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ISOLATION OF BIS(TRISDIMETHYLAMINOPHOSPHONIUMMETHYLIDE)-GOLD(I) CHLORIDE[1)]

Yoshihisa YAMAMOTO

Faculty of Pharmaceutical Science, HIGASHI-NIPPON-GAKUEN
UNIVERSITY, Ishikari-Tobetsu, Hokkaido 061-02

Bis(trisdimethylaminophosphoniummethylide)gold(I) chloride, $[(Me_2N)_3P\text{-}CH_2\text{-}Au\text{-}CH_2\text{-}P(NMe_2)_3]Cl$, has been isolated and characterized by analytical and spectroscopic methods. It is stable in water and air.

Although preparation of trimethylmethylenephosphorane-metal complexes[2)] and of triphenylmethylenephosphorane-metal complexes[3)] has already been reported, tris(dimethylamino)methylenephosphorane-metal complex has never been described because the tris(dimethylamino)methylenephosphorane (**L**) is very unstable. We have now isolated 2/1 complex of bis(trisdimethylaminophosphoniummethylide)gold(I) chloride (**1**) from **L** and triphenylphosphinegold(I) chloride. Complex **1** was prepared by the reaction of $(Me_2N)_3P{=}CH_2$[4)] (0.39 g, 2.2 mmol) and Ph_3PAuCl[5)] (0.36 g, 0.727 mmol) in 20 ml dry benzene. The reaction is written as;

$$\underset{\mathbf{L}}{3(Me_2N)_3P{=}CH_2} \quad + \quad (C_6H_5)_3PAuCl \longrightarrow \underset{\mathbf{1}}{[(Me_2N)_3P\text{-}CH_2\text{-}Au\text{-}CH_2\text{-}P(NMe_2)_3]Cl}.$$

They were dissolved in benzene and stirred for approximately 1 day at r.t. under nitrogen. White precipitates formed were filtered, washed with dry benzene, and dried in vacuum. Yield: 0.33 g (77.3%), mp 109-111 °C. Anal. Calcd for $C_{14}H_{40}N_6AuClP_2$ (MW 586.83): C, 28.65; H, 6.87. Found: C, 29.00; H, 6.86%. Complex **1** was soluble in water, benzene and chloroform. It was highly hygroscopic and was very stable both in water and air.

^{1}H- and ^{13}C-NMR spectra of **1** have been measured in D_2O at r.t.. The chemical shift (δPCH_2) of methylene protons was lower than that of **L** and the coupling constant ($^1J_{PC}$) was much smaller than that of **L** as is shown in Table. These spectra are similar to those of bis(triphenylphosphoniummethylide)-gold(I) chloride,[6)] $[(C_6H_5)_3P\text{-}CH_2\text{-}Au\text{-}CH_2\text{-}P(C_6H_5)_3]Cl$ (**2**). It seems that the ylide is attached to the metal atom through the carbanionic donor atom. The coupling constant ($^2J_{HCP}$) of **1** is different from that of **2**, i.e., the coupling constant of **2** is larger than that[7)] of the corresponding ylide. The ^{13}C-NMR spectrum of **L** has been measured in C_6D_6 at r.t. and the data are collected in Table.

Table. ^{1}H- and ^{13}C-NMR data of gold complex 1, ylide L and of related phosphorus complex L'

^{1}H

No	δNCH_3	$^3J_{HCNP}$	$\delta PCH_{2(3)}$	$^2J_{HCP}$	Solvent	Standard
L'	2.95 d(18H)	11.0	2.35 d(3H)	15.5	$CDCl_3$	int-TMS
L	2.49 d(18H)	10.0	0.1 d(2H)	13.5	C_6D_6	int-TMS
1	2.68 d(36H)	9.75	0.99 d(4H)	13.5	D_2O	int-DSS
	2.58 d(36H)	9.75	0.99 d(4H)	13.5	C_6D_6	int-TMS

^{13}C

No	δNCH_3	$^2J_{CNP}$	$\delta PCH_{2(3)}$	$^1J_{CP}$		
L'	36.62 d	3.9	9.04 d	112.3	$CDCl_3$	$CDCl_3$
L	37.6 d	2.93	-9.15 d	175.8	C_6D_6	C_6D_6
1	37.4 d	2.0	9.48 d	88.9	D_2O	int-dioxane
	37.2 d	2.93	7.9 d	87.9	$CDCl_3$	$CDCl_3$

L': $[(Me_2N)_3PCH_3]Br$. Standard: internal TMS (δ=0), $CDCl_3$ (77.1), C_6D_6 (128.0), dioxane (67.4), DSS (0 ppm).

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