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Gold(I) and Gold(III) Phosphine Complexes: Synthesis, Anticancer Activities Towards 2D and 3D Cancer Models, and Apoptosis Inducing Properties

Contents

Characterization of gold complexes (1-10)

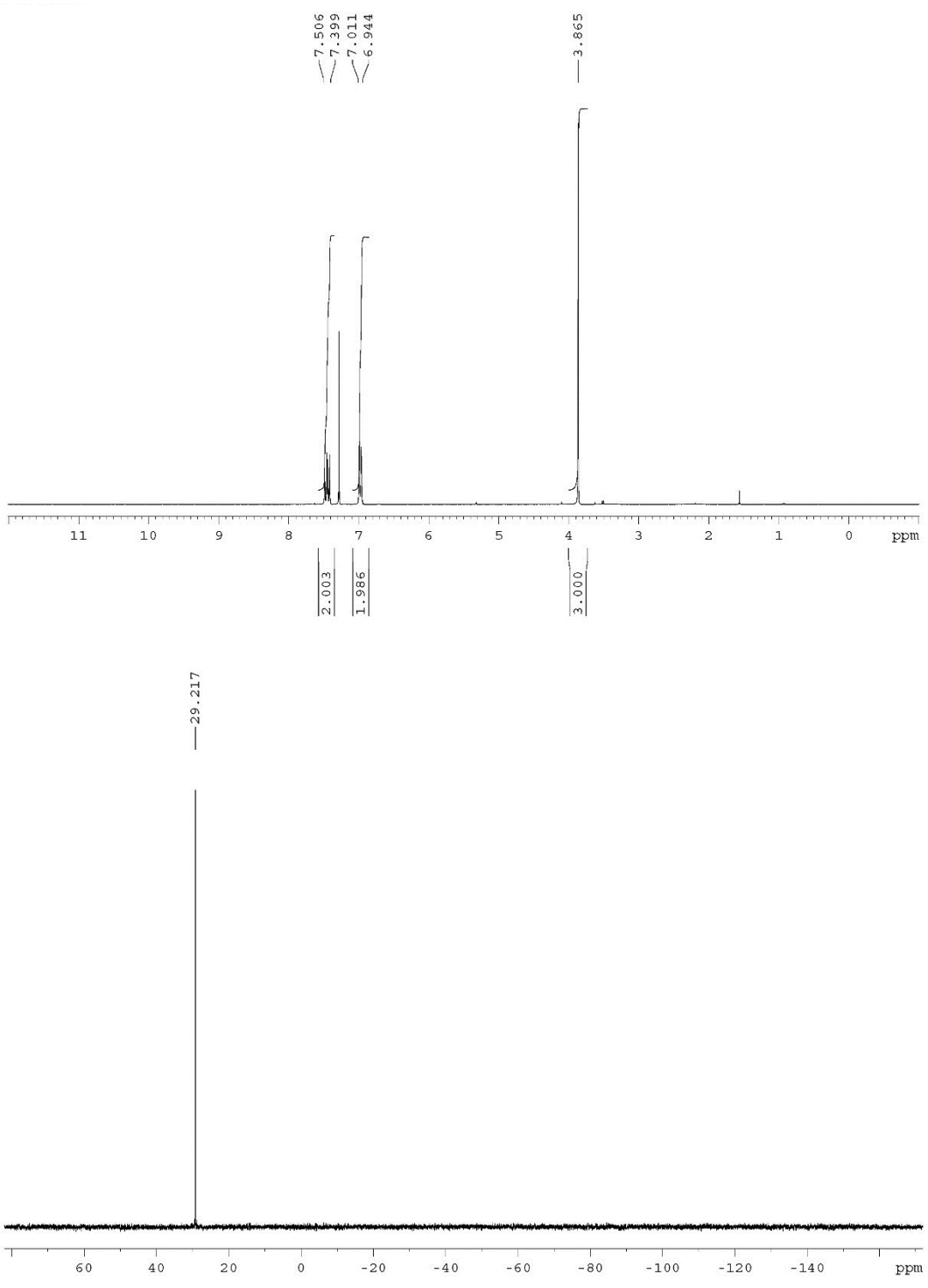
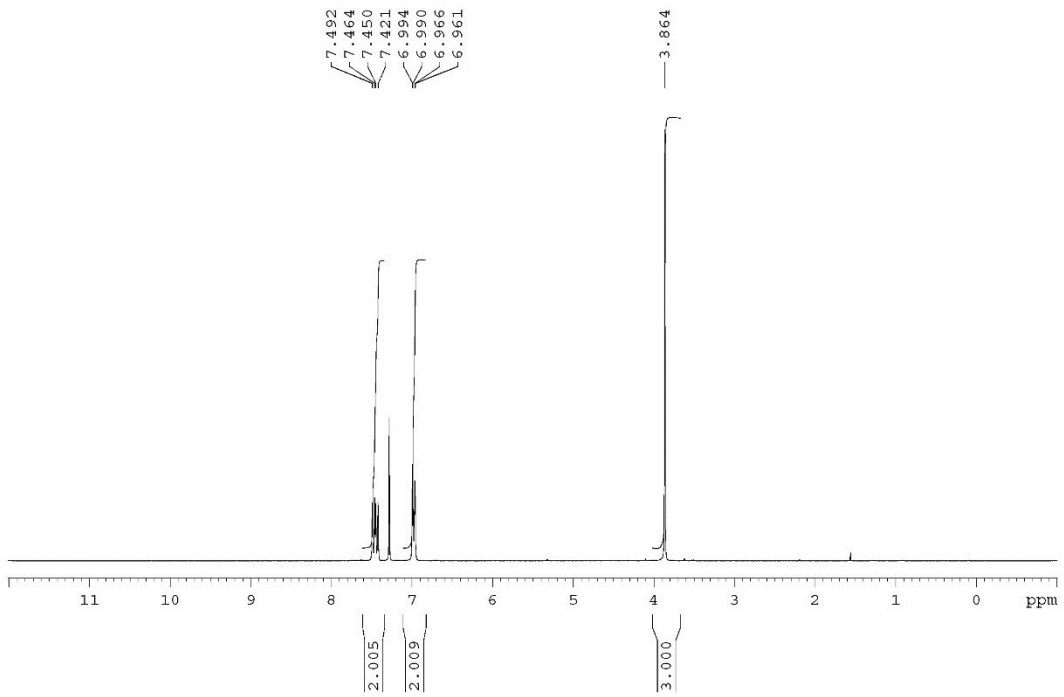


Figure S1. ^{1}H NMR and ^{31}P NMR spectra of complex **1**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7.421
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6.990
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9
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7
6
5
4
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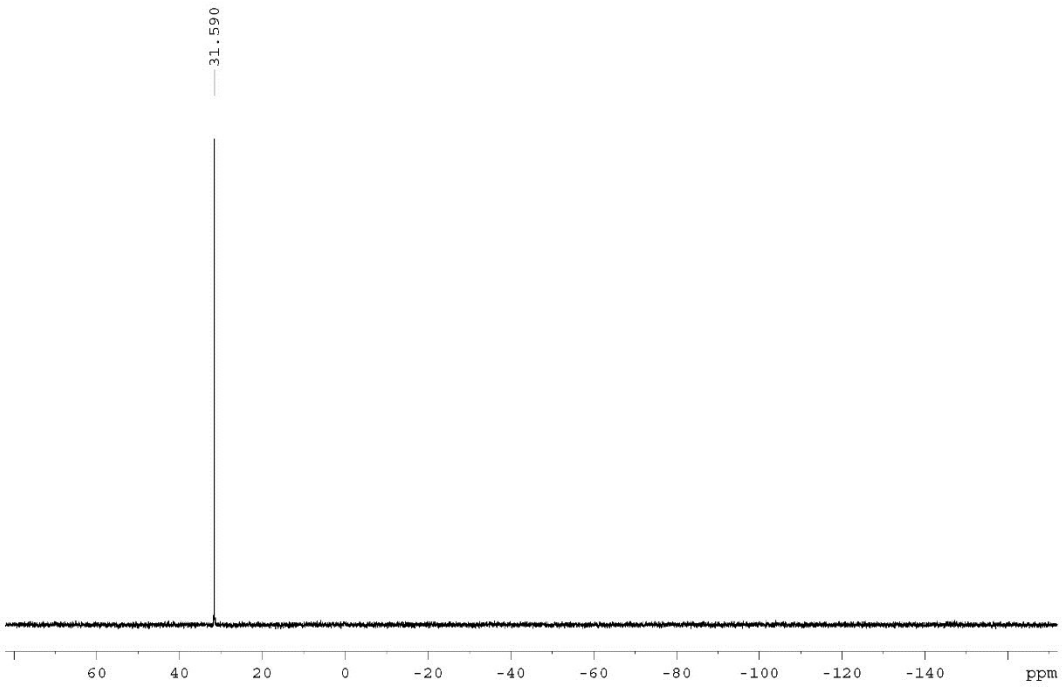
2
1
0
ppm
2.005
2.009
3.000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-20
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-60
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-100
-120
-140
ppm

