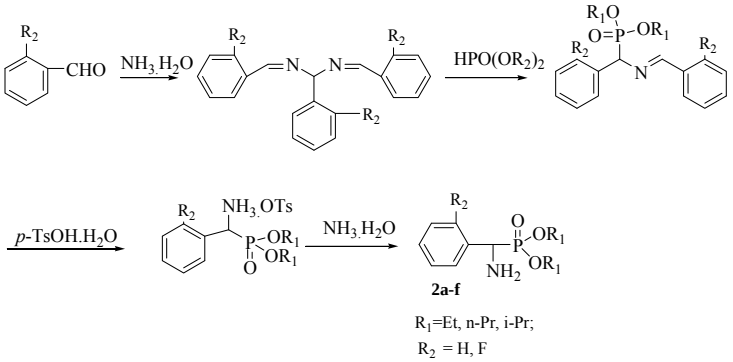
Rapid Synthesis and Antiviral Activity of (Quinazolin-4-Ylamino)Methyl-Phosphonates Through Microwave Irradiation

Supporting Information

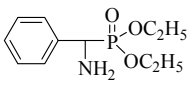
1. Instruments

The melting points of the products were determined on a XT-4 binocular microscope (Beijing Tech Instrument Co., China) and are uncorrected. IR spectra (KBr disks) were recorded on a Bruker Vector 22 spectrometer. ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded at room temperature on a JEOL-ECX 500 NMR spectrometer operating at 500 and 125 MHz, respectively, using TMS as an internal standard. Microwave irradiations were carried out in a Discovery^{TM} LabMate instrument. Elemental analysis was performed on an Elementar Vario-III CHN analyzer. Analytical TLC was performed on silica gel GF254. Column chromatographic purification was carried out using silica gel. All reagents were of analytical reagent grade or chemically pure. All solvents were dried, deoxygenated, and redistilled before use.

2. Synthesis of Intermediate 2a-f



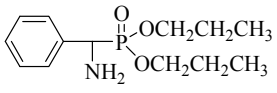
Diethyl amino(phenyl)methylphosphonate (2a)



The benzaldehyde (21.05 g, 0.2 mol) was slowly added to ammonium hydroxide (30%, 15 mL) and the mixture was stirred for 2 h under reflux. Then, a white precipitate was formed which was separated by filtration and dried. Intermediate 4 (6.02 g, 0.02 mol) was added to diethyl phosphonate (0.03 mol) and the mixture was stirred for 8 h at 75–85 °C. Chromatography on silica gel with EtOAc/Petroleum ether (1:1) gave the pure intermediate 5 as oils. *p*-Toluenesulfonic acid (3.46 g, 0.02 mol) in 60 mL THF was added to the intermediate 5 (6.65 g, 0.02 mol) and the reaction mixture was stirred for 2 h at 0 °C. The precipitate formed was removed by filtration and washed by THF (30 mL) several times to obtain white solid intermediate 6（5.24 g，0.013 mol. Ammonium hydroxide (30%, 80 mL) was slowly added to intermediate 6 and the mixture was stirred for 2 h at room temperature; the mixture was extracted with ethyl ether (2 × 20 mL), followed by evaporation to obtain crude products (2.02 g).

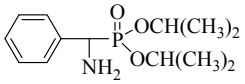
During this time, this products was chrom-atographed over a silica gel column using a solvent system (EtOAc/Petroleum ether = 1:10 to 1:0) to give the pure target intermediate 2a (1.79 g, 58.2%) as Colorless viscous liquid; nD^{20} = 1.4950; ^1H NMR ($CDCl_3$, 500 MHz) δ: 7.32–7.45 (m, 5H, Ar-2,3,4,5,6-H), 4.23 (d, *J* = 10.0 Hz, 1H, CH), 4.02~4.09 (m, 4H, $2CH_2$), 2.01 (s, 2H, NH_2), 1.16–1.25 (m, 6H, $2CH_3$), ^{13}C NMR ($CDCl_3$, 125MHz): *δ* 137.60, 128.35, 127.76, 127.73, 127.68, 77.40, 62.79, 62.67, 16.39, 16.35, ^{31}P NMR ($CDCl_3$, 500 MHz): δ 25.3; IR (KBr): ν 3379.08, 2981.34, 1238. 56, 1026.39; *Anal.* Calcd for $C_{11}H_{18}NO_3P$: C 54.32, H 7.46, N 5.76; Found. C 54.56, H 7.25, N 5.84.

Di-*n*-propyl amino(phenyl)methylphosphonate (2b)



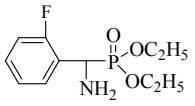
Intermediate **2b** was prepared by reacting intermediate **4** (6.02 g, 0.02 mol) with dipropyl phosphonate (0.03 mol) using a procedure similar to the preparation of **2a** to obtain colorless liquid of **2b** (1.83 g, 56.1%); nD^{20} = 1.4865; ^1H NMR: ($CDCl_3$, 500 MHz) δ: 7.35–7.46 (m, 5H, Ar-2,3,4,5,6-H), 4.24 (d, *J* = 10.0 Hz, 1H, CH), 3.93–4.12 (m, 4H, $2OCH_2$), 2.05 (s, 2H, NH_2), 0.94–1.26 (m , 4H, $2CH_2$), 0.87–0.93 (m, 6H, $2CH_3$), ^{13}C NMR ($CDCl_3$, 125 MHz): δ 137.86, 128.32, 77.72, 77.18, 68.01, 60.29, 54. 61, 53.43, 23.86, 20.94, 14.14, 9.97, 9.90, ^{31}P NMR ($CDCl_3$, 500 MHz): δ 24.7; IR (KBr): ν 3377.02, 2981.56, 1232.20, 1026.06); *Anal.* Calcd for $C_{13}H_{22}NO_3P$: C 57.55, H 8.17, N 5.16; Found. C 57.26, H 8.23, N 10.95.

Diispropyl amino(phenyl)methylphosphonate (2c)



Intermediate **2c** was prepared by contacting intermediate **4** (6.02 g, 0.02 mol) with disopropyl phosphonate (0.03 mol) using a procedure similar to the preparation of **2a** to obtain colorless liquid of **2c** (1.87 g, 56.7%); nD^{20} = 1.4867. ^1H NMR: ($CDCl_3$, 500 MHz) δ: 7.33–7.45 (m, 5H, Ar-2,3,4,5,6-H), 4.76 (d, *J* = 8.5Hz, 1H, CH), 4.59–4.66 (m, 1H, OCH), 4.48–4.54 (m, 1H, OCH), 2.04 (s , 2H, NH_2), 1.24–1.30 (m, 12H, $4CH_3$), ^{13}C NMR ($CDCl_3$,125MHz): δ 128.33, 127.94, 127.90, 127.76, 127.75, 77.30, 77.06, 76.80, 71.46, 55.05, 24.20, 24.19, 23.90, ^{31}P NMR ($CDCl_3$, 500 MHz): δ 23.9; IR (KBr, cm^{-1}): ν 3379.02, 2981.56, 1233.21, 1025.86; *Anal.* Calcd for $C_{13}H_{22}NO_3P$: C 57.55, H 8.17, N 5.16; Found. C 57.41, H 8.16, N 10.87.

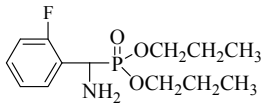
Diethyl amino(2-fluorophenyl)methylphosphonate (2d)



The 2-fluorobenzaldehyde (24.62 g, 0.2 mol) was slowly added to ammonium hydroxide (30%, 15 mL) and the mixture was stirred for 2 h under reflux.Then, a white precipitate was formed which

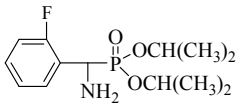
was separated by filtration and dried. Intermediate 7 (7.06 g, 0.02 mol) was added to diethyl phosphonate (0.03 mol) and the mixture was stirred for 8 h at 75–85 °C. Chromatography on silica gel with EtOAc/Petroleum ether (1:1) gave the pure intermediate **8** as oils. 4-Methylbenzenesulfonic acid(3.46 g, 0.02 mol) in 60 mL THF was added to the intermediate **8** (6.65 g, 0.02 mol) and the reaction mixture was stirred for 2 h at 0 °C. The precipitate formed was removed by filtration and washed by THF (30 mL) several times to obtain white solid intermediate of **9 (**4.35 g, 0.01 mol). Ammonium hydroxide (30%, 80 mL) was slowly added to intermediate **9** and the Mixture was stirred for 2h at room temperature; the mixture was extracted with ethyl ether (2 × 20 mL), followed by evaporation to obtain crude products (1.68 g). During this time, this products was chromatographed over a silica gel column using a solvent system (EtOAc/Petroleum ether = 1:10 to 1:0) to give Colorless viscous liquid of the pure target intermediate **2d** (1.40 g, 53.2%) as Colorless viscous liquid; n^{D20} = 1.4766; ^{1}H NMR: ($CDCl_3$, 500 MHz): δ 7.05–7.57 (m, 4H, Ar-3,4,5,6-H), 4.59 (d, *J* = 15.0 Hz, 1H, CH), 4.12–4.15 (m, 4H, $2CH_2$), 2.36 (s, 2H, NH_2), 1.15–1.32 (m, 6H, $2CH_3$), ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 137.72, 128.18, 127.64, 127.60, 77.59, 62.61, 62.56, 16.28, 16.23, 16.15, ^{31}P NMR ($CDCl_3$, 500 MHz): δ 24.7; IR (KBr): ν 3377.36, 2981.95, 1234.44, 1028.06; *Anal.* Calcd for $C_{11}H_{17}FNO_3P$: C 50.58, H 6.56, N 5.36; Found. C 50.41, H 6.27, N 5.74

Di-*n*-propyl amino(2-fluorophenyl)methylphosphonate (2e)



Intermediate **2e** was prepared by reacting intermediate **7** (7.06 g, 0.02 mol) with dipropyl phosphonate (5.10 g, 0.03 mol) using a procedure similar to the preparation of **2d** to obatin colorless liquid of **2e** (1.49 g, 52.7%); nD^{20} = 1.4826; ^{1}H NMR: ($CDCl_3$, 500 MHz): δ 7.04–7.57 (m, 4H, Ar-3,4,5,6-H), 4.62 (d, 1H, *J* = 15Hz, CH), 4.03–4.13 (m, 4H, $2OCH_2$), 2.05 (s, 2H, NH_2), 0.96–1.26 (m , 4H, $2CH_2$), 0.82–0.94 (m, 6H, $2CH_3$), ^{13}C NMR ($CDCl_3$, 125MHz): δ 129.42, 124.45, 77.38, 77.12, 68.44, 60.50, 47.05, 45.86, 22.98, 22.89, 21.15, 14.29, 9.94, ^{31}P NMR ($CDCl_3$, 500 MHz) δ: 24.7; IR (KBr, cm^{-1}): ν 3296.35, 2931.80, 1235.56, 1053.13; *Anal.* Calcd for $C_{13}H_{21}FNO_3P$: C 53.97, H 7.32, N 4.84; Found. C 54.21, H 7.16, N 4.87.

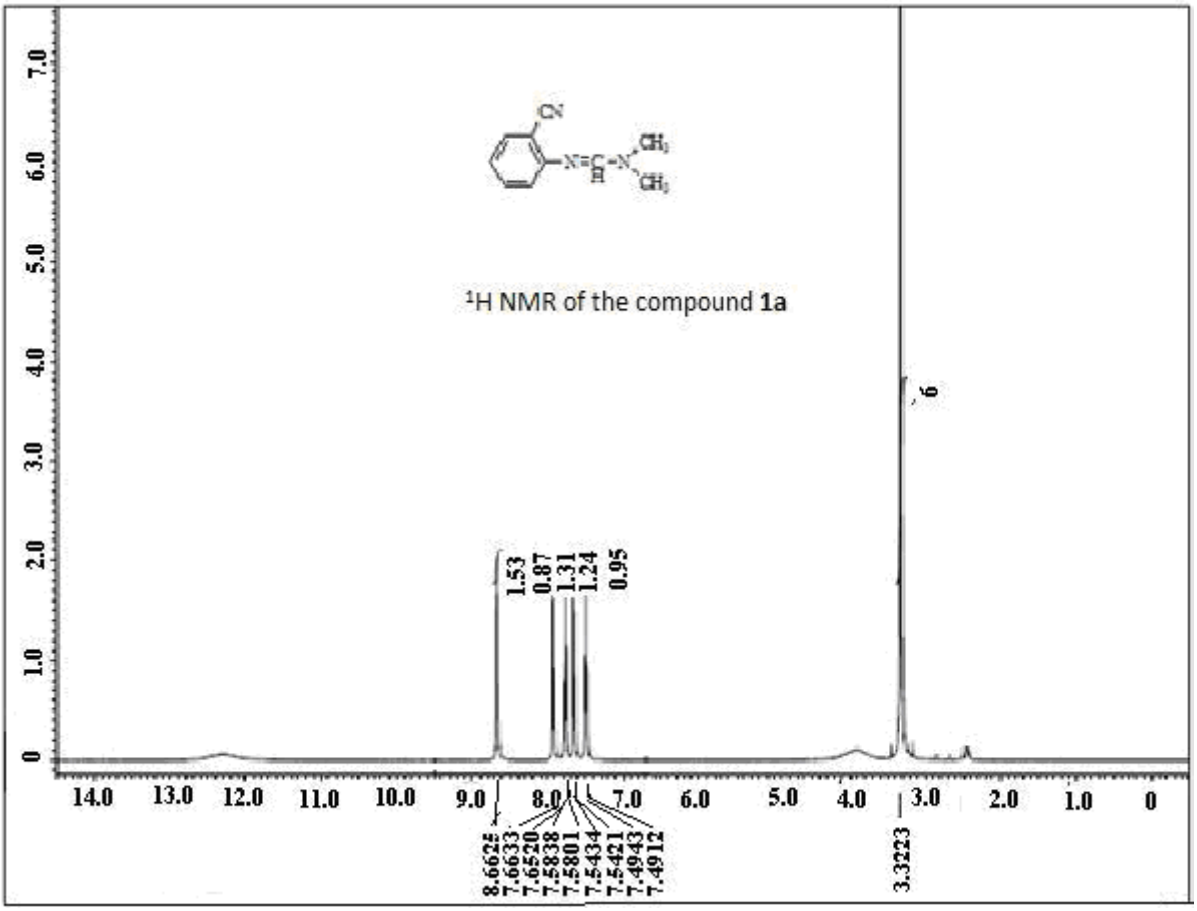
Diispropyl amino(2-fluorophenyl)methylphosphonate(2f)

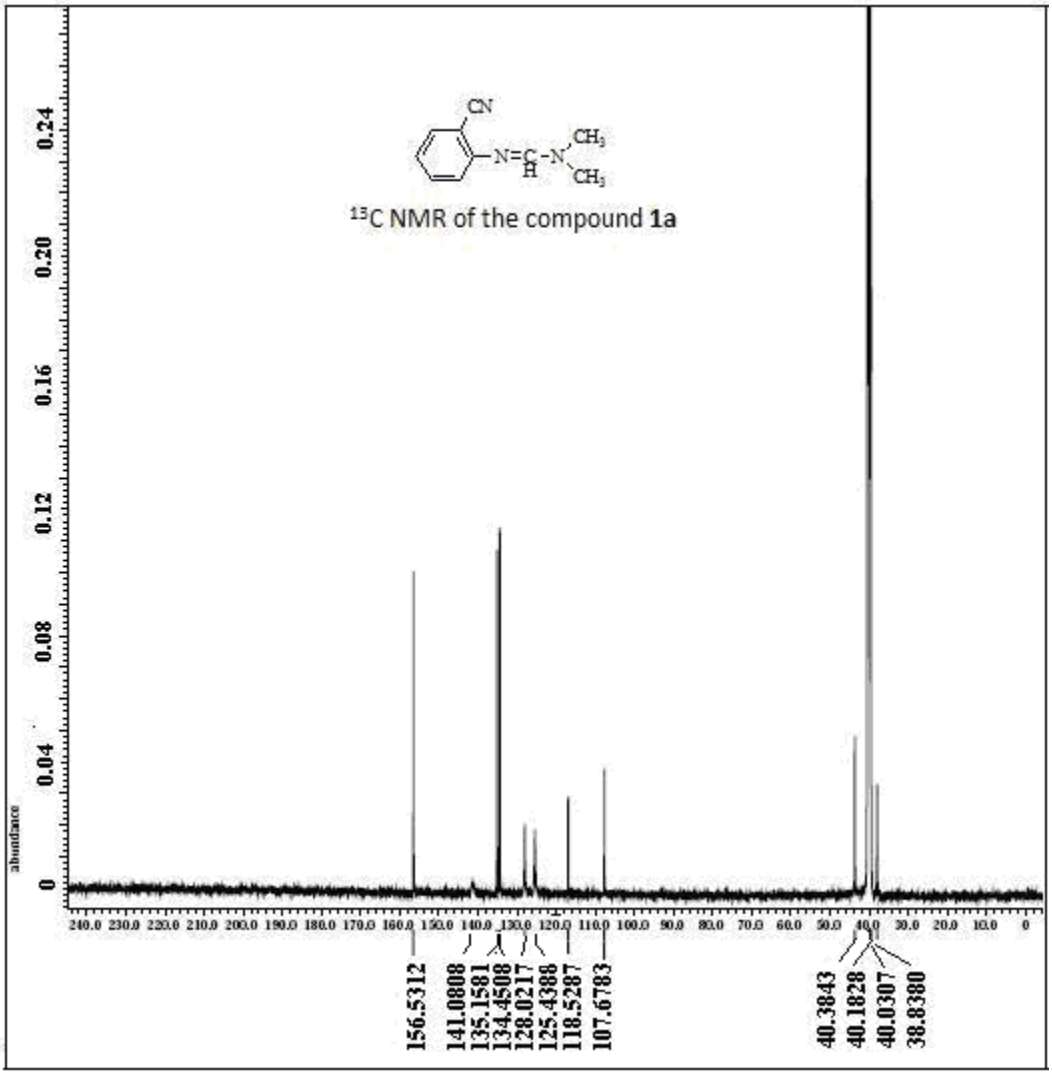


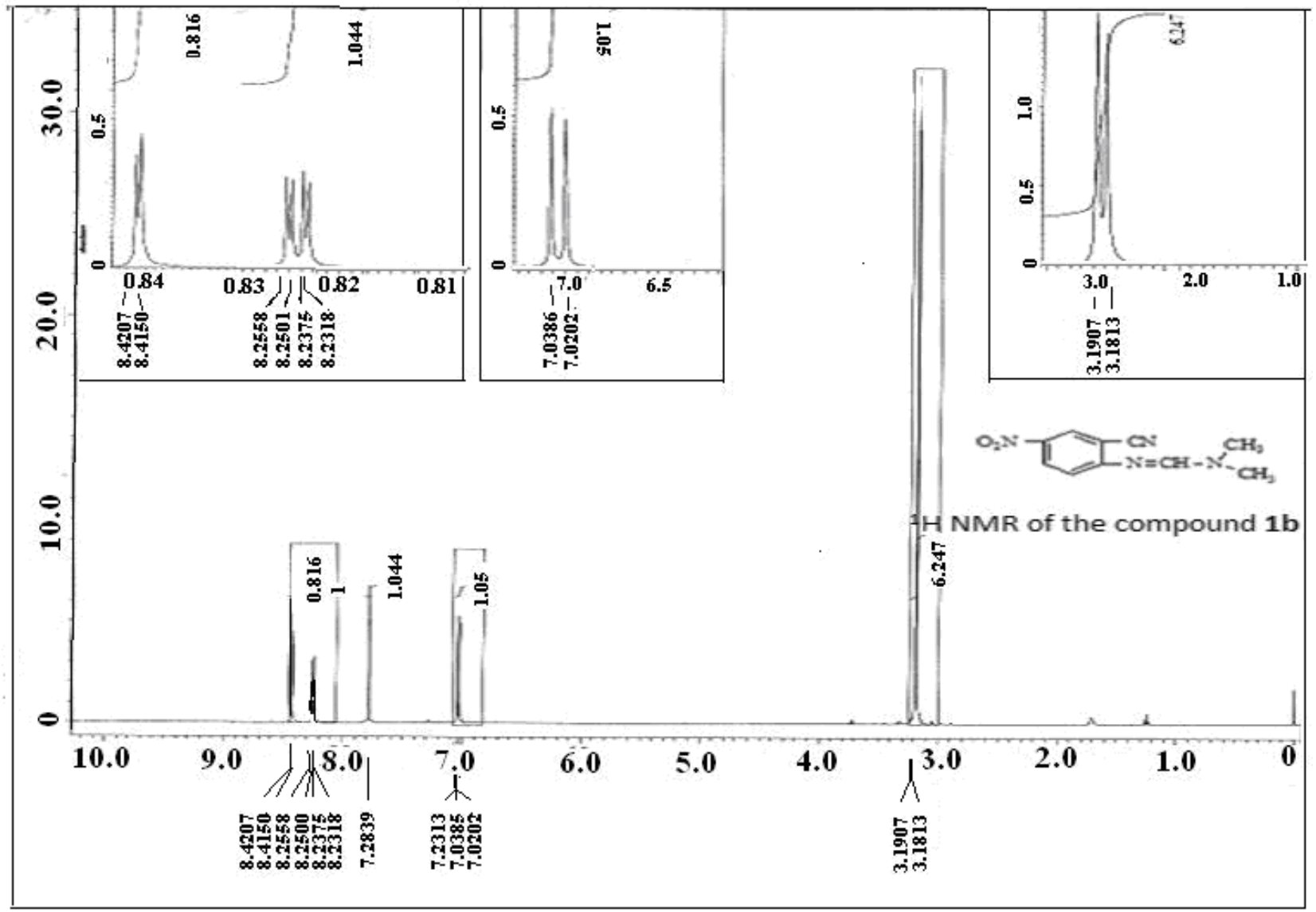
Intermediate **2f** was prepared by reacting intermediate **7** (7.06 g, 0.02 mol) with disopropyl phosphonate (5.10 g, 0.03 mol) using a procedure similar to the preparation of **2d** to obtain colorless liquid of **2f** (1.70 g, 52.1%); nD^{20} = 1.4826; ^{1}H NMR: ($CDCl_3$, 500 MHz): *δ* 7.05~7.59 (m, 4H, Ar-3,4,5,6-H), 4.72–4.74 (m, 2H,2OCH), 4.52 (d, *J* = 15Hz, 1H, CH), 2.17 (s, 2H, NH_2), 1.24–1.34 (m, 12H, $4CH_3$); ^{13}C NMR ($CDCl_3$,125MHz) δ: 128.97, 125.68, 125.57, 123.99, 115.01, 114.70, 77.50, 71.23, 45.87, 45.77, 23.70, 23.83, 23.57; ^{31}P NMR: ($CDCl_3$, 500 MHz) δ: 23.0; IR (KBr, cm^{-1}): ν 3346.16, 2951.73, 1223.90, 1015.46; *Anal.* Calcd for $C_{13}H_{21}FNO_3P$: C 53.97, H 7.32, N 4.84; Found.

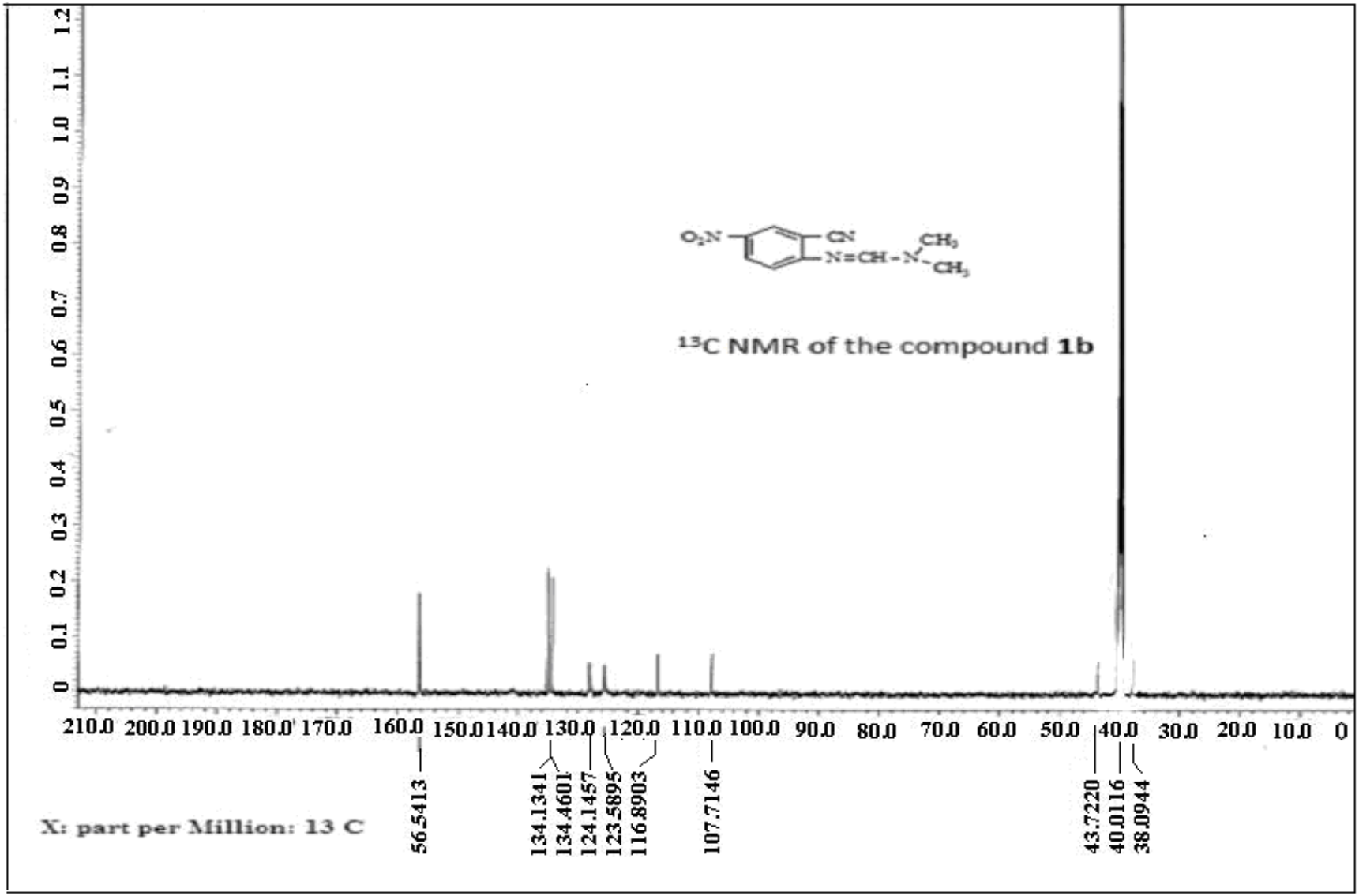
C 54.16, H 7.53, N 4.99.

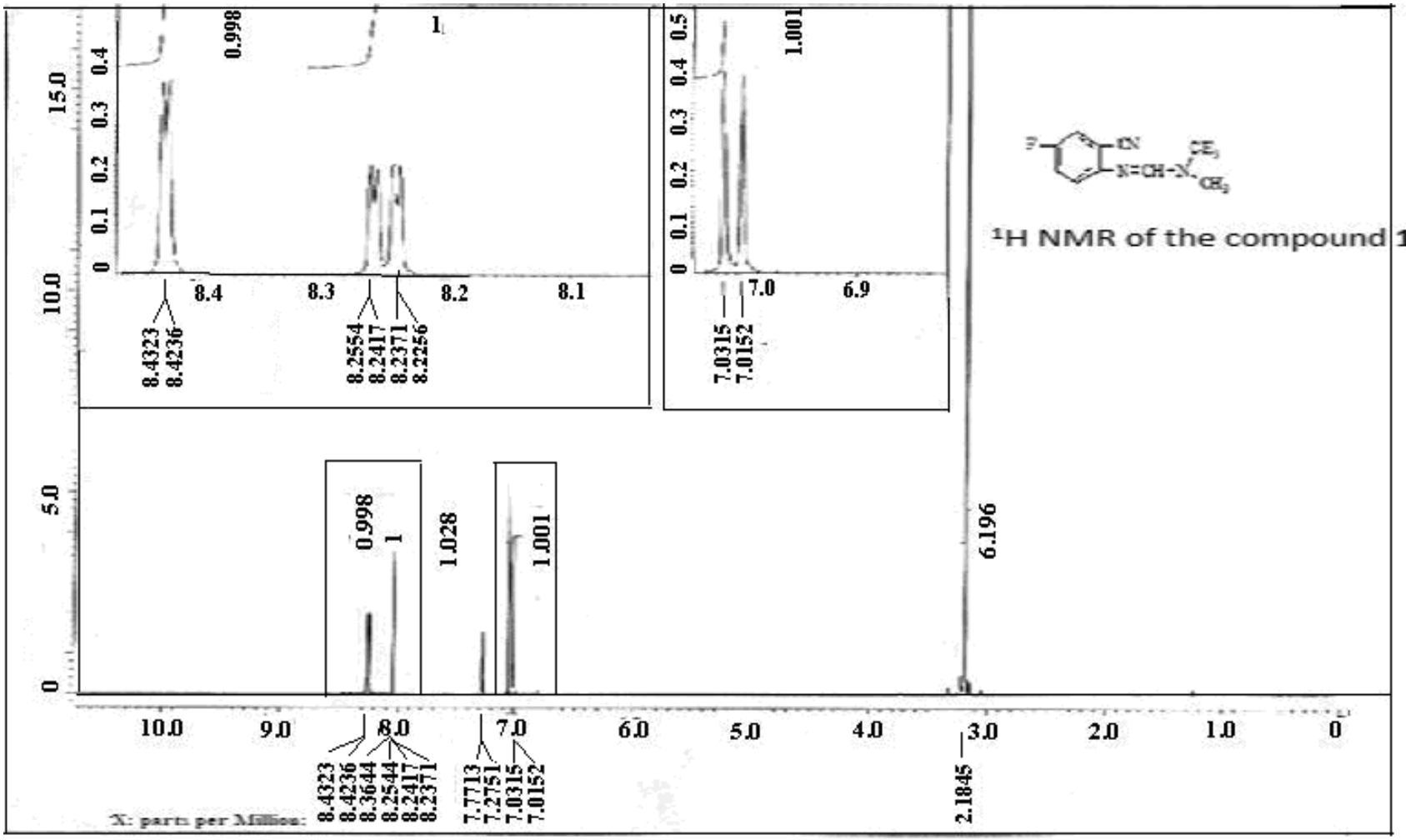
3. NMR Spectra (^{1}H, ^{13}C and ^{31}P) for 1a-d, 2a-f, 3a-3x

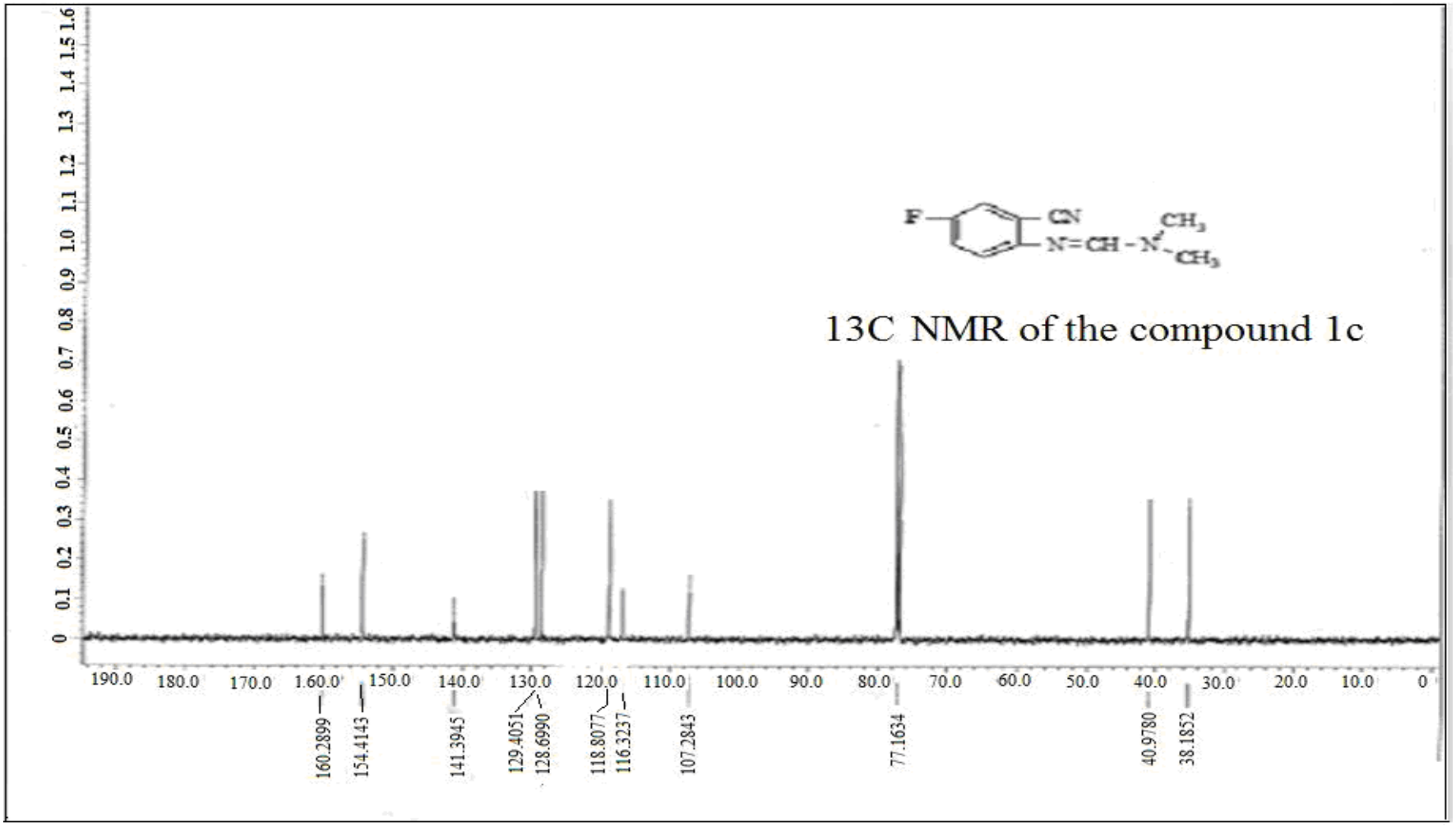


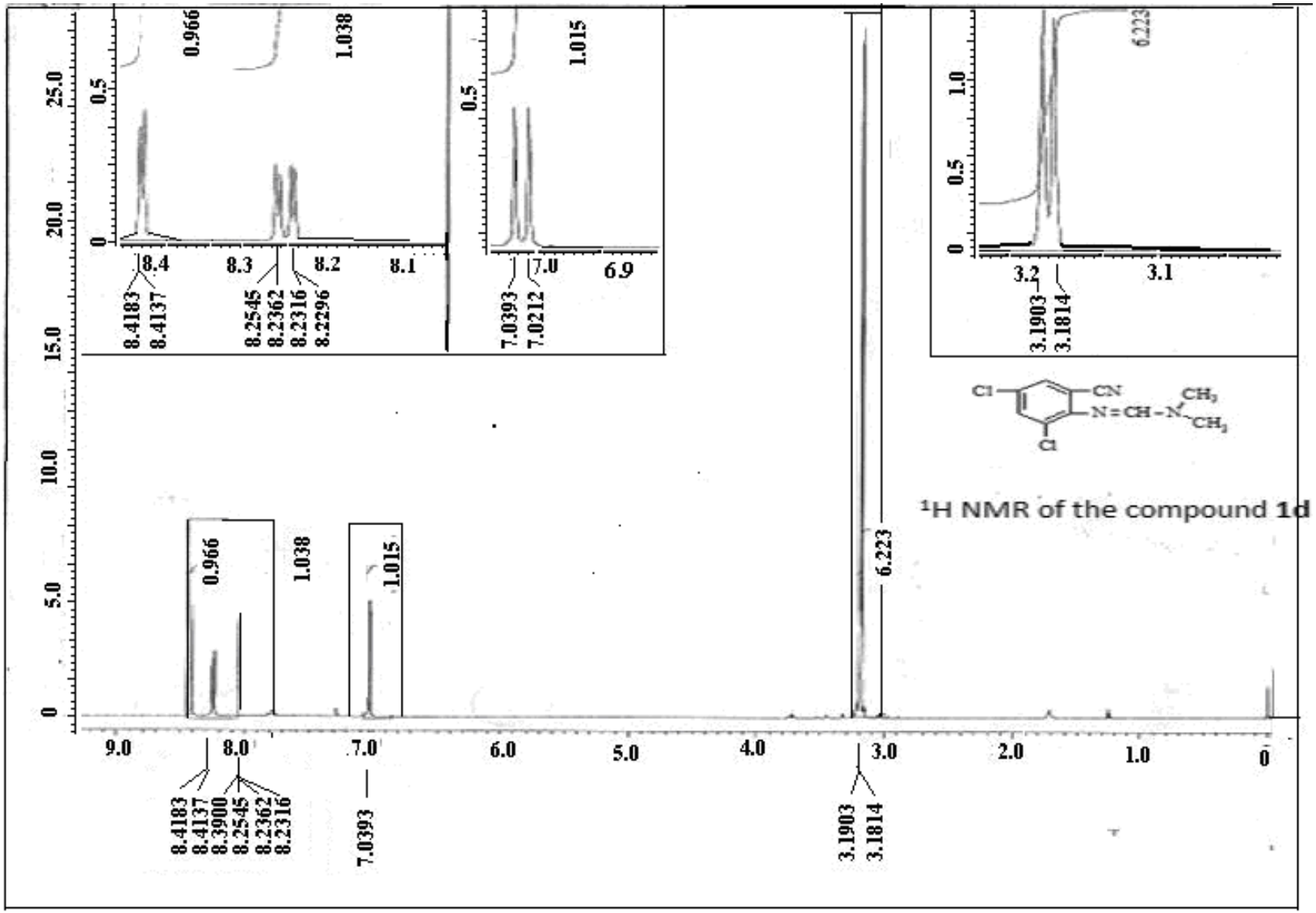

CN
N=C
H
N
CH3
CH3
13C NMR of the compound 1a
abundance
0
0.04
0.08
0.12
0.16
0.20
0.24
156.5312
141.0808
135.1581
134.4508
128.0217
125.4388
118.5287
107.6783
40.3843
40.1828
40.0307
38.8380

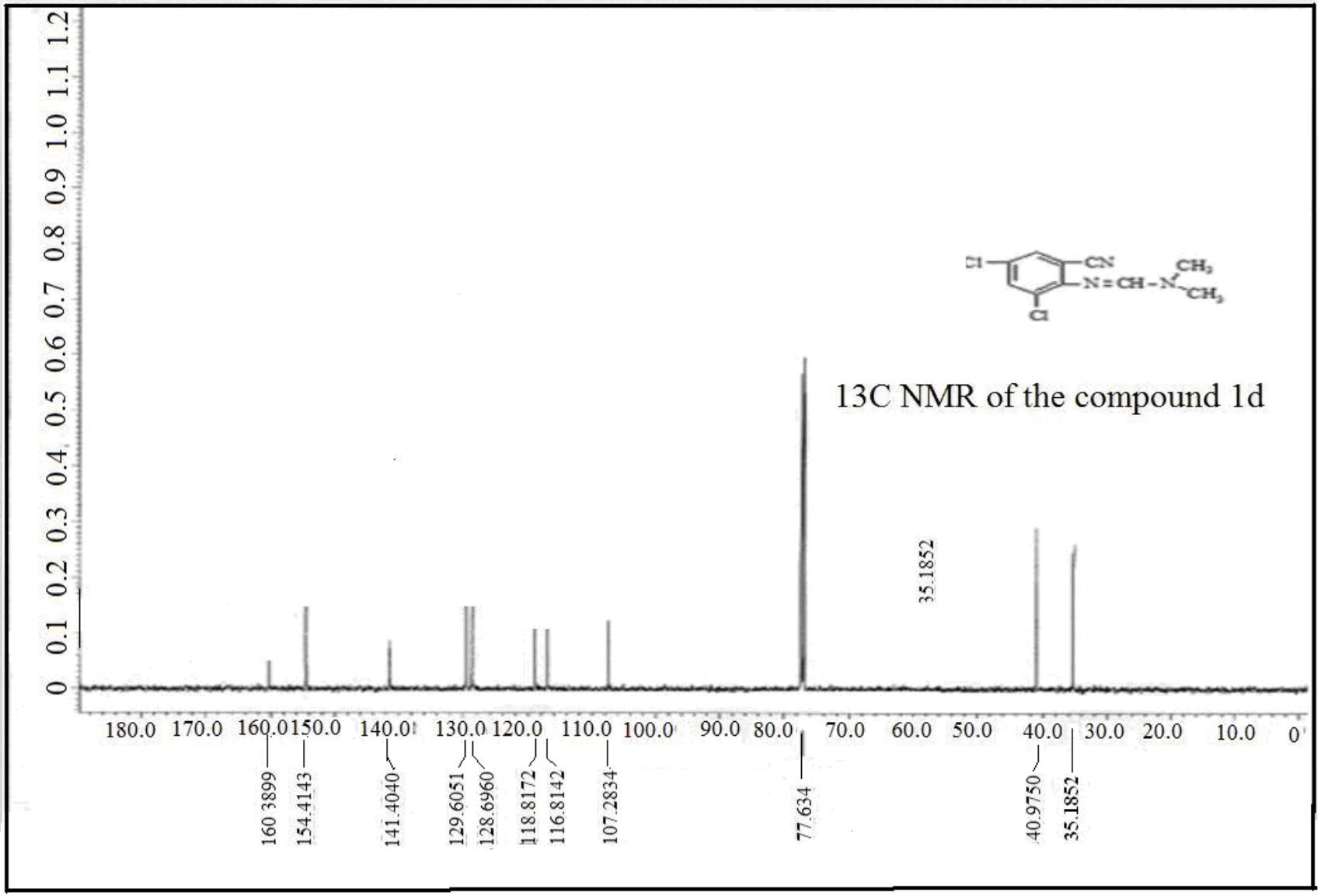

1H NMR of the compound 1b
O2N
CN
N=CH-N
CH3
CH3
8.4207
8.4150
8.2558
8.2500
8.2375
8.2318
7.2839
7.2313
7.0385
7.0202
3.1907
3.1813
0.816
1
1.044
1.05
6.247

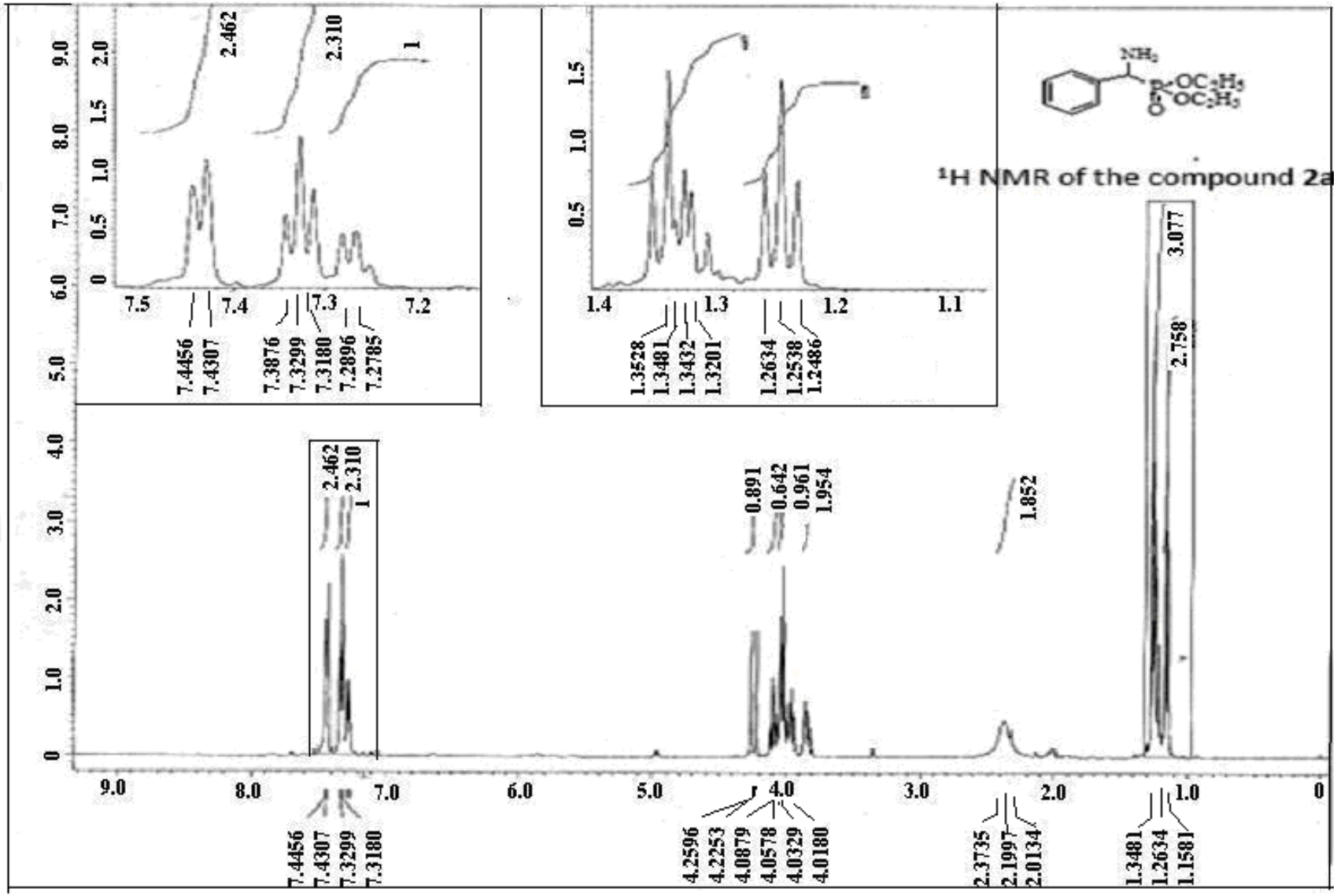

^{13}C NMR of the compound **1b**
X: part per Million: 13 C
56.5413
134.1341
134.4601
124.1457
123.5895
116.8903
107.7146
43.7220
40.0116
38.0944

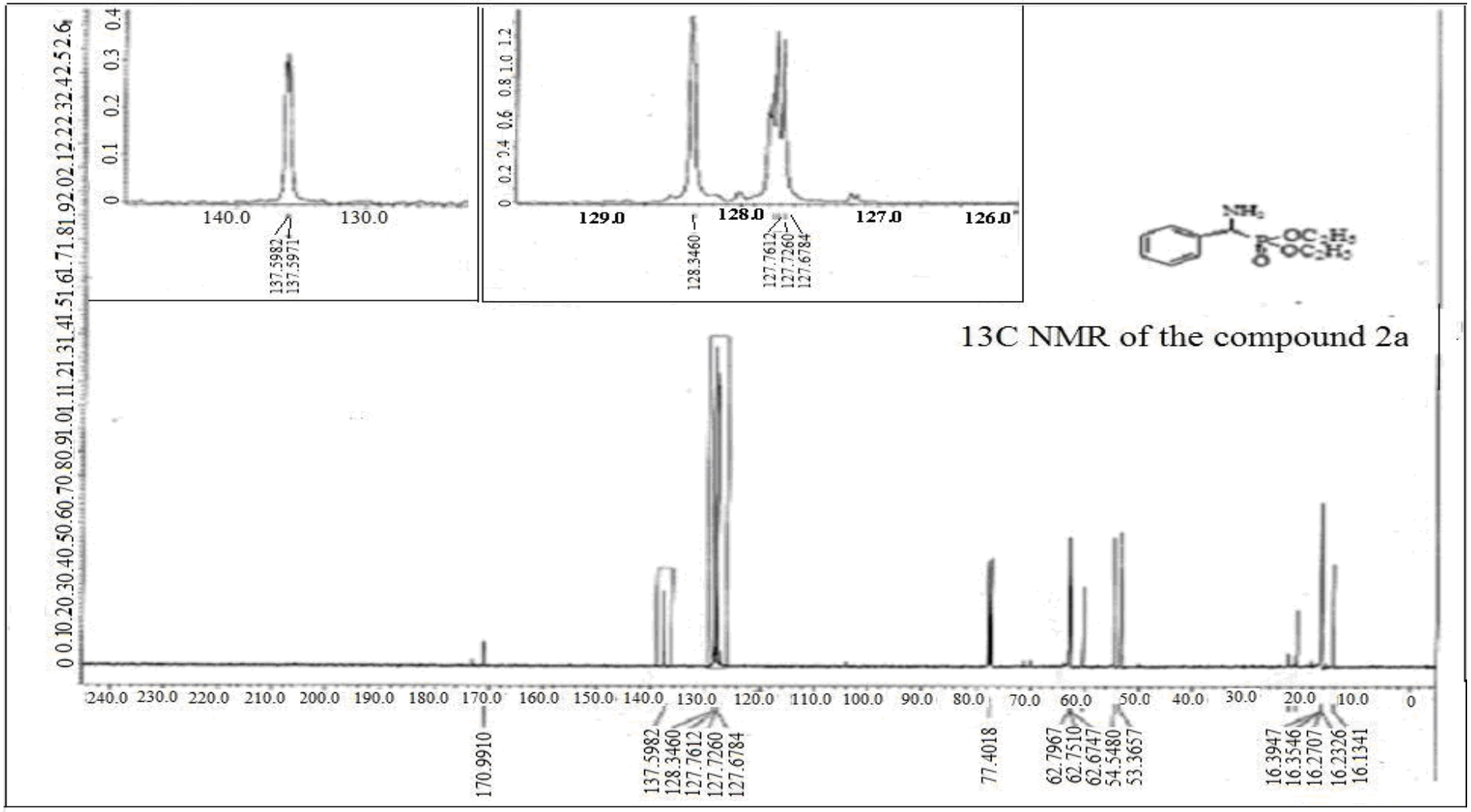

¹H NMR of the compound 1c
X: parts per Million:

