

VOLATILE ORGANOGOLD COMPOUNDS $[\text{AuR}(\text{CNR}^1)]$: THEIR POTENTIAL FOR CHEMICAL VAPOUR DEPOSITION OF GOLD

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Summary

The preparation and characterization of a series of novel volatile organogold compounds of the type $[\text{AuR}(\text{CNR}^1)]$, where R = methyl, phenyl, vinyl, 1-propynyl and 3,3-dimethyl-butynyl, and R^1 = methyl, ethyl, i-propyl, t-butyl, phenyl and cyclohexyl, are described. These compounds show sharply differing thermal stabilities. As a result of their volatility, the more stable members of the series have potential as chemical vapour deposition agents, selectively yielding essentially pure gold films under conditions of high vacuum and moderate temperature. This principle is demonstrated for these and other known volatile organogold compounds.

Introduction

Gold metal has found a special place in the rapidly growing microelectronics industry as a result of its high conductivity, corrosion resistance and excellent mechanical properties. Plating by gold and other precious metals is commonly used in manufacturing electronic contacts, connectors, printed circuit boards and a variety of other electronic components. The increasing cost and scarcity of precious metals, together with a rapid rise in the number of components produced, has stimulated the development of selective high speed deposition techniques. However, poor pattern definition is often encountered in plating procedures that do not employ physical masking. The techniques most commonly employed to produce gold films are sputtering and electrolysis. Sputtering yields gold films of high purity, but requires both masking and reclamation of the gold deposited on the mask to be cost-effective. Electrolysis requires the circuit board or other plating surface to be immersed in a gold-bearing electrolyte solution and a current passed through it. Precise deposition is again a problem and masking is usually required in such cases. An improvement on the electrolysis involves the directing of an argon laser beam through a lens into a jetstream of gold-bearing electrolyte, resulting in gold deposits of good metallurgical quality without the need for masking [1].

This paper describes the use of chemical vapour deposition for preparing gold films. The method requires suitable volatile gold compounds as precursors and these should decompose on heating under vacuum to give the gold film. Laser writing technology could then, in principle, be used to prepare gold microcircuits, or wafers could be coated with gold by using conventional heat sources [2,3].

Several constraints prevail in the choice of molecules suitable for these purposes:

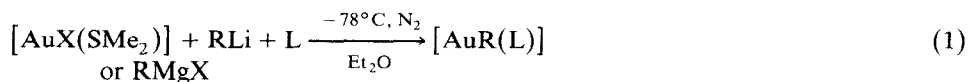
- (1) The organogold compound must be stable but at the same time volatile enough so that a sufficient vapour pressure can be obtained at moderate heating and high vacuum to ensure a high rate of plating.
- (2) The organogold compound must decompose to elemental gold well below the critical temperature for damage to the microelectronic device being plated.
- (3) The decomposition of the organogold complex should yield a pure form of elemental gold under these conditions, and the byproducts should not contain elements which could act as dopants for semiconductors or form stable compounds with gold.
- (4) Since pure gold is too soft to form robust devices, codeposition with other metals would be necessary in practice to form harder gold alloys.

Only a small number of reasonably stable yet volatile organogold compounds are known in the chemical literature. These include the trialkylphosphine compounds, $[\text{AuR}(\text{PR}_3)]$ and $[\text{AuR}_3(\text{PR}_3)]$, where R is a small hydrocarbon moiety [4–6].

Because the known derivatives contain phosphorus, which may be expected to form stable gold-phosphides and may also affect the conductivity of silicon or other semiconductors, we also set out to prepare a series of organo(isocyanide)gold(I) compounds of the formula $[\text{AuR}(\text{CNR}^1)]$. R and R^1 are again limited to small hydrocarbon units for increased volatility of the resulting organogold compound [2]. The general class of organogold(I) compounds $[\text{AuR}(\text{CNR}^1)]$, where R and R^1 are alkyl or aryl groups, is known and the use of these compounds in forming gold films by applying paints containing the compounds to substrates and subsequently heating the substrates to high temperatures to volatilize the organic fragments has been demonstrated [7–9]. However, the known examples contain substituted phenyl rings as R and R^1 in the compounds $[\text{AuR}(\text{CNR}^1)]$. As a result of these bulky substituents, the compounds would not be useful as vapour deposition vehicles.

