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SYNTHESIS OF MIXED DIARYLGOLD(III) COMPLEXES. CRYSTAL STRUCTURE OF *cis*-[2-(PHENYLAZO)PHENYL][2-{(DIMETHYLAMINO)METHYL}PHENYL]GOLD(III) TETRACHLOROAURATE *

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Summary

The reaction (1 : 1) between $[A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)Cl_2]$ and $[Hg(2\text{-}C_6H_4\text{-}N{=}NPh)_2]$ gives the complex $[(2\text{-}PhN{=}NC_6H_4)A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)Cl]$, which on treatment with $AgClO_4$ gives $[(2\text{-}Ph\overline{N{=}NC_6H_4)A}\overline{u(2\text{-}C_6H_4CH_2N}Me_2)]ClO_4$. From this complex, the species $[(2\text{-}PhN{=}NC_6H_4)A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)X]$ (X = CH_3COO, CN), or $[(2\text{-}PhN{=}NC_6H_4)A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)L]$ (L = PPh_3, py) are obtained by reaction with the corresponding KX salts or neutral ligands.

The crystal structure of $[(2\text{-}Ph\overline{N{=}NC_6H_4)A}\overline{u(2\text{-}C_6H_4CH_2N}Me_2)][AuCl_4]$ (obtained by metathesis between the corresponding perchlorate and tetramethylammonium salts) has been determined; the cation displays square-planar coordination with two *cis*-nitrogen (Au–N: 2.166(15), 2.140(14) Å) and two *cis*-carbon (Au–C: 2.021(16), 2.033(17) Å) atoms bonded to the gold atom.

Introduction

Novel gold complexes can be prepared using organomercury compounds; we reported such 'one-pot' syntheses of $[Au(R)Cl]^-$ (R = *o*-, *m*-, *p*-$C_6H_4NO_2$) [1], $[Au(R)_2Cl_2]^-$ (R = *o*-$C_6H_4NO_2$, 2-CH_3,6-$O_2NC_6H_3$) [2], $[A\overline{u(2\text{-}C_6H_4N{=}N}Ph)Cl_2]$ [3], $[A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)Cl_2]$ [4] or $[(2\text{-}PhN{=}NC_6H_4)A\overline{u(2\text{-}C_6H_4N{=}N}Ph)Cl]$ [5] from $[AuCl_2]^-$, $[AuCl_4]^-$ or $[AuCl_3(tht)]$ (tht = tetrahydrothiophene). The result of

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* Dedicated to Prof. Rafael Usón on the occasion of his 60th birthday.

these transmetallations depends mainly on the nature of the transferred organic group. Thus, using [Hg(o-$C_6H_4NO_2$)$_2$] we were only able to prepare diarylgold(III) [2] or platinum(II) [6] complexes, whereas [Hg(2-C_6H_4N=NPh)$_2$] reacts with [$AuCl_4$]$^-$ (2/1) to give first [Au(2-C_6H_4N=NPh)Cl_2] and then [(2-PhN=N-C_6H_4)Au(2-C_6H_4N=NPh)Cl] [5]. Since this is a two-step process, it should be possible to synthesize mixed diarylgold(III) complexes, until now only represented by *trans*-[Au(C_6F_5)(2,4,6-$C_6F_3H_2$)X_2]$^-$ (X = Br, I) [7].

