

## A NEW WAY OF SYNTHESISING DIARYLGOLD(III) COMPLEXES USING ORGANOMERCURY COMPOUNDS \*

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### Summary

Treatment of  $[\text{Me}_4\text{N}][\text{AuCl}_4]$  with di-organomercury derivatives  $\text{R}_2\text{Hg}$  in which R is an *ortho*-nitroaryl group ( $\text{R} = o\text{-O}_2\text{NC}_6\text{H}_4$  or  $2\text{-CH}_3, 6\text{-O}_2\text{NC}_6\text{H}_3$ ) gives anionic gold(III) complexes of the type *cis*- $\text{Me}_4\text{N}[\text{AuR}_2\text{Cl}_2]$ .

These react with (a) KCN, to give the anionic complexes *cis*- $\text{Me}_4\text{N}[\text{AuR}_2(\text{CN})_2]$ , (b) neutral monodentate ligands (L) to give the neutral complexes *cis*- $[\text{AuR}_2\text{Cl}(\text{L})]$  ( $\text{L} = \text{PPh}_3, \text{PCy}_3, \text{py}$ ), and (c) 1,2-bis(diphenylphosphino)ethane (dpe) in the presence of  $\text{NaClO}_4$  to give the cationic complexes *cis*- $[\text{AuR}_2(\text{dpe})]\text{ClO}_4$ . The complexes are thought to be the first transition metal complexes containing *ortho*-nitroaryl ligands.

### Introduction

The only reported methods for obtaining diarylgold(III) complexes involve the oxidation of (a) the gold(I) complexes  $[\text{AuXL}]$  ( $\text{X} = \text{Cl}, \text{Br}, \text{I}; \text{L} = \text{PPh}_3, \text{AsPh}_3, \text{Ph}_2\text{P}[(\text{CH}_2)_2]\text{PPh}_2$  and  $\text{Ph}_2\text{P}(\text{CH}_2)_4\text{PPh}_2$ ) with  $[\text{Ti}(\text{C}_6\text{F}_5)_2\text{X}]_2$  ( $\text{X} = \text{Cl}, \text{Br}, \text{I}$ ) to give  $[\text{Au}(\text{C}_6\text{F}_5)_2\text{XL}]$  complexes [1] or (b) the gold(I) anions  $[\text{AuR}_2]^-$  ( $\text{R} = \text{C}_6\text{F}_5, 2,4,6\text{-C}_6\text{F}_3\text{H}_2$ ) with halogens ( $\text{Cl}_2, \text{Br}_2, \text{I}_2$ ) or  $\text{TiCl}_3$  to give  $[\text{AuR}_2\text{X}_2]^-$  complexes [2]. Cationic complexes  $[\text{Au}(\text{C}_6\text{F}_5)_2(\text{PPh}_3)\text{L}]^+$  can be prepared by adding neutral ligands L to solutions of  $[\text{Au}(\text{C}_6\text{F}_5)_2\text{X}(\text{PPh}_3)]$  ( $\text{X} = \text{ClO}_4$  or  $1/2 \text{SO}_4$ ) [3]. These methods have been tested only for polyfluoroaryl, mainly  $\text{C}_6\text{F}_5$ , complexes.

We have also shown that an organotin compound can be used to prepare dibenzometalole complexes of gold(III) [4].

Recently we used chloro[2-(phenylazo)phenyl]mercury(II) for the synthesis of a monoarylgold(III) complex, *cis*-dichloro[2-(phenylazo)phenyl]gold(III) [5], through a transmetallation reaction. Organomercury compounds are being increasingly used as synthetic intermediates in organometallic chemistry [6], but in the case of gold only

\* Dedicated to Prof. Antonio Soler Martinez on the occasion of his retirement.

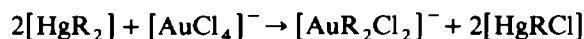
reactions of gold(I) complexes with organomercury compounds have been reported [7]. Our continuing interest in developing new routes to organogold complexes prompted us to examine the potential of the use of organomercury compounds.

We present here a new way for obtaining diaryl gold(III) complexes containing *ortho*-nitrophenyl groups. This type of group has been chosen for several reasons, one of them being the instability of the corresponding organolithium derivatives [8], which have been very little used [9]. In addition, it is well known that *ortho*-substituents exert a profound influence on the stability of arylmetal complexes, and furthermore, our interest in the synthesis of metallacycle complexes [4,5,11] suggests to us the possibility that the *ortho*-nitrophenyl group may act as a new type of chelate ligand to give a five-membered metallacycle.

