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Note

Quantifying the *trans* influence of triphenylarsine. Crystal and molecular structures of *cis*-$[PtCl_2(SMe_2)(AsPh_3)]$ and *cis*-$[PtCl_2(AsPh_3)_2]\cdot CHCl_3$

Stefanus Otto [1]*, Maria H. Johansson

Inorganic Chemistry 1, Center for Chemistry and Chemical Engineering, Lund University, PO Box 124, S-221 00 Lund, Sweden

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Abstract

Reaction between a mixture of *cis–trans*-$[PtCl_2(SMe_2)_2]$ and 1 equiv. $AsPh_3$ in chloroform gives *cis*-$[PtCl_2(SMe_2)(AsPh_3)]$ crystallizing in $P2_1/n$ with $a = 10.397(2)$, $b = 14.876(3)$, $c = 13.956(3)$ Å, $\beta = 90.86(3)°$ and $Z = 4$. Selected geometrical parameters are Pt–As 2.3531(10), Pt–S 2.262(2), Pt–Cl (*trans* to S) 2.301(2), Pt–Cl (*trans* to As) 2.328(2) Å and S–Pt–As 88.85(6), S–Pt–Cl(2) 90.77(8), As–Pt–Cl(1) 91.07(6) and Cl–Pt–Cl 89.42(7)°. *cis*-$[PtCl_2(AsPh_3)_2]\cdot CHCl_3$ crystallizes in $P2_1/c$ with $a = 20.557(4)$, $b = 9.5951(19)$, $c = 20.147(4)$ Å, $\beta = 96.77(3)°$ and $Z = 4$. Selected geometrical parameters are Pt–As(1) 2.3599(9), Pt–As(2) 2.3770(9), Pt–Cl(1) (*trans* to As(1)) 2.3515(18), Pt–Cl(2) (*trans* to As(2)) 2.3251(18) Å and As–Pt–As 97.87(3), As(1)–Pt–Cl(2) 88.67(5), As(2)–Pt–Cl(1) 84.30(5) and Cl–Pt–Cl 89.32(7)°. By comparison with related structures from the literature the following *trans* influence series was established $PMe_2Ph > PPh_3 > AsPh_3 \approx SbPh_3 > Me_2SO \approx SMe_2 \approx SPh_2 > NH_3 \approx \text{olefin} > Cl^- > MeCN$.

Keywords: Triphenylarsine; Platinum; *Trans* influence; Crystal structures

1. Introduction

It is important to distinguish between the terms *trans* effect and *trans* influence. The *trans* effect is defined as the ability of a certain ligand to labilize the substitution of the ligand *trans* to itself, i.e. it is concerned with the reactivity of the complex and is thus a kinetic effect dependant on the energy difference between the ground- and transition states. The *trans* influence on the other hand is a measure of the extent to which a ligand is able to weaken the coordinating ability of the ligand *trans* to itself and is thus only concerned with the ground state of the complex. Furthermore the high *trans* effect of certain ligands can be attributed to either ground state labilization as in the case of σ binding ligands [1] such as H^- and CH_3^-, or by transition state stabilization for π bonding ligands [2] such as C_2H_4. Ligands exerting a high *trans* effect due to their π bonding properties only show a moderate *trans* influence while ground state labilization is directly linked to a large *trans* influence and is thus in essence a measure of the σ bonding properties of a ligand.

Several ways of quantifying the *trans* influence of ligands are used today, amongst others IR- and NMR spectroscopy and X-ray crystallography. Probably the most generally accepted criterium is comparison of bond distances obtained from a series of related crystal structure determinations.

Although the chemistry of phosphine ligands have been extensively studied, including numerous crystallographic investigations to aid in our understanding of their chemical properties, the solid state chemistry of arsine and stibine ligands have received very limited interest. In order to determine the *trans* influence of a ligand the bond distances *trans* to these ligands, typically to a chloride, is compared for a related series of complexes such as $[PtCl_3(L)]^-$ or *cis*-$[PtCl_2(L)_2]$ (L = neutral ligand). The *cis* influence could in principle be determined from complexes such as *trans*-$[PtCl_2(L)_2]$,

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* Corresponding author. Tel.: +46-46-2228 116; fax: +46-46-2224 439.