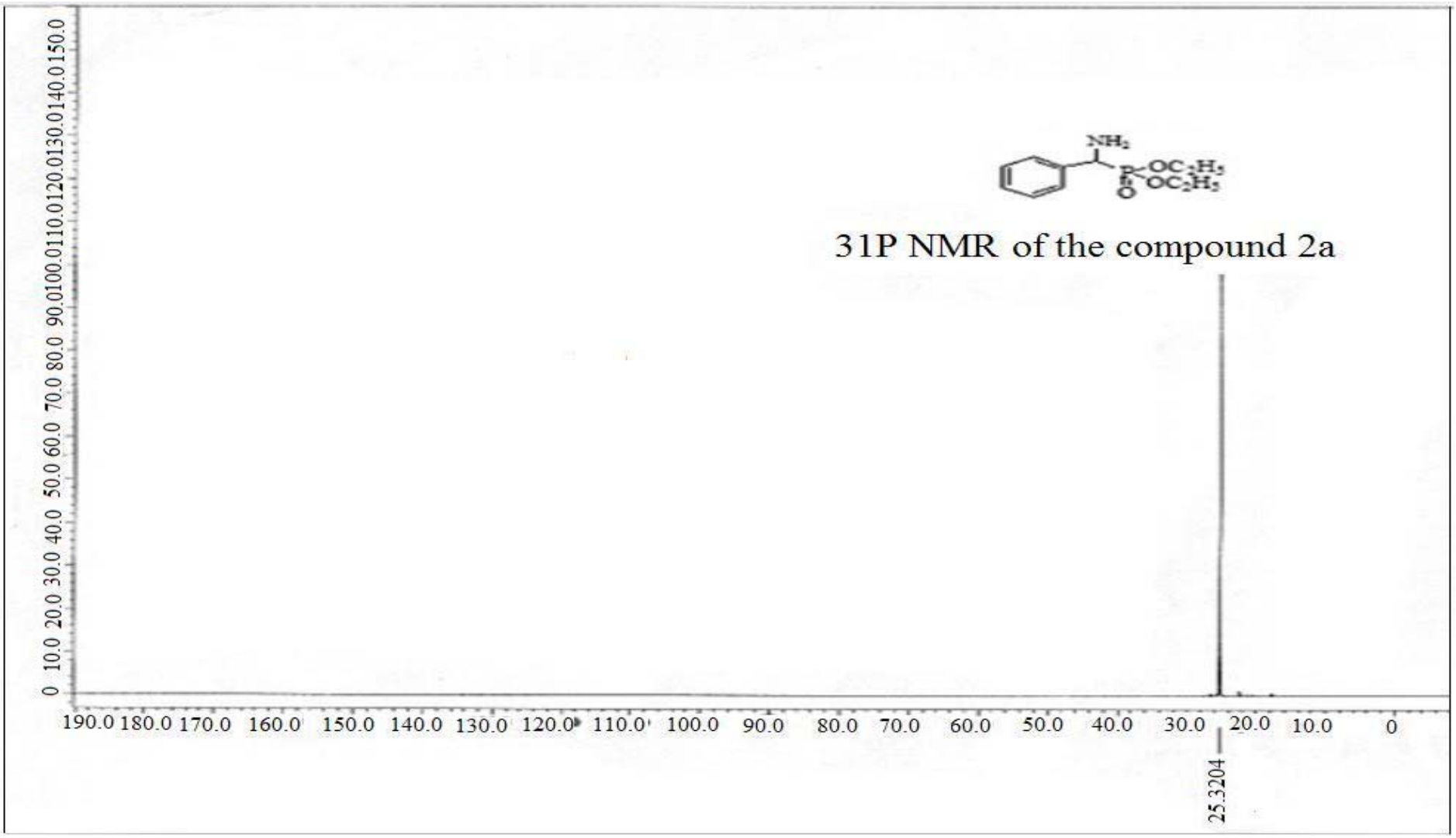

13C NMR of the compound 1c
160.2899
154.4143
141.3945
129.4051
128.6990
118.8077
116.3237
107.2843
77.1634
40.9780
38.1852

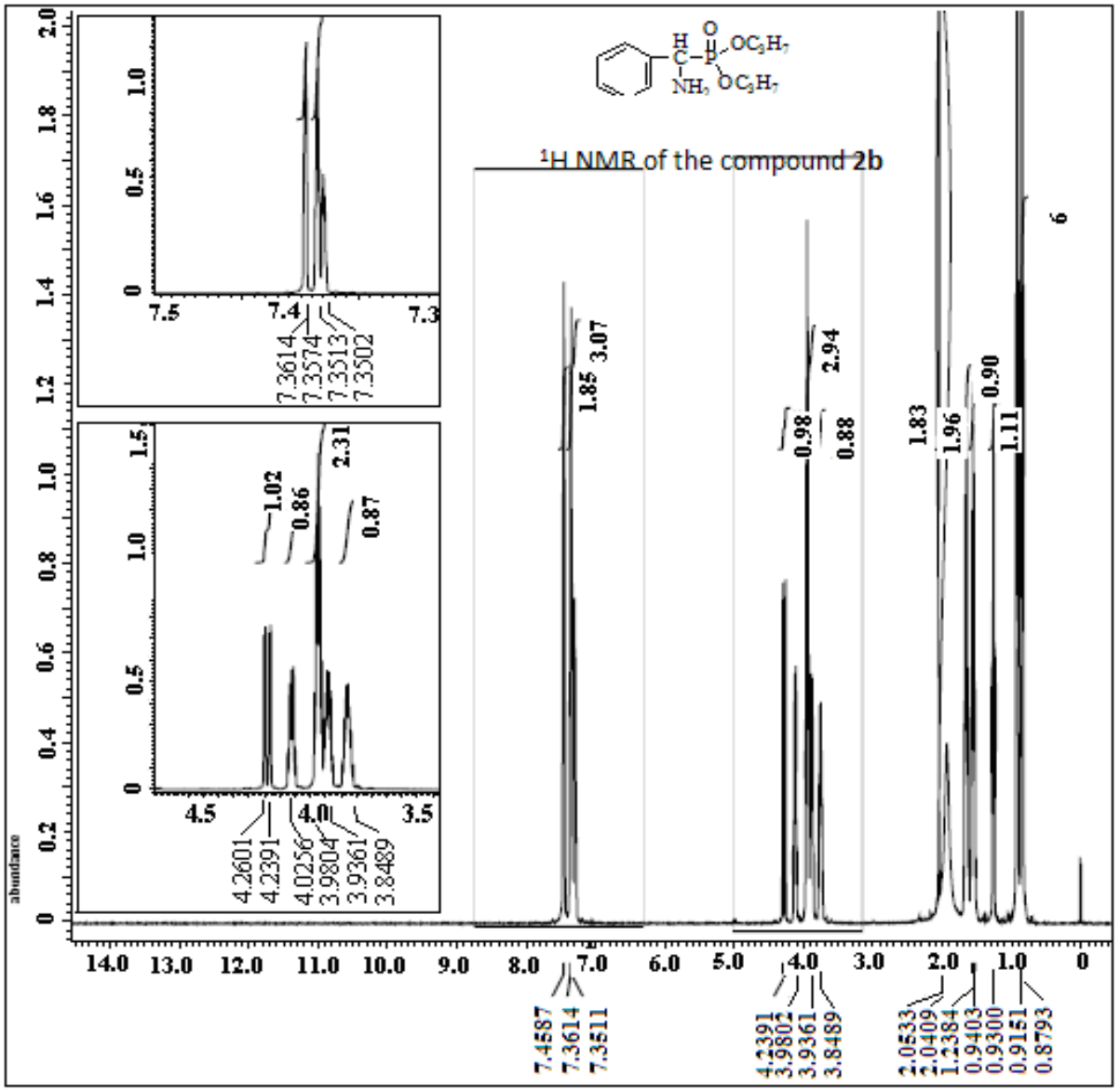

0.966
1.038
1.015
6.223
8.4183
8.4137
8.3900
8.2545
8.2362
8.2316
7.0393
7.0212
3.1903
3.1814
Cl
CN
N=CH-N
CH3
CH3
Cl
¹H NMR of the compound 1d

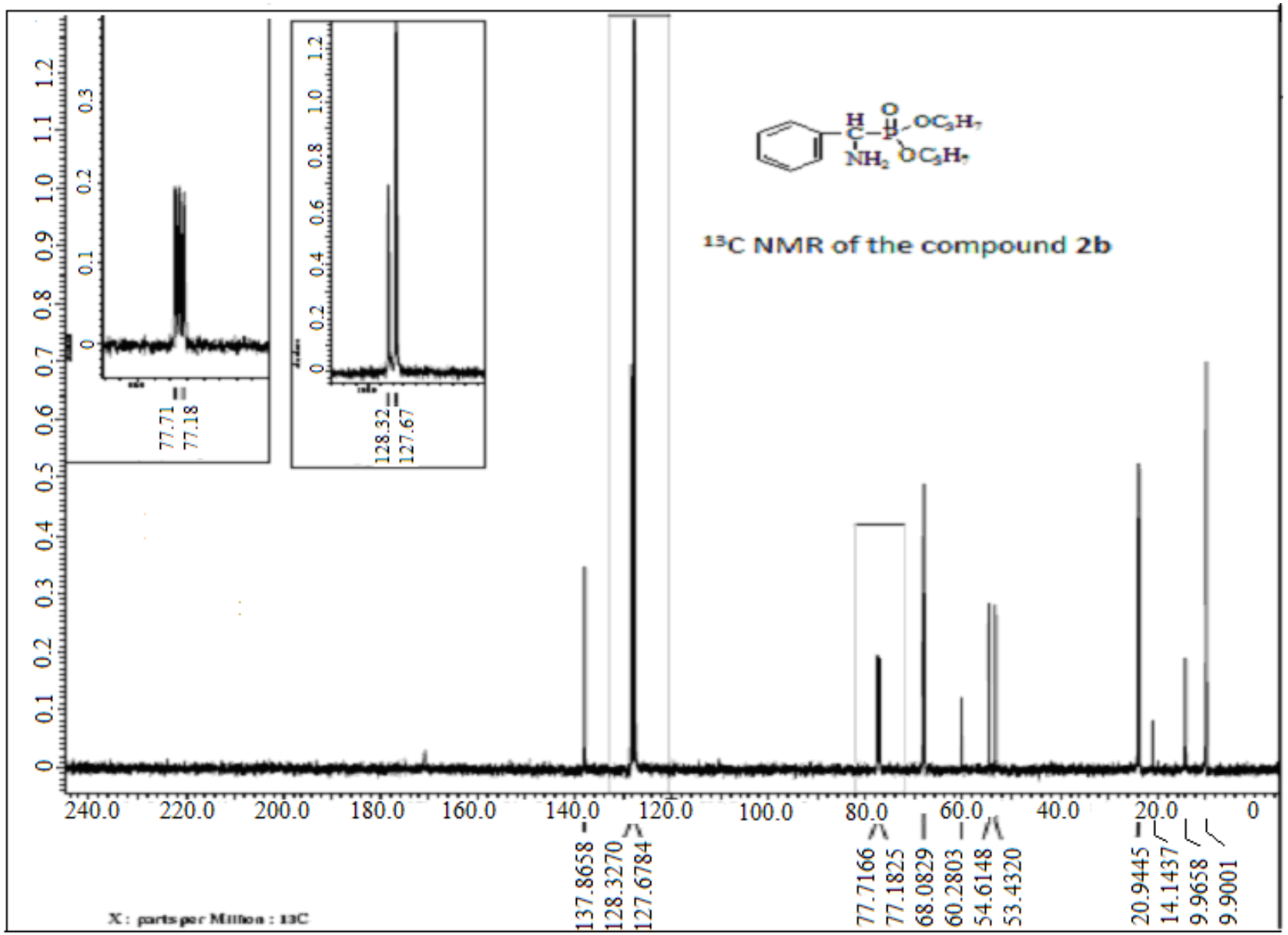

13C NMR of the compound 1d
35.1852
160.3899
154.4143
141.4040
129.6051
128.6960
118.8172
116.8142
107.2834
77.634
40.9750
35.1852

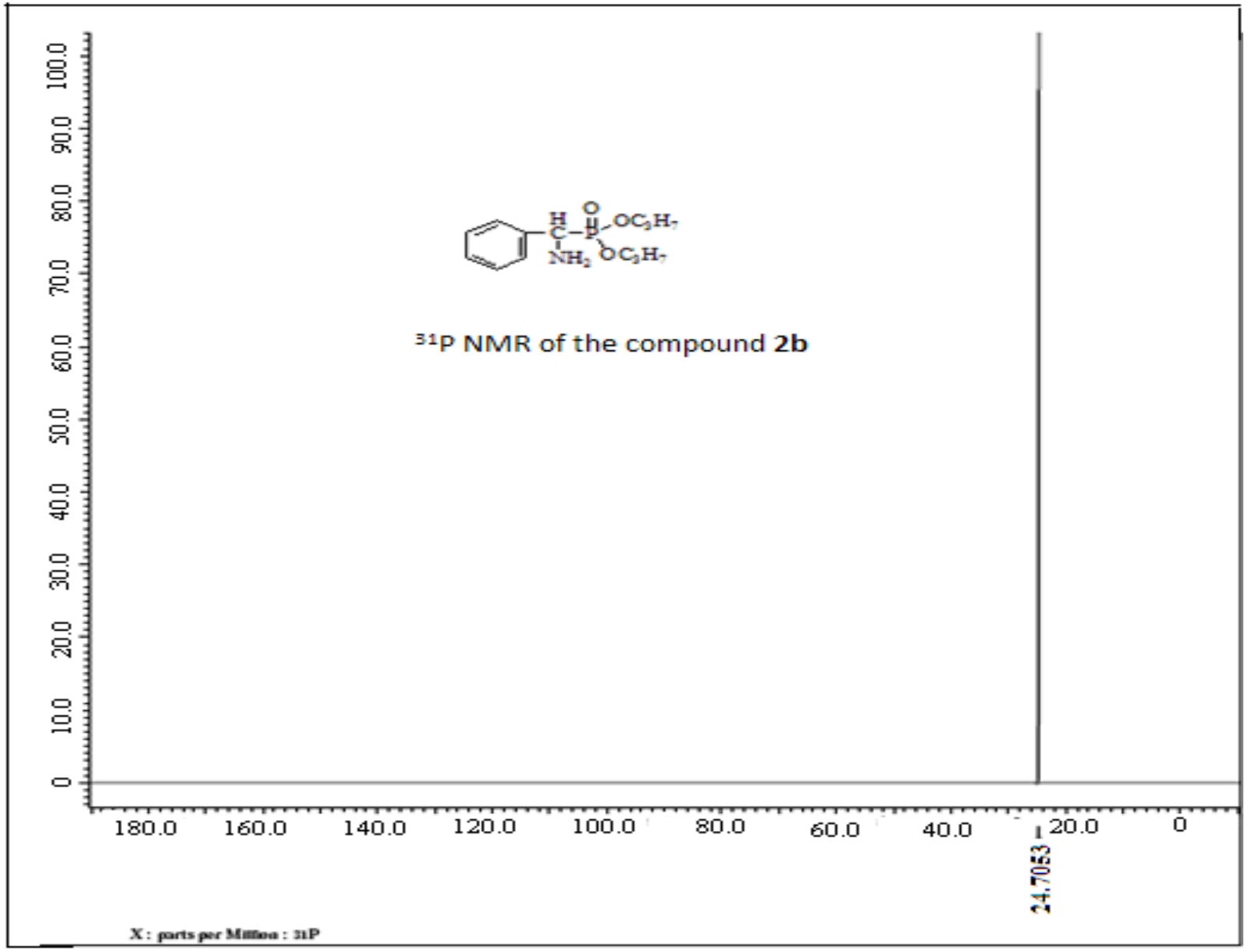

¹H NMR of the compound 2a


13C NMR of the compound 2a
137.5982
137.5971
128.3460
127.7612
127.7260
127.6784
170.9910
77.4018
62.7967
62.7510
62.6747
54.5480
53.3657
16.3947
16.3546
16.2707
16.2326
16.1341

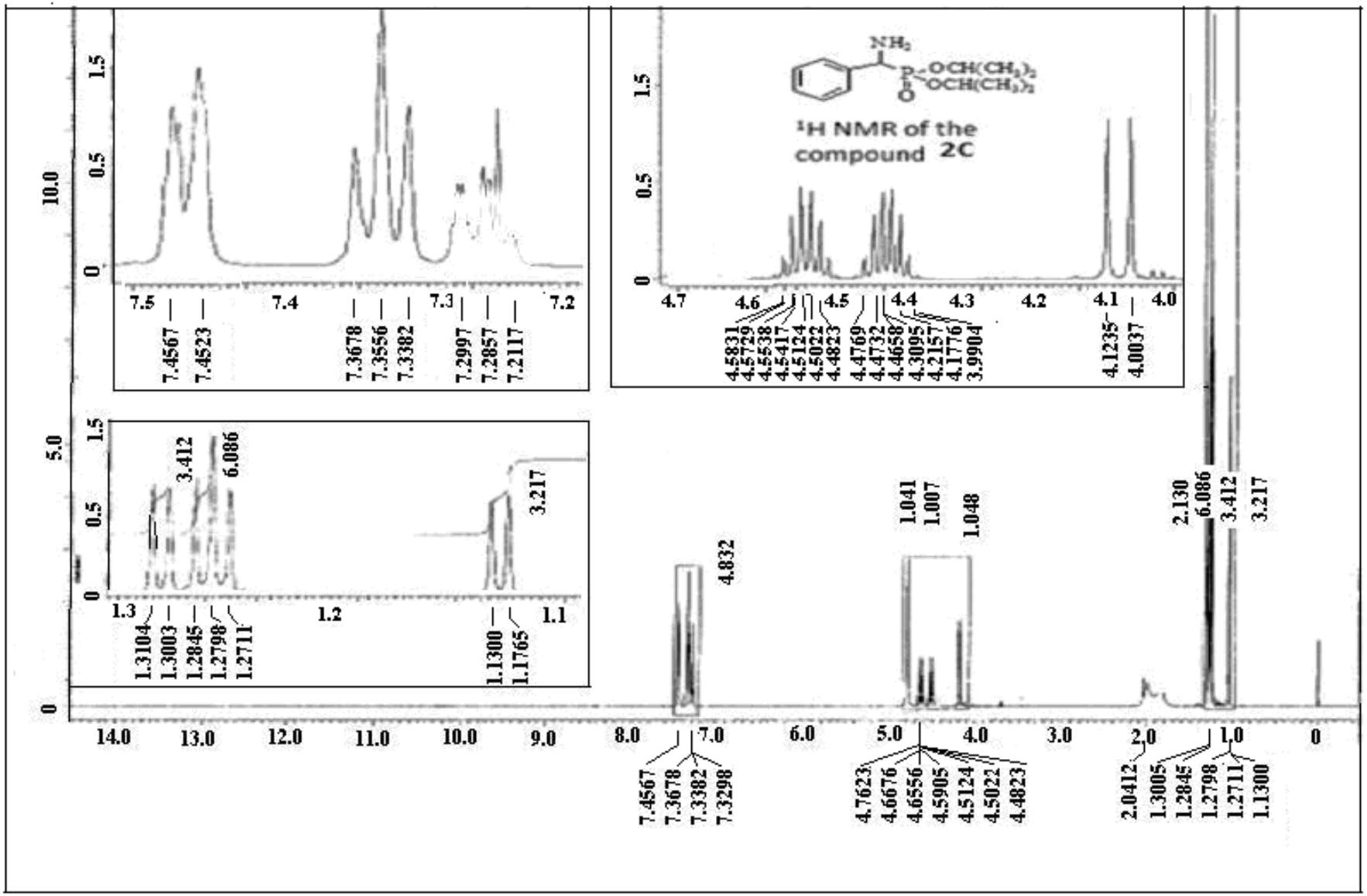

31P NMR of the compound 2a
25.3204

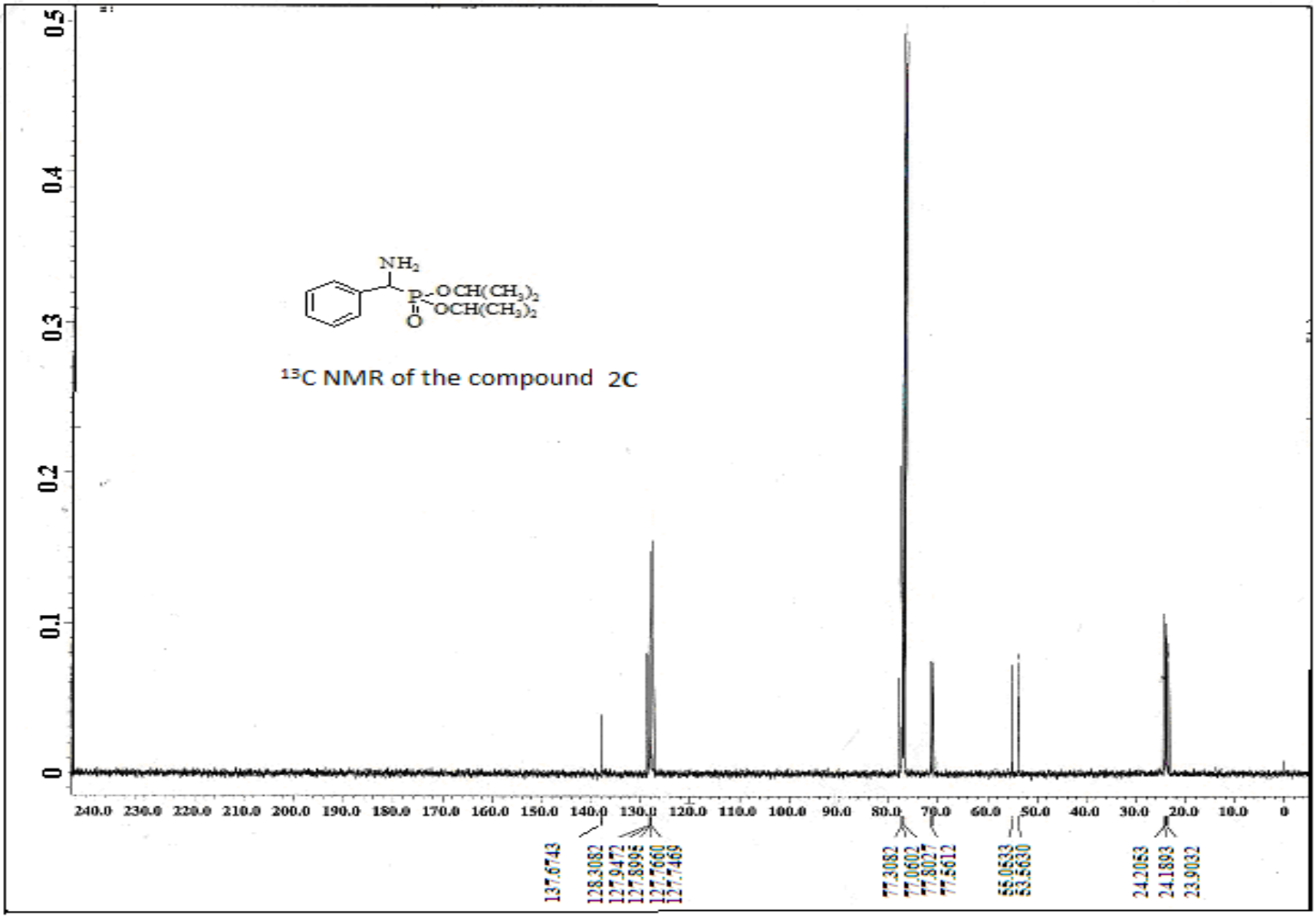

^{1}H NMR of the compound 2b
abundance
7.4587
7.3614
7.3511
4.2391
3.9804
3.9361
3.8489
2.0533
2.0409
1.2384
0.9403
0.9300
0.9151
0.8793
7.3614
7.3574
7.3513
7.3502
4.2601
4.2391
4.0256
3.9804
3.9361
3.8489
1.02
0.86
2.31
0.87
1.85
3.07
0.98
2.94
0.88
1.83
1.96
0.90
1.11
6

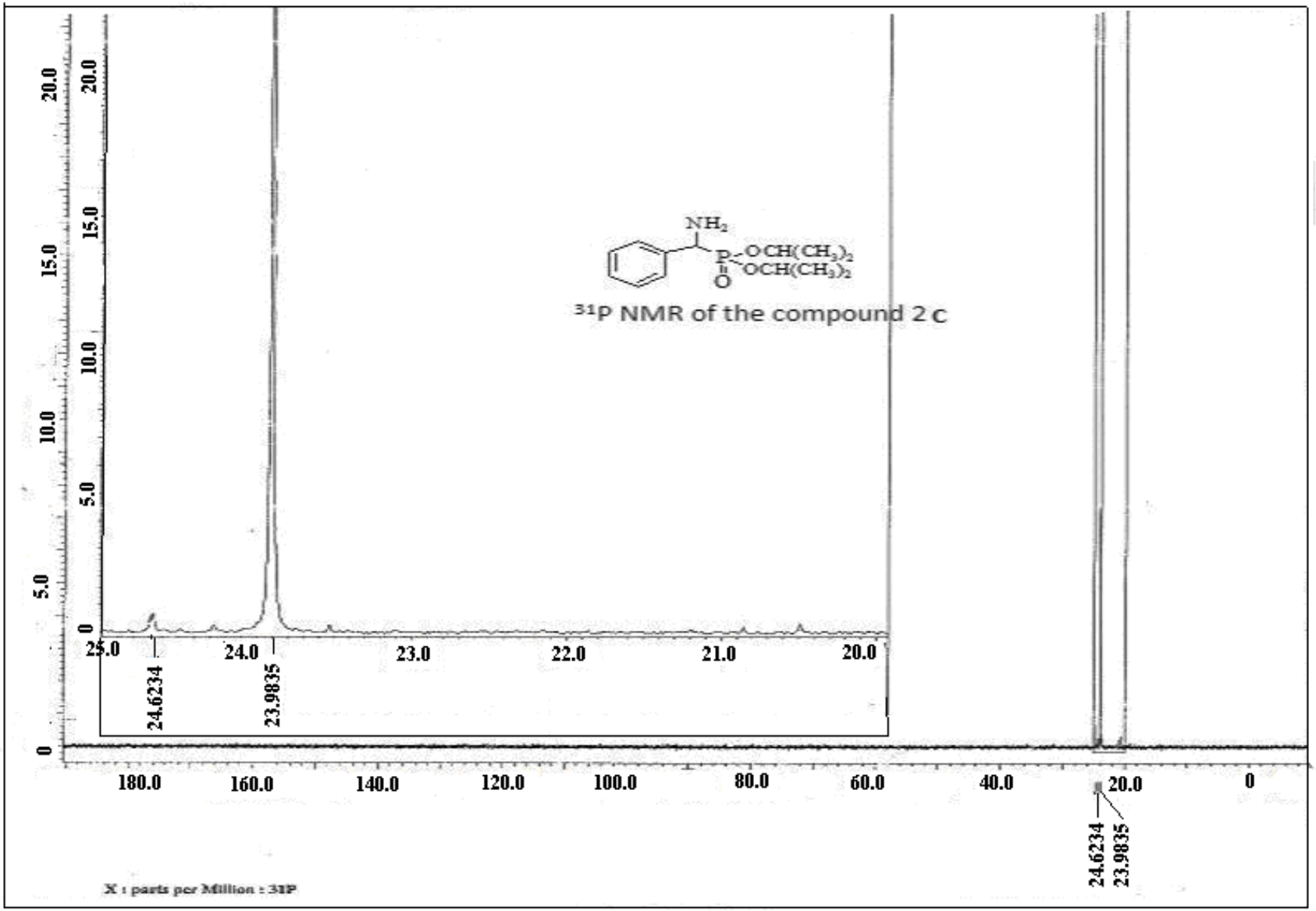

^{13}C NMR of the compound 2b
77.71
77.18
128.32
127.67
137.8658
128.3270
127.6784
77.7166
77.1825
68.0829
60.2803
54.6148
53.4320
20.9445
14.1437
9.9658
9.9001
X : parts per Million : 13C

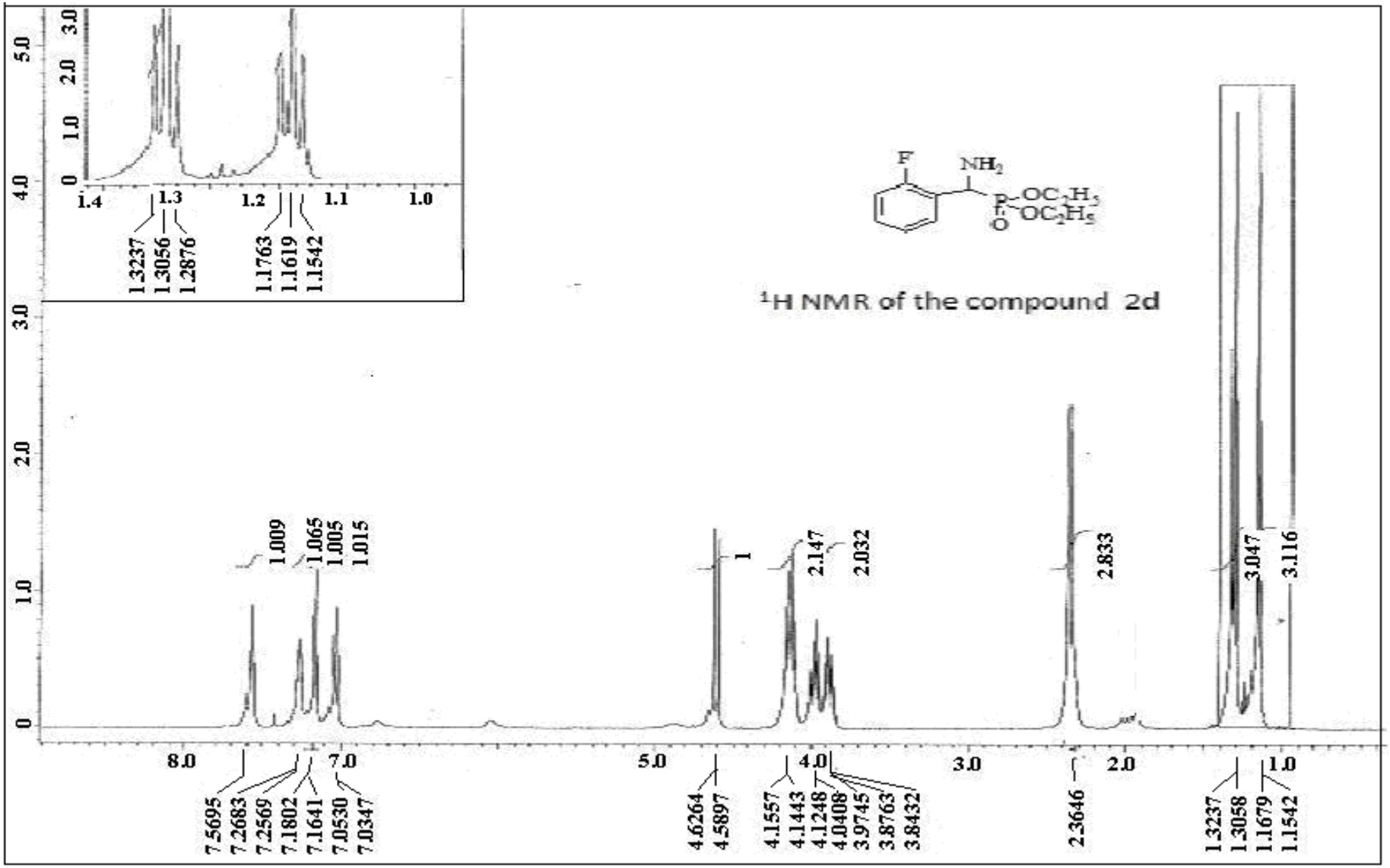


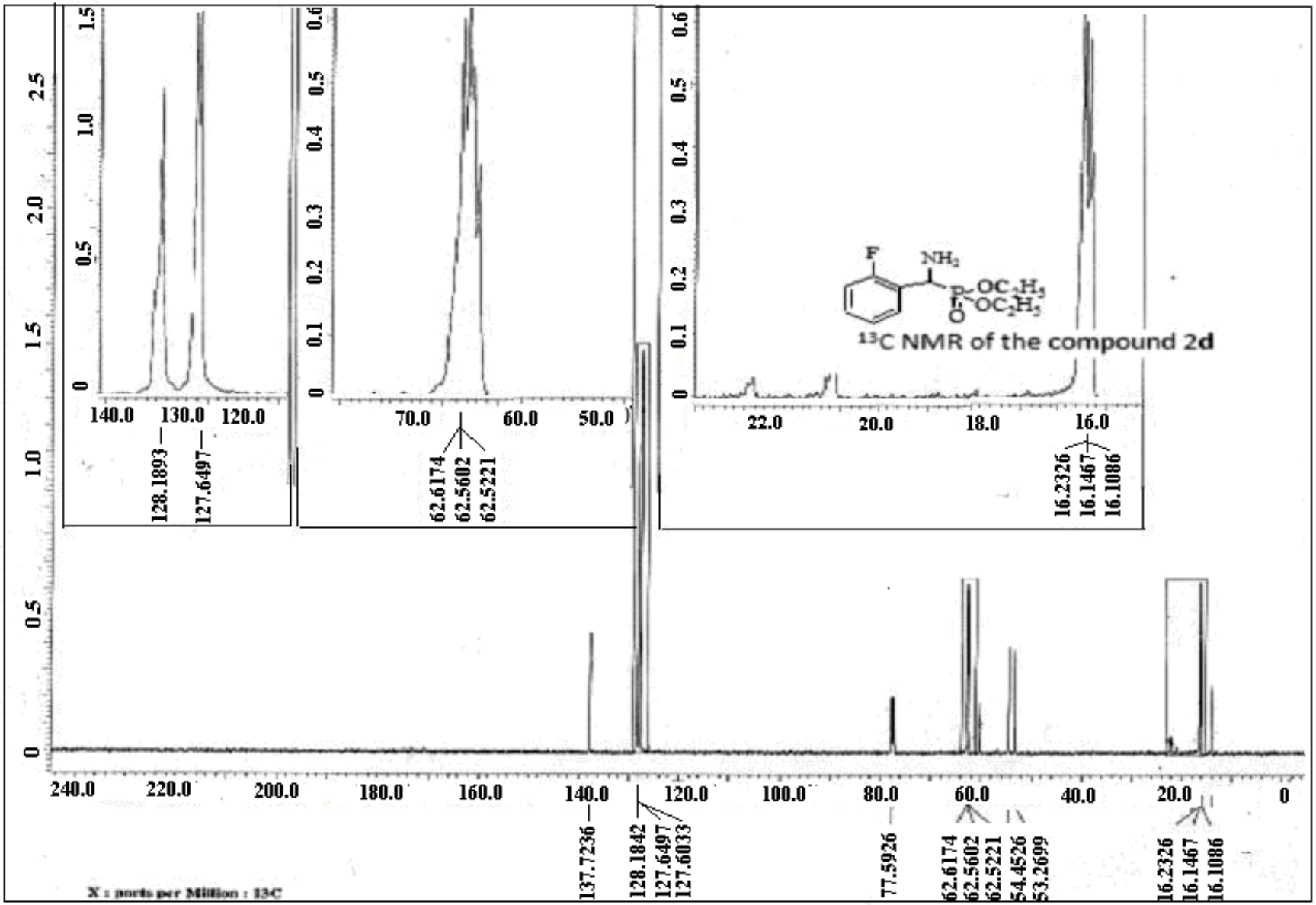
^{31}P NMR of the compound **2b**

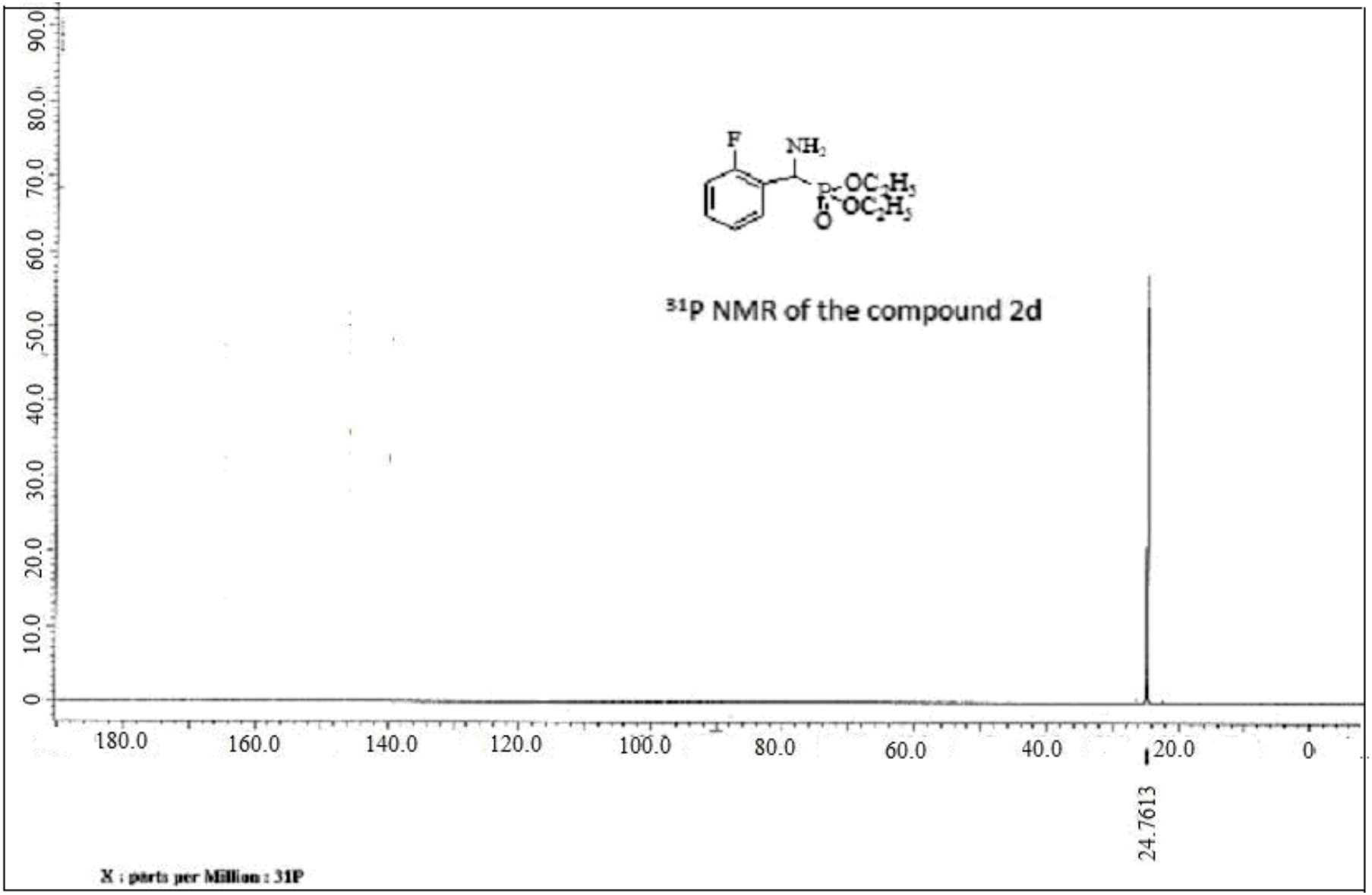

^{1}H NMR of the compound 2C

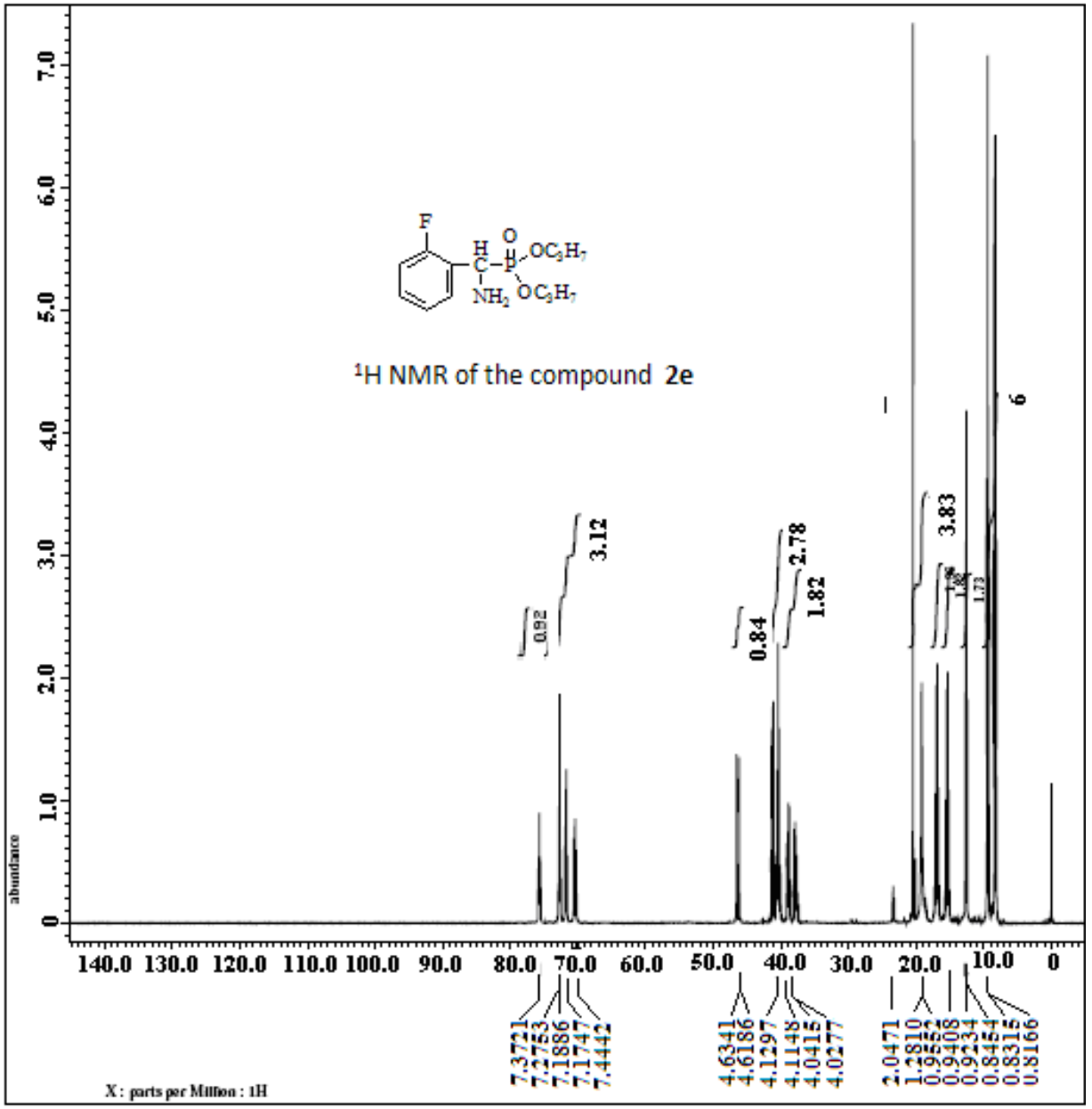

NH2
P
O
OCH(CH3)2
OCH(CH3)2
13C NMR of the compound 2C
0.5
0.4
0.3
0.2
0.1
0
240.0 230.0 220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
137.6743
128.3082
127.9472
127.7660
127.7469
77.3082
77.0602
77.8027
77.5612
55.0533
53.5630
24.2053
24.1893
23.9032

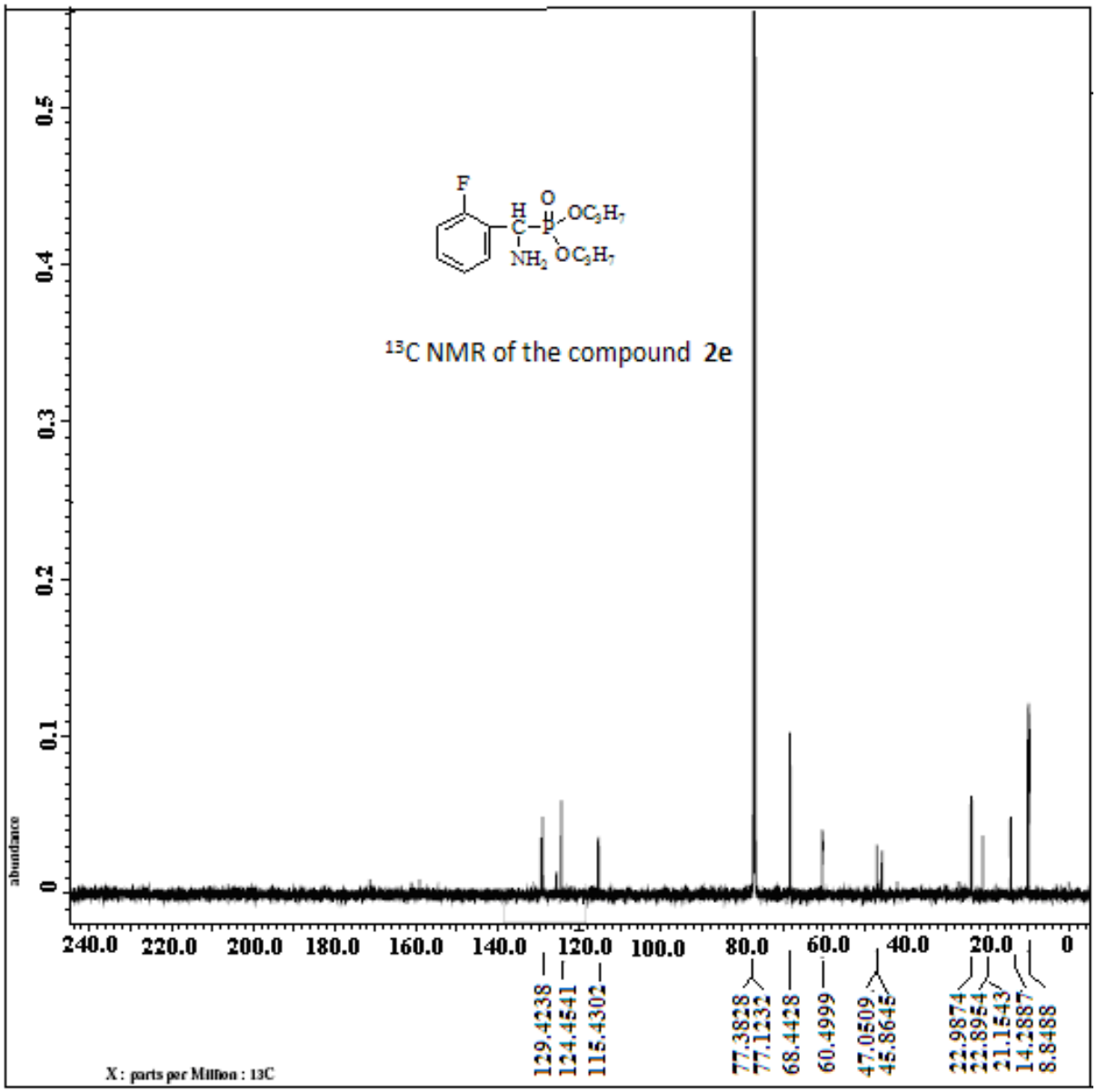

NH2
P
OCH(CH3)2
OCH(CH3)2
O
31P NMR of the compound 2c
24.6234
23.9835
X : parts per Million : 31P

