



# Crystal structures of chlorido[dihydroxybis(1-iminoethoxy)]arsanido- $\kappa^3N,As,N'$ ]platinum(II) and of a polymorph of chlorido[dihydroxybis(1-iminopropoxy)]arsanido- $\kappa^3N,As,N'$ ]platinum(II)

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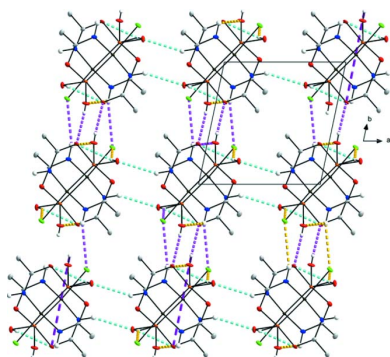
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Each central platinum(II) atom in the crystal structures of chlorido[dihydroxybis(1-iminoethoxy)arsanido- $\kappa^3N,As,N'$ ]platinum(II), [Pt(C<sub>4</sub>H<sub>10</sub>AsN<sub>2</sub>O<sub>4</sub>)Cl] (**1**), and of chlorido[dihydroxybis(1-iminopropoxy)arsanido- $\kappa^3N,As,N'$ ]platinum(II), [Pt(C<sub>6</sub>H<sub>14</sub>AsN<sub>2</sub>O<sub>4</sub>)Cl] (**2**), is coordinated by two nitrogen donor atoms, a chlorido ligand and to arsenic, which, in turn, is coordinated by two oxygen donor ligands, two hydroxyl ligands and the platinum(II) atom. The square-planar and trigonal-bipyramidal coordination environments around platinum and arsenic, respectively, are significantly distorted with the largest outliers being 173.90 (13) and 106.98 (14)° for platinum and arsenic in (**1**), and 173.20 (14)° and 94.20 (9)° for (**2**), respectively. One intramolecular and four classical intermolecular hydrogen-bonding interactions are observed in the crystal structure of (**1**), which give rise to an infinite three-dimensional network. A similar situation (one intramolecular and four classical intermolecular hydrogen-bonding interactions) is observed in the crystal structure of (**2**). Various  $\pi$ -interactions are present in (**1**) between the platinum(II) atom and the centroid of one of the five-membered rings formed by Pt, As, C, N, O with a distance of 3.7225 (7) Å, and between the centroids of five-membered (Pt, As, C, N, O) rings of neighbouring molecules with distances of 3.7456 (4) and 3.7960 (6) Å. Likewise, weak  $\pi$ -interactions are observed in (**2**) between the platinum(II) atom and the centroid of one of the five-membered rings formed by Pt, As, C, N, O with a distance of 3.8213 (2) Å, as well as between the Cl atom and the centroid of a symmetry-related five-membered ring with a distance of 3.8252 (12) Å. Differences between (**2**) and the reported polymorph [Miodragović *et al.* (2013). *Angew. Chem. Int. Ed.* **52**, 10749–10752] are discussed.

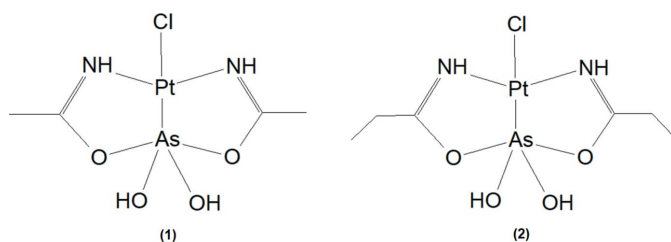


## 1. Chemical context

Platinum and arsenic compounds have shown great versatility in terms of applications in the biological and medicinal fields (Reedijk, 2009). Platinum compounds are still the most widely used drugs in the fight against cancer in spite of the serious side effects and the resistance of some types of cancers (Miller *et al.*, 2002; Basu & Krishnamurthy, 2010; Jakupec *et al.*, 2003; Kauffman *et al.*, 2010; Wheate *et al.*, 2010; Rosenberg *et al.*, 1965; Marino *et al.*, 2017; Aabo *et al.*, 1998; Kelland, 2007; Shi *et al.*, 2019). Tumoral malignancies have a high lethality rate and are among the most widespread and difficult diseases to treat. The need for the development of new drugs and treatment alternatives has increased as many of the available effective drugs are comparable and similar to each other (Ott, 2009; Burchenal, 1978). Platinum-based antitumour agents



have guided and constructed the current tumor chemotherapy treatment, but the side effects complicate and inhibit their clinical application (Rosenberg *et al.*, 1965; Marino *et al.*, 2017; Basu & Krishnamurthy, 2010; Aabo *et al.*, 1998; Kelland, 2007; Shi *et al.*, 2019). Drug resistance is a major limiting factor in terms of the range of tumours that can be treated and the improvement of the therapy (Marino *et al.*, 2017). Arsenic trioxide was approved by the FDA in 2000 for the treatment of acute promyelocytic leukemia, and since then several studies have shown that the combinatorial employment of arsenic and platinum-based cancer drugs has shown significant therapeutic potential (Wang *et al.*, 2004; Shen *et al.*, 2004; Emadi & Gore, 2010; Zhang *et al.*, 2009, 2010). These results led to the synthesis of complexes containing both platinum and arsenic (Swindell *et al.*, 2013; Miodragović *et al.*, 2013, 2019), which were called arsenoplatins. Initial results indicate that these complexes are able to bypass drug-resistance mechanisms that lower the effect of cisplatin and have higher cytotoxicity than cisplatin in some cases. To date, the studies of Miodragović *et al.* (2013, 2019) are the only crystallographic data available in the CCDC (Groom *et al.*, 2016).



The structures reported here,  $[\text{Pt}(\text{C}_4\text{H}_{10}\text{AsN}_2\text{O}_4)\text{Cl}]$  (**1**), and  $[\text{Pt}(\text{C}_6\text{H}_{14}\text{AsN}_2\text{O}_4)\text{Cl}]$ , (**2**), expand on this work and form part of an ongoing study on arsenoplatins, their solid- and solution-state behaviour and evaluation thereof.

## 2. Database survey

Two crystal structures similar to (**1**) were found after a search of the Cambridge Structure Database (CSD, Version 5.40, update of November 2019; Groom *et al.*, 2016), both of which (ODOHAS, ODOHEW) were reported by Miodragović *et al.* (2013, 2019). They consist of the same arsenoplatin complex as (**1**), accompanied by an acetamide hemihydrate and acetamide solvent species in the unit cell, and crystallize in the  $P\bar{1}$  and  $P2_1/n$  space groups, respectively. The search also revealed that

(**2**) represents a polymorph, with the first crystal structure determination (ODOGOF; Miodragović *et al.*, 2013) in the orthorhombic space group type  $Pbca$ , in contrast to space group type  $P2_1/c$  of (**2**).