E-mail address: stefanus.otto@inorg.lu.se (S. Otto).

[1] On leave from the Department of Chemistry, University of the Free State, Bloemfontein 9300, South Africa.

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but as it is much less pronounced than the *trans* influence crystallographically determined bond distances are normally not discriminative enough to make clear-cut conclusions possible.

The first crystal structures of *cis*- and *trans*-platinum(II)bisstibine complexes were only reported [3–5] recently, while the known platinum(II)bisarsine complexes are limited to complexes with a *trans* geometry [6]. In this paper we report the crystal structures of *cis*-[$PtCl_2(SMe_2)(AsPh_3)$] (**1**) and *cis*-[$PtCl_2(AsPh_3)_2$]·$CHCl_3$ (**2**) in order to quantify the *trans* influence of $AsPh_3$.

Table 1
Experimental details, crystal data and refinement parameters for **1** and **2**

	1 [a]	**2** [b]
Chemical formula	$PtCl_2[S(CH_3)_2]$-$[As(C_6H_5)_3]$	$PtCl_2[As(C_6H_5)_3]_2$·$CHCl_3$
Chemical formula weight	634.34	997.80
Crystal system	monoclinic	monoclinic
Space group	$P2_1/n$	$P2_1/c$
a (Å)	10.397(2)	20.557(4)
b (Å)	14.876(3)	9.5951(19)
c (Å)	13.956(3)	20.147(4)
β (°)	90.86(3)	96.77(3)
V (Å^3)	2158.3(8)	3946.2(14)
Z	4	4
D_x (g cm^{-3})	1.952	1.679
Number of reflections for cell parameters	4773	5629
θ Range (°)	2.38–25.07	2.5–25.01
μ (mm^{-1})	8.368	5.583
F(000)	1208	1928
Crystal size (mm)	$0.19 \times 0.30 \times 0.42$	$0.10 \times 0.15 \times 0.20$
T_{min}/T_{max}	0.085/0.160	0.420/0.525
Collected reflections	23686	31929
Unique reflections (R_{int})	6734 (0.0947)	12002 (0.0572)
Observed reflections ($I > 2\sigma(I)$)	3546	8316
Range of h, k, l	$-14 \rightarrow h \rightarrow 15$ $-21 \rightarrow k \rightarrow 20$ $-13 \rightarrow l \rightarrow 20$	$-29 \rightarrow h \rightarrow 29$ $-13 \rightarrow k \rightarrow 9$ $-25 \rightarrow l \rightarrow 28$
θ Range (°)	2.00–31.86	2.00–31.56
R_1 [$F^2 > 2\sigma(F^2)$] [c]	0.0499	0.0573
wR_2 (F^2) [d]	0.0966	0.1103
R_1 (all data)	0.1271	0.0979
wR_2 (all data)	0.1146	0.1277
S	0.922	1.135
Data/restraints/parameters	6734/0/229	12002/1/470
Extinction coefficient		0.00097(8)
$(\Delta/\sigma)_{max}$	0.000	0.003
$\Delta\rho_{max}/\Delta\rho_{min}$ (e Å^{-3})	2.36/−1.45	2.52/−1.37

[a] *cis*-[$PtCl_2(SMe_2)(AsPh_3)$].

[b] *cis*-[$PtCl_2(AsPh_3)_2$]·$CHCl_3$.

[c] $R_1 = [(\Sigma \Delta F)/(\Sigma F_o)]$.

[d] $wR_2 = \Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]^{1/2}$.

2. Experimental

2.1. Preparation of complexes

All solvents and chemicals used for the preparation of the complexes were of reagent grade and were used without further purification. All preparations were carried out under aerobic conditions and no special precautions were taken to exclude atmospheric moisture. The starting complex *cis–trans*-[$PtCl_2(SMe_2)_2$] and *cis*-[$PtCl_2(AsPh_3)_2$] were prepared according to literature procedures [7,8], spectroscopic data were consistent with that reported. White plates of **2**, were grown by slow evaporation of a chloroform solution of *cis*-[$PtCl_2(AsPh_3)_2$].

