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cis-Bis(dimethyl sulfide)dinitrato-platinum(II) at 295 and 150 K

Christian Hansson* and Åke Oskarsson

Organic Chemistry, Department of Chemistry, Lund University, PO Box 124, SE-221 00 Lund, Sweden
Correspondence e-mail: christian.hansson@organic.lu.se

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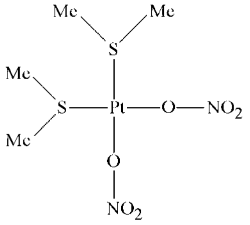
The title compound, $[Pt(NO_3)_2(C_2H_6S)_2]$, crystallizes in the $P2_1/n$ space group ($Z' = 2$), with pseudo-square-planar coordination geometry. The complex forms dimers with pseudosymmetry C_i arranged in columns along the *b*-axis direction, with Pt$\cdots$Pt distances of 6.3056 (3) and 4.2382 (2) Å (at 150 K). Each column is surrounded by six other columns in a honeycomb rod-like packing. The coordination mode of the nitrate ligands is monodentate, with Pt—O—N angles ranging from ~117 to ~118° and a tilt between the nitrate ligands and the coordination planes in the range ~63–70° (at 150 K). The coordination mode of the nitrate ligands is compared with that observed in reported $Pt(NO_3)_2L_2$ complexes (where L is a ligand with a donor atom from group 15 or 16), all of which are monodentate, with an average Pt—O—N angle of 118 (2)° and a tilt in the range 90 ± 30° (with two exceptions, in which the nitrates are approximately in the coordination plane).

Comment

The *cis*/*trans*-PtX_2L_2 complexes, where X is a halogen and L is a ligand with a donor atom from groups 14, 15 or 16, have been studied extensively in the solid state [Cambridge Structural Database (CSD), Version 5.28 of November 2006; Allen, 2002]. However, data for *cis*/*trans*-PtX_2L_2 with $X = NO_3$ are relatively scarce. We report here the structure of the title compound, (I), with emphasis on (*a*) the coordination mode of the nitrate ion in relation to other *cis*/*trans*-$Pt(NO_3)_2L_2$ structures and (*b*) the packing arrangement of the complex. As the crystal structure analysis showed some abnormal displacement ellipsoid parameters at 295 K, data were re-collected at 150 K.

cis-$Pt(NO_3)_2(Me_2S)_2$ (Fig. 1) crystallizes in $P2_1/n$ with two independent complexes that have angles around the Pt^{II} centres ranging from 86.24 (13) to 92.82 (4)° at 150 K. The Pt—S bond lengths range from 2.2390 (11) to 2.2499 (12) Å, and the Pt—O bond lengths are in the range 2.053 (3)–2.063 (3) Å. These values illustrate the different *trans* influences of the O and S atoms. The Pt—O distances are elongated (~0.05 Å) compared with those in $[Pt(NO_3)_4]^{2-}$ (Elding & Oskarsson, 1985), and the Pt—S distances are shortened by a similar amount when compared with $[Pt(Me_2S)_4]^{2+}$ (Bugarcic *et al.*, 1991). The parameters describing the coordination mode are defined in Fig. 2. The nitrate ions clearly coordinate in a monodentate fashion, with the second closest O atom located about 3.0 Å from the Pt^{II} atom (Table 1). This is further supported by the N—O distances, which for the coordinated O atoms are about 0.1 Å longer than for the uncoordinated O atoms, which are both similar. Both nitrate ions are located on the same side of the coordination plane, with Pt—O_A—N angles in a narrow range (~117–118°; Table 3). The Pt—O_A—N—O_B torsion angle is a measure of the deviation of the Pt atom from the nitrate plane; these angles are less than 5°, with the exception of the Pt1—O1—N1—O3 angle [18.7 (5)°]. The tilts between the nitrate plane and the coordination plane are ~63–70° (from the S—Pt—O_A—N torsion angles; see deposited material). The Me_2S



(I)

