Neutral, Cationic and Anionic 2-[(phenylazo)phenyl] Complexes of Gold(III)

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$[AuCl_3(tht)]$ (tht = tetrahydrothiophene) reacts with $[Hg(Az)Cl]$ [Az = 2-(phenylazo)phenyl, 2-$(C_6H_4N=NC_6H_5)$] in the presence of Me_4NCl to give dichloro-2[(phenylazo)phenyl]gold(III), $[Au(Az)Cl_2]$. By reacting this complex with neutral ligands, complexes of the types $[Au(Az)Cl_2L]$ (L = PPh_3, PCy_3, $P(o\text{-}MeOC_6H_4)_3$, PBu_3^n, $AsPh_3$) and $[(Az)Cl_2Au(LL)AuCl_2(Az)]$ (LL = $Ph_2P(CH_2)_2PPh_2$, dpe) have been obtained. Starting from $[Au(Az)Cl_2-(PCy_3)]$ it is possible to substitute one or two chloro- for iodo- ligands to give complexes $[Au(Az)ClI-(PCy_3)]$ and $[Au(Az)I_2(PCy_3)]$.

Cationic complexes $[Au(Az)ClLL']ClO_4$ have been synthesized by reacting $[Au(Az)Cl_2]$ or the corresponding neutral complexes $[Au(Az)ClL]$ with neutral monodentate (L = L' = PPh_3, PCy_3; L = PPh_3, L' = py, tht; L = PCy_3, L' = py) or bidentate ligands (LL' = dpe, phen) and $NaClO_4$.

The reaction of $[Au(Az)Cl_2]$ with $BzPh_3PCl$ gives the anionic complex $BzPh_3P[Au(Az)Cl_3]$.

Introduction

The auration reaction of aromatic compounds is a well-known process since the reports of Kharash [1]. This type of reaction seems to be limited by the need for a potentially vacant co-ordination site on gold, as in $[Au_2Cl_6]$. Thus, neither $H[AuCl_4]$ or $[Au_2Cl_6]$ in donor solvents react with benzene, nor does $[Au_2Cl_6]$ react with arenes if they have substituents which can co-ordinate to gold [1–5]. This explains the fact that *ortho*-metallation reactions had never been observed in the chemistry of gold(III), although decomposition of the adduct $[AuCl_3-(PhN_2Ph)]$ was thought likely to occur through the intermediacy of the *ortho*-metallated complex dichloro [2-(phenylazo)phenyl]gold(III) [3]. We report here the synthesis of this complex using chloro [2-(phenylazo)phenyl]mercury(II) as an intermediate, and its reactivity towards neutral and anionic ligands.

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Organomercury compounds have widely been used in the synthesis of other organometallic compounds [6–13]. In the chemistry of gold, only gold-(I) complexes have been reacted with organomercurials [14, 15]. The complex chloro[2(phenylazo)phenyl]mercury(II) has been used in the synthesis of some palladium(II), platinum(II), nickel-(II), manganese(I) [16, 17], tellurium(IV) [18] and tin(IV) [19] 2-(phenylazo)phenyl complexes.

Some of the complexes reported here have been the subject of a preliminary communication [20].

Results and Discussion

When a mixture of $[AuCl_3(tht)]$, $[Hg(Az)Cl]$ and Me_4NCl (molar ratio 1:1:1) is stirred in acetone for 24 hr an orange solution of $[Au(Az)Cl_2]$ (I) and a precipitate containing $Me_4N[HgCl_3]$ and (I) are obtained.
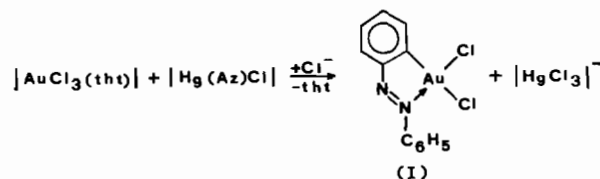
The i.r. spectrum, analytical data, conductivity, molecular weight (determined by osmometry) and reactivity of I allow the formulation as had been postulated previously [3], namely an intermediate in the decomposition of $[AuCl_3(PhN_2Ph)]$.

