Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Catalyst- and reagent- free 1,6-hydrophosphonylation of *p*-quinone methides: a practical approach for the synthesis of diarylmethyl phosphine oxides

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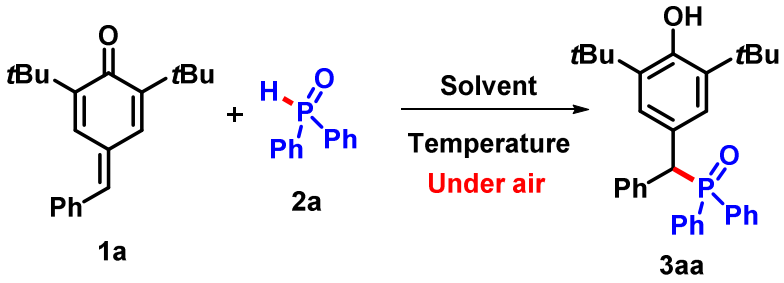
2. Experimental Procedure of the Optimization Study

To a screw capped vial with a spinvane triangular-shaped Teflon stirbar were added *p*-quinone methide **1a** (29.4 mg, 0.10 mmol), diphenylphosphine oxide **2a** (30.3 mg, 0.15 mmol), and solvent (0.6 mL). The reaction mixture was stirred at 80 °C for indicated time. (in case of H_2O as a solvent, after the heating the reaction mixture for indicated time, it was extracted with ethyl acetate (2 × 5 mL) and the extracts were dried over $NaSO_4$). Afterwards, the reaction mixture was concentrated under reduced pressure and crude yield was measured by ^{1}H NMR using an internal standard (1,1,2,2-tetrachloroethane).

Table S1. Optimization of the Catalyst- and Reagent- free 1,6-Hydrophosphonylation of p-Quinone Methides [a]



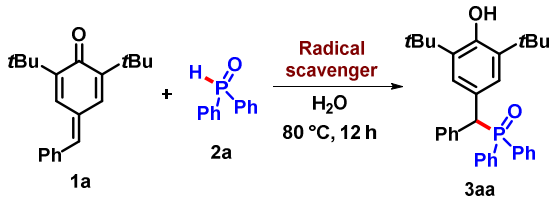
entry	solvent	temp (°C)	yield (%)[b]
1	1,2-DCE	80	86[c]
2	1,2-DCE	80	80
3	THF	80	82
4	CH_3CN	80	87
5	Toluene	80	85
6	1,4-Dioxane	80	96 (92)
7	MeOH	80	81
8	EtOAc	80	85
9	H_2O	80	68
10[d]	**H_2O**	**80**	**98 (94)**[e]
11[d]	H_2O	40	70

[a]Reaction conditions: **1a** (0.10 mmol), **2a** (0.15 mmol) in solvent (0.6 mL) at indicated temperature for 8 h. [b]Isolated yields are based on crude ^{1}H NMR (internal standard: 1,1,2,2 tetrachloroethane). [c]Under 1 atm of O_2 (ballon). [d]Reaction run for 12 h. [e]Yield in the parentheses indicates isolated yield.

3. Mechanistic Experiments

Radical Trap Experiments

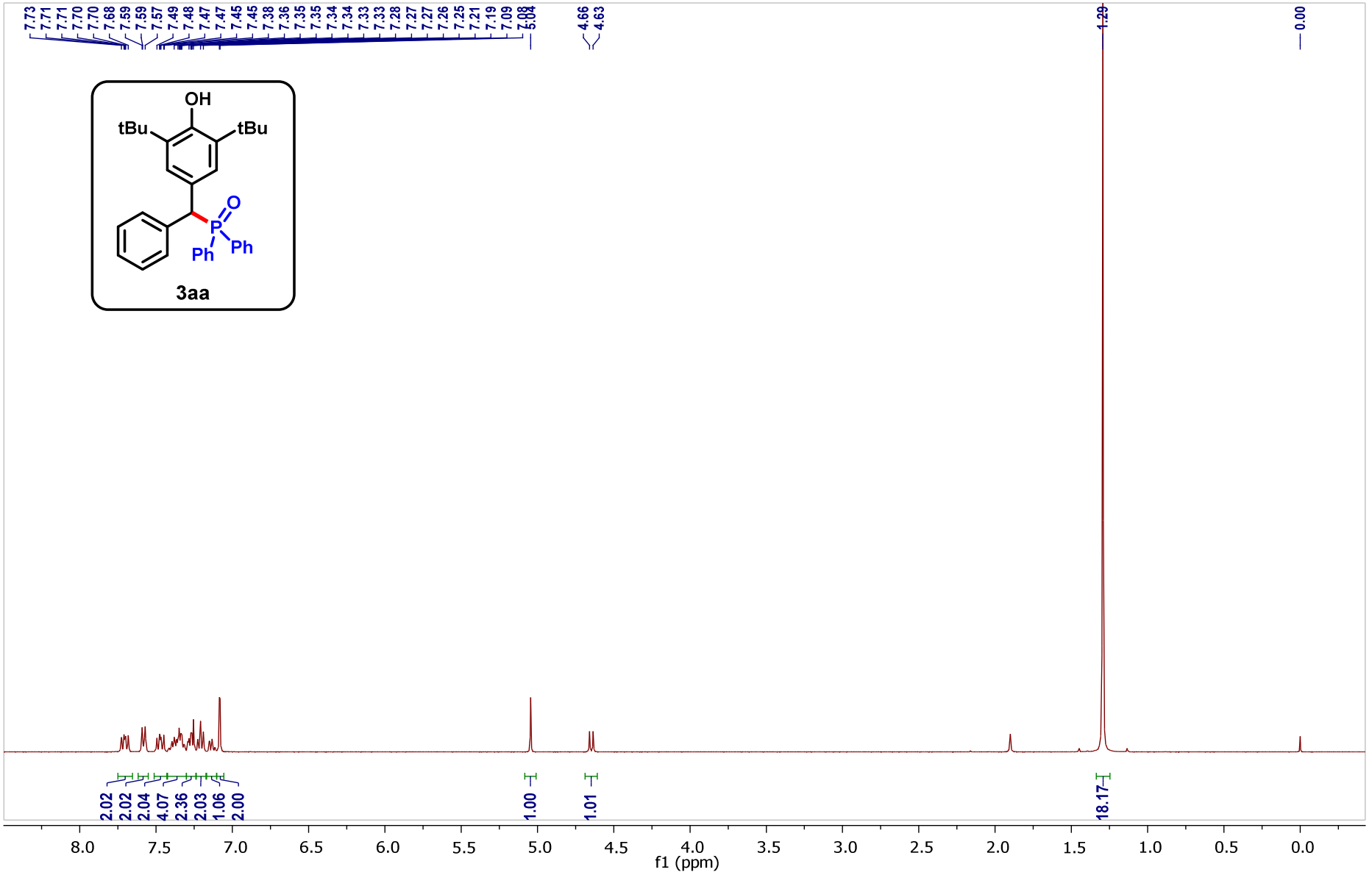
To a screw capped vial with a spinvane triangular-shaped Teflon stirbar were added *p*-quinone methide **1a** (29.4 mg, 0.10 mmol), diphenylphosphine oxide **2a** (30.3 mg, 0.15 mmol). Then Radical Scavengers such as BHT (44.0 mg, 0.2 mmol) or and 1,1-diphenylethylene (36.1 mg, 0.2 mmol), and H_2O (0.6 mL) were added. The reaction mixture was stirred at 80 ºC for 12 h. After indicated time, the reaction mixture was cooled to room temperature & extracted with ethyl acetate (2 × 10 mL) and the extract was dried over $NaSO_4$. Then the solvent was removed under reduced pressure and crude yield was measured by ^1H NMR using an internal standard (1,1,2,2-tetrachloroethane).



Radical scavenger (equiv.)	^1H NMR yield of **3aa**
1. BHT (2.0)	98%
2. 1,1-Diphenylethylene (2.0)	97%

Appendix

Spectral Copies of ^{1}H and ^{13}C NMR of Compounds Obtained in this study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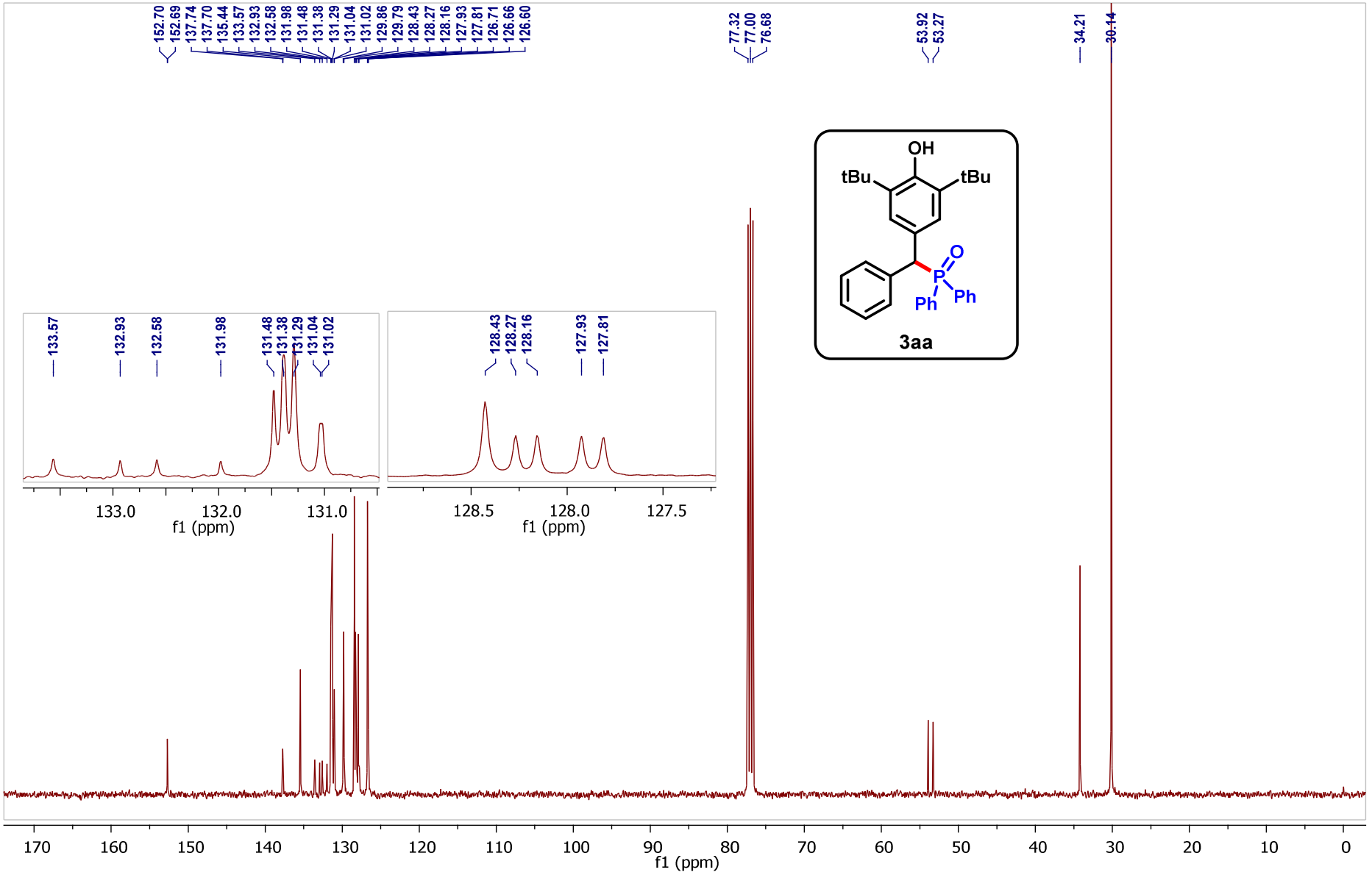
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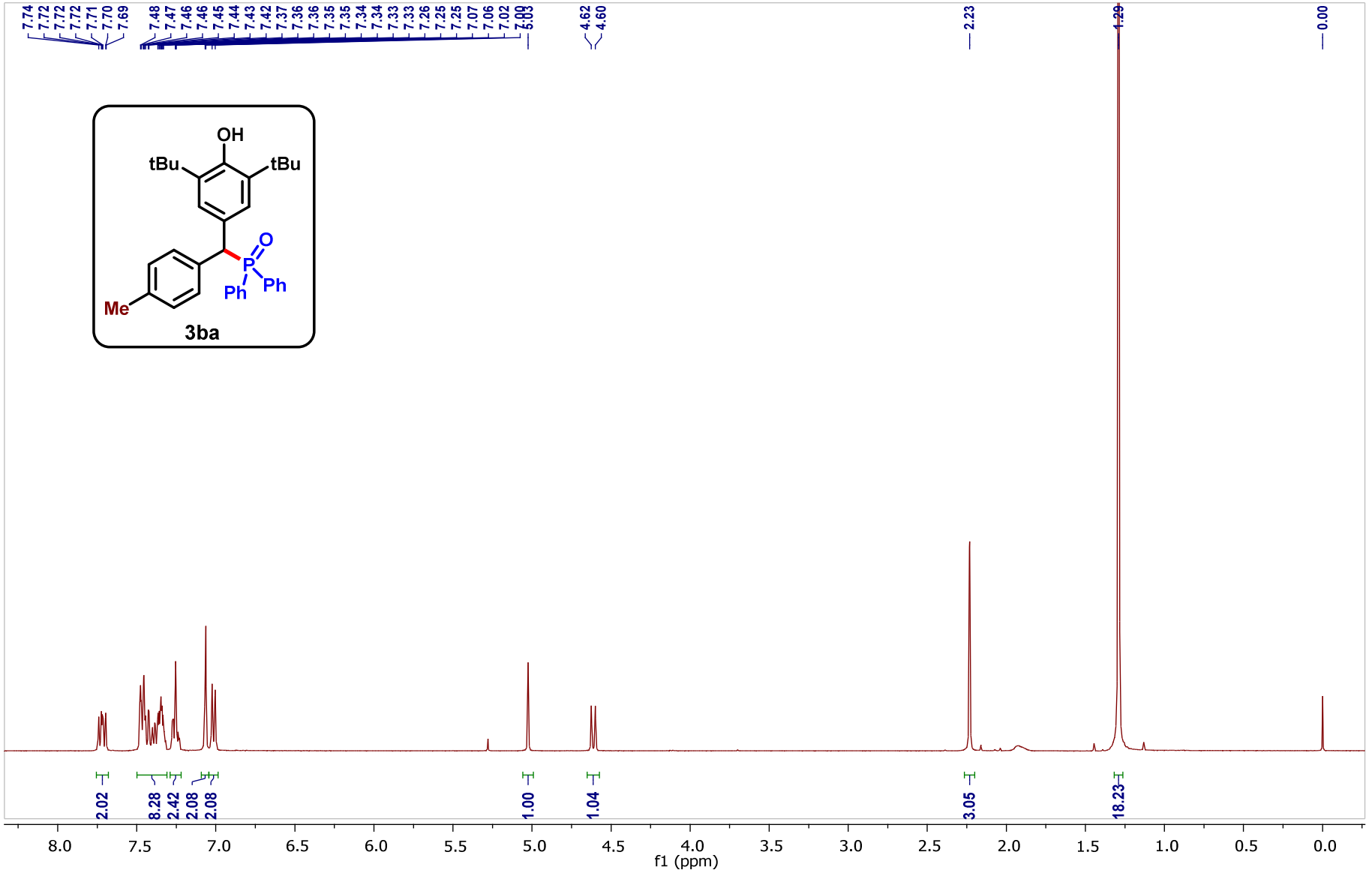
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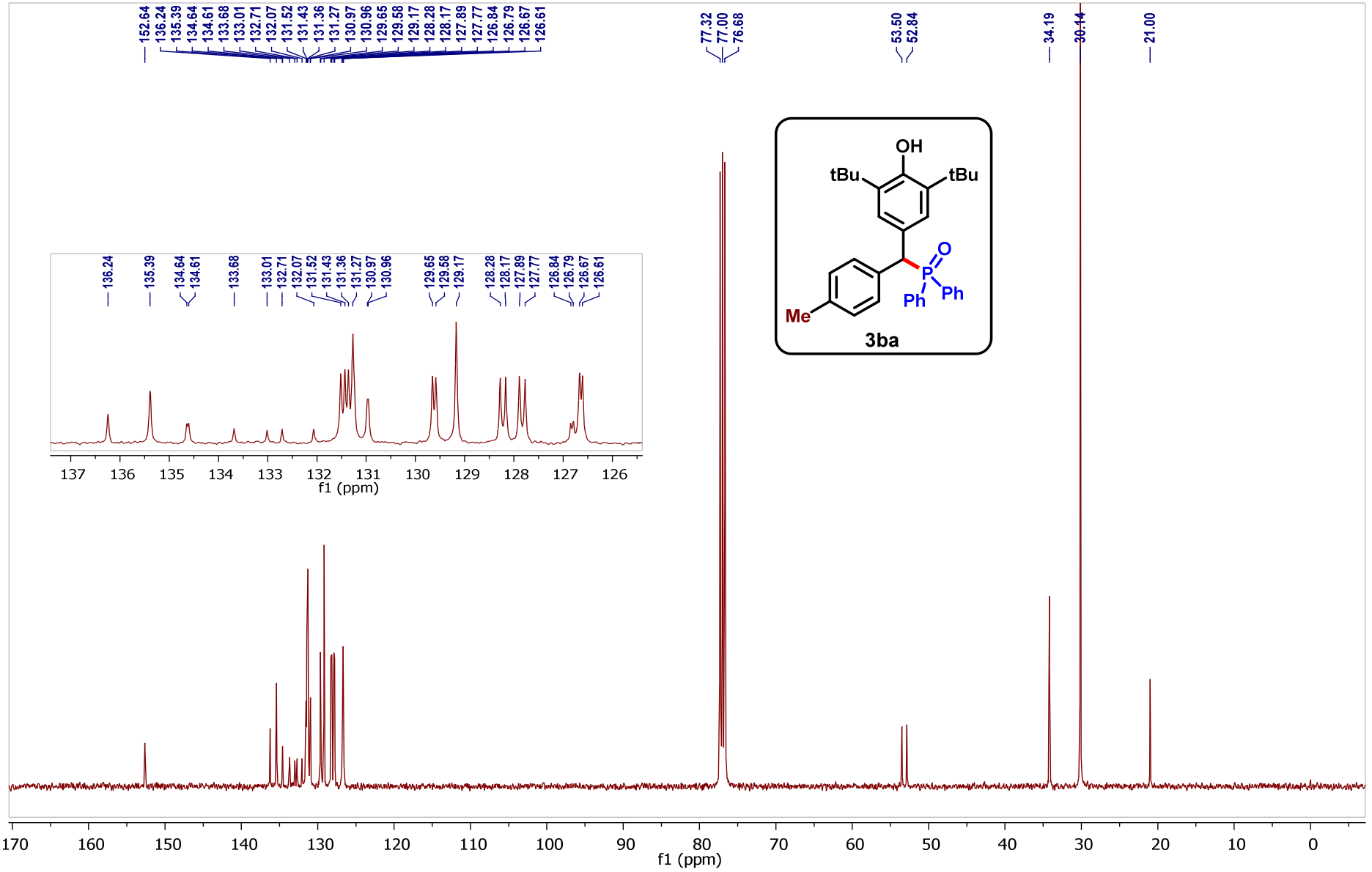
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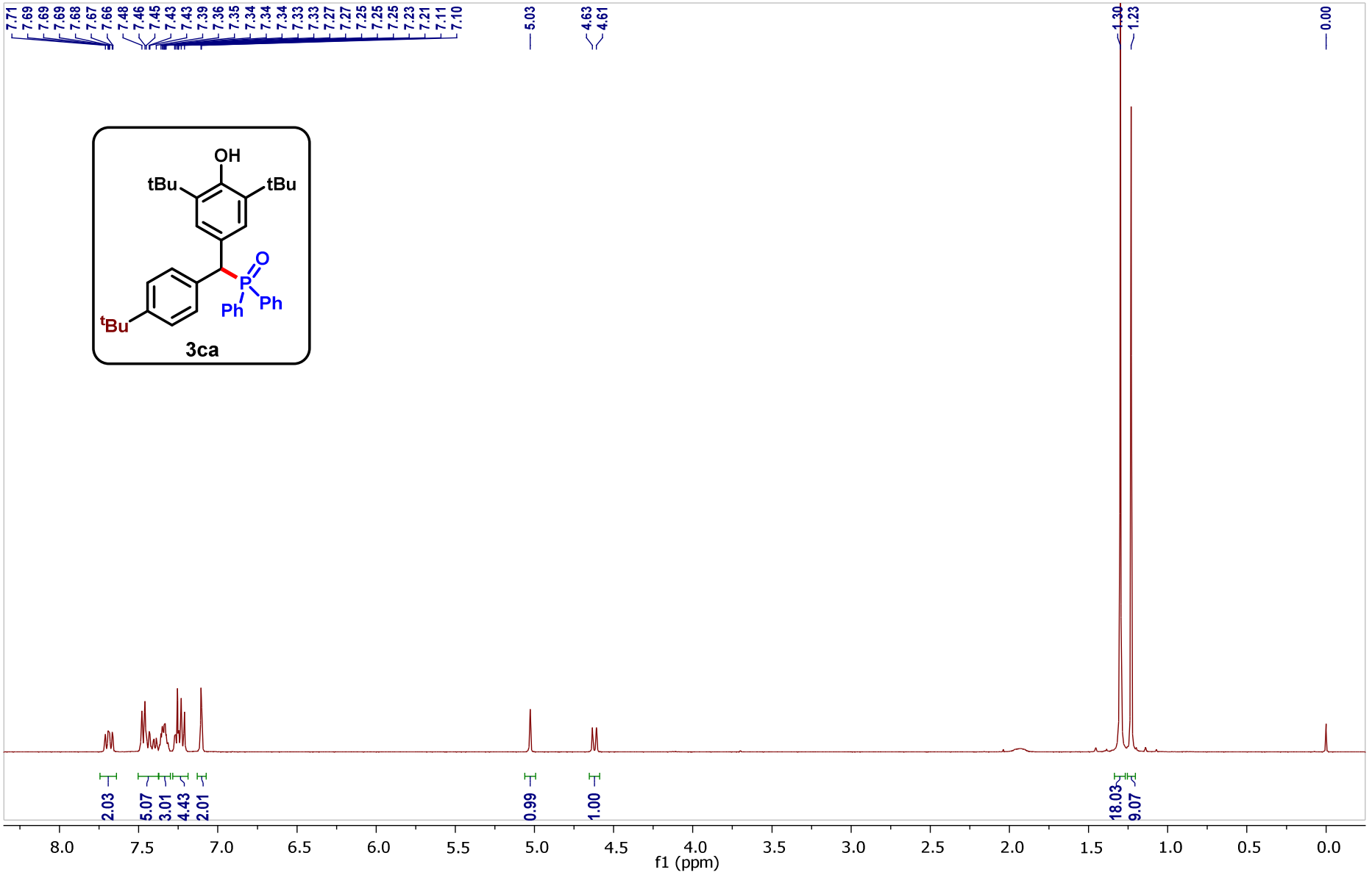
f1 (ppm)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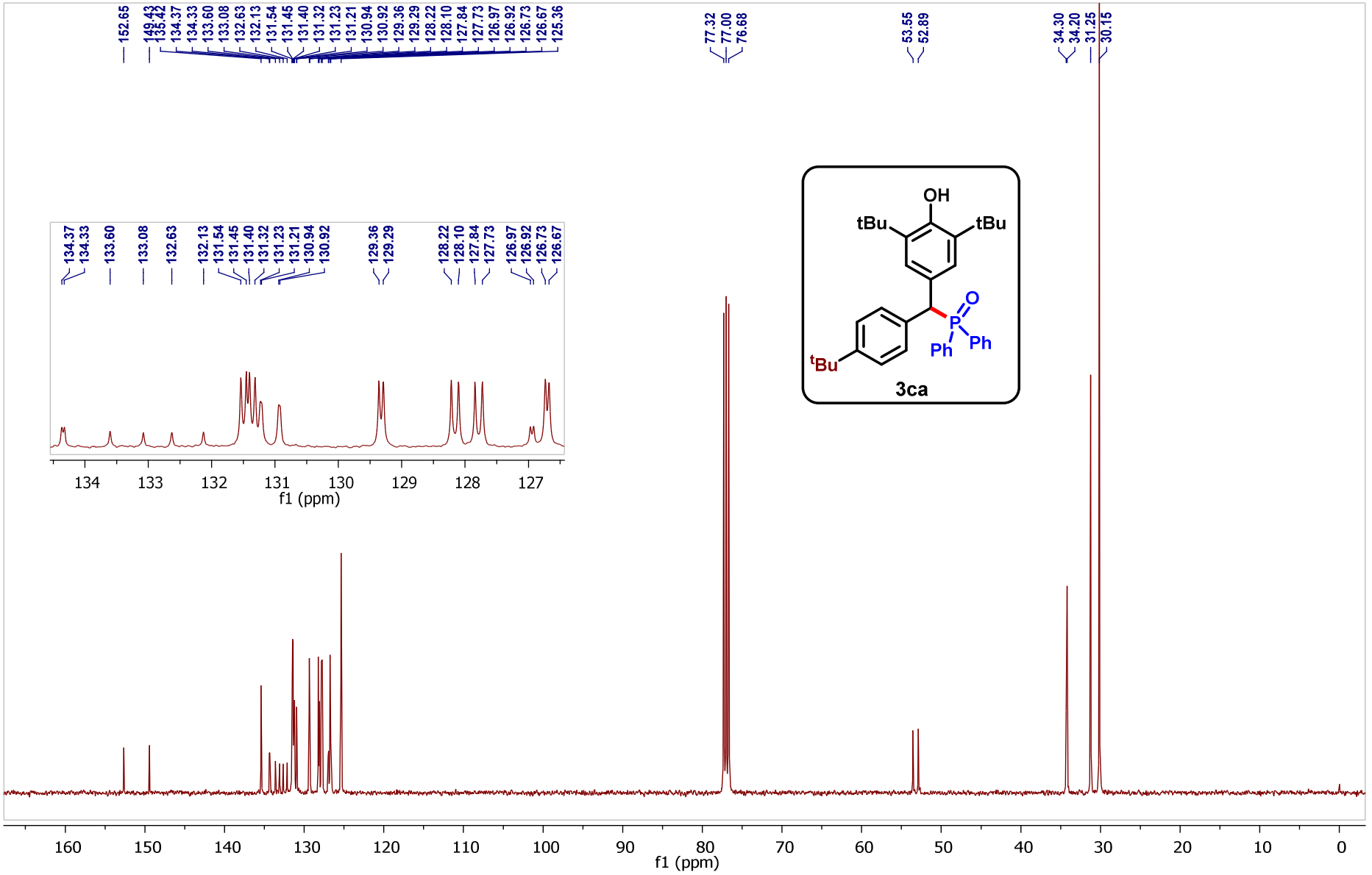
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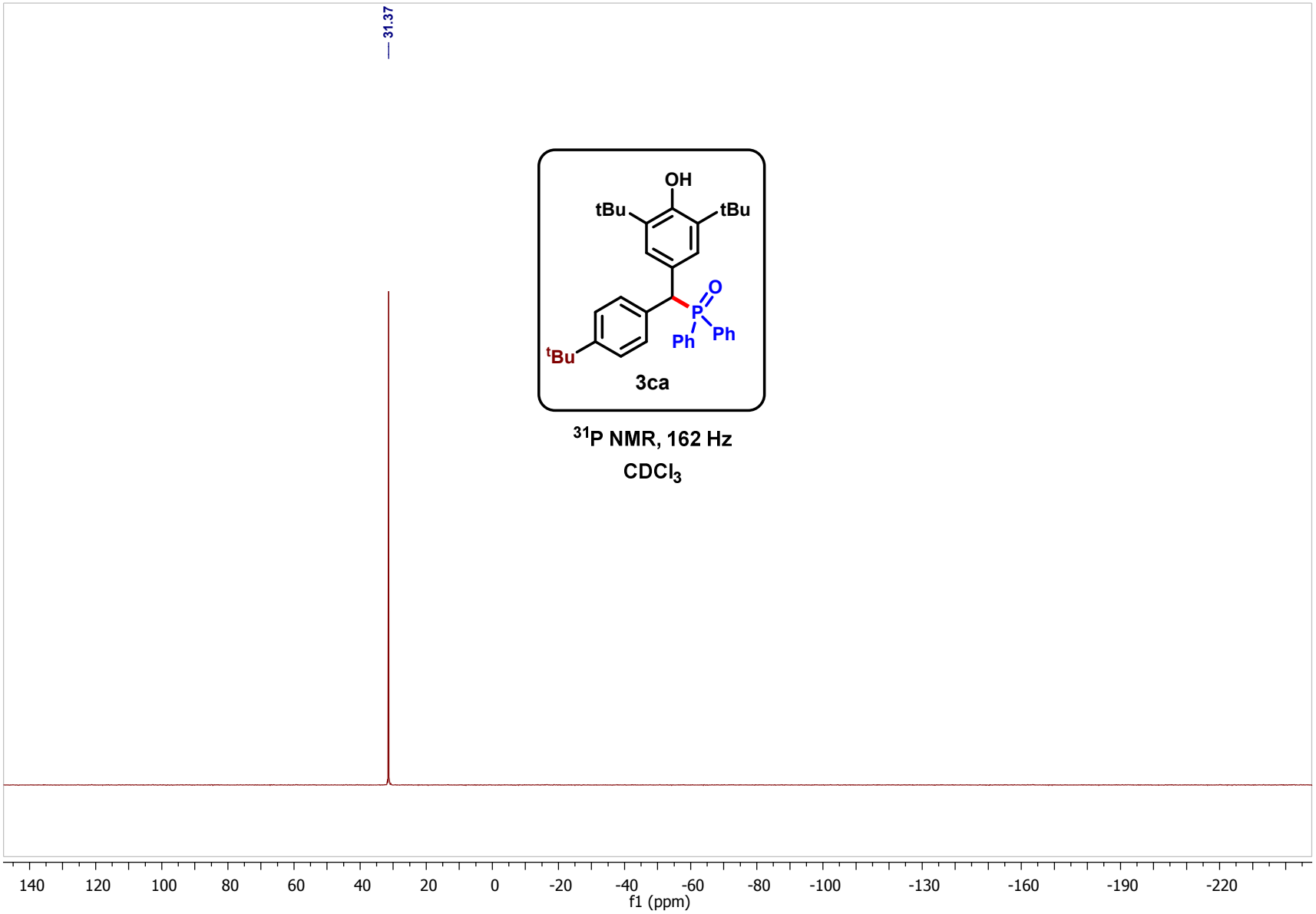
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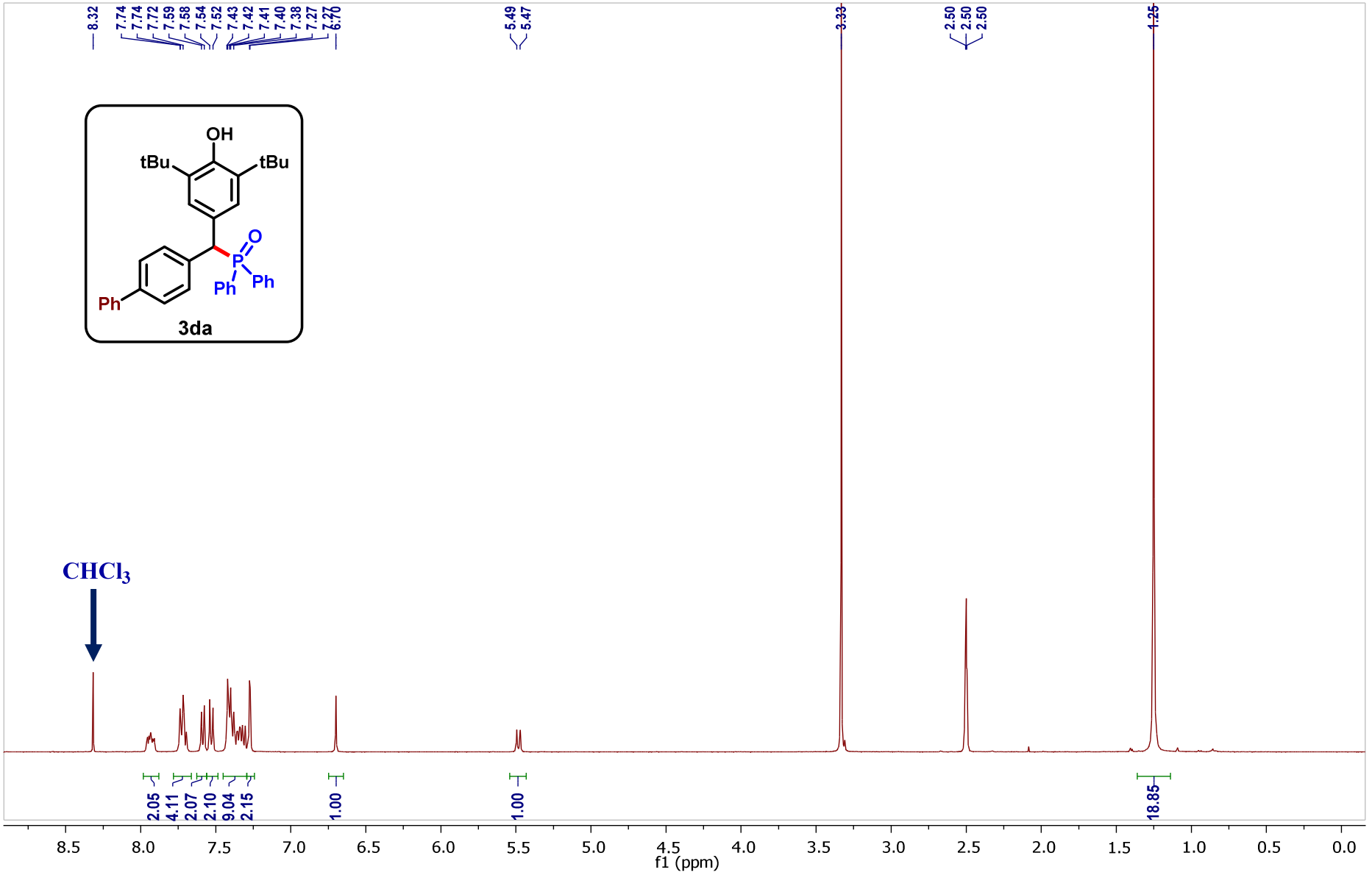
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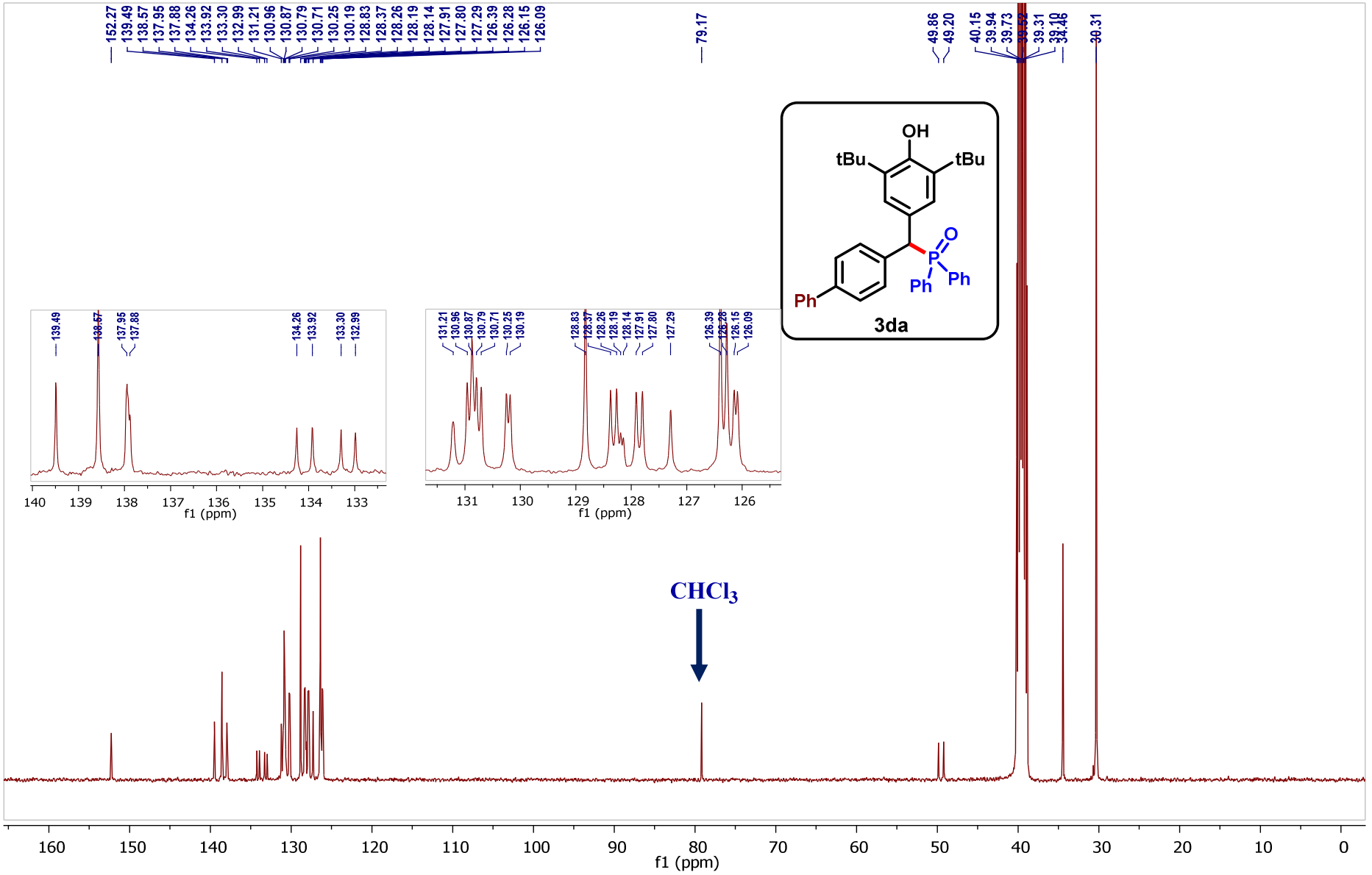
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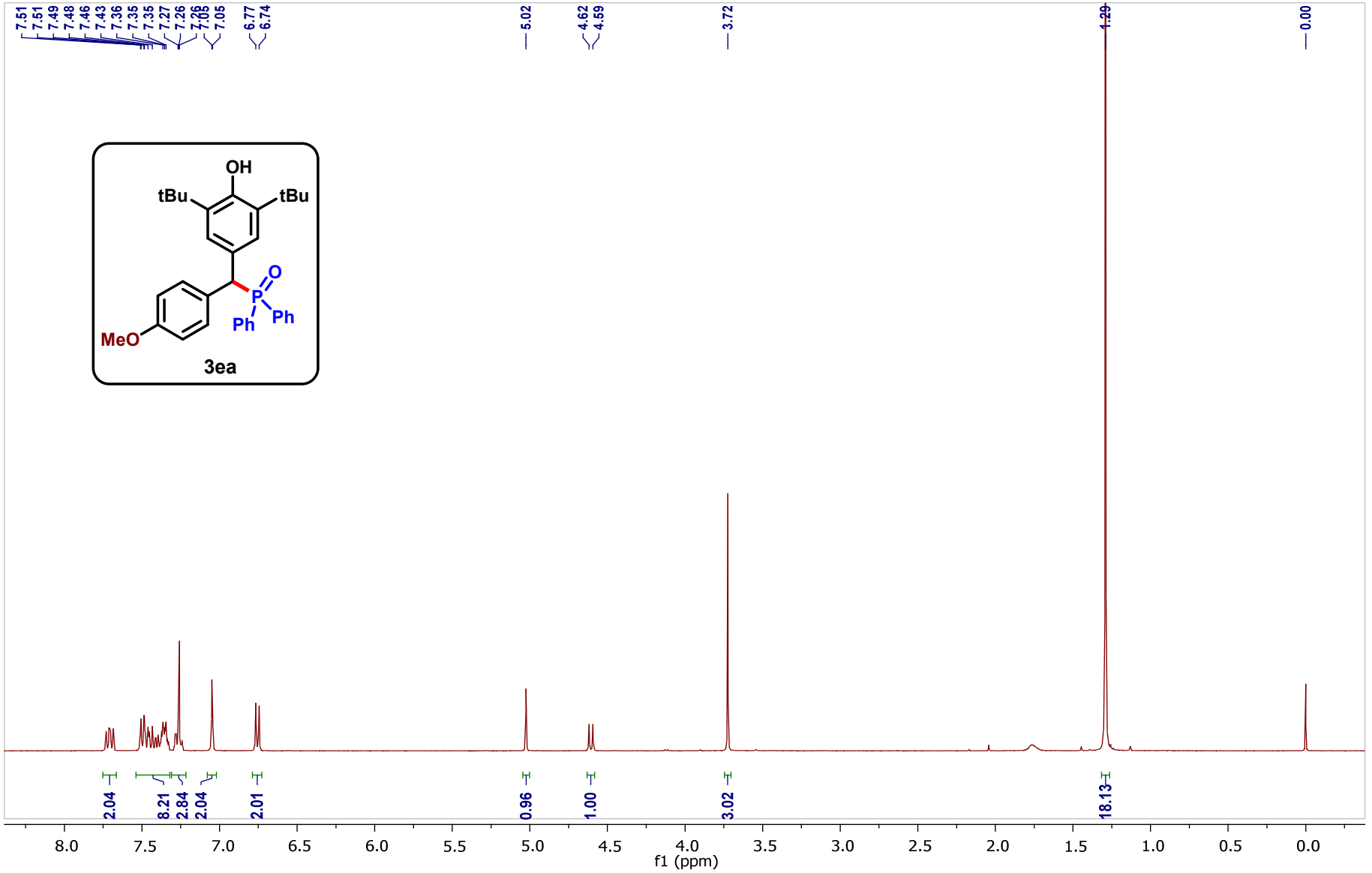
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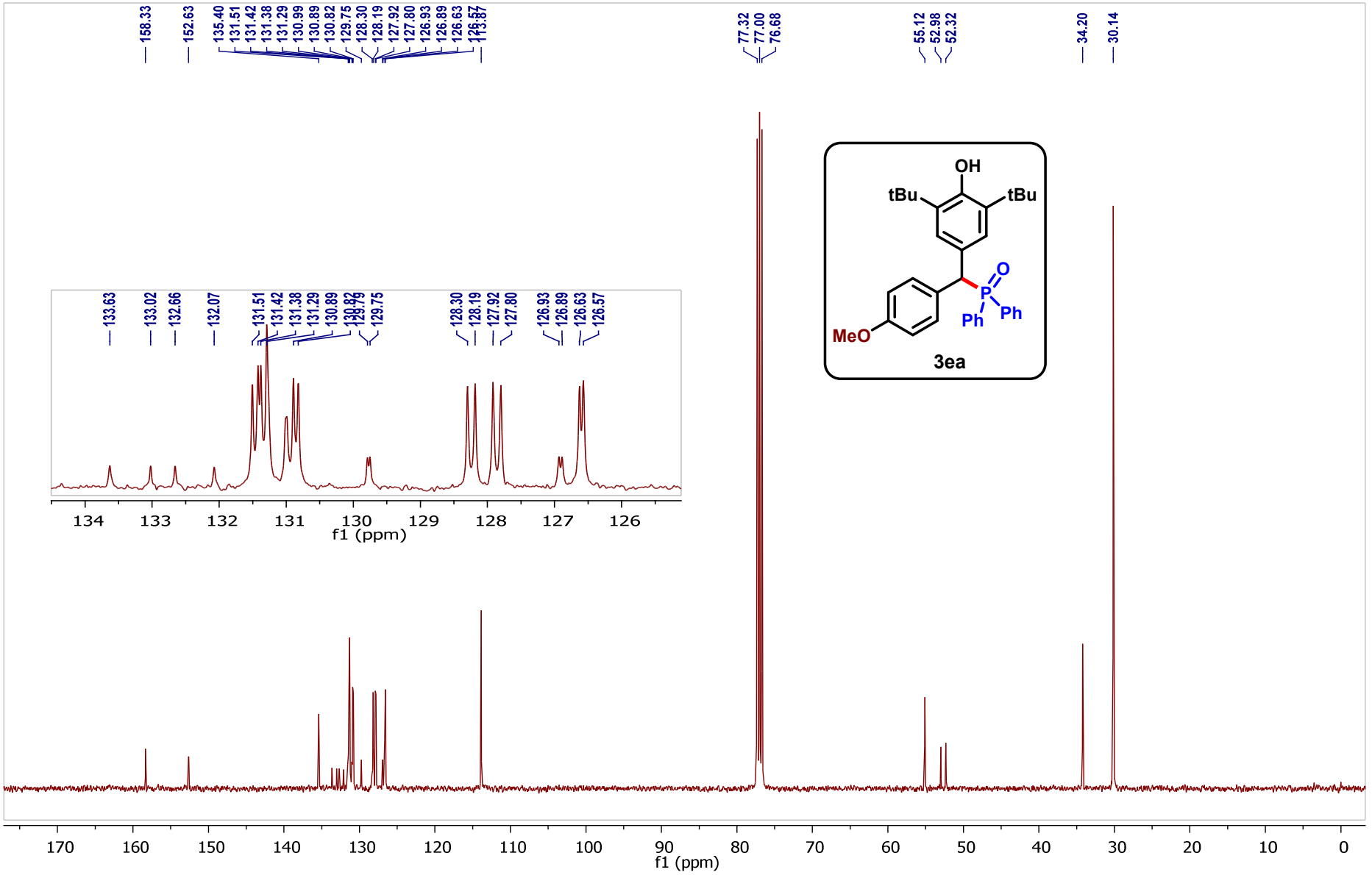
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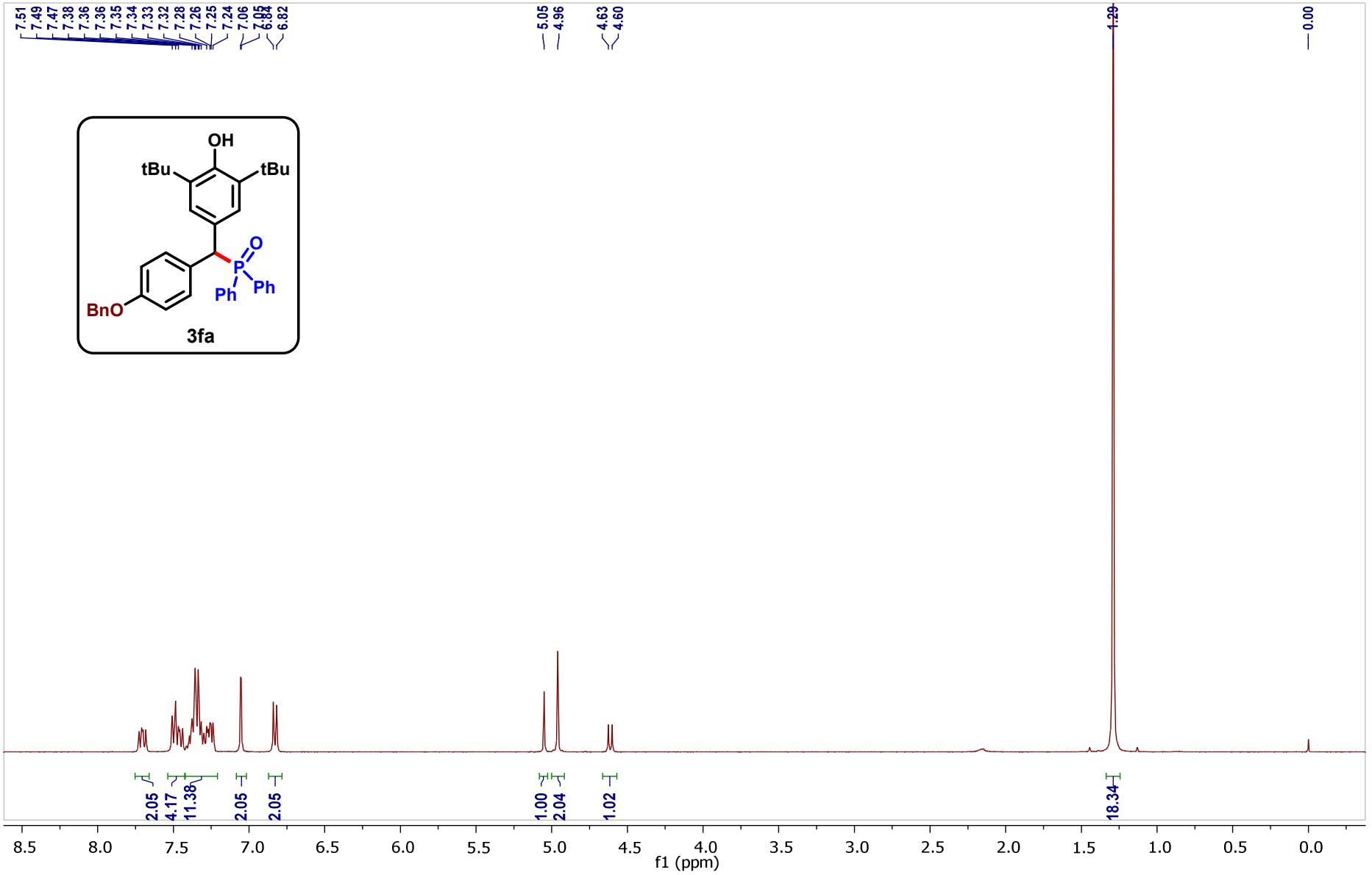
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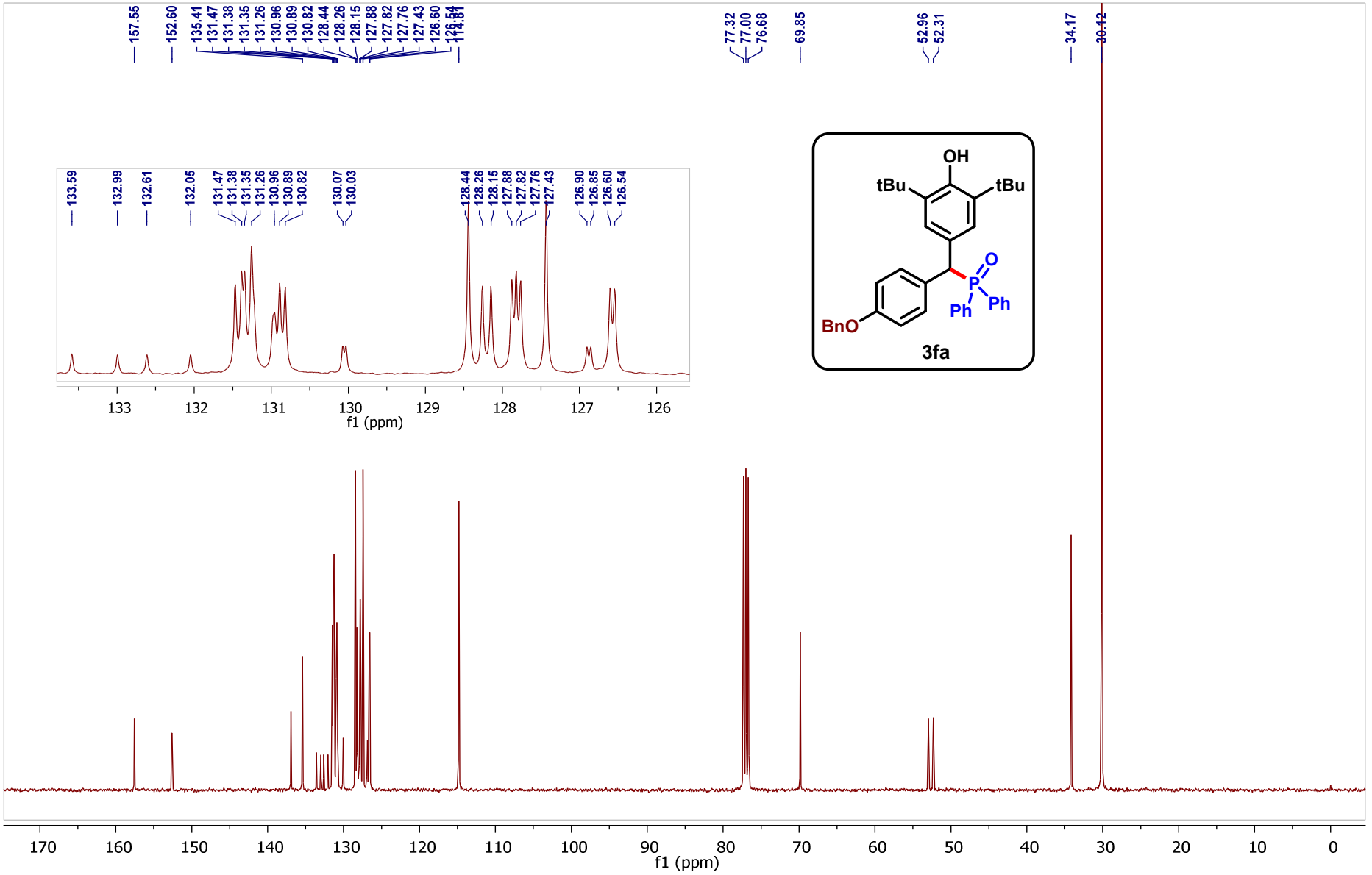
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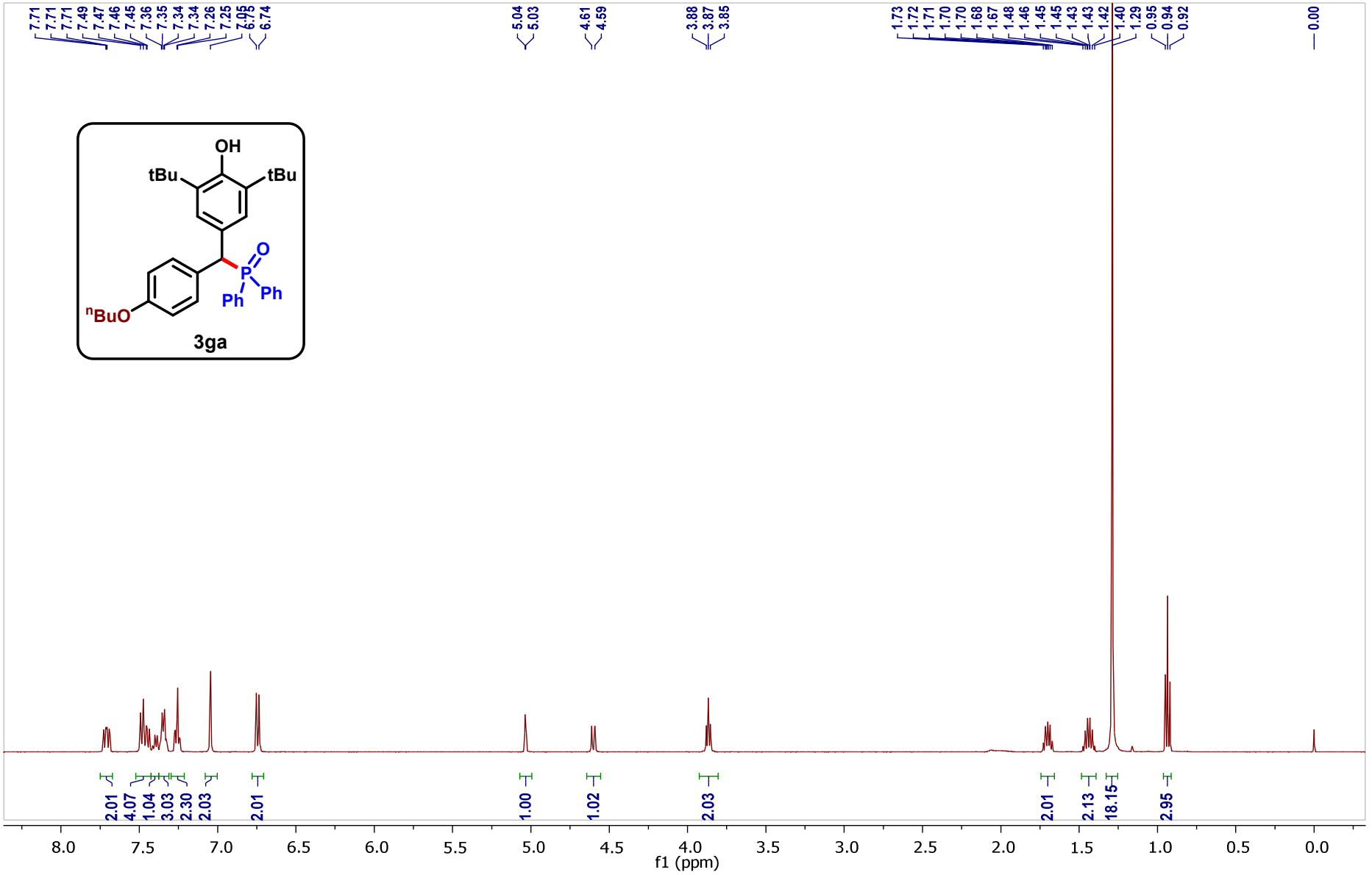
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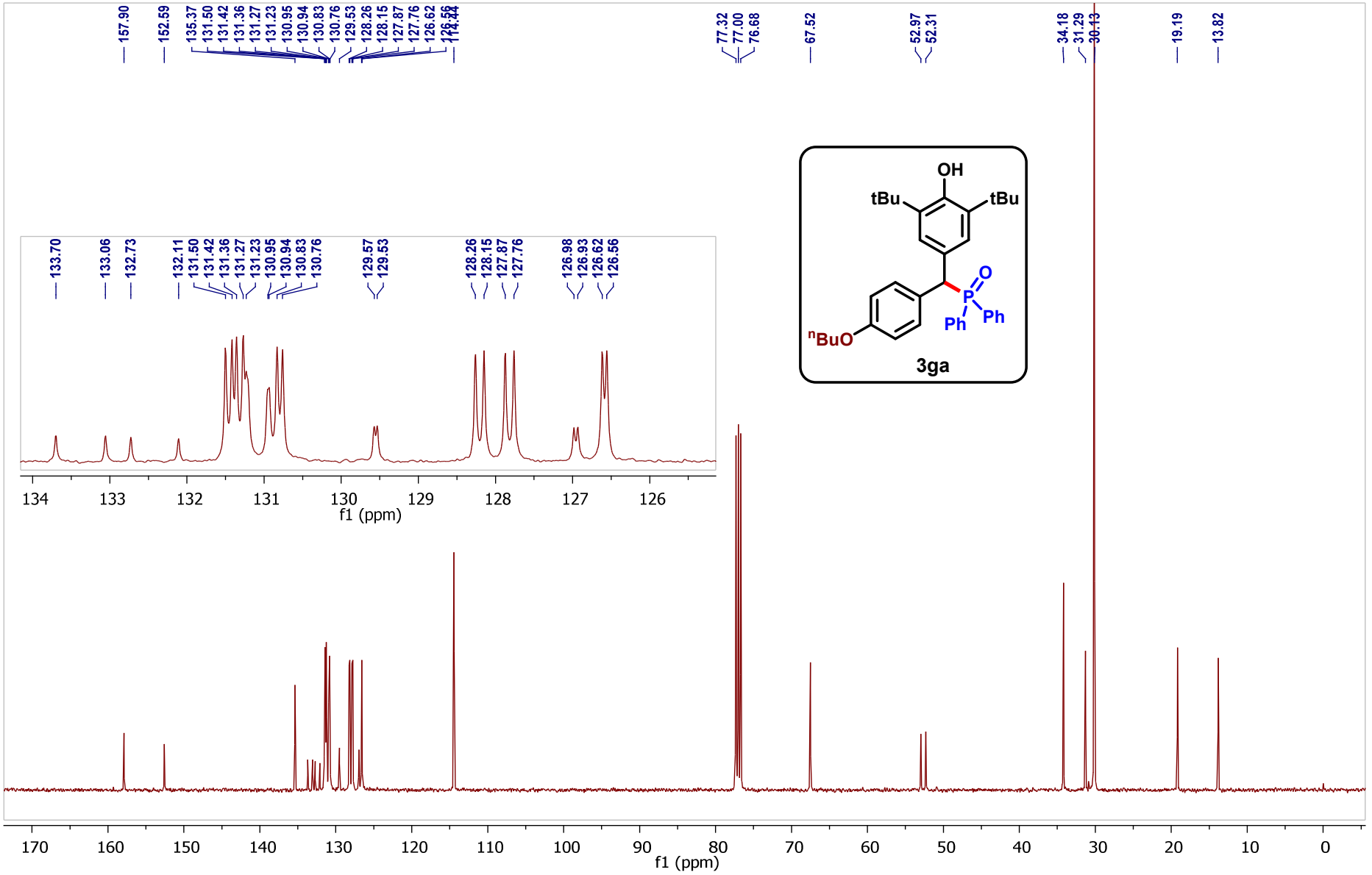
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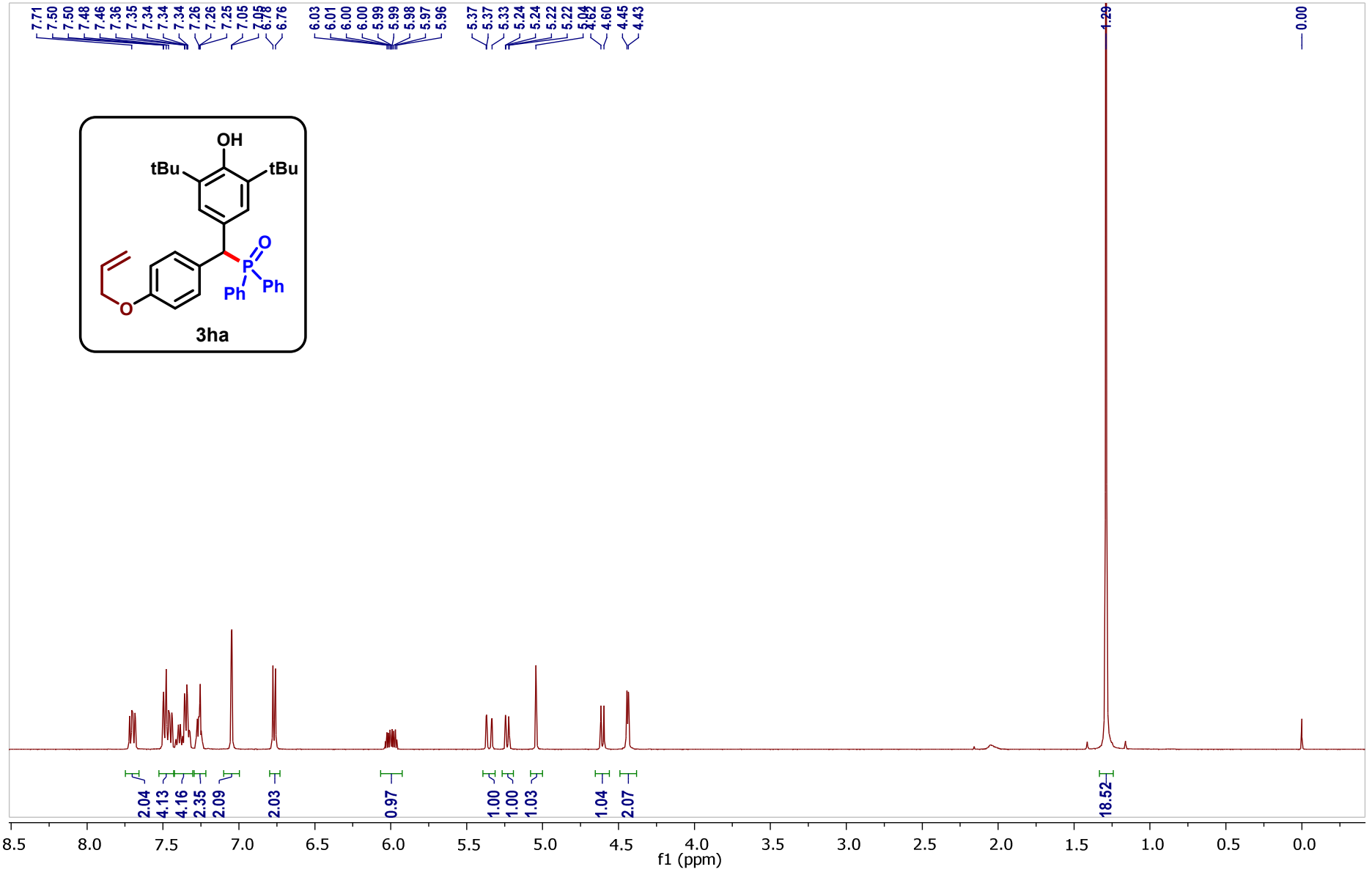
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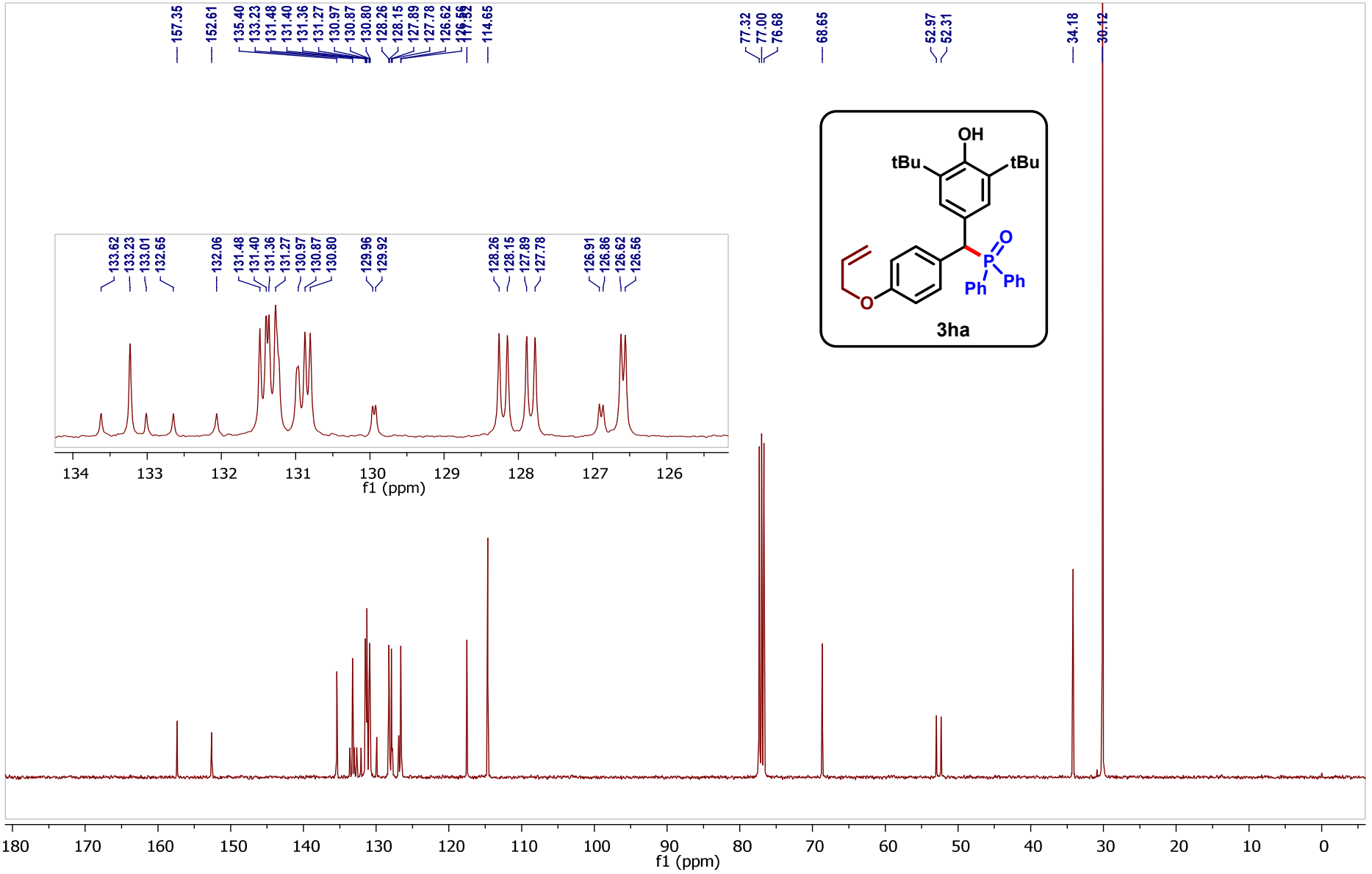
f1 (ppm)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77.32
