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Di- μ -chloro-bis[*cis,cis*- η^4 -1,5-cyclooctadiene]rhodium(I): a Redetermination

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Abstract

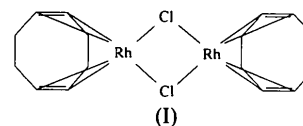
The structure of $[\text{Rh}_2\text{Cl}_2(\text{C}_8\text{H}_{12})_2]$ has been redetermined. The precision of this structure determination compared with other published data shows an improvement such that averaged bond distance and angle e.s.d.'s are smaller by a factor of two. The bond distances in the *cis,cis*-1,5-cyclooctadiene ring are compared to those of analogous compounds in the Cambridge Structural Database. The title compound has no molecular symmetry; the *cis,cis*-1,5-cyclooctadiene rings each have a boat conformation showing C_{2v} symmetry.

Comment

The crystal structure of the title compound (I) was reported for the first time by Ibers & Snyder (1962) who used the equi-inclination Weissenberg technique leading to $R = 0.10$ for 779 reflections. It was impossible for these authors to distinguish, on the basis of length, the single bonds from the double bonds in the *cis,cis*-1,5-cyclooctadiene rings (abbreviated hereafter as cc-cod).

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Since the reported crystal structure was apparently disordered, Boeyens, Denner, Orchard, Rencken & Rose (1986) reinvestigated the RhCl complex based on X-ray diffraction data recorded with a counter-tube detector. These authors observed a regular alternation of short and long bonds around the cc-cod rings, which they suggested could be the result of oxidation of the ligand producing cyclooctatetraene (cot) on the one hand and the occurrence of disorder on the other. The former explanation was refuted by decomposing the complex followed by gas-chromatographic comparison with authentic samples of both cc-cod and cot. The latter was supported by high values of the anisotropic displacement parameters associated with all of the $\text{C}(sp^3)$ atoms compared with the other atoms.

During the course of preparation of a series of new rhodium(I)-iminophosphorane complexes $[\text{RhL}_2\text{Cl}(\text{R}_3\text{P}=\text{NR}')]_2$ with $\text{L}_2 = (\text{CO})_2$ or cod (Imhoff, Elsevier & Stam, 1990), a crystal of a sample supposed to contain $[\text{Rh}(\text{cod})\text{Cl}(\text{Et}_3\text{P}=\text{N}-\text{SiMe}_3)]_2$ was investigated by X-ray diffraction and found to contain molecules of the title compound. Since the single bonds which are next-nearest neighbours of the double bonds in the cc-cod ring are significantly longer in the present crystal structure than those found by Boeyens *et al.* (1986), it is of interest to report these results.

The present crystal data are of better quality: mean e.s.d.'s of C-atom distances and angles are 0.01 Å and 0.6° here, compared with 0.02 Å and 1.1° in the earlier study. The R value is almost 10% lower than obtained by Boeyens *et al.* (1986) who report $R = 0.042$ for 2176 reflections and 200 variable parameters (the number of parameters refined by Boeyens *et al.* ought to be 253 since these authors refined their structure by a procedure similar to ours, the only difference being an isotropic extinction coefficient in our calculations).

The range of bond lengths of the different bond types in the cc-cod rings is smaller than reported by Boeyens *et al.* (1986): 1.388 (9)–1.401 (8) Å for the double bonds (henceforth D22), 1.50 (1)–1.52 (1) Å for the single bonds adjacent to the $\text{C}=\text{C}$ double bonds (henceforth S23) and 1.45 (1)–1.50 (1) Å for the single bonds adjacent to two single bonds (henceforth S33), compared to 1.38–1.40, 1.50–1.56 and 1.37–1.43 Å, respectively, reported by Boeyens *et al.* Since the double bond is significantly longer and the single bond between the two other single bonds is significantly shorter than the commonly reported values of 1.317 and 1.524 Å, respectively (Allen *et al.*, 1987), a search of the Cambridge Structural Database (Version 5.04, 1 October 1992, 102 525 entries) (Allen *et al.*, 1991) was per-