2.1.1. *cis-[$PtCl_2(SMe_2)(AsPh_3)$] (**1**)*

$AsPh_3$ (40 mg, 0.131 mmol) in chloroform was added to a chloroform solution of *cis–trans*-[$PtCl_2(SMe_2)_2$] (50 mg, 0.128 mmol). Slow evaporation of the reaction mixture yielded yellow crystals of **1** suitable for X-ray analysis.

^{1}H NMR ($CDCl_3$): 2.45 (6H, t, $^3J_{Pt-H} = 48$); 7.4–7.5 (9H, m); 7.6–7.7 (6H, m).

2.2. X-ray measurements and structure determinations

The data collections were performed at 293(1) K on a Bruker Smart diffractometer equipped with a rotating anode, Mo Kα ($\lambda = 0.71073$ Å) radiation. The intensities were merged and integrated using SAINT [9] and corrected for Lorentz polarization (L_p) and absorption effects with SADABS [10]. The structures were solved by Patterson and difference Fourier methods and refined by full-matrix least-squares calculations using the SHELXS97 and SHELXL97 programs [11] with $\Sigma(F_o^2 - F_c^2)^2$ being minimized. All non-hydrogen atoms were refined anisotropically and the hydrogen atom positions were calculated as riding on the adjacent carbon atom (aromatic C–H = 0.93 Å, methyl C–H = 0.96 Å) and refined with an overall thermal parameter. The graphics were done with DIAMOND 2.0 [12].

3. Results and discussion

3.1. Crystallography

Details of the data collection and refinement parameters are summarized in Table 1. The thermal displacement ellipsoids and numbering schemes for **1** and **2** are given in Figs. 1 and 3, packing diagrams in Figs. 2 and 4 and selected geometrical parameters in Tables 2 and 3, respectively.

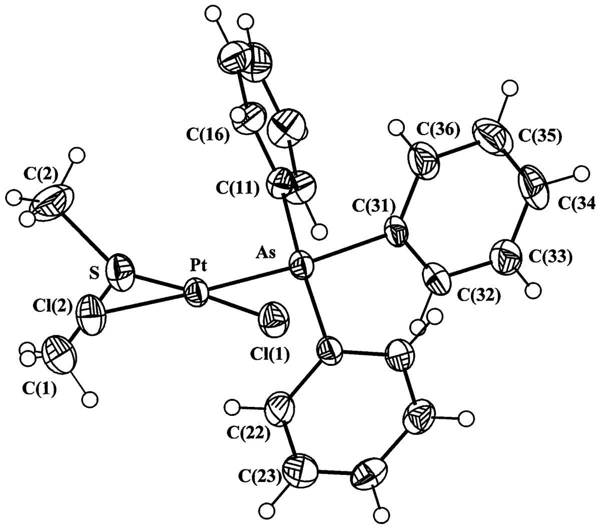

Fig. 1. DIAMOND drawing showing the numbering scheme and thermal ellipsoids (30% probability) for **1**. Hydrogen atoms are of arbitrary size.

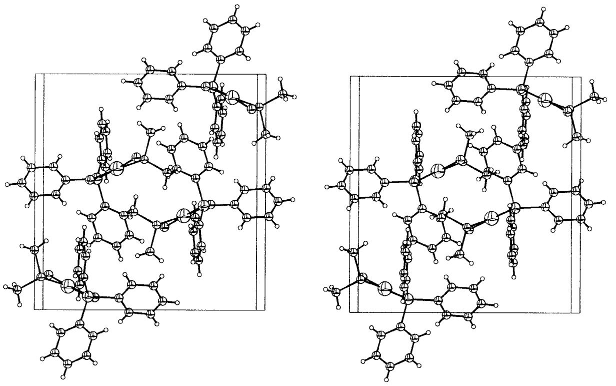

Fig. 2. Packing diagram of **1**.