Results and discussion

All the compounds examined as potential vapour deposition agents were prepared by the general synthetic route of equation (1). Previous methods include the two-step sequence of treating $[\text{AuX}(\text{SMe}_2)]$ with a ligand L to give $[\text{AuXL}]$ followed by reaction with organolithium or Grignard reagent to give $[\text{AuRL}]$. The two steps are combined in the method of eq. 1 [4–6,10].



The preparations involve suspension of $[\text{AuX}(\text{SMe}_2)]$, X = Cl or Br, in dry diethyl ether in a dinitrogen atmosphere, addition of an excess of the appropriate organolithium or Grignard reagent at -78°C , followed by addition of the organo-isocyanide or organophosphorus ligand. The gold(I) products are isolated as white

TABLE 1
SPECTROSCOPIC DATA FOR GOLD(I) COMPOUNDS

Complex	^1H NMR (δ (ppm), J (Hz))	IR ($\nu(\text{C}\equiv\text{N})$ (cm^{-1}))	Mass spectrum (m/e^+)
$\text{H}_3\text{CAuCNCH}_3$	0.295 [s, 3H, AuCH_3] 3.210 [t, $^2J(\text{HN})$ 1.9, 3H, AuCNCCH_3] ^a	2240 ^d	253,238,197,41
$\text{H}_3\text{CAuCNCH}_2\text{CH}_3$	0.250 [s, 3H, AuCH_3] 1.403 [m, 3H, CNCH_2CH_3] 3.478 [m, 2H, CNCH_2CH_3] ^b	2200 ^d	267,252,224,197,55
$\text{H}_3\text{CAuCNCH}(\text{CH}_3)_2$	0.297 [s, 3H, AuCH_3] 1.435 [dt, $^3J(\text{HH})$ 6.6, $^3J(\text{HN})$ 2.5, 6H, - $\text{CNCH}(\text{CH}_3)_2$] 3.851 [st, $^3J(\text{HH})$ 6.6, $^2J(\text{HN})$ 1.9, 1H, - $\text{CNCH}(\text{CH}_3)_2$] ^a	2200 ^d	281,266,224,69,43
$\text{H}_3\text{CAuCNC}(\text{CH}_3)_3$	0.302 [s, 3H, AuCH_3] 1.492 [t, $^3J(\text{HN})$ 2.2, 9H, $\text{CNC}(\text{CH}_3)_3$] ^b	2200 ^d	295,280,253,239,224,69
$\text{H}_3\text{CAuCNC}_6\text{H}_5$	0.343 [s, 3H, AuCH_3] 7.666 [m, 5H, CNC_6H_5] ^c	2200 ^d	315,300,103
$\text{H}_3\text{CAuCNC}_6\text{H}_{11}$	0.211 [s, 3H, AuCH_3] 1.426 [m, 4H, $\text{AuCNCCH}_2\text{CH}_2$] 1.741 [m, 4H, $\text{AuCNCCHCH}_2\text{CH}_2$] 1.919 [m, 2H, $\text{AuCNCCHCH}_2\text{CH}_2\text{CH}_2$] 3.732 [m, 1H, AuCNCCH] ^c	2220 ^d	321,306,224,197,109
$\text{H}_5\text{C}_6\text{AuCNCH}_3$	3.267 [t, $^2J(\text{HN})$ 2.5, 3H, AuCNCCH_3] 7.327 [m, 5H, AuC_6H_5] ^c	2210 ^d	306,260,230,154,77,41
$\text{CH}_2=\text{CHAuCNCH}_3$	3.579 [t, not resolved, 3H, AuCNCCH_3] 5.600 [dd, $^3J(\text{HH})_{\text{trans}}$ 20.7, $^2J(\text{HH})_{\text{gem}}$ 5.0, 1H, $\text{AuCH}=\text{CHH}$] 6.079 [dd, $^3J(\text{HH})_{\text{cis}}$ 14.2, $\text{AuCH}=\text{CHH}$] 7.146 [dd, 1H, $\text{AuCH}=\text{CH}_2$] ^b		
$\text{CH}_2\text{CHAuCNC}(\text{CH}_3)_3$	1.445 [t, not resolved, 9H, $\text{AuCNC}(\text{CH}_3)_3$] 5.214 [dd, $^3J(\text{HH})_{\text{trans}}$ 20.7, $^2J(\text{HH})_{\text{gem}}$ = 5.1, 1H, $\text{AuCH}=\text{CHH}$] 5.683 [dd, $^3J(\text{HH})_{\text{cis}}$ 14.2, 1H, $\text{AuCH}=\text{CHH}$] 6.773 [dd, 1H, $\text{AuCH}=\text{CHH}$] ^b		
$\text{H}_3\text{CC}\equiv\text{CAuCNCH}_3$	1.751 [s, 3H, $\text{AuC}\equiv\text{CCCH}_3$] 3.544 [t, $^2J(\text{HN})$ 2.7, 3H, AuCNCCH_3] ^c	2257 ^e	277,238,236 197,41
$(\text{CH}_3)_3\text{CC}\equiv\text{CAuCNCH}_3$	1.179 [s, 9H, $\text{AuC}\equiv\text{CC}(\text{CH}_3)_3$] 3.337 [t, $^2J(\text{HN})$ 2.5, 3H, AuCNCCH_3] ^c	2259 ^e	319,304,238
$\text{H}_3\text{CC}\equiv\text{CAuP}(\text{CH}_3)_3$	1.485 [d, $^2J(\text{PH})$ 10.2, 9H, $\text{AuP}(\text{CH}_3)_3$] 1.829 [s, 3H, $\text{AuC}\equiv\text{CCCH}_3$] ^c		312,273,76

