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Chemistry of phosphorus ylides. Part 27. Metal complexes of 4-hydroxyquinaldine, its Mannich base and phosphonium ylide

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The reaction of Hg^{2+}, Cd^{2+}, Co^{2+}, and Ni^{2+} ions with 1 mol of the ligands 2-methylquinoline-4-ol, 3[(diethylamino)methyl]-2-methylquinoline-4-ol, and methyl-3-(4-hyrdoxy-2-methylquinoline-3-yl)-2-(triphenylphosphoranylidene)propanoate, has been investigated to give the corresponding 1:1 metal:ligand complexes. Reaction of 2 mol of the ligand gave the corresponding 1:2 metal:ligand complexes. The coordination takes place only through the quinoline nitrogen atom. The spectroscopic and the physicochemical data of the new metal complexes are discussed.

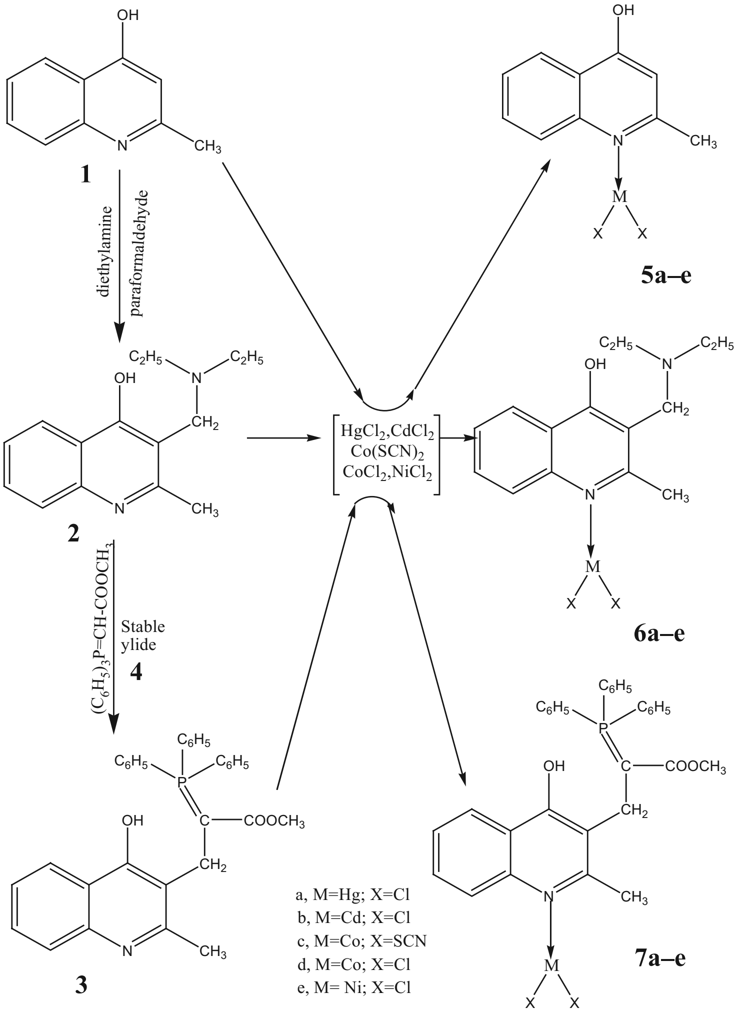
Keywords: quinaldine complexes, phosphoranes

Quinaldine and its derivatives constitute an important class of organic compounds with industrial and medicinal applications.[1-4] The have uses as dyes,[5] catalysis,[6] antioxidant precursors,[7] and corrosion inhibitors,[8] and are used as antitumor,[9] anti-HIV,[10] antimalarial,[11] antileishmanial,[12] antimicrobial,[13] antihistamine, anticholinergic, cardiovascular effects,[14] and also as molluscicides for the aquatic snails, *e.g. Biomphalaria glabrata* and *Biomphalaria alexandrina*.[15]

On the other hand, phosphonium ylide complexes attract interest from both the synthetic,[16,17] and biological viewpoints.[18] However, known methods for their preparation are difficult and lengthy.[19,20]

A simple route for the preparation of alkylated phosphonium ylide **3** derived from 2-methylquinoline-4-ol (**1**) has been reported.[21] This was achieved *via* transylidation of 3-[(diethylamino)methyl]-2-methylquinoline-4-ol (**2**) by stabilised phosphonium ylide **4** (see Scheme 1).

