

Reaction of 1-(2-Hydroxyphenyl)-3-Phenylpropane-1,3-Dione With Some Phosphorus Halides: A Simple Synthesis of Novel 1,2-Benzoxaphosphinines

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(A) Full experimental and spectroscopic data for the synthesized compounds :

General method for reaction compound 1 with phenyl phosphorus halides: Synthesis of 2-aryl-3-benzoyl-4-hydroxy-2H-1,2-benzoxaphosphinines 2-4.

P,P-Dichlorophenylphosphine, phenyl phosphonic dichloride and phenyl phosphorodichloridate (5 mmol) was added individually to a solution of compound 1 (5 mmol, 1.20 g) in dry toluene (30 mL) in the presence of triethylamine (10 mmol, 1.4 mL) as a base, under stirring for 30 minutes at 10 °C then heated under reflux for 8-12 hours. The formed solids were filtered off, washed with water and crystallized from diluted EtOH.

3-Benzoyl-4-hydroxy-2-phenyl-2H-1,2-benzoxaphosphinine (2). Orange solid in 59% yield; mp 226–228 °C; IR (KBr), ν 3390 (OH), 3057 (C–H_{Ar}), 1660 (C=O), 1619, 1591 (C=C), 1024 (O–C); ¹H-NMR (400 MHz, DMSO-*d*₆): δ 7.44 (t, 1H, *J*=7.6 Hz, H–6), 7.55–7.70 (m, 6H, Ph–H), 7.79 (d, 1H, *J*=6.8 Hz, H–8), 7.89–7.94 (m, 2H, Ph–H), 8.00 (t, 1H, *J*=6.0 Hz, H–7), 8.12 (d, 1H, *J*=8.0 Hz, H–5), 8.16 (d, 2H, *J*=7.2 Hz, Ph–H), 12.63 (br, 1H, OH); ¹³C-NMR (100 MHz, DMSO-*d*₆): δ 117.3 (C–8), 119.5 (d, *J*=97 Hz, C–1^{''}), 122.1 (C–4[^]), 127.3 (C–4a), 128.2 (C–3[^], 5[^]), 128.5 (C–5), 129.3 (d, *J*=132 Hz, C–3), 129.4 (C–2[^], 6[^]), 130.5 (C–3^{''}, 5^{''}), 131.3 (C–1[^]), 132.2 (C–6), 133.2 (C–2^{''}, 6^{''}), 133.5 (C–7), 137.3 (C–4^{''}), 156.9 (C–8a), 159.5 (C–4), 166.6 (C=O); MS *m/z*, (%) 346 (M⁺, 2%). Anal. Calcd for C₂₁H₁₅O₃P (346.33): C, 72.83; H, 4.37%. Found: C, 72.51; H, 4.09%.

3-Benzoyl-4-hydroxy-2-oxido-2-phenyl-2H-1,2-benzoxaphosphinine (3). Beige solid in 72% yield; mp 149–151 °C. IR (KBr), ν 3008 (br, OH), 1687 (C=O), 1601, 1581 (C=C), 1292

(P=O), 1026, 1071 (O–C); ¹H-NMR (400 MHz, DMSO-*d*₆): δ 7.47 (t, 1H, *J*=7.6 Hz, H–6), 7.55–7.70 (m, 7H, H–8 and Ph–H), 7.90–7.94 (m, 3H, H–7 and Ph–H), 8.13 (d, 1H, *J*=7.6 Hz, H–5), 8.17 (d, 2H, *J*=7.2 Hz, Ph–H), 12.92 (br, 1H, OH); ¹³C-NMR (100 MHz, DMSO-*d*₆): δ 117.3 (C–8), 121.8 (C–4'), 124.6 (d, *J*=86 Hz, C–1''), 127.3 (C–4a), 128.3 (C–5), 128.8 (d, *J*=60 Hz, C–3), 128.9 (C–3',5'), 129.4 (C–1'), 129.7 (C–2',6'), 130.0 (C–3'',5''), 130.5 (C–6), 131.1 (C–7), 133.2 (C–2'',6''), 137.3 (C–4''), 156.9 (C–8a), 159.3 (C–4), 167.7 (C=O). ³¹P-NMR (162 MHz, DMSO-*d*₆): δ 38.42; MS *m/z*, (%) 362 (M⁺, 16%). Anal. Calcd for C₂₁H₁₅O₄P (362.33): C, 69.62; H, 4.17. Found: C, 69.23; H, 3.88%.

3-Benzoyl-4-hydroxy-2-oxido-2-phenoxy-2*H*-1,2-benzoxaphosphinine (4). Beige solid in 66% yield; mp 149–151 °C. IR (KBr), ν 3158 (br, OH), 3091, 3024 (C–HAr), 1646 (C=O), 1600, 1581 (C=C), 1281 (P=O), 1012, 1039 (O–C); ¹H-NMR (400 MHz, DMSO-*d*₆): δ 6.90–6.93 (m, 2H, Ph-H), 7.48 (t, 1H, *J*=7.2 Hz, H–6), 7.58–7.71 (m, 6H, and Ph–H), 7.77 (d, 1H, *J*=8.0 Hz, H–8), 7.93 (t, 1H, *J*=8.0 Hz, H–7), 8.13 (d, 1H, *J*=8.0 Hz, H–5), 8.18 (d, 2H, *J*=7.2 Hz, Ph–H), 12.15 (br, 1H, OH); ¹³C-NMR (100 MHz, DMSO-*d*₆): δ 117.3 (C–8), 117.5 (C–3'',5''), 119.6 (C–4'), 127.3 (C–4a), 128.2 (C–3',5'), 128.5 (C–5), 129.4 (C–2',6'), 129.8 (d, *J*=165 Hz, C–3), 130.5 (C–6), 131.7 (C–1'), 133.1 (C–4''), 136.1 (C–7), 137.3 (C–2'',6''), 159.4 (C–8a), 156.9 (C–1''), 161.5 (C–4), 172.3 (C=O); MS *m/z*, (%) 378 (M⁺, 10%). Anal. Calcd for C₂₁H₁₅O₅P (378.32): C, 66.67; H, 4.00. Found: C, 66.34; H, 3.71%.

Conversion of compound 2 into the corresponding oxide analogue 3.

A solution of compound **2** (0.1 g) in tetrahydrofuran (10 ml) and aqueous hydrogen peroxide (30%, 0.25 mL) was stirred for 8 hours at room temperature. The reaction mixture was concentrated to its third volume, and then added diethyl ether (20 mL). The isolated precipitate **3** was filtered off and dried to give beige solid in 48% yield; mp 150–151 °C.

Synthesis of 3-benzoyl-2,4-dihydroxy-2-oxido-2*H*-1,2-benzoxaphosphinine (5).

Method A. A solution of phosphorus oxychloride (5 mmol, 0.6 mL) in dry toluene, was added to a solution of compound **1** (5 mmol, 1.20 g) in dry toluene (30 mL) in the presence of triethylamine (10 mmol, 1.4 mL) as a base, under stirring for 30 minutes at 10 °C then heated

under reflux for 10 hours. The oily product was heated in distilled water (20 mL). The formed solid was filtered off, washed with water and crystallized from methanol to give green solid in 85% yield; mp 91–93 °C.

Method B. Compound **6** (0.1 g) was warmed in aqueous solution of sodium carbonate (10 mL, 10%) for 1 hour. The formed solid was filtered off, washed with water and crystallized from methanol to give green solid in 54% yield; mp 91–92 °C. IR (KBr), ν 3389, 3148 (br, OH), 3056, 3002 (C–HAr), 1666 (C=O), 1619, 1592 (C=C), 1189 (P=O), 1024 (O–C); ¹H-NMR (400 MHz, DMSO-*d*₆): δ 3.54 (s, 1H, OH), 7.16 (t, 1H, *J*=7.6 Hz, H–6), 7.48–7.63 (m, 3H, Ph–H), 7.82 (t, 1H, *J*=8.0 Hz, H–7), 7.88 (d, 1H, *J*=8.4 Hz, H–8), 7.92 (d, 1H, *J*=6.8 Hz, H–5), 8.13–8.17 (m, 2H, Ph–H), 12.93 (s, 1H, OH); ¹³C-NMR (100 MHz, DMSO-*d*₆): δ 119.6 (C–8), 120.5 (C–4'), 123.1 (C–4a), 126.3 (C–5), 127.4 (C–3', 5'), 128.7 (d, *J*=86 Hz, C–3), 129.3 (C–2', 6'), 131.8 (C–1'), 132.5 (C–6), 133.0 (C–7), 152.9 (C–8a), 164.8 (C=O), 171.5 (C=O); MS *m/z*, (%) 302 (M⁺, 24%). Anal. Calcd for C₁₅H₁₁O₅P (302.23): C, 59.61; H, 3.67. Found: C, 59.29; H, 3.32%.

Synthesis of 2,2-dichloro-3-benzoyl-4*H*-1,2λ⁵-benzoxaphosphinin-4-one (6).

A solution of phosphorus pentachloride (5 mmol, 1.04 g) in dry toluene, was added to a solution of compound **1** (5 mmol, 1.20 g) in dry toluene (30 mL) in the presence of triethylamine (15 mmol, 2.1 mL) as a base, under stirring for 30 minutes at 10 °C then heated under reflux for 10 hours. The formed solid was filtered off, washed with ether and crystallized from petroleum ether to give yellow solid in 61% yield; mp 96–97 °C. IR (KBr), ν 3079 (C–HAr), 1695, 1657 (C=O), 1605, 1593 (C=C), 1072 (O–C); ¹H-NMR (400 MHz, DMSO-*d*₆): δ 7.49 (t, 1H, *J*=8.0 Hz, H–6), 7.55–7.61 (m, 3H, Ph–H), 7.78 (d, 1H, *J*=8.4 Hz, H–8), 7.83 (t, 1H, *J*=7.2 Hz, H–7), 8.04 (d, 1H, *J*=7.6 Hz, H–5), 8.10 (d, 2H, *J*=8.0 Hz, Ph–H); ¹³C-NMR (100 MHz, DMSO-*d*₆): δ 119.0 (C–8), 121.7 (C–4'), 124.5 (d, *J*=153 Hz, C–3), 125.2 (C–4a), 126.0 (C–5), 126.8 (C–3', 5'), 129.7 (C–2', 6'), 131.6 (C–1'), 132.2 (C–6), 134.6 (C–7), 156.1 (C–8a), 162.9 (C=O), 177.5 (C=O); MS *m/z*, (%) 339 (M⁺, 9%). Anal. Calcd for C₁₅H₉Cl₂O₃P (339.12): C, 53.13; H, 2.68. Found: C, 52.87; H, 2.32%.

Synthesis of 3-benzoyl-4*H*-1,2-benzoxaphosphinin-4-one (7).

A solution of phosphorus tribromide (5 mmol, 0.5 mL) in dry toluene, was added to a solution of compound **1** (5 mmol, 1.20 g) in dry toluene (30 mL) in the presence of triethylamine (15 mmol, 2.1 mL) **as a base**, under stirring for 30 minutes at 10 °C then heated under reflux for 10 hours. The formed solid was filtered off, washed with water and crystallized from diluted ethanol to give green solid in 54% yield; mp 99–101 °C IR (KBr), ν 3059 (C–H_{Ar}), 1694, 1649 (C=O), 1613, 1592 (C=C), 1027 (O–C); ¹H-NMR (400 MHz, DMSO-*d*₆): δ 7.48 (t, 1H, *J*=6.8 Hz, H–6), 7.55–7.62 (m, 3H, Ph–H), 7.78 (d, 1H, *J*=8.0 Hz, H–8), 7.83 (t, 1H, *J*=8.4 Hz, H–7), 8.04 (d, 1H, *J*=7.6 Hz, H–5), 8.10 (d, 2H, *J*=8.0 Hz, Ph–H); ¹³C-NMR (100 MHz, DMSO-*d*₆): δ 119.0 (C–8), 122.5 (C–4'), 124.4 (d, *J*=125 Hz, C–3), 125.2 (C–4a), 126.0 (C–5), 126.8 (C–3',5'), 129.5 (C–2',6'), 131.6 (C–1'), 132.4 (C–6), 134.8 (C–7), 156.1 (C–8a), 163.1 (C=O), 177.6 (C=O); ³¹P-NMR (162 MHz, DMSO-*d*₆): δ 210.02; MS *m/z*, (%) 268 (M⁺, 4%). Anal. Calcd for C₁₅H₉O₃P (268.21): C, 67.17; H, 3.38. Found: C, 68.86; H, 3.04%.

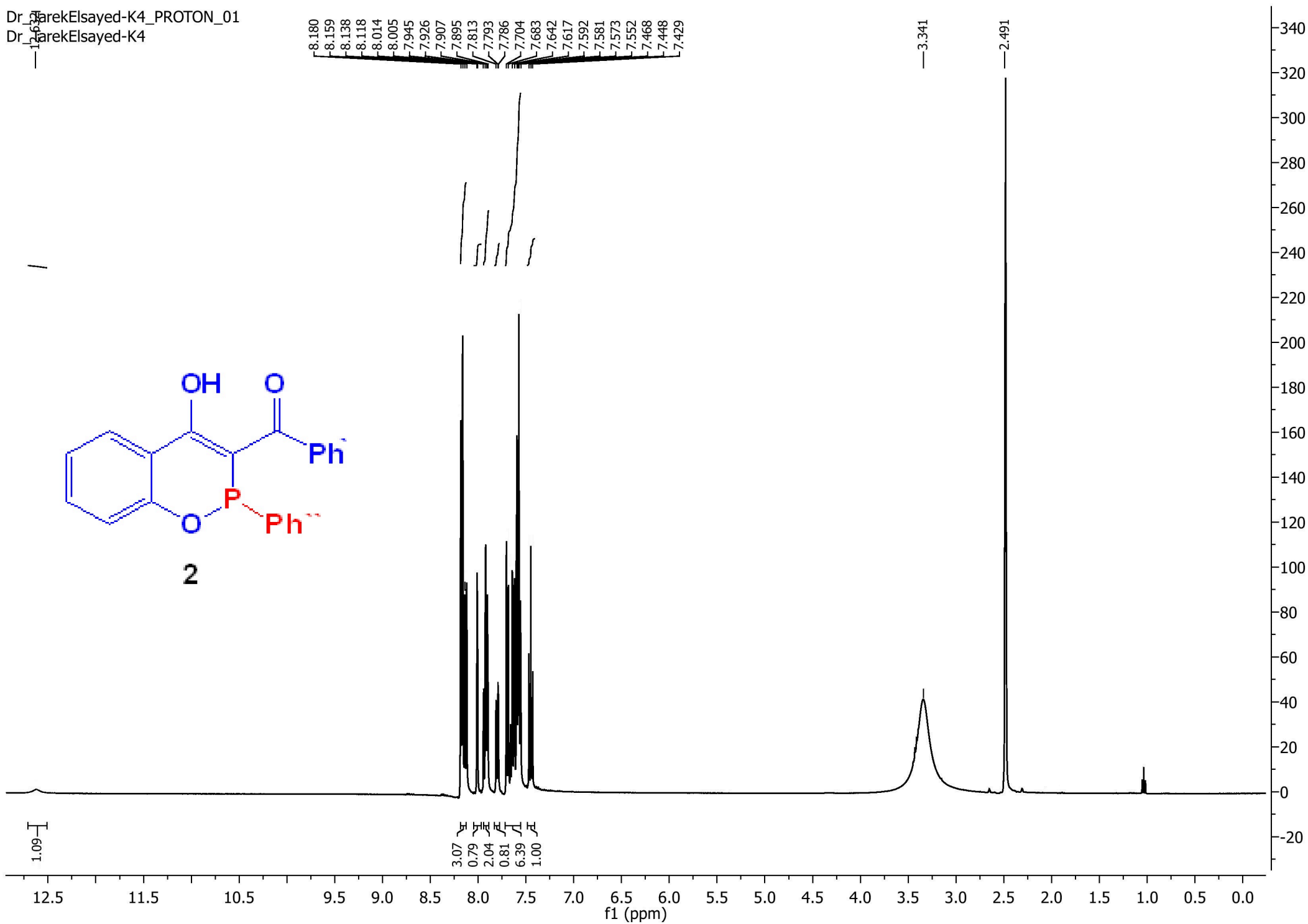
General method for reaction compound **1** with phosphonium salts: Synthesis of the products **8** and **9**.

Acetyl triphenylphosphonium bromide and benzyl triphenyl phosphonium chloride (5 mmol) was added individually to each a solution of compound **1** (5 mmol, 1.20 g) in dry dioxane (30 mL) in the presence of sodium hydride (10 mmol, 0.24 g) **as a base**, then heated under reflux for 10-12 hours. The mixtures were concentrated into their half volumes and poured into cold water then extracted with ethyl acetate. The evaporation of ethyl acetate gave solids **8** and **9**, respectively.

