

Dichlorido-1 κ^2 Cl- μ -[(1,2,5,6- η :3,4,7,8- η)-1,3,5,7-cyclooctatetraene]dimethyl-2 κ^2 C-diplatinum(II)

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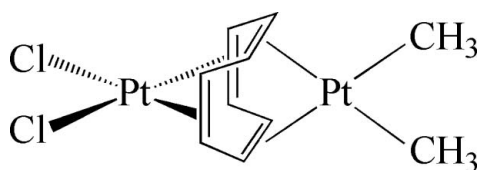
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.020$ Å; R factor = 0.058; wR factor = 0.145; data-to-parameter ratio = 19.5.

The title complex, $[\text{Pt}_2(\text{CH}_3)_2\text{Cl}_2(\text{C}_8\text{H}_8)]$, consists of PtCl_2 and $\text{Pt}(\text{CH}_3)_2$ groups bridged by a 1,3,5,7-cyclooctatetraene ligand, and is disposed about a mirror plane passing through the two Pt atoms, the methyl groups and the centre of the ligand. The coordination geometry around each Pt^{II} centre is essentially square planar.

Related literature

For related literature, see: Doyle & Baenziger (1995); Doyle *et al.* (1961); Elschenbroich & Salzer (1992); Fritz & Keller (1962); Fritz & Sellmann (1967); Jensen (1953); Kistner *et al.* (1963); Kunkely & Vogler (2006); Song *et al.* (2006); Tresoldi *et al.* (1982).



Experimental

Crystal data

$[\text{Pt}_2(\text{CH}_3)_2\text{Cl}_2(\text{C}_8\text{H}_8)]$	$V = 1260.7(2)$ Å ³
$M_r = 595.29$	$Z = 4$
Orthorhombic, $Pnma$	Mo $K\alpha$ radiation
$a = 7.8478(8)$ Å	$\mu = 22.55$ mm ⁻¹
$b = 10.3924(10)$ Å	$T = 293(2)$ K
$c = 15.4582(16)$ Å	$0.25 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART 1000 CCD diffractometer	7010 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2000)	1363 independent reflections
$T_{\min} = 0.574$, $T_{\max} = 1.000$	1226 reflections with $I > 2\sigma(I)$
(expected range = 0.060–0.105)	$R_{\text{int}} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$	70 parameters
$wR(F^2) = 0.145$	H-atom parameters constrained
$S = 1.20$	$\Delta\rho_{\text{max}} = 1.77$ e Å ⁻³
1363 reflections	$\Delta\rho_{\text{min}} = -1.95$ e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

Pt1—C1	2.256 (16)	Pt2—C5	2.195 (17)
Pt1—C2	2.267 (16)	Pt2—C3	2.249 (15)
Pt1—Cl1	2.460 (4)	Pt2—C4	2.257 (15)
Pt2—C6	2.137 (19)		
Cl1—Pt1—Cl1 ⁱ	83.3 (2)	C6—Pt2—C5	85.1 (7)

 Symmetry code: (i) $x, -y + \frac{3}{2}, z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SK3133).

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supplementary materials

Acta Cryst. (2007). E63, m1878 [doi:10.1107/S1600536807028036]

Dichlorido- $1\kappa^2\text{Cl}$ - μ -[(1,2,5,6-*M*:3,4,7,8-*M*)-1,3,5,7-cyclooctatetraene]dimethyl- $2\kappa^2\text{C}$ -diplatinum(II)

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Comment

1,3,5,7-Cyclooctatetraene (cot) is a widely utilized and versatile ligand in organometallic chemistry (Elschenbroich & Salzer, 1992). The cot ligand can coordinate metal atoms as a tub-shaped tetraene or as a planar aromatic anion $\text{C}_8\text{H}_8^{2-}$, and its coordination modes are quite variable, *viz.* η^2 , η^3 , η^4 , η^5 , η^6 and η^8 . Numerous cot-metal complexes are known, however, relatively few mono- and dinuclear Pt compounds with cot are synthesized and studied (Jensen, 1953; Doyle *et al.*, 1961; Fritz & Keller, 1962; Kistner *et al.*, 1963; Fritz & Sellmann, 1967; Tresoldi *et al.*, 1982; Kunkely & Vogler, 2006). The decomposition of the mononuclear complexes, [(cot)PtR₂] (R = CH₃ or C₆H₅), lead to the formation of the dinuclear complexes, [R₂Pt(cot)PtR₂], in which cot acts as a bridging ligand between two Pt atoms. The NMR spectra of the dinuclear compounds were examined for potential long-range coupling by the ¹⁹⁵Pt nuclei (Kistner *et al.*, 1963), and only the crystal structure of the dinuclear complex with CH₃ was reported (Doyle & Baenziger, 1995; Song *et al.*, 2006). The X-ray structure analysis reveals that the complex contains a twofold axis passing through the Pt atoms and the center of the cot ligand. The dinuclear title complex, (I), was formed by the reaction of [(cot)Pt(CH₃)₂] with K₂PtCl₄ and its structure is reported here.

