Acta Cryst. (1999). C**55**, 29–30

The $[Ru_3(CO)_{11}]^{2-}$ dianion

Jianping Liu, Edwin P. Boyd and Sheldon G. Shore

Department of Chemistry, The Ohio State University, 120 W 18th Avenue, Columbus, OH 43210, USA. E-mail: shore.1@osu.edu

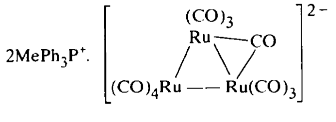
(Received 26 February 1998; accepted 26 June 1998)

Abstract

The structure of the $[Ru_3(CO)_{11}]^{2-}$ dianion was determined from an X-ray single-crystal structure determination of bis(methyltriphenylphosphonium) μ-carbonyl-1:2κ^2C-decacarbonyl-1κ^3C,2κ^3C,3κ^4C-triruthenium-(3 *Ru—Ru*) dichloromethane solvate, $[(C_6H_5)_3(CH_3)P]_2$-$[Ru_3(CO)_{11}]\cdot CH_2Cl_2$, and was found to contain a single bridging CO ligand between two Ru atoms. This result supports the IR and ^{13}C NMR spectroscopic studies reported earlier [Nagel, Bricker, Alway & Shore (1981). *J. Organomet. Chem.* **219**, C9–12; Bhattacharyya, Nagel & Shore (1983). *Organometallics*, **2**, 1187–1193].

Comment

The structure of the $[Fe_3(CO)_{11}]^{2-}$ dianion was shown to contain both bridging and capping CO ligands (Lo *et al.*, 1980). However, its closest analogue $[Ru_3(CO)_{11}]^{2-}$ appears to contain only a single bridging CO ligand based on IR and ^{13}C NMR spectroscopic studies (Nagel *et al.*, 1981; Bhattacharyya *et al.*, 1983). The structure of $[Ru_3(CO)_{11}]^{2-}$ has now been determined and it supports the interpretations of the spectroscopic data. We report herein the structure of $[Ph_3(CH_3)P]_2$-$[Ru_3(CO)_{11}]\cdot CH_2Cl_2$, (I), obtained from a single-crystal X-ray diffraction analysis.



(I)

The molecular structure of the $[Ru_3(CO)_{11}]^{2-}$ dianion is shown in Fig. 1. It consists of a single symmetrically bridging CO ligand between atoms Ru1 and Ru3. The Ru1 and Ru3 atoms each have three terminal CO ligands. In contrast, Ru2 has four terminal CO ligands. The bridging *CB*—*OB* bond distance is 1.200 (5) Å, which is similar to that observed in the monoanion $[(\mu\text{-H})Ru_3(CO)_{11}]^-$ [*CB*—*OB* 1.180 (12) Å; Johnson *et al.*, 1979]. Due to the bridging CO ligand, the distance between Ru1 and Ru3 is 2.7759 (6) Å, which is shorter than those for Ru1—Ru2 [2.8787 (6) Å] and Ru2—Ru3 [2.8744 (6) Å], two non-bridged Ru—Ru bonds. The asymmetrically capping CO ligand observed in $[Fe_3(CO)_{11}]^{2-}$ is obtained from an Fe atom that has no bridging CO ligand connected to it. In the case of $[Ru_3(CO)_{11}]^{2-}$, this CO ligand is in a terminal position. The $[Ru_3(CO)_{11}]^{2-}$ dianion is isoelectric and isostructural with the neutral $Co_2Ru(CO)_{11}$ complex (Roland & Vahrenkamp, 1985).

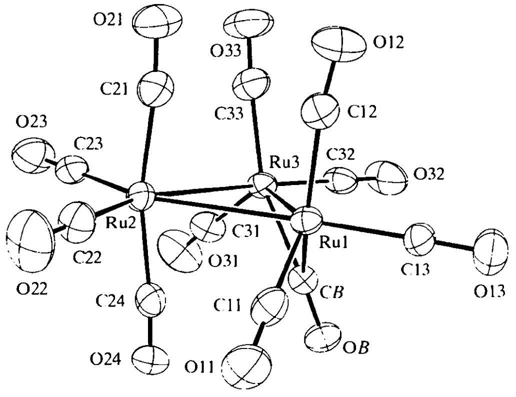


Fig. 1. The molecular structure of the $[Ru_3(CO)_{11}]^{2-}$ dianion shown with 50% probability displacement ellipsoids.

Experimental

$K_2[Ru_3(CO)_{11}]$ was prepared according to the literature method of Bhattacharyya *et al.* (1983). $[Ph_3(CH_3)P]_2$-$[Ru_3(CO)_{11}]\cdot CH_2Cl_2$ was synthesized by the metathesis reaction of $K_2[Ru_3(CO)_{11}]$ with $[Ph_3(CH_3)P]Br$ in mixed CH_2Cl_2/THF solvent. Single crystals were obtained from a hexane-layered methylene chloride/THF solution at 238 K.