77.00
76.68
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52.97
52.31
34.18
30.12
OH
tBu
tBu
O
P
Ph
Ph
O
3ha
133.62
133.23
133.01
132.65
132.06
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127.89
127.78
126.91
126.86
126.62
126.56
134
133
132
131
130
129
128
127
126
f1 (ppm)
180
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50
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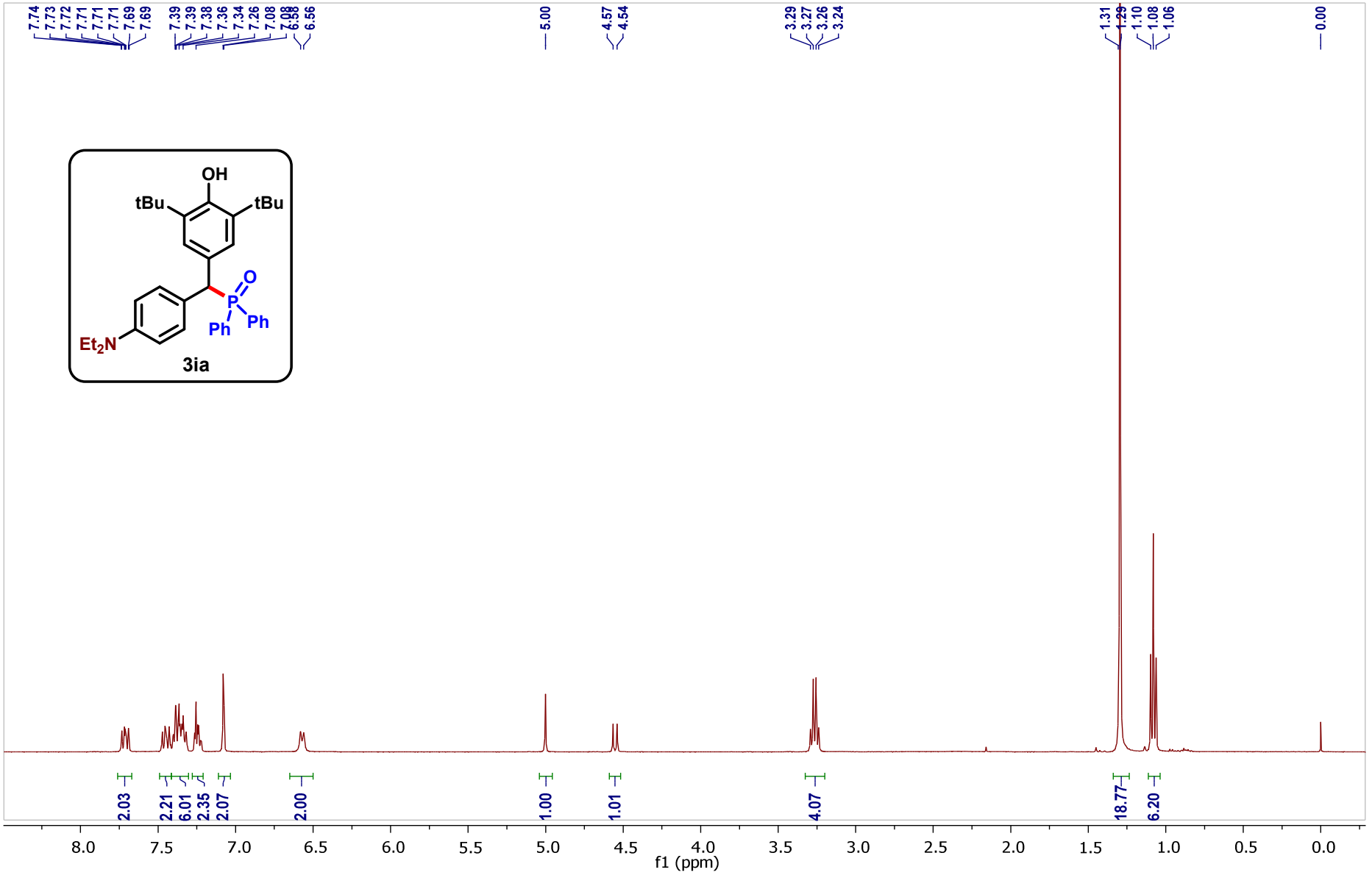
f1 (ppm)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7.34
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7.08
6.58
6.56
5.00
4.57
4.54
3.29
3.27
3.26
3.24
1.31
1.29
1.10
1.08
1.06
0.00
OH
tBu
tBu
O
P
Ph
Ph
Et2N
3ia
2.03
2.21
6.01
2.35
2.07
2.00
1.00
1.01
4.07
18.77
6.20
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7.0
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6.0
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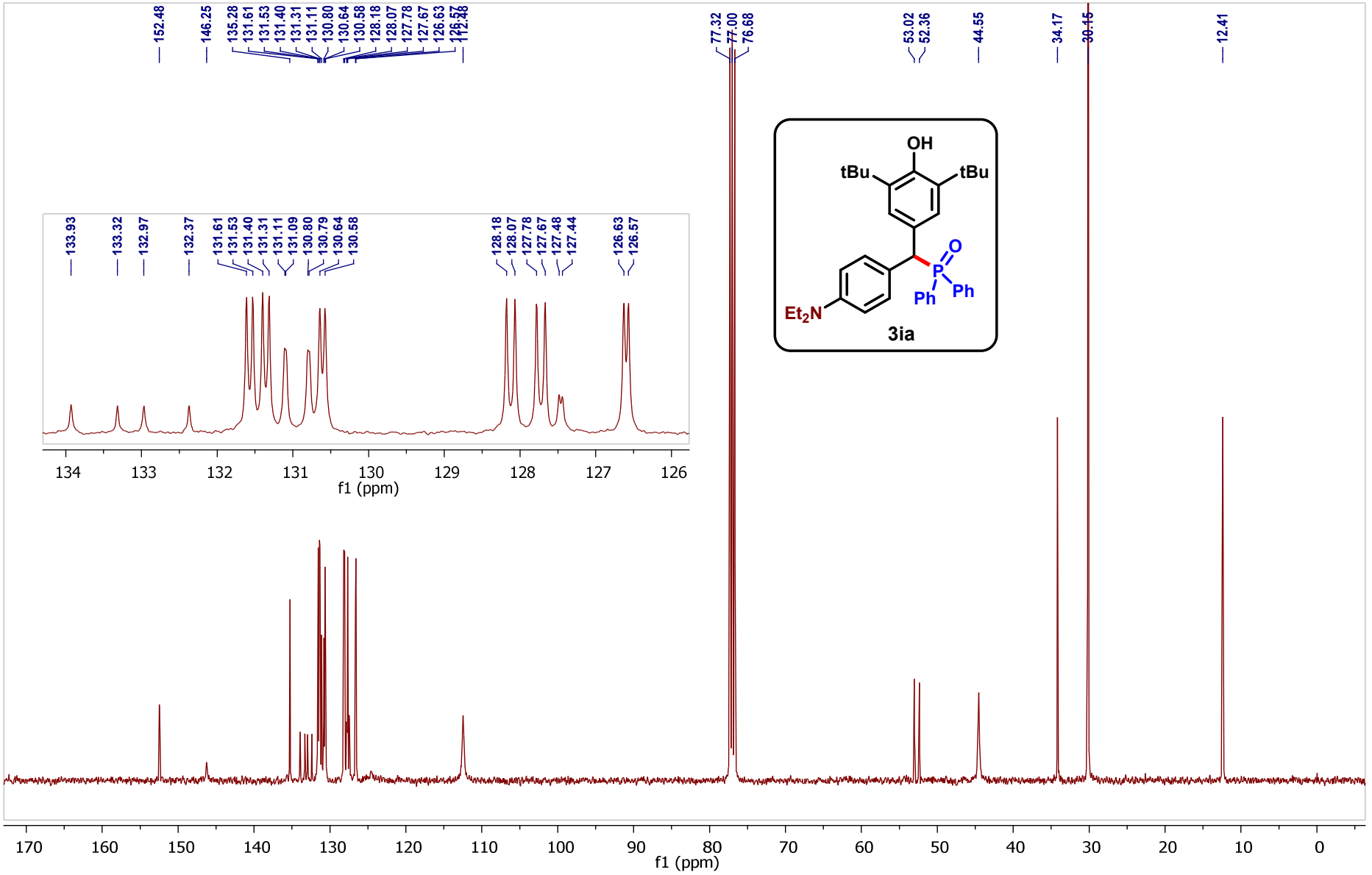
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77.00
76.68
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126.57
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133
132
131
130
129
128
127
126
f1 (ppm)
OH
tBu
tBu
O
P
Ph
Ph
Et2N
3ia
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110
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90
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50
40
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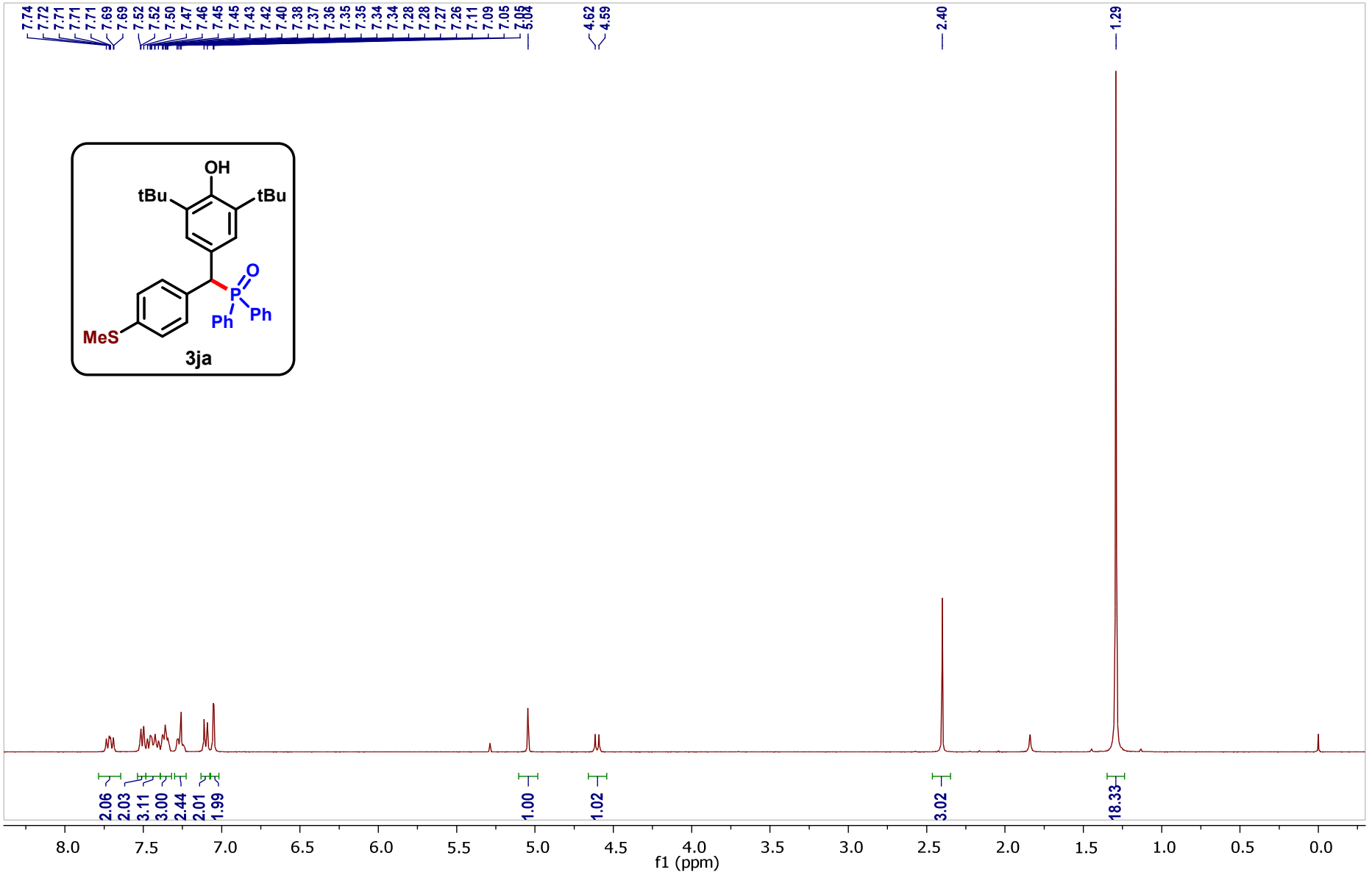
10
0
f1 (ppm)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7.46
7.45
7.45
7.43
7.42
7.40
7.38
7.37
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7.35
7.35
7.34
7.34
7.28
7.28
7.27
7.26
7.11
7.09
7.05
5.04
4.62
4.59
2.40
1.29
OH
tBu
tBu
O
P
Ph
Ph
MeS
3ja
2.06
2.03
3.11
3.00
2.44
2.01
1.99
1.00
1.02
3.02
18.33
8.0
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7.0
6.5
6.0
5.5
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2.5
2.0
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1.0
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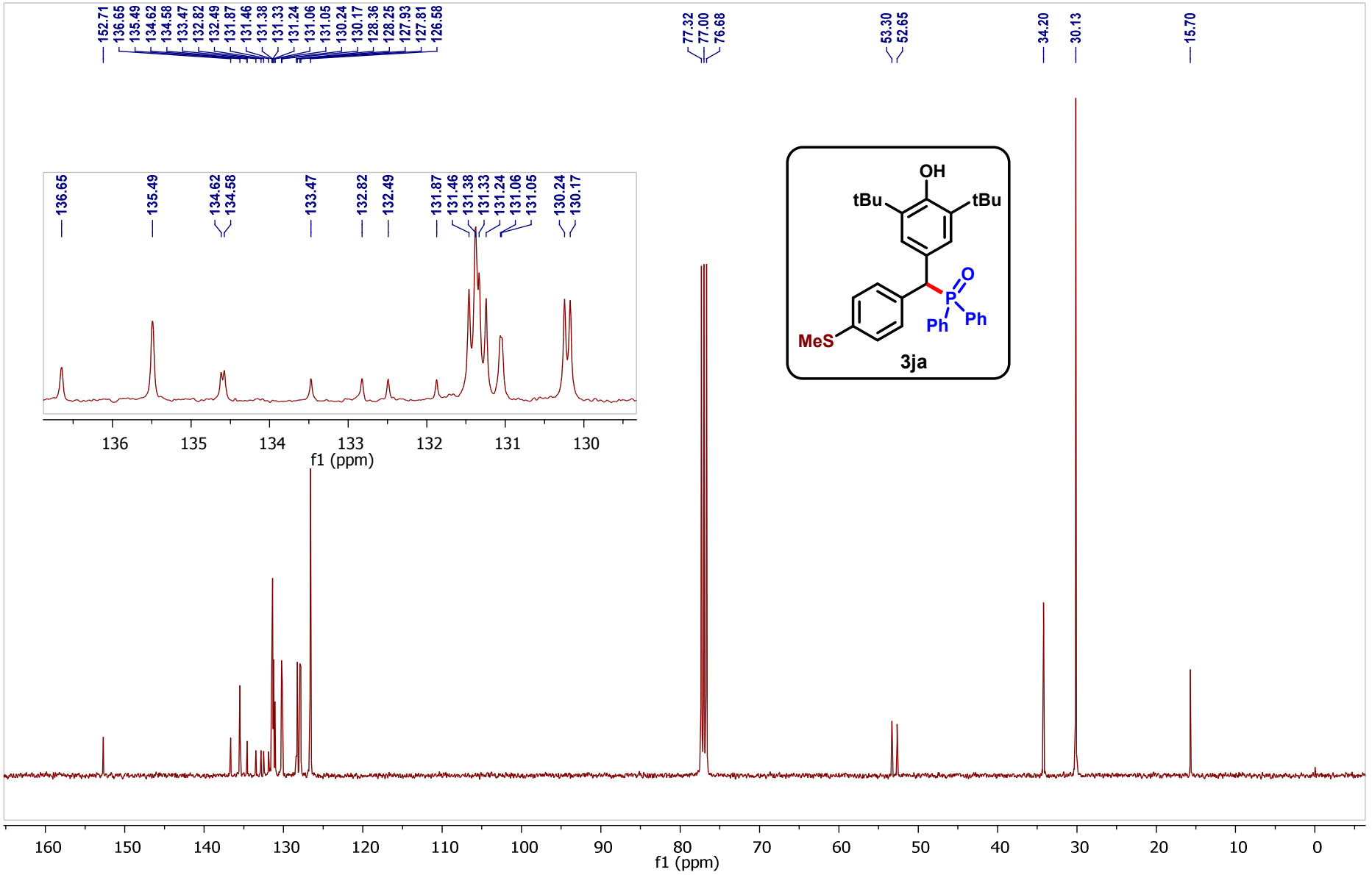
0.0
f1 (ppm)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131.24
131.06
131.05
130.24
130.17
128.36
128.25
127.93
127.81
126.58
77.32
77.00
76.68
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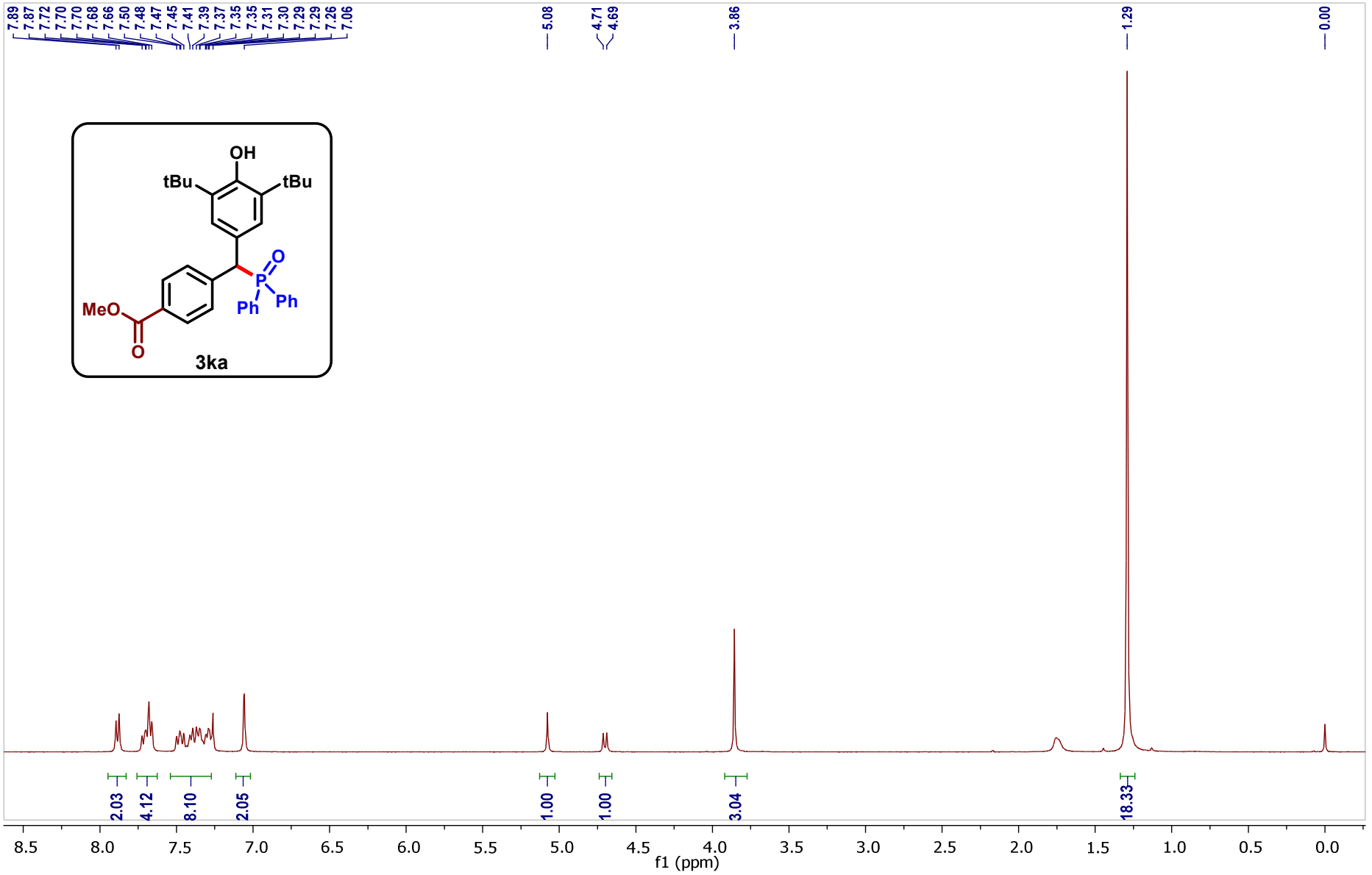
30.13
15.70
f1 (ppm)
OH
tBu
tBu
O
P
Ph
Ph
MeS
3ja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7.37
7.35
7.35
7.31
7.30
7.29
7.29
7.26
7.06
5.08
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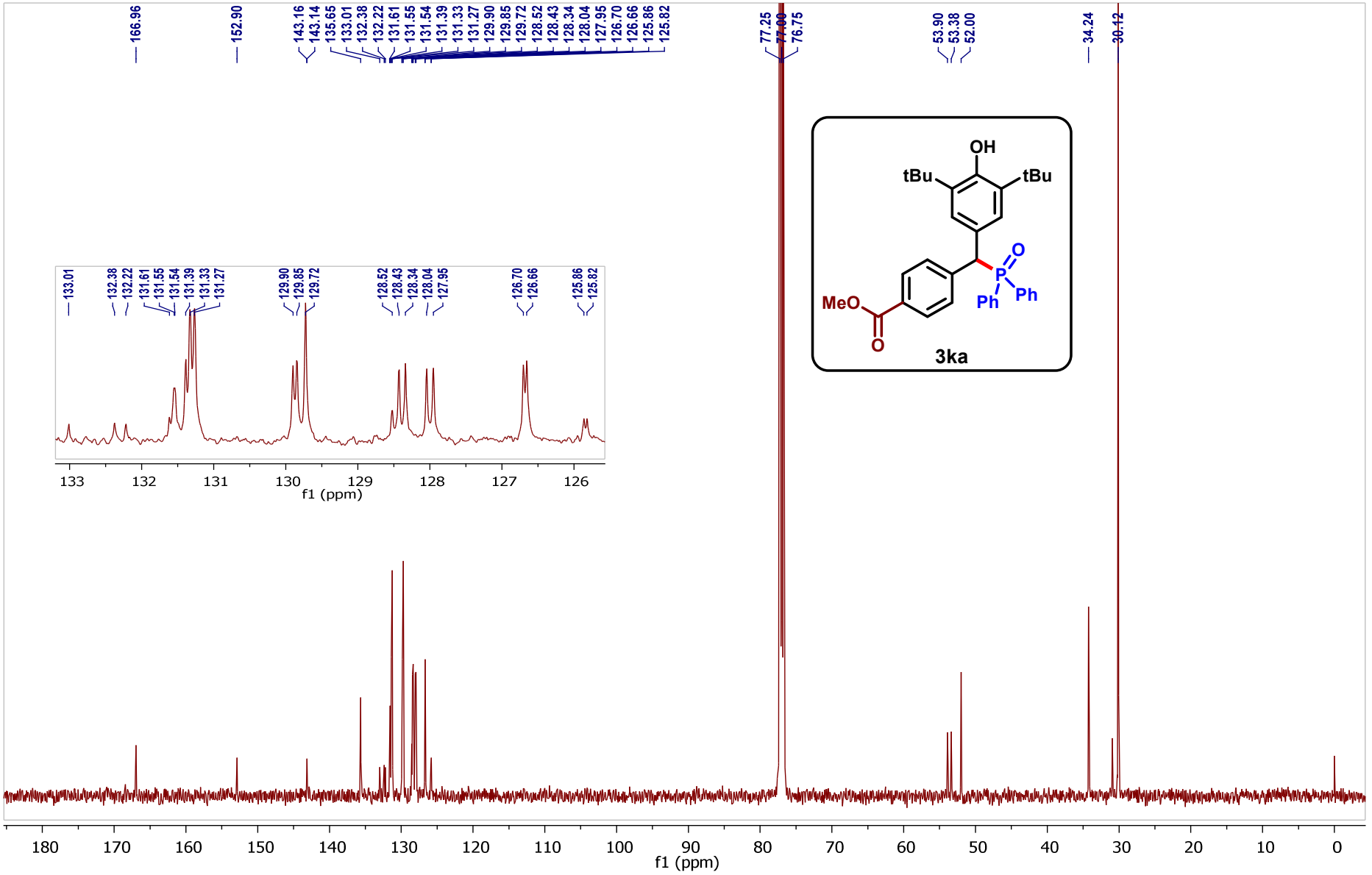
4.69
3.86
1.29
0.00
OH
tBu
tBu
O
P
Ph
Ph
MeO
O
3ka
2.03
4.12
8.10
2.05
1.00
1.00
3.04
18.33
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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129.90
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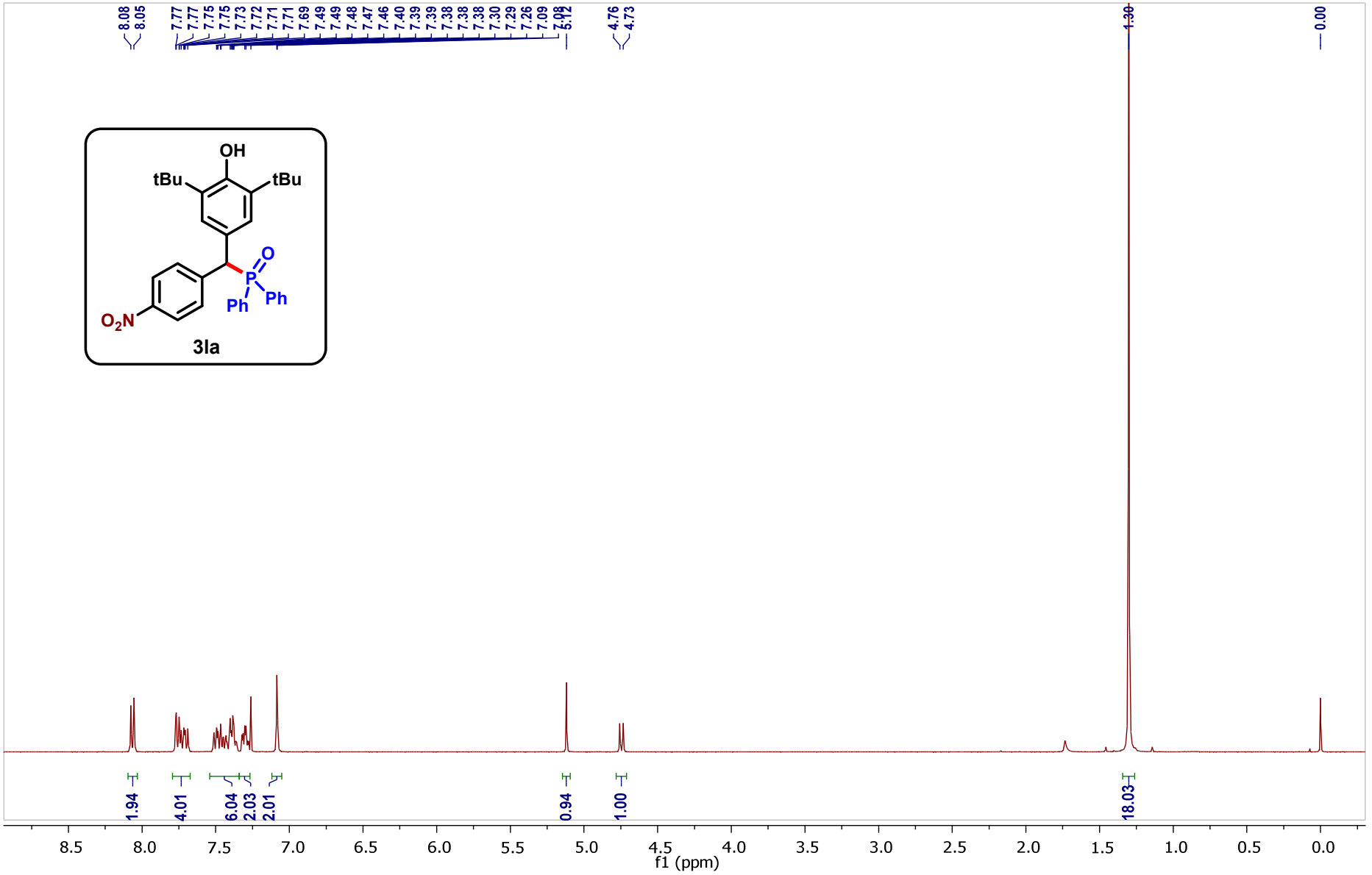
128.43
128.34
128.04
127.95
126.70
126.66
125.86
125.82
77.25
77.00
76.75
53.90
53.38
52.00
34.24
30.12
OH
tBu
tBu
O
P
Ph
Ph
MeO
O
3ka
f1 (ppm)
f1 (ppm)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7.48
7.47
7.46
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7.39
7.38
7.38
7.38
7.30
7.29
7.26
7.09
7.08
5.12
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4.73
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OH
tBu
tBu
O
P
Ph
Ph
O2N
3la
1.94
4.01
6.04
2.03
2.01
0.94
1.00
18.03
8.5
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7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
1.5
1.0
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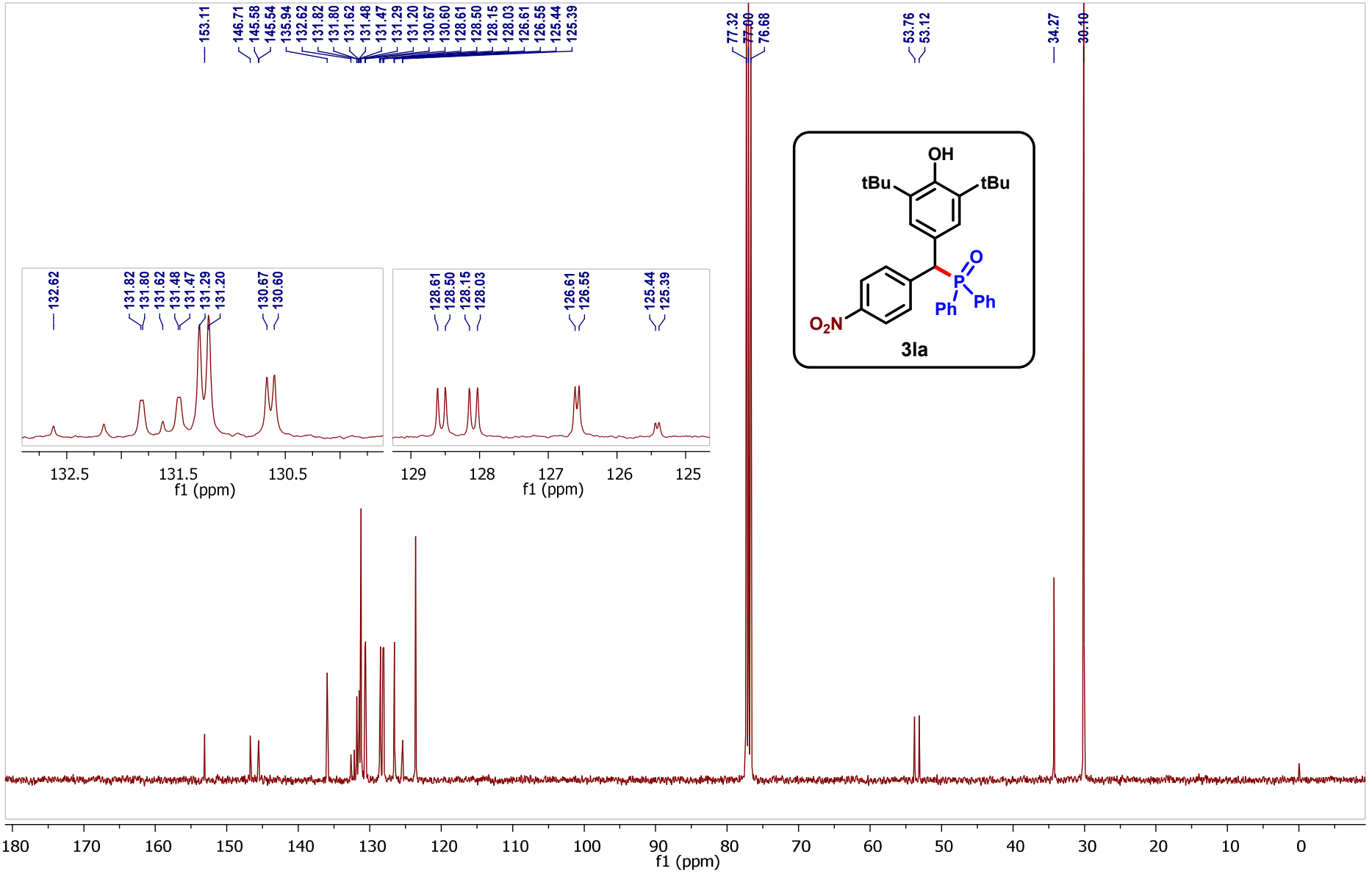
0.0
f1 (ppm)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128.61
128.50
128.15
128.03
126.61
126.55
125.44
125.39
77.32
77.00
76.68
53.76
53.12
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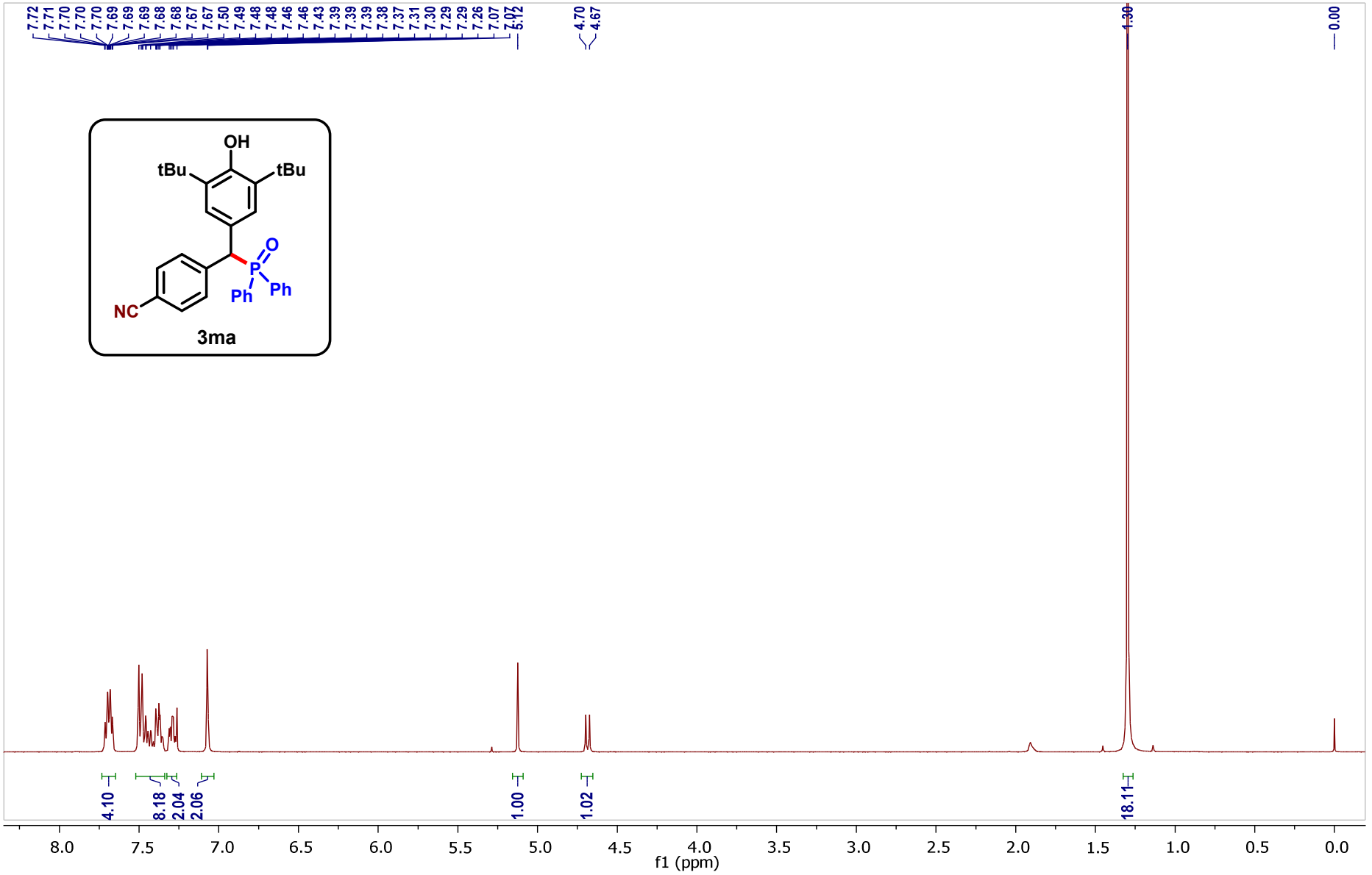
tBu
O
P
Ph
Ph
O2N
3la
f1 (ppm)
f1 (ppm)
f1 (ppm)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7.30
7.29
7.29
7.26
7.07
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0.00
OH
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tBu
O
P
Ph
Ph
NC
3ma
4.10
8.18
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2.06
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1.02
