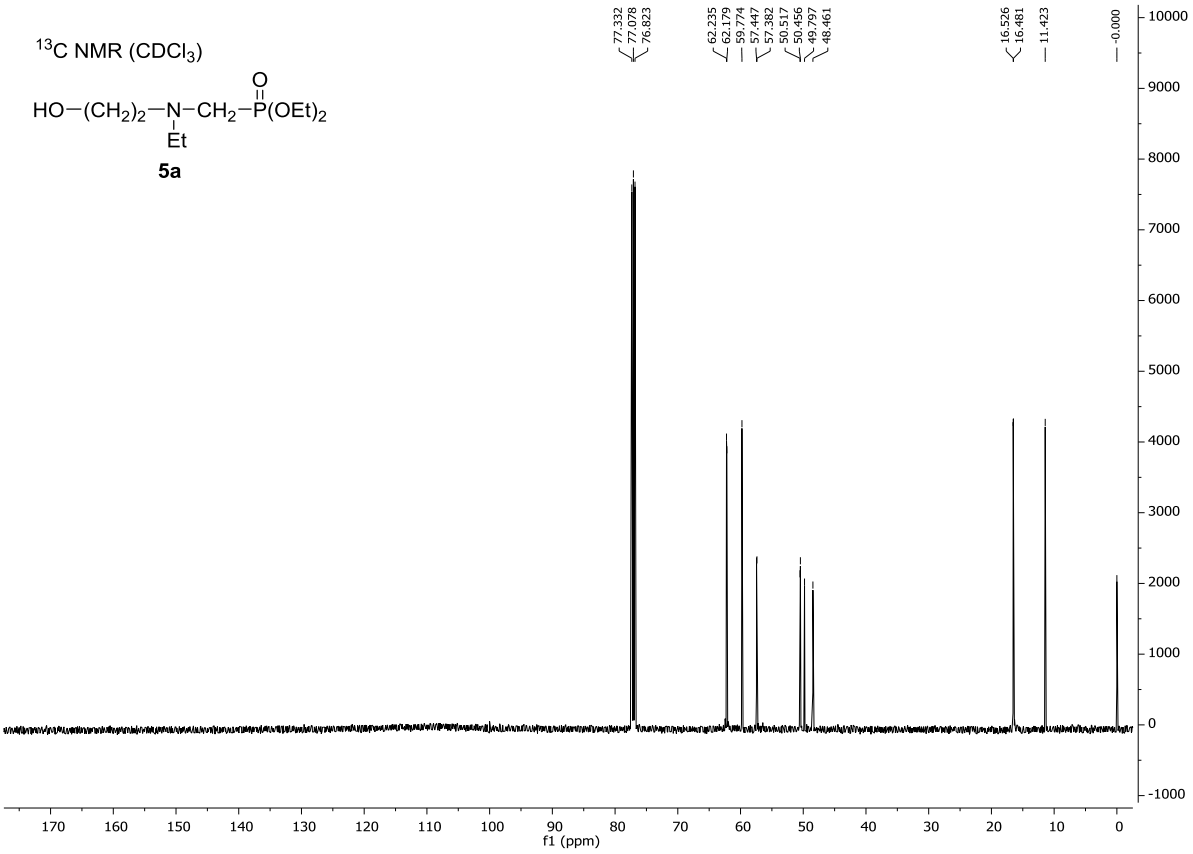
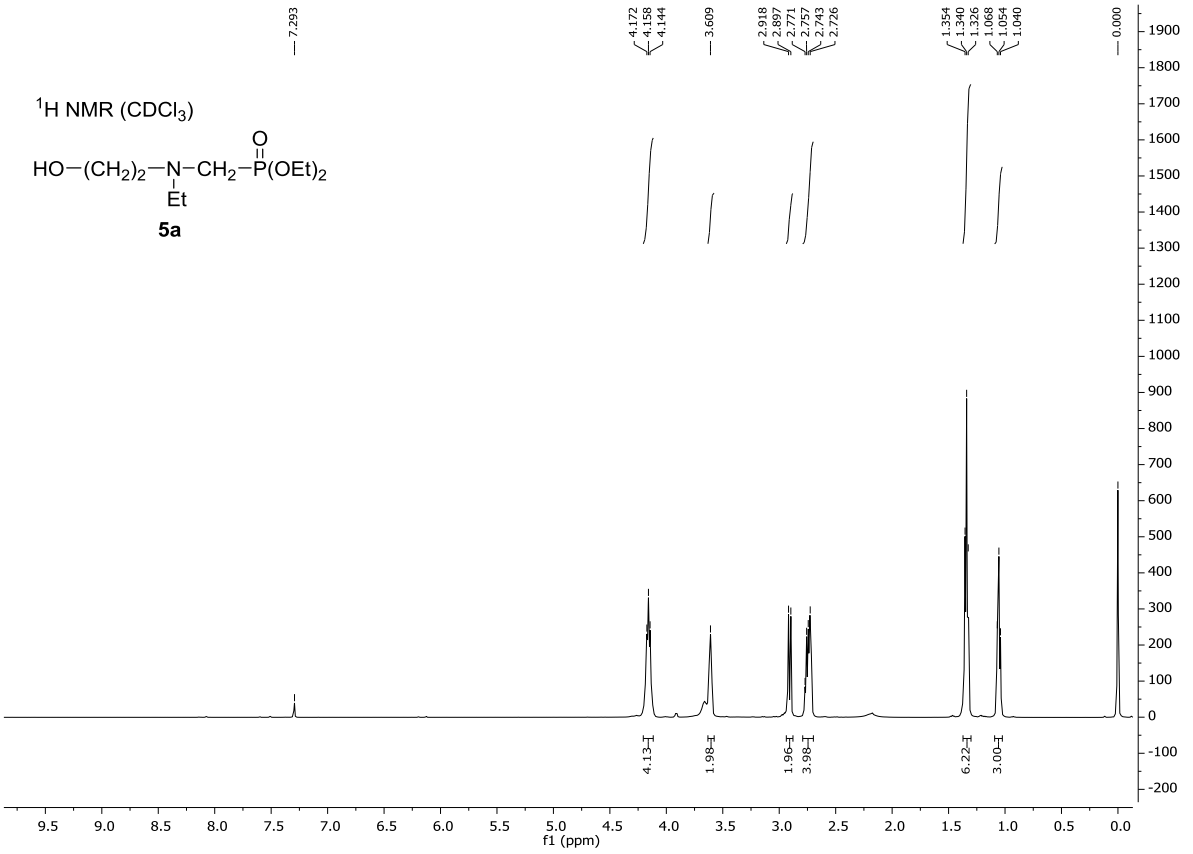
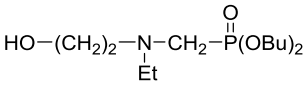

7.839 7.836 7.833 7.826 7.822 7.819 7.816 7.813 7.810 7.804 7.800 7.797 7.599 7.597 7.584 7.581 7.577 7.573 7.569 7.567 7.564 7.527 7.524 7.520 7.514 7.512 7.508 7.505 7.497 7.491 7.489 7.275 4.167 4.153 4.147 4.139 4.133 4.119 3.958 3.944 3.938 3.930 3.924 3.909 3.536 3.533 3.525 3.520 3.516 3.513 3.508 3.503 3.495 3.492 3.041 3.020 3.010 2.989 2.976 2.966 2.963 2.945 2.932 2.665 2.657 2.651 2.643 2.639 2.631 2.622 2.614 2.609 2.601 2.596 2.588 2.583 2.357 1.334 1.320 1.306 -0.000
1H NMR (CDCl3)
HO−(CH2)2−N(Me)−CH2−P(=O)(OEt)Ph
4c
1.94 1.07 2.18 1.05 0.96 1.93 2.26 2.12 2.93 3.00
f1 (ppm)


31P NMR (CDCl3)
HO−(CH2)2−N(Et)−CH2−P(=O)(OEt)2
5a
26.408
f1 (ppm)


13C NMR (CDCl3)
HO−(CH2)2−N(Et)−CH2−P(=O)(OEt)2
5a
77.332
77.078
76.823
62.235
62.179
59.774
57.447
57.382
50.517
50.456
49.797
48.461
16.526
16.481
11.423
-0.000
f1 (ppm)

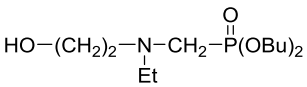

1H NMR (CDCl3)
HO−(CH2)2−N(Et)−CH2−P(=O)(OEt)2
5a
7.293
4.172
4.158
4.144
3.609
2.918
2.897
2.771
2.757
2.743
2.726
1.354
1.340
1.326
1.068
1.054
1.040
0.000
4.13
1.98
1.96
3.98
6.22
3.00
f1 (ppm)

^{31}P NMR ($CDCl_3$)



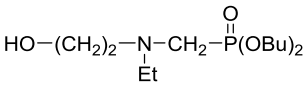
5b

^{13}C NMR ($CDCl_3$)



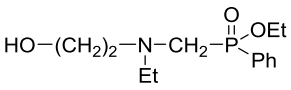
5b

^{1}H NMR ($CDCl_3$)

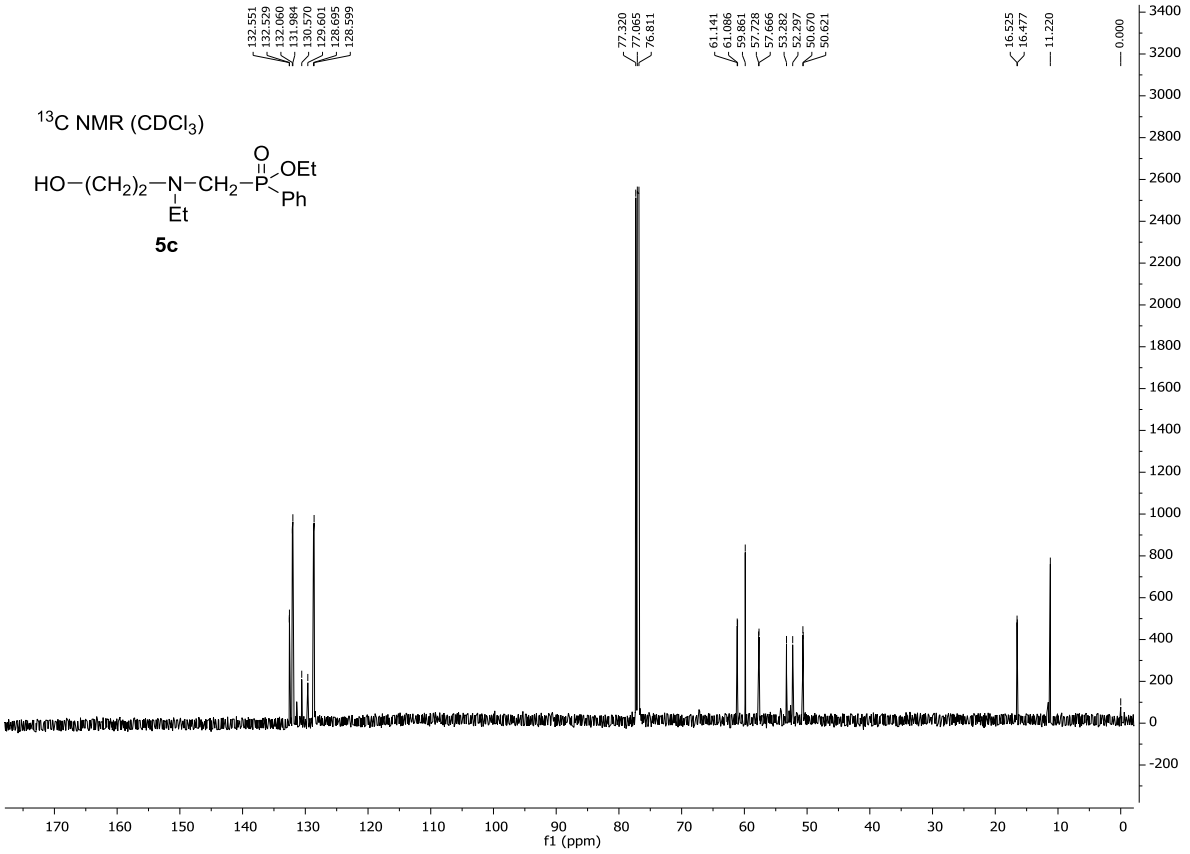


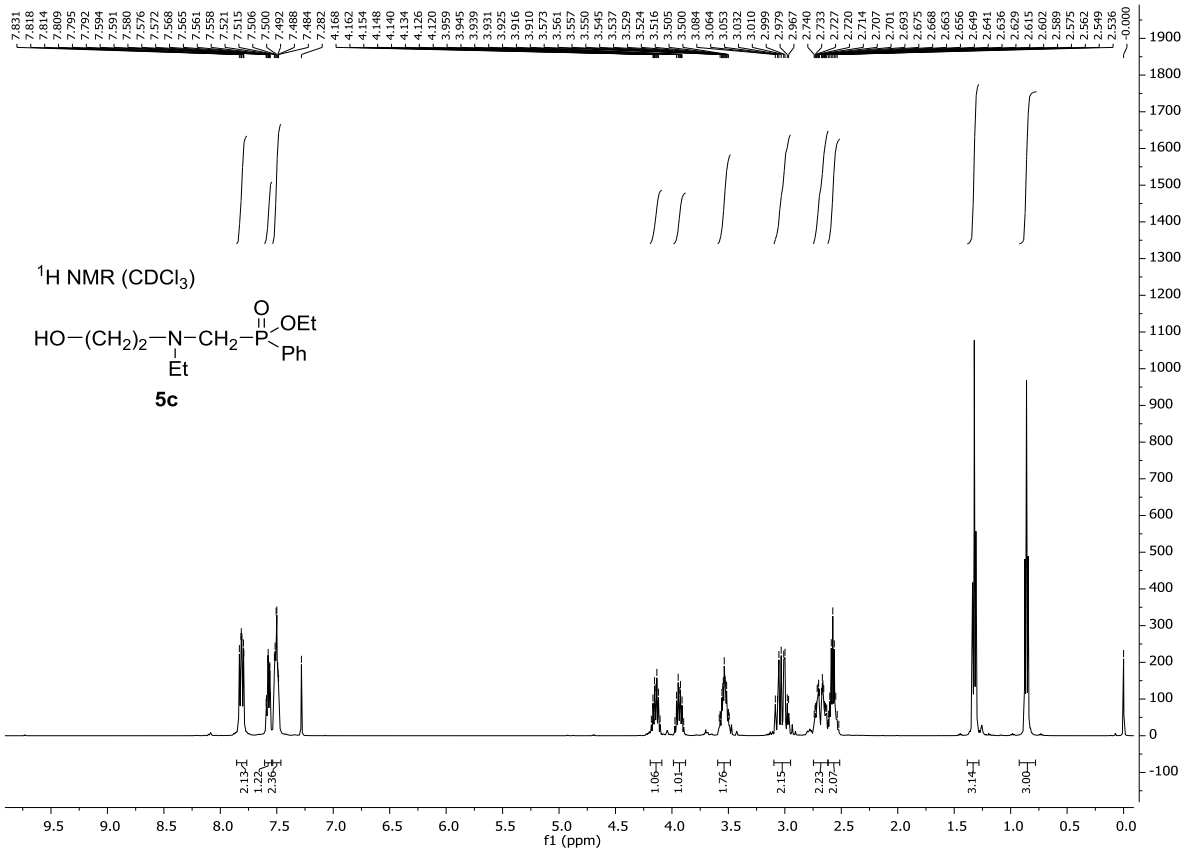
5b

^{31}P NMR ($CDCl_3$)