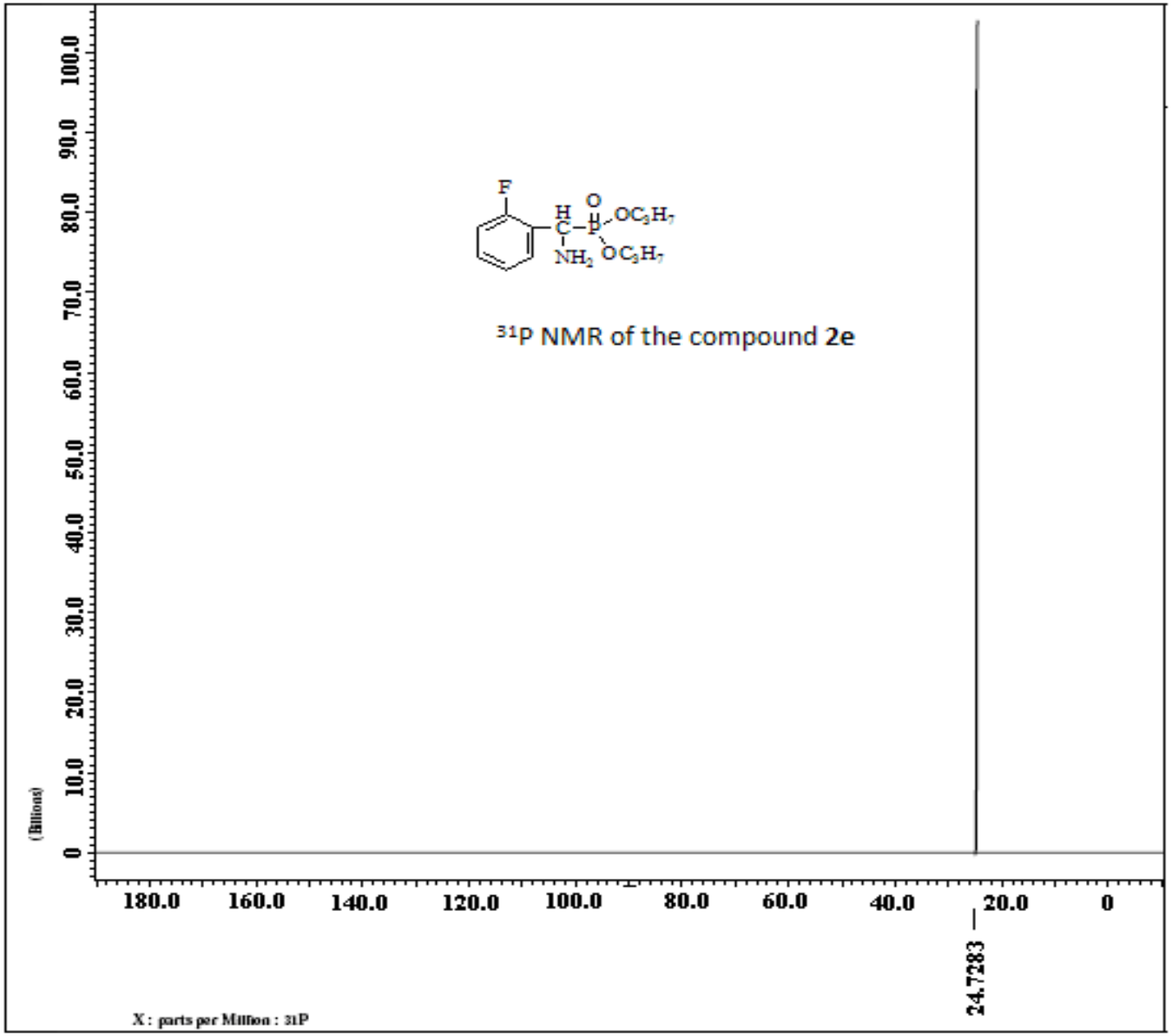

^{1}H NMR of the compound 2d
F
NH2
P
OC2H5
OC2H5
O
1.3237
1.3056
1.2876
1.1763
1.1619
1.1542
1.009
1.065
1.005
1.015
1
2.147
2.032
2.833
3.047
3.116
7.5695
7.2683
7.2569
7.1802
7.1641
7.0530
7.0347
4.6264
4.5897
4.1557
4.1443
4.1248
4.0408
3.9745
3.8763
3.8432
2.3646
1.3237
1.3058
1.1679
1.1542

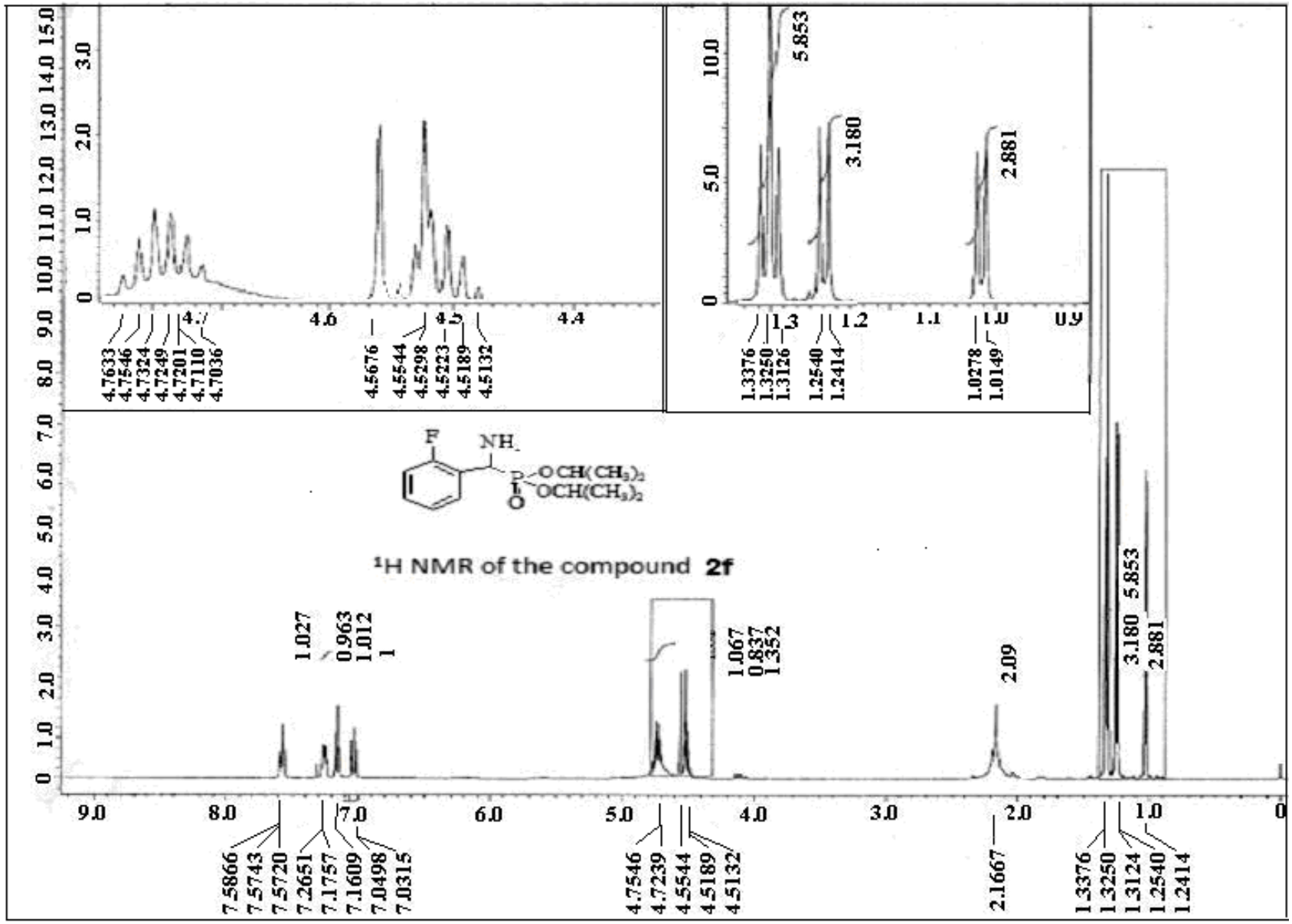

^{13}C NMR of the compound 2d
X : parts per Million : 13C
137.7236
128.1842
127.6497
127.6033
77.5926
62.6174
62.5602
62.5221
54.4526
53.2699
16.2326
16.1467
16.1086
128.1893
127.6497
62.6174
62.5602
62.5221
16.2326
16.1467
16.1086

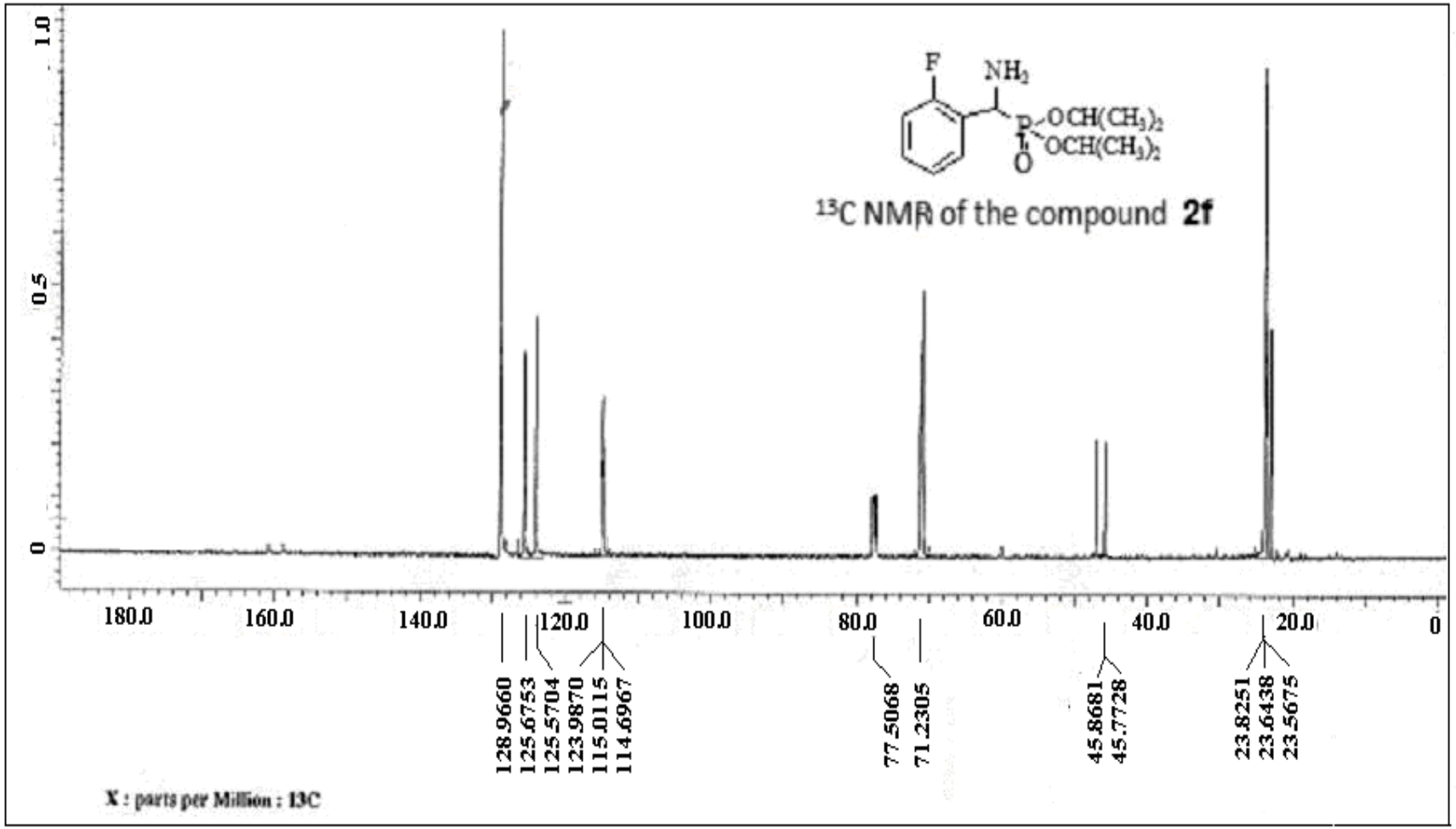

F
NH2
P
OC2H5
OC2H5
O
31P NMR of the compound 2d
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0
24.7613
X : parts per Million : 31P

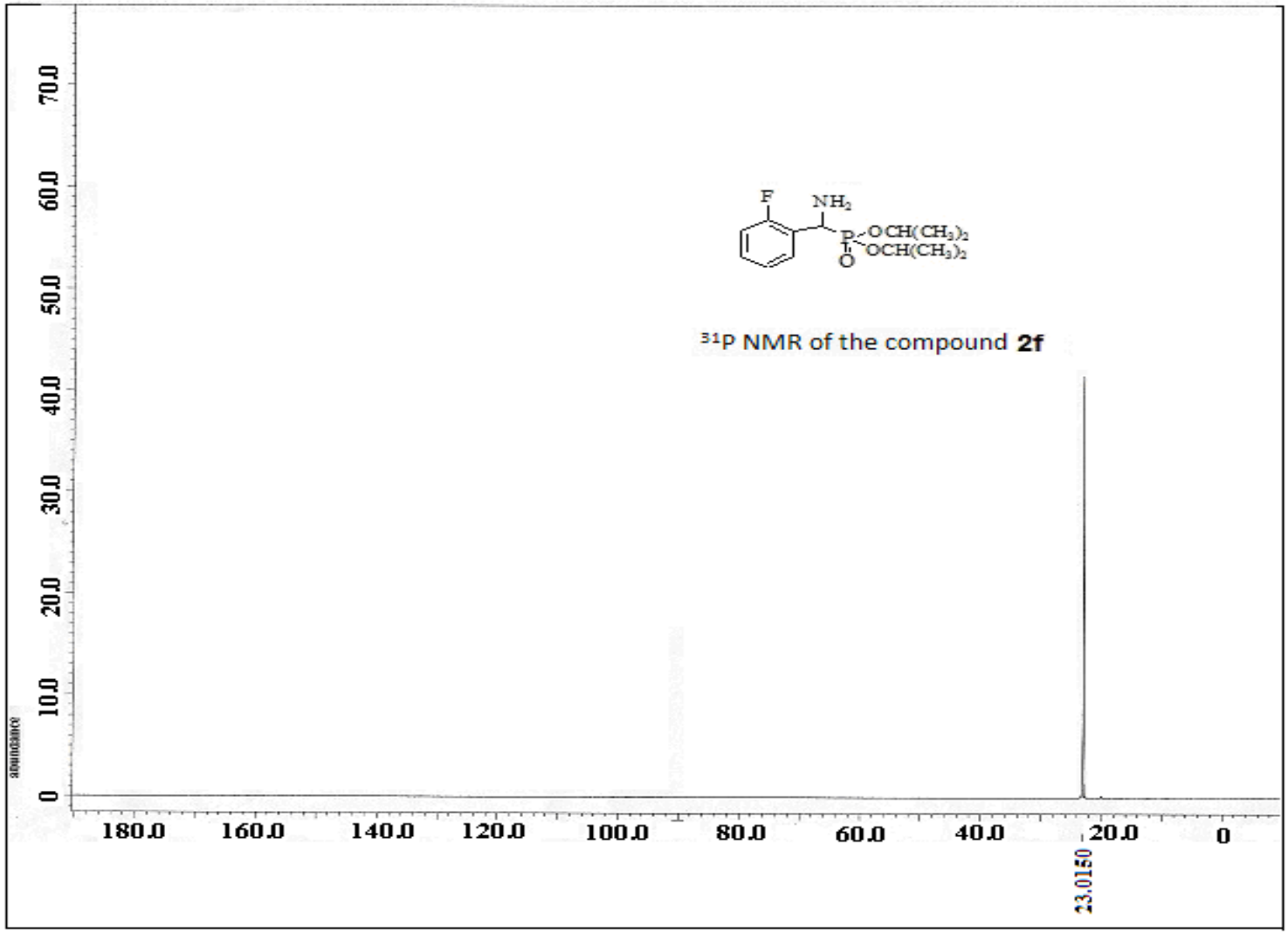

F
H
C
P
O
OC2H7
NH2
OC2H7
1H NMR of the compound 2e
abundance
X : parts per Million : 1H
3.12
0.84
2.78
1.82
3.83
6
7.3721
7.2753
7.1886
7.1747
7.4442
4.6341
4.6186
4.1297
4.1148
4.0415
4.0277
2.0471
1.2810
0.9552
0.9408
0.9234
0.8454
0.8315
0.8166

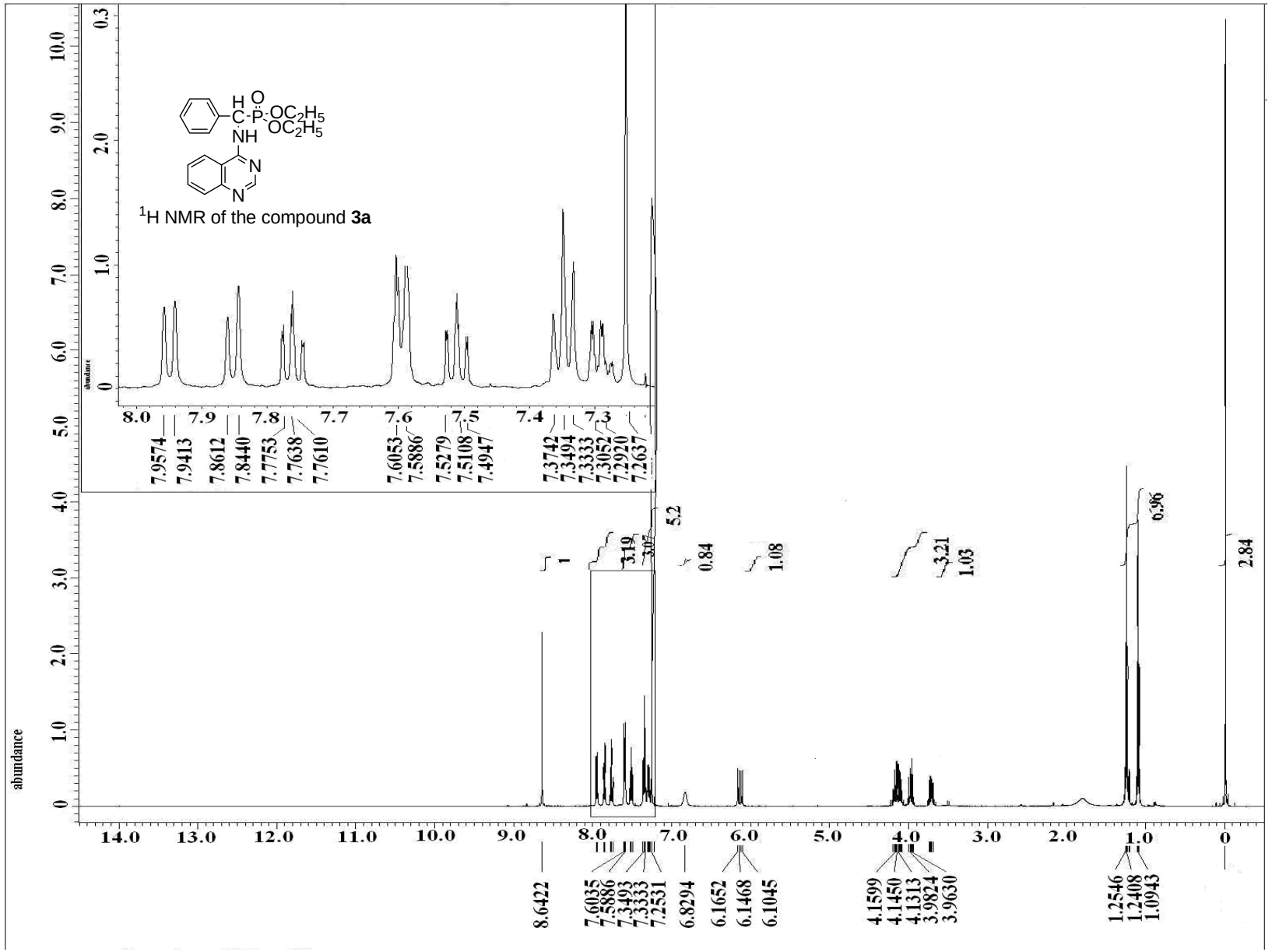

F
H
O
C
P
OC3H7
NH2
OC3H7
13C NMR of the compound 2e
abundance
0
0.1
0.2
0.3
0.4
0.5
240.0
220.0
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0
129.4238
124.4541
115.4302
77.3828
77.1232
68.4428
60.4999
47.0509
45.8645
22.9874
22.8954
21.1543
14.2887
8.8488
X : parts per Million : 13C

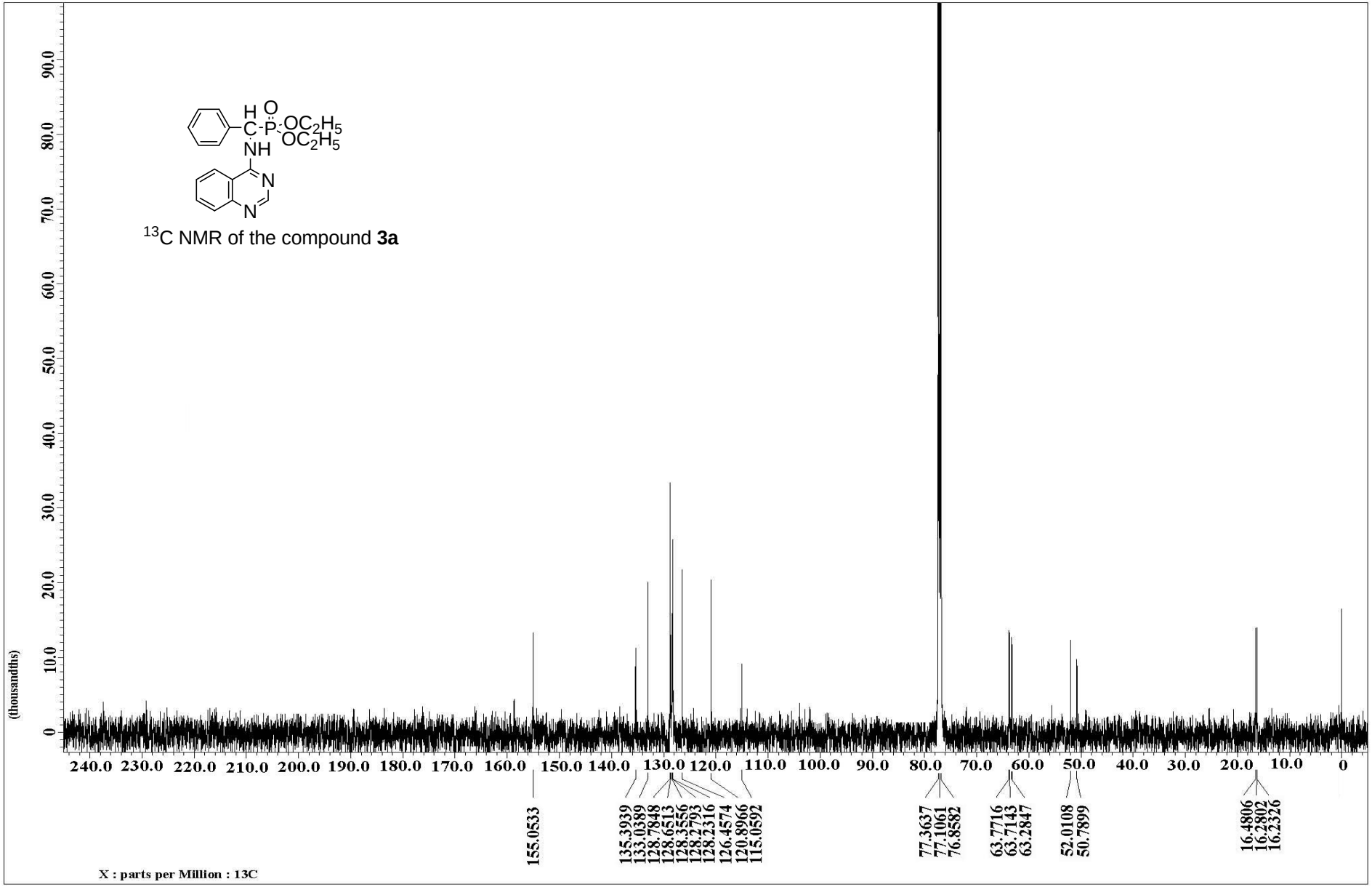

F
H
O
C
P
OC2H7
NH2
OC2H7
31P NMR of the compound 2e
(Billions)
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0
24.7283
X : parts per Million : 31P

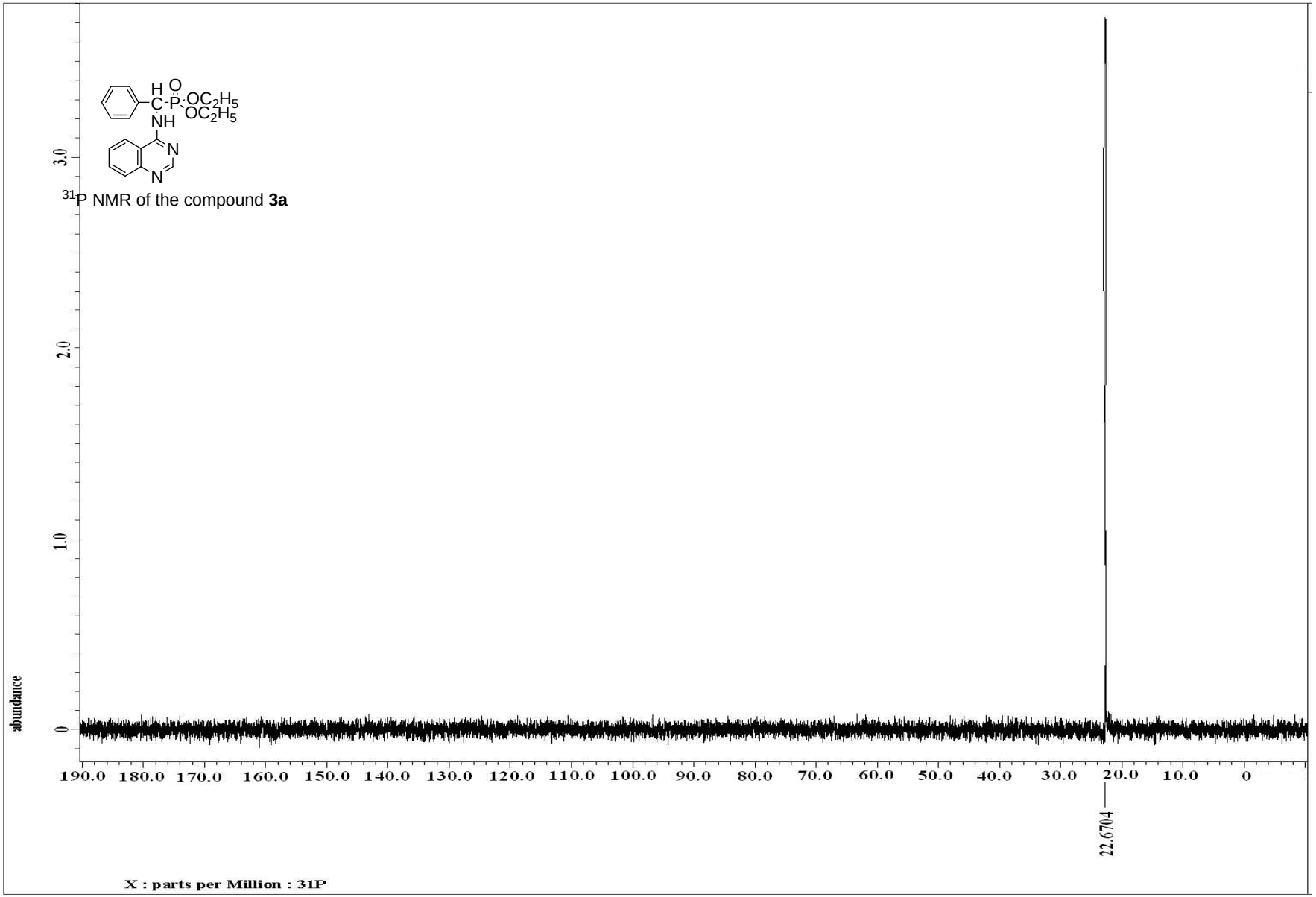

F
NH
OCH(CH3)2
OCH(CH3)2
O
1H NMR of the compound 2f
1.027
0.963
1.012
1
1.067
0.837
1.352
2.09
3.180
5.853
2.881
7.5866
7.5743
7.5720
7.2651
7.1757
7.1609
7.0498
7.0315
4.7546
4.7239
4.5544
4.5189
4.5132
2.1667
1.3376
1.3250
1.3124
1.2540
1.2414
4.7633
4.7546
4.7324
4.7249
4.7201
4.7110
4.7036
4.5676
4.5544
4.5298
4.5223
4.5189
4.5132
1.3376
1.3250
1.3126
1.2540
1.2414
1.0278
1.0149

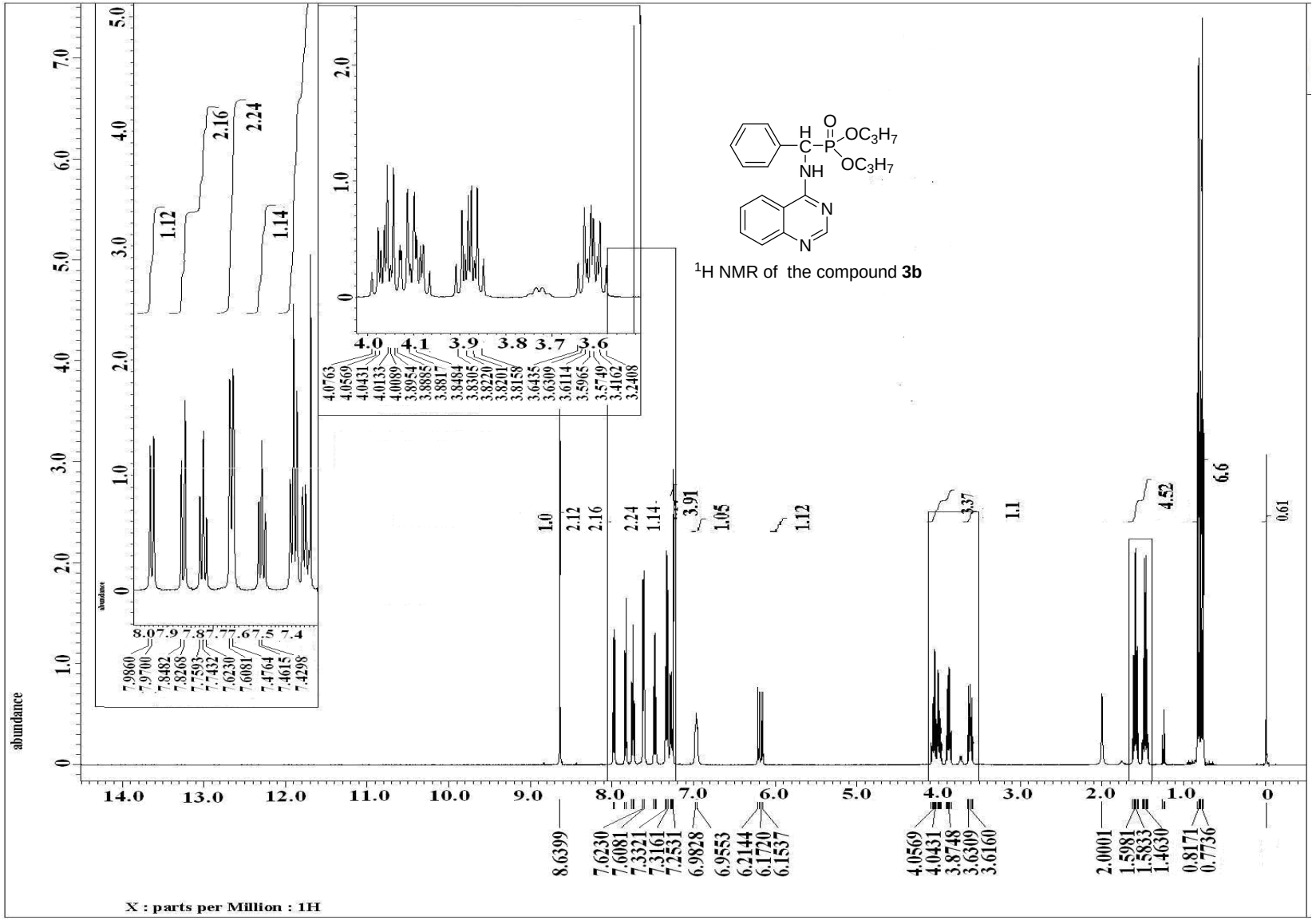

F
NH2
P
OCH(CH3)2
OCH(CH3)2
O
13C NMR of the compound 2f
1.0
0.5
0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0
128.9660
125.6753
125.5704
123.9870
115.0115
114.6967
77.5068
71.2305
45.8681
45.7728
23.8251
23.6438
23.5675
X : parts per Million : 13C


F
NH2
P
O
OCH(CH3)2
OCH(CH3)2
31P NMR of the compound 2f
abundance
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0
23.0150

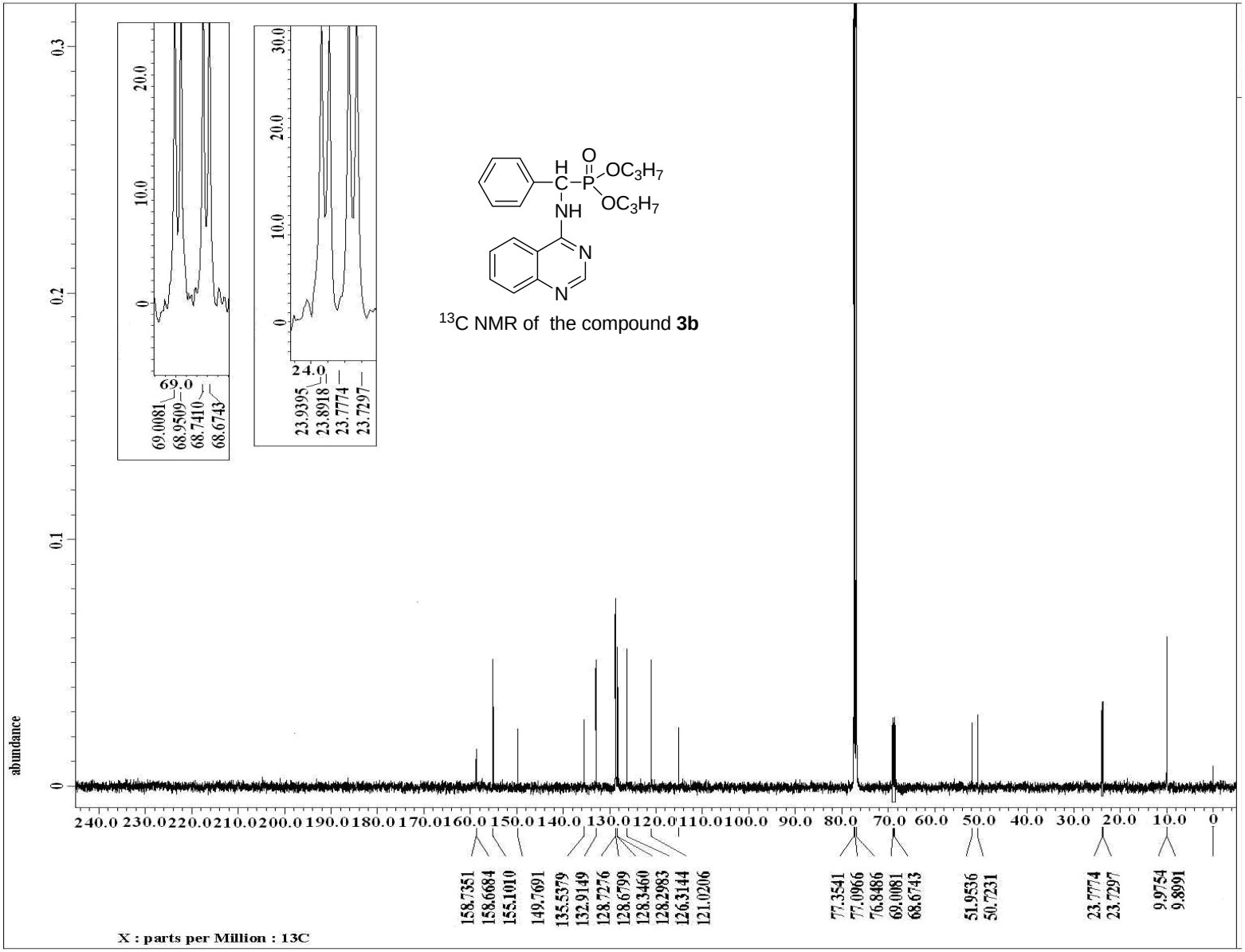

^{1}H NMR of the compound **3a**
abundance
7.9574
7.9413
7.8612
7.8440
7.7753
7.7638
7.7610
7.6053
7.5886
7.5279
7.5108
7.4947
7.3742
7.3494
7.3333
7.3052
7.2920
7.2637
8.6422
7.6035
7.5886
7.3493
7.3333
7.2531
6.8294
6.1652
6.1468
6.1045
4.1599
4.1450
4.1313
3.9824
3.9630
1.2546
1.2408
1.0943
5.2
3.19
3.07
0.84
1.08
3.21
1.03
0.96
2.84


^{13}C NMR of the compound **3a**
(thousandths)
X : parts per Million : 13C
155.0533
135.3939
133.0389
128.7848
128.6513
128.3556
128.2793
128.2316
126.4574
120.8966
115.0592
77.3637
77.1061
76.8582
63.7716
63.7143
63.2847
52.0108
50.7899
16.4806
16.2802
16.2326

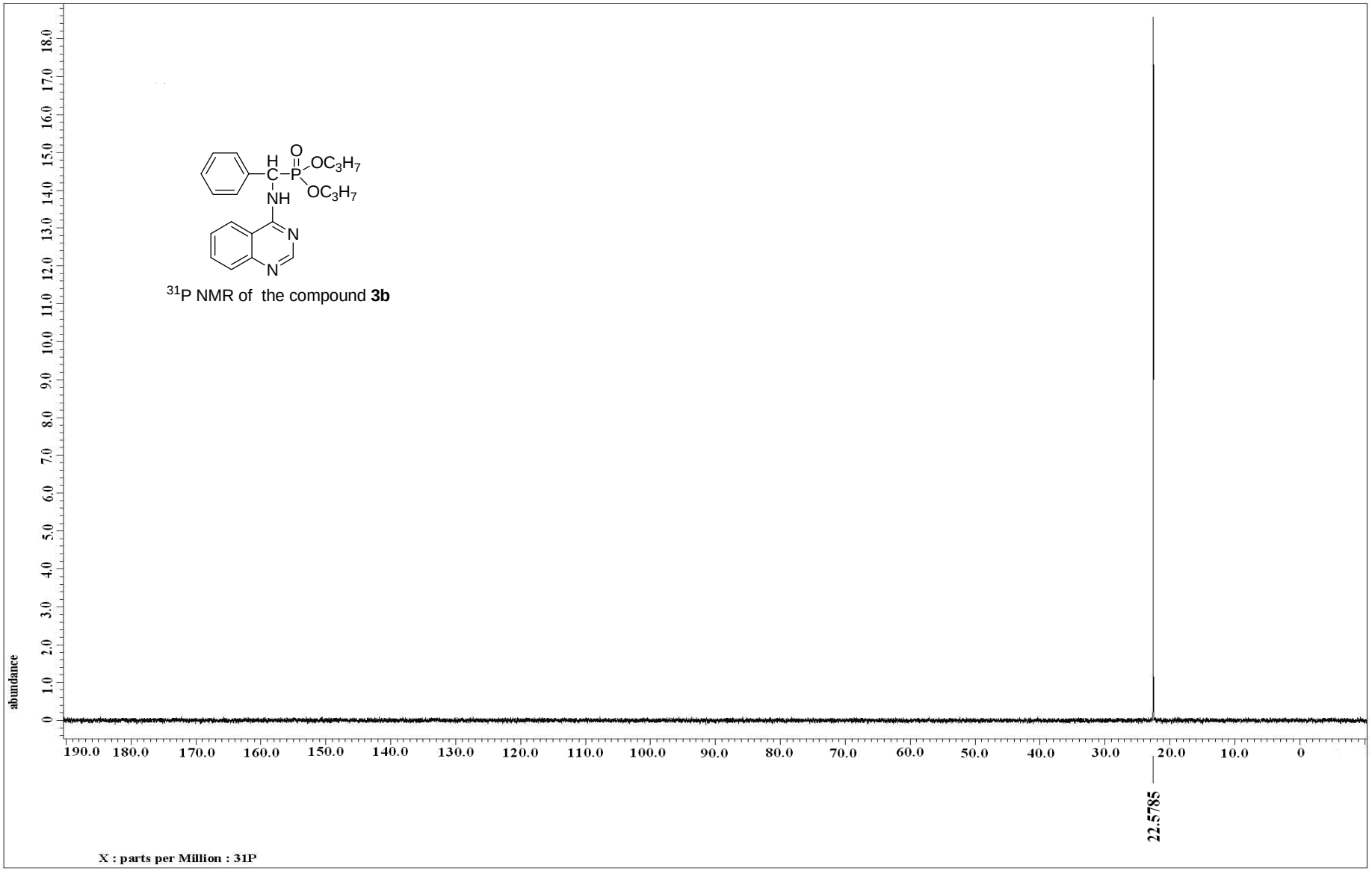

H
O
C
P
OC2H5
OC2H5
NH
N
N
31P NMR of the compound 3a
abundance
3.0
2.0
1.0
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
22.6704
X : parts per Million : 31P



