



# Crystallization experiments with the dinuclear chelate ring complex di- $\mu$ -chlorido-bis[( $\eta^2$ -2-allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy)phenyl- $\kappa C^1$ )platinum(II)]

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Crystallization experiments with the dinuclear chelate ring complex di- $\mu$ -chlorido-bis[( $\eta^2$ -2-allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy)phenyl- $\kappa C^1$ )platinum(II)], [Pt<sub>2</sub>(C<sub>15</sub>H<sub>19</sub>O<sub>4</sub>)<sub>2</sub>Cl<sub>2</sub>], containing a derivative of the natural compound eugenol as ligand, have been performed. Using five different sets of crystallization conditions resulted in four different complexes which can be further used as starting compounds for the synthesis of Pt complexes with promising anticancer activities. In the case of vapour diffusion with the binary chloroform–diethyl ether or methylene chloride–diethyl ether systems, no change of the molecular structure was observed. Using evaporation from acetonitrile (at room temperature), dimethylformamide (DMF, at 313 K) or dimethyl sulfoxide (DMSO, at 313 K), however, resulted in the displacement of a chloride ligand by the solvent, giving, respectively, the mononuclear complexes (acetonitrile- $\kappa N$ )( $\eta^2$ -2-allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy)phenyl- $\kappa C^1$ )chloridoplatinum(II) monohydrate, [Pt(C<sub>15</sub>H<sub>19</sub>O<sub>4</sub>)Cl(CH<sub>3</sub>CN)]·H<sub>2</sub>O, ( $\eta^2$ -2-allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy)phenyl- $\kappa C^1$ )chlorido(dimethylformamide- $\kappa O$ )platinum(II), [Pt(C<sub>15</sub>H<sub>19</sub>O<sub>4</sub>)Cl(C<sub>2</sub>H<sub>7</sub>NO)], and ( $\eta^2$ -2-allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy)phenyl- $\kappa C^1$ )chlorido(dimethyl sulfoxide- $\kappa S$ )platinum(II), determined as the analogue ( $\eta^2$ -2-allyl-4-methoxy-5-[(ethoxycarbonyl)methoxy]phenyl- $\kappa C^1$ )chlorido(dimethyl sulfoxide- $\kappa S$ )platinum(II), [Pt(C<sub>14</sub>H<sub>17</sub>O<sub>4</sub>)Cl(C<sub>2</sub>H<sub>6</sub>OS)]. The crystal structures confirm that acetonitrile interacts with the Pt<sup>II</sup> atom *via* its N atom, while for DMSO, the S atom is the coordinating atom. For the replacement, the longest of the two Pt–Cl bonds is cleaved, leading to a *cis* position of the solvent ligand with respect to the allyl group. The crystal packing of the complexes is characterized by dimer formation *via* C–H···O and C–H··· $\pi$  interactions, but no  $\pi$ – $\pi$  interactions are observed despite the presence of the aromatic ring.

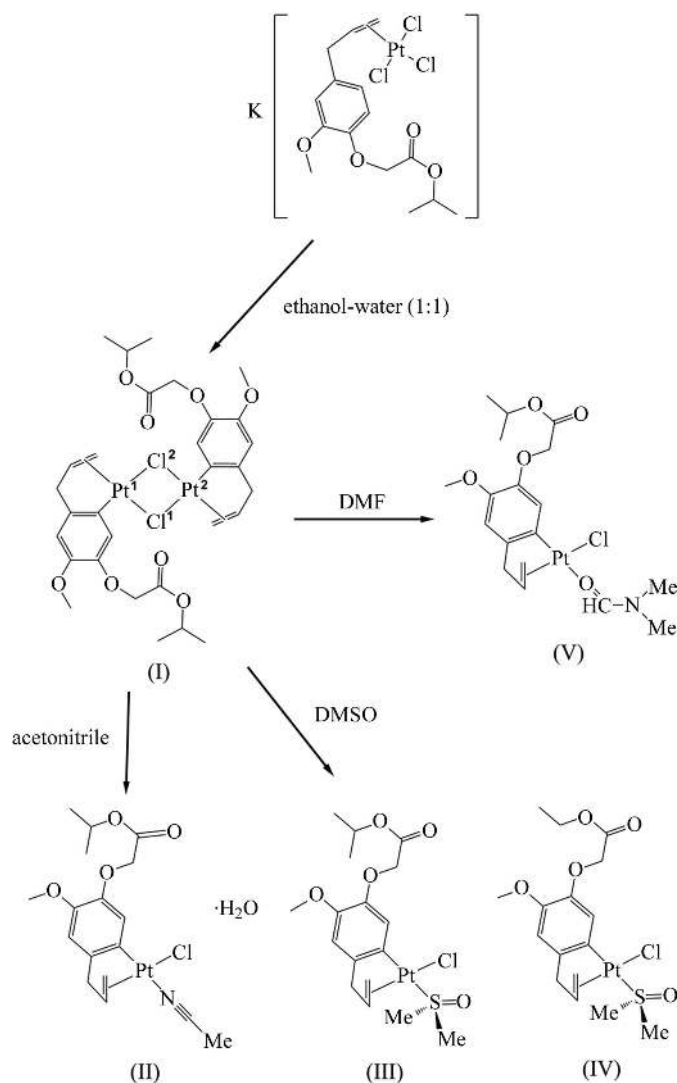
## 1. Introduction

Platinum-based drugs are irreplaceable in cancer chemotherapy, including three globally approved generations, namely cisplatin, carboplatin and oxaliplatin. Due to their high toxicity, these drugs still exert undesirable side effects (Klein & Hambley, 2009; Wilson & Lippard, 2014). This induces scientists to look for more effective platinum complexes. Recently, the strategy of introducing a natural compound as ligand in Pt<sup>II</sup> complexes in order to reduce their toxicity has attracted much attention (Sally & John, 2002; Romerosa *et al.*, 2004; Leite *et al.*, 2004; Da *et al.*, 2015). Olefins, such as safrole extracted from sassafras oil and some derivatives of eugenol extracted from clove basil oil, for instance, have been used to prepare dinuclear complexes of



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the type  $[\text{Pt}_2(\text{olefin-1H})_2\text{Cl}_2]$  (where olefin-1H indicates the monoanionic form of the parent olefin), which are critical in the production of many other  $\text{Pt}^{\text{II}}$  complexes with promising anticancer activities (Da *et al.*, 2010, 2012, 2015; Thong & Chi, 2014). Nevertheless, no crystallographic data are available for these key complexes. In this paper, we describe crystallization experiments with the sterically bulky complex di- $\mu$ -chlorido-bis[ $(\eta^2$ -2-allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy)phenyl- $\kappa^{\text{C}^1}$ ]platinum(II)],  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$ , (I), under five different sets of conditions, namely vapour diffusion



with the binary  $\text{CHCl}_3$ - $\text{Et}_2\text{O}$  and  $\text{CH}_2\text{Cl}_2$ - $\text{Et}_2\text{O}$  systems, and slow evaporation from  $\text{CH}_3\text{CN}$ , dimethylformamide (DMF) or dimethyl sulfoxide (DMSO) solution. The crystal structures, as well as the molecular arrangements, of the resulting compounds crystallized from  $\text{CHCl}_3$ - $\text{Et}_2\text{O}$  and  $\text{CH}_3\text{CN}$  are analyzed, *i.e.* compounds (I) and (II), respectively. To verify the product of the crystallization of  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$  from DMSO, the related complex  $[\text{Pt}(\text{Eteug-1H})\text{Cl}(\text{DMSO})]$  [Eteug is ethyl 2-(4-allyl-2-methoxyphenoxy)acetate], (IV) (see Scheme), is also investigated for comparison.

## 2. Experimental

### 2.1. Synthesis and crystallization

A lemon-yellow powder of the complex  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$ , (I), was synthesized according to the method of Thong & Chi (2014). Crystals of the complex were grown under different conditions, namely vapour diffusion using  $\text{CHCl}_3$ - $\text{Et}_2\text{O}$  and  $\text{CH}_2\text{Cl}_2$ - $\text{Et}_2\text{O}$ , and slow evaporation from  $\text{CH}_3\text{CN}$ , DMSO and DMF, to give four different compounds, namely  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$ , (I),  $[\text{Pt}(\text{isoPreug-1H})\text{Cl}(\text{CH}_3\text{CN})]\cdot\text{H}_2\text{O}$ , (II),  $[\text{Pt}(\text{isoPreug-1H})\text{Cl}(\text{DMSO})]$ , (III), the ethyl analogue of (III), *i.e.*  $[\text{Pt}(\text{Eteug-1H})\text{Cl}(\text{DMSO})]$ , (IV), and  $[\text{Pt}(\text{isoPreug-1H})\text{Cl}(\text{DMF})]$ , (V).

**2.1.1. Crystallization of  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$ , (I).** A saturated solution of the  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$  powder in chloroform was filtered to obtain a clear solution. The solution was left under an atmosphere of diethyl ether at room temperature and lemon-yellow single crystals suitable for X-ray diffraction analysis appeared after 5 h. The platinum analysis and analysis of the ESI-MS, IR and  $^1\text{H}$  NMR spectra of (I) were described by Thong & Chi (2014).

A saturated solution of the  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$  powder in  $\text{CH}_2\text{Cl}_2$  was filtered to obtain a clear solution. The solution was left under an atmosphere of diethyl ether at room temperature and lemon-yellow small crystals appeared after 7 h. The platinum analysis and the IR and ESI-MS spectra were the same as those of (I) above.

**2.1.2. Crystallization of  $[\text{Pt}(\text{isoPreug-1H})\text{Cl}(\text{CH}_3\text{CN})]\cdot\text{H}_2\text{O}$ , (II).** A saturated solution of the  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$  powder in acetonitrile was filtered to obtain a clear solution. Pale-yellow crystals were obtained after slow evaporation of the solvent under ambient conditions for 12 h and these crystals were suitable for X-ray diffraction.

%Pt (calculated/found): 35.32/35.40. Positive ESI-MS:  $m/z$  1010  $[2M - 2\text{H}_2\text{O} - 2\text{CH}_3\text{CN} + \text{Na}]^+$ . IR (KBr,  $\text{cm}^{-1}$ ): 3577, 3500 ( $\nu_{\text{OH}}$ ); 2986, 2922 ( $\nu_{\text{CH}}$ ); 2001 ( $\nu_{\text{CN}}$ ); 1731 ( $\nu_{\text{C=O}}$ ), 1642 ( $\delta_{\text{HOH}}$ ), 1590, 1490 ( $\nu_{\text{C=C}}$ ).  $^1\text{H}$  NMR (500 MHz,  $\text{CD}_3\text{CN}$ ):  $\delta$  6.79 (1H, *s*,  $^3J_{\text{Pt-H}} = 45$  Hz, Ar-H), 6.68 (1H, *s*, Ar-H), 5.05 [1H, *m*,  $\text{CH}(\text{CH}_3)_2$ ], 5.01 (1H, *m*,  $^2J_{\text{Pt-H}} = 70$  Hz,  $\text{CH}=\text{CH}_2$ ), 4.60 (2H, *s*,  $\text{OCH}_2$ ), 4.18 (1H, *d*,  $^3J = 7.5$ ,  $^2J_{\text{Pt-H}} = 75$  Hz,  $\text{CH}=\text{CH}_2$ ), 3.90 (1H, *d*,  $^3J = 13.0$ ,  $^2J_{\text{Pt-H}} = 74$  Hz,  $\text{CH}=\text{CH}_2$ ), 3.79 (4H, *ov*,  $\text{OCH}_3$ ,  $\text{CH}_2\text{CH}$ ), 2.70 (1H, *d*,  $^2J = 17.0$ ,  $^3J_{\text{Pt-H}} = 110$  Hz,  $\text{CH}_2\text{CH}$ ), 1.95 (3H, *ov*,  $\text{CH}_3\text{CN}$ ), 1.25 [6H, *d*,  $^3J = 7.0$  Hz,  $\text{CH}(\text{CH}_3)_2$ ].

**2.1.3. Crystallization of  $[\text{Pt}(\text{isoPreug-1H})\text{Cl}(\text{DMSO})]$ , (III).**  $[\text{Pt}_2(\text{isoPreug-1H})_2\text{Cl}_2]$  powder was dissolved in DMSO to afford a clear solution and small light-yellow crystals were obtained after evaporating the solvent at 313 K for 36 h.