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7.0
6.5
6.0
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2.5
2.0
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1.0
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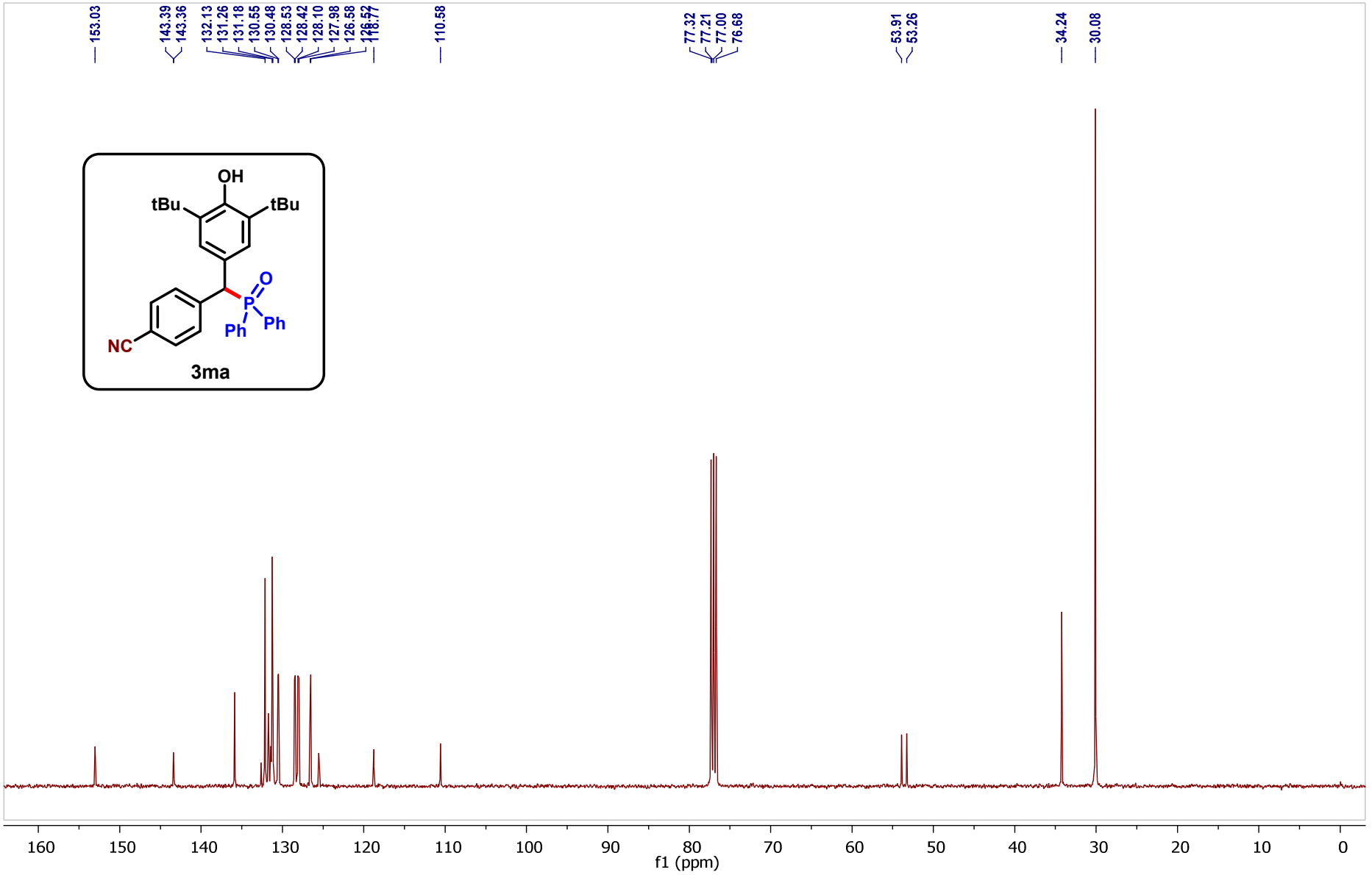
f1 (ppm)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77.32
77.21
77.00
76.68
53.91
53.26
34.24
30.08
OH
tBu
tBu
O
P
Ph
Ph
NC
3ma
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130
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110
100
90
80
70
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50
40
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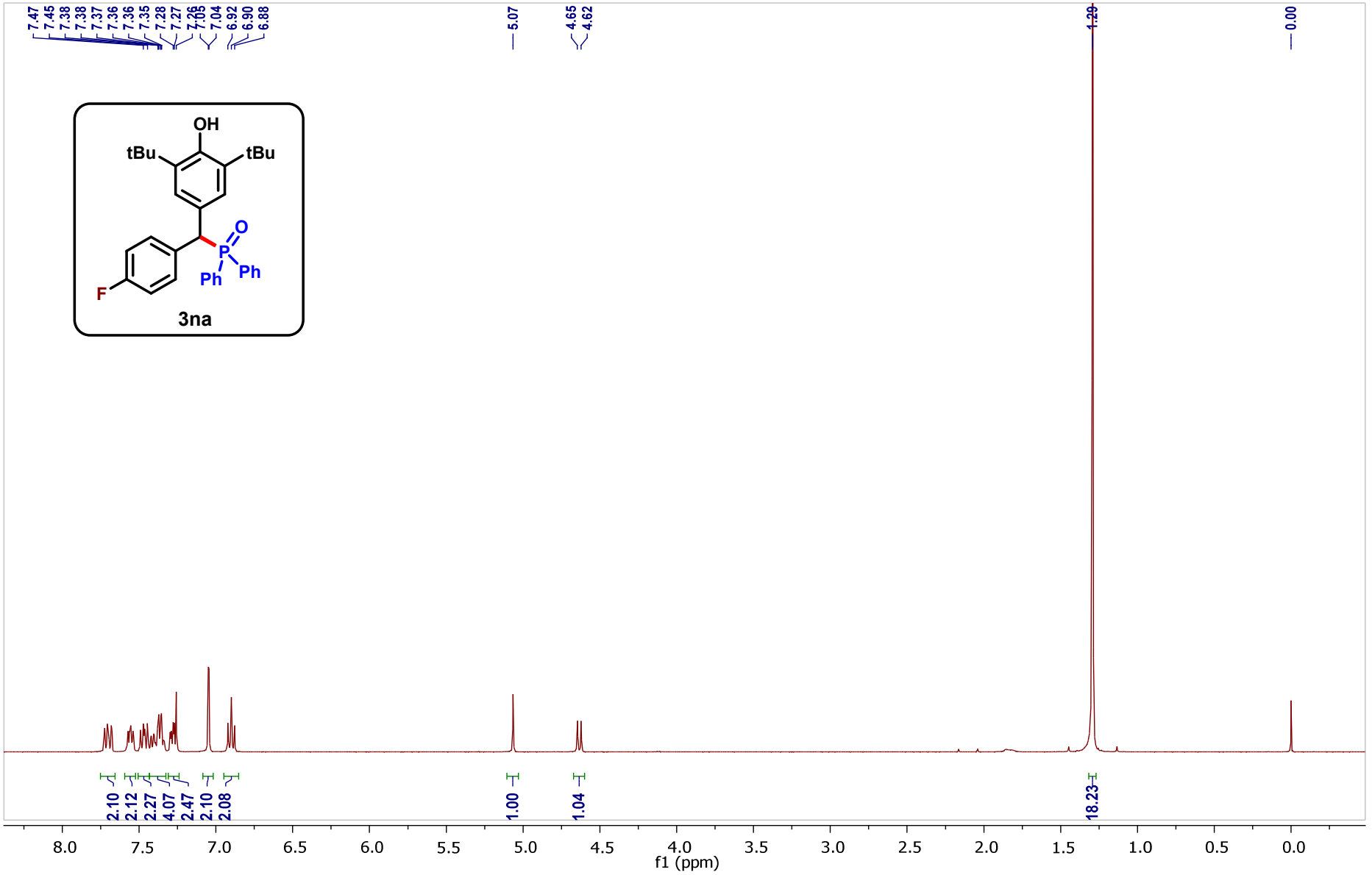
10
0
f1 (ppm)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6.88
5.07
4.65
4.62
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tBu
O
P
Ph
Ph
F
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2.10
2.12
2.27
4.07
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2.10
2.08
1.00
1.04
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6.5
6.0
5.5
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2.0
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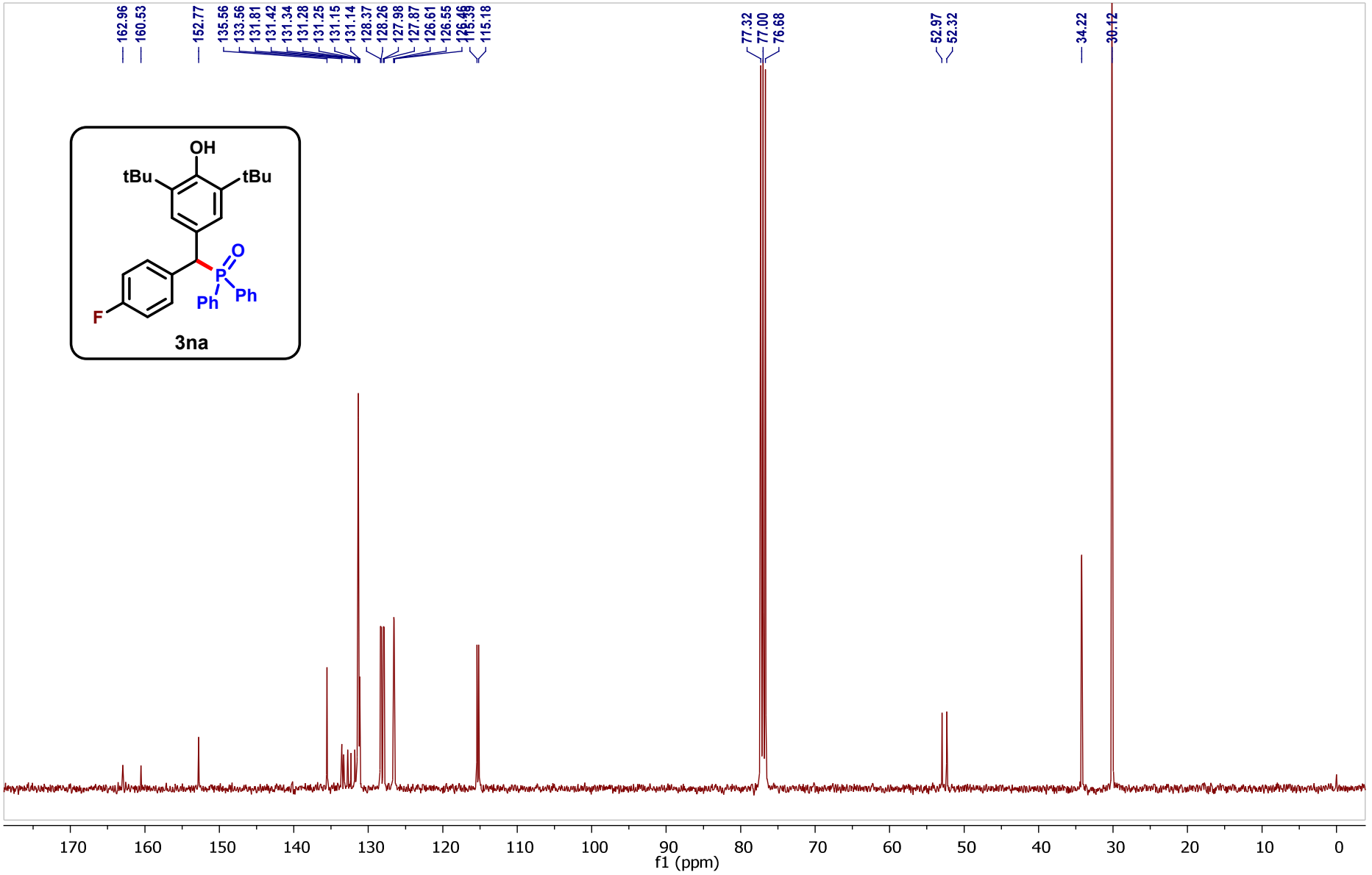
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77.00
76.68
52.97
52.32
34.22
30.12
OH
tBu
tBu
O
P
Ph
Ph
F
3na
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110
100
90
80
70
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50
40
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10
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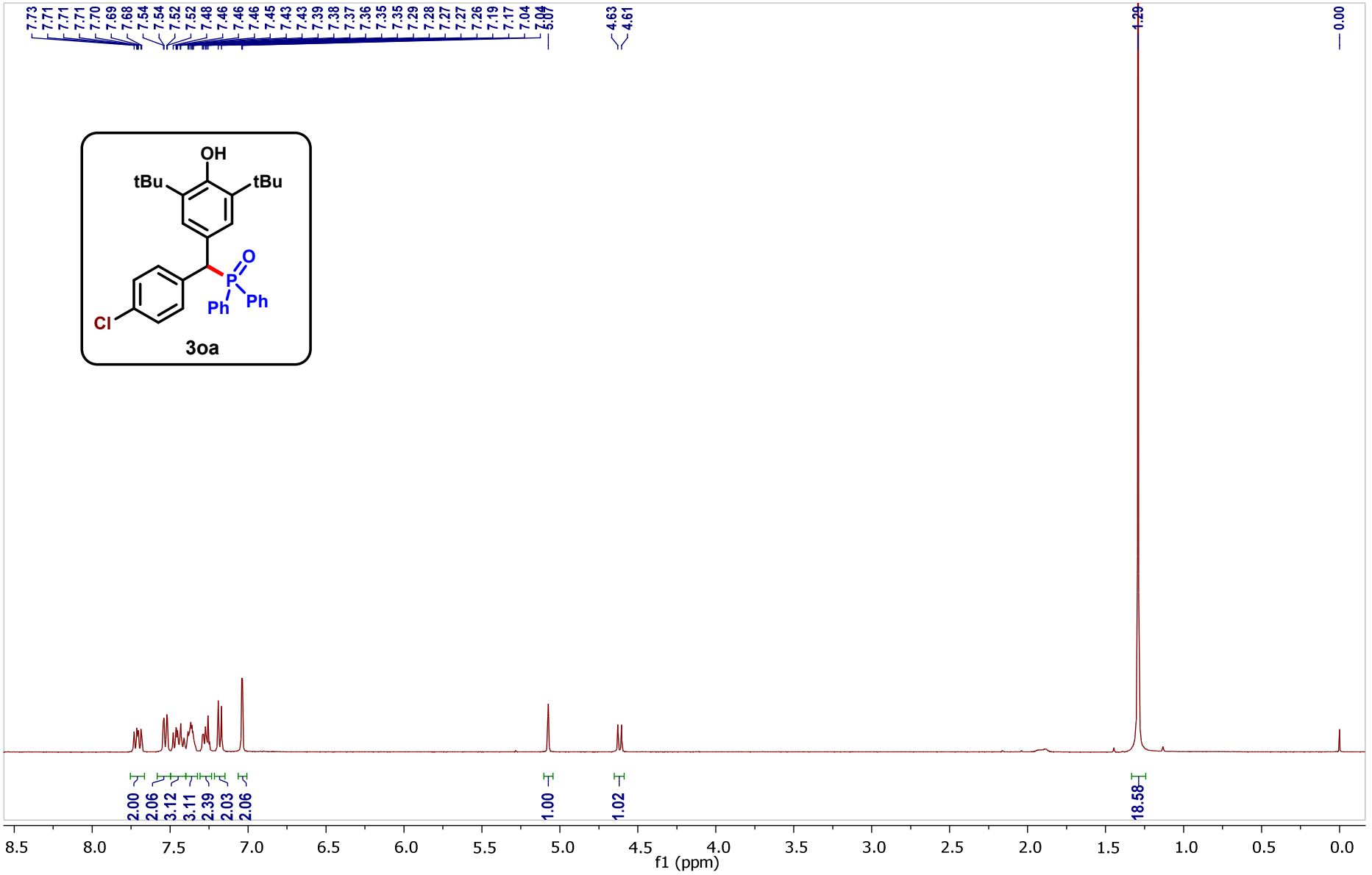
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7.04
5.07
4.63
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1.29
0.00
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tBu
tBu
O
P
Ph
Ph
Cl
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2.06
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2.03
2.06
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1.02
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6.0
5.5
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2.5
2.0
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1.0
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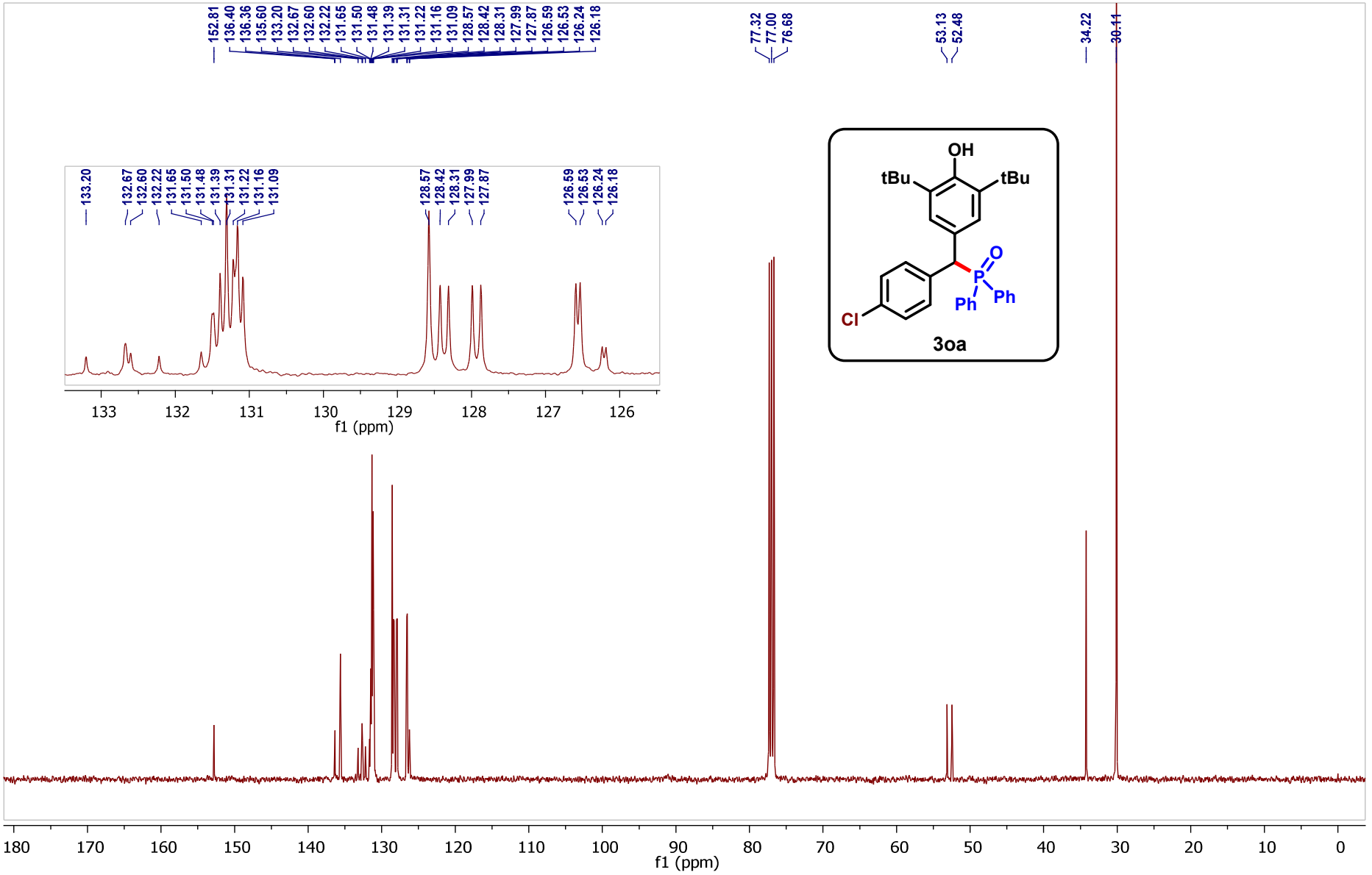
0.0
f1 (ppm)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77.00
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53.13
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OH
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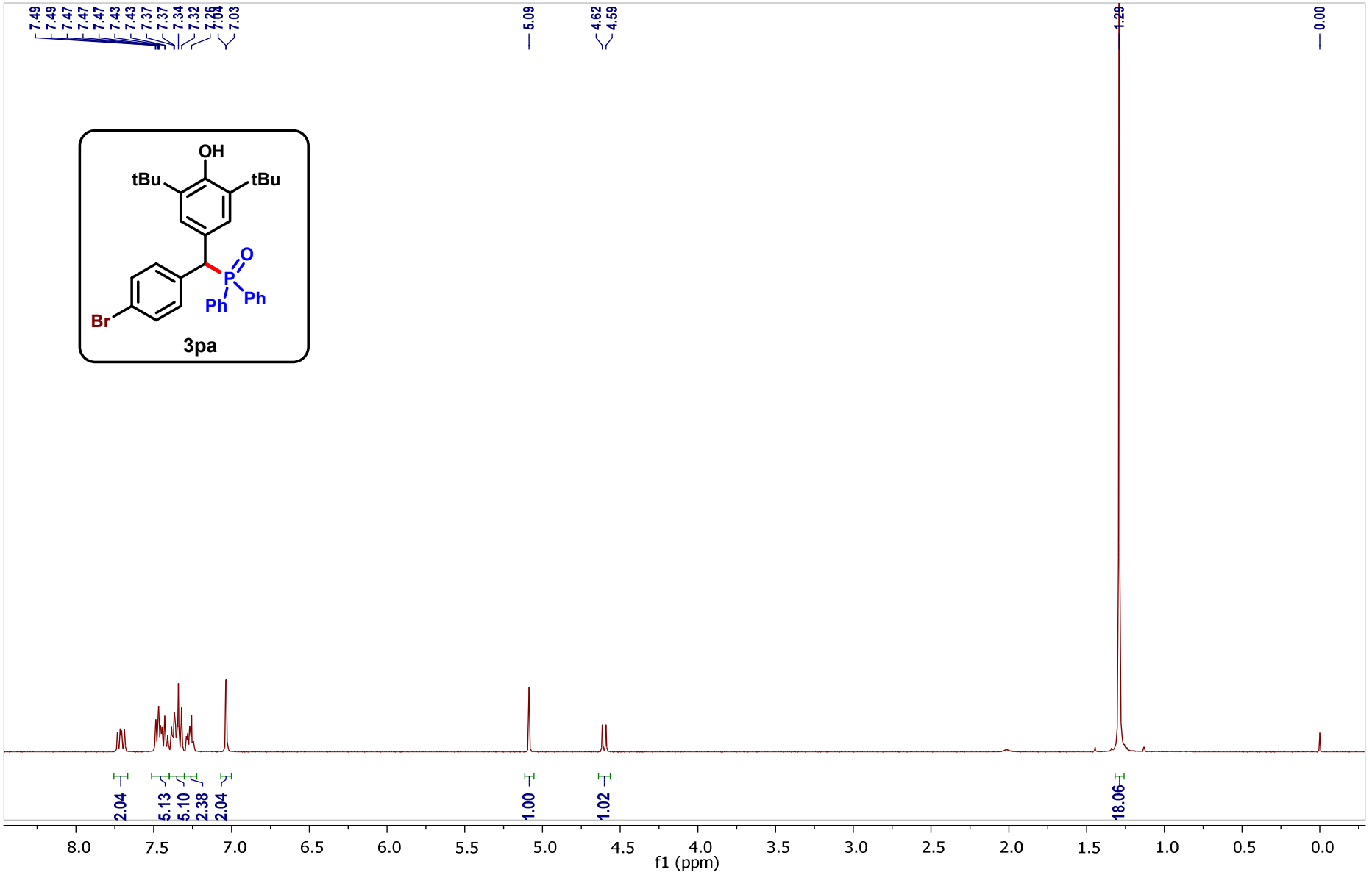
tBu
O
P
Ph
Ph
Cl
3oa
f1 (ppm)
f1 (ppm)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7.03
5.09
4.62
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tBu
O
P
Ph
Ph
Br
3pa
2.04
5.13
5.10
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2.04
1.00
1.02
18.06
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6.5
6.0
5.5
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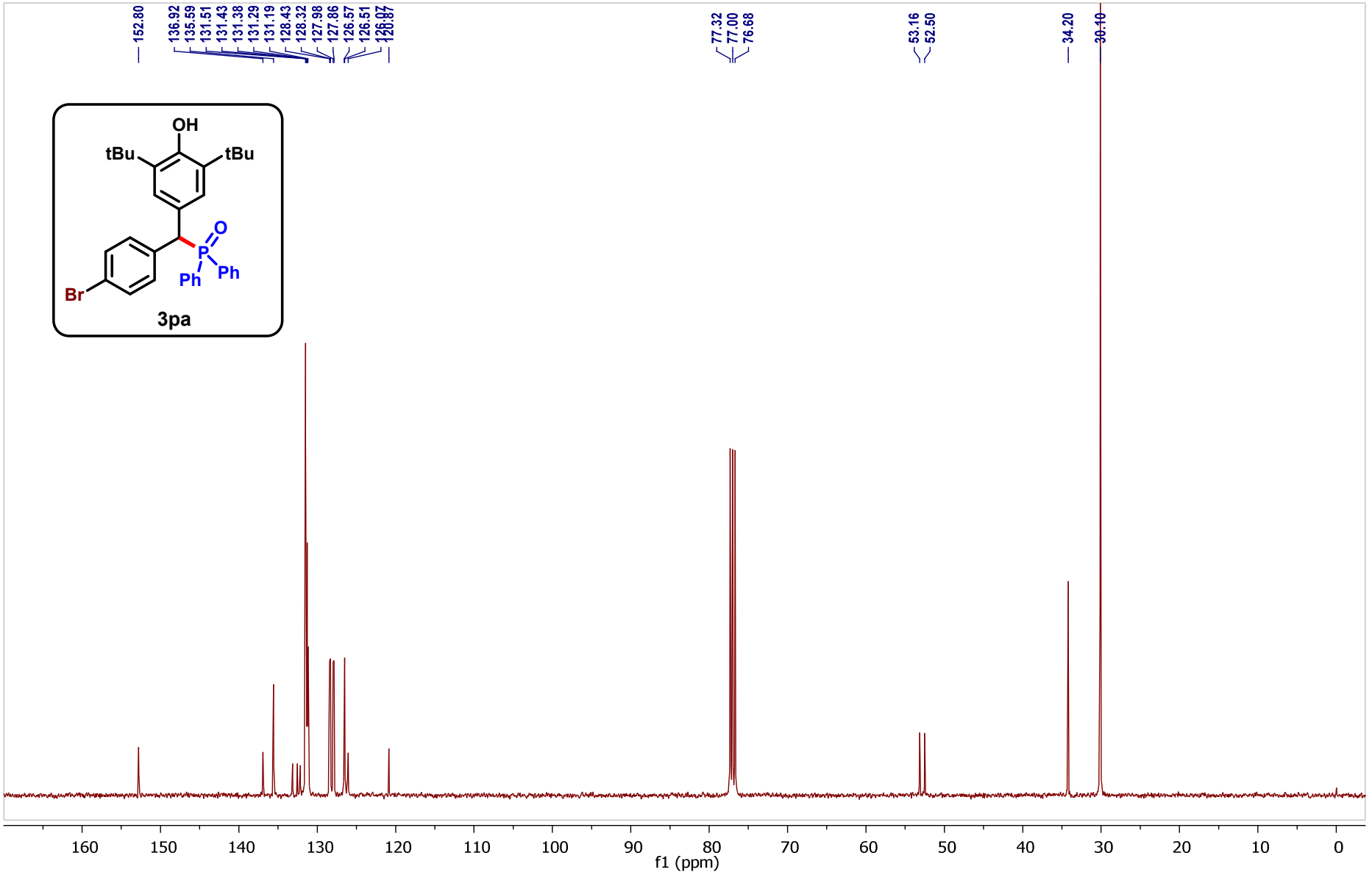
f1 (ppm)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77.00
76.68
53.16
52.50
34.20
30.10
OH
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tBu
O
P
Ph
Ph
Br
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50
40
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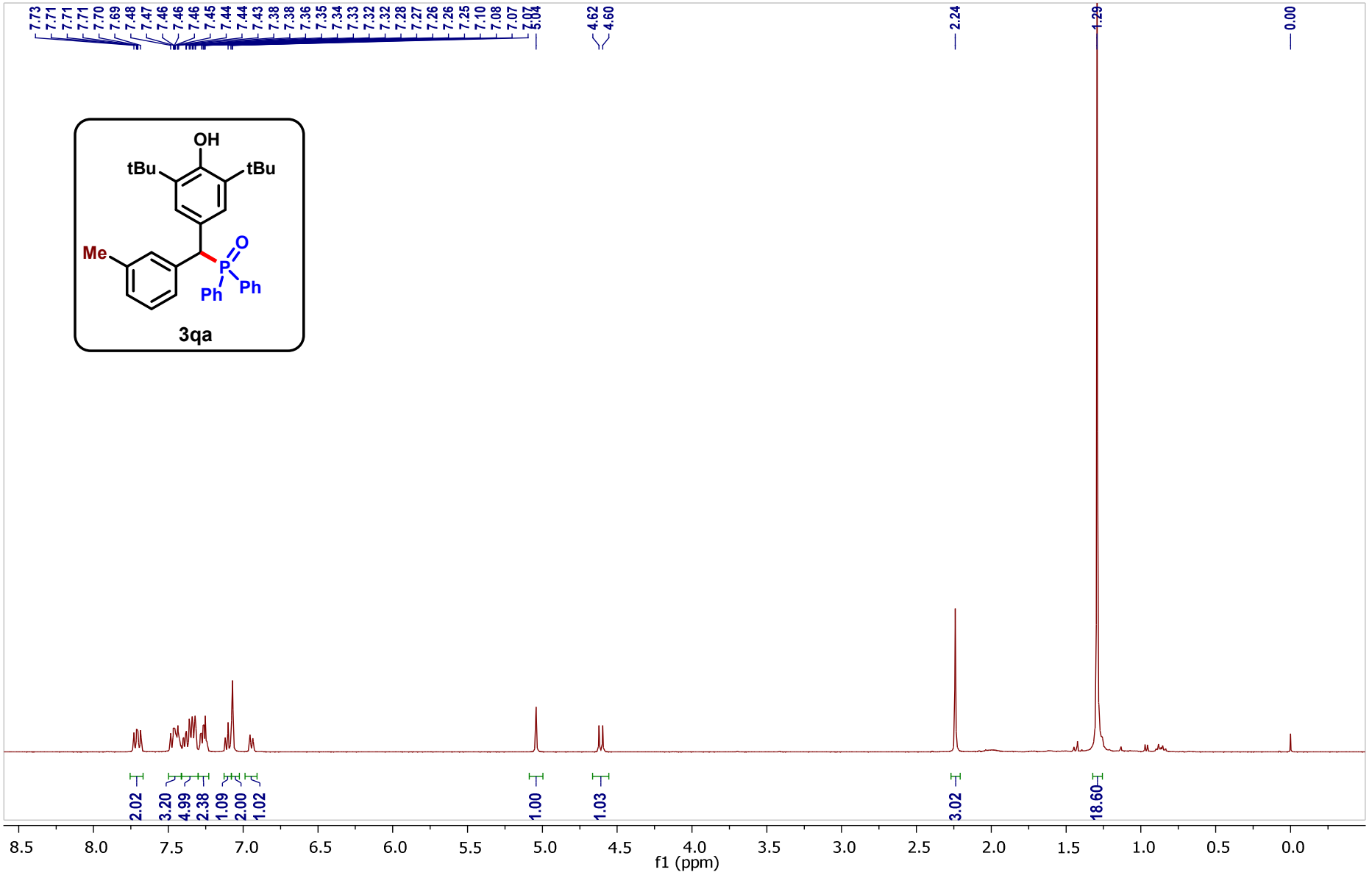
10
0
f1 (ppm)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7.34
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7.32
7.32
7.28
7.27
7.26
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7.25
7.10
7.08
7.07
5.04
4.62
4.60
2.24
1.29
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tBu
tBu
Me
O
P
Ph
Ph
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2.02
3.20
4.99
2.38
1.09
2.00
1.02
1.00
1.03
3.02
18.60
8.5
8.0
7.5
7.0
6.5
6.0
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2.5
2.0
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1.0
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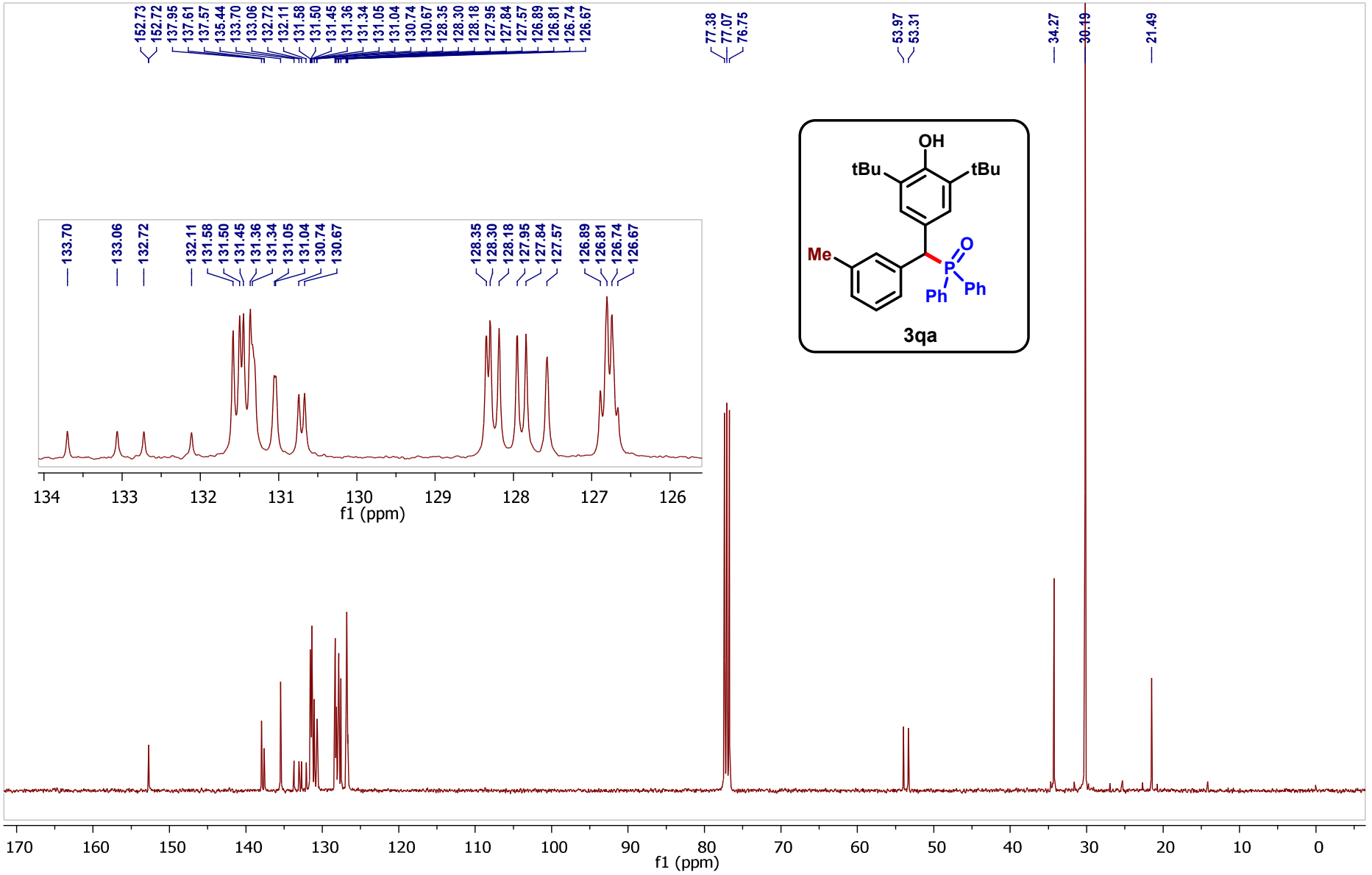
0.0
f1 (ppm)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126.67
77.38
77.07
76.75
53.97
53.31
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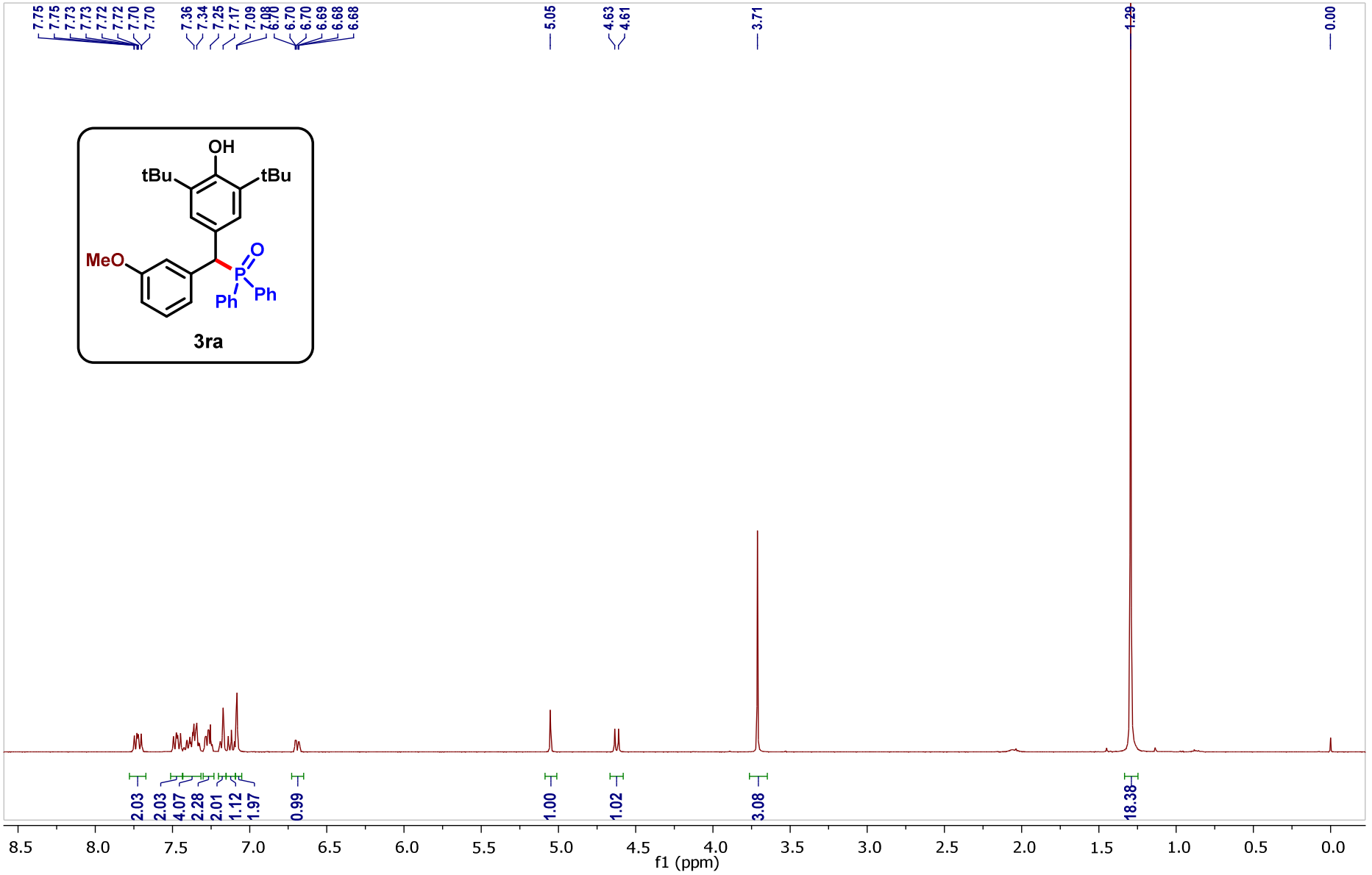
21.49
OH
tBu
tBu
Me
O
P
Ph
Ph
3qa
f1 (ppm)