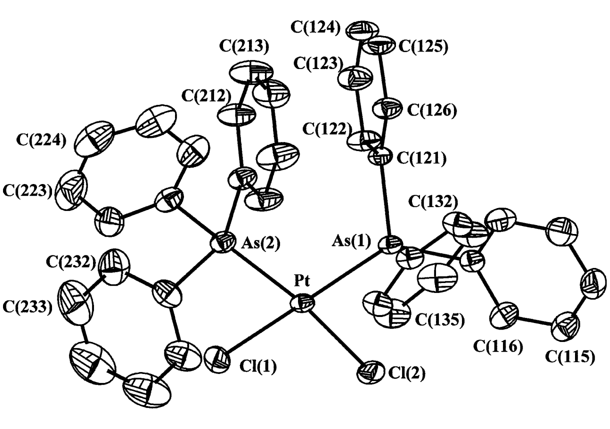

Fig. 3. DIAMOND drawing showing the numbering scheme and thermal ellipsoids (30% probability) for **2**. Hydrogen atoms and the chloroform solvent molecule are omitted for clarity.

3.1.1. Crystal structure of *cis*-[$PtCl_2(SMe_2)(AsPh_3)$]

The compound crystallizes as mononuclear complexes with a pseudo square planar coordination geometry with the chloride atoms in a *cis* configuration.

The Pt–S bond distance of 2.262(2) Å is within the normal range for a bond of this nature [14,19]. No crystallographic data are available for Pt–As bonds *trans* to a chloro ligand, but the distance of 2.3531(10) Å obtained during this study is slightly shorter than the sum of the covalent radii of As and Pt, 1.210 and 1.370 Å, respectively, [10]. The difference in the Pt–Cl distances of 2.301(2) and 2.328(2) Å *trans* to S and As, respectively, is indicative of the difference in the *trans* influences of these ligands, with that of $AsPh_3$ being larger than for SMe_2.

All angles within the platinum coordination sphere are close to 90°, ranging from 88.85(6) for As–P–S to 91.07(6)° for As–Pt–Cl(1). One would expect the angle between the two most bulky ligands, i.e. As–Pt–S, to be the largest within the coordination sphere, the opposite was surprisingly observed. This may be the result of the two methyl substituents on the dimethylsulfide ligands directed away from the bulky arsine ligand towards Cl(2), resulting in a S–Pt–Cl(2) angle of 90.77(8)°. The Cl(2)–Pt–S–C(1) and Cl(2)–Pt–S–C(2) torsion angles of $-53.3(4)$ and 52.1(4)° indicate an almost perfect staggered orientation of the methyl groups with respect to the coordination plane. On the other hand the small Cl(1)–Pt–As–C(31) torsion angle of $-26.1(3)°$ indicate that phenyl ring 3 is directed towards Cl(1) resulting in the slightly larger As–Pt–Cl(1) angle of 91.07(6)°. An almost identical coordination geometry was observed in *cis*-[$PtCl_2(SMe_2)(PMe_2Ph)$] [14].

The C–As–C angles range between 101.3(3) and 105.5(3)° while the C–As–Pt angles range from 111.5(2) to 121.2(2)°. The compression of the C–As–C angles (L = Group 15 atom) is normal for this type of ligands [13].

The packing within the unit cell (Fig. 2) seems to be governed by van der Waals forces alone, with no strong intermolecular interactions detected between the individual molecules. The only possible intermolecular interactions present are $\pi-\pi$ stacking between phenyl ring 3 in the arsine ligand with the same ring in two adjacent molecules. Ring 3 (symmetry x, y, z) has a distance of 5.315 and 5.550 Å to ring 3 ($1-x$, $1-y$, $-z$) and ring 3 ($-x$, $1-y$, $-z$) respectively. Both have an angle of 0.02° with respect to the first ring.

3.1.2. Crystal structure of *cis*-[$PtCl_2(AsPh_3)_2$]·$CHCl_3$

The compound crystallizes as mononuclear complexes with a pseudo square planar coordination geometry with the chloride atoms in a *cis* configuration. Each platinum complex is accompanied by a severely disordered chloroform solvent molecule.