5c


132.551
132.529
132.060
131.984
130.570
129.601
128.695
128.599
77.320
77.065
76.811
61.141
61.086
59.861
57.728
57.666
53.282
52.297
50.670
50.621
16.525
16.477
11.220
0.000
^{13}C NMR ($CDCl_3$)
HO−(CH2)2−N(Et)−CH2−P(=O)(OEt)Ph
5c
f1 (ppm)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7.809
7.795
7.792
7.594
7.591
7.580
7.576
7.572
7.568
7.565
7.561
7.558
7.521
7.515
7.506
7.500
7.492
7.488
7.484
7.282
4.168
4.162
4.154
4.148
4.140
4.134
4.126
4.120
3.959
3.945
3.939
3.931
3.925
3.916
3.910
3.573
3.561
3.557
3.550
3.545
3.537
3.529
3.524
3.516
3.505
3.500
3.084
3.064
3.053
3.032
3.010
2.999
2.979
2.967
2.740
2.733
2.727
2.720
2.714
2.707
2.701
2.693
2.675
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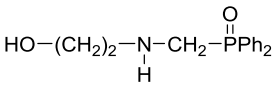
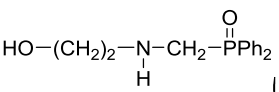
2.656
2.649
2.641
2.636
2.629
2.615
2.602
2.589
2.575
2.562
2.549
2.536
-0.000
^{1}H NMR ($CDCl_3$)
HO−(CH2)2−N(Et)−CH2−P(=O)(OEt)Ph
5c
2.13
1.22
2.36
1.06
1.01
1.76
2.15
2.23
2.07
3.14
3.00
f1 (ppm)

^{31}P NMR ($CDCl_3$)

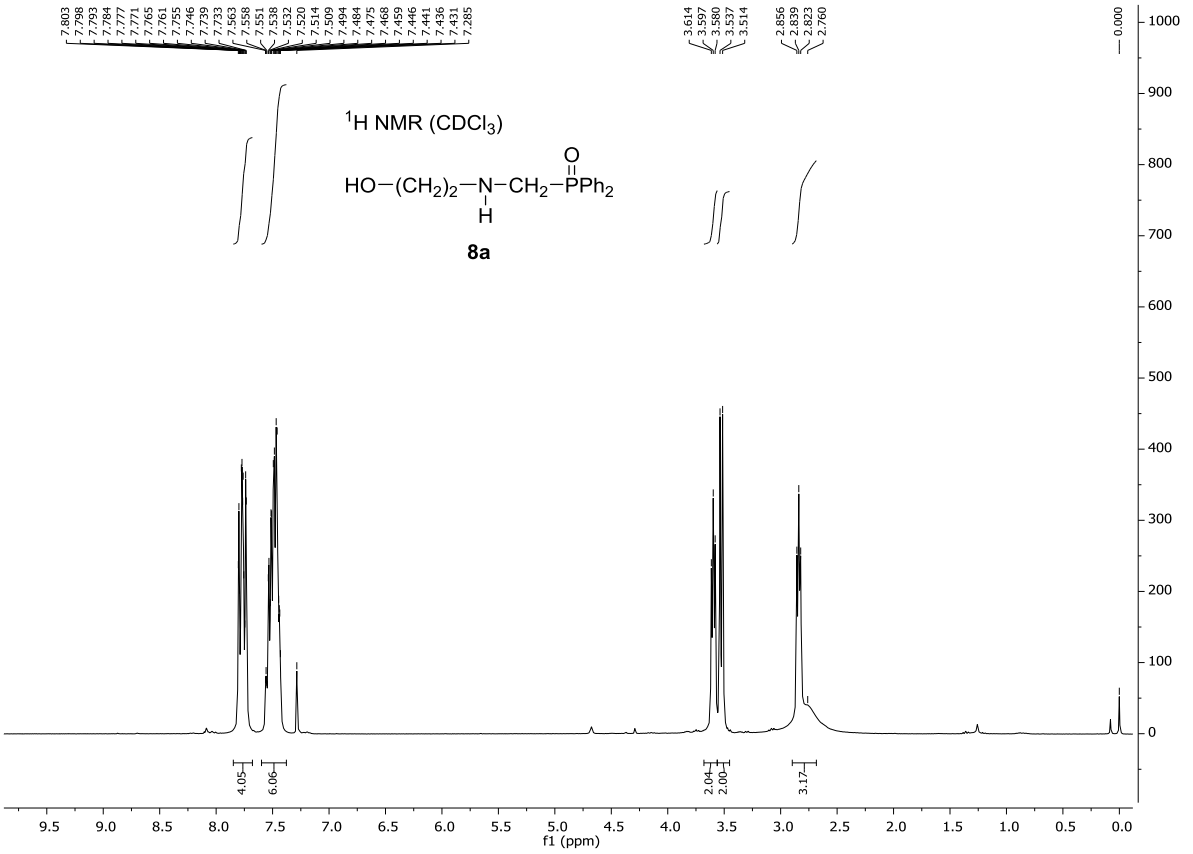


8a

^{13}C NMR ($CDCl_3$)



8a


7.803
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7.793
7.784
7.777
7.771
7.765
7.761
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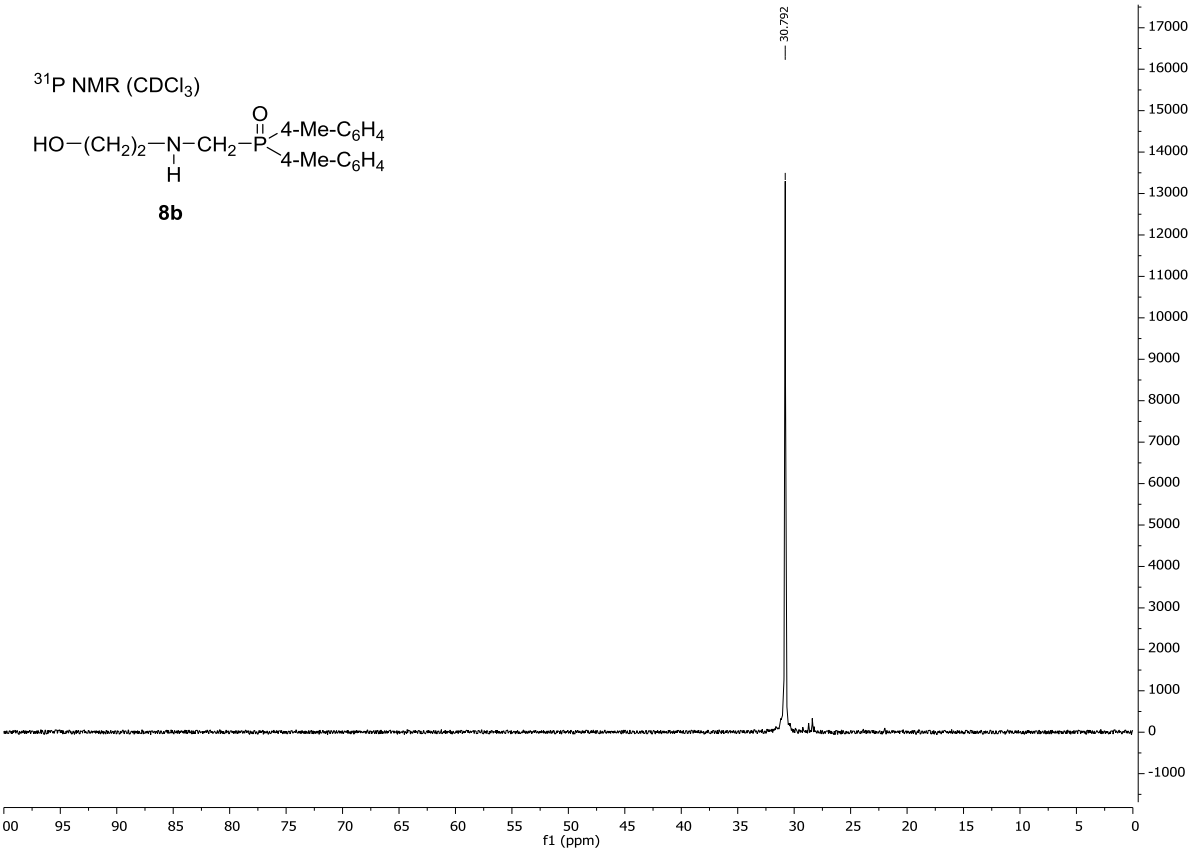
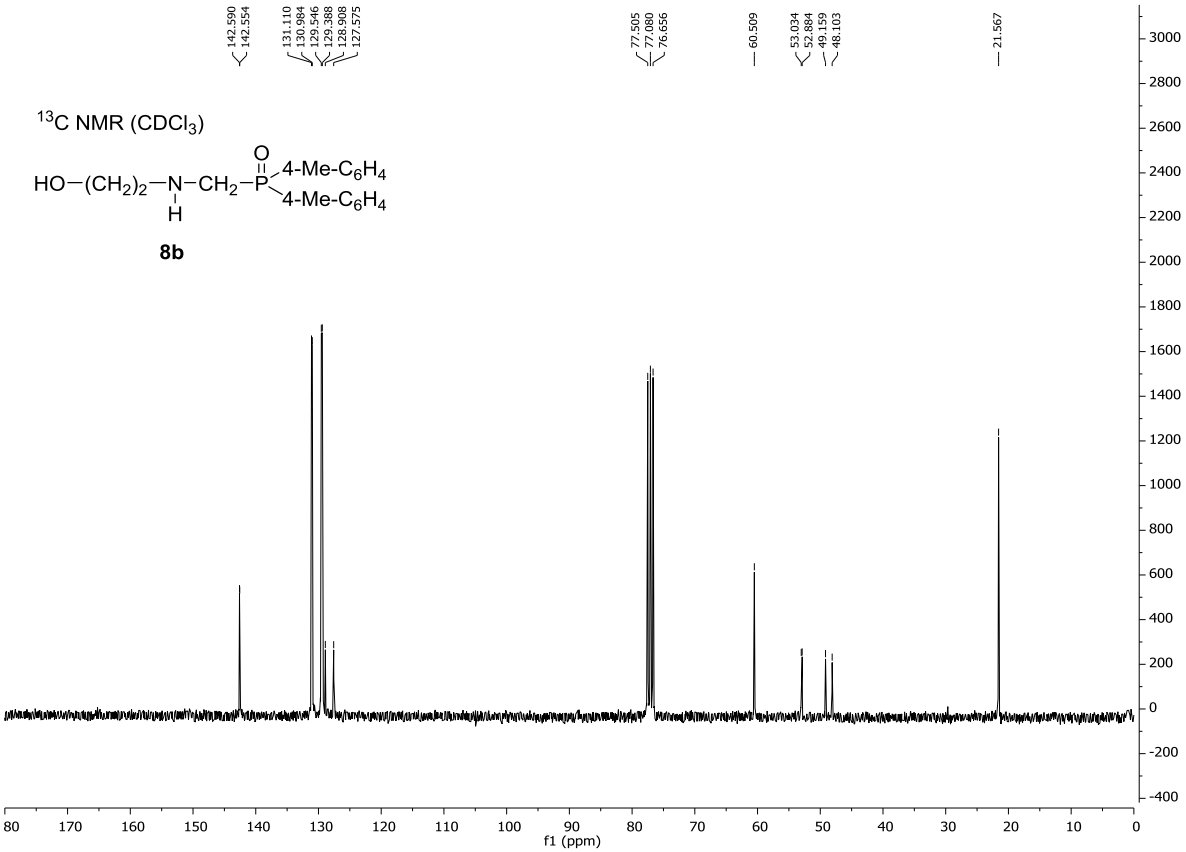
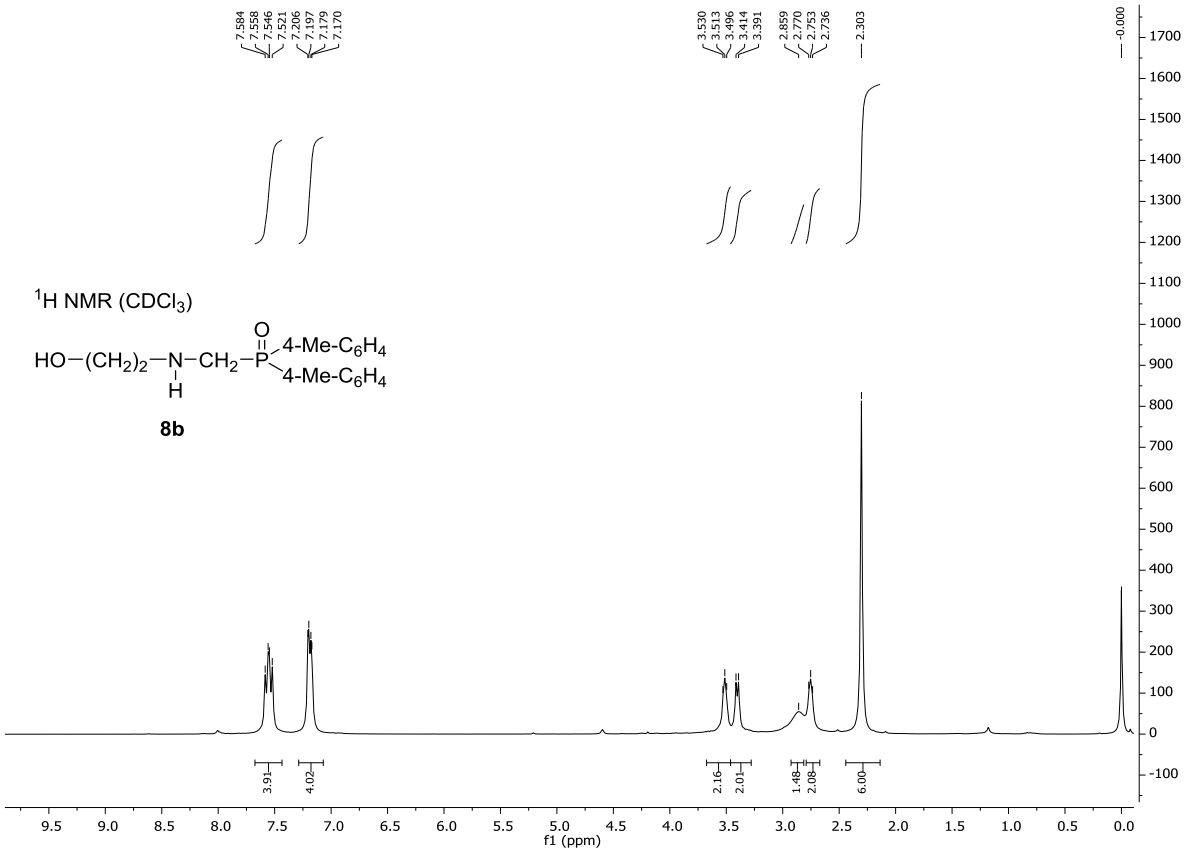
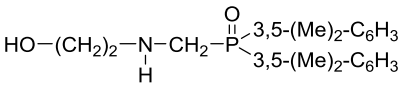
7.746
7.739
7.733
7.563
7.558
7.551
7.538
7.532
7.520
7.514
7.509
7.494
7.484
7.475
7.468
7.459
7.446
7.441
7.436
7.431
7.285
3.614
3.597
3.580
3.537
3.514
2.856
2.839
2.823
2.760
0.000
1H NMR (CDCl3)
HO−(CH2)2−N(H)−CH2−P(=O)Ph2
8a
4.05
6.06
2.04
2.00
3.17
f1 (ppm)


30.792
31P NMR (CDCl3)
HO−(CH2)2−N(H)−CH2−P(=O)(4-Me-C6H4)(4-Me-C6H4)
8b
f1 (ppm)


142.590
142.554
131.110
130.984
129.546
129.388
128.908
127.575
77.505
77.080
76.656
60.509
53.034
52.884
49.159
48.103
21.567
13C NMR (CDCl3)
HO−(CH2)2−N(H)−CH2−P(=O)(4-Me-C6H4)(4-Me-C6H4)
8b
f1 (ppm)

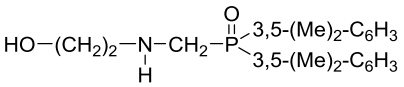

7.584
7.558
7.546
7.521
7.206
7.197
7.179
7.170
3.530
3.513
3.496
3.414
3.391
2.859
2.770
2.753
2.736
2.303
-0.000
1H NMR (CDCl3)
HO−(CH2)2−N(H)−CH2−P(=O)(4-Me-C6H4)(4-Me-C6H4)
8b
3.91
4.02
2.16
2.01
1.48
2.08
6.00
f1 (ppm)

^{31}P NMR ($CDCl_3$)

