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SYNTHESIS AND REACTIVITY OF DIBENZOMETALLOLE COMPLEXES OF GOLD(III) AND PLATINUM(II)

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Summary

Gold(III) and platinum(II) metallacycles, $[(C_{12}H_8)AuCl]_n$, $[(C_{12}H_8)AuX_2]^-$ and $[(C_{12}H_8)Pt(COD)]$ can be prepared by treating $[AuCl_3(tht)]$, $[AuX_4]^-$ or $[PtCl_2(COD)]$ with $(C_{12}H_8)Sn$-n-Pr_2 ($C_{12}H_8$ = 2,2′-biphenyl, COD = 1,5-cyclooctadiene, tht = tetrahydrothiophen). The reaction of $[(C_{12}H_8)AuCl]_n$ with neutral monodentate ligands L (L = PPh_3, py or tht) or bidentate ligands LL (LL = phen or bipy) leads to complexes of the types $[(C_{12}H_8)AuClL]$ or $[(C_{12}H_8)AuLL]^+$ $[(C_{12}H_8)AuCl_2]^-$, whilst treatment with anionic ligands (X = CN) gives polynuclear $[(C_{12}H_8)AuX]_m$ or mononuclear species $[(C_{12}H_8)Au(acac)]$ (X = acac = acetylacetonate), respectively. The bromo ligands of $[(C_{12}H_8)AuBr_2]^-$ can be replaced by other halogens or pseudohalogens (X = I, CN, SCN) to give anionic complexes of the type $[(C_{12}H_8)AuX_2]^-$. The reactions of $[(C_{12}H_8)Pt(COD)]$ with neutral monodentate ligands (L = PPh_3 or t-BuNC) lead to complexes of the type $[(C_{12}H_8)PtL_2]$.

Introduction

Only a few metallacyclic complexes containing Au as the heteroatom are known, viz. the pentamethylene derivatives $[(CH_2)_5AuBr]_2$ [1] and several dimeric and monomeric tetraphenylaurapentadienegold complexes [2,3]. Although dibenzometallole complexes of post-transition [4] and transition [5] metals have been prepared by using 2,2′-dilithium-biphenyl [6], the transition metal derivatives were only obtained in low yields, (e.g. 4.8% yield for $[(C_{12}H_8)Pt(norbornadine)]$, and no gold derivative has hitherto been reported.

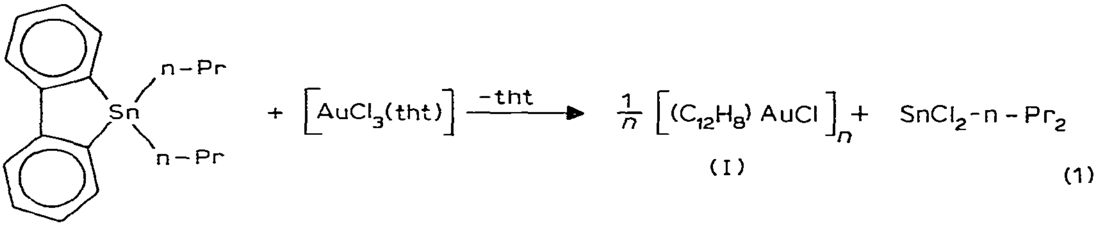
In the present paper we show that the reaction of a stannole with $[AuCl_3(tht)]$, which we have previously used [3] for the preparation of tetraphenylauracyclopentadienegold(III) derivatives, can also be adapted for the synthesis of gold(III) and platinum(II) dibenzometallole complexes by using 5,5-di-n-propyldibenzostannole as the source of the $C_{12}H_8$ group.

While aryltin compounds have been extensively used in forming aryl—platinum bonds [7], to our knowledge this is the first time that a dibenzostannole compound has been used as intermediate in the synthesis of organic complexes of transition metals.

Results and discussion

Gold(III) complexes

The reaction of 5,5-di-n-propyldibenzostannole with [$AuCl_3(tht)$] in dichloromethane according to eq. 1 leads to the precipitation of a slightly grey-tinged product (contaminated with traces of metallic gold). Because of its insolubility it was not possible to determine its conductivity and molecular weight. However, the analytical data (see Table 1) are in good agreement with the formula [$(C_{12}H_8)AuCl]_n$ (I), n probably being 2, as was found for all the analogous complexes previously described [8].