Results and discussion

Synthesis and reactivity

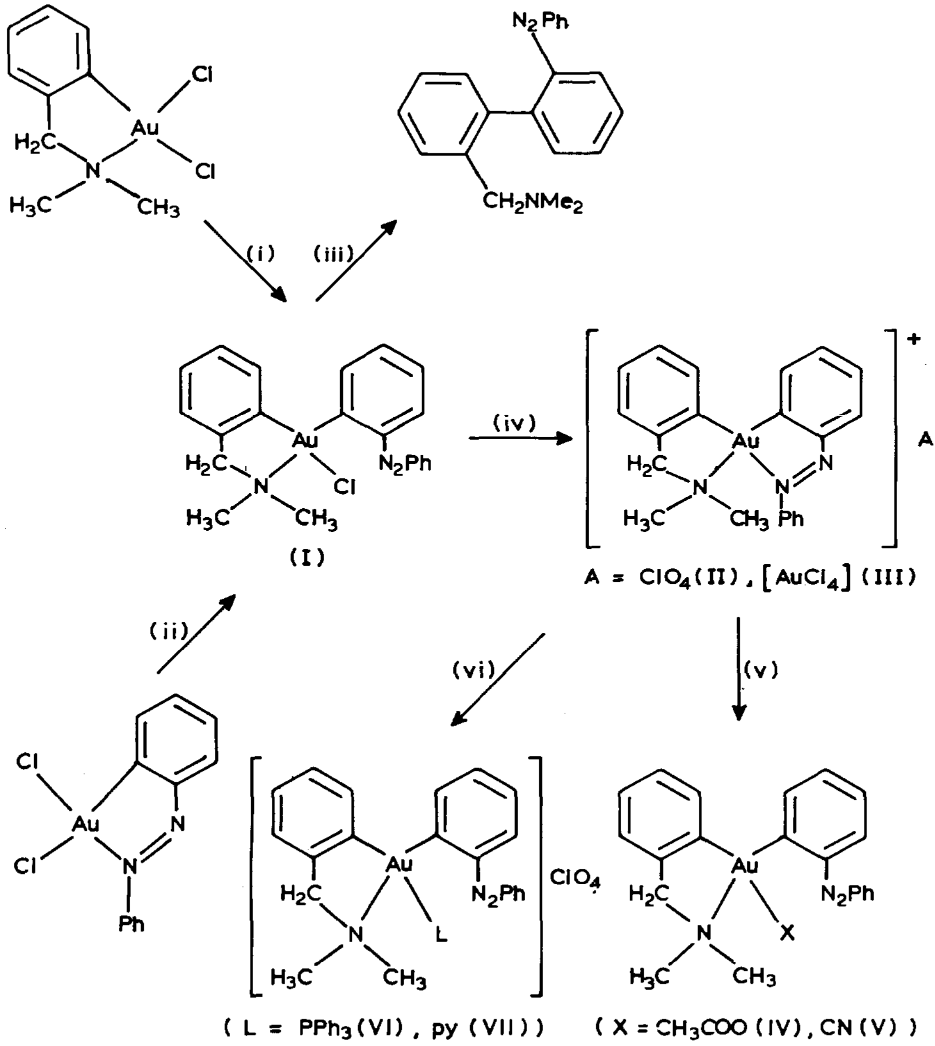
The mixed diarylgold(III) complex [(2-PhN=NC_6H_4)Au(2-$C_6H_4CH_2NMe_2$)Cl] (I) can be obtained, along with metallic gold, by the reaction of [Au(2-C_6H_4N=NPh)Cl_2] with [Hg(2-$C_6H_4CH_2NMe_2$)$_2$] (molar ratio 1/1 in diethyl ether at room temperature, 3 h, 40% yield). Under the same conditions [Au(2-$C_6H_4CH_2$-NMe_2)Cl_2] does not react with [Hg(2-C_6H_4N=NPh)$_2$], but in acetone a slow reaction (requiring 6 h) does occur, to give I in substantially better yield (85%) because no metallic gold is formed.

Complex I reacts with PPh_3 to give, through a reductive elimination step, [AuCl(PPh_3)]. Similar behaviour is observed for [(2-PhN=NC_6H_4)Au(2-C_6H_4-N=NPh)Cl] [5]. It is possible to extract from the mother liquor with n-hexane, a product formulated as $C_6H_4(CH_2NMe_2)C_6H_4(N_2Ph)$ from GC/MS evidence (*m/z*: 315 (11%) M^+, 271 (100%) $(M - NMe_2)^+$). It is likely that this biphenyl compound is a 2,2′-derivative resulting from an intramolecular coupling (see Scheme 1).