We now report a study of the reaction of some metal salts with 2-methylquinoline-4-ol (**1**), 3[(diethylamino)methyl]-2-methylquinoline-4-ol (**2**), and/or methyl-3-(4-hyrdoxy-



Scheme 1

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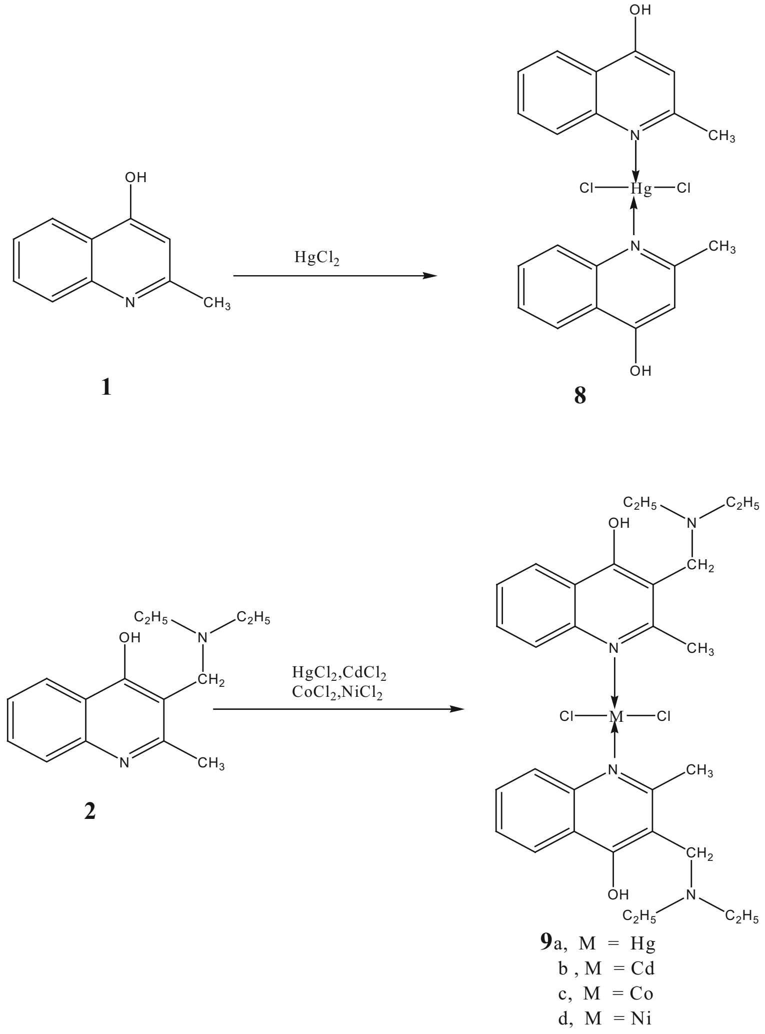
2-methylquinoline-3-yl)-2-(triphenylphosphoranylidene) propanoate (**3**), to prepare new metallic complexes of potential biological interest. The metal cations selected in the current study were Hg^{2+}, Cd^{2+}, Co^{2+}, and Ni^{2+}. The product obtained in each case was isolated and identified by elemental microanalysis, and by physicochemical and spectroscopic methods (IR, ^{1}H NMR, MS).