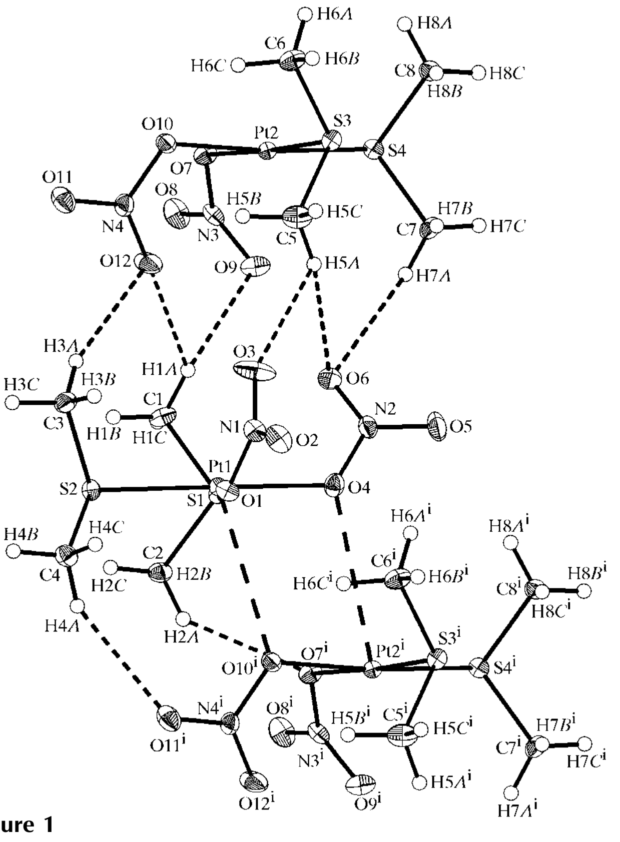


Figure 1
The atomic numbering scheme and the two pseudocentrosymmetric dimers, *viz.* Pt1$\cdots$Pt2 and Pt1$\cdots$Pt2^i, for (I) at 150 K. Dashed lines denote either H$\cdots$O or Pt$\cdots$O interactions. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (i) $x, y - 1, z$.]

molecules coordinate to Pt^{II} in a staggered or nearly staggered fashion with respect to the coordination plane. The two sulfide ligands are oriented in the same direction, and the Me_2S molecule with the largest deviation from the staggered mode is the S2 ligand, which has C3—S2—Pt1—O1 and C4—S2—Pt1—O1 torsion angles of ~66 and ~−41°, respectively, at both temperatures.

Neither of the complexes show pseudosymmetry, although (I) may adopt molecular point group symmetries C_2, C_s or C_{2v}. Density functional theory (DFT) calculations for the complex in the gas phase using the observed geometry as starting parameters result in an energy only 6 kJ mol^{-1} higher than that for a C_s geometry with the Me_2S ligands pointing away from one another.

Intramolecular S···O interactions are present [S1···O6 = 3.046 (3) Å and S4···O9 = 3.091 (5) Å at 150 K], as well as an intramolecular C—H···O interaction [H4*C*···O1 = 2.61 Å in the Pt1 complex, and H6*C*···O10 = 2.58 Å in the Pt2 complex (at 150 K)].

The difference in geometry (excluding H atoms) between the two complexes was analysed by an r.m.s. overlay (Fig. 3), yielding 0.177 Å at 150 K and 0.153 Å at 295 K. The difference between the complexes at the two temperatures has been analysed by a half-normal probability plot. The 28 bonds involving non-H atoms in each asymmetric unit were used in the analysis. Plotting observed values of δm_i *versus* the values α_i expected for a half-normal distribution of errors (*International Tables for X-ray Crystallography*, 1974, Vol. IV) gives a straight line $\delta m_i = 3.08\alpha_i + 0.102$ with $R = 0.986$, indicating negligible systematic differences but overly optimistic s.u. values in one or both structure analyses, with no outliers.