The reaction between I and $AgClO_4$ gives the complex [(2-PhN=NC_6H_4)Au(2-$C_6H_4CH_2NMe_2$)]ClO_4 (II). When II is treated with $Me_4N[AuCl_4]$ the complex [(2-PhN=NC_6H_4)Au(2-$C_6H_4CH_2NMe_2$)][$AuCl_4$] (III) can be isolated. II reacts with KX salts (X = Br, I, CH_3COO or CN) to give complexes [(2-PhN=NC_6H_4)Au(2-$C_6H_4CH_2NMe_2$)X]. However, these could be isolated analytically pure only for X = CH_3COO (IV) or CN (V). Complexes IV and V can also be obtained by treating I with the corresponding KX salts. Gold analyses of complex V were always lower than expected; when heated, the aqueous hydrazine suspensions give off yellow fumes and it is possible that V partially vaporizes before it decomposes; it has a very low melting point (75 °C).

Complex II reacts with PPh_3 (1/1) or py (excess) to give [(2-PhN=NC_6H_4)Au(2-$C_6H_4CH_2NMe_2$)L]ClO_4 (L = PPh_3 (VI), py (VII)) but with $AsPh_3$ (1/1) it gives [Au($AsPh_3$)$_4$]ClO_4 and unchanged II, and probably also the above-mentioned 2,2′-biphenyl derivative. The reaction between [Au(2-C_6H_4N=NPh)$_2$]ClO_4 and pyridine or PPh_3 (1/1) does not give pure [(2-PhN=NC_6H_4)Au(2-C_6H_4-N=NPh)(L)]ClO_4 complexes [5], probably because reductive coupling occurs more easily. However the pyridine complex was prepared by treating the acetato complex with [pyH]ClO_4. No reaction was observed between IV and [pyH]ClO_4 or between IV and 8-hydroxyquinoline.

The reaction between II and 1,10-phenanthroline (1/1) results in immediate precipitation of metallic gold. However, 2,2′-bipyridine reacts (1/1) to give a yellow solid that, from its IR spectrum, molar conductivity (131 Ω^{-1} cm^2 mol^{-1}) and reaction with KCN to give V, can be formulated as [(2-PhN=NC_6H_4)Au(2-



SCHEME 1. (i) + [Hg(2-$C_6H_4N{=}NC_6H_5$)], −[(Hg(2-$C_6H_4N{=}NC_6H_5$)Cl]. (ii) + [(Hg(2-$C_6H_4CH_2NMe_2$)$_2$], −[(Hg(2-$C_6H_4CH_2NMe_2$)Cl]. (iii) + PPh_3, −[AuCl(PPh_3)]. (iv) + $AgClO_4$, −AgCl (A = ClO_4); + $AgClO_4$, −AgCl, + $Me_4N[AuCl_4]$, −(Me_4N)ClO_4 (A = [$AuCl_4$]). (v) + KX, −$KClO_4$. (vi) + L.

$C_6H_4CH_2NMe_2$)(bipy)]ClO_4. Its instability in solution prevented us from obtaining an analytically pure sample.

Table 1 gives the analytical results and other data for complexes I–VII.

Structure of the complexes

The structure of complex III was determined by X-ray diffraction and is shown in Fig. 1. The gold atom displays *cis* square-planar coordination. The 'bite' of the ligands (mean 79.0°) and the Au–C bond lengths (2.021(16), 2.033(17) Å) are normal [5,8–10]. The Au–N bond lengths (2.140(14), 2.166(15) Å) are similar to

TABLE 1

ANALYTICAL AND OTHER DATA FOR COMPLEXES I–VII

Complex [a]	M.p. (°C)	Λ_M [b]	Analytical data (Found (calcd.) (%))				Yield (%)
			C	H	N	Au	
[(Az)Au(Bz)Cl] (I)	90 (dec.)	2	46.18 (46.04)	4.33 (3.86)	7.30 (7.67)	35.67 (35.95)	86
[(Az)Au(Bz)ClO$_4$ (II)	200 (dec.)	129	41.39 (41.22)	3.39 (3.46)	6.40 (6.67)	32.18 (32.19)	96
[(Az)Au(Bz)][AuCl$_4$] (III)	185 (dec.)	118	29.36 (29.63)	2.33 (2.49)	4.76 (4.94)	46.89 (46.28)	93
[(Az)Au(Bz)(OOCCH$_3$)] (IV)	160 (dec.)	0	47.71 (48.35)	3.96 (4.23)	7.39 (7.35)	33.75 (34.47)	42
[(Az)Au(Bz)CN)] (V)	75	1	49.13 (49.08)	3.73 (3.93)	10.21 (10.41)	[c]	50
[(Az)Au(Bz)(PPh$_3$)]ClO$_4$ (VI)	120	92	53.02 (53.59)	4.19 (4.15)	5.07 (4.81)	23.01 (22.53)	80
[(Az)Au(Bz)(py)]ClO$_4$ (VII)	185 (dec.)	104	44.87 (45.20)	3.59 (3.79)	7.81 (8.11)	28.50 (28.51)	72