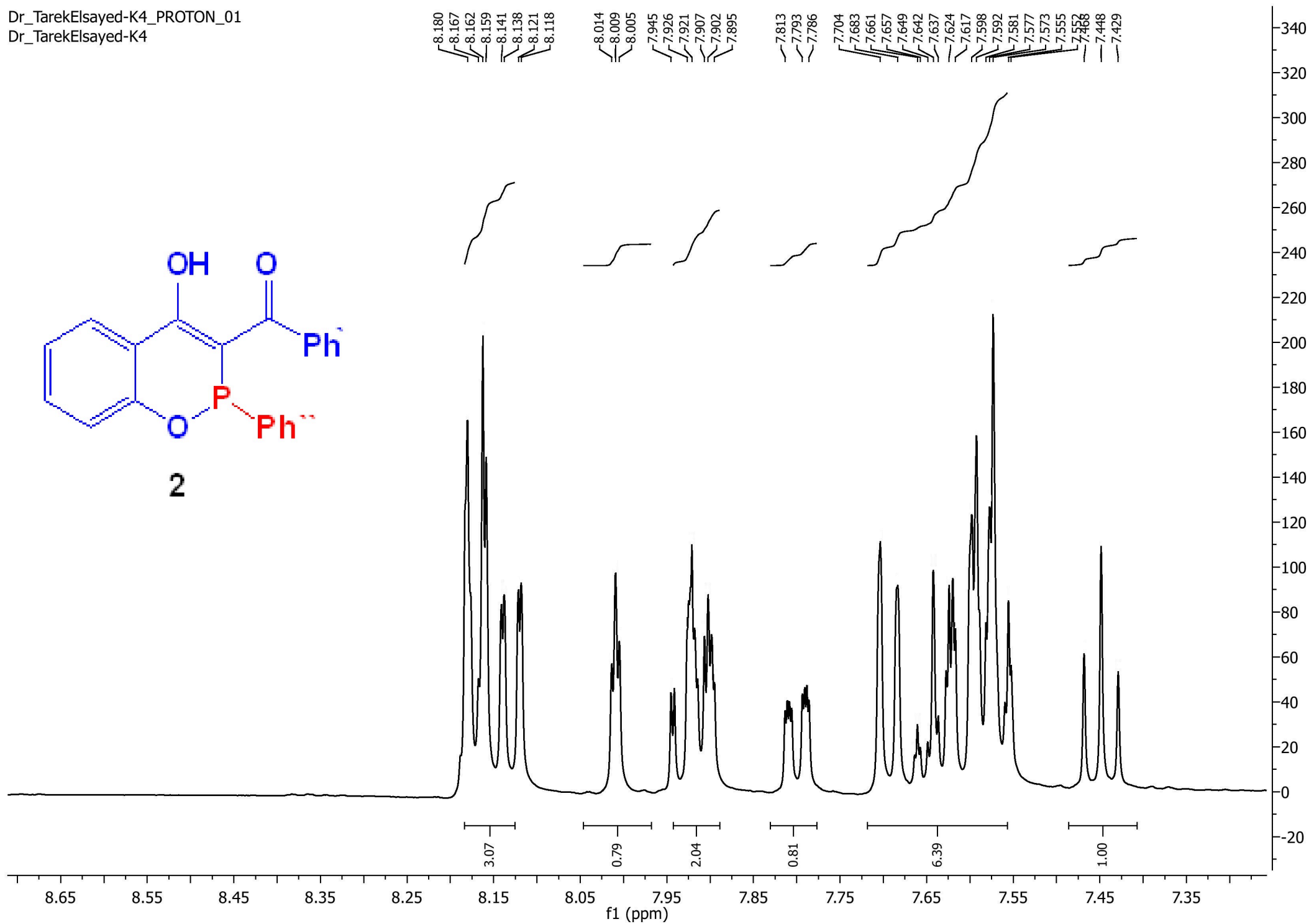
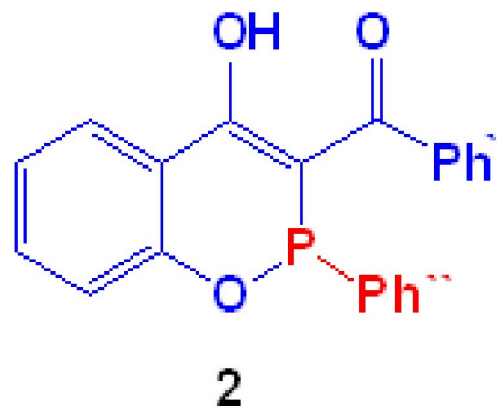
4-(1-Phenylethanoyl)-3-acetyl-2,2,2-triphenyl-2*H*-1,2λ⁵-benzoxaphosphinine (8). Beige solid in 44% yield; mp 132–134 °C. IR (KBr), ν 3040, 3010 (C–H_{Ar}), 2901, 2820 (C–H_{aliph}), 1765, 1702 (C=O), 1612, 1572 (C=C), 1026, 1011 (O–C); ¹H-NMR (400 MHz, DMSO-*d*₆): δ 2.33 (s, 3H, CH₃), 5.72 (d, 2H, *J*=16 Hz, CH₂), 7.55–7.66 (m, 7H, Ph–H), 7.71–7.78 (m, 10H, Ph–H), 7.83 (t, 2H, *J*=6.4 Hz, Ph–H), 7.92 (t, 2H, *J*=8.0 Hz, Ph–H), 8.13 (d, 1H, *J*=7.6 Hz,

H-5), 8.17 (d, 2H, $J=8.4$ Hz, Ph-H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$): δ 32.0 (CH_3), 38.3 (d, $J=58$ Hz, CH_2), 117.3 (C-8), 119.3 (d, $J=88$ Hz, C-1 $''$), 120.3 (C-4), 123.4 (C-5), 126.2 (C-4a), 127.4 (C-1 $'$), 128.2 (C-3 $'$, 5 $'$), 128.5 (C-4 $'$), 129.4 (d, $J=86$ Hz, C-3), 129.5 (C-2 $'$, 6 $'$), 130.5 (C-3 $''$, 5 $''$), 133.2 (C-2 $''$, 6 $''$), 134.1 (C-4 $''$), 135.2 (C-6), 137.3 (C-7), 156.9 (C-8a), 159.3 (C=O), 201.5 (C=O); MS m/z , (%) 463 (M^+ -Ph, 7%). Anal. Calcd for $\text{C}_{36}\text{H}_{29}\text{O}_3\text{P}$ (540.60): C, 79.98; H, 5.41. Found: C, 79.69; H, 5.07%.

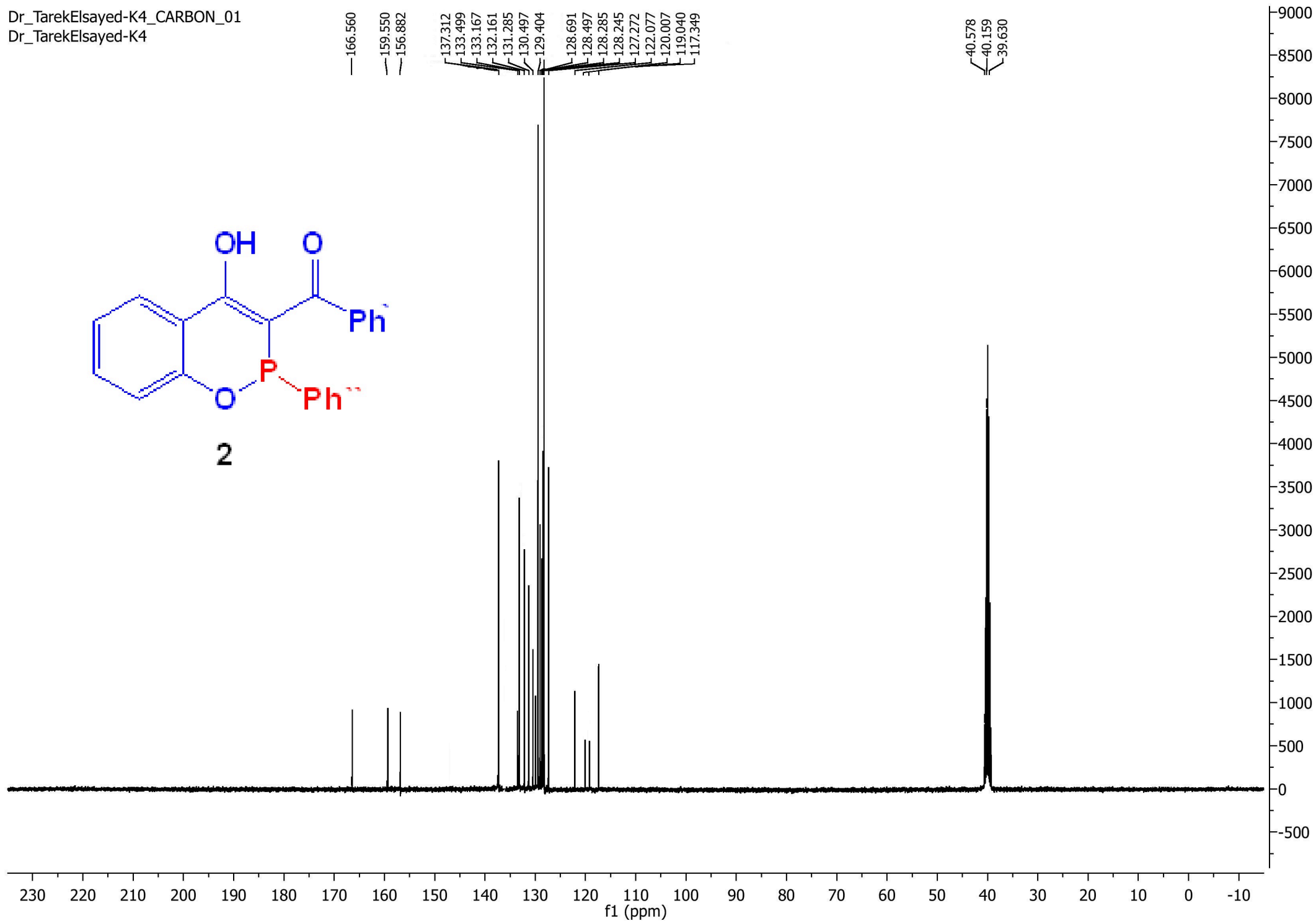
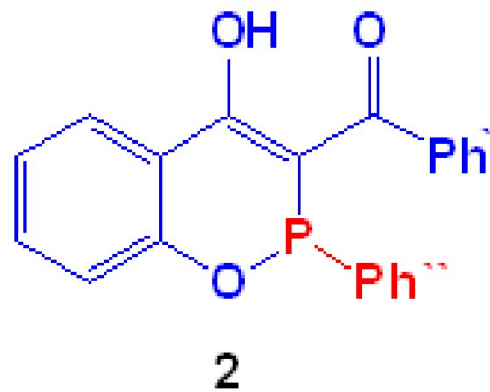
4-(1-Phenylethanoyl)-2,2,2,3-tetraphenyl-2H-1,2 λ^5 -benzoxaphosphinine (9). Beige solid in 53% yield; mp 101–103 °C. IR (KBr), ν 3076, 3057 (C-H_{Ar}), 2989, 2873 (C-H_{aliph}), 1765 (C=O), 1613, 15991 (C=C), 1029 (O-C). $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$): δ 5.22 (d, 2H, $J=14.6$ Hz, CH_2), 6.97 (d, 2H, $J=7.6$ Hz, Ph-H), 7.20 (t, 1H, $J=7.6$ Hz, H-6), 7.25–7.29 (m, 3H, Ph-H), 7.56–7.74 (m, 17H, H-8 and Ph-H), 7.86–7.89 (m, 3H, Ph-H), 7.94 (t, 1H, $J=8.0$ Hz, H-7), 8.14 (d, 1H, $J=8.0$ Hz, H-5), 8.16–8.18 (m, 1H, Ph-H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$): δ 50.0 (d, $J=56$ Hz, CH_2), 117.4 (C-8), 118.3 (d, $J=85$ Hz, C-1 $''$), 121.0 (C-4), 124.7 (C-5), 126.0 (C-4a), 127.4 (C-1 $'''$), 128.2 (C-3 $'$, 5 $'$), 128.4 (C-4 $'$), 128.5 (d, $J=85$ Hz, C-3), 129.1 (C-4 $'''$), 129.2 (C-3 $'''$, 5 $'''$), 129.5 (C-2 $'''$, 6 $'''$), 130.5 (C-3 $''$, 5 $''$), 131.3 (C-2 $'$, 6 $'$), 132.4 (C-1 $'$), 133.2 (C-6), 134.4 (C-2 $''$, 6 $''$), 135.5 (C-4 $''$), 137.3 (C-7), 156.8 (C-8a), 159.3 (C=O); $^{31}\text{P-NMR}$ (162 MHz, $\text{DMSO-}d_6$): δ 24.31; MS m/z , (%) 497 (M^+ -Ph, 12%). Anal. Calcd for $\text{C}_{40}\text{H}_{31}\text{O}_2\text{P}$ (574.67): C, 83.60; H, 5.44. Found: C, 83.32; H, 5.11%.