%Pt (calculated/found) 34.09/34.20. Positive ESI-MS:  $m/z$  574  $[M + \text{H}]^+$ . IR (KBr,  $\text{cm}^{-1}$ ): 3000, 2900, 2830 ( $\nu_{\text{CH}}$ ); 1750 ( $\nu_{\text{C=O}}$ ); 1590, 1480 ( $\nu_{\text{C=C}}$ ), 1190 ( $\nu_{\text{S=O}}$ ).  $^1\text{H}$  NMR [500 MHz,  $(\text{CD}_3)_2\text{SO}$ ]:  $\delta$  6.75 (2H, *ov*, Ar-H), 5.09 (1H, *br*,  $^2J_{\text{Pt-H}} = 70$  Hz,  $\text{CH}=\text{CH}_2$ ), 4.99 [1H, *m*,  $\text{CH}(\text{CH}_3)_2$ ], 4.55 (1H, *d*,  $^2J = 16.5$  Hz,  $\text{OCH}_2$ ), 4.52 (1H, *d*,  $^2J = 16.5$  Hz,  $\text{OCH}_2$ ), 4.33 (1H, *br*,  $^2J_{\text{Pt-H}} = 73$  Hz,  $\text{CH}=\text{CH}_2$ ), 3.93 (1H, *d*,  $^3J = 13.5$ ,  $^2J_{\text{Pt-H}} = 72$  Hz,  $\text{CH}=\text{CH}_2$ ), 3.70 (4H, *ov*,  $\text{OCH}_3$ ,  $\text{CH}_2\text{CH}$ ), 2.80 (1H, *d*,  $^2J = 17.0$ ,  $^3J_{\text{Pt-H}} = 112$  Hz,  $\text{CH}_2\text{CH}$ ), 2.55 [6H, *s*,  $(\text{CH}_3)_2\text{SO}$ ], 1.23

**Table 1**  
Experimental details.

	(I)	(II)	(IV)
Crystal data			
Chemical formula	[Pt <sub>2</sub> (C <sub>15</sub> H <sub>19</sub> O <sub>4</sub> ) <sub>2</sub> Cl <sub>2</sub> ]	[Pt(C <sub>15</sub> H <sub>19</sub> ClO <sub>4</sub> )Cl(C <sub>2</sub> H <sub>3</sub> N)]·H <sub>2</sub> O	[Pt(C <sub>14</sub> H <sub>17</sub> O <sub>4</sub> )Cl(C <sub>2</sub> H <sub>6</sub> OS)]
<i>M<sub>r</sub></i>	987.66	552.91	557.94
Crystal system, space group	Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>n</i>	Triclinic, <i>P</i> $\bar{1}$	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	200	100	101
<i>a</i> , <i>b</i> , <i>c</i> (Å)	13.5600 (3), 7.98332 (19), 15.8057 (4)	8.3747 (4), 10.5732 (5), 11.0883 (4)	8.7920 (3), 12.1383 (4), 17.4745 (6)
$\alpha$ , $\beta$ , $\gamma$ (°)	90, 104.815 (3), 90	76.670 (4), 89.496 (3), 82.641 (4)	87.030 (3), 82.074 (3), 87.651 (3)
<i>V</i> (Å <sup>3</sup> )	1654.15 (7)	947.30 (7)	1843.52 (12)
<i>Z</i>	2	2	4
Radiation type	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	8.66	7.57	7.89
Crystal size (mm)	0.21 × 0.07 × 0.05	0.45 × 0.25 × 0.2	0.22 × 0.15 × 0.12
Data collection			
Diffractometer	Agilent SuperNova (single source at offset, Eos detector)	Agilent SuperNova (single source at offset, Eos detector)	Agilent SuperNova (single source at offset, Eos detector)
Absorption correction	Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku Oxford Diffraction, 2015)	Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku Oxford Diffraction, 2015)	Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku Oxford Diffraction, 2015)
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.542, 1.000	0.533, 1.000	0.851, 1.000
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	32760, 3368, 3107	12209, 3871, 3646	13831, 7504, 6746
<i>R</i> <sub>int</sub>	0.027	0.029	0.023
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.625	0.625	0.625
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>	0.015, 0.037, 1.08	0.021, 0.051, 1.09	0.024, 0.051, 1.05
No. of reflections	3368	3871	7504
No. of parameters	193	245	441
No. of restraints	0	2	0
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.66, -0.32	0.92, -1.50	1.11, -1.10

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXS97* (Sheldrick, 2008), *SHELXL2015* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

[3H, *d*, <sup>3</sup>*J* = 6.0 Hz, CH(CH<sub>3</sub>)<sub>2</sub>], 1.22 [3H, *d*, <sup>3</sup>*J* = 6.0 Hz, CH(CH<sub>3</sub>)<sub>2</sub>].

**2.1.4. Crystallization of [Pt(Eteug-1H)Cl(DMSO)], (IV).** The powder of [Pt<sub>2</sub>(Eteug-1H)<sub>2</sub>Cl<sub>2</sub>], prepared according to the synthetic protocol of Da *et al.* (2010), was dissolved in DMSO to give a clear solution. Single crystals suitable for X-ray diffraction analysis were obtained after evaporating the solvent at 313 K for 48 h.

%Pt (calculated/found) 34.94/34.85. Positive ESI-MS: *m/z* 559 [*M* + H]<sup>+</sup>. IR (KBr, cm<sup>-1</sup>): 2995, 2909, 2833 ( $\nu_{\text{CH}}$ ); 1754 ( $\nu_{\text{C=O}}$ ); 1571, 1479 ( $\nu_{\text{C=C}}$ ), 1186 ( $\nu_{\text{S=O}}$ ). <sup>1</sup>H NMR [500 MHz, (CD<sub>3</sub>)<sub>2</sub>SO]:  $\delta$  6.75 (2H, *ov*, Ar-*H*), 5.10 (1H, *br*, <sup>2</sup>*J*<sub>Pt-H</sub> = 72 Hz, CH=CH<sub>2</sub>), 4.59 (1H, *d*, <sup>2</sup>*J* = 16.0 Hz, OCH<sub>2</sub>), 4.56 (1H, *d*, <sup>2</sup>*J* = 16.0 Hz, OCH<sub>2</sub>), 4.33 (1H, *br*, <sup>2</sup>*J*<sub>Pt-H</sub> = 73 Hz, CH=CH<sub>2</sub>), 4.17 (2H, *m*, CH<sub>2</sub>CH<sub>3</sub>), 3.94 (1H, *d*, CH=CH<sub>2</sub>, <sup>3</sup>*J* = 13.0, <sup>2</sup>*J*<sub>Pt-H</sub> = 74 Hz), 3.70 (4H, *ov*, OCH<sub>3</sub>, CH<sub>2</sub>CH), 2.80 (1H, *d*, CH<sub>2</sub>CH, <sup>3</sup>*J*<sub>Pt-H</sub> = 110 Hz), 2.55 [6H, *s*, (CH<sub>3</sub>)<sub>2</sub>SO], 1.23 (3H, *t*, <sup>3</sup>*J* = 7.0 Hz, CH<sub>2</sub>CH<sub>3</sub>).

**2.1.5. Crystallization of [Pt(isoPreug-1H)Cl(DMF)], (V).** [Pt<sub>2</sub>(isoPreug-1H)<sub>2</sub>Cl<sub>2</sub>] powder was dissolved in DMF to afford a clear solution and small light-yellow crystals were obtained after evaporating the solvent at 313 K for 30 h.

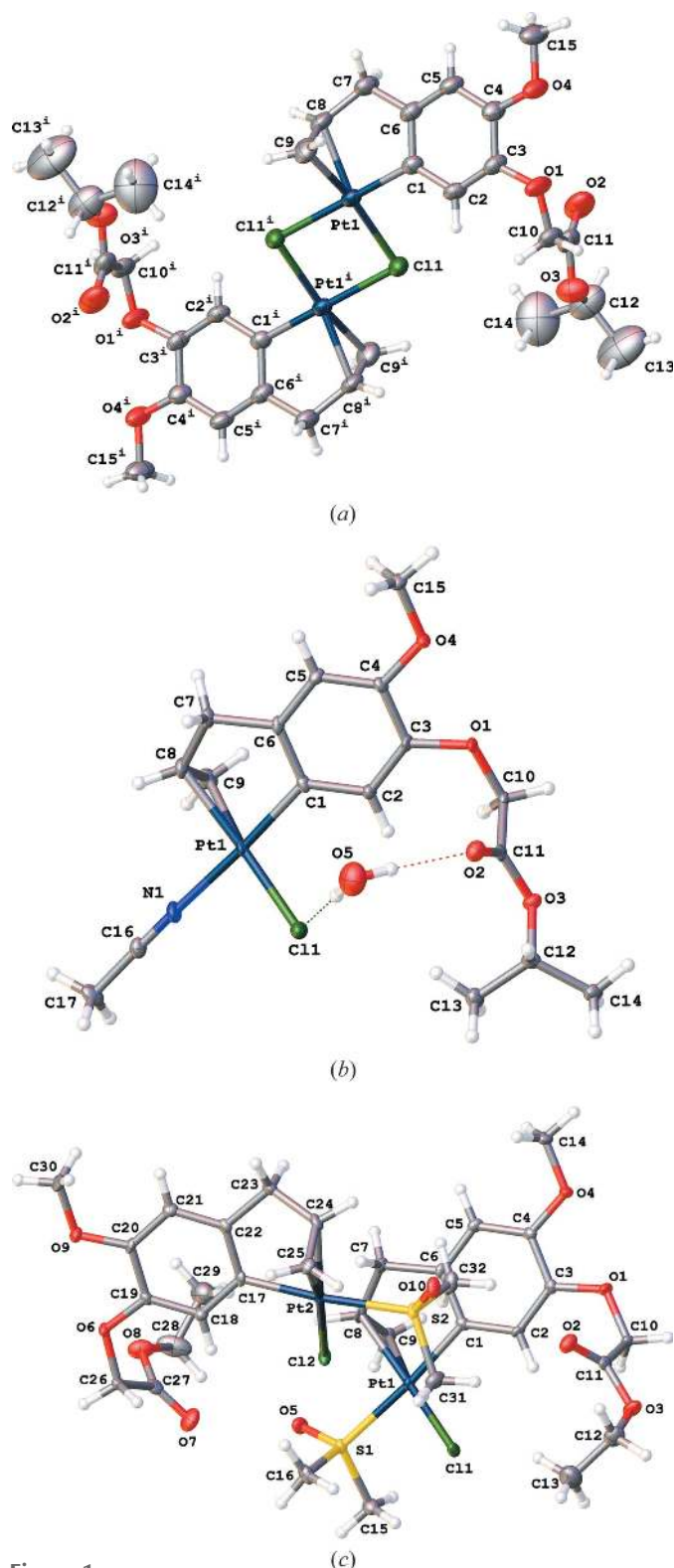
%Pt (calculated/found) 34.42/34.25. Negative ESI-MS: *m/z* 1022 [2*M* - 2DMF + Cl]<sup>-</sup>. IR (KBr, cm<sup>-1</sup>): 2978, 2900, 2800 ( $\nu_{\text{CH}}$ ); 1746, 1641 ( $\nu_{\text{C=O}}$ ); 1590, 1490 ( $\nu_{\text{C=C}}$ ).

## 2.2. Structure solution and refinement

Crystal data, data collection and structure refinement details for (I), (II) and (IV) are summarized in Table 1. All H atoms were placed in idealized positions and refined in riding mode, with *U*<sub>iso</sub>(H) values assigned as 1.2 times *U*<sub>eq</sub> of the parent atoms (1.5 times for methyl groups), with C–H distances of 0.93 (aromatic), 0.96 (methyl), 0.97 (methylene) and 0.97 Å (methine) for (I), 0.95 (aromatic), 0.98 (methyl), 0.99 (methylene) and 0.99 Å (methine) for (II) and (IV), and with O–H distances of 0.84 (2) Å for (II).

## 3. Results and discussion

It is remarkable that the crystallization of the powder of [Pt<sub>2</sub>(isoPreug-1H)<sub>2</sub>Cl<sub>2</sub>] [isoPreug-1H is (2-allyl-4-methoxy-5-[[propan-2-yloxy]carbonyl]methoxy)phenyl] from five different solvent systems, namely CHCl<sub>3</sub>–Et<sub>2</sub>O, CH<sub>2</sub>Cl<sub>2</sub>–Et<sub>2</sub>O, CH<sub>3</sub>CN, DMF and DMSO, afforded four different compounds. The data from platinum analysis (%Pt) and the IR, ESI-MS and <sup>1</sup>H NMR spectra (see *Experimental*) have elucidated that the compounds are [Pt<sub>2</sub>(isoPreug-1H)<sub>2</sub>Cl<sub>2</sub>], (I), [Pt(isoPreug-1H)Cl(CH<sub>3</sub>CN)]·H<sub>2</sub>O, (II), [Pt(isoPreug-1H)Cl(DMSO)], (III), and [Pt(isoPreug-1H)Cl(DMF)], (V). This means that the dimeric complex (I) is easily cleaved by the coordinating



**Figure 1**  
Views of the molecular structures of (a) (I), (b) (II) and (c) (IV), showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 50% probability level. Dotted lines show hydrogen bonds. [Symmetry code: (i)  $-x, -y + 1, -z$ .]

solvents ( $\text{CH}_3\text{CN}$ , DMSO and DMF) to form the monomeric complexes (II), (III) and (V). The nature of complexes (I) and (II) was further confirmed by X-ray diffraction. As it was not

**Table 2**  
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ) for (I).

Cg1 is the centroid of the C1–C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10–H10B $\cdots$ O1 <sup>i</sup>	0.99	2.57	3.124 (4)	116
C10–H10B $\cdots$ O4 <sup>i</sup>	0.99	2.44	3.294 (4)	144
C7–H7A $\cdots$ Cg1 <sup>ii</sup>	0.99	2.63	3.554 (3)	155

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

**Table 3**  
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ) for (II).

Cg1 is the centroid of the C1–C6 ring.