## 3. Structural commentary

In (**1**) the square-planar coordination environment around platinum(II) is defined by two nitrogen donor atoms, a chlorido ligand and the coordination to arsenic. In turn, arsenic is coordinated by two oxygen donor atoms, two hydroxyl ligands and by platinum(II), completing a trigonal-bipyramidal coordination sphere (Fig. 1). The first (ODOHAS) of the other two structure reports with a chlorido[dihydroxybis(1-iminoethoxy)]arsanido]platinum(II) molecule (Miodragović *et al.*, 2013) is different from (**1**) because of an acetamide solvent molecule in the unit cell and a different space group ( $P2_1/n$ ), and the second (ODOHEW) crystallizes in the same space group as (**1**) ( $P\bar{1}$ ) but with acetamide and hemihydrate solvent molecules in the unit cell. The bond lengths in the title compound compare very well with those in the two structures in literature. The Pt–As bond length of 2.2730 (12) Å and the Pt–Cl bond length of 2.3401 (15) Å are similar to 2.2732 (3) and 2.3272 (8) Å for ODOHEW, and 2.2729 (2) and 2.3328 (6) Å for ODOHAS. The Pt–N bond lengths vary between 1.999 (4) and 2.005 (4) Å, the As–O bond lengths between 1.898 (3) and 2.107 (3) Å, and the As–OH bond lengths between 1.722 (3) and 1.738 (3) Å. Overall, these molecular structures compare well. When comparing the Pt–As and Pt–Cl bond lengths to those of other platinum(II) complexes where As and Cl are in *trans* positions, it is clear that the Pt–As bond lengths do not vary significantly and range between 2.3333 (6) and 2.3599 (2) Å, while for the Pt–Cl bond lengths a greater variation is seen, in a range from 2.2917 (4) to 2.3927 (5) Å (Reinholdt & Bendix, 2017; Clegg, 2016; Dube *et al.*, 2016; Imoto *et al.*, 2017; Muessig *et al.*, 2019). While the Pt–Cl length in (**1**) compares well with these *trans* complexes, the Pt–As bond length is somewhat smaller. The square-planar coordination around the central platinum(II) atom is distorted with N1–Pt1–N2 and N1–Pt1–As1 being 173.90 (13) and 85.18 (11)°, respectively, deviating from the expected 180 and 90°. The trigonal-bipyramidal coordination around the arsenic atom is significantly distorted with O4–As1–O6, O1–As1–O2 and O1–As1–Pt1 being 106.98 (14), 174.04 (11) and 95.35 (10)°, deviating from the ideal 120, 180 and 90°, respec-

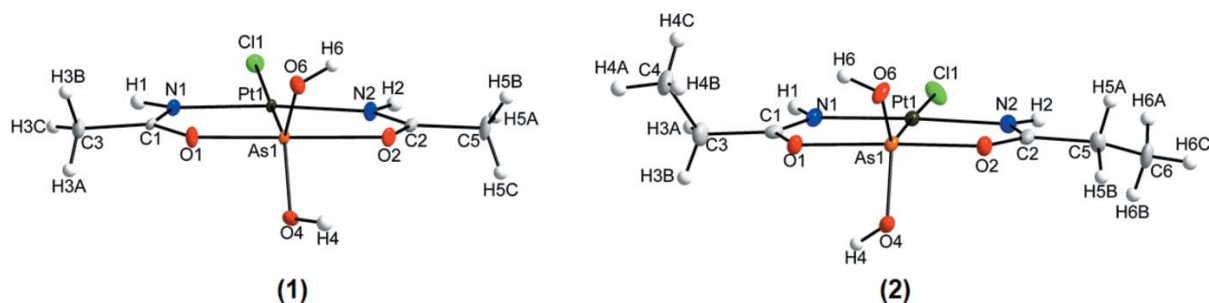


Figure 1

Molecular structures of (**1**) and (**2**), indicating the numbering schemes. Displacement ellipsoids are drawn at a probability level of 50%.

tively. Considering arsenic with a coordination number of 5, the index  $\tau_5$  parameter can be used to calculate any potential distortion (Addison *et al.*, 1984). The  $\tau_5$  parameter is defined as  $(\beta - \alpha)/60^\circ$  with  $\beta$  the largest and  $\alpha$  the second largest angle in the coordination sphere and was calculated as 0.794 for **(1)**, suggesting a significantly distorted trigonal-bipyramidal shape around arsenic ( $\tau_5 = 0$  for an ideal square pyramid and 1 for an ideal trigonal bipyramid).

The coordination environments of the platinum and arsenic atoms in **(2)** are the same as in **(1)**, *i.e.* Pt is coordinated by a chlorido ligand, two nitrogen donor atoms and arsenic, that is additionally bonded to two hydroxyl ligands and two oxygen donor atoms (Fig. 1). The Pt–As and Pt–Cl bond lengths of 2.2672 (8) Å and 2.3387 (11) Å in **(2)** are virtually identical with the bond lengths of 2.2687 (4) Å and 2.3361 (9) Å, respectively, in the orthorhombic polymorph reported by Miodragović *et al.* (2013). Again, these Pt–As and Pt–Cl bond lengths fit well into the ranges reported for other structures where As and Cl are in *trans* positions. The square-planar coordination environment around the platinum(II) atom is similarly distorted in the structures of the two polymorphs, with the ideal  $180^\circ$  (N–Pt–N) and  $90^\circ$  (N–Pt–Cl) angles deviating at  $173.59$  (13) and  $94.68$  (9)° for the structure determined by Miodragović *et al.* (2013) and  $173.20$  (14) (N1–Pt1–N2) and  $94.16$  (11)° (N1–Pt1–Cl1) for **(2)**, respectively. The largest deviation of the trigonal-bipyramidal coordination sphere of the arsenic atom in the polymorphic structures pertains to the Pt–As–OH angle, with reported values of  $129.78$  (10) and  $124.67$  (9)° for the orthorhombic structure (Miodragović *et al.*, 2013) and of  $130.05$  (11) and  $124.46$  (9)° for **(2)**. The  $\tau_5$  parameter for **(2)** is calculated as 0.711.

When comparing the molecules of **(1)** and **(2)**, it is clear that they do not differ much in terms of bond lengths and angles, with the only structural difference being the alkyl substituent on the ligand, *viz.* in **(1)** an ethyl and in **(2)** a propyl chain. The

bond lengths around platinum are all similar (Pt–As, Pt–Cl, Pt–N) as well as the two pairs of As–OH distances. There is a slight variation in the As–O bond lengths, 1.898 (3) and 2.107 (3) Å for **(1)** and 1.946 (3) and 1.979 (3) Å for **(2)**. The N1–Pt1–N2 bond angles are similar [ $173.90$  (13)° for **(1)** and  $173.20$  (14)° for **(2)**] while there are slight differences for the N–Pt1–As1 and N–Pt–Cl1 bond angles:  $85.18$  (11) and  $89.42$  (11)° for **(1)**, and  $87.25$  (10) and  $86.05$  (10)° for **(2)** (N1–Pt1–As1, N2–Pt1–As1), and  $93.28$  (11) and  $92.34$  (11)° for **(1)** and  $94.16$  (11) and  $92.53$  (10)° for **(2)** (N1–Pt1–Cl1, N2–Pt1–Cl1). The As1–Pt1–Cl1 bond angles also vary being  $174.79$  (3) and  $178.51$  (3)° for **(1)** and **(2)**. The bond angles around arsenic are all in a similar range but have more variation in some of the angles, for instance  $126.42$  (10) and  $130.05$  (11)° (O6–As1–Pt1),  $90.24$  (9) and  $94.20$  (9)° (O2–As1–Pt1), and  $95.35$  (10) and  $93.12$  (8)° (O1–As1–Pt1) for **(1)** and **(2)**, respectively. The trigonal-bipyramidal coordination environment around arsenic is distorted in both molecules with a  $\tau_5$  parameter value of 0.794 and 0.711 for **(1)** and **(2)**. Thus, the As atom in **(2)** shows a slightly higher distortion than in **(1)**.