The complex consists of PtCl₂ and Pt(CH₃)₂ groups bridged by an 1,3,5,7-cyclooctatetraene ligand (Fig. 1), and is disposed about a mirror plane passing through the two Pt atoms and the center of the ligand (Fig. 2). Each Pt atom is essentially in a square-planar environment defined by the two midpoints of the π -coordinated double bonds of the cot ligand and the two Cl atoms or the two C atoms of methyl groups. The midpoints, the Pt and Cl or C atoms form two coordination planes with the largest deviations 0.002 Å (Pt1) from the least-squares planes. The bond angles lie in the range of 83.3°–95.5° (<C11—Pt1—C11ⁱ = 83.3 (2)°, <M1—Pt1—M1ⁱ = 85.7°, <C11—Pt1—M1 = 95.5°, <C5—Pt2—C6 = 85.1 (7)°, <M2—Pt2—M2ⁱ = 85.3°, <C6—Pt2—M1 = 94.2°, <C5—Pt2—M2 = 95.4°; symmetry code *i*: *x*, 1.5 - *y*, *z*; M1 and M2 denote the centroids of the olefinic bonds C1—C2 and C3—C3ⁱ, respectively). The coordination planes are perfectly perpendicular to each other (90.0°). The Pt—C(cot) bond lengths range from 2.249 (15) Å to 2.267 (16) Å, and are slightly longer than the Pt—C(methyl) bond (2.195 (17) Å and 2.137 (19) Å). The distances between the Pt atom and the midpoints are 2.156 Å (M1), 2.150 Å (M2) and 2.147 Å (M2ⁱ). The Pt1—C1/C2 bonds *trans* to Cl atom are, on an average, almost equal to the Pt2—C3/C4 bonds *trans* to methyl group (the mean lengths: Pt1—C1/C2 = 2.262 Å and Pt2—C3/C4 = 2.253 Å). The cot ligand coordinates the two Pt atoms very symmetrically in the tub conformation. The distance between the Pt atoms is 4.1407 (4) Å. The four C atoms (C1, C2, C1ⁱ and C2ⁱ) coordinated to Pt1 and the four C atoms (C3, C4, C3ⁱ and C4ⁱ) coordinated to Pt2 lie on a plane, respectively, with the torsion angles <C1—C2—C2ⁱ—C1ⁱ = 0° and <C3—C3ⁱ—C4ⁱ—C4 = 0°. The Pt atoms are displaced by 1.581 (12) Å (Pt1) from the plane C1/C2/C2ⁱ/C1ⁱ and 1.580 (10) Å (Pt2) from the plane C3/C3ⁱ/C4ⁱ/C4, respectively. The planes are nearly parallel to each other with dihedral angle 0.3(1.9)°. The cot ring angles lie in the range of 121.1 (15)°–122.6 (9)° in the complex.

Experimental

To a solution of cyclooctatetraenedimethylplatinum(II) (0.0724 g, 0.22 mmol) in CH_2Cl_2 (15 ml) and EtOH (15 ml) was added a solution of K_2PtCl_4 (0.1018 g, 0.25 mmol) in H_2O (10 ml), and stirred for 24 h at room temperature. The formed precipitate was collected by filtration, washed with CH_2Cl_2 and H_2O , and dried, giving a orange powder (0.0277 g). Needle-shaped crystals suitable for an X-ray structure analysis were obtained *via* slow evaporation from a CHCl_3 solution.

Refinement

All H atoms were positioned geometrically and allowed to ride on their respective carrier atoms [$\text{C}-\text{H} = 0.93$ or 0.96 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$].

Figures

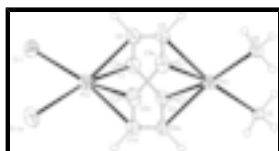


Fig. 1. The structure of (I), showing the atom-numbering scheme (symmetry code a: $x, 1.5 - y, z$). Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary size.



Fig. 2. A packing diagram for (I).