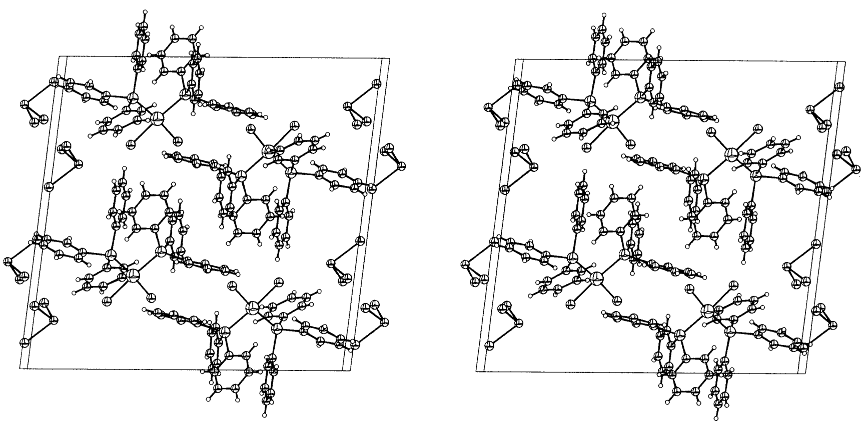

Fig. 4. Packing diagram of **2**.

The Pt atom is situated on a general position in the unit cell resulting in the arsine- and chloro ligands, which are chemically equivalent, to be crystallographically different. The individual Pt–As and Pt–Cl distances vary by 0.017(1) and 0.026(3) Å, respectively, suggesting possible solvent interactions. The shortest distances are comparable to those (Cl *trans* to As) found in **1**.

The As(1)–Pt–As(2) angle of 97.87(3)° deviates significantly from 90° indicating steric strain between these two bulky ligands. Interestingly enough the angles mainly compensating for this distortion is not the Cl–Pt–Cl angle, but in fact the Cl–Pt–As angles. The phenyl substituents on the two AsPh$_3$ ligands are similarly orientated, one ring above-, one below and one almost parallel to the coordination plane. Phenyl ring 1 on As(2) is pointing towards As(1), while the corresponding ring of As(1) is directed towards Cl(2). This results in a Cl(1)–Pt–As(2) angle of 84.30(5)° and a Cl(2)–Pt–As(1) angle of 89.32(7)°, indicative of larger steric strain in the latter.

Table 2
Selected bond distances (Å), angles (°) and torsion angles (°) for **1**

Bond distances			
Pt–As	2.3531(10)	Pt–S	2.262(2)
Pt–Cl(1)	2.301(2)	Pt–Cl(2)	2.328(2)
As–C(11)	1.946(7)	S–C(1)	1.803(9)
As–C(21)	1.927(7)	S–C(2)	1.792(10)
As–C(31)	1.936(7)		
Bond angles			
Cl(1)–Pt–Cl(2)	89.42(7)	As–Pt–S	88.85(6)
Cl(1)–Pt–As	91.07(6)	Cl(1)–Pt–S	176.73(8)
Cl(2)–Pt–As	178.03(6)	Cl(2)–Pt–S	90.77(8)
C(11)–As–C(21)	105.5(3)	C(1)–S–C(2)	99.5(5)
C(11)–As–C(31)	101.3(3)	C(1)–S–Pt	106.4(3)
C(21)–As–C(31)	103.7(3)	C(2)–S–Pt	106.6(4)
C(11)–As–Pt	111.5(2)	C(31)–As–Pt	121.2(2)
C(21)–As–Pt	112.1(2)		
Torsion angles			
Cl(1)–Pt–As–C(11)	−145.1(2)	Cl(2)–Pt–S–C(1)	−53.3(4)
Cl(1)–Pt–As–C(21)	96.9(2)	Cl(2)–Pt–S–C(2)	52.1(4)
Cl(1)–Pt–As–C(31)	−26.1(3)		

The tetrahedral angles around the two arsine atoms in **2** are very similar ranging from 100.4(3) to 109.5(3)° for C–As(1)–C and 97.7(3)–105.8(4)° for C–As(2)–C. The C–As(1)–Pt angles range from 111.44(18) to 118.71(17)° and C–As(2)–Pt from 112.0(2) to 122.3(5)°. These values are in agreement with that found in **1** indicating very similar geometries for the three triphenylarsine ligands in the two studies.