Some of the complexes reported were described in a preliminary communication [12].

## Results and discussion

When acetone solutions containing  $[\text{HgR}_2]$  and  $\text{Me}_4\text{N}[\text{AuCl}_4]$  in 2/1 molar ratios are refluxed the anionic complexes  $\text{Me}_4\text{N}[\text{AuR}_2\text{Cl}_2]$  are obtained ( $\text{R} = o\text{-O}_2\text{NC}_6\text{H}_4$  (I),  $2\text{-CH}_3$ ,  $6\text{-O}_2\text{NC}_6\text{H}_3$  (II)).



The reactions are slow, especially when the aryl group is the more sterically demanding nitrotolyl. Thus, I is obtained in yields of 12, 45, 89 and 92% after reflux for 6, 12, 20 and 30 h, respectively, whereas II can be isolated under similar conditions only in 50% yield after 50 h. Because of the similar solubilities of II and  $[\text{HgR}_2]$  it is advisable to use a 1/1 molar ratio of the reagents, since the excess of  $\text{Me}_4\text{N}[\text{AuCl}_4]$  can easily be removed washing with ethanol and the yield is increased for shorter reaction times (e.g. 70% after 18 h). When  $[\text{Hg}(\text{C}_6\text{H}_5)_2]$  was used as arylating agent metallic gold was formed rapidly and exclusively, providing further evidence for the special stability which *ortho*-substituents confer on aryl complexes.

Complexes I and II each show two strong bands in their IR spectra at 280 and 300  $\text{cm}^{-1}$  and at 285 and 305  $\text{cm}^{-1}$ , respectively, that can be assigned to  $\nu(\text{AuCl})$  ( $\text{A}_1$ ) and ( $\text{B}_1$ ) for a *cis*-geometry ( $\text{C}_{2v}$ ). A *trans*-geometry ( $\text{D}_{2h}$ ) would give only a band ( $\text{B}_{2u}$ ) in the region 340–360  $\text{cm}^{-1}$  due to the lower *trans*-influence of a chloro with respect to an aryl ligand [2]. It is known that *cis*-dihalodiarylgold(III) complexes are always obtained when the *trans*-isomers are heated in solution [2].

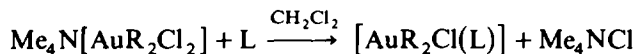
The  $^1\text{H}$  NMR ( $(\text{CD}_3)_2\text{SO}$ ,  $\text{Me}_4\text{Si}$ ) of complex II contains a multiplet centered at 7.18 ppm (6H, phenyl protons) and two singlets at 3.09 ppm (12H,  $\text{N}(\text{CH}_3)_4$ ) and 2.52 ppm (6H,  $\text{CH}_3$ -aryl).

When complexes I or II are treated in acetone at room temperature with an excess of KCN, substitution of both chloro ligands takes place to give anionic complexes  $\text{Me}_4\text{N}[\text{AuR}_2(\text{CN})_2]$  ( $\text{R} = o\text{-O}_2\text{NC}_6\text{H}_4$  (III);  $2\text{-CH}_3$ ,  $6\text{-O}_2\text{NC}_6\text{H}_3$  (IV)); these each show two bands, at 2165 and 2159 and at 2169 and 2162  $\text{cm}^{-1}$ , respectively. This allows the formulation of both complexes as *cis*- $[\text{AuR}_2(\text{CN})_2]^-$ .

All the anionic complexes I–IV show molar conductivities in acetone solutions in the range 100–115  $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$ , as expected for a 1/1 electrolyte (see Table 1).

When suspensions of complexes I or II in dichloromethane are treated with neutral ligands a precipitate of  $\text{Me}_4\text{NCl}$  and solutions of neutral complexes

$[\text{AuR}_2\text{Cl}(\text{L})]$  are obtained ( $\text{R} = o\text{-O}_2\text{NC}_6\text{H}_4$ ,  $\text{L} = \text{PPh}_3$  (V),  $\text{PCy}_3$  (VI),  $\text{py}$  (VII);  $\text{R} = 2\text{-CH}_3$ ,  $6\text{-O}_2\text{NC}_6\text{H}_3$ ,  $\text{L} = \text{PPh}_3$  (VIII),  $\text{PCy}_3$  (IX),  $\text{py}$  (X)).