The reaction of one mol of the ligand 2-methylquinoline-4-ol (**1**) with one mol of the previously mentioned transition metal cations in ethanol, afforded the complexes **5a–e**. The assignments of the proposed structures were based on the following points. The IR and Far IR spectra for the isolated metal complexes **5a–e** were determined and compared with the IR spectrum of the ligand **1**. The OH stretching vibrations absorb around ν = 3500–3560 cm^{-1}, while the other stretching vibration modes in the ligand are much less affected by adduct formation. The Far IR spectra in the range 200–600 cm^{-1} were examined and observed bands in the region 200–300 cm^{-1} for complexes **5a,b,d,e** were attributed to the metal chloride (M-Cl) bands.[22,23] The ^{1}H NMR spectra for the complexes **5a–e** showed the phenolic OH proton at δ = 11.2–11.9 ppm which disappeared upon the addition of D_2O, and the CH_3 protons at δ = 3.3–4.0 ppm. The presence of the OH proton in transition metal complexes suggested that the coordination takes place through the N atom while the OH proton is not affected. The ^{1}H NMR spectra of Co and Ni complexes are broad and less sharp than those of Hg and Cd compounds. The magnetic susceptibility data showed the values of μ = 3.2, 3.5 B.M. for Co complexes **5c,d** and μ = 2.5 B.M. for Ni complex **5e**, which indicate their paramagnetic and tetrahedral properties.[24,25] In the mass spectrum of 2-methylquinoline-4-ol mercuric chloride complex (**5a**), taken as an example, the molecular ion M^+, which would appear at 430, was not recorded. The spectrum showed peaks at *m/z* 393 (6%) (M^+ – Cl), and at *m/z* 365 (1.7%) (M^+– Cl, CO), which undergo fragmentation to a cation with the most abundant peak at *m/z* 130, (2-methyl-3H-indole-3-ylium). On the other hand, compound **5a** showed also peaks at *m/z* 159 (59.3%) (M^+– HgCl), which undergo fragmentation to the same cation with the most abundant peak at *m/z* 130 (100%), and a mercuric chloride peak at *m/z* 272 (17.7%), which on further fragmentation gave a mercury peak at *m/z* 202 (14%).

When the ligand **1** was allowed to react with ($HgCl_2$) in molar ratio (2 : 1), a white product was isolated, which gave physical data suggesting the general formula **8** (Scheme 2). The IR spectrum of the complex **8** showed the OH group at ν = 3521 cm^{-1} and the C–H (aliphatic) at ν = 2912 cm^{-1}, the C–N stretching mode at ν = 1632 cm^{-1}. The ^{1}H NMR spectrum for compound **8** showed the OH peak at δ = 11.5 (exchangeable with D_2O), the aromatic protons at δ = 7.2–8.1 (m), CH proton at δ = 5.6 (s), and CH_3 protons at δ = 2.2 (s).

The reactions of 3-[(diethylamino)methyl]-2-methylquinoline-4-ol, (**2**) with the metal cations Hg^{2+}, Cd^{2+}, Co^{2+} and Ni^{2+} were also studied, (Scheme 1). The IR spectra for the isolated products **6a–e** showed the OH stretching vibrations around ν = 3393–3530 cm^{-1}, CH_3 around ν = 2900–2976 cm^{-1},



Scheme 2

while the other stretching vibration modes in the ligand are much less affected by adduct formation. Moreover, the (SCN) group appeared at ν = 2065 cm^{-1} in the complex **6c**. The far IR spectra of the complexes **6a,b,d,e** were observed in the region 216–307 cm^{-1} and were assigned to the appropriate metal halogen bands. The ^{1}H NMR spectra for the complexes **6a–e** showed the phenolic OH proton at δ = 9.2–11.9 ppm, which disappeared upon the addition of D_2O and the CH_3 protons were found at δ = 2.5–3.4 ppm, The presence of the OH proton in transition metal complexes suggested that the coordination takes place also through the N atom while the OH proton is not affected. In the mass spectrum of **6b** for example, the cleavage of the molecular ion of 3-[(diethylamino)methyl]-2-methylquinoline-4-ol cadmium chloride complex showed the appearance of the Mannich quinaldine ligand **2** peak at *m/z* 244 (1.2%) (M^+– $CdCl_2$) and the most abundant peak at *m/z* 171 (98.1%) [M^+– $CdCl_2$, $NH(C_2H_5)_2$], which loses CO to give a peak at *m/z* 143 (38.3%), then undergoes further fragmentation to the phenyl cation peak at *m/z* 77 (12.9%). The magnetic susceptibility data for complexes **6c,d** showed the values of μ = 3.1, 3.2 B.M, for Co^{2+} complexes and for Ni^{2+} complex **6e** μ = 2.3 B.M. When two mols of the ligand **2** were reacted with one mol of the metal chloride salts of Hg^{2+}, Cd^{2+}, Co^{2+}, and Ni^{2+} in ethanol, the isolated solid products obtained had the suggested formula **9a–d** (Scheme 2). In the mass spectroscopy, the molecular ion of the compound **9c**, as an example, undergoes the following fragmentation. Its cleavage is shown by the appearance of a peak at *m/z* 475 (2.98%) [M^+-$2N(C_2H_5)_2$], characteristic to the 2 mol addition, and also the 1 mol addition peak at *m/z* 372 (5.75%) [M^+– mol