formed on a fragment composed of one Rh atom connected to one cod ring. On the 158 entries thus obtained the *GSTAT* program was run with the following restrictions: no disorder, error-free, $R \leq 0.05$ and diffractometer data only. This resulted in 117 hits showing the following average values: 1.381 for D22, 1.512 for S23 and 1.493 Å for S33 (CSD Refcodes and the short references have been deposited as supplementary material). The present structural results agree well with these mean values. Boeyens *et al.* (1986) attribute the general feature of a shortening of the S33 bonds mainly to disorder, as well as to electronic factors that might contribute to this phenomenon. The lengthening of the double bonds (D22) in the rhodium complexes could relate to a more efficient π back-bonding in d^8 systems, according to Albinati, Meille & Carturan (1979).

When following the convention that the double bonds rather than the individual $C(sp^2)$ atoms define the coordination sites, each Rh atom is in an approximately square-planar configuration. The Cl atoms show the largest deviation from the best plane through the Rh and Cl atoms and the double-bond centres *M1–M4*: 0.260 (2) for Cl(2) and 0.176 (2) Å for Cl(1). Ibers & Snyder (1962) indicated that the double bonds are almost perpendicular to this plane without giving numerical values. The values found in this study are 85.30 (1), 85.80 (1), 82.77 (1) and 85.77 (1)° for C(1)=C(2), C(5)=C(6), C(9)=C(10) and C(13)=C(14), respectively.

The cod rings each have a boat conformation with approximate C_{2v} symmetry. The mirror planes are defined by the lines passing through the midpoints of the D22 bonds and the S33 bonds, respectively; the twofold axis passes through the geometric centre of gravity defined by the eight C atoms of each ring. The deviation

from C_{2v} symmetry has been judged by calculating the differences between the distances of nearly symmetry-equivalent atom pairs from the two mirror planes [e.g. C(1) and C(2) for the D22 mirror plane or C(1) and C(6) for the S33 mirror plane] and from the twofold axis [e.g. C(1)–C(5)]. The maximum differences between distances of nearly symmetry-equivalent atom pairs from the three symmetry operators are 0.0231 (14) Å for the C(1)–C(8) ring and 0.0332 (8) Å for the C(9)–C(16) ring.

The rhodium complex has no molecular symmetry, in contrast to the analogous copper complex described in the same paper by Boeyens *et al.* (1986); the latter complex is centrosymmetric.

Experimental

In the synthesis of the [Rh(cc-cod)Cl]₂ complex, reactions were carried out in an atmosphere of purified nitrogen. Benzene was carefully dried and distilled prior to use. [Rh(cc-cod)Cl]₂ was synthesized using the literature procedure given by Chatt & Venanzi (1957). A mixture of 618.1 mg of [Rh(cc-cod)Cl]₂ (1.25 mmol) and 515.1 mg of Et₃P=N–SiMe₃ (2.51 mmol) was dissolved in 15 ml of benzene. The solution was filtered. The solvent was evaporated slowly under a moderate nitrogen stream for 25 d until 60% of its original volume remained, yielding orange crystals. The remaining benzene was decanted and the crystals were washed twice with 1.5 ml of benzene and dried *in vacuo* giving 392.4 mg of [Rh(cc-cod)Cl]₂. Density was measured pycnometrically (Ibers & Snyder, 1962).

Crystal data

[Rh₂Cl₂(C₈H₁₂)₂]
 $M_r = 493.08$
 Monoclinic
 $P2_1/n$
 $a = 9.056 (1) \text{ \AA}$
 $b = 25.475 (6) \text{ \AA}$
 $c = 7.309 (1) \text{ \AA}$
 $\beta = 91.79 (1)^\circ$
 $V = 1685.4 (5) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.943 \text{ Mg m}^{-3}$
 $D_m = 1.93 (2) \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.71069 \text{ \AA}$
 Cell parameters from 23 reflections
 $\theta = 19.9\text{--}21.6^\circ$
 $\mu = 2.24 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Block
 $0.43 \times 0.28 \times 0.25 \text{ mm}$
 Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
 $T_{\min} = 0.837$, $T_{\max} = 1.118$
 7601 measured reflections
 7348 independent reflections