In space group $P2_1/n$, with $Z' = 2$, there are more than 14 possible choices for the pair of complexes in the asymmetric unit. Some result in a close contact of the two complexes and may be called a dimer, and some are more remote. Fig. 1 shows two reasonable choices for the asymmetric unit. The Pt1 and Pt2 complexes form a sandwiched dimer with pseudosymmetry C_i across (0.51, 0.51, 0.76) [$\sim(\frac{1}{2}, \frac{1}{2}, \frac{3}{4})$], stabilized by C—H···O interactions (Table 2), and have a Pt1···Pt2 distance of 6.3056 (3) Å. A second choice is the complexes Pt1 and $Pt2^i$, which also form a sandwiched dimer with pseudosymmetry C_i but now across (0.51, 0.00, 0.76) [$\sim(\frac{1}{2}, 0, \frac{3}{4})$], stabilized by $Pt1\cdots O10^i$ and $Pt2^i\cdots O4$ interactions, and with a $Pt1\cdots Pt2^i$ distance of 4.2382 (2) Å (symmetry code as in Table 1). DFT calculations on this second dimer show a stabilizing energy of ~70 kJ (mole monomer)$^{-1}$ in the gas phase, which seems too high. The dimers are stacked in columns along the *b* axis. The centres of pseudosymmetry, *viz.* $(\frac{1}{2}, \frac{1}{2}, \frac{3}{4})$ and $(\frac{1}{2}, 0, \frac{3}{4})$, are as far away as possible from the space group operators, 2_1, *n* and $\overline{1}$, in accordance with the findings of Collins (2006) for molecular pairs in $Z' = 2$ structures. Each column is surrounded by six other columns in a pseudo-honeycomb rod-like packing (O'Keeffe & Andersson, 1977) stabilized by C—H···O (H···O = 2.47–2.69 Å and C—H···O = 117–161°) and C—H···S (H···S = 2.96 Å and C—H···S = 149°) interactions and S···O contacts [3.228 (5) and 3.255 (4) Å] (Fig. 4). The H···O distances are long but still shorter than the average CH_2—H···O distance [2.761 (6) Å] retrieved from 767 structures in the CSD (Steiner & Desiraju, 1998).

The symmetry centres create voids of different sizes. Rings of four molecules are formed around the inversion centres at $(0, \frac{1}{2}, \frac{1}{2})$ with $H8B^{iv}\cdots S1^{v}$ and $H8A^{iv}\cdots O5^{iii}$ interactions; at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ with $H5A\cdots O3$ and $H4C^{ii}\cdots O11$ interactions; at $(0, \frac{1}{2}, 0)$ with $H4A^{iii}\cdots O11^{iv}$ and $H4C^{iii}\cdots O11^{vi}$ interactions; and at $(\frac{1}{2}, \frac{1}{2}, 0)$ with $H1A^{ii}\cdots O9^{ii}$ and $H8B^{vii}\cdots S1^{ii}$ interactions [symmetry codes: (ii) $-x + 1, -y + 1, -z + 1$; (iii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (iv) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$; (v) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$; (vi) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (vii) $x, y, z - 1$].

A search of the CSD for *cis*/*trans*-$Pt^{II}(NO_3)_2L_2$ complexes, where *L* is a ligand with a donor atom from groups 14, 15 or 16, using the *ConQuest* software (Bruno *et al.*, 2002), resulted in 11 *cis* and two *trans* compounds (Table 3). The dominance of the *cis* complexes may indicate a preference for *cis* complexes in the solid state, in contrast to the result of DFT calculations in the gas phase. The energies of two complexes reported as both *cis* and *trans* in the CSD are 30–40 kJ mol^{-1} lower for the corresponding *trans* complexes. All complexes listed in Table 3 have $l_2 - l_1 > 0.75$ Å, thus fulfilling the criterion for monodentate coordination for nitrate ligands, *viz.*

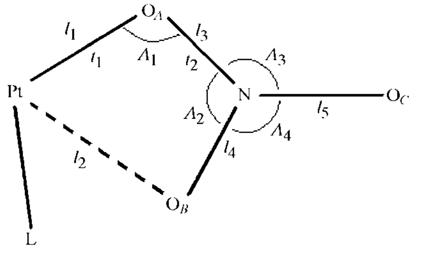


Figure 2
A schematic representation of the parameters defining the coordination mode of the nitrate ligands; *l* are distances, *A* angles and *t* the torsion angles $L(cis)$—Pt—O_A—N and Pt—O_A—N—O_B.