#### 4. Supramolecular features

In the crystal structure of **(1)**, six hydrogen-bonding interactions are observed (Table 1), five intermolecular (N1–H1···O4<sup>i</sup>, N2–H2···O6<sup>ii</sup>, O4–H4···O2<sup>iii</sup>, O6–H6···Cl1<sup>iv</sup>, C3–H3B···Cl<sup>v</sup>) and one intramolecular (O6–H6···O2), as illustrated in Fig. 2. Bifurcation creates inter- and intramolecular interactions that can contribute to the stability of the structure. One of the donor hydrogen atoms (H6) takes part in hydrogen-bonding interactions to an oxygen atom (O2) and a chloride atom (Cl1) and forms an unsymmetrical bifurcated bond. Overall, the four stronger intermolecular hydrogen-bonding interactions sustain an infinite three-

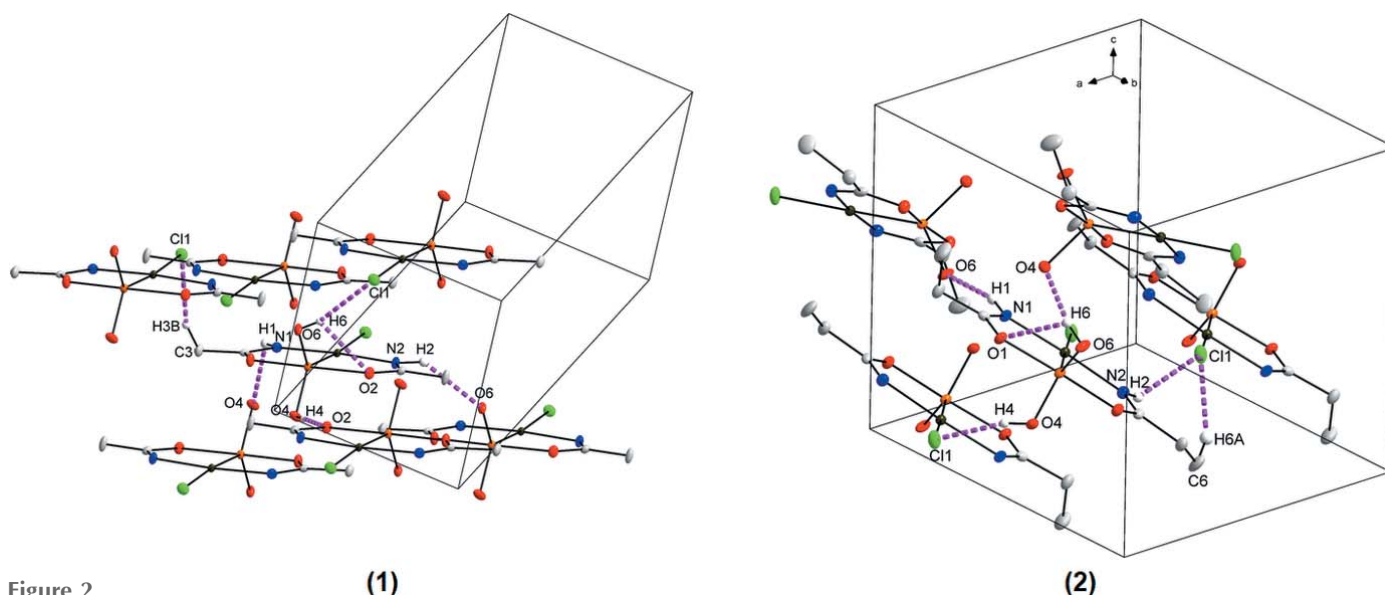


Figure 2

Hydrogen-bonding interactions (indicated by purple dashed lines) observed in the structures of **(1)** and **(2)**. Hydrogen atoms not involved in hydrogen-bonding interactions were omitted for clarity.