4822 observed reflections
 $[I > 2.5\sigma(I)]$
 $\theta_{\max} = 34.88^\circ$
 $h = -14 \rightarrow 14$
 $k = 0 \rightarrow 41$
 $l = 0 \rightarrow 11$
 2 standard reflections
 frequency: 83 min
 intensity variation: 2.3%

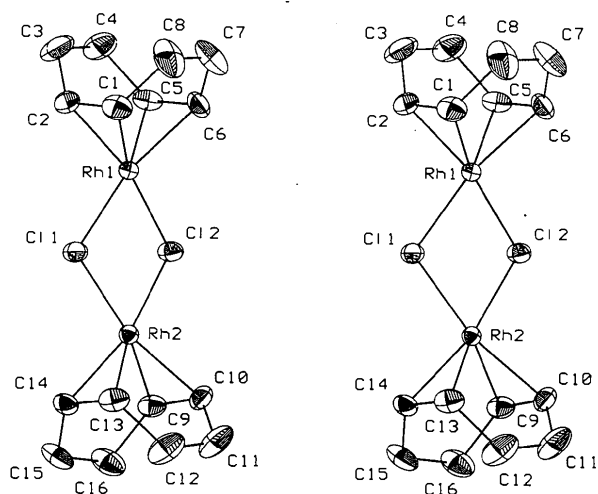


Fig. 1. Stereoscopic *PEANUT* (Hummel, Hauser & Bürgi, 1990) drawing of the title compound showing the atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level; H atoms have been omitted for clarity.

Refinement

Refinement on F $R = 0.038$ $wR = 0.063$ $S = 1.057$

4820 reflections

254 parameters

Only coordinates of H atoms

refined

 $w = 1/[\sigma^2(F) + 0.02F^2]$ $(\Delta/\sigma)_{\max} = 0.508$ $\Delta\rho_{\max} = 1.479 \text{ e } \text{Å}^{-3}$ $\Delta\rho_{\min} = -1.931 \text{ e } \text{Å}^{-3}$

Extinction correction:

Zachariasen (1967)

Extinction coefficient:

 $g = 2(2) \times 10^{-6}$

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol IV, Tables 2.2B

and 2.3.1)

Table 1. Fractional coordinates and equivalent isotropic displacement parameters (Å^2) $M(1)$, $M(2)$, $M(3)$ and $M(4)$ are the midpoints of the double bonds $C(1)=C(2)$, $C(5)=C(6)$, $C(9)=C(10)$ and $C(13)=C(14)$, respectively.

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^*$$

	x	y	z	U_{eq}
Rh(1)	0.35804 (4)	0.58628 (1)	0.36508 (4)	0.0308 (2)
Rh(2)	0.61175 (3)	0.67537 (1)	0.56567 (4)	0.0289 (1)
Cl(1)	0.5763 (2)	0.63197 (6)	0.2757 (2)	0.0482 (6)
Cl(2)	0.3728 (1)	0.63964 (6)	0.6381 (2)	0.0446 (6)
C(1)	0.3931 (7)	0.5247 (3)	0.1796 (9)	0.055 (3)
C(2)	0.3071 (8)	0.5633 (3)	0.0949 (7)	0.052 (3)
C(3)	0.1437 (9)	0.5597 (4)	0.056 (1)	0.084 (5)
C(4)	0.0497 (8)	0.5621 (4)	0.218 (1)	0.076 (5)
C(5)	0.1332 (6)	0.5686 (2)	0.3997 (9)	0.048 (3)
C(6)	0.2191 (7)	0.5293 (2)	0.4846 (8)	0.052 (3)
C(7)	0.242 (1)	0.4757 (3)	0.403 (1)	0.088 (6)
C(8)	0.332 (1)	0.4731 (3)	0.243 (1)	0.098 (7)
C(9)	0.6084 (6)	0.7310 (2)	0.7778 (8)	0.048 (3)
C(10)	0.6763 (7)	0.6851 (3)	0.8417 (7)	0.051 (3)
C(11)	0.8400 (9)	0.6783 (4)	0.873 (1)	0.088 (6)
C(12)	0.9303 (7)	0.6842 (4)	0.706 (1)	0.082 (5)
C(13)	0.8392 (6)	0.6850 (3)	0.5276 (9)	0.053 (3)
C(14)	0.7639 (6)	0.7290 (2)	0.4619 (7)	0.045 (3)
C(15)	0.7586 (8)	0.7813 (3)	0.556 (1)	0.068 (4)
C(16)	0.6912 (9)	0.7808 (3)	0.737 (1)	0.072 (5)
$M(1)$	0.3501	0.5440	0.1373	
$M(2)$	0.1762	0.5490	0.4422	
$M(3)$	0.6424	0.7081	0.8098	
$M(4)$	0.8016	0.7070	0.4948	

