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SYNTHESIS AND CHARACTERIZATION OF TRIARYLGOLDZINC COMPOUNDS *

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Summary

The reactions of diphenylzinc and di-*p*-tolylzinc with gold(I) and gold(III) salts yield dimeric species of the type $(Ar_3AuZn)_2$. On the basis of spectroscopic data and physical measurements a cyclic structure is proposed which contains both aryl groups exclusively bound to zinc and aryl groups bridging between gold and zinc. Upon reaction with bidentate nitrogen-containing ligand molecules, complexes of the type Ar_3AuZnL are obtained which probably have an "ate"-like structure, $ArZn^+LAu^-Ar_2$. In reactions with phosphorus-containing ligand molecules, the Ar_3AuZn compounds are cleaved to give ArAuL complexes. This difference in behaviour is discussed in terms of the differing affinities of gold and zinc for N- and P-donor molecules.

Introduction

Arylgold compounds have been made in several ways. The oldest method, direct auration of aromatic compounds, yielding arylgold(III) derivatives [1] is rather limited in scope [2]. More recently, the use of arylmagnesium and aryllithium compounds as arylating agents for gold was reported [3,4,5]. Only arylgold(I) derivatives can be made with these reagents, because of their strongly reducing properties. Diarylmercury compounds have been used by Liddle and Parkin [6] to synthesize arylgold(III) complexes. Van Koten and Noltes described complex compounds of the type $R_4Au_2Li_2$ which could be converted into simple arylgold(I) compounds [7,8]. Each of these methods has its limitations.

To extend the scope of this type of reaction, we decided to investigate the suitability of arylzinc compounds as arylating agents for gold. In this paper, we describe the synthesis and properties of triarylgoldzinc compounds, the unex-

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* Dedicated to Prof. Dr. L. Birkofer, University of Dusseldorf, at the occasion of his 65th anniversary.

pected complex products formed in the reaction of gold salts with diarylzinc compounds *.

Results

When an ether solution of gold(I) carbonylchloride, $Au(CO)Cl$, was added at —40° C to an ether solution containing a more than twofold excess of diphenylzinc, a brilliant red precipitate was formed which analyzed correctly for Ph_3AuZn(I). Carbon monoxide was liberated during the addition and the supernatant solution contained phenylzinc chloride:

$$2Ph_2Zn + Au(CO)Cl \xrightarrow[-40°C]{Et_2O} \underset{(I)}{Ph_3AuZn\downarrow} + PhZnCl + CO$$

I was obtained also by treating a suspension of gold(III) chloride, $AuCl_3$, in ether at —70° C with a large excess of diphenylzinc. Part of the zinc compound in that case may be used to reduce gold(III) chloride to monovalent gold, as shown by the concomitant formation of one mole of biphenyl for every mole of $AuCl_3$ consumed:

$$4Ph_2Zn + AuCl_3 \xrightarrow[-70°C]{Et_2O} \underset{(I)}{Ph_3AuZn\downarrow} + 3PhZnCl + Ph—Ph$$

The production of I by this second route may proceed as well via formation of triphenylgold, which then eliminates biphenyl in a reductive elimination step. We were unable, however, to isolate Ph_3Au from the reaction mixture or demonstrate its presence in any other way. Red tri-*p*-tolylgoldzinc(II) was prepared similarly from $Au(CO)Cl$ and di-*p*-tolylzinc. On the other hand, some other substituted diarylzinc compounds (viz. bis(2,6-dimethoxyphenyl)zinc, bis(2-methoxyphenyl)zinc, bis(2-vinylphenyl)zinc and bis(2-methylphenyl)zinc) yielded cream-coloured products with the composition $Ar_2AuZnCl$. These compounds will be described separately, together with the corresponding compounds in which zinc is replaced by cadmium and mercury.