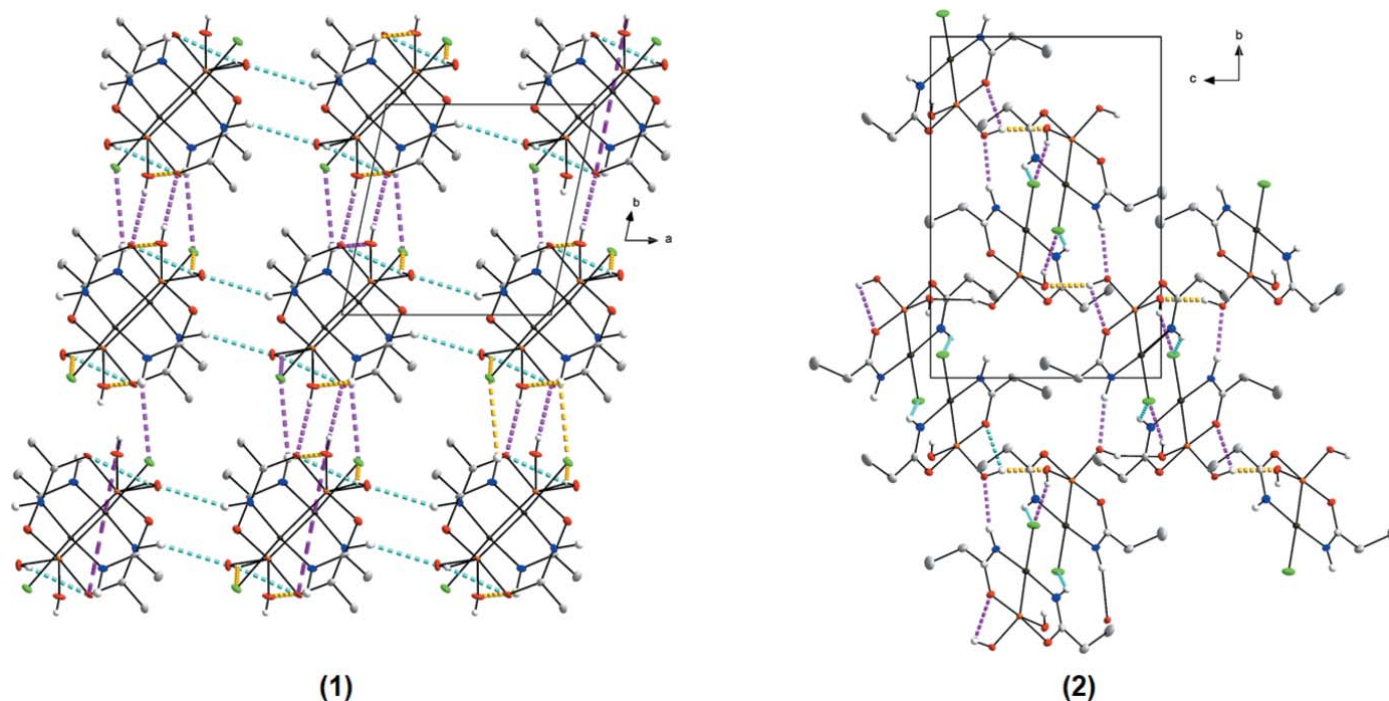


Figure 3

Illustration of the infinite three-dimensional frameworks formed by the hydrogen-bonding interactions in (1) and (2). Blue dashed lines indicate the infinite networks along the *a* axes, purple dashed lines along the *b* axes and gold dashed lines along the *c* axes. Hydrogen atoms not involved in the interactions were omitted for clarity.

dimensional framework (Fig. 3). An intermolecular cluster is formed from the strongest hydrogen-bonding interaction [ $O4 \cdots O2^{iii} = 2.750$  (4) Å], which generates an infinite chain along the *c*-axis direction (as can be seen in Fig. 3). Various  $\pi$ -interactions are also observed in (1), defined by the platinum(II) atom of one molecule to the centroid of the (Pt1,As1,C2,N2,O2) ring with a Pt $\cdots$ centroid distance of 3.7225 (7) Å, by the centroid of the (Pt1,As1,C2,N2,O2) ring to the centroid of the (Pt1,As1,C2,N2,O2) ( $-x, -y, 1-z$ ) ring

of an adjacent molecule with a distance of 3.7456 (4) Å, and by the centroid of the (Pt1,As1,C1,N1,O1) ring to the centroid of another (Pt1,As1,C1,N1,O2) ( $-x, -y, -z$ ) ring with a distance of 3.7960 (6) Å (Fig. 4). When viewed along the *c* axis, individual molecules pack in 'column-like' structures in an alternating head-to-tail fashion, as illustrated in Fig. 3.

The crystal structure of (2) is likewise stabilized by one intramolecular ( $O6-H6 \cdots O1$ ) and five intermolecular ( $N1-H1 \cdots O6^i$ ,  $N2-H2 \cdots Cl1^{iii}$ ,  $O4-H4 \cdots Cl1^{iii}$ ,  $O6-H6 \cdots O4^{iv}$ ,

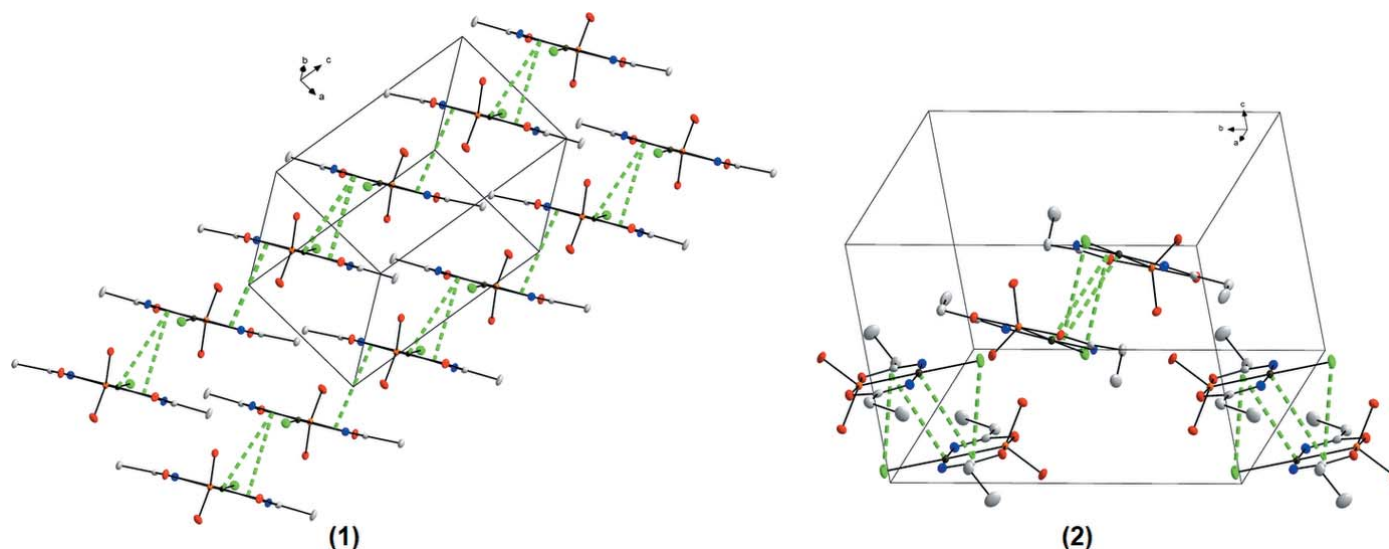


Figure 4

$\pi$ -interactions observed in the crystal structures of (1) and (2). Hydrogen atoms were omitted for clarity.

**Table 1**  
Hydrogen-bond geometry (Å, °) for **1**.

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
N1—H1···O4 <sup>i</sup>	0.901 (19)	2.50 (5)	3.077 (5)	122 (4)
N2—H2···O6 <sup>ii</sup>	0.90 (2)	2.51 (4)	3.280 (5)	143 (5)
O4—H4···O2 <sup>iii</sup>	0.821 (19)	1.93 (2)	2.750 (4)	173 (6)
O6—H6···O2	0.827 (19)	2.27 (5)	2.643 (4)	108 (4)
O6—H6···Cl1 <sup>iv</sup>	0.827 (19)	2.45 (3)	3.191 (3)	149 (5)
C3—H3B···Cl1 <sup>v</sup>	0.96	2.72	3.613 (5)	155

Symmetry codes: (i)  $-x, -y, -z$ ; (ii)  $x+1, y, z$ ; (iii)  $-x, -y-1, -z+1$ ; (iv)  $-x, -y, -z+1$ ; (v)  $x-1, y, z$ .

**Table 2**  
Hydrogen-bond geometry (Å, °) for **2**.

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
N1—H1···O6 <sup>i</sup>	0.88 (5)	2.19 (5)	3.041 (4)	163 (5)
N2—H2···Cl1 <sup>ii</sup>	0.854 (19)	2.71 (4)	3.408 (4)	140 (4)
O4—H4···Cl1 <sup>iii</sup>	0.82	2.28	3.071 (3)	161
O6—H6···O1	0.82	2.34	2.608 (4)	100
O6—H6···O4 <sup>iv</sup>	0.82	1.92	2.712 (4)	162
C6—H6A···Cl1 <sup>ii</sup>	0.96	2.82	3.735 (5)	159

Symmetry codes: (i)  $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$ ; (ii)  $-x, -y, -z$ ; (iii)  $-x+1, -y, -z$ ; (iv)  $x, -y+\frac{1}{2}, z+\frac{1}{2}$ .