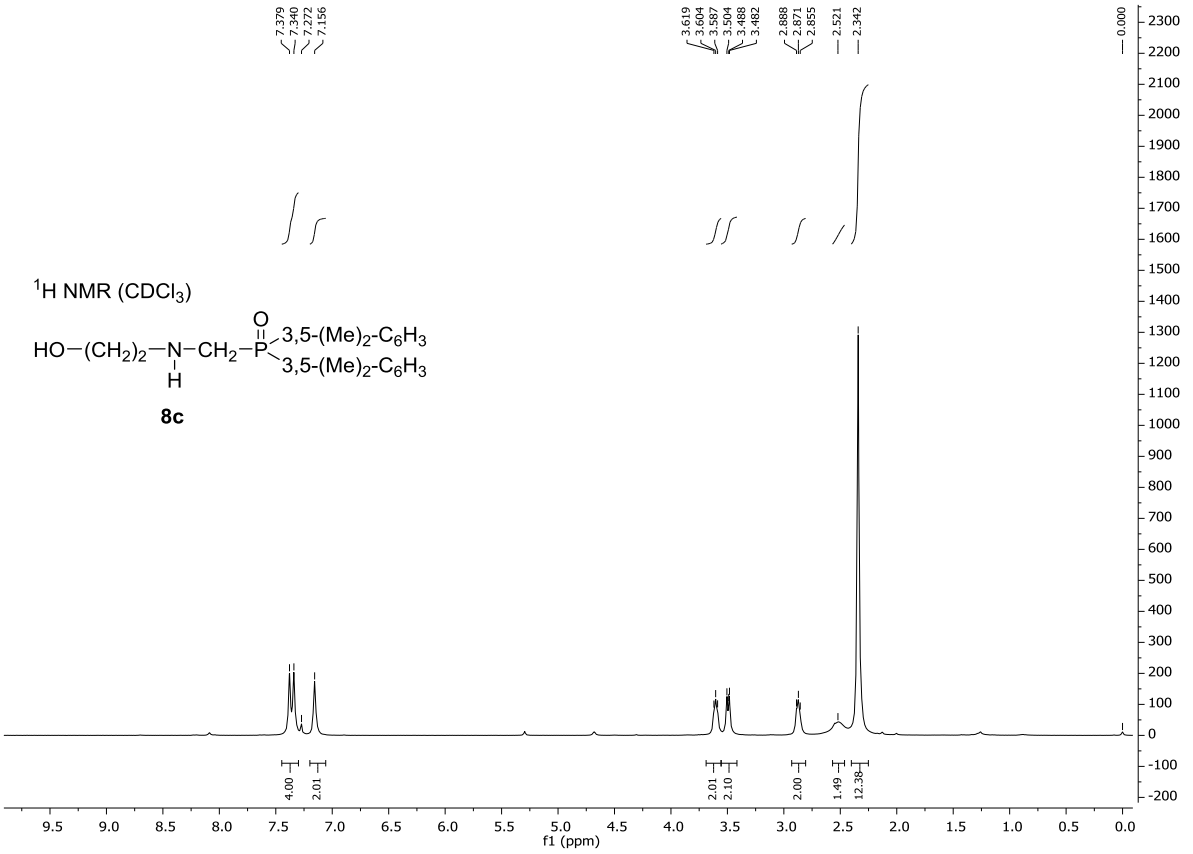


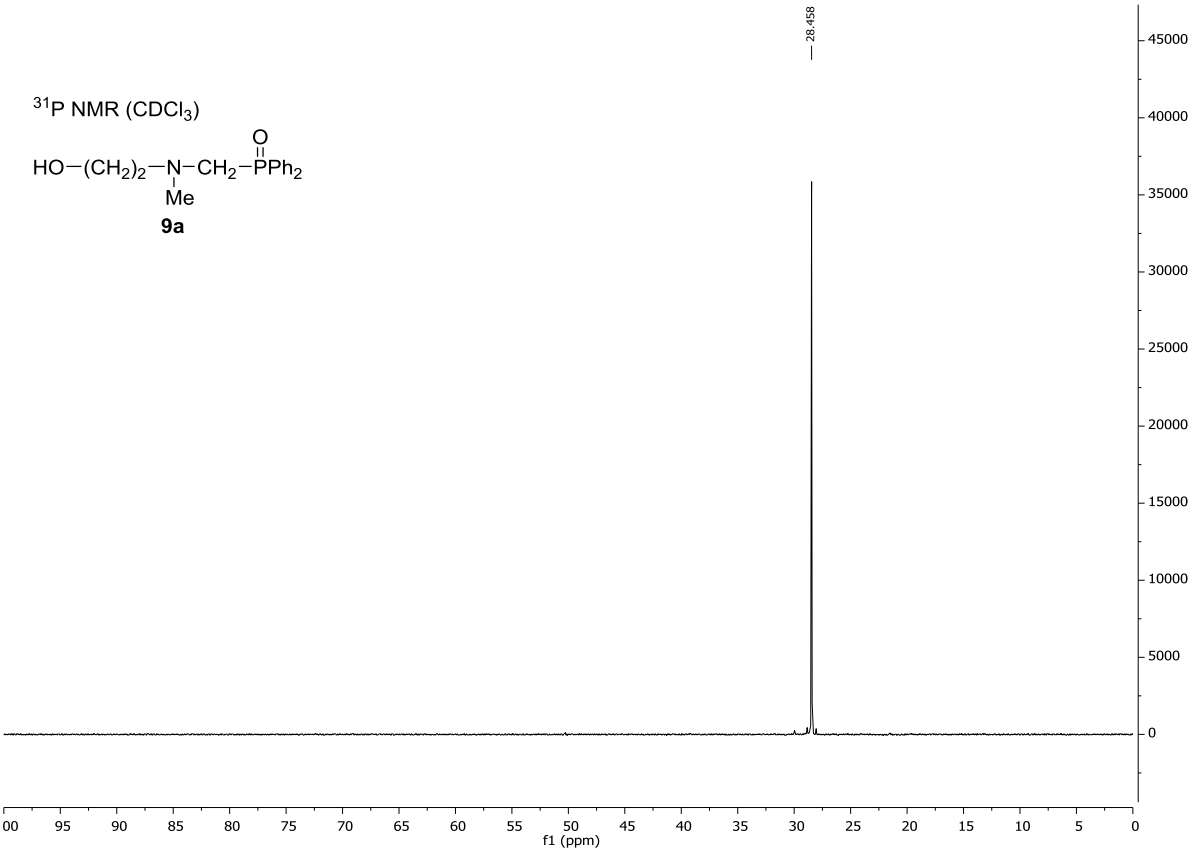
8c

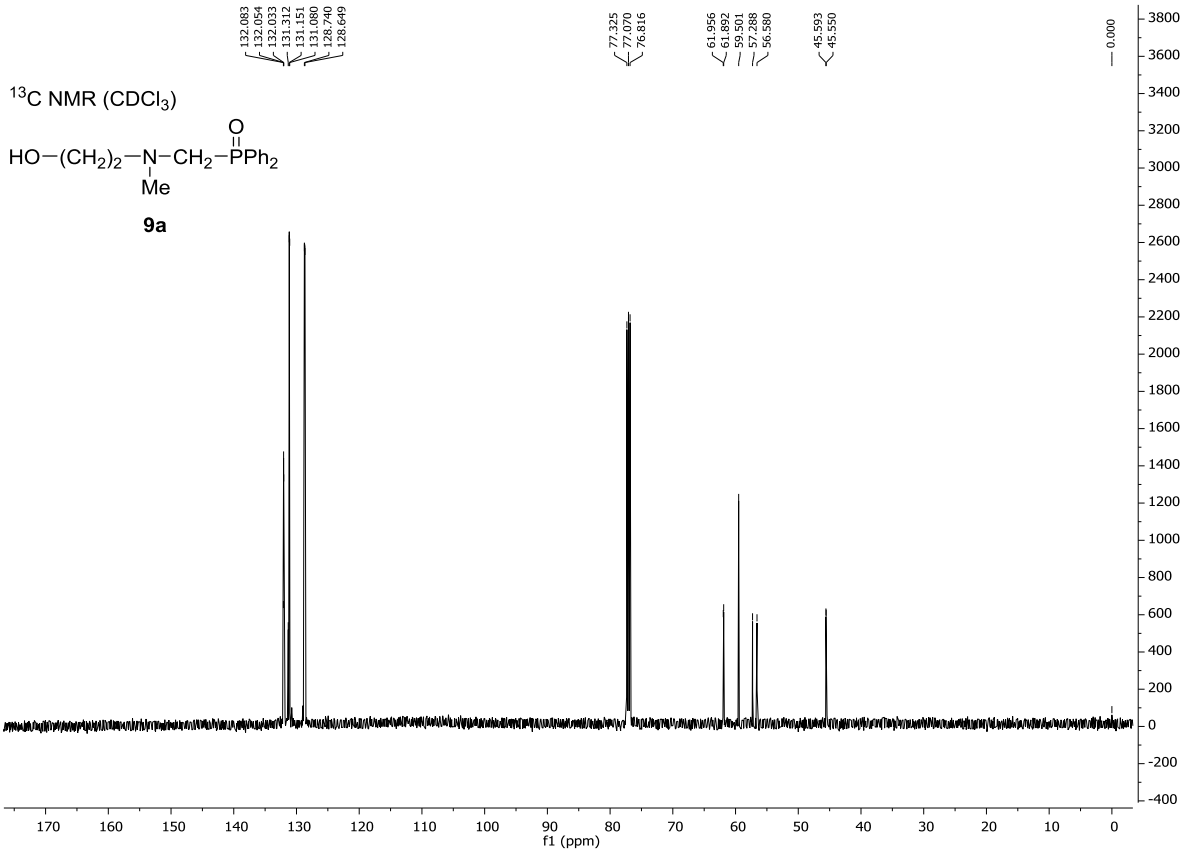
^{13}C NMR ($CDCl_3$)

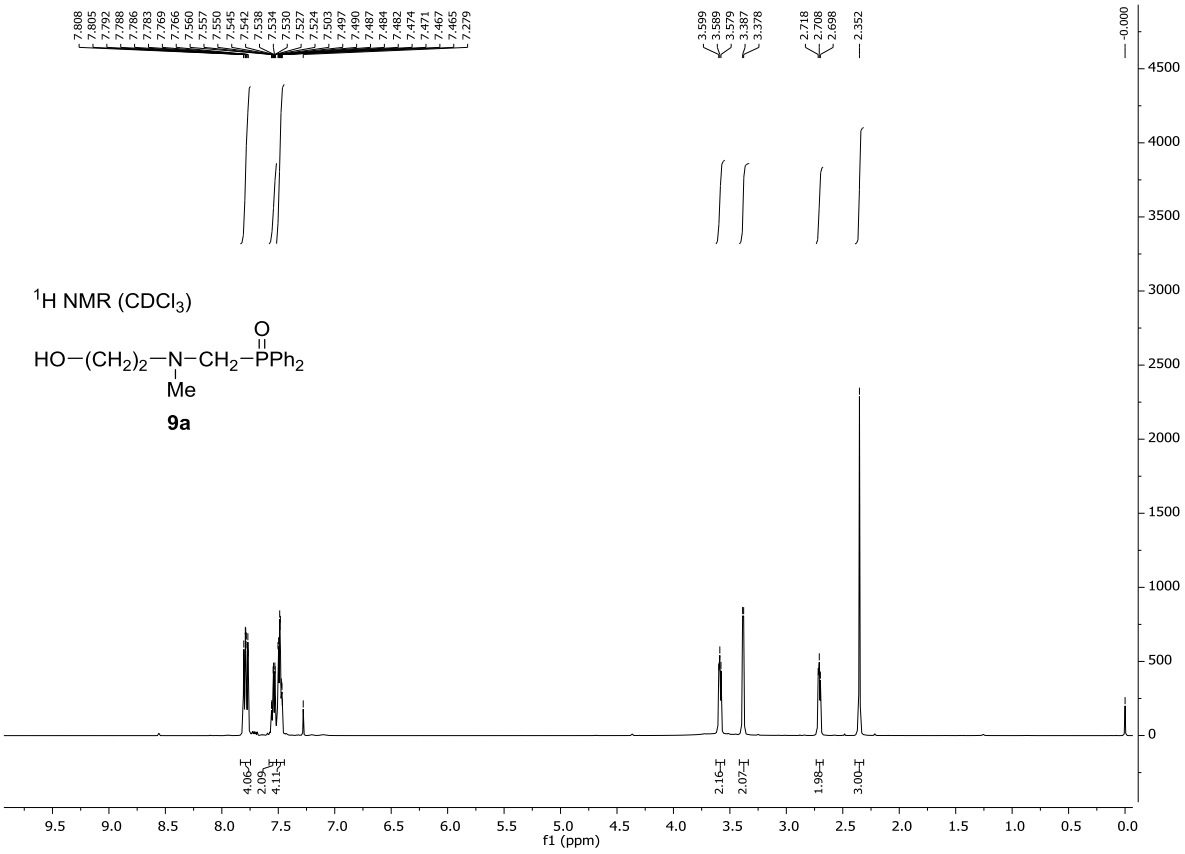


8c

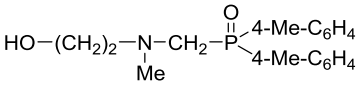

7.379
7.340
7.272
7.156
3.619
3.604
3.587
3.504
3.488
3.482
2.888
2.871
2.855
2.521
2.342
0.000
^{1}H NMR ($CDCl_3$)
HO−(CH$_2$)$_2$−N(H)−CH$_2$−P(=O)(3,5-(Me)$_2$-C$_6$H$_3$)$_2$
8c
4.00
2.01
2.01
2.10
2.00
1.49
12.38
f1 (ppm)


28.458
^{31}P NMR ($CDCl_3$)
HO−(CH$_2$)$_2$−N(Me)−CH$_2$−P(=O)Ph$_2$
9a
f1 (ppm)


132.083
132.054
132.033
131.312
131.151
131.080
128.740
128.649
77.325
77.070
76.816
61.956
61.892
59.501
57.288
56.580
45.593
45.550
0.000
13C NMR (CDCl3)
HO−(CH2)2−N(Me)−CH2−P(=O)Ph2
9a
f1 (ppm)

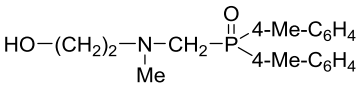

7.808
7.805
7.792
7.788
7.786
7.783
7.769
7.766
7.560
7.557
7.550
7.545
7.542
7.538
7.534
7.530
7.527
7.524
7.503
7.497
7.490
7.487
7.484
7.482
7.474
7.471
7.467
7.465
7.279
3.599
3.589
3.579
3.387
3.378
2.718
2.708
2.698
2.352
-0.000
1H NMR (CDCl3)
HO−(CH2)2−N(Me)−CH2−P(=O)Ph2
9a
4.06
2.09
4.11
2.16
2.07
1.98
3.00
f1 (ppm)

^{31}P NMR ($CDCl_3$)

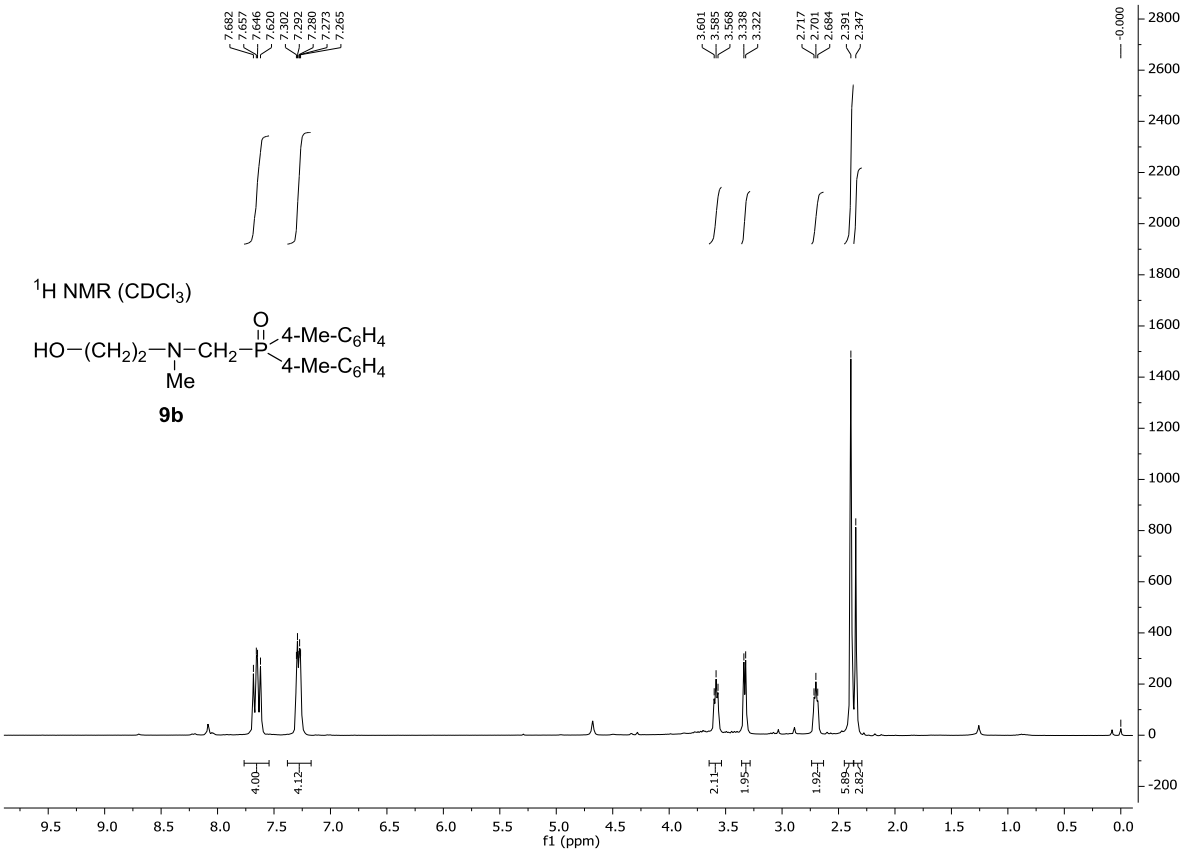


9b

^{13}C NMR ($CDCl_3$)