Figure S2. ^{1}H NMR and ^{31}P NMR spectra of complex **2**

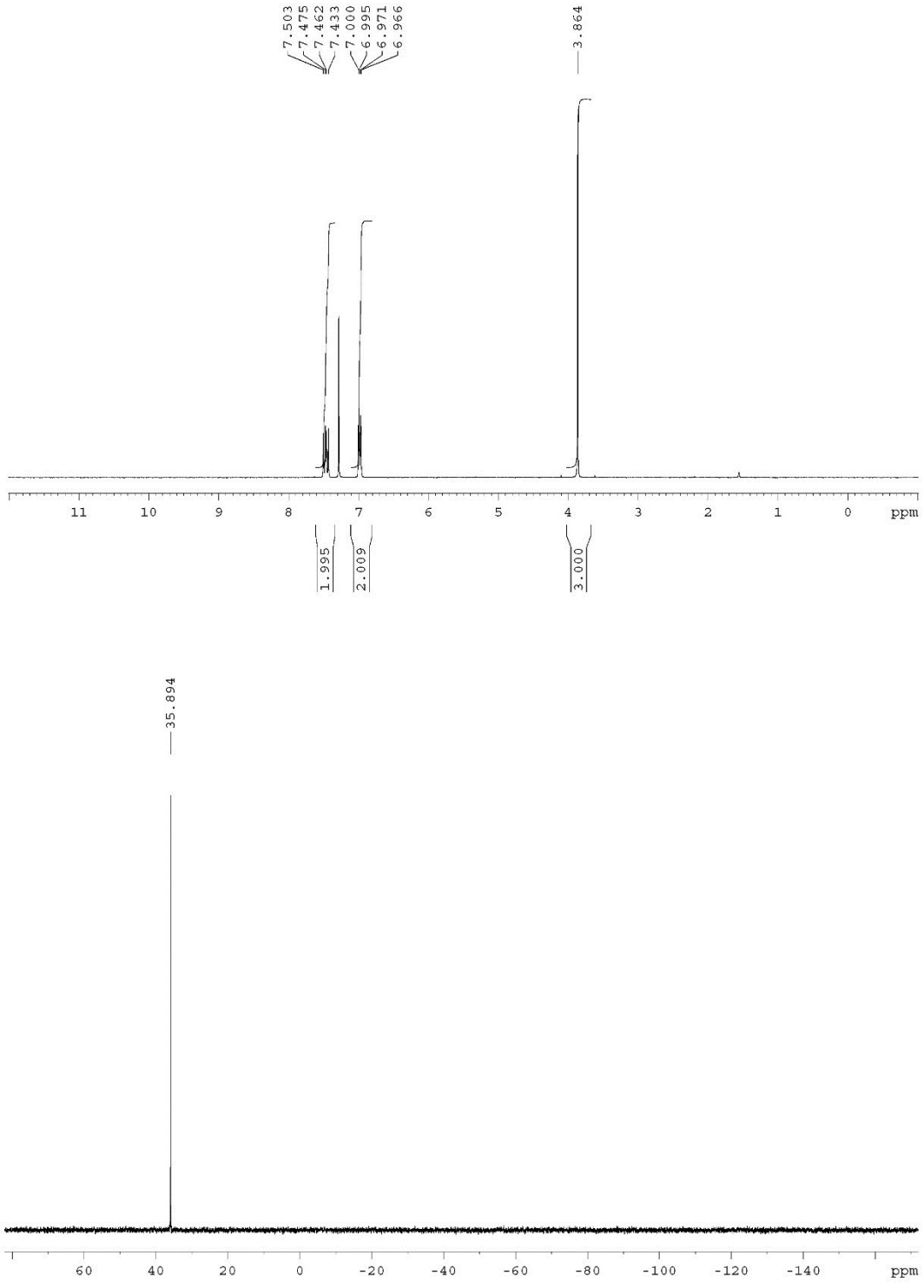

Figure S3. ^{1}H NMR and ^{31}P NMR spectra of complex **3**

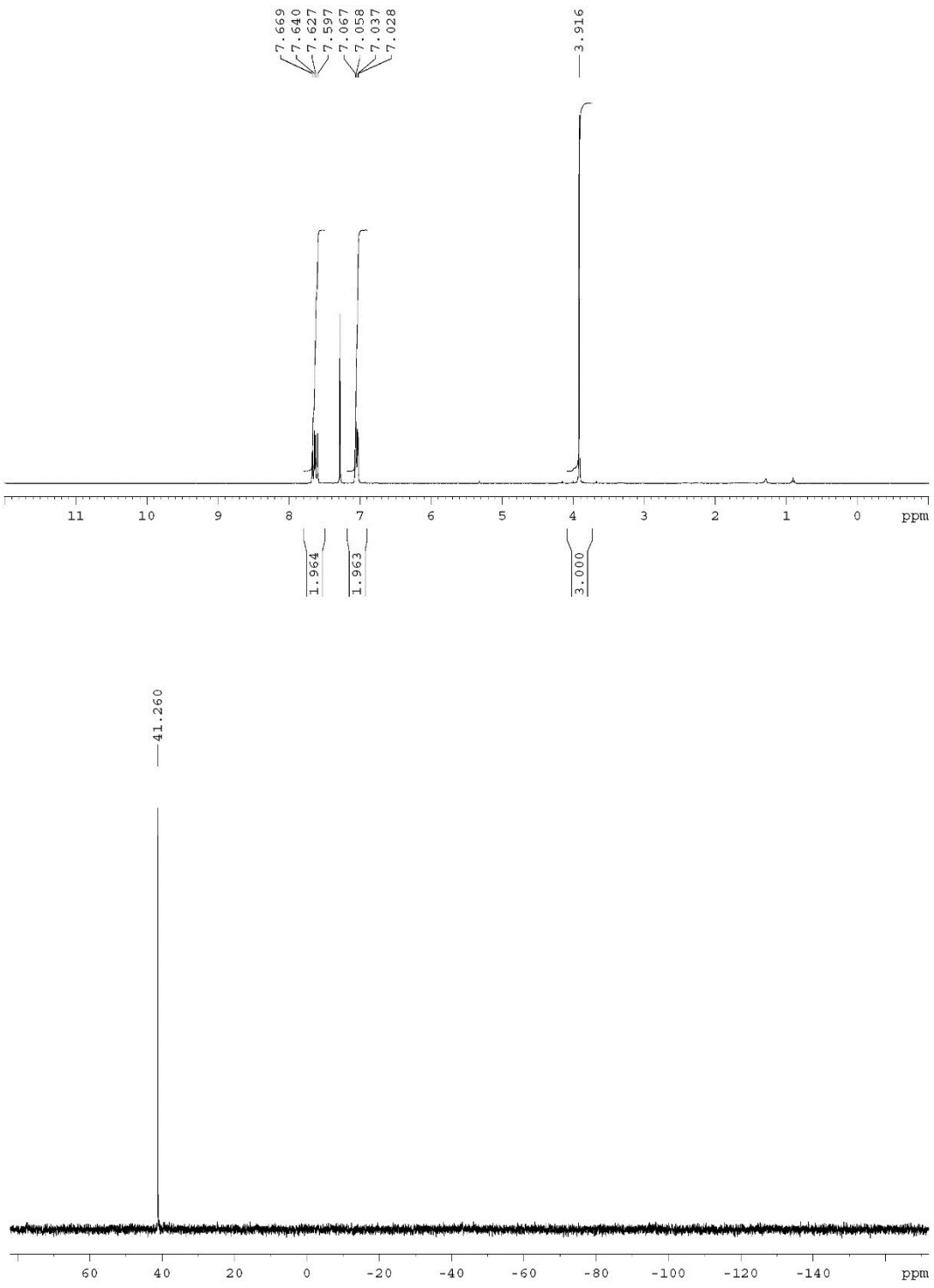

Figure S4. ^{1}H NMR and ^{31}P NMR spectra of complex **4**

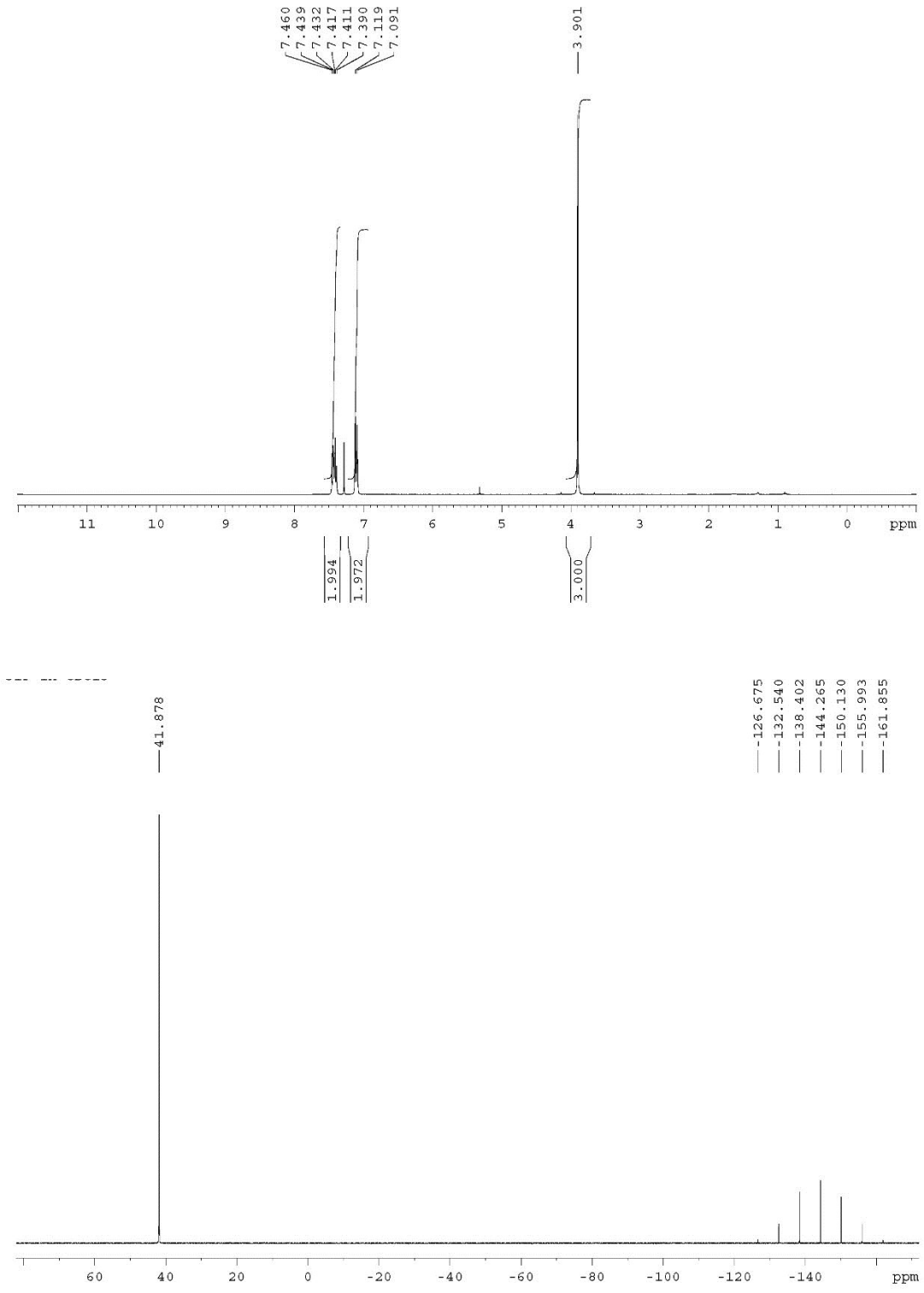

Figure S5. ^{1}H NMR and ^{31}P NMR spectra of complex **5**

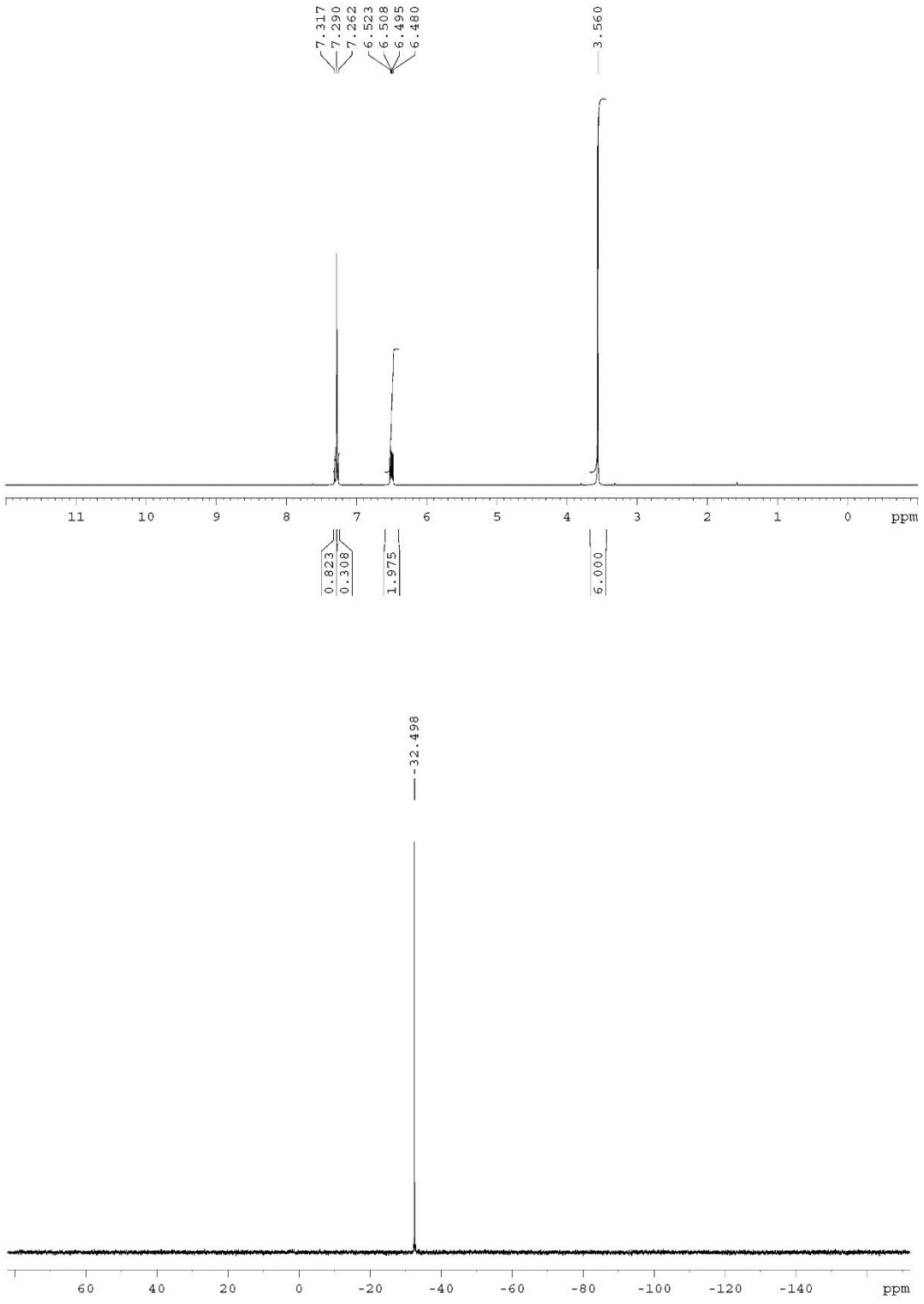

Figure S6. ^{1}H NMR and ^{31}P NMR spectra of complex **6**

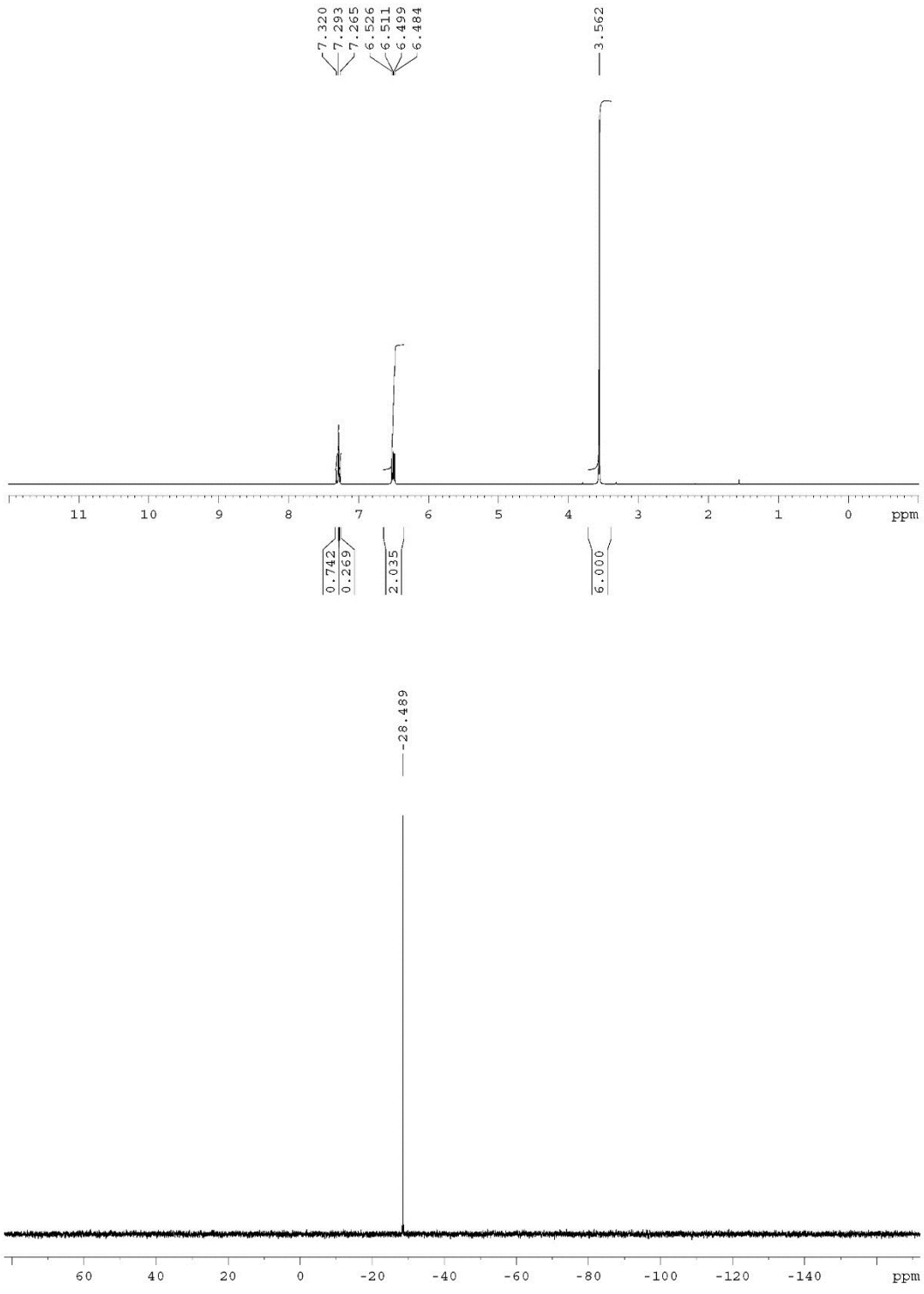

Figure S7. ^{1}H NMR and ^{31}P NMR spectra of complex **7**

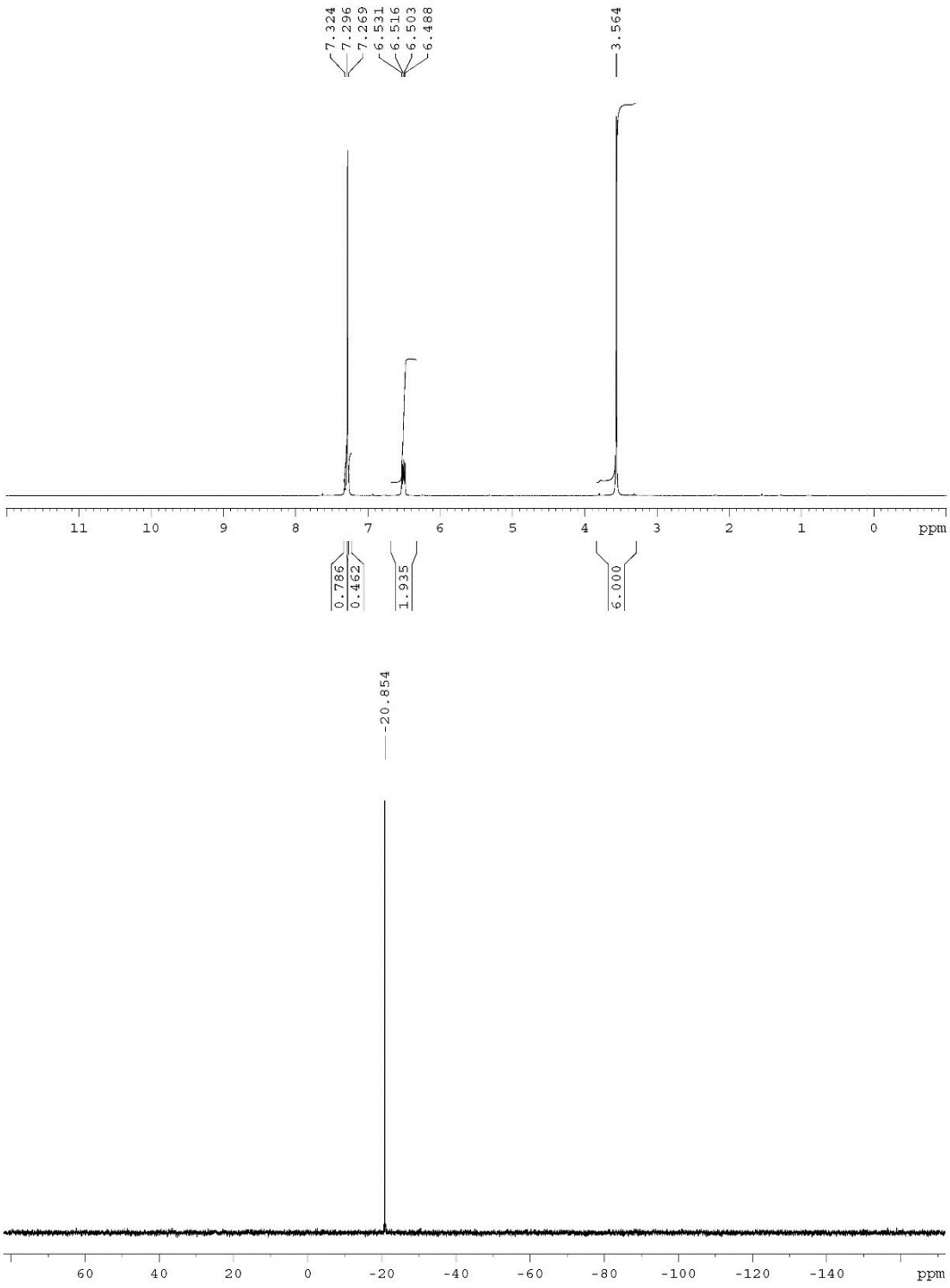

Figure S8. ^{1}H NMR and ^{31}P NMR spectra of complex **8**

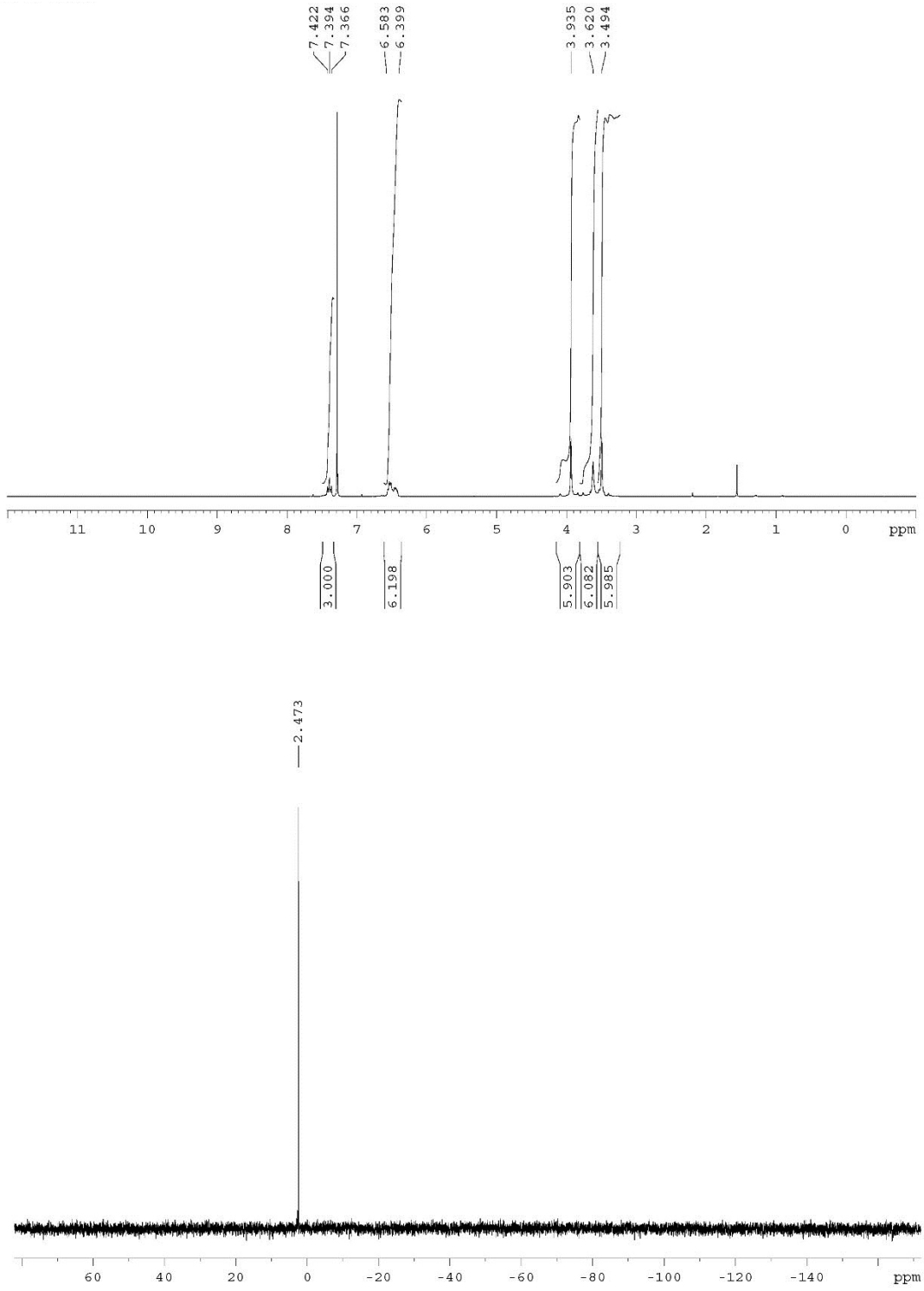

Figure S9. ^{1}H NMR and ^{31}P NMR spectra of complex **9**

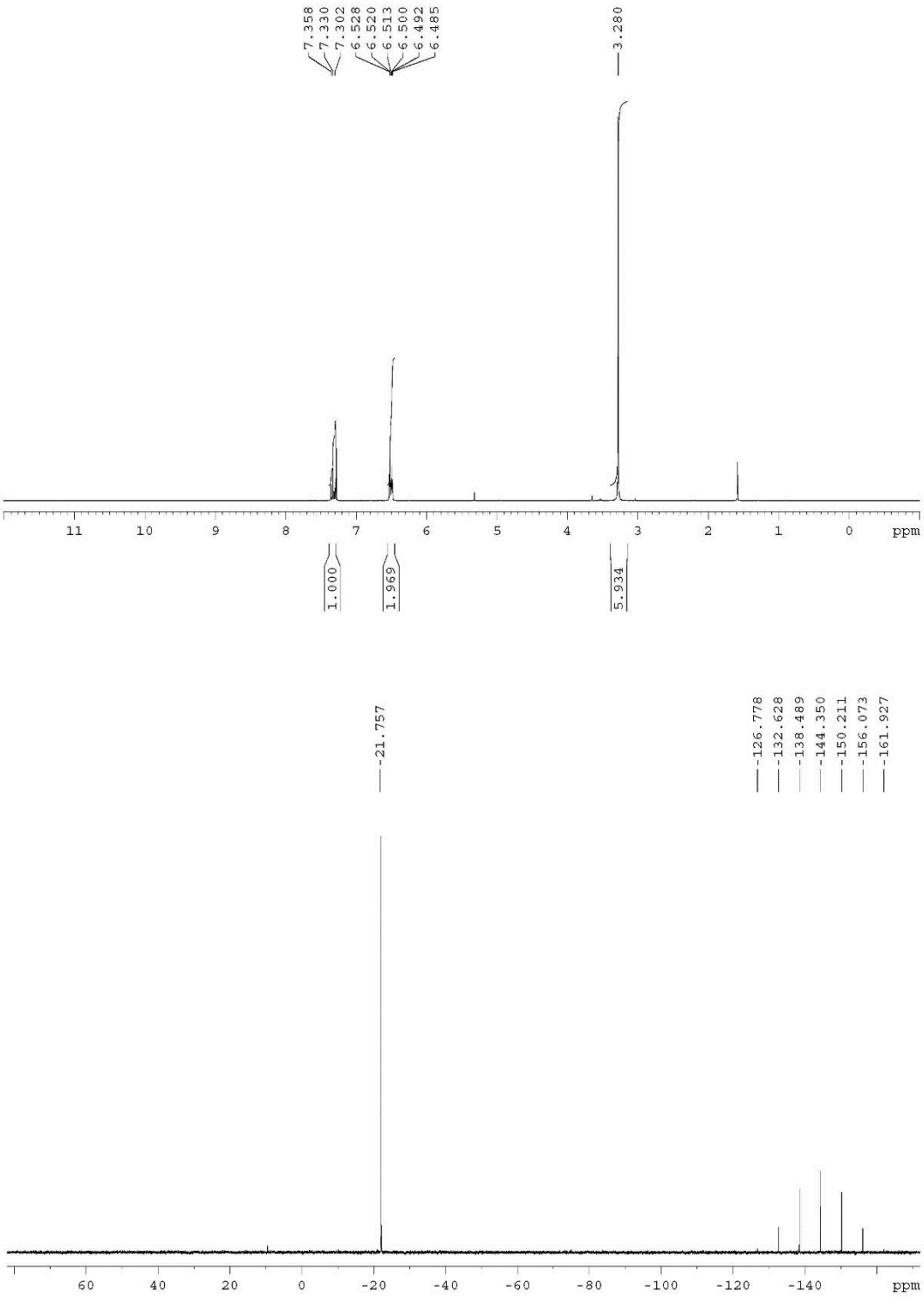

Figure S10. ^{1}H NMR and ^{31}P NMR spectra of complex **10**

Table S1. Crystal and structure refinement data for complexes **4-10**

Compound	**4**	**5**
Formula	$[AuCl_3\{P(C_6H_4\text{-}4\text{-}OMe)_3\}]$	$[Au\{P(C_6H_4\text{-}4\text{-}OMe)_3\}_2]PF_6$
fw	655.66	1131.56
Crystal system	triclinic	triclinic
Space group	P-1	P-1
a, Å	9.943(2)	9.5056(15)
b, Å	10.966(3)	11.8716(19)
c, Å	11.238(3)	20.347(3)
α, deg	72.292(5)	92.365(3)
β, deg	74.571(5)	95.981(3)
γ, deg	76.643(5)	102.177(4)
V, Å^3	1110.1(5)	2227.5(6)
Z	2	2
Cryst dimens (mm)	0.23 × 0.05 × 0.04	0.19 × 0.13 × 0.07
ρ_{calc} (g/cm^3)	1.962	1.687
μ (mm^{-1})	7.080	3.602
T, K	150(2)	150(2)
Radiation	Mo Kα (λ = 0.71073 Å)	Mo Kα (λ = 0.71073 Å)
2θ range for data collection	3.892 to 59.996	3.518 to 60
Index ranges	-13 ≤ h ≤ 13, -15 ≤ k ≤ 15, -15 ≤ l ≤ 15	-13 ≤ h ≤ 13, -16 ≤ k ≤ 16, -28 ≤ l ≤ 28
Reflections collected	45188	81312