Both I and II show appreciable thermal stability (decomposition after 3 h at 50°, after 30 s at 70—75°; explosive decomposition at 130° and 150° C, respectively), but are sensitive towards air and moisture. The compounds are stable at room temperature in aromatic hydrocarbons or carbon disulfide, but decompose slowly in polar solvents like THF and DMF. In view of the unexpected formation of these bimetallic gold-zinc compounds, we also investigated the reactions of diphenylzinc with silver and copper salts. The reaction with silver salts did not yield corresponding silver-zinc compounds, but has been developed into an excellent preparative route for phenylsilver [10]. Likewise, cuprous chloride reacted with diphenylzinc to give phenylcopper, no well-defined copper-zinc compounds being formed.

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* A preliminary account of part of this work has already appeared [9].

Characterization

As a general structure in solution for compounds of the type Ar_3AuZn, we propose a dimeric associate as shown in Fig. 1 for Ar = phenyl. This structure, which is characterized by bridging aryl groups between gold and zinc, is suggested on the basis of the following chemical evidence:

(*i*) Molecular weight determinations by cryoscopy in benzene showed triphenylgoldzinc to be a non-dissociating dimer, $(Ph_3AuZn)_2$.

(*ii*) Proton-noise decoupled ^{13}C NMR spectra, recorded in carbon disulfide solution, revealed the presence of two types of aryl groups. The chemical shifts of one set of signals correspond closely with those of the parent diarylzinc compound, whereas the other set, which has greater intensity, has different chemical shifts (Table 1). The intensities of the two sets roughly indicate that the two kinds of aryl groups are present in a 1 : 2 ratio.

(*iii*) The ^1H NMR spectra confirm the presence of two kinds of aryl groups in a 1 : 2 ratio (Table 2). In the spectra of I, the single signal which, from the integration, must be attributed to aryl groups exclusively bound to zinc (cf. Fig. 1), is split into two multiplets at temperatures below −25° C. This does not occur for the signals attributed to bridging aryl groups, suggesting that the bridging aryl groups are more rigidly fixed in the molecular framework. In the spectra of II, a similar temperature dependence is observed. In this case also, the low-intensity signals show an *AA'BB'* pattern changing upon cooling to −50° C into one broad line. On the other hand the high-intensity signals (also an *AA'BB'* pattern) do not change over the temperature range +30° to −80° C.

Any doubt regarding the inertness of these triarylgoldzinc compounds towards carbon disulfide was removed by recording ^1H NMR spectra of I in toluene-*d*8. The spectra were identical to those recorded in CS_2, but were of lower quality for solubility reasons.

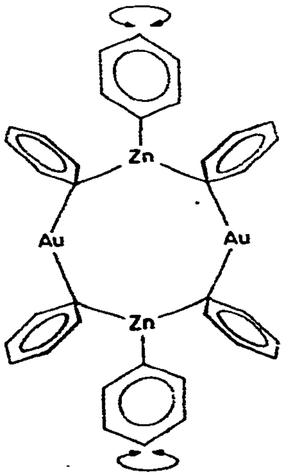
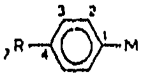


TABLE 1

^{13}C NMR RESONANCES OF Ar_3AuZn AND Ar_2Zn [a]

	C_1^A [b]	C_2^A	C_3^A	C_4^A	C_7^A	C_1^B	C_2^B	C_3^B	C_4^B	C_7
Ph_3AuZn	146.7	137.7	127.6	128.8		146.7	132.0	129.5	127.3	
Ph_2Zn	148.6	138.1	128.3	128.8						
$pTol_3AuZn$	146.6	141.5	128.4	135.5	22.3	147.5	141.8	128.2	130.1	22.7
$pTol_2Zn$	144.1	141.7	129.7	134.9	22.2					

[a] In CS_2, in ppm relative to TMS, gold compounds at −20°, zinc compounds at 35°C. [b] A and B relate to Fig. 1.