The reactions proceed quickly as shown by the rapid dissolution of the starting complexes and immediate precipitation of  $\text{Me}_4\text{NCl}$ . Even if a great excess of  $\text{py}$  is used a mixture of I and VII is always isolated, but can be separated due to the better solubility of VII in diethyl ether. Thus the lower yield in the preparation of VII can be accounted for by assuming that the reaction is reversible. Surprisingly, the corresponding reaction between II and pyridine gives X in high yield, and no unchanged II is present in the reaction products.

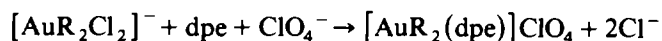
Complex IX ( $\text{R} = 2\text{-CH}_3, 6\text{-O}_2\text{NC}_6\text{H}_3$ ) shows an appreciable molar conductivity in acetone ( $33 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$ ), which could be due to the existence of an equilibrium such as:



the dissociation probably being favoured by the presence of the very bulky ligands. In the cationic complex, an oxygen atom of the nitro group might occupy the vacant coordination site and we are investigating this possibility.

Complexes V–X show a medium intensity band in the  $310\text{--}320 \text{ cm}^{-1}$  region (V, 320; VI, 320; VII, 315; VIII, 310; IX, 312; X,  $320 \text{ cm}^{-1}$ ) similar to that found for other neutral *cis*-diarylhalogold(III) complexes containing P-, N- and S-donor neutral ligands [1,4]. Furthermore  $\nu(\text{AuCl})$  *trans* to pyridine has been assigned at  $363 \text{ cm}^{-1}$  in  $[\text{Au}(\text{CN})_2\text{Cl}(\text{py})]$  [13]. Complex VIII shows in its  $^1\text{H}$  NMR spectra ( $\text{CDCl}_3$ ,  $\text{Me}_4\text{Si}$ ) a multiplet centered at 7.62 ppm (2H, phenyl protons) and two singlets at 2.69 (3H) and 2.18 ppm (3H) corresponding to the two different *ortho*-methyl groups. For these reasons we assign the bands observed in the IR spectra to  $\nu(\text{AuCl})$  *trans* to the phenyl groups, and this implies a *cis*-geometry for complexes V–X.

If a bidentate ligand such as 1,2-[bis(diphenyl)phosphino]ethane (dpe) is treated with I or II in the presence of  $\text{NaClO}_4$ , cationic complexes  $[\text{AuR}_2(\text{dpe})]\text{ClO}_4$  ( $\text{R} = o\text{-O}_2\text{NC}_6\text{H}_4$  (XI);  $2\text{-CH}_3, 6\text{-O}_2\text{NC}_6\text{H}_3$  (XII)) are obtained.



The mononuclear nature of the cationic complexes XI and XII has been confirmed by measuring the molar conductivities of solutions of several concentrations (range of equivalent concentrations  $4 \times 10^{-3}$  to  $3 \times 10^{-4}$  and  $3 \times 10^{-3}$  to  $9 \times 10^{-5}$ , respectively) in nitromethane. The equations representing the equivalent conductivity as a function of the equivalent concentration are  $\Lambda_e = 79.4\text{--}242.6 \text{ c}^{1/2}$  and  $\Lambda_e = 67.7\text{--}183.9 \text{ c}^{1/2}$ . The slopes are in the range observed for other 1/1 electrolytes in nitromethane [14].

All the complexes reported show a band in each of the regions  $1510\text{--}1495$ ,  $1345\text{--}1320$  and  $903\text{--}875 \text{ cm}^{-1}$ , which could be tentatively assigned to  $\nu_{\text{asym}}(\text{NO}_2)$ ,  $\nu_{\text{sym}}(\text{NO}_2)$  and  $\nu(\text{CN})$ , respectively. They appear in the ranges expected for nitroaromatic groups, which might suggest that there is no appreciable O...Au interaction. We have recently prepared an *ortho*-nitrophenylpalladium complex in which this type of bonding is present, and in that case there is an important change, mainly in the absorption corresponding to  $\nu_{\text{sym}}(\text{NO}_2)$  [15]. Other bands in the region