Table 2. Selected geometric parameters (Å , $^\circ$)

Rh(1)—Cl(1)	2.402 (1)	Rh(2)—Cl(1)	2.403 (1)
Rh(1)—Cl(2)	2.415 (1)	Rh(2)—Cl(2)	2.421 (1)
Rh(1)—C(1)	2.104 (7)	Rh(2)—C(9)	2.102 (6)
Rh(1)—C(2)	2.097 (6)	Rh(2)—C(10)	2.098 (5)
Rh(1)—C(5)	2.108 (5)	Rh(2)—C(13)	2.101 (5)
Rh(1)—C(6)	2.126 (6)	Rh(2)—C(14)	2.099 (6)
Rh(1)— $M(1)$	1.982	Rh(2)— $M(3)$	1.981
Rh(1)— $M(2)$	1.997	Rh(2)— $M(4)$	1.982
C(1)—C(2)	1.388 (9)	C(9)—C(10)	1.395 (9)
C(2)—C(3)	1.50 (1)	C(10)—C(11)	1.50 (1)
C(3)—C(4)	1.48 (1)	C(11)—C(12)	1.50 (1)
C(4)—C(5)	1.52 (1)	C(12)—C(13)	1.52 (1)
C(5)—C(6)	1.401 (8)	C(13)—C(14)	1.389 (9)
C(6)—C(7)	1.51 (1)	C(14)—C(15)	1.502 (9)
C(7)—C(8)	1.45 (1)	C(15)—C(16)	1.48 (1)
C(1)—C(8)	1.51 (1)	C(9)—C(16)	1.51 (1)
Cl(1)—Rh(1)—Cl(2)	85.83 (4)	Cl(1)—Rh(2)—Cl(2)	85.66 (5)
Cl(1)—Rh(1)—C(1)	92.5 (2)	Cl(1)—Rh(2)—C(9)	163.0 (2)
Cl(1)—Rh(1)—C(2)	92.2 (2)	Cl(1)—Rh(2)—C(10)	158.2 (2)
Cl(1)—Rh(1)—C(5)	160.4 (2)	Cl(1)—Rh(2)—C(13)	92.4 (2)
Cl(1)—Rh(1)—C(6)	160.9 (2)	Cl(1)—Rh(2)—C(14)	93.0 (2)
Cl(2)—Rh(1)—C(1)	161.2 (2)	Cl(2)—Rh(2)—C(9)	93.4 (2)
Cl(2)—Rh(1)—C(2)	160.1 (2)	Cl(2)—Rh(2)—C(10)	93.3 (2)