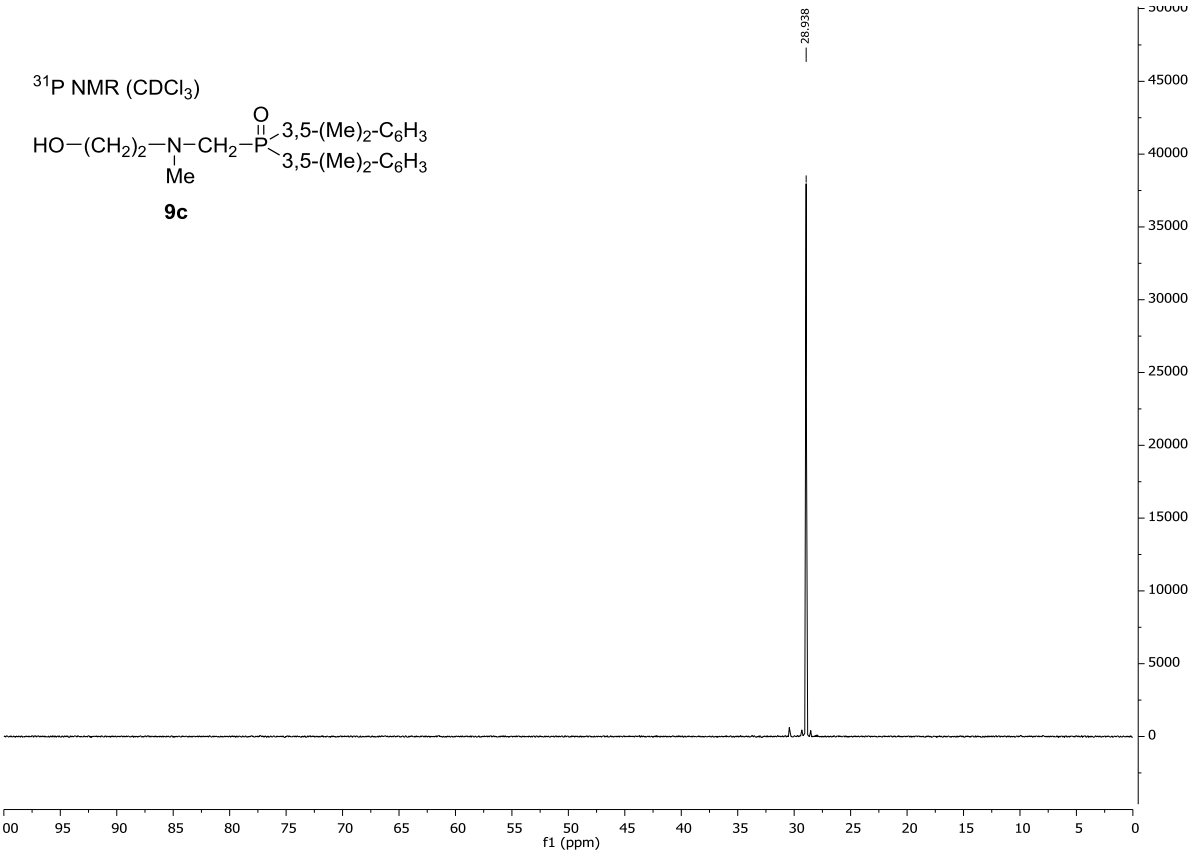


9b



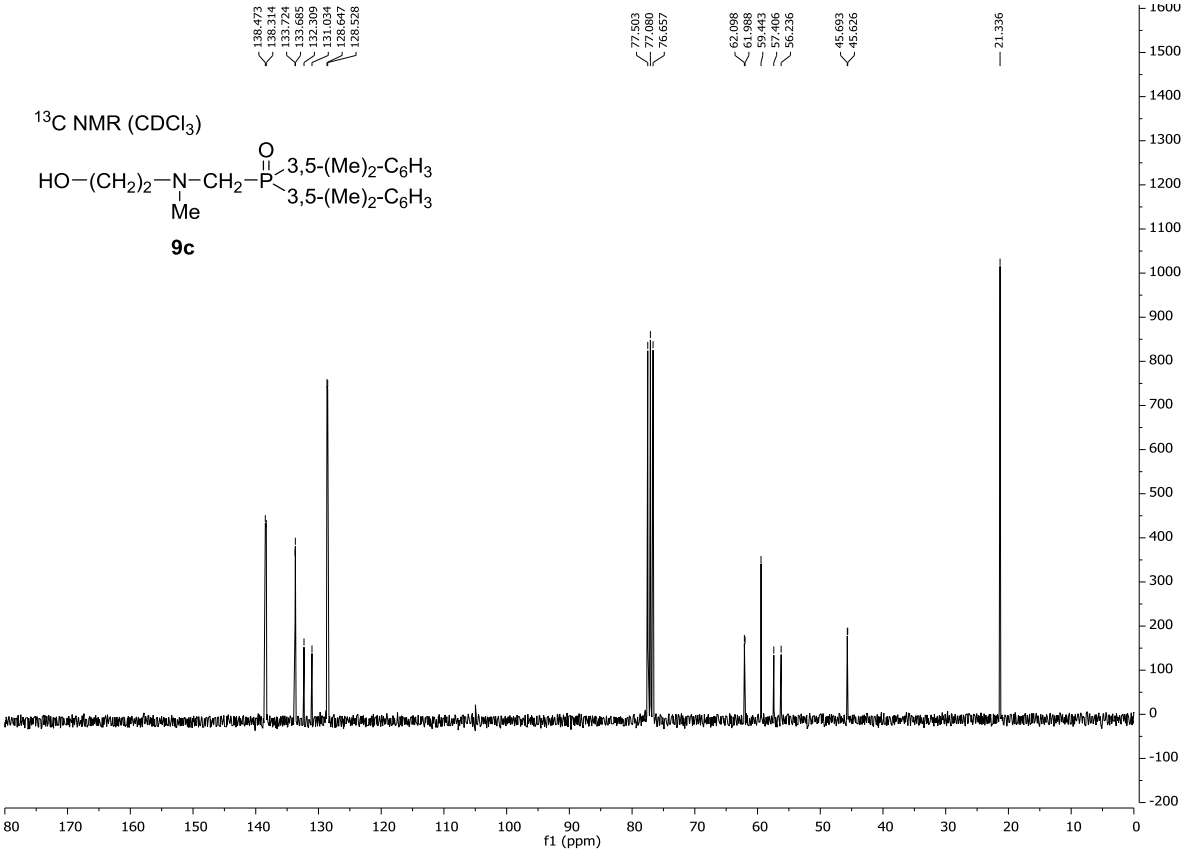
^{1}H NMR ($CDCl_3$)

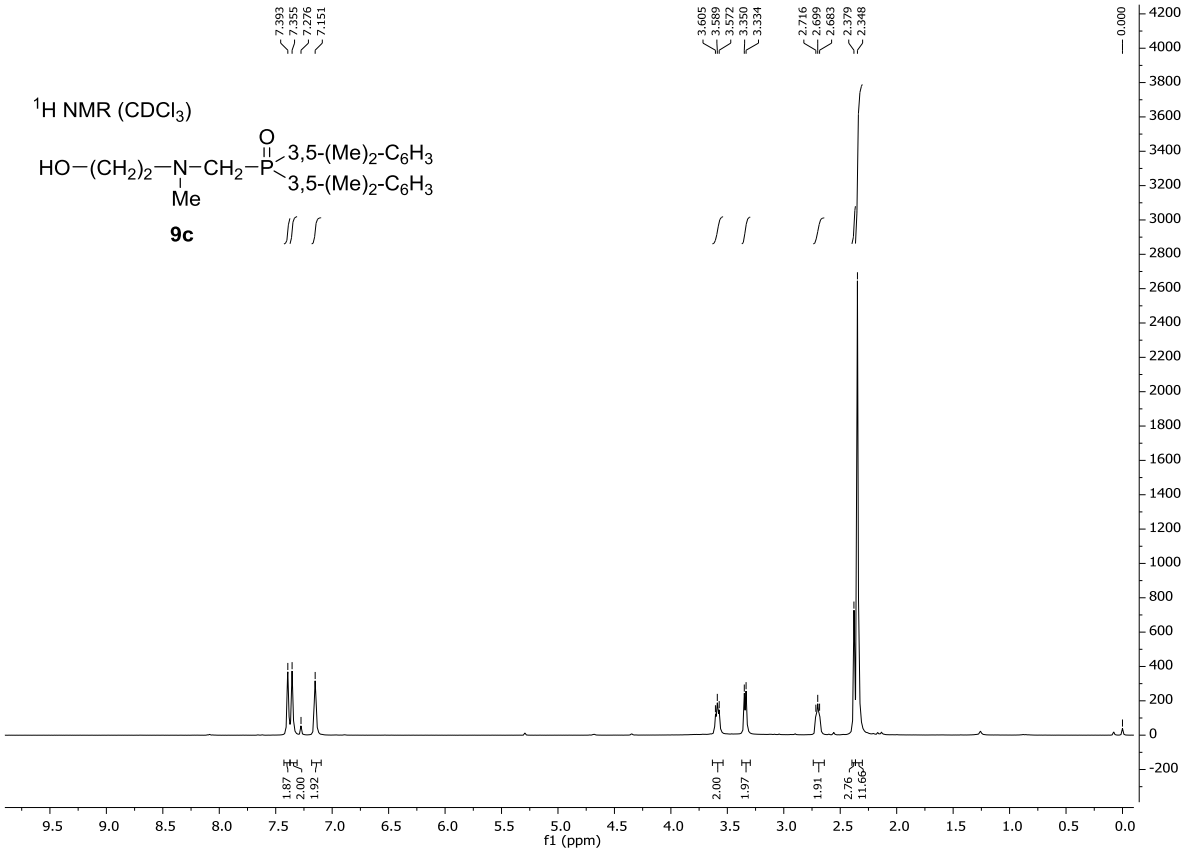
9b



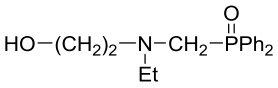
^{31}P NMR ($CDCl_3$)

9c


138.473
138.314
133.724
133.685
132.309
131.034
128.647
128.528
77.503
77.080
76.657
62.098
61.988
59.443
57.406
56.236
45.693
45.626
21.336
13C NMR (CDCl3)
HO−(CH2)2−N(Me)−CH2−P(=O)(3,5-(Me)2-C6H3)(3,5-(Me)2-C6H3)
9c
f1 (ppm)

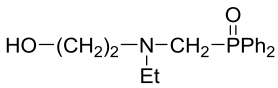

7.393
7.355
7.276
7.151
3.605
3.589
3.572
3.350
3.334
2.716
2.699
2.683
2.379
2.348
0.000
1H NMR (CDCl3)
HO−(CH2)2−N(Me)−CH2−P(=O)(3,5-(Me)2-C6H3)(3,5-(Me)2-C6H3)
9c
1.87
2.00
1.92
2.00
1.97
1.91
2.76
11.66
f1 (ppm)

^{31}P NMR ($CDCl_3$)

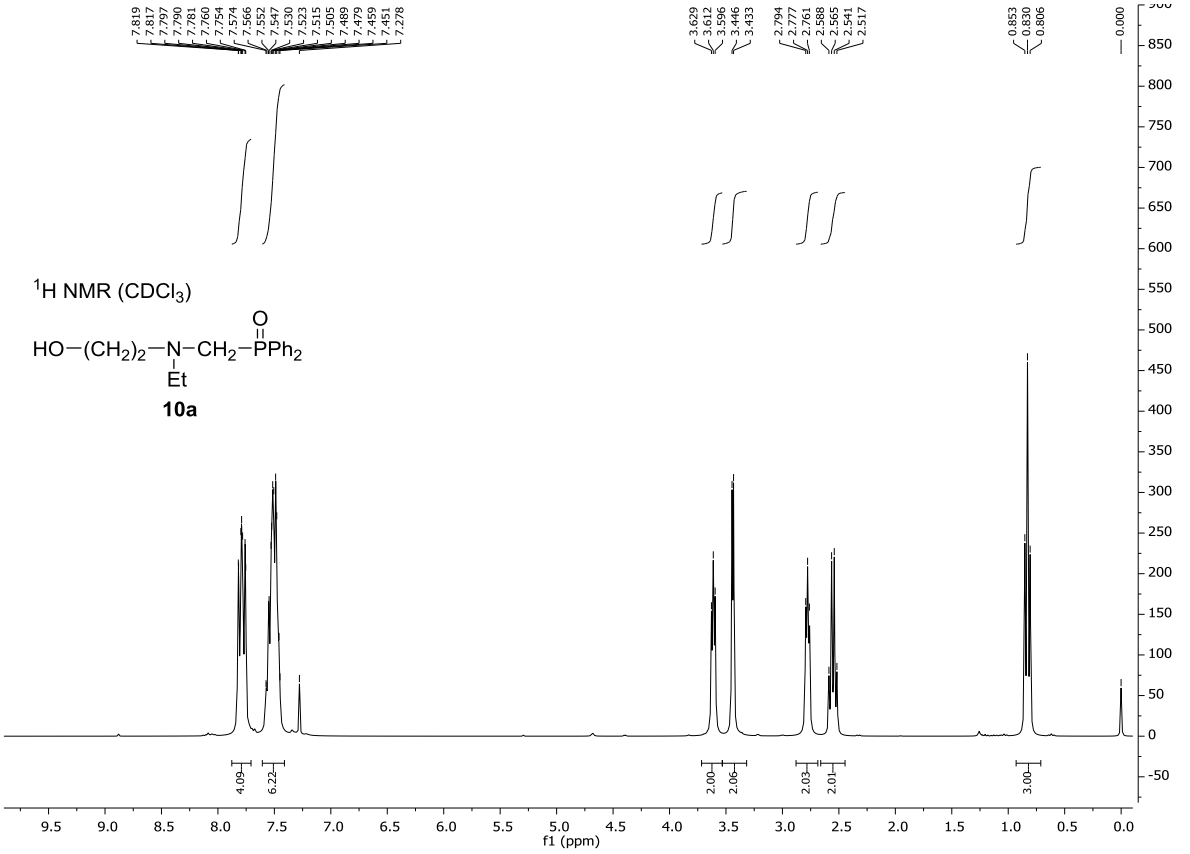


10a

^{13}C NMR ($CDCl_3$)

