

**This is an electronic reprint of the original article.
This reprint *may differ* from the original in pagination and typographic detail.**

Author(s): Tskhovrebov, Alexander; Haukka, Matti

Title: cis-Dichloridobis(2-isocyanophenyl 4-methoxybenzoate)palladium(II) chloroform monosolvate

Year: 2012

Version:

Please cite the original version:

Tskhovrebov, A., & Haukka, M. (2012). cis-Dichloridobis(2-isocyanophenyl 4-methoxybenzoate)palladium(II) chloroform monosolvate. *Acta Crystallographica Section E*, E68(12), m1476-m1477. <https://doi.org/10.1107/S1600536812045801>

All material supplied via JYX is protected by copyright and other intellectual property rights, and duplication or sale of all or part of any of the repository collections is not permitted, except that material may be duplicated by you for your research use or educational purposes in electronic or print form. You must obtain permission for any other use. Electronic or print copies may not be offered, whether for sale or otherwise to anyone who is not an authorised user.

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

cis-Dichloridobis(2-isocyanophenyl 4-methoxybenzoate)palladium(II) chloroform monosolvate

Alexander Tskhovrebov^a and Matti Haukka^{b*}

^aDepartment of Chemistry, Saint-Petersburg State University, Universitetsky Pr. 26, 198504 Stary Petergof, Russian Federation, and ^bUniversity of Jyväskylä, Department of Chemistry, PO Box 35, 40014 University of Jyväskylä, Finland
Correspondence e-mail: matti.o.haukka@jyu.fi

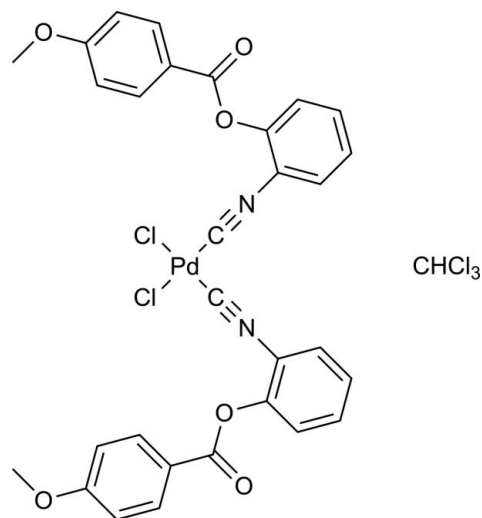
Received 25 September 2012; accepted 6 November 2012

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.037; wR factor = 0.067; data-to-parameter ratio = 22.6.

In the title compound, $[\text{PdCl}_2(\text{C}_{15}\text{H}_{11}\text{NO}_3)_2] \cdot \text{CHCl}_3$, the Pd^{II} atom adopts a slightly distorted square-planar coordination geometry composed of two Cl atoms in *cis* positions and two C atoms from isocyanophenyl ligands. The molecular conformation is stabilized by π - π stacking interactions [shortest centroid-centroid distance = 3.600 (1) Å] between substituted benzene rings of different ligands. The crystal packing is characterized by C-H...O and C-H...Cl interactions involving the chloroform solvent molecules.

Related literature

For further information on acyclic diaminocarbenes, see: Slaughter (2012); Boyarskiy *et al.* (2012). For background to the Passerini reaction, see: Banfi & Riva (2005). For novel metal-mediated coupling as a route to cyclic carbenes and aminocarbene complexes, see: Luzyanin *et al.* (2009*a,b*); Tskhovrebov *et al.* (2011); Chay *et al.* (2012). For related structures, see: Davies *et al.* (1996); Bertani *et al.* (1991); Bonati & Minghetti (1970); Luzyanin *et al.* (2009*a,b*); Michelin *et al.* (1988*a,b*); Rourke (2007). For bond lengths in coordination complexes, see: Orpen *et al.* (1989).