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Crystal data

$[\text{Pt}_2(\text{CH}_3)_2\text{Cl}_2(\text{C}_8\text{H}_8)]$

$M_r = 595.29$

Orthorhombic, $Pnma$

Hall symbol: $-P\ 2ac\ 2n$

$a = 7.8478$ (8) \AA

$b = 10.3924$ (10) \AA

$c = 15.4582$ (16) \AA

$V = 1260.7$ (2) \AA^3

$Z = 4$

$F_{000} = 1056$

$D_x = 3.136 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1985 reflections

$\theta = 2.4\text{--}25.0^\circ$

$\mu = 22.55 \text{ mm}^{-1}$

$T = 293$ (2) K

Needle, orange

$0.25 \times 0.12 \times 0.10 \text{ mm}$

Data collection

Bruker SMART 1000 CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

1363 independent reflections

1226 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$T = 293(2)$ K $\theta_{\max} = 26.4^\circ$
 φ and ω scans $\theta_{\min} = 2.4^\circ$
 Absorption correction: Multi-scan
 (SADABS; Bruker, 2000) $h = -7 \rightarrow 9$
 $T_{\min} = 0.574$, $T_{\max} = 1.000$ $k = -12 \rightarrow 12$
 7010 measured reflections $l = -19 \rightarrow 17$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.058$ H-atom parameters constrained
 $wR(F^2) = 0.145$ $w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 15.8923P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.20$ $(\Delta/\sigma)_{\max} < 0.001$
 1363 reflections $\Delta\rho_{\max} = 1.77 \text{ e } \text{\AA}^{-3}$
 70 parameters $\Delta\rho_{\min} = -1.94 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.82988 (12)	0.7500	-0.11111 (6)	0.0463 (3)
Pt2	0.43629 (11)	0.7500	0.06729 (6)	0.0388 (3)
Cl1	1.0022 (6)	0.5927 (5)	-0.1916 (3)	0.0579 (11)
C1	0.624 (2)	0.6080 (14)	-0.0752 (12)	0.045 (4)
H1	0.6470	0.5587	-0.1240	0.054*
C2	0.738 (2)	0.6098 (16)	-0.0088 (11)	0.051 (4)
H2	0.8374	0.5611	-0.0135	0.061*
C3	0.7102 (19)	0.6866 (15)	0.0704 (10)	0.044 (4)
H3	0.6917	0.6437	0.1224	0.053*
C4	0.4650 (18)	0.6831 (16)	-0.0708 (9)	0.041 (4)
H4	0.3615	0.6397	-0.0680	0.049*
C5	0.157 (2)	0.7500	0.0657 (12)	0.026 (4)

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H5A	0.1171	0.7500	0.0069	0.039*
H5B	0.1152	0.8254	0.0947	0.039*
C6	0.410 (2)	0.7500	0.2049 (12)	0.036 (5)
H6A	0.5209	0.7500	0.2312	0.054*
H6B	0.3487	0.6746	0.2227	0.054*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.0460 (6)	0.0553 (6)	0.0377 (6)	0.000	0.0031 (4)	0.000
Pt2	0.0409 (5)	0.0423 (5)	0.0331 (5)	0.000	0.0005 (4)	0.000
Cl1	0.060 (3)	0.070 (3)	0.043 (3)	0.016 (2)	0.0070 (19)	-0.001 (2)
C1	0.060 (10)	0.025 (7)	0.049 (11)	0.000 (7)	0.016 (8)	-0.004 (7)
C2	0.060 (10)	0.047 (9)	0.045 (10)	0.005 (8)	0.011 (8)	0.008 (8)
C3	0.048 (9)	0.045 (8)	0.038 (9)	0.014 (7)	0.006 (7)	0.013 (7)
C4	0.034 (7)	0.063 (9)	0.027 (8)	-0.007 (7)	0.003 (6)	-0.003 (7)
C5	0.022 (9)	0.035 (10)	0.020 (10)	0.000	-0.006 (7)	0.000
C6	0.029 (10)	0.063 (13)	0.015 (10)	0.000	-0.009 (8)	0.000

Geometric parameters (\AA , $^\circ$)