TABLE 2

^{1}H NMR DATA FOR Ar_3AuZn AND Ar_2Zn AT 100 MHz [a]

Compound	Aryl A	Aryl B	CH_3
Ph_3AuZn [b] (−18°C)	7.42s [d] (5H)	8.20m (4H) 7.84m (6H)	
Ph_2Zn [c]	7.32s		
p-Tol_3AuZn [b] (−20°C)	6.58, 6.65, 6.72, 6.80 [e] (4H)	7.00, 7.08, 7.52, 7.60 [e] (8H)	2.14s (3H) 2.33s (6H)
p-Tol_2Zn [b]	6.84, 6.97, 7.21, 7.34 [e]		2.24s

[a] In ppm relative to TMS. [b] In CS_2. [c] In $C_6D_5CD_3$. [d] s = singlet, m = multiplet. [e] $AA'BB'$ pattern.

TABLE 3

X-SENSITIVE BANDS OF Ph_3AuZn AND RELATED COMPOUNDS (cm^{-1})

Mode [a]	$PhAuCl_2$ [b]	Ph_3AuZn	Ph_2Zn
q	1052m	1070m 1054m	1072m
r	662s	677s	676m
y	473m	450m 470s	438s

[a] Symbols according to ref. 11. [b] Prepared by auration, ref. 2.

(*iv*) IR frequencies of I could not be assigned unambiguously, because the phenyl groups in this compound do not necessarily have the C_{2v} symmetry needed for an analysis by Whiffen's procedure [11]. In hexaphenyldialuminium and tetramethyldiphenyldialuminium the bridging phenyl groups are known to deviate from C_{2v} symmetry. Therefore, only a tentative assignment of the X-sensitive frequencies (X = Au or Zn) is given in Table 3.

(*v*) Triphenylgoldzinc was titrated with ligand molecules in benzene solution using the microwave titration technique developed by Adema and Schrama [13] (vide infra). In this method titrations are followed by measuring the changes in the transmission curve of a microwave cavity in which the complexation takes place. It appeared that triphenylgoldzinc alone did not dampen the microwave cavity to a measurable extent. This implies that the compound has a very small or zero dipole moment since the damping is correlated with the dipole moment of the solute if a non-polar solvent such as benzene is used [13]. This observation is in accord with the symmetric structure proposed for the triphenylgoldzinc dimer.

Reactions with nitrogen- and phosphorus-containing ligand molecules

In general, organozinc compounds form more stable complexes with N-donor than with P-donor molecules. In organogold chemistry the reverse is true. This difference in behaviour is apparent in the reactions of arylgoldzinc compounds with both types of ligands. Our initial aim was the cleavage of Ph_3AuZn into a diphenylzinc-complex and phenylgold, since simple, uncomplexed arylgold(I) species are unknown, but this goal was not realized.

TABLE 4

COMPLEXES FORMED BY TREATING $(Ph_3AuZn)_2$ WITH LIGAND MOLECULES

Ligands	Number of inflexion points	Final ratio $(Ph_3AuZn)_2$: L	Isolated products
Bipy	2	1 : 2	Ph_3AuZnbipy
TMED	1	1 : 2	Ph_3AuZnTMED
PPh_3	1	1 : 2	$PhAuPPh_3$
Diphos	1	1 : 1	$(PhAu)_2$diphos

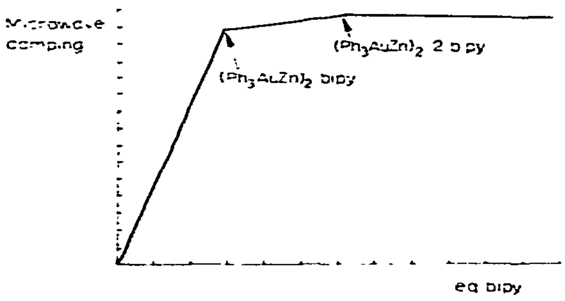


Fig. 2.

