Table 4. *Selected geometric parameters (Å, °) for* (II)

Os—F1	1.941 (3)	N2—C21	1.351 (6)
Os—F2	1.976 (3)	N2—C25	1.340 (6)
Os—F3	1.946 (3)	C11—C12	1.369 (9)
Os—Cl1	2.2782 (13)	C12—C13	1.367 (10)
Os—Cl2	2.3039 (15)	C13—C14	1.387 (10)
Os—Cl3	2.3108 (15)	C14—C15	1.365 (8)
N1—C11	1.358 (7)	C21—C22	1.369 (7)
N1—C15	1.341 (6)	C22—C23	1.380 (8)
N1—*CM*	1.469 (6)	C23—C24	1.369 (8)
N2—*CM*	1.492 (6)	C24—C25	1.367 (7)
F1—Os—F3	178.35 (12)	Cl1—Os—Cl2	91.13 (5)
F1—Os—F2	88.07 (13)	F1—Os—Cl3	90.66 (10)
F3—Os—F2	90.28 (13)	F3—Os—Cl3	89.24 (11)
F1—Os—Cl1	92.64 (9)	F2—Os—Cl3	89.06 (10)
F3—Os—Cl1	89.01 (9)	Cl1—Os—Cl3	91.61 (5)
F2—Os—Cl1	179.02 (9)	Cl2—Os—Cl3	177.09 (5)
F1—Os—Cl2	90.17 (10)	C15—N1—C11	121.5 (5)
F3—Os—Cl2	89.85 (11)	N1—*CM*—N2	109.2 (4)
F2—Os—Cl2	88.18 (10)		

For both compounds, data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structures: *SIR*92 (Altomare *et al.*, 1992); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*93.

Financial support from Fonds der Chemischen Industrie and Deutsche Forschungsgemeinschaft is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1030). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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μ-(1,2,5,6-η:3,4,7,8-η)-1,3,5,7-Cyclooctatetraene-bis[dimethylplatinum(II)]

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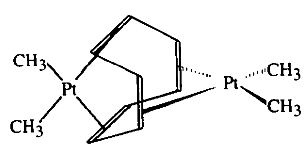
Abstract

μ-(1,2,5,6-η:3,4,7,8-η)-1,3,5,7-Cyclooctatetraene-bis[dimethylplatinum(II)], $[Pt_2(C_8H_8)(CH_3)_4]$, consists of two $Pt(CH_3)_2$ groups bridged by a single cyclooctatetraene molecule. The coordination about each Pt atom is approximately square planar, if each olefinic group is considered as a single ligand. The molecule contains a twofold axis passing through the Pt atoms and the center of the cyclooctatetraene molecule.

Comment

As part of a study concerned with the synthesis and properties of metal–olefin compounds, the NMR spectrum of the title compound, (I), was examined for potential long-range coupling by the Pt^{195} nuclei. Here we report its crystal structure.



(I)

The molecular structure and atom-numbering scheme are shown in Fig. 1. The molecule consists of two dimethylplatinum groups bridged by a cyclooctatetraene molecule. The molecule displays a twofold axis passing through the Pt atoms and the center of the cyclooctatetraene molecule. The coordination about each Pt atom is approximately square planar with the third and fourth bonds of the platinum directed towards the midpoints

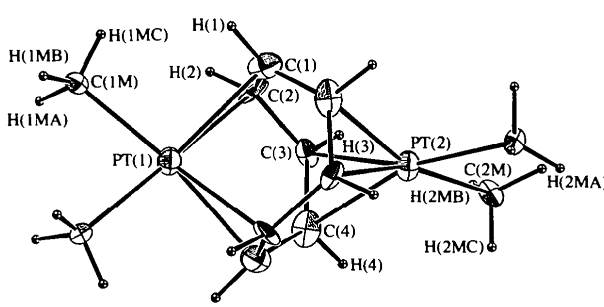


Fig. 1. *ORTEP* (Johnson, 1965) view of the title compound with displacement ellipsoids plotted at the 25% probability level.

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of the olefinic bonds (denoted *M*1 and *M*2) of the cyclooctatetraene. The square-planar arrays of ligands about the Pt atoms are perpendicular to each other. The cyclooctatetraene molecule is slightly distorted from the free olefin configuration and displays a small increase in the double-bond lengths compared with the uncoordinated olefin.

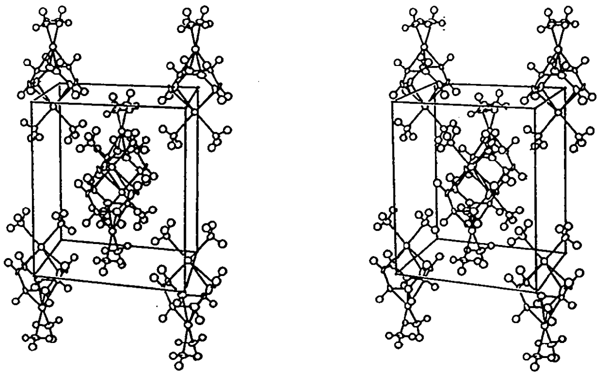

Fig. 2. Stereoview (*ORTEP*; Johnson, 1965) of the unit cell of the title compound looking towards the centered face of the unit cell.