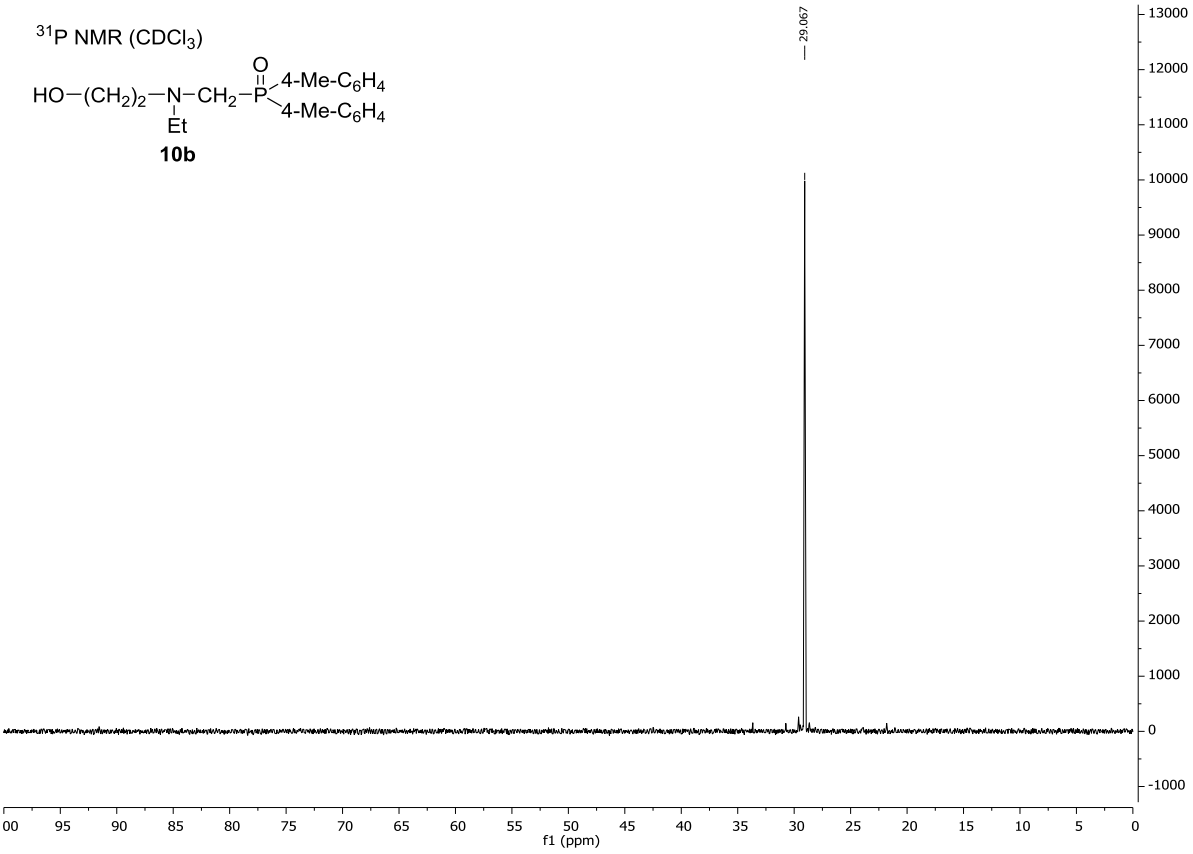


10a



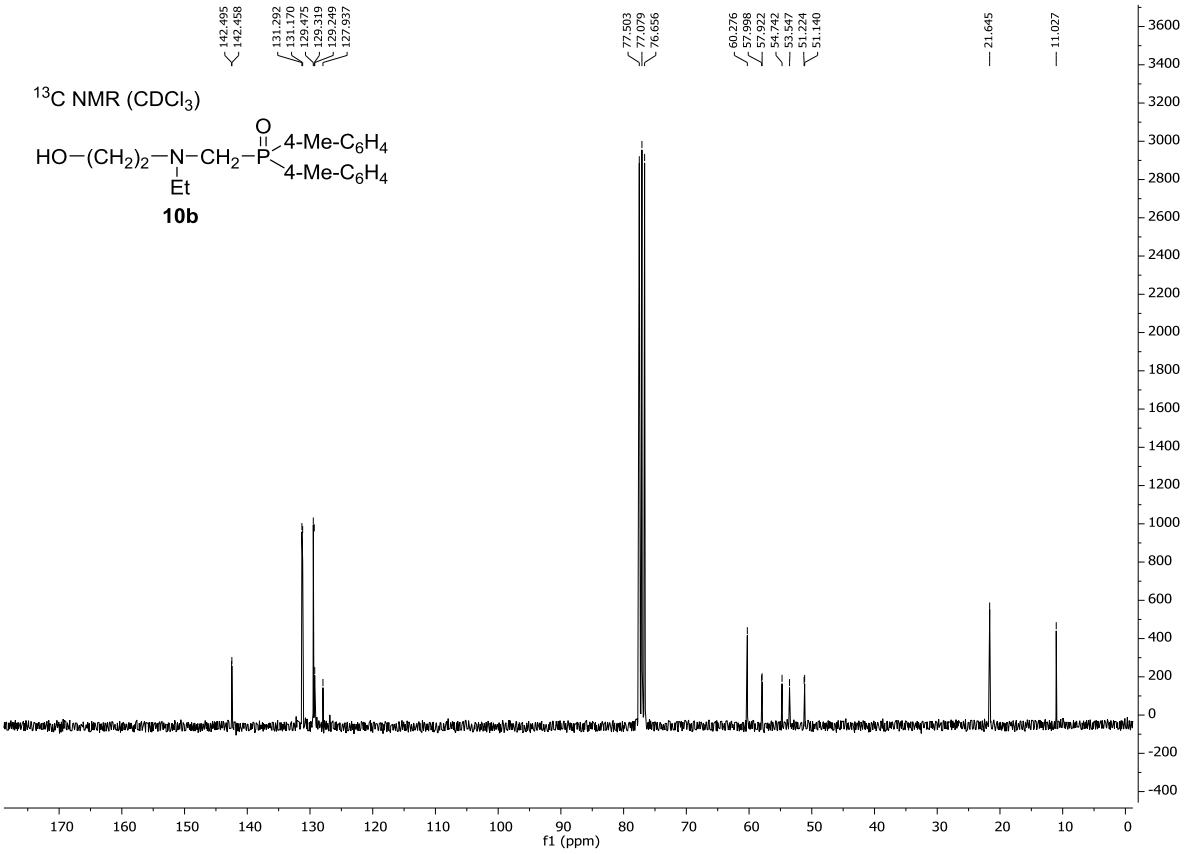
^{1}H NMR ($CDCl_3$)

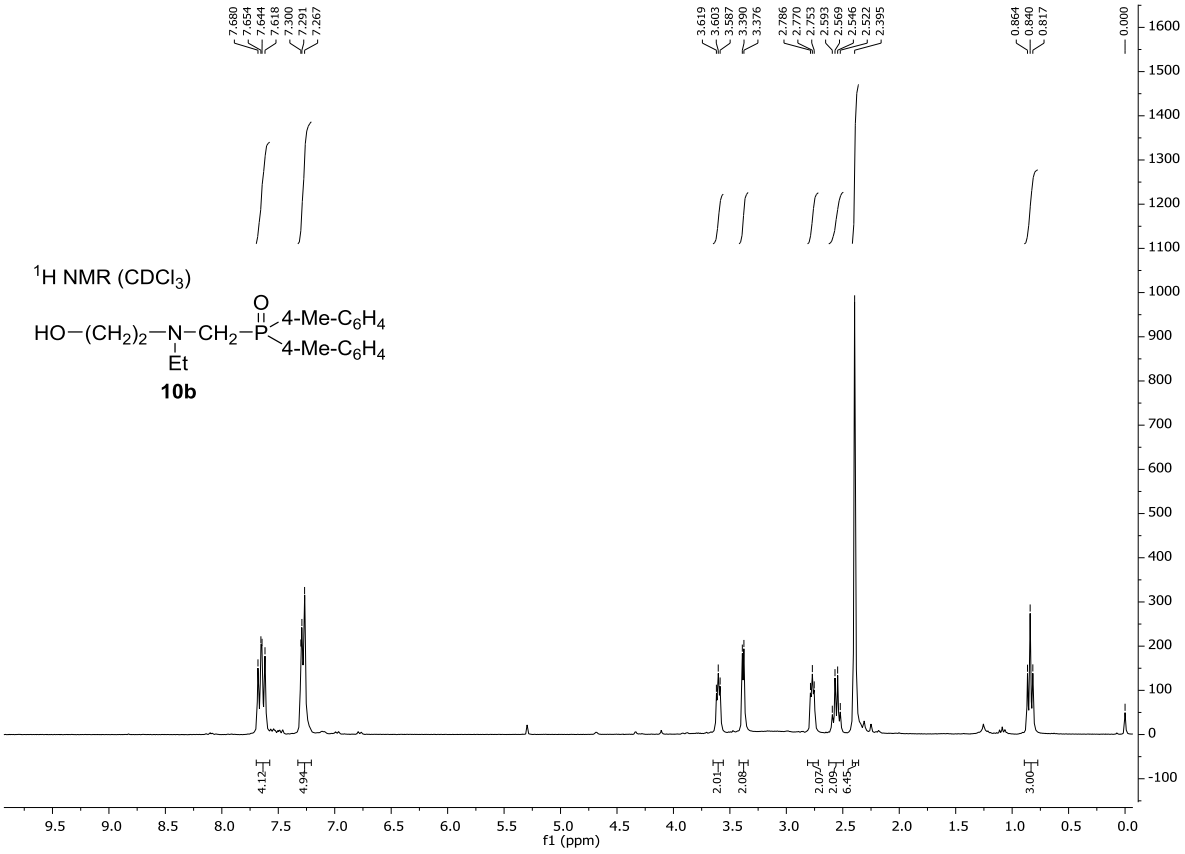
10a



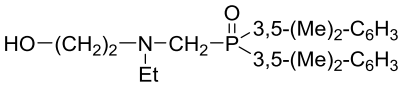
^{31}P NMR ($CDCl_3$)

10b


142.495
142.458
131.292
131.170
129.475
129.316
129.249
127.937
77.503
77.079
76.656
60.276
57.998
57.922
54.742
53.547
51.224
51.140
21.645
11.027
13C NMR (CDCl3)
HO−(CH2)2−N(Et)−CH2−P(=O)(4-Me-C6H4)(4-Me-C6H4)
10b
f1 (ppm)

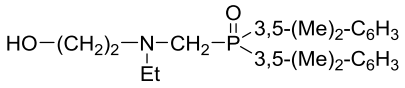

7.680
7.654
7.644
7.618
7.300
7.291
7.267
3.619
3.603
3.587
3.390
3.376
2.786
2.770
2.753
2.593
2.569
2.546
2.522
2.395
0.864
0.840
0.817
0.000
1H NMR (CDCl3)
HO−(CH2)2−N(Et)−CH2−P(=O)(4-Me-C6H4)(4-Me-C6H4)
10b
4.12
4.94
2.01
2.08
2.07
2.09
6.45
3.00
f1 (ppm)

^{31}P NMR ($CDCl_3$)

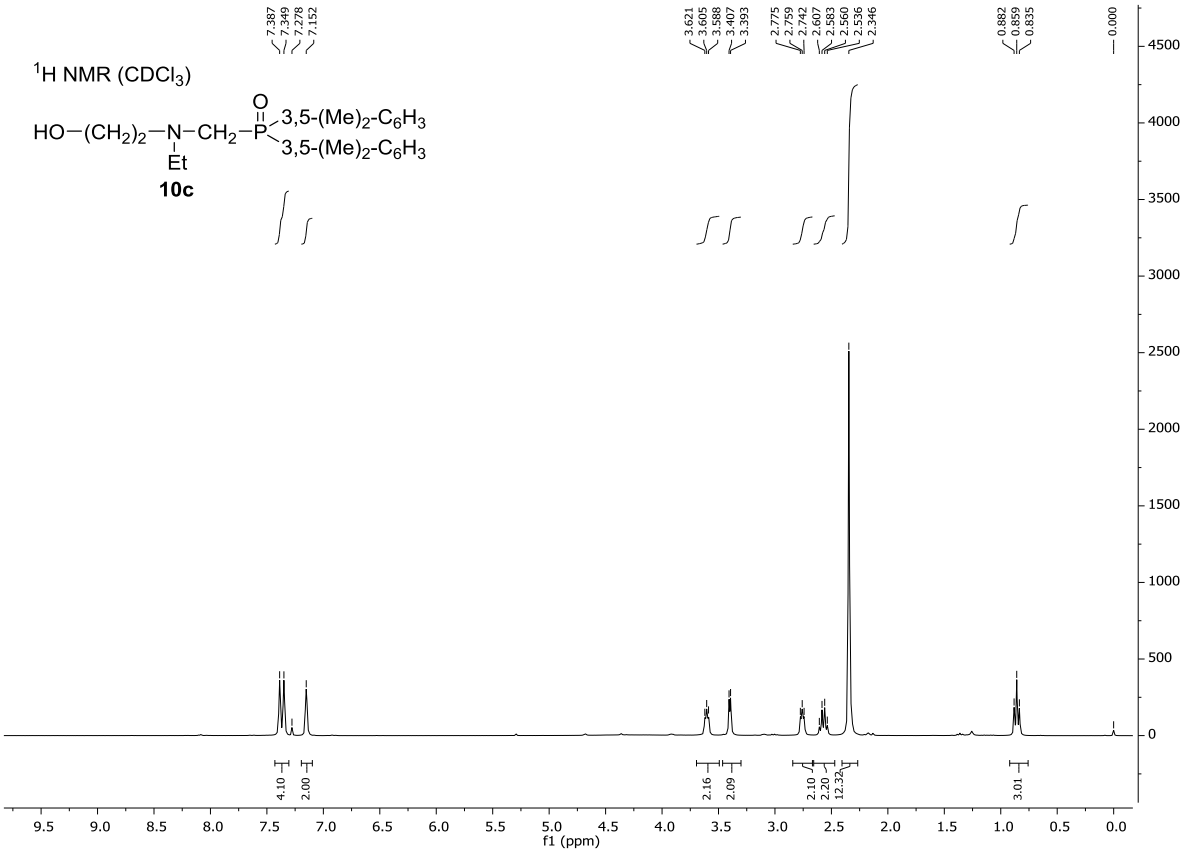


10c

^{13}C NMR ($CDCl_3$)