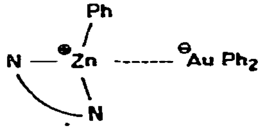
The reactions of triphenylgoldzinc with N- or P-containing ligands were studied by the microwave titration technique described above. The complexes formed were also made on a preparative scale and, as far as thermal stability permitted, characterized by elemental analysis, and by IR and NMR spectroscopy. Table 4 summarizes these results and Fig. 2 shows a typical microwave titration curve.

Bipy was found to react with red, dimeric triphenylgoldzinc in a two-step reaction, yielding initially a 1 : 1 complex which reacted further to give a final colourless 1 : 2 complex in which the zinc : Bipy ratio is 1 : 1.

Both the initial 1 : 1 complex and the final 1 : 2 complex (the latter was also made on a preparative scale) show a very large microwave damping, indicating that appreciable charge separation occurs in these complexes. Since it is very difficult to visualize a strongly asymmetrical dimeric structure for the final complex, remembering that Bipy always acts as a bidentate chelating ligand, we propose a monomeric "ate"-like structure for $Ph_3AuZnBipy$ (Fig. 3).

Unfortunately, the insolubility of the final complex prevented a molecular weight determination and further proof of its molecular structure cannot be offered at present.

TMED reacted with I in a one-step reaction resulting directly in a complex with a zinc : TMED ratio of 1 : 1. This is remarkable, since organozinc compounds usually react with TMED in a two-step reaction in which initially TMED-bridged complexes with a zinc : TMED ratio of 2 : 1 are formed [14]. The ^1H NMR spectrum of the complex obtained on a preparative scale showed CH_2N- and CH_3N-signals at 2.52 and 2.20 ppm, respectively (both singlets). These chemical shifts, which are very similar to those observed in the compounds $Ph_2Zn.TMED$ and $PhZnCl.TMED$ (which certainly contain chelating TMED ligands) sug-



(N—N = Bipy)

Fig. 3.

gest the presence of a chelating TMED ligand in the gold/zinc TMED complex. Furthermore, this complex shows a very large microwave damping and so has a large dipole moment. On the basis of arguments similar to those used for the Bipy complex we suggest a corresponding monomeric "ate"-like structure for Ph_3Au-$Zn.TMED$. Although this complex is soluble in benzene its thermal stability in solution is too low to permit molecular weight determination. This structural proposal is supported by the results of ^1H and ^{13}C NMR spectroscopy. Firstly, the presence of two different kinds of phenyl groups appears both from the ^1H and ^{13}C NMR spectra. As in the starting compound the ratio of these groups is still 1 : 2. Secondly, when comparing the ^{13}C NMR chemical shifts of Ph_3AuZn and $Ph_3AuZn.TMED$ (cf. Table 4), it is obvious that in the latter case the signals attributed to the formerly bridging phenyl groups (B) are shifted to lower field much more than those attributed to the zinc-bound phenyl groups (A). This larger shift, which can be interpreted in terms of a higher negative charge on the formerly bridging phenyl groups, which become exclusively bound to gold upon complexation, is in accord with the high negative charge on the gold in the "ate" complex.

The microwave titration of dimeric triphenylgoldzinc with triphenylphosphine showed that each Ph_3AuZn unit reacts with only one PPh_3 ligand. The microwave damping of the resulting colourless solution was small, indicating that only species with small dipole moments had formed. When this reaction was carried out on a preparative scale using a 1 : 1 ratio of PPh_3 : Ph_3AuZn, a thermally stable colourless solution was obtained. Upon blowing air through this solution, a precipitate of an oxidized zinc species was formed. The colourless filtrate upon evaporation yielded $PhAuPPh_3$ quantitatively. A similar reaction with 1,2-bis(diphenylphosphino)ethane, (diphos), yielded a stable complex analyzing for $(PhAu)_2$diphos. This behaviour of triphenylgoldzinc towards phosphine ligands contrasts strongly with that towards nitrogen-containing ligands. In the latter case, oxidation of the resulting solutions caused complete decomposition as indicated by the formation of metallic gold. Moreover, these solutions were only stable up to 10° C.