Cl(2)—Rh(1)—C(5)	92.8 (2)	Cl(2)—Rh(2)—C(13)	163.6 (2)
Cl(2)—Rh(1)—C(6)	93.6 (2)	Cl(2)—Rh(2)—C(14)	157.7 (2)
C(1)—Rh(1)—C(2)	38.6 (3)	C(9)—Rh(2)—C(10)	38.2 (2)
C(1)—Rh(1)—C(5)	94.8 (2)	C(9)—Rh(2)—C(13)	93.2 (2)
C(1)—Rh(1)—C(6)	81.9 (2)	C(9)—Rh(2)—C(14)	81.5 (2)
C(2)—Rh(1)—C(5)	82.4 (3)	C(10)—Rh(2)—C(13)	82.5 (2)
C(2)—Rh(1)—C(6)	94.6 (2)	C(10)—Rh(2)—C(14)	96.0 (2)
C(5)—Rh(1)—C(6)	38.6 (2)	C(13)—Rh(2)—C(14)	38.6 (2)
Rh(1)—Cl(1)—Rh(2)	94.08 (5)	Rh(1)—Cl(2)—Rh(2)	93.32 (4)
$M(1)$ —Rh(1)— $M(2)$	88.3	$M(3)$ —Rh(2)— $M(4)$	88.1
$M(1)$ —Rh(1)—Cl(1)	92.5	$M(3)$ —Rh(2)—Cl(1)	177.5
$M(1)$ —Rh(1)—Cl(2)	178.2	$M(3)$ —Rh(2)—Cl(2)	93.6
$M(2)$ —Rh(1)—Cl(1)	179.2	$M(4)$ —Rh(2)—Cl(1)	92.8
$M(2)$ —Rh(1)—Cl(2)	93.4	$M(4)$ —Rh(2)—Cl(2)	176.7
Rh(1)—C(1)—C(2)	70.5 (4)	Rh(2)—C(9)—C(10)	70.5 (3)
Rh(1)—C(1)—C(8)	112.8 (5)	Rh(2)—C(9)—C(16)	113.8 (4)
C(2)—C(1)—C(8)	123.5 (7)	C(10)—C(9)—C(16)	123.6 (6)
Rh(1)—C(2)—C(1)	71.0 (4)	Rh(2)—C(10)—C(9)	70.8 (3)
Rh(1)—C(2)—C(3)	112.4 (5)	Rh(2)—C(10)—C(11)	112.0 (4)
C(1)—C(2)—C(3)	125.1 (7)	C(9)—C(10)—C(11)	124.8 (7)
C(2)—C(3)—C(4)	116.0 (6)	C(10)—C(11)—C(12)	115.3 (6)
C(3)—C(4)—C(5)	114.8 (6)	C(11)—C(12)—C(13)	113.9 (6)
Rh(1)—C(5)—C(4)	112.1 (4)	Rh(2)—C(13)—C(12)	113.1 (4)
Rh(1)—C(5)—C(6)	71.4 (3)	Rh(2)—C(13)—C(14)	70.6 (3)
C(4)—C(5)—C(6)	124.3 (6)	C(12)—C(13)—C(14)	123.7 (6)
Rh(1)—C(6)—C(5)	70.0 (3)	Rh(2)—C(14)—C(13)	70.8 (3)
Rh(1)—C(6)—C(7)	111.5 (5)	Rh(2)—C(14)—C(15)	112.4 (4)
C(5)—C(6)—C(7)	123.7 (6)	C(13)—C(14)—C(15)	125.5 (5)
C(6)—C(7)—C(8)	116.5 (7)	C(14)—C(15)—C(16)	115.0 (6)
C(1)—C(8)—C(7)	115.5 (7)	C(9)—C(16)—C(15)	114.0 (6)

The structure was determined by Patterson methods and subsequent difference Fourier synthesis with the aid of *Xtal3.0* (Hall & Stewart, 1990). The positions of several H atoms could be found in difference maps, but all were placed in geometrically calculated positions and refined in the structure-factor calculations as riding atoms, each restrained at 1.09 Å from their carrier atom with a fixed $U = 0.10 \text{ Å}^2$. The anomalous dispersion for the non-H atoms was accounted for in the structure-factor calculations. Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CELCON*, comparable to *Xtal LATCON* (Hall & Stewart, 1990). Data reduction: *Xtal ADDREF*. Program(s) used to refine structure: *Xtal CRYLSQ*. Molecular graphics: *PEANUT* (Hummel, Hauser & Bürgi, 1990). Software used to prepare material for publication: *Xtal BONDLA* and *Xtal CIFIO*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and CSD Refcodes have been deposited with the IUCr (Reference: SH1088). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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***N*¹,*N*¹-Dimethyl-3-oxopiperazinium(1+)
Trichloro(dimethyl sulfoxide-*S*)-
platinate(1-), (C₆H₁₃N₂O)[PtCl₃(C₂H₆OS)]**