Experimental

The compound was prepared according to the procedure described previously by Doyle, Hutchinson, Baenziger & Tresselt (1961) and recrystallized from a dichloromethane–hexane mixture yielding pale yellow needle-shaped crystals.

Crystal data

$[Pt_2(C_8H_8)(CH_3)_4]$
$M_r = 554.49$
Monoclinic
$C2/c$
$a = 10.9538\,(6)$ Å
$b = 11.1908\,(8)$ Å
$c = 10.770\,(5)$ Å
$\beta = 111.21\,(16)°$
$V = 1231\,(3)$ Å^3
$Z = 4$
$D_x = 2.992\,(7)$ Mg m^{-3}
$D_m = 3.06\,(5)$ Mg m^{-3}
D_m measured by pycnometer

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 (back-reflection Weissenberg for a,b) and 10 (precession, calibrated by a,b to determine c) reflections
$\theta = 60$–$82$6o (Cu $K\alpha$)
$\mu = 23.6$ mm^{-1}
$T = 295$ K
Needle
$0.190\,(001) \times 0.088\,(1\bar{1}0) \times 0.056\,(110)$ mm
Pale yellow

Data collection

Picker diffractometer
Step scan data from θ–2θ scans (Baenziger *et al.*, 1977)
Absorption correction: analytical (Templeton & Templeton, 1973)
$T_{min} = 0.247$, $T_{max} = 0.665$
5047 measured reflections
808 independent reflections

667 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.057$
$\theta_{max} = 23°$
$h = -11 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -11 \rightarrow 10$
3 standard reflections monitored every 60 reflections
intensity decay: 12%

Refinement

Refinement on F
$R = 0.047$
$wR = 0.073$
$S = 1.24$
808 reflections
66 parameters
H-atom positions were fixed and not refined
Weighting scheme according to Killean & Lawrence (1969)

$(\Delta/\sigma)_{max} = 0.23$
$\Delta\rho_{max} = 2.2$ e Å^{-3}
$\Delta\rho_{min} = -1.4$ e Å^{-3}
Extinction correction: Zachariasen (1963)
Extinction coefficient: $6.6\,(2) \times 10^{-7}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

*M*1 and *M*2 are the midpoints of C(1)=C(2) and C(3)=C(4), respectively. $B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Pt(1)	0	−0.1155 (1)	1/4	3.04 (3)
Pt(2)	0	0.25248 (9)	1/4	3.20 (3)
C(1)	0.132 (2)	0.025 (2)	0.211 (2)	4.0 (5)
C(1M)	0.135 (2)	−0.255 (2)	0.291 (2)	3.3 (5)
C(2)	0.153 (2)	0.026 (2)	0.346 (2)	3.2 (4)
C(2M)	0.021 (2)	0.398 (2)	0.388 (1)	2.7 (4)
C(3)	0.086 (2)	0.117 (2)	0.404 (2)	2.7 (4)
C(4)	−0.048 (2)	0.113 (2)	0.370 (2)	3.6 (5)
*M*1	0.1425	0.0255	0.2785	
*M*2	0.019	0.115	0.387	

Table 2. *Selected geometric parameters* (Å, °)

Pt(1)···Pt(2)	4.118 (2)	C(1)—C(2)	1.39 (3)
Pt(1)—C(1)	2.28 (3)	C(1)—C(4)	1.42 (4)
Pt(1)—C(2)	2.26 (2)	C(2)—C(3)	1.51 (3)
Pt(1)—C(1M)	2.08 (2)	C(3)—C(4)	1.38 (4)
Pt(2)—C(3)	2.19 (2)	Pt(1)—*M*1	2.16
Pt(2)—C(4)	2.21 (2)	Pt(2)—*M*2	2.09
Pt(2)—C(2M)	2.16 (2)		
C(1)—Pt(1)—C(1^i)	93 (1)	*M*2—Pt(2)—*M*2^i	85.2
C(1)—Pt(1)—C(2)	35.8 (9)	C(2M)—Pt(2)—C(3)	95.3 (8)
C(1)—Pt(1)—C(2^i)	81.2 (8)	C(2M)—Pt(2)—C(3^i)	162 (1)
C(1)—Pt(1)—C(1M)	96.1 (9)	C(2M)—Pt(2)—C(4^i)	161 (1)
C(1)—Pt(1)—C(1M^i)	158 (1)	C(2M)—Pt(2)—C(4)	96.6 (8)
C(1M)—Pt(1)—C(1M^i)	83 (1)	C(3)—Pt(2)—C(3^i)	92 (1)
C(1M)—Pt(1)—*M*1	95.4	C(3)—Pt(2)—C(4^i)	80.0 (8)
*M*1—Pt(1)—*M*1^i	86.2	C(3)—Pt(2)—C(4)	36.4 (9)
C(1M)—Pt(1)—C(2)	94.3 (8)	C(4)—Pt(2)—C(4^i)	90 (1)
C(1M)—Pt(1)—C(2^i)	166 (1)	C(2)—C(1)—C(4^i)	118 (2)
C(2)—Pt(1)—C(2^i)	92 (1)	C(1)—C(2)—C(3)	121 (2)
C(2M)—Pt(2)—C(2M^i)	82 (1)	C(2)—C(3)—C(4)	120 (2)
C(2M)—Pt(2)—*M*2	96.2	C(1)—C(4)—C(3)	125 (2)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

The structure was solved by Patterson methods and difference electron density maps. All H atoms were located from electron density difference maps.