The platinum complexes are packed in two layers parallel to the *y*, *z*-plane in the middle of the unit cell. On the outside of the platinum complex layers the chloroform molecules are packed along the *y*, *z*-plane. The two layers are rotated 180° with respect to each

Table 3
Selected bond distances (Å), angles (°) and torsion angles (°) for **2**

Bond distances			
Pt–As(1)	2.3599(9)	Pt–As(2)	2.3770(9)
Pt–Cl(1)	2.3515(18)	Pt–Cl(2)	2.3251(18)
As(1)–C(1)	1.950(7)	As(2)–C(1)	1.951(7)
As(1)–C2	1.926(6)	As(2)–C(2)	1.951(8)
As(1)–C3	1.932(6)	As(2)–C(3)	1.938(8)
Bond angles			
Cl(1)–Pt–Cl(2)	89.32(7)	As(1)–Pt–As(2)	97.87(3)
Cl(1)–Pt–As(1)	175.75(5)	Cl(2)–Pt–As(2)	173.09(5)
Cl(2)–Pt–As(1)	88.67(5)	Cl(1)–Pt–As(2)	84.30(5)
C(111)–As(1)–C(121)	101.8(3)	C(211)–As(2)–C(221)	105.0(3)
C(111)–As(1)–C(131)	100.4(3)	C(211)–As(2)–C(231)	97.7(3)
C(121)–As(1)–C(131)	109.5(3)	C(221)–As(2)–C(231)	105.8(4)
C(111)–As(1)–Pt	118.71(17)	C(211)–As(2)–Pt	122.3(5)
C(121)–As(1)–Pt	111.44(18)	C(221)–As(2)–Pt	112.0(2)
C(131)–As(1)–Pt	113.8(2)	C(231)–As(2)–Pt	112.1(3)
Torsion angles			
Cl(1)–Pt–As(2)–C(211)	172.6(3)	Cl(2)–Pt–As(1)–C(111)	−14.0(2)
Cl(1)–Pt–As(2)–C(221)	61.6(3)	Cl(2)–Pt–As(1)–C(121)	−131.7(2)
Cl(1)–Pt–As(2)–C(231)	−57.2(3)	Cl(2)–Pt–As(1)–C(131)	103.9(2)

Table 4
Geometrical parameters for *cis*-$[Pt(Cl)_2(L_1)(L_2)]$ complexes (Cl_n *trans* to L_n, $n = 1$ or 2)

Complex	Pt–L_1 (Å)	Pt–Cl_1 (Å)	Pt–L_2 (Å)	Pt–Cl_2 (Å)	Pt–Cl_{avg} (Å) [a]	References
cis-$[PtCl_2(Sme_2)(Me_2SO)]$	2.294(3)	2.299(3)	2.211(4)	2.320(4)		[14]
cis-$[PtCl_2(Sme_2)(AsPh_3)]$	2.262(2)	2.301(2)	2.3531(10)	2.328(2)		TW [b]
cis-$[PtCl_2(Sme_2)(PMe_2Ph)]$	2.270(3)	2.299(3)	2.224(3)	2.367(3)		[14]
$(NH_4)_2[PtCl_4]$	2.305(5)				2.305(5)	[15]
cis-$[PtCl_2(MeCN)_2]$	1.978(5)	2.266(2)	1.969(5)	2.271(2)	2.269(4)	[16]
cis-$[PtCl_2(NH_3)_2]$	2.00(2)	2.315(7)	2.08(2)	2.306(7)	2.310(7)	[17]
cis-$[PtCl_2(COD)]$ [c]	2.172(6)	2.315(1)	2.176(6)	2.309(1)	2.312(4)	[18]
	2.154(5)		2.178(6)			
cis-$[Pt(Cl)_2(SPh_2)_2]$	2.2801(19)	2.319(2)	2.2832(18)	2.295(2)	2.307(17)	[19]
cis-$[Pt(Cl)_2(SMe_2)_2]$	2.269(1)	2.319(1)	2.272(1)	2.315(1)	2.317(3)	[20]
cis-$[Pt(Cl)_2(Me_2SO)_2]$	2.251(3)	2.313(3)	2.238(3)	2.319(4)	2.316(4)	[21]
cis-$[Pt(Cl)_2(SbPh_3)_2]$	2.491(1)	2.354(3)	2.510(1)	2.326(4)	2.338(12)	[4]
	2.497(1)	2.337(4)	2.512(1)	2.333(3)		
cis-$[Pt(Cl)_2(AsPh_3)_2]$	2.3599(9)	2.3515(18)	2.3770(9)	2.3251(18)	2.338(19)	TW
cis-$[Pt(Cl)_2(PPh_3)_2]$	2.267(3)	2.329(3)	2.244(3)	2.360(3)	2.35(2)	[22]
cis-$[Pt(Cl)_2(PMe_2Ph)_2]$	2.245(1)	2.355(1)	2.242(1)	2.359(1)	2.357(3)	[23]