f1 (ppm)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4.61
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0.00
OH
tBu
tBu
MeO
O
P
Ph
Ph
3ra
2.03
2.03
4.07
2.28
2.01
1.12
1.97
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1.00
1.02
3.08
18.38
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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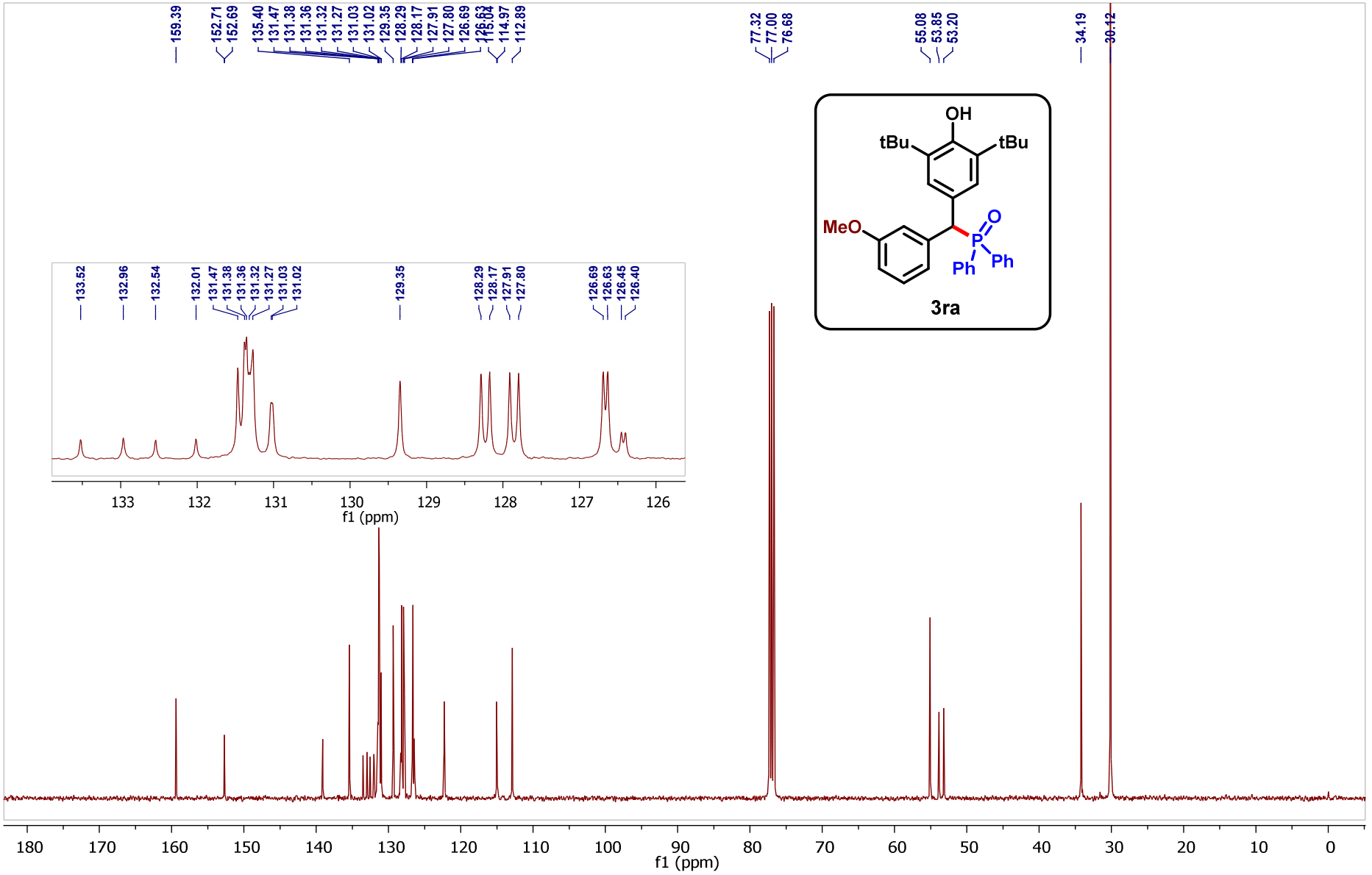
f1 (ppm)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126.45
126.40
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114.97
112.89
77.32
77.00
76.68
55.08
53.85
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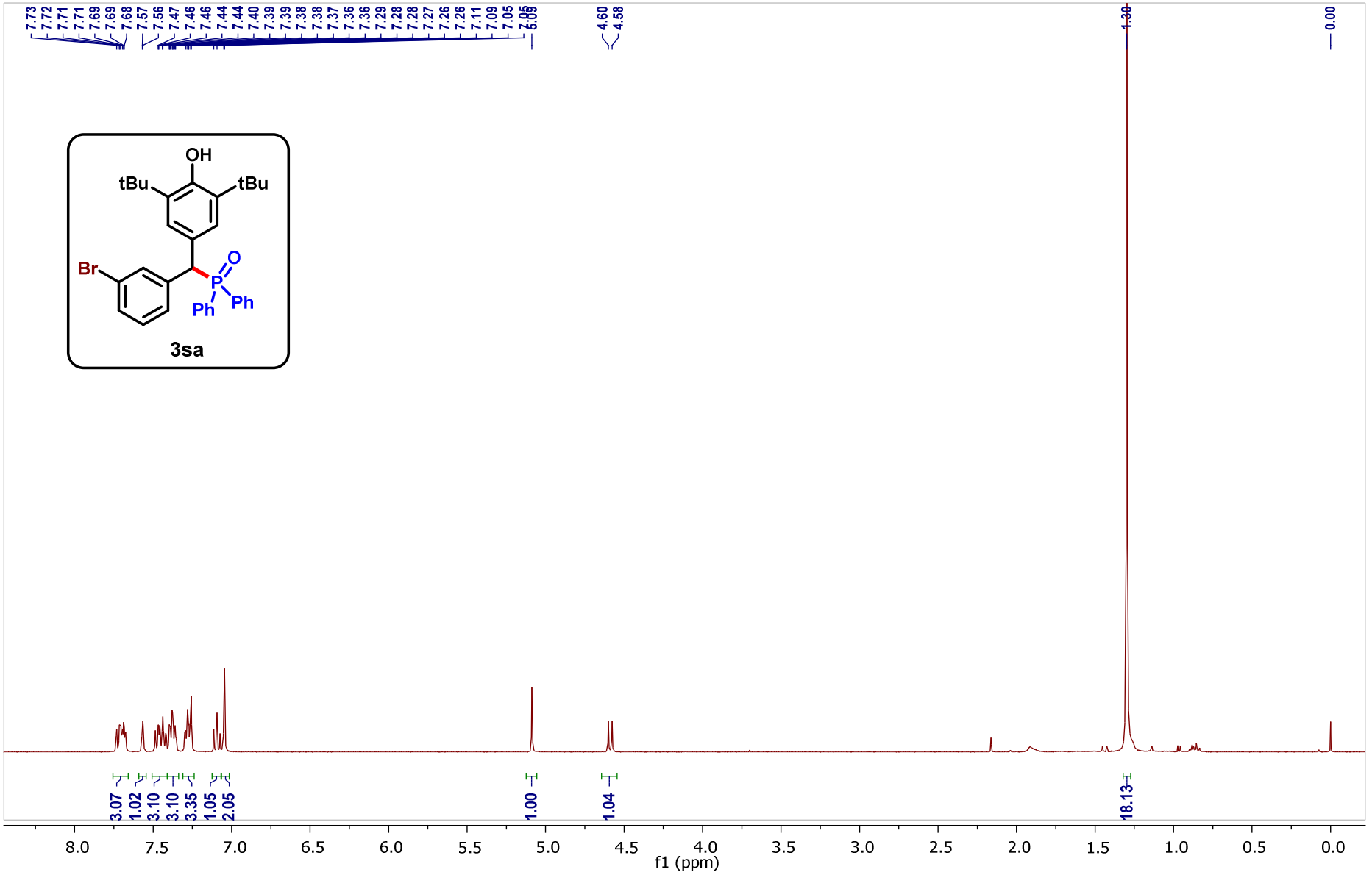
OH
tBu
tBu
MeO
O
P
Ph
Ph
3ra
f1 (ppm)
f1 (ppm)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7.28
7.28
7.27
7.26
7.26
7.11
7.09
7.05
5.09
4.60
4.58
1.30
0.00
OH
tBu
tBu
Br
O
P
Ph
Ph
3sa
3.07
1.02
3.10
3.10
3.35
1.05
2.05
1.00
1.04
18.13
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7.0
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6.0
5.5
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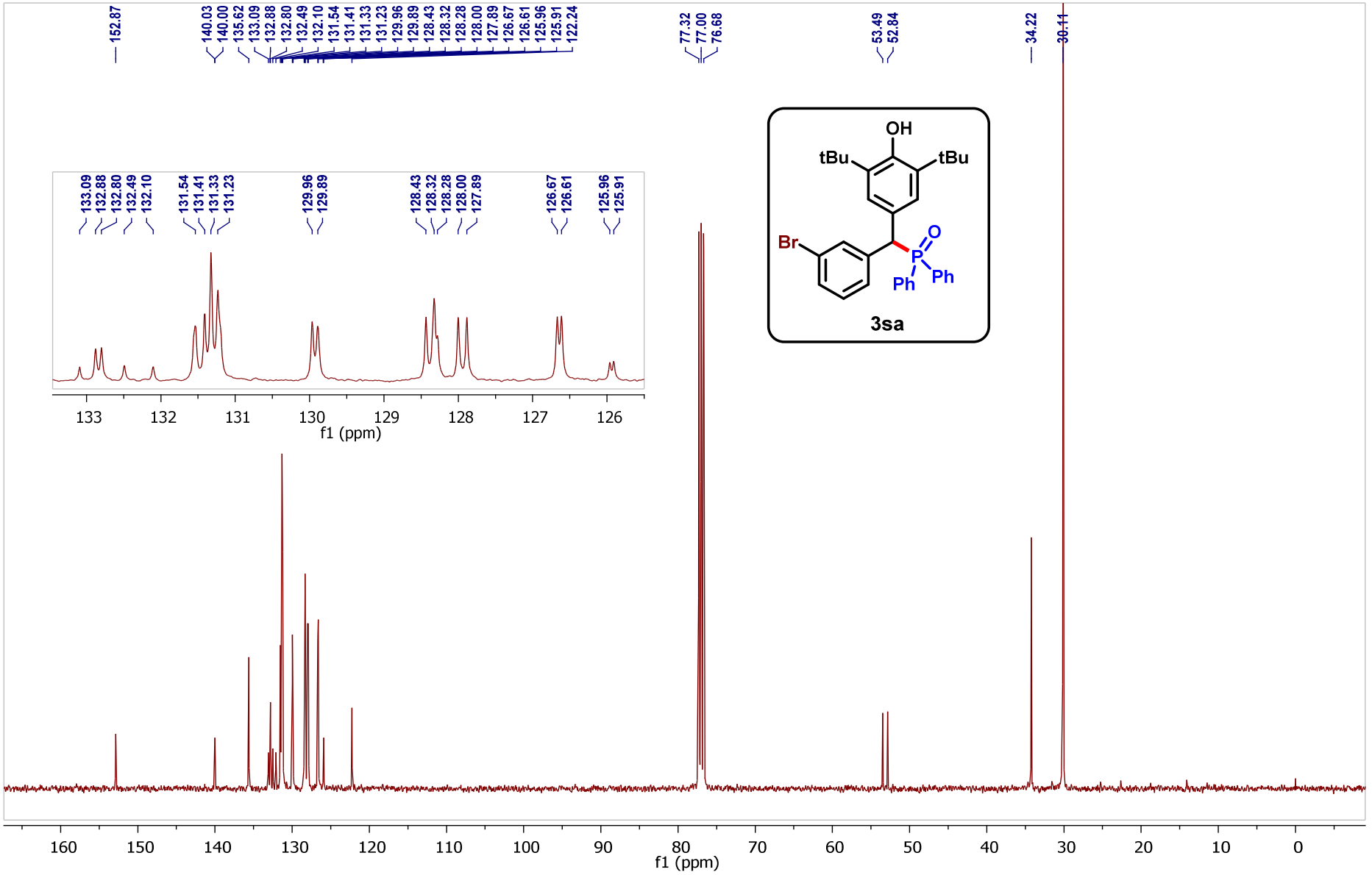
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128.32
128.28
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126.67
126.61
125.96
125.91
122.24
77.32
77.00
76.68
53.49
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OH
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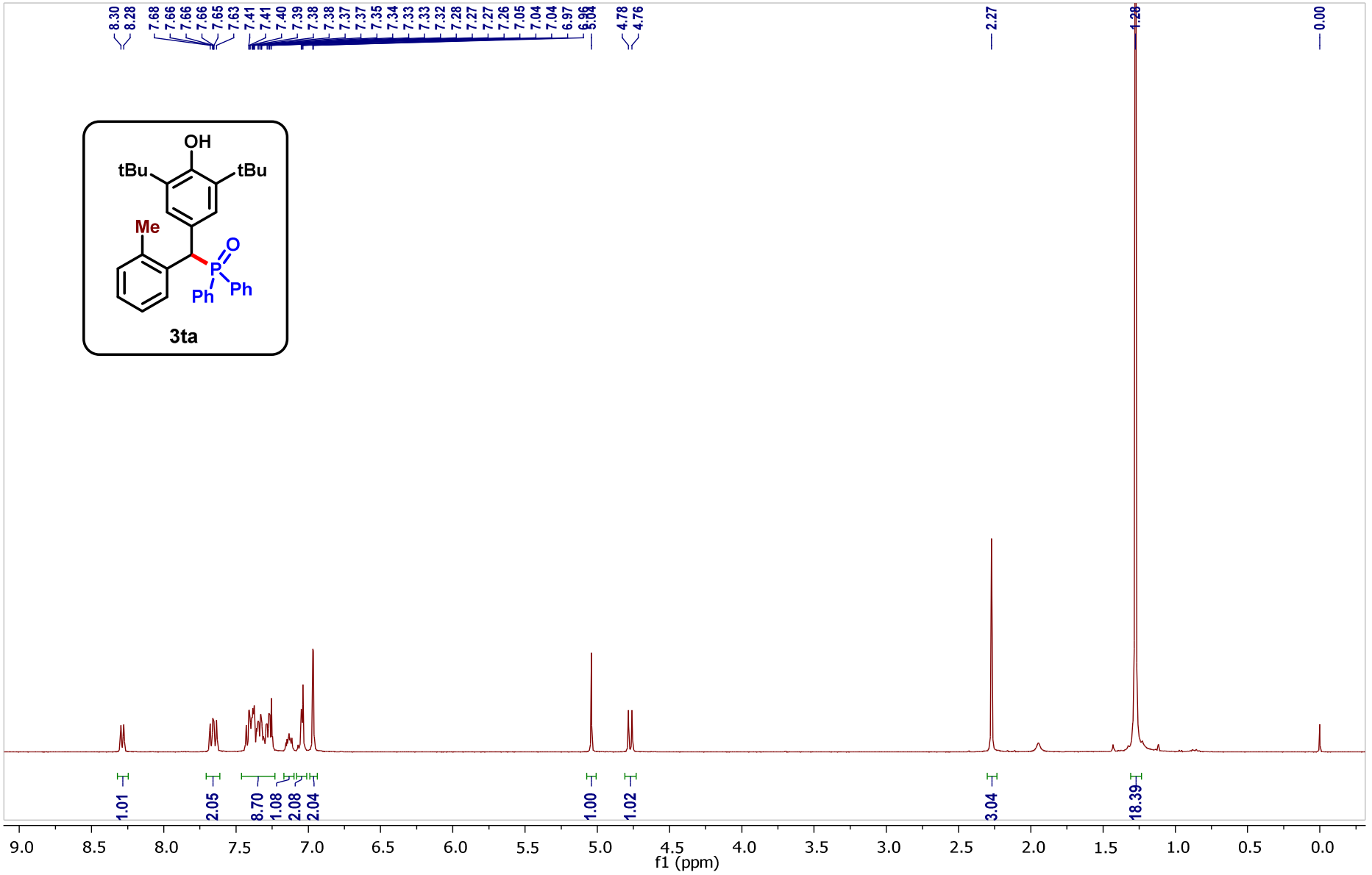
tBu
Br
O
P
Ph
Ph
3sa
f1 (ppm)
f1 (ppm)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7.37
7.37
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7.34
7.33
7.33
7.32
7.28
7.27
7.27
7.26
7.05
7.04
7.04
6.97
5.04
4.78
4.76
2.27
1.28
0.00
OH
tBu
tBu
Me
O
P
Ph
Ph
3ta
1.01
2.05
8.70
1.08
2.08
2.04
1.00
1.02
3.04
18.39
9.0
8.5
8.0
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7.0
6.5
6.0
5.5
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2.5
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1.0
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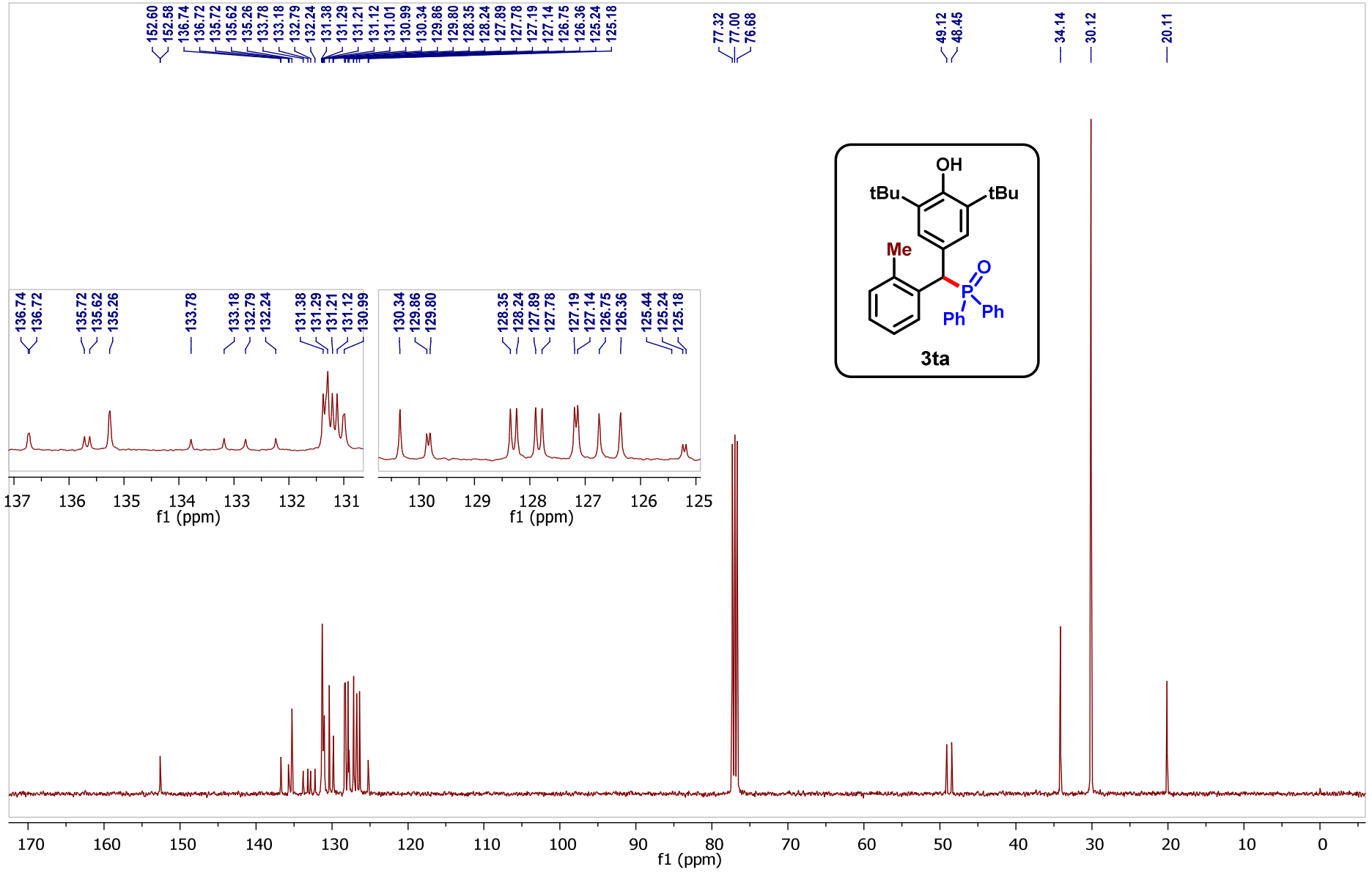
f1 (ppm)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131.01
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130.34
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128.24
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125.18
77.32
77.00
76.68
49.12
48.45
34.14
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20.11
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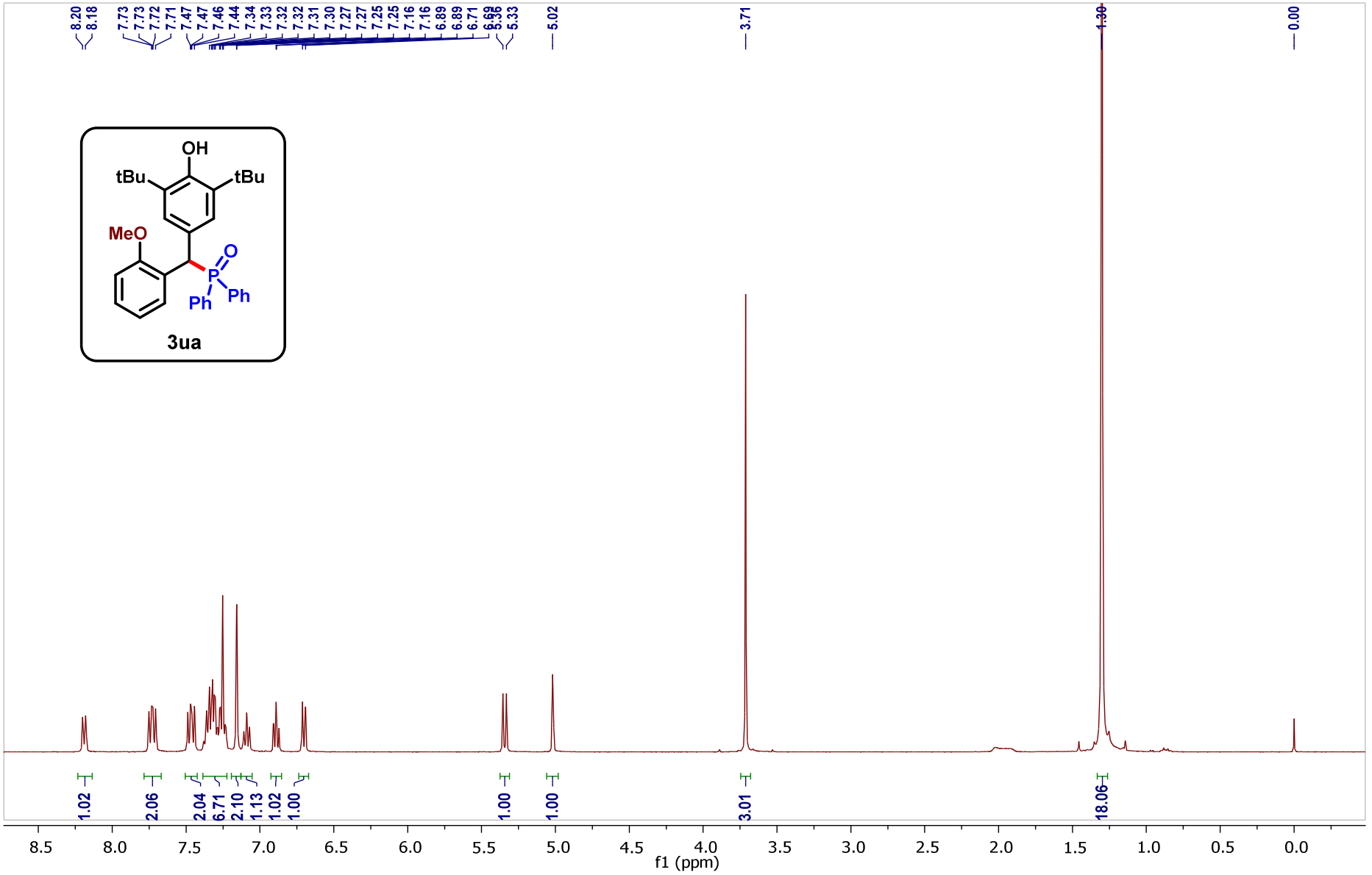
tBu
tBu
Me
O
P
Ph
Ph
3ta
f1 (ppm)
f1 (ppm)
f1 (ppm)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7.34
7.33
7.32
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7.31
7.30
7.27
7.27
7.25
7.25
7.16
7.16
6.89
6.89
6.71
5.33
5.02
3.71
1.30
0.00
OH
tBu
tBu
MeO
O
P
Ph
Ph
3ua
1.02
2.06
2.04
6.71
2.10
1.13
1.02
1.00
1.00
1.00
3.01
18.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
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2.0
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1.0
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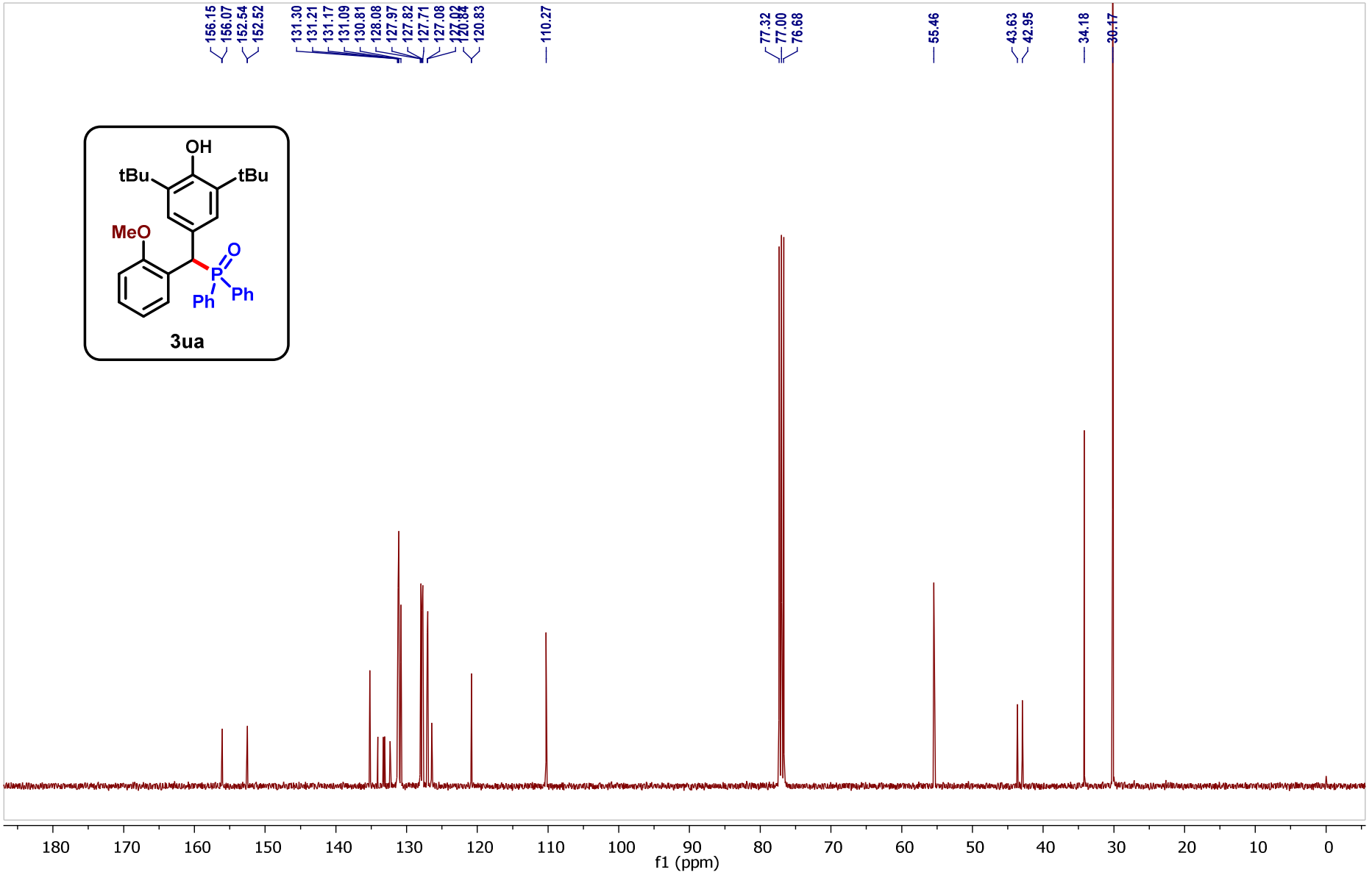
f1 (ppm)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127.71
127.08
120.84
120.83
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77.32
77.00
76.68
55.46
43.63
42.95
34.18
30.17
OH
tBu
tBu
MeO
O
P
Ph
Ph
3ua
180
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130
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110
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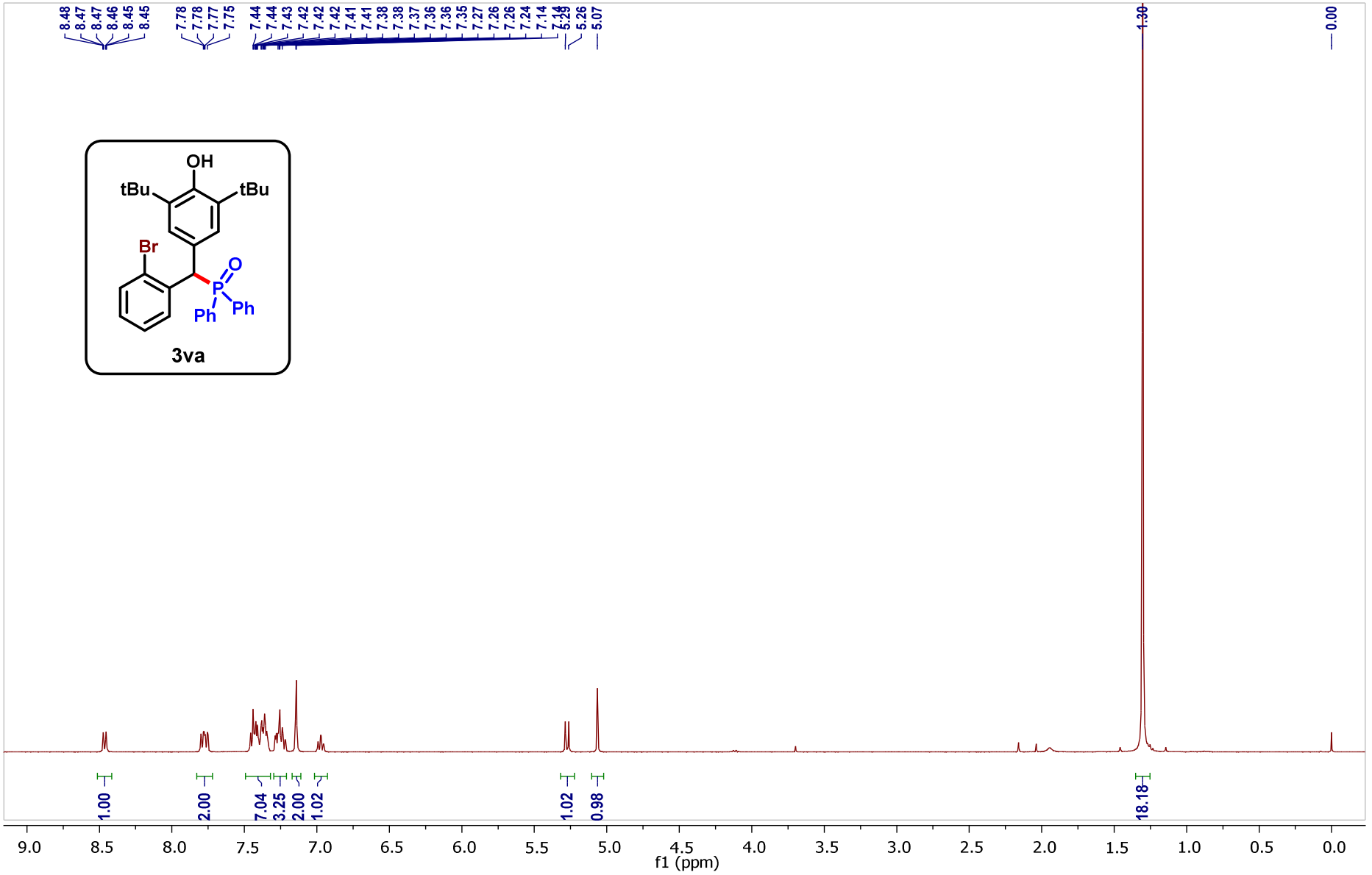
f1 (ppm)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7.78
7.77
7.75
7.44
7.44
7.43
7.42
7.42
7.42
7.41
7.41
7.38
7.38
7.37
7.36
7.36
7.35
7.27
7.26
7.26
7.24
7.14
5.26
5.07
1.30
0.00
OH
tBu
tBu
Br
O
P
Ph
Ph
3va
1.00
2.00
7.04
3.25
2.00
1.02
1.02
0.98
18.18
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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3.5
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2.5
2.0
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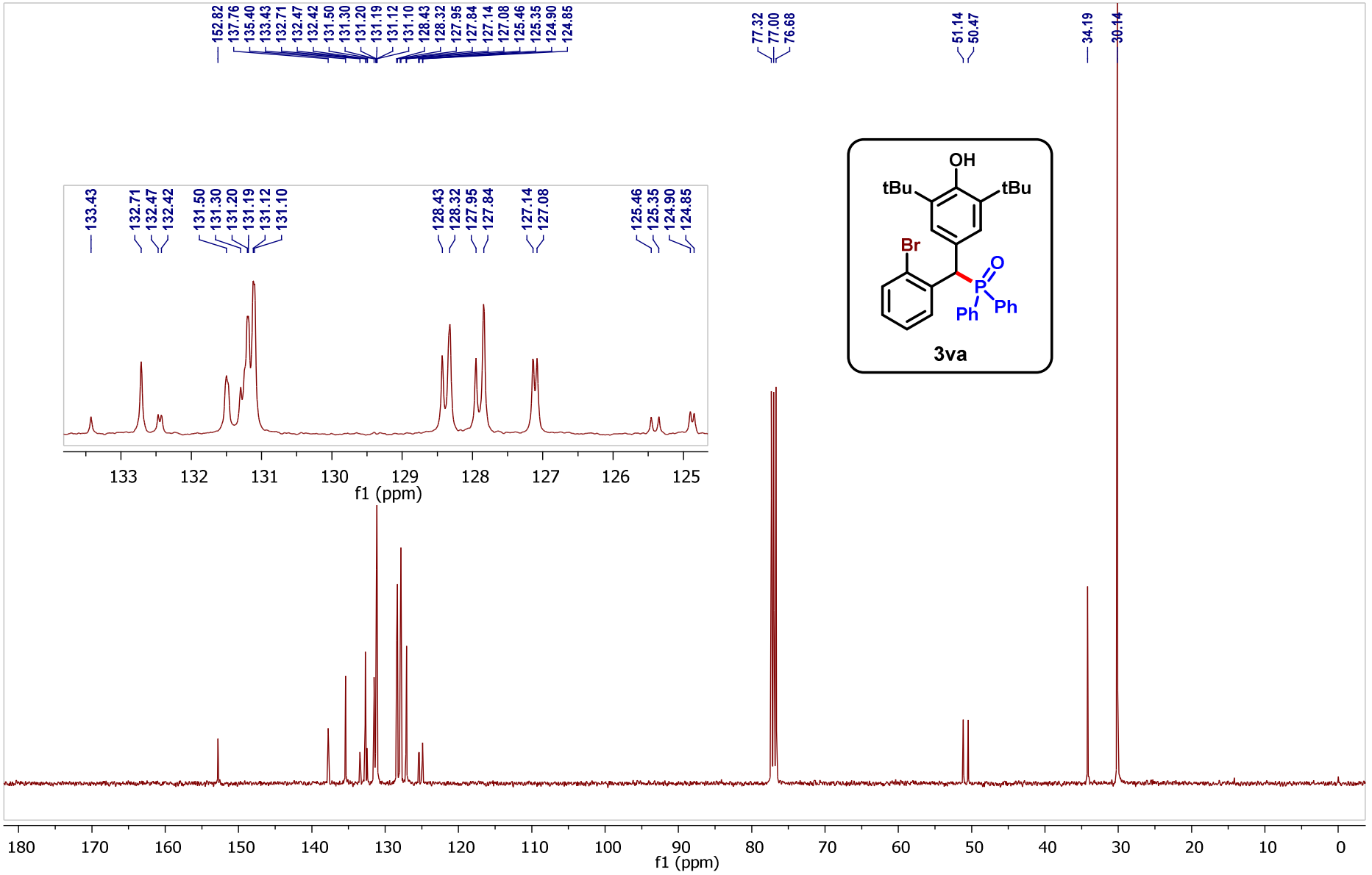
1.0
0.5
0.0
f1 (ppm)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128.32
127.95
127.84
127.14
127.08
125.46
125.35
124.90
124.85
77.32
77.00
76.68
51.14