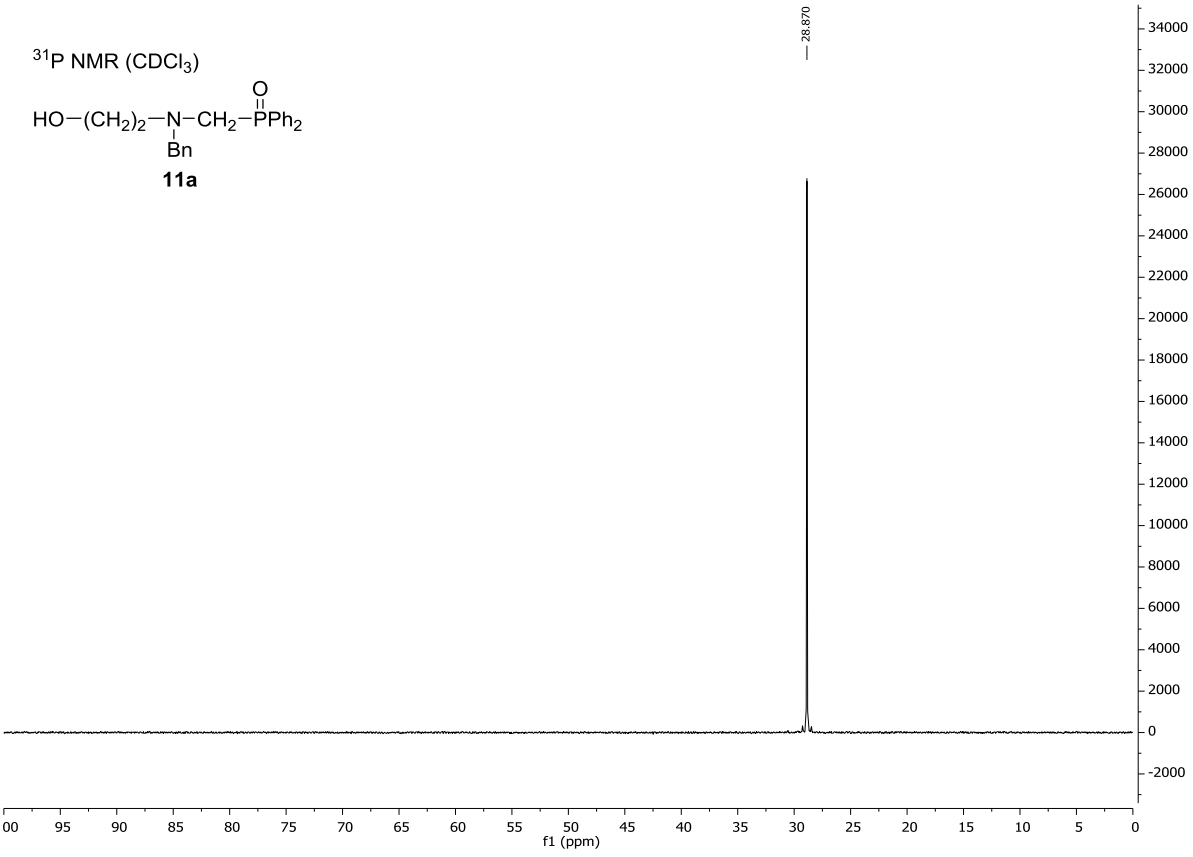


10c



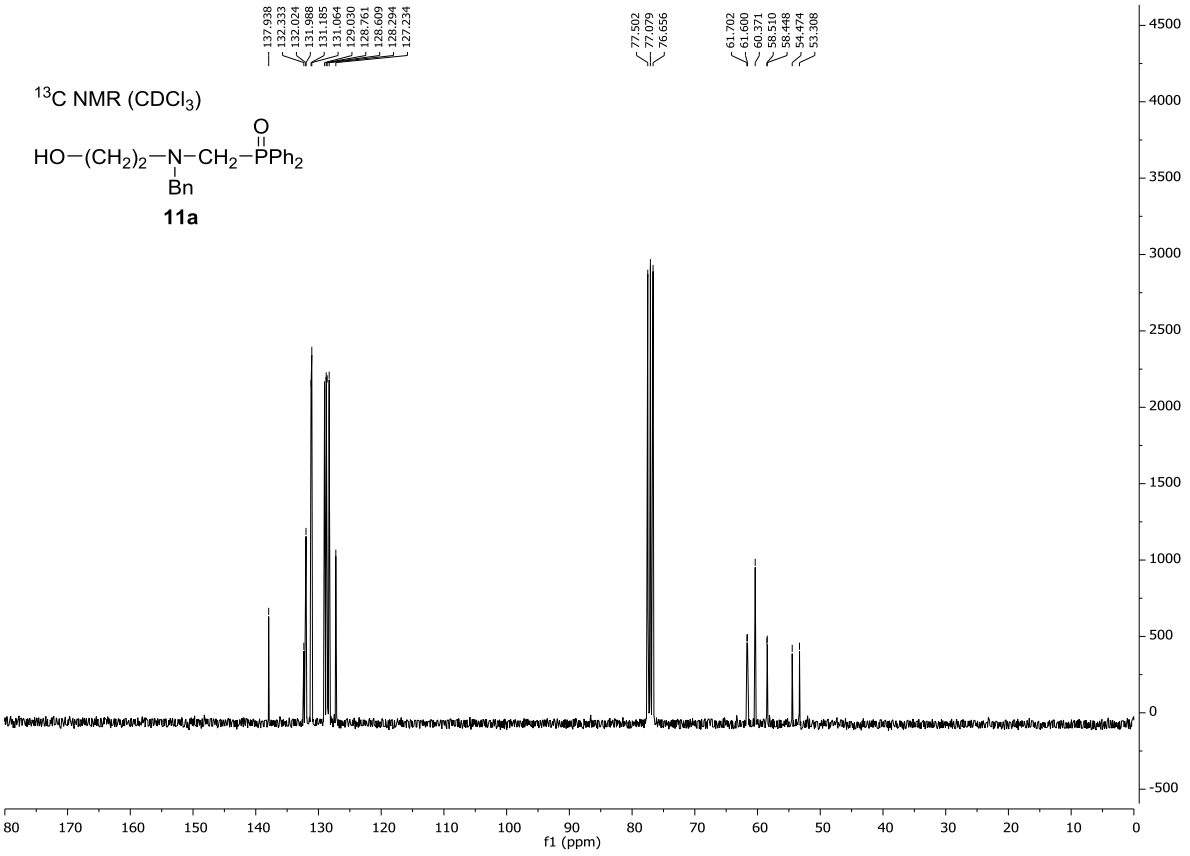
^{1}H NMR ($CDCl_3$)

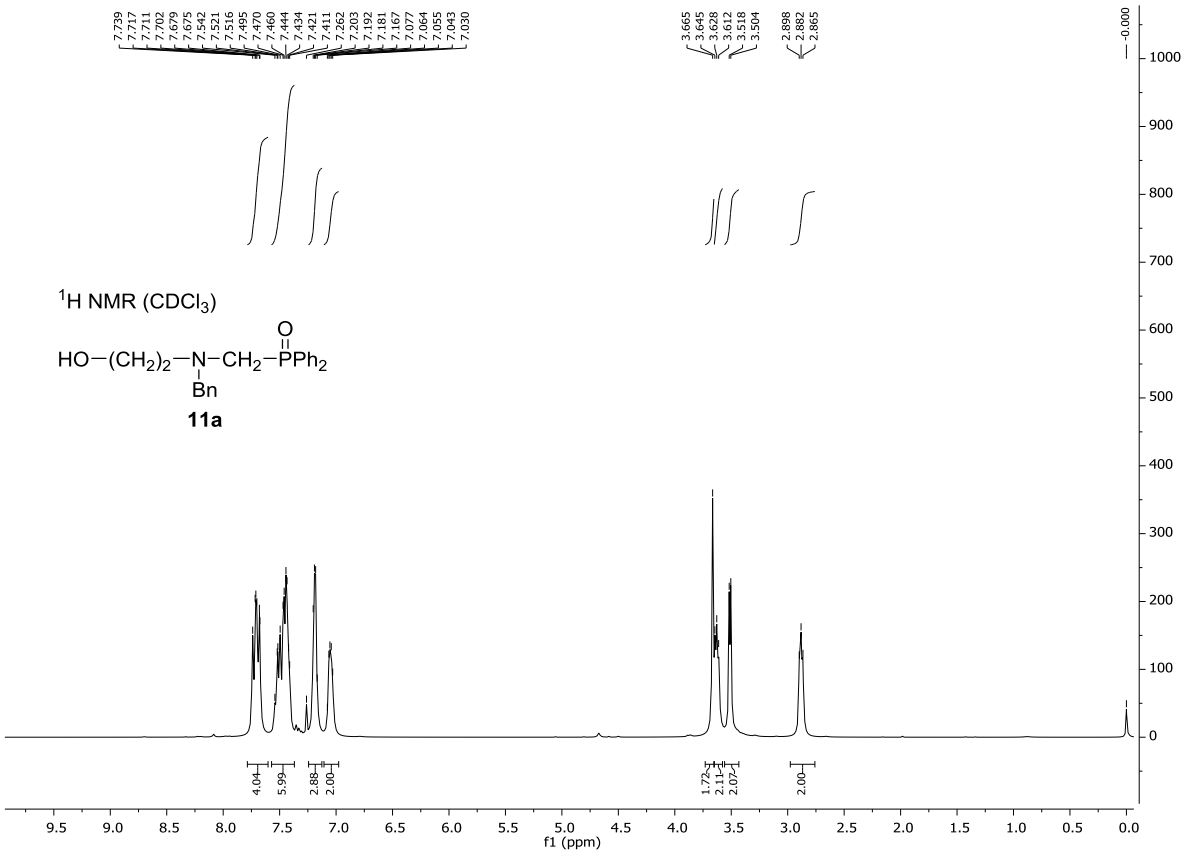
10c



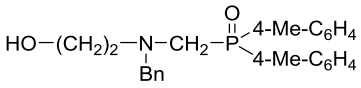
^{31}P NMR ($CDCl_3$)

11a


137.938
132.333
132.024
131.988
131.185
131.064
129.030
128.761
128.609
128.294
127.234
77.502
77.079
76.656
61.702
61.600
60.371
58.510
58.448
54.474
53.308
13C NMR (CDCl3)
HO−(CH2)2−N(Bn)−CH2−P(=O)Ph2
11a
f1 (ppm)

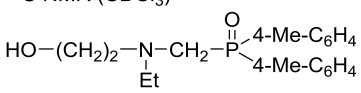

7.739
7.717
7.711
7.702
7.679
7.675
7.542
7.521
7.516
7.495
7.470
7.460
7.444
7.434
7.421
7.411
7.262
7.203
7.192
7.181
7.167
7.077
7.064
7.055
7.043
7.030
3.665
3.645
3.628
3.612
3.518
3.504
2.898
2.882
2.865
-0.000
1H NMR (CDCl3)
HO−(CH2)2−N(Bn)−CH2−P(=O)Ph2
11a
4.04
5.99
2.88
2.00
1.72
2.11
2.07
2.00
f1 (ppm)

^{31}P NMR ($CDCl_3$)

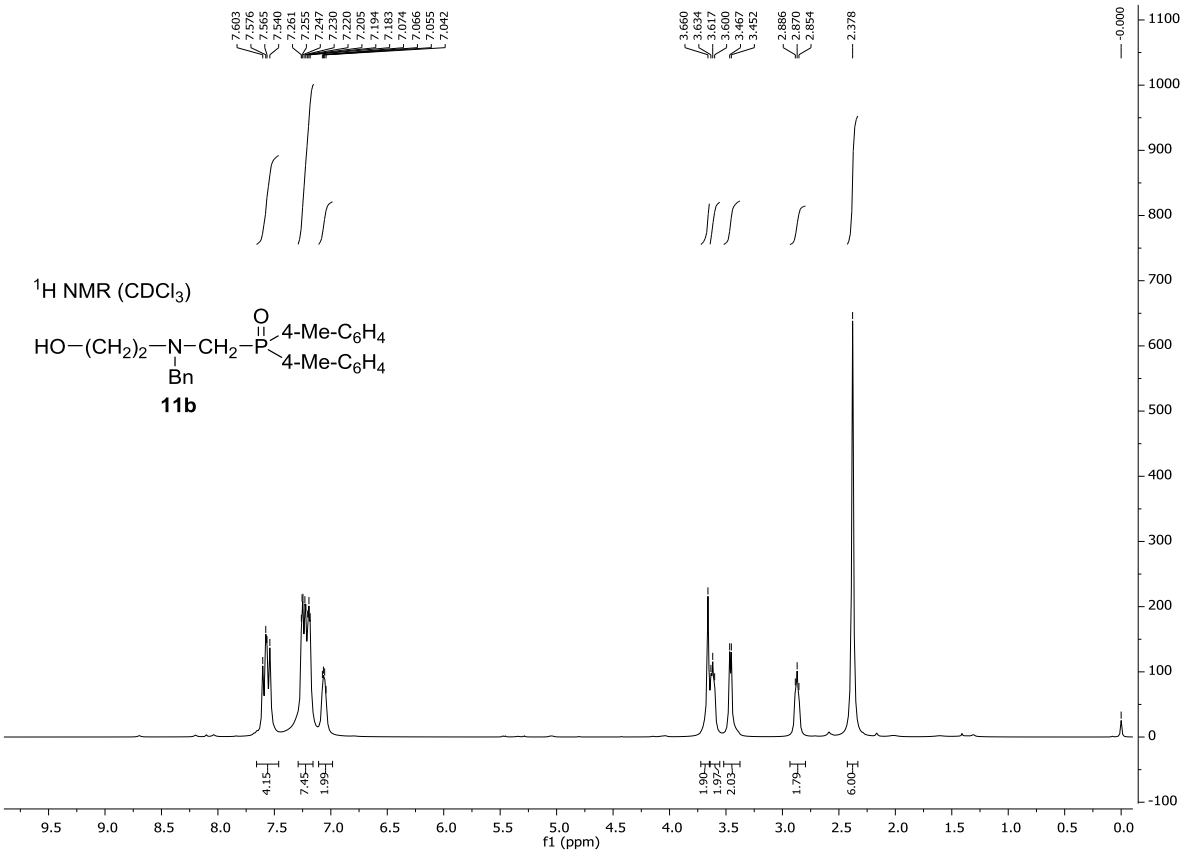


11b

^{13}C NMR ($CDCl_3$)



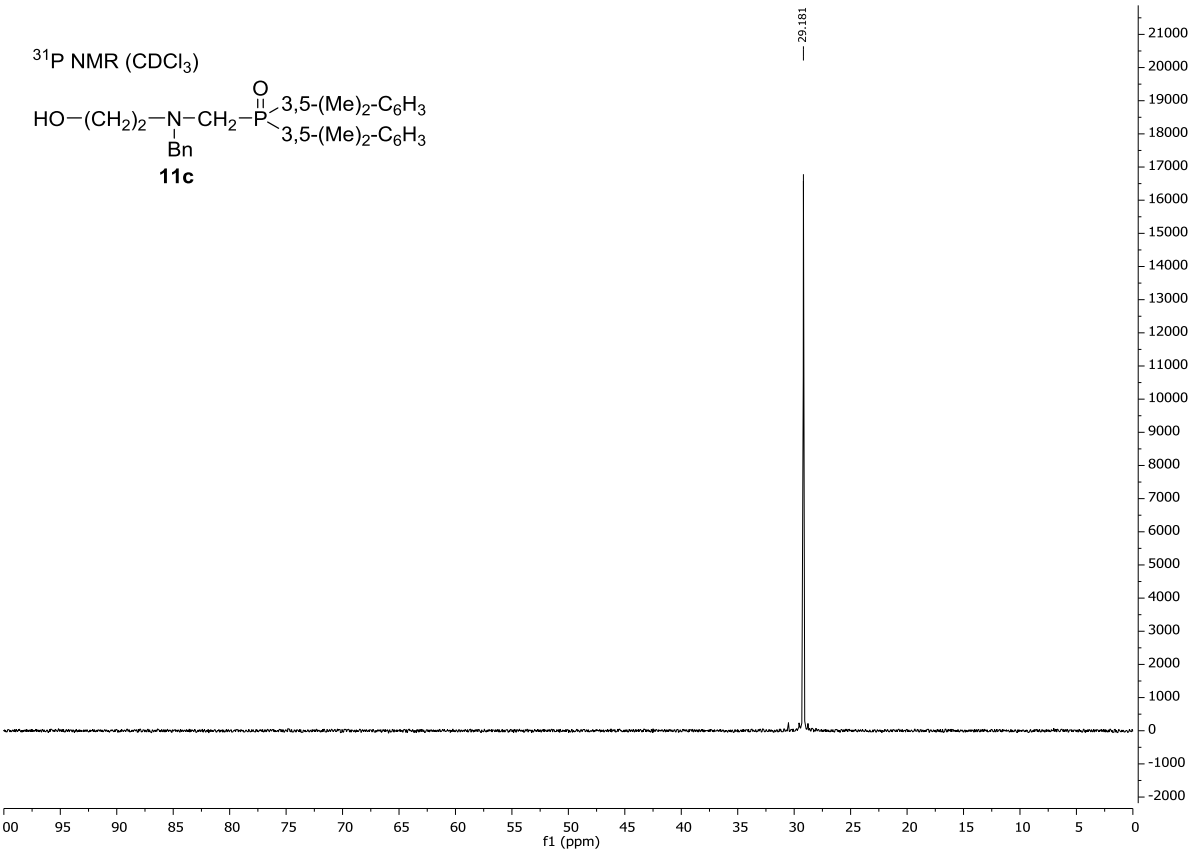
11b



^{1}H NMR ($CDCl_3$)

HO−$(CH_2)_2$−N(Bn)−CH_2−P(=O)(4-Me-C_6H_4)(4-Me-C_6H_4)