C6—H6A···Cl1<sup>ii</sup>) hydrogen-bonding interactions (Table 2, Fig. 2), again with an unsymmetrical bifurcated hydrogen bond involving atom H6 (bonding to O1 and O4) and a resulting three-dimensional network structure, as illustrated in

**Table 3**  
Experimental details.

	(1)	2
Crystal data		
Chemical formula	[PtCl(C <sub>4</sub> H <sub>10</sub> AsN <sub>2</sub> O <sub>4</sub> )]	[Pt(C <sub>6</sub> H <sub>14</sub> AsN <sub>2</sub> O <sub>4</sub> )Cl]
<i>M<sub>r</sub></i>	455.59	483.65
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$	Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>c</i>
Temperature (K)	100	100
<i>a, b, c</i> (Å)	7.272 (1), 8.099 (1), 9.350 (2)	8.9009 (3), 14.1270 (5), 9.6438 (3)
$\alpha, \beta, \gamma$ (°)	66.588 (5), 83.993 (5), 76.737 (5)	90, 98.243 (2), 90
<i>V</i> (Å <sup>3</sup> )	491.81 (14)	1200.11 (9)
<i>Z</i>	2	4
Radiation type	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	17.86	14.65
Crystal size (mm)	0.39 × 0.29 × 0.14	0.55 × 0.42 × 0.08
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2012)	Multi-scan ( <i>SADABS</i> ; Bruker, 2012)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.003, 0.090	0.001, 0.301
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	8305, 2435, 2345	41298, 2891, 2700
<i>R<sub>int</sub></i>	0.039	0.047
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.667	0.661
Refinement		
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.020, 0.052, 1.18	0.022, 0.081, 1.05
No. of reflections	2411	2891
No. of parameters	136	148
No. of restraints	4	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ , $\Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.79, -2.05	1.53, -1.98

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz, 2005) and *WinGX* (Farrugia, 2012).

Fig. 3. In addition, Cl1 is the acceptor of two hydrogen-bonding interactions. Two weak  $\pi$ -interactions are also observed in (**2**), one from Pt1 to the centroid of Pt1,As1,O1,Cl1,N1 with a distance of 3.8213 (2) Å and the other from Cl1 to a symmetry-related centroid (Pt1,As1,O1,Cl1,N1; 1 - *x*, -*y*, -*z*) with a distance of 3.8252 (12) Å (Fig. 4).

In comparison, molecules in (**1**) and (**2**) pack differently due to the presence of different alkyl chains (Fig. 3).

## 5. Synthesis and crystallization

### Synthesis of 1

K<sub>2</sub>PtCl<sub>4</sub> (416 mg, 1 mmol) was added to a 125 ml solution of 9:1 (*v:v*) CH<sub>3</sub>CN/H<sub>2</sub>O. The mixture was stirred at 363 K. Once the K<sub>2</sub>PtCl<sub>4</sub> had dissolved, As<sub>2</sub>O<sub>3</sub> (405 mg, 2.05 mmol) was added to the solution and refluxed at 363 K for 48 h. The mixture was then filtered, and the filtrate was left to stand at room temperature. Crystals suitable for X-ray crystallography were obtained by slow evaporation. Yield: 301 mg (66%). <sup>1</sup>H NMR (300.18 MHz, dimethyl sulfoxide-*d*<sub>6</sub>):  $\sigma$  = 7.30 (OH, 2H,*s*), 6.69 (NH, 2H,*s*), 1.75 (CH<sub>3</sub>, 6H, *s*) ppm. <sup>13</sup>C NMR (150.95 MHz, dimethyl sulfoxide-*d*<sub>6</sub>):  $\sigma$  = 172 (CN), 23 (CH<sub>3</sub>) ppm. <sup>195</sup>Pt NMR (242.99 MHz, dimethyl sulfoxide-*d*<sub>6</sub>):  $\sigma$  = -3590.62 ppm. UV/Vis =  $\lambda$  = 285 nm,  $\epsilon$  = 4029 dm<sup>3</sup> mol<sup>-1</sup> cm<sup>-1</sup>. Analysis calculated: C, 10.55; H, 2.21; N, 6.15. Found: C, 10.64; H, 2.22; N, 6.09%.

### Synthesis of **2**

$K_2PtCl_4$  (422 mg, 1.02 mmol) was added to a 50 ml solution of 9:1 (v:v)  $H_2O/CH_3CH_2CN$ . The mixture was stirred at room temperature. Once the  $K_2PtCl_4$  had dissolved,  $As_2O_3$  (400 mg, 2.02 mmol) was added to the solution and was stirred at room temperature for 96 h. The solution was cooled in an ice bath and then filtered. The filtrate was left to stand at room temperature. Crystals suitable for X-ray crystallography were obtained by slow evaporation. Yield: 278 mg (56%).  $^1H$  NMR (600.28 MHz, dimethyl sulfoxide- $d_6$ ):  $\delta$  = 8.89 (OH, 2H, s), 7.99 (NH, 2H, s), 2.48 ( $CH_2$ , 4H, q), 1.04 ( $CH_3$ , 6H, t).  $^{13}C$  NMR (150.95 MHz, dimethyl sulfoxide- $d_6$ ):  $\delta$  = 176.18 (CN), 24.53 ( $CH_2$ ), 11.70 ( $CH_3$ ).  $^{195}Pt$  NMR (242.99 MHz, dimethyl sulfoxide- $d_6$ ) =  $-3591.52$ . UV/Vis:  $\lambda_{max}$  = 270 nm,  $\epsilon$  =  $4231 L mol^{-1} cm^{-1}$ . Analysis calculated C, 14.90; H, 2.92; N, 5.79. Found: C, 14.82; H, 2.91; N, 5.76.

### 6. Refinement

Crystal data and details of data collections and structure refinements are summarized in Table 3. Methyl and methylene hydrogen atoms were placed in geometrically idealized positions ( $C-H = 0.95-0.97 \text{ \AA}$ ) and constrained to ride on their parent atoms [ $U_{iso}(H) = 1.5U_{eq}(C)$  and  $1.2U_{eq}(C)$ ]. The OH and NH hydrogen atoms were located in a difference-Fourier map and their positional parameters were constrained with  $O-H = 0.84$  (2)  $\text{ \AA}$  and  $N-H = 0.89$  (2)  $\text{ \AA}$  for (**1**), and  $N-H = 0.87$  (2)  $\text{ \AA}$  for (**2**) with  $O-H$  distances fixed at 0.82  $\text{ \AA}$  and with  $U_{iso}(H) = 1.5U_{eq}(O)$ . For (**2**), the  $F_c$  versus  $F_o$  plot proved ten reflections to be outliers, and they were removed from the refinement as systematic errors.