Experimental

Crystal data

 $[\text{PdCl}_2(\text{C}_{15}\text{H}_{11}\text{NO}_3)_2] \cdot \text{CHCl}_3$
 $M_r = 803.16$ Orthorhombic, $P2_12_12_1$ $a = 7.4457$ (1) Å $b = 12.1352$ (4) Å $c = 36.1109$ (11) Å $V = 3262.80$ (15) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 1.02$ mm⁻¹ $T = 100$ K $0.35 \times 0.23 \times 0.10$ mm

Data collection

Nonius KappaCCD diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 2008)

 $T_{\text{min}} = 0.717$, $T_{\text{max}} = 0.903$

24908 measured reflections

9228 independent reflections

7397 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.046$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$ $wR(F^2) = 0.067$ $S = 1.01$

9228 reflections

408 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.61$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.79$ e Å⁻³

Absolute structure: Flack (1983),

3936 Friedel pairs

Flack parameter: -0.011 (17)

Table 1

Selected bond lengths (Å).

Pd1—C16	1.935 (3)	Pd1—Cl2	2.2979 (7)
Pd1—Cl1	1.947 (3)	Pd1—Cl1	2.2994 (7)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C4—H4...O2 ⁱ	0.95	2.53	3.193 (4)	127
C6—H6...O6 ⁱⁱ	0.95	2.53	3.433 (4)	158
C19—H19...O5 ⁱⁱⁱ	0.95	2.37	3.182 (3)	143
C20—H20...Cl1 ^{iv}	0.95	2.80	3.622 (3)	145
C31—H31...Cl1 ^v	1.00	2.77	3.607 (3)	141
C31—H31...Cl2 ^v	1.00	2.67	3.513 (3)	142

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

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2009); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Russian Fund for Basic Research (grant 12–03–00076).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2184).

References

- Banfi, L. & Riva, R. (2005). *Org. React.* pp. 1–140.
- Bertani, R., Mozzon, M., Michelin, R. A., Benetollo, F., Bombieri, G., Castilho, T. J. & Pombeiro, A. J. L. (1991). *Inorg. Chim. Acta*, **189**, 175–187.
- Bonati, F. & Minghetti, G. (1970). *J. Organomet. Chem.* **24**, 251–256.
- BoyarSKIY, V. P., Luzyanin, K. V. & Kukushkin, V. Y. (2012). *Coord. Chem. Rev.* **256**, 2029–2056.
- Brandenburg, K. (2009). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Chay, R. S., Luzyanin, K. V., Kukushkin, V. Y., Guedes da Silva, M. F. C. & Pombeiro, A. J. L. (2012). *Organometallics*, **31**, 2379–2387.
- Davies, J. A., Hockensmith, C. M., Kukushkin, V. Y. & Kukushkin, Y. N. (1996). In *Synthetic Coordination Chemistry: Principles and Practice*. Singapore: World Scientific.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Luzyanin, K. V., Tskhovrebov, A. G., Carias, M. C., Guedes da Silva, M. F. C., Pombeiro, A. J. L. & Kukushkin, V. Y. (2009a). *Organometallics*, **28**, 6559–6566.
- Luzyanin, K. V., Tskhovrebov, A. G., Guedes da Silva, M. F. C., Haukka, M., Pombeiro, A. J. L. & Kukushkin, V. Y. (2009b). *Chem. Eur. J.* **15**, 5969–5978.
- Michelin, R. A., Zanutto, L., Braga, D., Sabatino, P. & Angelici, R. J. (1988a). *Inorg. Chem.* **27**, 85–92.
- Michelin, R. A., Zanutto, L., Braga, D., Sabatino, P. & Angelici, R. J. (1988b). *Inorg. Chem.* **27**, 93–99.
- Nonius (1997). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1989). *J. Chem. Soc. Dalton Trans.* pp. S1–S83.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Rourke, J. P. (2007). In *Comprehensive Organometallic Chemistry III*, 1st ed., edited by A. Canty, Vol. 8, ch. 8.07, pp. 405–444. Oxford: Elsevier.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Slaughter, L. M. (2012). *ACS Catal.* **2**, 1802–1816.
- Tskhovrebov, A. G., Luzyanin, K. V., Dolgushin, F. M., Guedes da Silva, M. F. C., Pombeiro, A. J. L. & Kukushkin, V. Y. (2011). *Organometallics*, **30**, 3362–3370.

supporting information

Acta Cryst. (2012). E68, m1476–m1477 [doi:10.1107/S1600536812045801]

cis-Dichloridobis(2-isocyanophenyl 4-methoxybenzoate)palladium(II) chloroform monosolvate**Alexander Tskhovrebov and Matti Haukka****S1. Comment**