[a] Az = 2-PhN=NC$_6$H$_4$; $\overline{\text{Az}}$ = 2-PhN=$\overline{\text{NC}_6\text{H}_4}$; $\overline{\text{Bz}}$ = $\overline{\text{2-C}_6\text{H}_4\text{CH}_2\text{N}}Me_2$. [b] In ~$10^{-4}$ *M* solution in acetone. (Ω^{-1} cm^2 mol^{-1}). [c] See text.

those in *cis*-[Au(2-C$_6$H$_4$N=NPh)$_2$ClO$_4$ (2.17(2) Å mean) and [Au(2-C$_6$H$_4$-CH$_2$NMe$_2$)(PPh$_3$)(phen)](ClO)$_4$)$_2$ (2.151(12) Å) [8] but longer than those in [Au(2-C$_6$H$_4$CH$_2$NMe$_2$)(py)$_2$]$^{2+}$ [9] (2.069(7) Å), [(8-NC$_9$H$_6$O)Au(2-C$_6$H$_4$CH$_2$NMe$_2$)]$^+$ (8-NC$_9$H$_6$O = 8-hydroxyquinolinato; 2.054(6) Å) or in [(2-SC$_6$H$_4$NH$_2$)Au(2-

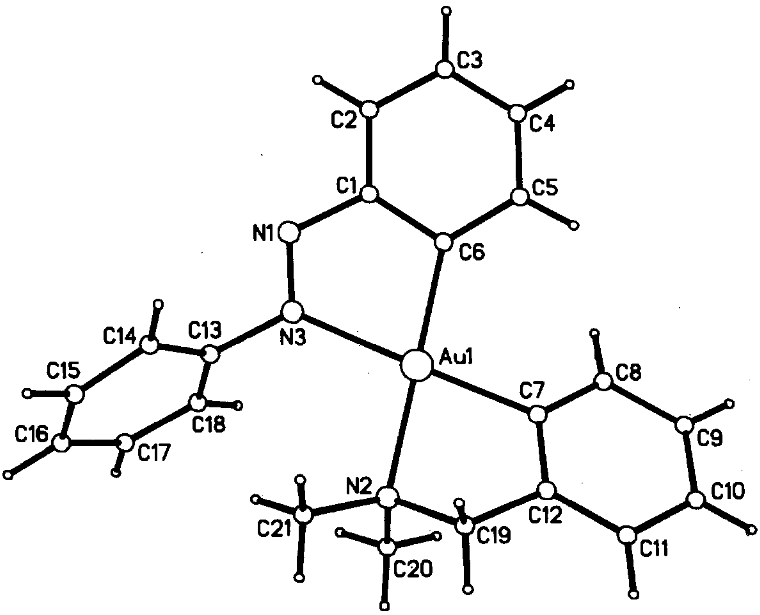


Fig. 1. The cation of III, showing the atom numbering scheme. Radii arbitrary.

$\overline{C_6H_4CH_2N}Me_2)]^+$ [8] (2.118(4) Å). These data give the following order of *trans*-influence: $8\text{-}NC_9H_6O < py < 2\text{-}SC_6H_4NH_2 < 2\text{-}C_6H_4N_2Ph \cong PPh_3$.

The coordinated moiety of the 2-(phenylazo)phenyl ligand (C(1)–C(6), N(1), N(3)) also lies in the ligand plane (r.m.s. deviation of 9 atoms for the least-squares plane = 0.02 Å), the free phenyl ring being twisted 70° around the C–N bond. The aromatic ring of the other ligand, however, makes an angle of 30° with the ligand plane.