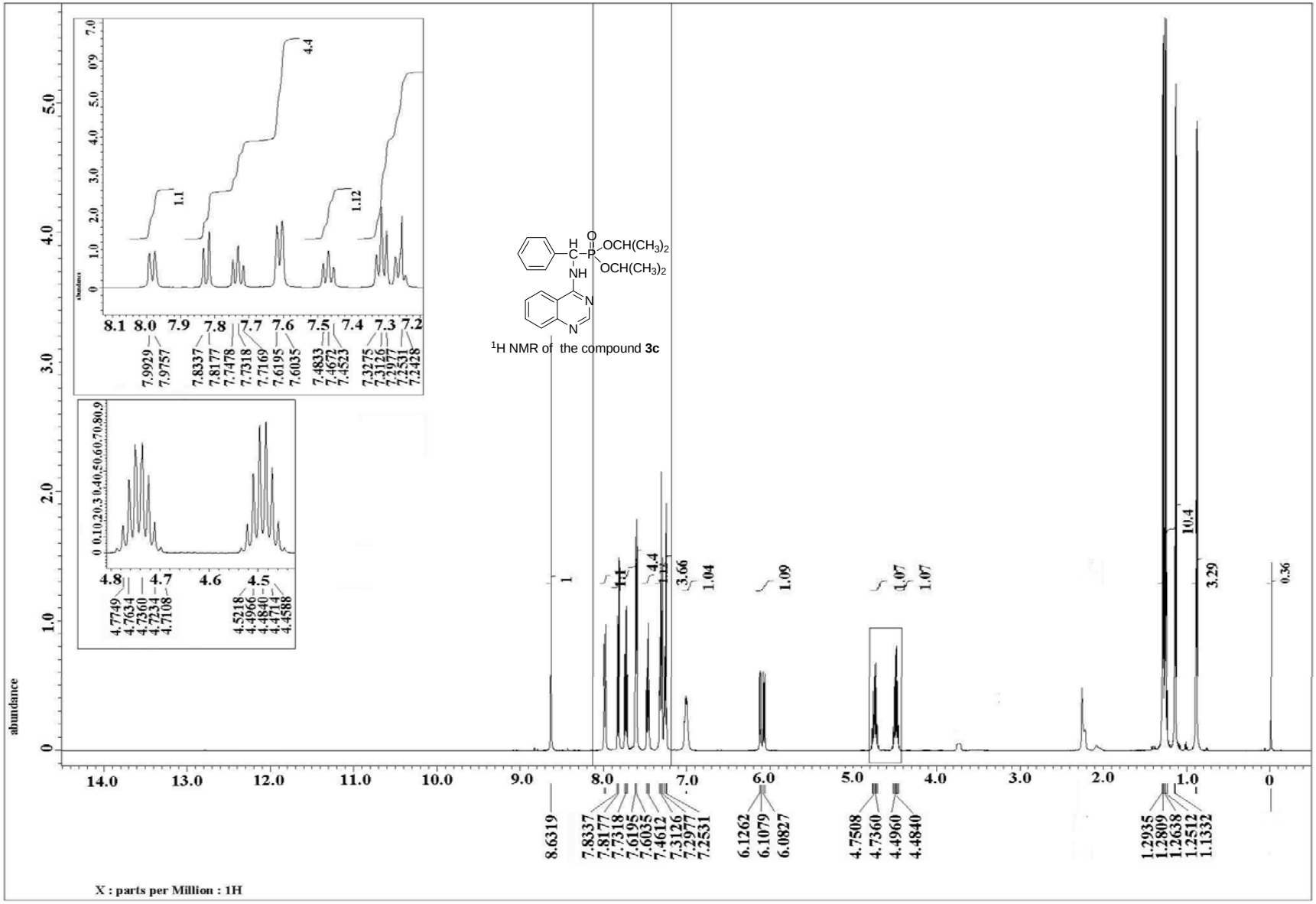
^{1}H NMR of the compound **3b**

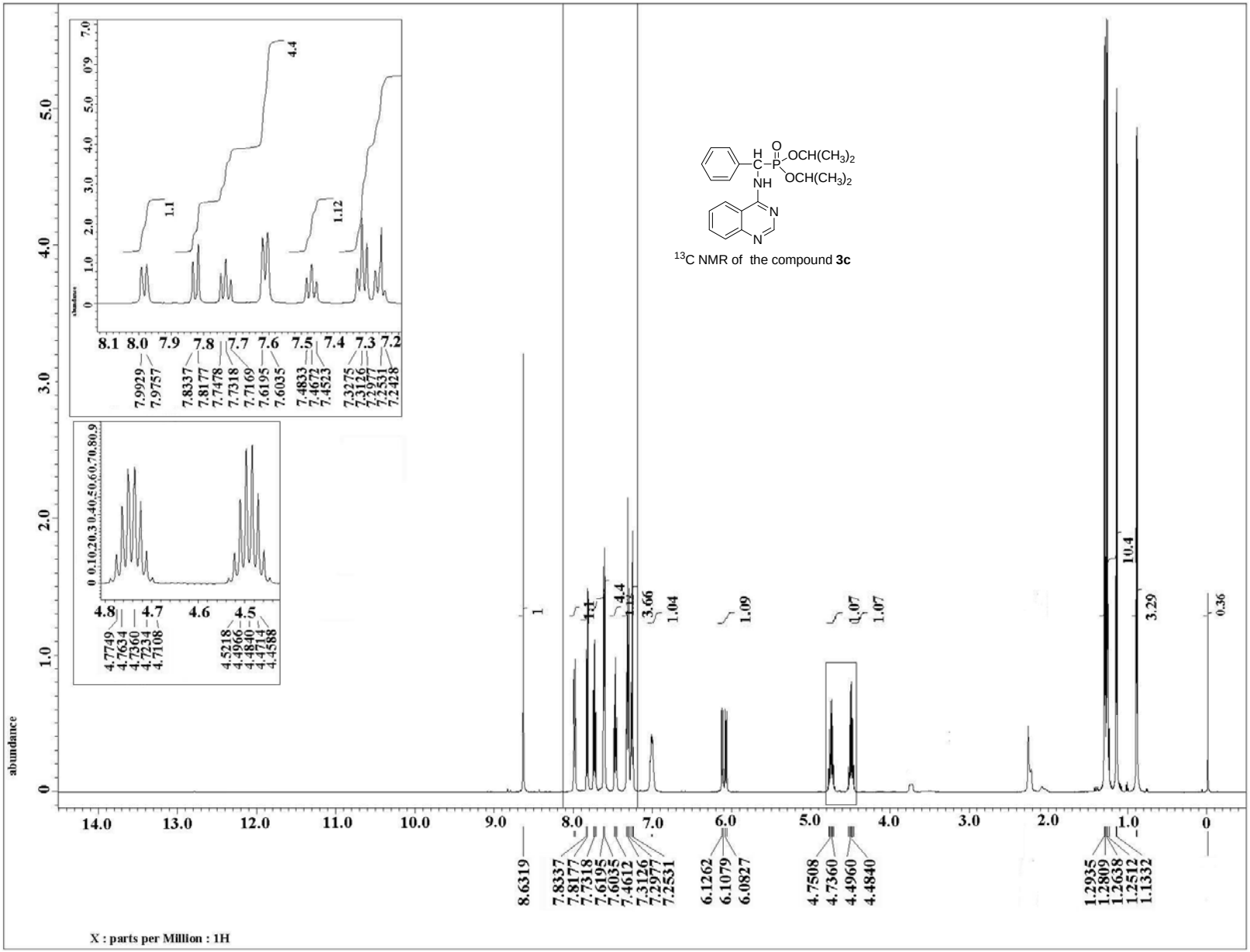


^{13}C NMR of the compound **3b**

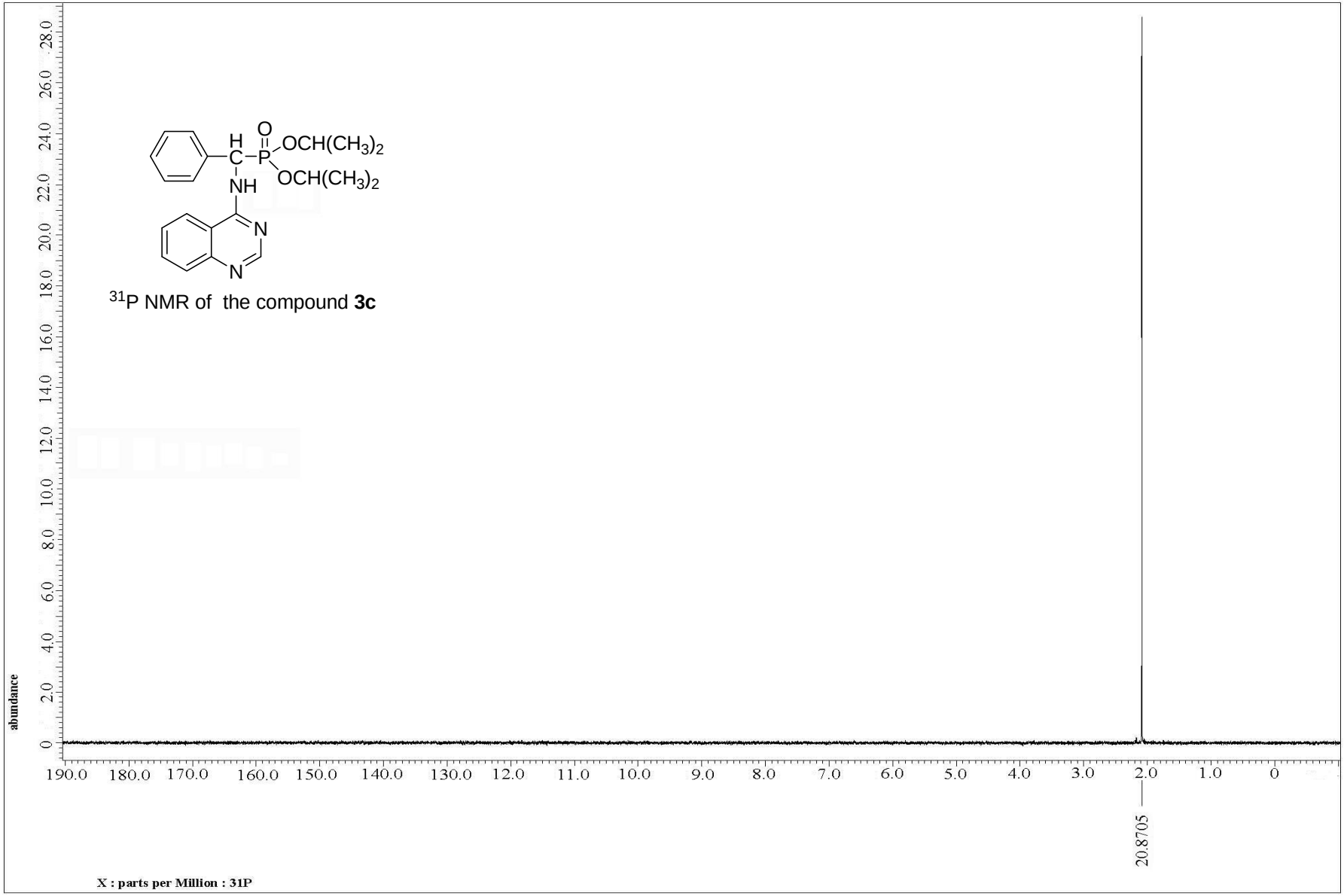


^{31}P NMR of the compound **3b**

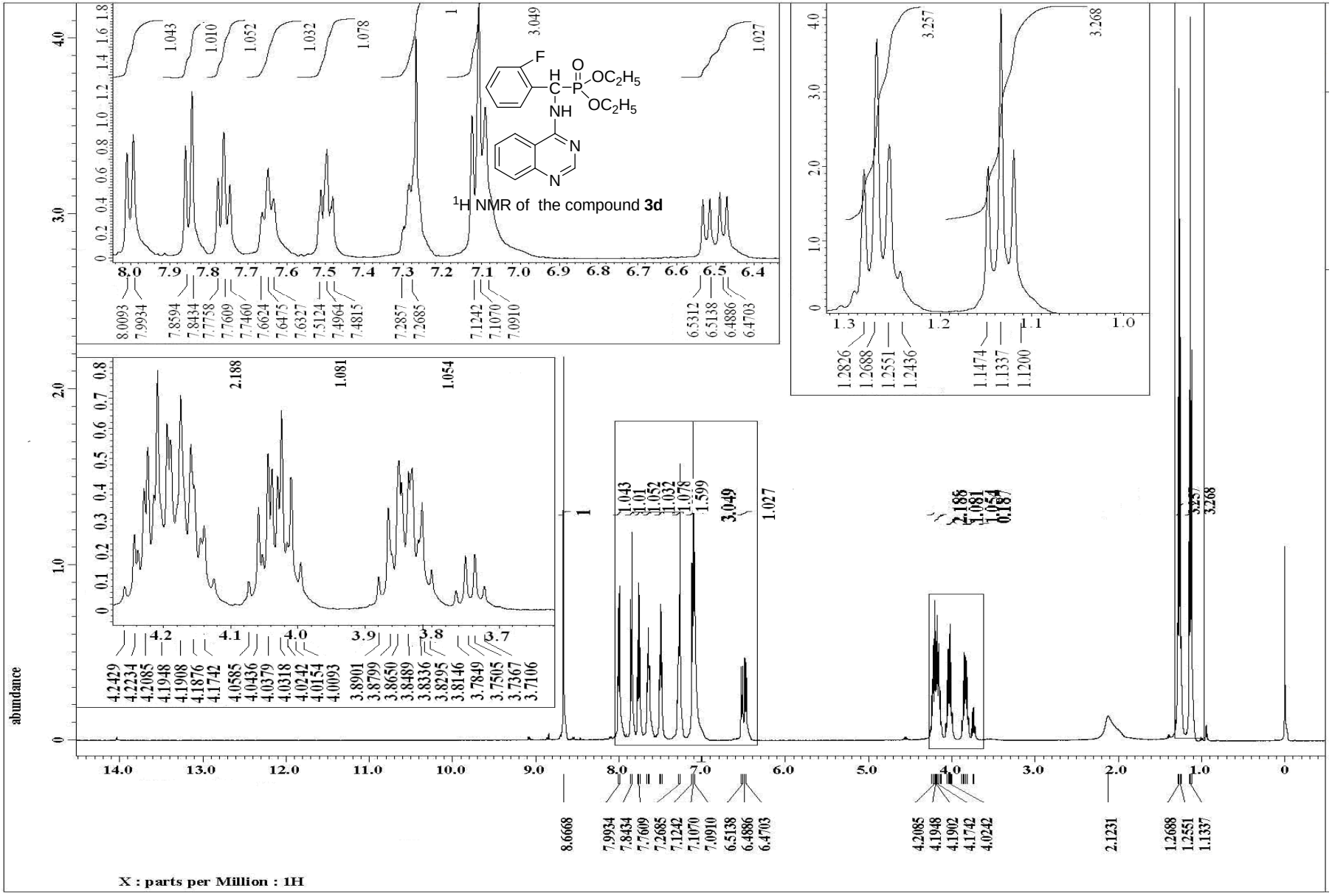

OCH(CH3)2
OCH(CH3)2
NH
N
N
1H NMR of the compound 3c
abundance
X : parts per Million : 1H

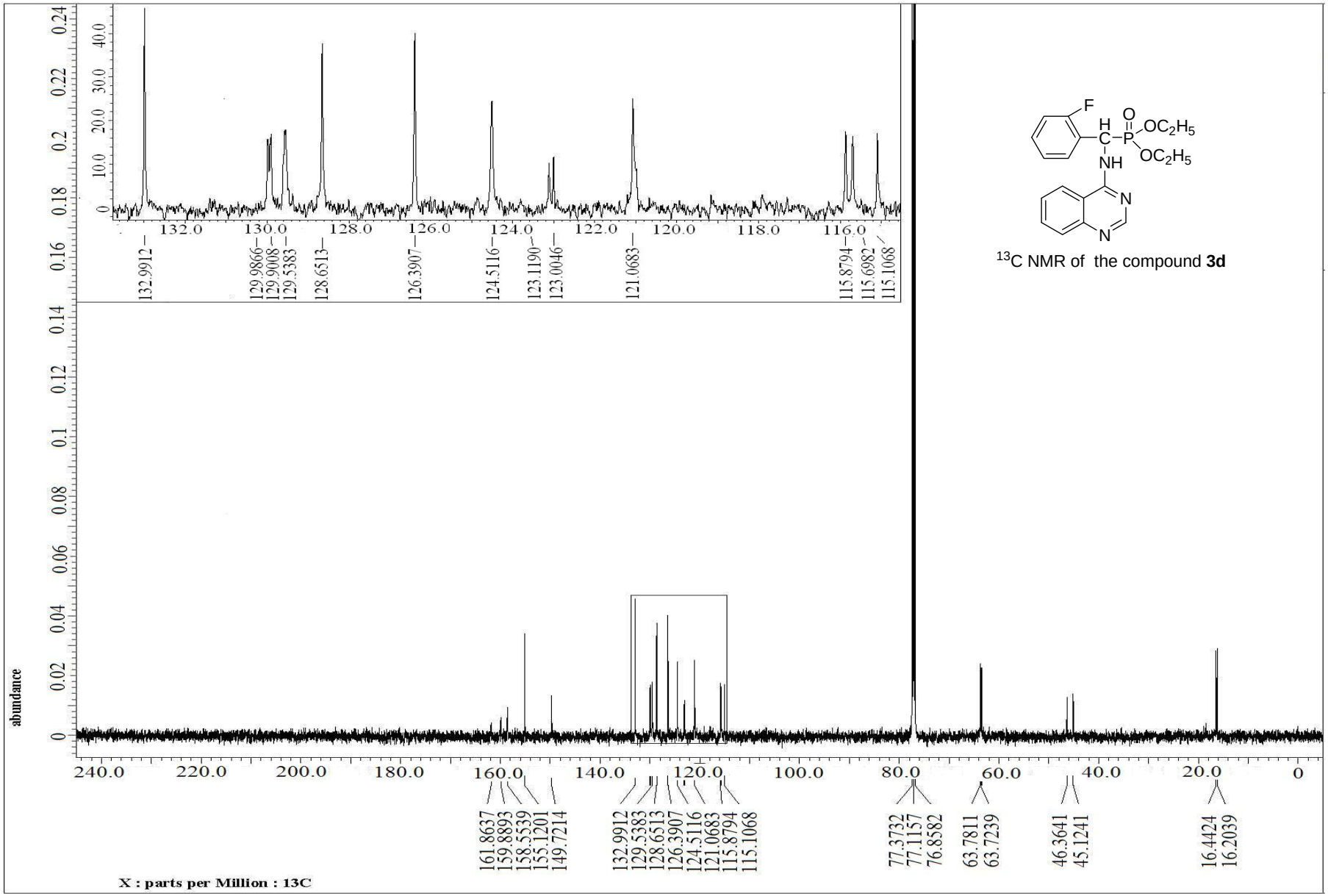


^{13}C NMR of the compound **3c**

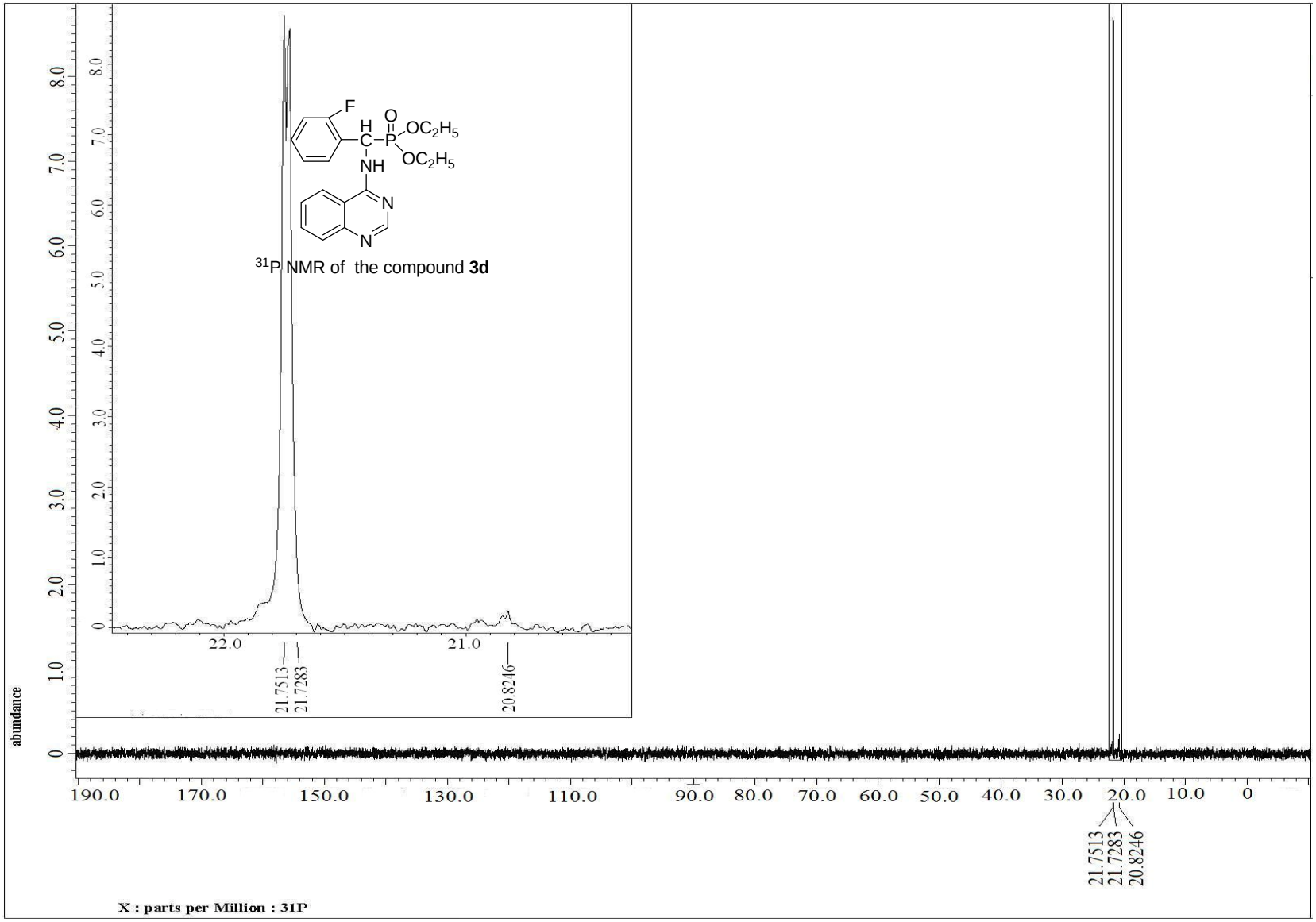


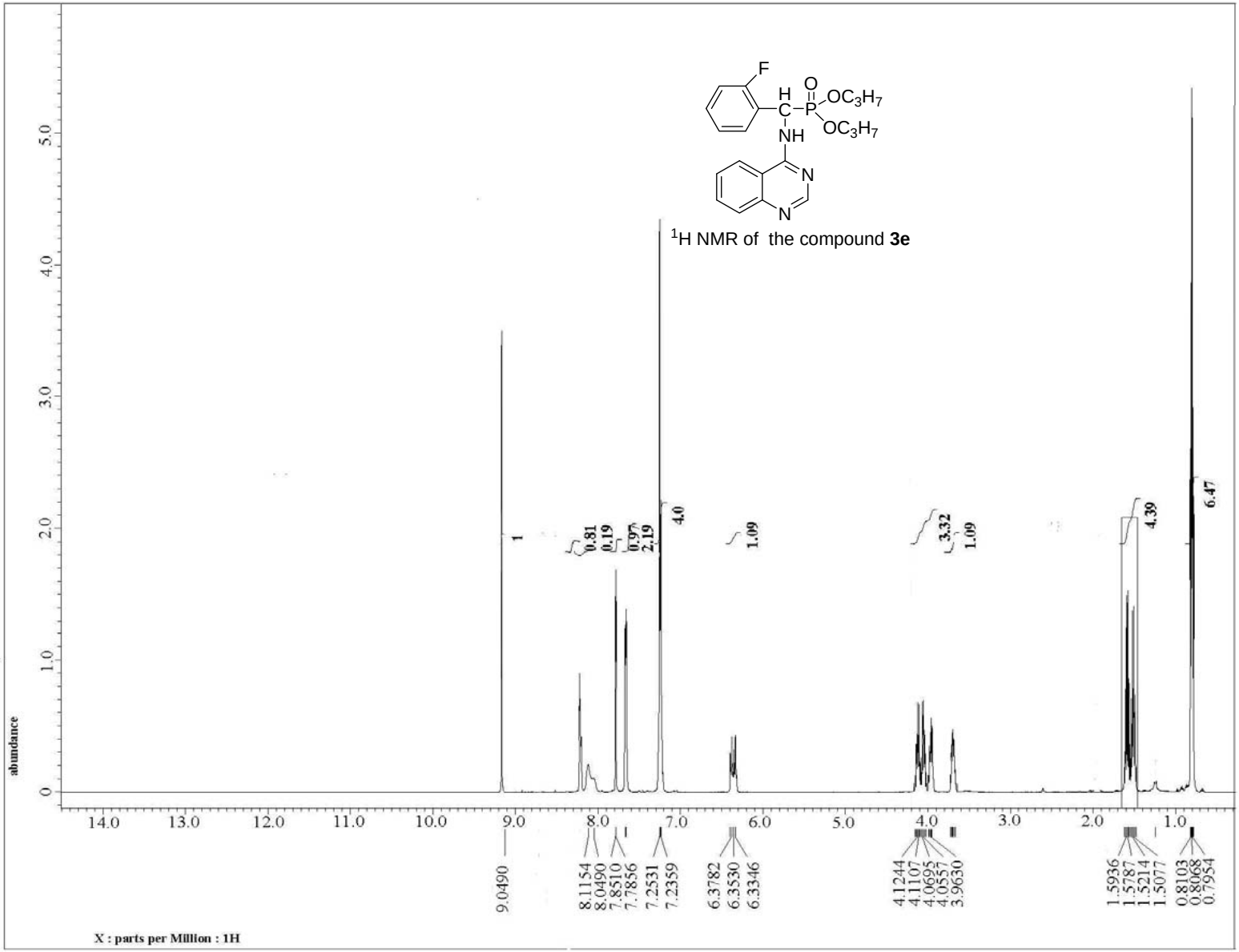
^{31}P NMR of the compound **3c**


abundance
X : parts per Million : 1H
1H NMR of the compound 3d
F
H
C
P
O
OC2H5
OC2H5
NH
N
N

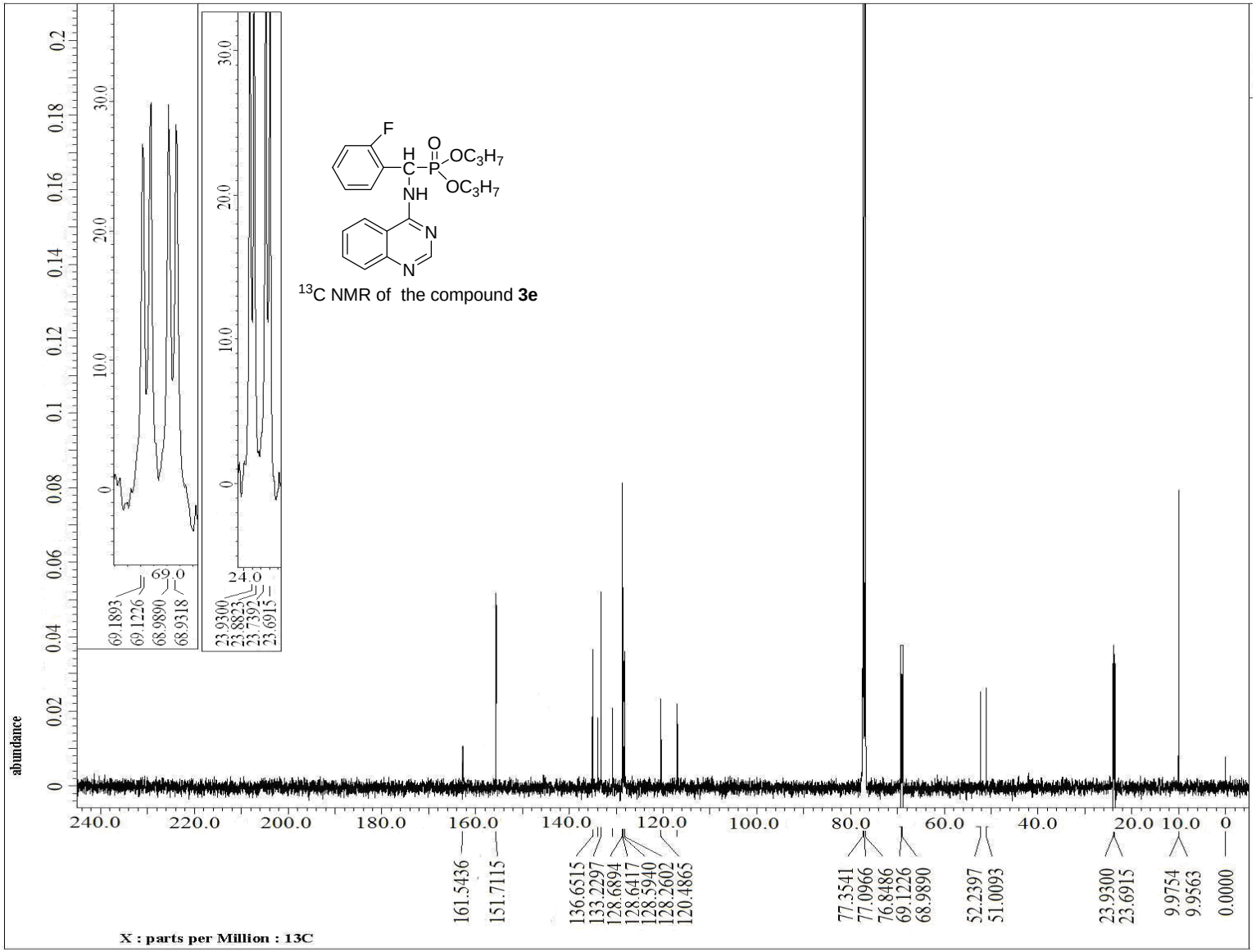


^{13}C NMR of the compound **3d**

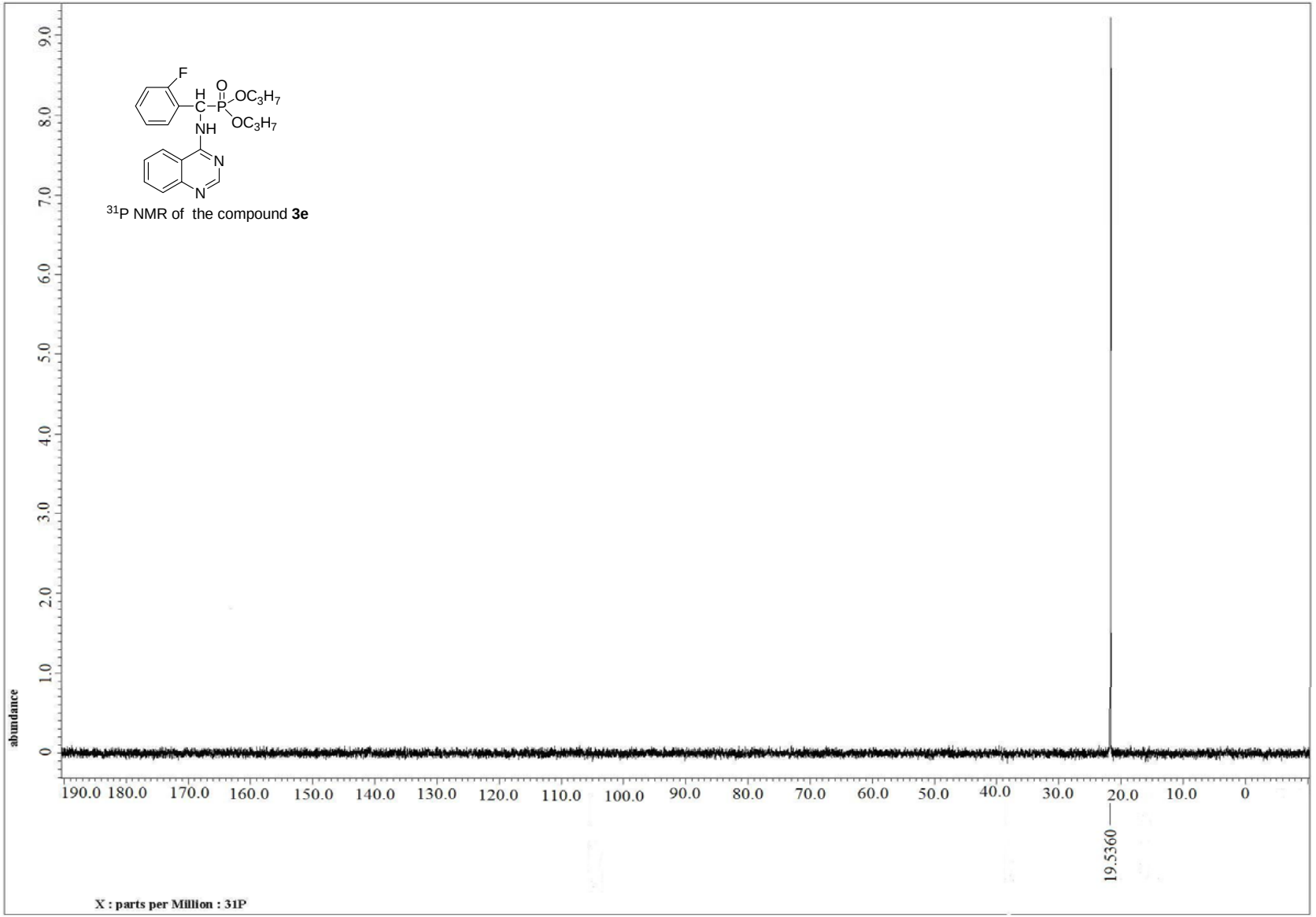

F
H
C
P
O
OC_2H_5
OC_2H_5
NH
N
N
^{31}P NMR of the compound **3d**
abundance
21.7513
21.7283
20.8246
X : parts per Million : 31P

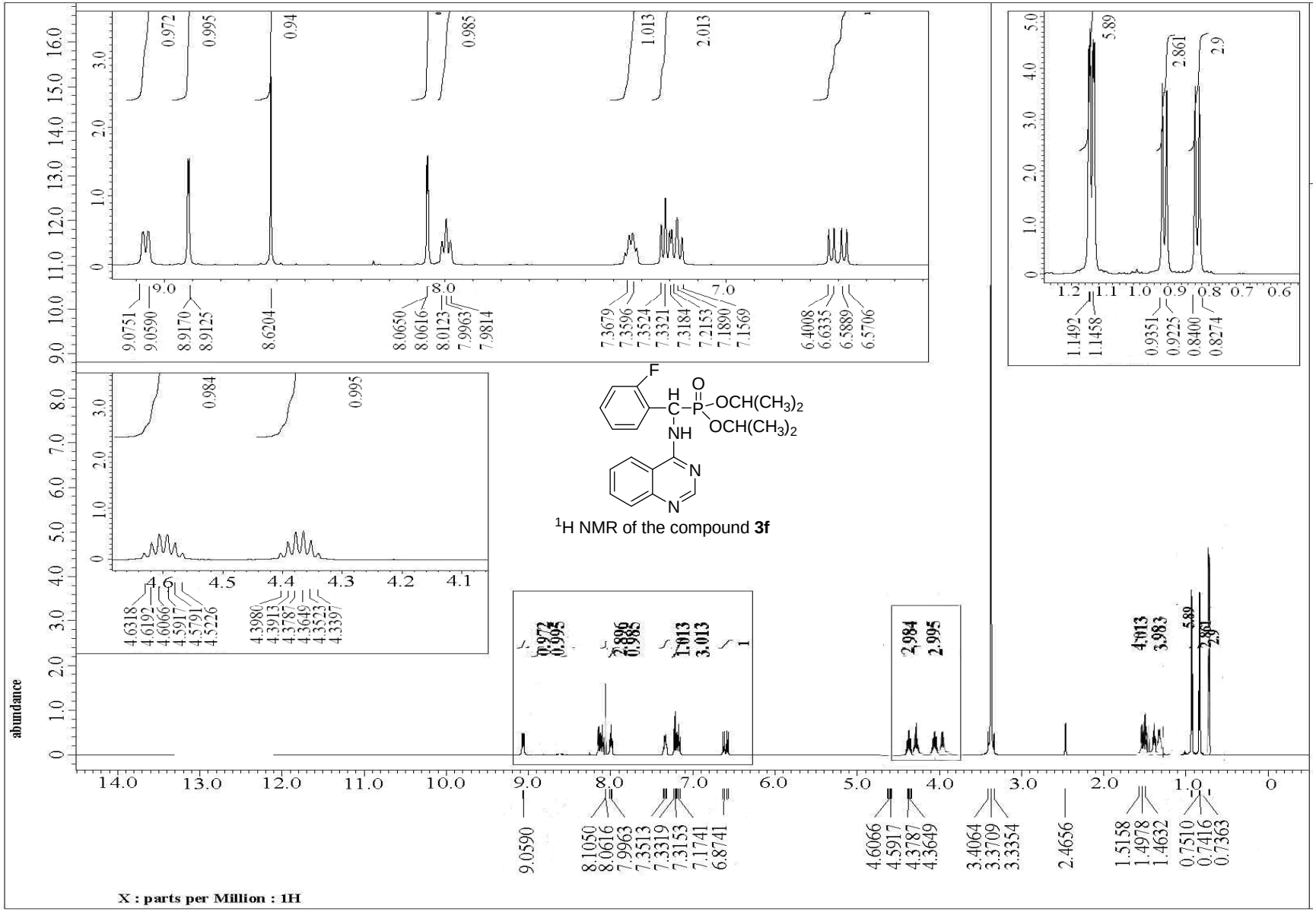


^{1}H NMR of the compound **3e**

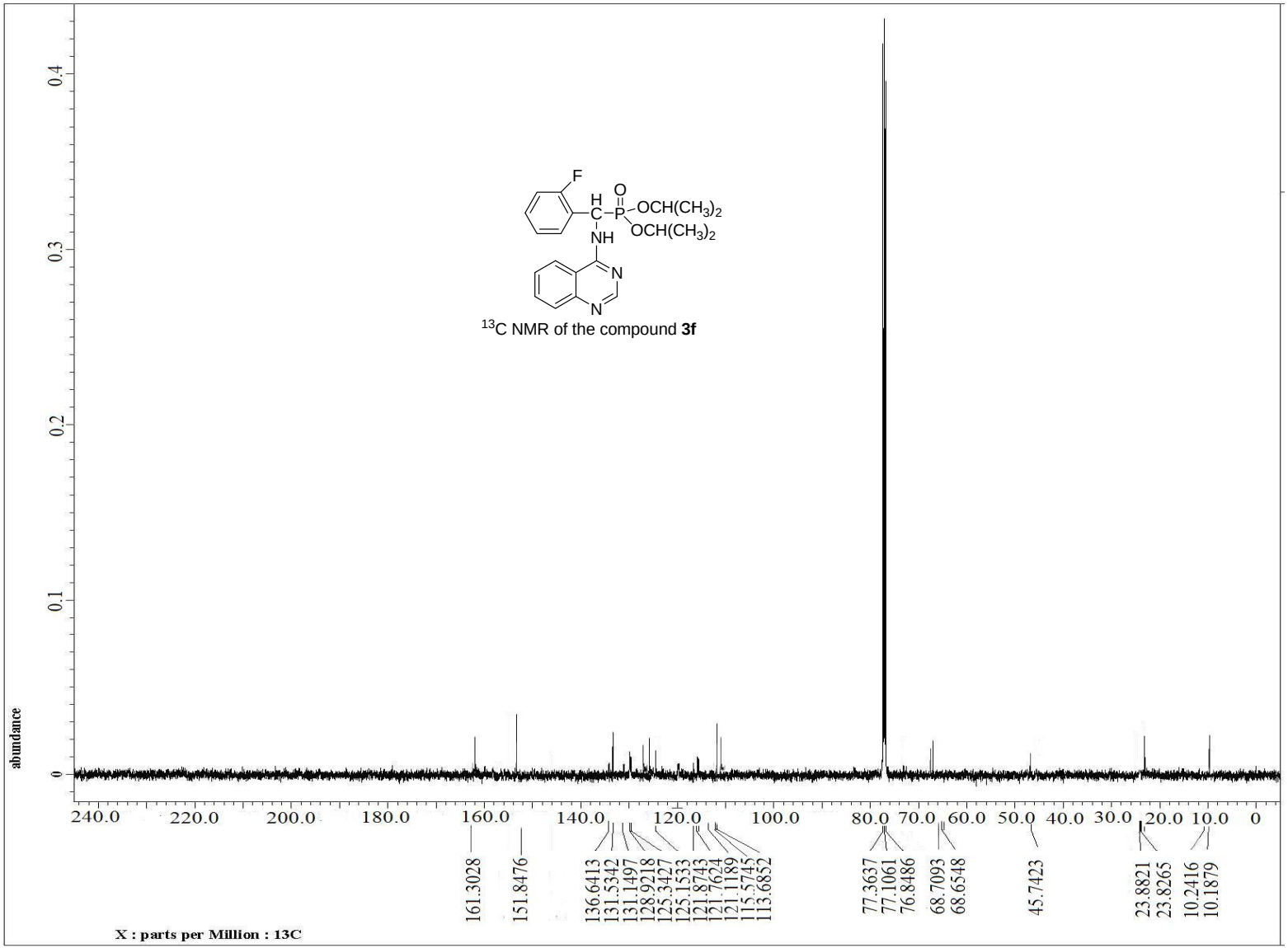


^{13}C NMR of the compound **3e**

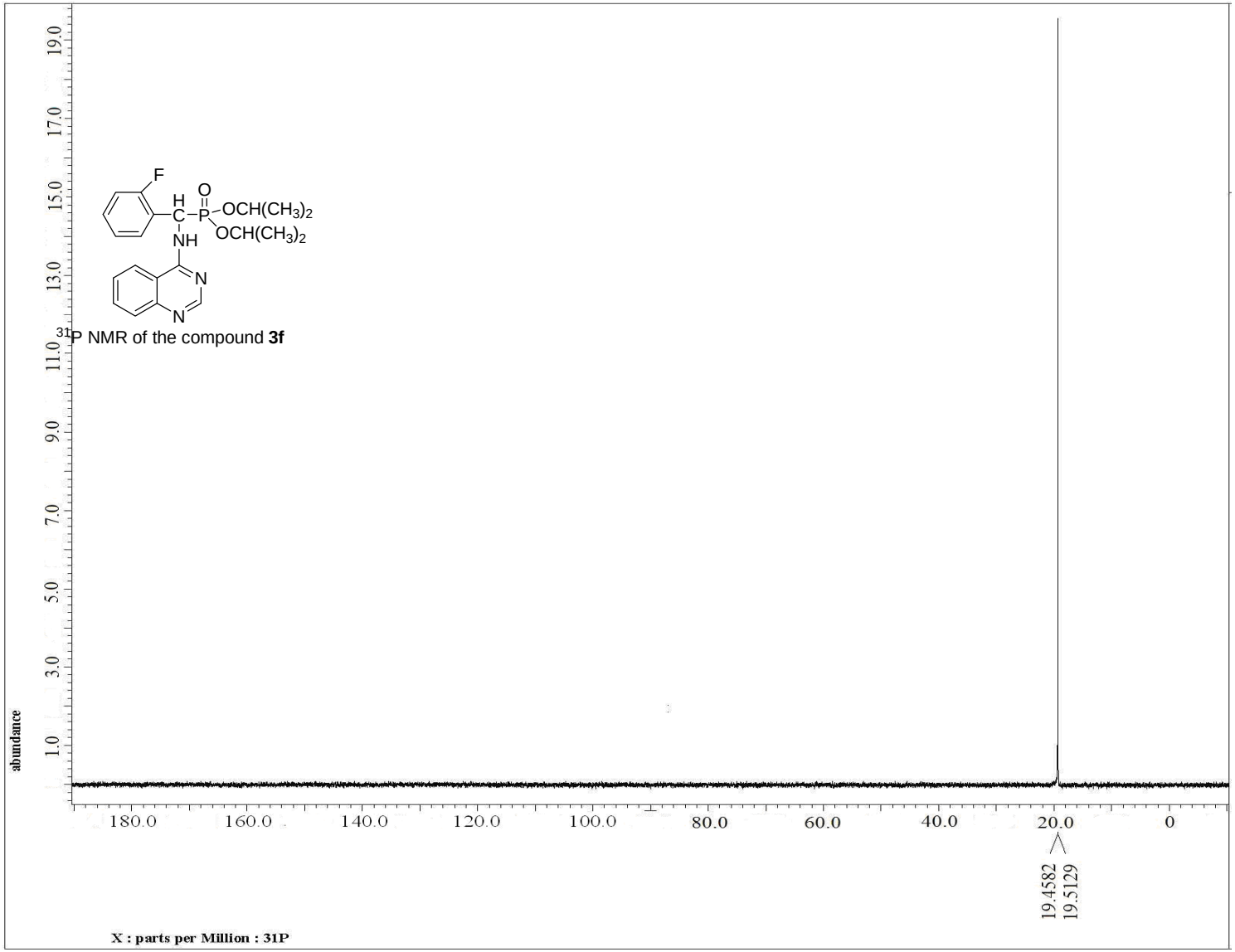

F
H
O
C
P
OC3H7
OC3H7
NH
N
N
31P NMR of the compound 3e
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
19.5360
X : parts per Million : 31P