Isocyanides are important organic reagents used in multicomponent reactions such as, *e.g.*, Ugi and Passerini reactions (Banfi & Riva, 2005). Metal complexes of isocyanides could be used as precursors for the generation of coordinated N-heterocyclic carbenes (NHC's) and acyclic diaminocarbenes (ADC's) (Slaughter, 2012). In turn, Pd^{II}-NHC and Pd^{II}-ADC systems are particularly interesting since they are used as catalysts in a wide range of cross-coupling reactions (Boyarskiy *et al.*, 2012). Recently, it was observed that the coupling of Pd^{II}-bound isocyanides and various nucleophiles leads to the formation of cyclic carbenes (Luzyanin *et al.*, 2009*b*) and ADC complexes (Luzyanin *et al.*, 2009*a*; Tskhovrebov *et al.*, 2011; Chay *et al.*, 2012), which could not be obtained by the common methods for the generation of metal carbenes. Here we report the structure of a new isocyanide complex that could be used as a starting material for generation of various palladium carbenes.

In the title compound, the isocyanide ligands are mutually in the *cis*-position (Fig. 1) insofar as the ligated RNC species exhibit higher *trans*-effect than the chlorides (Davies *et al.*, 1996). The fragments C–N–C–Pd in both complexes are almost linear, *viz.*, the angles N1–C1–Pd1 and N2–C16–Pd1 are 174.2 (2)° and 177.4 (3)°, respectively. The angles C2–N1–C1 and C17–N2–C16 are 174.3 (3)° and 172.0 (3)°, correspondingly. In the isocyanide moieties, the C≡N triple bonds [C1–N1 1.141 (3) Å and C16–N2 1.150 (3) Å] are close to those in some other palladium-isocyanide complexes (Bertani *et al.*, 1991; Bonati & Minghetti, 1970; Luzyanin *et al.*, 2009*a,b*; Michelin *et al.*, 1988*a,b*; Orpen *et al.*, 1989; Rourke, 2007). The molecular conformation is stabilized by π - π stacking interactions [shortest centroid-centroid distance = 3.600 (1) Å] between the substituted benzene rings C9–C15 and C17–C22 of different ligands. The crystal packing is characterized by intermolecular C–H⋯O and C–H⋯Cl interactions involving the chloroform solvent molecules (Table 1).

S2. Experimental

The title compound was synthesized by the addition of 2 equiv of 2-isocyanophenyl-4-methoxybenzoate into a chloroform solution of [PdCl₂(MeCN)₂]. The solid product was dissolved and recrystallized by slow evaporation from a solution of Et₂O/CHCl₃ (1:1, *v/v*).

S3. Refinement

All H atoms were positioned geometrically and constrained to ride on their parent atoms, with C–H = 0.95 Å and $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms, with C–H = 1.00 Å and $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for methine H atoms, and with C–H = 0.98 Å and $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms. The highest peak is located 1.28 Å from atom Cl6 and the deepest hole is located 0.78 Å from atom Pd1.

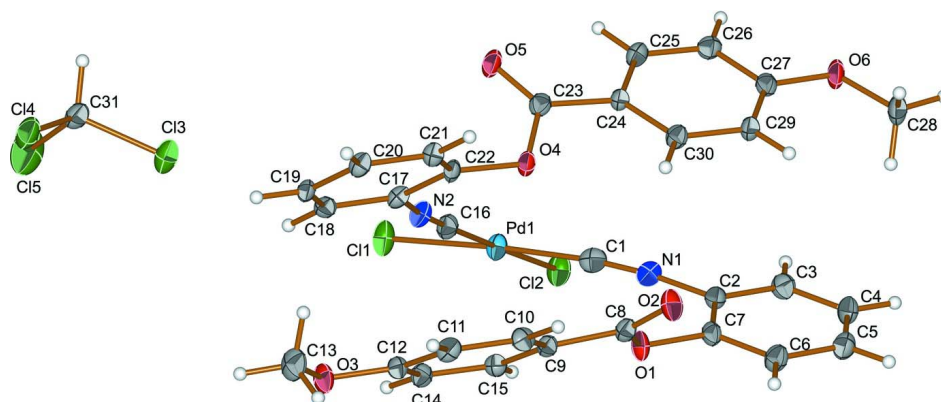


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

cis-Dichloridobis(2-isocyanophenyl 4-methoxybenzoate)palladium(II) chloroform monosolvate