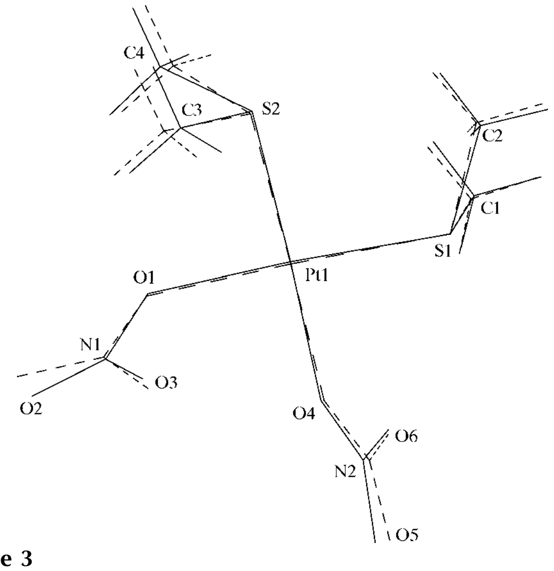


Figure 3
An r.m.s. overlay plot of the two molecules of (I) in the asymmetric unit.

$l_2 - l_1 > 0.6$, suggested by Kleywegt *et al.* (1985) (see Fig. 2 for definition of parameters describing the coordination mode). This is further supported by the l_3 distances, which have an average of 1.31 (2) Å, where the error is calculated from $\langle[\sum(L - L_{av})^2]/(n - 1)\rangle^{1/2}$, compared with l_4 and l_5 which are similar and placed together give an average of 1.22 (2) Å. The angle A_1 is slightly below 120°, as expected for a simple bonding model with O_A sp^2-hybridized, with an average of 118 (3)°. The angle A_4 is consistently greater than 120°, with an average of 123.8 (12)°, correlating well with the elongated l_3 distance. The torsion angle t_1 gives the orientation of the nitrate ions with respect to the coordination plane; nitrate ions located on opposite sides of the coordination plane have the same sign on t_1. Out of 14 *cis* complexes, seven have nitrate ions on opposite sides, five on the same side and two in the plane. With the exception of the ions roughly in the coordination plane, the nitrate ions form an angle of 90 ± 30° with the coordination plane. The Pt atom is normally close to the nitrate plane, as shown by t_2, but exceptions with t_2 as large as −33.3° have been observed.

The *cis* complexes also give information about the *trans* influence of the donor atom *trans* to the nitrate ligand. The Pt—O distances are 2.02–2.05 Å in the *N*-donor complexes, 2.04–2.06 Å in the *S*-donor complexes and 2.08–2.14 Å in the *P*-donor complexes, suggesting the following *trans* influence series between the donor atoms: N < S < P. Although the number of complexes is small, the series is in agreement with previous findings (Otto & Johansson, 2002).

The Kitaigordosky packing indices (KPI), calculated as described by Hansson (2007), are 0.72 and 0.74 at 295 and 150 K, respectively, compared with 0.65 and 0.68 for *cis*-$PtCl_2(Et_2S)_2$ (Hansson, 2007). Compound (I) is thus more close packed, which is further supported by the different thermal expansion coefficients [$1/V\,dV/dT$; 1.5×10^{-4} K^{-1} for (I) and 2.3×10^{-4} K^{-1} for *cis*-$PtCl_2(Et_2S)_2$].

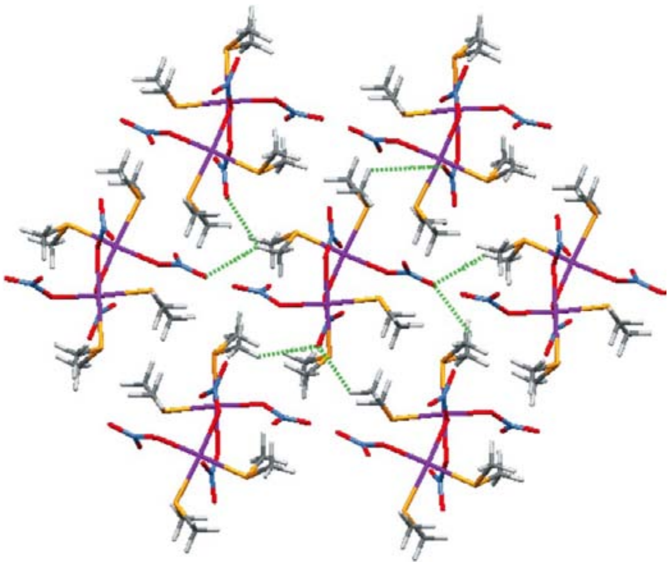


Figure 4

The pseudo-honeycomb rod-like packing of (I), viewed along the *b*-axis direction. Dashed lines denote C—H···O interactions.