The addition of different neutral ligands to suspensions of (I) (molar ratio 1:1) in acetone gives solutions of complexes $[Au(Az)Cl_2L]$ [L = PPh_3 (II), PCy_3 (III), $P(o\text{-}MeOC_6H_4)_3$ (IV), PBu_3^n (V) and $AsPh_3$ (VI)].
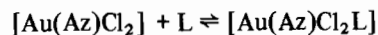
$$[Au(Az)Cl_2] + L \rightleftharpoons [Au(Az)Cl_2L]$$

The use of pyridine or tetrahydrothiophene as ligands leads to solutions from which mixtures of (I) and, probably, the corresponding complexes

[Au(Az)Cl₂L], are obtained. Upon recrystallizing the mixtures, only (I) can be isolated.

The process is likely to be an equilibrium that goes to the left, due to the poorer donor ability of these ligands and to the scarce solubility of (I).
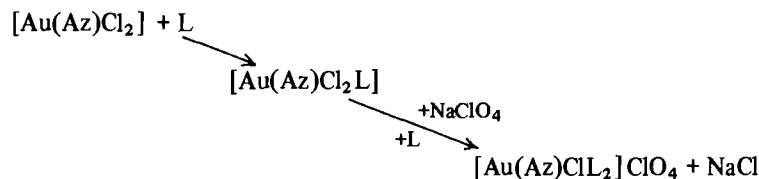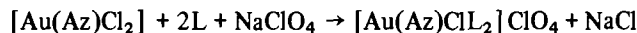
When a bidentate ligand such as 1,2-bis(diphenyl-phosphino)ethane (dpe) is reacted with (I) (molar ratio 1:2) a dinuclear complex [(Az)Cl₂Au(dpe)-AuCl₂(Az)] (VII) is obtained.

When [Au(Az)Cl₂(PCy₃)] (III) is reacted with NaI in acetone two different complexes, [Au(Az)-ClI(PCy₃)] (VIII) and [Au(Az)I₂(PCy₃)] (IX), are obtained depending on the molar ratio (III):NaI.
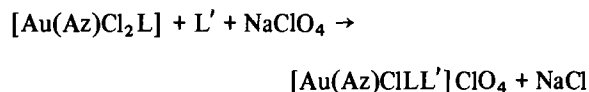
Even if an excess of PPh₃ or AsPh₃ is added to (I) (molar ratios $\geqslant$2:1) only complexes (II) or (VI) can be isolated although equilibria of the type

$$[Au(Az)Cl_2L] + L \rightleftharpoons [Au(Az)ClL_2]^+ + Cl^-$$
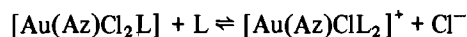
must occur in solution, as has been proved by measuring the conductivity of a 4.55×10^{-4} M solution of (II) to which successive amounts of PPh₃ were added. Molar conductivities of these solutions, assuming no change in volume, are presented in Table I. These results suggest the feasibility of isolating cationic complexes if chloride is substituted in the equilibrium by a poorer donor ligand (such as perchlorate). In fact, cationic complexes [Au(Az)ClL₂]⁺ [L = PPh₃ (X), PCy₃ (XI)] have been prepared starting from (I) in one or two steps:

$$[Au(Az)Cl_2] + 2L + NaClO_4 \rightarrow [Au(Az)ClL_2]ClO_4 + NaCl$$

[Au(Az)Cl₂] + L

[Au(Az)Cl₂L]

+NaClO₄
+L

[Au(Az)ClL₂]ClO₄ + NaCl

The two steps lead to the possibility of introducing two different ligands:

$$[Au(Az)Cl_2L] + L' + NaClO_4 \rightarrow$$

$$[Au(Az)ClLL']ClO_4 + NaCl$$

if L' is a weaker donor ligand than L to avoid substitution reactions such as

$$[Au(Az)Cl_2L] + L' \rightarrow [Au(Az)Cl_2L'] + L$$
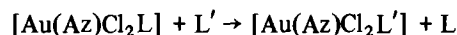
In fact, complexes of the type [Au(Az)ClLL']⁺ have been synthesized when L = PPh₃, L' = py (XII), tht (XIII) and L = PCy₃, L' = py (XIV).