Crystal data

$(C_{19}H_{18}P)_2[Ru_3(CO)_{11}]$·-
CH_2Cl_2
$M_r = 1250.85$
Monoclinic
$P2_1/n$
$a = 9.486 (1)$ Å
$b = 21.276 (4)$ Å
$c = 25.266 (4)$ Å
$\beta = 94.92 (1)^\circ$
$V = 5081 (2)$ Å^3
$Z = 4$
$D_x = 1.635$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12–15^\circ$
$\mu = 1.106$ mm^{-1}
$T = 213 (1)$ K
Rectangular
0.3 × 0.3 × 0.2 mm
Maroon

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction:
ψ scan (North *et al.*, 1968)
$T_{min} = 0.718$, $T_{max} = 0.801$

7020 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 25^\circ$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 25$
$l = -29 \rightarrow 29$

9685 measured reflections
8908 independent reflections

4 standard reflections
frequency: 180 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.033$
$wR(F^2) = 0.084$
$S = 1.049$
8854 reflections
613 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 11.2195P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.726$ e Å^{-3}
$\Delta\rho_{\min} = -0.618$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Ru1—Ru3	2.7759 (6)	Ru2—C23	1.898 (5)
Ru1—Ru2	2.8787 (6)	Ru2—C22	1.902 (5)
Ru2—Ru3	2.8744 (6)	Ru2—C24	1.936 (5)
Ru1—C*B*	2.069 (4)	Ru2—C21	1.937 (5)
Ru3—C*B*	2.068 (4)	Ru3—C32	1.847 (5)
Ru1—C13	1.860 (4)	Ru3—C31	1.892 (5)
Ru1—C11	1.882 (5)	Ru3—C33	1.944 (5)
Ru1—C12	1.946 (5)	C*B*—O*B*	1.200 (5)
Ru3—Ru1—Ru2	61.075 (15)	O*B*—C*B*—Ru3	137.7 (3)
Ru3—Ru2—Ru1	57.697 (14)	O*B*—C*B*—Ru1	137.6 (3)
Ru1—Ru3—Ru2	61.228 (15)	C13—Ru1—C*B*	90.8 (2)
Ru3—C*B*—Ru1	84.29 (15)	C11—Ru1—C*B*	98.1 (2)
C*B*—Ru1—Ru2	83.91 (11)	C12—Ru1—C*B*	143.2 (2)
C*B*—Ru1—Ru3	47.83 (11)	C32—Ru3—C*B*	89.8 (2)
C*B*—Ru3—Ru1	47.88 (11)	C31—Ru3—C*B*	96.9 (2)
C*B*—Ru3—Ru2	84.05 (11)	C33—Ru3—C*B*	147.1 (2)

No extinction correction was applied. All non-H atoms were refined anisotropically. The H-atom positional parameters were calculated assuming ideal geometries around C atoms. The isotropic displacement parameters for H atoms were set to $U_{iso}(H) = 1.2U_{iso}(C)$ for CH groups in phenyl rings and the CH_2 group in CH_2Cl_2, and to $U_{iso} = 1.5U_{iso}(C)$ for CH_3 groups. The positional and displacement parameters were fixed during the refinement.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *XPREP* in *SHELXTL* (Siemens, 1994). Program(s) used to solve structure: *XS* in *SHELXTL*. Program(s) used to refine structure: *XL* in *SHELXTL*. Molecular graphics: *XP* in *SHELXTL*. Software used to prepare material for publication: *XCIF* in *SHELXTL*.

This work is supported by the National Science Foundation through grant CHE97-00394.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1022). Services for accessing these data are described at the back of the journal.

References

Bhattacharyya, A. A., Nagel, C. C. & Shore, S. G. (1983). *Organometallics*, **2**, 1187–1193.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Johnson, B. F. G., Lewis, J., Raithby, P. R. & Suss, G. (1979). *J. Chem. Soc. Dalton Trans.* pp. 1356–1361.

Lo, F. Y.-K., Longoni, G., Chini, P., Lower, L. D. & Dahl, L. F. (1980). *J. Am. Chem. Soc.* **102**, 7691–7701.

Nagel, C. C., Bricker, J. C., Alway, D. G. & Shore, S. G. (1981). *J. Organomet. Chem.* **219**, C9–12.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Roland, E. & Vahrenkamp, H. (1985). *Chem. Ber.* **118**, 1133–1142.

Siemens (1994). *SHELXTL*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1999). C**55**, 30–32

$[PPh_4]_2[Pd\{Se_2C_2(COOCH_3)_2\}_2]$

CRAIG C. MCLAUCHLAN AND JAMES A. IBERS

Department of Chemistry, Northwestern University, 2145 Sheridan Rd, Evanston, IL 60208-3113, USA. E-mail: ibers@chem.nwu.edu

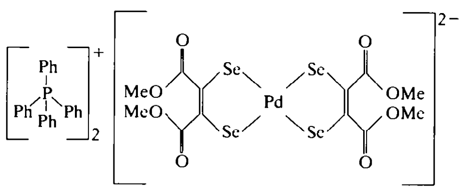
(*Received 2 April 1998; accepted 4 August 1998*)

Abstract

The title compound, bis(tetraphenylphosphonium) bis(*cis*-1,2-dicarbomethoxyethylene-1,2-diseleno)palladate(II) {alternative name: bis(tetraphenylphosphonium) bis[dimethyl 2,3-bis(selenido)but-2-ene-1,4-dioato-*Se*,*Se*′]palladate(II)}, $(C_{24}H_{20}P)_2[Pd(C_6H_6O_4Se_2)_2]$, shows a similar structure to the telluro, seleno, and sulfido analogues with other transition metals.

Comment

The reaction of activated acetylenes with polychalcogenide chains is well documented (Bolinger & Rauchfuss, 1982). One reaction of interest is that of dimethyl acetylenedicarboxylate with selenometalates (Ansari *et al.*, 1989). Whereas many reactions of this type have been attempted, few of the products have been structurally characterized (Ansari *et al.*, 1990). In addition, no reactions had been attempted with the use of Group X selenometalates. By reacting dimethyl acetylenedicarboxylate with a selenopalladate species, the title compound, (I), was synthesized.



(I)

This structure possesses similar bond lengths to the other known *cis*-1,2-dicarbomethoxyethylene-1,2-di-