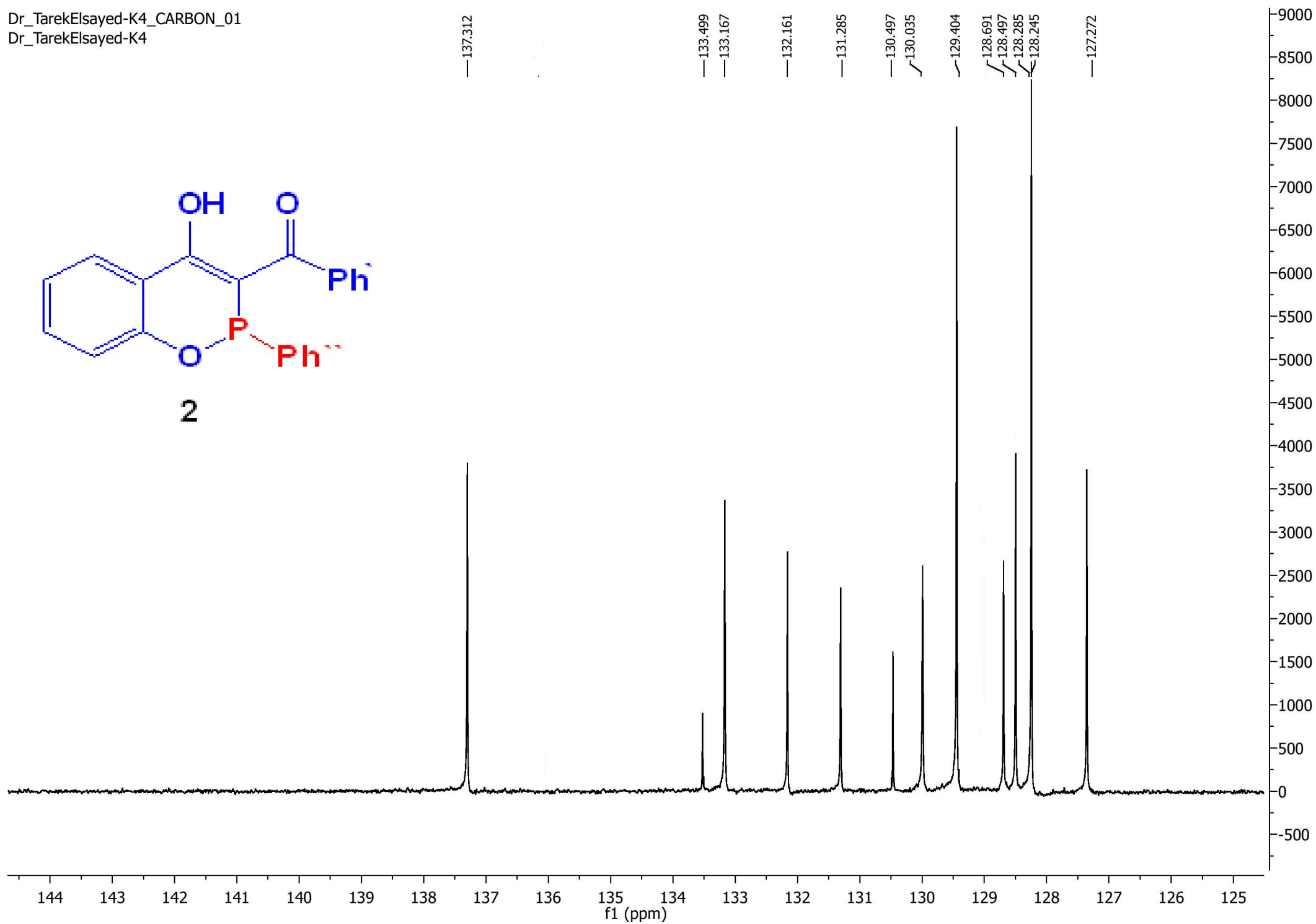
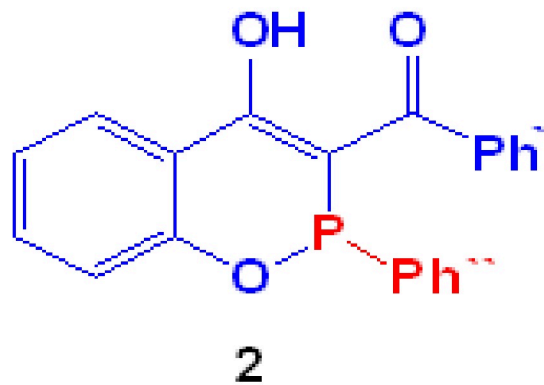
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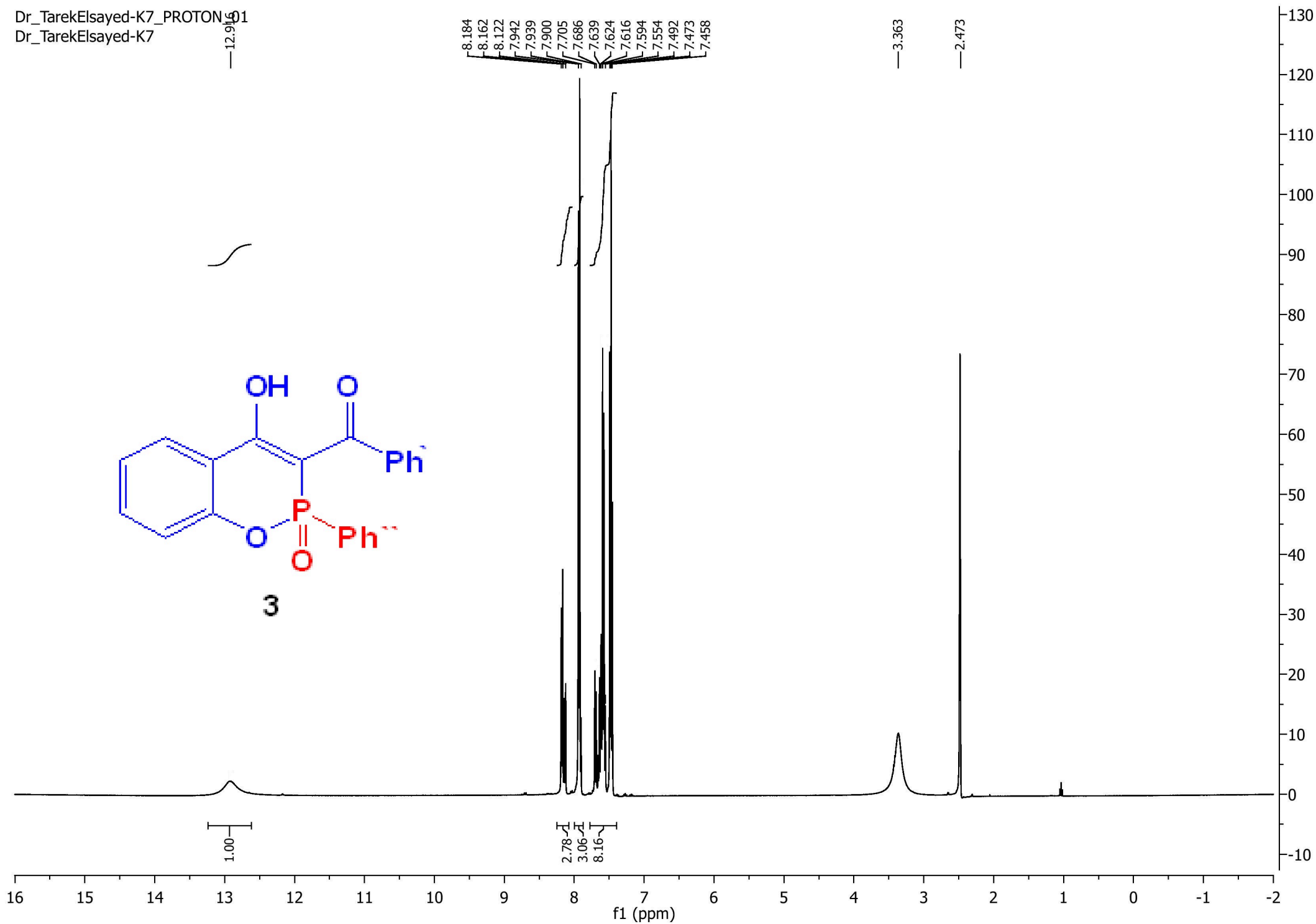
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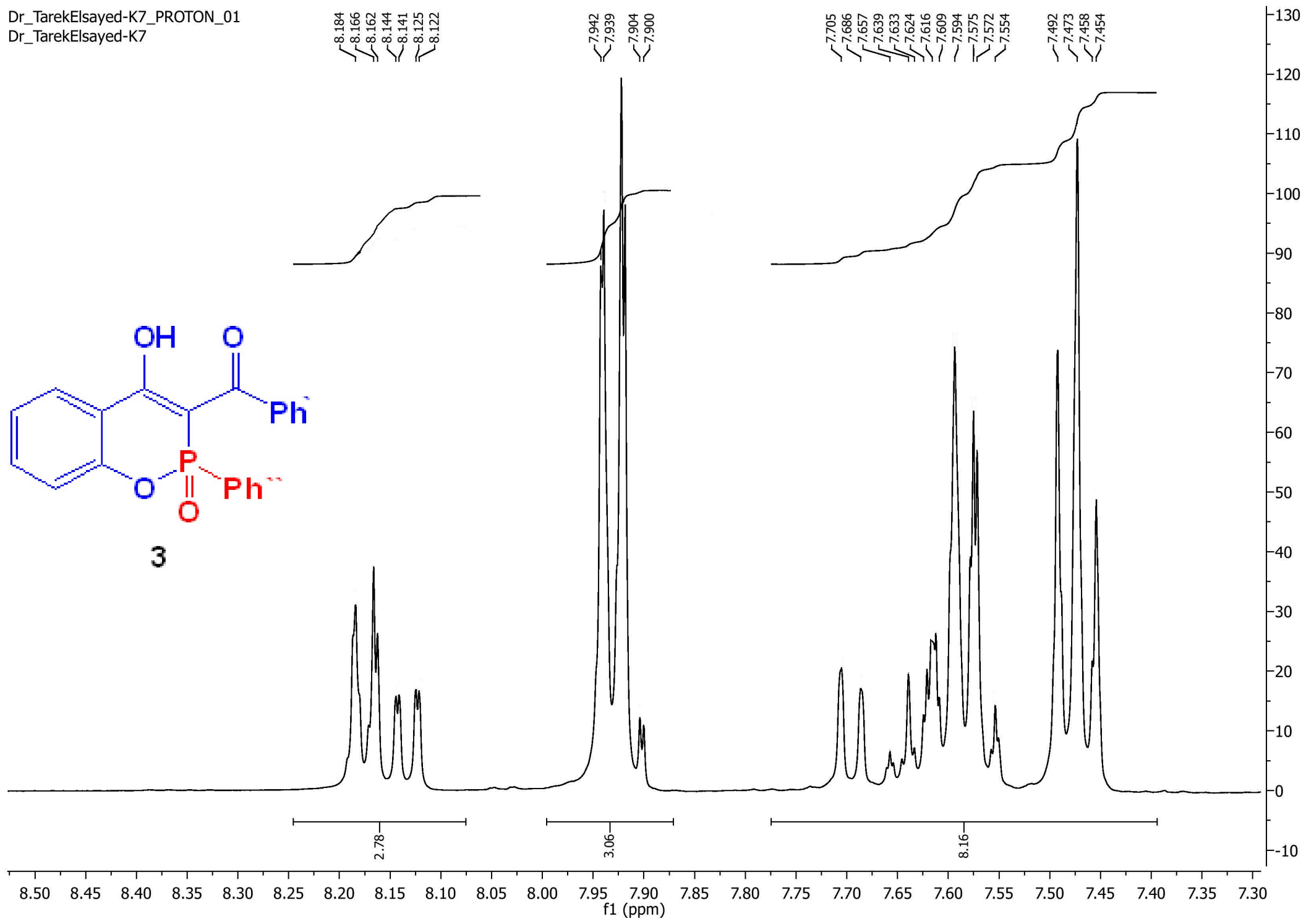
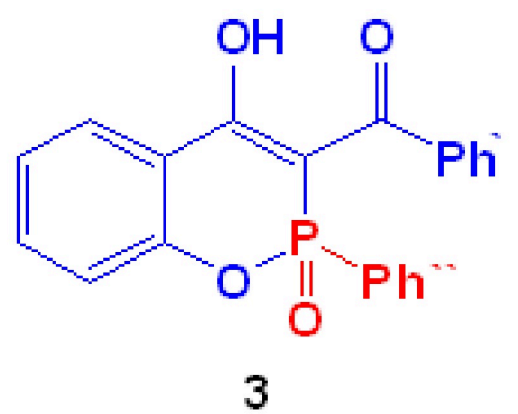
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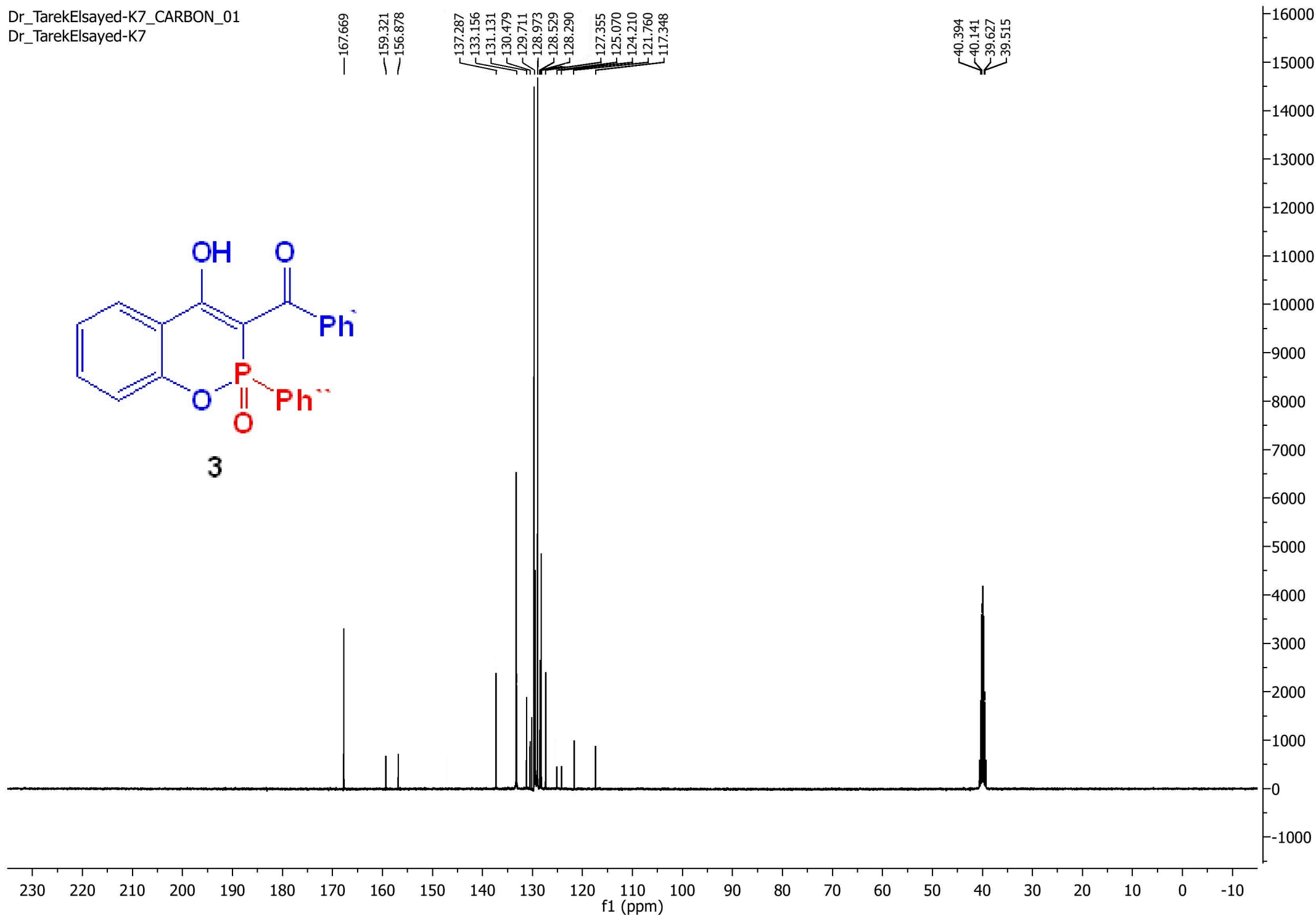
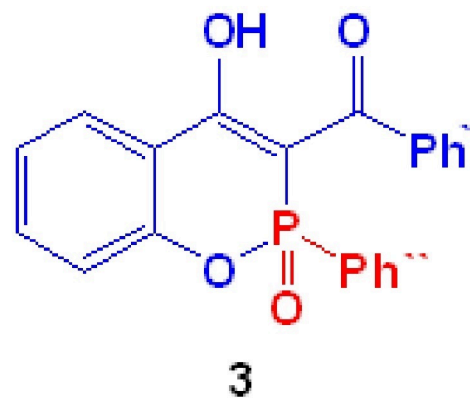
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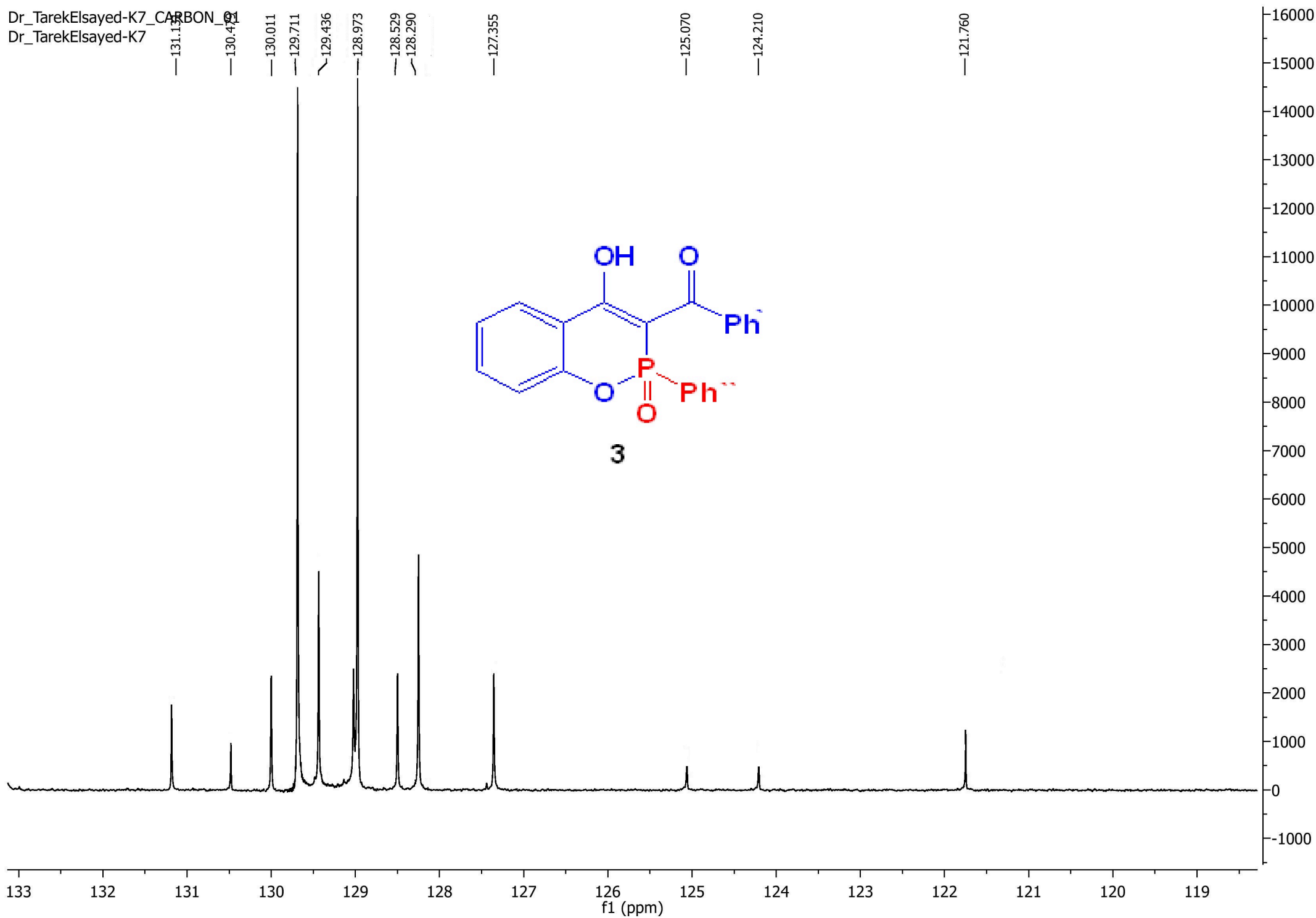
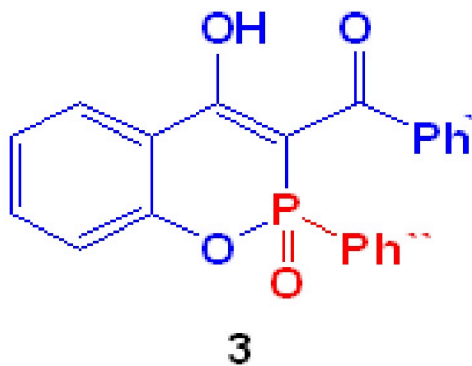


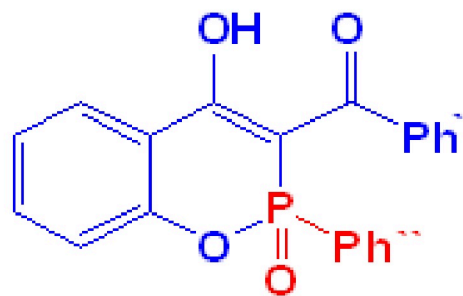
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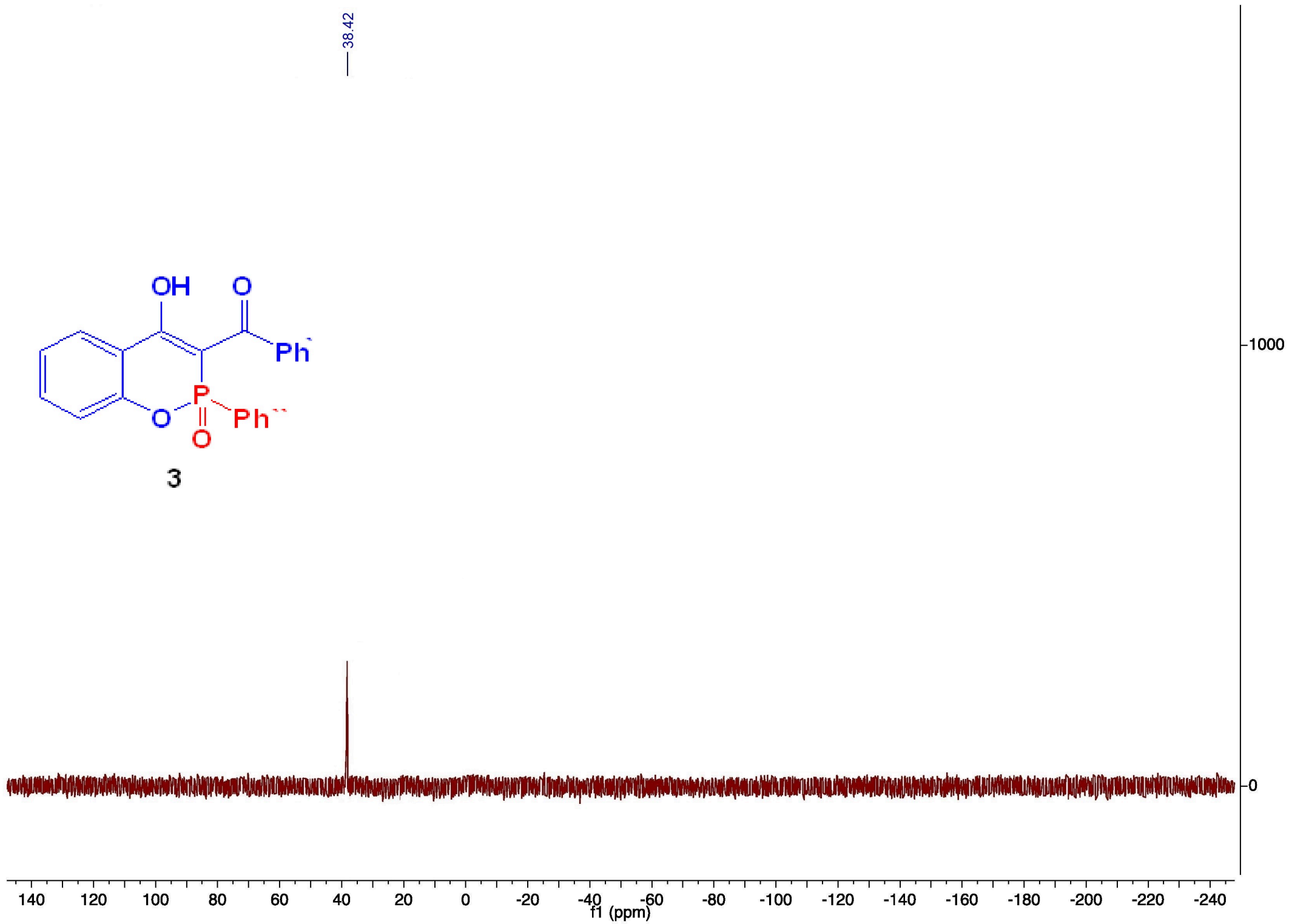
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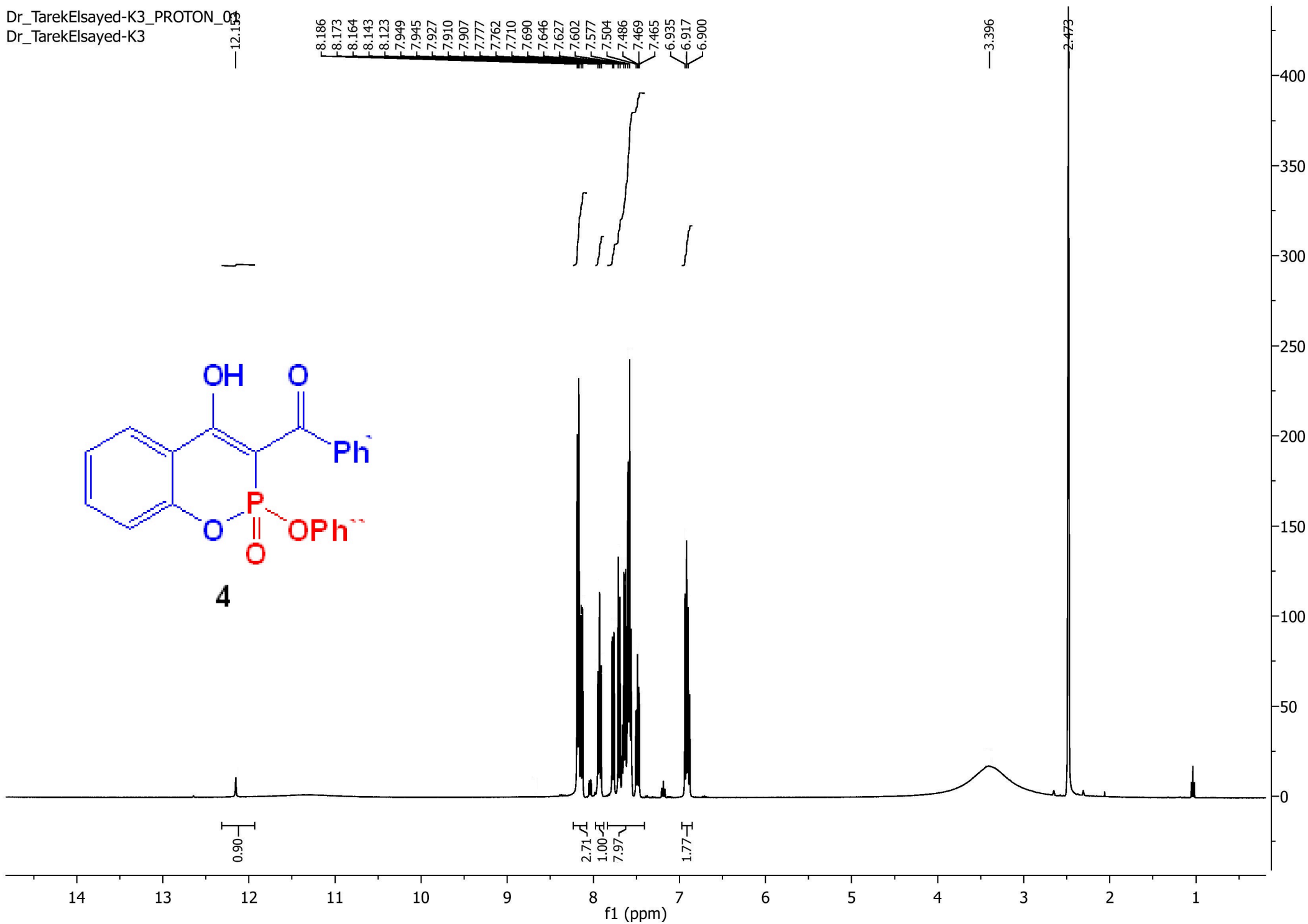