Table 1 Physical and microanalytical data for metal complexes **5a–e**, **6a–e**, **7a–e**, **8** and **9a–d**

Comp. No.	M.p./ °C^a	Colour	Yield/%	Mol. Formula (MWt)	Anal. Calcd/ Found%						
					C	H	N	Cl	M	S	P
5a	230	White Yellow	95	$C_{10}H_9NO.HgCl_2$	27.87	2.09	3.25	16.46	46.58	—	
				(430.5)	27.83	2.06	3.14	16.25	46.36	—	
5b	>300	White	88	$C_{10}H_9NO.CdCl_2$	35.05	2.62	4.09	20.70	32.82	—	
				(342.3)	35.00	2.58	3.97	20.08	31.86	—	
5c	285	Blue	76	$C_{10}H_9NO.Co(SCN)_2$	43.11	2.69	12.57	—	17.66	19.18	
				(334)	43.00	2.63	11.95		16.92	18.89	
5d	>300	Blue	84	$C_{10}H_9NO.CoCl_2.H_20$	39.11	3.59	4.56	23.14	19.23	—	
				(306.8)	39.01	3.88	4.16	22.97	19.08	—	
5e	>300	Green	66	$C_{10}H_9NO.NiCl_2$	41.58	3.11	4.85	24.55	20.32	—	
				(288.6)	41.55	3.01	3.92	23.82	19.97	—	
6a	>300	Pale Yellow	90	$C_{15}H_{20}N_2O.HgCl_2$	34.95	3.88	5.43	13.75	38.89	—	
				(515)	34.90	3.83	4.83	12.91	38.03	—	
6b	265	White	85	$C_{15}H_{20}N_2O.CdCl_2$	42.12	4.68	6.55	16.58	26.29	—	
				(427.3)	42.01	4.56	6.36	15.79	25.97	—	
6c	>300	Green	75	$C_{15}H_{20}N_2O.Co(SCN)_2$	48.69	4.77	13.36	—	14.05	15.29	
				(419)	48.81	4.68	12.96	—	13.66	14.86	
6d	260	Blue	88	$C_{15}H_{20}N_2O.CoCl_2.H_20$	45.94	5.62	7.14	18.08	15.03	—	
				(391.8)	45.83	5.00	6.87	17.78	14.95	—	
6e	>300	Green	60	$C_{15}H_{20}N_2O.NiCl_2$	48.17	5.35	7.49	18.96	15.70	—	
				(373.6)	48.03	5.26	6.97	17.57	15.22	—	
7a	185	White	92	$C_{32}H_{28}NO_3P.HgCl_2$	49.45	3.60	1.80	9.13	25.81	—	3.99
				(776.5)	49.40	3.58	1.37	8.51	25.33	—	3.59
7b	230	White	87	$C_{32}H_{28}NO_3P.CdCl_2$	55.78	4.06	2.03	10.29	16.32	—	4.50
				(688.3)	55.71	4.01	1.91	09.57	16.22	—	3.88
7c	150	Violet	74	$C_{32}H_{28}NO_3P.Co(SCN)_2$	60.00	4.11	6.17	—	8.67	9.41	4.55
				(680)	60.29	4.00	6.30	—	8.15	8.90	4.24
7d	185	Blue	65	$C_{32}H_{28}NO_3P.CoCl_2.H_20$	58.82	4.59	2.20	11.16	9.28	—	4.87
				(652.8)	58.75	4.95	1.93	11.07	8.59	—	3.97
7e	>300	Blue	77	$C_{32}H_{28}NO_3P.\ NiCl_2$	60.51	4.41	2.21	11.16	9.24	—	4.88
				634.6	60.30	4.20	2.10	10.66	8.64	—	4.24
8	250	White	88	$(C_{10}H_9NO)_2.HgCl_2$	40.74	3.05	4.75	12.02	34.01	—	—
				589	40.65	3.00	3.95	11.88	33.90	—	—
9a	228	Yellow	90	$(C_{15}H_{20}N_2O)_2.HgCl_2$	47.43	5.27	7.37	9.33	26.39	—	—
				759	47.21	5.02	6.86	9.01	25.91	—	—
9b	260	White	85	$(C_{15}H_{20}N_2O)_2.CdCl_2$	53.65	5.96	8.34	10.55	16.73	—	—
				671	53.50	5.87	7.96	10.01	16.06	—	—
9c	260	Blue	66	$(C_{15}H_{20}N_2O)_2.CoCl_2.H_20$	56.62	6.29	8.80	11.16	9.27	—	—
				635.8	56.50	6.19	8.86	10.91	8.96	—	—
9d	230	Green	69	$(C_{15}H_{20}N_2O)_2.NiCl_2$	58.29	6.48	9.50	11.49	9.49	—	—
				617.6	58.09	6.34	8.96	10.81	9.06	—	—