When dpe and NaClO₄ are reacted with (I) (molar ratio 1:1:1) a cationic complex [(Az)ClAu(dpe)₂-AuCl(Az)](ClO₄)₂ (XV) is obtained. Its dinuclear

TABLE I. Λ_M of Solutions of Complex (II) when PPh₃ is added.

Molar ratio PPh₃:(II)	Λ_M^* Ω^{-1} cm² mol⁻¹
0	2.2
1	18.2
3	30.7
7.4	59.3
18	94.4

*In nitromethane solution.

nature has been corroborated using the method of Feltham [25]. Measuring the molar conductivities of six different solutions (range of equivalent concentrations 4×10^{-3} to 4×10^{-5} M) of (XV) in nitromethane, the equation representing the equivalent conductivity as a function of the equivalent concentration is $\Lambda_e = 79.0 - 435.4 \sqrt{c}$ (correlation coefficient = 0.99). The slope is in the range observed for other 2:1 electrolytes in nitromethane (-400 to -510).

The possibility of 2[(phenylazo)phenyl] being a bridging ligand has been rejected for complex (X), for which $\Lambda_e = 66.6 - 150 \sqrt{c}$ (correlation coefficient = 0.97, slopes for other 1:1 electrolytes go from -150 to -220).

When 1,10-phenanthroline (phen) and NaClO₄ are reacted with (I) (molar ratio 1:1:1) a cationic complex [Au(Az)Cl(phen)]ClO₄ (XVI) is obtained.

As far as we know complexes (X)–(XVI) are the first cationic gold(III) complexes with only one gold—carbon bond [26].

Addition of Me₄NCl to an acetone suspension of (I) (1:1) results in a solution from which only (I) is recovered. Nevertheless, when a bigger cation such as BzPh₃PCl is used, the anionic BzPh₃P[Au(Az)Cl₃] (XVII) can be isolated.

Table II presents the analytical data for complexes (I)–(XVII).

Iodine reacts with the starting organomercurial [Hg-(Az)Cl] to give 2-iodoazobenzene [27]. By refluxing chloroform solutions of (I), (II) or (X) with iodine for 2 to 12 hr the only organic compound isolated was 2-iodoazobenzene characterized by m.p. and i.r.

TABLE II. Analytical and Other Data for Complexes (I)–(XVII).