(1)

The chemical behaviour of complex I is summarized in Scheme 1.

(a) I reacts with monodentate neutral ligands to give mononuclear neutral complexes of the type [$(C_{12}H_8)AuClL$] (II, L = PPh_3; III, L = py; IV, L = tht).

(b) I reacts with bidentate neutral ligands to give complexes of the type $[(C_{12}H_8)Au(LL)]^+$ $[(C_{12}H_8)AuCl_2]^-$ (VI, LL = phen; VII, L = bipy) as previously reported for $[(CH_3)_2AuX]_2$ [8]).

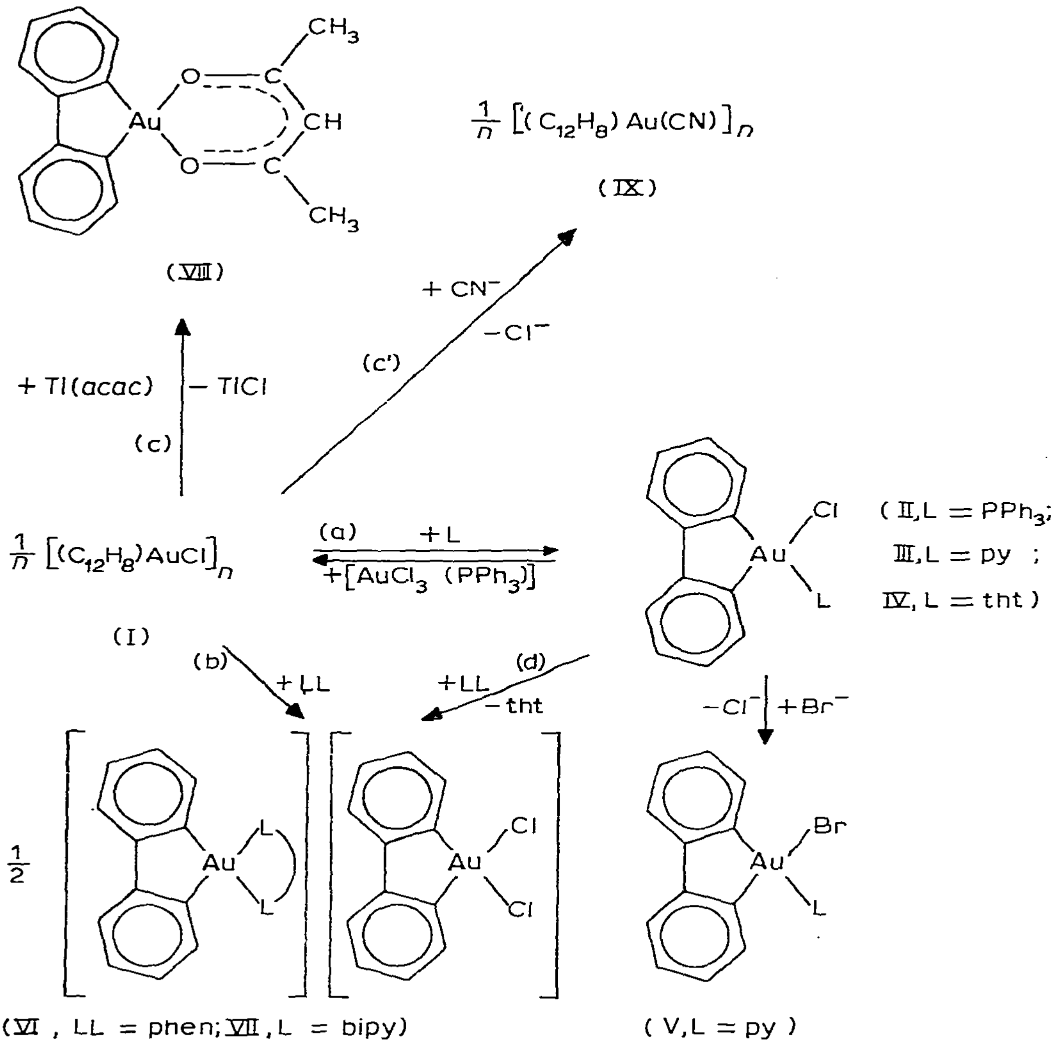
(c) I reacts with anionic chelating ligands, such as acetylacetonate (acac), with displacement of the halogen and cleavage of the bridges to give the mononuclear complex [$(C_{12}H_8)Au(acac)$] (VIII). The reaction with CN^- (c′) leads to an insoluble polymeric product (IX), which may be tetrameric, by analogy with $[(CH_3)_2Au(CN)]_4$ [9] (see IR spectra).