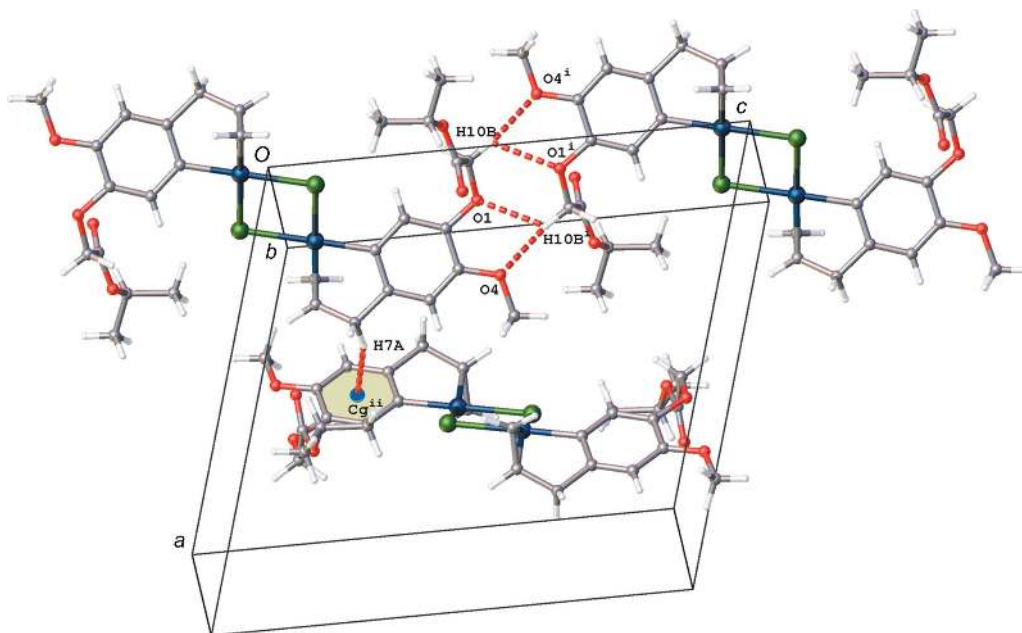
$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5–H5A $\cdots$ O2	0.89 (4)	1.95 (4)	2.848 (4)	178 (6)
O5–H5B $\cdots$ Cl1	0.89 (2)	2.33 (3)	3.213 (3)	174 (6)
C8–H8 $\cdots$ Cl1 <sup>i</sup>	0.99 (4)	2.83 (4)	3.701 (4)	148 (3)
C10–H10A $\cdots$ O1 <sup>ii</sup>	0.99	2.54	3.245 (4)	128
C10–H10A $\cdots$ O4 <sup>ii</sup>	0.99	2.41	3.334 (4)	155
C15–H15B $\cdots$ O5 <sup>iii</sup>	0.98	2.37	3.313 (5)	160
C17–H17B $\cdots$ O2 <sup>iv</sup>	0.98	2.50	3.387 (4)	151
C17–H17C $\cdots$ O5 <sup>i</sup>	0.98	2.42	3.152 (5)	131
C13–H13B $\cdots$ Cg1 <sup>v</sup>	0.98	2.68	3.481 (4)	139

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

possible to obtain good quality crystals for (III), a structural analog, *i.e.*  $[\text{Pt}(\text{Eteug-1H})\text{Cl}(\text{DMSO})]$ , (IV), was synthesized and crystallized in order to confirm the Pt coordination.

Complex (I) crystallizes in the space group  $P2_1/n$ . The asymmetric unit consists of a half complex, the second half being generated by an inversion centre (Fig. 1a). The Pt<sup>II</sup> metal atoms display a distorted square-planar coordination, with the Pt<sup>II</sup> atom coordinated by two Cl atoms, and by a phenyl C atom and the C=C double bond of the isoPreug ligand. Both Pt–Cl distances are different [Pt1–Cl1 = 2.3527 (7)  $\text{\AA}$  and Pt1–Cl1( $-x, -y + 1, -z$ ) = 2.4773 (7)  $\text{\AA}$ ], but still in the range observed in the Cambridge Structural Database (CSD, Version 5.37, last update May 2016; Groom *et al.*, 2016) for Pt–Cl distances in Pt<sub>2</sub>Cl<sub>2</sub> dimers (2.307–2.612  $\text{\AA}$ ). The Cl atoms are in *cis* positions and make an angle of 85.75 (3) $^\circ$  (77.33–88.12 $^\circ$  in the CSD). The complexes form chains along the *c* axis *via* C–H $\cdots$ O interactions between atoms O1 and O4, and atom H10B (Fig. 2 and Table 2). C–H $\cdots$  $\pi$  interactions further link parallel chains (Fig. 2 and Table 2). The isopropyl groups fill a void between neighboring molecules, resulting in higher displacement parameters for these atoms compared to the rest of the complex.

Complex (II) crystallizes in the space group  $P\bar{1}$  with an additional water molecule in the asymmetric unit (Fig. 1b). The Pt<sup>II</sup> atom exhibits the usual square-planar coordination and is surrounded by one Cl atom [Pt1–Cl1 = 2.3232 (9)  $\text{\AA}$ ], by a C atom and the C=C double bond of the isoPreug ligand, and by the N atom of the acetonitrile ligand. The water molecule bridges the Cl1 atom and acyl atom O2 (Table 3). In the crystal packing, the complexes form two different types of dimers. The first kind is the result of C–H $\cdots$ O interactions between atoms O1 and O4, and atom H10A, and was also

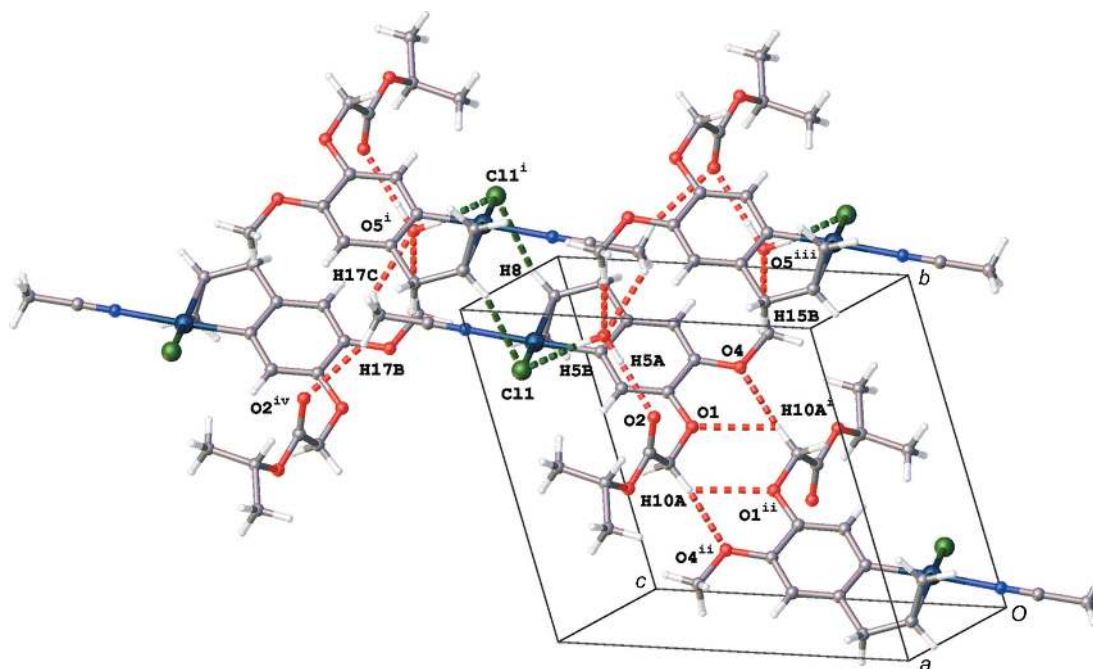


**Figure 2**  
A partial packing diagram of complex (I). Chains along the *c* axis are formed by C–H···O hydrogen bonding (dotted lines) between the isoPreug ligands and the chains interact with parallel ones through C–H··· $\pi$  interactions (*Cg*1 is the centroid of the C1–C6 ring). [Symmetry codes: (i)  $-x, -y + 1, -z + 1$ ; (ii)  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ ]

observed in the packing of (I). At the other side of the complex, dimer formation is realised *via* C–H···Cl interactions (Fig. 3 and Table 3). Other C–H···O and C–H··· $\pi$  interactions facilitate the formation of a three-dimensional network.

As it was not possible to obtain well-diffracting crystals for complex (III), crystals of the ethyl analogue (IV) were used to

verify the nature of the complex. The compound crystallizes in the space group  $P\bar{1}$  with two molecules in the asymmetric unit (Fig. 1c). The two molecules differ only in the orientation of the ethoxycarbonyl group, as illustrated by the torsion angles O1–C10–C11–O3 [ $-172.8(3)^\circ$ ] and O6–C26–C27–O8 [ $-39.0(4)^\circ$ ] (r.m.s. deviation fit = 1.039 Å). The Pt<sup>II</sup> atom exhibits a square-planar coordination and is surrounded by



**Figure 3**  
A partial packing diagram of complex (II). Two types of dimers are formed by (a) C–H···O hydrogen bonding (red dotted lines) between the isoPreug ligands and (b) C–H···Cl interactions (green dotted lines). [Symmetry codes: (i)  $-x + 1, -y + 2, -z + 2$ ; (ii)  $-x + 1, -y + 1, -z + 1$ ; (iii)  $-x + 1, -y + 2, -z + 1$ ; (iv)  $x, y, z + 1$ .]

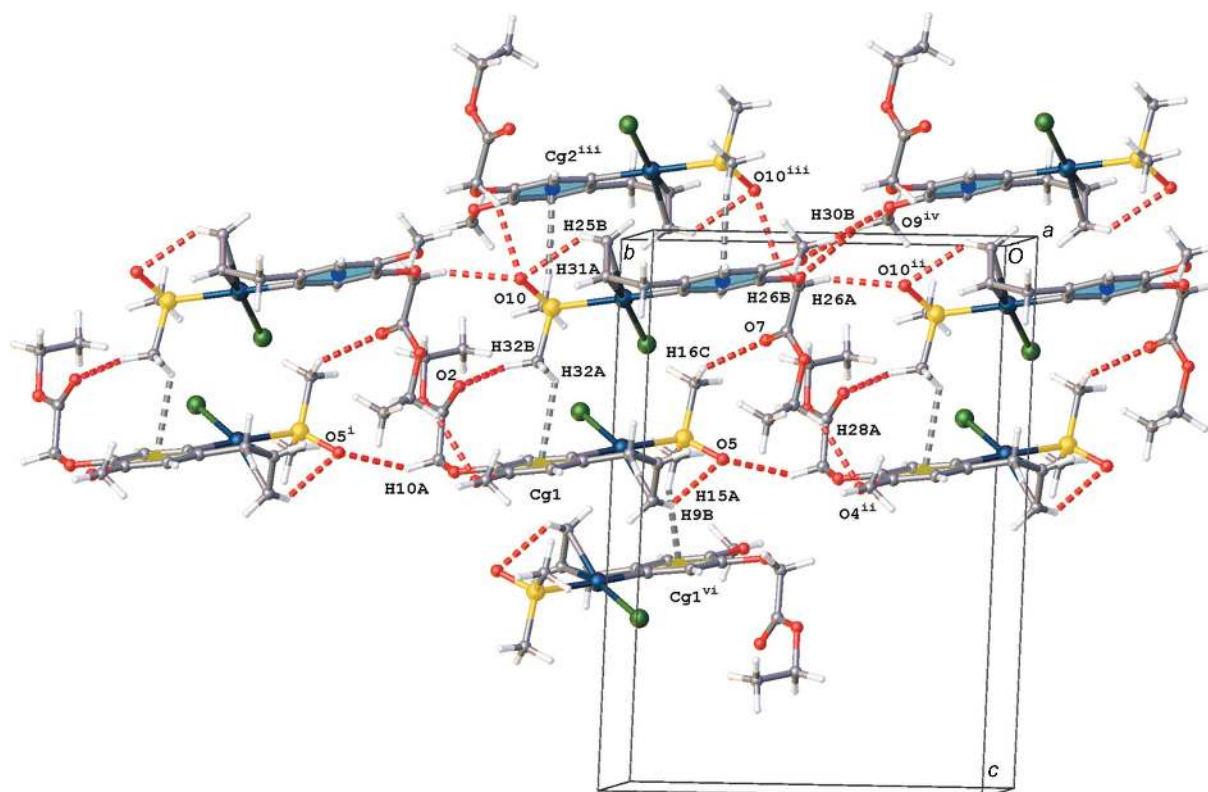


Figure 4

A partial packing diagram of complex (IV). Red dotted lines show C—H...O hydrogen bonds and gray dotted lines show C—H... $\pi$  interactions. Cg1 and Cg2 are the centroids of the C1–C6 and C17–C22 rings, respectively. [Symmetry codes: (i)  $x, y + 1, z$ ; (ii)  $x, y - 1, z$ ; (iii)  $-x + 1, -y + 2, -z$ ; (iv)  $-x + 2, -y + 1, -z$ ; (v)  $x + 1, y, z$ ; (vi)  $-x, -y + 2, -z + 1$ .]

Table 4

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ) for (IV).

Cg1 and Cg2 are the centroids of C1–C6 and C17–C22 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9–H9B...O5	0.95	2.40	3.092 (4)	130
C10–H10A...O5 <sup>i</sup>	0.99	2.40	3.230 (4)	142
C16–H16C...O7	0.98	2.42	3.213 (5)	138
C25–H25B...O10	0.95	2.41	3.129 (4)	132
C26–H26A...O10 <sup>ii</sup>	0.99	2.42	3.337 (4)	153
C26–H26B...O10 <sup>iii</sup>	0.99	2.46	3.289 (4)	141
C28–H28A...O4 <sup>ii</sup>	0.99	2.45	3.346 (6)	150
C30–H30B...O9 <sup>iv</sup>	0.98	2.50	3.222 (5)	130
C30–H30C...O7 <sup>v</sup>	0.98	2.40	3.334 (6)	160
C32–H32B...O2	0.98	2.41	3.242 (5)	143
C15–H15A...Cg1 <sup>vi</sup>	0.98	2.49	3.405 (5)	155
C31–H31A...Cg2 <sup>iii</sup>	0.98	2.77	3.732 (5)	166
C32–H32A...Cg1	0.98	2.66	3.269 (4)	120

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

one Cl atom [Pt–Cl = 2.3449 (9) and 2.3284 (9)  $\text{\AA}$ ], by a C atom and the C=C double bond of the isoPreug ligand, and by the S atom of the DMSO ligand. The molecules in the asymmetric unit form a dimer *via* a C—H... $\pi$  interaction and C—H...O interactions between a DMSO methyl H atom and acyl atoms O2 and O7 (Table 4). Chains of dimers are formed by C—H...O interactions involving atoms O4 (methoxy group), O5 and O10 (both DMSO) (Fig. 4 and Table 4).

Parallel chains are further linked to each other by C—H...O and C—H... $\pi$  interactions.

Despite the presence of the aromatic ring in the isoPreug-1H ligand, no  $\pi$ – $\pi$  interactions are observed in the packing of the three complexes. A common motif present in (I) and (II) is the dimer formation through C—H...O interactions between both oxygen-containing substituents of isoPreug-1H. The packing of (IV) is mainly determined by the presence of DMSO, which induces a different type of dimer *via* C—H... $\pi$  and C—H...O interactions between the two molecules present in the asymmetric unit.