Experimental

$PtI_2(Me_2S)_2$ (0.301 g, 0.526 mmol) was dissolved in acetone (15 ml). $AgNO_3$ (0.179 g, 1.052 mmol) was added and the reaction mixture was stirred for 90 min. A yellow precipitate of AgI was then removed by filtration. The pale-yellow acetone solution was left to evaporate slowly, which resulted in pale-yellow crystals suitable for X-ray diffraction experiments. The reaction between $PtI_2(Me_2S)_2$ and $AgNO_3$ is believed to be faster than the corresponding reaction with $PtCl_2(Me_2S)_2$ (Souchard *et al.*, 1990). The solubility of $AgNO_3$ in acetone is very limited, but still the reaction is rather fast. A change in colour, from light red to pale yellow, indicates when the reaction is finished. DFT calculations were performed at the s-VWN level with the basis sets def-TZVPP for Pt, TZVPP for O and N, and 6–31G* for C and H atoms, using the software *TURBOMOLE* (Version 5.5; Alrichs *et al.*, 1989).

Compound (I) at 295 K

Crystal data

$[Pt(NO_3)_2(C_2H_6S)_2]$
$M_r = 443.37$
Monoclinic, $P2_1/n$
$a = 16.0762$ (11) Å
$b = 9.5937$ (5) Å
$c = 16.1552$ (11) Å
$\beta = 106.402$ (4)°
$V = 2390.2$ (3) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 12.10$ mm^{-1}
$T = 295$ (1) K
0.25 × 0.08 × 0.05 mm

Data collection

Oxford Diffraction Xcalibur3 diffractometer
Absorption correction: numerical (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min} = 0.405$, $T_{max} = 0.771$
23666 measured reflections
8172 independent reflections
4952 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.120$
$S = 0.95$
8172 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 3.82$ e Å^{-3}
$\Delta\rho_{min} = -2.66$ e Å^{-3}

Compound (I) at 150 K

Crystal data

$[Pt(NO_3)_2(C_2H_6S)_2]$
$M_r = 443.37$
Monoclinic, $P2_1/n$
$a = 15.9689$ (7) Å
$b = 9.5020$ (3) Å
$c = 16.0399$ (7) Å
$\beta = 105.940$ (3)°
$V = 2340.26$ (16) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 12.36$ mm^{-1}
$T = 150$ (1) K
0.25 × 0.08 × 0.05 mm

Data collection

Oxford Diffraction Xcalibur3 diffractometer
Absorption correction: numerical (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min} = 0.374$, $T_{max} = 0.771$
23004 measured reflections
8007 independent reflections
6244 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.077$
$S = 0.97$
8007 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 2.62$ e Å^{-3}
$\Delta\rho_{min} = -2.34$ e Å^{-3}

Table 1
Selected interatomic distances (Å) for (I) at 150 K.

Pt1—O1	2.053 (3)	Pt2—O7	2.053 (3)
Pt1—O4	2.061 (3)	Pt2—O10	2.063 (3)
Pt1—S1	2.2411 (11)	Pt2—S3	2.2390 (11)
Pt1—S2	2.2424 (11)	Pt2—S4	2.2499 (12)
Pt1···Pt2	6.3056 (3)	Pt1···O3	2.969 (4)
Pt1···Pt2^{i}	4.2382 (2)	Pt1···O6	2.985 (3)
Pt1···O10^{i}	3.325 (3)	Pt2···O9	2.999 (3)
Pt2^{i}···O4	3.331 (3)	Pt2···O12	2.951 (5)