(d) The Cl of the neutral complexes II—IV can be replaced by other anions: thus reaction of III with Br^- leads to complex V, though with copious precipitation of metallic gold. The reaction of IV with bidentate ligands leads to the previously mentioned complexes VI and VII. Complex VII, which is insoluble in organic solvents, is best prepared by this route, since it avoids the contamination with metallic gold generally contained in complex I.

It seems of interest that the reaction of II with [$AuCl_3(PPh_3)$] yields complex I as a completely white solid, and can therefore be used for the purification of this compound, which cannot be recrystallized because of its insolubility. Doubtless [$AuCl_3(PPh_3)$] abstracts the phosphine ligand from II, according to eq. 2.

$$[(C_{12}H_8)AuCl(PPh_3)] + [AuCl_3(PPh_3)] \rightarrow [AuCl(PPh_3)] + PCl_2PPh_3 + [(C_{12}H_8)AuCl]_n \quad (2)$$

SCHEME 1



Substituting $R_4N[AuX_4]$ for $AuCl_3(tht)$ in eq. 1 leads to a novel type of derivatives $R_4N[(C_{12}H_8)AuX_2]$ (X, R = Me, X = Cl; XI, R = n-Bu, X = Br) as may be seen from eq. 3.

$$(C_{12}H_8)Sn\text{-}n\text{-}Pr_2 + R_4N[AuX_4] \rightarrow R_4N[(C_{12}H_8)AuX_2] + SnX_2\text{-}n\text{-}Pr_2 \qquad (3)$$

The reaction of XI with NaI, KCN or KSCN leads to the corresponding substitution products $R_4N[(C_{12}H_8)AuY_2]$ (R = n-Bu, XII, Y = I; XIII, Y = CN, XIV, Y = NCS). The cyano complex XIII can also be obtained by treating IX with $[n\text{-}Bu_4N]CN$.

Platinum(II) complexes

The reaction of the dibenzostannole compound with $[PtCl_2(COD)]$ in reflux-

TABLE 1

PHYSICAL AND ANALYTICAL DATA OF COMPLEXES I—XVII

	Complex	Yield (%)	M.p. (°C)	Λ_M [a] (Ω^{-1} cm^2 mol^{-1})	Analytical data (Found (calcd.) (%))				Mol. wt. (Found calcd.)
					C	H	N	Au	
(I)	$[(C_{12}H_8)AuCl]_2$	70—75	>300	[c]	37.12 (37.41)	2.19 (2.10)		51.00 (51.21)	b
(II)	$[(C_{12}H_8)AuCl(PPh_3)]$	80	247	4.9	55.06 (55.70)	3.10 (3.58)		29.10 (30.45)	613 [c] (647)
(III)	$[(C_{12}H_8)AuCl(py)]$	55	200	1.8	43.98 (44.08)	2.62 (2.83)	3.01 (3.02)	42.97 (42.48)	481 [e] (464)
(IV)	$[(C_{12}H_8)AuCl(tht)]$	42	300	0.9	40.85 (40.65)	3.51 (3.40)		40.92 (41.66)	
(V)	$[(C_{12}H_8)AuBr(py)]$	17	180d	0.6	40.20 (40.18)	2.58 (2.57)	3.01 (2.75)	38.28 (38.76)	526 [c] (508)
(VI)	$[(C_{12}H_8)Au(phen)][(C_{12}H_8)AuCl_2]$	90	300	23.0 [d]	44.97 (45.54)	2.43 (2.54)	3.08 (2.96)	42.20 (41.49)	
(VII)	$[(C_{12}H_8)Au(bipy)][(C_{12}H_8)AuCl_2]$	80	275d	14.1 [d]	43.34 (44.12)	2.63 (2.61)	3.08 (3.02)	42.90 (42.75)	
(VIII)	$[(C_{12}H_8)Au(acac)]$	15	270	0.9 [d]	45.16 (45.55)	3.35 (3.37)		41.66 (43.94)	418 [c] (448)
(IX)	$[(C_{12}H_8)Au(CN)]_n$	80	296d	[b]	40.29 (41.62)	2.27 (2.15)	3.42 (3.73)	52.14 (52.20)	[b]
(X)	$Me_4N[(C_{12}H_8)AuCl_2]$	30	161d	140	39.38 (38.89)	4.16 (4.08)	3.17 (2.83)	38.50 (39.85)	
(XI)	$n\text{-}Bu_4N[(C_{12}H_8)AuBr_2]$	85	85d	118	44.31 (44.76)	6.05 (5.90)	2.09 (1.86)	26.79 (26.22)	
(XII)	$n\text{-}Bu_4N[(C_{12}H_8)AuI_2]$	55	116	116	40.46 (39.78)	5.28 (5.25)	1.90 (1.66)	22.63 (23.30)	
(XIII)	$n\text{-}Bu_4N[(C_{12}H_8)Au(CN)_2]$	83	144	113	55.10 (55.98)	6.82 (6.89)	6.01 (6.53)	30.77 (30.60)	
(XIV)	$n\text{-}Bu_4N[(C_{12}H_8)Au(NCS)_2]$	73	83	117	50.71 (50.91)	6.09 (6.27)	5.91 (5.94)	28.63 (27.83)	
(XV)	$[(C_{12}H_8)Pt(COD)]$	80	243d	1.5	53.66 (52.74)	4.59 (4.43)			463 [c] (455)
(XVI)	$[(C_{12}H_8)Pt(PPh_3)_2]$	81	213d	1.7	67.23 (66.12)	4.59 (4.39)			936 [c] (872)
(XVII)	$[(C_{12}H_8)Pt(t\text{-}BuNC)_2]$	71	184	0.3	50.47 (51.45)	5.06 (5.10)	5.23 (5.45)		530 [c] (513)