The coordination of acetonitrile and DMSO has been observed in other Pt<sup>II</sup> complexes. The CSD contains 104 structures containing the Pt...N $\equiv$ C—CH<sub>3</sub> fragment, with Pt...N distances ranging from 1.883 to 2.208  $\text{\AA}$  (mean distance = 2.021  $\text{\AA}$ ) and of which 14 have at least one Cl atom as an extra ligand. Four complexes have one Cl ligand bound to the square-planar-coordinated Pt<sup>II</sup> atom, with the N and Cl atoms in *cis* positions, which is also the case for (II). For DMSO, two coordinations, *i.e.* *via* S or O, are possible with a Pt<sup>II</sup> atom. In the CSD, the majority (352 structures) of the Pt...DMSO complexes coordinate *via* the S atom, with Pt...S distances ranging from 2.157 to 2.340  $\text{\AA}$  (mean distance = 2.220  $\text{\AA}$ ). Of the 110 complexes having one Cl ligand, the majority (107 structures) present the Cl and S atoms in a *cis* conformation. In five complexes, DMSO coordinates *via* its O atom.

In (II), (III) and (IV), the CH<sub>3</sub>CN and DMSO ligands are in a *cis* position with respect to the allyl group. This means that both solvents cleave the Pt<sup>1</sup>–Cl<sup>2</sup> (or Pt<sup>2</sup>–Cl<sup>1</sup>) bond, but not the Pt<sup>1</sup>–Cl<sup>1</sup> (or Pt<sup>2</sup>–Cl<sup>2</sup>) bond in the diplatinum complex (I) (see Scheme). This is in good agreement with the longer bond length observed for Pt1–Cl1(–*x*, –*y* + 1, –*z*) [2.4773 (7) Å], as compared to Pt1–Cl1 [2.3527 (7) Å]. However, the solvents used for the vapour-diffusion crystallization of (I), *i.e.* chloroform, methylene chloride and diethyl ether, are not able to replace a Cl atom as ligand. The CSD contains no structures where chloroform is directly bound to a Pt<sup>II</sup> atom, for methylene chloride two structures are included and for diethyl ether four structures.

Despite many trials, no crystals suitable for XRD diffraction could be obtained for (V) and the structure was confirmed by spectral and analytical methods. The determined platinum percentage of 34.25% is consistent with the formula [Pt(isoPreug-1H)Cl(DMF)] containing 34.42% platinum. In the negative-mode ESI–MS spectrum of (V), the base peak at *m/z* 1022.1 au (relative intensity 100%) is consistent with the pseudo-molecular ion [2M – 2DMF + Cl<sup>–</sup>], *i.e.* [Pt<sub>2</sub>(isoPreug-1H)<sub>2</sub>Cl<sub>3</sub>]<sup>–</sup>. The IR spectra of (V) show bands for the presence of isoPreug and DMF. The reduced  $\nu_{\text{C=O}}$  wavenumber (1640 cm<sup>–1</sup>) compared to 1670 cm<sup>–1</sup> for noncoordinated DMF suggests that DMF coordinates with Pt<sup>II</sup> *via* the O atom. In addition, the absence of a medium band at 1620 cm<sup>–1</sup> typical for the C=C double bond of the allyl group in noncoordinated isoPreug indicates that the allyl group coordinates in a  $\eta^2$  manner.

When growing crystals of K[PtCl<sub>3</sub>(isoPreug)] in ethanol–water (1:1 *v/v*), we also obtained complex (I) by chance. It is noted that the starting powder, *i.e.* [Pt<sub>2</sub>(isoPreug-1H)<sub>2</sub>Cl<sub>2</sub>], is insoluble in ethanol, water or an ethanol–water mixture and can be prepared by interaction between K[PtCl<sub>3</sub>(isoPreug)] and a mixture of ethanol and water at about 333 K and precipitates at this temperature in the reaction solution. Consequently, ethanol and water could not displace the Cl atom in (I) like CH<sub>3</sub>Cl and CH<sub>2</sub>Cl<sub>2</sub>, and the moderate heating which was used in the case of slow evaporation from DMSO and DMF is not necessary for Cl displacement. In conclusion,

the polarity of the solvent systems used for the crystallization experiments might be one of the factors affecting the displacement. However, it can not be the only factor as (I) is not soluble in the more polar solvents H<sub>2</sub>O and EtOH. Complexes (I)–(V) can be used as starting compounds in the synthesis of other Pt<sup>II</sup> complexes with potential anticancer activity as the bonds between Pt and Cl, acetonitrile or DMSO are also easily cleaved.

### Acknowledgements

The authors thank VLIR–UOS (project ZEIN2014Z182) and Vietnam National Foundation for Science and Technology Development NAFOSTED (grant No. 104.03–2015.83) for financial support and the Hercules Foundation for supporting the purchase of the diffractometer through project AKUL/09/0035.

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## supporting information

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## Crystallization experiments with the dinuclear chelate ring complex di- $\mu$ -chlorido-bis[( $\eta^2$ -2-allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy}phenyl- $\kappa$ C<sup>1</sup>)platinum(II)]

**Chi Nguyen Thi Thanh, Thong Pham Van, Hai Le Thi Hong and Luc Van Meervelt**

### Computing details

For all compounds, data collection: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); cell refinement: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); data reduction: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2015* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

### (I) Di- $\mu$ -chlorido-bis[( $\eta^2$ -2-allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy}phenyl- $\kappa$ C<sup>1</sup>)platinum(II)]

#### Crystal data

[Pt<sub>2</sub>(C<sub>15</sub>H<sub>19</sub>O<sub>4</sub>)<sub>2</sub>Cl<sub>2</sub>]  
*M<sub>r</sub>* = 987.66  
 Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 13.5600 (3) Å  
*b* = 7.98332 (19) Å  
*c* = 15.8057 (4) Å  
 $\beta$  = 104.815 (3)°  
*V* = 1654.15 (7) Å<sup>3</sup>  
*Z* = 2

*F*(000) = 944  
*D<sub>x</sub>* = 1.983 Mg m<sup>-3</sup>  
 Mo *K* $\alpha$  radiation,  $\lambda$  = 0.71073 Å  
 Cell parameters from 18257 reflections  
 $\theta$  = 3.4–28.9°  
 $\mu$  = 8.66 mm<sup>-1</sup>  
*T* = 200 K  
 Block, yellow  
 0.21 × 0.07 × 0.05 mm

#### Data collection

Agilent SuperNova (single source at offset, Eos detector) diffractometer  
 Radiation source: SuperNova (Mo) X-ray Source  
 Mirror monochromator  
 Detector resolution: 15.9631 pixels mm<sup>-1</sup>  
 $\omega$  scans

Absorption correction: multi-scan (CrysAlis PRO; Rigaku Oxford Diffraction, 2015)  
 $T_{\min}$  = 0.542,  $T_{\max}$  = 1.000  
 32760 measured reflections  
 3368 independent reflections  
 3107 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}}$  = 0.027  
 $\theta_{\max}$  = 26.4°,  $\theta_{\min}$  = 2.9°  
 $h$  = -16→16  
 $k$  = -9→9  
 $l$  = -19→19



Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.015$   
 $wR(F^2) = 0.037$   
 $S = 1.08$   
 3368 reflections  
 193 parameters  
 0 restraints

Primary atom site location: structure-invariant  
 direct methods  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0155P)^2 + 1.4928P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.66 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.06825 (2)	0.63620 (2)	0.08285 (2)	0.02999 (4)
Cl1	-0.02626 (6)	0.38782 (9)	0.07869 (5)	0.03904 (17)
O1	0.01296 (17)	0.5844 (3)	0.41263 (14)	0.0503 (6)
C1	0.1027 (2)	0.6554 (3)	0.21281 (18)	0.0307 (6)
O2	0.05762 (18)	0.2519 (4)	0.40238 (19)	0.0701 (8)
C2	0.0395 (2)	0.6040 (4)	0.26528 (19)	0.0347 (6)
H2	-0.0247	0.5542	0.2392	0.042*
O3	-0.10704 (18)	0.1958 (3)	0.33764 (17)	0.0563 (6)
C3	0.0702 (2)	0.6254 (3)	0.3550 (2)	0.0360 (7)
O4	0.18747 (17)	0.7136 (3)	0.48295 (13)	0.0483 (6)
C4	0.1646 (2)	0.6996 (4)	0.39382 (19)	0.0362 (6)
C5	0.2274 (2)	0.7497 (4)	0.3420 (2)	0.0380 (7)
H5	0.2915	0.7999	0.3681	0.046*
C6	0.1970 (2)	0.7268 (4)	0.2517 (2)	0.0358 (6)
C7	0.2650 (2)	0.7654 (4)	0.1919 (2)	0.0419 (7)
H7A	0.3043	0.8688	0.2120	0.050*
H7B	0.3139	0.6725	0.1942	0.050*
C8	0.2018 (2)	0.7884 (4)	0.0993 (2)	0.0400 (7)
H8	0.2391	0.7791	0.0525	0.048*
C9	0.1145 (3)	0.8878 (4)	0.0797 (2)	0.0477 (8)
H9A	0.0973	0.9429	0.0217	0.057*
H9B	0.1042	0.9609	0.1273	0.057*
C10	-0.0677 (2)	0.4687 (4)	0.3849 (2)	0.0413 (7)
H10A	-0.1117	0.5049	0.3277	0.050*
H10B	-0.1099	0.4676	0.4276	0.050*
C11	-0.0287 (2)	0.2947 (5)	0.3763 (2)	0.0461 (8)
C12	-0.0847 (4)	0.0230 (5)	0.3190 (3)	0.0775 (13)
H12	-0.0158	-0.0134	0.3542	0.093*
C13	-0.1678 (5)	-0.0850 (6)	0.3355 (5)	0.134 (3)

H13A	-0.1613	-0.0916	0.3986	0.201*
H13B	-0.1625	-0.1976	0.3124	0.201*
H13C	-0.2342	-0.0365	0.3064	0.201*
C14	-0.0936 (6)	0.0161 (7)	0.2205 (4)	0.136 (3)
H14A	-0.1590	0.0643	0.1887	0.204*
H14B	-0.0897	-0.1007	0.2025	0.204*
H14C	-0.0377	0.0802	0.2073	0.204*
C15	0.2803 (3)	0.7966 (5)	0.5240 (2)	0.0550 (9)
H15A	0.3378	0.7348	0.5123	0.083*
H15B	0.2878	0.8015	0.5873	0.083*
H15C	0.2791	0.9106	0.5008	0.083*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pt1	0.02253 (6)	0.03176 (7)	0.03563 (7)	-0.00193 (4)	0.00735 (4)	0.00490 (4)
Cl1	0.0372 (4)	0.0423 (4)	0.0355 (4)	-0.0121 (3)	0.0054 (3)	0.0075 (3)
O1	0.0415 (13)	0.0714 (15)	0.0446 (12)	-0.0268 (12)	0.0230 (10)	-0.0142 (11)
C1	0.0232 (13)	0.0320 (14)	0.0380 (15)	-0.0001 (11)	0.0098 (11)	0.0006 (11)
O2	0.0339 (13)	0.083 (2)	0.097 (2)	0.0068 (13)	0.0237 (13)	0.0267 (16)
C2	0.0229 (13)	0.0390 (16)	0.0431 (16)	-0.0067 (12)	0.0098 (12)	-0.0015 (13)
O3	0.0429 (13)	0.0445 (13)	0.0797 (17)	0.0042 (11)	0.0125 (12)	-0.0072 (13)
C3	0.0313 (15)	0.0381 (16)	0.0443 (17)	-0.0086 (12)	0.0198 (13)	-0.0040 (12)
O4	0.0456 (13)	0.0639 (15)	0.0391 (12)	-0.0266 (11)	0.0175 (10)	-0.0136 (11)
C4	0.0348 (15)	0.0379 (16)	0.0376 (16)	-0.0074 (13)	0.0121 (12)	-0.0072 (13)
C5	0.0303 (15)	0.0408 (16)	0.0445 (17)	-0.0131 (12)	0.0128 (13)	-0.0061 (13)
C6	0.0305 (15)	0.0336 (15)	0.0464 (17)	-0.0070 (12)	0.0157 (13)	0.0005 (13)
C7	0.0327 (16)	0.0519 (19)	0.0440 (17)	-0.0132 (14)	0.0149 (13)	-0.0044 (14)
C8	0.0342 (16)	0.0415 (17)	0.0467 (17)	-0.0156 (14)	0.0146 (13)	0.0028 (14)
C9	0.057 (2)	0.0331 (17)	0.053 (2)	-0.0100 (15)	0.0142 (16)	0.0042 (14)
C10	0.0290 (15)	0.0538 (19)	0.0456 (17)	-0.0110 (14)	0.0178 (13)	-0.0023 (15)
C11	0.0325 (17)	0.060 (2)	0.0518 (19)	-0.0018 (15)	0.0208 (14)	0.0126 (16)
C12	0.074 (3)	0.046 (2)	0.122 (4)	0.015 (2)	0.042 (3)	0.002 (2)
C13	0.133 (6)	0.049 (3)	0.238 (8)	0.004 (3)	0.080 (6)	0.001 (4)
C14	0.200 (7)	0.081 (4)	0.142 (6)	0.046 (4)	0.073 (5)	-0.018 (4)
C15	0.047 (2)	0.074 (2)	0.0447 (19)	-0.0268 (18)	0.0130 (15)	-0.0189 (18)

*Geometric parameters (Å, °)*

Pt1—Cl1	2.3527 (7)	C7—H7A	0.9900
Pt1—Cl1 <sup>i</sup>	2.4773 (7)	C7—H7B	0.9900
Pt1—C1	1.993 (3)	C7—C8	1.507 (4)
Pt1—C8	2.141 (3)	C8—H8	1.0000
Pt1—C9	2.108 (3)	C8—C9	1.393 (5)
Cl1—Pt1 <sup>i</sup>	2.4773 (7)	C9—H9A	0.9900
O1—C3	1.379 (3)	C9—H9B	0.9900
O1—C10	1.413 (3)	C10—H10A	0.9900
C1—C2	1.397 (4)	C10—H10B	0.9900