Independent reflections	6462 [R_{int} = 0.0326, R_{sigma} = 0.0169]	13011 [R_{int} = 0.0358, R_{sigma} = 0.0221]
Data/restraints/parameters	6462/0/265	13011/42/572
Goodness-of-fit on F^2	1.048	1.033
Final R indexes [I>=2σ (I)]	R_1 = 0.0142, wR_2 = 0.0364	R_1 = 0.0236, wR_2 = 0.0585
Final R indexes [all data]	R_1 = 0.0155, wR_2 = 0.0370	R_1 = 0.0280, wR_2 = 0.0608
Largest diff. peak/hole / e Å^{-3}	1.88/-0.47	0.99/-1.04

Compound	**6**	**7**
Formula	$[AuCl\{P(C_6H_3\text{-}2,6\text{-}\{OMe\}_2)_3\}]$	$[AuBr\{P(C_6H_3\text{-}2,6\text{-}\{OMe\}_2)_3\}]$
fw	674.84	804.23
Crystal system	triclinic	orthorhombic
Space group	P-1	$Pna2_1$
a, Å	10.874(2)	14.422(3)
b, Å	14.940(3)	16.702(3)
c, Å	16.308(3)	11.860(2)
α, deg	107.912(4)	90
β, deg	91.326(4)	90
γ, deg	93.609(4)	90
V, Å^3	2513.4(9)	2856.9(9)
Z	4	4
Cryst dimens (mm)	0.29 × 0.06 × 0.04	0.27 × 0.08 × 0.08
ρ_{calc} (g/cm^3)	1.783	1.870
μ (mm^{-1})	6.060	6.828
T, K	150(2)	150(2)
Radiation	Mo Kα (λ = 0.71073 Å)	Mo Kα (λ = 0.71073 Å)
2θ range for data collection	3.238 to 65	3.732 to 64.998
Index ranges	-16 ≤ h ≤ 16, -22 ≤ k ≤ 22, -24 ≤ l ≤ 24	-21 ≤ h ≤ 21, -25 ≤ k ≤ 25, -17 ≤ l ≤ 17
Reflections collected	125615	73847
Independent reflections	18205 [R_{int} = 0.0333, R_{sigma} = 0.0207]	10314 [R_{int} = 0.0282, R_{sigma} = 0.0275]
Data/restraints/parameters	18205/0/607	10314/8/348
Goodness-of-fit on F^2	1.015	0.947
Final R indexes [I>=2σ (I)]	R_1 = 0.0170, wR_2 = 0.0354	R_1 = 0.0129, wR_2 = 0.0295
Final R indexes [all data]	R_1 = 0.0229, wR_2 = 0.0370	R_1 = 0.0151, wR_2 = 0.0300
Largest diff. peak/hole / e Å^{-3}	0.67/-0.56	0.59/-0.51

Compound	**8**	**9**
Formula	[[AuI{P(C_6H_3-2,6-{OMe}$_2$)$_3$}]	[$AuCl_3${P(C_6H_3-2,6-{OMe}$_2$)$_3$}]
fw	851.22	830.67
Crystal system	orthorhombic	monoclinic