^a CDCl_3 at r.t. ^b CDCl_3 at -20°C . ^c CD_2Cl_2 at r.t. ^d Nujol mull on NaCl plates. ^e CH_2Cl_2 solution.

TABLE 2
ELEMENTAL ANALYSES FOR GOLD(I) COMPOUNDS

Complex	Analysis: Found (calc) (%)		
	C	H	N
$\text{H}_3\text{CC}\equiv\text{CAuCNCH}_3$	21.5(21.7)	2.4(2.2)	5.4(5.1)
$(\text{CH}_3)_3\text{CC}\equiv\text{CAuCNCH}_3$	29.9(30.1)	3.8(3.8)	4.1(4.4)
$\text{H}_3\text{CC}\equiv\text{CAuP}(\text{CH}_3)_3$	23.3(23.1)	4.3(3.9)	

or pale yellow solids from the organic layers after hydrolysis, and can be recrystallized from hydrocarbon solvents such as acetone, diethyl ether or dichloromethane.

The products were analyzed by the usual spectroscopic methods, and ^1H NMR and mass spectroscopy proved the most useful characterization tools (Table 1). For the least stable compounds, the spectra were recorded at low temperatures. A characteristic feature of the proton NMR spectra is well-resolved H–N coupling within the isocyanide ligands. Parent ion signals were observed in almost all of the mass spectra recorded. The $\text{C}\equiv\text{N}$ infrared stretching frequencies of the isocyanide ligands are listed for those compounds of which the infrared spectra could be recorded at room temperature. Even the more stable members of the class of compounds deteriorated when stored at room temperature and, as a result, satisfactory elemental analyses could not be obtained. The novel alkynylgold(I) compounds proved much more stable than their alkylgold(I) counterparts, and elemental analyses were obtained, see Table 2.