Neutral chelated 2-(phenylazo)phenylgold(III) complexes react with neutral and anionic ligands with cleavage of the N–Au bond, whereas chelated 2-{(dimethylamino)methyl}phenylgold(III) complexes react with the same ligands to give substitution products while maintaining the chelated structure. Since III is the first gold(III) complex containing both ligands, its crystal structure gave us the opportunity of relating the different N–Au reactivities (or inertness) with structural parameters. However, the difference between the N–Au distances is scarcely significant and is anyway in the opposite sense to that which might be expected. The phenylazo chelate ring appears to be more strained than the {(dimethylamino)methyl}phenyl chelate ring (the bond angles deviate more from idealized values), which may account for its greater lability, although steric factors could also account for the relative inertness of the latter.

Complex I shows a band at 300 m cm^{-1} that may be assigned to ν(Au–Cl) *trans* to a phenyl group. If the chloro ligand were *trans* to any of the nitrogen donor atoms, ν(Au–Cl) would be observed at 350–370 cm^{-1} [3–5]. The *cis*-geometry of I is in accordance with that of III, $[\overline{Au(2\text{-}C_6H_4N{=}N}Ph)_2]^+$ [5] and most diarylgold(III) complexes [11]. Although pentacoordination in I cannot be ruled out with certainty, we believe that the 2-(phenylazo)phenyl ligand is monocoordinated (see Scheme 1) because (i) gold (III) shows a great tendency to form tetracoordinated complexes; (ii) ν(AuCl) is sensitive to an Au–N axial interaction even if it is very weak; thus in $[\overline{Au\{C(Ph){=}C(Ph)C(Ph){=}C}(Ph)\}Cl(py)]$ the ν(AuCl) appears at 300 cm^{-1} whereas in the very distorted five-coordinated $[\overline{Au\{C(Ph){=}C(Ph)C(Ph){=}C}(Ph)\}Cl(phen)]$, which contains a weak axial Au–N bond, ν(AuCl) appears at 280 cm^{-1} [12]. For I ν(AuCl) has the same frequency as ν(AuCl) *trans* to the phenyl group in $[\overline{Au(2\text{-}C_6H_4CH_2N}Me_2)Cl_2]$ [4]; (iii) the chemical reactivity of the neutral 2-(phenylazo)phenylgold(III) complexes has been interpreted on the assumption that the first process is always the cleavage of the Au–N bond [3–5]; and (iv), several 2-(phenylazo)phenyl complexes have been formulated in which this ligand is monocoordinated [13], and the crystal structure of *trans*-$[Pd(2\text{-}C_6H_4N_2Ph)Cl(PEt_3)_2]$ shows this type of coordination [14].

As shown in Scheme 1, both processes leading to I involve replacement of the chloro ligand *trans* to each nitrogen atom. However, an additional isomerization process is required when the starting material is $[\overline{Au(2\text{-}C_6H_4N{=}N}Ph)Cl_2]$. It is probable that the observed decomposition to metallic gold is connected with the isomerization.

The IR spectrum of IV shows the $\nu_{asym}(CO_2)$ and $\nu_{sym}(CO_2)$ at 1635s and 1300s cm^{-1}. The position of these bands [15], their wavenumber difference [16] and the above comments allow us tentatively to assign to the acetato ligand the same position as the chloro ligand in I. The ν(CN) band is observed in V at 2160w cm^{-1}.

The molar conductivities of complexes I–VII (see Table 1) are in accordance with the structures given in Scheme 1.

Experimental

Recording of infrared spectra, the C, H, N and Au analyses, conductance measurements and melting point determinations were performed as described elsewhere [15]. The GC/MS spectrum was obtained with a Hewlett Packard 5995 system. Reactions were carried out at room temperature with magnetic stirring and without special precautions to exclude moisture except where otherwise stated. The starting mercury [17,18] and gold [3,4] complexes were obtained as reported. All complexes are orange in colour.