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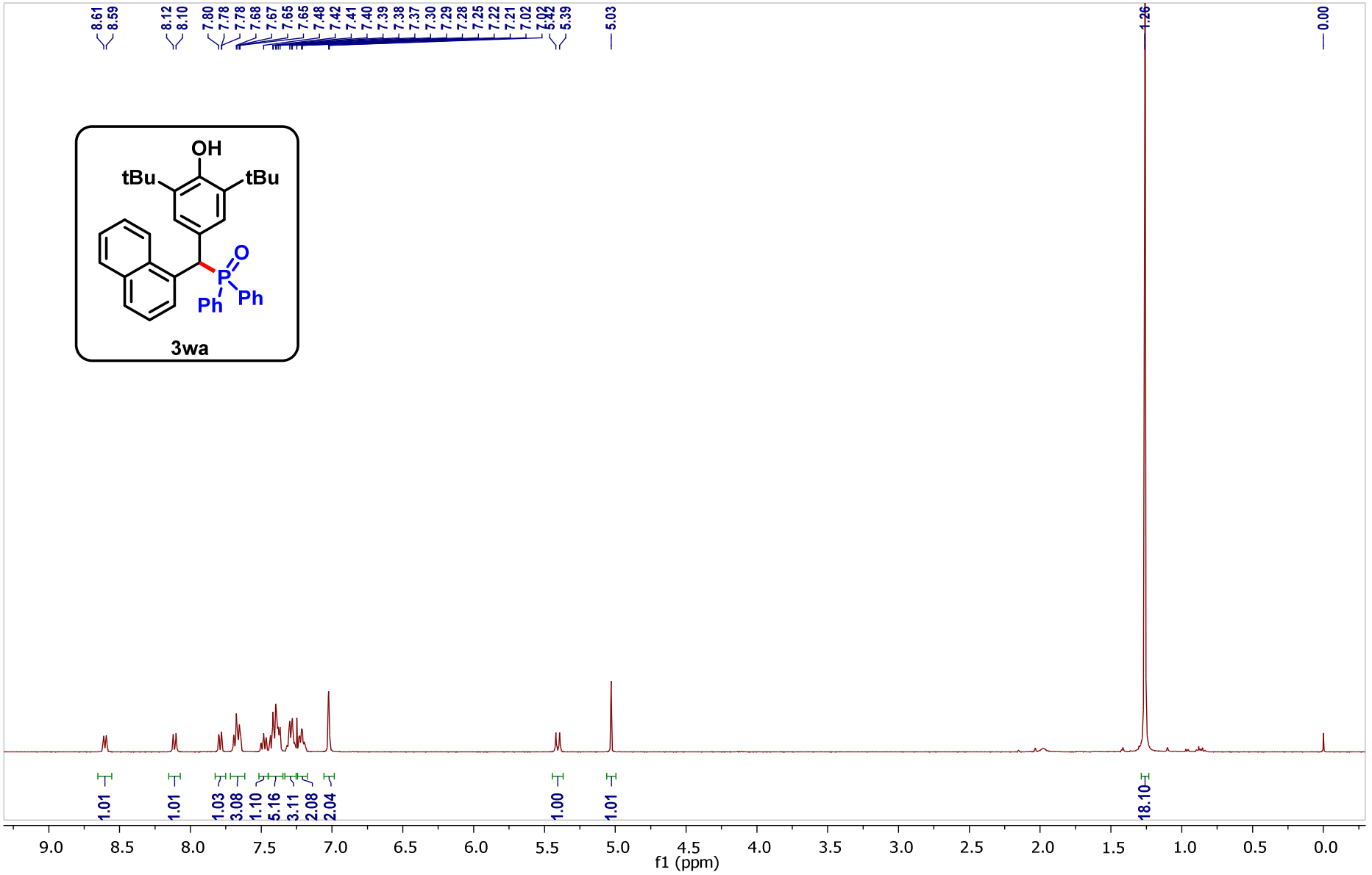
30.14
OH
tBu
tBu
Br
O
P
Ph
Ph
3va
f1 (ppm)
f1 (ppm)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7.48
7.42
7.41
7.40
7.39
7.38
7.37
7.30
7.29
7.28
7.25
7.22
7.21
7.02
7.02
5.42
5.39
5.03
1.26
0.00
OH
tBu
tBu
O
P
Ph
Ph
3wa
1.01
1.01
1.03
3.08
1.10
5.16
3.11
2.08
2.04
1.00
1.01
18.10
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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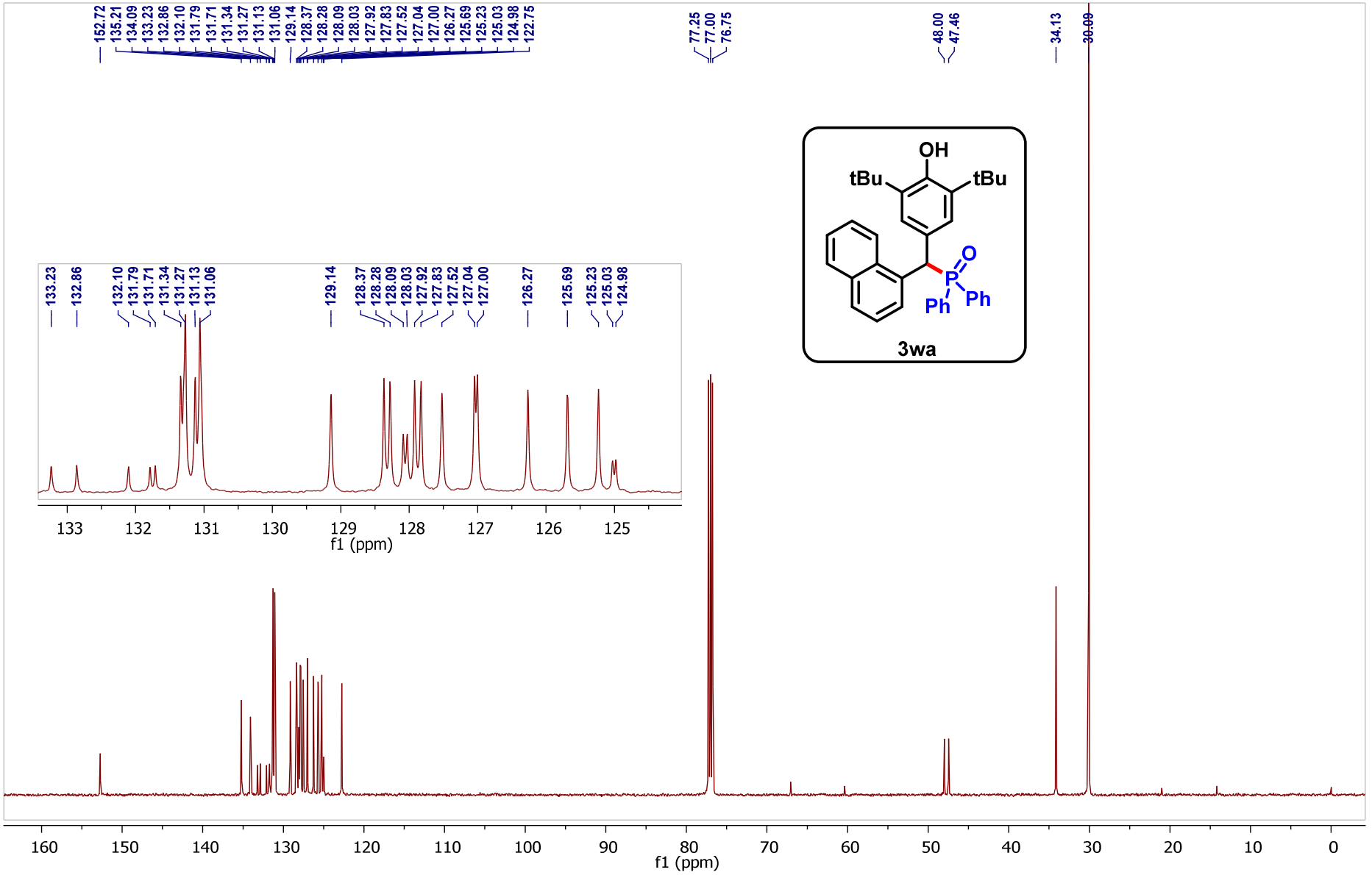
0.0
f1 (ppm)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128.28
128.09
128.03
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127.83
127.52
127.04
127.00
126.27
125.69
125.23
125.03
124.98
122.75
77.25
77.00
76.75
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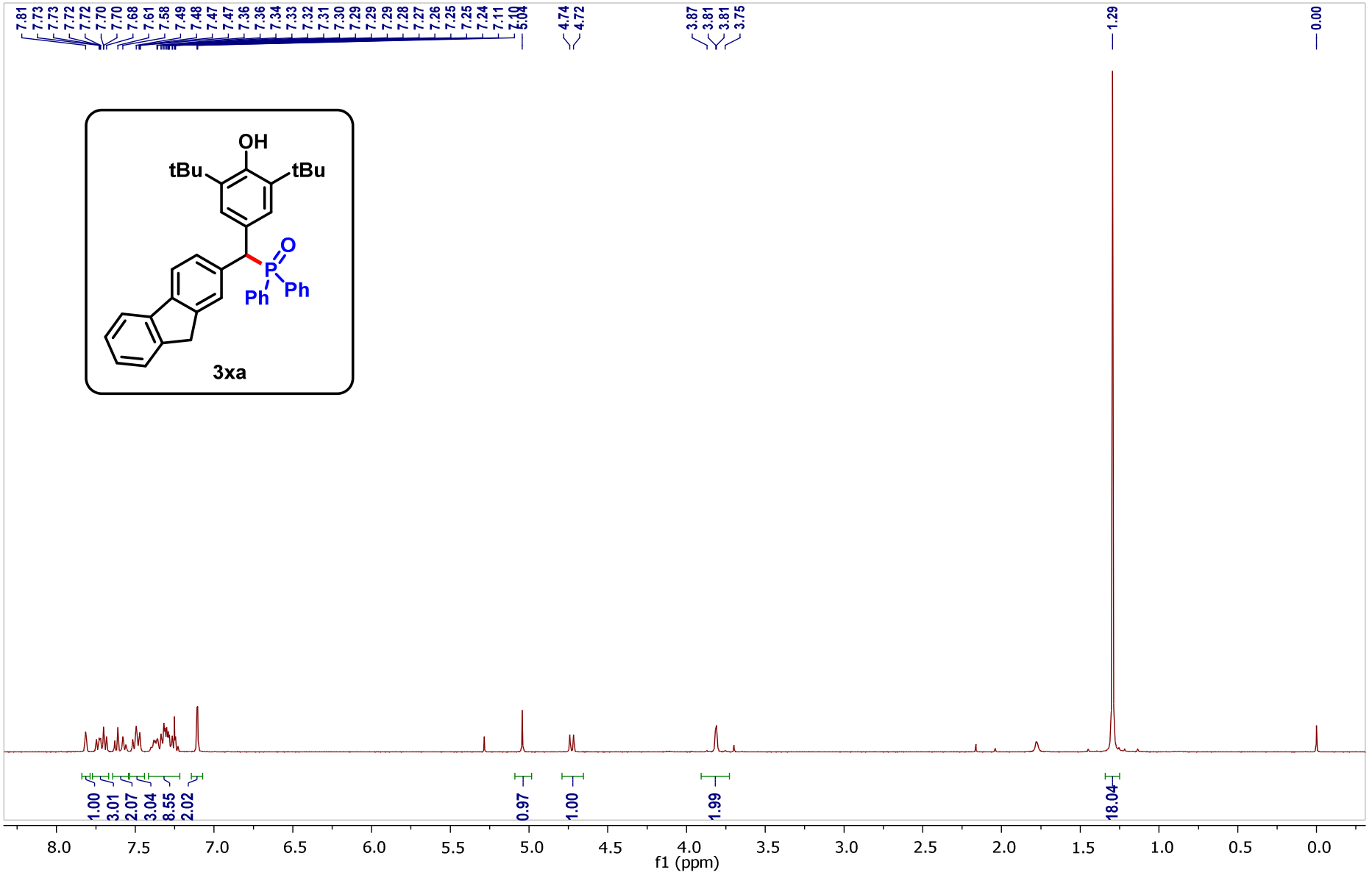
30.09
OH
tBu
tBu
O
P
Ph
Ph
3wa
f1 (ppm)
f1 (ppm)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7.26
7.25
7.25
7.24
7.11
7.10
5.04
4.74
4.72
3.87
3.81
3.81
3.75
1.29
0.00
OH
tBu
tBu
O
P
Ph
Ph
3xa
1.00
3.01
2.07
3.04
8.55
2.02
0.97
1.00
1.99
18.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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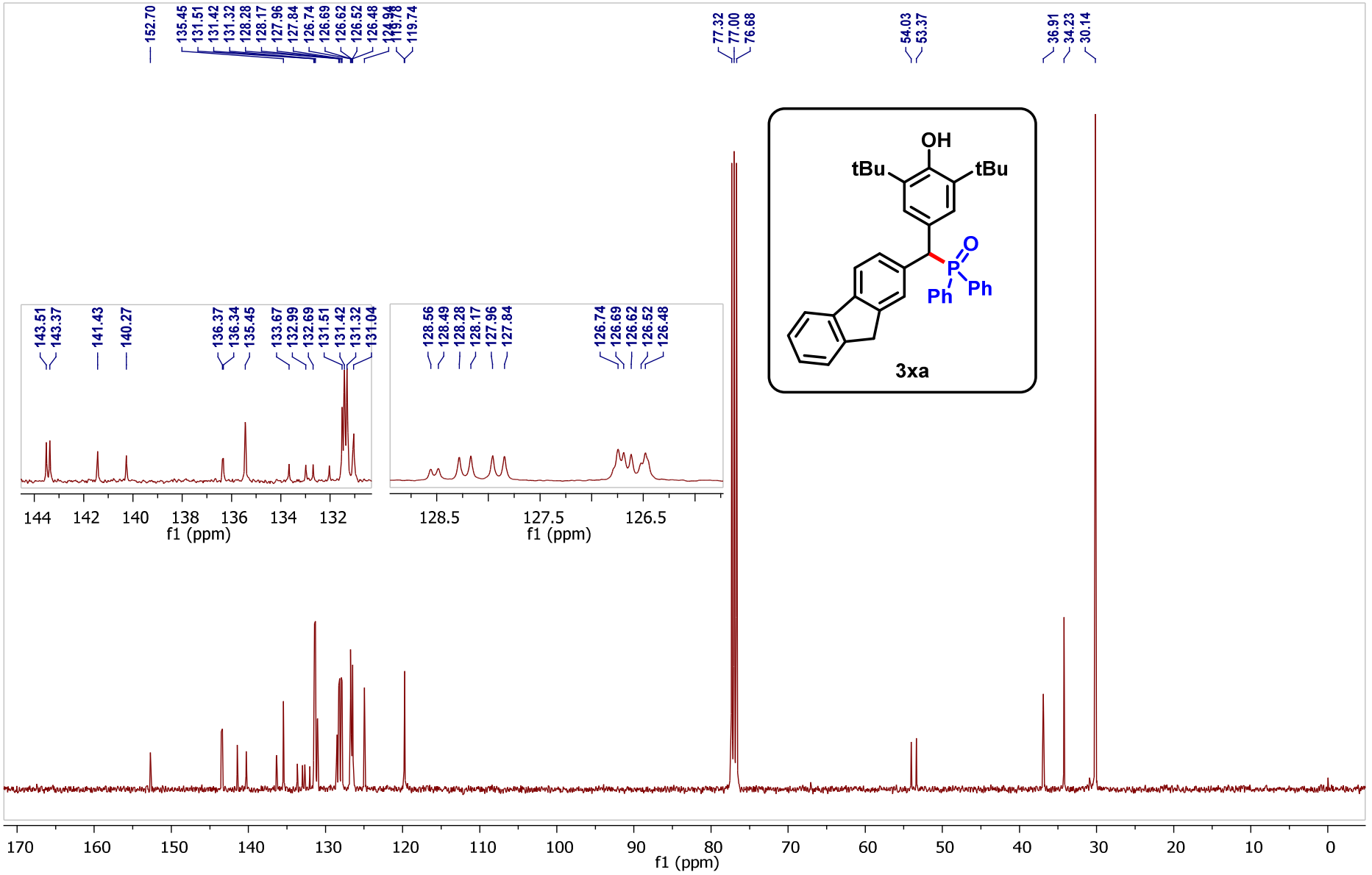
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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77.32
77.00
76.68
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36.91
34.23
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143.37
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126.52
126.48
144
142
140
138
136
134
132
f1 (ppm)
128.5
127.5
126.5
f1 (ppm)
OH
tBu
tBu
O
P
Ph
Ph
3xa
170
160
150
140
130
120
110
100
90
80
70
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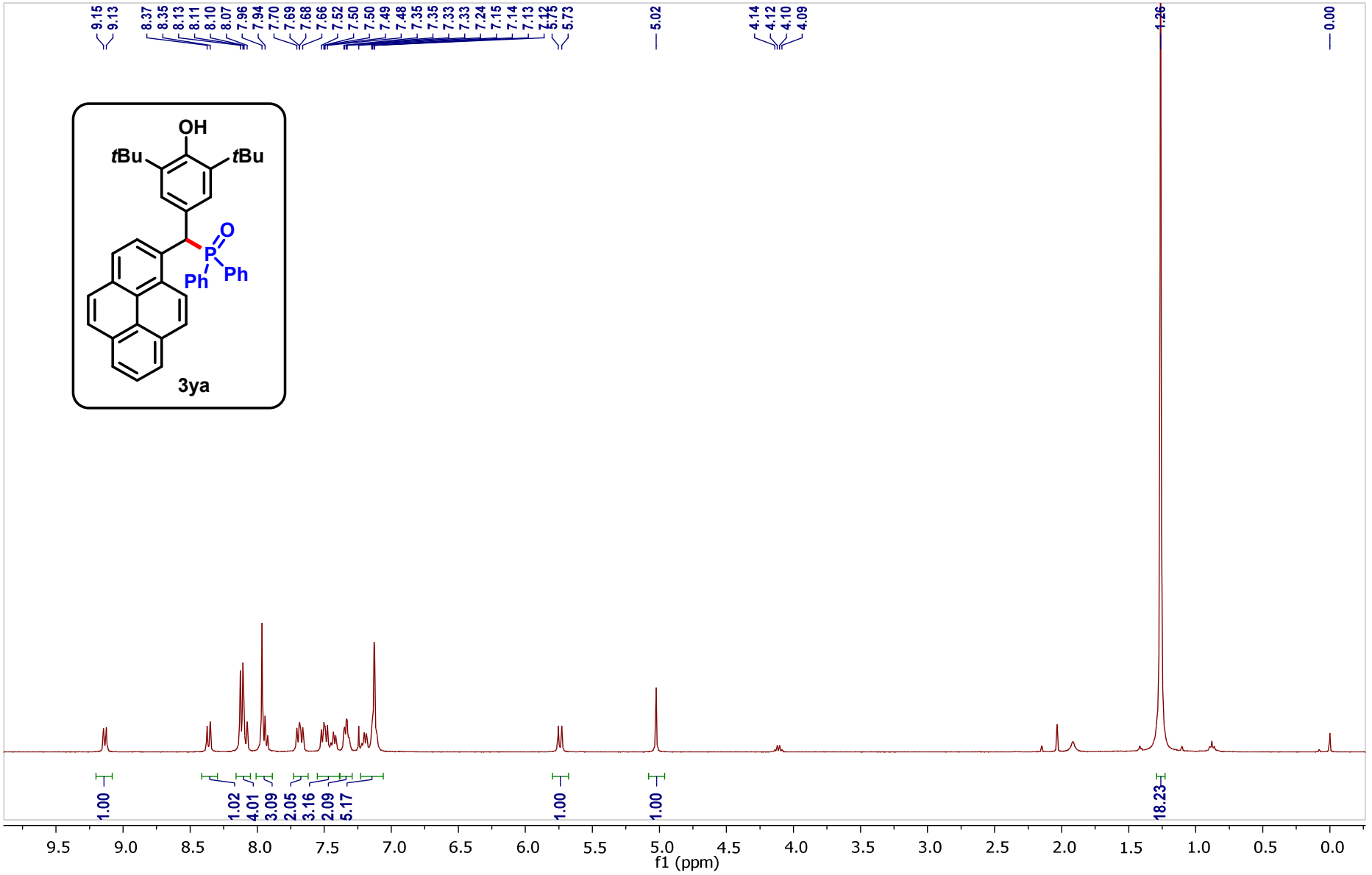
50
40
30
20
10
0
f1 (ppm)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7.94
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7.66
7.52
7.50
7.50
7.49
7.48
7.35
7.35
7.33
7.33
7.24
7.15
7.14
7.13
5.75
5.73
5.02
4.14
4.12
4.10
4.09
1.26
0.00
OH
tBu
tBu
O
P
Ph
Ph
3ya
1.00
1.02
4.01
3.09
2.05
3.16
2.09
5.17
1.00
1.00
18.23
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
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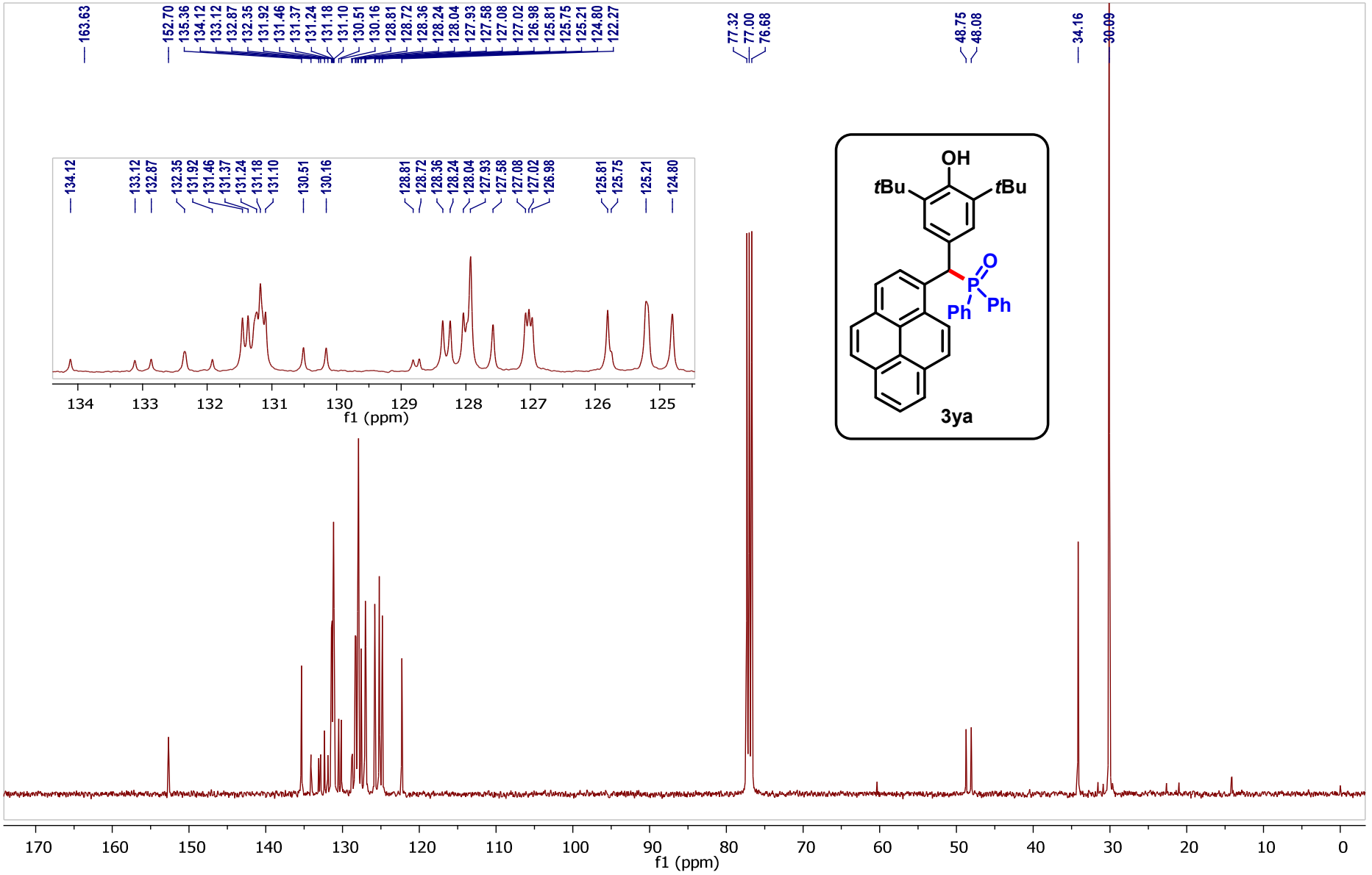
1.0
0.5
0.0
f1 (ppm)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130.16
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128.24
128.04
127.93
127.58
127.08
127.02
126.98
125.81
125.75
125.21
124.80
122.27
77.32
77.00
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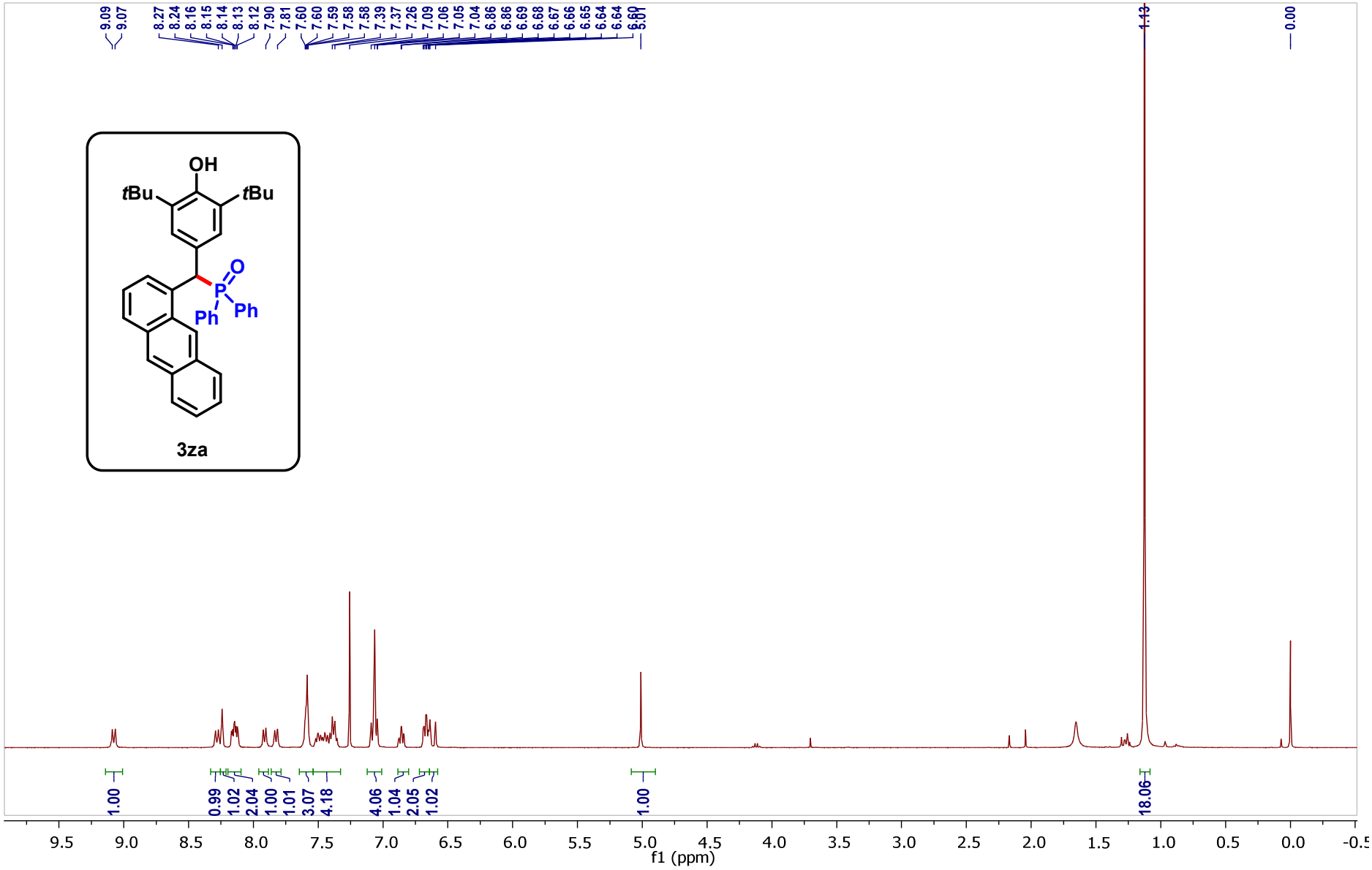
48.08
34.16
30.09
f1 (ppm)
OH
tBu
tBu
O
P
Ph
Ph
3ya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8.13
8.12
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7.81
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7.60
7.59
7.58
7.58
7.39
7.37
7.26
7.09
7.06
7.05
7.04
6.86
6.86
6.69
6.68
6.67
6.66
6.65
6.64
6.64
6.60
5.01
1.13
0.00
OH
tBu
tBu
O
P
Ph
Ph
Ph
3za
1.00
0.99
1.02
2.04
1.00
1.01
3.07
4.18
4.06
1.04
2.05
1.02
1.00
18.06
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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3.0
2.5
2.0
1.5
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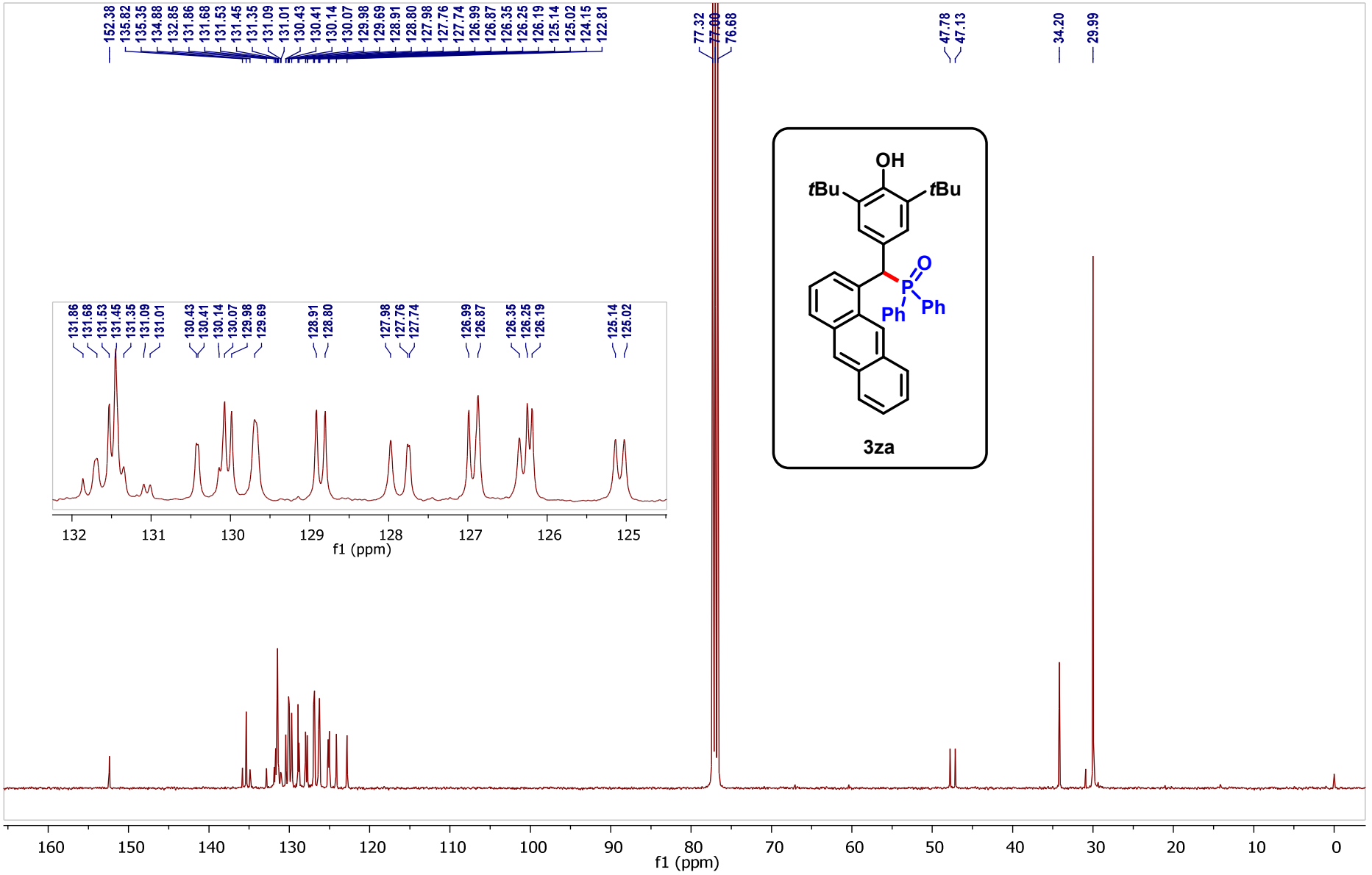
0.5
0.0
-0.5
f1 (ppm)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130.14
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129.69
128.91
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127.98
127.76
127.74
126.99
126.87
126.35
126.25
126.19
125.14
125.02
124.15
122.81
77.32
77.00
76.68
47.78
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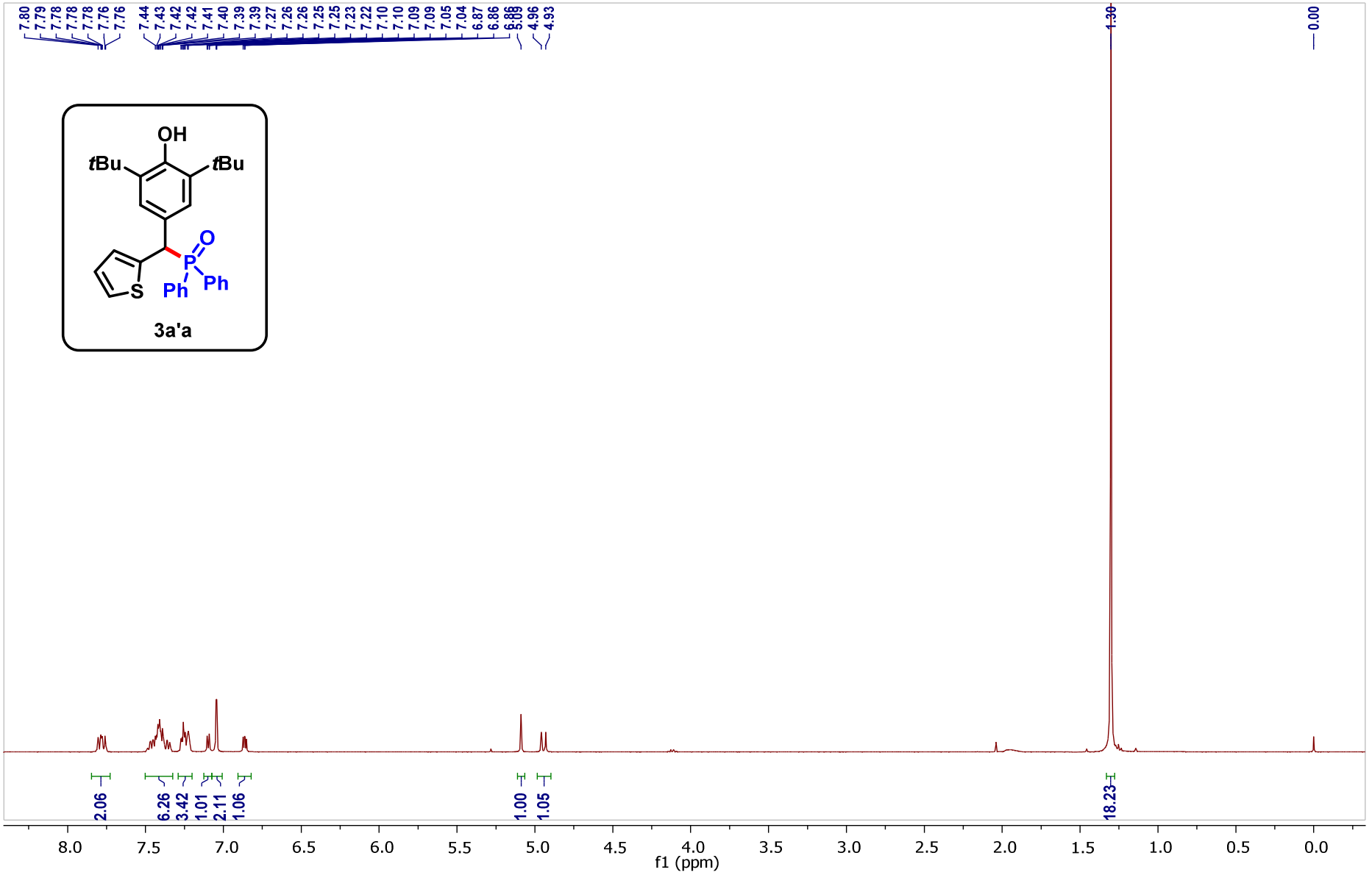
29.99
OH
tBu
tBu
O
P
Ph
Ph
3za
f1 (ppm)
f1 (ppm)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7.40
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7.39
7.27
7.26
7.26
7.25
7.25
7.23
7.22
7.10
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7.09
7.09
7.05
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4.93
1.30
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OH
tBu
tBu
O
P
Ph
Ph
S
3a'a
2.06
6.26
3.42
1.01
2.11
1.06
1.00
1.05
18.23
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
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1.0