Complex	M.p. °C	Colour	$\Lambda_M{}^a$ Ω^{-1} cm^2 mol^{-1}	Analysis Found (Calc.) (%)				Yield
				C	H	N	Au	
[AuAzCl$_2$] (I)	218(d)	brick-red	4	32.59 (32.09)	2.12 (2.02)	6.03 (6.24)	44.59 (43.86)	64
[Au(Az)Cl$_2$(PPh$_3$)] (II)	181(d)	orange	2.2	50.99 (50.56)	3.97 (3.40)	3.83 (3.94)	27.13 (27.69)	84
[Au(Az)Cl$_2$(PCy$_3$)] (III)	219(d)	orange	2.73	49.81 (49.39)	5.49 (5.80)	3.95 (3.84)	27.51 (26.99)	68
[Au(Az)Cl$_2$(P(o-MeOC$_6$H$_4$)$_3$] (IV)	121	orange	6.4	50.11 (49.46)	3.96 (3.77)	3.16 (3.50)	24.77 (24.57)	47
[Au(Az)Cl$_2$(PBu$_3$)] (V)	148	orange	2.25	43.67 (44.21)	5.40 (5.57)	3.90 (4.30)	30.81 (30.23)	66
[Au(Az)Cl$_2$(AsPh$_3$)] (VI)	175	orange	1.7	48.41 (47.70)	3.57 (3.20)	3.74 (3.71)	27.26 (26.08)	69
[Au$_2$(Az)$_2$Cl$_4$(dpe)] (VII)	169	yellow	2.43	45.84 (46.32)	3.36 (3.27)	4.10 (4.32)	30.98 (30.38)	83
[Au(Az)ClI(PCy$_3$)] (VIII)	214(d)	orange	7.65	44.39 (43.89)	4.49 (5.16)	3.77 (3.42)	23.71 (24.00)	80
[Au(Az)I$_2$(PCy$_3$)] (IX)	174(d)	orange	0.22	40.67 (39.49)	4.83 (4.64)	2.99 (3.07)	20.90 (21.59)	90
[Au(Az)Cl(PPh$_3$)$_2$]ClO$_4$ (X)	136(d)	pale-orange	123.5	54.93 (55.56)	3.95 (3.79)	2.56 (2.69)	19.70 (18.98)	87
[Au(Az)Cl(PCy$_3$)$_2$]ClO$_4$ (XI)	204	yellow	123.5	54.21 (53.68)	7.11 (7.03)	2.59 (2.60)	18.03 (18.34)	80
[Au(Az)Cl(PPh$_3$)(py)]ClO$_4$ (XII)	193(d)	orange	146.2	49.78 (49.19)	3.84 (3.42)	5.16 (4.92)	23.70 (23.05)	84
[Au(Az)Cl(PPh$_3$)(tht)]ClO$_4$ (XIII)	144	orange	132	48.26 (47.31)	3.72 (3.73)	3.21 (3.24)	22.31 (22.82)	43
[Au(Az)Cl(PCy$_3$)(py)]ClO$_4$ (XIV)	145(d)	orange	126.6	47.50 (48.17)	5.65 (5.43)	4.37 (4.81)	21.89 (22.58)	74
[Au$_2$(Az)$_2$Cl$_2$(dpe)$_2$[(ClO$_4$)$_2$ (XV)	137(d)	orange	237	49.35 (50.07)	3.64 (3.65)	2.87 (3.07)	22.04 (21.61)	48
[Au(Az)Cl(phen)]ClO$_4$ (XVI)	199(d)	orange	126.3	42.41 (41.57)	2.87 (2.46)	7.81 (8.08)	27.86 (28.41)	57
BzPh$_3$P[Au(Az)Cl$_3$] (XVII)	83	orange	133	53.51 (53.03)	3.79 (3.72)	2.82 (3.34)	22.59 (23.50)	76

^{a}In acetone solution.

I.R. Spectra

The i.r. spectrum of complex (I) shows two strong bands at 305 and 370 cm^{-1} that can be assigned to ν(AuCl) *trans* to the phenyl ring and to the nitrogen of the azo-group, respectively. Such a great difference in position of the bands is in accordance with the stronger *trans*-influence of phenyl with respect to N-donor ligands. In the related palladium(II) complex Et$_4$N[Pd(C$\overset{\frown}{N}$)Cl$_2$] (C$\overset{\frown}{N}$ = *ortho*-metallated acetophenone hydrazone), two strong bands (263, 346 cm^{-1}) are also observed [28]. In complexes of the type *cis*-[Au(Ar)X$_2$L] (Ar = C$_6$F$_5$, C$_6$Cl$_5$, C$_6$Br$_5$; X = Cl; L = Et$_3$, PPh$_3$) the bands ν(AuCl) *trans* to Ar have been observed in the region 295–300 cm^{-1} [29]. For the anionic complexes R$_4$N[Au-(C$_6$H$_5$)Cl$_3$] the band assigned to ν(AuCl) *trans*

to C$_6$H$_5$ is at 289 (R = Et) or 287 (R = Bu^n) cm^{-1} [30].

In *trans*-[Au(CN)$_2$Cl(py)] the band at 363 cm^{-1} has been assigned to ν(AuCl) *trans* to pyridine [31] which can be supposed to have a *trans*-influence similar to that of the azo-group.

In the neutral complexes (II)–(VII) two bands are also observed. Those appearing in the 290–298 cm^{-1} region (290 (II), 294 (III), 290 (IV), 297 (V), 297 (VI), 298 (VII) cm^{-1}) can be assigned to ν(AuCl) *trans* to the phenyl ring, while those in the 310–315 cm^{-1} region [315 (II), (VII); 310 (III), (IV), (V); 312 (VI)] can be assigned to ν(AuCl) *trans* to the neutral ligands according to their minor *trans* influence with respect to the phenyl group.