Space group	$Pna2_1$	$P2_1/n$
a, Å	14.496(7)	10.764(2)
b, Å	16.885(8)	20.118(4)
c, Å	11.951(5)	14.621(3)
α, deg	90	90
β, deg	90	107.320(4)
γ, deg	90	90
V, Å^3	2925(2)	3022.4(11)
Z	4	4
Cryst dimens (mm)	0.31 × 0.16 × 0.09	0.18 × 0.11 × 0.06
ρ_{calc} (g/cm^3)	1.933	1.826
μ (mm^{-1})	6.358	5.400
T, K	150(2)	150(2)
Radiation	Mo Kα (λ = 0.71073 Å)	Mo Kα (λ = 0.71073 Å)
2θ range for data collection	3.702 to 54.99	3.552 to 65
Index ranges	-18 ≤ h ≤ 18, -19 ≤ k ≤ 21, -15 ≤ l ≤ 15	-16 ≤ h ≤ 14, -30 ≤ k ≤ 30, -22 ≤ l ≤ 22
Reflections collected	42149	73853
Independent reflections	6638 [R_{int} = 0.0249, R_{sigma} = 0.0270]	10938 [R_{int} = 0.0333, R_{sigma} = 0.0209]
Data/restraints/parameters	6638/8/361	10938/0/349
Goodness-of-fit on F^2	0.943	1.017
Final R indexes [I>=2σ (I)]	R_1 = 0.0100, wR_2 = 0.0258	R_1 = 0.0213, wR_2 = 0.0444
Final R indexes [all data]	R_1 = 0.0103, wR_2 = 0.0265	R_1 = 0.0281, wR_2 = 0.0467
Largest diff. peak/hole / e Å^{-3}	0.33/-0.43	1.84/-1.21

Compound	**10**
Formula	[Au{P(C_6H_3-2,6-{OMe}$_2$)$_3$}$_2$]PF_6
fw	1311.71
Crystal system	triclinic
Space group	P-1
a, Å	13.737(4)
b, Å	14.454(5)
c, Å	16.002(5)
α, deg	68.210(7)
β, deg	75.034(7)
γ, deg	62.753(6)
V, Å^3	2607.9(14)
Z	2
Cryst dimens (mm)	0.21 × 0.12 × 0.04
ρ_{calc} (g/cm^3)	1.670
μ (mm^{-1})	3.097
T, K	150(2)
Radiation	Mo Kα (λ = 0.71073 Å)
2θ range for data collection	2.756 to 54.998
Index ranges	-17 ≤ h ≤ 17, -18 ≤ k ≤ 18, -20 ≤ l ≤ 20
Reflections collected	79211
Independent reflections	11987 [R_{int} = 0.0772, R_{sigma} = 0.0419]
Data/restraints/parameters	11987/56/671
Goodness-of-fit on F^2	1.066
Final R indexes [I>=2σ (I)]	R_1 = 0.0307, wR_2 = 0.0720
Final R indexes [all data]	R_1 = 0.0348, wR_2 = 0.0739
Largest diff. peak/hole / e Å^{-3}	1.63/-1.12