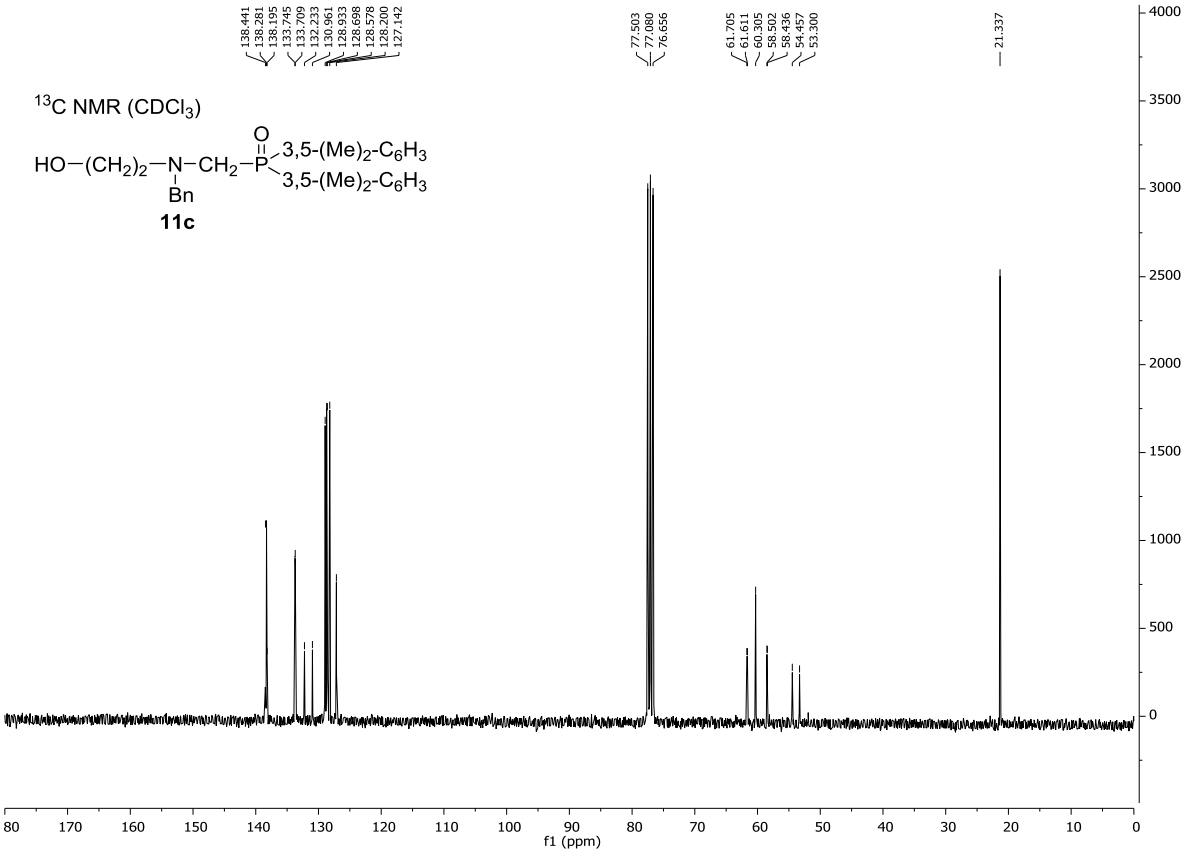
11b

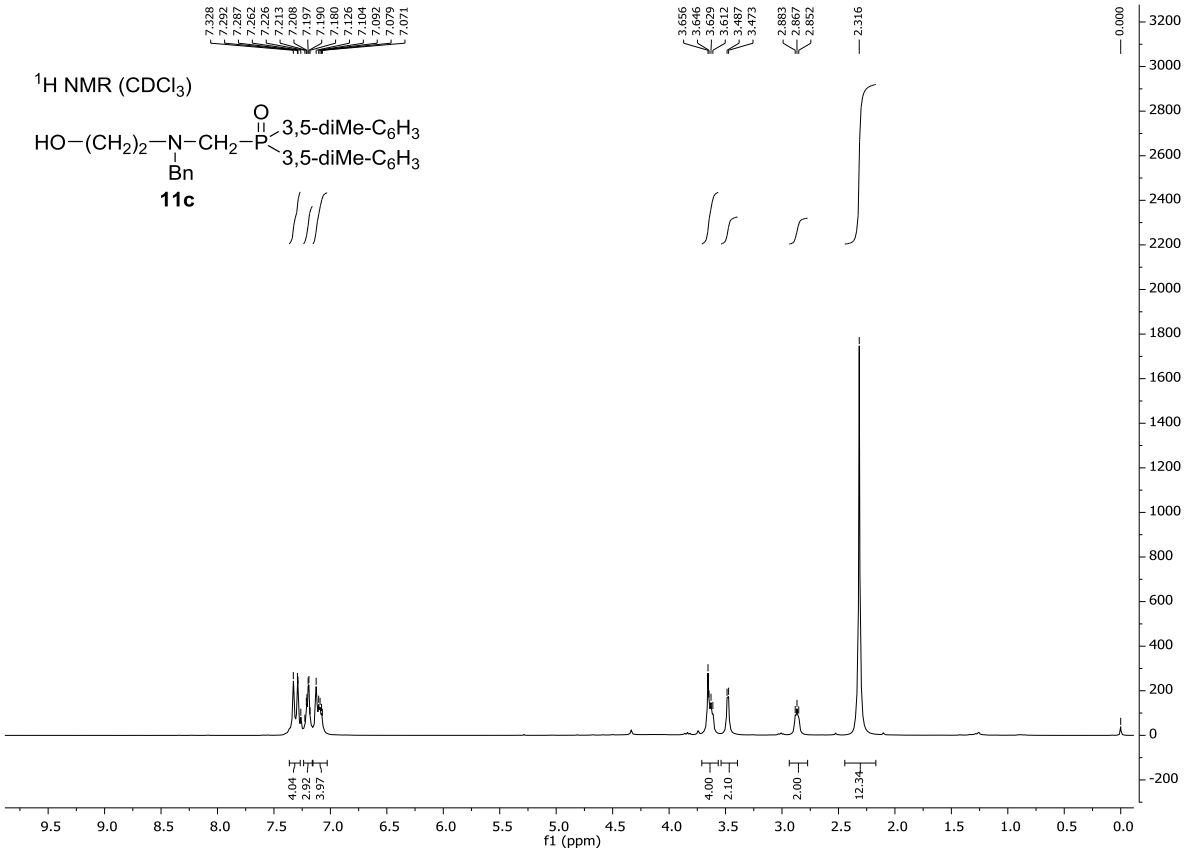


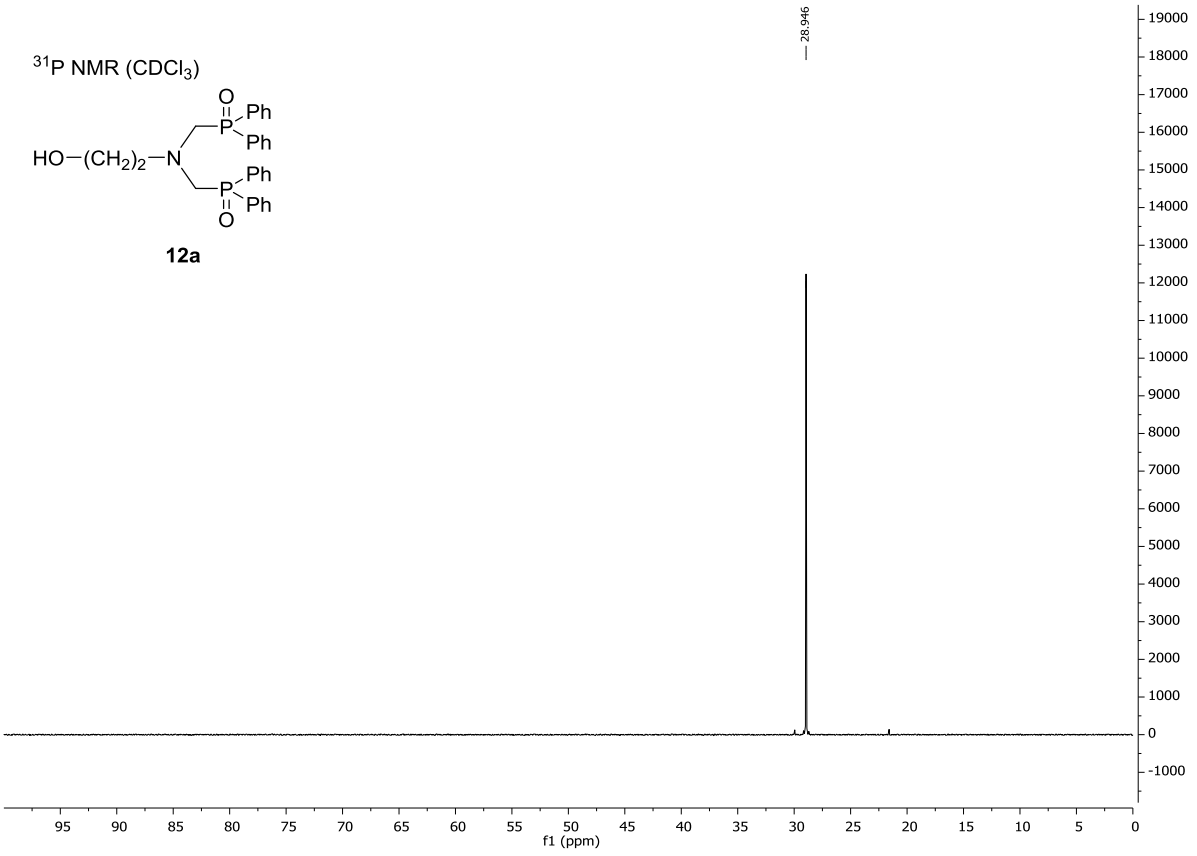
^{31}P NMR ($CDCl_3$)

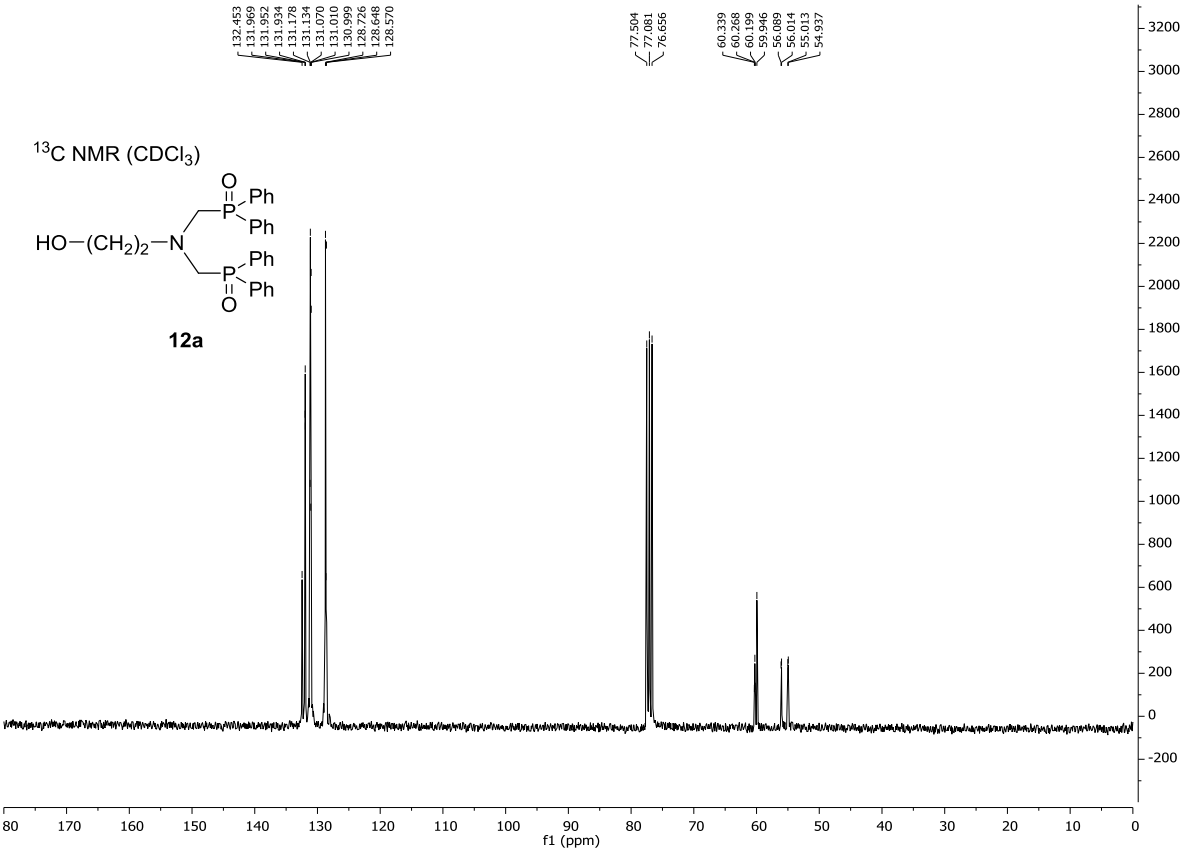
HO−$(CH_2)_2$−N(Bn)−CH_2−P(=O)(3,5-$(Me)_2$-C_6H_3)(3,5-$(Me)_2$-C_6H_3)