In the afore-mentioned cis-[Au(Ar)X$_2$L], bands in the 350–318 cm^{-1} region have also been assigned to ν(AuCl) trans to different phosphines [29].

Although penta-coordination is not excluded, i.r. spectra are as expected for cis-square planar complexes (assuming the cleavage of the Au–N bond). Such cleavages have also been observed for other similar complexes of palladium(II) and platinum(II) [16, 21–24]. The i.r. spectra for a trans-square planar geometry would show a strong band at 360–370 cm^{-1}, due to assymetric ν(AuCl$_2$) [29–34]. The i.r. spectrum of complex [Au(Az)ClI$_2$(PCy$_3$)] (VIII) only shows a strong band assignable to ν(AuCl). Unfortunately, its position (at 305 cm^{-1}) is intermediate between those assigned to ν(AuCl) trans to phenyl and trans to phosphine, precluding any suggestion about the structure of (VIII). Complex [Au(Az)I$_2$(PCy$_3$)] (IX) does not show any strong band in the low frequency region of our spectrophotometer.

The trans configuration of [Au(Az)Cl(PPh$_3$)$_2$]·ClO$_4$ (X) is confirmed by ^{31}P n.m.r. (a singlet is observed at 34.9 p.p.m. (PO$_4$H$_3$) in CCl$_3$D). The product of the reaction between [Pd(Az)Cl]$_2$ and PEt$_3$ is also trans-[Pd(Az)Cl(PEt$_3$)$_2$] [24]. Furthermore the trans-geometry of (X) is in accordance with the better trans effect of PPh$_3$ with respect to the phenyl group [35]. Therefore, the strong band observed at 305 cm^{-1} must be assigned to ν(AuCl) trans to phenyl. The difference between this value and that of complex [Au(Az)Cl$_2$(PPh$_3$)] (II) ($\Delta\nu$ = 15 cm^{-1}) could be partially due to the cationic nature of complex (X) that would enhance the π-donor ability of the chloro ligand strengthening the Au–Cl bond. A weak band at 545 cm^{-1} can also be related to the trans geometry of (X) if observations made for bis(triphenylphosphine) complexes of Pt(II) [36] are extended to Au(III) derivatives [37].

The mononuclear cationic complexes (XI)–(XIV) present also a band in the 302–310 cm^{-1} region (302 (XI); 310 (XII), (XIII); 305 (XIV) cm^{-1}) and tentatively a trans geometry could be assigned. In fact, [Au(Az)Cl(PPh$_3$)(tht)]ClO$_4$ (XIII) reacts with PPh$_3$ to give (X).

In [(Az)ClAu(dpe)$_2$AuCl(Az)](ClO$_4$)$_2$ (XV), the band appearing at 320 cm^{-1} could tentatively be assigned to ν(AuCl) trans to the phosphine. In complex [Au(Az)Cl(phen)]ClO$_4$ (XVI), which must be cis, the corresponding ν(AuCl) band is at 360 cm^{-1}.

The anionic BzPh$_3$P[Au(Az)Cl$_3$] (XVII) presents two medium bands at 358 and 280 cm^{-1}. The former has been assigned to ν_{asym}(AuCl$_2$) in other [Au(Ph)Cl$_3$]$^-$ complexes (359 cm^{-1}) [30]. The corresponding ν_{sym} (AuCl$_2$) is weaker and sometimes is not observed, or is masked by the asymmetric one, as probably happens in (XVII) [29]. The last one

has been assigned to ν(AuCl) trans to the phenyl group in [Au(Ph)Cl$_3$]$^-$ complexes (287–289 cm^{-1}) [30].

The bands assigned to ν(AuCl) trans to the phenyl group appear at lower frequencies for the anionic complex (280 cm^{-1}), intermediate for the neutral (290–305 cm^{-1}) and higher for the cationic ones (302–310 cm^{-1}).