Methyl(methyl isocyanide)gold(I) was one of the most stable members of this class of compounds, and, as expected, one of the most volatile. It melts with decomposition at 95°C , can be sublimed under vacuum and can be stored for extended periods at low temperature.

A surprising observation is that the corresponding gold(III) complex, trimethyl(methyl isocyanide)gold(III), could not be prepared using the commonly employed reagent mixture, MeLi and MeI with $[\text{AuCl}(\text{SMe}_2)]$ and MeNC [11]. Under these conditions only the monomethylgold(I) product is isolated. The simple oxidative addition of MeI to the gold(I) centre could also not be effected at room temperature over several days. Clearly, the isocyanide ligand favors gold(I) rather than gold(III) compared to phosphine ligands.

Methyl(ethyl isocyanide)gold(I) is considerably less stable than the analogous methylgold(I) complex, and the solid begins decomposing at around 0°C . However, a parent ion signal is observed in the mass spectrum at $m/e^+ 267$ when the sample probe of the mass spectrometer is gradually warmed from -20°C . The ^1H NMR spectrum, recorded at -20°C , confirms the above formulation.

Methyl(*i*-propyl isocyanide)gold(I) melts at 30°C , and decomposes below 40°C . The ^1H NMR spectrum is shown in Fig. 1. The six proton resonance of the two methyl groups in the isocyanide ligand appears at δ 1.43 ppm, as a doublet of triplets with $^3J(\text{HN})$ 2.5 Hz. The single Me_2CH proton of the isopropyl group resonates as a septet of triplets at δ 3.85 ppm, with $^2J(\text{HN})$ 1.9 Hz.

Methyl(*t*-butyl isocyanide)gold(I) melts with decomposition at 62°C . The nine equivalent methyl protons of the isocyanide ligand appear as the expected 1:1:1 triplet, $^3J(\text{HN})$ 2.2 Hz, in the ^1H NMR spectrum, as a result of coupling to nitrogen.

Methyl(phenyl isocyanide)gold(I) (m.p. 102°C with decomposition), and methyl(cyclohexyl isocyanide)gold(I) (m.p. 88°C with decomposition), were also easily characterized by their spectroscopic data (Table 1).

The reaction of $[\text{AuX}(\text{SMe}_2)]$ with phenyllithium in the presence of excess methylisocyanide yielded a mixture of phenyl(methyl isocyanide)gold(I) and polymeric phenylgold.

Vinyl(methyl isocyanide)gold(I) is thermally very unstable and could only be characterized by low temperature ^1H NMR spectroscopy (Fig. 2). The triplet resulting from the methyl protons of the isocyanide ligand, coupled to nitrogen, is not well-resolved at this temperature. Treatment of vinyl(methyl isocyanide)gold(I)

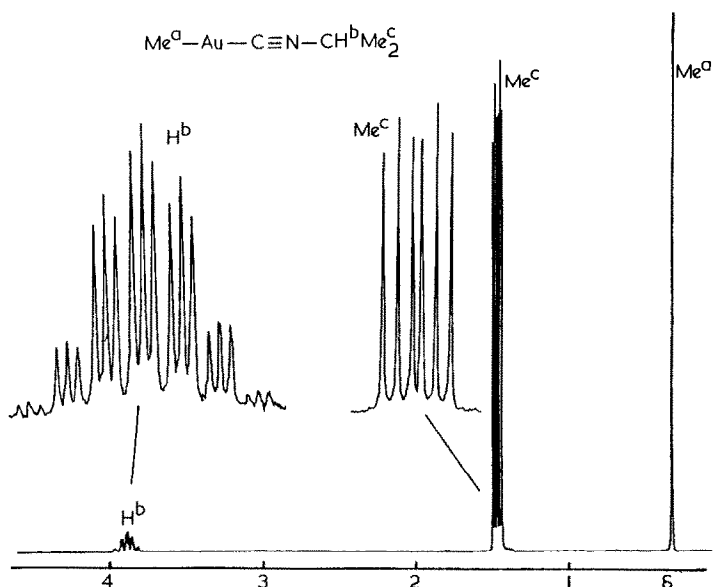


Fig. 1. ^1H NMR spectrum (200 MHz) of $[\text{AuMe}(\text{i-PrNC})]$.

with *t*-butyl isocyanide at low temperature resulted in quantitative conversion to vinyl(*t*-butyl isocyanide)gold(I). This compound has equally low thermal stability.