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

Table 2
Hydrogen-bond geometry (Å, °) for (I) at 150 K.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O12	0.96	2.40	3.146 (6)	134
C1—H1*A*···O9	0.96	2.65	3.358 (6)	131
C2—H2*A*···O7^{i}	0.96	2.54	3.288 (6)	135
C3—H3*A*···O12	0.96	2.68	3.624 (6)	168
C4—H4*A*···O11^{i}	0.96	2.50	3.398 (6)	156
C5—H5*A*···O3	0.96	2.56	2.997 (7)	108
C6—H6*B*···O5^{v}	0.96	2.52	3.209 (6)	128
C8^{iv}—H8*A*^{iv}···O5^{iii}	0.96	2.47	3.393 (6)	161

Symmetry codes: (i) $x, y-1, z$; (v) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Table 3
The coordination mode of the nitrate ion for *cis*-$Pt(NO_3)_2L_2$ compounds found in the CSD.

A complete table with all parameters defined in Fig. 2 is deposited.

Refcode	Donor	l_1	l_2	l_3	A_1	A_4
(I)[a]	S	2.053 (3)	2.969 (4)	1.312 (5)	117.2 (2)	124.2 (4)
	S	2.061 (3)	2.985 (3)	1.320 (5)	117.9 (3)	124.9 (4)
	S	2.053 (3)	2.99 (3)	1.326 (5)	118.1 (3)	124.4 (4)
	S	2.063 (3)	2.951 (5)	1.317 (5)	117.3 (3)	124.2 (4)
APUMON[b]	P/P	2.10/2.13	2.90/2.91	1.33/1.30	113.5/114.7	126.8/124.7
ICUVEH[c]	N/N	2.02/2.03	2.96/2.98	1.34/1.27	117.0/121.1	123.5/122.7
ICUVO*R*[c]	N/N	2.02/2.03	2.94/2.92	1.32/1.30	117.1/117.4	125.3/123.9
ICUVU*X*[c]	N/N	2.03/2.04	2.98/2.99	1.31/1.32	119.0/119.5	125.3/125.1
LALNIV[d]	P/P	2.11/2.14	2.99/3.02	1.31/1.29	118.2/118.0	124.1/123.3
NARQA*Z*[e]	N/N	2.04/2.05	2.98/2.98	1.32/1.32	117.7/117.6	124.9/124.4
QIHTO*Q*[f]	P/P	2.11/2.12	2.91/2.95	1.28/1.26	113.6/116.2	121.6/122.2
TAZZOJ[g]	S/S	2.04/2.06	3.07/2.95	1.36/1.31	115.8/115.7	123.1/124.3
WIJWE*R*[h]	P/P	2.09/2.10	2.98/2.91	1.26/1.29	119.5/115.9	122.7/123.0
XABZAC[i]	P/P	2.08/2.10	3.09/3.15	1.33/1.32	121.6/121.9	124.6/122.5
	P/P	2.09/2.10	3.13/3.14	1.32/1.31	122.4/122.3	121.1/122.5
XIKJAC[j]	P/P	2.13/2.13	2.88/2.88	1.31/1.31	111.8/111.8	123.6/123.6

Notes: (*a*) this work; (*b*) Longato *et al.* (2003); (*c*) Tessier & Rochon (2001); (*d*) Suzuki *et al.* (1993); (*e*) Grabner *et al.* (2005); (*f*) Arendse *et al.* (2001); (*g*) Boström *et al.* (1991); (*h*) Redwine *et al.* (2000); (*i*) Anandhi *et al.* (2003); (*j*) Kuehl *et al.* (2001).

trans-$Pt(NO_3)_2L_2$ compounds found in the CSD.

Refcode	Donor	l_1	l_2	l_3	A_1	A_4
ICUVAD[a]	N	2.01	2.96	1.31	118.7	123.5
ICUVI*L*[a]	N	2.02	2.93	1.32	117.9	125.0

Note: (*a*) Tessier & Rochon (2001).

H atoms were treated as riding on the adjacent C atoms [C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$]. A residual electron density of 3.8 e Å^{-3} at 295 K, located in the coordination plane and 1.6 Å from two C atoms, reduces to 1.4 e Å^{-3} at 150 K.

For both determinations, data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2000) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003) and *enCIFer* (Allen *et al.*, 2004).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3113). Services for accessing these data are described at the back of the journal.

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