Data collection: locally written programs (Baenziger *et al.*, 1977). Program(s) used to solve structure: local programs. Program(s) used to refine structure: *MolEN* (Fair, 1990) and *SDP* (B. A. Frenz & Associates, Inc., 1985). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *MolEN* and *SDP*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hexacarbonyl-1κ^4C,2κ^2C-[μ-1κC^6:2(η^5)-6-methylfulvene]diruthenium(*Ru—Ru*), $[Ru_2(C_5H_4CHCH_3)(CO)_6]$

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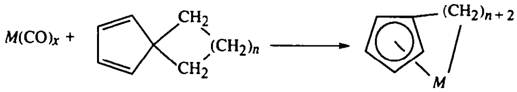
Abstract

The reaction of spiro[2.4]hepta-4,6-diene with $[Ru_3(CO)_{12}]$ leads to the title compound, a diruthenium–fulvene complex which contains a μ-1κC:2(η^5)- (or, simplistically, μ-η^1:η^5-) $C_5H_4CHCH_3$ bridging ligand. The Ru—Ru distance is 2.7954 (8) Å, while Ru–ligand distances are: Ru—C(η^1) 2.232 (8) Å, average Ru—C(Cp) (Cp = cyclopentadienyl) 2.237 Å, average Ru—C(CO) 1.945/1.879 Å with/without *trans* influence. Excepting the methyl group, the molecule has approximate *m* point symmetry.

Comment

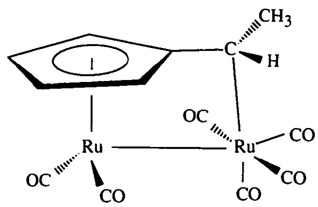
Reaction (1) below has been shown to be quite general for spiro[2.4]heptadiene (*A*) and spiro[4.4]nonadiene (*B*) with a variety of metal carbonyls (Eilbracht & Dahler, 1977; Braun, Dahler & Eilbracht, 1978). In an attempt to establish a new synthetic route for the insertion of Cp rings into Ru—Ru bonds, a reaction analogous to (1) between *A* and $[Ru_3(CO)_{12}]$ was studied. We found that the insertion does take place, but with C—C bond cleavage resulting in the formation of a diruthenium fulvene complex and elimination of a $Ru(CO)_4$ unit.



(*A*) $n = 0$
(*B*) $n = 2$

(1)

The structure analysis of the reaction product showed that the crystal contains discrete molecules of $[Ru_2(C_5H_4CHCH_3)(CO)_6]$, (I), which contain a μ-1κC:2(η^5)- (or, simplistically, μ-η^1:η^5-) C_5H_4CH moiety (Fig. 1). Excepting the methyl group, the molecule has approximate *m* point symmetry; the plane of symmetry defined by the Ru(1)—Ru(2)—C(1)—C(6) metallacycle makes a dihedral angle of 89.8° with the plane of the Cp ring.



The Ru(2)—C(1) distance is slightly, but not significantly, longer than the other four Ru—C(Cp) bond lengths. This elongation may be due to the strain imposed on the η^5 interaction by the Ru(2)—Ru(1)—C(6) bridge. A similar structural feature, with larger differences, has been observed in other Ru complexes containing a μ-η^1:η^5-C_5H_4 moiety, such as $[Ru_2$(6,6-diphenylfulvene)$(CO)_5(SbPh_3)]$, (II) (Töfke, Haupt & Beherens, 1986), the only other Ru μ-η^1:η^5-fulvene complex structurally characterized so far, and in isolobal trinuclear analogues, such as $[Ru_3(C_5H_4)(CO)_9(PPh_3)]$, (III) (Heineke & Vahrenkamp, 1993), and $[Ru_3(C_5H_4)(CO)_{10}]$, (IV) (Arce, De Sanctis, Manzur & Capparelli, 1994). The Ru(1)—C(6) distance is intermediate between the equivalent bond length in (II) [2.29 (1) Å] and the Ru—C bond lengths observed for ligands with secondary alkyl substituents (*ca* 2.14–2.18 Å; Orpen *et al.*, 1989).

In the Cp ring the C(3)—C(4) distance is the shortest of the five C—C bonds. Although in the present study this shortening is not significant, it probably corresponds to a structural feature of the ring, since in (III), (IV) and binuclear complexes with a μ-η^1:η^5-C_5H_4 unit (Hoxmeier, Knobler & Kaesz, 1979, and references therein; Herrmann, Kriechbaum, Bauer, Guggolz & Ziegler, 1981) it was also observed that the C—C bond opposite to the σ-bonded C atom was the shortest of the ring. The C(1)—C(6) distance is consistent with partial double-bond character, when compared with the value

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