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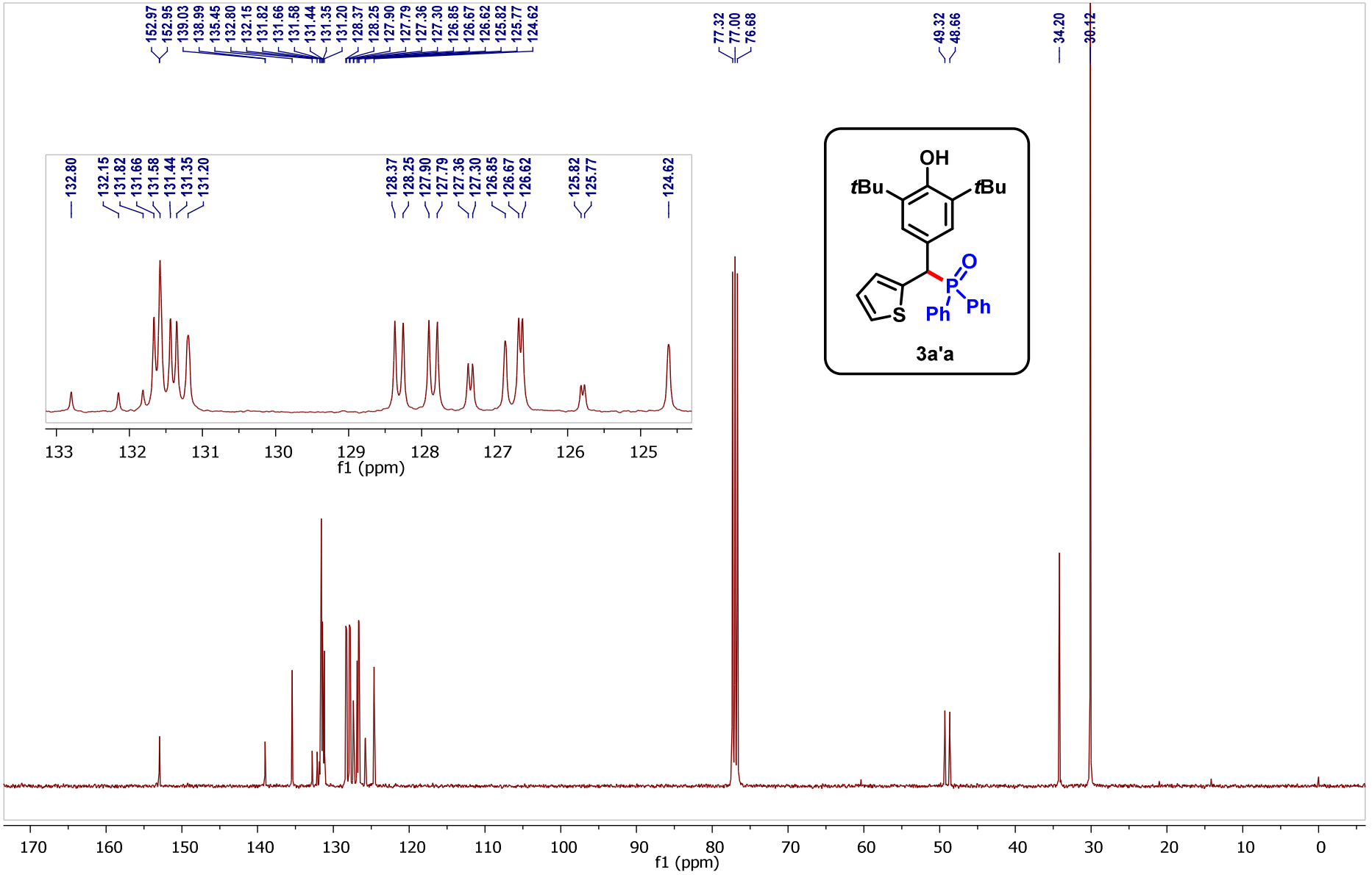
0.0
f1 (ppm)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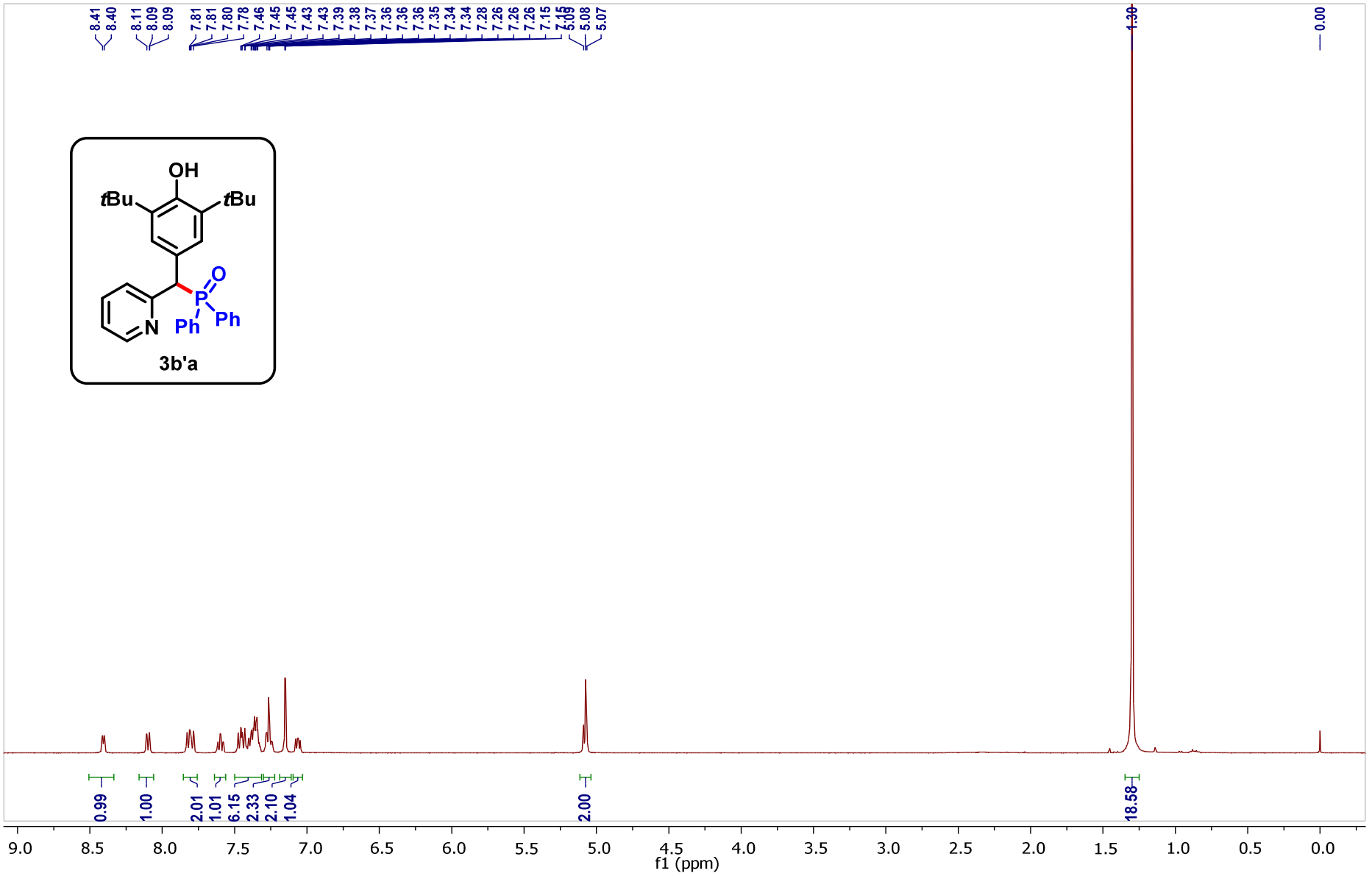
126.62
125.82
125.77
124.62
77.32
77.00
76.68
49.32
48.66
34.20
30.12
OH
tBu
tBu
O
P
Ph
Ph
S
3a'a
f1 (ppm)
f1 (ppm)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7.45
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7.43
7.39
7.38
7.37
7.36
7.36
7.36
7.35
7.34
7.34
7.28
7.26
7.26
7.26
7.15
5.09
5.08
5.07
1.30
0.00
OH
tBu
tBu
O
P
Ph
Ph
N
3b'a
0.99
1.00
2.01
1.01
6.15
2.33
2.10
1.04
2.00
18.58
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.5
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1.0
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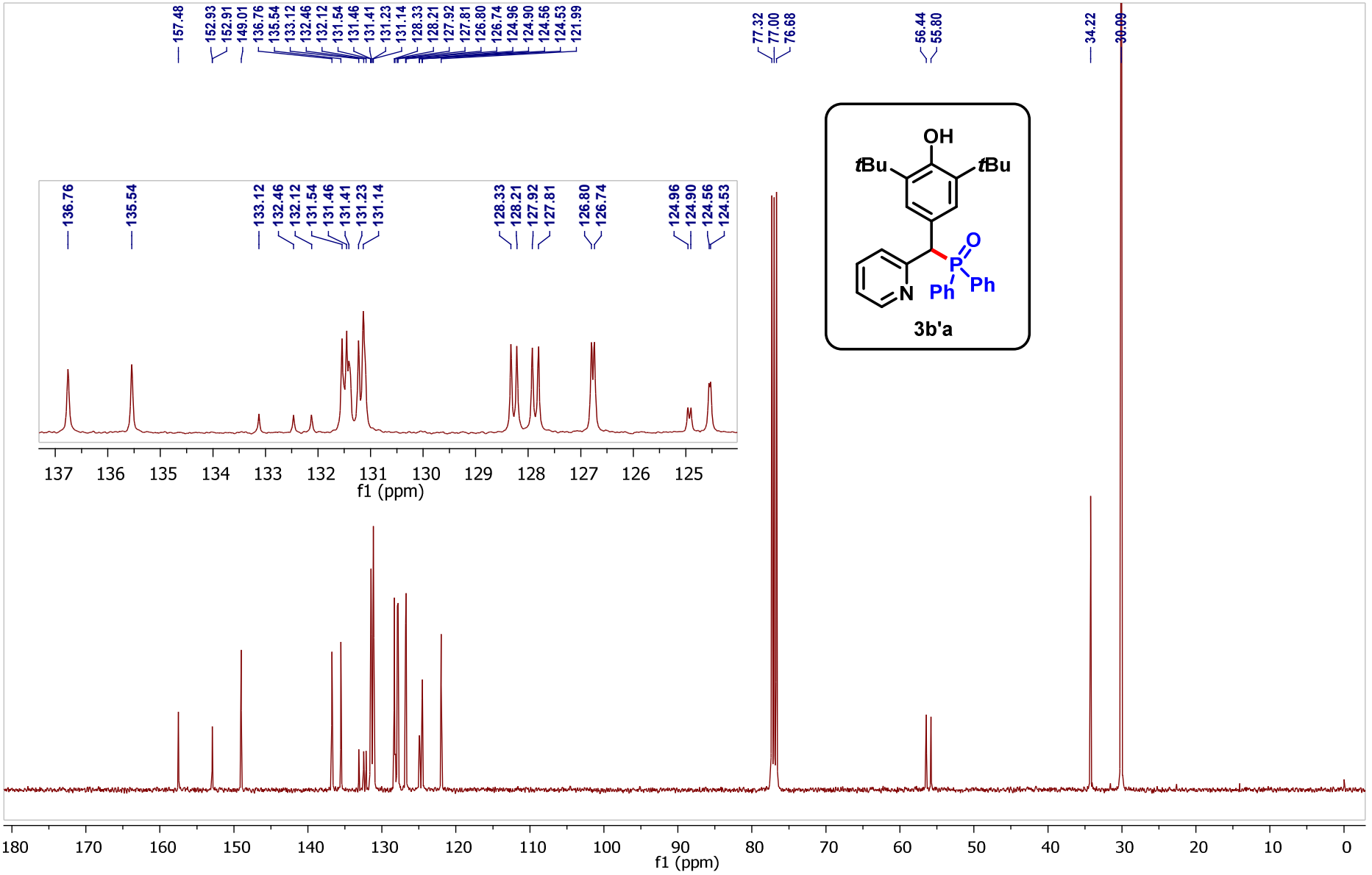
f1 (ppm)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128.33
128.21
127.92
127.81
126.80
126.74
124.96
124.90
124.56
124.53
121.99
77.32
77.00
76.68
56.44
55.80
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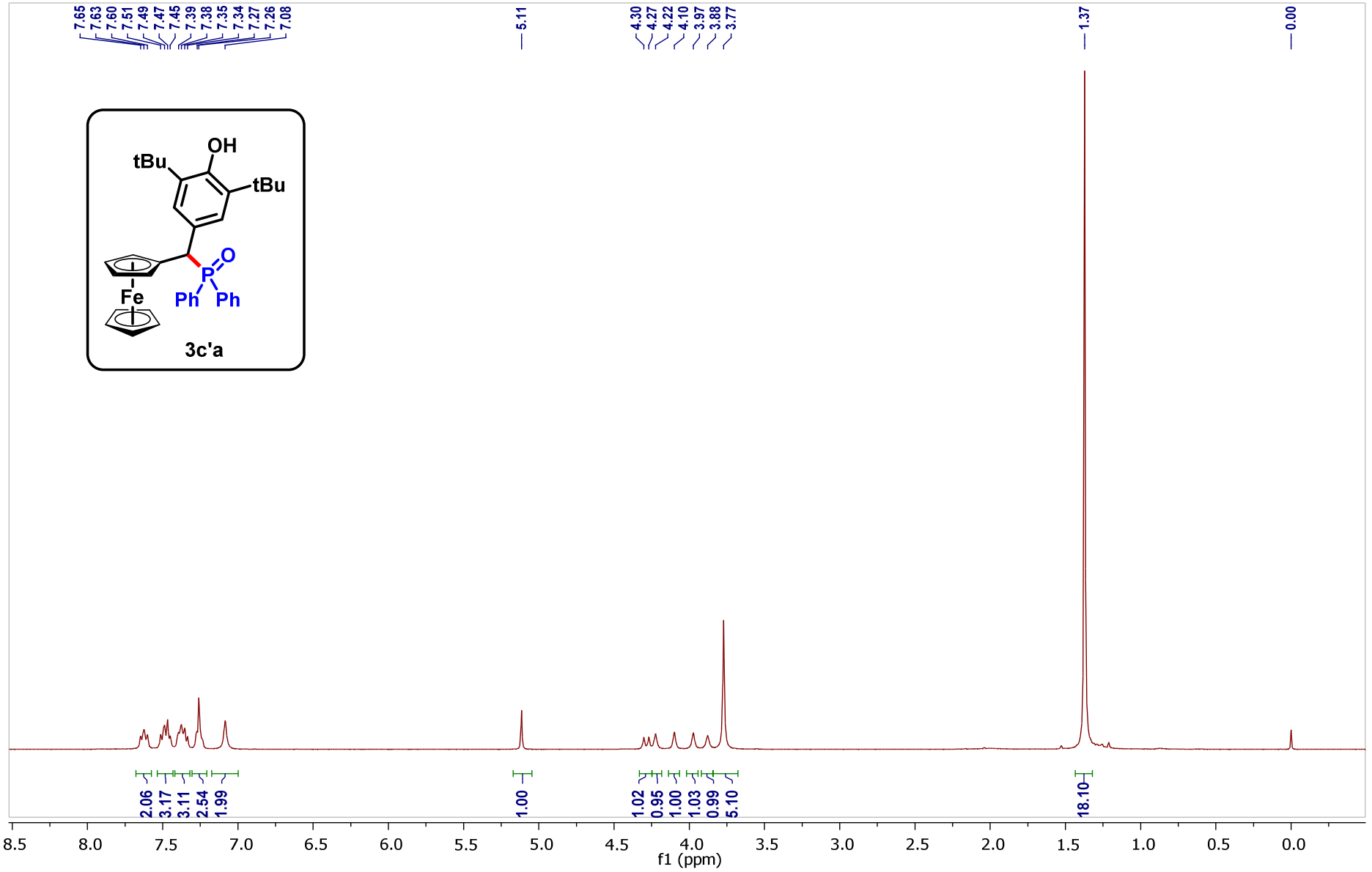
OH
tBu
tBu
O
P
Ph
Ph
N
3b'a
f1 (ppm)
f1 (ppm)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7.34
7.27
7.26
7.08
5.11
4.30
4.27
4.22
4.10
3.97
3.88
3.77
1.37
0.00
tBu
OH
tBu
O
P
Ph
Ph
Fe
3c'a
2.06
3.17
3.11
2.54
1.99
1.00
1.02
0.95
1.00
1.03
0.99
5.10
18.10
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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3.5
3.0
2.5
2.0
1.5
1.0
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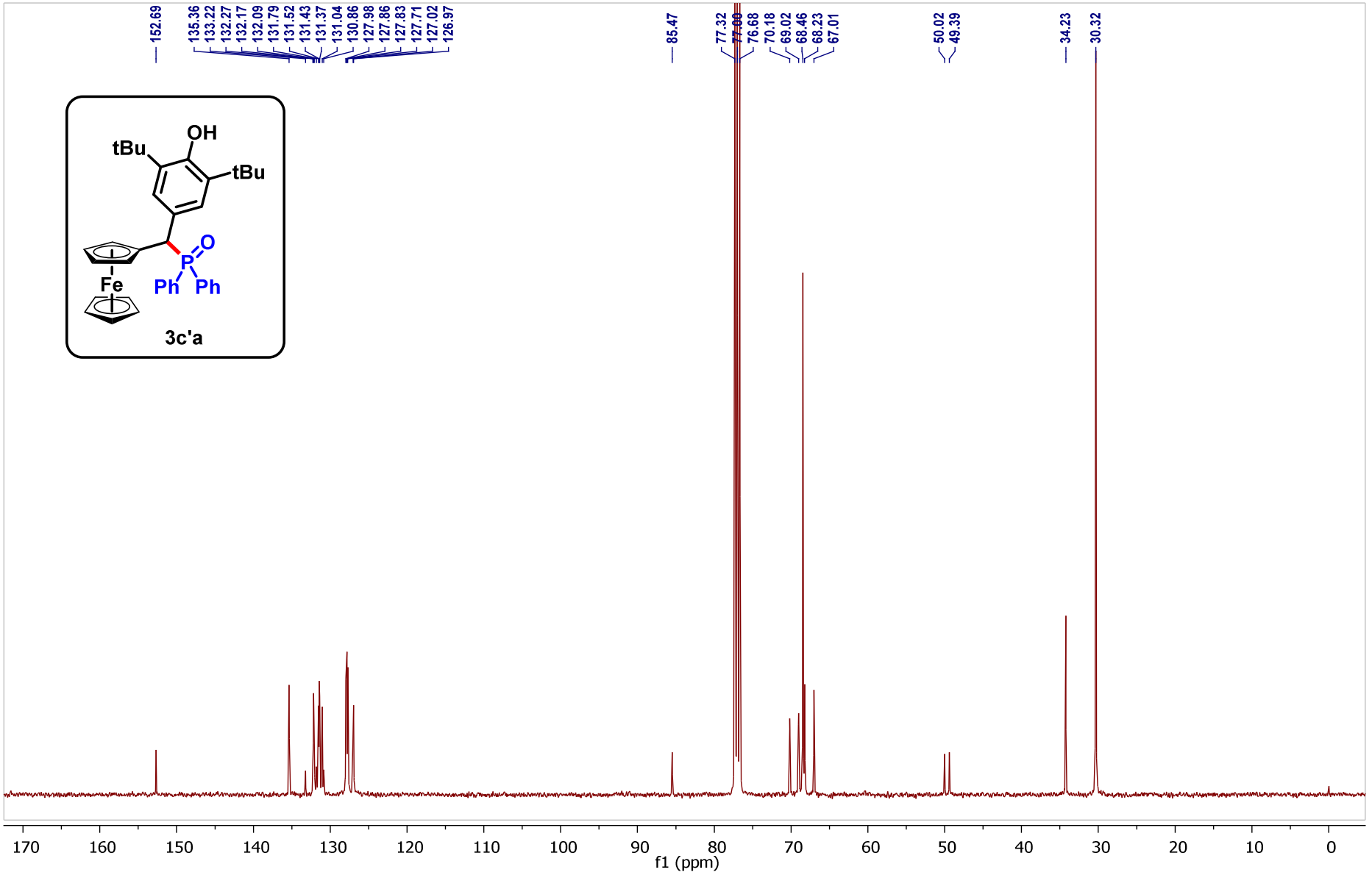
0.0
f1 (ppm)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131.37
131.04
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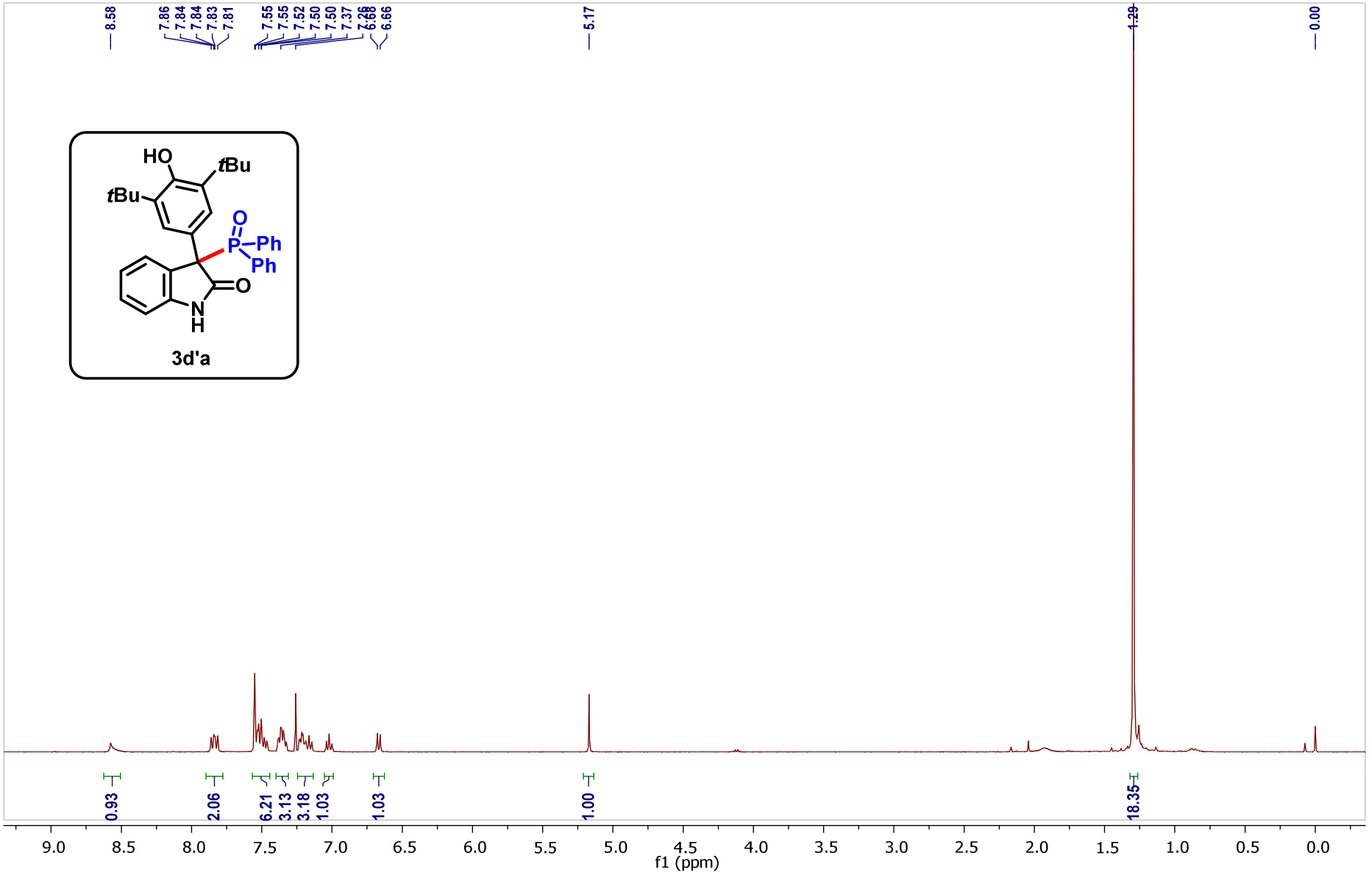
127.86
127.83
127.71
127.02
126.97
85.47
77.32
77.00
76.68
70.18
69.02
68.46
68.23
67.01
50.02
49.39
34.23
30.32
OH
tBu
tBu
O
P
Ph
Ph
Fe
3c'a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.84
7.84
7.83
7.81
7.55
7.55
7.52
7.50
7.50
7.37
7.26
6.68
6.66
5.17
1.29
0.00
HO
tBu
tBu
O
P–Ph
Ph
O
N
H
3d'a
0.93
2.06
6.21
3.13
3.18
1.03
1.03
1.00
18.35
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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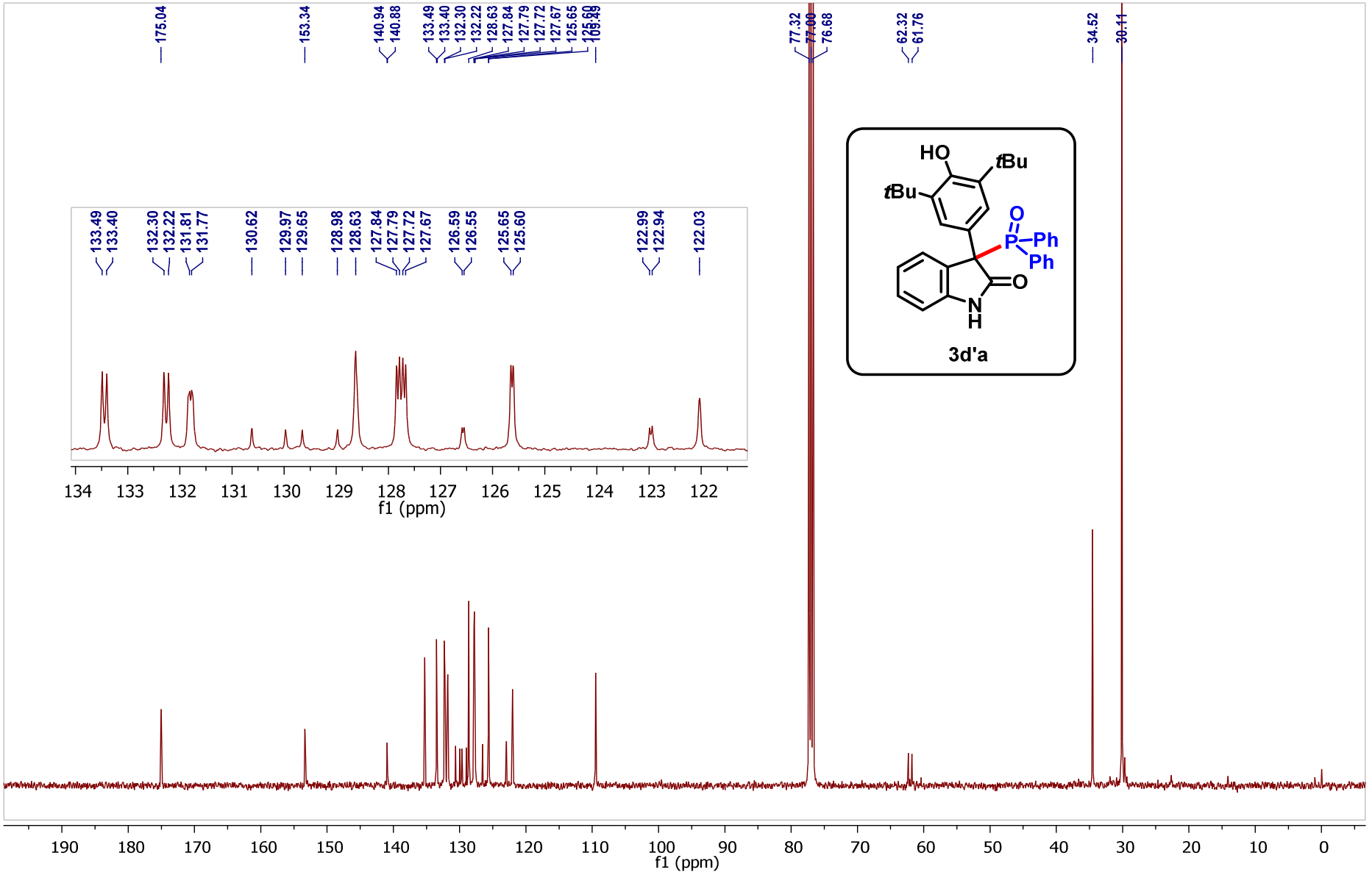
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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140.88
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133.40
132.30
132.22
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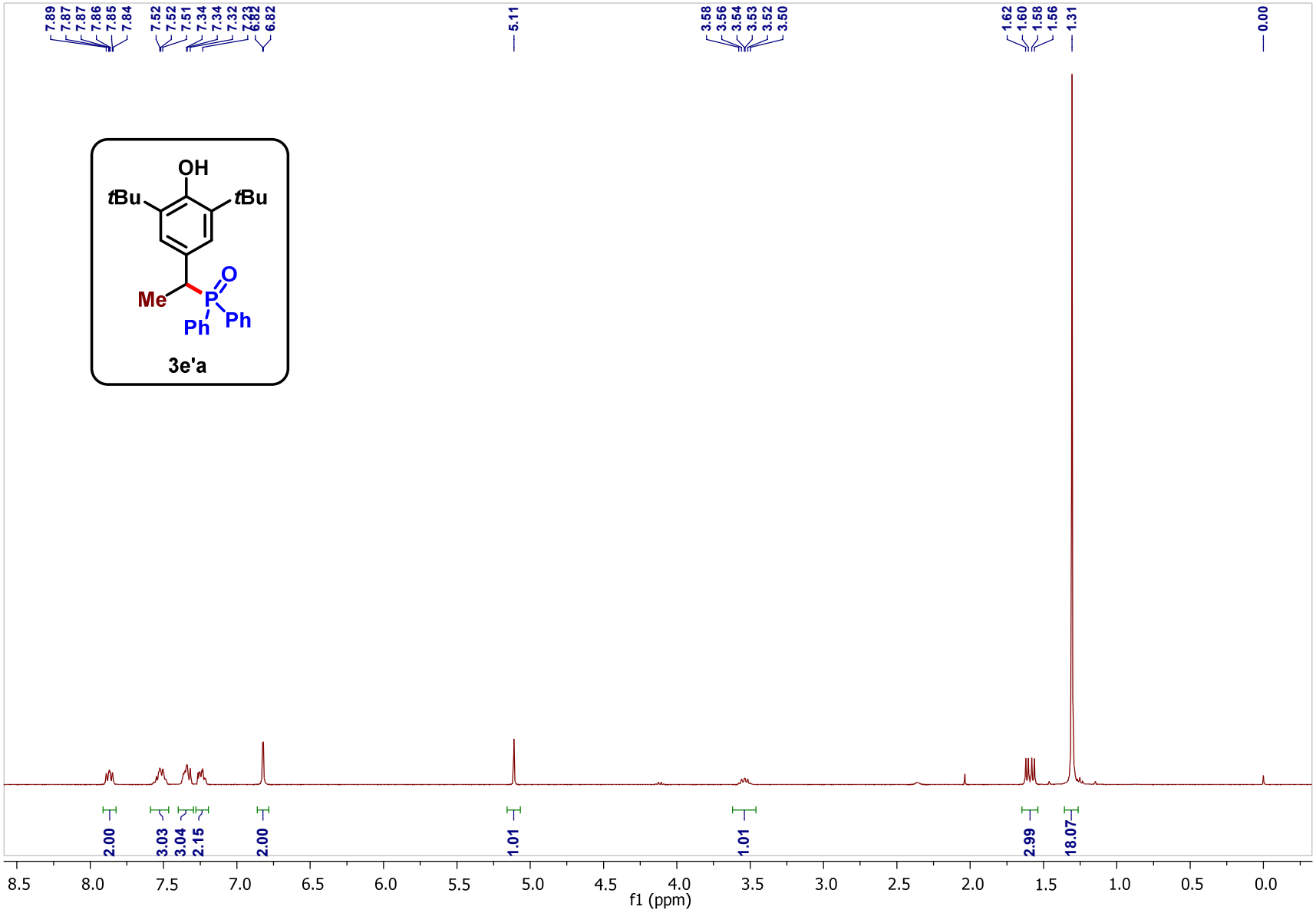
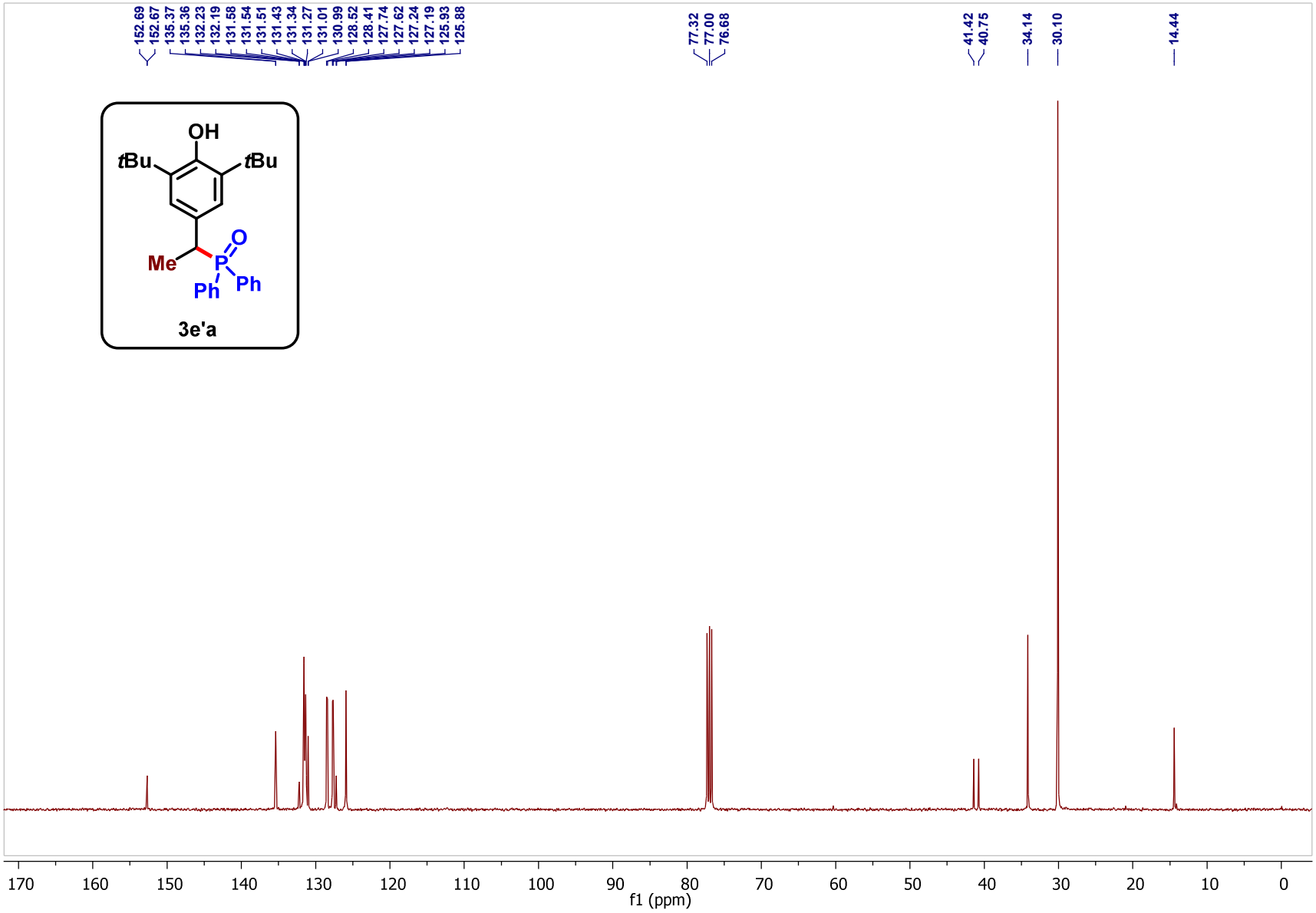
127.84
127.79
127.72
127.67
125.65
125.60
125.49
77.32
77.00
76.68
62.32
61.76
34.52
30.11
HO
tBu
tBu
O
P
Ph
Ph
O
N
H
3d'a
133.49
133.40
132.30
132.22
131.81
131.77
130.62
129.97
129.65
128.98
128.63
127.84
127.79
127.72
127.67
126.59
126.55
125.65
125.60
122.99
122.94
122.03
134 133 132 131 130 129 128 127 126 125 124 123 122
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.89
7.87
7.87
7.86
7.85
7.84
7.52
7.52
7.51
7.34
7.34
7.32
7.23
6.82
5.11
3.58
3.56
3.54
3.53
3.52
3.50
1.62
1.60
1.58
1.56
1.31
0.00
OH
tBu
tBu
Me
P
O
Ph
Ph
3e'a
2.00
3.03
3.04
2.15
2.00
1.01
1.01
2.99
18.07
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
f1 (ppm)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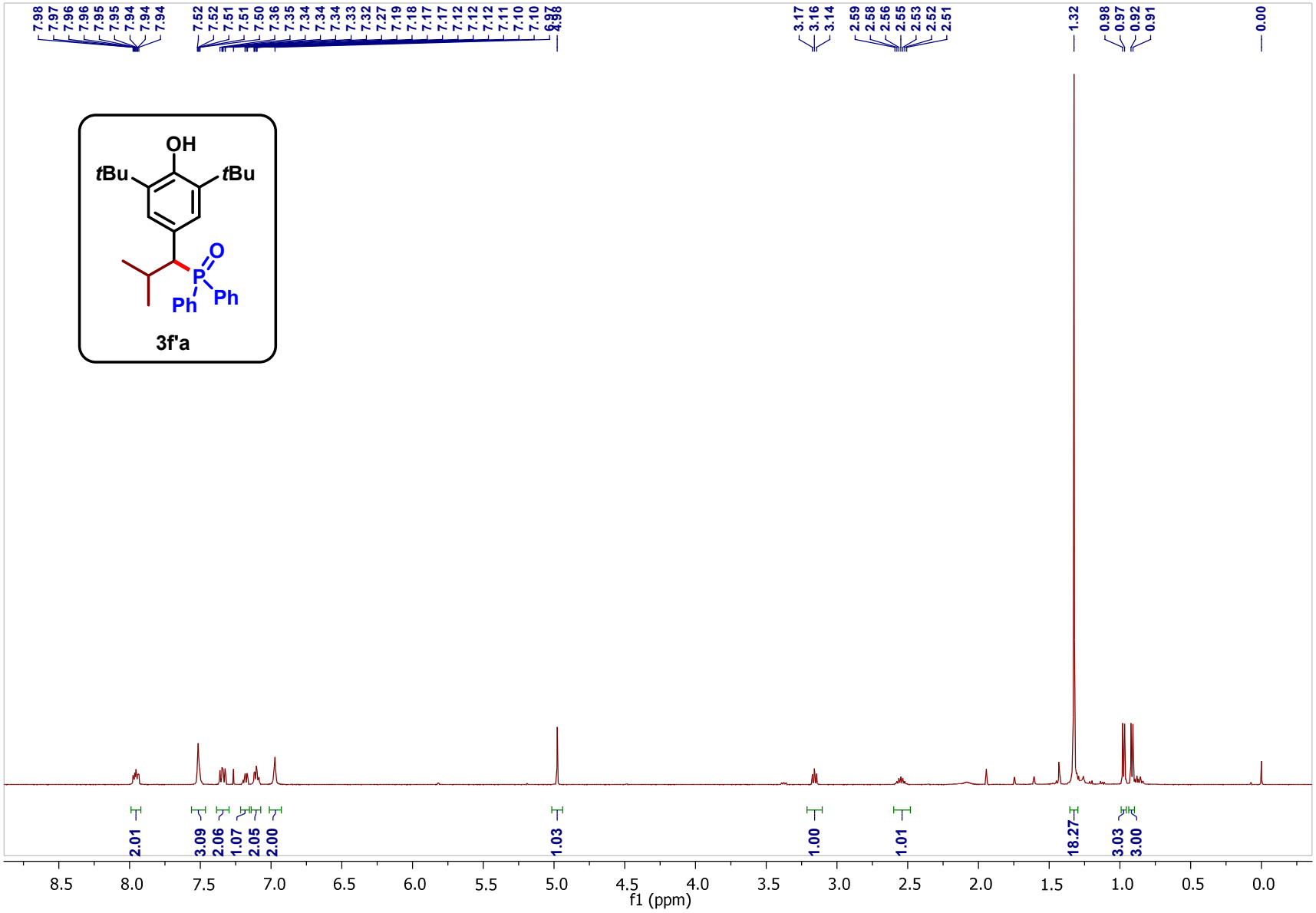
132.19
131.58
131.54
131.51
131.43
131.34
131.27
131.01
130.99
128.52
128.41
127.74
127.62
127.24
127.19
125.93
125.88
77.32
77.00
76.68
41.42
40.75
34.14
30.10
14.44
OH
tBu
tBu
Me
P
O
Ph
Ph
3e'a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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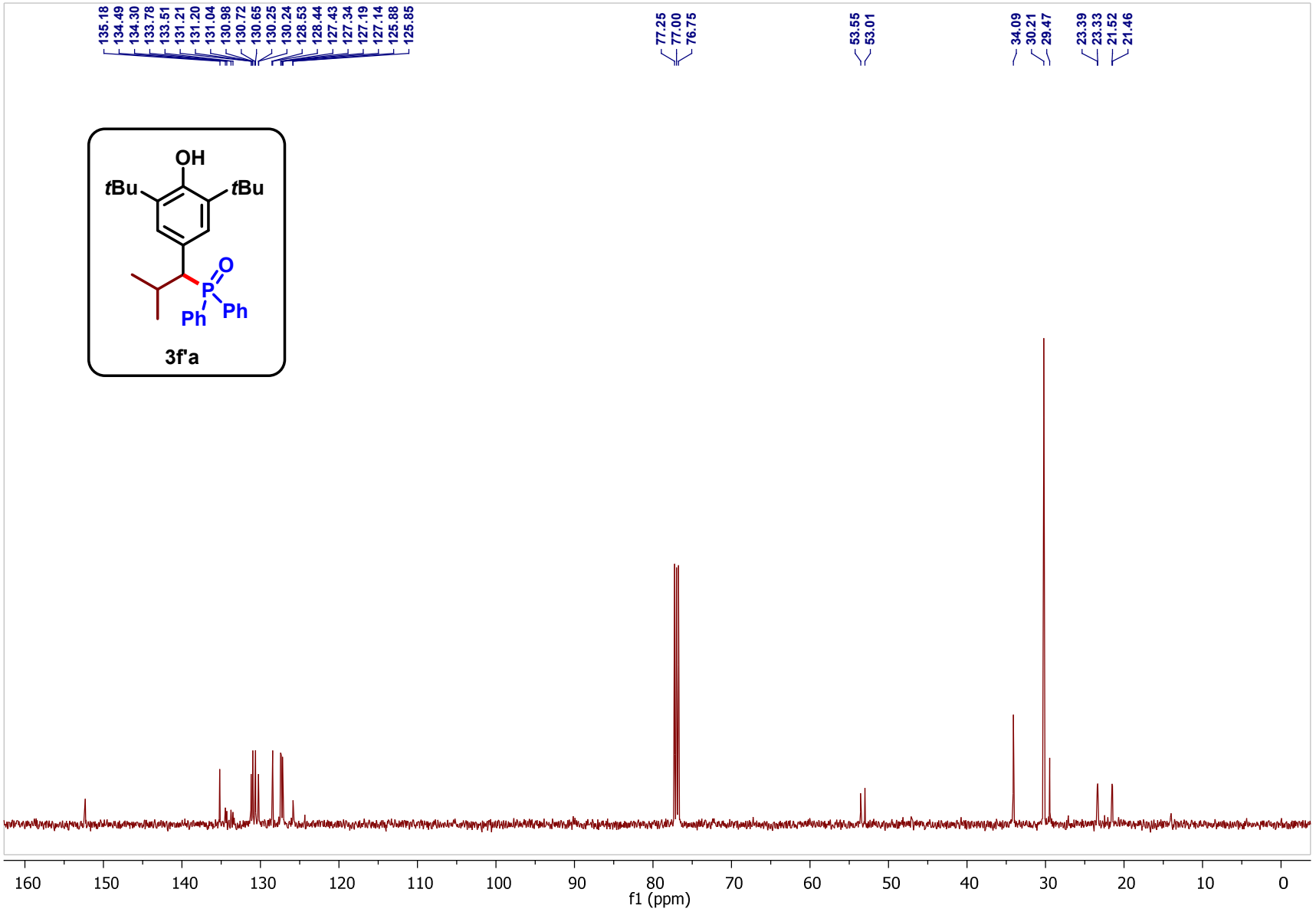
3.17 3.16 3.14
2.59 2.58 2.56 2.55 2.53 2.52 2.51
1.32
0.98 0.97 0.92 0.91
0.00
OH
tBu
tBu
O
P
Ph
Ph
3f'a
2.01 3.09 2.06 1.07 2.05 2.00 1.03 1.00 1.01 18.27 3.03 3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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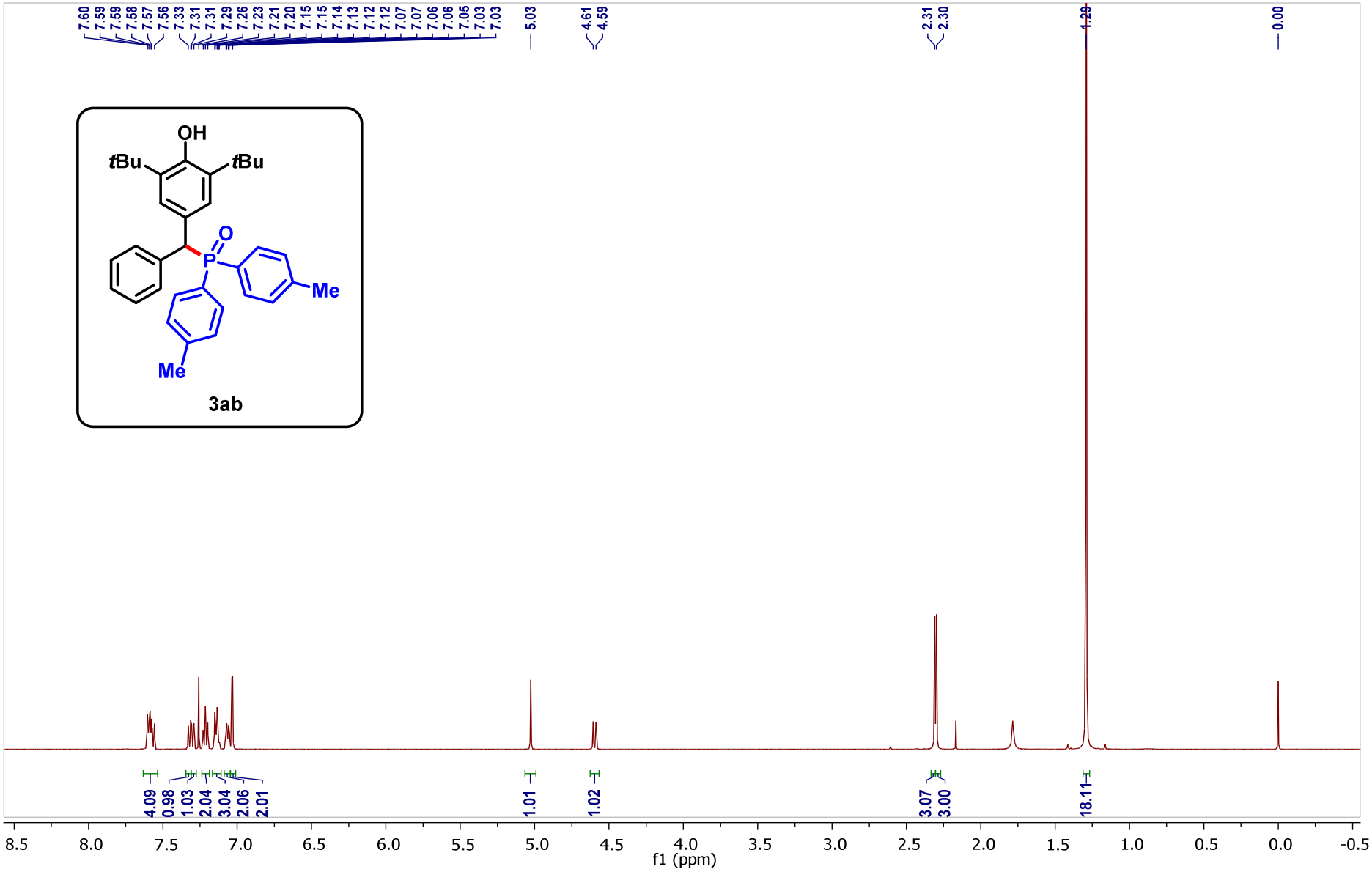
130.24
128.53
128.44
127.43
127.34
127.19
127.14
125.88
125.85
77.25
77.00
76.75
53.55
53.01
34.09
30.21
29.47
23.39
23.33
21.52
21.46
OH
tBu
tBu
O
P
Ph
Ph
3f'a
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.58
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7.29