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## supporting information

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## Crystal structures of chlorido[dihydroxybis(1-iminoethoxy)]arsanido- $\kappa^3N,As,N'$ platinum(II) and of a polymorph of chlorido[dihydroxybis(1-iminopropoxy)]arsanido- $\kappa^3N,As,N'$ platinum(II)

Nina R. Marogo, D.V. Kama, Hendrik G. Visser and M. Schutte-Smith

### Computing details

For both structures, data collection: *APEX2* (Bruker, 2012); cell refinement: *S SAINT* (Bruker, 2012); data reduction: *S SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

### Chlorido[dihydroxybis(1-iminoethoxy)]arsanido- $\kappa N,As,N'$ platinum(II) (1)

#### Crystal data

[Pt(C<sub>4</sub>H<sub>10</sub>AsN<sub>2</sub>O<sub>4</sub>)Cl]

$M_r = 455.59$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 7.272$  (1) Å

$b = 8.099$  (1) Å

$c = 9.350$  (2) Å

$\alpha = 66.588$  (5)°

$\beta = 83.993$  (5)°

$\gamma = 76.737$  (5)°

$V = 491.81$  (14) Å<sup>3</sup>

$Z = 2$

$F(000) = 416$

$D_x = 3.076$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å

Cell parameters from 6890 reflections

$\theta = 3.6$ – $28.3$ °

$\mu = 17.86$  mm<sup>-1</sup>

$T = 100$  K

Cuboid, colourless

$0.39 \times 0.29 \times 0.14$  mm

#### Data collection

Bruker APEXII CCD  
diffractometer

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2012)

$T_{\min} = 0.003$ ,  $T_{\max} = 0.090$

8305 measured reflections

2435 independent reflections

2345 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 28.3$ °,  $\theta_{\min} = 3.8$ °

$h = -9 \rightarrow 9$

$k = -8 \rightarrow 10$

$l = -12 \rightarrow 12$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.020$

$wR(F^2) = 0.052$

$S = 1.18$

2411 reflections

136 parameters

4 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent  
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.017P)^2 + 1.0064P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.79 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -2.04 \text{ e } \text{\AA}^{-3}$$

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.07024 (2)	0.05975 (2)	0.24814 (2)	0.00525 (6)
As1	-0.10555 (5)	-0.15921 (5)	0.31382 (4)	0.00631 (9)
Cl1	0.22859 (14)	0.30050 (13)	0.18937 (12)	0.01233 (19)
O4	-0.0662 (4)	-0.3392 (4)	0.2529 (3)	0.0115 (6)
O1	-0.2849 (4)	-0.0205 (4)	0.1539 (3)	0.0113 (6)
C3	-0.3872 (6)	0.2425 (6)	-0.0760 (5)	0.0130 (8)
H3B	-0.508229	0.285642	-0.037137	0.019*
H3A	-0.400172	0.162257	-0.125455	0.019*
H3C	-0.339248	0.345439	-0.150284	0.019*
C5	0.3527 (6)	-0.3940 (6)	0.6461 (5)	0.0136 (8)
H5A	0.451272	-0.333216	0.646809	0.02*
H5C	0.407126	-0.505519	0.630569	0.02*
H5B	0.285185	-0.422631	0.743999	0.02*
O2	0.0724 (4)	-0.3296 (4)	0.5035 (3)	0.0098 (6)
N2	0.2483 (5)	-0.1101 (5)	0.4204 (4)	0.0098 (7)
O6	-0.2912 (4)	-0.1890 (4)	0.4486 (3)	0.0109 (6)
N1	-0.1134 (5)	0.2051 (5)	0.0754 (4)	0.0099 (7)
C1	-0.2535 (6)	0.1403 (5)	0.0559 (4)	0.0083 (7)
C2	0.2192 (6)	-0.2701 (5)	0.5165 (5)	0.0090 (7)
H2	0.355 (5)	-0.091 (8)	0.448 (6)	0.025 (15)*
H1	-0.113 (8)	0.314 (4)	-0.004 (5)	0.025 (15)*
H6	-0.236 (7)	-0.202 (7)	0.527 (4)	0.017 (13)*
H4	-0.059 (8)	-0.438 (4)	0.328 (5)	0.026 (15)*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pt1	0.00569 (9)	0.00337 (9)	0.00559 (9)	-0.00199 (6)	-0.00017 (6)	-0.00001 (6)
As1	0.00695 (19)	0.00417 (19)	0.00666 (18)	-0.00267 (14)	-0.00050 (14)	0.00001 (14)
Cl1	0.0149 (5)	0.0096 (4)	0.0132 (4)	-0.0071 (4)	0.0013 (4)	-0.0029 (4)
O4	0.0210 (15)	0.0055 (13)	0.0089 (13)	-0.0049 (11)	-0.0006 (11)	-0.0024 (11)
O1	0.0107 (14)	0.0101 (14)	0.0100 (13)	-0.0045 (11)	-0.0032 (11)	0.0013 (11)
C3	0.0101 (19)	0.014 (2)	0.0105 (18)	0.0002 (15)	-0.0032 (15)	-0.0015 (15)
C5	0.0084 (18)	0.0122 (19)	0.0143 (19)	-0.0030 (15)	-0.0055 (15)	0.0029 (15)
O2	0.0107 (14)	0.0059 (13)	0.0098 (13)	-0.0049 (10)	-0.0003 (11)	0.0016 (11)
N2	0.0068 (16)	0.0103 (17)	0.0109 (16)	-0.0039 (13)	-0.0017 (13)	-0.0010 (13)



O6	0.0062 (13)	0.0161 (14)	0.0095 (13)	-0.0038 (11)	0.0006 (11)	-0.0034 (11)
N1	0.0128 (17)	0.0059 (16)	0.0091 (16)	-0.0024 (13)	-0.0009 (13)	-0.0005 (13)
C1	0.0108 (18)	0.0048 (17)	0.0065 (17)	0.0022 (14)	-0.0007 (14)	-0.0013 (14)
C2	0.0082 (18)	0.0074 (18)	0.0092 (17)	0.0002 (14)	0.0004 (14)	-0.0021 (14)