Crystal data

$[\text{PdCl}_2(\text{C}_{15}\text{H}_{11}\text{NO}_3)_2] \cdot \text{CHCl}_3$

$M_r = 803.16$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.4457$ (1) Å

$b = 12.1352$ (4) Å

$c = 36.1109$ (11) Å

$V = 3262.80$ (15) Å³

$Z = 4$

$F(000) = 1608$

$D_x = 1.635$ Mg m⁻³

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

Cell parameters from 24908 reflections

$\theta = 3.2\text{--}30.0^\circ$

$\mu = 1.02$ mm⁻¹

$T = 100$ K

Block, colourless

$0.35 \times 0.23 \times 0.10$ mm

Data collection

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed tube

Horizontally mounted graphite crystal

monochromator

Detector resolution: 9 pixels mm⁻¹

φ scans and ω scans with κ offset

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2008)

$T_{\min} = 0.717$, $T_{\max} = 0.903$

24908 measured reflections

9228 independent reflections

7397 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.046$

$\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.2^\circ$

$h = -10 \rightarrow 10$

$k = -17 \rightarrow 15$

$l = -50 \rightarrow 41$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.067$

$S = 1.01$

9228 reflections

408 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 0.61$ e Å⁻³

$\Delta\rho_{\min} = -0.79$ e Å⁻³

Absolute structure: Flack (1983), 3936 Friedel pairs

Absolute structure parameter: -0.011 (17)

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	0.70069 (3)	0.468290 (17)	0.888077 (5)	0.01742 (5)
Cl1	0.77179 (10)	0.36814 (6)	0.940105 (18)	0.02588 (17)
Cl2	0.78655 (11)	0.32749 (5)	0.849385 (18)	0.02437 (15)
Cl3	0.19677 (12)	1.19482 (6)	0.947449 (18)	0.02811 (16)
Cl4	0.16546 (10)	1.10483 (7)	0.873361 (19)	0.03021 (18)
Cl5	0.30198 (14)	1.32462 (7)	0.88439 (2)	0.0512 (2)
O1	0.7349 (2)	0.81404 (15)	0.82187 (5)	0.0201 (4)
O2	0.5584 (2)	0.96590 (18)	0.81736 (5)	0.0243 (4)
O3	0.8859 (2)	0.99125 (16)	0.98090 (5)	0.0236 (5)
O4	0.3493 (2)	0.79408 (15)	0.89629 (5)	0.0176 (4)
O5	0.1775 (3)	0.65489 (16)	0.91802 (5)	0.0243 (5)
O6	0.0757 (3)	0.58547 (17)	0.74567 (5)	0.0231 (5)
N1	0.6421 (3)	0.60184 (19)	0.81643 (6)	0.0197 (5)
N2	0.5755 (3)	0.66170 (19)	0.93709 (6)	0.0181 (5)
C1	0.6542 (3)	0.5533 (2)	0.84336 (8)	0.0202 (6)
C2	0.6443 (3)	0.6656 (2)	0.78406 (7)	0.0181 (6)
C3	0.6047 (4)	0.6183 (3)	0.75010 (7)	0.0213 (6)
H3	0.5760	0.5422	0.7484	0.026*
C4	0.6076 (4)	0.6834 (3)	0.71872 (8)	0.0242 (7)
H4	0.5802	0.6522	0.6953	0.029*
C5	0.6502 (4)	0.7939 (3)	0.72142 (8)	0.0246 (7)
H5	0.6505	0.8383	0.6997	0.030*
C6	0.6925 (4)	0.8410 (2)	0.75529 (7)	0.0220 (6)
H6	0.7245	0.9166	0.7567	0.026*
C7	0.6877 (4)	0.7772 (2)	0.78685 (7)	0.0175 (6)
C8	0.6627 (4)	0.9124 (2)	0.83496 (7)	0.0175 (6)
C9	0.7281 (3)	0.9349 (2)	0.87254 (7)	0.0158 (6)
C10	0.6814 (3)	1.0340 (2)	0.88931 (7)	0.0187 (5)
H10	0.6137	1.0867	0.8758	0.022*
C11	0.7323 (3)	1.0570 (2)	0.92547 (7)	0.0186 (6)
H11	0.7003	1.1252	0.9366	0.022*
C12	0.8307 (3)	0.9794 (2)	0.94535 (7)	0.0185 (6)
C13	0.8259 (4)	1.0869 (2)	1.00095 (7)	0.0293 (7)
H13A	0.8686	1.1536	0.9884	0.044*
H13B	0.8739	1.0847	1.0262	0.044*