$[(2\text{-}PhN{=}NC_6H_4)A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)Cl]$ (I)

To a suspension of $[Au(2\text{-}C_6H_4CH_2NMe_2)Cl_2]$ (84.8 mg, 0.21 mmol) in acetone (10 cm^3), solid $[Hg(2\text{-}C_6H_4N{=}NPh)_2]$ (112.6 mg, 0.21 mmol) and some more acetone (5 cm^3) were added. After 6 h the resulting solution was evaporated to dryness and diethyl ether (15 cm^3) added to give a suspension; this was filtered, yielding an orange solid ($[Hg(2\text{-}C_6H_4N{=}NPh)Cl]$) and an orange solution. This solution was concentrated (1 cm^3), and slow addition of n-hexane (15 cm^3) gave complex I.

$[(2\text{-}Ph\overline{N{=}NC_6H_4)A}\overline{u(2\text{-}C_6H_4CH_2N}Me_2]^+\ A^-$ ($A = ClO_4$ (II), $AuCl_4$ (III))

To a solution of I (145.2 mg, 0.27 mmol) in acetone (15 cm^3), solid $AgClO_4$ (56 mg, 0.27 mmol) was added and the resulting suspension stirred for 24 h in the dark and then evaporated to dryness. The residue was extracted with dichloromethane (3 × 10 cm^3) and filtered through celite; the orange filtrate was concentrated (1 cm^3) and diethyl ether (15 cm^3) was added to give II.

Suspensions of II (95 mg, 0.16 mmol) and $Me_4N[AuCl_4]$ (66.1 mg, 0.16 mmol) were mixed and stirred at 0 to −5°C for 3.5 h, giving a suspension. After filtration through celite, the solution was concentrated (1 cm^3) and diethyl ether (10 cm^3) added to give a solid, which was recrystallized from dichloromethane/n-hexane to give III. Single crystals of III were obtained by diffusion of diethyl ether into a solution of III in dichloromethane.

$[(2\text{-}PhN{=}NC_6H_4)A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)(OOCCH_3)]$ (IV)

Solid $KOOCCH_3$ (25.5 mg, 0.26 mmol) was added to a suspension of II (158 mg, 0.26 mmol) in acetone (15 cm^3) and the resulting suspension was stirred for 15 h and then evaporated to dryness. The residue was extracted with dichloromethane (15 cm^3), the extract was filtered through celite, and the filtrate concentrated (1 cm^3). Diethyl ether (15 cm^3) was added, and cooling (0°C) then gave a solid, which was recrystallized from diethyl ether/n-hexane to give IV.

$[(2\text{-}PhN{=}NC_6H_4)A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)(CN)]$ (V)

Solid KCN (19.5 mg, 0.3 mmol) was added to a solution of I (66.6 mg, 0.12 mmol) in acetone (10 cm^3) and the suspension was stirred for 3 d and then evaporated to dryness. The residue was extracted as for IV, except that n-hexane (15 cm^3) was added to the concentrated dichloromethane solution (1 cm^3) to give V.

$[(2\text{-}(PhN{=}NC_6H_4)A\overline{u(2\text{-}C_6H_4CH_2N}Me_2)(L)]ClO_4$ ($L = PPh_3$ (VI), py (VII))

Solid PPh_3 (7.9 mg, 0.03 mmol) or pyridine (0.05 cm^3) was added to a suspension of II (20 mg, 0.03 mmol) in dichloromethane (10 cm^3) and the resulting solution was