*Geometric parameters (Å, °)*

Pt1—N1	1.999 (4)	C3—H3A	0.96
Pt1—N2	2.005 (4)	C3—H3C	0.96
Pt1—As1	2.2730 (12)	C5—C2	1.501 (5)
Pt1—C11	2.3401 (15)	C5—H5A	0.96
As1—O4	1.722 (3)	C5—H5C	0.96
As1—O6	1.738 (3)	C5—H5B	0.96
As1—O1	1.898 (3)	O2—C2	1.302 (5)
As1—O2	2.107 (3)	N2—C2	1.301 (5)
O4—H4	0.821 (19)	N2—H2	0.90 (2)
O1—C1	1.316 (5)	O6—H6	0.827 (19)
C3—C1	1.491 (5)	N1—C1	1.306 (5)
C3—H3B	0.96	N1—H1	0.901 (19)
N1—Pt1—N2	173.90 (13)	H3B—C3—H3C	109.5
N1—Pt1—As1	85.18 (11)	H3A—C3—H3C	109.5
N2—Pt1—As1	89.42 (11)	C2—C5—H5A	109.5
N1—Pt1—C11	93.28 (11)	C2—C5—H5C	109.5
N2—Pt1—C11	92.34 (11)	H5A—C5—H5C	109.5
As1—Pt1—C11	174.79 (3)	C2—C5—H5B	109.5
O4—As1—O6	106.98 (14)	H5A—C5—H5B	109.5
O4—As1—O1	90.06 (14)	H5C—C5—H5B	109.5
O6—As1—O1	88.71 (14)	C2—O2—As1	116.5 (2)
O4—As1—O2	88.29 (13)	C2—N2—Pt1	122.5 (3)
O6—As1—O2	86.29 (13)	C2—N2—H2	109 (4)
O1—As1—O2	174.04 (11)	Pt1—N2—H2	129 (4)
O4—As1—Pt1	126.37 (10)	As1—O6—H6	100 (4)
O6—As1—Pt1	126.42 (10)	C1—N1—Pt1	121.7 (3)
O1—As1—Pt1	95.35 (10)	C1—N1—H1	109 (4)
O2—As1—Pt1	90.24 (9)	Pt1—N1—H1	130 (4)
As1—O4—H4	111 (4)	N1—C1—O1	121.2 (4)
C1—O1—As1	116.4 (3)	N1—C1—C3	122.7 (4)
C1—C3—H3B	109.5	O1—C1—C3	116.1 (4)
C1—C3—H3A	109.5	N2—C2—O2	121.1 (4)
H3B—C3—H3A	109.5	N2—C2—C5	121.7 (4)
C1—C3—H3C	109.5	O2—C2—C5	117.2 (3)
O4—As1—O1—C1	-123.0 (3)	As1—O1—C1—C3	175.8 (3)
O6—As1—O1—C1	130.0 (3)	Pt1—N2—C2—O2	1.2 (6)
Pt1—As1—O1—C1	3.5 (3)	Pt1—N2—C2—C5	-179.1 (3)
Pt1—N1—C1—O1	3.2 (6)	As1—O2—C2—N2	2.8 (5)
Pt1—N1—C1—C3	-177.2 (3)	As1—O2—C2—C5	-177.0 (3)

As1—O1—C1—N1                      -4.6 (5)

*Hydrogen-bond geometry* (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1···O4 <sup>i</sup>	0.901 (19)	2.50 (5)	3.077 (5)	122 (4)
N2—H2···O6 <sup>ii</sup>	0.90 (2)	2.51 (4)	3.280 (5)	143 (5)
O4—H4···O2 <sup>iii</sup>	0.821 (19)	1.93 (2)	2.750 (4)	173 (6)
O6—H6···O2	0.827 (19)	2.27 (5)	2.643 (4)	108 (4)
O6—H6···C11 <sup>iv</sup>	0.827 (19)	2.45 (3)	3.191 (3)	149 (5)
C3—H3B···C11 <sup>v</sup>	0.96	2.72	3.613 (5)	155

Symmetry codes: (i)  $-x, -y, -z$ ; (ii)  $x+1, y, z$ ; (iii)  $-x, -y-1, -z+1$ ; (iv)  $-x, -y, -z+1$ ; (v)  $x-1, y, z$ .**Chlorido[dihydroxybis(1-iminopropoxy)arsanido- $\kappa N, As, N'$ ]platinum(II) (2)***Crystal data*[Pt(C<sub>6</sub>H<sub>14</sub>AsN<sub>2</sub>O<sub>4</sub>)Cl] $M_r = 483.65$ Monoclinic,  $P2_1/c$ 

Hall symbol: -P 2ybc

 $a = 8.9009$  (3) Å $b = 14.1270$  (5) Å $c = 9.6438$  (3) Å $\beta = 98.243$  (2)° $V = 1200.11$  (9) Å<sup>3</sup> $Z = 4$  $F(000) = 896$  $D_x = 2.677$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å

Cell parameters from 9966 reflections

 $\theta = 3.6$ – $28.4$ ° $\mu = 14.65$  mm<sup>-1</sup> $T = 100$  K

Plate, colourless

 $0.55 \times 0.42 \times 0.08$  mm*Data collection*

Bruker APEXII CCD

diffractometer

 $\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Bruker, 2012)

 $T_{\min} = 0.001$ ,  $T_{\max} = 0.301$ 

41298 measured reflections

2891 independent reflections

2700 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.047$  $\theta_{\max} = 28.0$ °,  $\theta_{\min} = 4.4$ ° $h = -11$ → $11$  $k = -18$ → $18$  $l = -12$ → $12$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.022$  $wR(F^2) = 0.081$  $S = 1.05$ 

2891 reflections

148 parameters

1 restraint

Primary atom site location: heavy-atom method

Hydrogen site location: mixed

H atoms treated by a mixture of independent

and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 1.201P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 1.53$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -1.97$  e Å<sup>-3</sup>*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.33235 (2)	0.06675 (2)	0.08616 (2)	0.00918 (9)
As1	0.47791 (4)	0.19900 (3)	0.11318 (4)	0.00918 (11)
Cl1	0.17867 (12)	-0.06796 (6)	0.05304 (12)	0.0183 (2)
O4	0.6100 (3)	0.2309 (2)	0.0038 (3)	0.0127 (5)
H4	0.676165	0.190233	0.007601	0.019*
O1	0.6421 (3)	0.13513 (19)	0.2392 (3)	0.0127 (5)
O6	0.4810 (3)	0.2897 (2)	0.2331 (3)	0.0150 (6)
H6	0.524135	0.27179	0.309457	0.022*
C5	0.1062 (5)	0.2938 (3)	-0.1667 (4)	0.0168 (8)
H5B	0.167052	0.336047	-0.21515	0.02*
H5A	0.047602	0.332619	-0.110998	0.02*
C2	0.2107 (5)	0.2323 (3)	-0.0697 (4)	0.0127 (7)
N2	0.1856 (4)	0.1445 (2)	-0.0450 (3)	0.0134 (6)
C4	0.7743 (6)	0.0425 (4)	0.5043 (5)	0.0297 (10)
H4B	0.79075	0.109321	0.495994	0.045*
H4A	0.861994	0.014145	0.557876	0.045*
H4C	0.68723	0.032054	0.550648	0.045*
O2	0.3329 (3)	0.27611 (19)	-0.0081 (3)	0.0130 (5)
C1	0.6203 (5)	0.0463 (3)	0.2667 (4)	0.0119 (7)
C3	0.7473 (5)	-0.0015 (3)	0.3597 (4)	0.0180 (8)
H3B	0.8395	0.002883	0.317329	0.022*
H3A	0.723211	-0.068067	0.368096	0.022*
N1	0.4959 (4)	0.0020 (2)	0.2166 (3)	0.0129 (6)
C6	-0.0037 (5)	0.2407 (3)	-0.2757 (5)	0.0260 (10)
H6B	0.052289	0.198648	-0.327478	0.039*
H6C	-0.058897	0.285263	-0.338787	0.039*
H6A	-0.073555	0.204823	-0.229549	0.039*
H1	0.489 (6)	-0.057 (4)	0.244 (6)	0.026 (17)*
H2	0.106 (4)	0.120 (4)	-0.090 (5)	0.023 (14)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pt1	0.01054 (13)	0.00700 (12)	0.00989 (12)	-0.00034 (4)	0.00112 (8)	-0.00025 (4)
As1	0.0127 (2)	0.00657 (19)	0.00790 (18)	-0.00061 (13)	0.00022 (15)	-0.00002 (12)
Cl1	0.0126 (5)	0.0103 (5)	0.0318 (6)	-0.0023 (3)	0.0029 (4)	-0.0018 (3)
O4	0.0132 (14)	0.0138 (12)	0.0109 (11)	-0.0004 (10)	0.0009 (10)	0.0029 (10)
O1	0.0149 (14)	0.0097 (12)	0.0127 (12)	-0.0006 (10)	-0.0008 (10)	0.0028 (10)
O6	0.0258 (16)	0.0084 (13)	0.0092 (12)	0.0020 (11)	-0.0026 (11)	-0.0014 (9)
C5	0.014 (2)	0.0172 (19)	0.0180 (18)	0.0012 (15)	-0.0003 (15)	0.0040 (15)
C2	0.0152 (19)	0.0142 (18)	0.0086 (15)	0.0040 (15)	0.0014 (13)	-0.0010 (13)
N2	0.0125 (16)	0.0132 (16)	0.0143 (15)	-0.0009 (13)	0.0008 (13)	0.0009 (12)
C4	0.032 (3)	0.035 (3)	0.019 (2)	0.010 (2)	-0.0082 (19)	-0.001 (2)
O2	0.0139 (14)	0.0104 (12)	0.0139 (12)	-0.0002 (10)	-0.0010 (10)	0.0018 (10)
C1	0.016 (2)	0.0103 (16)	0.0107 (16)	0.0016 (15)	0.0053 (14)	-0.0001 (13)