Crystal structure diagrams of the complexes

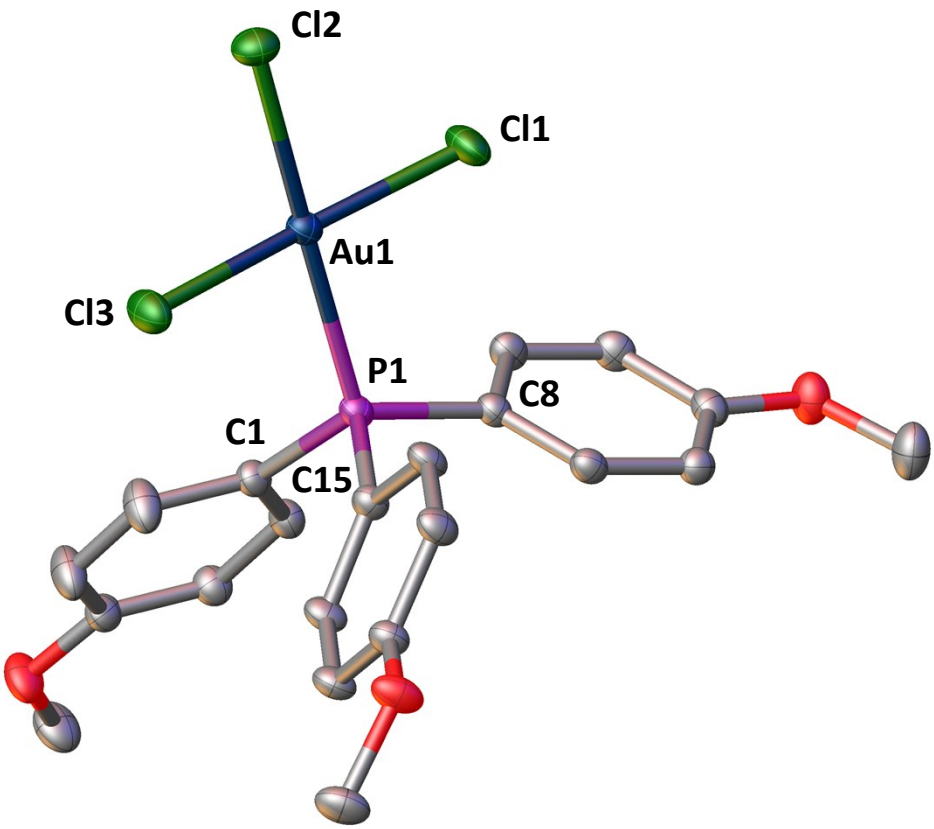

Figure S11. Molecular structure of $[AuCl_3\{P(C_6H_4\text{-}4\text{-}OMe)_3\}]$ (**4**). Ellipsoids show 50% probability levels and hydrogen atoms have been omitted for clarity. Selected bond distances (Å) and angles (°): Au(1)-P(1) 2.3394(6), Au(1)-Cl(1) 2.2766(7), Au(1)-Cl(2) 2.3668(6), Au(1)-Cl(3) 2.2907(7), Cl(2)-Au(1)-P(1) 176.687(16).

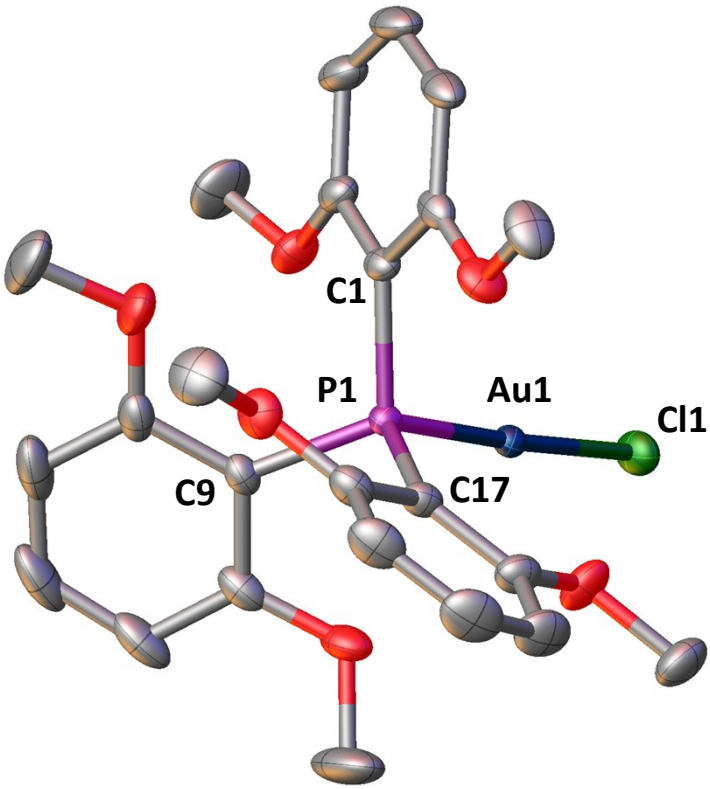

Figure S12. Molecular structure of [AuCl{P(C_6H_3-2,6-{OMe}$_2$)$_3$}] (**6**). Ellipsoids show 50% probability levels and hydrogen atoms have been omitted for clarity. Selected bond distances (Å) and angles (°): Au(1)-P(1) 2.2416(5), Au(1)-Cl1(1) 2.2965(5), Cl(1)-Au(1)-P(1) 177.099(15).

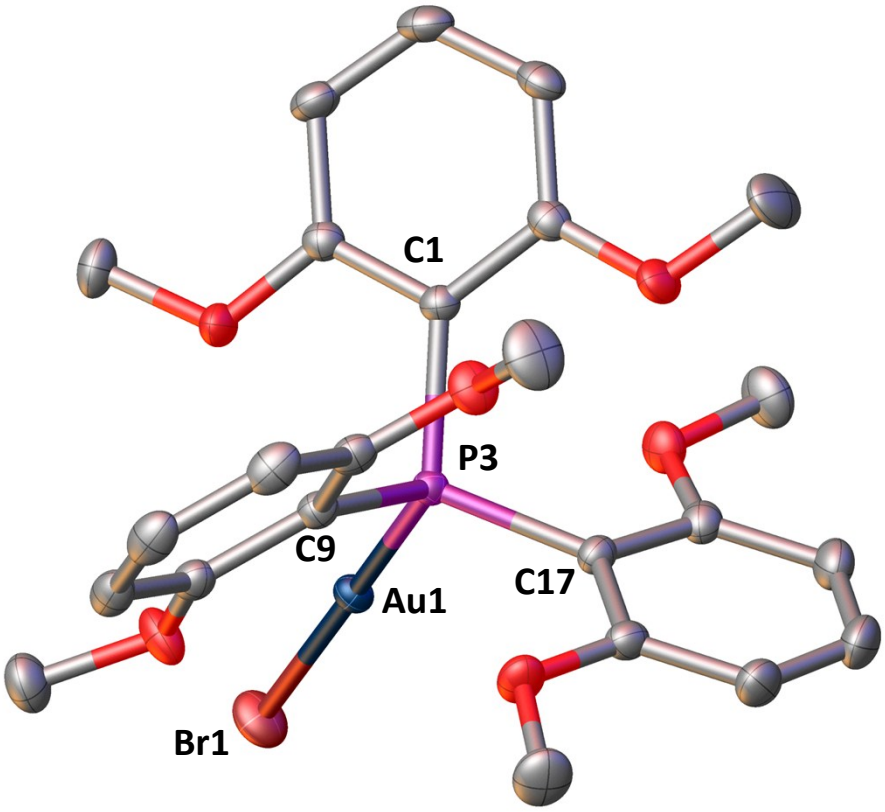

Figure S13. Molecular structure of [AuBr{P(C_6H_3-2,6-{OMe}$_2$)$_3$}] (**7**). Ellipsoids show 50% probability levels and hydrogen atoms have been omitted for clarity. Selected bond distances (Å) and angles (°): Au(1)-P(3) 2.2530(7), Au(1)-Br1(1) 2.4227(5), Br(1)-Au(1)-P(3) 175.510(15).

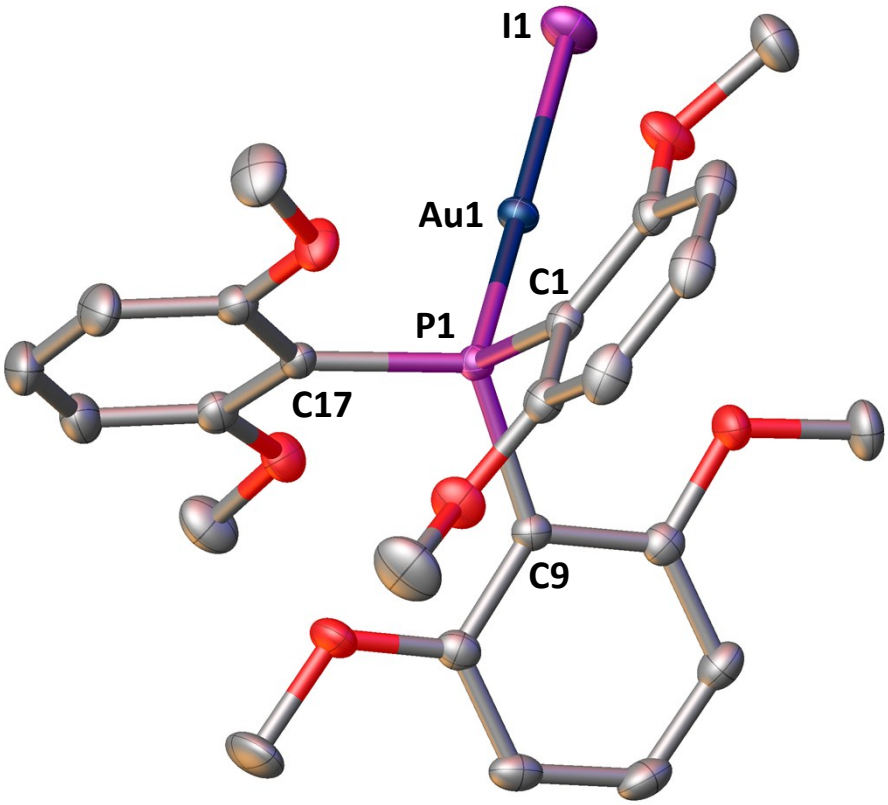

Figure S14. Molecular structure of [AuI{P(C_6H_3-2,6-{OMe}$_2$)$_3$}] (**8**). Ellipsoids show 50% probability levels and hydrogen atoms have been omitted for clarity. Selected bond distances (Å) and angles (°): Au(1)-P(1) 2.2662(12), Au(1)-I1(1) 2.5687(10), I(1)-Au(1)-P(1) 174.268(18).

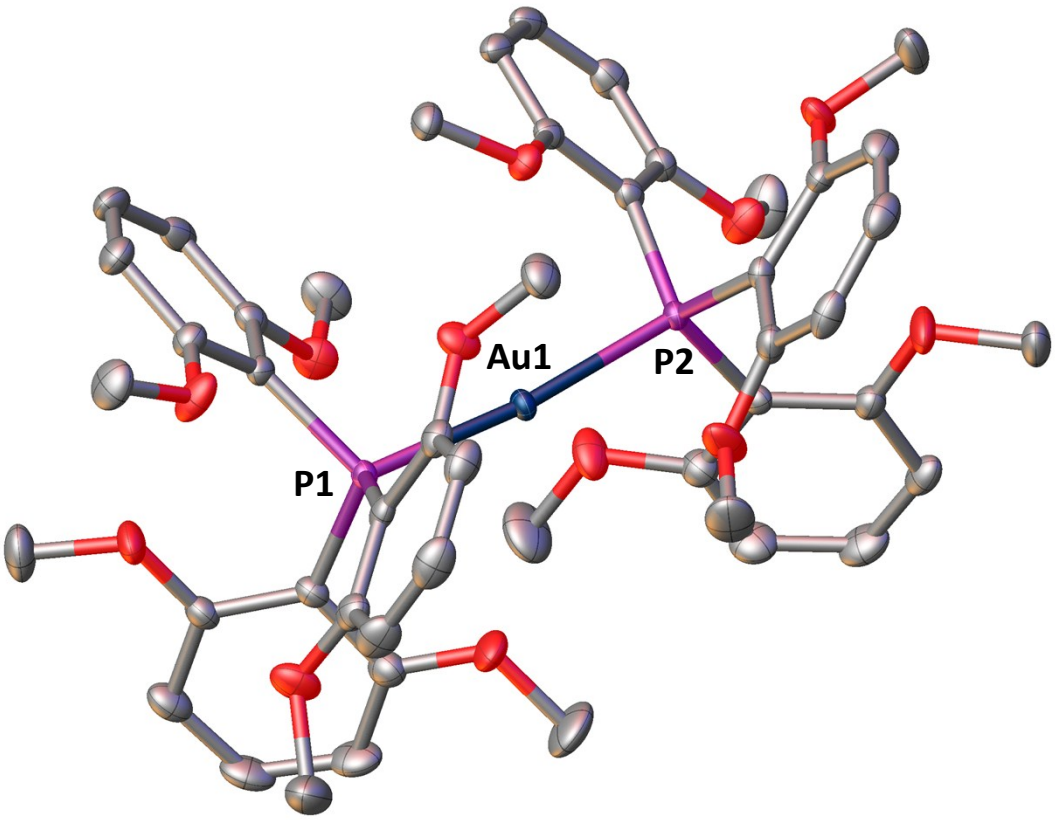

Figure S15. Molecular structure of $[Au\{P(C_6H_3\text{-}2,6\text{-}\{OMe\}_2)_3\}_2]PF_6$ (**10**). Ellipsoids show 50% probability levels. Hydrogen atoms and PF_6 counter ion have been omitted for clarity. Selected bond distances (Å) and angles (°): Au(1)-P(1) 2.3250(10), Au(1)-P(2) 2.3381(10), P(1)-Au(1)-P(2) 173.56(3).