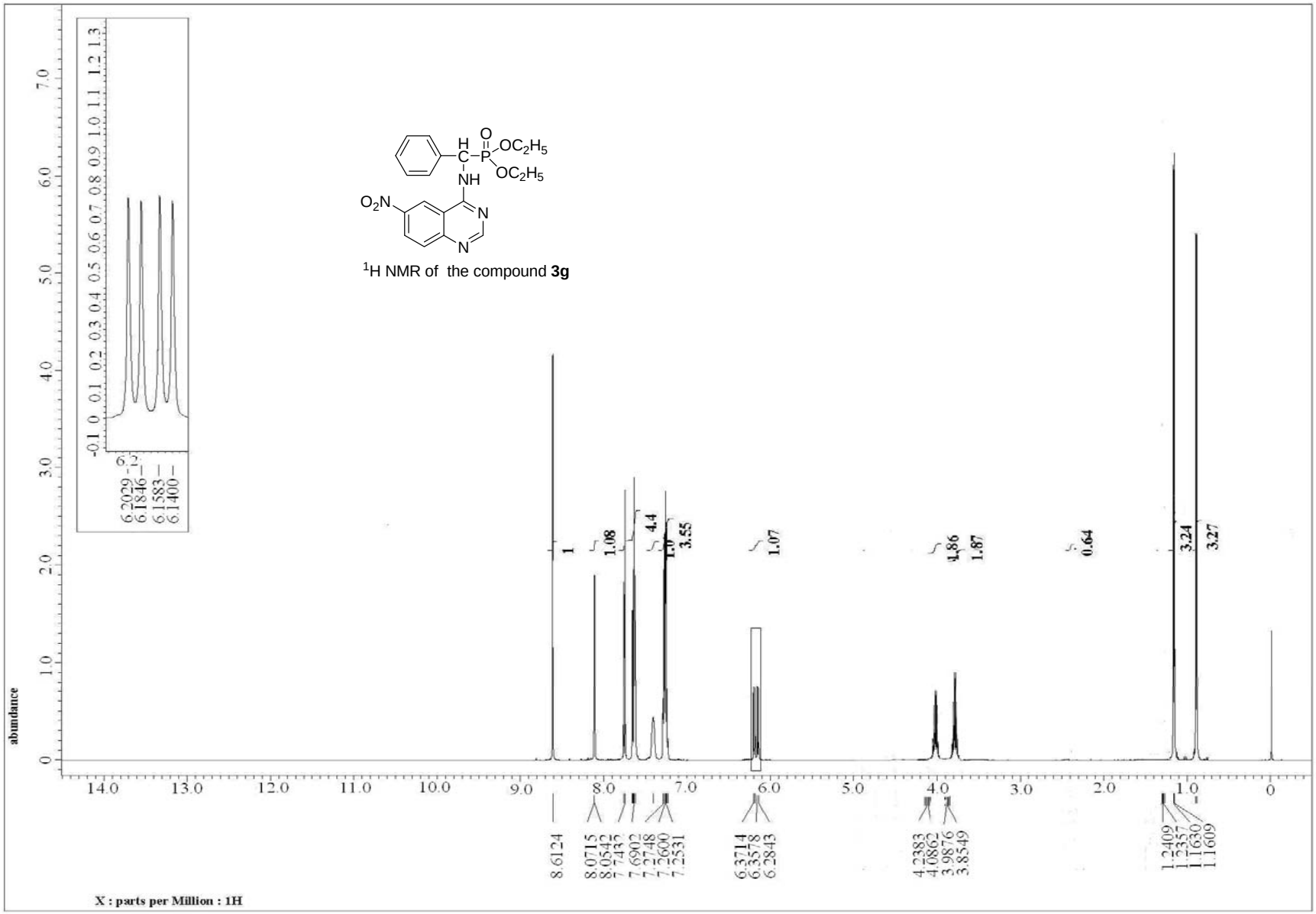
^{1}H NMR of the compound **3f**



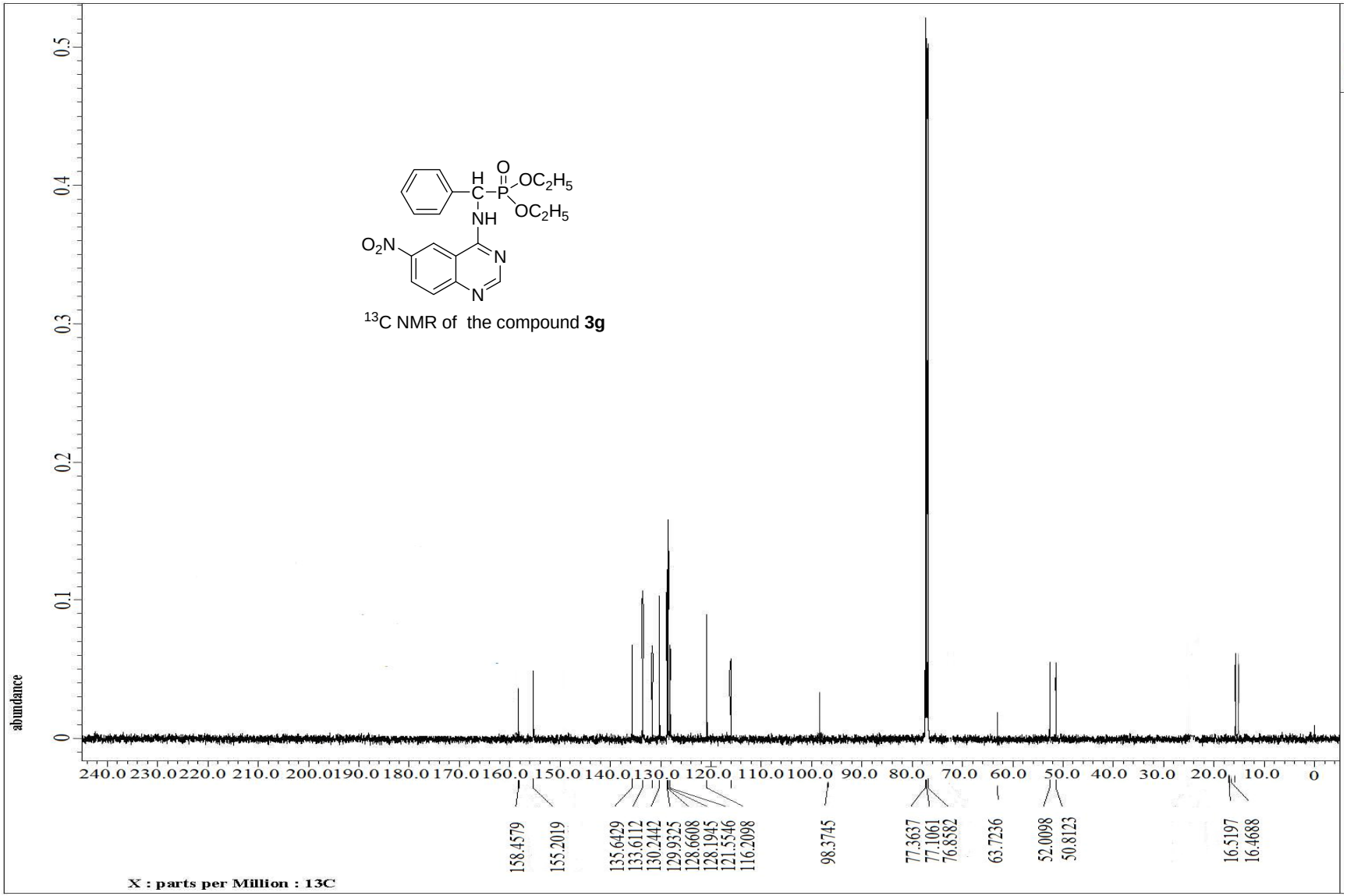
^{13}C NMR of the compound **3f**



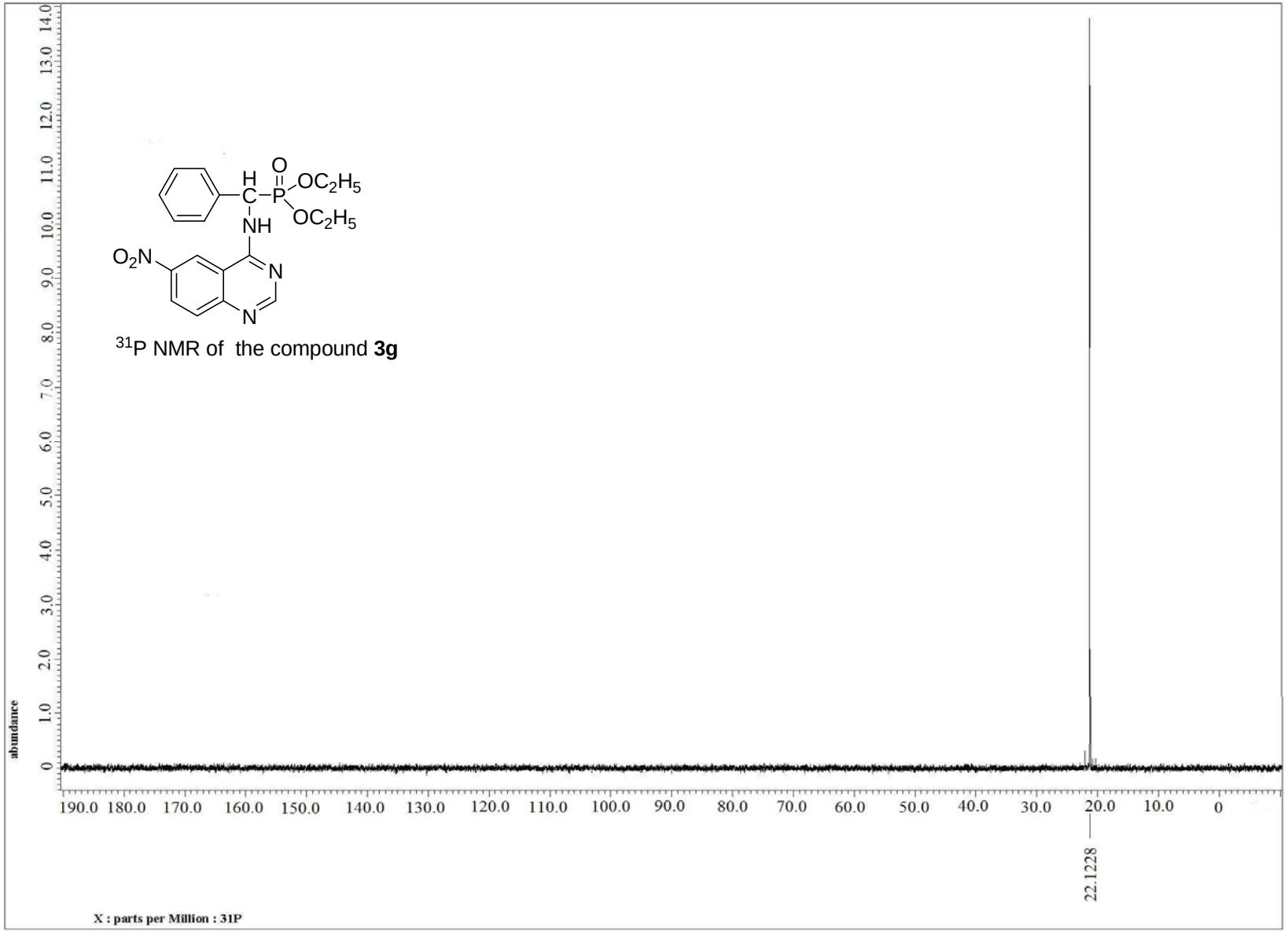
^{31}P NMR of the compound **3f**

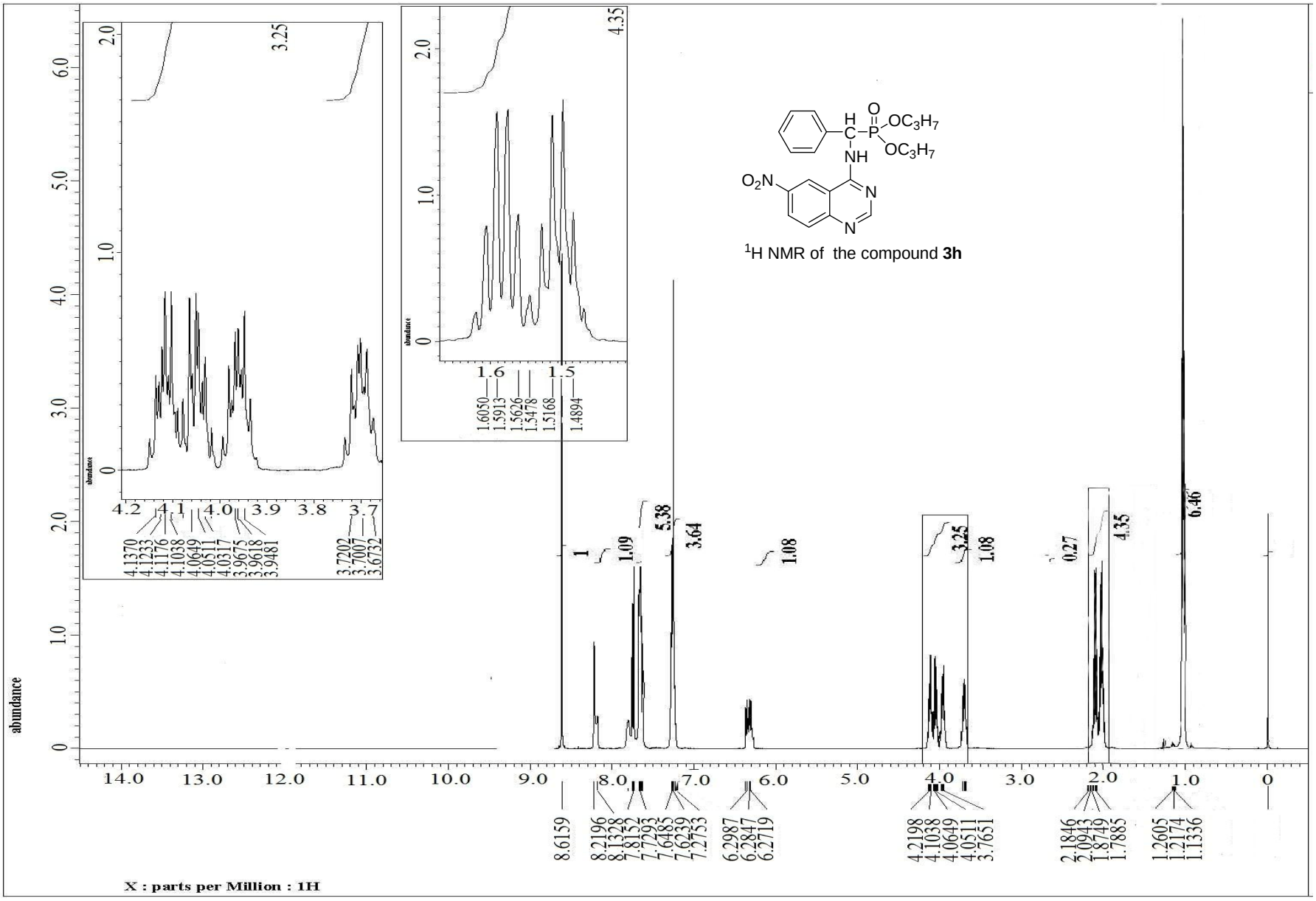


^{1}H NMR of the compound **3g**

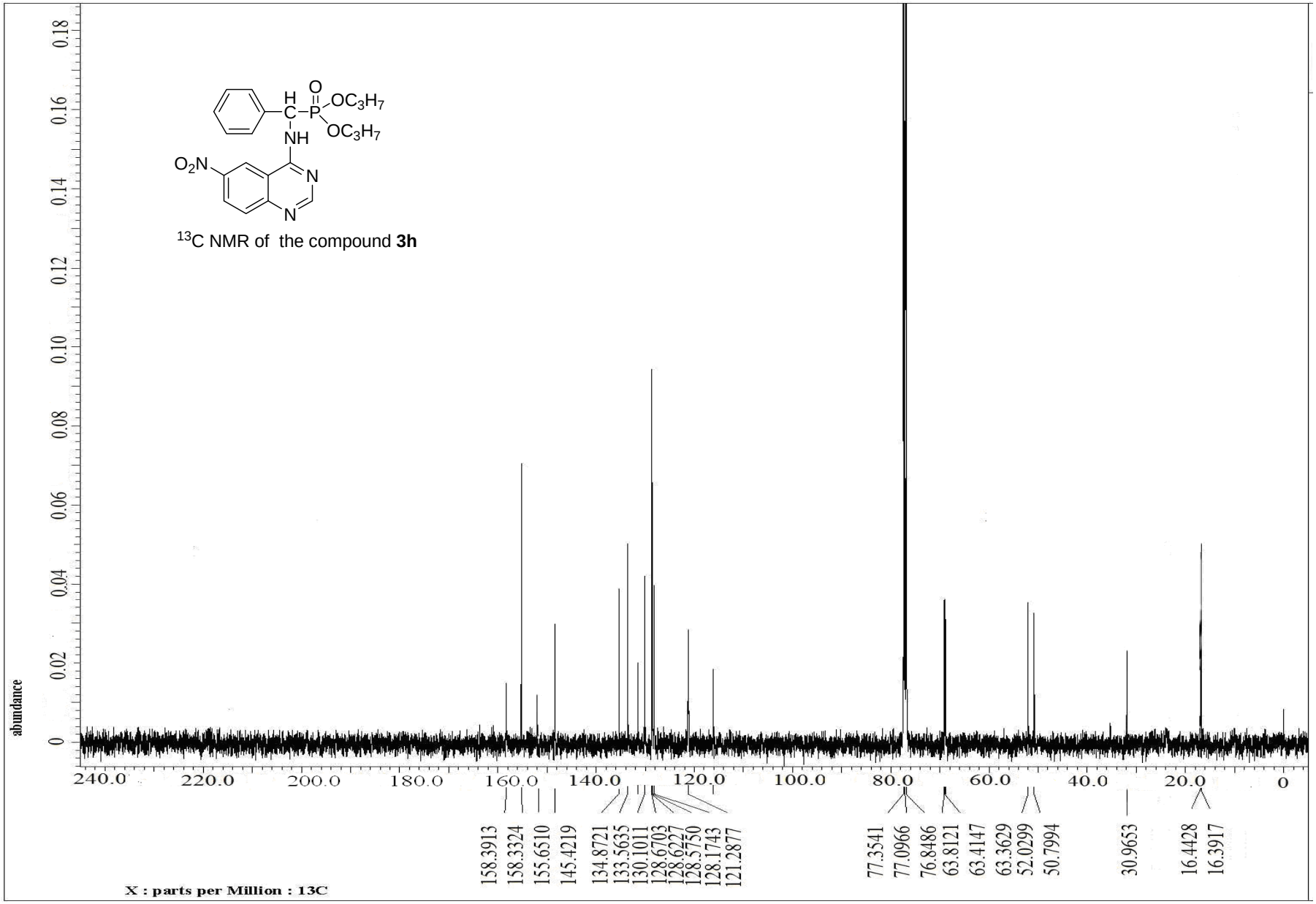


^{13}C NMR of the compound **3g**

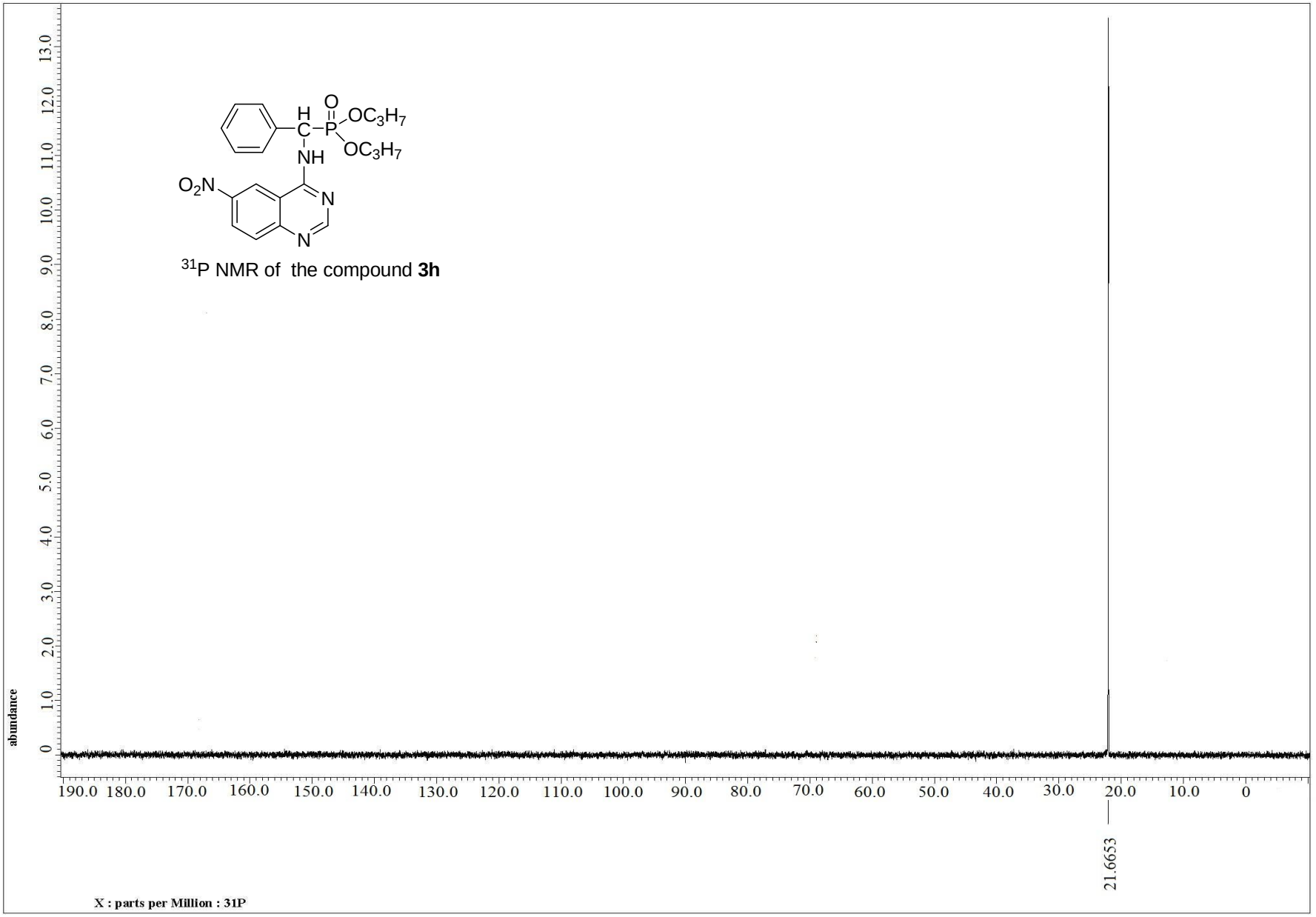

O
H
C
P
OC2H5
OC2H5
NH
O2N
N
N
31P NMR of the compound 3g
abundance
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
22.1228
X : parts per Million : 31P

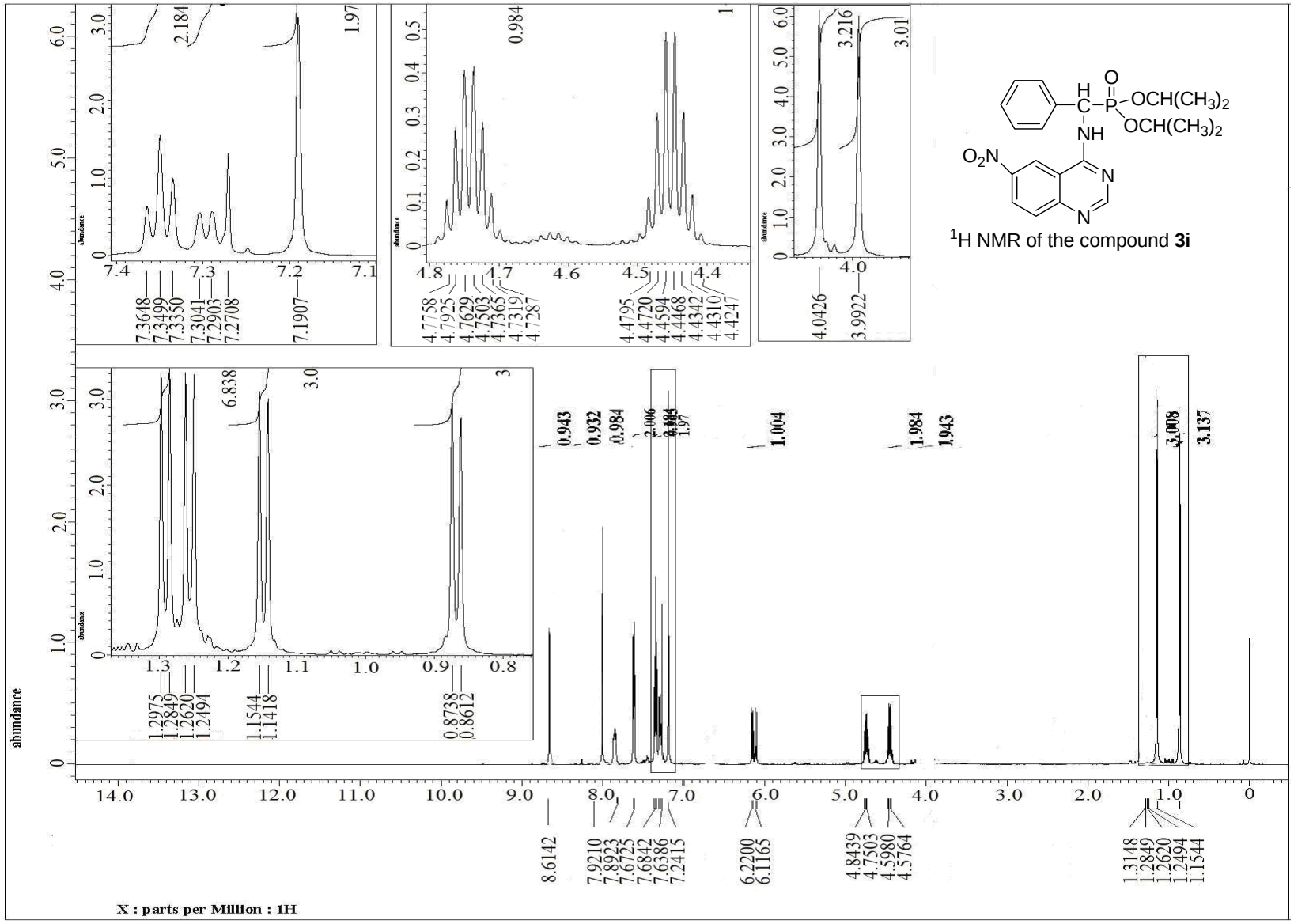


^{1}H NMR of the compound **3h**

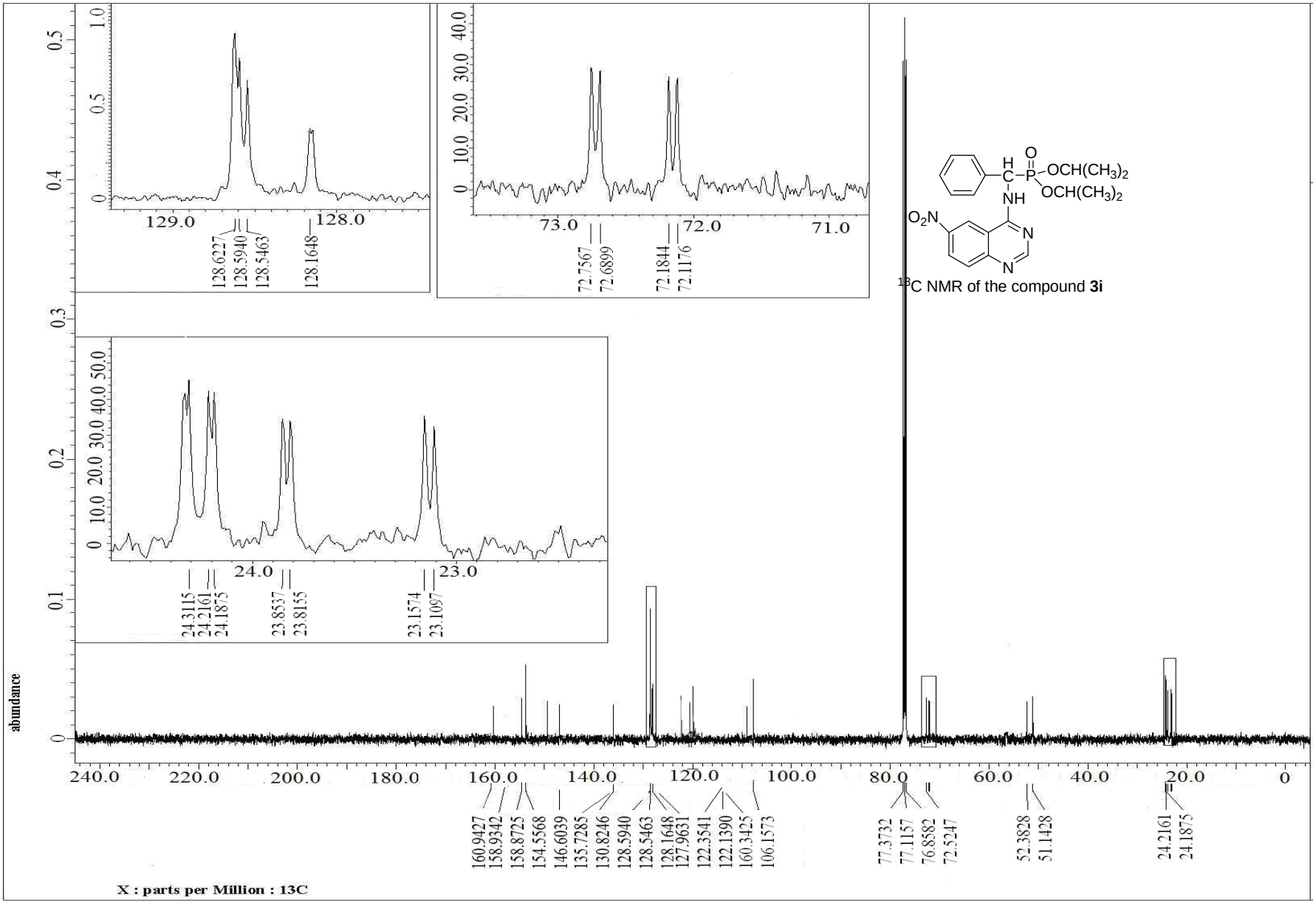


^{13}C NMR of the compound **3h**

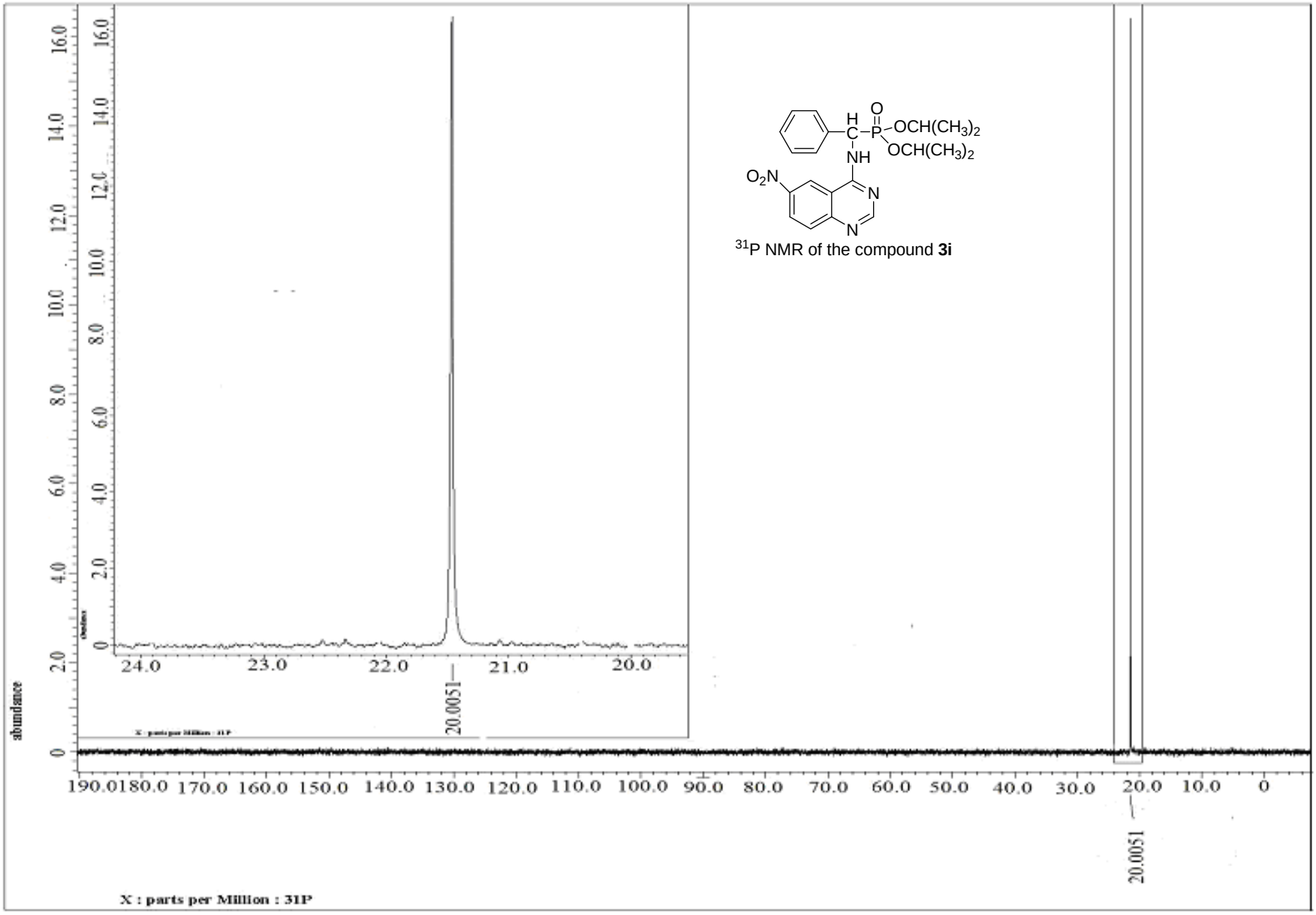

O
H
C
P
OC3H7
OC3H7
NH
O2N
N
N
31P NMR of the compound 3h
abundance
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
21.6653
X : parts per Million : 31P



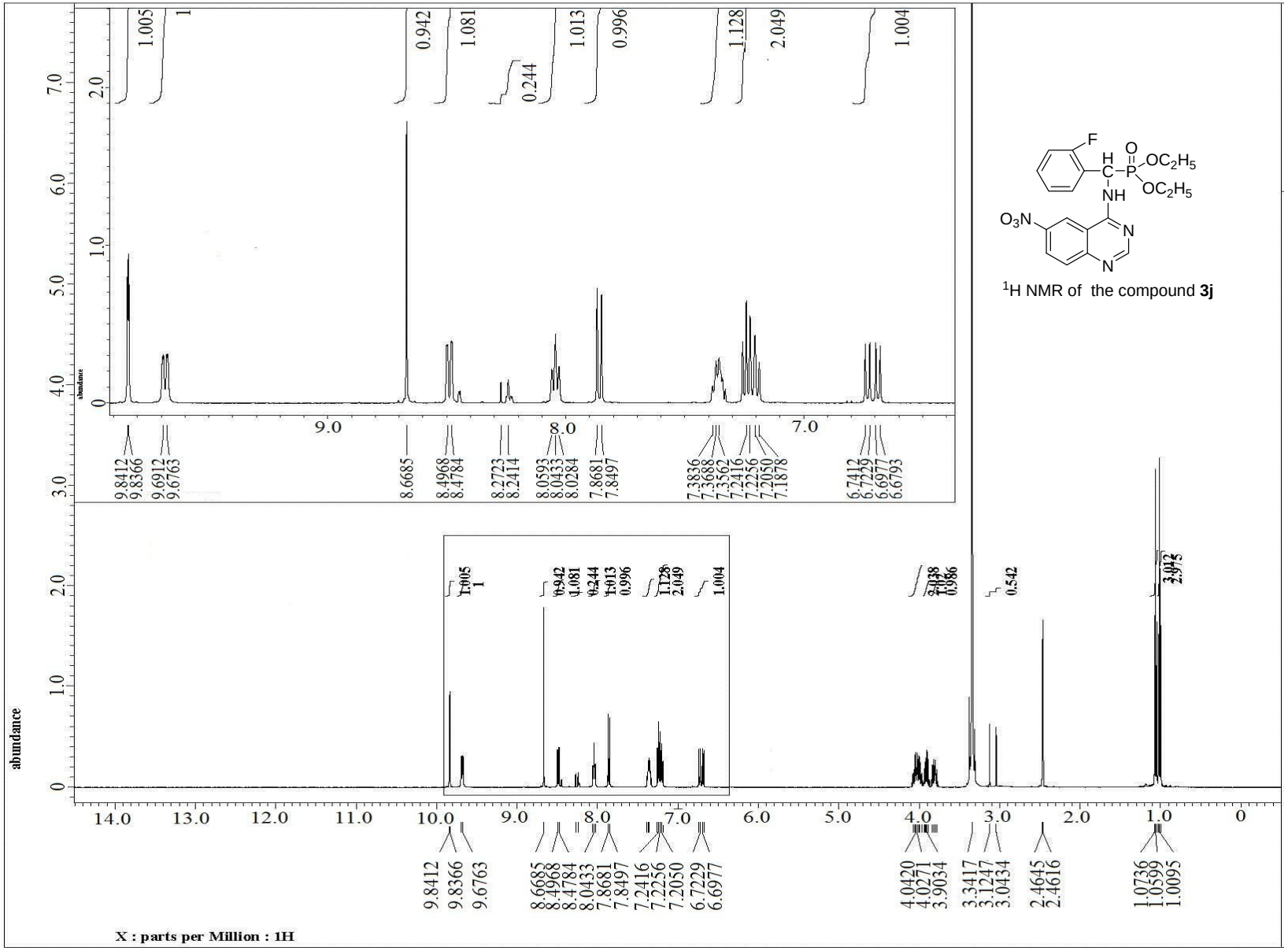
^{1}H NMR of the compound **3i**

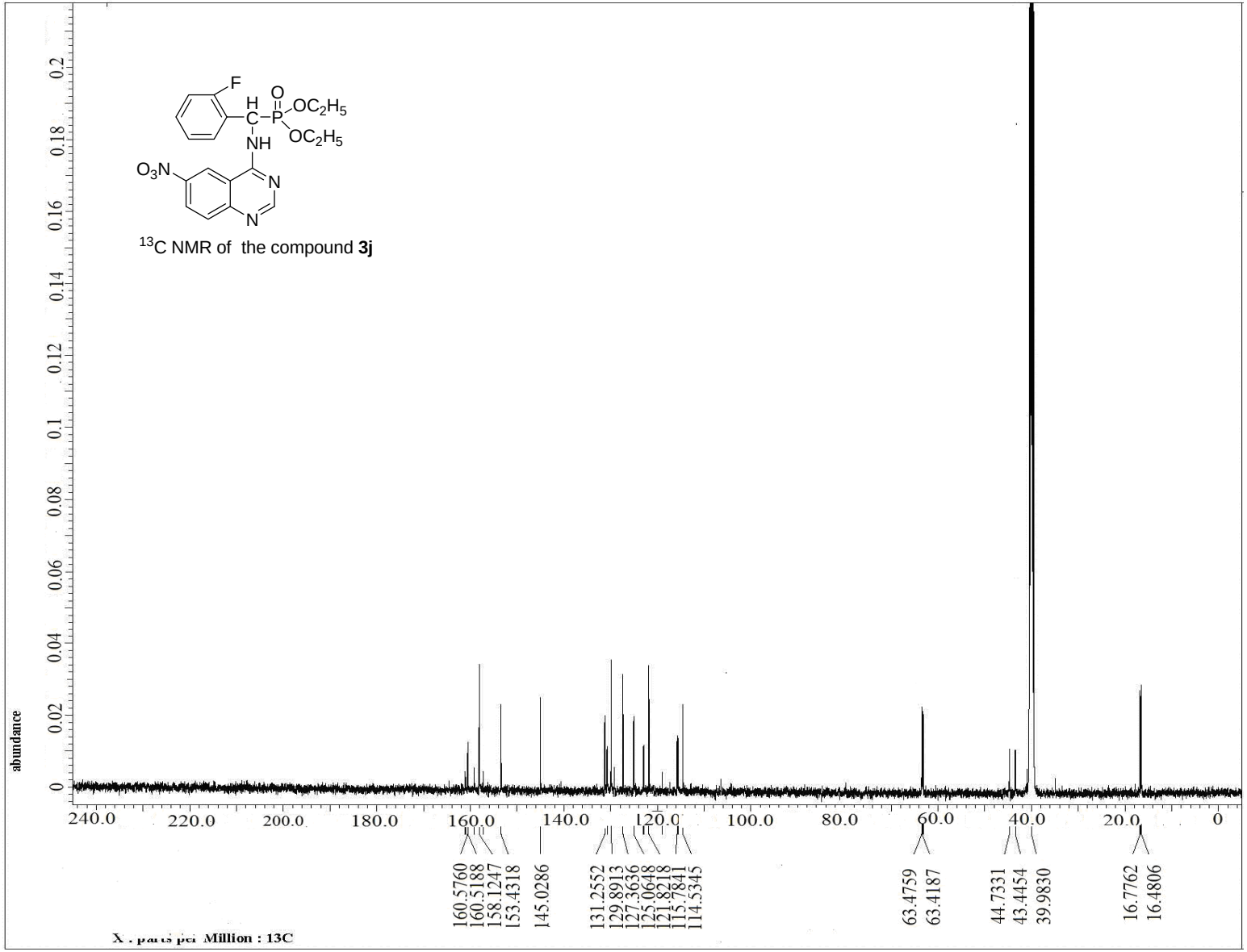


^{13}C NMR of the compound **3i**

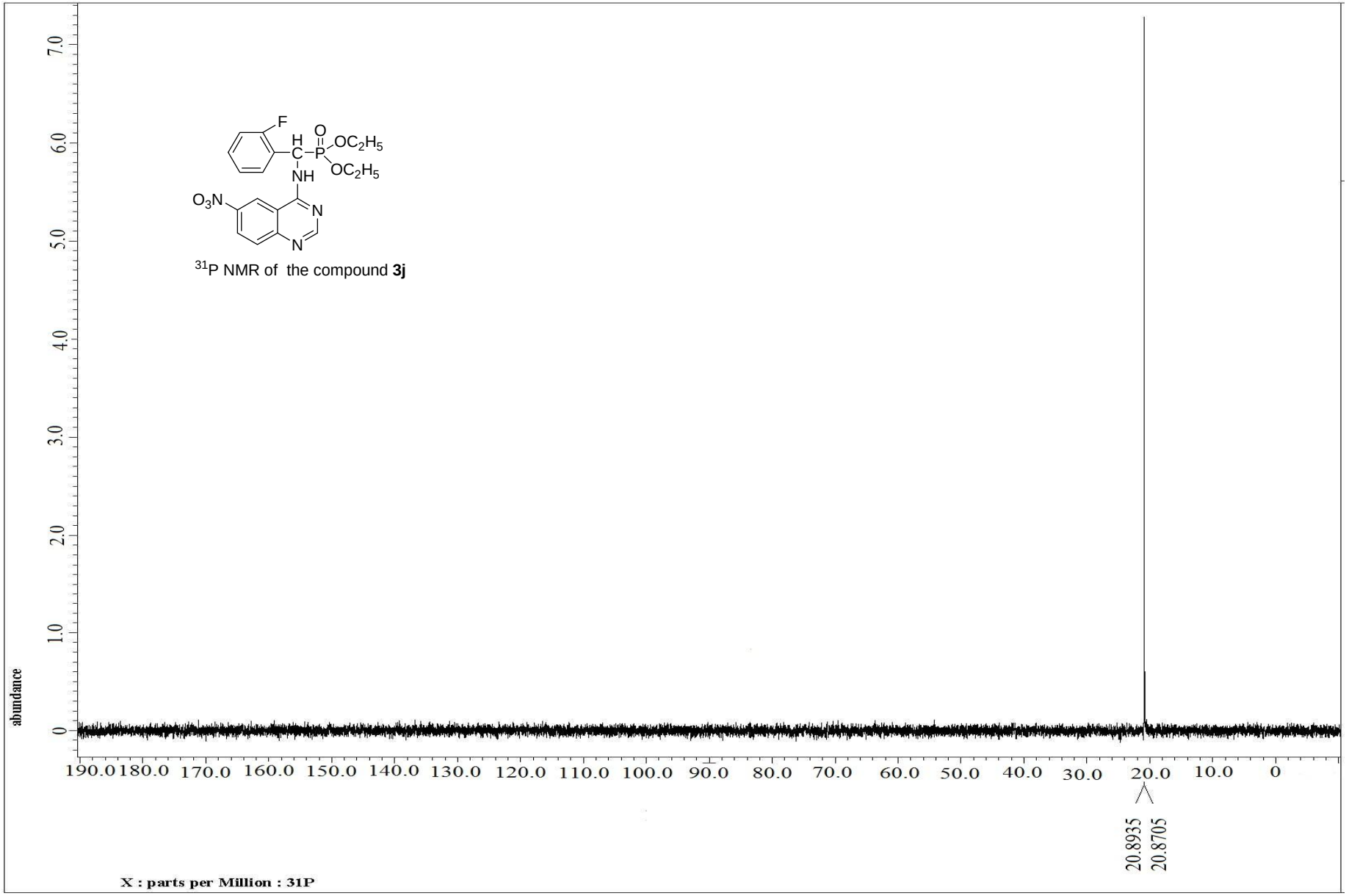


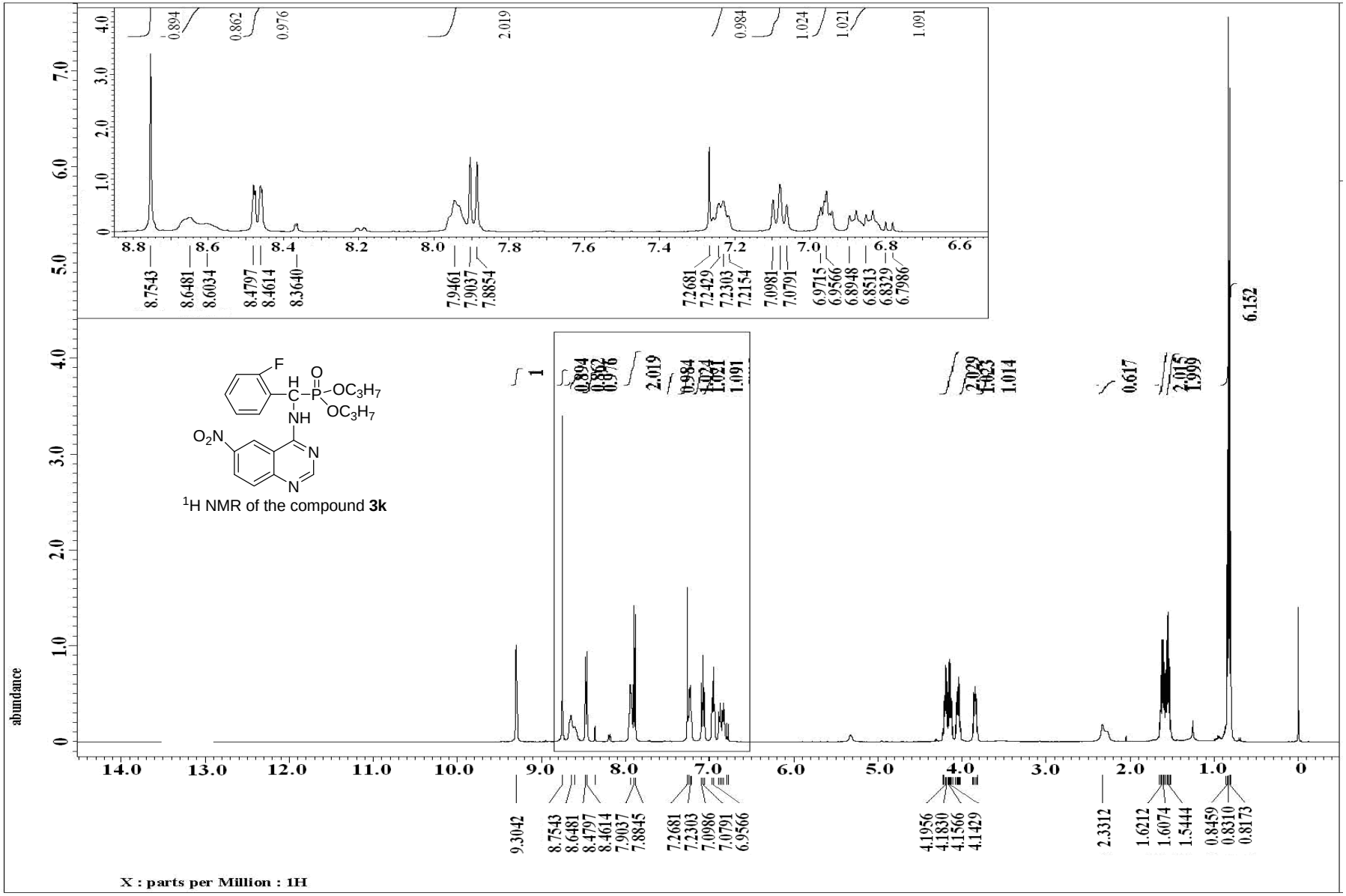
^{31}P NMR of the compound **3i**


F
H
C
P
O
OC2H5
OC2H5
NH
O3N
N
N
1H NMR of the compound 3j
abundance
X : parts per Million : 1H

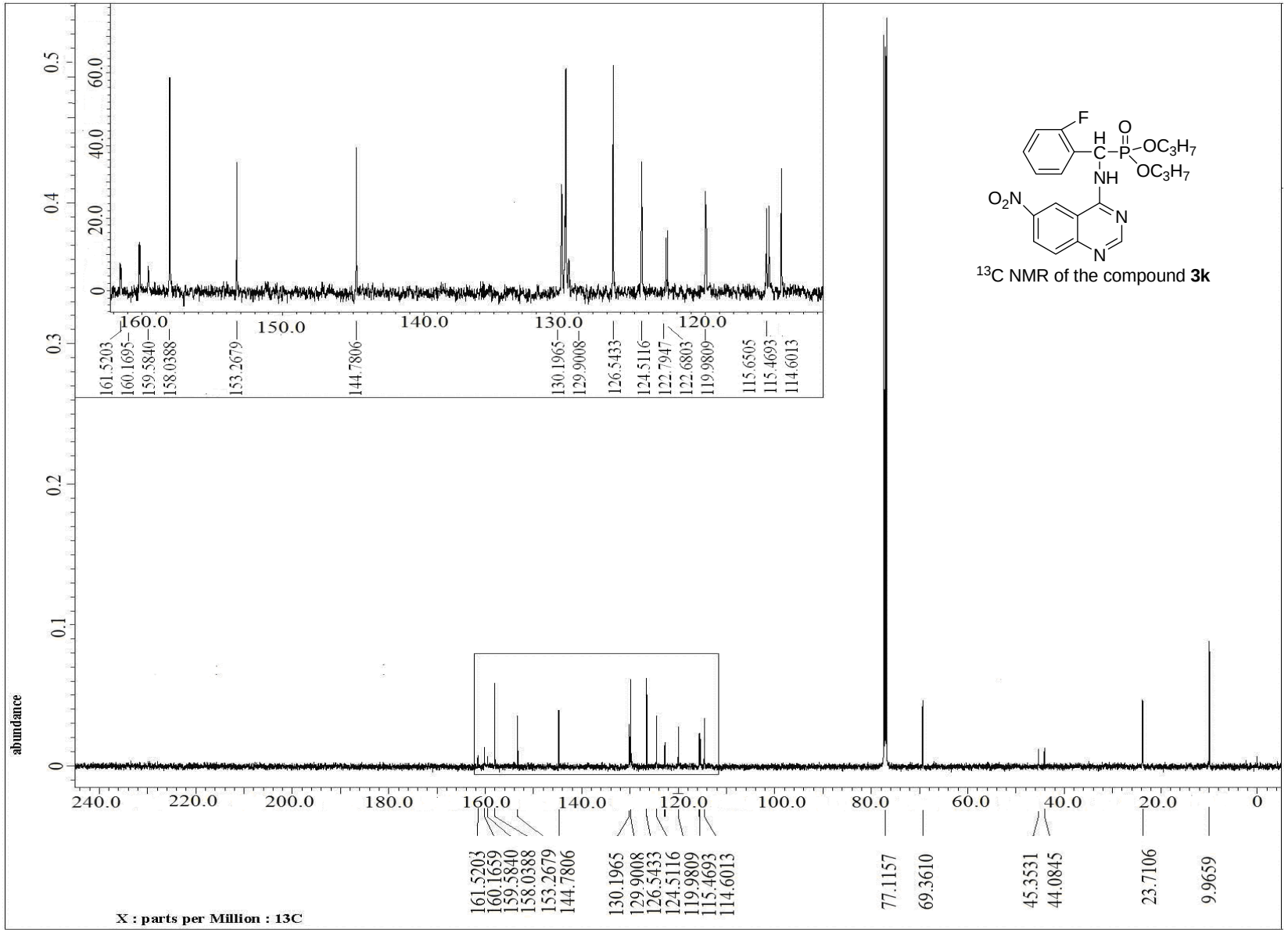


^{13}C NMR of the compound **3j**

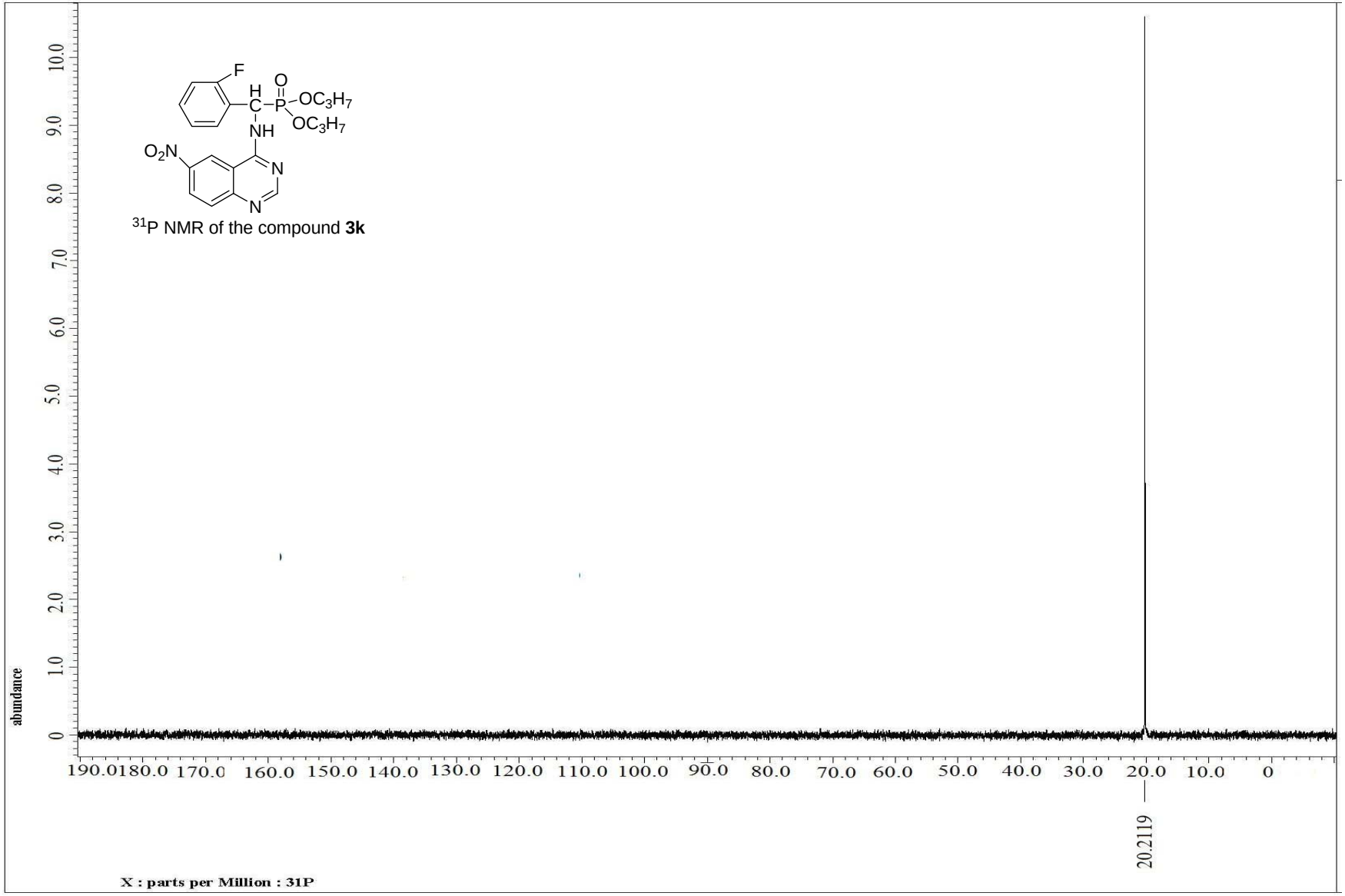

F
H
C
P
O
OC_2H_5
OC_2H_5
NH
O_3N
N
N
^{31}P NMR of the compound **3j**
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
20.8935
20.8705
X : parts per Million : 31P