[a] The error in Pt–Cl_{avg} is calculated as: $\sqrt{\Sigma_n(d_n-\bar{d})^2/(n-1)}$.

[b] TW, this work.

[c] COD, *cis,cis*-1,5-cyclooctadiene.

other with Cl(2) (short Pt–Cl distance) pointing towards the platinum complexes in the adjacent layer and Cl(1) (long Pt–Cl bond distance) are facing the solvent molecules. Since the solvent molecules are disordered and no hydrogen atom is placed on the chloroform carbon, no formal hydrogen bonding- or other interactions between Cl(1) and the solvent molecules could be established, no intermolecular π–π interactions of the phenyl rings were observed either.

3.2. *trans* Influence series

Table 4 shows a summary of bond distances for related complexes found in the literature. Both the average Pt–As and Pt–Cl bond distances determined for **2** are slightly longer than the Pt–As and Pt–Cl(2) bond distances found in **1**. The lengthening of the Pt–As bonds may be a result of the larger extent of steric crowding in **2** due to the presence of two bulky $AsPh_3$ ligands in the coordination sphere, as evident also from the large As–Pt–As angle of 97.87(3)°. The long Pt–Cl(1) distance found in **2** is most probably a result of interaction with the solvent molecule as it deviates significantly from the other two Pt–Cl bonds *trans* to As in **1** and **2**.

From Table 4 it is clear that in the mixed complexes *cis*-$[PtCl_2(SMe_2)(L)]$ ($L = SMe_2$, Me_2SO, $AsPh_3$, PMe_2Ph) the *trans* influence of $AsPh_3$ is slightly larger than that of SMe_2 and Me_2SO, but significantly smaller than that of PMe_2Ph. In the series of complexes *cis*-$[PtCl_2(L)_2]$ ($L = Cl^-$, MeCN, NH_3, COD, SPh_2, SMe_2, Me_2SO, $SbPh_3$, $AsPh_3$, PPh_3 and PMe_2Ph) the *trans* influence of $AsPh_3$ is comparable to that of $SbPh_3$, larger than that of Cl^-, all the S–, N– and olefin donor ligands, but still slightly smaller than that of PPh_3 and PMe_2Ph. The overall *trans* influence series established is, $PMe_2Ph > PPh_3 > AsPh_3 \approx SbPh_3 > Me_2SO \approx SMe_2 \approx SPh_2 > NH_3 \approx$ olefin $> Cl^- >$ MeCN. It should be noted that the errors in Pt–Cl_{avg} in Table 4 have not been used to discriminate between the *trans* influence of the ligands since these errors mainly reflect the intermolecular effect on the Pt–Cl bonds as discussed by Johansson et al. [6].

4. Supplementary material

Tables of the atomic coordinates, the anisotropic temperature factors for non-hydrogen atoms, equivalent isotropic temperature factors for the hydrogen atoms and observed and calculated structure factors for **1** and **2** were deposited at the Cambridge Crystallographic Database.

Acknowledgements

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