The alkynylgold(I) compounds, 1-propynyl(methyl isocyanide)gold(I), 3,3-dimethyl-1-butynyl(methyl isocyanide)gold(I) and 1-propynyl(trimethylphosphine)-gold(I) are markedly more stable, with melting/decomposition points at 140°C (dec), $180\text{--}181^\circ\text{C}$ (dec) and 100°C (dec) respectively. The $\nu(\text{C}\equiv\text{C})$ bands in the infrared spectra were observed as weak bands in the region $2200\text{--}1900\text{ cm}^{-1}$.

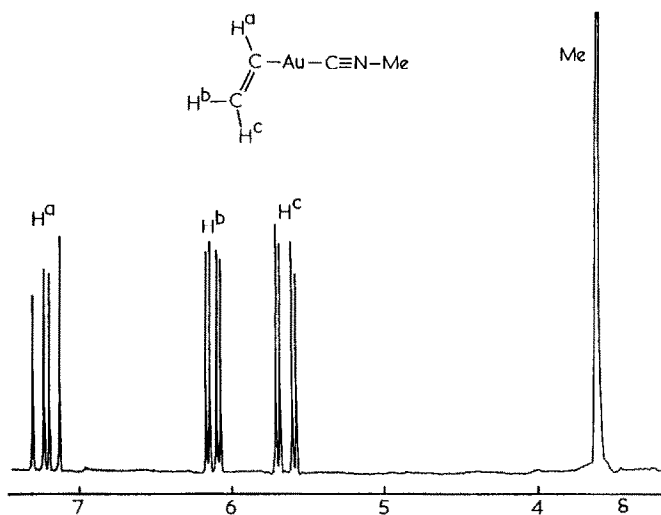


Fig. 2. ^1H NMR of $[\text{Au}(\text{CH}=\text{CH}_2)(\text{MeNC})]$ at -40°C .

Vapour deposition studies

The potential of the more stable organo(isocyanide)gold(I) derivatives, as well as other known volatile organo-gold(I) and -gold(III) compounds for producing gold films, was investigated. This was achieved by vaporizing the organogold compound under high vacuum and contacting the vapour with a target surface heated to at least the decomposition temperature of the organogold compound.

In a typical simple experiment [AuMe(CNMe)] (m.p. 95°C (dec)) was vaporized from a round-bottomed flask under dynamic vacuum (4×10^{-3} mbar), by gently warming the organogold compound to 30°C. The vapour of the organogold compound was passed along a glass tube containing a glass disk. The tube was heated by a furnace to 200°C (i.e. above the decomposition temperature of the organogold compound). Decomposition of the organogold compound occurred upon passage of the vapour through the glass tube, resulting in the deposition of gold metal onto the heated target disk and walls. After termination of the experiment the deposited gold film was peeled from the glass surface of the target disk, and the composition of the film was analyzed by X-ray photoelectron spectroscopy (XPS). The results for the deposition using [AuMe(CNMe)] are included in Table 3.

This example illustrated that any heat source capable of temperatures higher than the decomposition temperature of the organogold compound, can be used in the preparation of gold films from volatile organogold compounds. Similarly, selective plating of the target disk can be achieved by selective heating of the target disk only. Any solid material capable of withstanding temperatures of 150°C or greater can be