[a] In acetone unless otherwise stated. [b] Not soluble enough for determination. [c] In chloroform. [d] In *N,N*-dimethylformamide. [e] In benzene.

ing $CHCl_3$ leads to XV (eq. 4).

$$(C_{12}H_8)Sn\text{-}n\text{-}Pr_2 + [PtCl_2(COD)] \rightarrow \underset{(XV)}{[(C_{12}H_8)Pt(COD)]} \qquad (4)$$

The diolefine group of (XV) can be readily replaced to give $[(C_{12}H_8)PtL_2]$ (XVI, L = PPh_3; XVII, L = t-BuNC).

Analytical and other data for the novel complexes are listed in Table 1. Most of them show conductivities in accordance with the proposed formulae. However, conductivities of VI and VII are rather low, and indicate the presence of ions in equilibrium with neutral species.

IR spectra

On comparing the IR spectra of the novel complexes and the dibenzostannole starting compound the following bands can tentatively be assigned to the dibenzometallole group: 3060—3040; 1580—1550 (two or three bands); 1310; 1250; 1160—1150; 1120—1110; 1060—1040; 1020—1010; 1000—990; 950—925; 750—740; 660—640; 620—610; 480—470 and 420—410 cm^{-1}.

The chlorogold(III) complexes show bands assignable to ν(Au—Cl) in the 350—200 cm^{-1} region. Complex I shows absorptions at 316 and 250 cm^{-1} and the chloro complexes II—IV exhibit a strong or medium absorption at approx. 310 cm^{-1}. VI, VII and X show two bands at 316 and 272 cm^{-1}. The similar spectra of these three complexes support the proposed formulae. The absorption at ~200 cm^{-1} observed for the bromo complex could not be resolved, which indicates that the band due to ν(Au—Br) must be located slightly below the range of our instrument. The absorptions due to ν(Au—I) in complex XII must be located at still lower energies.

The acac complex VIII shows three very strong bands at 1580, 1554 and 1518 cm^{-1}, as previously observed for $[Me_2Au(acac)]$ [10] in which the acac ligand also acts as a chelating group.

The dicyano complex XIII exhibits a weak band at 2156 cm^{-1}, instead of the two expected absorptions due to ν(CN); a chance coincidence of the frequencies could be the explanation of this. Similar observations have been reported for some cyanopalladium complexes [11]. The cyano complex IX shows a strong band at 2200 cm^{-1} which coincides with that reported for $[Me_2AuCN]_4$ [12].