H13C	0.6944	1.0875	1.0019	0.044*
C14	0.8794 (3)	0.8798 (2)	0.92857 (7)	0.0176 (6)
H14	0.9465	0.8267	0.9421	0.021*
C15	0.8303 (3)	0.8586 (2)	0.89253 (7)	0.0173 (6)
H15	0.8662	0.7916	0.8811	0.021*
C16	0.6242 (4)	0.5885 (2)	0.91953 (7)	0.0194 (6)
C17	0.5048 (4)	0.7558 (2)	0.95387 (7)	0.0165 (6)
C18	0.5512 (3)	0.7851 (2)	0.98970 (7)	0.0176 (6)
H18	0.6306	0.7403	1.0038	0.021*
C19	0.4803 (3)	0.8805 (2)	1.00472 (7)	0.0173 (6)
H19	0.5105	0.9015	1.0293	0.021*
C20	0.3652 (3)	0.9456 (2)	0.98394 (7)	0.0176 (6)
H20	0.3170	1.0110	0.9944	0.021*
C21	0.3197 (4)	0.9165 (2)	0.94812 (7)	0.0171 (6)
H21	0.2407	0.9617	0.9341	0.021*
C22	0.3893 (3)	0.8223 (2)	0.93303 (7)	0.0152 (6)
C23	0.2352 (3)	0.7055 (2)	0.89214 (8)	0.0182 (6)
C24	0.1980 (4)	0.6801 (2)	0.85274 (7)	0.0153 (5)
C25	0.1211 (4)	0.5787 (2)	0.84476 (7)	0.0198 (6)
H25	0.0940	0.5293	0.8644	0.024*
C26	0.0834 (3)	0.5485 (2)	0.80881 (7)	0.0203 (6)
H26	0.0349	0.4778	0.8035	0.024*
C27	0.1174 (3)	0.6232 (2)	0.78018 (7)	0.0185 (6)
C28	0.1047 (4)	0.6582 (3)	0.71486 (7)	0.0269 (7)
H28A	0.0354	0.7260	0.7185	0.040*
H28B	0.0658	0.6221	0.6920	0.040*
H28C	0.2327	0.6763	0.7131	0.040*
C29	0.1902 (4)	0.7258 (2)	0.78777 (7)	0.0185 (6)
H29	0.2117	0.7765	0.7682	0.022*
C30	0.2316 (3)	0.7546 (2)	0.82398 (7)	0.0182 (6)
H30	0.2825	0.8247	0.8292	0.022*
C31	0.1513 (4)	1.2255 (2)	0.90068 (7)	0.0237 (7)
H31	0.0267	1.2557	0.8988	0.028*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.02268 (10)	0.01504 (10)	0.01454 (9)	0.00299 (10)	0.00058 (9)	-0.00068 (9)
Cl1	0.0409 (4)	0.0209 (4)	0.0159 (3)	0.0074 (3)	-0.0020 (3)	-0.0008 (3)
Cl2	0.0353 (4)	0.0195 (3)	0.0184 (3)	0.0051 (4)	0.0003 (3)	-0.0039 (3)
Cl3	0.0448 (4)	0.0220 (4)	0.0175 (3)	0.0023 (4)	-0.0028 (4)	0.0009 (3)
Cl4	0.0401 (4)	0.0340 (4)	0.0165 (3)	0.0044 (4)	-0.0049 (3)	-0.0018 (3)
Cl5	0.0628 (5)	0.0512 (6)	0.0397 (5)	-0.0301 (5)	-0.0031 (6)	0.0165 (4)
O1	0.0291 (11)	0.0193 (10)	0.0119 (9)	0.0024 (9)	-0.0041 (8)	-0.0030 (8)
O2	0.0310 (11)	0.0232 (11)	0.0186 (11)	0.0062 (11)	-0.0058 (8)	0.0005 (10)
O3	0.0283 (10)	0.0287 (13)	0.0139 (10)	0.0011 (9)	-0.0028 (8)	-0.0068 (9)
O4	0.0247 (10)	0.0177 (10)	0.0104 (10)	-0.0043 (8)	-0.0010 (7)	0.0006 (7)
O5	0.0324 (11)	0.0296 (12)	0.0109 (9)	-0.0083 (10)	0.0024 (9)	-0.0012 (8)