7.26
7.23
7.21
7.20
7.15
7.15
7.14
7.13
7.12
7.12
7.07
7.07
7.06
7.06
7.05
7.03
7.03
5.03
4.61
4.59
2.31
2.30
1.29
0.00
OH
tBu
tBu
O
P
Me
Me
3ab
4.09
0.98
1.03
2.04
3.04
2.06
2.01
1.01
1.02
3.07
3.00
18.11
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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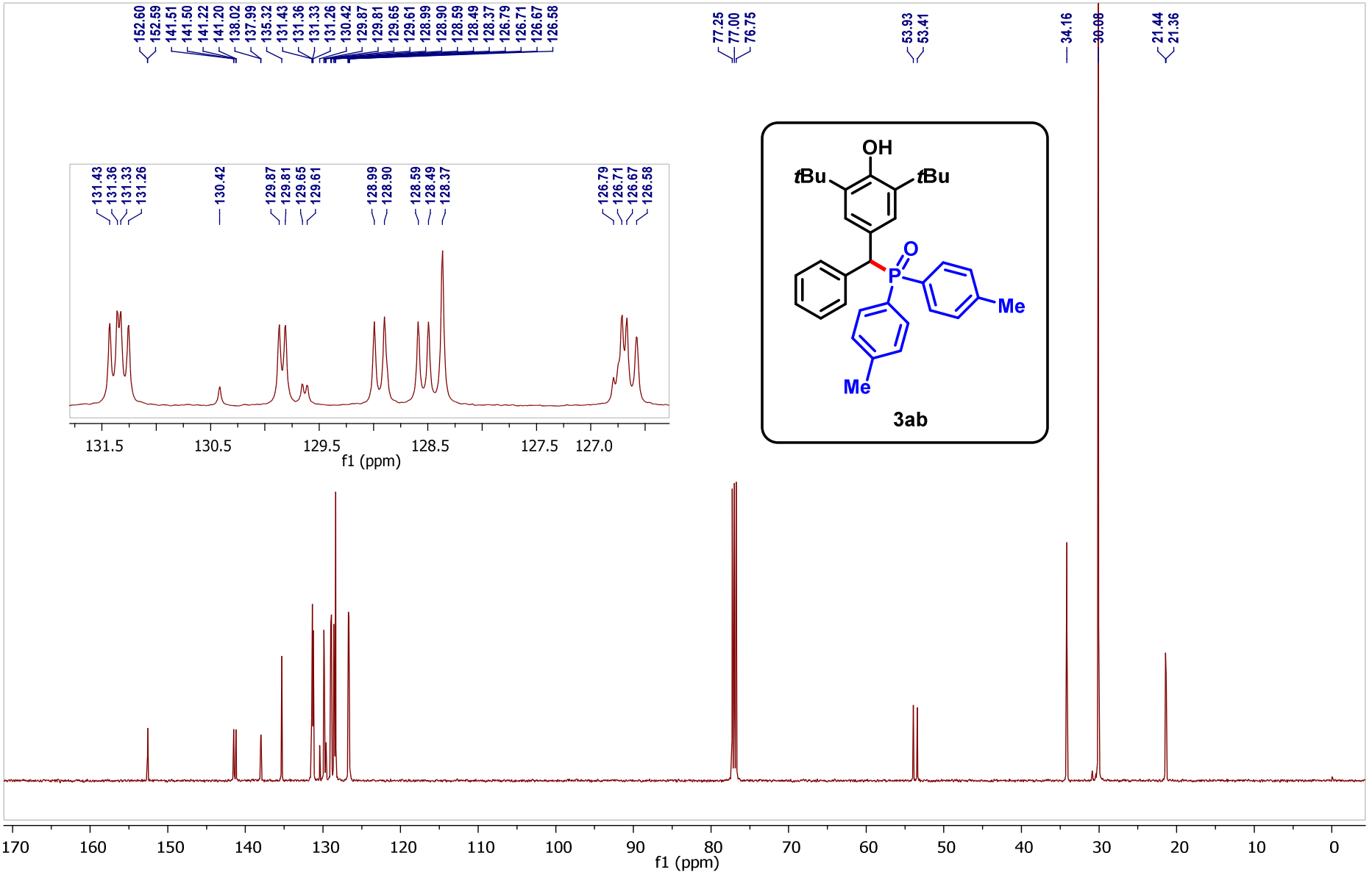
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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141.20
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137.99
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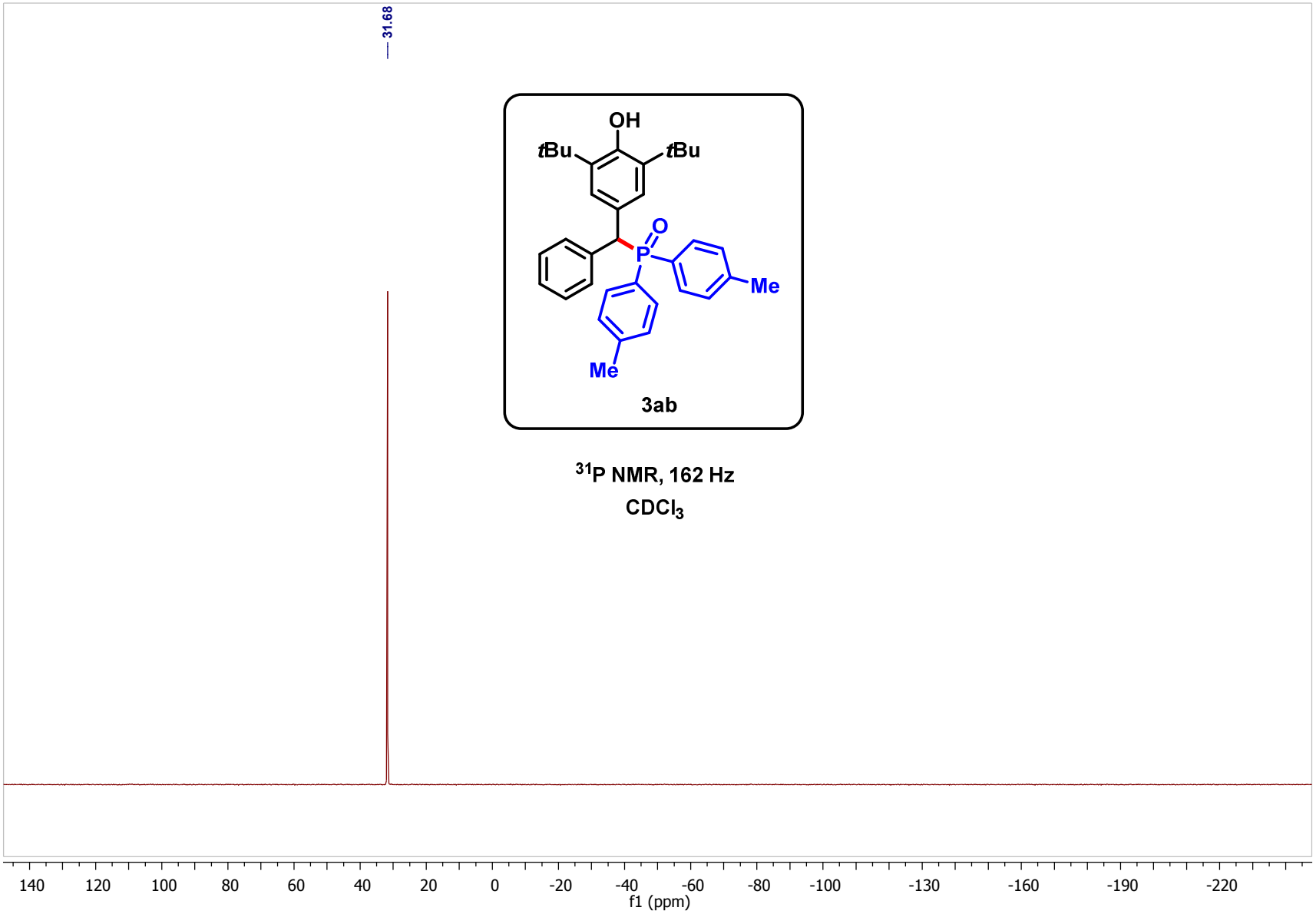
131.43
131.36
131.33
131.26
130.42
129.87
129.81
129.65
129.61
128.99
128.90
128.59
128.49
128.37
126.79
126.71
126.67
126.58
77.25
77.00
76.75
53.93
53.41
34.16
30.08
21.44
21.36
131.5
130.5
129.5
128.5
127.5
127.0
f1 (ppm)
OH
tBu
tBu
O
P
Me
Me
3ab
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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tBu
O
P
Me
Me
3ab
31P NMR, 162 Hz
CDCl3
140
120
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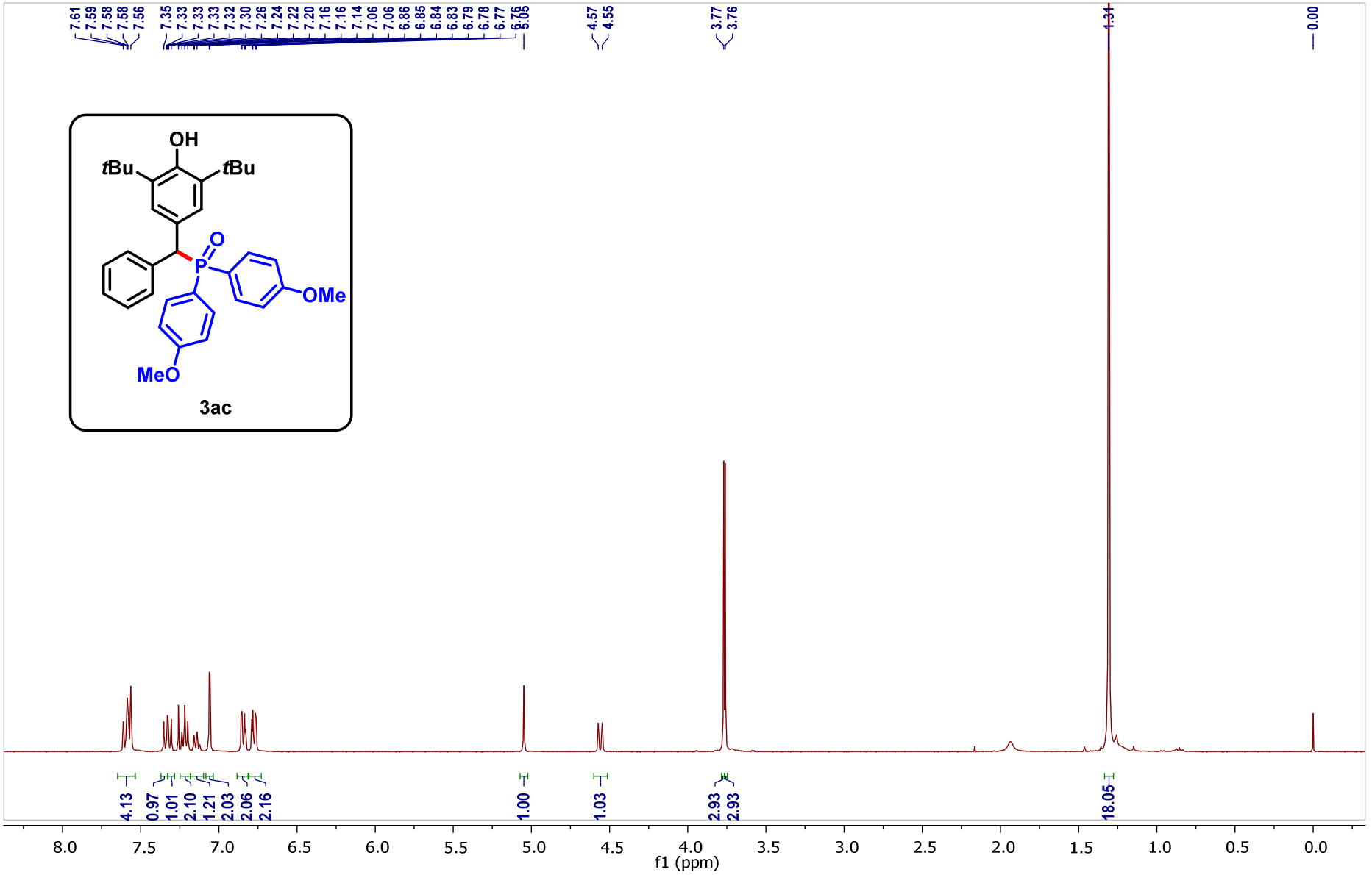
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)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7.58
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7.33
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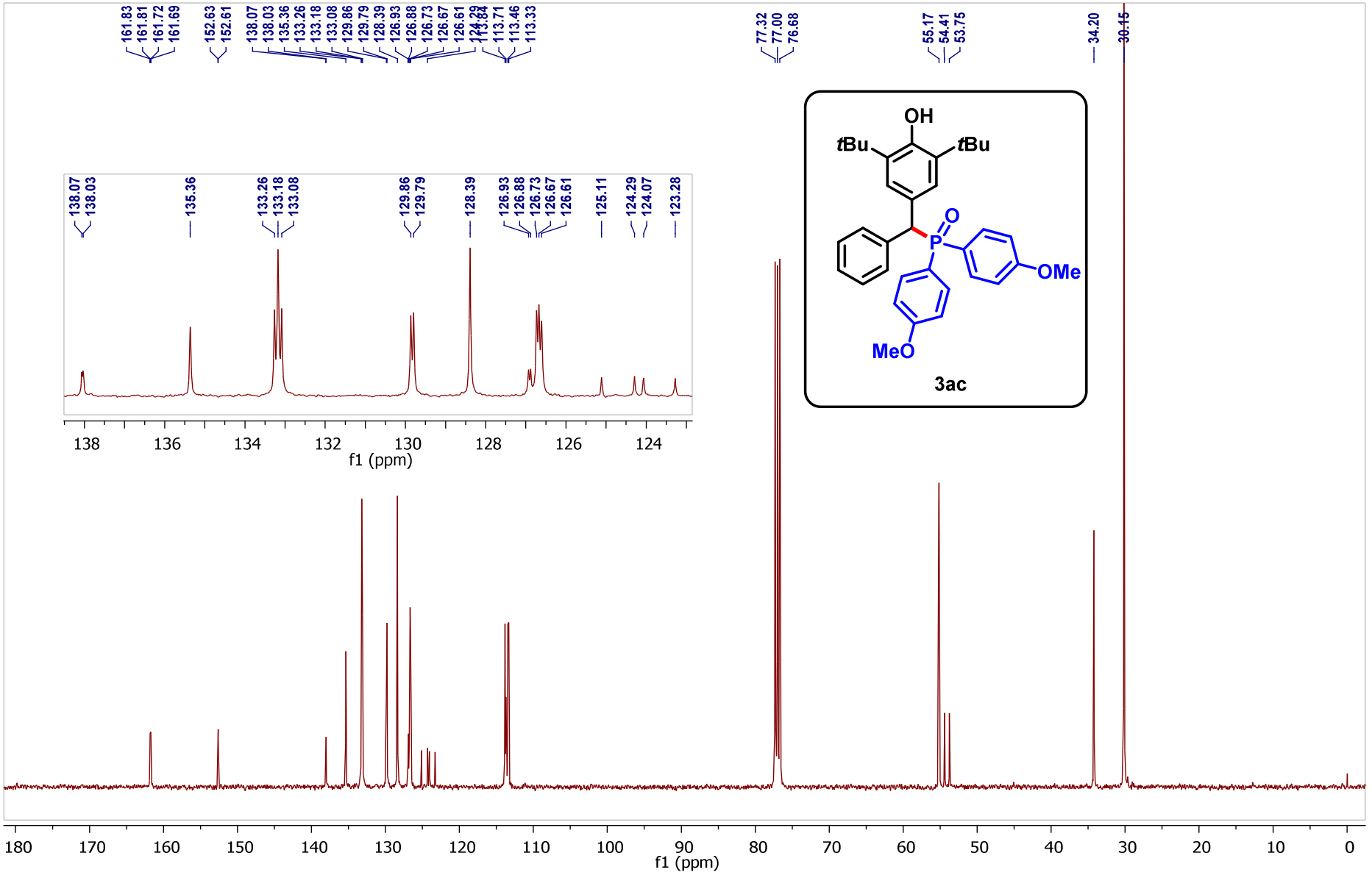
7.32
7.30
7.26
7.24
7.22
7.20
7.16
7.16
7.14
7.06
7.06
6.86
6.85
6.84
6.83
6.79
6.78
6.77
6.76
6.75
4.57
4.55
3.77
3.76
1.34
0.00
OH
tBu
tBu
O
P
OMe
MeO
3ac
4.13
0.97
1.01
2.10
1.21
2.03
2.06
2.16
1.00
1.03
2.93
2.93
18.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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152.61
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133.18
133.08
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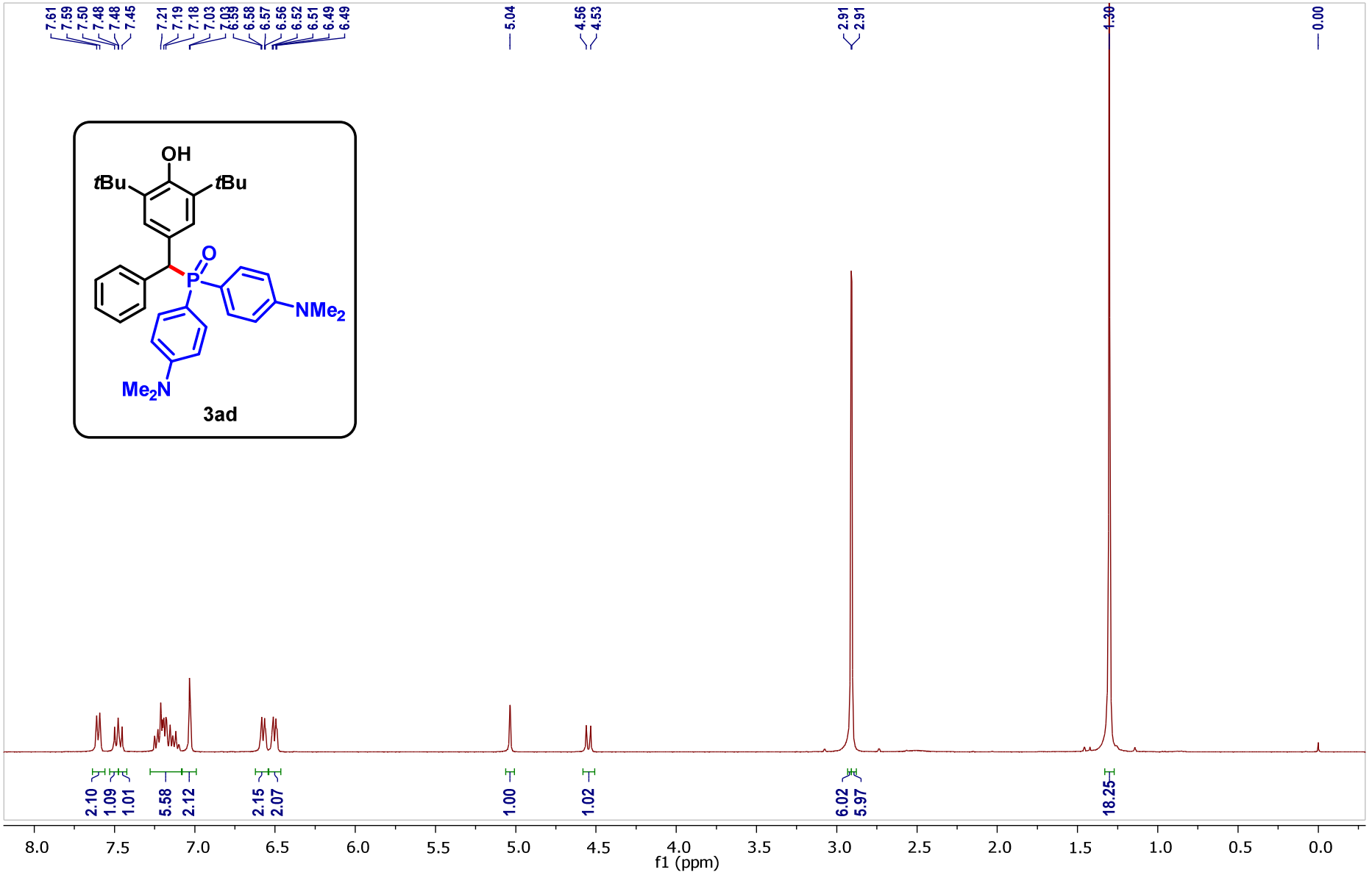
129.79
128.39
126.93
126.88
126.73
126.67
126.61
125.11
124.29
124.07
123.28
113.84
113.71
113.46
113.33
77.32
77.00
76.68
55.17
54.41
53.75
34.20
30.15
OH
tBu
tBu
O
P
OMe
MeO
3ac
f1 (ppm)
f1 (ppm)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7.18
7.03
6.59
6.58
6.57
6.56
6.52
6.51
6.49
6.49
5.04
4.56
4.53
2.91
2.91
1.30
0.00
OH
tBu
tBu
O
P
NMe2
Me2N
3ad
2.10
1.09
1.01
5.58
2.12
2.15
2.07
1.00
1.02
6.02
5.97
18.25
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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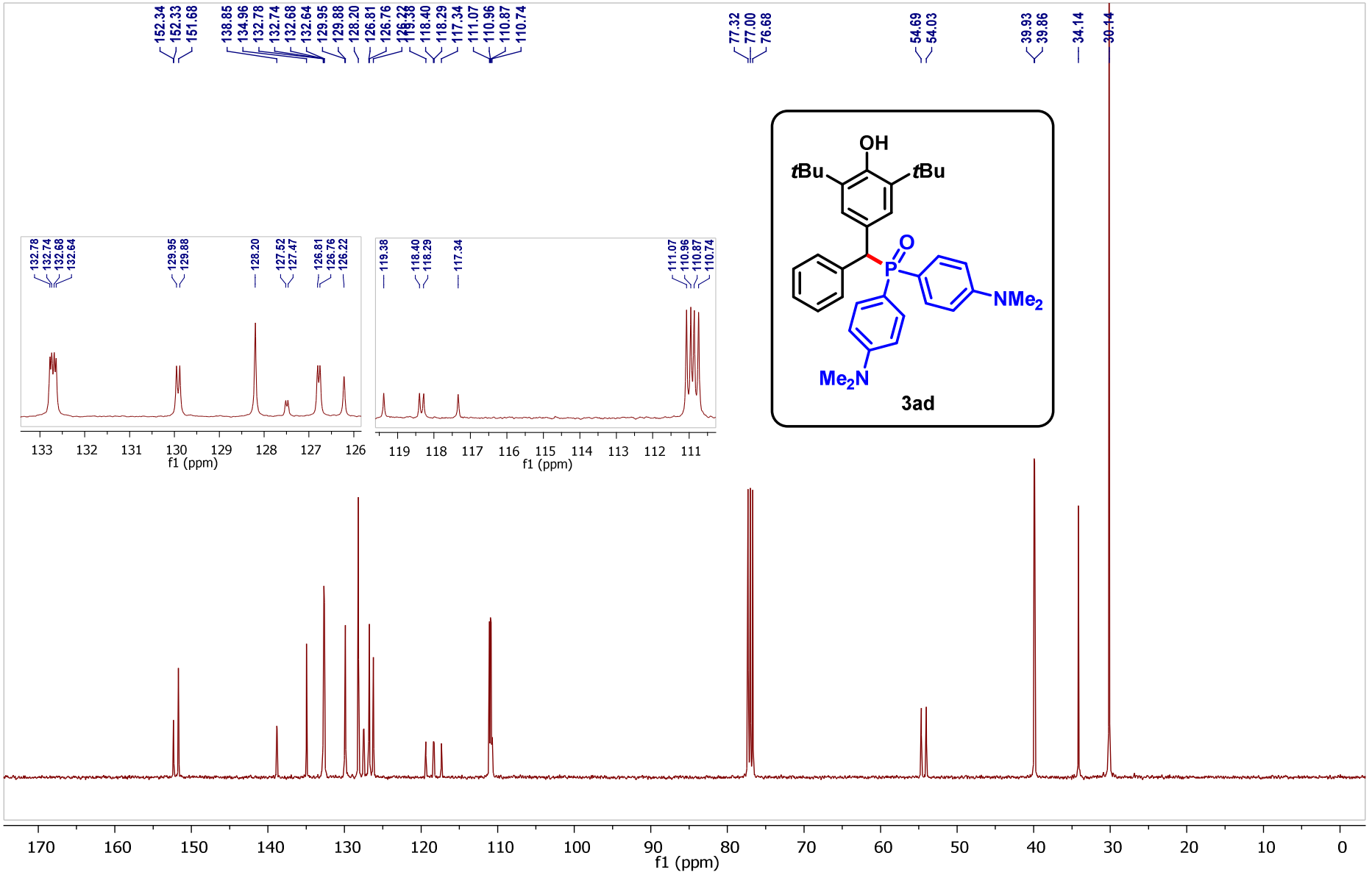
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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126.81
126.76
126.22
119.38
118.40
118.29
117.34
111.07
110.96
110.87
110.74
77.32
77.00
76.68
54.69
54.03
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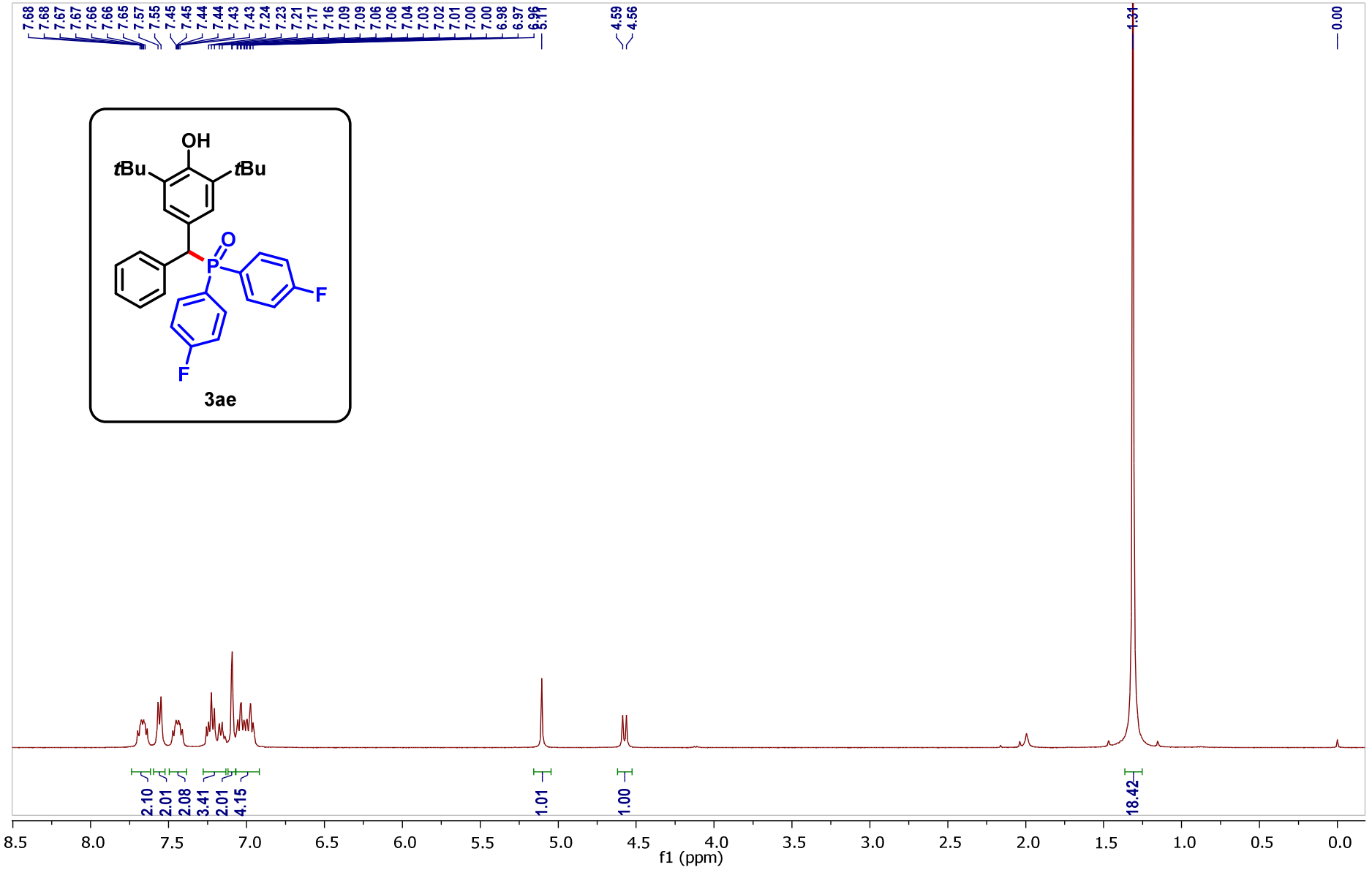
34.14
30.14
OH
tBu
tBu
O
P
NMe2
Me2N
3ad
f1 (ppm)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7.44
7.43
7.43
7.24
7.23
7.21
7.17
7.16
7.09
7.09
7.06
7.06
7.04
7.03
7.02
7.01
7.00
7.00
6.98
6.97
6.96
6.96
4.59
4.56
1.31
0.00
OH
tBu
tBu
O
P
F
F
3ae
2.10
2.01
2.08
3.41
2.01
4.15
1.01
1.00
18.42
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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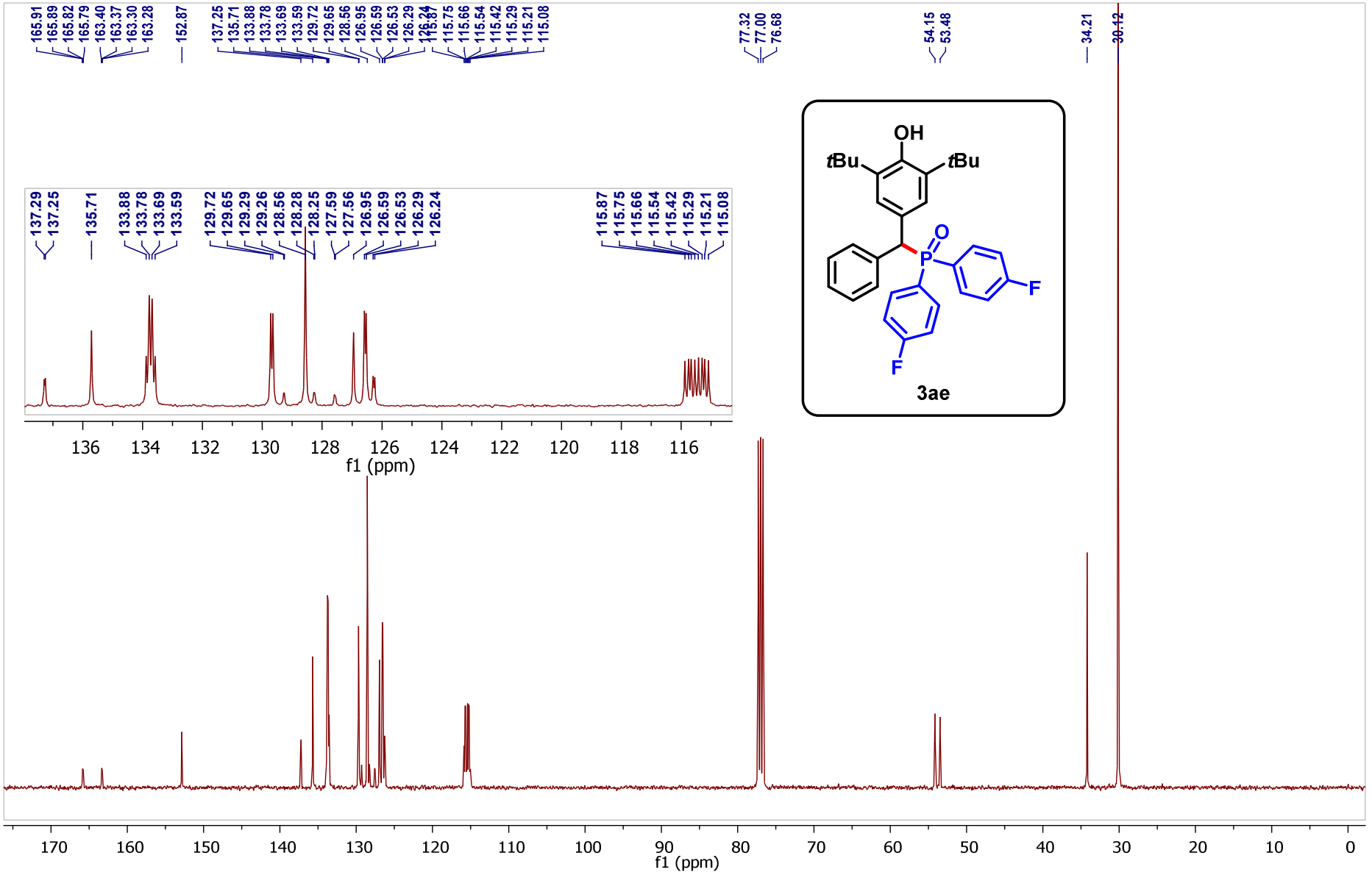
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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127.59
127.56
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126.53
126.29
126.24
115.87
115.75
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115.54
115.42
115.29
115.21
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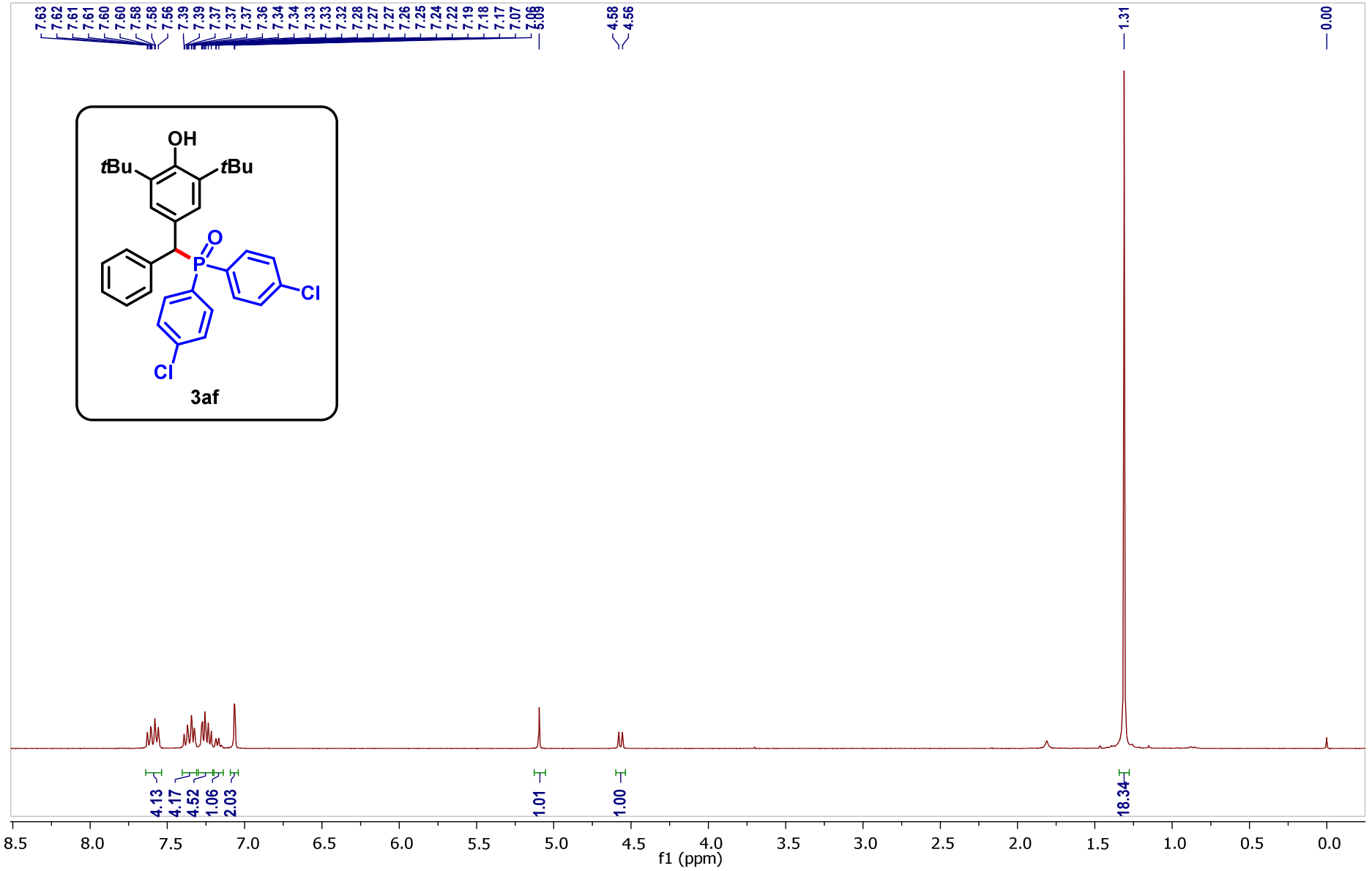
O
P
F
F
3ae
f1 (ppm)
f1 (ppm)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7.24
7.22
7.19
7.18
7.17
7.07
5.09
4.58
4.56
1.31
0.00
OH
tBu
tBu
O
P
Cl
Cl
3af
4.13
4.17
4.52
1.06
2.03
1.01
1.00
18.34
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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2.5
2.0
1.5
1.0
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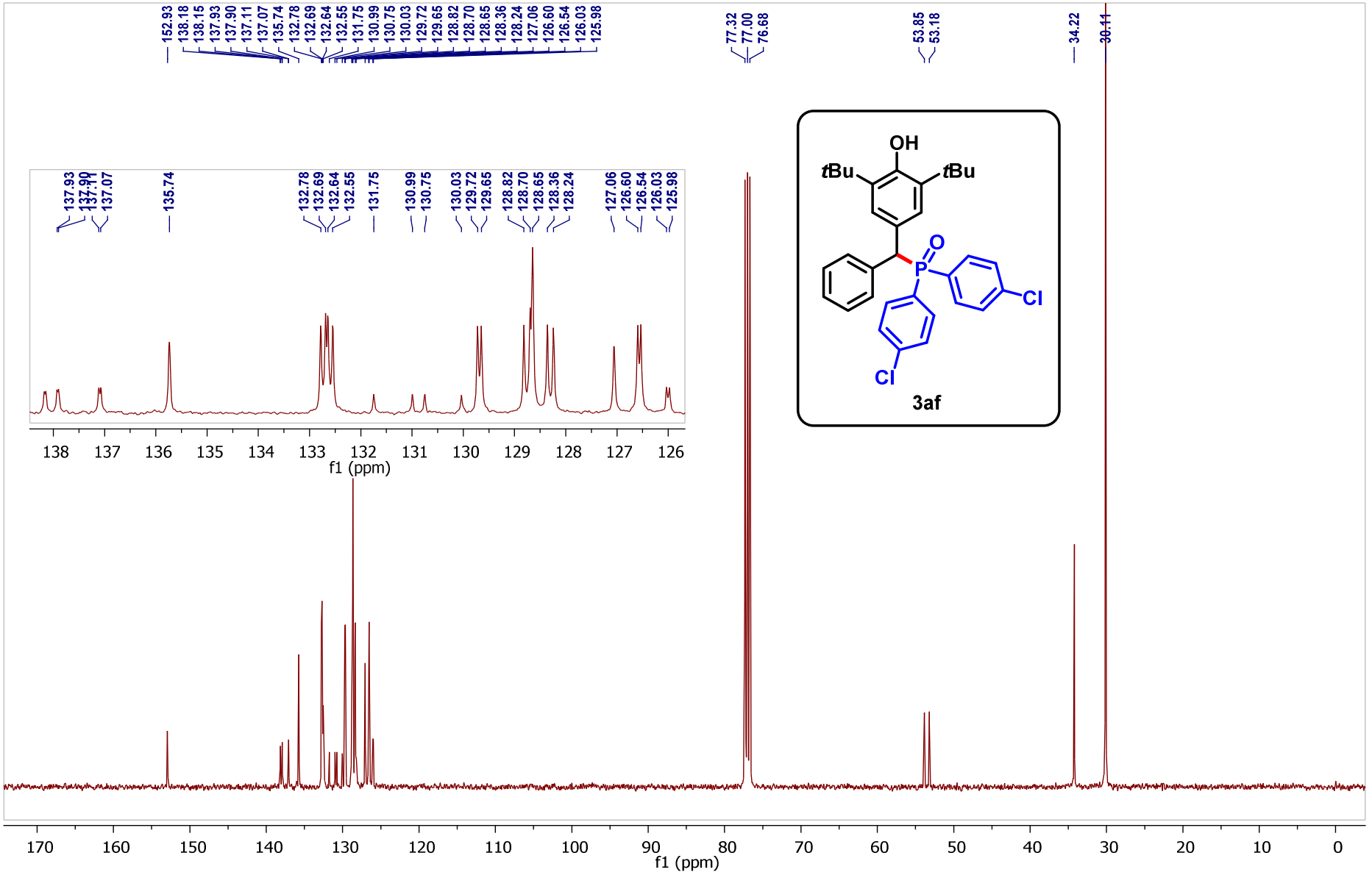
f1 (ppm)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138.15
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135.74
132.78
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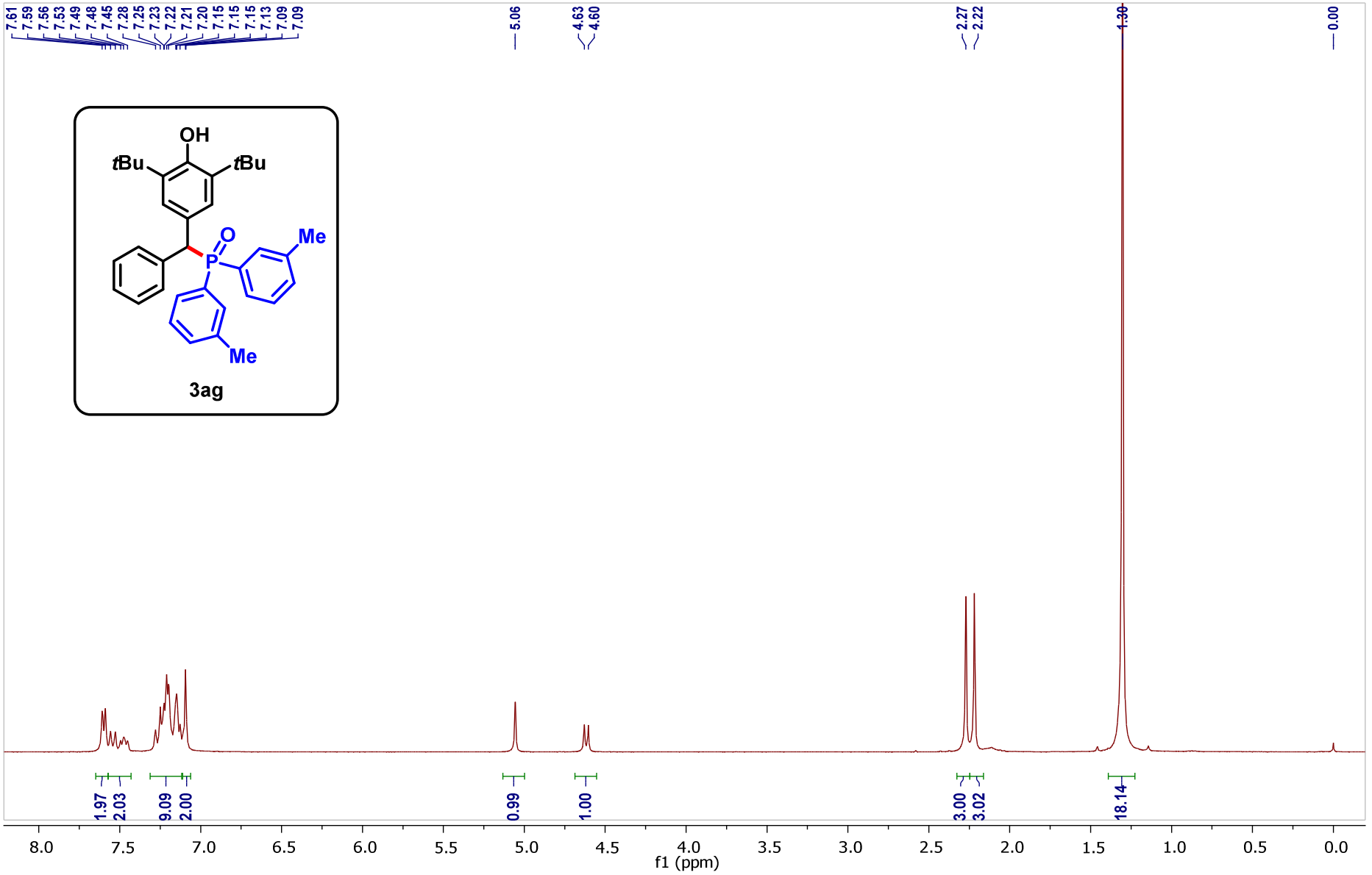