Azobenzene and 2-iodoazobenzene have two strong bands at approx. 770 and 690 cm^{-1}. In addition, 2-iodoazobenzene has a strong band at 710 cm^{-1} that could be due to the ortho-substitution of one of the rings. Complex (I) shows the same pattern as 2-iodoazobenzene in the 680–780 cm^{-1} region, and the same applies to those complexes in which ligands do not present bands in that region [e.g. complexes (III) and (V)].

Experimental

I.r. spectra were recorded over the range 4000–250 cm^{-1} on a Perkin-Elmer 457 spectrophotometer using Nujol mulls between polyethylene sheets or KBr pellets. Conductivities were measured in 5×10^{-4} M solutions with a Phillips 9501 conductimeter. Melting points were measured on a Reichert apparatus and are uncorrected. C, H and N analyses were carried out with a Perkin-Elmer 240C microanalyzer. Au was determined by ashing the samples together with an aqueous solution of hydrazine.

The starting complexes [AuCl$_3$(tht)] [33] and Hg(Az)Cl [27] were prepared by literature methods. The other reagents were obtained from commercial sources; solvents were distilled before use. Unless otherwise stated the reactions were carried out at room temperature; under magnetic stirring and without special precautions against daylight or moisture.

Complexes (I), (II) and (VI) were prepared as described in our preliminary communication [20].

Preparation of Neutral Complexes (III)–(V) and (VII)

To suspensions of (I) in acetone (10 ml) stoichiometric amounts of the ligands were added. After 4 hr stirring the resulting suspensions were concentrated (1ml) and diethyl ether (10 ml) added to assist in the precipitation of complexes (III)–(V) and (VII).

Preparation of Neutral Iodo Complex (VIII)

To a suspension of (III) (86 mg, 0.12 mmol) in acetone (15 ml) NaI (18 mg, 0.12 mmol) was added and the suspension refluxed for 4 hr. It was then concentrated to dryness and extracted with methylene chloride (3 × 6 ml). Evaporation of the

solvent (3 ml) and addition of diethyl ether (15 ml) gave (VIII).

Preparation of Neutral Iodo Complex (IX)

A suspension of (VIII) (160 mg, 0.19 mmol) in acetone (15 ml) was treated with NaI (80 mg, 0.53 mmol) and refluxed for 1 hr. It was then concentrated to dryness and the solid washed with water (5 X 5 ml), 50% acetone/water (3 X 3 ml) and diethyl ether (3 X 3 ml), and dried to give (IX).

Preparation of Cationic Complexes (X) and (XI)

To suspensions of (I) in acetone PPh₃ or PCy₃ (L: (I) = 2:1) and NaClO₄ (1:1) were successively added. After 6 hr stirring the suspensions were concentrated to dryness, extracted with methylene chloride (2 X 10 ml), NaCl filtered off, the solutions concentrated (1 ml) and diethyl ether (10 ml) added to precipitate complexes (X) or (XI).

Preparation of Cationic Complexes (XII)–(XIV)

To suspensions of complexes (II) or (III) in acetone (10 ml) excess of the ligands py or tht and NaClO₄ (1:1) were added. After 4 hr stirring the mixtures were concentrated to dryness, extracted with methylene chloride, NaCl filtered off, the solutions concentrated (1 ml), and diethyl ether (10 ml) added to precipitate complexes (XII)–(XIV).

Preparation of Cationic Complexes (XV) and (XVI)

To suspensions of (I) in acetone (10 ml) dpe or phen (1:1) and NaClO₄ (1:1) were added. After 4 hr stirring the suspensions were concentrated to dryness, extracted with methylene chloride (2 X 5 ml), NaCl filtered off, the solutions concentrated (1 ml), and diethyl ether added to precipitate complexes (XV) or (XVI).

Preparation of Anionic Complex (XVII)

To a suspension of (I) (100 mg, 0.22 mmol) in acetone (10 ml) BzPh₃PCl (86.6 mg, 0.22 mmol) was added. After 30 min stirring the solution was concentrated to dryness, extracted with methylene chloride (1ml), and diethyl ether (15 ml) added to precipitate (XVII).

Acknowledgement

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