O6	0.0312 (11)	0.0284 (12)	0.0097 (10)	-0.0021 (10)	-0.0025 (8)	-0.0009 (9)
N1	0.0214 (12)	0.0170 (13)	0.0207 (13)	0.0035 (10)	0.0028 (10)	0.0013 (10)
N2	0.0240 (12)	0.0146 (13)	0.0157 (12)	0.0036 (10)	0.0030 (10)	0.0016 (10)
C1	0.0197 (14)	0.0167 (16)	0.0240 (15)	0.0023 (11)	0.0033 (11)	-0.0053 (12)
C2	0.0170 (13)	0.0228 (16)	0.0145 (14)	0.0005 (12)	0.0000 (11)	0.0000 (12)
C3	0.0226 (14)	0.0219 (16)	0.0194 (15)	-0.0021 (13)	0.0001 (12)	-0.0042 (13)
C4	0.0263 (15)	0.0324 (19)	0.0138 (15)	-0.0011 (14)	0.0004 (12)	-0.0041 (13)
C5	0.0286 (16)	0.0288 (17)	0.0165 (15)	-0.0035 (14)	0.0008 (12)	0.0008 (13)
C6	0.0283 (14)	0.0202 (15)	0.0176 (14)	-0.0023 (15)	0.0017 (14)	-0.0017 (11)
C7	0.0188 (13)	0.0208 (14)	0.0128 (12)	0.0011 (13)	-0.0010 (12)	-0.0037 (11)
C8	0.0210 (15)	0.0163 (14)	0.0150 (13)	-0.0017 (12)	0.0016 (11)	0.0010 (11)
C9	0.0185 (13)	0.0153 (13)	0.0137 (12)	-0.0027 (11)	0.0008 (11)	-0.0002 (10)
C10	0.0196 (12)	0.0157 (12)	0.0207 (13)	0.0019 (13)	-0.0011 (13)	0.0053 (13)
C11	0.0232 (14)	0.0135 (14)	0.0190 (13)	-0.0018 (11)	0.0029 (11)	-0.0015 (10)
C12	0.0182 (13)	0.0235 (15)	0.0138 (13)	-0.0040 (13)	-0.0001 (10)	-0.0022 (12)
C13	0.0363 (18)	0.0322 (18)	0.0192 (15)	-0.0030 (16)	0.0014 (14)	-0.0105 (13)
C14	0.0184 (13)	0.0176 (15)	0.0169 (14)	0.0011 (12)	-0.0009 (11)	0.0029 (12)
C15	0.0167 (13)	0.0175 (14)	0.0177 (14)	-0.0002 (11)	0.0003 (11)	-0.0001 (11)
C16	0.0210 (14)	0.0214 (16)	0.0158 (14)	0.0002 (13)	0.0001 (11)	0.0043 (12)
C17	0.0188 (13)	0.0155 (14)	0.0152 (14)	0.0003 (12)	0.0039 (11)	0.0017 (12)
C18	0.0159 (13)	0.0190 (15)	0.0180 (15)	0.0010 (12)	0.0002 (11)	0.0040 (12)
C19	0.0202 (14)	0.0214 (16)	0.0103 (13)	-0.0052 (13)	0.0006 (11)	-0.0004 (12)
C20	0.0220 (13)	0.0147 (15)	0.0160 (13)	0.0004 (11)	0.0026 (11)	-0.0011 (11)
C21	0.0196 (13)	0.0166 (14)	0.0153 (13)	0.0003 (12)	0.0002 (11)	0.0005 (11)
C22	0.0204 (13)	0.0166 (15)	0.0087 (13)	-0.0034 (12)	0.0002 (10)	0.0009 (11)
C23	0.0189 (13)	0.0194 (14)	0.0162 (14)	0.0025 (11)	0.0020 (11)	0.0006 (12)
C24	0.0158 (12)	0.0183 (13)	0.0118 (12)	-0.0001 (13)	-0.0002 (12)	0.0015 (10)
C25	0.0218 (14)	0.0222 (16)	0.0154 (14)	-0.0002 (12)	0.0018 (11)	0.0032 (12)
C26	0.0214 (13)	0.0196 (17)	0.0198 (14)	-0.0043 (13)	0.0008 (11)	0.0002 (13)
C27	0.0157 (13)	0.0248 (16)	0.0151 (14)	0.0033 (13)	-0.0006 (11)	-0.0011 (12)
C28	0.0300 (16)	0.038 (2)	0.0127 (14)	0.0025 (15)	-0.0032 (13)	0.0035 (13)
C29	0.0196 (13)	0.0231 (15)	0.0128 (12)	0.0003 (14)	-0.0006 (12)	0.0032 (11)
C30	0.0205 (14)	0.0184 (14)	0.0157 (13)	-0.0004 (12)	0.0012 (11)	-0.0024 (11)
C31	0.0267 (15)	0.0252 (17)	0.0192 (15)	0.0005 (13)	-0.0039 (12)	0.0044 (12)