C1—C6	1.391 (4)	C10—C11	1.505 (5)
O2—C11	1.188 (4)	C12—H12	1.0000
C2—H2	0.9500	C12—C13	1.494 (7)
C2—C3	1.382 (4)	C12—C14	1.531 (7)
O3—C11	1.339 (4)	C13—H13A	0.9800
O3—C12	1.459 (5)	C13—H13B	0.9800
C3—C4	1.401 (4)	C13—H13C	0.9800
O4—C4	1.368 (3)	C14—H14A	0.9800
O4—C15	1.424 (4)	C14—H14B	0.9800
C4—C5	1.383 (4)	C14—H14C	0.9800
C5—H5	0.9500	C15—H15A	0.9800
C5—C6	1.394 (4)	C15—H15B	0.9800
C6—C7	1.510 (4)	C15—H15C	0.9800
C11—Pt1—C11 <sup>i</sup>	85.76 (2)	C9—C8—C7	121.6 (3)
C1—Pt1—C11	94.37 (8)	C9—C8—H8	116.1
C1—Pt1—C11 <sup>i</sup>	179.74 (8)	Pt1—C9—H9A	116.4
C1—Pt1—C8	82.13 (12)	Pt1—C9—H9B	116.4
C1—Pt1—C9	87.59 (12)	C8—C9—Pt1	72.14 (17)
C8—Pt1—C11 <sup>i</sup>	97.66 (9)	C8—C9—H9A	116.4
C8—Pt1—C11	156.86 (9)	C8—C9—H9B	116.4
C9—Pt1—C11 <sup>i</sup>	92.35 (10)	H9A—C9—H9B	113.4
C9—Pt1—C11	164.87 (10)	O1—C10—H10A	109.3
C9—Pt1—C8	38.27 (13)	O1—C10—H10B	109.3
Pt1—C11—Pt1 <sup>i</sup>	94.25 (2)	O1—C10—C11	111.7 (3)
C3—O1—C10	118.2 (2)	H10A—C10—H10B	107.9
C2—C1—Pt1	124.9 (2)	C11—C10—H10A	109.3
C6—C1—Pt1	115.7 (2)	C11—C10—H10B	109.3
C6—C1—C2	119.5 (3)	O2—C11—O3	125.8 (4)
C1—C2—H2	119.9	O2—C11—C10	124.9 (3)
C3—C2—C1	120.2 (3)	O3—C11—C10	109.2 (3)
C3—C2—H2	119.9	O3—C12—H12	112.0
C11—O3—C12	118.0 (3)	O3—C12—C13	107.8 (4)
O1—C3—C2	125.2 (3)	O3—C12—C14	105.8 (4)
O1—C3—C4	114.6 (3)	C13—C12—H12	112.0
C2—C3—C4	120.2 (3)	C13—C12—C14	106.9 (5)
C4—O4—C15	116.7 (2)	C14—C12—H12	112.0
O4—C4—C3	115.3 (3)	C12—C13—H13A	109.5
O4—C4—C5	125.0 (3)	C12—C13—H13B	109.5
C5—C4—C3	119.6 (3)	C12—C13—H13C	109.5
C4—C5—H5	119.9	H13A—C13—H13B	109.5
C4—C5—C6	120.2 (3)	H13A—C13—H13C	109.5
C6—C5—H5	119.9	H13B—C13—H13C	109.5
C1—C6—C5	120.2 (3)	C12—C14—H14A	109.5
C1—C6—C7	116.5 (3)	C12—C14—H14B	109.5
C5—C6—C7	123.1 (3)	C12—C14—H14C	109.5
C6—C7—H7A	109.6	H14A—C14—H14B	109.5
C6—C7—H7B	109.6	H14A—C14—H14C	109.5

H7A—C7—H7B	108.1	H14B—C14—H14C	109.5
C8—C7—C6	110.3 (2)	O4—C15—H15A	109.5
C8—C7—H7A	109.6	O4—C15—H15B	109.5
C8—C7—H7B	109.6	O4—C15—H15C	109.5
Pt1—C8—H8	116.1	H15A—C15—H15B	109.5
C7—C8—Pt1	107.57 (19)	H15A—C15—H15C	109.5
C7—C8—H8	116.1	H15B—C15—H15C	109.5
C9—C8—Pt1	69.60 (17)		
Pt1—C1—C2—C3	-179.0 (2)	O4—C4—C5—C6	178.9 (3)
Pt1—C1—C6—C5	178.5 (2)	C4—C5—C6—C1	0.9 (5)
Pt1—C1—C6—C7	-5.5 (3)	C4—C5—C6—C7	-174.9 (3)
O1—C3—C4—O4	2.7 (4)	C5—C6—C7—C8	-159.6 (3)
O1—C3—C4—C5	-178.4 (3)	C6—C1—C2—C3	0.6 (4)
O1—C10—C11—O2	11.3 (5)	C6—C7—C8—Pt1	-29.7 (3)
O1—C10—C11—O3	-170.8 (3)	C6—C7—C8—C9	46.8 (4)
C1—C2—C3—O1	177.8 (3)	C7—C8—C9—Pt1	-98.6 (3)
C1—C2—C3—C4	0.3 (4)	C10—O1—C3—C2	20.1 (5)
C1—C6—C7—C8	24.5 (4)	C10—O1—C3—C4	-162.3 (3)
C2—C1—C6—C5	-1.2 (4)	C11—O3—C12—C13	142.0 (4)
C2—C1—C6—C7	174.8 (3)	C11—O3—C12—C14	-103.9 (5)
C2—C3—C4—O4	-179.6 (3)	C12—O3—C11—O2	-5.3 (5)
C2—C3—C4—C5	-0.7 (5)	C12—O3—C11—C10	176.9 (3)
C3—O1—C10—C11	69.7 (4)	C15—O4—C4—C3	-176.7 (3)
C3—C4—C5—C6	0.1 (5)	C15—O4—C4—C5	4.4 (5)

Symmetry code: (i)  $-x, -y+1, -z$ .

#### Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ )

Cg1 is the centroid of the C1—C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10—H10B $\cdots$ O1 <sup>ii</sup>	0.99	2.57	3.124 (4)	116
C10—H10B $\cdots$ O4 <sup>ii</sup>	0.99	2.44	3.294 (4)	144
C7—H7A $\cdots$ Cg1 <sup>iii</sup>	0.99	2.63	3.554 (3)	155

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

#### (II) (Acetonitrile- $\kappa N$ )( $\eta^2$ -2-allyl-4-methoxy-5-[[propan-2-yloxy]carbonyl]methoxy}phenyl- $\kappa C^1$ )chloridoplatinum(II) monohydrate

##### Crystal data

$[\text{Pt}(\text{C}_{15}\text{H}_{19}\text{ClO}_4)\text{Cl}(\text{C}_2\text{H}_3\text{N})]\cdot\text{H}_2\text{O}$

$M_r = 552.91$

Triclinic,  $P\bar{1}$

$a = 8.3747$  (4)  $\text{\AA}$

$b = 10.5732$  (5)  $\text{\AA}$

$c = 11.0883$  (4)  $\text{\AA}$

$\alpha = 76.670$  (4) $^\circ$

$\beta = 89.496$  (3) $^\circ$

$\gamma = 82.641$  (4) $^\circ$

$V = 947.30$  (7)  $\text{\AA}^3$

$Z = 2$

$F(000) = 536$

$D_x = 1.938$   $\text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$   $\text{\AA}$

Cell parameters from 7067 reflections

$\theta = 3.0\text{--}29.2^\circ$

$\mu = 7.57 \text{ mm}^{-1}$   
 $T = 100 \text{ K}$

Block, yellow  
 $0.45 \times 0.25 \times 0.2 \text{ mm}$

*Data collection*

Agilent SuperNova (single source at offset, Eos detector) diffractometer  
 Radiation source: SuperNova (Mo) X-ray Source  
 Mirror monochromator  
 Detector resolution:  $15.9631 \text{ pixels mm}^{-1}$   
 $\omega$  scans

Absorption correction: multi-scan (CrysAlis PRO; Rigaku Oxford Diffraction, 2015)  
 $T_{\min} = 0.533$ ,  $T_{\max} = 1.000$   
 12209 measured reflections  
 3871 independent reflections  
 3646 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$   
 $\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 3.0^\circ$   
 $h = -10 \rightarrow 10$   
 $k = -13 \rightarrow 12$   
 $l = -13 \rightarrow 13$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.021$   
 $wR(F^2) = 0.051$   
 $S = 1.09$   
 3871 reflections  
 245 parameters  
 2 restraints

Primary atom site location: structure-invariant direct methods  
 Hydrogen site location: mixed  
 H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 0.1035P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.002$   
 $\Delta\rho_{\max} = 0.92 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -1.50 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.43075 (2)	0.81119 (2)	0.99883 (2)	0.01389 (5)
Cl1	0.70613 (11)	0.77252 (9)	0.96959 (8)	0.0229 (2)
O1	0.4806 (3)	0.5871 (2)	0.5975 (2)	0.0149 (5)
N1	0.4768 (4)	0.8473 (3)	1.1735 (3)	0.0191 (7)
C1	0.3746 (4)	0.7822 (3)	0.8337 (3)	0.0122 (7)
O2	0.7820 (3)	0.6636 (2)	0.6011 (2)	0.0189 (5)
C2	0.4613 (4)	0.6899 (3)	0.7754 (3)	0.0124 (7)
H2	0.5557	0.6372	0.8143	0.015*
O3	0.8844 (3)	0.4599 (2)	0.7031 (2)	0.0160 (5)
C3	0.4088 (4)	0.6766 (3)	0.6620 (3)	0.0117 (7)
O4	0.2306 (3)	0.7327 (2)	0.4903 (2)	0.0143 (5)
C4	0.2702 (4)	0.7548 (3)	0.6027 (3)	0.0125 (7)
C5	0.1845 (4)	0.8440 (3)	0.6605 (3)	0.0134 (7)
H5	0.0904	0.8967	0.6213	0.016*
C6	0.2357 (4)	0.8572 (3)	0.7763 (3)	0.0124 (7)

C7	0.1452 (4)	0.9519 (3)	0.8431 (3)	0.0159 (7)
H7A	0.0279	0.9517	0.8331	0.019*
H7B	0.1704	1.0417	0.8070	0.019*
C8	0.1920 (4)	0.9132 (4)	0.9793 (3)	0.0178 (8)
H8	0.191 (5)	0.985 (4)	1.023 (3)	0.021*
C9	0.1896 (5)	0.7859 (4)	1.0479 (3)	0.0207 (8)
H9A	0.195 (5)	0.762 (4)	1.138 (4)	0.025*
H9B	0.158 (5)	0.723 (4)	1.008 (4)	0.025*
C10	0.6095 (4)	0.4941 (3)	0.6561 (3)	0.0131 (7)
H10A	0.6225	0.4193	0.6155	0.016*
H10B	0.5835	0.4603	0.7439	0.016*
C11	0.7654 (4)	0.5513 (3)	0.6503 (3)	0.0144 (7)
C12	1.0447 (4)	0.5012 (4)	0.7124 (3)	0.0159 (7)
H12	1.0585	0.5761	0.6411	0.019*
C13	1.0550 (4)	0.5442 (4)	0.8322 (3)	0.0207 (8)
H13A	0.9675	0.6147	0.8341	0.031*
H13B	1.1588	0.5761	0.8382	0.031*
H13C	1.0454	0.4698	0.9021	0.031*
C14	1.1658 (4)	0.3847 (4)	0.7047 (3)	0.0201 (8)
H14A	1.1500	0.3104	0.7732	0.030*
H14B	1.2750	0.4072	0.7103	0.030*
H14C	1.1510	0.3608	0.6256	0.030*
C15	0.0954 (4)	0.8166 (3)	0.4241 (3)	0.0168 (7)
H15A	0.0766	0.7908	0.3465	0.025*
H15B	0.1179	0.9077	0.4055	0.025*
H15C	-0.0006	0.8085	0.4751	0.025*
C16	0.5056 (4)	0.8661 (3)	1.2673 (3)	0.0175 (8)
C17	0.5457 (5)	0.8895 (4)	1.3880 (3)	0.0230 (9)
H17A	0.6319	0.9454	1.3784	0.035*
H17B	0.5818	0.8056	1.4457	0.035*
H17C	0.4502	0.9331	1.4206	0.035*
O5	0.7623 (4)	0.9013 (3)	0.6829 (3)	0.0433 (8)
H5B	0.752 (7)	0.870 (5)	0.764 (2)	0.065*
H5A	0.766 (7)	0.827 (3)	0.657 (5)	0.065*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pt1	0.01872 (8)	0.01490 (9)	0.00909 (8)	0.00034 (5)	0.00076 (5)	-0.00629 (6)
Cl1	0.0184 (4)	0.0297 (5)	0.0231 (4)	0.0046 (4)	-0.0044 (4)	-0.0152 (4)
O1	0.0139 (12)	0.0200 (13)	0.0124 (11)	0.0053 (10)	-0.0025 (10)	-0.0111 (10)
N1	0.0247 (17)	0.0201 (17)	0.0134 (15)	0.0015 (13)	0.0018 (13)	-0.0081 (13)
C1	0.0175 (17)	0.0111 (17)	0.0088 (15)	-0.0039 (13)	0.0027 (13)	-0.0030 (13)
O2	0.0197 (13)	0.0179 (14)	0.0175 (12)	-0.0008 (10)	0.0032 (10)	-0.0016 (11)
C2	0.0115 (16)	0.0149 (18)	0.0115 (15)	0.0001 (13)	0.0007 (13)	-0.0054 (14)
O3	0.0121 (12)	0.0166 (13)	0.0181 (12)	-0.0002 (10)	-0.0004 (10)	-0.0027 (10)
C3	0.0102 (16)	0.0142 (18)	0.0116 (15)	0.0005 (13)	0.0044 (13)	-0.0062 (14)
O4	0.0154 (12)	0.0174 (13)	0.0103 (11)	0.0037 (10)	-0.0031 (9)	-0.0065 (10)