^{a}All the new metal complexes were crystallised from absolute ethanol.

of ligand **2**], which undergoes further fragmentation to give the most abundant peak at *m/z* 171 (100%) [quinonemethylene], and a peak at *m/z* 144 [quinonemethylene – CO] (32.94%). The magnetic susceptibility data for compound **9c** showed the value of $\mu = 3.1$ B.M. for the Co^{2+} complex and $\mu = 2.6$ B.M. for the Ni complex **9d**.

Finally, the reaction of quinaldine phosphonium ylide, namely methyl-3-(4-hyrdoxy-2-methylquinoline-3-yl)-2-(triphenylphosphoranylidene) propanoate, ligand **3**, with the same metal cations was performed to give **7a–e**. In their IR spectra, the OH stretching vibrations absorbed around ν = 3443–3600 cm^{-1}, and P-C stretching modes around ν = 1366–1439 cm^{-1}, while the other stretching vibration modes in the ligand are much less affected by adduct formation. Moreover, the (SCN) group appears at ν = 2070 cm^{-1} in the complex **7c**. Far IR spectra were observed in the region 200–300 cm^{-1} for complexes **7a,b,d,e** and are attributed to the metal halogen bands. The ^{1}H NMR spectra for the complexes **7a–e** showed the phenolic OH proton at δ = 11.1–11.8 ppm which disappeared upon the addition of D$_2$O, the CH$_3$ protons at δ = 2.1–2.5 ppm and the ester CH$_3$ protons at δ = 3.4–3.7 ppm. Generally, the Co^{2+} and Ni^{2+} complexes which are paramagnetic gave broad NMR spectra. In the mass spectrum of the isolated complex **7a**, the cleavage of the molecular ion of the quinaldine phosphonium mercuric chloride complex moiety, was indicated by the appearance of the quinaldine ylide peak at *m/z* 505 (0.18%) (M$^+$– HgCl$_2$), and at *m/z* 272 (12.33%) for HgCl$_2$ which undergo fragmentation to the mercuric peak at *m/z* 199 (7.61%). Moreover, cleavage of M$^+$ **7a** also, gave a peak at *m/z* 444 (0.13%) [M$^+$– phosphonium ylide], and the ylide peaks at *m/z* = 333 (35%), which fragmented to the most abundant peak at *m/z* = 262 (100%) for triphenylphosphine. The magnetic susceptibility data for compounds **7c,d** showed the values of μ = 3.2, 3.5 B.M. for Co complexes and **7e** showed the value of μ = 2.1 B.M. for Ni complex, which indicates tetrahedral structures.

From the magnetic susceptibility data, it could be concluded that the paramagnetic Co^{2+} and Ni^{2+} are tetrahedral. The Hg^{2+} and Cd^{2+} complexes are diamagnetic and also tetrahedral.[24,25]

Conductometric titration for the complexes

Further insight concerning the products of reactions of **1**, **2**, and **3**, with metal cations was gleaned from a consideration of conductometric measurements. Conductometric titrations were performed by titrating the metal cation solution against the ligand solution of **1**, **2**, and/or **3**. The titration curves are smooth straight lines for all the points and the well-defined breaks are coincident with the stoichometric ratio of the complexes formed in solution. The obtained data are in a good agreement with the 1 : 1 and 2 : 1 (L : M) molar ratio suggested for the previously mentioned complexes.