Geometric parameters (Å, °)

Pd1—C16	1.935 (3)	C10—H10	0.9500
Pd1—C1	1.947 (3)	C11—C12	1.393 (4)
Pd1—Cl2	2.2979 (7)	C11—H11	0.9500
Pd1—Cl1	2.2994 (7)	C12—C14	1.400 (4)
Cl3—C31	1.762 (3)	C13—H13A	0.9800
Cl4—C31	1.768 (3)	C13—H13B	0.9800
Cl5—C31	1.747 (3)	C13—H13C	0.9800
O1—C7	1.387 (3)	C14—C15	1.376 (4)
O1—C8	1.392 (3)	C14—H14	0.9500
O2—C8	1.195 (3)	C15—H15	0.9500
O3—C12	1.356 (3)	C17—C18	1.386 (4)

O3—C13	1.439 (3)	C17—C22	1.400 (4)
O4—C23	1.379 (3)	C18—C19	1.383 (4)
O4—C22	1.402 (3)	C18—H18	0.9500
O5—C23	1.198 (3)	C19—C20	1.386 (4)
O6—C27	1.364 (3)	C19—H19	0.9500
O6—C28	1.437 (3)	C20—C21	1.383 (3)
N1—C1	1.141 (3)	C20—H20	0.9500
N1—C2	1.402 (3)	C21—C22	1.368 (3)
N2—C16	1.150 (3)	C21—H21	0.9500
N2—C17	1.395 (3)	C23—C24	1.482 (3)
C2—C3	1.386 (4)	C24—C25	1.387 (4)
C2—C7	1.396 (4)	C24—C30	1.400 (3)
C3—C4	1.382 (4)	C25—C26	1.378 (3)
C3—H3	0.9500	C25—H25	0.9500
C4—C5	1.381 (4)	C26—C27	1.398 (4)
C4—H4	0.9500	C26—H26	0.9500
C5—C6	1.386 (4)	C27—C29	1.385 (4)
C5—H5	0.9500	C28—H28A	0.9800
C6—C7	1.378 (3)	C28—H28B	0.9800
C6—H6	0.9500	C28—H28C	0.9800
C8—C9	1.467 (3)	C29—C30	1.388 (3)
C9—C10	1.391 (3)	C29—H29	0.9500
C9—C15	1.399 (4)	C30—H30	0.9500
C10—C11	1.388 (3)	C31—H31	1.0000
C16—Pd1—C1	92.00 (12)	C14—C15—C9	120.6 (3)
C16—Pd1—Cl2	178.37 (8)	C14—C15—H15	119.7
C1—Pd1—Cl2	86.52 (8)	C9—C15—H15	119.7
C16—Pd1—Cl1	89.24 (8)	N2—C16—Pd1	177.4 (3)
C1—Pd1—Cl1	176.91 (8)	C18—C17—N2	121.4 (2)
Cl2—Pd1—Cl1	92.28 (3)	C18—C17—C22	120.5 (3)
C7—O1—C8	119.2 (2)	N2—C17—C22	118.1 (2)
C12—O3—C13	117.9 (2)	C19—C18—C17	119.1 (3)
C23—O4—C22	115.11 (19)	C19—C18—H18	120.5
C27—O6—C28	117.8 (2)	C17—C18—H18	120.5
C1—N1—C2	174.3 (3)	C18—C19—C20	120.0 (3)
C16—N2—C17	172.0 (3)	C18—C19—H19	120.0
N1—C1—Pd1	174.2 (2)	C20—C19—H19	120.0
C3—C2—C7	121.0 (3)	C21—C20—C19	120.8 (3)
C3—C2—N1	120.5 (3)	C21—C20—H20	119.6
C7—C2—N1	118.5 (2)	C19—C20—H20	119.6
C4—C3—C2	119.1 (3)	C22—C21—C20	119.5 (2)
C4—C3—H3	120.5	C22—C21—H21	120.2
C2—C3—H3	120.5	C20—C21—H21	120.2
C5—C4—C3	120.1 (3)	C21—C22—C17	120.0 (2)
C5—C4—H4	120.0	C21—C22—O4	120.1 (2)
C3—C4—H4	120.0	C17—C22—O4	119.9 (2)
C4—C5—C6	120.9 (3)	O5—C23—O4	122.4 (3)