Pt1—C1	2.256 (16)	C1—C4	1.47 (2)
Pt1—C1 ⁱ	2.256 (16)	C1—H1	0.9300
Pt1—C2 ⁱ	2.267 (16)	C2—C3	1.48 (2)
Pt1—C2	2.267 (16)	C2—H2	0.9300
Pt1—Cl1	2.460 (4)	C3—C3 ⁱ	1.32 (3)
Pt1—Cl1 ⁱ	2.460 (4)	C3—H3	0.9300
Pt2—C6	2.137 (19)	C4—C4 ⁱ	1.39 (3)
Pt2—C5	2.195 (17)	C4—H4	0.9300
Pt2—C3 ⁱ	2.249 (15)	C5—H5A	0.9600
Pt2—C3	2.249 (15)	C5—H5B	0.9600
Pt2—C4	2.257 (15)	C6—H6A	0.9600
Pt2—C4 ⁱ	2.257 (15)	C6—H6B	0.9600
C1—C2	1.36 (3)		
C1—Pt1—C1 ⁱ	81.7 (8)	C2—C1—C4	121.1 (15)
C1—Pt1—C2 ⁱ	91.3 (6)	C2—C1—Pt1	72.9 (10)
C1 ⁱ —Pt1—C2 ⁱ	35.1 (6)	C4—C1—Pt1	105.8 (10)
C1—Pt1—C2	35.1 (6)	C2—C1—H1	119.5
C1 ⁱ —Pt1—C2	91.3 (6)	C4—C1—H1	119.5
C2 ⁱ —Pt1—C2	80.0 (9)	Pt1—C1—H1	91.3
C1—Pt1—Cl1	94.8 (4)	C1—C2—C3	122.2 (16)
C1 ⁱ —Pt1—Cl1	162.3 (5)	C1—C2—Pt1	72.1 (10)
C2 ⁱ —Pt1—Cl1	162.6 (5)	C3—C2—Pt1	106.2 (10)
C2—Pt1—Cl1	95.7 (5)	C1—C2—H2	118.9
C1—Pt1—Cl1 ⁱ	162.3 (5)	C3—C2—H2	118.9

C1 ⁱ —Pt1—C11 ⁱ	94.8 (4)	Pt1—C2—H2	91.7
C2 ⁱ —Pt1—C11 ⁱ	95.7 (5)	C3 ⁱ —C3—C2	122.6 (9)
C2—Pt1—C11 ⁱ	162.6 (5)	C3 ⁱ —C3—Pt2	73.0 (4)
C11—Pt1—C11 ⁱ	83.3 (2)	C2—C3—Pt2	106.4 (11)
C6—Pt2—C5	85.1 (7)	C3 ⁱ —C3—H3	118.7
C6—Pt2—C3 ⁱ	94.0 (6)	C2—C3—H3	118.7
C5—Pt2—C3 ⁱ	163.0 (4)	Pt2—C3—H3	90.6
C6—Pt2—C3	94.0 (6)	C4 ⁱ —C4—C1	121.9 (9)
C5—Pt2—C3	163.0 (4)	C4 ⁱ —C4—Pt2	72.1 (4)
C3 ⁱ —Pt2—C3	34.1 (8)	C1—C4—Pt2	106.9 (10)
C6—Pt2—C4	162.0 (4)	C4 ⁱ —C4—H4	119.0
C5—Pt2—C4	95.1 (6)	C1—C4—H4	119.0
C3 ⁱ —Pt2—C4	90.9 (5)	Pt2—C4—H4	91.0
C3—Pt2—C4	80.5 (6)	Pt2—C5—H5A	109.5
C6—Pt2—C4 ⁱ	162.0 (4)	Pt2—C5—H5B	109.5
C5—Pt2—C4 ⁱ	95.1 (6)	H5A—C5—H5B	109.5
C3 ⁱ —Pt2—C4 ⁱ	80.5 (6)	Pt2—C6—H6A	109.5
C3—Pt2—C4 ⁱ	90.9 (5)	Pt2—C6—H6B	109.5
C4—Pt2—C4 ⁱ	35.9 (9)	H6A—C6—H6B	109.5
C1 ⁱ —Pt1—C1—C2	-104.3 (10)	C4—Pt2—C3—C3 ⁱ	106.0 (4)
C2 ⁱ —Pt1—C1—C2	-70.5 (12)	C4 ⁱ —Pt2—C3—C3 ⁱ	71.5 (4)
C11—Pt1—C1—C2	93.2 (10)	C6—Pt2—C3—C2	148.9 (10)
C11 ⁱ —Pt1—C1—C2	176.1 (11)	C5—Pt2—C3—C2	62 (3)
C1 ⁱ —Pt1—C1—C4	14.0 (14)	C3 ⁱ —Pt2—C3—C2	-119.9 (9)
C2 ⁱ —Pt1—C1—C4	47.8 (12)	C4—Pt2—C3—C2	-13.9 (10)
C2—Pt1—C1—C4	118.3 (16)	C4 ⁱ —Pt2—C3—C2	-48.4 (11)
C11—Pt1—C1—C4	-148.5 (10)	C2—C1—C4—C4 ⁱ	66.8 (17)
C11 ⁱ —Pt1—C1—C4	-65.6 (18)	Pt1—C1—C4—C4 ⁱ	-12.4 (12)
C4—C1—C2—C3	0(2)	C2—C1—C4—Pt2	-12.3 (18)
Pt1—C1—C2—C3	98.2 (15)	Pt1—C1—C4—Pt2	-91.5 (9)
C4—C1—C2—Pt1	-98.4 (14)	C6—Pt2—C4—C4 ⁱ	-178 (2)
C1 ⁱ —Pt1—C2—C1	73.5 (11)	C5—Pt2—C4—C4 ⁱ	91.7 (2)
C2 ⁱ —Pt1—C2—C1	106.9 (9)	C3 ⁱ —Pt2—C4—C4 ⁱ	-72.4 (4)
C11—Pt1—C2—C1	-90.2 (9)	C3—Pt2—C4—C4 ⁱ	-105.0 (4)
C11 ⁱ —Pt1—C2—C1	-176.0 (11)	C6—Pt2—C4—C1	-60 (2)
C1—Pt1—C2—C3	-119.3 (16)	C5—Pt2—C4—C1	-149.5 (10)
C1 ⁱ —Pt1—C2—C3	-45.8 (12)	C3 ⁱ —Pt2—C4—C1	46.5 (11)
C2 ⁱ —Pt1—C2—C3	-12.4 (15)	C3—Pt2—C4—C1	13.9 (10)
C11—Pt1—C2—C3	150.5 (11)	C4 ⁱ —Pt2—C4—C1	118.9 (10)
C11 ⁱ —Pt1—C2—C3	65 (2)	C1—C2—C1 ⁱ —C2 ⁱ	180.000 (3)
C1—C2—C3—C3 ⁱ	-67.4 (18)	C3—C4 ⁱ —C3 ⁱ —C4	0.000 (5)