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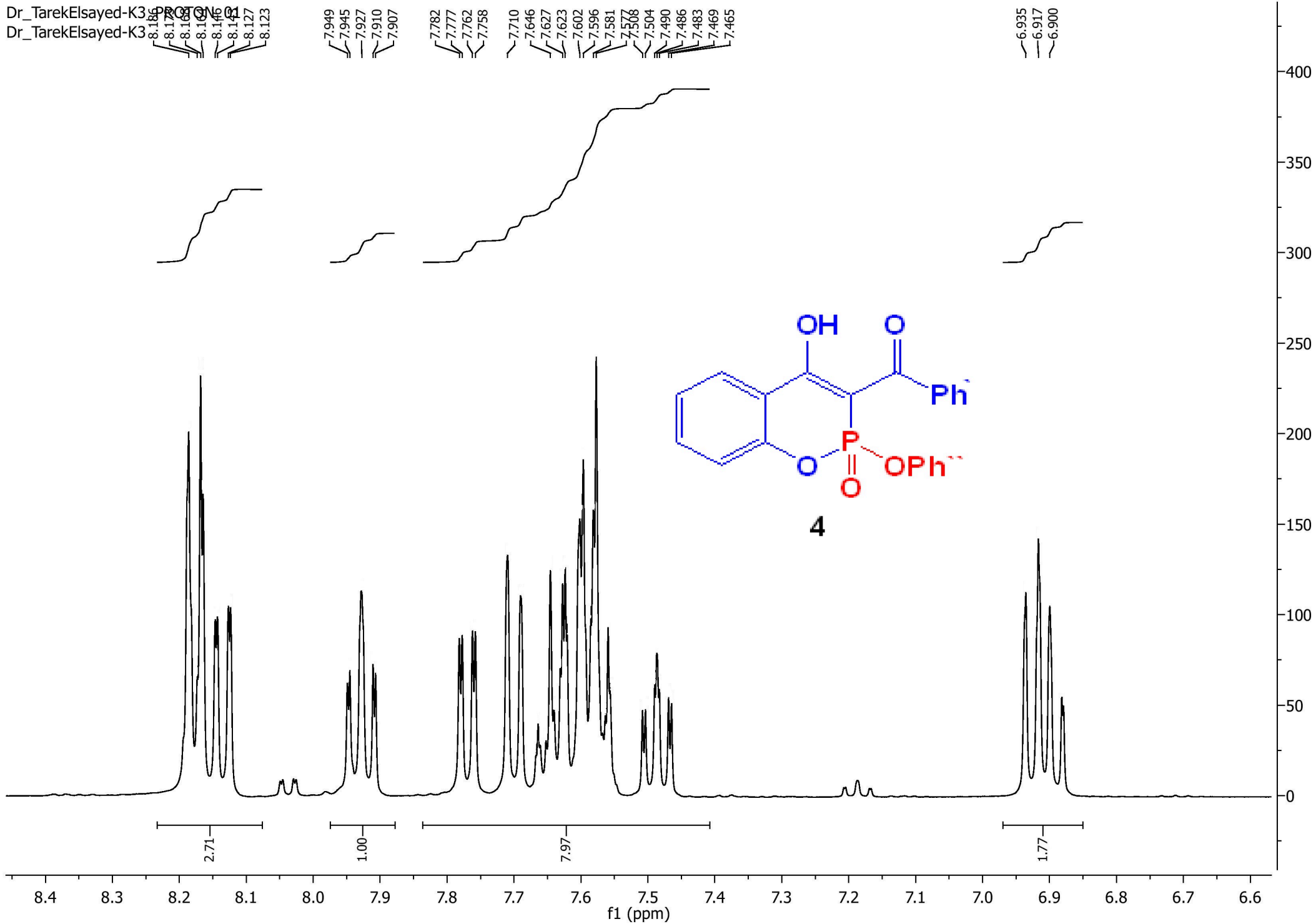
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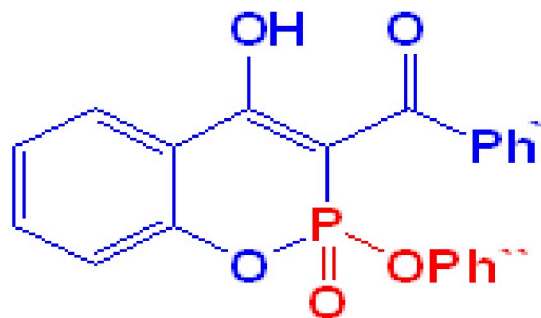
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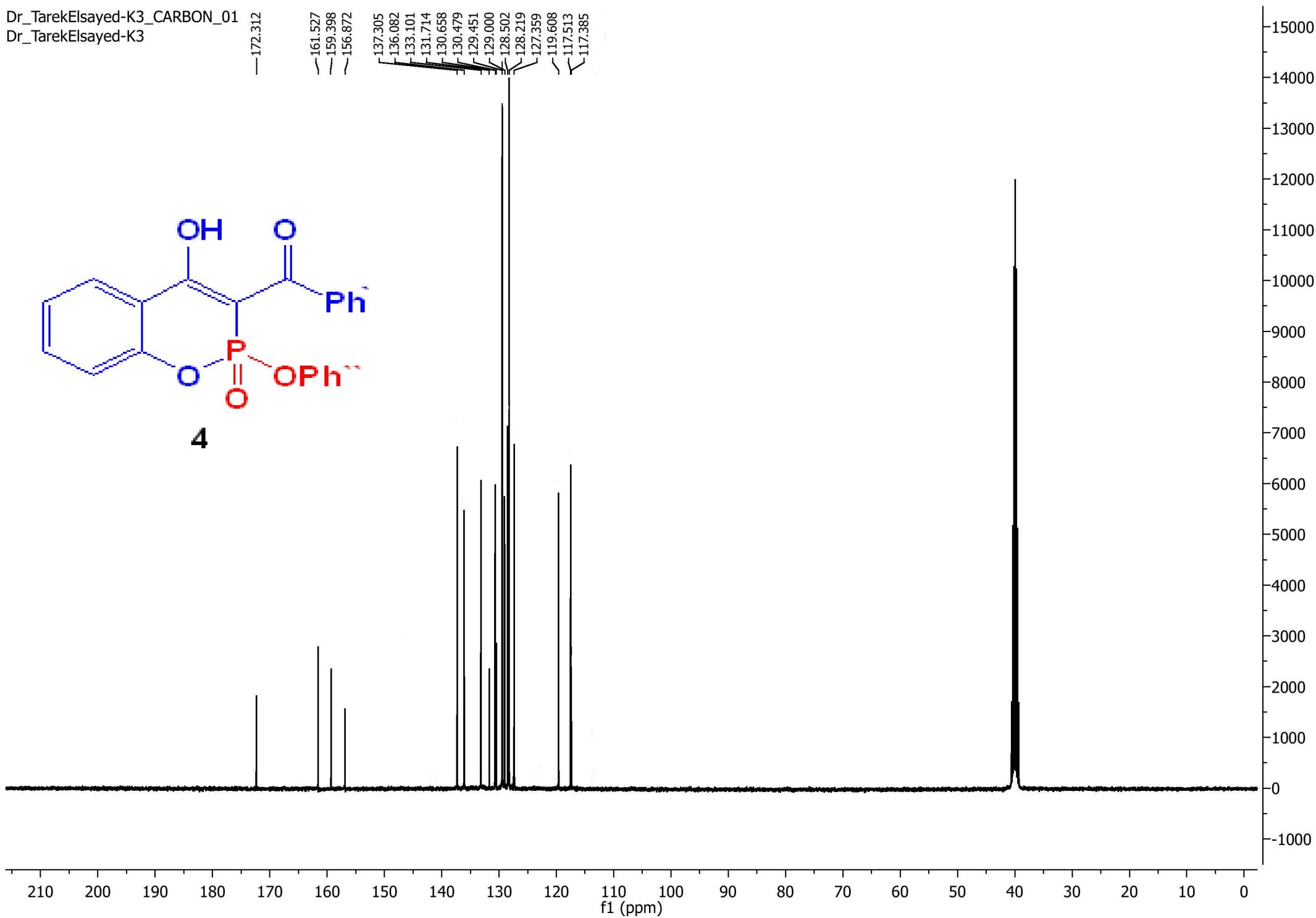
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f1 (ppm)

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4



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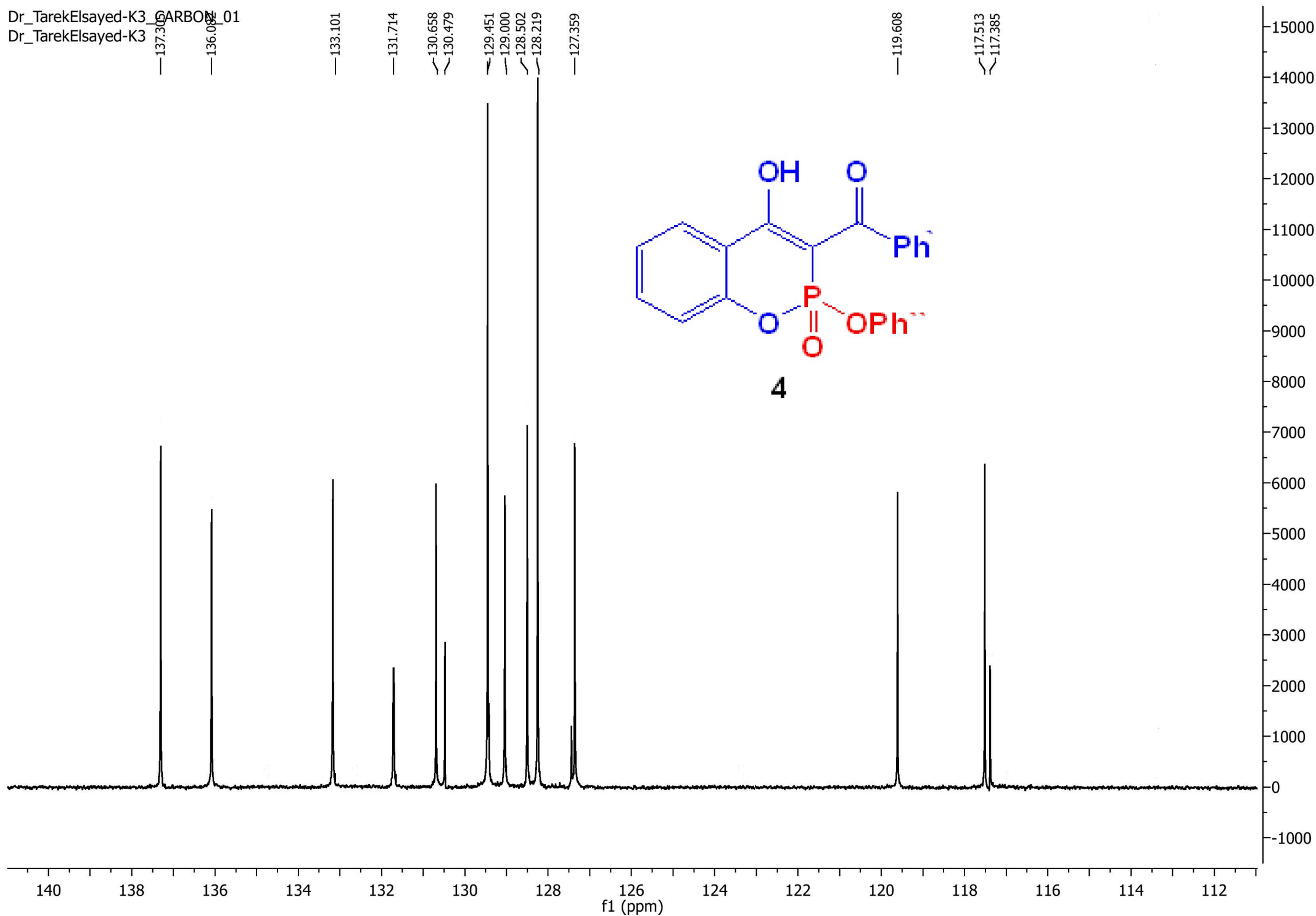
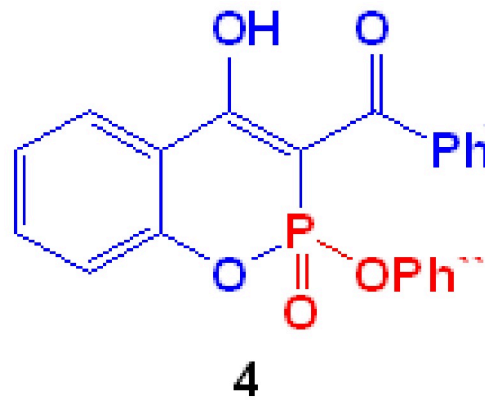
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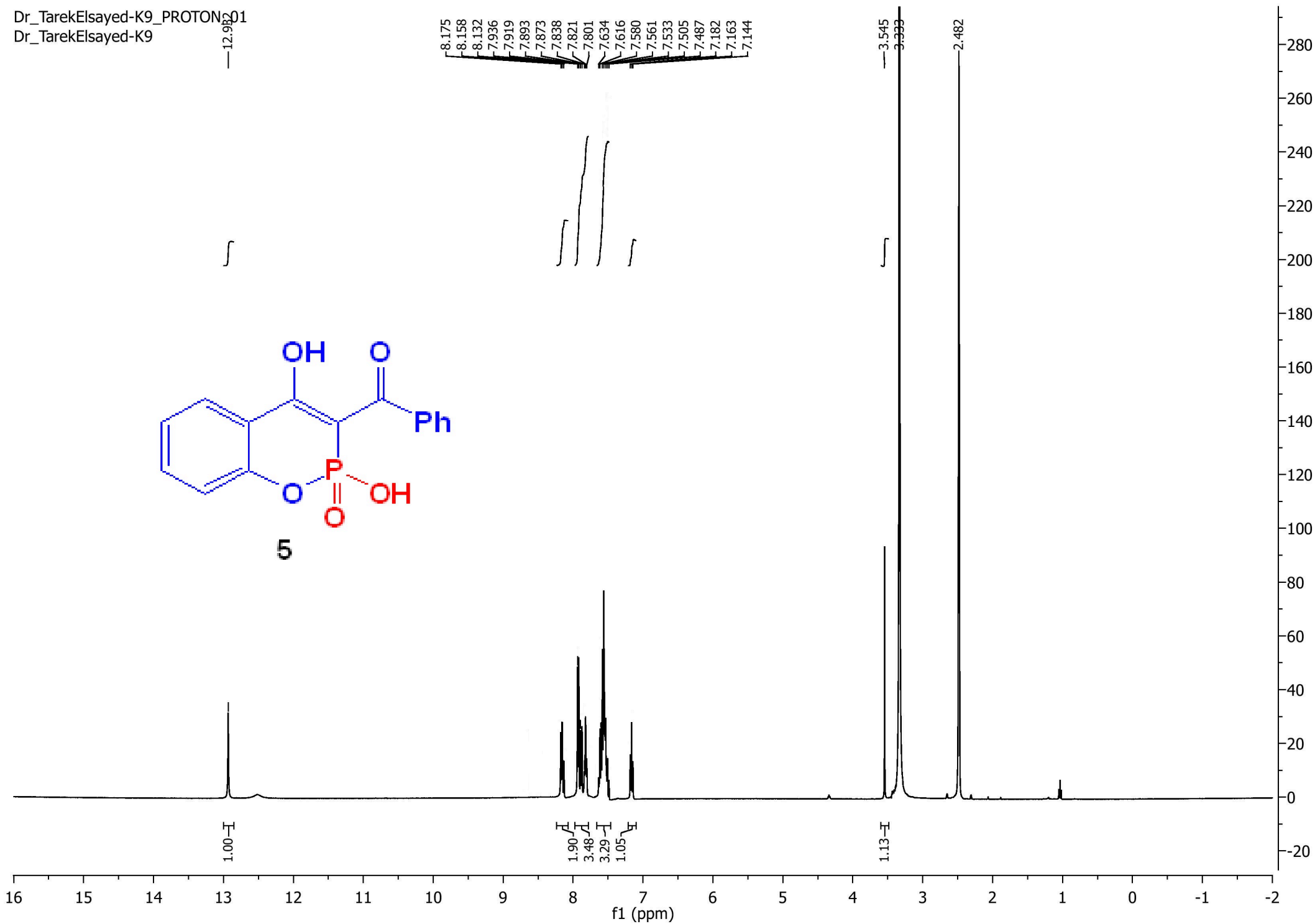
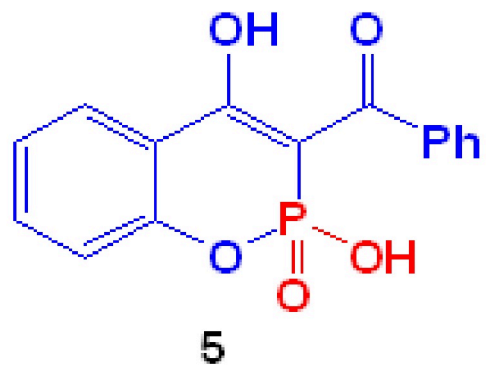
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Dr_TarekElsayed-K9



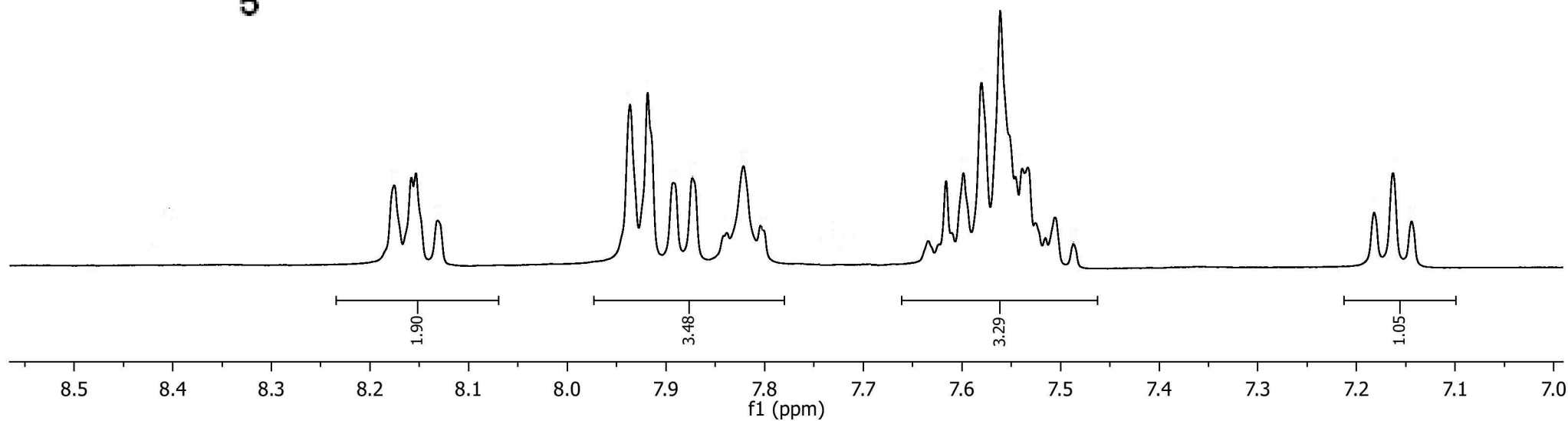
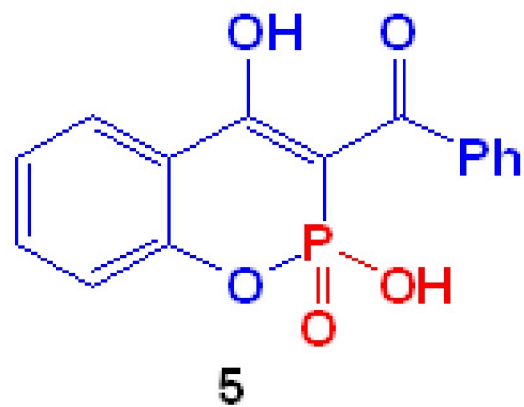
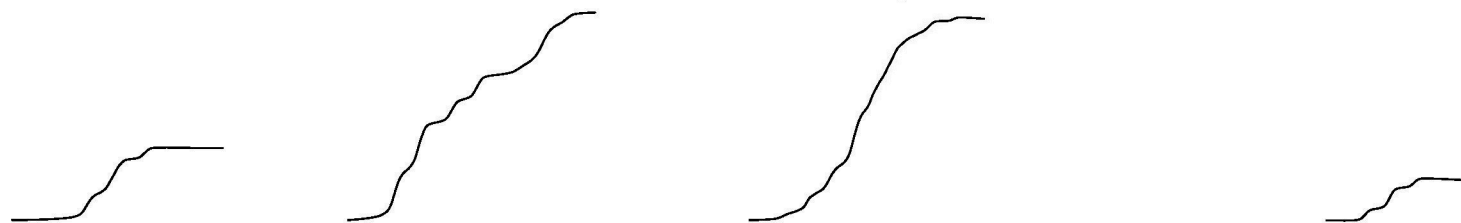
Dr_TarekElsayed-K9_PROTON_01
Dr_TarekElsayed-K9