In conclusion, from our experimental results it is evident that ligands **1**, **2**, and **3** reacted with the metal salt cations (Hg^{2+}, Cd^{2+}, Co^{2+}, Ni^{2+}) and the coordination takes place only through the quinoline nitrogen atom, since the OH group is not a true phenolic hydroxyl group, due to the resonance structure of quinoline, and also due to the steric hindrance of the diethyl amine group in Mannich ligand **2**, and of the triphenyl phosphonium group in the phosphonium ylide ligand **3**. This is in contrast to the coordination of 8-hydroxyquinoline as the two functional groups in 4-hydroxyquinoline are located at opposite sides of the aromatic ring.[26] The data obtained from the conductance titration curves showed that the ligands reacted with the metal salts in 1:1 and 2:1 molar ratio (L : M). The paramagnetic Co(II) and Ni(II) complexes **5–9** appear to have tetrahedral geometry, as do the diamagnetic Hg and Cd complexes.

Experimental

All melting points were measured on a Gallenkamp electrothermal melting point apparatus and are uncorrected. The IR spectra were recorded in KBr pellets on a Pye-Unicam SP 3300 and FTIR 8101PC Shimadzu IR spectrometers. NMR spectra were obtained in DMSO on a Varian MERCURY (1H: 300 MHz) spectrometer using (TMS) as an internal reference. ^{31}P NMR spectra were run on the same spectrometer using, H$_3$PO$_4$ (85%) as external reference. Mass spectra were recorded on a Shimadzu GC-MS QP 1000 Ex spectrometer at (E I, 70 eV). All the new compounds were crystallised from absolute ethanol. Elemental analyses were carried out at the microanalytical centre of National Research Centre, Cairo. Their results were in agreement with the calculated values. Physical, microanalytical data and spectroscopic data (IR, NMR and MS) of the collected complexes are reported in Tables 1 and 2.

Reaction of metal salts with 2-methylquinoline-4-ol (**1**) *in (1/1 molar ratio). Preparation of 2-methylquinoline-4-ol transition metal complexes* **5a–e**

A solution of the metal salts (HgCl$_2$, CdCl$_2$, CoCl$_2$, Co(SCN)$_2$, or NiCl$_2$) (0.01 mol) in 50 mL absolute ethanol was added dropwise

Table 2 IR, 1H NMR, and 31P NMR data for metal complexes **5a–e** , **6a–e** , **7a–e** and **9a–d**

No.	IR (ν)/cm^{-1}					^{1}H NMR(δ)/ppm			
Compd	OH	CH$_3$	C–N	Aromatic	M(SCN)	OH	(4H, aromatics, m)	(1H,CH, s)	(3H,CH$_3$-, s)
1						10.6	7.0–8.2	5.4	3.2
5a	3550	2915	1630	1580–1490 –1464	—	11.5	7.2–8.0	5.9	3.3
5b	3500	2987	1635	1594–1513 –1446	—	11.5	7.2–8.1	5.7	3.6
5c	3556	2965	1631	1599–1558 –1549	2076	11.4	7.2–8.0	5.8	3.3
5d	3521	3321	1631	1599–1496 –1466	—	11.2	7.2–8.3	4.8	4
5e	3560	3392	1632	1548–1470 –1409	—	11.9	7.0–8.2	5.8	3.3

Table 2 *(cont.)*

No.	IR (ν)/cm^{-1}					^{1}H NMR(δ)/ppm.					
						OH	(4H,aromatic,m)	(2H,CH$_2$-N,s)	(3H,CH$_3$-,s)	(2H,**CH$_2$**-CH$_3$,q)	(3H,CH$_2$-**CH$_3$**,t)
2						9.8	7.1–8.1	3.4	2.5	2.3	1.0
6a	3447	2972	1612	1579–1521–1440		9.2	7.2–8.2	3.3	2.9	2.5	1.0
6b	3530	2976	1614	1581–1498		11.4	7.2–8.0	3.3	2.6	2.4	1.0
6c	3743	2970	1631	1557–1510–1469	2065	11.4	7.2–8.0	3.5	3.4	2.4	1.0
6d	3433	2950	1629	1586–1553–1529		11.9	7.2–8.2	3.5	2.7	2.5	1.0
6e	3393	2900	1627	1523–1475		11.5	7.0–8.0	3.3	2.5	2.3	1.1