Stability studies in DMSO-d_6

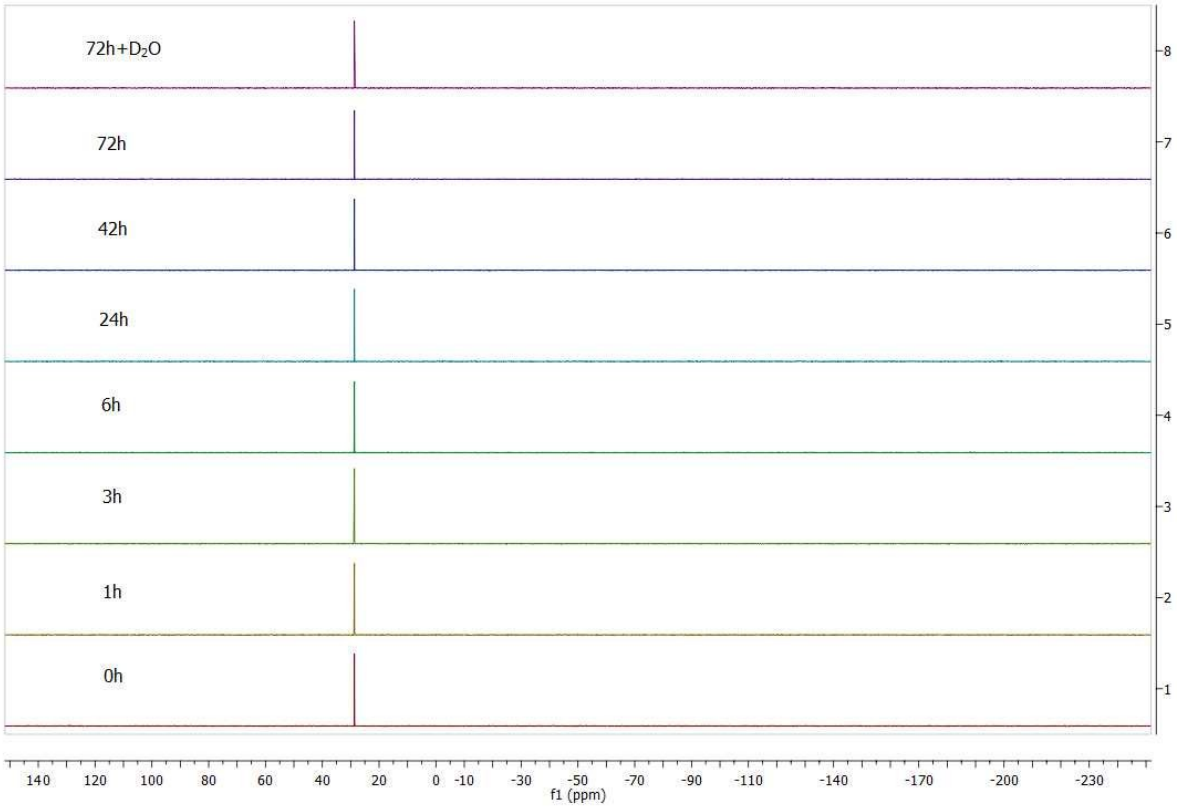

Figure S16. ^{31}P NMR spectrum of **1** in DMSO-d_6 over a 72 h time period

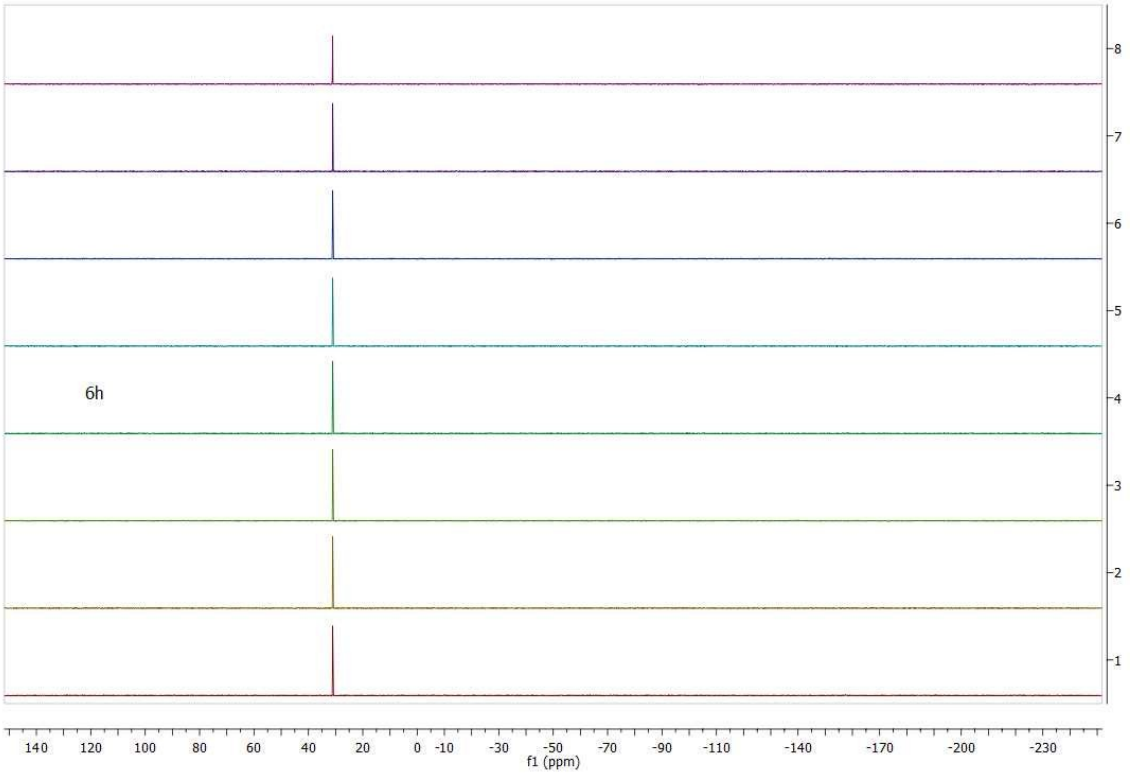

Figure S17. ^{31}P NMR spectrum of **2** in DMSO-d_6 over a 72 h time period

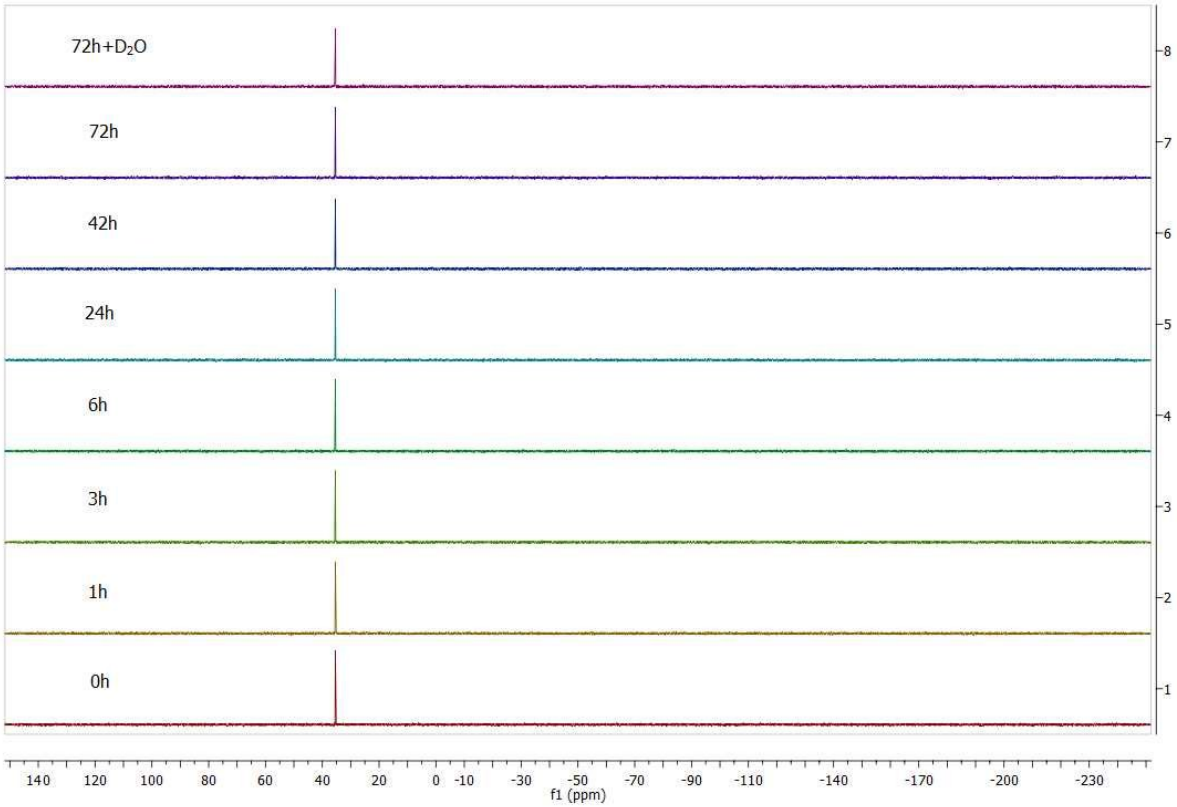

Figure S18. ^{31}P NMR spectrum of **3** in DMSO-d_6 over a 72 h time period

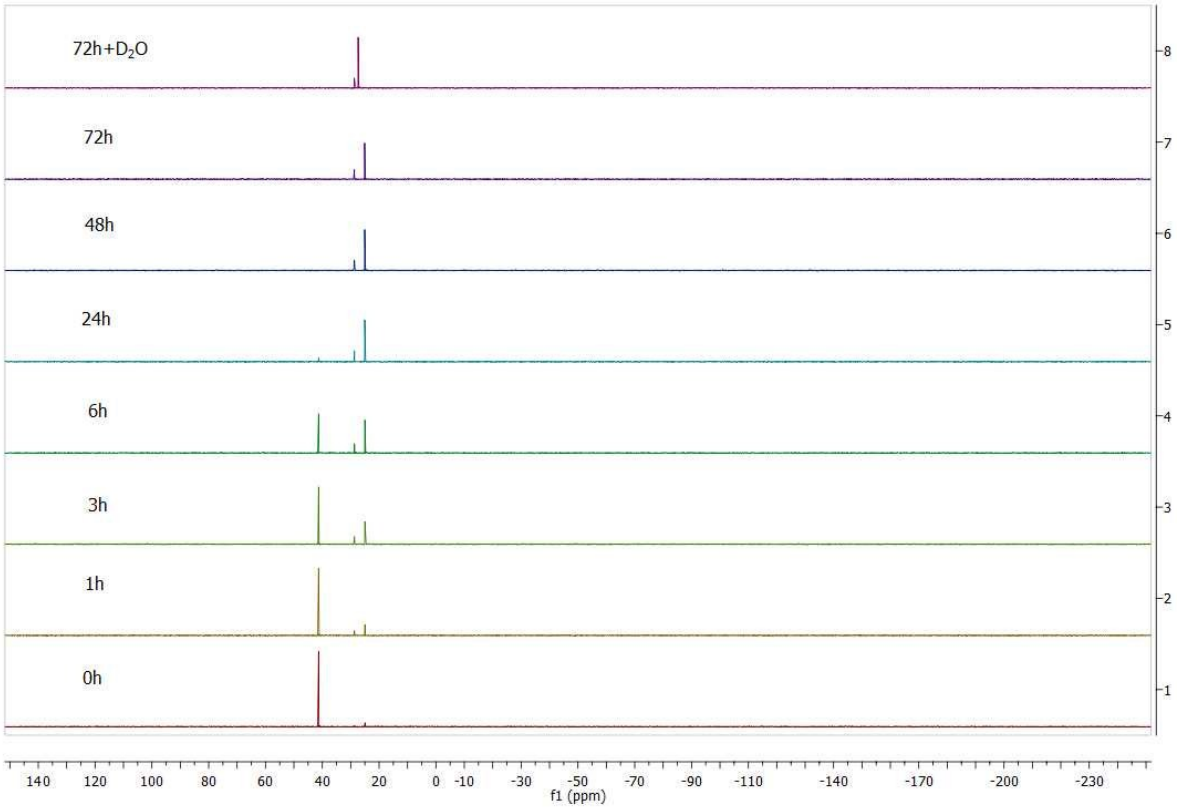

Figure S19. ^{31}P NMR spectrum of **4** in DMSO-d_6 over a 72 h time period