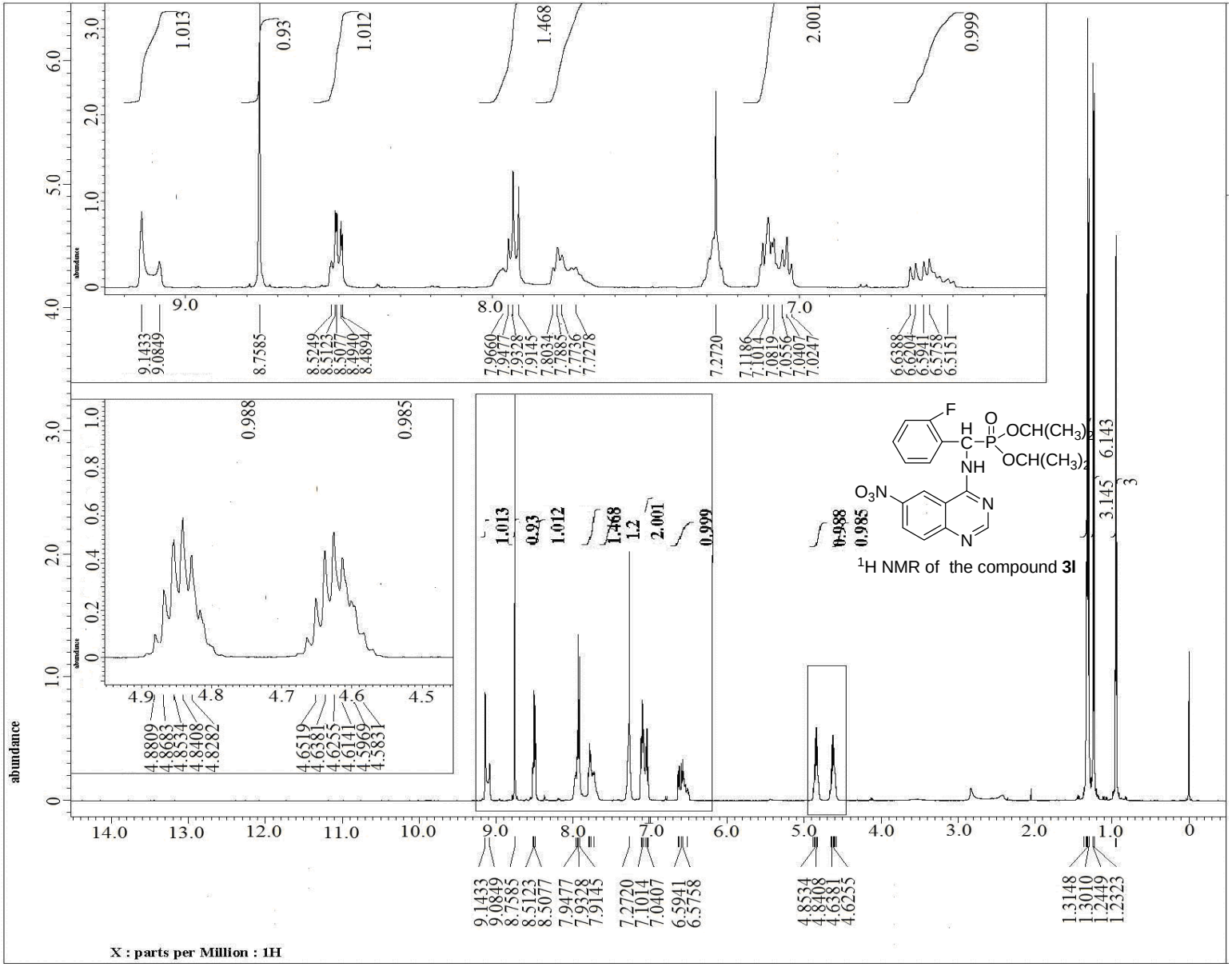


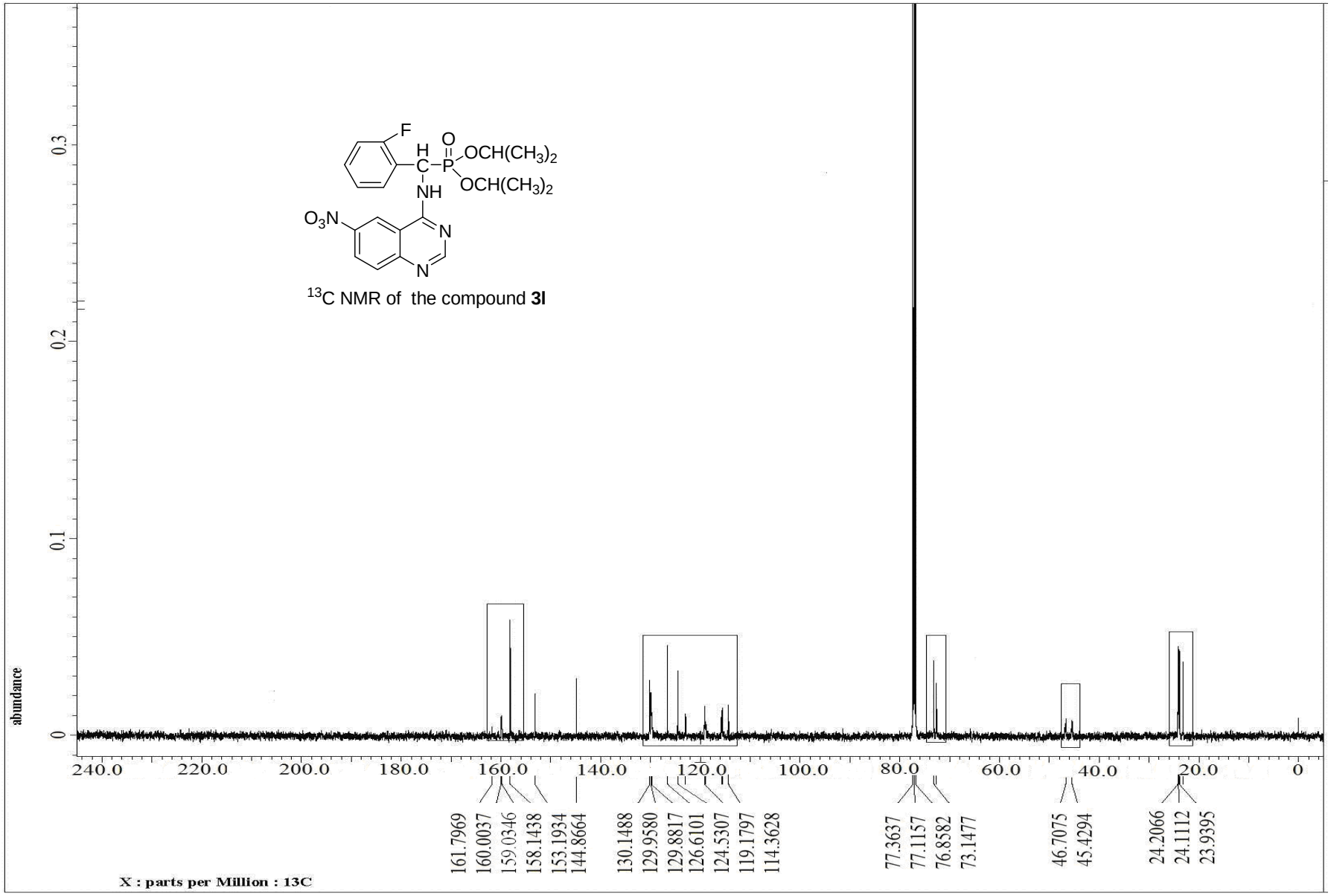
^{1}H NMR of the compound **3k**



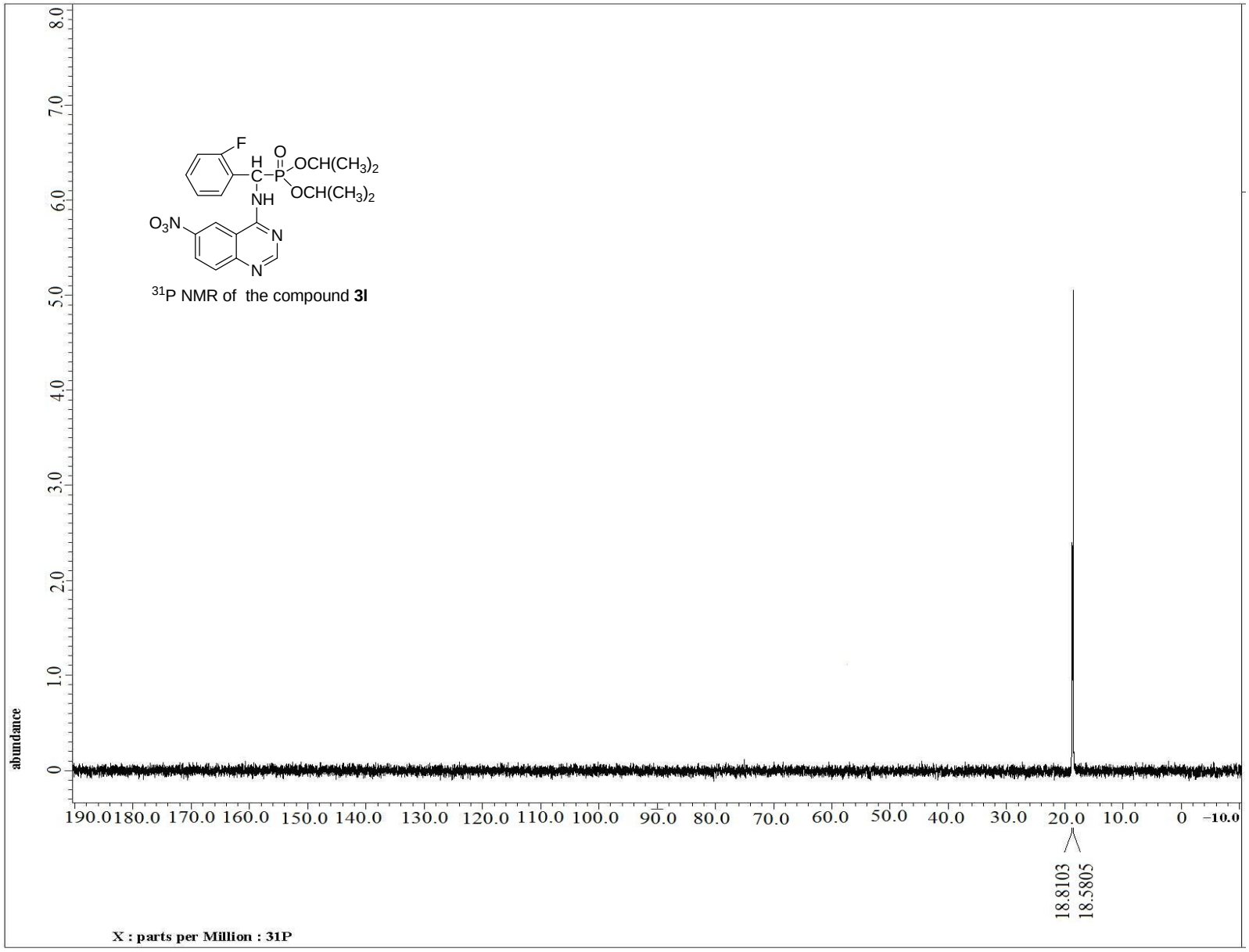
^{13}C NMR of the compound **3k**

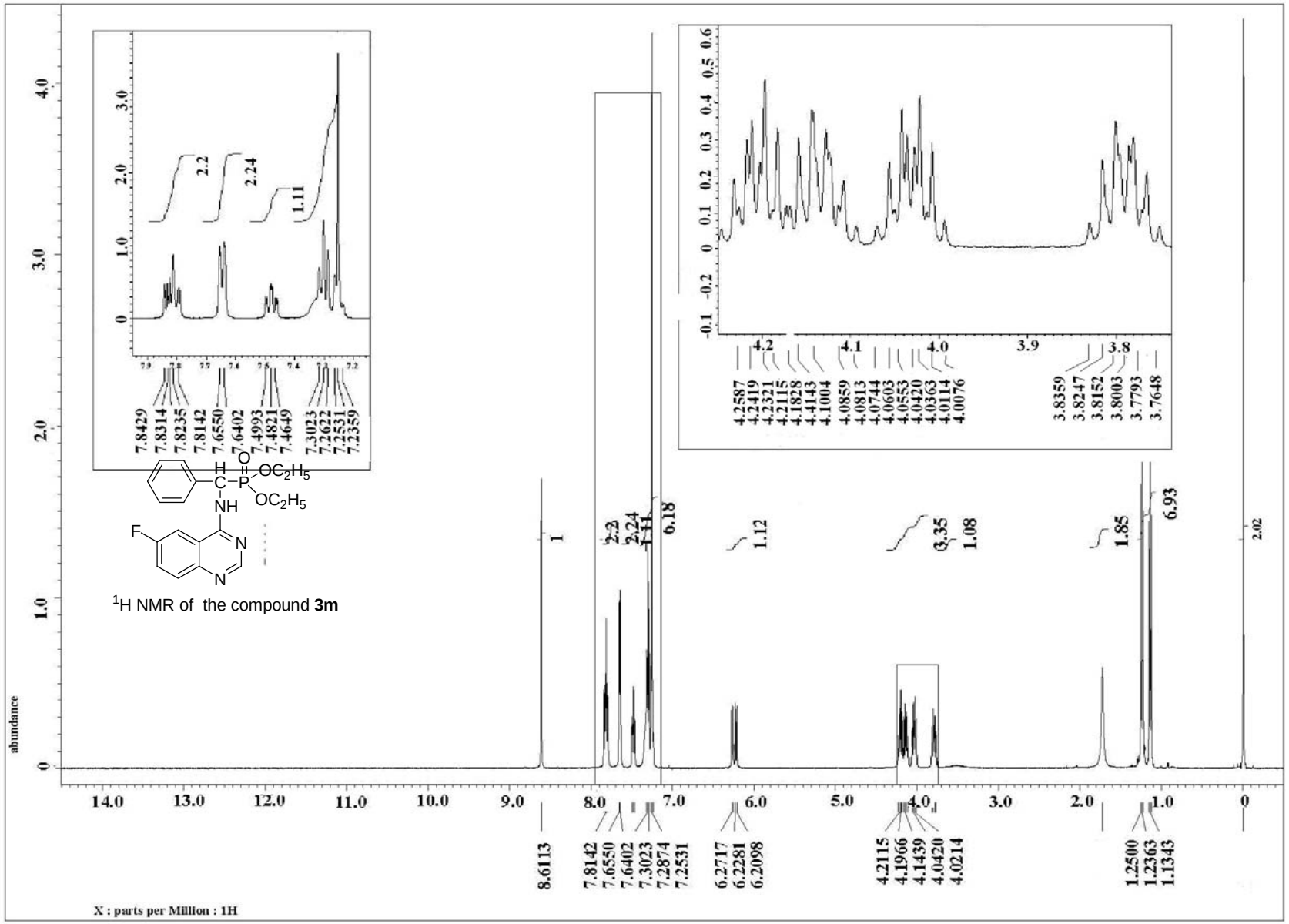

F
H
C
O
P
OC3H7
OC3H7
NH
O2N
N
N
31P NMR of the compound 3k
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
20.2119
X : parts per Million : 31P


1.013
0.93
1.012
1.468
2.001
0.999
0.988
0.985
6.143
3.145
3
F
H
C
P
O
OCH(CH3)2
OCH(CH3)2
NH
O3N
N
N
1H NMR of the compound 3l
abundance
X : parts per Million : 1H

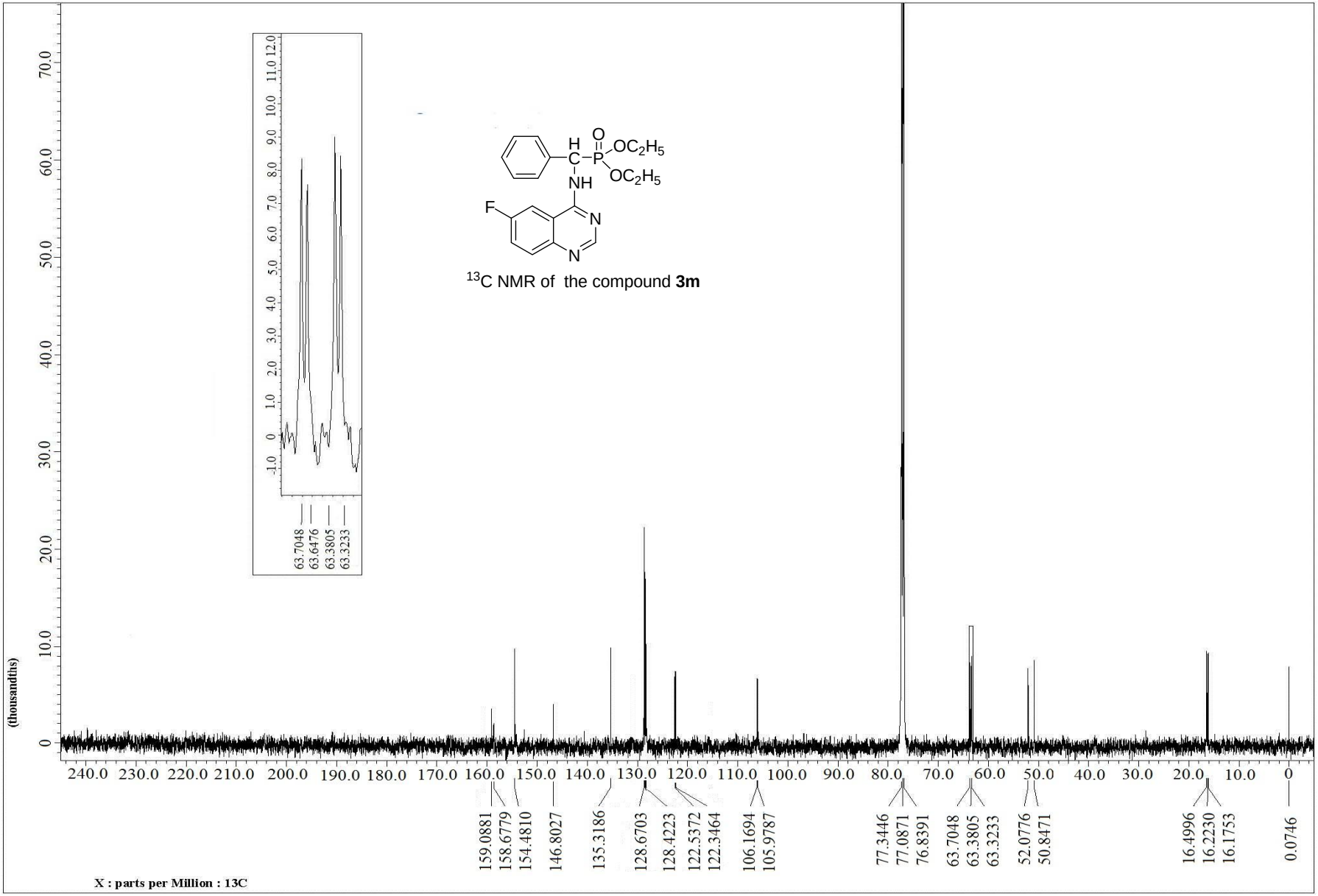


^{13}C NMR of the compound **3l**

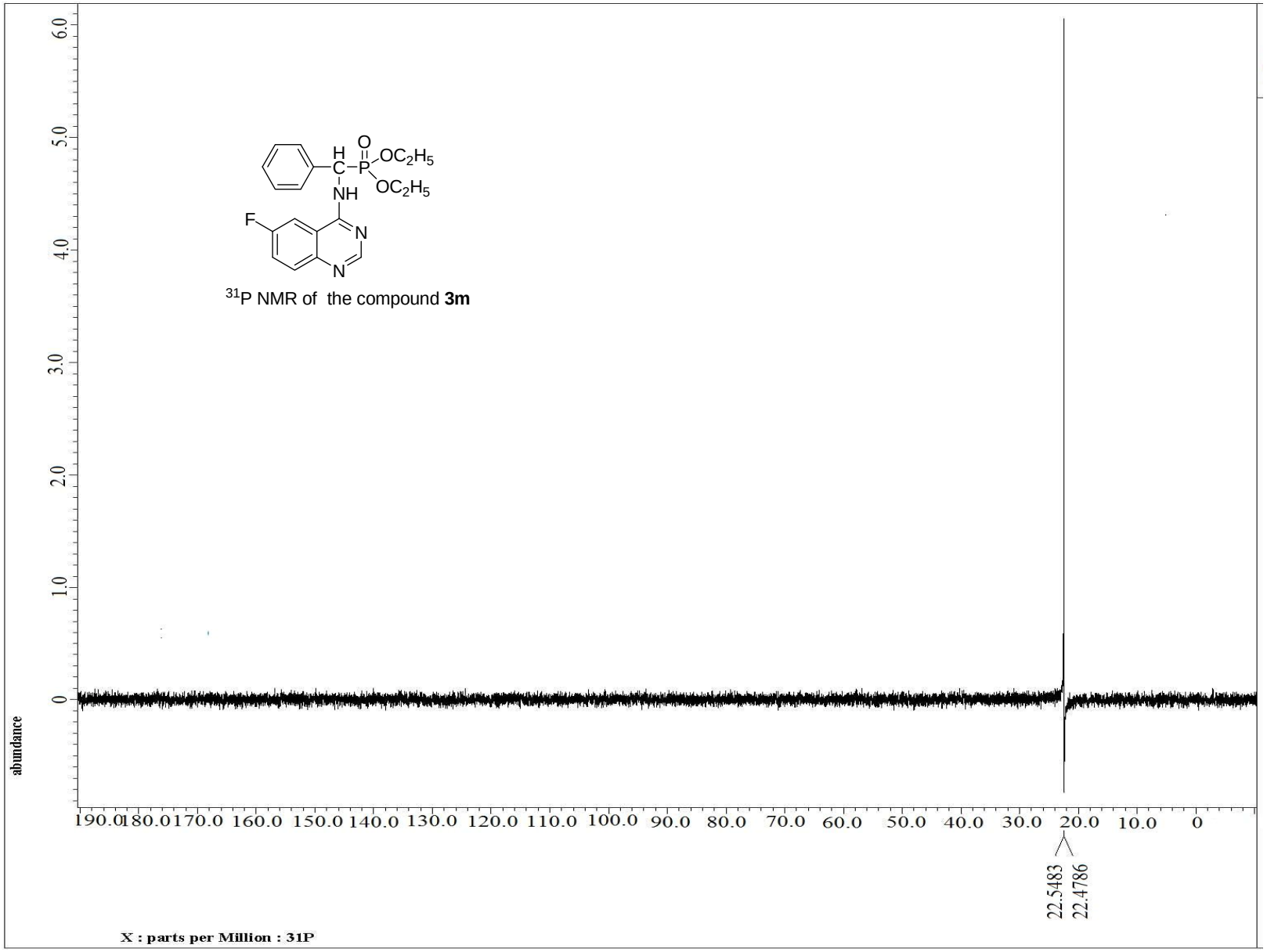

F
H
C
P
O
OCH(CH3)2
OCH(CH3)2
NH
O3N
N
N
31P NMR of the compound 3l
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
18.8103
18.5805
X : parts per Million : 31P



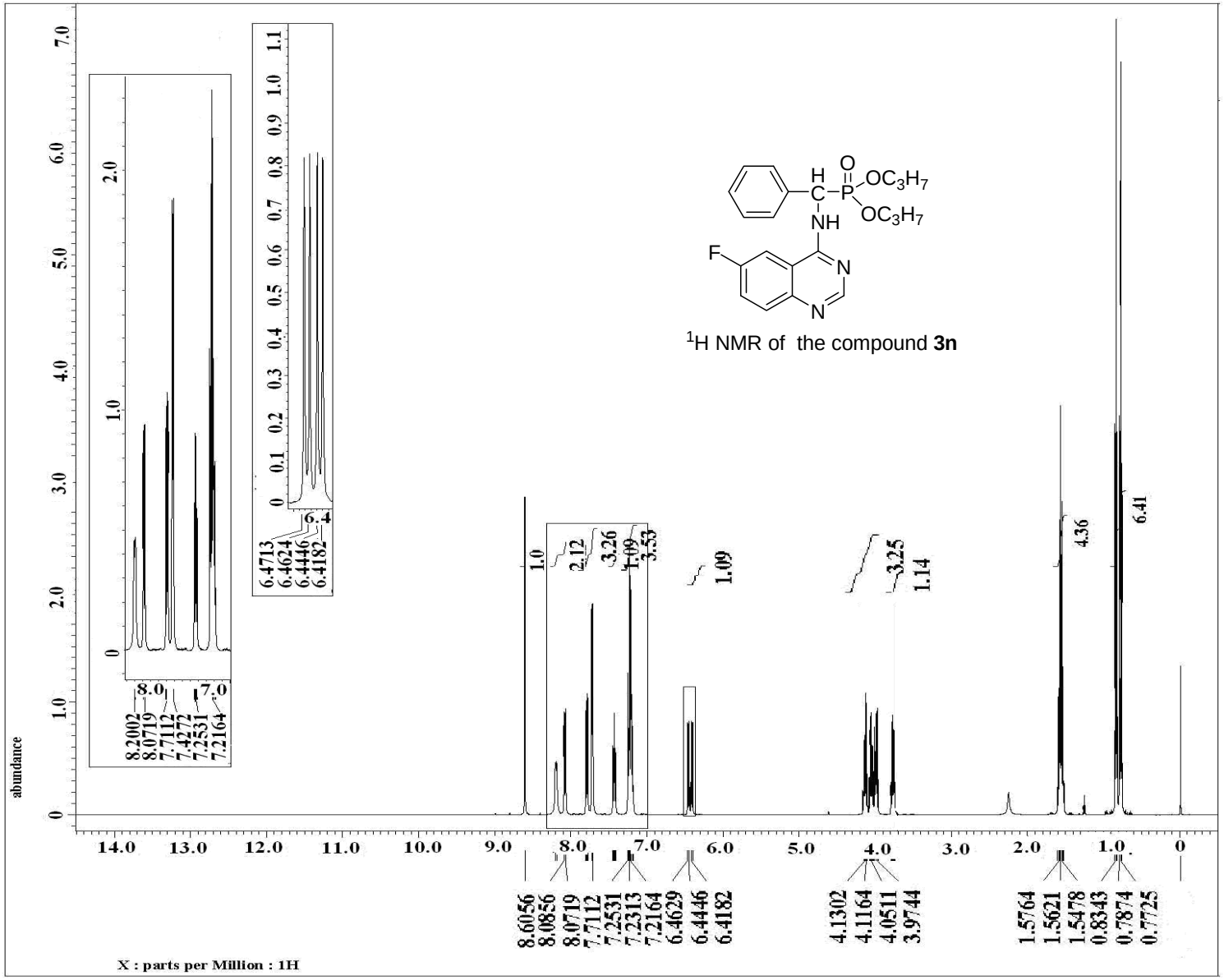
^{1}H NMR of the compound **3m**



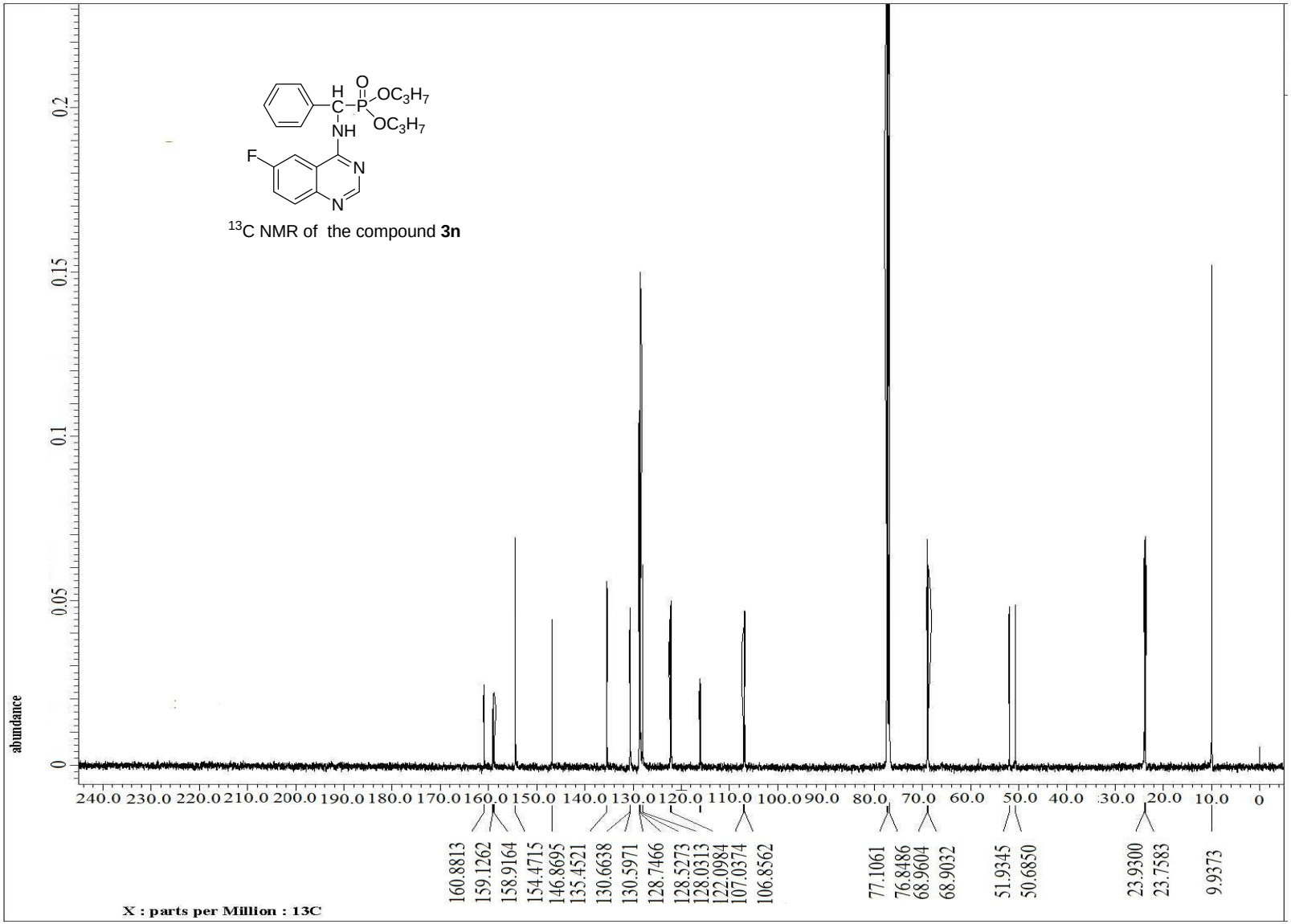
^{13}C NMR of the compound **3m**



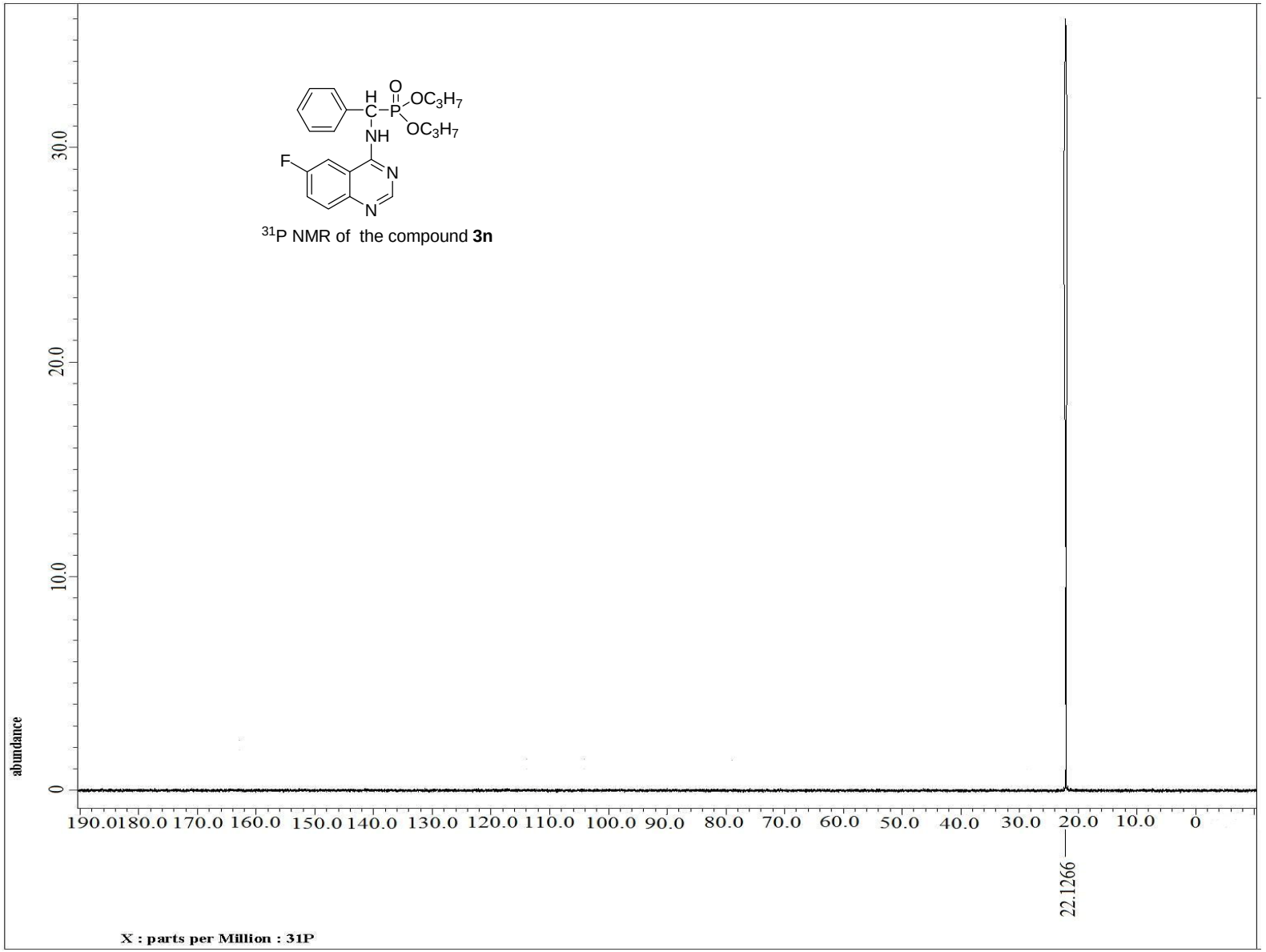
^{31}P NMR of the compound **3m**



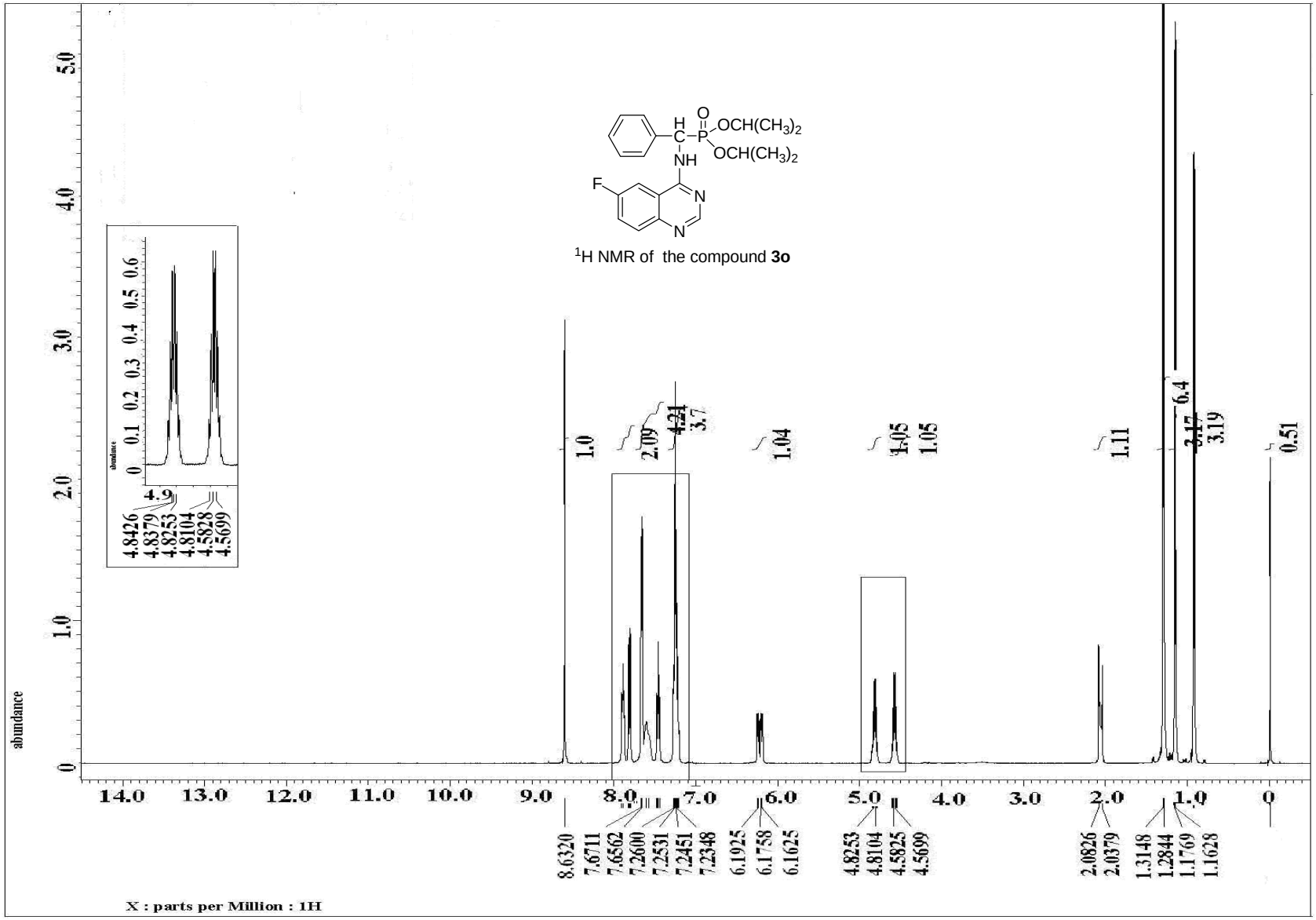
^{1}H NMR of the compound **3n**

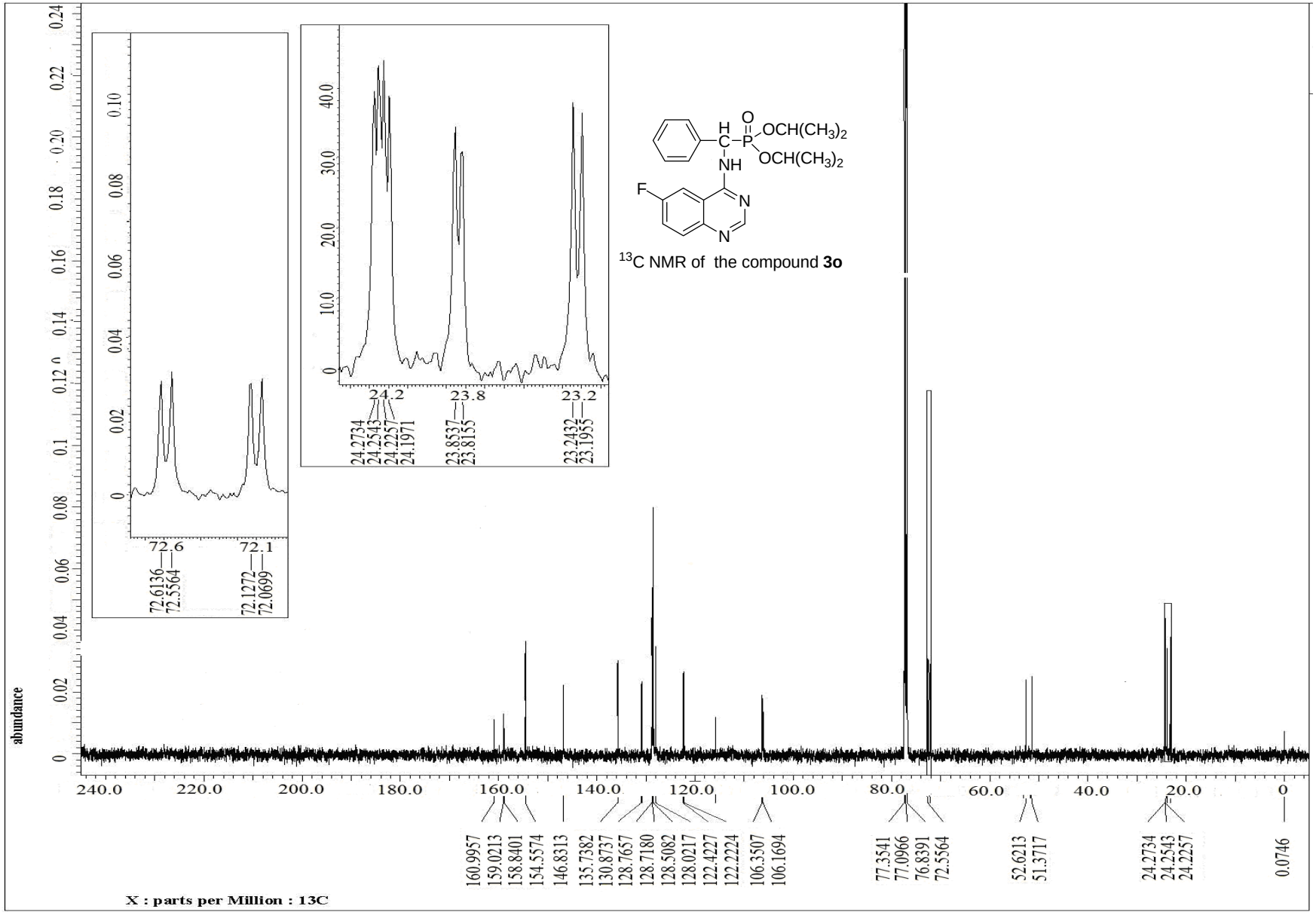


^{13}C NMR of the compound **3n**

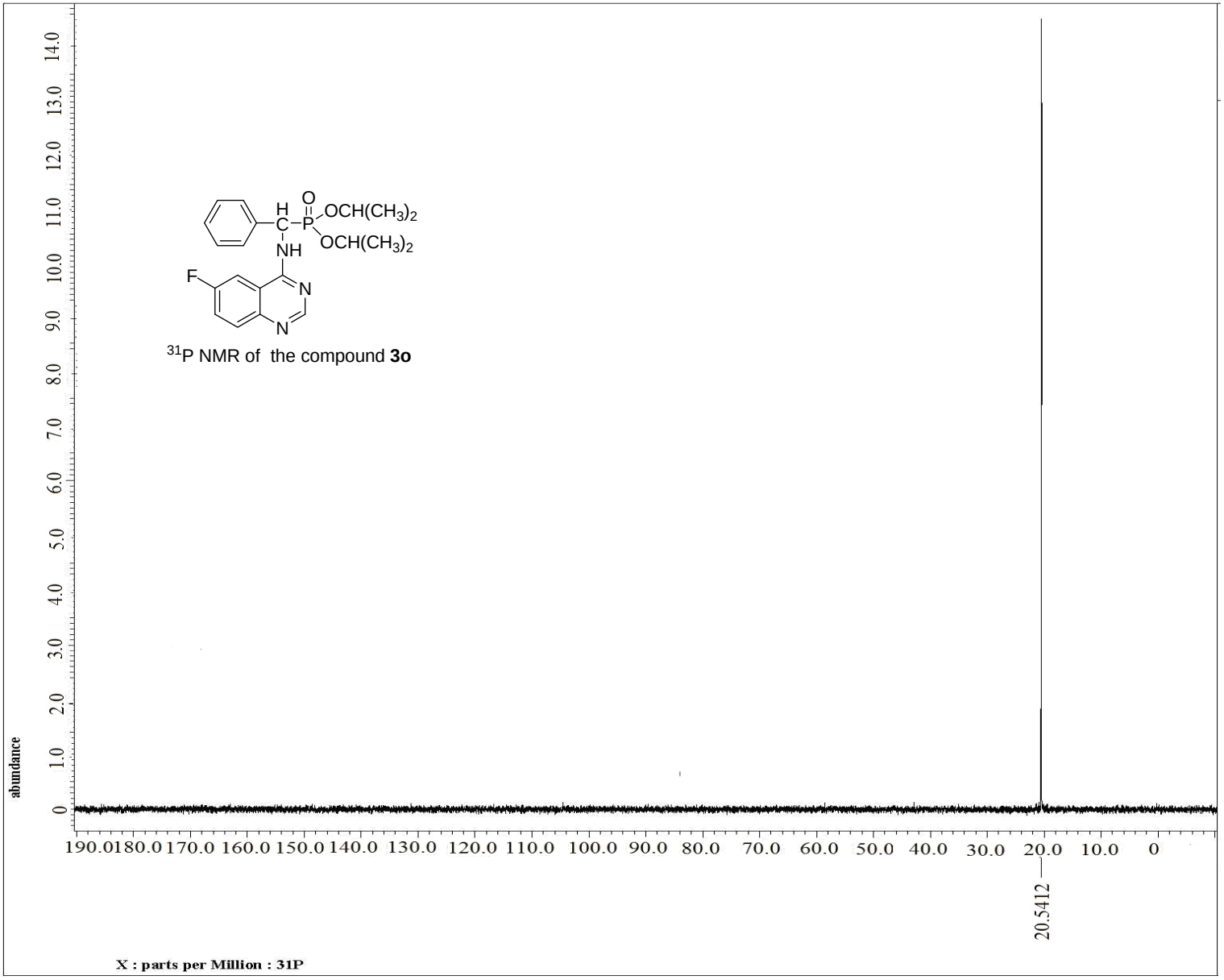


^{31}P NMR of the compound **3n**

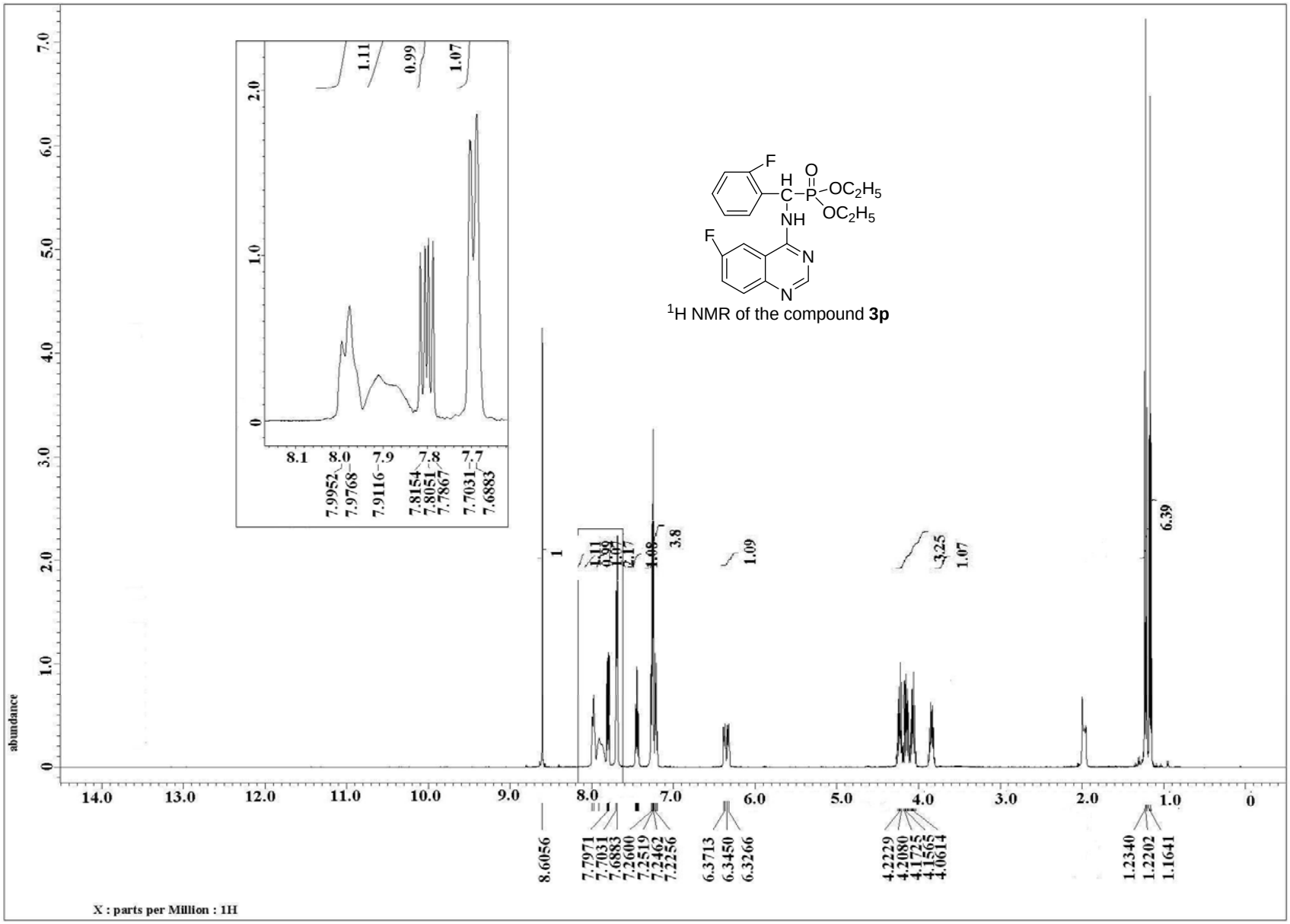

O
H
C
P
OCH(CH3)2
NH
OCH(CH3)2
F
N
N
1H NMR of the compound 3o
abundance
X : parts per Million : 1H
1.0
2.09
4.21
3.7
1.04
1.05
1.05
1.11
6.4
3.17
3.19
0.51
8.6320
7.6711
7.6562
7.2600
7.2531
7.2451
7.2348
6.1925
6.1758
6.1625
4.8253
4.8104
4.5825
4.5699
2.0826
2.0379
1.3148
1.2844
1.1769
1.1628
4.8426
4.8379
4.8253
4.8104
4.5828
4.5699