As far as we know, all the gold(III) complexes containing SCN^- as a ligand are S-bonded: thus the anionic derivatives $[Au(SCN)_4]^-$ [13], *trans*-$[Au(CN)_2(SCN)_2]^-$ [14], $[Au(C_6F_5)(SCN)_2Br]$ [15] and $[Au(C_6F_5)_3(SCN)]^-$ [16] show the ν(CN) absorptions at about 2144—2130 cm^{-1}, whilst the neutral complex $[Au(CH_3)_2(SCN)L]$ [17] exhibits it at 2120 cm^{-1}. The absorption due to ν(CN) of complex XIV is observed at lower frequencies (2100(vs) cm^{-1}) and the determination of its internal standard ratio [18] gives 4.7. All this points to a N-bonded structure for the complex.

The *cis*-configuration of the platinum complexes is confirmed by the presence of a strong band at 536 cm^{-1} (XVI) [19] or two very strong bands at 2200 and 2180 cm^{-1} (XVII).

Experimental

IR spectra were recorded (over the range 4000—200 cm^{-1}) on a Perkin—Elmer 577 spectrophotometer using Nujol mulls between polyethylene sheets. Conductivities were measured in 5×10^{-4} *M* solution with a Philips PW 9501/01 conductimeter. Molecular weights were determined with a Hitachi Perkin—Elmer 115 Osmometer. C, H and N analyses were carried out with a Perkin—Elmer 240 microanalyzer. Au was determined by ashing the samples together with an aqueous solution of hydrazine.

Unless otherwise stated, all the reactions were carried out at room temperature.

The starting complexes [$(C_{12}H_8)$Sn-n-Pr$_2$] [4b], [Cl$_3$Au(tht)] [20], R$_4$N-[AuX$_4$] [21] and [PtCl$_2$(COD)] [22] were prepared as described in the literature. Calculations were based on $n = 2$ in I.

[$(C_{12}H_8)$AuCl]$_n$ (I). Addition of [AuCl$_3$(tht)] (220 mg, 0.56 mmol) to a dichloromethane (20 ml) solution of $(C_{12}H_8)$Sn-n-Pr$_2$ (200 mg, 0.56 mmol) led to immediate precipitation of a grey solid. After 45 min. stirring the solvent was evaporated and the residue was washed with diethyl ether (15 ml). The resulting complex I is insoluble in dichloromethane, diethyl ether, acetone, benzene and *N,N*-dimethylformamide.

Pure I was obtained by treating [$(C_{12}H_8)$AuCl(PPh$_3$)] (II) (110 mg, 0.17 mmol) in dichloromethane (20 ml) with [AuCl$_3$(PPh$_3$)] (96 mg, 0.17 mmol). The white precipitate was filtered off and washed with diethyl ether (15 ml) (63% yield). Addition of ethanol (10 ml) to the filtrate and concentration to ~15 ml gave [AuCl(PPh$_3$)].

[$(C_{12}H_8)$AuXL] (II—V). To a dichloromethane suspension of I the relevant ligand was added in 2/1 ratio (PPh$_3$) or drop by drop (py, tht) until a solution was formed. The solution stirred for 1 h at room temperature and filtered through a column containing Celite and anhydrous MgSO$_4$ to remove the colloidal gold. Concentration of the filtrate (to ca. 5 ml) and addition of diethyl ether (20 ml) gave white (II, III) or orange-coloured (IV) solids, which were filtered off and washed with diethyl ether (15 ml). II—IV are soluble in dichloromethane and acetone, and III is also soluble in benzene. They are insoluble in diethyl ether and n-hexane.

Addition of NaBr (30 mg, 0.30 mmol) to a solution of complex III (86 mg, 0.20 mmol) in 29 ml of acetone gave a suspension, which was stirred for 14 h, evaporated to dryness, extracted with dichloromethane (2 × 10 ml), filtered, concentrated (to ca. 5 ml), and treated with diethyl ether (5 ml). The formed metallic gold was removed by filtration through Celite and anhydrous MgSO$_4$ and the filtrate was cooled to —70°C. Complex V crystallized out as a yellow solid, which was filtered off and washed with diethyl ether.