C4	0.0139 (17)	0.0160 (18)	0.0088 (15)	-0.0033 (13)	0.0012 (13)	-0.0048 (14)
C5	0.0131 (17)	0.0135 (18)	0.0116 (16)	0.0021 (13)	0.0010 (13)	-0.0011 (14)
C6	0.0166 (17)	0.0114 (17)	0.0099 (15)	-0.0012 (13)	0.0043 (13)	-0.0040 (13)
C7	0.0182 (18)	0.0157 (19)	0.0152 (17)	-0.0005 (14)	0.0040 (14)	-0.0073 (15)
C8	0.0194 (19)	0.020 (2)	0.0155 (17)	0.0006 (15)	0.0040 (14)	-0.0088 (15)
C9	0.023 (2)	0.029 (2)	0.0124 (17)	-0.0057 (16)	0.0046 (15)	-0.0082 (16)
C10	0.0134 (17)	0.0135 (18)	0.0131 (16)	0.0035 (14)	-0.0014 (14)	-0.0072 (14)
C11	0.0163 (18)	0.019 (2)	0.0088 (15)	0.0029 (14)	0.0009 (13)	-0.0084 (14)
C12	0.0132 (17)	0.020 (2)	0.0152 (17)	-0.0050 (14)	0.0018 (14)	-0.0041 (15)
C13	0.0185 (19)	0.025 (2)	0.0207 (18)	-0.0038 (15)	0.0038 (15)	-0.0102 (16)
C14	0.0128 (18)	0.028 (2)	0.0198 (18)	-0.0003 (15)	0.0004 (15)	-0.0087 (16)
C15	0.0168 (18)	0.0180 (19)	0.0145 (17)	0.0013 (14)	-0.0025 (14)	-0.0033 (15)
C16	0.0195 (19)	0.018 (2)	0.0156 (18)	-0.0024 (15)	0.0043 (14)	-0.0061 (15)
C17	0.026 (2)	0.033 (2)	0.0134 (17)	-0.0102 (17)	0.0028 (15)	-0.0088 (17)
O5	0.050 (2)	0.0308 (19)	0.049 (2)	-0.0073 (16)	0.0093 (18)	-0.0087 (16)

*Geometric parameters (Å, °)*

Pt1—C11	2.3232 (9)	C8—H8	0.99 (4)
Pt1—N1	2.106 (3)	C8—C9	1.388 (5)
Pt1—C1	1.994 (3)	C9—H9A	0.98 (4)
Pt1—C8	2.134 (4)	C9—H9B	0.94 (4)
Pt1—C9	2.121 (4)	C10—H10A	0.9900
O1—C3	1.388 (4)	C10—H10B	0.9900
O1—C10	1.415 (4)	C10—C11	1.502 (5)
N1—C16	1.137 (4)	C12—H12	1.0000
C1—C2	1.414 (4)	C12—C13	1.507 (5)
C1—C6	1.390 (5)	C12—C14	1.510 (5)
O2—C11	1.212 (4)	C13—H13A	0.9800
C2—H2	0.9500	C13—H13B	0.9800
C2—C3	1.379 (4)	C13—H13C	0.9800
O3—C11	1.331 (4)	C14—H14A	0.9800
O3—C12	1.475 (4)	C14—H14B	0.9800
C3—C4	1.407 (5)	C14—H14C	0.9800
O4—C4	1.370 (4)	C15—H15A	0.9800
O4—C15	1.437 (4)	C15—H15B	0.9800
C4—C5	1.383 (5)	C15—H15C	0.9800
C5—H5	0.9500	C16—C17	1.465 (5)
C5—C6	1.400 (4)	C17—H17A	0.9800
C6—C7	1.504 (4)	C17—H17B	0.9800
C7—H7A	0.9900	C17—H17C	0.9800
C7—H7B	0.9900	O5—H5B	0.893 (19)
C7—C8	1.512 (5)	O5—H5A	0.897 (19)
N1—Pt1—C11	89.72 (9)	C8—C9—Pt1	71.5 (2)
N1—Pt1—C8	95.03 (12)	C8—C9—H9A	123 (2)
N1—Pt1—C9	90.55 (13)	C8—C9—H9B	119 (2)
C1—Pt1—C11	93.36 (10)	H9A—C9—H9B	117 (3)

C1—Pt1—N1	176.75 (12)	O1—C10—H10A	109.2
C1—Pt1—C8	81.72 (14)	O1—C10—H10B	109.2
C1—Pt1—C9	86.81 (14)	O1—C10—C11	112.2 (3)
C8—Pt1—C11	158.63 (10)	H10A—C10—H10B	107.9
C9—Pt1—C11	163.01 (11)	C11—C10—H10A	109.2
C9—Pt1—C8	38.06 (14)	C11—C10—H10B	109.2
C3—O1—C10	118.2 (2)	O2—C11—O3	124.6 (3)
C16—N1—Pt1	178.3 (3)	O2—C11—C10	124.9 (3)
C2—C1—Pt1	125.1 (2)	O3—C11—C10	110.4 (3)
C6—C1—Pt1	115.6 (2)	O3—C12—H12	109.5
C6—C1—C2	119.2 (3)	O3—C12—C13	108.2 (3)
C1—C2—H2	120.1	O3—C12—C14	106.2 (3)
C3—C2—C1	119.9 (3)	C13—C12—H12	109.5
C3—C2—H2	120.1	C13—C12—C14	113.7 (3)
C11—O3—C12	117.6 (3)	C14—C12—H12	109.5
O1—C3—C4	114.0 (3)	C12—C13—H13A	109.5
C2—C3—O1	125.2 (3)	C12—C13—H13B	109.5
C2—C3—C4	120.8 (3)	C12—C13—H13C	109.5
C4—O4—C15	116.5 (3)	H13A—C13—H13B	109.5
O4—C4—C3	115.3 (3)	H13A—C13—H13C	109.5
O4—C4—C5	125.4 (3)	H13B—C13—H13C	109.5
C5—C4—C3	119.3 (3)	C12—C14—H14A	109.5
C4—C5—H5	119.8	C12—C14—H14B	109.5
C4—C5—C6	120.4 (3)	C12—C14—H14C	109.5
C6—C5—H5	119.8	H14A—C14—H14B	109.5
C1—C6—C5	120.4 (3)	H14A—C14—H14C	109.5
C1—C6—C7	117.4 (3)	H14B—C14—H14C	109.5
C5—C6—C7	122.2 (3)	O4—C15—H15A	109.5
C6—C7—H7A	109.7	O4—C15—H15B	109.5
C6—C7—H7B	109.7	O4—C15—H15C	109.5
C6—C7—C8	109.8 (3)	H15A—C15—H15B	109.5
H7A—C7—H7B	108.2	H15A—C15—H15C	109.5
C8—C7—H7A	109.7	H15B—C15—H15C	109.5
C8—C7—H7B	109.7	N1—C16—C17	179.0 (4)
Pt1—C8—H8	107 (2)	C16—C17—H17A	109.5
C7—C8—Pt1	108.5 (2)	C16—C17—H17B	109.5
C7—C8—H8	117 (2)	C16—C17—H17C	109.5
C9—C8—Pt1	70.5 (2)	H17A—C17—H17B	109.5
C9—C8—C7	121.2 (3)	H17A—C17—H17C	109.5
C9—C8—H8	119 (2)	H17B—C17—H17C	109.5
Pt1—C9—H9A	103 (2)	H5B—O5—H5A	99 (5)
Pt1—C9—H9B	107 (2)		
Pt1—C1—C2—C3	178.5 (2)	O4—C4—C5—C6	179.3 (3)
Pt1—C1—C6—C5	-179.6 (2)	C4—C5—C6—C1	1.0 (5)
Pt1—C1—C6—C7	0.8 (4)	C4—C5—C6—C7	-179.3 (3)
O1—C3—C4—O4	-1.5 (4)	C5—C6—C7—C8	160.6 (3)
O1—C3—C4—C5	177.4 (3)	C6—C1—C2—C3	0.9 (5)



O1—C10—C11—O2	-0.4 (4)	C6—C7—C8—Pt1	27.4 (3)
O1—C10—C11—O3	-178.2 (2)	C6—C7—C8—C9	-50.5 (5)
C1—C2—C3—O1	-178.0 (3)	C7—C8—C9—Pt1	100.3 (3)
C1—C2—C3—C4	0.6 (5)	C10—O1—C3—C2	5.7 (5)
C1—C6—C7—C8	-19.7 (4)	C10—O1—C3—C4	-172.9 (3)
C2—C1—C6—C5	-1.7 (5)	C11—O3—C12—C13	88.6 (3)
C2—C1—C6—C7	178.6 (3)	C11—O3—C12—C14	-148.9 (3)
C2—C3—C4—O4	179.8 (3)	C12—O3—C11—O2	5.8 (4)
C2—C3—C4—C5	-1.3 (5)	C12—O3—C11—C10	-176.4 (3)
C3—O1—C10—C11	-79.1 (4)	C15—O4—C4—C3	-176.3 (3)
C3—C4—C5—C6	0.5 (5)	C15—O4—C4—C5	4.8 (5)

#### Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of ring C1-C6.

<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>
O5—H5 <i>A</i> ...O2	0.89 (4)	1.95 (4)	2.848 (4)	178 (6)
O5—H5 <i>B</i> ...C11	0.89 (2)	2.33 (3)	3.213 (3)	174 (6)
C8—H8...C11 <sup>i</sup>	0.99 (4)	2.83 (4)	3.701 (4)	148 (3)
C10—H10 <i>A</i> ...O1 <sup>ii</sup>	0.99	2.54	3.245 (4)	128
C10—H10 <i>A</i> ...O4 <sup>ii</sup>	0.99	2.41	3.334 (4)	155
C15—H15 <i>B</i> ...O5 <sup>iii</sup>	0.98	2.37	3.313 (5)	160
C17—H17 <i>B</i> ...O2 <sup>iv</sup>	0.98	2.50	3.387 (4)	151
C17—H17 <i>C</i> ...O5 <sup>i</sup>	0.98	2.42	3.152 (5)	131
C13—H13 <i>B</i> ...Cg1 <sup>v</sup>	0.98	2.68	3.481 (4)	139

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

#### (IV) ( $\eta^2$ -2-Allyl-4-methoxy-5-[(propan-2-yloxy)carbonyl]methoxy}phenyl- $\kappa^1$ )chlorido(dimethyl sulfoxide- $\kappa^N$ )platinum(II)

##### Crystal data

[Pt(C<sub>14</sub>H<sub>17</sub>O<sub>4</sub>)Cl(C<sub>2</sub>H<sub>6</sub>OS)]

$M_r = 557.94$

Triclinic,  $P\bar{1}$

$a = 8.7920$  (3) Å

$b = 12.1383$  (4) Å

$c = 17.4745$  (6) Å

$\alpha = 87.030$  (3)°

$\beta = 82.074$  (3)°

$\gamma = 87.651$  (3)°

$V = 1843.52$  (12) Å<sup>3</sup>

$Z = 4$

$F(000) = 1080$

$D_x = 2.010$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 7952 reflections

$\theta = 2.8$ – $29.0$ °

$\mu = 7.89$  mm<sup>-1</sup>

$T = 101$  K

Block, colourless

$0.22 \times 0.15 \times 0.12$  mm

##### Data collection

Agilent SuperNova (single source at offset, Eos detector)

diffractometer

Radiation source: SuperNova (Mo) X-ray

Source

Mirror monochromator

Detector resolution: 15.9631 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan

(CrysAlis PRO; Rigaku Oxford Diffraction, 2015)

$T_{\min} = 0.851$ ,  $T_{\max} = 1.000$

13831 measured reflections

7504 independent reflections

6746 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.023$   
 $\theta_{\text{max}} = 26.4^\circ$ ,  $\theta_{\text{min}} = 2.8^\circ$

$h = -10 \rightarrow 10$   
 $k = -15 \rightarrow 14$   
 $l = -21 \rightarrow 20$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.051$   
 $S = 1.05$   
 7504 reflections  
 441 parameters  
 0 restraints

Primary atom site location: structure-invariant  
 direct methods  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0132P)^2 + 1.6196P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 1.11 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -1.10 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.11532 (2)	0.98567 (2)	0.38029 (2)	0.01055 (4)
Cl1	-0.06390 (11)	1.08305 (7)	0.31254 (5)	0.01287 (19)
S1	0.01065 (11)	0.81710 (8)	0.35908 (6)	0.0141 (2)
O1	0.1887 (3)	1.4250 (2)	0.43020 (16)	0.0143 (6)
C1	0.2228 (4)	1.1230 (3)	0.4015 (2)	0.0112 (8)
O2	0.1517 (3)	1.4180 (2)	0.27435 (17)	0.0207 (7)
C2	0.1563 (4)	1.2297 (3)	0.4075 (2)	0.0112 (8)
H2	0.0515	1.2424	0.4009	0.013*
O3	-0.0878 (3)	1.4922 (2)	0.30078 (16)	0.0210 (7)
C3	0.2427 (4)	1.3167 (3)	0.4229 (2)	0.0119 (8)
O4	0.4707 (3)	1.3899 (2)	0.45167 (16)	0.0158 (6)
C4	0.3971 (4)	1.2989 (3)	0.4339 (2)	0.0120 (8)
O5	0.0797 (3)	0.7189 (2)	0.39671 (18)	0.0215 (7)
C5	0.4646 (4)	1.1942 (3)	0.4272 (2)	0.0131 (8)
H5	0.5694	1.1819	0.4340	0.016*
C6	0.3779 (4)	1.1062 (3)	0.4103 (2)	0.0120 (8)
C7	0.4477 (4)	0.9922 (3)	0.3971 (2)	0.0145 (9)
H7A	0.5246	0.9753	0.4327	0.017*
H7B	0.5007	0.9892	0.3434	0.017*
C8	0.3243 (4)	0.9076 (3)	0.4106 (2)	0.0148 (9)
H8	0.3526	0.8339	0.3887	0.018*
C9	0.2128 (4)	0.9081 (3)	0.4760 (2)	0.0164 (9)
H9A	0.2136	0.9625	0.5131	0.020*
H9B	0.1363	0.8541	0.4832	0.020*
C10	0.0441 (4)	1.4546 (3)	0.4063 (2)	0.0140 (8)
H10A	0.0129	1.5300	0.4226	0.017*