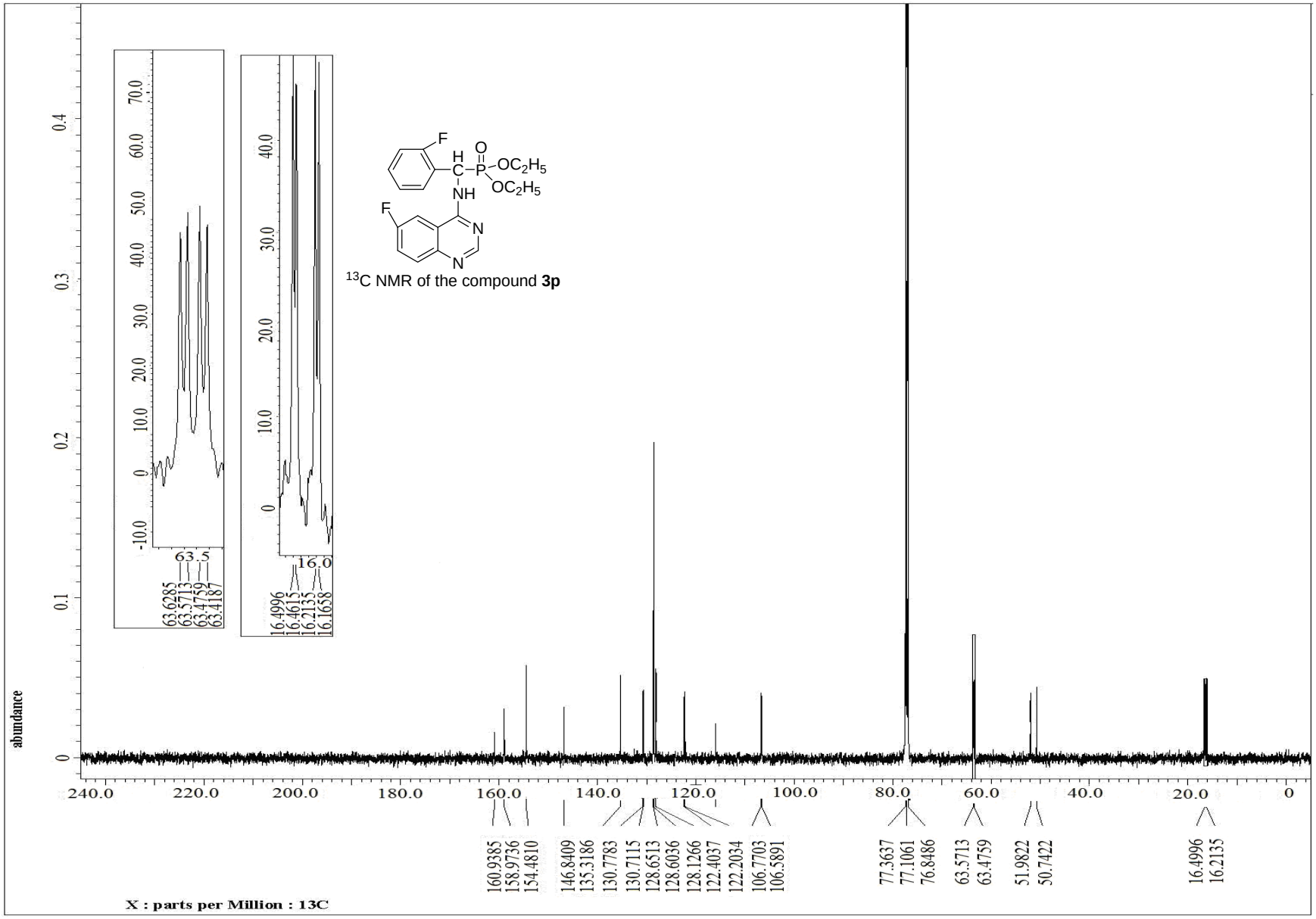
^{13}C NMR of the compound **3o**



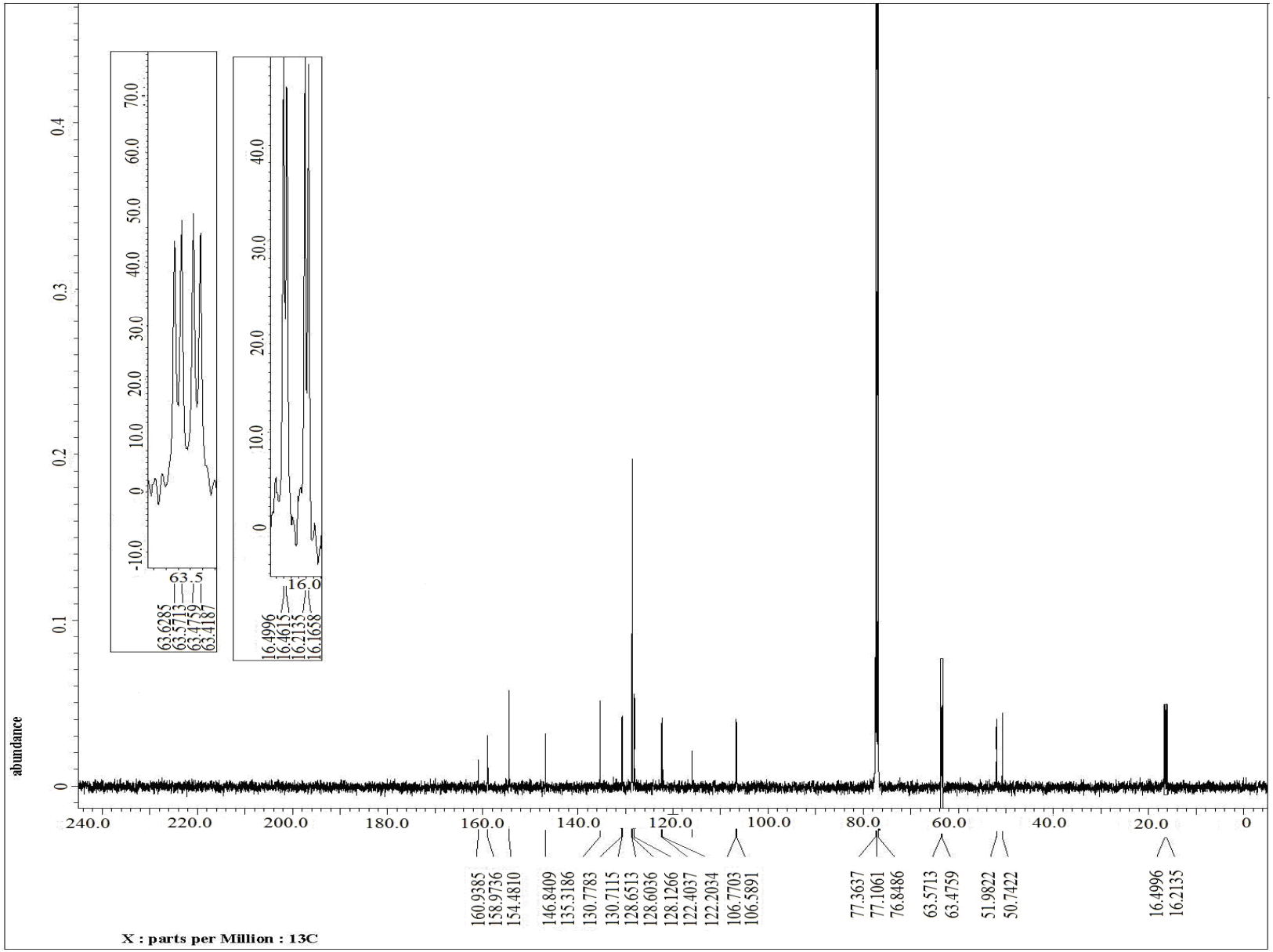
^{31}P NMR of the compound **3o**

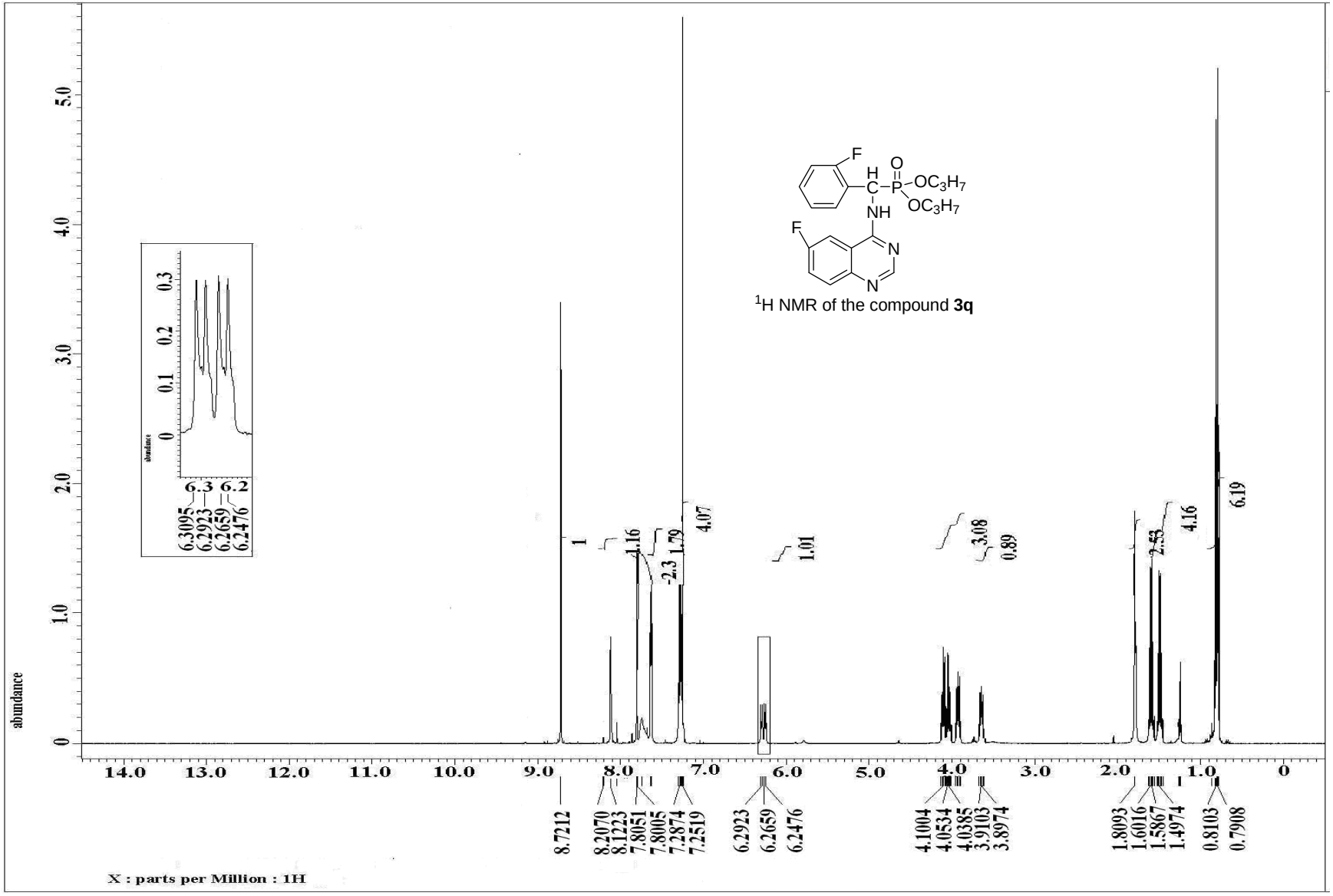


^{1}H NMR of the compound **3p**

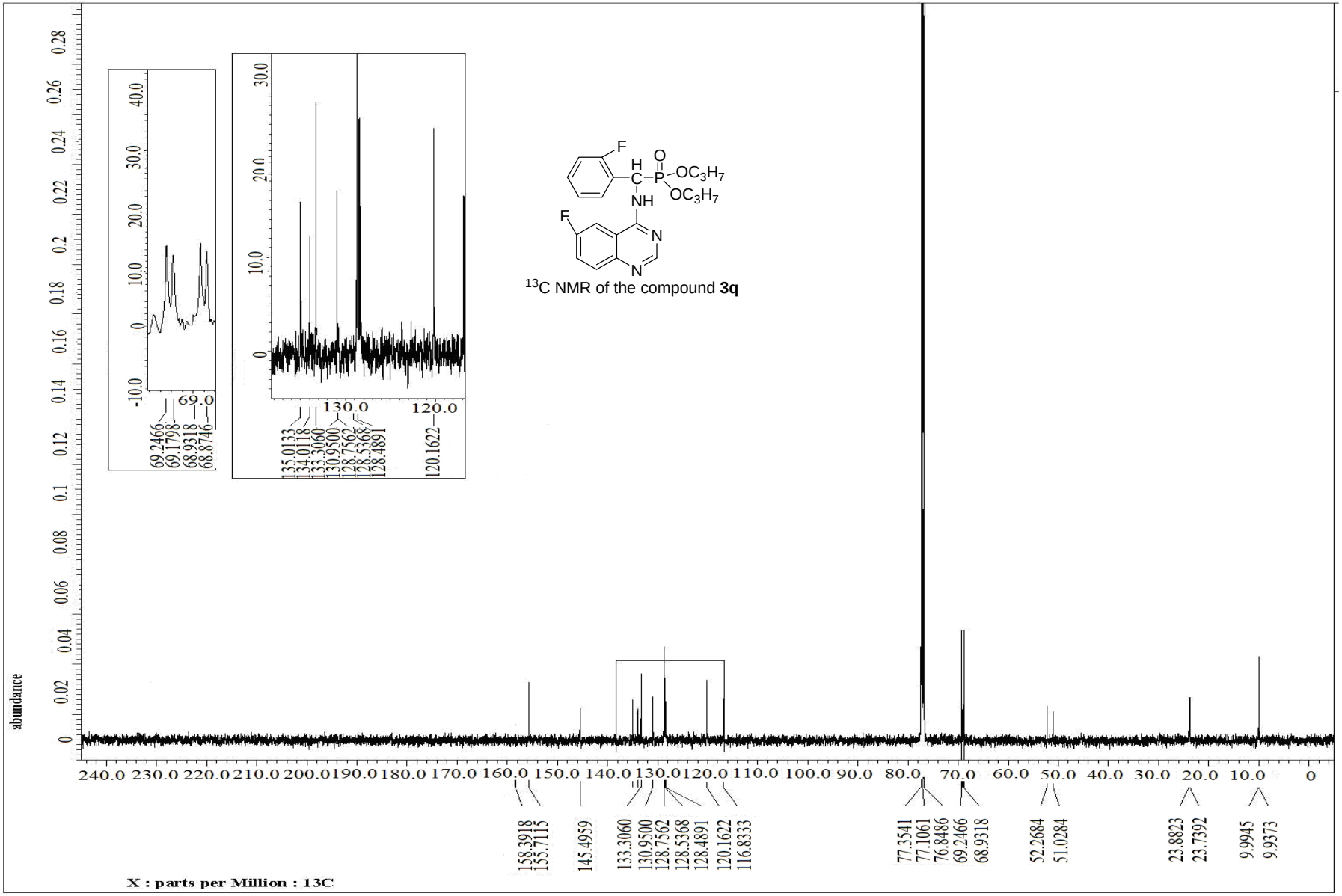


^{13}C NMR of the compound **3p**

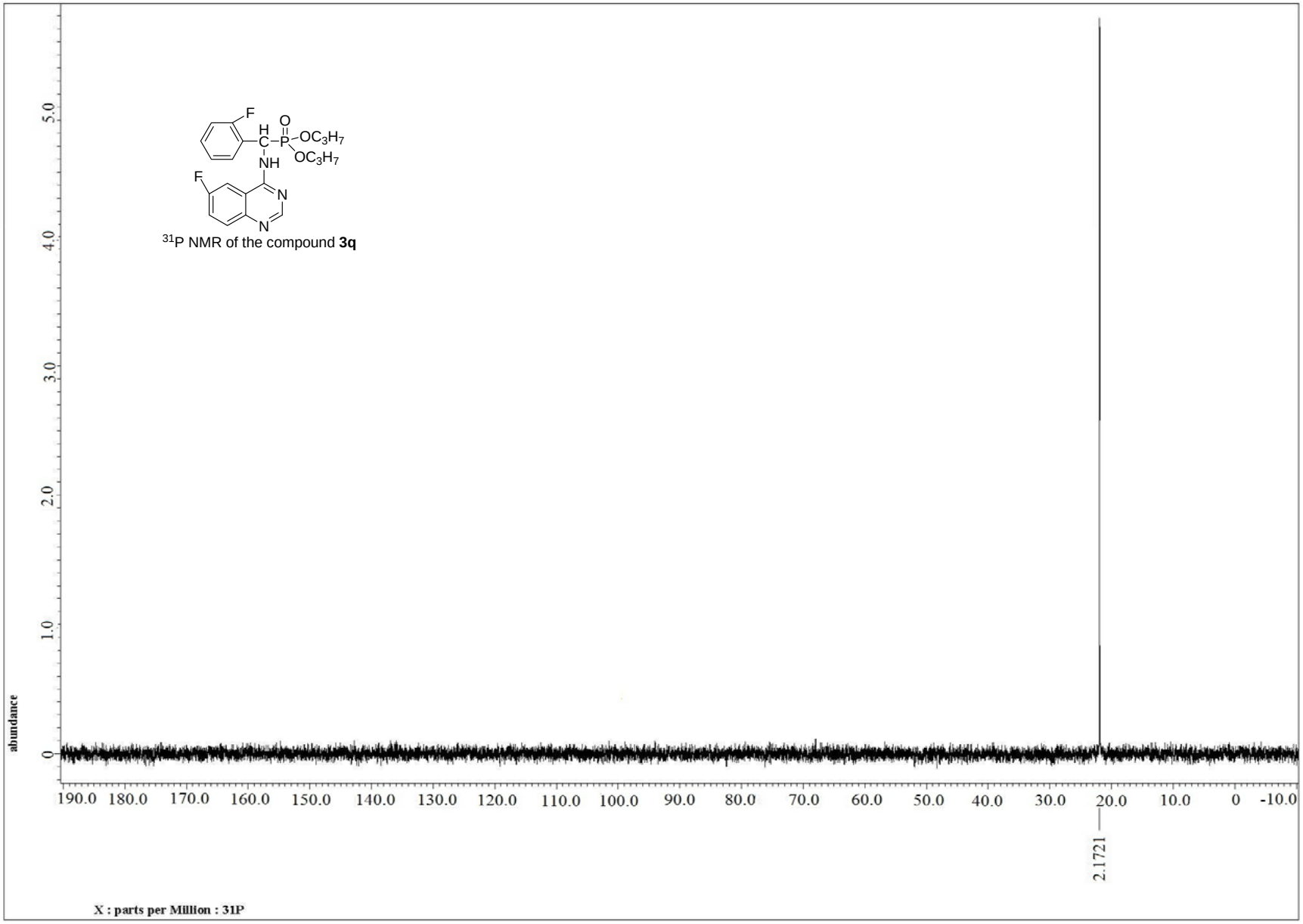

abundance
X : parts per Million : 13C
160.9385
158.9736
154.4810
146.8409
135.3186
130.7783
130.7115
128.6513
128.6036
128.1266
122.4037
122.2034
106.7703
106.5891
77.3637
77.1061
76.8486
63.5713
63.4759
51.9822
50.7422
16.4996
16.2135
63.6285
63.5713
63.4759
63.4187
16.4996
16.4615
16.2135
16.1658