TABLE 2

ATOM COORDINATES ($\times 10^4$) FOR III

Atom	x	y	z
Au(1)	639.4(.5)	1083.2(.2)	1201.9(.5)
Au(2)	2207(1)	2500	531(1)
Au(3)	0	0	0
Cl(1)	2235(4)	3138(2)	527(4)
Cl(2)	1660(10)	2500	−1328(7)
Cl(3)	2659(10)	2500	2422(8)
Cl(4)	1243(4)	−396(2)	−703(5)
Cl(5)	340(4)	−197(2)	1831(4)
N(1)	1289(11)	945(4)	−1187(11)
N(2)	−501(10)	1504(4)	1699(11)
N(3)	579(10)	1118(4)	−641(11)
C(19)	−61(15)	1650(6)	2795(15)
C(1)	1978(12)	743(5)	−462(14)
C(2)	2761(13)	546(6)	−1008(14)
C(3)	3453(15)	344(7)	−345(16)
C(4)	3346(14)	344(6)	824(15)
C(5)	2589(13)	544(5)	1385(15)
C(6)	1834(11)	741(4)	742(14)
C(7)	721(12)	1043(5)	2951(14)
C(8)	1027(14)	716(6)	3530(15)
C(9)	987(15)	718(7)	4781(16)
C(10)	610(19)	1027(7)	5333(18)
C(11)	240(16)	1323(6)	4714(14)
C(12)	317(15)	1333(6)	3518(14)
C(13)	−153(13)	1324(6)	−1363(13)
C(14)	184(16)	1643(6)	−1939(16)
C(15)	−589(20)	1834(7)	−2509(19)
C(16)	−1593(19)	1721(8)	−2580(21)
C(17)	−1854(20)	1391(8)	−2013(22)
C(18)	−1168(16)	1190(5)	−1382(17)
C(20)	−1520(13)	1344(6)	1935(16)
C(21)	−645(18)	1822(5)	888(15)
C(100)	1322(25)	2500	4934(73)
Cl(6)	172(11)	2500	4851(11)
Cl(7)	2211(20)	2344(20)	5578(13)

stirred for 21 or 24 h, respectively. It was then concentrated (1 cm^3) and diethyl ether (10 cm^3) was added to precipitate a solid. Recrystallization from dichloromethane/diethyl ether gave VI or VII, respectively.

Crystal structure determination of III

Crystal data. $[C_{21}H_{21}AuN_3]^+[AuCl_4]^- \cdot 0.5CH_2Cl_2$, $M = 893.63$. Orthorhombic, *Pnma*, *a* 12.743(5), *b* 35.62(3), *c* 11.580(7) Å, *U* 5257 Å^3, $Z = 8$, D_x 2.26 g cm^{-3}, λ(Mo-K_α) 0.71069 Å, μ(Mo-K_α) 11.6 mm^{-1}, $F(000) = 3320$.

Data collection and reduction. Stoe-Siemens four-circle diffractometer, monochromated Mo-K_α radiation, $2\theta_{max}$ 50°; 4693 independent reflections measured in profile-fitting mode [19], 2884 with $F > 4\sigma(F)$ used for all calculations. Absorption correction based on ψ-scans; crystal size $0.35 \times 0.35 \times 0.15$ mm, transmission 0.26–1.00. Cell constants refined from 2θ values of 41 reflections in the range 20–23°.