supplementary materials

Pt1—C2—C3—C3 ⁱ	11.3 (13)	C4—C1—C2—C3	0(2)
C1—C2—C3—Pt2	12.6 (19)	C4 ⁱ —C1 ⁱ —C2 ⁱ —C3 ⁱ	0(2)
Pt1—C2—C3—Pt2	91.3 (10)	C1—C4—C3 ⁱ —C2 ⁱ	93.6 (12)
C6—Pt2—C3—C3 ⁱ	-91.2 (2)	C1 ⁱ —C4 ⁱ —C3—C2	-93.6 (12)
C5—Pt2—C3—C3 ⁱ	-178 (2)		

Symmetry codes: (i) $x, -y+3/2, z$.

Fig. 1

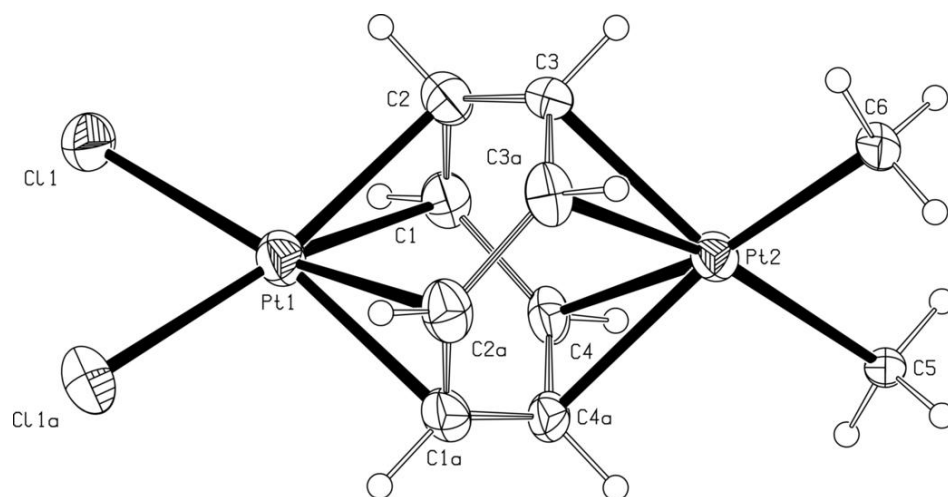


Fig. 2