We interpret these results in terms of the difference in the affinities of gold and zinc towards N-donor and P-donor ligand molecules. With N-donor ligands, the Ph_3AuZn unit reacts to give complexes with nitrogen coordinated to zinc, which have an ate-like structure. In these complexes, the positive charge on zinc is stabilized by electron-donation from nitrogen. Because of the small af-

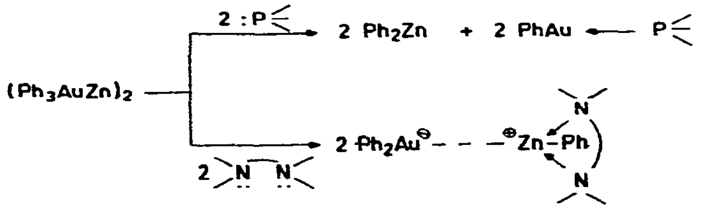


Fig. 4.

finity of zinc for phosphorus, P-donor ligands are unable to stabilize Ph_3AuZn units as "ate" complexes. Instead, the P-donor ligands coordinate to gold, thereby destabilizing Ph_3AuZn and cleaving it into a PhAu-phosphine complex and diphenylzinc.

A schematic representation of these reactions is given in Fig. 4.

Experimental

Unless stated otherwise all experiments were carried out under dry, oxygen-free nitrogen. Solvents were purified and stored under nitrogen. Solvents, solutions and liquid reagents were handled by syringes.

IR investigations were carried out using a Perkin-Elmer 457 double beam grating spectrometer. Spectra were recorded between 4000 and 250 cm^{-1} from nujol suspensions between polyethylene films or KBr discs; polystyrene was used for calibration. Frequencies given are believed to be accurate within 3 cm^{-1}. Samples were cooled to 0°C by blowing evaporating liquid nitrogen along their surfaces.

NMR spectra were recorded using Varian A-60, EM360, XL100, or CFT-20 spectrometers by Messrs. D. Seykens, A.V.E. George and J. Schuring. ^1H NMR 100 MHz spectra were recorded by Miss T. Volp on a Varian HA-100. The ^1H values are believed to be accurate within 2Hz, ^{13}C values within 0.1 ppm. Decomposition temperatures (±5°C) and melting points (±1°C) were determined on a Kofler Heizbank. Temperatures given pertain to immediate black colouration, or, when stated, to the time after which a first blackening was observed.

Analyses were carried out by the Element Analytical Section of the Institute for Organic Chemistry TNO, Utrecht, under supervision of Mr. W.J. Buis. Molecular weights were measured by cryometry with a Knaur "Kryoskopie-gerät" or by ebulliometry using a Gallenkamp MW 125, both modified for operation in a nitrogen atmosphere.

Starting materials

Diarylzinc compounds were prepared from arylmagnesium bromide and dry zinc chloride in procedures related to those of Sheverdina et al. [15].

Diphenylzinc. A 10% solution of dry $ZnCl_2$ in ether was added dropwise to the Grignard reagent prepared from Mg (12.2 g, 500 mmol) and PhBr (81 g, 515 mmol) in ether until a Gilman test was negative. The mixture was transferred to a separation funnel and added in small portions to an evacuated distillation apparatus fitted with an air-cooled condenser. When all solvent was thus removed, Ph_2Zn was distilled from the mixture into the condenser at a bath-temperature of 250—300°C (b.p. 180°C/10^{-4} mmHg), yielding 20.4 g (93 mmol, 37% overall yield) of pure product after washing with pentane (5 × 50 ml).

Preparation of Ph_2Zn by addition of $ZnCl_2$ to PhLi in ether, followed by extraction of the crude product with benzene and precipitation with pentane, resulted in lower yields than those from the procedure described above.