TABLE I  
ANALYTICAL AND OTHER DATA FOR COMPLEXES I-XII

Complex <sup>a</sup>	M.p. (°C)	Colour	$\Delta_M^b$	Analytical data (Found(calcd.) (%))				Yield (%)
				C	H	N	Au	
<i>cis</i> -Me <sub>4</sub> N[AuR <sub>2</sub> Cl <sub>2</sub> ] (I)	186	cream	115	32.82 (32.78)	3.50 (3.44)	7.00 (7.17)	33.30 (33.60)	92
<i>cis</i> -Me <sub>4</sub> N[AuR <sub>2</sub> Cl <sub>2</sub> ] (II)	200 (d)	white	108	34.74 (35.20)	4.04 (3.94)	6.86 (6.84)	31.40 (32.06)	70
<i>cis</i> -Me <sub>4</sub> N[AuR <sub>2</sub> (CN) <sub>2</sub> ] (III)	193	cream	106	38.51 (38.10)	3.71 (3.55)	12.59 (12.34)	34.51 (34.72)	78
<i>cis</i> -Me <sub>4</sub> N[AuR <sub>2</sub> (CN) <sub>2</sub> ] (IV)	223 (d)	white	100	39.13 (40.34)	3.93 (4.06)	10.73 (11.76)	33.38 (33.08)	82
<i>cis</i> -[AuR <sub>2</sub> Cl(PPh <sub>3</sub> )] (V)	151	cream	19	47.92 (48.76)	3.92 (3.14)	3.79 (3.79)	25.76 (26.66)	85
<i>cis</i> -[AuR <sub>2</sub> Cl(PCy <sub>3</sub> )] (VI)	155	cream	7	48.33 (47.60)	5.74 (5.46)	3.39 (3.70)	26.53 (26.02)	75
<i>cis</i> -[AuR <sub>2</sub> Cl(py)] (VII)	120 (d)	cream	15	36.84 (36.74)	3.09 (2.36)	7.76 (7.56)	35.01 (35.44)	38
<i>cis</i> -[AuR <sub>2</sub> Cl(PPh <sub>3</sub> )] (VIII)	165 (d)	cream	1	50.22 (50.11)	3.74 (3.55)	3.56 (3.65)	25.17 (25.68)	77
<i>cis</i> -[AuR <sub>2</sub> Cl(PCy <sub>3</sub> )] (IX)	164 (d)	white	33	47.73 (48.95)	5.64 (5.78)	3.67 (3.57)	24.05 (25.08)	56
<i>cis</i> -[AuR <sub>2</sub> Cl(py)] (X)	207 (d)	white	0	38.89 (39.09)	3.13 (2.93)	7.00 (7.19)	32.40 (33.74)	76
<i>cis</i> -[AuR <sub>2</sub> (dpe)ClO <sub>4</sub> ] (XI)	205	white	119	48.59 (48.60)	3.17 (3.43)	2.82 (2.98)	20.53 (20.98)	82
<i>cis</i> -[AuR <sub>2</sub> (dpe)ClO <sub>4</sub> ] (XII)	221 (d)	white	129	50.75 (49.68)	3.77 (3.75)	2.50 (2.90)	20.05 (20.37)	87

<sup>a</sup> R = *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>; R' = 2-CH<sub>3</sub>-6-O<sub>2</sub>NC<sub>6</sub>H<sub>3</sub>. <sup>b</sup> On approx. 10<sup>-4</sup> M solution in acetone ( $\Omega^{-1}$  cm<sup>2</sup> mol<sup>-1</sup>).

800–650  $\text{cm}^{-1}$  are difficult to assign since the absorptions are due to interaction of  $\text{NO}_2$  and C–H out-of-plane bending frequencies [16].

All the reported complexes are thermally stable in the solid state and in solution, and stable also towards atmospheric moisture and daylight.

Table 1 presents analytical and other data for complexes I–XII.

## Experimental

IR spectra were recorded in the range 4000–250  $\text{cm}^{-1}$  on a Perkin–Elmer 457 spectrophotometer using Nujol mulls between polyethylene sheets or KBr pellets. The spectra of complexes III and IV were also recorded on a Nicolet MX-1 spectrophotometer in order to distinguish clearly between the two  $\nu(\text{CN})$  bands. Conductivities were measured on  $5 \times 10^{-4}$  M solutions with a Phillips 9501 conductimeter. Melting points were determined 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 with an aqueous solution of hydrazine.

The starting organomercury compounds and  $\text{Me}_4\text{N}[\text{AuCl}_4]$  were prepared by literature methods [17,18]. Other reagents were obtained from commercial sources. Solvents were distilled before use. Unless otherwise stated the reactions were carried out with magnetic stirring and without special precautions against daylight or moisture. Complexes I, V and VIII were prepared as described in our preliminary communication [12].