8.175
8.158
8.153
8.132

7.936
7.919
7.893
7.873
7.838
7.821
7.801

7.634
7.616
7.598
7.580
7.561
7.539
7.533
7.505
7.487

7.182
7.163
7.144



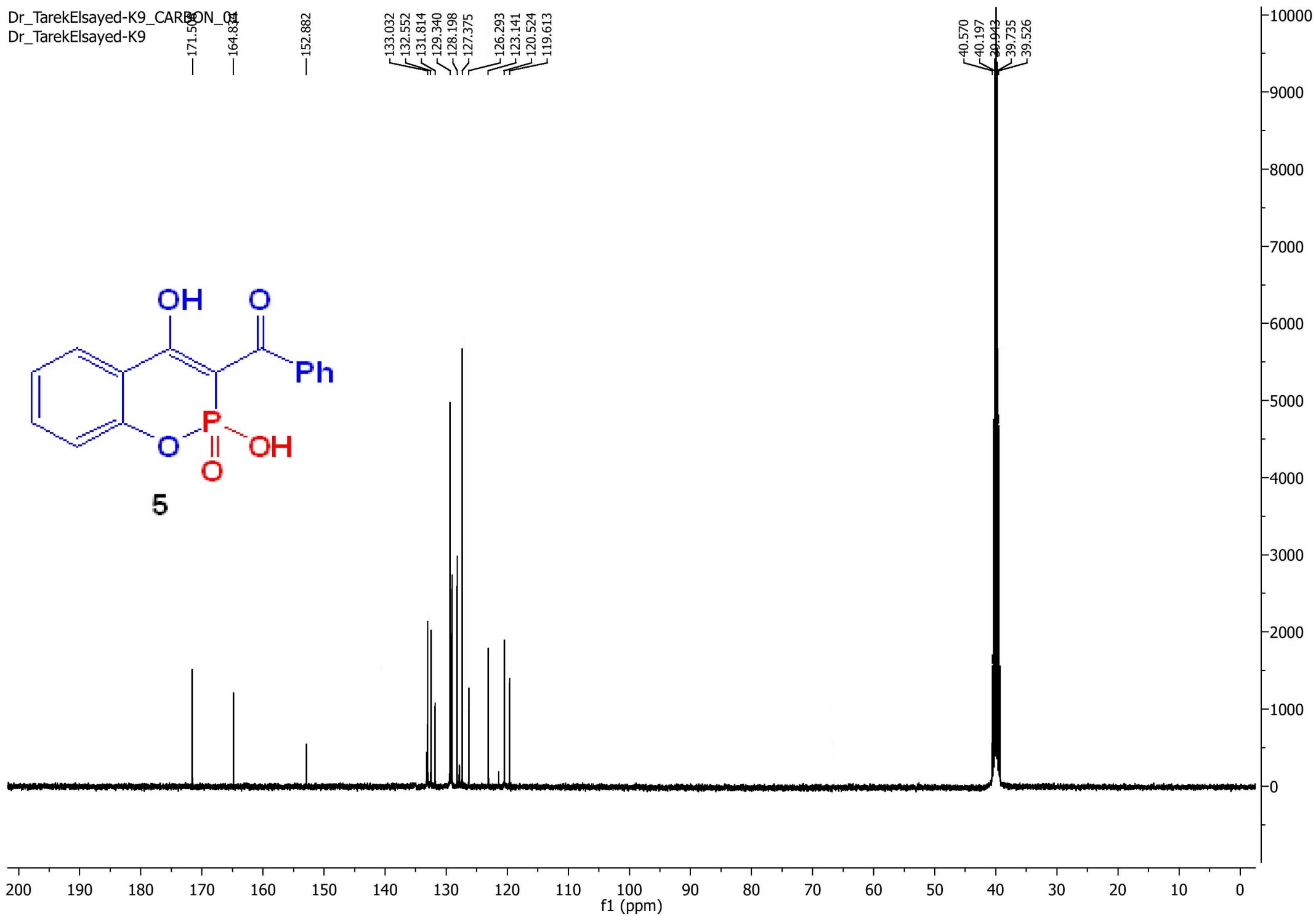
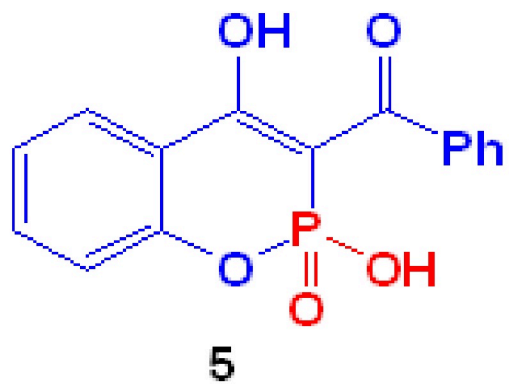
Dr_TarekElsayed-K9_CARBON_01
Dr_TarekElsayed-K9

171.500
164.814

152.882

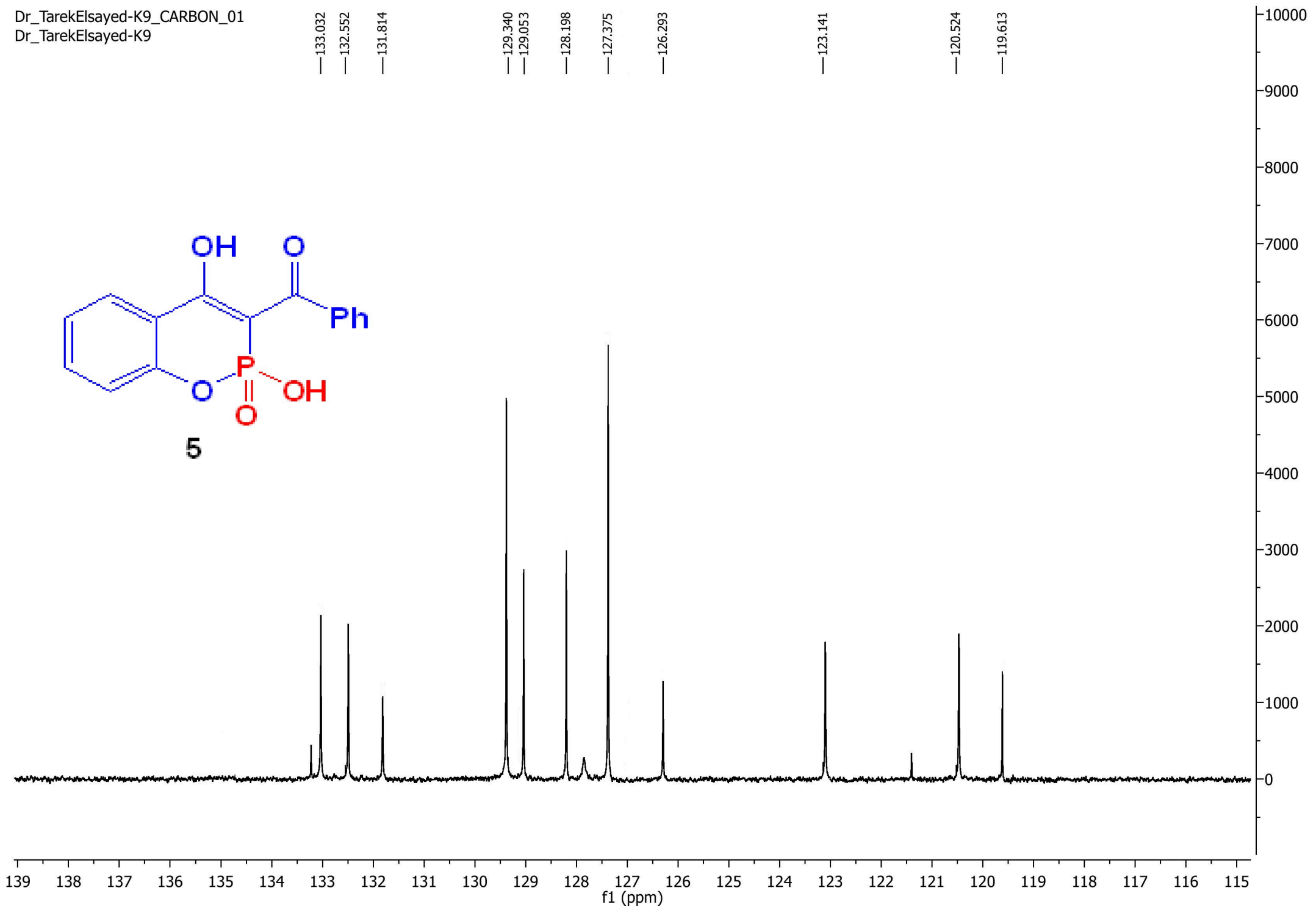
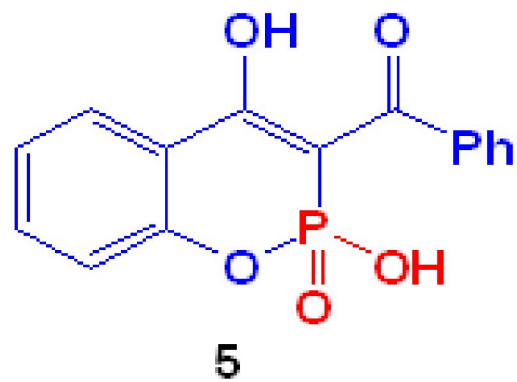
133.032
132.552
131.814
129.340
128.198
127.375
126.293
123.141
120.524
119.613

40.570
40.197
39.943
39.735
39.526



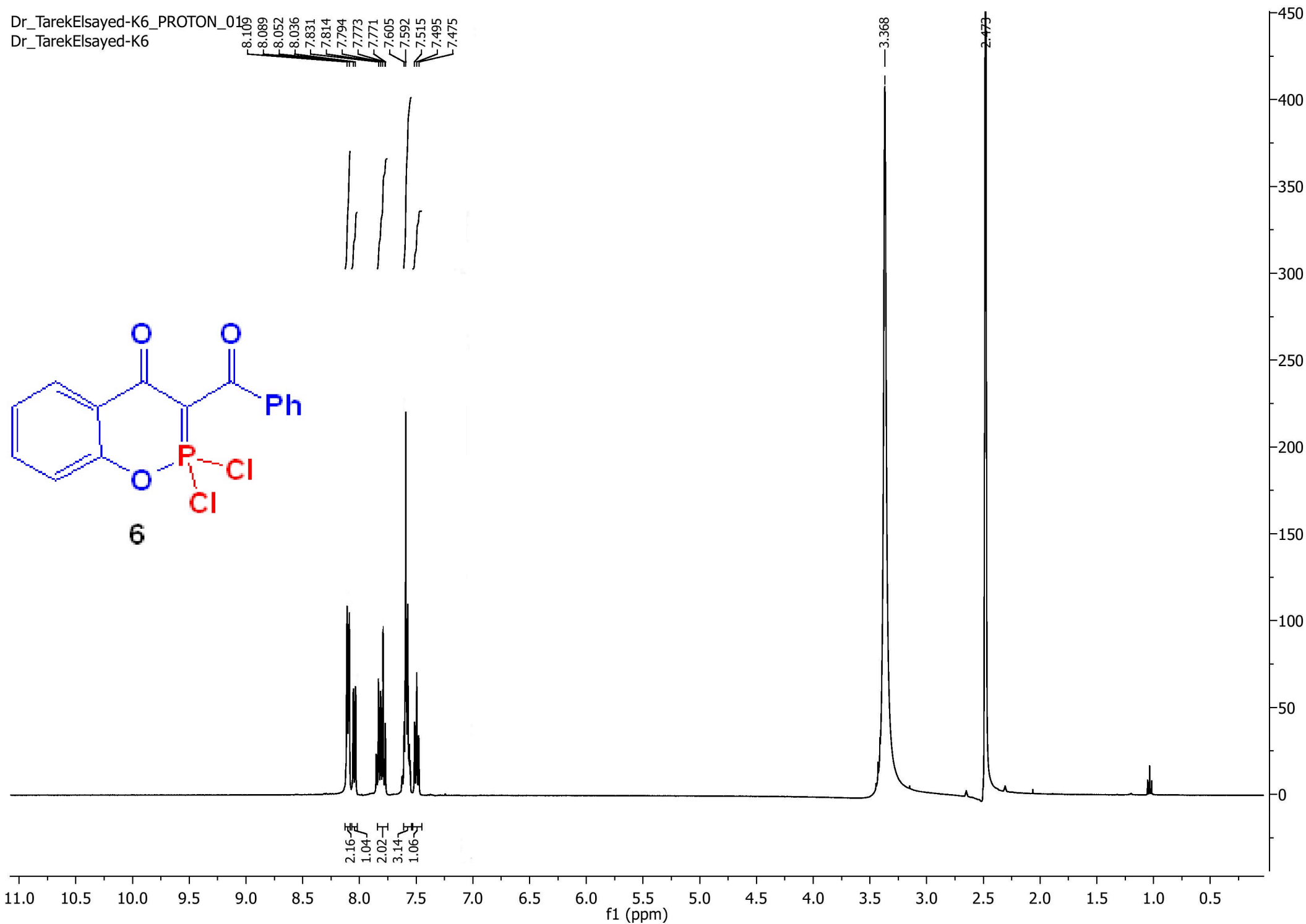
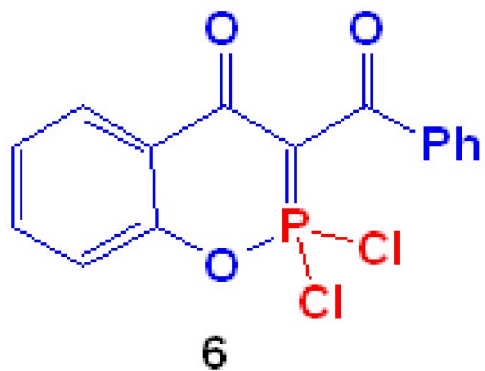
Dr_TarekElsayed-K9_CARBON_01
Dr_TarekElsayed-K9

—133.032 —132.552 —131.814 —129.340 —129.053 —128.198 —127.375 —126.293 —123.141 —120.524 —119.613



Dr_TarekElsayed-K6_PROTON_01
Dr_TarekElsayed-K6

8.109
8.089
8.052
8.036
7.831
7.814
7.794
7.773
7.771
7.605
7.592
7.515
7.495
7.475

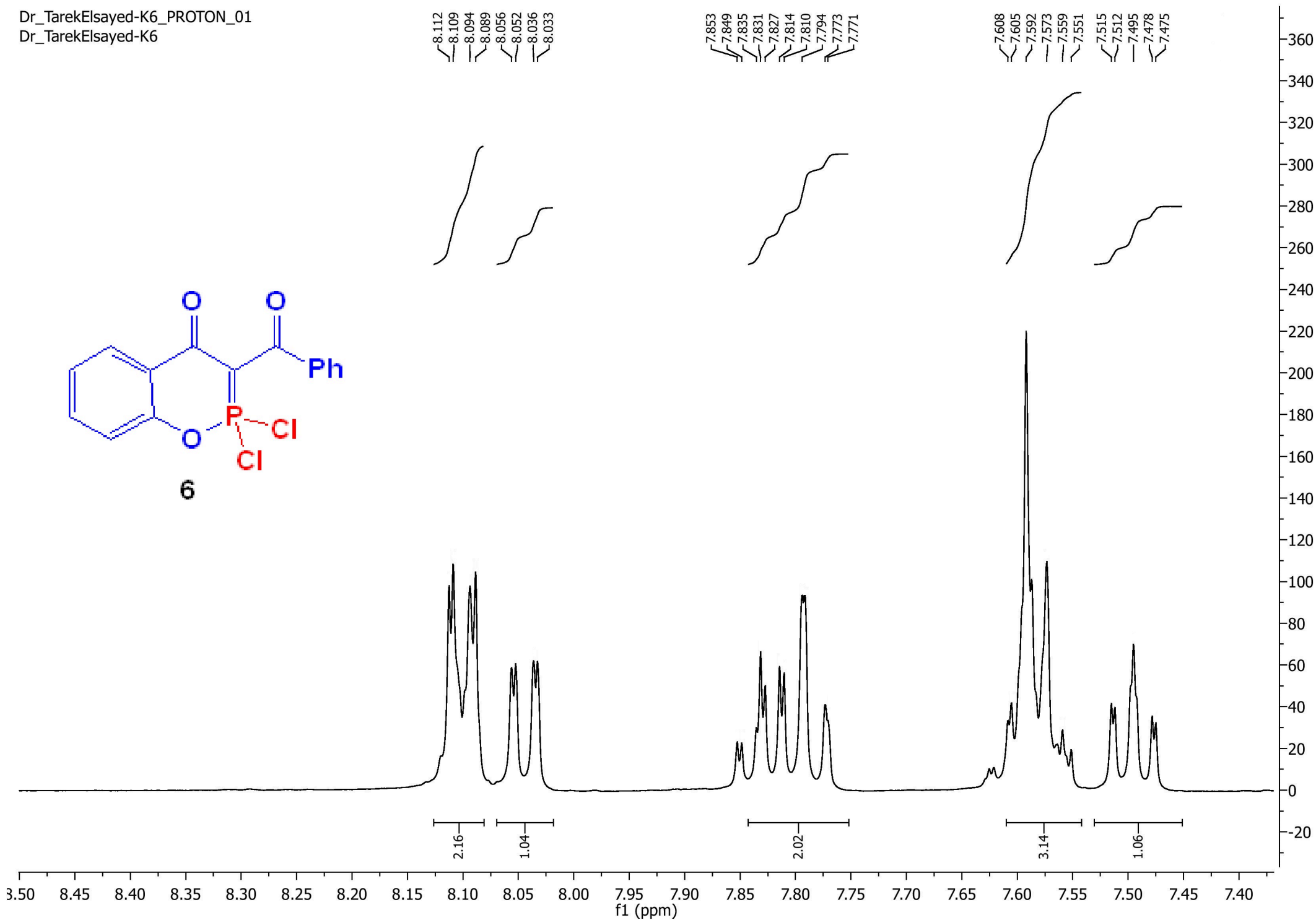
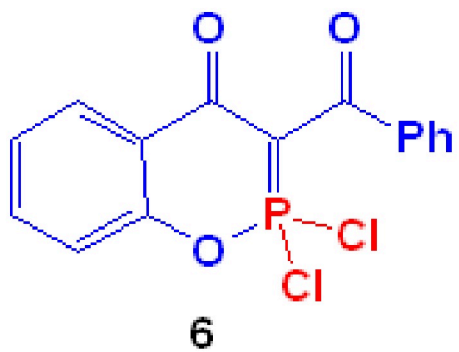