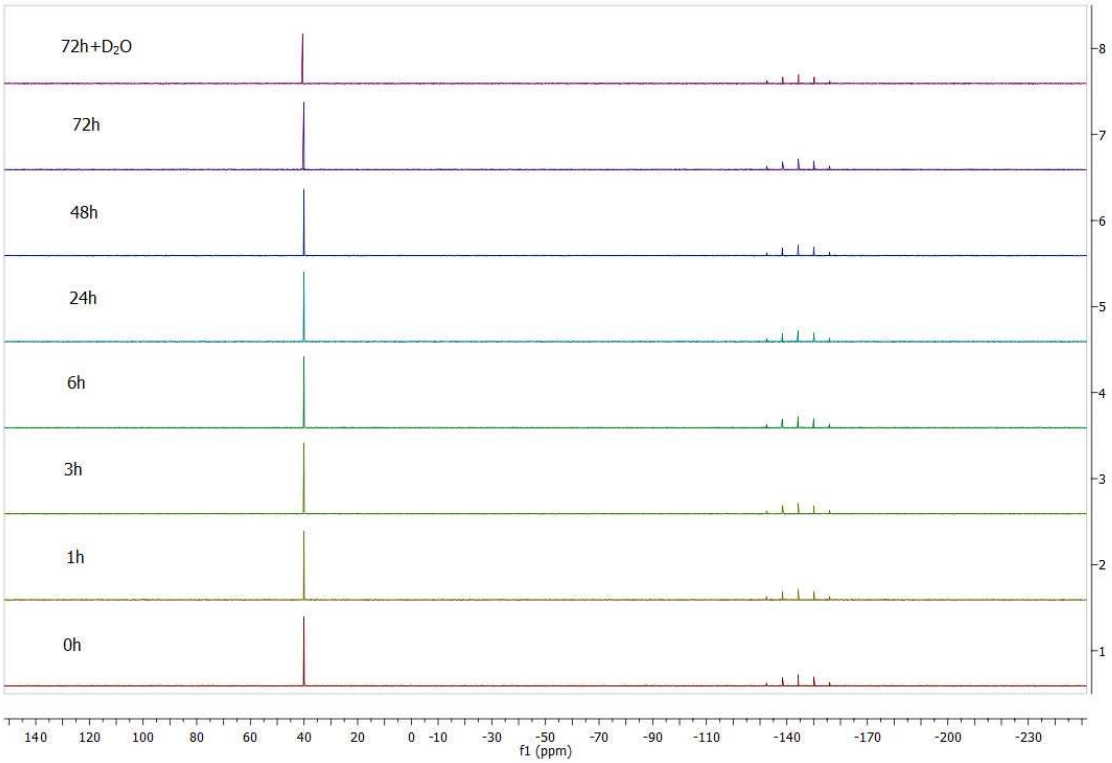

Figure S20. ^{31}P NMR spectrum of **5** in DMSO-d_6 over a 72 h time period

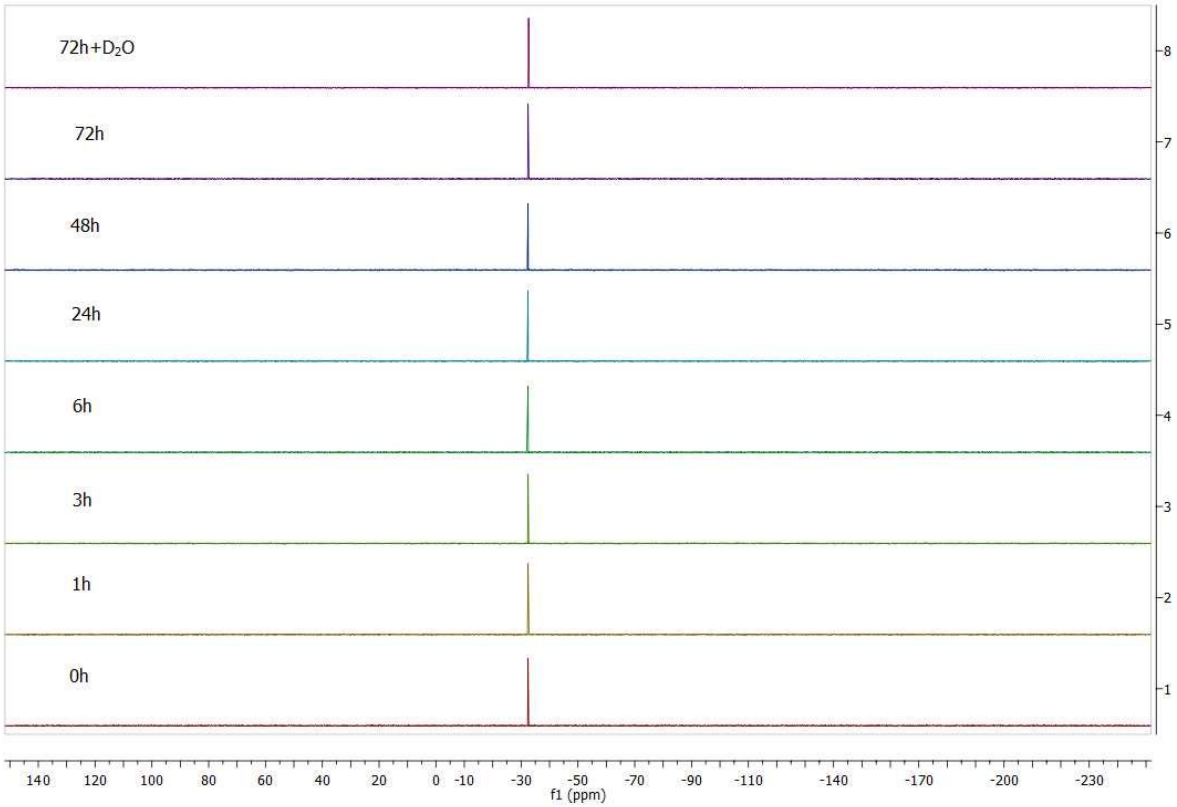

Figure S21. ^{31}P NMR spectrum of **6** in DMSO-d_6 over a 72 h time period

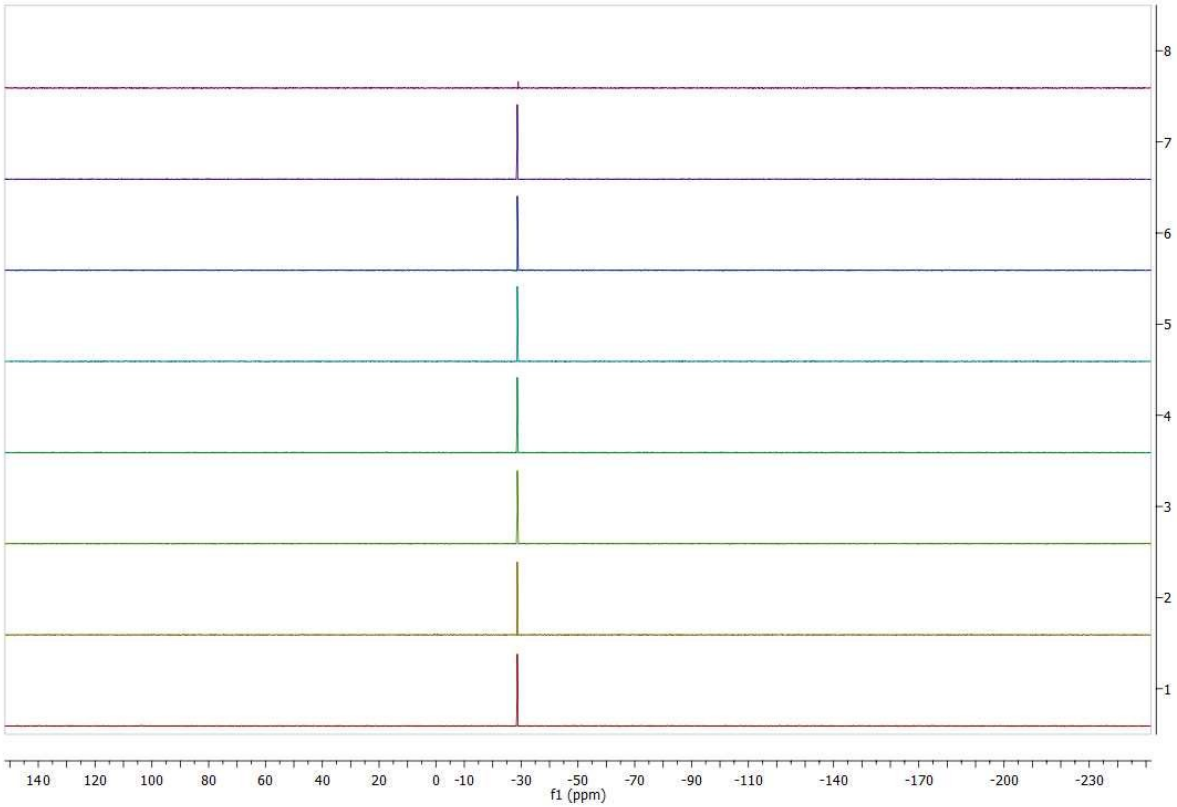

Figure S22. ^{31}P NMR spectrum of **7** in DMSO-d_6 over a 72 h time period

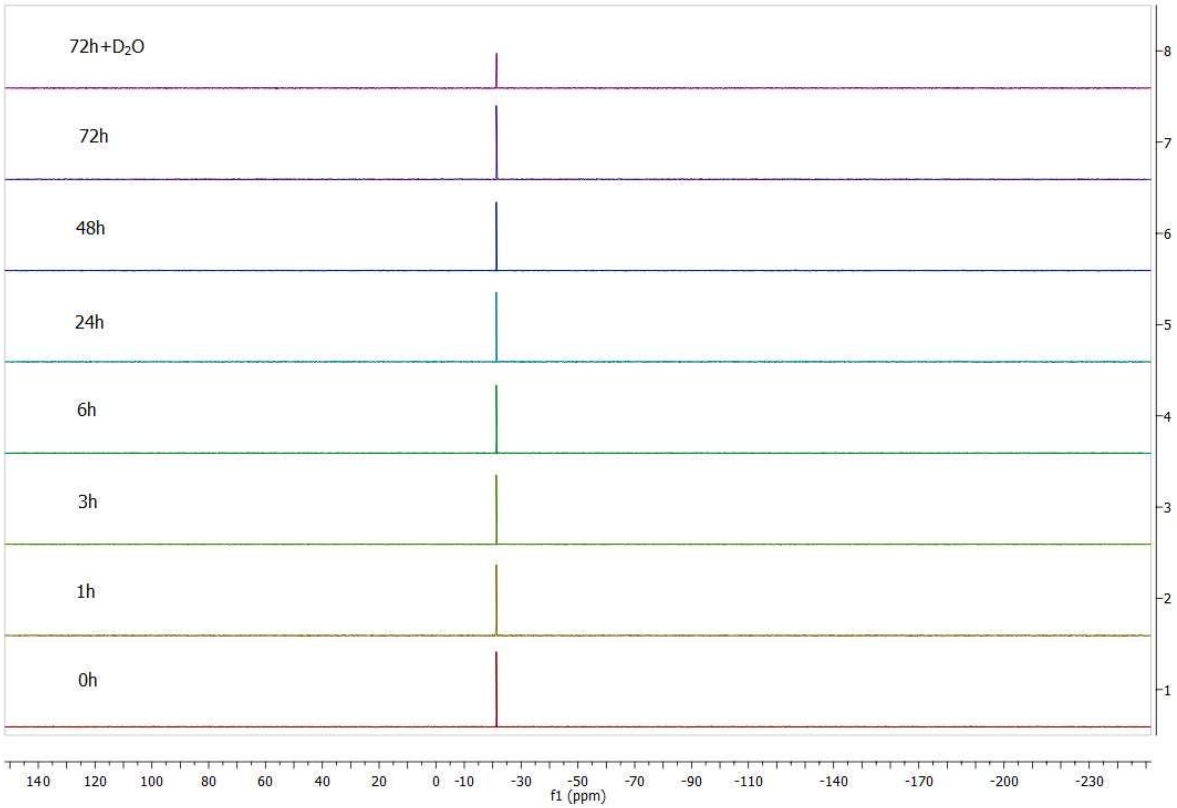