132.64
132.55
131.75
130.99
130.75
130.03
129.72
129.65
128.82
128.70
128.65
128.36
128.24
127.06
126.60
126.54
126.03
125.98
77.32
77.00
76.68
53.85
53.18
34.22
30.11
OH
tBu
tBu
O
P
Cl
Cl
3af
f1 (ppm)
f1 (ppm)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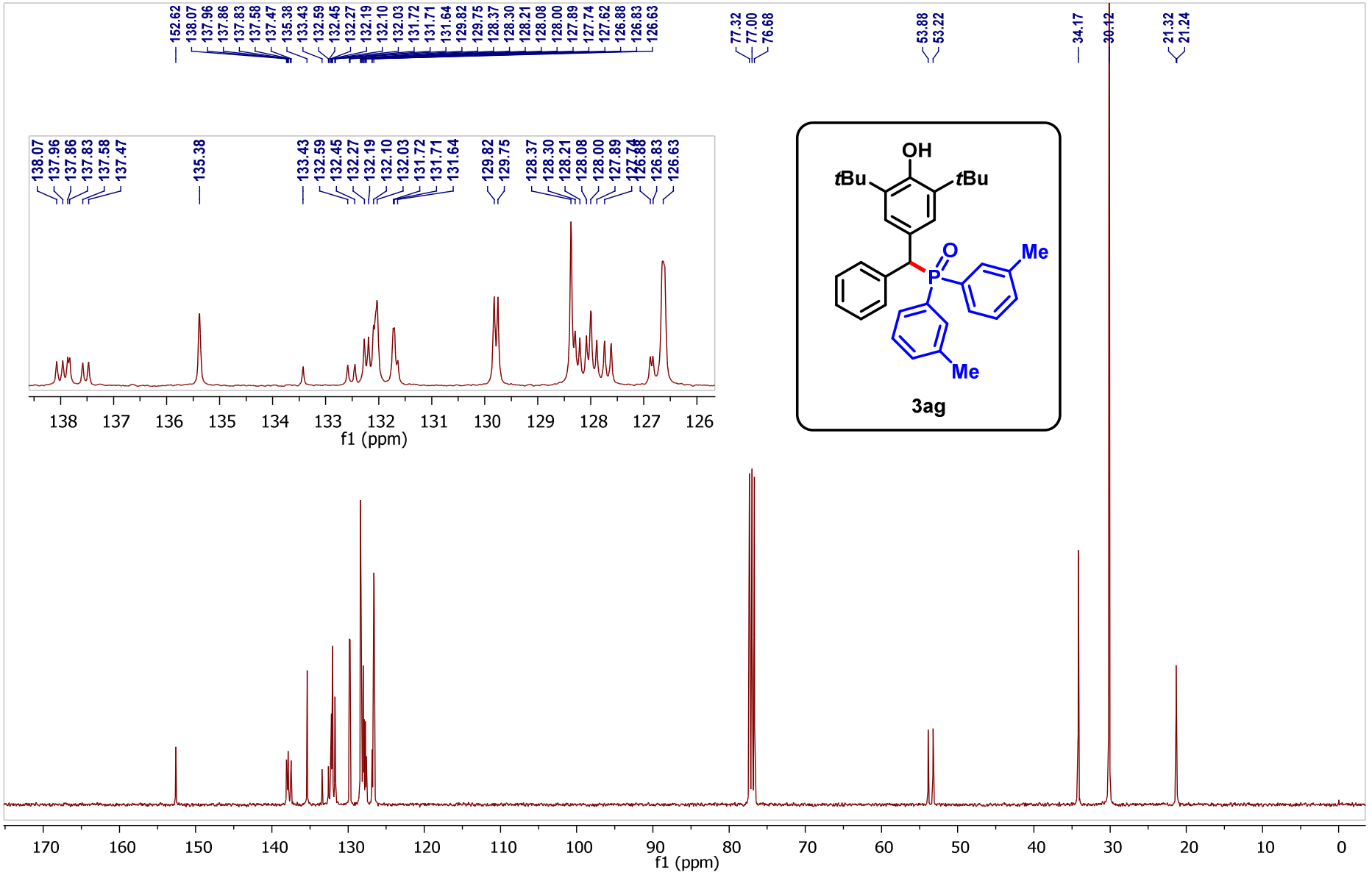
7.48
7.45
7.28
7.25
7.23
7.22
7.21
7.20
7.15
7.15
7.15
7.13
7.09
7.09
5.06
4.63
4.60
2.27
2.22
1.30
0.00
OH
tBu
tBu
O
P
Me
Me
3ag
1.97
2.03
9.09
2.00
0.99
1.00
3.00
3.02
18.14
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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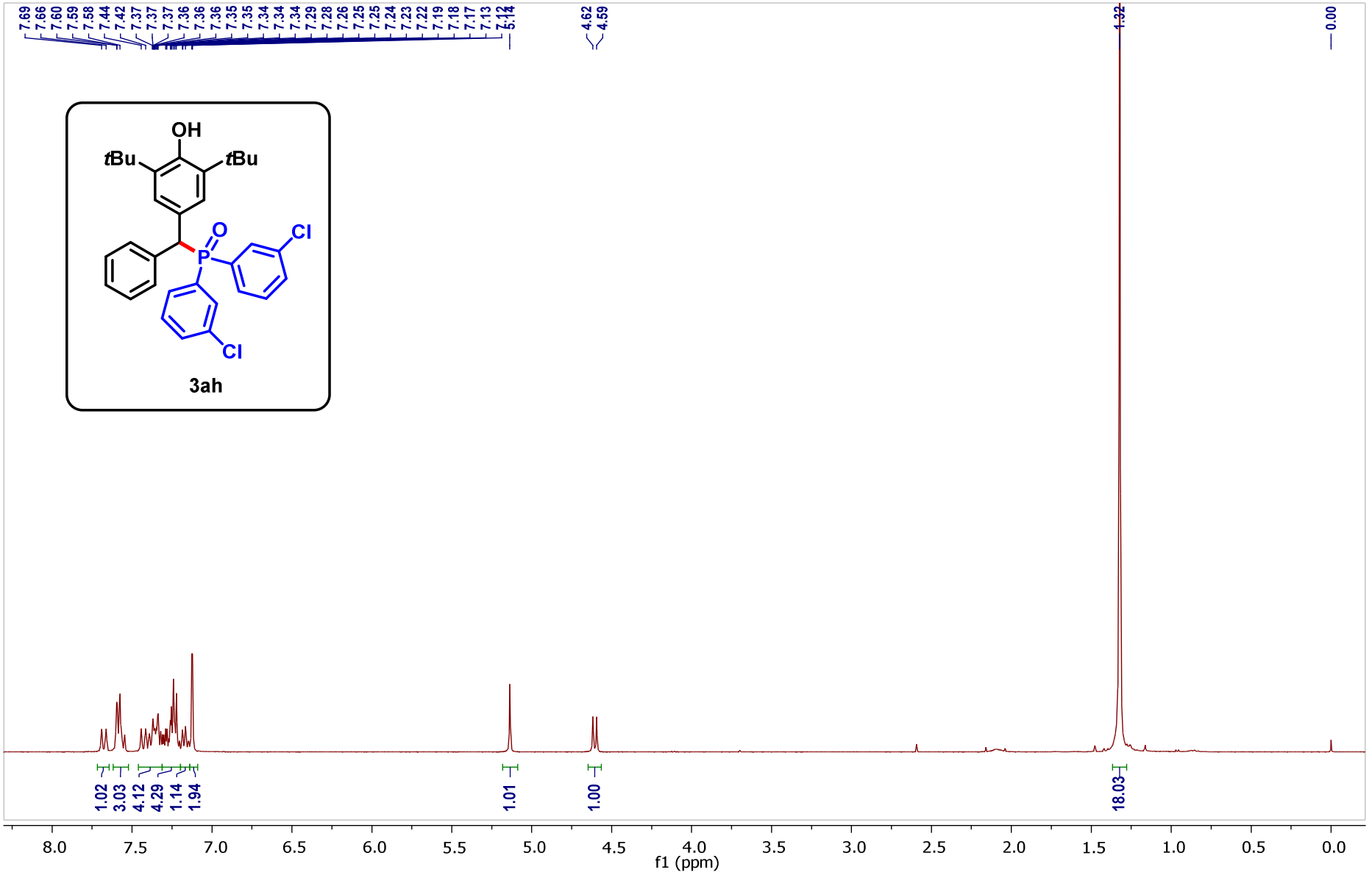
132.59
132.45
132.27
132.19
132.10
132.03
131.72
131.71
131.64
129.82
129.75
128.37
128.30
128.21
128.08
128.00
127.89
127.74
127.62
126.88
126.83
126.63
77.32
77.00
76.68
53.88
53.22
34.17
30.12
21.32
21.24
f1 (ppm)
OH
tBu
tBu
O
P
Me
Me
3ag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7.60
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7.58
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7.42
7.37
7.37
7.37
7.36
7.36
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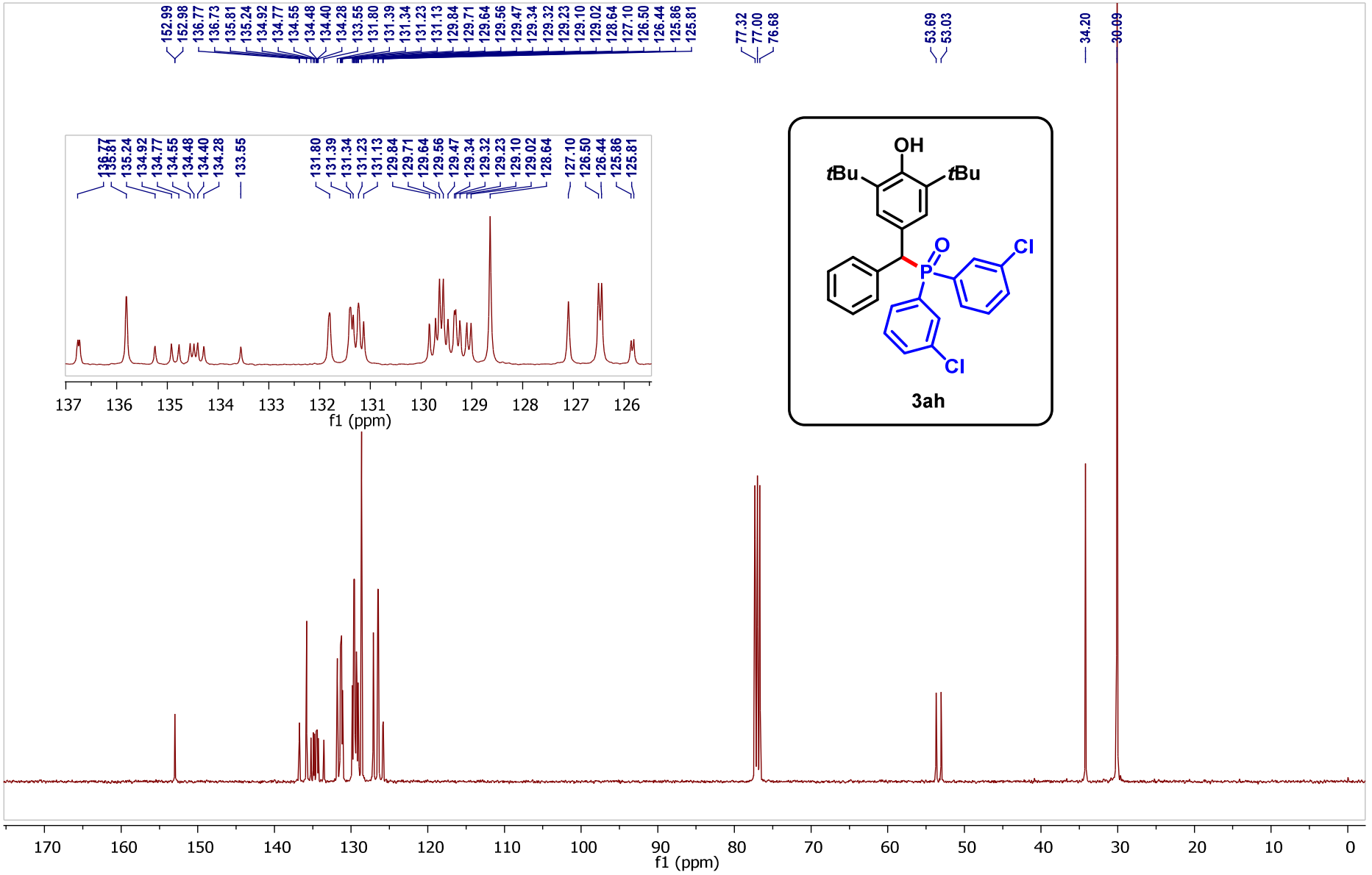
7.35
7.35
7.34
7.34
7.34
7.29
7.28
7.26
7.25
7.25
7.24
7.23
7.22
7.19
7.18
7.17
7.13
7.12
5.14
4.62
4.59
1.32
0.00
OH
tBu
tBu
O
P
Cl
Cl
3ah
1.02
3.03
4.12
4.29
1.14
1.94
1.01
1.00
18.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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135.24
134.92
134.77
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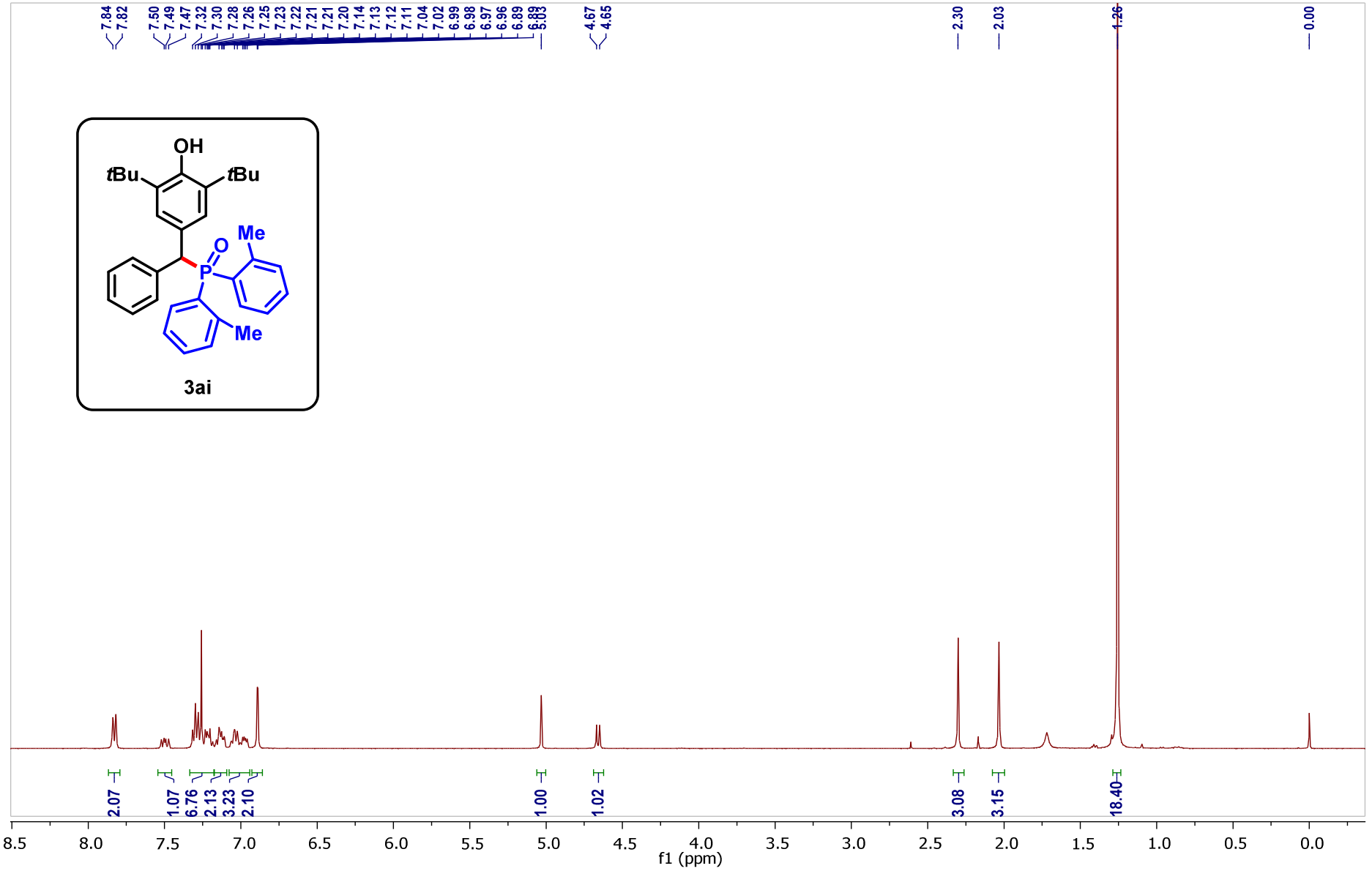
134.48
134.40
134.28
133.55
131.80
131.39
131.34
131.23
131.13
129.84
129.71
129.64
129.56
129.47
129.34
129.32
129.23
129.10
129.02
128.64
127.10
126.50
126.44
125.86
125.81
77.32
77.00
76.68
53.69
53.03
34.20
30.09
f1 (ppm)
OH
tBu
tBu
O
P
Cl
Cl
3ah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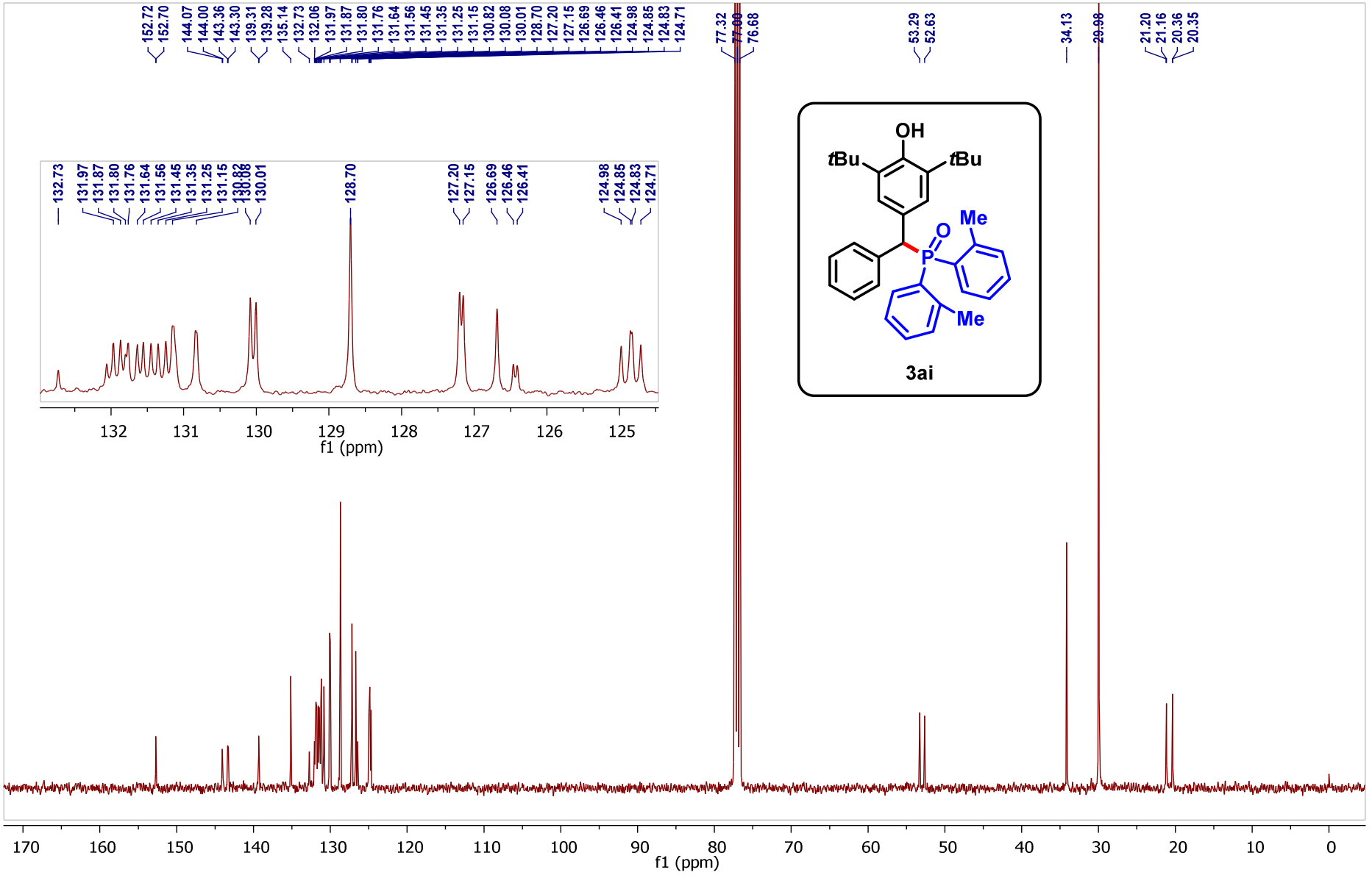
7.82
7.50
7.49
7.47
7.32
7.30
7.28
7.26
7.25
7.23
7.22
7.21
7.21
7.20
7.14
7.13
7.12
7.11
7.04
7.02
6.99
6.98
6.97
6.96
6.89
6.89
6.83
4.67
4.65
2.30
2.03
1.26
0.00
OH
tBu
tBu
Me
O
P
Me
3ai
2.07
1.07
6.76
2.13
3.23
2.10
1.00
1.02
3.08
3.15
18.40
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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143.36
143.30
139.31
139.28
135.14
132.73
132.06
131.97
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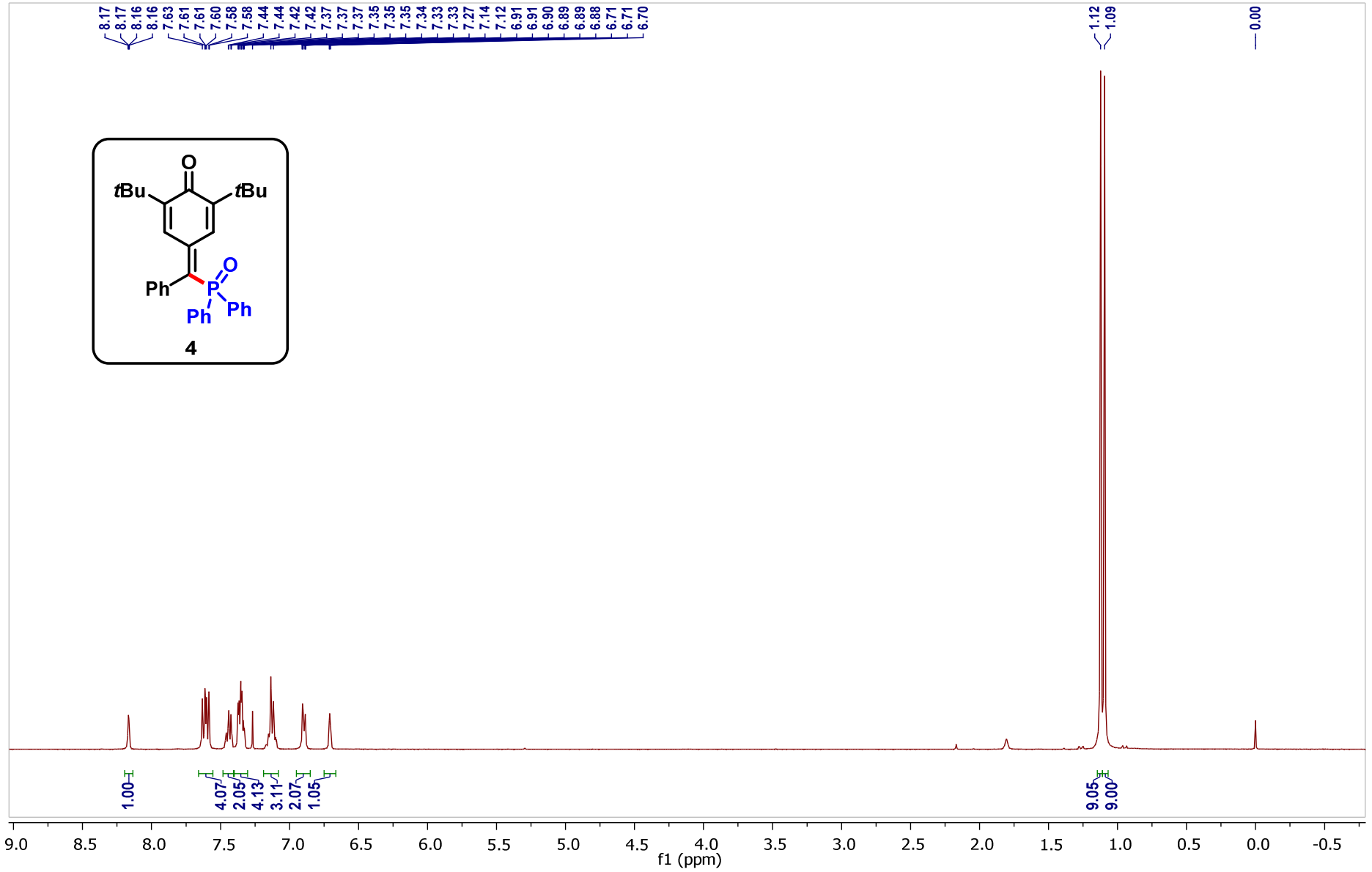
131.80
131.76
131.64
131.56
131.45
131.35
131.25
131.15
130.82
130.08
130.01
128.70
127.20
127.15
126.69
126.46
126.41
124.98
124.85
124.83
124.71
77.32
77.00
76.68
53.29
52.63
34.13
29.98
21.20
21.16
20.36
20.35
OH
tBu
tBu
Me
Me
O
P
3ai
f1 (ppm)
f1 (ppm)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8.16
8.16
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7.61
7.61
7.60
7.58
7.58
7.44
7.44
7.42
7.42
7.37
7.37
7.37
7.35
7.35
7.34
7.33
7.33
7.27
7.14
7.12
6.91
6.91
6.90
6.89
6.89
6.88
6.71
6.71
6.70
1.12
1.09
0.00
tBu
tBu
O
Ph
P
O
Ph
Ph
4
1.00
4.07
2.05
4.13
3.11
2.07
1.05
9.05
9.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
1.5
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0.5
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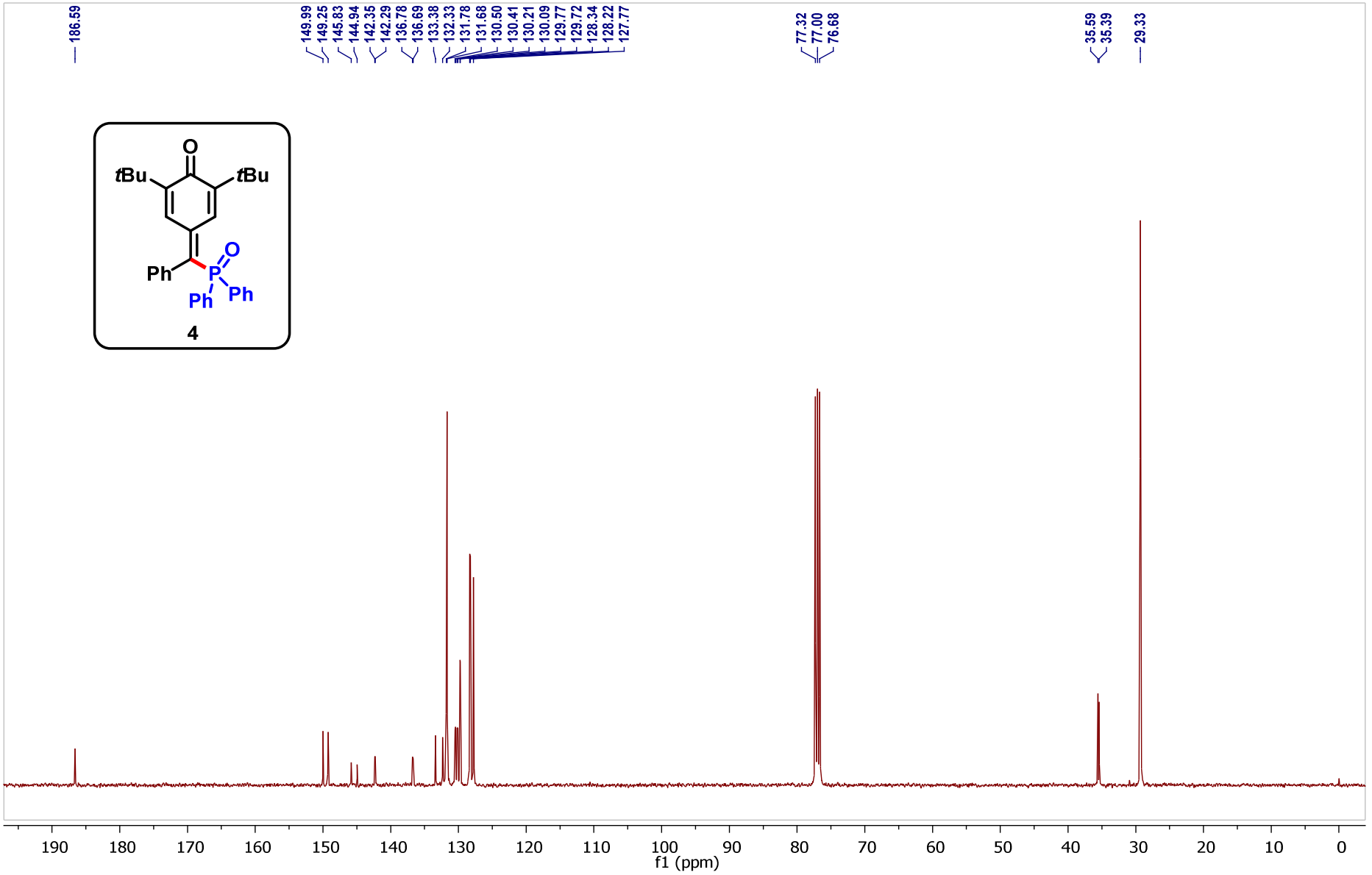
f1 (ppm)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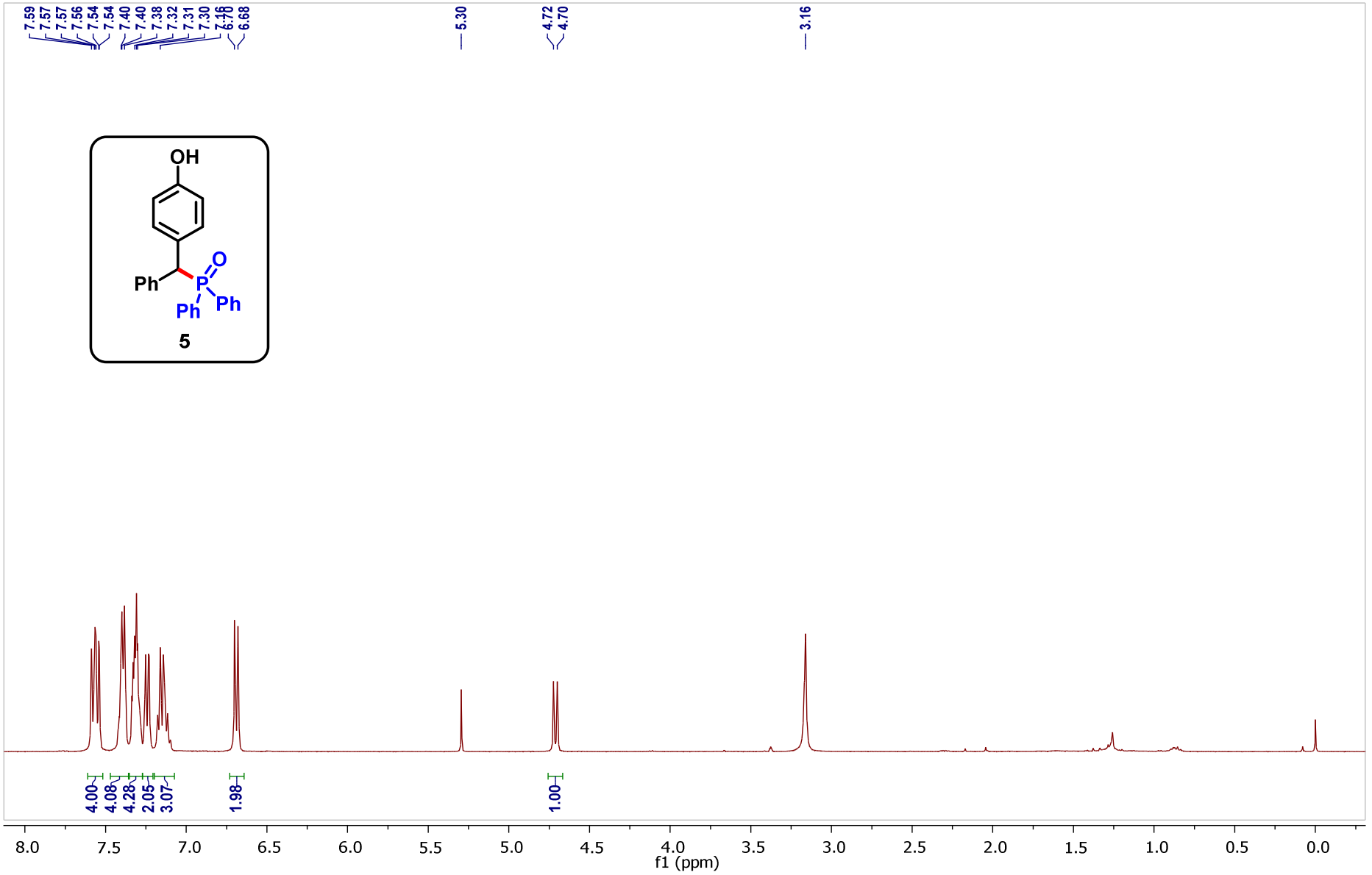
145.83
144.94
142.35
142.29
136.78
136.69
133.38
132.33
131.78
131.68
130.50
130.41
130.21
130.09
129.77
129.72
128.34
128.22
127.77
77.32
77.00
76.68
35.59
35.39
29.33
tBu
tBu
O
Ph
P
O
Ph
Ph
4
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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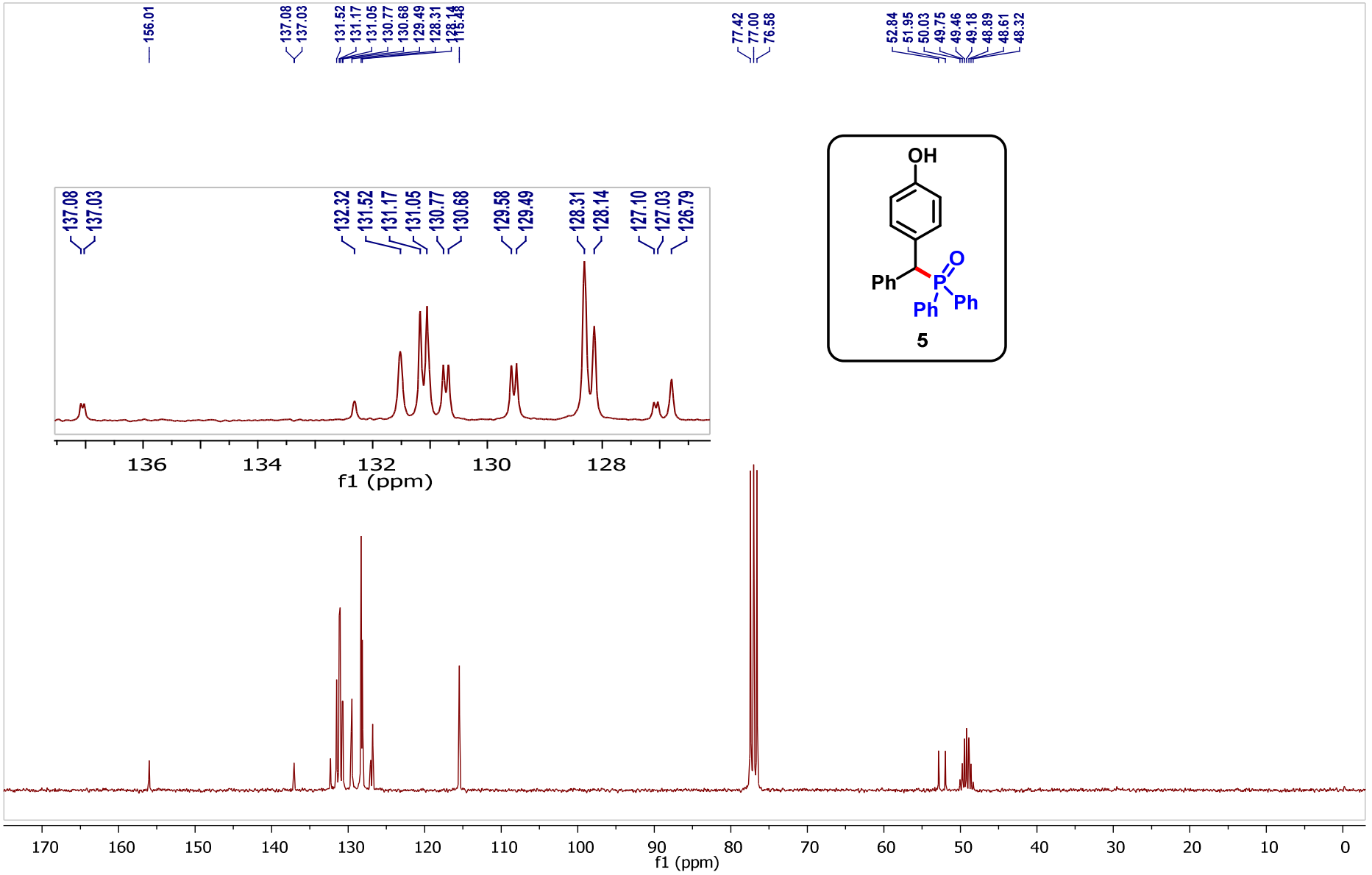
7.57
7.57
7.56
7.54
7.54
7.40
7.40
7.38
7.32
7.31
7.30
7.16
7.16
6.68
5.30
4.72
4.70
3.16
OH
Ph
O
P
Ph
Ph
5
4.00
4.08
4.28
2.05
3.07
1.98
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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131.05
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130.68
129.49
128.31
128.14
115.48
77.42
77.00
76.58
52.84
51.95
50.03
49.75
49.46
49.18
48.89
48.61
48.32
132.32
127.10
127.03
126.79
OH
Ph
O
P
Ph
Ph
5
136
134
132
130
128
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)