Bis(p-tolyl)zinc. A 10% solution of $ZnCl_2$ in ether was added dropwise to a Grignard reagent prepared from Mg (12.2 g, 500 mmol) and *p*-bromotoluene (85.6 g, 500 mmol) in ether until a Gilman-test was negative. All solvent was removed by suction (70°C/0.1 mmHg) and the residue was extracted exhaustively with toluene. Evaporation of the toluene yielded impure p-Tol_2Zn, which was recrystallized from toluene. Yield: 60 g, (48%).

The reaction procedure suggested in ref. 15, consisting of immobilizing the magnesium salts present in the crude reaction mixture by addition of dioxane followed by extraction of the soluble Ar_2Zn-dioxanate with benzene, proved cumbersome due to the volume of the precipitate formed, and yields of only 20% were obtained.

Bis(o-vinylphenyl)zinc. This was prepared in 30% yield from o-bromostyrene in a synthesis on 110 mmol scale analogous to the preparation of p-Tol_2Zn.

Bis(o-tolyl)zinc. This was prepared as described for the p-tolyl compound, from o-MePhMgBr and $ZnCl_2$ in THF. It crystallized from toluene with 2 molecules of THF.

Bis(2,6-dimethoxyphenyl)zinc. Bis(2,6-dimethoxyphenyl)zinc was prepared from 2,6-diMeOPhLi (100 mmol) in ether/hexane and $ZnCl_2$ at —20° C. Extraction with benzene gave the product in 53% yield.

Preparation of triarylgoldzinc compounds

Triphenylgoldzinc. A solution of Au(CO)Cl (210 mg, 0.8 mmol) in ether (100 ml) was slowly added to a solution of Ph_2Zn (670 mg, 3.1 mmol) in ether (100 ml) at —40° C. The mixture turned yellow after the first few drops: further addition resulted in the formation of a red precipitate. After 20 min stirring at —40° C, the supernatant liquid was decanted and the precipitate was washed three times with ether (50 ml). After drying at —20° C/0.1 mmHg, 370 mg (0.7 mmol, 90%) of Ph_3AuZn was obtained. Decomposition (DTA): 127° C at 10°/min, 114° C at 5°/min. Analysis: Found: C, 38.1; H, 3.0; Au, 40.0; Zn, 13.8. $C_{18}H_{15}AuZn$ calcd.: C, 43.80; H, 3.06; Au, 39.90; Zn, 13.24.

The preparation from $AuCl_3$ was carried out at —70° C, starting with a suspension of $AuCl_3$ (0.31 g, 1.0 mmol) in ether (30 ml), to which a chilled solution of Ph_2Zn (0.70 g, 3.2 mmol) in ether was added in 5 min. The mixture turned blcak immediately, but after 30 min at —70° C the slurry slowly became orange/red. The slurry was then decanted from some black resinous material. When the precipitate settled out, it was washed with ether and pentane, followed by drying at 0° C/0.1 mmHg. The red product was identical with the Ph_3AuZn obtained from Au(CO)Cl according to IR and NMR. Yield: 210 mg, 40%.

Relatively large amounts of PhPh were isolated from the washing solutions. No PhCl or 4-ClPhPh were present (GLC, IR).

When this reaction was carried out in the presence of PPh_3, no Ph_3AuPPh_3 was formed. Reaction of $AuCl_3$ (1.6 mmol), Ph_2Zn (4.8 mmol) and PPh_3 (1.6 mmol) afforded $PhAuPPh_3$, which was isolated by evaporation of the supernatant after 2 h at —70° C. The IR spectrum was identical with that of an authentic $PhAuPPh_3$ sample. Analysis: Found: C, 52.2; H, 4.2; P, 5.7; Au, 37. $C_{24}H_{20}AuP$ calcd.: C, 53.74; H, 3.76; P, 5.77; Au, 36.72.