C4—C5—H5	119.5	O5—C23—C24	125.2 (2)
C6—C5—H5	119.5	O4—C23—C24	112.4 (2)
C7—C6—C5	119.5 (3)	C25—C24—C30	119.5 (2)
C7—C6—H6	120.2	C25—C24—C23	117.4 (2)
C5—C6—H6	120.2	C30—C24—C23	123.0 (2)
C6—C7—O1	124.5 (2)	C26—C25—C24	121.1 (3)
C6—C7—C2	119.4 (2)	C26—C25—H25	119.5
O1—C7—C2	115.9 (2)	C24—C25—H25	119.5
O2—C8—O1	122.4 (2)	C25—C26—C27	119.1 (3)
O2—C8—C9	127.3 (3)	C25—C26—H26	120.4
O1—C8—C9	110.2 (2)	C27—C26—H26	120.4
C10—C9—C15	118.9 (2)	O6—C27—C29	124.9 (2)
C10—C9—C8	118.7 (2)	O6—C27—C26	114.6 (2)
C15—C9—C8	122.3 (2)	C29—C27—C26	120.5 (2)
C11—C10—C9	121.0 (2)	O6—C28—H28A	109.5
C11—C10—H10	119.5	O6—C28—H28B	109.5
C9—C10—H10	119.5	H28A—C28—H28B	109.5
C10—C11—C12	119.5 (2)	O6—C28—H28C	109.5
C10—C11—H11	120.3	H28A—C28—H28C	109.5
C12—C11—H11	120.3	H28B—C28—H28C	109.5
O3—C12—C11	125.1 (2)	C27—C29—C30	120.0 (2)
O3—C12—C14	115.0 (2)	C27—C29—H29	120.0
C11—C12—C14	119.8 (2)	C30—C29—H29	120.0
O3—C13—H13A	109.5	C29—C30—C24	119.8 (3)
O3—C13—H13B	109.5	C29—C30—H30	120.1
H13A—C13—H13B	109.5	C24—C30—H30	120.1
O3—C13—H13C	109.5	C15—C31—C13	110.17 (15)
H13A—C13—H13C	109.5	C15—C31—C14	110.14 (15)
H13B—C13—H13C	109.5	C13—C31—C14	110.39 (16)
C15—C14—C12	120.1 (3)	C15—C31—H31	108.7
C15—C14—H14	119.9	C13—C31—H31	108.7
C12—C14—H14	119.9	C14—C31—H31	108.7

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>
C4—H4...O2 ⁱ	0.95	2.53	3.193 (4)	127
C6—H6...O6 ⁱⁱ	0.95	2.53	3.433 (4)	158
C19—H19...O5 ⁱⁱⁱ	0.95	2.37	3.182 (3)	143
C20—H20...C11 ^{iv}	0.95	2.80	3.622 (3)	145
C31—H31...C11 ^v	1.00	2.77	3.607 (3)	141
C31—H31...C12 ^v	1.00	2.67	3.513 (3)	142

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$; (ii) $-x+1, y+1/2, -z+3/2$; (iii) $x+1/2, -y+3/2, -z+2$; (iv) $x-1/2, -y+3/2, -z+2$; (v) $x-1, y+1, z$.