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H10B	-0.0336	1.4035	0.4325	0.017*
C11	0.0463 (5)	1.4511 (3)	0.3196 (2)	0.0152 (9)
C12	-0.1070 (5)	1.4918 (4)	0.2193 (3)	0.0265 (11)
H12A	-0.0062	1.5010	0.1870	0.032*
H12B	-0.1760	1.5545	0.2064	0.032*
C13	-0.1733 (6)	1.3861 (4)	0.2021 (3)	0.0408 (14)
H13A	-0.1935	1.3894	0.1483	0.061*
H13B	-0.2696	1.3747	0.2366	0.061*
H13C	-0.1003	1.3248	0.2102	0.061*
C14	0.6328 (4)	1.3781 (3)	0.4526 (3)	0.0212 (10)
H14A	0.6831	1.3566	0.4017	0.032*
H14B	0.6542	1.3212	0.4919	0.032*
H14C	0.6724	1.4485	0.4649	0.032*
C15	-0.1904 (4)	0.8205 (3)	0.3894 (3)	0.0204 (9)
H15A	-0.2106	0.8335	0.4448	0.031*
H15B	-0.2324	0.7498	0.3797	0.031*
H15C	-0.2392	0.8801	0.3605	0.031*
C16	0.0145 (5)	0.7945 (3)	0.2591 (2)	0.0205 (9)
H16A	-0.0342	0.8581	0.2341	0.031*
H16B	-0.0415	0.7279	0.2533	0.031*
H16C	0.1212	0.7849	0.2349	0.031*
Pt2	0.55218 (2)	1.03314 (2)	0.11676 (2)	0.01112 (4)
Cl2	0.34837 (11)	0.95154 (8)	0.19582 (6)	0.0149 (2)
S2	0.43337 (11)	1.20883 (8)	0.13383 (6)	0.0133 (2)
O6	0.6563 (3)	0.5879 (2)	0.07660 (16)	0.0140 (6)
O7	0.2914 (3)	0.6369 (3)	0.18347 (18)	0.0281 (7)
O8	0.5009 (3)	0.5530 (3)	0.22136 (17)	0.0279 (8)
O9	0.9494 (3)	0.6089 (2)	0.04786 (16)	0.0167 (6)
O10	0.5067 (3)	1.3000 (2)	0.08416 (16)	0.0188 (6)
C17	0.6739 (4)	0.8886 (3)	0.1021 (2)	0.0124 (8)
C18	0.6118 (4)	0.7856 (3)	0.0980 (2)	0.0129 (8)
H18	0.5035	0.7787	0.1065	0.015*
C19	0.7057 (4)	0.6932 (3)	0.0818 (2)	0.0115 (8)
C20	0.8667 (4)	0.7033 (3)	0.0675 (2)	0.0119 (8)
C21	0.9285 (4)	0.8049 (3)	0.0737 (2)	0.0141 (8)
H21	1.0369	0.8115	0.0666	0.017*
C22	0.8334 (4)	0.8976 (3)	0.0905 (2)	0.0125 (8)
C23	0.8944 (4)	1.0105 (3)	0.0996 (2)	0.0145 (9)
H23A	0.9172	1.0164	0.1531	0.017*
H23B	0.9908	1.0210	0.0638	0.017*
C24	0.7760 (4)	1.0985 (3)	0.0820 (2)	0.0159 (9)
H24	0.7914	1.1737	0.0998	0.019*
C25	0.6979 (4)	1.0953 (3)	0.0170 (2)	0.0164 (9)
H25A	0.7191	1.0366	-0.0173	0.020*
H25B	0.6240	1.1518	0.0074	0.020*
C26	0.4943 (4)	0.5749 (3)	0.0873 (2)	0.0133 (8)
H26A	0.4735	0.4992	0.0739	0.016*
H26B	0.4481	0.6271	0.0509	0.016*

C27	0.4160 (5)	0.5940 (3)	0.1689 (2)	0.0169 (9)
C28	0.4414 (6)	0.5700 (5)	0.3024 (3)	0.0437 (15)
H28A	0.4375	0.4986	0.3326	0.052*
H28B	0.3363	0.6037	0.3066	0.052*
C29	0.5460 (6)	0.6441 (4)	0.3321 (3)	0.0381 (13)
H29A	0.5146	0.6521	0.3877	0.057*
H29B	0.5419	0.7166	0.3050	0.057*
H29C	0.6512	0.6128	0.3236	0.057*
C30	1.1126 (4)	0.6181 (4)	0.0286 (3)	0.0231 (10)
H30A	1.1345	0.6717	-0.0150	0.035*
H30B	1.1591	0.5460	0.0143	0.035*
H30C	1.1556	0.6428	0.0734	0.035*
C31	0.2328 (5)	1.2145 (4)	0.1273 (3)	0.0251 (10)
H31A	0.2168	1.2034	0.0739	0.038*
H31B	0.1889	1.2867	0.1429	0.038*
H31C	0.1825	1.1564	0.1616	0.038*
C32	0.4317 (5)	1.2398 (3)	0.2321 (2)	0.0211 (10)
H32A	0.3681	1.1871	0.2654	0.032*
H32B	0.3893	1.3148	0.2402	0.032*
H32C	0.5369	1.2344	0.2450	0.032*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pt1	0.01251 (8)	0.00795 (8)	0.01134 (8)	0.00155 (6)	-0.00278 (6)	-0.00031 (6)
Cl1	0.0175 (5)	0.0087 (4)	0.0138 (5)	0.0015 (4)	-0.0079 (4)	0.0003 (4)
S1	0.0154 (5)	0.0101 (5)	0.0172 (5)	0.0005 (4)	-0.0030 (4)	-0.0018 (4)
O1	0.0112 (14)	0.0105 (14)	0.0220 (16)	0.0021 (11)	-0.0064 (12)	0.0001 (12)
C1	0.014 (2)	0.013 (2)	0.0076 (19)	-0.0029 (16)	-0.0023 (15)	0.0012 (16)
O2	0.0182 (16)	0.0217 (16)	0.0206 (16)	0.0041 (13)	0.0001 (13)	0.0026 (14)
C2	0.0085 (19)	0.013 (2)	0.013 (2)	0.0021 (15)	-0.0034 (15)	0.0027 (17)
O3	0.0178 (16)	0.0297 (18)	0.0167 (16)	0.0068 (13)	-0.0082 (12)	-0.0033 (14)
C3	0.015 (2)	0.012 (2)	0.0091 (19)	0.0005 (16)	-0.0014 (15)	-0.0008 (16)
O4	0.0104 (14)	0.0124 (14)	0.0260 (16)	-0.0008 (11)	-0.0057 (12)	-0.0043 (13)
C4	0.011 (2)	0.015 (2)	0.0098 (19)	-0.0015 (16)	-0.0005 (15)	-0.0020 (17)
O5	0.0228 (17)	0.0113 (15)	0.0325 (18)	0.0015 (12)	-0.0123 (14)	0.0016 (14)
C5	0.012 (2)	0.016 (2)	0.011 (2)	0.0013 (16)	-0.0023 (15)	0.0028 (17)
C6	0.015 (2)	0.0093 (19)	0.0113 (19)	0.0018 (15)	-0.0016 (16)	0.0029 (16)
C7	0.015 (2)	0.011 (2)	0.017 (2)	0.0027 (16)	-0.0030 (16)	0.0010 (17)
C8	0.018 (2)	0.0076 (19)	0.018 (2)	0.0069 (16)	-0.0065 (17)	0.0031 (17)
C9	0.019 (2)	0.013 (2)	0.018 (2)	0.0022 (17)	-0.0090 (17)	0.0021 (18)
C10	0.011 (2)	0.011 (2)	0.021 (2)	0.0013 (15)	-0.0069 (16)	-0.0015 (17)
C11	0.018 (2)	0.0085 (19)	0.021 (2)	-0.0001 (16)	-0.0074 (18)	0.0011 (18)
C12	0.029 (3)	0.030 (3)	0.022 (2)	0.005 (2)	-0.010 (2)	0.002 (2)
C13	0.045 (3)	0.040 (3)	0.042 (3)	0.004 (3)	-0.021 (3)	-0.012 (3)
C14	0.013 (2)	0.019 (2)	0.033 (3)	-0.0041 (17)	-0.0050 (18)	-0.005 (2)
C15	0.017 (2)	0.021 (2)	0.023 (2)	-0.0037 (18)	0.0020 (18)	-0.005 (2)
C16	0.029 (3)	0.018 (2)	0.015 (2)	-0.0038 (19)	-0.0016 (18)	-0.0051 (19)

Pt2	0.01299 (8)	0.00863 (8)	0.01108 (8)	-0.00005 (6)	0.00062 (6)	-0.00056 (6)
Cl2	0.0151 (5)	0.0100 (5)	0.0172 (5)	-0.0016 (4)	0.0066 (4)	-0.0005 (4)
S2	0.0166 (5)	0.0098 (5)	0.0122 (5)	0.0013 (4)	0.0014 (4)	-0.0003 (4)
O6	0.0114 (14)	0.0083 (14)	0.0218 (16)	-0.0002 (11)	-0.0003 (11)	-0.0023 (12)
O7	0.0241 (18)	0.0320 (19)	0.0250 (18)	0.0075 (15)	0.0061 (14)	-0.0023 (15)
O8	0.0264 (18)	0.040 (2)	0.0173 (16)	-0.0047 (15)	-0.0061 (13)	0.0073 (15)
O9	0.0133 (15)	0.0135 (15)	0.0226 (16)	0.0023 (11)	0.0000 (12)	-0.0047 (13)
O10	0.0254 (17)	0.0120 (15)	0.0171 (15)	0.0016 (12)	0.0028 (12)	0.0016 (13)
C17	0.016 (2)	0.012 (2)	0.0084 (19)	0.0031 (16)	-0.0016 (16)	-0.0037 (16)
C18	0.012 (2)	0.014 (2)	0.012 (2)	-0.0013 (16)	-0.0001 (15)	-0.0004 (17)
C19	0.016 (2)	0.0100 (19)	0.0093 (19)	-0.0021 (16)	-0.0034 (15)	-0.0005 (16)
C20	0.013 (2)	0.012 (2)	0.0104 (19)	0.0047 (16)	0.0001 (15)	-0.0022 (17)
C21	0.013 (2)	0.016 (2)	0.013 (2)	-0.0021 (16)	0.0001 (16)	0.0010 (17)
C22	0.015 (2)	0.014 (2)	0.0091 (19)	-0.0021 (16)	-0.0008 (15)	-0.0020 (17)
C23	0.013 (2)	0.014 (2)	0.017 (2)	-0.0027 (16)	-0.0034 (16)	-0.0016 (18)
C24	0.020 (2)	0.010 (2)	0.018 (2)	-0.0045 (16)	0.0005 (17)	-0.0035 (17)
C25	0.014 (2)	0.020 (2)	0.014 (2)	0.0008 (17)	0.0031 (16)	-0.0012 (18)
C26	0.012 (2)	0.0100 (19)	0.019 (2)	-0.0016 (15)	-0.0049 (16)	-0.0011 (17)
C27	0.019 (2)	0.013 (2)	0.019 (2)	-0.0073 (17)	-0.0025 (17)	0.0033 (18)
C28	0.046 (3)	0.067 (4)	0.020 (3)	-0.025 (3)	-0.010 (2)	0.010 (3)
C29	0.034 (3)	0.045 (3)	0.035 (3)	-0.002 (2)	-0.003 (2)	-0.010 (3)
C30	0.014 (2)	0.020 (2)	0.035 (3)	0.0038 (18)	0.0011 (19)	-0.011 (2)
C31	0.018 (2)	0.024 (2)	0.033 (3)	0.0032 (19)	-0.006 (2)	0.003 (2)
C32	0.028 (2)	0.019 (2)	0.016 (2)	0.0045 (19)	0.0003 (18)	-0.0027 (19)

*Geometric parameters (Å, °)*

Pt1—Cl1	2.3449 (9)	Pt2—Cl2	2.3284 (9)
Pt1—S1	2.3448 (10)	Pt2—S2	2.3507 (10)
Pt1—C1	2.025 (4)	Pt2—C17	2.027 (4)
Pt1—C8	2.151 (4)	Pt2—C24	2.149 (4)
Pt1—C9	2.140 (4)	Pt2—C25	2.140 (4)
S1—O5	1.480 (3)	S2—O10	1.482 (3)
S1—C15	1.773 (4)	S2—C31	1.781 (4)
S1—C16	1.778 (4)	S2—C32	1.775 (4)
O1—C3	1.385 (4)	O6—C19	1.378 (4)
O1—C10	1.419 (4)	O6—C26	1.425 (4)
C1—C2	1.402 (5)	O7—C27	1.194 (5)
C1—C6	1.399 (5)	O8—C27	1.326 (5)
O2—C11	1.201 (5)	O8—C28	1.462 (6)
C2—H2	0.9500	O9—C20	1.365 (4)
C2—C3	1.384 (5)	O9—C30	1.435 (5)
O3—C11	1.337 (5)	C17—C18	1.394 (5)
O3—C12	1.457 (5)	C17—C22	1.397 (5)
C3—C4	1.403 (5)	C18—H18	0.9500
O4—C4	1.373 (4)	C18—C19	1.383 (5)
O4—C14	1.429 (4)	C19—C20	1.412 (5)
C4—C5	1.384 (5)	C20—C21	1.384 (5)