Dr_TarekElsayed-K6_PROTON_01
Dr_TarekElsayed-K6

8.112
8.109
8.094
8.089
8.056
8.052
8.036
8.033

7.853
7.849
7.835
7.831
7.827
7.814
7.810
7.794
7.773
7.771

7.608
7.605
7.592
7.573
7.559
7.551
7.515
7.512
7.495
7.478
7.475

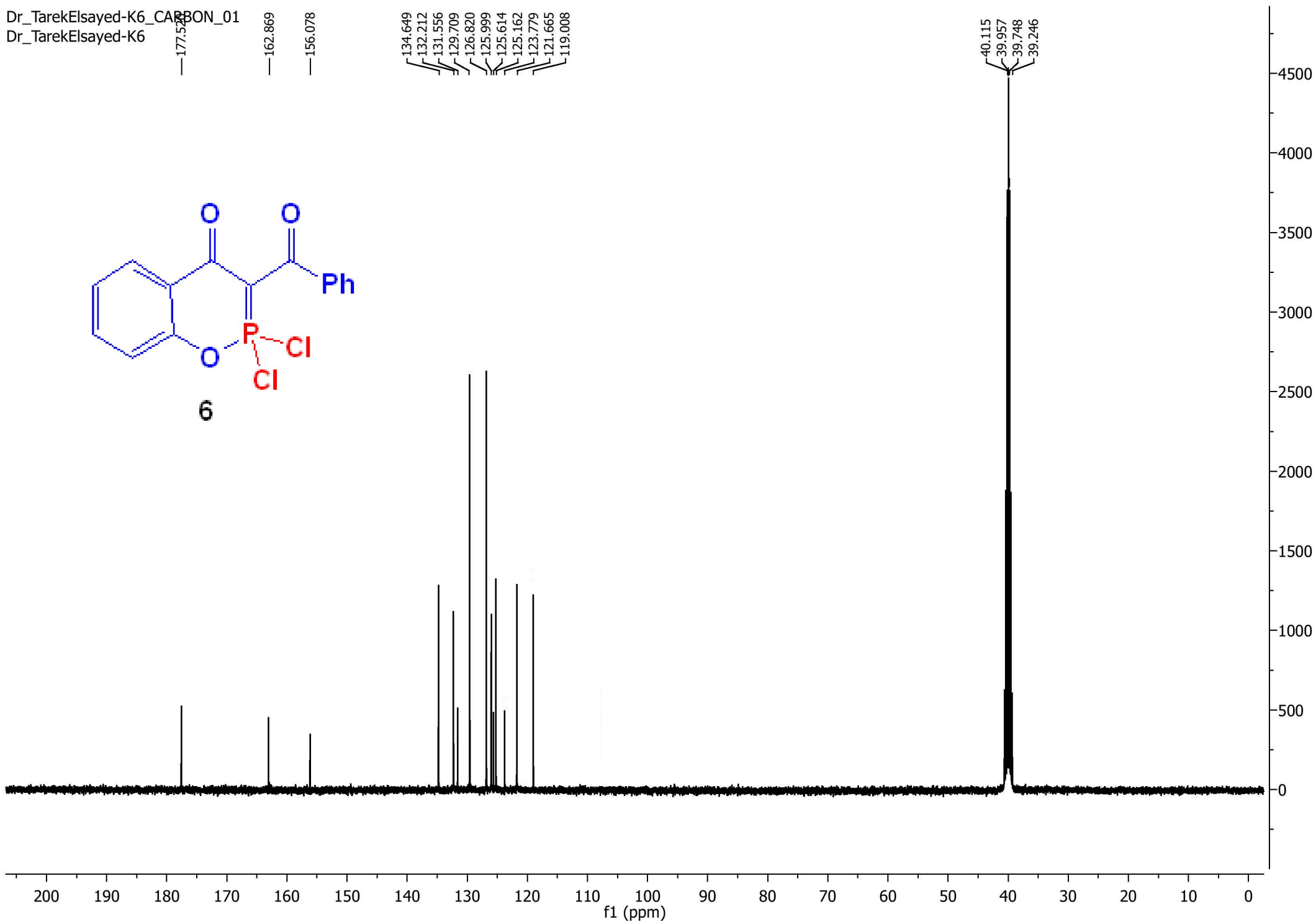
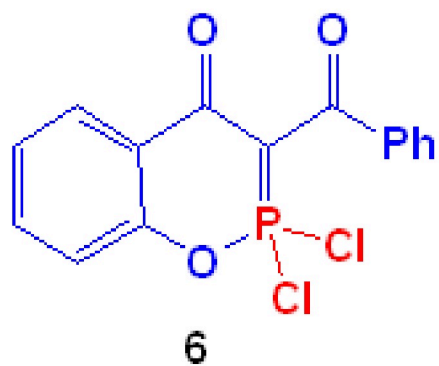


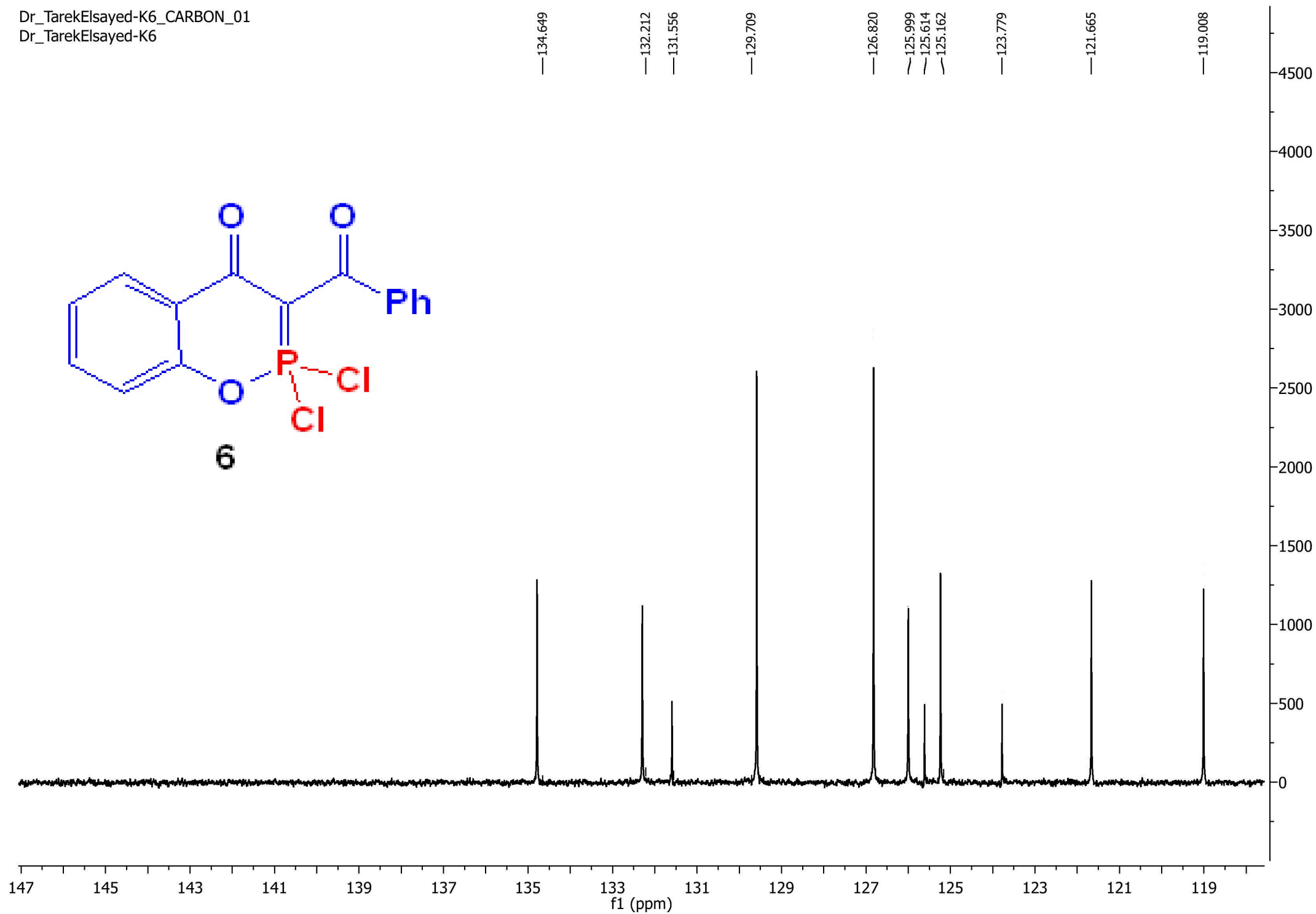
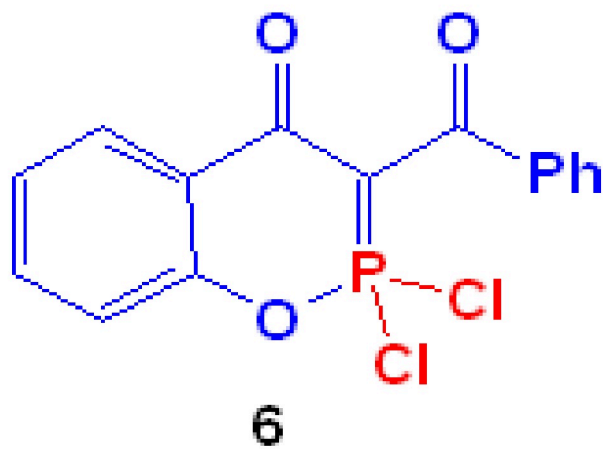
Dr_TarekElsayed-K6_CARBON_01
Dr_TarekElsayed-K6

177.528
162.869
156.078

134.649
132.212
131.556
129.709
126.820
125.999
125.614
125.162
123.779
121.665
119.008

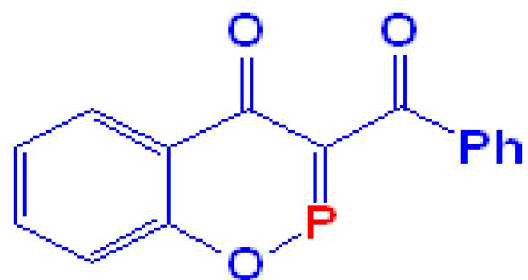
40.115
39.957
39.748
39.246



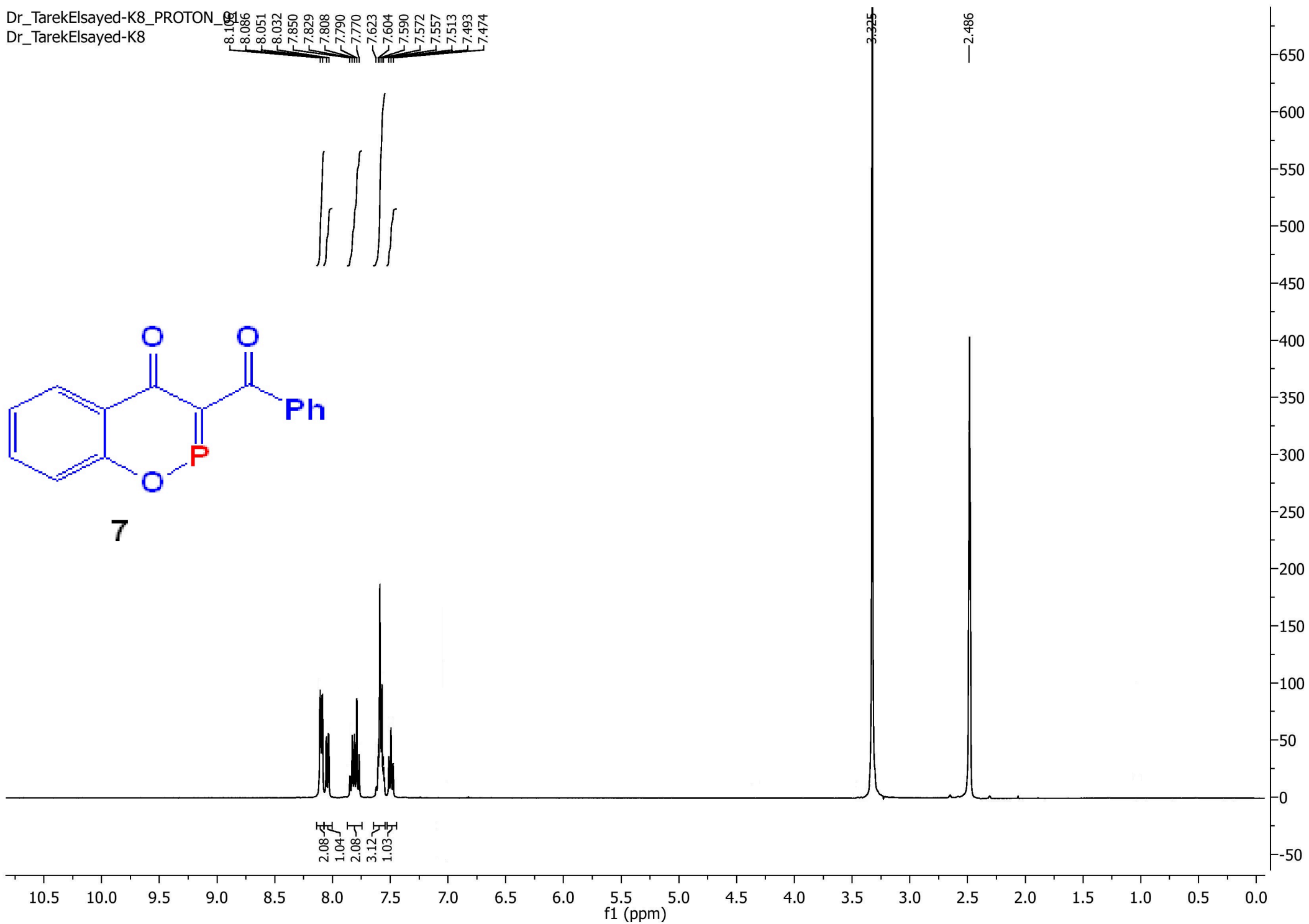


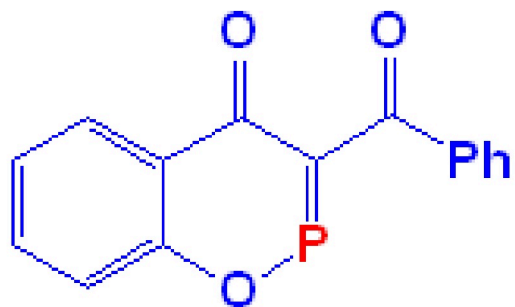
Dr_TarekElsayed-K8_PROTON_01
Dr_TarekElsayed-K8

8.101
8.086
8.051
8.032
7.850
7.829
7.808
7.790
7.770
7.623
7.604
7.590
7.572
7.557
7.513
7.493
7.474