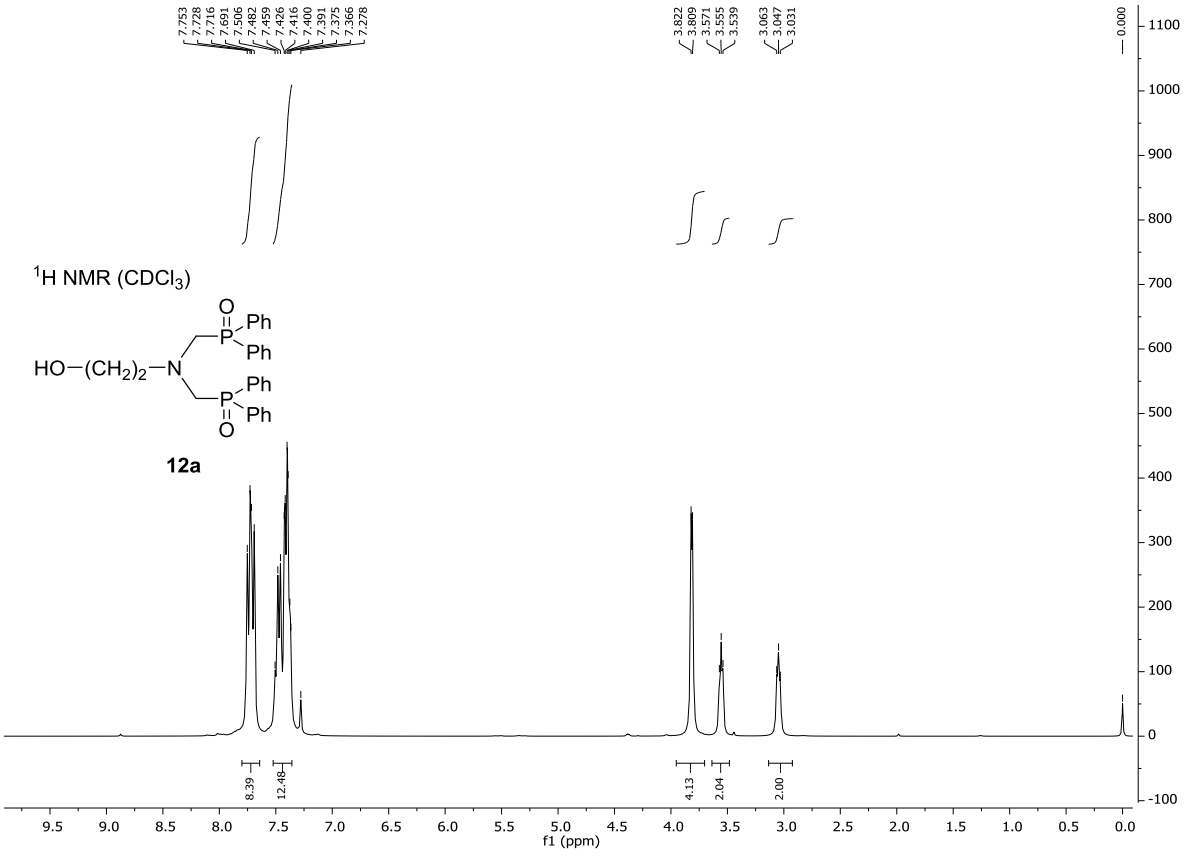
11c

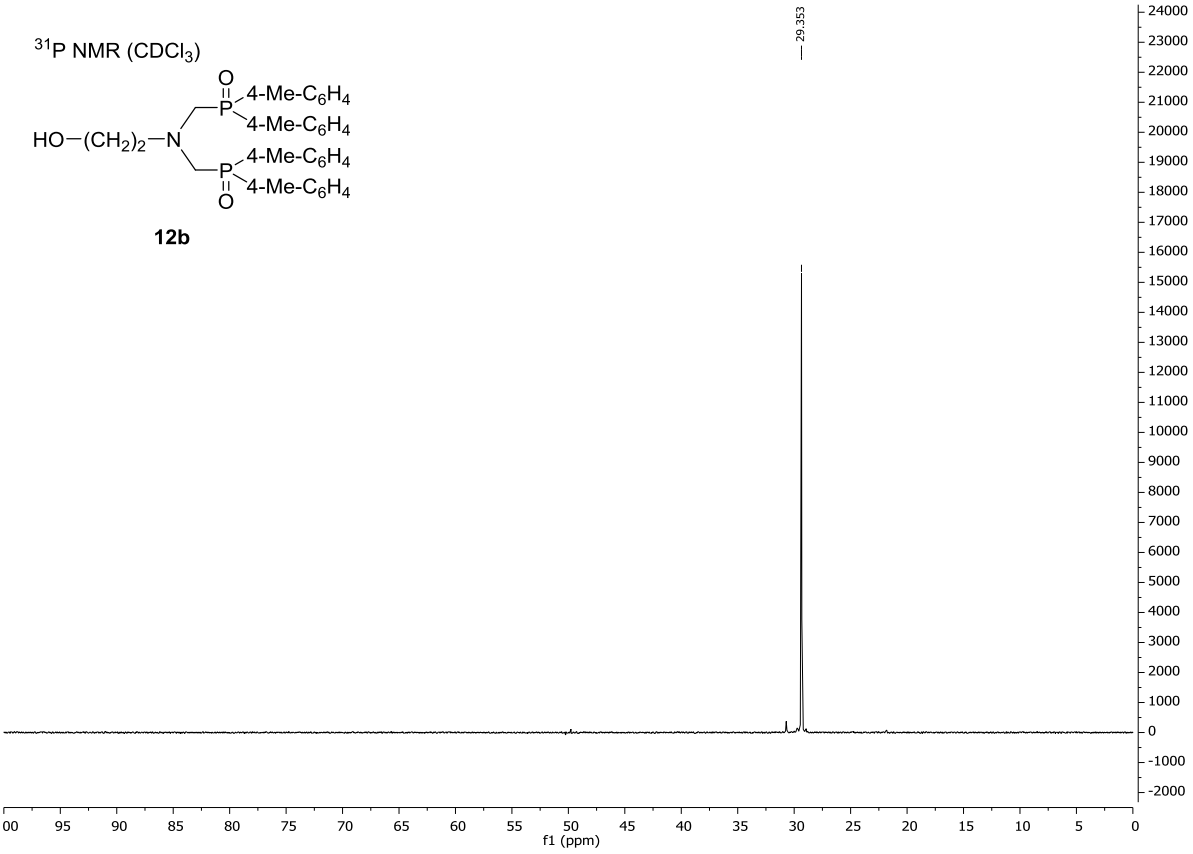

138.441
138.281
138.195
133.745
133.709
132.233
130.961
128.933
128.698
128.578
128.200
127.142
77.503
77.080
76.656
61.705
61.611
60.305
58.502
58.436
54.457
53.300
21.337
^{13}C NMR ($CDCl_3$)
HO−(CH2)2−N(Bn)−CH2−P(=O)(3,5-(Me)2-C6H3)(3,5-(Me)2-C6H3)
11c
f1 (ppm)

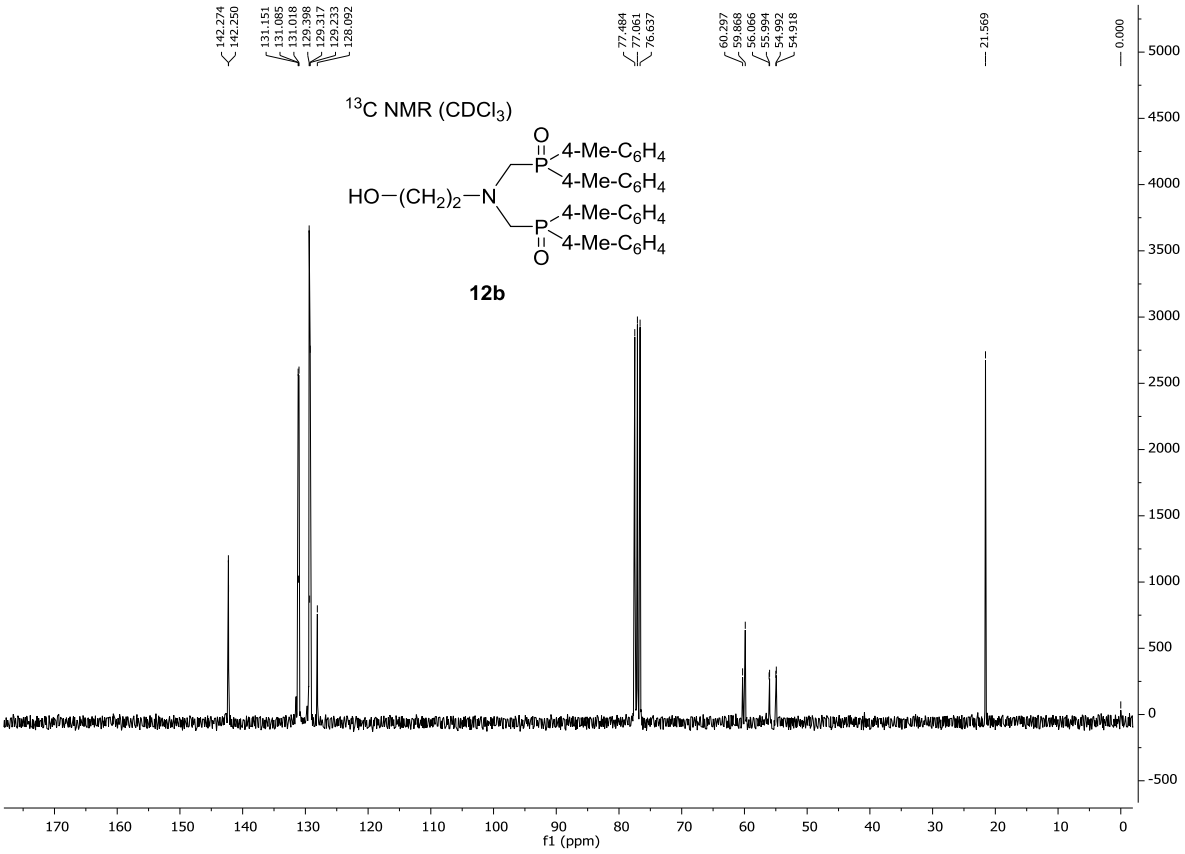

7.328
7.292
7.287
7.262
7.226
7.213
7.208
7.197
7.190
7.180
7.126
7.104
7.092
7.079
7.071
3.656
3.646
3.629
3.612
3.487
3.473
2.883
2.867
2.852
2.316
0.000
^{1}H NMR ($CDCl_3$)
HO−(CH2)2−N(Bn)−CH2−P(=O)(3,5-diMe-C6H3)(3,5-diMe-C6H3)
11c
4.04
2.92
3.97
4.00
2.10
2.00
12.34
f1 (ppm)

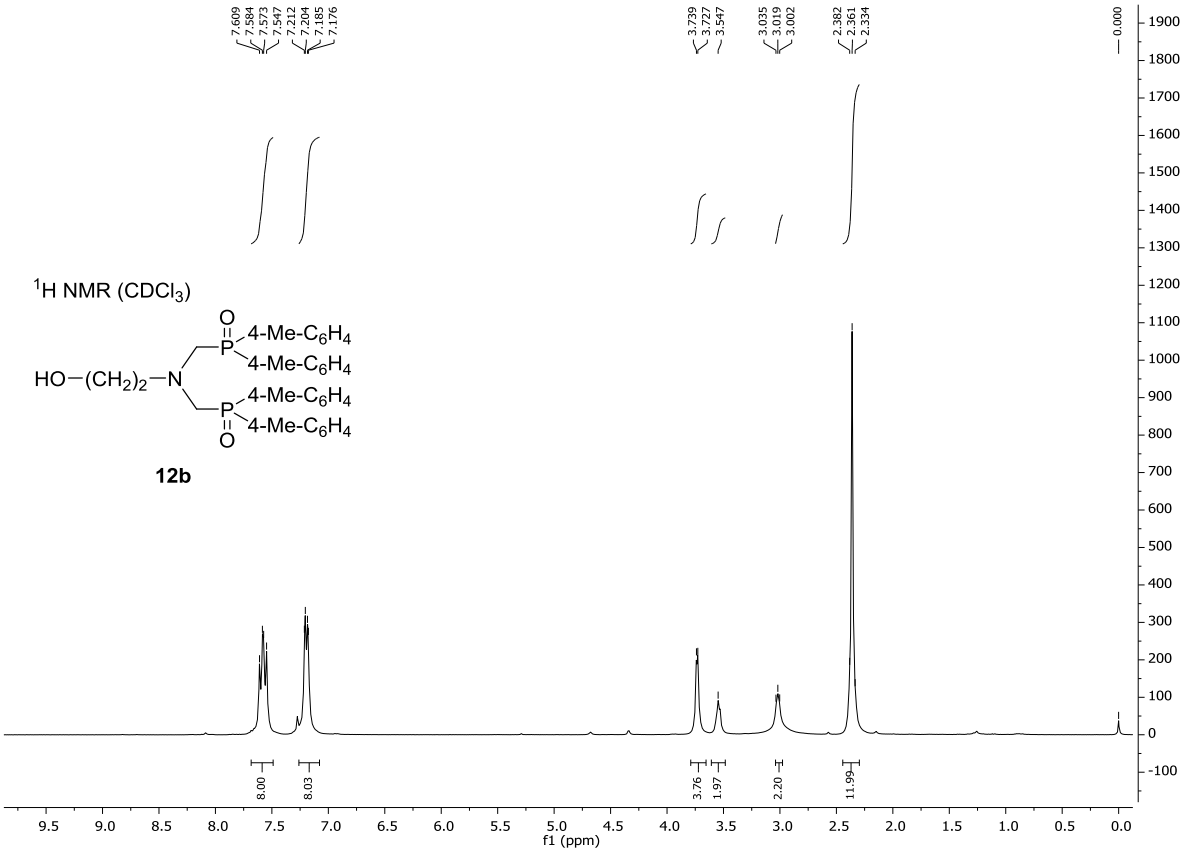

31P NMR (CDCl3)
HO−(CH2)2−N
Ph
Ph
Ph
Ph
O
P
12a
28.946
f1 (ppm)


13C NMR (CDCl3)
HO−(CH2)2−N
Ph
Ph
Ph
Ph
O
P
12a
132.453
131.969
131.952
131.934
131.178
131.134
131.070
131.010
130.999
128.726
128.648
128.570
77.504
77.081
76.656
60.339
60.268
60.199
59.946
56.089
56.014
55.013
54.937
f1 (ppm)

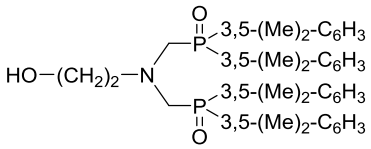

7.753
7.728
7.716
7.691
7.506
7.482
7.459
7.426
7.416
7.400
7.391
7.375
7.366
7.278
3.822
3.809
3.571
3.555
3.539
3.063
3.047
3.031
0.000
1H NMR (CDCl3)
HO−(CH2)2−N
Ph
Ph
Ph
Ph
12a
8.39
12.48
4.13
2.04
2.00
f1 (ppm)


29.353
31P NMR (CDCl3)
HO−(CH2)2−N
4-Me-C6H4
4-Me-C6H4
4-Me-C6H4
4-Me-C6H4
12b
f1 (ppm)


142.274
142.250
131.151
131.085
131.018
129.398
129.317
129.233
128.092
77.484
77.061
76.637
60.297
59.868
56.066
55.994
54.992
54.918
21.569
0.000
13C NMR (CDCl3)
HO−(CH2)2−N
4-Me-C6H4
12b
f1 (ppm)

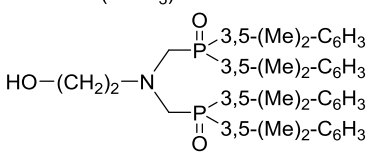

7.609
7.584
7.573
7.547
7.212
7.204
7.195
7.176
3.739
3.727
3.547
3.035
3.019
3.002
2.382
2.361
2.334
0.000
1H NMR (CDCl3)
HO−(CH2)2−N
4-Me-C6H4
12b
8.00
8.03
3.76
1.97
2.20
11.99
f1 (ppm)

^{31}P NMR ($CDCl_3$)

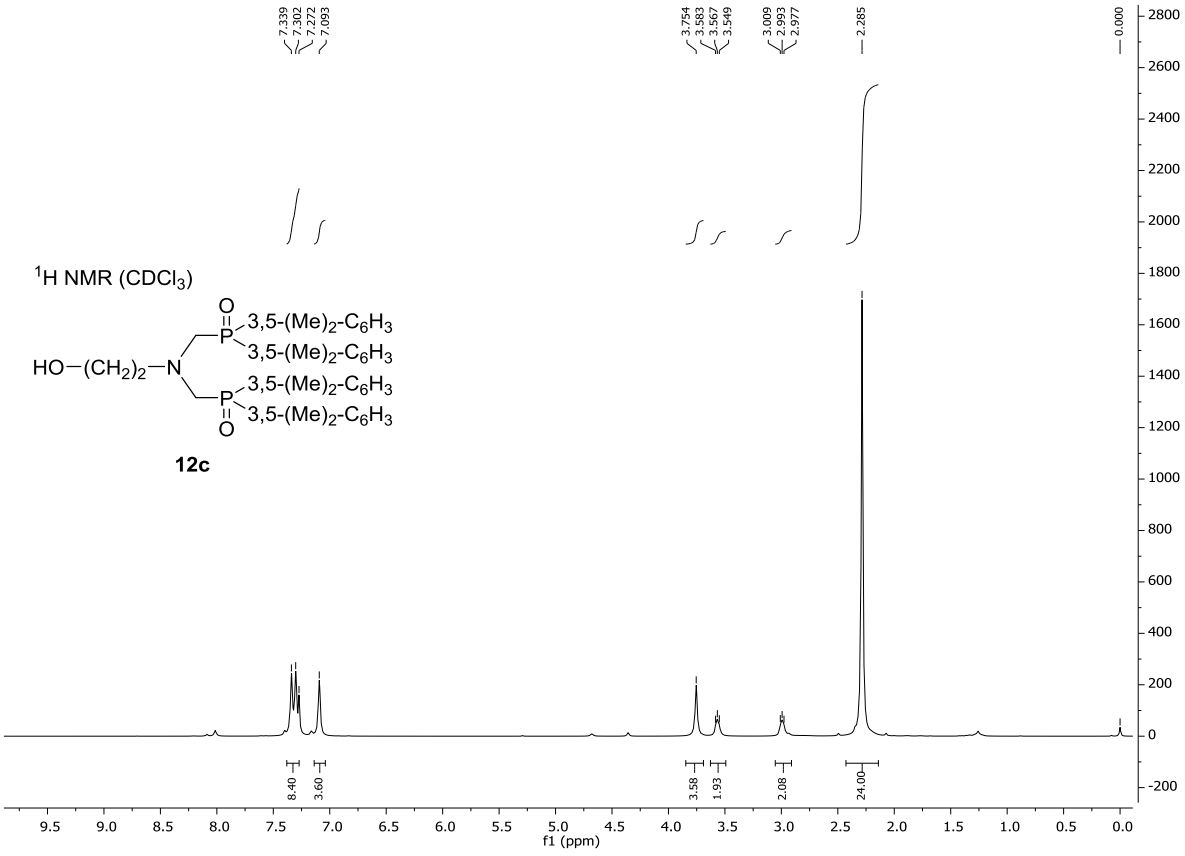


12c

^{13}C NMR ($CDCl_3$)



12c


7.339
7.302
7.272
7.093
3.754
3.583
3.567
3.549
3.009
2.993
2.977
2.285
0.000
^{1}H NMR ($CDCl_3$)
HO−$(CH_2)_2$−N
P
O
3,5-$(Me)_2$-C_6H_3
12c
8.40
3.60
3.58
1.93
2.08
24.00
f1 (ppm)