[$(C_{12}H_8)$Au(phen)] [$(C_{12}H_8)$AuCl$_2$] (VI). By the procedure described above (phen/I in 1/1 molar ratio) complex VI was obtained as a cream-coloured solid, which is soluble in dichloromethane and *N,N*-dimethylformamide but insoluble in diethyl ether, nitromethane, acetone and benzene.

[$(C_{12}H_8)$Au(bipy)] [$(C_{12}H_8)$AuCl$_2$] (VII). Addition of 2,2'-bipyridyl (50 mg, 0.16 mmol) to a solution of IV (150 mg, 0.32 mmol) in dichloromethane (20 ml) caused immediate precipitation of VII which, after 30 min. stirring, was

isolated as a yellow solid. Further amounts of the complex were obtained by cooling the filtrate to −30°C. VIII is soluble only in *N,N*-dimethylformamide.

$[(C_{12}H_8)Au(acac)]$ (VIII). Tl(acac) (120 mg, 0.40 mmol) was added to a suspension of I (150 mg, 0.20 mmol) in 20 ml of dichloromethane, and the mixture was stirred for 24 h and filtered. The filtrate was concentrated to 5 ml and addition of diethyl ether (15 ml) caused precipitation of a white solid which was washed with diethyl ether. VIII is soluble in acetone and dichloromethane, but insoluble in diethyl ether, benzene and ethanol.

$[(C_{12}H_8)Au(CN)]_n$ (IX). KCN (110 mg, 1.69 mmol) was added to a suspension of I (650 mg, 0.84 mmol) in 20 ml of acetone, and the mixture was stirred for 90 min. and filtered. The filtrate was evaporated to dryness and the residue was washed with methanol (3 × 20 ml) filtered off, and vacuum dried. IX is insoluble in organic solvents.

$Me_4N[(C_{12}H_8)AuCl_2]$ (X). To a solution of $Me_4N[AuCl_4]$ (232 mg, 0.56 mmol) in 20 ml of acetone was added $(C_{12}H_8)$Sn-n-Pr_2 (200 mg, 0.56 mmol). After 1 h stirring at room temperature and 1 h refluxing the solution was concentrated to 5 ml and filtered. Addition of 15 ml of diethyl ether caused the precipitation of the white X, which is soluble in acetone and dichloromethane, but insoluble in diethyl ether and benzene.

n-$Bu_4N[(C_{12}H_8)AuBr_2]$ (XI). Starting from n-$Bu_4N[AuBr_4]$ and following the procedure described above, gave, after 20 h stirring, a colourless solution, from which XI was isolated as a white solid. Its solubility was similar to that of X.

n-$Bu_4N[(C_{12}H_8)AuY_2]$ (XII—XIV). To an acetone solution of XI was added a stoichiometric amount of MY (NaI, KCN or KSCN) and the suspension was stirred for ca. 2 h then it was concentrated to dryness. The residue was extracted with dichloromethane, and the extract was concentrated. Addition of diethyl ether caused precipitation of the yellow complex XII or white complexes XIII or XIV. Their solubilities were similar to that of complex X.

The dicyano complex XIII can also be obtained by treating the cyano complex IX with the stoichiometric amount of [n-Bu_4N]CN (prepared in dichloromethane solution by treating KCN with n-Bu_4NBr) and then following the procedure described above (83% yield).

$[(C_{12}H_8)Pt(COD)]$ (XV). $(C_{12}H_8)$Sn-n-Pr_2 (357 mg, 0.91 mmol) was added to a solution of $[PtCl_2(COD)]$ (228 mg, 0.61 mmol) in 20 ml of chloroform and the mixture was refluxed for 24 h. The resulting yellow solution was evaporated to dryness and the residue was washed with hexane (2 × 10 ml) and diethyl ether (2 × 10 ml) to give the yellow complex XIV, which is soluble in dichloromethane, chloroform and acetone, but insoluble in diethyl ether and n-hexane.

$[(C_{12}H_8)PtL_2]$ (XVI—XVII). A stoichiometric amount of L (PPh_3 or t-BuNC) was added to a dichloromethane solution of XV (20 ml) and the mixture was stirred overnight. Evaporation to dryness and washing of the residue with diethyl ether (2 × 10 ml) gave a first fraction of the required complex. A further amount was obtained by adding n-hexane to the filtrate and concentrating the solution. The white complexes XVI and XVII are soluble in dichloromethane and acetone, but insoluble in diethyl ether and n-hexane.

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