### *cis*- $\text{Me}_4\text{N}[\text{AuR}_2\text{Cl}_2]$ ( $R = 2\text{-CH}_3, 6\text{-O}_2\text{NC}_6\text{H}_3$ ) (II)

To a solution of  $\text{Me}_4\text{N}[\text{AuCl}_4]$  (384 mg, 0.93 mmol) in acetone (20 ml) was added solid  $[\text{HgR}_2]$  (440 mg, 0.93 mmol). The mixture was refluxed for 18 h then the solution was concentrated (5 ml) and ethanol (15 ml) slowly added to give a solid, which was filtered off and washed with diethyl ether to give II.

### *cis*- $\text{Me}_4\text{N}[\text{AuR}_2(\text{CN})_2]$ ( $R = o\text{-O}_2\text{NC}_6\text{H}_4$ ) (III)

To a solution of I (53 mg, 0.09 mmol) in acetone (20 ml) was added KCN (25 mg, 0.38 mmol). After 14 h at room temperature the suspension was filtered and the solution was concentrated (3 ml) and diethyl ether (30 ml) slowly added. The resulting solid was filtered off and washed with diethyl ether to give III.

### *cis*- $\text{Me}_4\text{N}[\text{AuR}_2(\text{CN})_2]$ ( $R = 2\text{-CH}_3, 6\text{-O}_2\text{NC}_6\text{H}_3$ ) (IV)

To a suspension of II (88 mg, 0.14 mmol) in acetone (8 ml) was added KCN (48 mg, 0.74 mmol). After 5 h at room temperature the solvent was removed and the residue extracted with dichloromethane (10 ml). The solution was filtered and concentrated (5 ml), and diethyl ether (10 ml) was added to give a solid, which was filtered off and washed with diethyl ether to give IV.

### *cis*- $[\text{AuR}_2\text{Cl}(\text{PCy}_3)]$ ( $R = o\text{-O}_2\text{NC}_6\text{H}_4$ (VI), $2\text{-CH}_3, 6\text{-O}_2\text{NC}_6\text{H}_3$ (IX))

Complex I or II and the equivalent amount of  $\text{PCy}_3$  were mixed in dichloromethane (25 ml). After 2 h at room temperature  $\text{Me}_4\text{NCl}$  was removed by filtration, and the solution was concentrated (3 ml) and diethyl ether was added slowly to give a solid which was filtered off and washed with diethyl ether to give VI or IX.

*cis*[AuR<sub>2</sub>Cl(py)] (R = *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>) (VII)

To a suspension of I (100 mg, 0.17 mmol) in dichloromethane (25 ml) was added anhydrous pyridine (0.5 ml). After 12 h at room temperature Me<sub>4</sub>NCl was removed by filtration, and the solution was concentrated (3 ml) and diethyl ether (25 ml) was added to precipitate unchanged I. The solution was filtered, then concentrated (5 ml), and n-hexane (25 ml) added to give a solid, which was filtered off and washed with n-hexane to give VII.

*cis*-[AuR<sub>2</sub>Cl(py)] (R = 2-CH<sub>3</sub>,6-O<sub>2</sub>NC<sub>6</sub>H<sub>3</sub>) (X)

Starting from II the method was similar to that described for VII, except that the reaction time was 15 min and that complex X separated when diethyl ether was added.

*cis*-[AuR<sub>2</sub>(dpe)]ClO<sub>4</sub> (R = *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>) (XI)

To a suspension of I (130 mg, 0.22 mmol) in dichloromethane (18 ml) was added solid dpe (115 mg, 0.29 mmol). After 7 h at room temperature Me<sub>4</sub>NCl was removed by filtration and the solvent evaporated to dryness. Acetone (25 ml) and NaClO<sub>4</sub> (40 mg, 0.33 mmol) were added and after 16 h at room temperature the solvent was removed and the residue was extracted with dichloromethane (10 ml). The solution was concentrated (3 ml) and diethyl ether slowly added (30 ml) to give a solid, which was filtered off and washed with diethyl ether to give XI.

*cis*-[AuR<sub>2</sub>(dpe)]ClO<sub>4</sub> (R = 2-CH<sub>3</sub>,6-O<sub>2</sub>NC<sub>6</sub>H<sub>3</sub>) (XII)

To a suspension of II (86 mg, 0.14 mmol) in acetone (12 ml) were added solid dpe (76 mg, 0.19 mmol) and solid NaClO<sub>4</sub> (28 mg, 0.20 mmol). After 10 h refluxing and 24 h at room temperature the suspension was filtered and the solvent removed. The residue was extracted with dichloromethane and the solution was treated as described for XI to give XII.

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