Tris(p-tolyl)goldzinc. Addition of a solution of 2.3 mmol of Au(CO)Cl in ether to a solution of 5.8 mmol of p-Tol_2Zn in ether at —40° C gave a red solution and a finely divided red precipitate. After 2 days at —70° C, the solution was nearly colourless and the precipitate had settled. Washing with ether/pentane at —50° C afforded red p-Tol_3AuZn in 50% yield. Analysis: Found: C, 43.8; H, 3.9; Au, 36.5; Zn, 12.2; Cl, <0.2. $C_{21}H_{21}AuZn$ calcd.: C, 47.08, H, 3.95; Au, 36.76, Zn, 12.00; Cl, 0. From the washing solutions $TolZnCl2Et_2O$ crystallized after addition of extra pentane. An NMR spectrum at 100 MHz in CS_2 showed the aryl protons in an *AA′BB′* pattern at 7.43, 7.50, 7.82 and 7.90 ppm.

Reactions of arylgoldzinc compounds

Reaction of triphenylgoldzinc with bipy. When a solution of Ph_3AuZn (0.29 mmol) in benzene (30 ml) was treated with 0.6 mmol of bipy in benzene at 10°C, a white flocculent precipitate was obtained. After washing with benzene and drying, this was shown to be Ph_3AuZnbipy (0.3 mmol), decomposing at 140°C (1 sec), 80°C (5 min). The product is only slightly soluble in coordinating solvents such as pyridine and DMF. Analysis: Found: C, 52.0; H, 3.9; Au, 29.6; Zn, 11.3; N, 4.9. $C_{28}H_{23}AuZnN_2$ calcd.: C, 51.75; H, 3.56; Au, 30.31; Zn, 10.7, N, 4.59.

Reaction of triphenylgoldzinc with TMED. Ph_3AuZnTMED was obtained when slightly more than 1 equivalent of TMED was added to a solution of Ph_3AuZn in toluene at −20°C. After 1 h a colourless solution resulted from which a white precipitate was obtained by addition of pentane. The washed (pentane) and dried product analyzed correctly for Ph_3AuZnTMED. Decomposition: 55°C (1 sec). Analysis: Found: C, 45.5; H, 5.6; Au, 31.2; Zn, 11.3; N, 4.9. $C_{24}H_{31}AuZnN_2$ calcd.: C, 47.28; H, 5.12; Au, 32.2; Zn, 10.7; N, 4.59.

Reaction of triphenylgoldzinc with triphenylphosphine. $PhAuPPh_3$ was obtained when 0.3 mmol of Ph_3AuZn was stirred at −60°C with 3.3 mmol of PPh_3 in ether, by bubbling dry air through the opaque solution formed during 20 min. Evaporation of the clear supernatant solution yielded 0.2 mmol of $PhAuPPh_3$ as a colourless microcrystalline product after washing with pentane and recrystallization from ether. Melting point: 166° (lit [2]: 164°C). Analysis: Found: C, 53.7; H, 3.9; P, 5.9; Au, 37.2. $C_{24}H_{20}AuP$ calcd.: C, 53.74; H, 3.76; P, 5.77; Au, 36.72.

Reaction of triphenylgoldzinc with diphos. Addition of a solution of diphos (0.6 mmol) in 45 ml of diethyl ether to a solution of Ph_3AuZn (0.6 g, 1.2 mmol) in toluene (100 ml) at −60°C, caused disappearance of the yellow colour. Dry air was bubbled through at 25°C during 1 h, yielding a white precipitate. The supernatant liquid was decanted and cooled to −30°C, which yielded a white precipitate. After washing with pentane, this analyzed correctly for $(phAu)_2$diphos. The compound is poorly soluble in ether and chloroform and melts at 240°C with decomposition. Analysis: Found: C, 48.2; H, 3.9; P, 6.3; Zn, 1.5; rest (Au), 40.0. $C_{38}H_{34}P_2Au_2$ calcd.: C, 48.22; H, 3.62; P, 6.54; Zn, 0; rest (Au), 41.62.

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