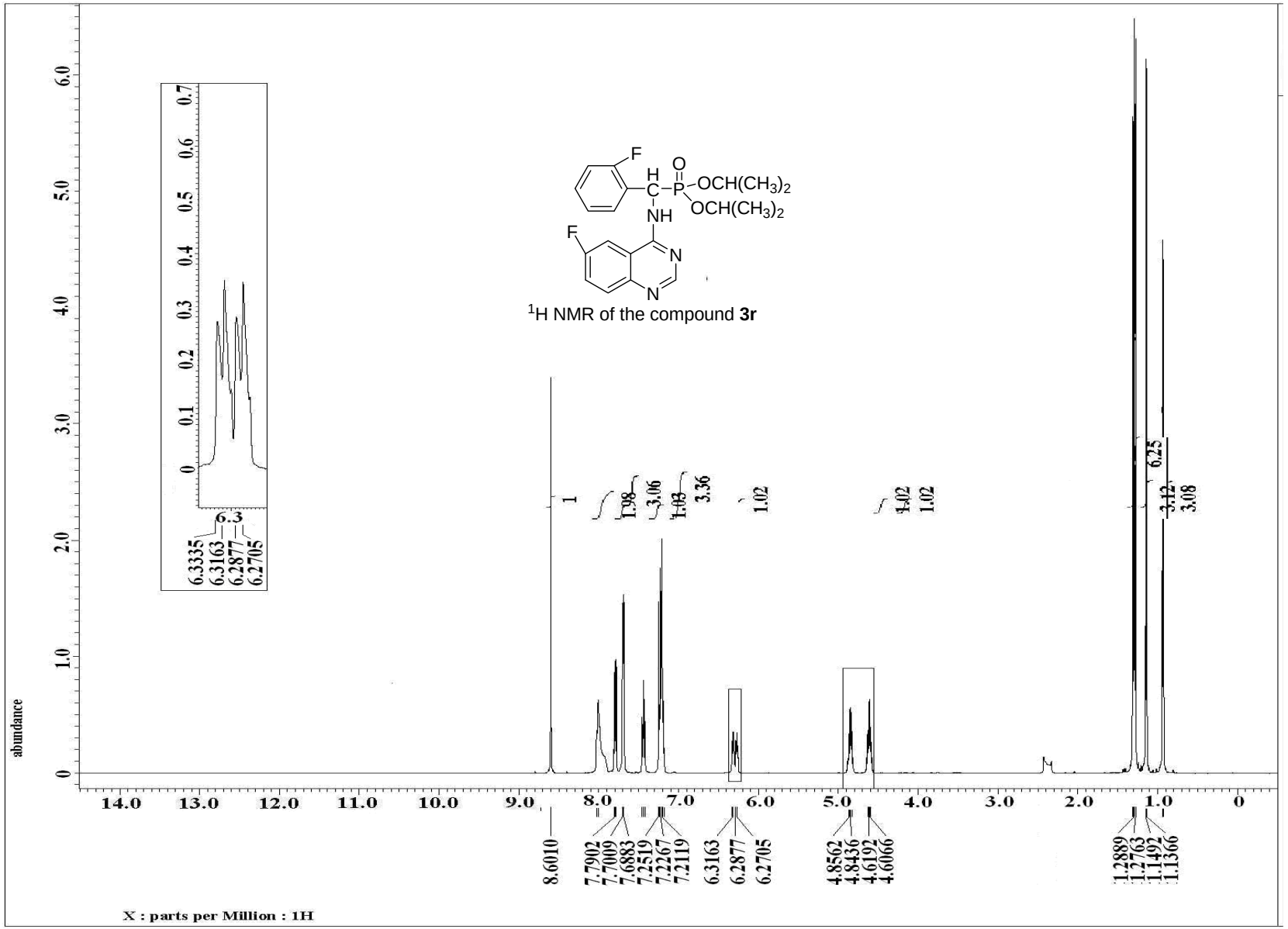
^{1}H NMR of the compound **3q**



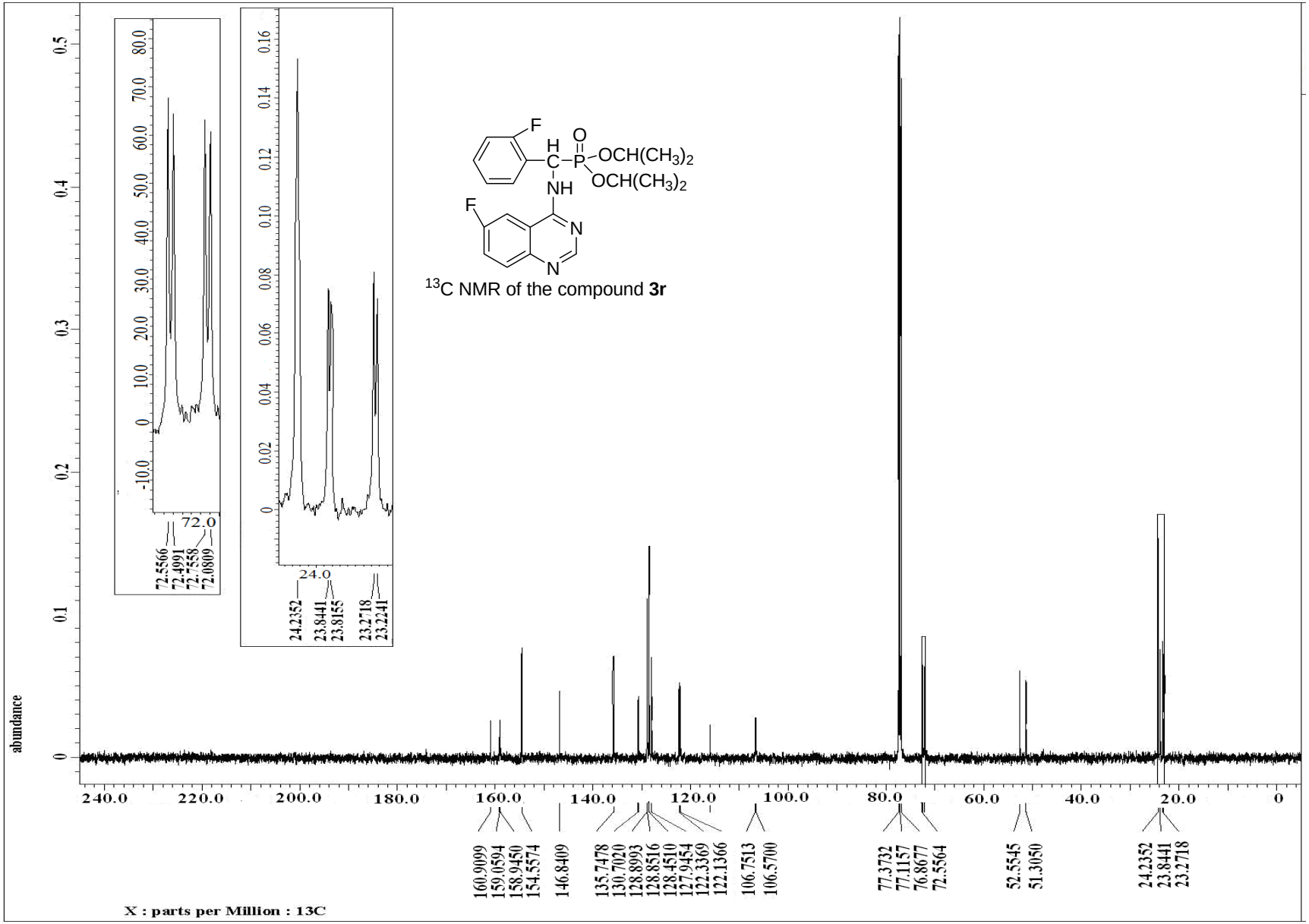
^{13}C NMR of the compound **3q**



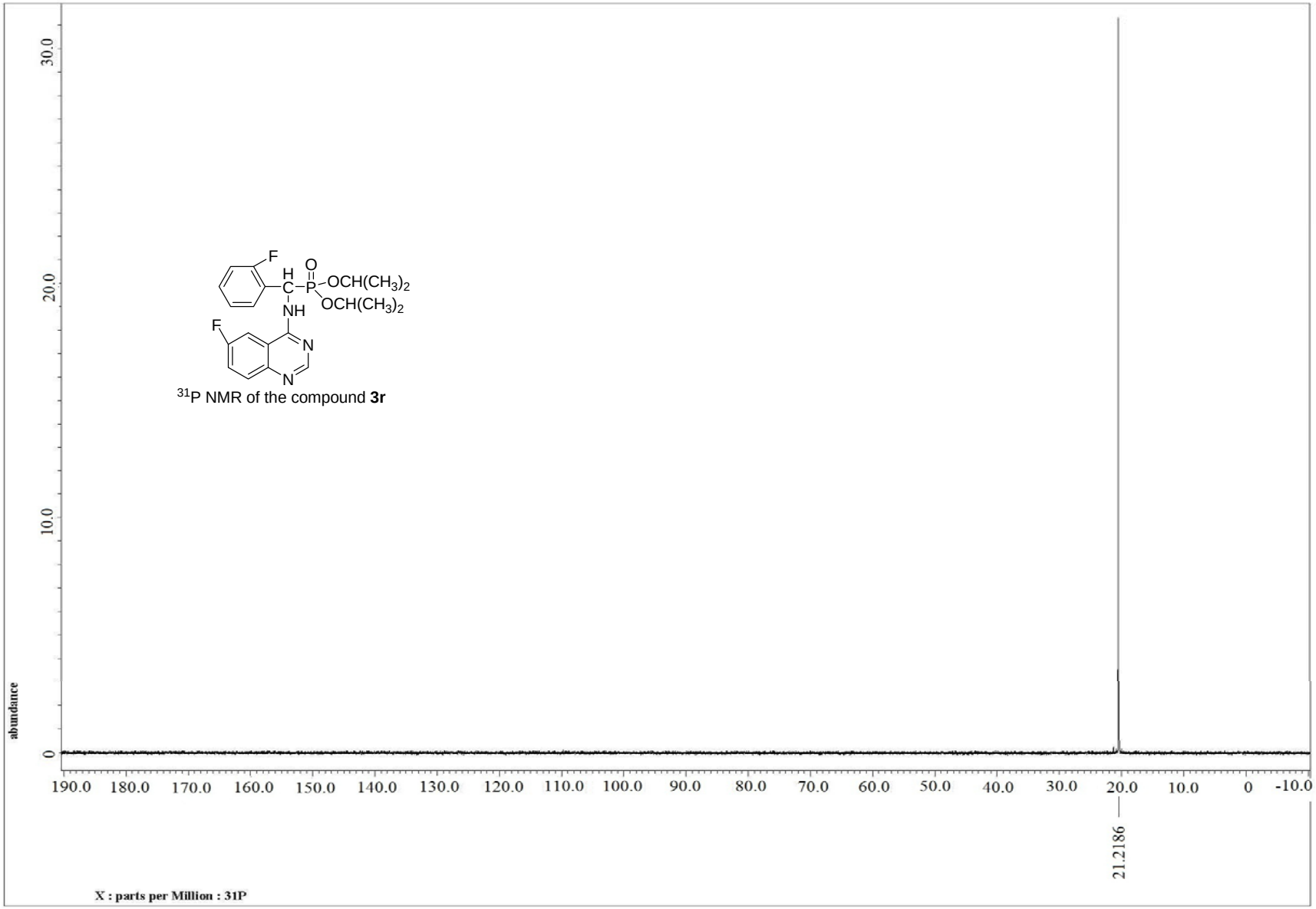
^{31}P NMR of the compound **3q**



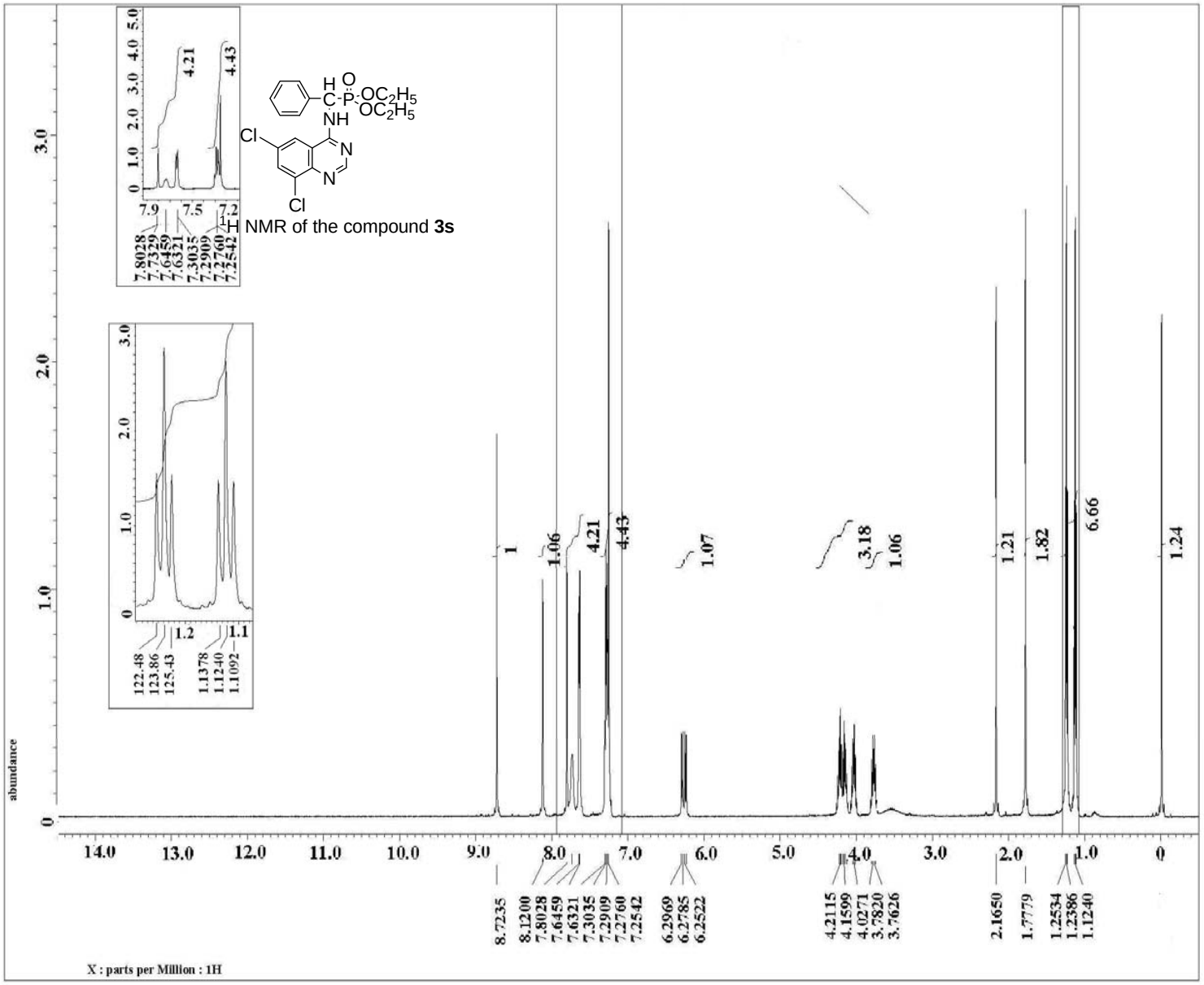
^{1}H NMR of the compound **3r**



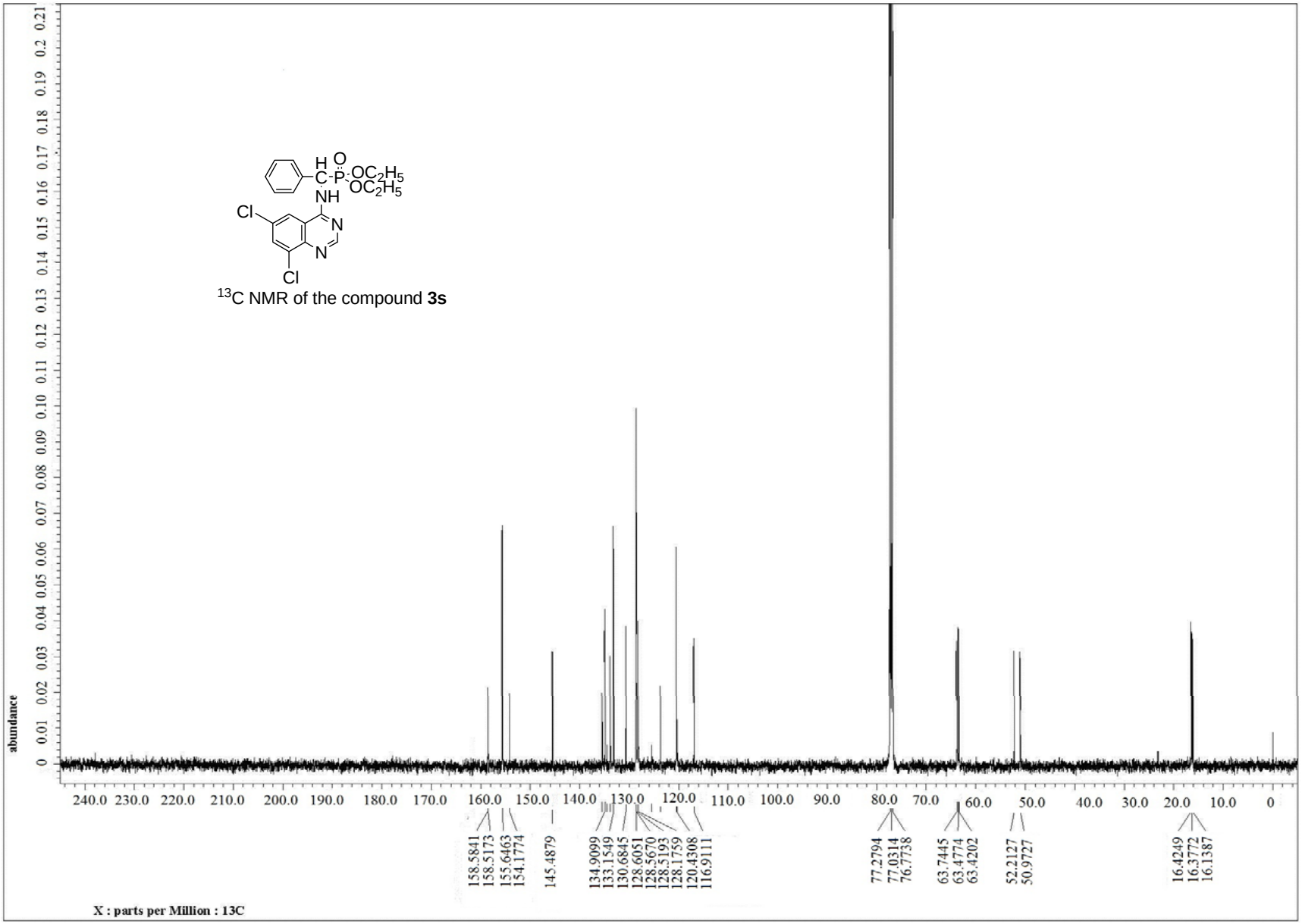
^{13}C NMR of the compound **3r**



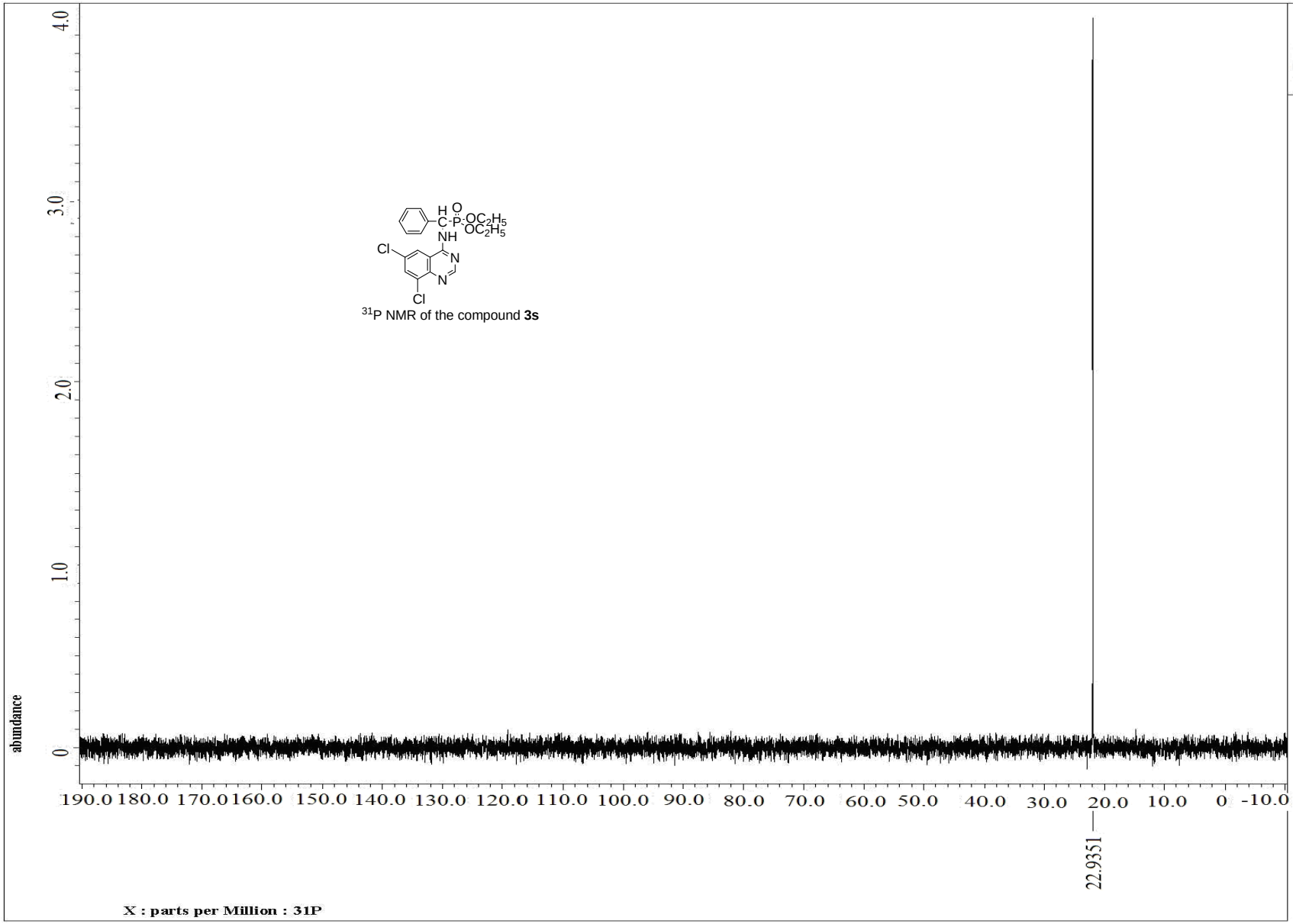
^{31}P NMR of the compound **3r**



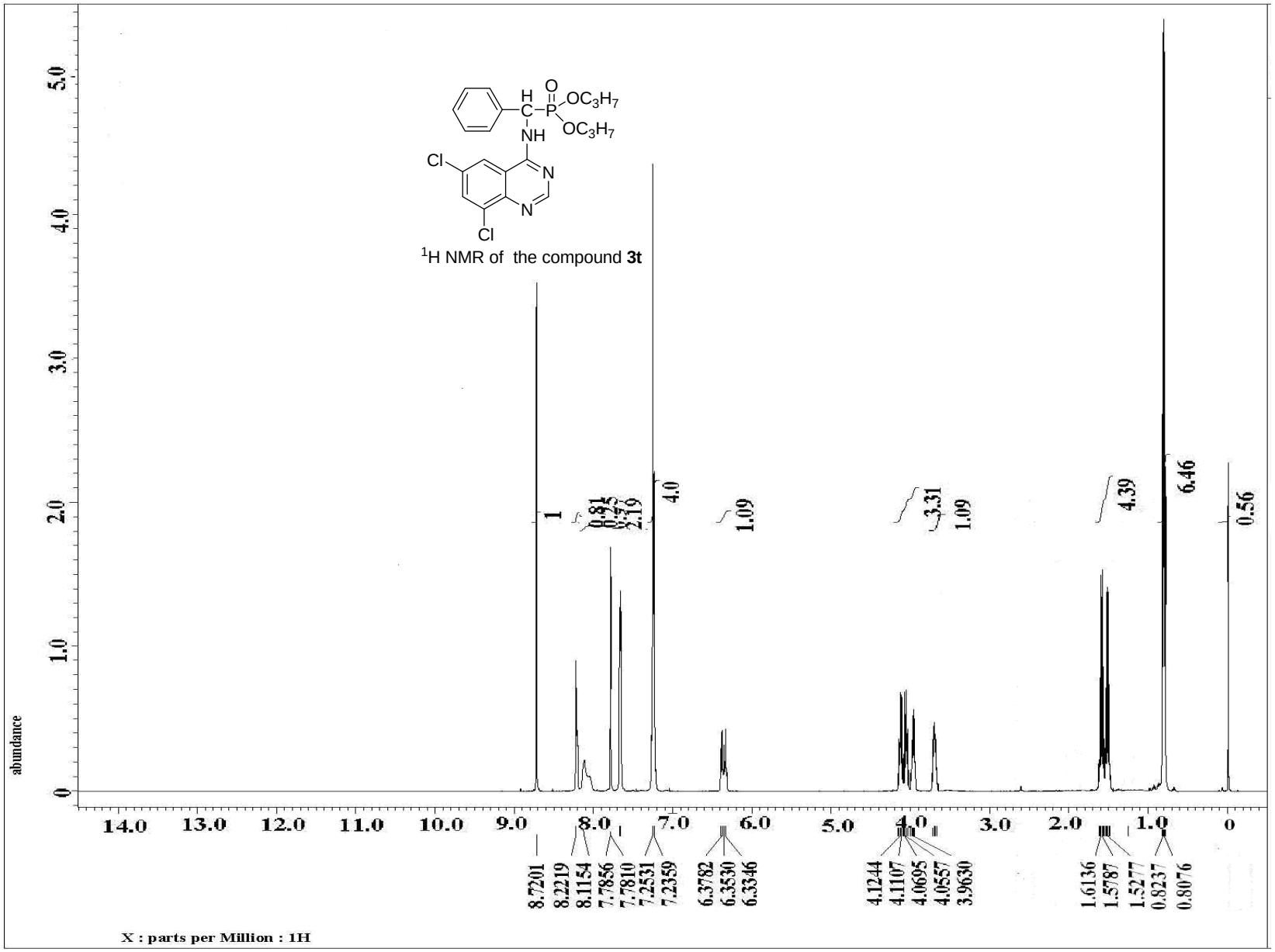
^{1}H NMR of the compound **3s**



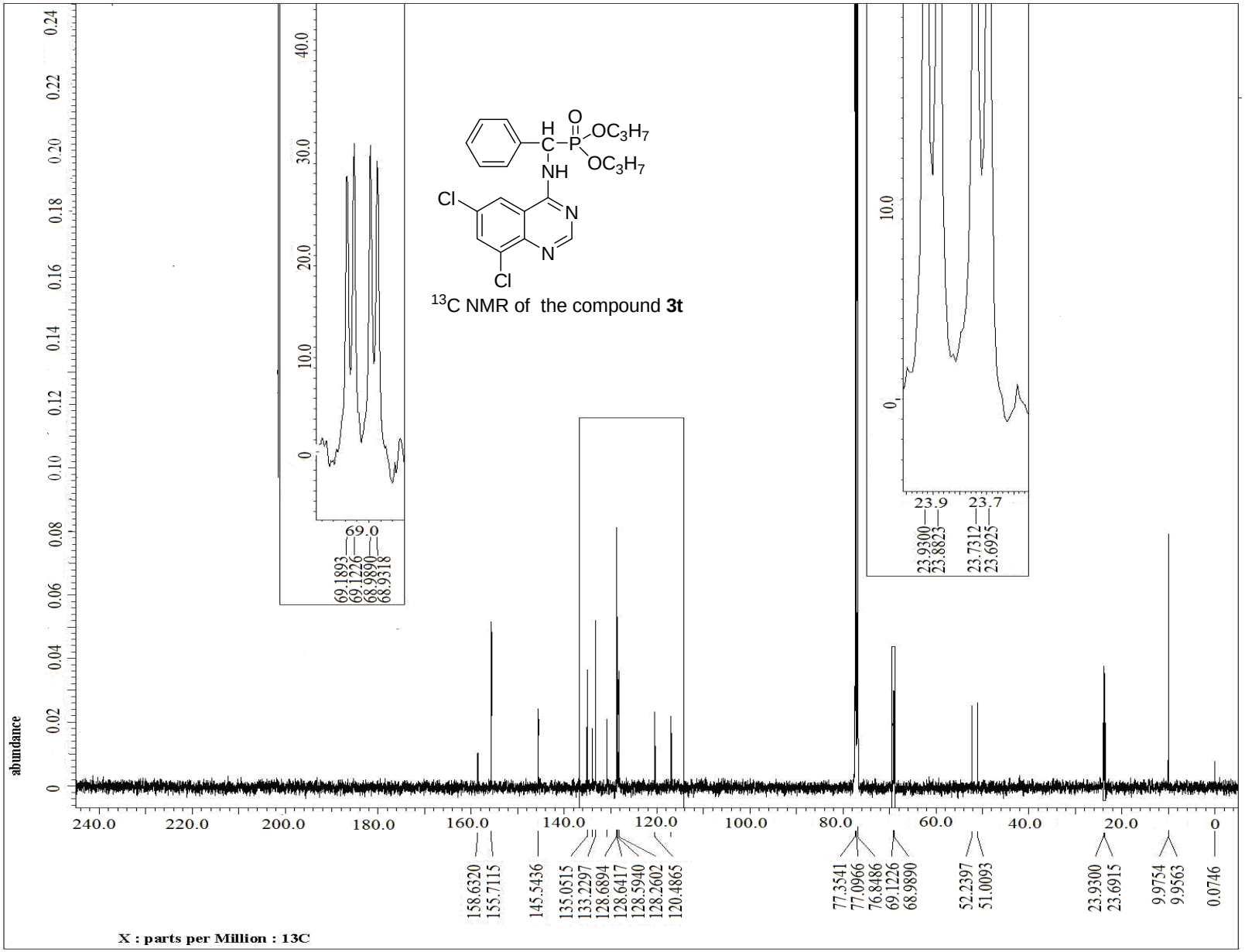
^{13}C NMR of the compound **3s**



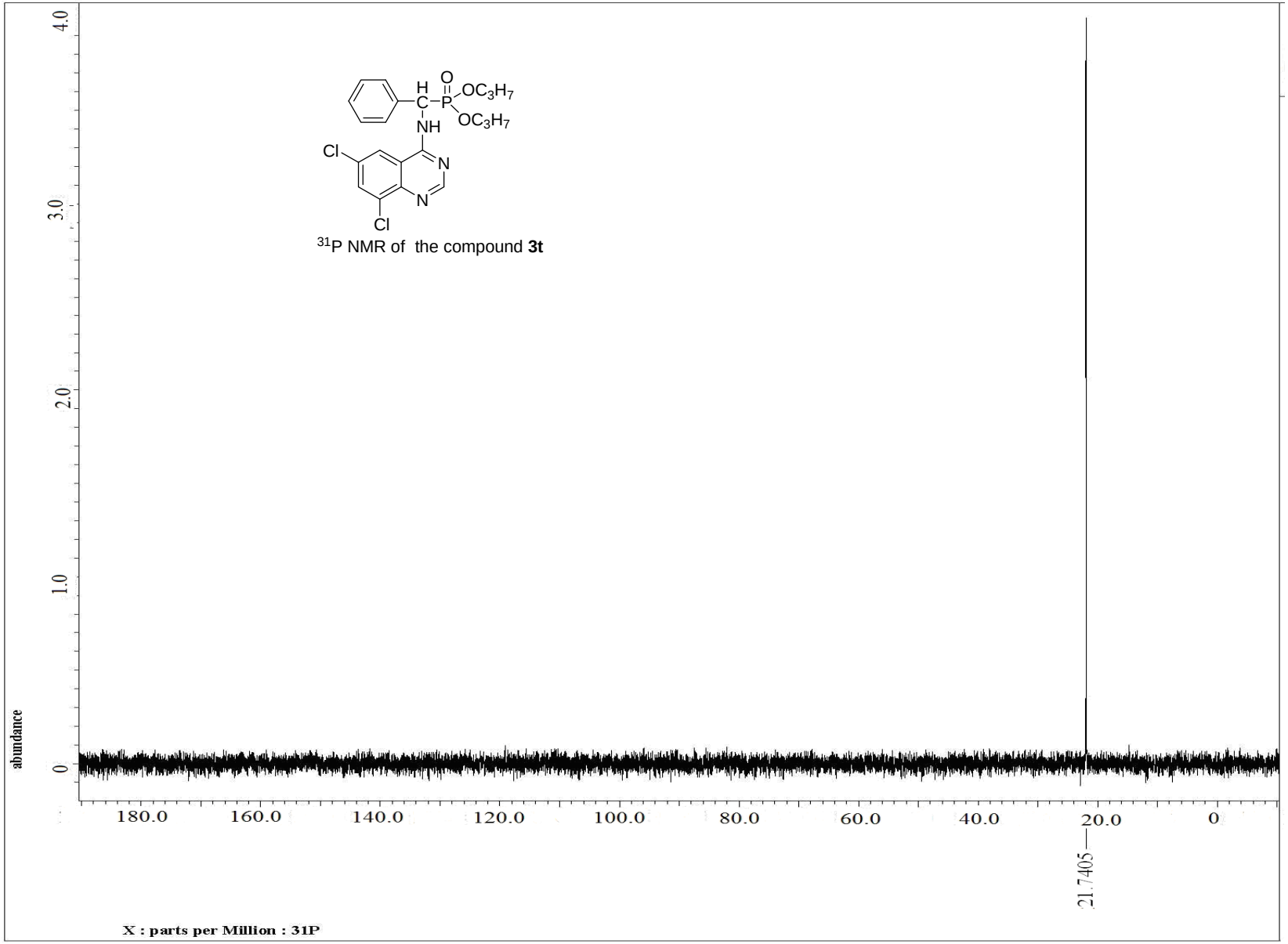
^{31}P NMR of the compound **3s**



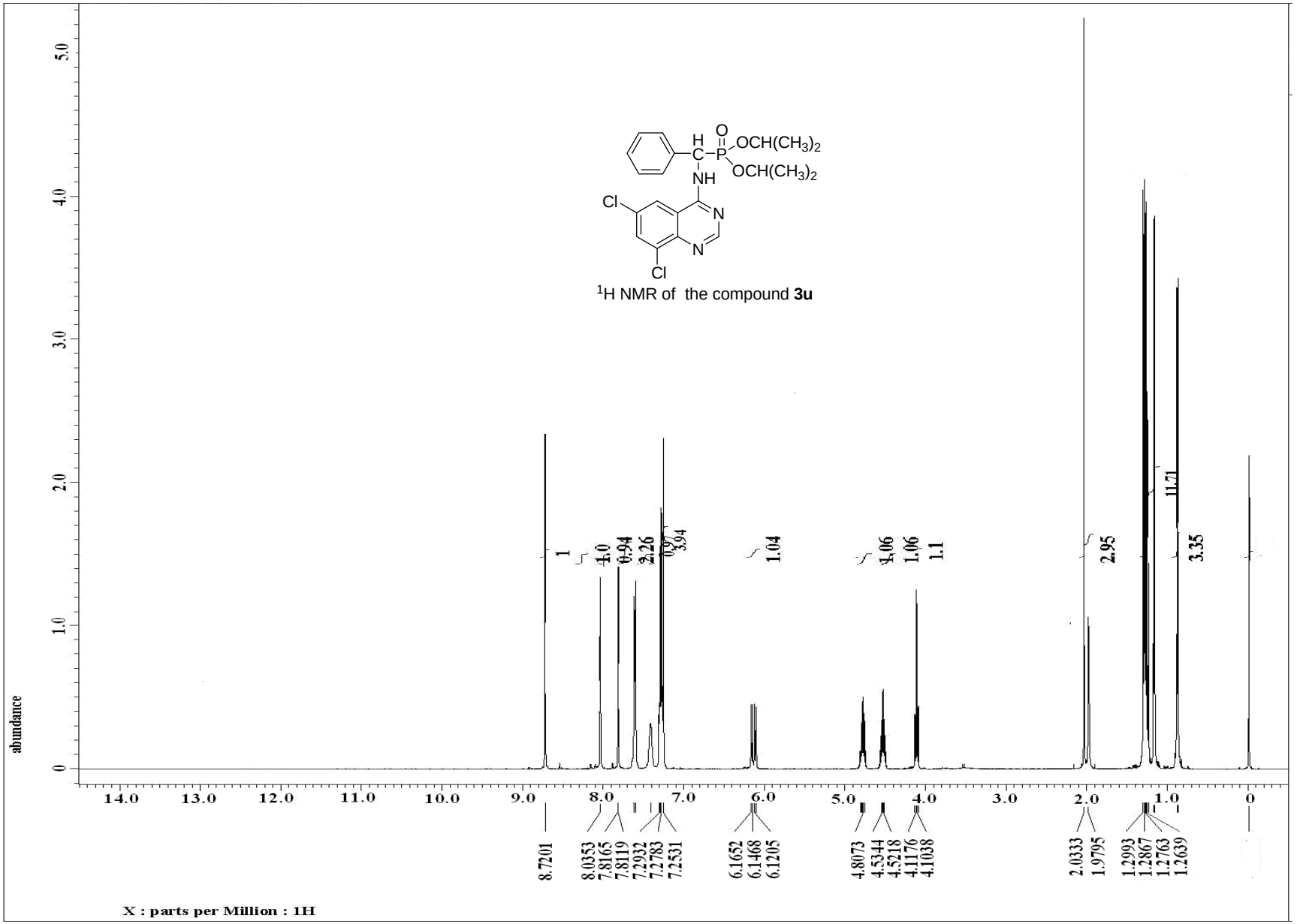
^{1}H NMR of the compound **3t**



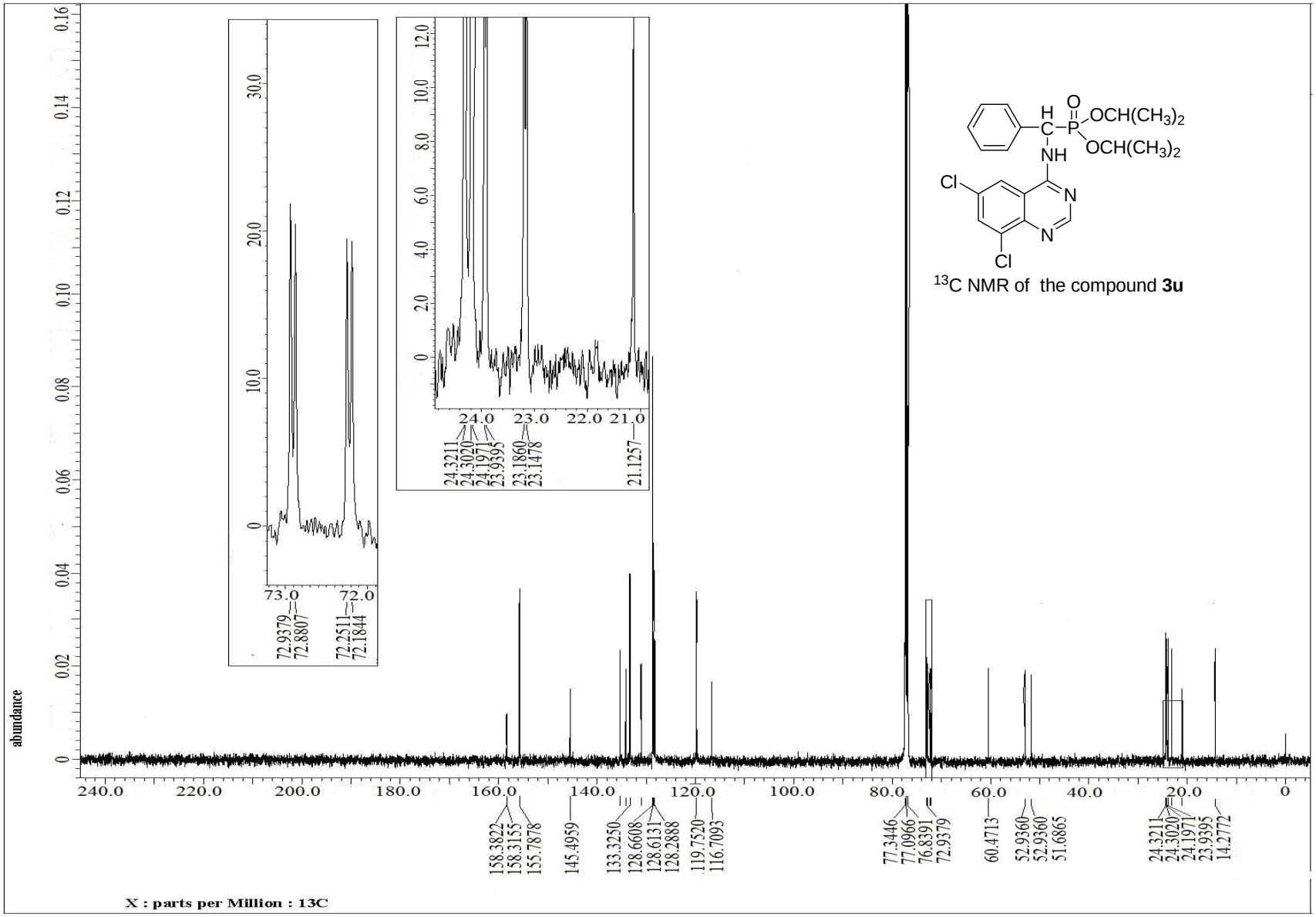
^{13}C NMR of the compound **3t**



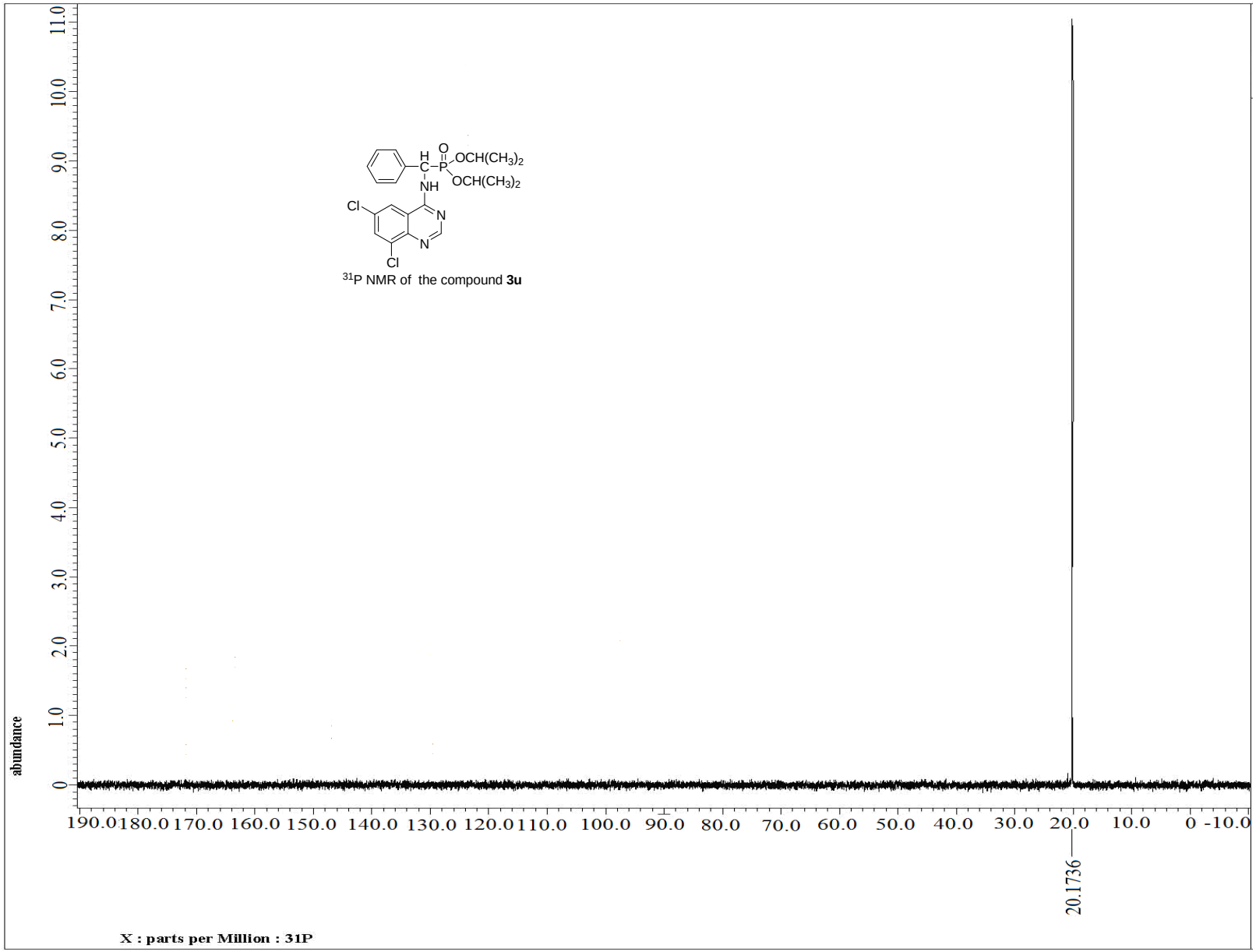
^{31}P NMR of the compound **3t**



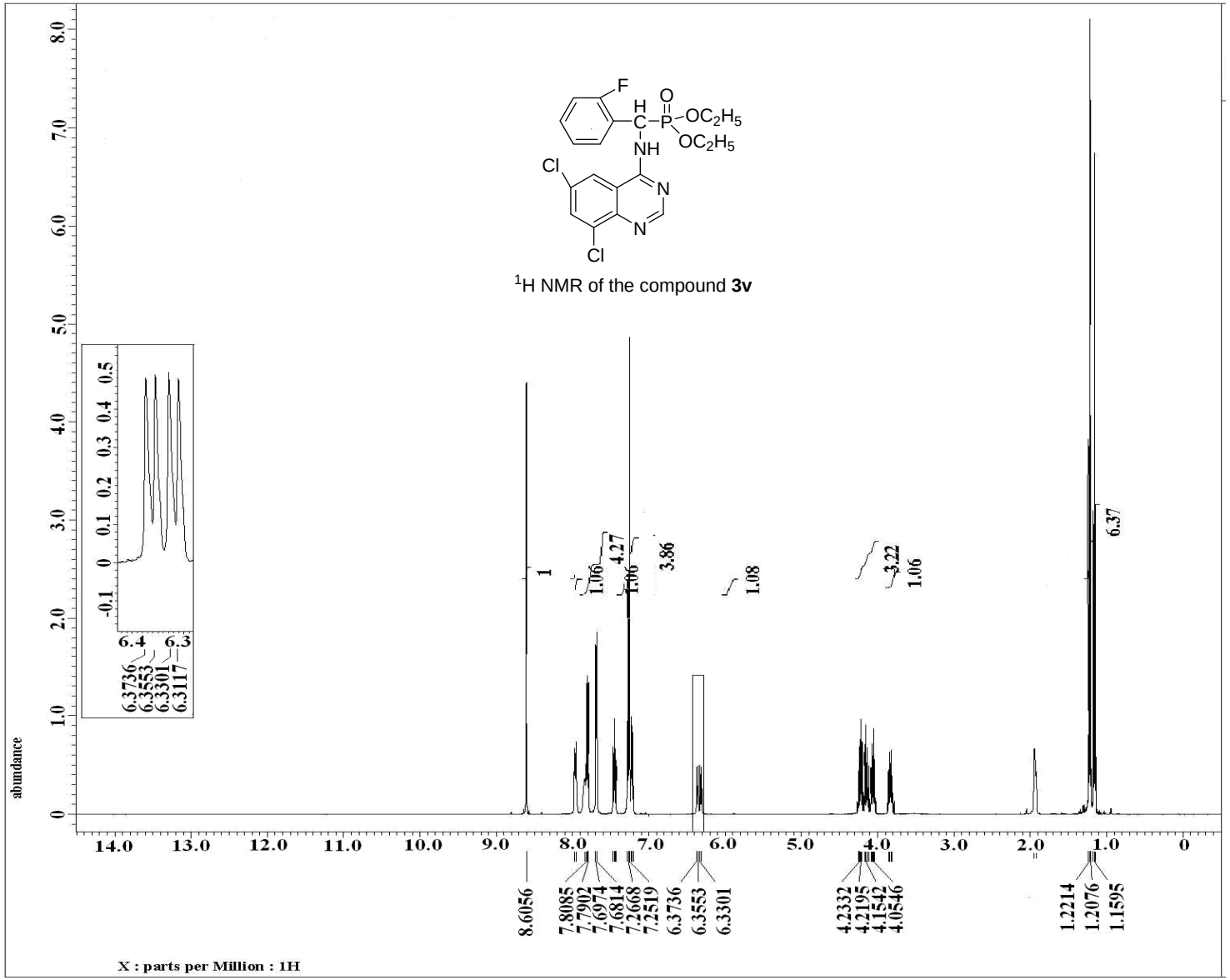
^{1}H NMR of the compound **3u**



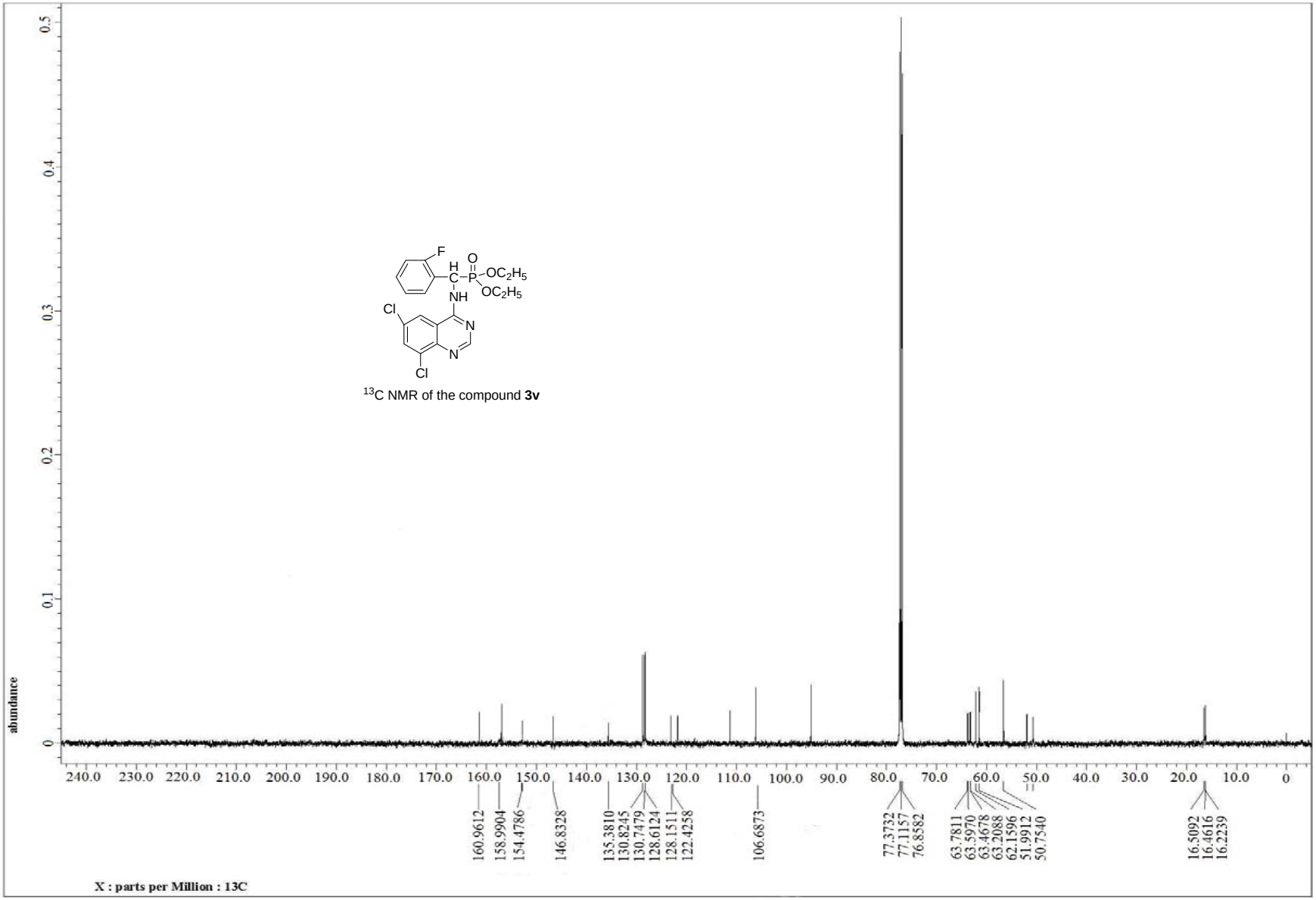
^{13}C NMR of the compound **3u**



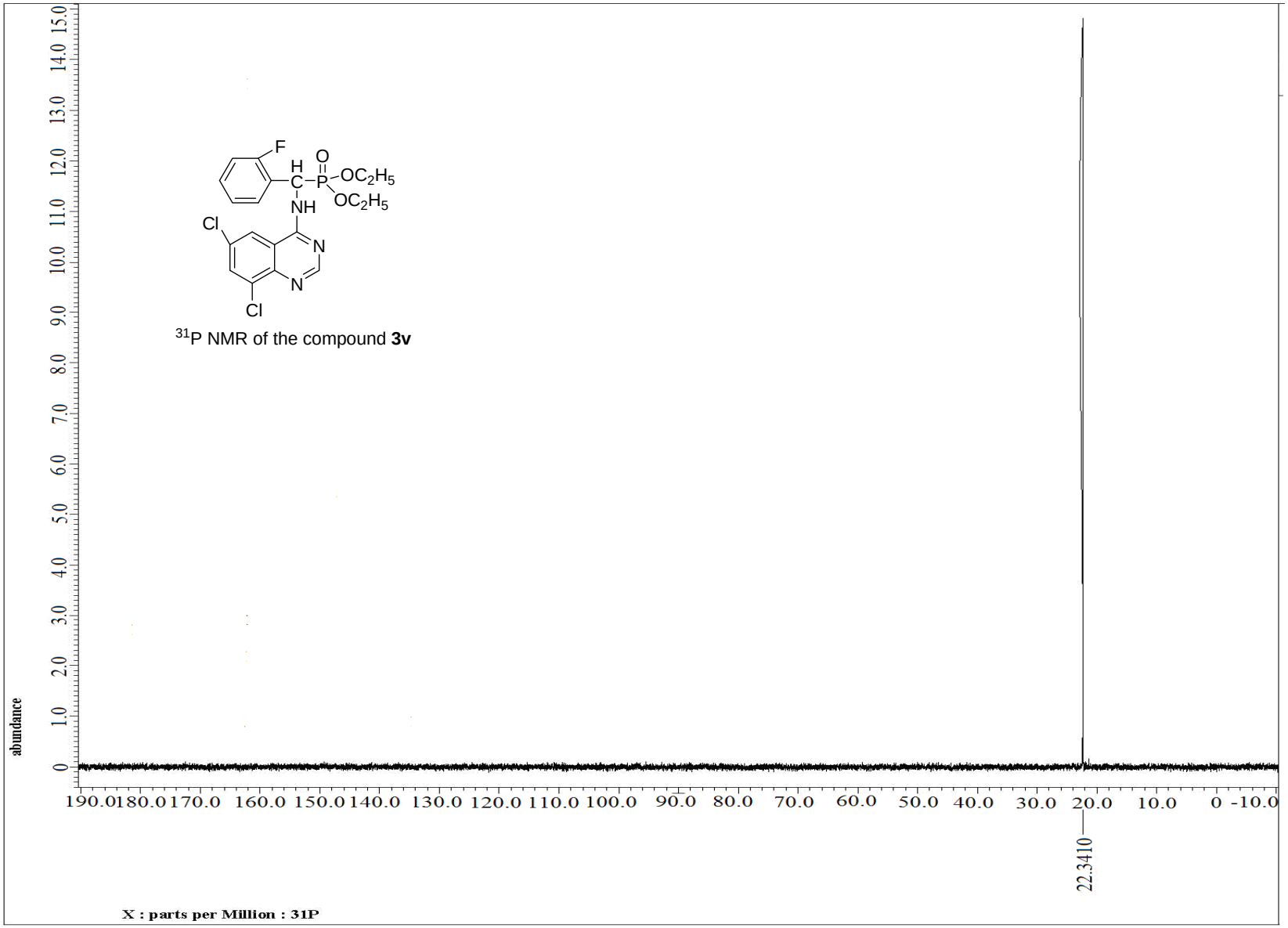
^{31}P NMR of the compound **3u**



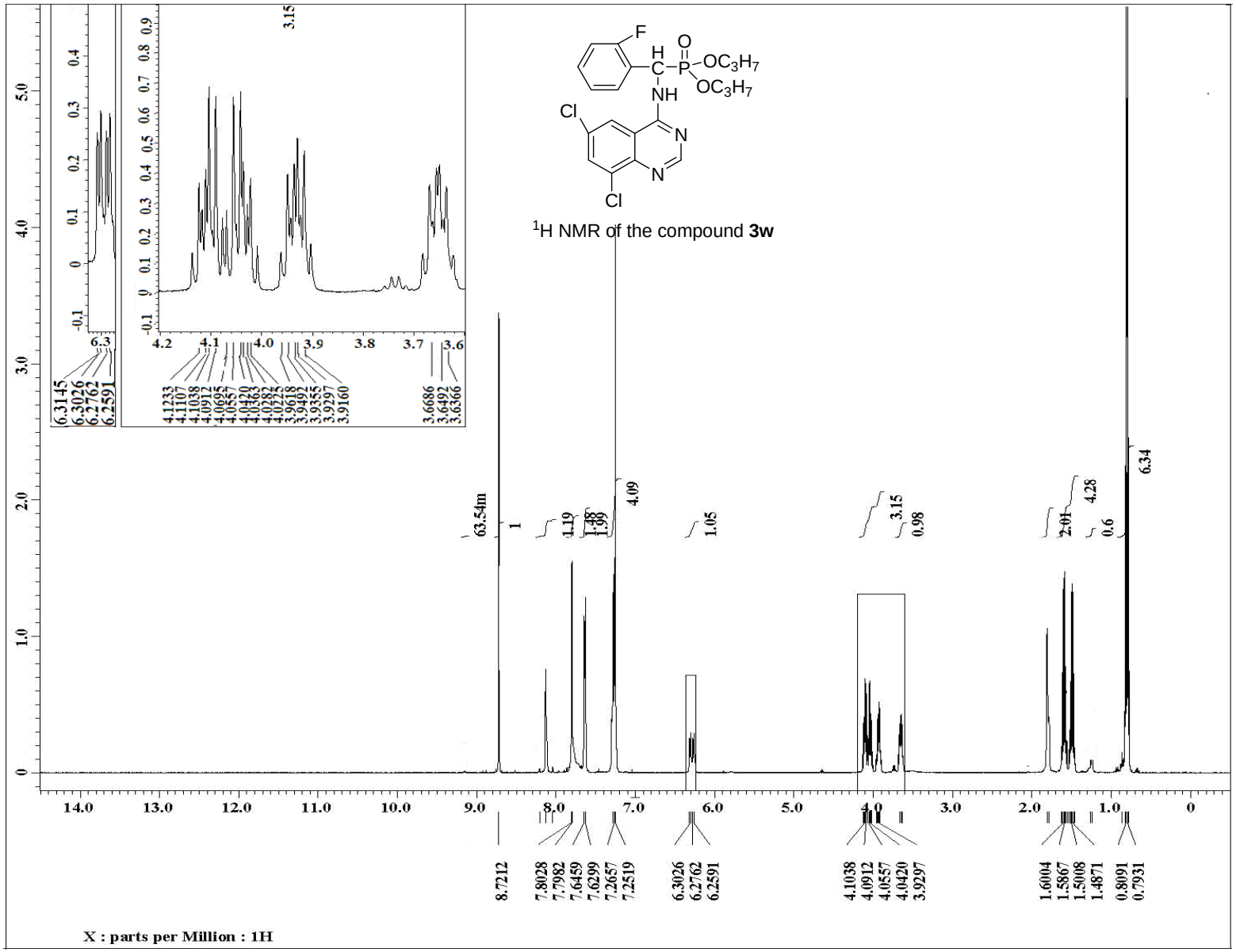
^{1}H NMR of the compound **3v**



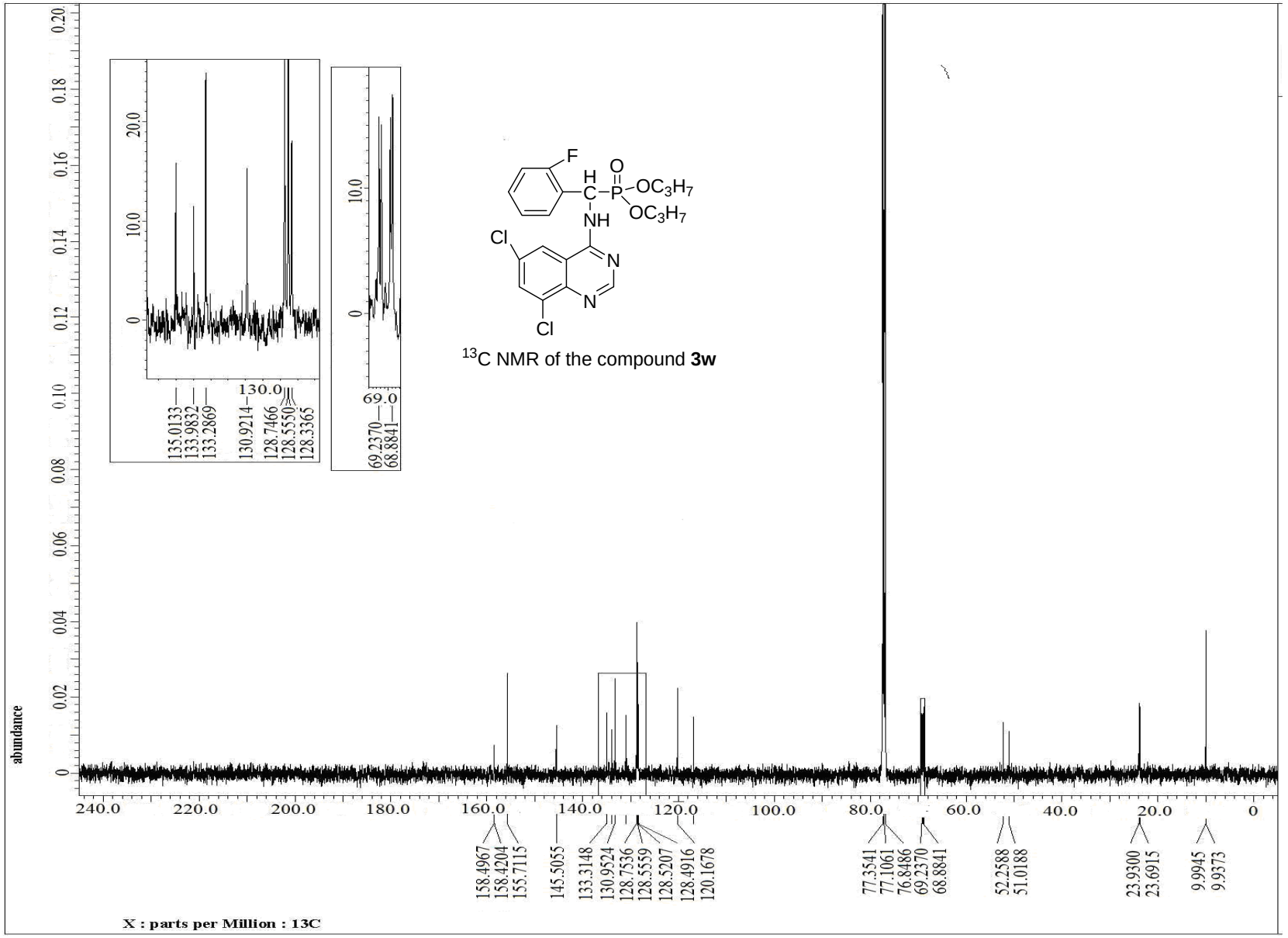
^{13}C NMR of the compound **3v**



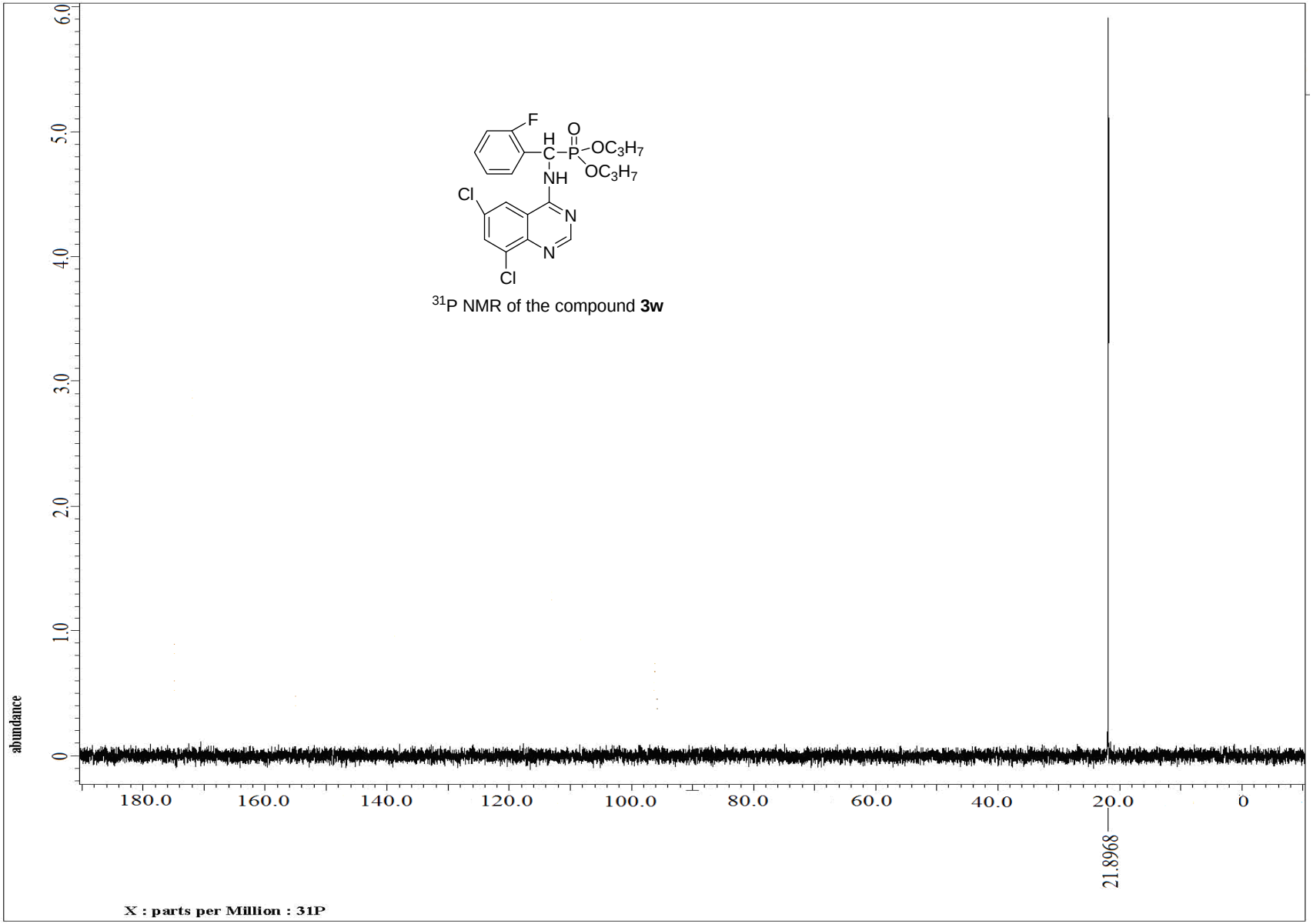
^{31}P NMR of the compound **3v**



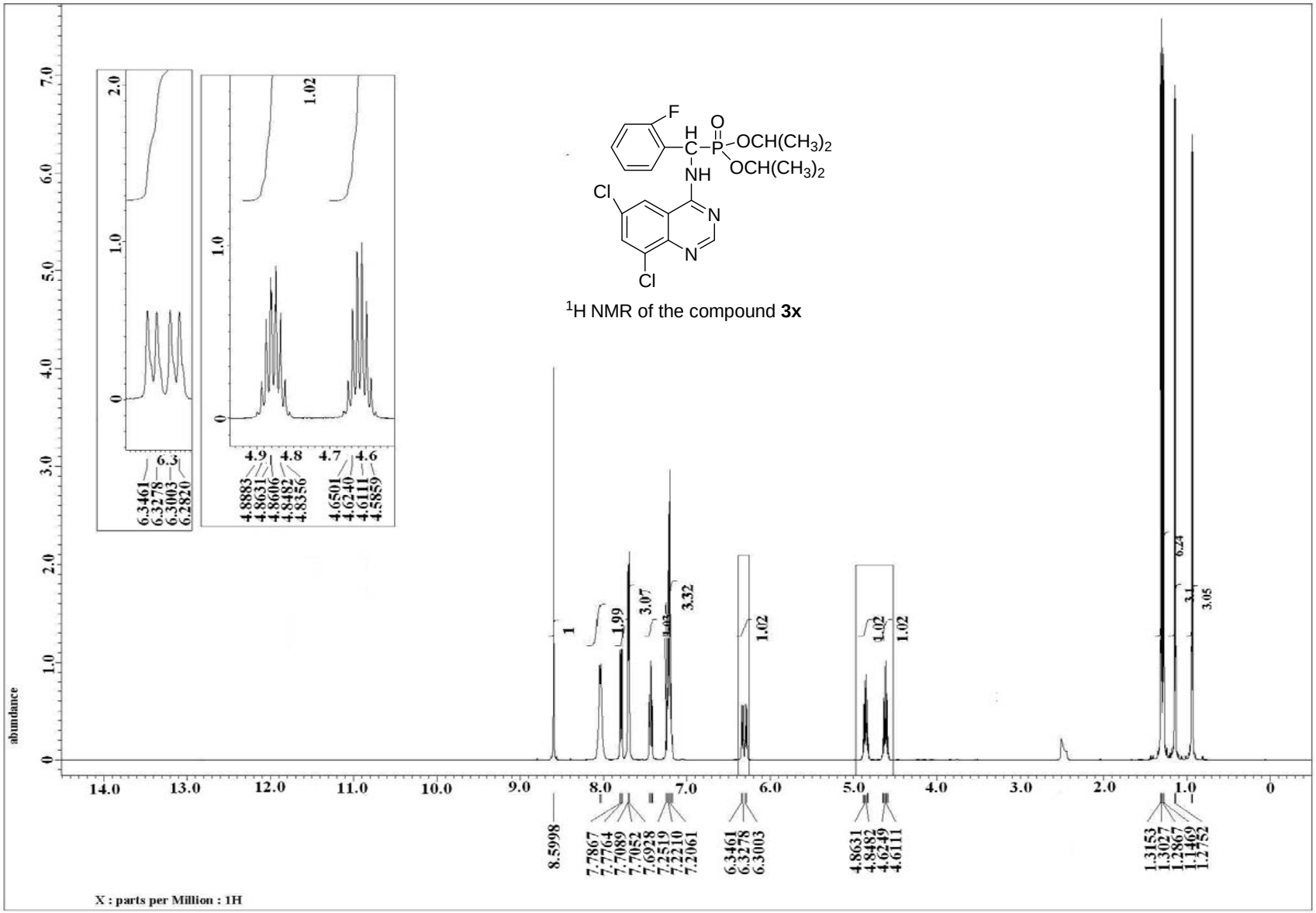
^{1}H NMR of the compound **3w**



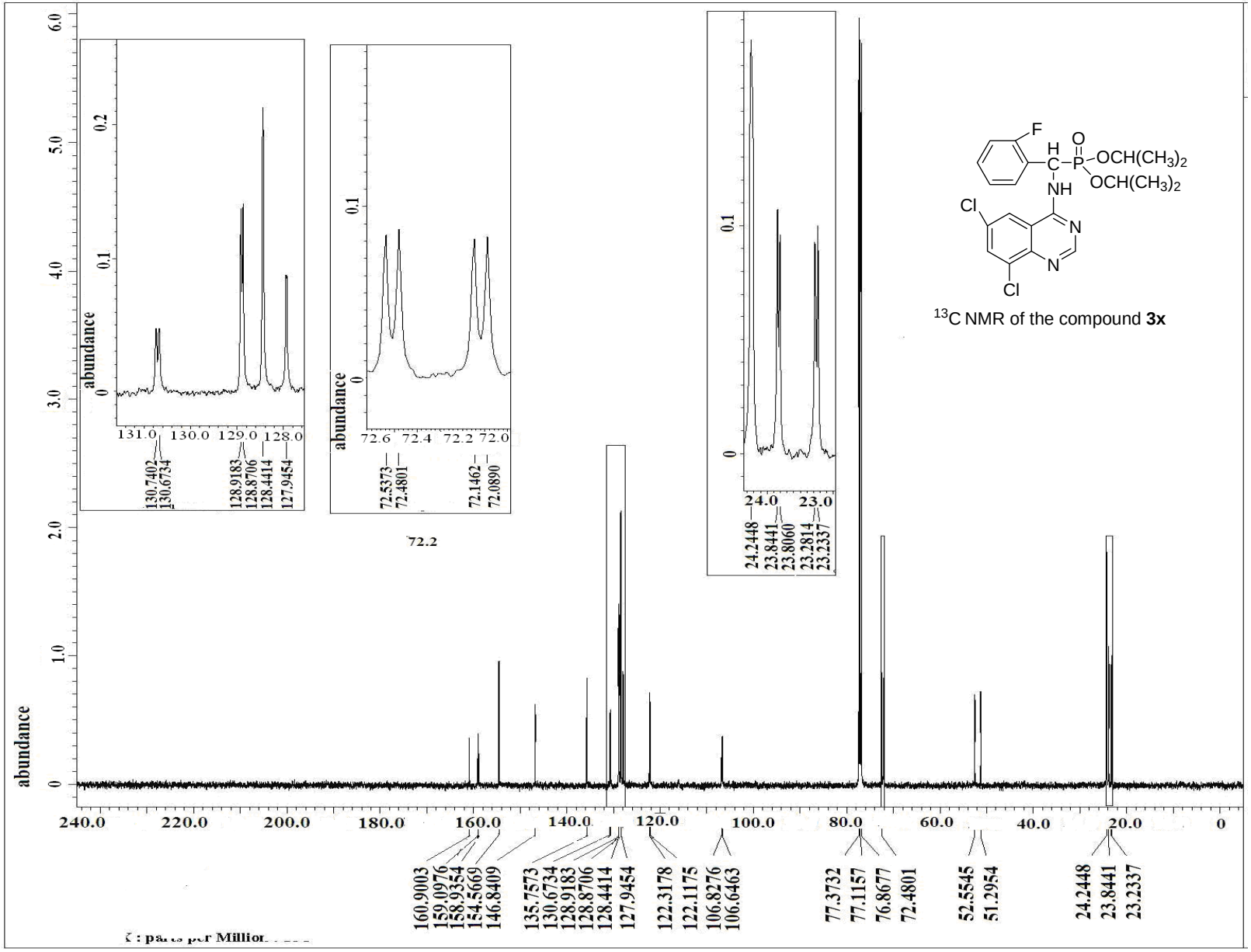
^{13}C NMR of the compound **3w**



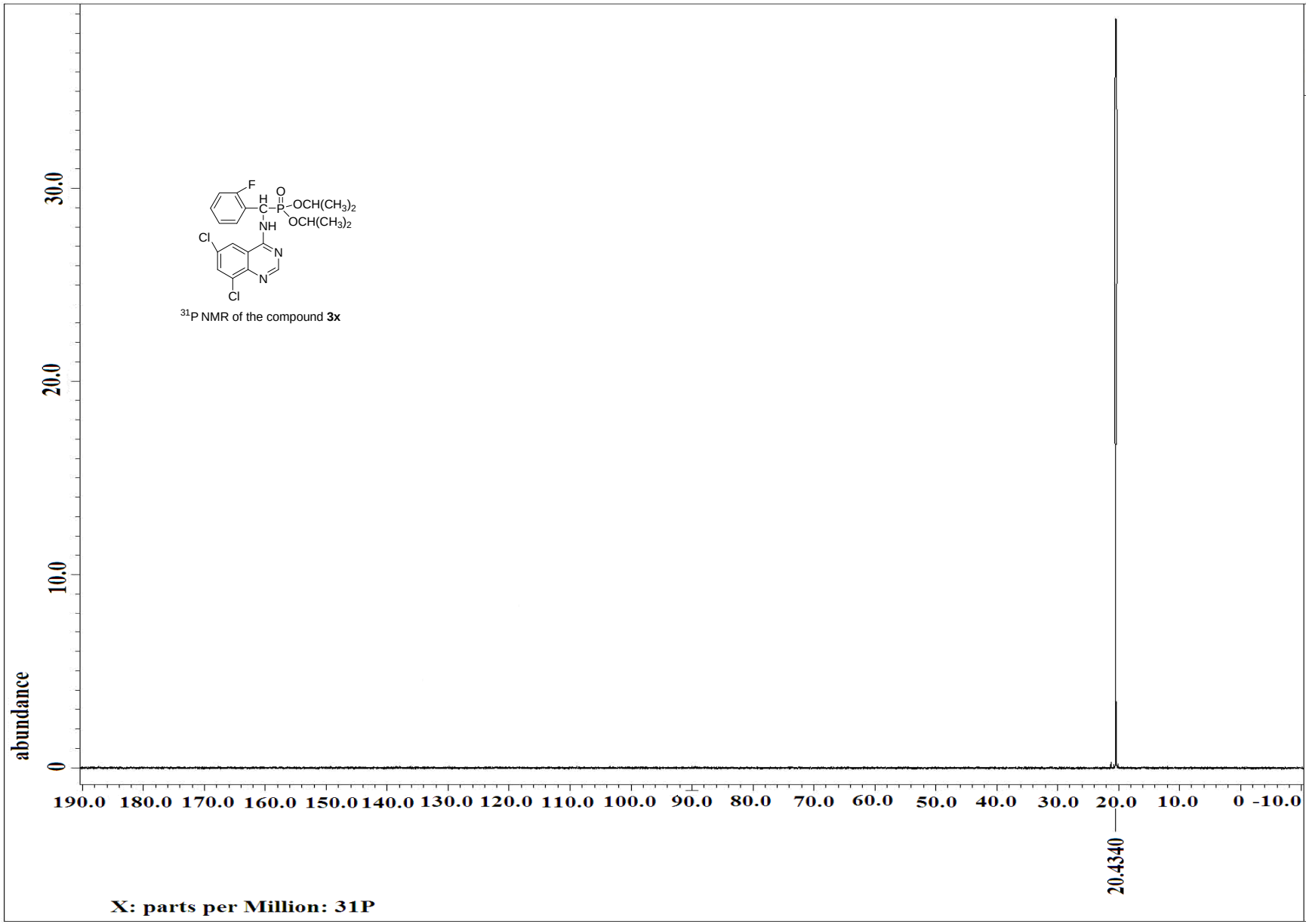
^{31}P NMR of the compound **3w**



^{1}H NMR of the compound **3x**



^{13}C NMR of the compound **3x**



^{31}P NMR of the compound **3x**