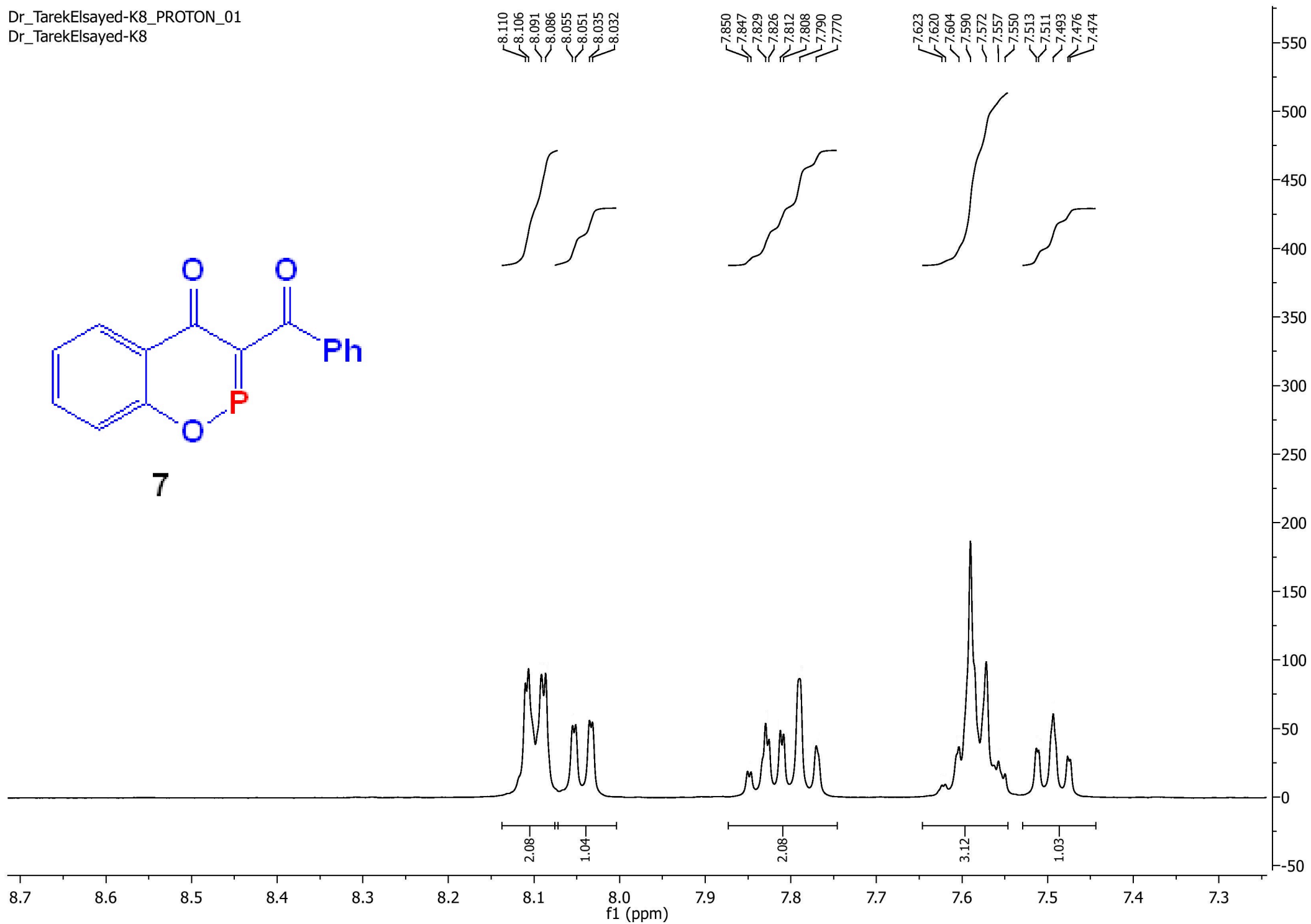


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7

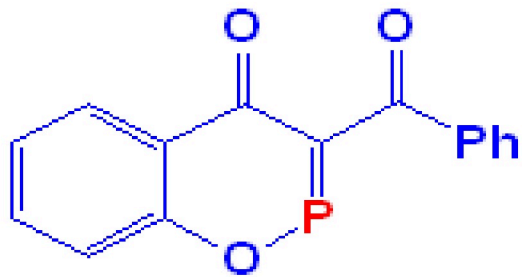


Dr_TarekElsayed-K8_CARBON_04
Dr_TarekElsayed-K8

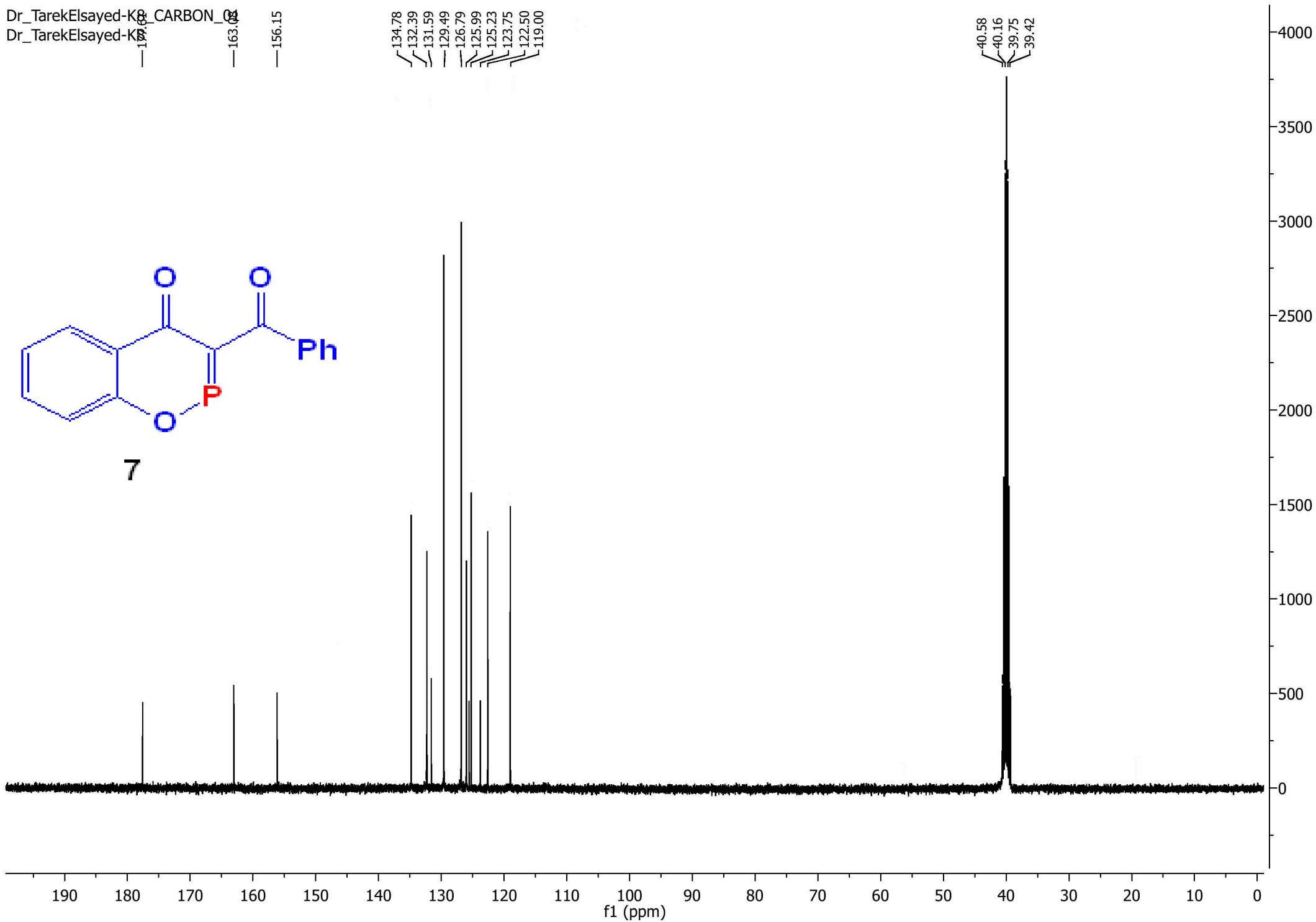
163.04
156.15

134.78
132.39
131.59
129.49
126.79
125.99
125.23
123.75
122.50
119.00

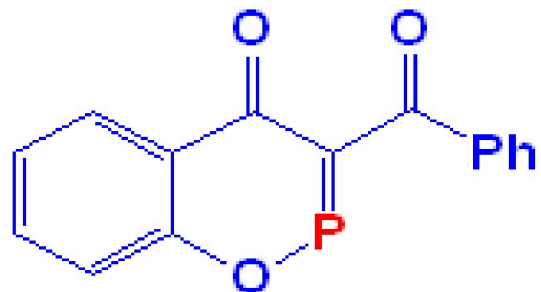
40.58
40.16
39.75
39.42



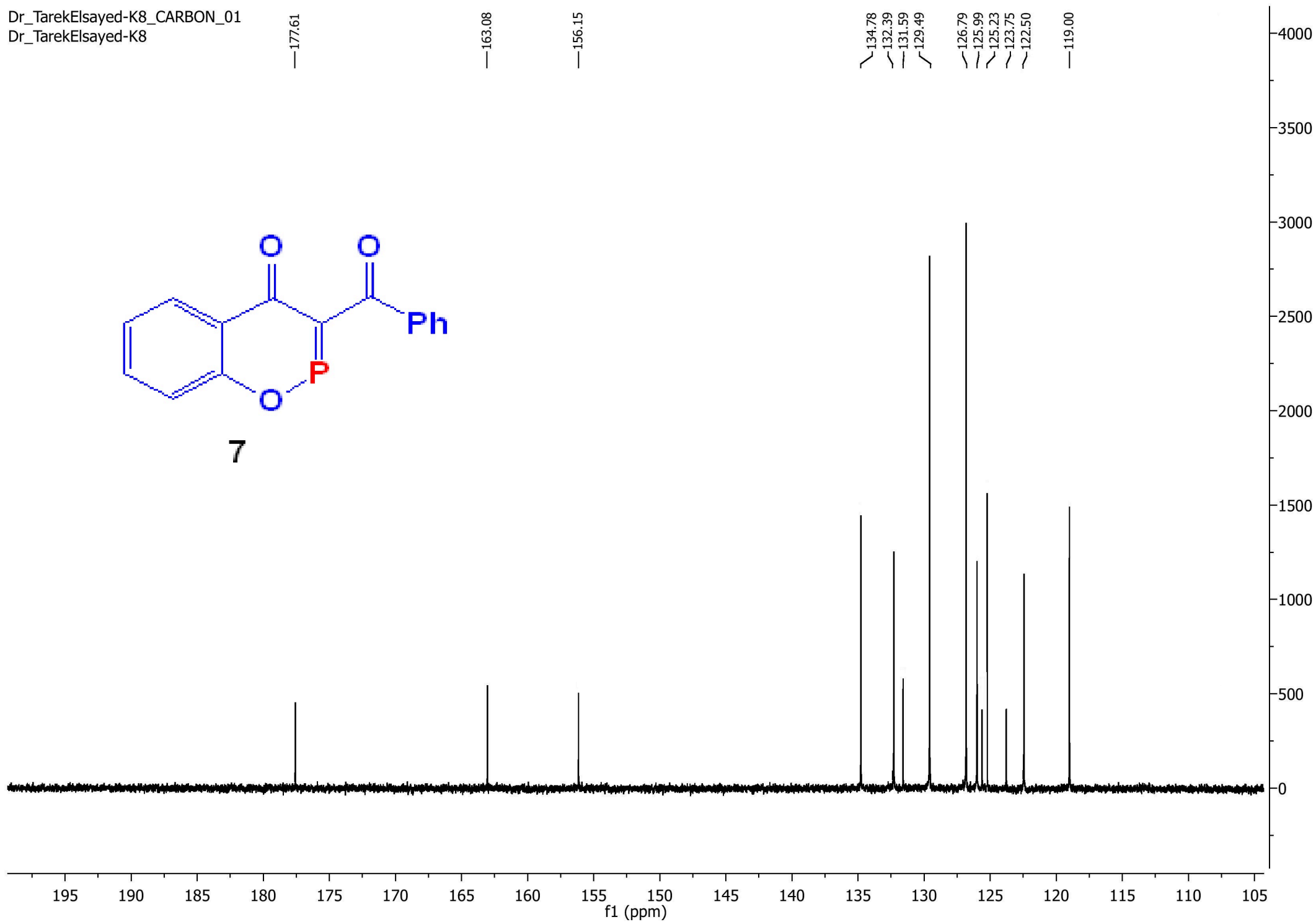
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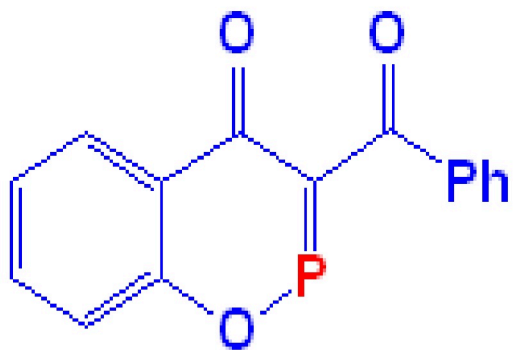
Dr_TarekElsayed-K8_CARBON_01
Dr_TarekElsayed-K8



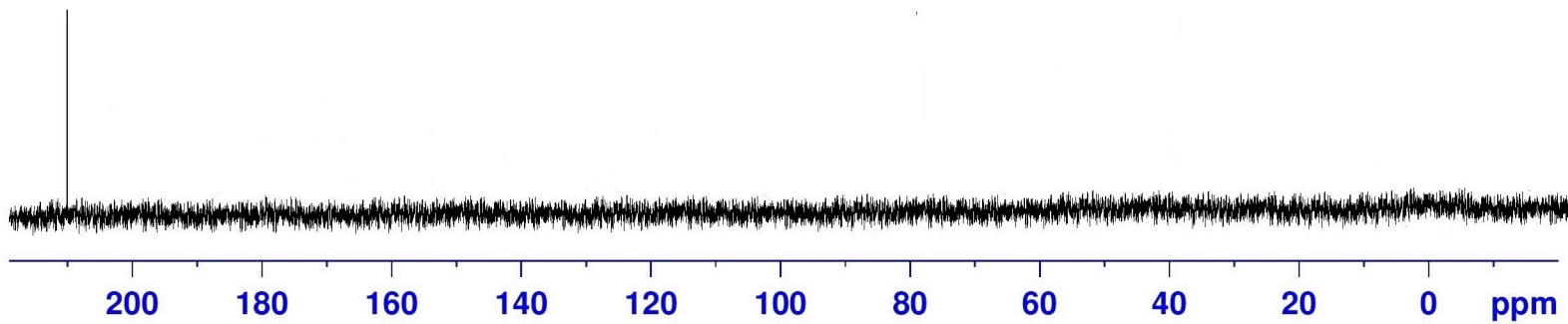
7



— 210.02



7



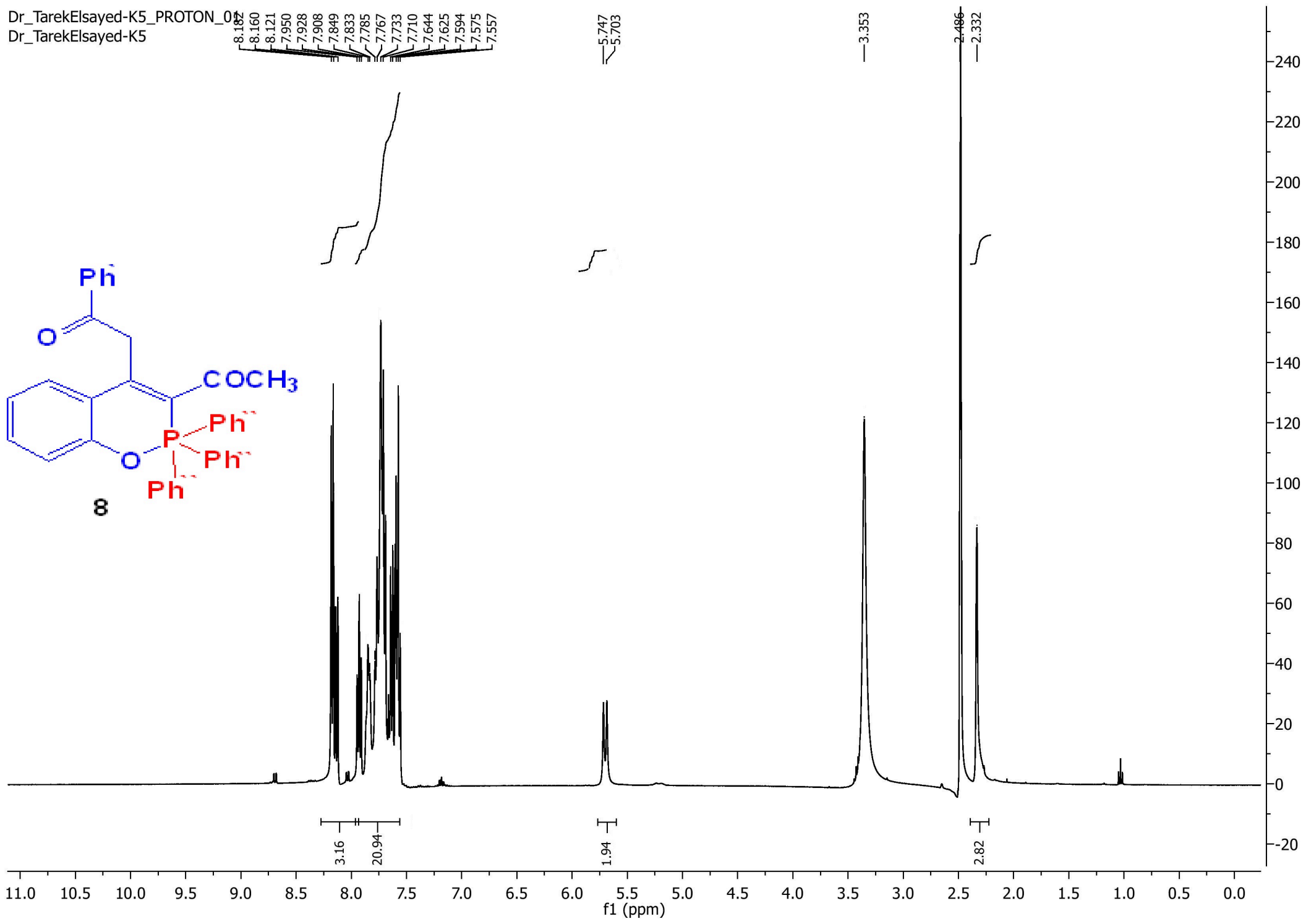
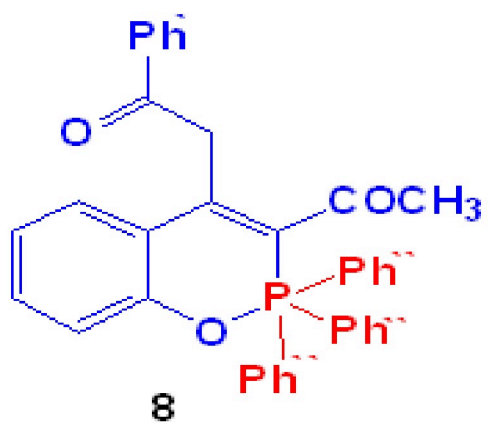
Dr_TarekElsayed-K5_PROTON_004
Dr_TarekElsayed-K5

8.182
8.160
8.121
7.950
7.928
7.908
7.849
7.833
7.785
7.767
7.733
7.710
7.644
7.625
7.594
7.575
7.557

5.747
5.703

3.353

2.486
2.332



Dr_TarekElsayed-K5_PROTON_01
Dr_TarekElsayed-K5

8.182
8.164
8.160
8.143
8.140
8.124
8.121

7.950
7.946
7.928
7.911
7.908

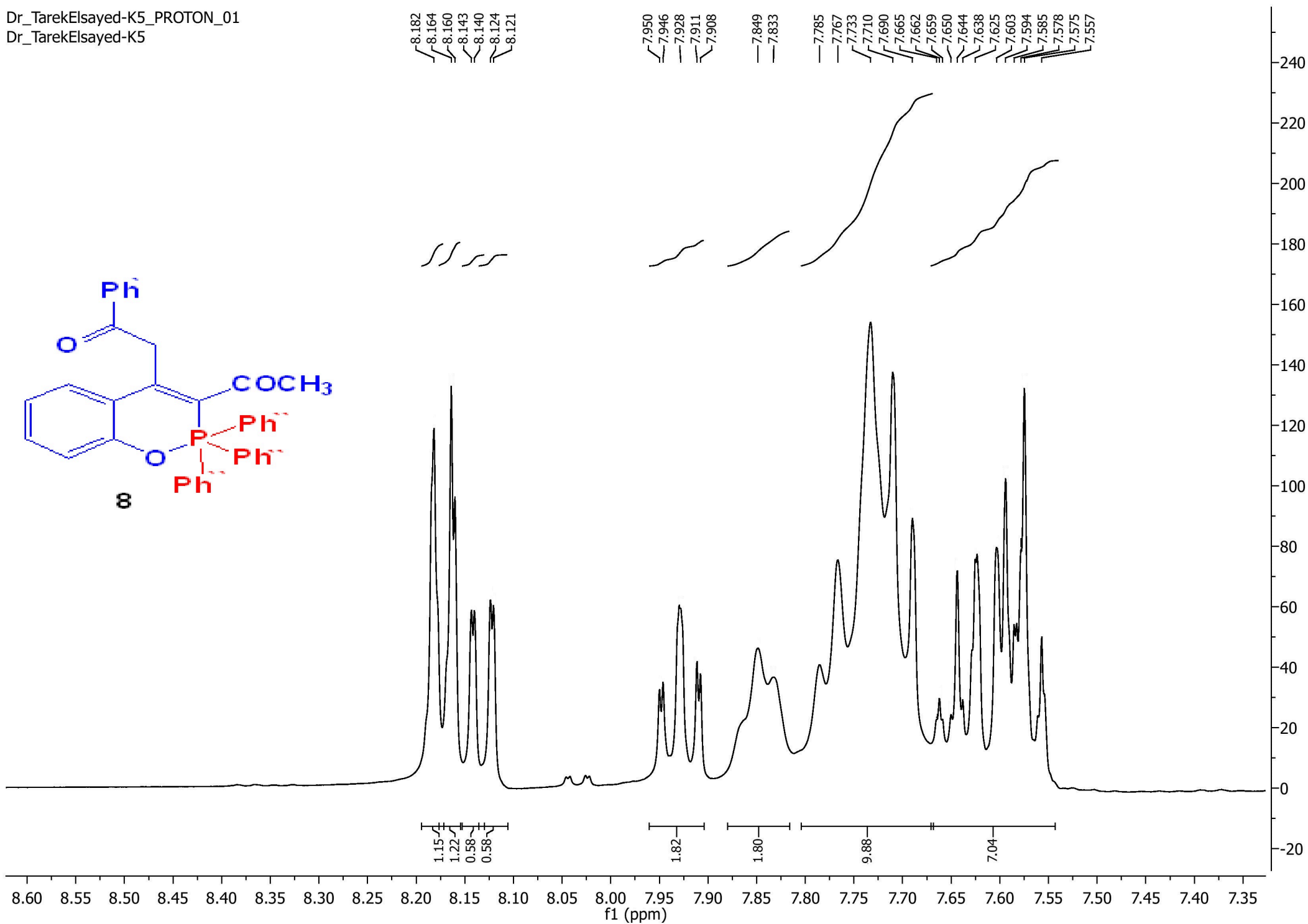
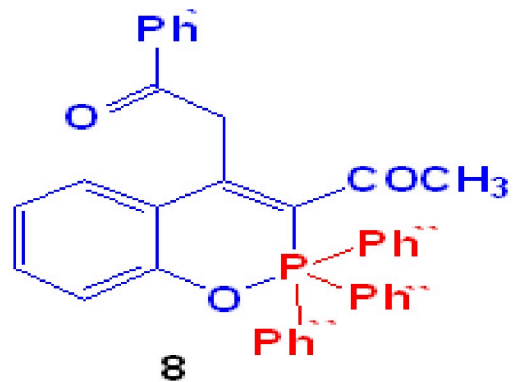
7.849
7.833