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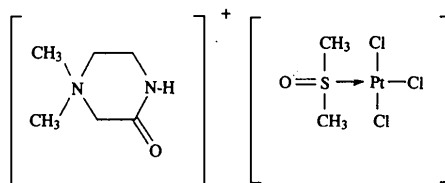
Abstract

The title compound, *N*¹,*N*¹-dimethyl-3-oxopiperazinium trichloro(dimethyl sulfoxide-*S*)platinate(II), (C₆H₁₃N₂O)[PtCl₃(C₂H₆OS)], was obtained from the reaction of PtCl₂(dms_o)₂ and the chloride salt of *N*¹,*N*¹-dimethyl-3-oxopiperazinium (dmpd). The dmpd cation is observed in a conjugated amide form and is hydrogen bonded to the anion via an N—H···O bridge [N···O = 3.14 (2) Å].

Comment

As part of a program of studies directed towards the preparation of new chelating amine ligands for platinum(II), with the aim of making novel platinum antitumour compounds, the reaction between bromoacetic acid and dimethylethylenediamine followed by crystallization in HCl was carried out. This reaction was found to yield *N*¹,*N*¹-dimethyl-3-oxopiperazinium chloride (dmpdCl), and not the

expected *N*¹,*N*¹-dimethylethylenediamine-*N*-acetic acid (Fujii, Kyuno & Tsuchiya, 1970). It was considered of interest to investigate whether this cation would undergo a ring-opening reaction with HCl, to allow a reaction with PtCl₂. Apparently the dmpd ring is quite stable and, in fact, ionic *N*¹,*N*¹-dimethyl-3-oxopiperazinium(1+) trichloro(dimethyl sulfoxide-*S*)platinate(1-) (I) was isolated, as shown by the present X-ray study (Fig. 1).



The structure consists of an ionic lattice comprising the unprecedented cation dmpd and the well known anion [PtCl₃(dms_o)]⁻. Comparison of the anionic species with those of previous determinations (see *e.g.* Cantoni, Tiripicchio, Tiripicchio-Camellini & Annibale, 1991; Khodadad & Rodier, 1987; Walba, Richards, Hermsmeier & Haltiwanger, 1987) shows that in the present structure the Pt—Cl bonds are all long, especially the *trans* bond [2.388 (4) Å]. This may be caused by substitutional disorder with Br, probably originating from the bromoacetic acid (this was, however, not pursued any further). For Pt—Cl bonds in the literature, *trans*-chlorine bonds are also seen to be slightly longer than the others. The N2—C3—O1 part of the dmpd cation has a conjugated amide form. Weak hydrogen bonding is observed between the N—H group and the O atom of dms_o [O2···N2 = 3.14 (2) Å].

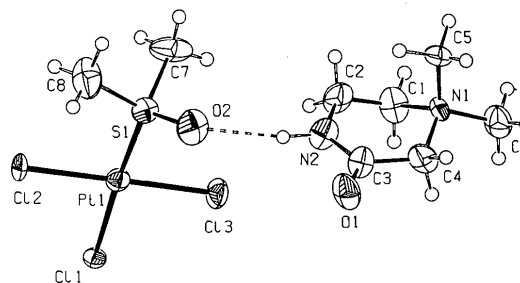


Fig. 1. Thermal motion ellipsoid plot (50% probability level).

Experimental

The title compound was crystallized from the reaction mixture of an aqueous chloride solution of the dmpd cation and PtCl₂(dms_o)₂ in MeOH.

Crystal data

(C₆H₁₃N₂O)[PtCl₃-(C₂H₆OS)]

Cu Kα radiation
λ = 1.54184 Å