TABLE 3

X-RAY PHOTOELECTRON SPECTROSCOPY ANALYSIS; SURFACE COMPOSITION TABLE

Compound	Element	Atom(%)
Sputtered gold standard	Au 4f	64.19
	C 1s	22.42
	O 1s	11.42
[AuCH ₃ (P(CH ₃) ₃)] - After bombarding with Ar ions for 3 min	Au 4f	71.22
	C 1s	28.78
	Au 4f	95.50
	C 1s	3.80
	O 1s	0.69
[Au(CH ₃) ₃ (P(CH ₃) ₃)] - After bombarding with Ar ions for 3 min	Au 4f	65.61
	C 1s	32.81
	O 1s	1.59
	Au 4f	95.32
	C 1s	4.68
[AuCH ₃ (CNCH ₃)]	Au 4f	52.93
	C 1s	36.81
	O 1s	5.25
	N 1s	5.01
[AuC≡CC(CH ₃) ₃ (P(CH ₃) ₃)]	Au 4f	37.64
	C 1s	59.70
	O 1s	2.66

plated in this way, including semiconductor materials such as silicon or gallium arsenide. Substitution of the glass target disk by a silicon wafer resulted in selective coating of the wafer prior to the surrounding glass walls. The purity of the gold films generated in this way was again confirmed by ESCA.

For the purpose of gold deposition from the vapour phase, the more volatile organogold compounds are preferred. They yield a higher vapour pressure at moderate temperature and high vacuum, and thus a high rate of plating occurs, once decomposition is induced. Generally, volatility increases as the size of the organic ligands decreases. As expected, compounds such as $[\text{AuMe}(\text{CNMe})]$, $[\text{AuMe}_3(\text{PMe}_3)]$ and $[\text{AuMe}(\text{PMe}_3)]$ proved very efficient vapour deposition vehicles (Table 3), generating strong gold films within a very short period of time.

If increased size of the organic ligands is accompanied by markedly increased thermal stability of the organogold compound, the materials also have good potential as deposition vehicles. In such cases, the organogold compound can be heated to a higher temperature prior to decomposition, and this compensates for the decreased volatility resulting from bulkier ligands. In the general class of alkyl(isocyanide)gold(I) compounds $[\text{AuR}(\text{L})]$, thermal stability does not increase significantly with increase in the sizes of R and/or L; it does, however, increase for the alkynylgold(I) compounds, and $[\text{Au}(\text{C}\equiv\text{CMe})(\text{CNMe})]$, $[\text{Au}(\text{C}\equiv\text{CBu}^t)(\text{CNMe})]$, $[\text{Au}(\text{C}\equiv\text{CMe})(\text{PMe}_3)]$ and $[\text{Au}(\text{C}\equiv\text{CBu}^t)(\text{PMe}_3)]$ [4] which proved to be useful gold deposition vehicles (Table 3).

The thickness of the gold films formed as a result of vapour deposition can be varied and depends obviously on the deposition time allowed, and on the vapour pressure achieved by the specific organogold compound under the experimental conditions employed. In all of the examples listed in Table 3 even the very thin films proved robust enough to be cut manually. The films do not adhere strongly to glass but do adhere well to silicon or gallium arsenide wafers.

In order to evaluate composition analyses of the gold films obtained from the volatile organogold compounds, data for a sputtered gold standard is also included in Table 3. The exposure of pure gold films to the atmosphere results in immediate coating of the films with carbon and oxygen impurities. The gold films can be cleaned by bombarding the surfaces with argon ion beams, resulting in a drastic drop in the percentage impurities adsorbed on the surface. This is illustrated in Table 3 for the films prepared using $[\text{AuMe}(\text{PMe}_3)]$ and $[\text{AuMe}_3(\text{PMe}_3)]$. The residual carbon is simply a function of the vacuum limits of the XPS system. The results in Table 3 clearly indicate the very high metallurgical purity of the gold films prepared by vapour deposition of volatile organogold compounds.

Experimental procedures

The reactions were carried out using standard Schlenk Tube techniques. Diethyl ether was dried by refluxing over LiAlH_4 for 3 h under dinitrogen and then distilled. ^1H NMR spectra were recorded on a Varian XL200 spectrometer. Typical examples of syntheses are given below.