C5—H5	0.9500	C21—H21	0.9500
C5—C6	1.403 (5)	C21—C22	1.393 (5)
C6—C7	1.506 (5)	C22—C23	1.517 (5)
C7—H7A	0.9900	C23—H23A	0.9900
C7—H7B	0.9900	C23—H23B	0.9900
C7—C8	1.511 (5)	C23—C24	1.510 (5)
C8—H8	1.0000	C24—H24	1.0000
C8—C9	1.399 (6)	C24—C25	1.409 (5)
C9—H9A	0.9500	C25—H25A	0.9500
C9—H9B	0.9500	C25—H25B	0.9500
C10—H10A	0.9900	C26—H26A	0.9900
C10—H10B	0.9900	C26—H26B	0.9900
C10—C11	1.515 (5)	C26—C27	1.521 (6)
C12—H12A	0.9900	C28—H28A	0.9900
C12—H12B	0.9900	C28—H28B	0.9900
C12—C13	1.492 (6)	C28—C29	1.474 (6)
C13—H13A	0.9800	C29—H29A	0.9800
C13—H13B	0.9800	C29—H29B	0.9800
C13—H13C	0.9800	C29—H29C	0.9800
C14—H14A	0.9800	C30—H30A	0.9800
C14—H14B	0.9800	C30—H30B	0.9800
C14—H14C	0.9800	C30—H30C	0.9800
C15—H15A	0.9800	C31—H31A	0.9800
C15—H15B	0.9800	C31—H31B	0.9800
C15—H15C	0.9800	C31—H31C	0.9800
C16—H16A	0.9800	C32—H32A	0.9800
C16—H16B	0.9800	C32—H32B	0.9800
C16—H16C	0.9800	C32—H32C	0.9800
S1—Pt1—C11	91.13 (3)	Cl2—Pt2—S2	90.82 (3)
C1—Pt1—C11	94.05 (11)	C17—Pt2—Cl2	93.56 (11)
C1—Pt1—S1	174.66 (11)	C17—Pt2—S2	174.56 (11)
C1—Pt1—C8	81.65 (15)	C17—Pt2—C24	81.44 (15)
C1—Pt1—C9	86.40 (15)	C17—Pt2—C25	86.30 (16)
C8—Pt1—C11	162.70 (11)	C24—Pt2—Cl2	159.15 (11)
C8—Pt1—S1	93.01 (11)	C24—Pt2—S2	93.34 (11)
C9—Pt1—C11	158.91 (11)	C25—Pt2—Cl2	162.12 (11)
C9—Pt1—S1	89.25 (11)	C25—Pt2—S2	90.53 (11)
C9—Pt1—C8	38.06 (15)	C25—Pt2—C24	38.35 (14)
O5—S1—Pt1	115.04 (12)	O10—S2—Pt2	115.65 (12)
O5—S1—C15	108.52 (19)	O10—S2—C31	108.77 (19)
O5—S1—C16	108.95 (19)	O10—S2—C32	108.61 (19)
C15—S1—Pt1	110.52 (14)	C31—S2—Pt2	114.39 (15)
C15—S1—C16	100.2 (2)	C32—S2—Pt2	107.55 (14)
C16—S1—Pt1	112.51 (14)	C32—S2—C31	100.7 (2)
C3—O1—C10	118.1 (3)	C19—O6—C26	116.5 (3)
C2—C1—Pt1	126.1 (3)	C27—O8—C28	116.8 (4)
C6—C1—Pt1	114.9 (3)	C20—O9—C30	116.8 (3)

C6—C1—C2	119.0 (3)	C18—C17—Pt2	125.6 (3)
C1—C2—H2	119.8	C18—C17—C22	119.1 (4)
C3—C2—C1	120.3 (3)	C22—C17—Pt2	115.1 (3)
C3—C2—H2	119.8	C17—C18—H18	119.5
C11—O3—C12	116.3 (3)	C19—C18—C17	120.9 (4)
O1—C3—C4	114.4 (3)	C19—C18—H18	119.5
C2—C3—O1	125.1 (3)	O6—C19—C18	125.6 (3)
C2—C3—C4	120.4 (3)	O6—C19—C20	114.6 (3)
C4—O4—C14	117.0 (3)	C18—C19—C20	119.8 (3)
O4—C4—C3	115.7 (3)	O9—C20—C19	115.6 (3)
O4—C4—C5	124.5 (3)	O9—C20—C21	125.1 (3)
C5—C4—C3	119.8 (3)	C21—C20—C19	119.3 (3)
C4—C5—H5	120.1	C20—C21—H21	119.7
C4—C5—C6	119.9 (3)	C20—C21—C22	120.6 (4)
C6—C5—H5	120.1	C22—C21—H21	119.7
C1—C6—C5	120.5 (3)	C17—C22—C23	116.8 (3)
C1—C6—C7	117.1 (3)	C21—C22—C17	120.2 (3)
C5—C6—C7	122.3 (3)	C21—C22—C23	123.0 (3)
C6—C7—H7A	109.6	C22—C23—H23A	109.8
C6—C7—H7B	109.6	C22—C23—H23B	109.8
C6—C7—C8	110.2 (3)	H23A—C23—H23B	108.2
H7A—C7—H7B	108.1	C24—C23—C22	109.4 (3)
C8—C7—H7A	109.6	C24—C23—H23A	109.8
C8—C7—H7B	109.6	C24—C23—H23B	109.8
Pt1—C8—H8	116.0	Pt2—C24—H24	115.8
C7—C8—Pt1	107.7 (2)	C23—C24—Pt2	108.0 (3)
C7—C8—H8	116.0	C23—C24—H24	115.8
C9—C8—Pt1	70.6 (2)	C25—C24—Pt2	70.4 (2)
C9—C8—C7	121.5 (4)	C25—C24—C23	121.9 (4)
C9—C8—H8	116.0	C25—C24—H24	115.8
Pt1—C9—H9A	107.0	Pt2—C25—H25A	107.3
Pt1—C9—H9B	91.5	Pt2—C25—H25B	91.4
C8—C9—Pt1	71.4 (2)	C24—C25—Pt2	71.2 (2)
C8—C9—H9A	120.0	C24—C25—H25A	120.0
C8—C9—H9B	120.0	C24—C25—H25B	120.0
H9A—C9—H9B	120.0	H25A—C25—H25B	120.0
O1—C10—H10A	109.0	O6—C26—H26A	108.7
O1—C10—H10B	109.0	O6—C26—H26B	108.7
O1—C10—C11	112.8 (3)	O6—C26—C27	114.4 (3)
H10A—C10—H10B	107.8	H26A—C26—H26B	107.6
C11—C10—H10A	109.0	C27—C26—H26A	108.7
C11—C10—H10B	109.0	C27—C26—H26B	108.7
O2—C11—O3	125.0 (4)	O7—C27—O8	124.7 (4)
O2—C11—C10	125.9 (4)	O7—C27—C26	124.0 (4)
O3—C11—C10	109.1 (3)	O8—C27—C26	111.3 (4)
O3—C12—H12A	109.6	O8—C28—H28A	110.2
O3—C12—H12B	109.6	O8—C28—H28B	110.2
O3—C12—C13	110.4 (4)	O8—C28—C29	107.3 (4)

H12A—C12—H12B	108.1	H28A—C28—H28B	108.5
C13—C12—H12A	109.6	C29—C28—H28A	110.2
C13—C12—H12B	109.6	C29—C28—H28B	110.2
C12—C13—H13A	109.5	C28—C29—H29A	109.5
C12—C13—H13B	109.5	C28—C29—H29B	109.5
C12—C13—H13C	109.5	C28—C29—H29C	109.5
H13A—C13—H13B	109.5	H29A—C29—H29B	109.5
H13A—C13—H13C	109.5	H29A—C29—H29C	109.5
H13B—C13—H13C	109.5	H29B—C29—H29C	109.5
O4—C14—H14A	109.5	O9—C30—H30A	109.5
O4—C14—H14B	109.5	O9—C30—H30B	109.5
O4—C14—H14C	109.5	O9—C30—H30C	109.5
H14A—C14—H14B	109.5	H30A—C30—H30B	109.5
H14A—C14—H14C	109.5	H30A—C30—H30C	109.5
H14B—C14—H14C	109.5	H30B—C30—H30C	109.5
S1—C15—H15A	109.5	S2—C31—H31A	109.5
S1—C15—H15B	109.5	S2—C31—H31B	109.5
S1—C15—H15C	109.5	S2—C31—H31C	109.5
H15A—C15—H15B	109.5	H31A—C31—H31B	109.5
H15A—C15—H15C	109.5	H31A—C31—H31C	109.5
H15B—C15—H15C	109.5	H31B—C31—H31C	109.5
S1—C16—H16A	109.5	S2—C32—H32A	109.5
S1—C16—H16B	109.5	S2—C32—H32B	109.5
S1—C16—H16C	109.5	S2—C32—H32C	109.5
H16A—C16—H16B	109.5	H32A—C32—H32B	109.5
H16A—C16—H16C	109.5	H32A—C32—H32C	109.5
H16B—C16—H16C	109.5	H32B—C32—H32C	109.5
Pt1—C1—C2—C3	179.5 (3)	Pt2—C17—C18—C19	174.8 (3)
Pt1—C1—C6—C5	-178.4 (3)	Pt2—C17—C22—C21	-175.0 (3)
Pt1—C1—C6—C7	4.5 (4)	Pt2—C17—C22—C23	6.9 (4)
O1—C3—C4—O4	1.3 (5)	O6—C19—C20—O9	2.3 (5)
O1—C3—C4—C5	-179.2 (3)	O6—C19—C20—C21	-177.9 (3)
O1—C10—C11—O2	6.4 (6)	O6—C26—C27—O7	143.6 (4)
O1—C10—C11—O3	-172.8 (3)	O6—C26—C27—O8	-39.0 (5)
C1—C2—C3—O1	-179.8 (3)	O9—C20—C21—C22	176.8 (3)
C1—C2—C3—C4	-0.9 (6)	C17—C18—C19—O6	179.9 (4)
C1—C6—C7—C8	-24.6 (5)	C17—C18—C19—C20	-1.4 (6)
C2—C1—C6—C5	1.9 (6)	C17—C22—C23—C24	-27.0 (5)
C2—C1—C6—C7	-175.2 (3)	C18—C17—C22—C21	1.1 (6)
C2—C3—C4—O4	-177.7 (3)	C18—C17—C22—C23	-177.0 (3)
C2—C3—C4—C5	1.8 (6)	C18—C19—C20—O9	-176.6 (3)
C3—O1—C10—C11	-67.9 (4)	C18—C19—C20—C21	3.2 (6)
C3—C4—C5—C6	-0.7 (6)	C19—O6—C26—C27	-67.5 (4)
O4—C4—C5—C6	178.7 (3)	C19—C20—C21—C22	-2.9 (6)
C4—C5—C6—C1	-1.1 (6)	C20—C21—C22—C17	0.8 (6)
C4—C5—C6—C7	175.8 (4)	C20—C21—C22—C23	178.8 (4)
C5—C6—C7—C8	158.4 (4)	C21—C22—C23—C24	155.0 (4)



C6—C1—C2—C3	-0.9 (5)	C22—C17—C18—C19	-0.8 (6)
C6—C7—C8—Pt1	30.8 (4)	C22—C23—C24—Pt2	32.2 (4)
C6—C7—C8—C9	-46.8 (5)	C22—C23—C24—C25	-45.5 (5)
C7—C8—C9—Pt1	99.3 (3)	C23—C24—C25—Pt2	99.6 (3)
C10—O1—C3—C2	-12.4 (5)	C26—O6—C19—C18	1.5 (5)
C10—O1—C3—C4	168.6 (3)	C26—O6—C19—C20	-177.4 (3)
C11—O3—C12—C13	89.2 (5)	C27—O8—C28—C29	-113.4 (5)
C12—O3—C11—O2	2.6 (6)	C28—O8—C27—O7	-5.1 (6)
C12—O3—C11—C10	-178.2 (3)	C28—O8—C27—C26	177.5 (3)
C14—O4—C4—C3	-171.6 (3)	C30—O9—C20—C19	176.5 (3)
C14—O4—C4—C5	9.0 (5)	C30—O9—C20—C21	-3.3 (6)

*Hydrogen-bond geometry (Å, °)*

Cg1 and Cg2 are the centroids of C1–C6 and C17–C22 rings, respectively.

<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>
C9—H9 <i>B</i> ...O5	0.95	2.40	3.092 (4)	130
C10—H10 <i>A</i> ...O5 <sup>i</sup>	0.99	2.40	3.230 (4)	142
C16—H16 <i>C</i> ...O7	0.98	2.42	3.213 (5)	138
C25—H25 <i>B</i> ...O10	0.95	2.41	3.129 (4)	132
C26—H26 <i>A</i> ...O10 <sup>ii</sup>	0.99	2.42	3.337 (4)	153
C26—H26 <i>B</i> ...O10 <sup>iii</sup>	0.99	2.46	3.289 (4)	141
C28—H28 <i>A</i> ...O4 <sup>ii</sup>	0.99	2.45	3.346 (6)	150
C30—H30 <i>B</i> ...O9 <sup>iv</sup>	0.98	2.50	3.222 (5)	130
C30—H30 <i>C</i> ...O7 <sup>v</sup>	0.98	2.40	3.334 (6)	160
C32—H32 <i>B</i> ...O2	0.98	2.41	3.242 (5)	143
C15—H15 <i>A</i> ...Cg1 <sup>vi</sup>	0.98	2.49	3.405 (5)	155
C31—H31 <i>A</i> ...Cg2 <sup>iii</sup>	0.98	2.77	3.732 (5)	166
C32—H32 <i>A</i> ...Cg1	0.98	2.66	3.269 (4)	120

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