Figure S23. ^{31}P NMR spectrum of **8** in DMSO-d_6 over a 72 h time period

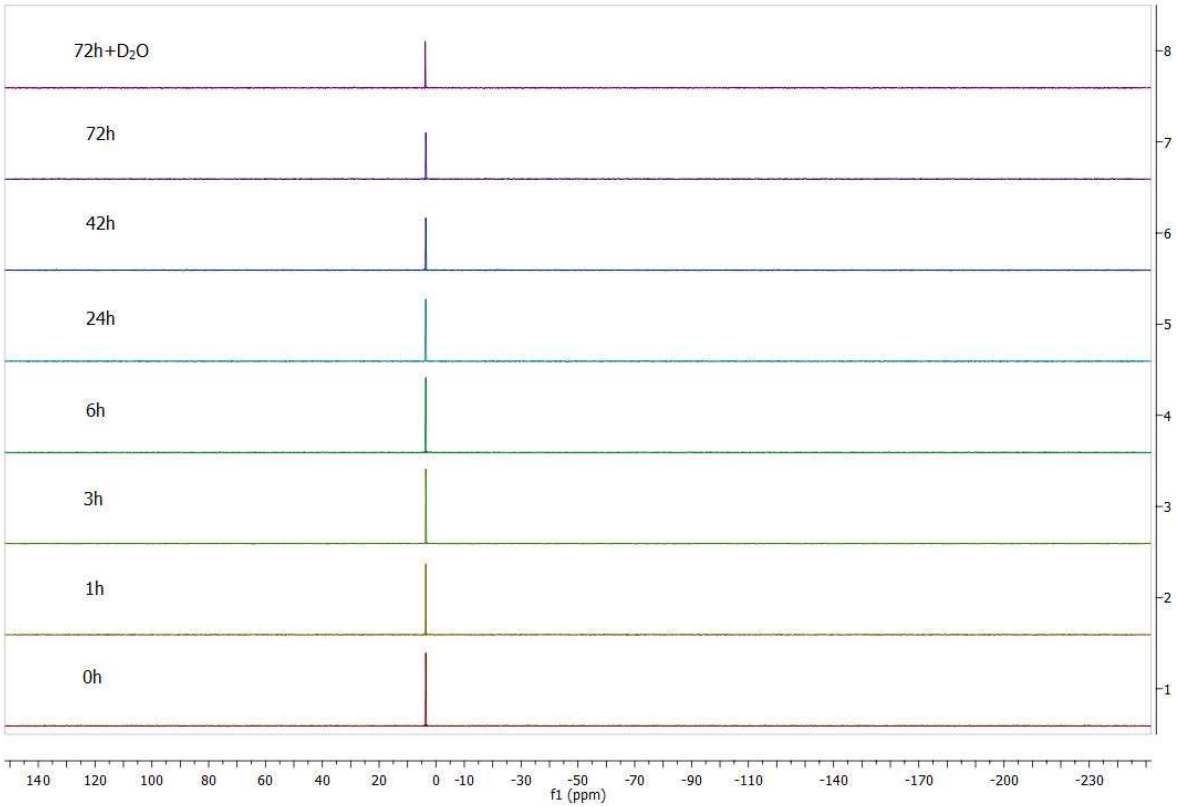

Figure S24. ^{31}P NMR spectrum of **9** in DMSO-d_6 over a 72 h time period

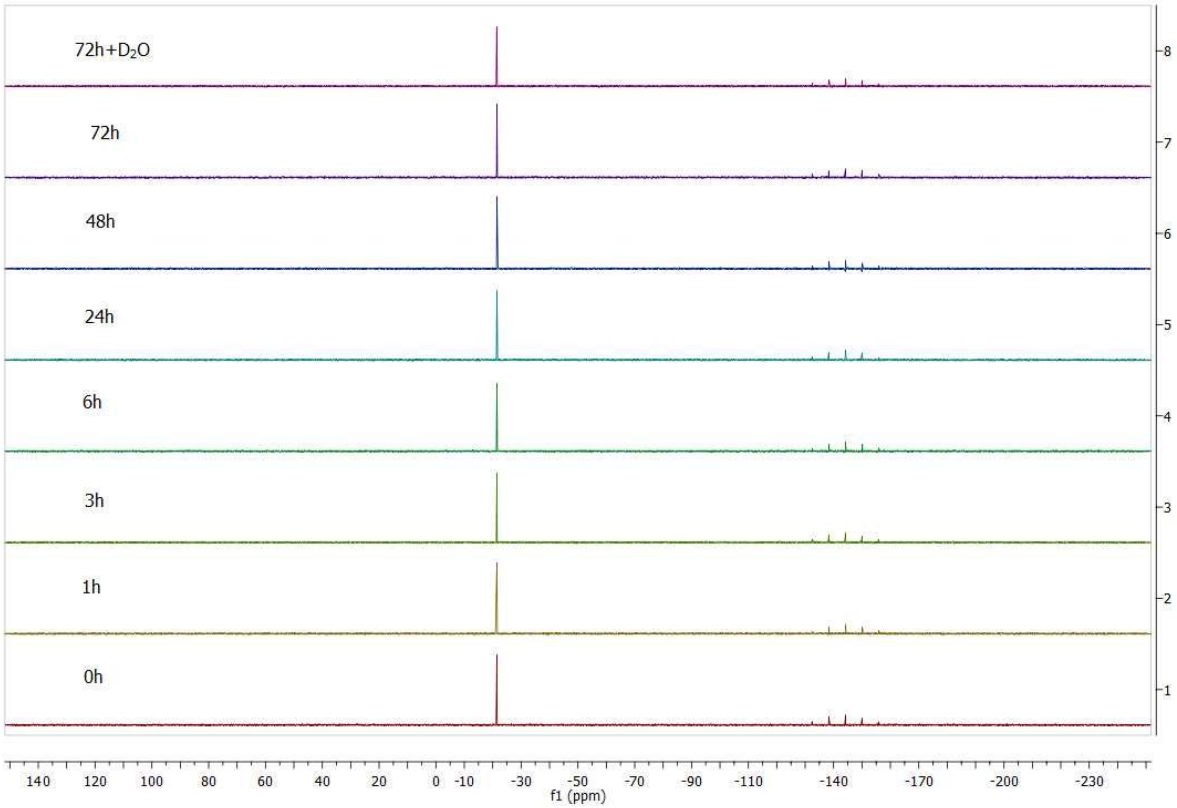

Figure S25**.** ^{31}P NMR spectrum of **10** in DMSO-d_6 over a 72 h time period

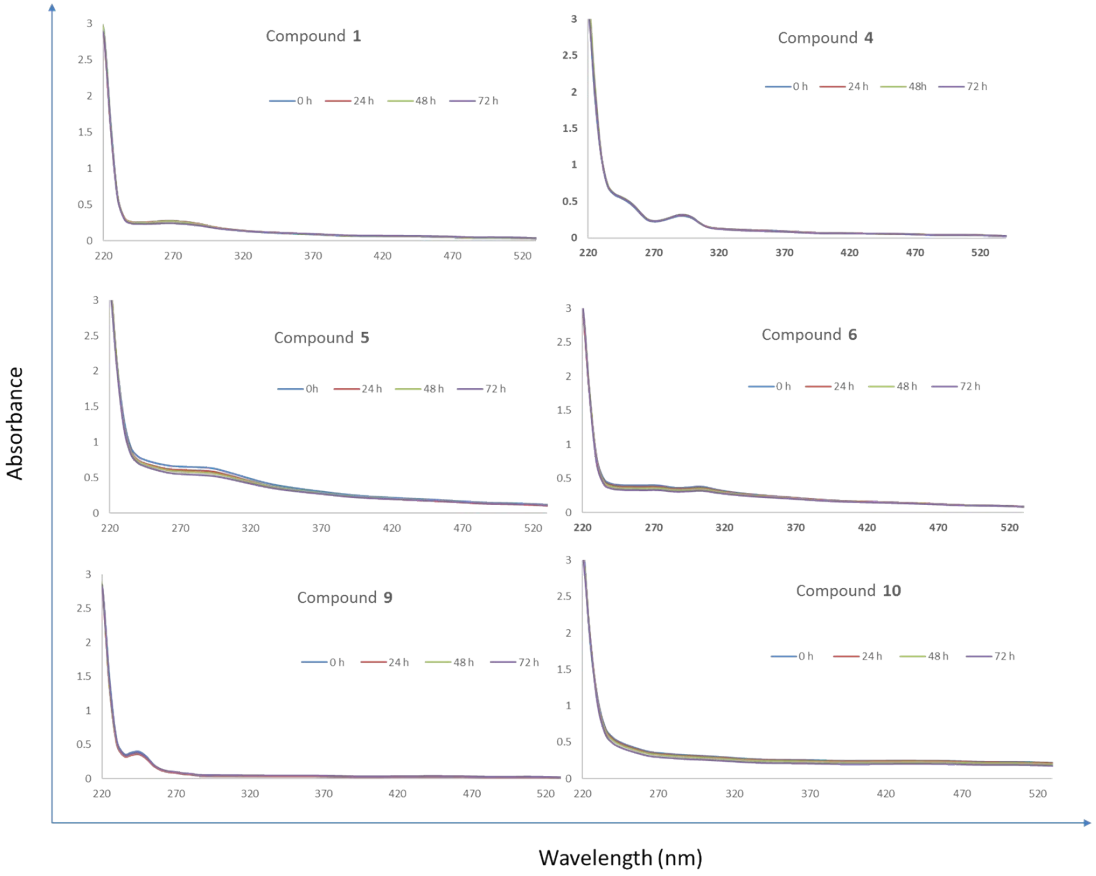

Figure S26. UV-Vis absorption spectra of the gold(I) and gold (III) complexes (25 μM) in (50 mM) Tris-(150 mM) NaCl buffer (pH 7.2) over 72 h.