7.785
7.767
7.733

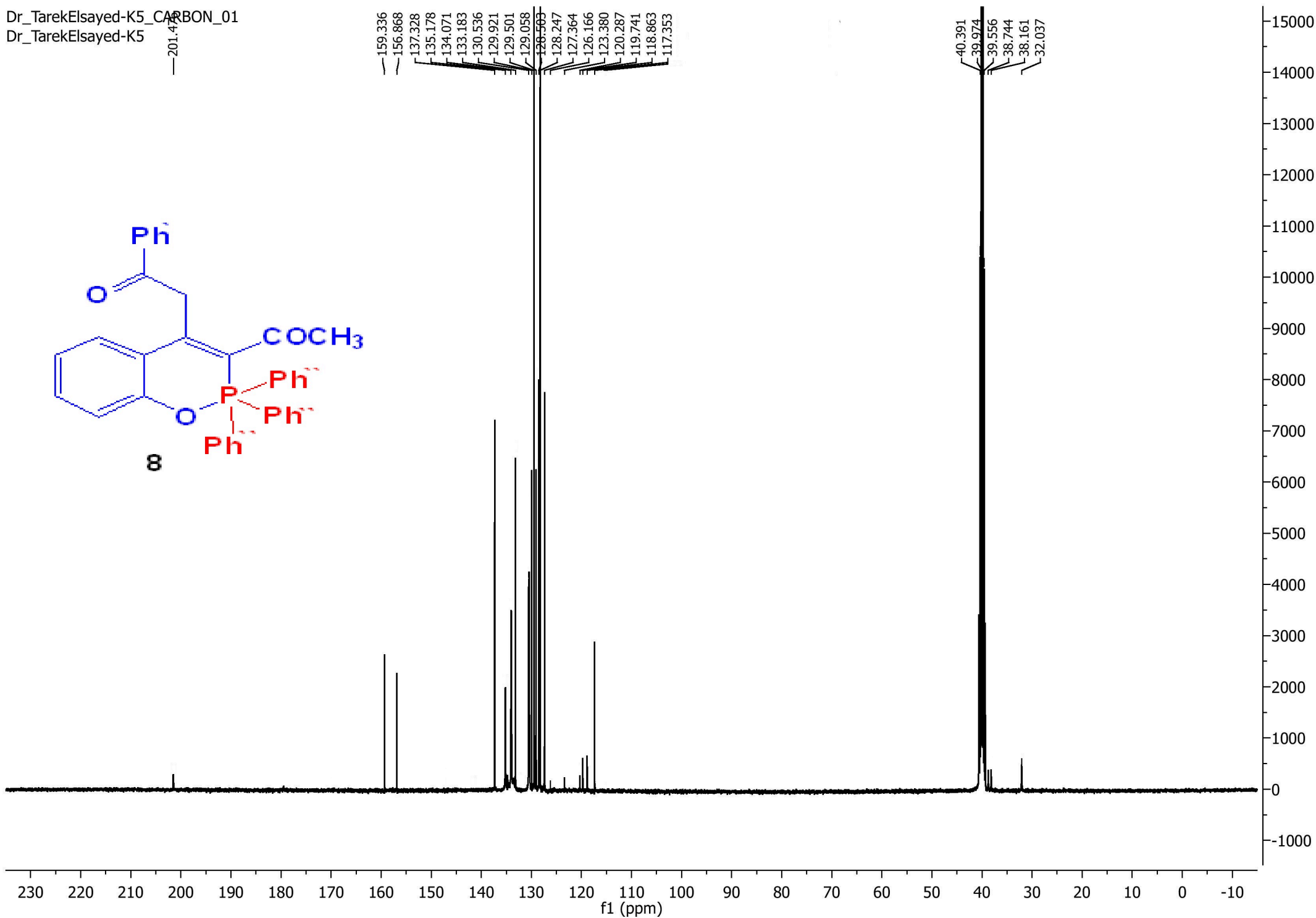
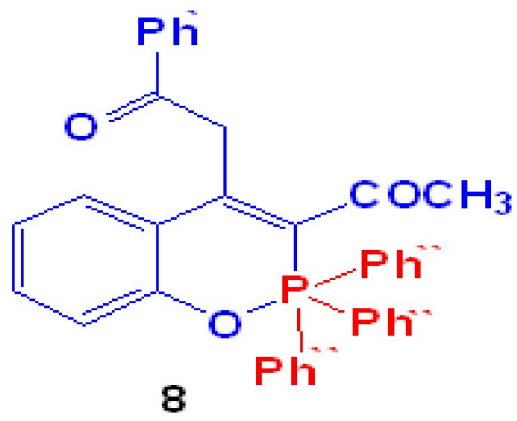
7.690
7.665
7.662
7.659

7.650
7.644
7.638
7.625
7.603

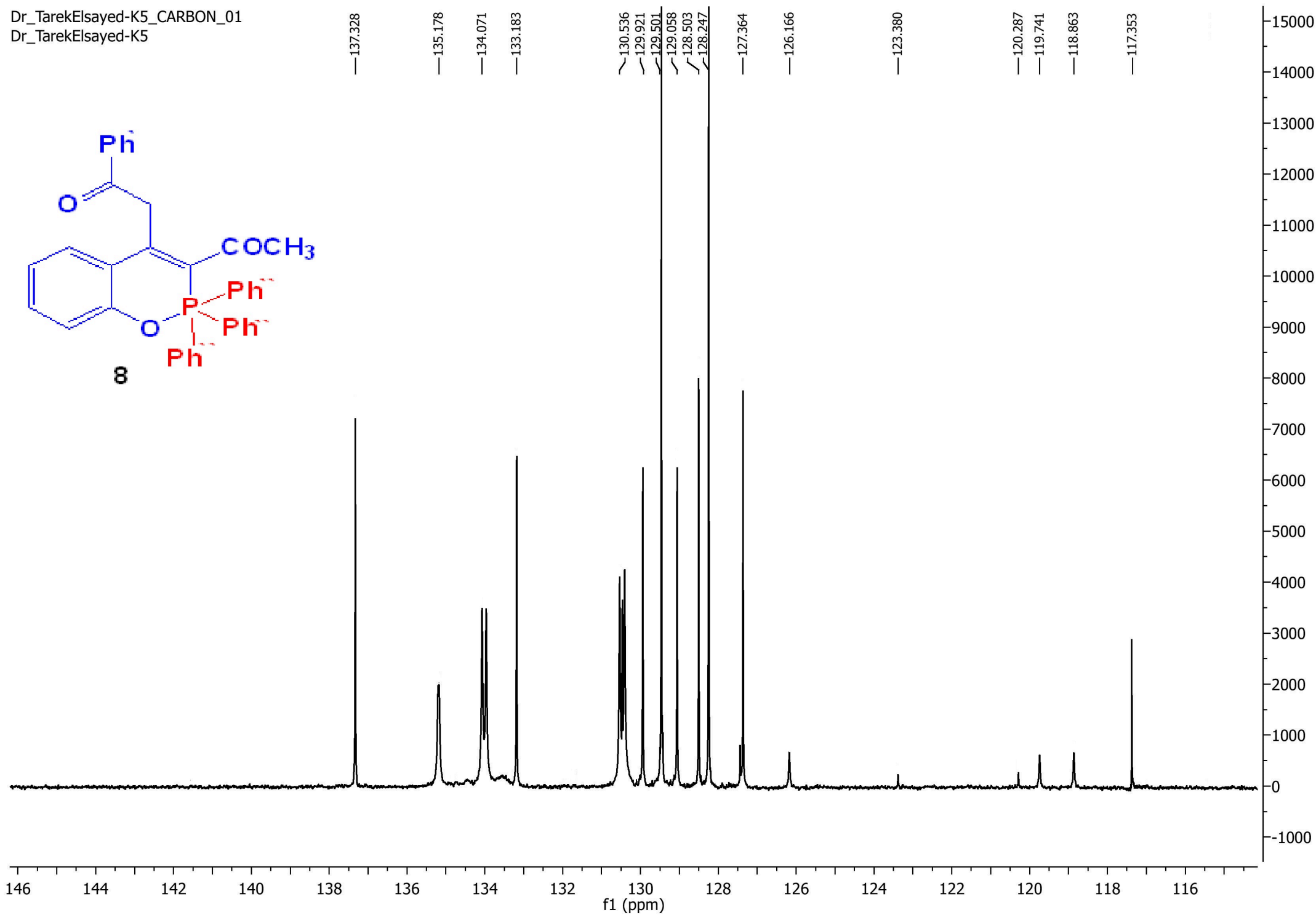
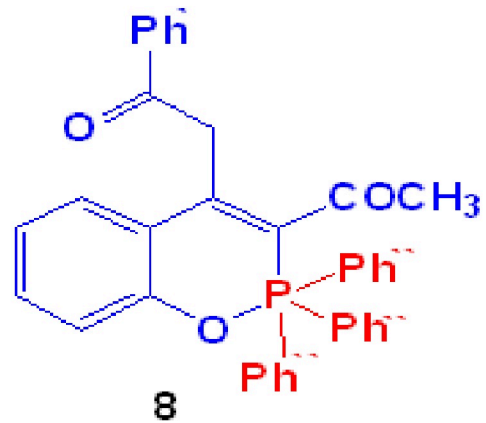
7.594
7.585
7.578
7.575
7.557



Dr_TarekElsayed-K5_CARBON_01
Dr_TarekElsayed-K5



Dr_TarekElsayed-K5_CARBON_01
Dr_TarekElsayed-K5



Dr_TarekElsayed-K2_PROTON_01
Dr_TarekElsayed-K2

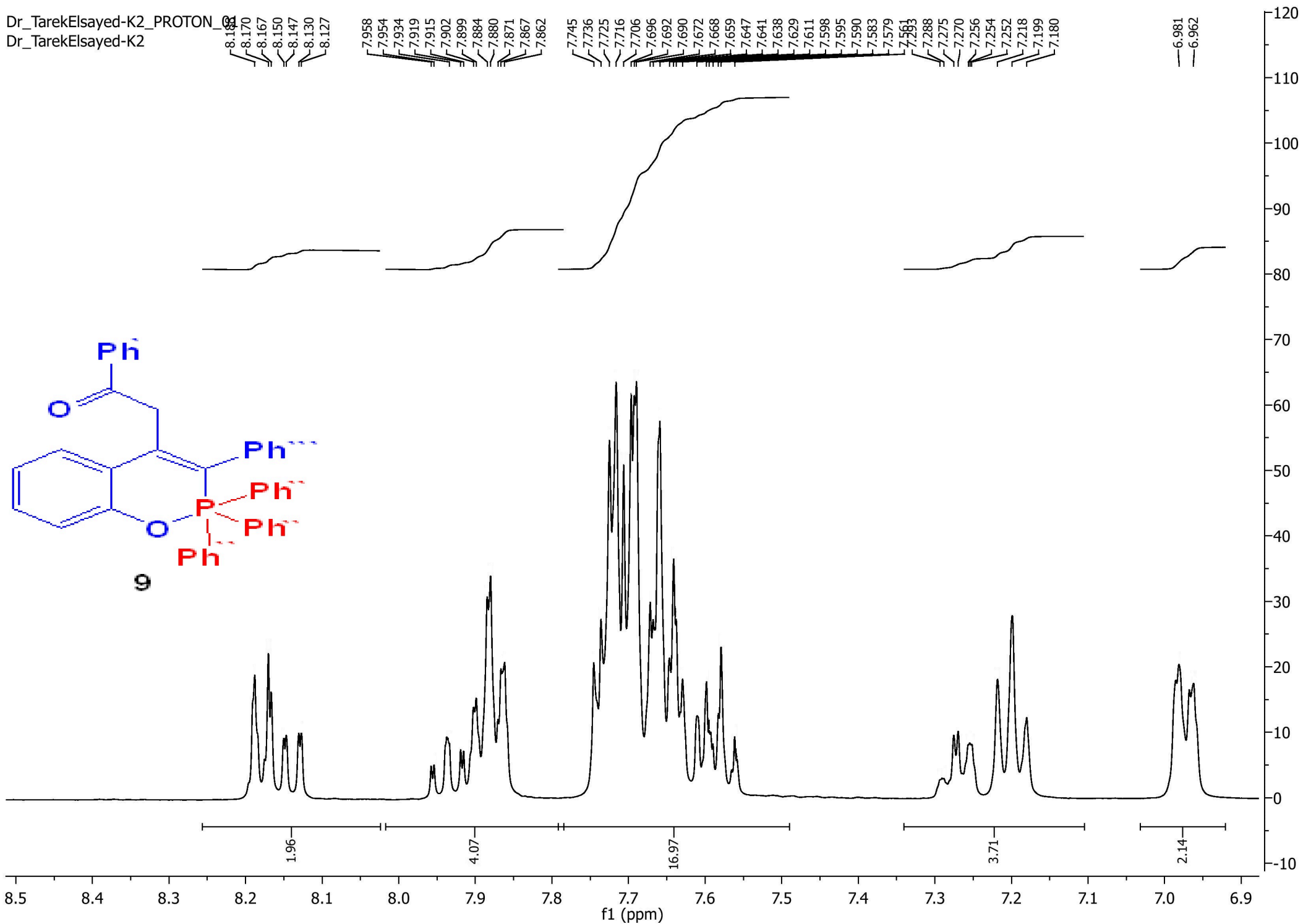
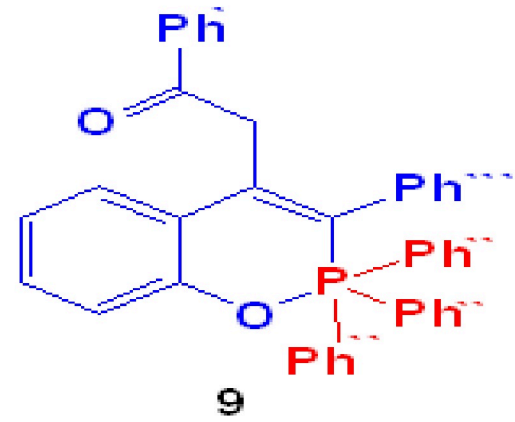
8.188
8.170
8.167
8.150
8.147
8.130
8.127

7.958
7.954
7.934
7.919
7.915
7.902
7.899
7.884
7.880
7.871
7.867
7.862

7.745
7.736
7.725
7.716
7.706
7.696
7.692
7.690
7.672
7.668
7.659
7.647
7.641
7.638
7.629
7.611
7.598
7.595
7.590
7.583
7.579
7.561
7.553

7.288
7.275
7.270
7.256
7.254
7.252
7.218
7.199
7.180

6.981
6.962

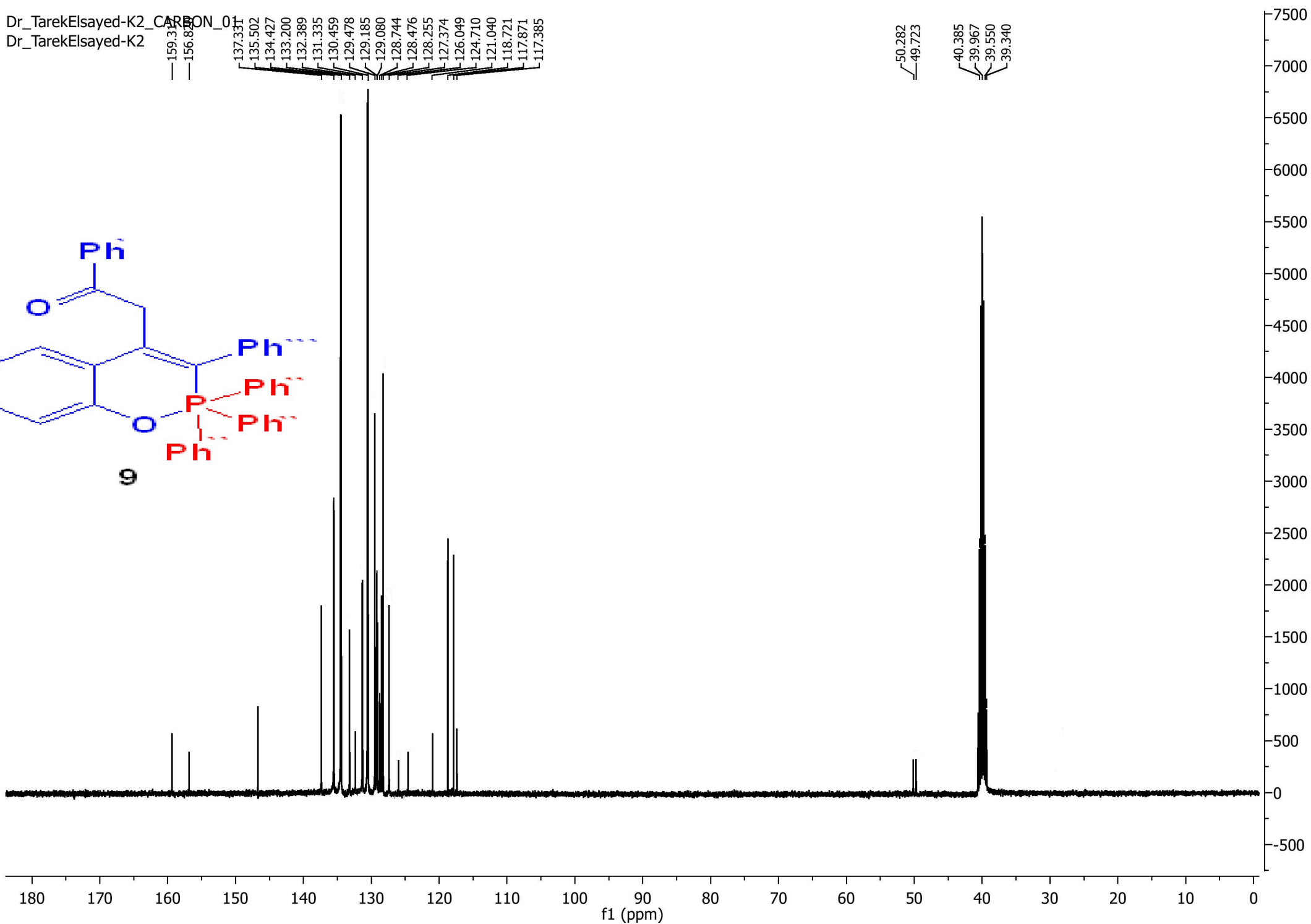
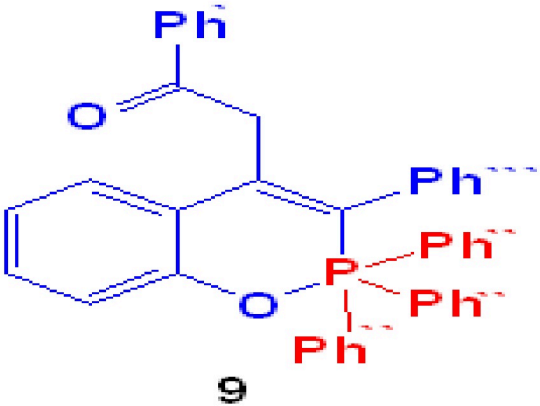


Dr_TarekElsayed-K2_CARBON_01
Dr_TarekElsayed-K2

159.315
156.872
137.331
135.502
134.427
133.200
132.389
131.335
130.459
129.478
129.185
129.080
128.744
128.476
128.255
127.374
126.049
124.710
121.040
118.721
117.871
117.385

50.282
49.723

40.385
39.967
39.550
39.340



Dr_TarekElsayed-K2_CARBON_01
Dr_TarekElsayed-K2