	OH	CH$_3$	C=O	Aromatic	P–C	OH	(19H, armatic,m)	(2H,CH$_2$,2d)	(3HCOOCH$_3$,s)	(3H,CH3, s)	^{31}P
3						10.3	7.6–8.3	2.8,3.2	3.6	2.1	
7a	3572	2934	1611	1549–1527	1436	11.7	7.3–8.3	2.5,3.3	3.6	2.2	25.7
7b	3457	2974	1622	1579–1501	1366	11.1	7.1–8.1	2.5,3.3	3.7	2.1	25.9
7c	3443	2924	1627	1523–1471	1433	11.3	7.1–8.1	2.4,3.5	3.5	2.2	25.7
7d	3588	2975	1750	1583–1499	1439	11.7	7.9	2.4,3.5	3.5	2.4	25.4
7e	3600	3100	1739	1523–1474	1437	11.8	7.2–8.0	2.7,2.9	3.4	2.5	25.4

	OH	CH3	C-N	Aromatic		OH	(4H,aromatic,m)	(2H,CH$_2$-N,s)	(3H,CH$_3$-,s)	(3H,**CH$_2$**-CH3,q)	(3H,CH$_2$-**CH$_3$**,t)
9a	3424	2927	1632	1579–1548–1497		11.5	7.3–8.1	3.6	2.9	2.5	1.0
9b	3522	2974	1622	1579–1501–1367		11.4	7.2–8.1	3.5	2.7	2.4	1.0
9c	3552	3059	1630	1580–1541–607		11.4	7.2–8.0	3.6	2.7	2.5	1.0
9d	3422	2986	1629	1527–1475		11.2	7.3–8.1	3.3	2.5	2.3	1.0

to a well-stirred solution of **1** (0.16 g, 0.01 mol) in absolute ethanol (50 mL). After complete addition of the metal salt the reaction mixture was heated under reflux for two hours, and then the solvent was evaporated under reduced pressure to give the metal complexes **5a–e**.

Reaction of mercuric chloride with 2-methylquinoline-4-ol **(1)** *in (1/2 molar ratio)*
A solution of the mercuric chloride (0.27 g, 0.01 mol) in absolute ethanol (50 mL) was added dropwise to a well-stirred solution of **1** (0.32 g, 0.02 mol) in absolute ethanol (50 mL). After complete addition of the metal salt the reaction mixture was heated under reflux for two hours, and then the solvent was evaporated under vacuum to give white crystals of **8**.

Reaction of metal salts with 3-[(diethylamino) methyl]-2-methylquinoline-4-ol **(2)** *in (1/1 molar ratio) Preparation of 3-[(diethylamino) methyl]-2-methylquinoline-4-ol transition metal complexes* **6a–e**
A solution of the metal salts (HgCl$_2$, CdCl$_2$, CoCl$_2$, Co(SCN)$_2$, or NiCl$_2$) (0.01 mol) in absolute ethanol (50 mL) was added dropwise

to a well-stirred solution of **2** (0.16 g, 0.01 mol) in absolute ethanol (50 mL). After complete addition of the metal salt the reaction mixture was heated under reflux for two hours, then the solvent was evaporated under reduced pressure to give the metal complexes **6a–e**.

Reaction of metal salts with 3-[(diethylamino) methyl]-2-methylquinoline-4-ol **(2)** *in (1/2 molar ratio)*

A solution of the metal salts ($HgCl_2$, $CdCl_2$, $CoCl_2$, or $NiCl_2$) (0.01 mol) in absolute ethanol (50 mL) was added dropwise to a well-stirred solution of two mols of the Mannich base **2** (0.5 g, 0.02 mol) in absolute ethanol (50 mL). After complete addition of the metal salt the reaction mixture was heated under reflux for two hours, then the solvent was evaporated under reduced pressure to give the metal complexes **9a–d**.

Reaction of metal salts with methyl-3-(4-hyrdoxy-2-methylquinoline-3-yl)-2-(triphenylphosphoranyli-dene) propanoate **(3)** *in (1/1 molar ratio).*

A solution of the metal salts ($HgCl_2$, $CdCl_2$, $CoCl_2$, $Co(SCN)_2$, or $NiCl_2$) (0.01 mol) in absolute ethanol (50 mL) was added dropwise to a well-stirred solution of **3** (0.5 g, 0.01 mol) in absolute ethanol (50 mL). After complete addition of the metal salt, the reaction mixture was heated under reflux for two hours; the solvent was evaporated under vacuum to give the metal complexes **7a–e**.

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