TABLE 3

BOND LENGTHS (Å) AND ANGLES (°) FOR III

Au(1)–N(2)	2.166(15)	Au(1)–N(3)	2.140(14)
Au(1)–C(6)	2.021(16)	Au(1)–C(7)	2.033(17)
Au(2)–Cl(1)	2.272(7)	Au(2)–Cl(2)	2.263(10)
Au(2)–Cl(3)	2.264(10)	Au(3)–Cl(4)	2.273(6)
Au(3)–Cl(5)	2.276(6)	N(1)–N(3)	1.265(21)
C(19)–N(2)	1.482(23)	C(1)–N(1)	1.411(23)
C(1)–C(2)	1.375(26)	C(1)–C(6)	1.406(24)
C(2)–C(3)	1.371(28)	C(3)–C(4)	1.361(26)
C(4)–C(5)	1.364(27)	C(5)–C(6)	1.405 (24)
C(7)–C(8)	1.399(27)	C(8)–C(9)	1.449(27)
C(10)–C(9)	1.359(34)	C(10)–C(11)	1.359(33)
C(11)–C(12)	1.389(24)	C(12)–C(19)	1.488(29)
C(12)–C(7)	1.328(28)	C(13)–N(3)	1.451(23)
C(13)–C(14)	1.384(29)	C(13)–C(18)	1.380(28)
C(14)–C(15)	1.366(33)	C(15)–C(16)	1.345(36)
C(16)–C(17)	1.385(41)	C(17)–C(18)	1.345(34)
C(20)–N(2)	1.444(23)	C(21)–N(2)	1.482(24)
N(2)–Au(1)–N(3)	101.6(6)	N(2)–Au(1)–C(6)	173.0(7)
N(3)–Au(1)–C(6)	78.4(7)	N(2)–Au(1)–C(7)	79.6(7)
N(3)–Au(1)–C(7)	178.9(7)	C(6)–Au(1)–C(7)	100.5(8)
Cl(1)–Au(2)–Cl(2)	90.1(2)	Cl(1)–Au(2)–Cl(3)	89.9(2)
Cl(2)–Au(2)–Cl(3)	176.8(5)	Cl(1)–Au(2)–Cl(1a)	178.2(4)
Cl(4)–Au(3)–Cl(5)	90.5(3)	Cl(4)–Au(3)–Cl(4b)	180.0
Cl(5)–Au(3)–Cl(4b)	89.5(3)	Cl(5)–Au(3)–Cl(5b)	180.0
N(3)–N(1)–C(1)	113.4(14)	Au(1)–N(2)–C(19)	102.5(11)
Au(1)–N(2)–C(20)	112.4(13)	C(19)–N(2)–C(20)	108.4(14)
Au(1)–N(2)–C(21)	116.3(12)	C(19)–N(2)–C(21)	108.7(15)
C(20)–N(2)–C(21)	108.1(15)	Au(1)–N(3)–N(1)	116.4(11)
Au(1)–N(3)–C(13)	128.8(11)	N(1)–N(3)–C(13)	114.8(14)
N(2)–C(19)–C(12)	109.7(17)	N(1)–C(1)–C(2)	116.0(16)
N(1)–C(1)–C(6)	120.7(15)	C(2)–C(1)–C(6)	123.2(16)
C(1)–C(2)–C(3)	118.5(17)	C(2)–C(3)–C(4)	119.5(20)
C(3)–C(4)–C(5)	123.0(19)	C(4)–C(5)–C(6)	119.5(17)
Au(1)–C(6)–C(1)	110.9(12)	Au(1)–C(6)–C(5)	132.6(13)
C(1)–C(6)–C(5)	116.0(15)	Au(1)–C(7)–C(8)	123.4(14)
Au(1)–C(7)–C(12)	114.7(14)	C(8)–C(7)–C(12)	121.3(17)
C(7)–C(8)–C(9)	117.7(18)	C(8)–C(9)–C(10)	119.2(21)
C(9)–C(10)–C(11)	120.1(20)	C(10)–C(11)–C(12)	121.5(21)
C(19)–C(12)–C(7)	116.1(16)	C(19)–C(12)–C(11)	123.9(20)
C(7)–C(12)–C(11)	120.0(20)	N(3)–C(13)–C(14)	119.6(17)
N(3)–C(13)–C(18)	115.9(17)	C(14)–C(13)–C(18)	124.5(18)
C(13)–C(14)–C(15)	114.7(20)	C(14)–C(15)–C(16)	124.5(25)
C(15)–C(16)–C(17)	116.9(24)	C(16)–C(17)–C(18)	123.5(24)
C(13)–C(18)–C(17)	115.7(20)		

Symmetry operators: (a) $x, 0.5-y, z$
(b) $-x, -y, -z$

Structure solution and refinement. Heavy-atom method, refinement on F to R 0.062, R_w 0.056. Non-H atoms (excluding solvent C) anisotropic, H atoms (excluding solvent) included using riding model (C–H 0.96 Å, H–C–H 109.5°, $U(\mathrm{H})=$

1.2U(C). Weighting scheme $w^{-1} = \sigma^2(F) + 0.0009\ F^2$; 294 parameters. Final atom coordinates and derived dimensions are given in Tables 2 and 3 *.

There are two independent $AuCl_4^-$ anions, one with crystallographic m symmetry and one with i symmetry. The solvent molecules are disordered across the mirror plane $y = 0.25$.

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* Further crystallographic data (temperature factors, H atom coordinates, structure factors) have been deposited with the Fachinformationszentrum Energie Physik Mathematik, 7514 Eggenstein-Leopoldshafen 2 (F.R.G.), whence they may be obtained on request; please quote reference no. CSD 51845 and the full literature citation.