C3	0.020 (2)	0.0132 (18)	0.0191 (18)	0.0038 (15)	-0.0020 (16)	0.0029 (14)
N1	0.0178 (17)	0.0085 (15)	0.0122 (14)	0.0028 (12)	0.0013 (12)	0.0019 (12)
C6	0.023 (2)	0.029 (2)	0.022 (2)	0.0019 (18)	-0.0081 (18)	0.0108 (18)

*Geometric parameters (Å, °)*

Pt1—N1	2.003 (3)	C2—N2	1.289 (5)
Pt1—N2	2.010 (3)	C2—O2	1.316 (5)
Pt1—As1	2.2672 (8)	N2—H2	0.854 (19)
Pt1—Cl1	2.3387 (11)	C4—C3	1.514 (6)
As1—O6	1.724 (3)	C4—H4B	0.96
As1—O4	1.747 (3)	C4—H4A	0.96
As1—O2	1.946 (3)	C4—H4C	0.96
As1—O1	1.979 (3)	C1—N1	1.303 (5)
O4—H4	0.82	C1—C3	1.500 (5)
O1—C1	1.303 (4)	C3—H3B	0.97
O6—H6	0.82	C3—H3A	0.97
C5—C2	1.498 (5)	N1—H1	0.88 (5)
C5—C6	1.527 (6)	C6—H6B	0.96
C5—H5B	0.97	C6—H6C	0.96
C5—H5A	0.97	C6—H6A	0.96
N1—Pt1—N2	173.20 (14)	C2—N2—Pt1	121.9 (3)
N1—Pt1—As1	87.25 (10)	C2—N2—H2	117 (4)
N2—Pt1—As1	86.05 (10)	Pt1—N2—H2	121 (4)
N1—Pt1—Cl1	94.16 (11)	C3—C4—H4B	109.5
N2—Pt1—Cl1	92.53 (10)	C3—C4—H4A	109.5
As1—Pt1—Cl1	178.51 (3)	H4B—C4—H4A	109.5
O6—As1—O4	105.44 (14)	C3—C4—H4C	109.5
O6—As1—O2	86.12 (13)	H4B—C4—H4C	109.5
O4—As1—O2	86.48 (13)	H4A—C4—H4C	109.5
O6—As1—O1	89.28 (12)	C2—O2—As1	116.2 (2)
O4—As1—O1	89.27 (13)	O1—C1—N1	122.0 (4)
O2—As1—O1	172.68 (11)	O1—C1—C3	115.7 (4)
O6—As1—Pt1	130.05 (11)	N1—C1—C3	122.2 (4)
O4—As1—Pt1	124.46 (9)	C1—C3—C4	111.8 (4)
O2—As1—Pt1	94.20 (9)	C1—C3—H3B	109.3
O1—As1—Pt1	93.12 (8)	C4—C3—H3B	109.3
As1—O4—H4	109.5	C1—C3—H3A	109.3
C1—O1—As1	116.5 (2)	C4—C3—H3A	109.3
As1—O6—H6	109.5	H3B—C3—H3A	107.9
C2—C5—C6	115.1 (3)	C1—N1—Pt1	121.1 (3)
C2—C5—H5B	108.5	C1—N1—H1	116 (4)
C6—C5—H5B	108.5	Pt1—N1—H1	123 (4)
C2—C5—H5A	108.5	C5—C6—H6B	109.5
C6—C5—H5A	108.5	C5—C6—H6C	109.5
H5B—C5—H5A	107.5	H6B—C6—H6C	109.5
N2—C2—O2	121.5 (3)	C5—C6—H6A	109.5



N2—C2—C5	124.4 (4)	H6B—C6—H6A	109.5
O2—C2—C5	114.1 (3)	H6C—C6—H6A	109.5
C6—C5—C2—N2	-21.3 (6)	As1—O1—C1—N1	-1.4 (5)
C6—C5—C2—O2	159.6 (3)	As1—O1—C1—C3	178.6 (3)
O2—C2—N2—Pt1	-1.3 (5)	O1—C1—C3—C4	63.1 (5)
C5—C2—N2—Pt1	179.8 (3)	N1—C1—C3—C4	-116.9 (4)
N2—C2—O2—As1	3.5 (5)	O1—C1—N1—Pt1	2.7 (5)
C5—C2—O2—As1	-177.5 (2)	C3—C1—N1—Pt1	-177.4 (3)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ O6 <sup>i</sup>	0.88 (5)	2.19 (5)	3.041 (4)	163 (5)
N2—H2 $\cdots$ C11 <sup>ii</sup>	0.854 (19)	2.71 (4)	3.408 (4)	140 (4)
O4—H4 $\cdots$ C11 <sup>iii</sup>	0.82	2.28	3.071 (3)	161
O6—H6 $\cdots$ O1	0.82	2.34	2.608 (4)	100
O6—H6 $\cdots$ O4 <sup>iv</sup>	0.82	1.92	2.712 (4)	162
C6—H6A $\cdots$ C11 <sup>ii</sup>	0.96	2.82	3.735 (5)	159

Symmetry codes: (i)  $-x+1, y-1/2, -z+1/2$ ; (ii)  $-x, -y, -z$ ; (iii)  $-x+1, -y, -z$ ; (iv)  $x, -y+1/2, z+1/2$ .