Methyl(methyl isocyanide)gold(I)

$[\text{AuBr}(\text{SMe}_2)]$ (1.0 g, 2.95 mmol) was suspended in dry diethyl ether (100 ml) under dinitrogen atmosphere. The mixture was stirred rapidly and cooled to

-78°C . A solution of MeLi (1.6 M) in ether (5.0 ml, 9.0 mmol) was added dropwise, followed by methyl isocyanide (328 mg, 8.0 mmol). The reaction mixture was allowed to warm gradually to room temperature over 1 h. The excess MeLi was hydrolyzed at 0°C by slow addition of saturated ammonium chloride solution (~ 15 ml). The organic layer was separated, dried over magnesium sulfate and filtered free of residual solids. The volatile components of the reaction mixture were removed under reduced pressure at 0°C , to give the product as a white solid in 74% yield.

Vinyl(methyl isocyanide)gold(I)

[AuBr(SMe₂)] (1.038 g, 3.06 mmol) was suspended in dry diethyl ether (100 ml) under dinitrogen at -78°C . A solution of vinylmagnesium bromide in tetrahydrofuran (5.0 ml, 1.2 M, 6.00 mmol) was added dropwise, followed by methyl isocyanide (287 mg, 7.00 mmol). The reaction mixture was allowed to warm to -5°C , and stirred for 1 h. Some decomposition to metallic gold was observed. The excess Grignard reagent was hydrolyzed at -5°C by slow addition of saturated ammonium chloride solution (~ 15 ml). The organic layer was decanted onto magnesium sulfate, dried and filtered free of residual solids while maintaining the solution at -5°C . The pale yellow solution was concentrated to 10 ml (at -5°C), cold pentane (10 ml) was added, and the solution was cooled to -78°C . White crystals deposited over 24 h, and these were isolated at -78°C , washed with cold pentane and dried in vacuo. Estimated yield 20%. An NMR sample was prepared and the spectrum recorded at -20°C . Rapid decomposition took place when the sample was allowed to warm to room temperature.

3,3-Dimethyl-1-butyne(methyl isocyanide)gold(I)

[AuBr(SMe₂)] (1.0 g, 2.95 mmol) was suspended in dry diethyl ether (100 ml) under dinitrogen. Meanwhile, 3,3-dimethyl-1-butyne (1.0 ml, 8.12 mmol) was added at 0°C to a solution of MeLi (15 ml, 7.5 mmol) in diethyl ether. The latter mixture was warmed to room temperature, and after 15 min it was added dropwise to the suspension containing the organogold complex at -78°C . The reaction mixture was allowed to warm to room temperature over 1 h. The excess organolithium reagent was hydrolyzed at 0°C by slow addition of saturated ammonium chloride solution (15 ml). The organic layer was separated and a second extraction of product with diethyl ether (~ 100 ml) was performed. The combined ether solutions were dried over magnesium sulfate and filtered free of residual solids. The volatile components were removed under reduced pressure at 0°C , giving the product as a white crystalline solid in 79% yield.

1-Propyne(trimethylphosphine)gold(I)

1-Propyne gas was bubbled through a solution of MeLi (6.8 mmol) in dry diethyl ether (15 ml) at 0°C under dinitrogen. A white suspension of CH₃C $\equiv$ CLi formed. This suspension was transferred dropwise into a reaction mixture containing [AuCl(SMe₂)] (1.0 g, 3.39 mmol) and PMe₃ (532 mg, 7.0 mmol) at -78°C in dry diethyl ether (~ 100 ml). The reaction mixture was allowed to warm to room temperature over 1 h. The excess organolithium reagent was hydrolyzed at 0°C by slow addition of saturated ammonium chloride solution (~ 15 ml). The diethyl ether layer was separated, and the product was further extracted by adding CH₂Cl₂ (~ 150 ml) to the remaining water layer. The combined organic extracts were dried

over MgSO_4 , and filtered free of residual solids. The volatile components were removed under reduced pressure, and the resulting solid was recrystallized from CH_2Cl_2 . The white product was obtained in 84% yield.

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