from *International Tables for X-ray Crystallography* (1974). Final R was 0·06. The ratio of maximum least-squares shift to e.s.d. in final refinement cycle was 0·009, maximum electron density in final difference Fourier synthesis was 0·62 e Å^{-3}.

Discussion. Final parameters are shown in Table 1.* The molecular structure of the complex including the atomic numbering scheme is illustrated in Fig. 1. Associated bond parameters for the structure and selected torsion angles are present in Table 2.

The Cu atoms have a highly distorted octahedral coordination geometry with the p-phdta ligand and the OW1 water molecule at the vertices of the square and the OW2 water molecule and the O11 (through the symmetry operation $x + \frac{1}{2}$, $-y$, z) occupying the apical positions. The Cu···O11($x + \frac{1}{2}$, $-y$, z) bond distance is 2·442 (5) Å and the angle O(OW2)··· Cu^{II}···O11($x + \frac{1}{2}$, $-y$, z) is 160·7 (2)°. The Cu^{II} −OW2 and the Cu^{II} −O11($x + \frac{1}{2}$, $-y$, z) axial bonds are, respectively, 0·33 and 0·47 Å longer than the average Cu^{II}—O in-plane bond length, owing to the Jahn–Teller effect. The Cu^{II}—OW1 bond distance, 2·007 Å, *trans* to N4, is shorter than those observed for Cu—OW2, 2·298 (5) Å. The Cu—N bond length, 2·078 (5) Å, compares well with that observed in methylenetriaminepentaacetate

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52759 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

cuprate(II) monohydrate (Seccombe, Lee & Henry, 1975) [average 2·049 (2) Å]. The Cu—O—C—C—N five-membered chelate rings have envelope conformations with the common N atom (N4) out of plane.

The hydrate water molecule (OW3) participates in intermolecular hydrogen bonds: OW3···O7 (x, y, z) 2·702 (5) Å and OW3···OW1 ($x - \frac{1}{2}$, $-y + \frac{1}{2}$, $-z + \frac{1}{2}$) 2·666 (5) Å. The OW2 is hydrogen bonded: OW2···O8 ($x + \frac{1}{2}$, $-y + \frac{1}{2}$, $-z + \frac{1}{2}$) 2·764 (5) Å.

References

AZUMA, S., NAKASUKA, N. & TANAKA, M. (1986). *Acta Cryst.* C42, 673–677.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
MCCANDLISH, E. F. K., MICHAEL, T. K., NEAL, J. A., LINGAFELTER, E. C. & ROSE, N. J. (1978). *Inorg. Chem.* 17, 1383–1394.
MEDEROS, A., FELIPE, J. M., BRITO, F. & BAZDIKIAN, K. (1986). *J. Coord. Chem.* 14, 285–291.
NAKASUKA, N., AZUMA, S., KATAYAMA, C., HONDA, M., TANAKA, J. & TANAKA, M. (1985). *Acta Cryst.* C41, 1176–1179.
NAKASUKA, N., AZUMA, S. & TANAKA, M. (1986). *Acta Cryst.* C42, 1482–1485.
SECCOMBE, C., LEE, B. & HENRY, G. M. (1975). *Inorg. Chem.* 14, 1147–1531.
SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). *SHELXS86.* Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.
STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1980). The *XRAY*80 system. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.
WALKER, N. & STUART, D. (1983). *Acta Cryst.* A39, 158–166.

Structure of Tetraphenylphosphonium Tetracyanooxo[N-o-tolylhydroxylaminato-(2 −)-O,N]molybdate(VI)

By Fouad Ridouane, José Sanchez, Henri Arzoumanian and Marcel Pierrot

Laboratoire de Cristallochimie - ESIPSOI-UA126, Faculté des Sciences, St Jérôme, 13397 Marseille, France

Abstract. [P(C$_6$H$_5$)$_4$]$_2$[Mo(CN)$_4$(O)(C$_7$H$_7$NO)], M_r = 1016, monoclinic, $P2_1/n$, $a = 11·332$ (4), $b = 13·698$ (4), $c = 36·06$ (1) Å, $\beta = 92·04$ (5)°, $V = 5594$ (6) Å^3, $Z = 4$, $D_x = 1·21$ g cm^{-3}, $F(000) = 2096$, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 3·26$ cm^{-1}, $T = 293$ K, $R = 0·085$ for 1743 unique diffractometer data with $I > 3\sigma(I)$. The seven-coordinate metal structure is compared to diverse oxo–peroxo or oxaziridine Mo complexes. Crystals of the title compound are composed of [Mo(CN)$_4$(O)(C$_7$H$_7$NO)]$^{2-}$ anions and [P(C$_6$H$_5$)$_4$]$^+$ cations, packed without any remarkable features.

Introduction. Transition-metal-catalyzed reduction of nitric oxide (NO) is important for industrial applications (*Ullmanns Encyklopädie der Technischen Chemie*, 1981) such as exhaust-gas treatment as well as for understanding enzymatic reactions (Hughes,

1981; Murphy, Takenchi, Barley & Meyer, 1986) in the transformation of nitrate to ammonia. Key intermediates in such reductions are O,N-coordinated hydroxylamino(1 −) complexes which are usually obtained in aqueous medium by treatment of the appropriate metal center with a hydroxylamine (Wieghardt, 1984). They can also be obtained by protonation (Wieghardt & Quilitzsch, 1981) or reduction by hydrazine (Sellmann, Seubert, Moll & Knoch, 1988) of the metal-coordinated NO. Many have been isolated and their structures determined by X-ray diffraction (Wieghardt, 1984; Sellmann *et al.*, 1988).

Complexes bearing an O,N-coordinated hydroxylaminato(2 −) ligand in which the hydroxylamine plays the role of a dianion are scarcer (Liebeskind, Sharpeless, Wilson & Ibers, 1978; Muccigrosso, Jacobsen, Apgar & Mares, 1978; Wieghardt & Holzbach, 1979). Most of them are obtained from a hydroxylamine and a metal oxo compound. They have been shown to be important intermediates in the metal-catalyzed reduction of NO but few have been isolated and only one was characterized by X-ray diffraction (Liebeskind *et al.*, 1978). In this latter case it has also been shown that oxidative addition of an arylnitroso compound onto a reduced metal center affords analogous results.

In the course of a study of dioxygen activation by molybdenum oxo complexes we have recently encountered a compound (1) capable of being peroxidized by a facile oxidative addition of O_2 to give the tetraphenylphosphonium tetracyanooxoperoxomolybdate(VI), compound (2) (Arzoumanian, Petrignani, Pierrot, Ridouane & Sanchez, 1988). It seemed desirable to study the reactivity of compound (1) towards arylnitroso compounds as a synthetic route to O,N-coordinated hydroxylaminato(2 −) complexes. We thus reacted compound (1) with o-nitrosotoluene and obtained quantitatively the desired tetraphenylphosphonium tetracyanooxo(N-o-tolylhydroxylaminato(2 −)-O,N]molybdate(VI), compound (3).
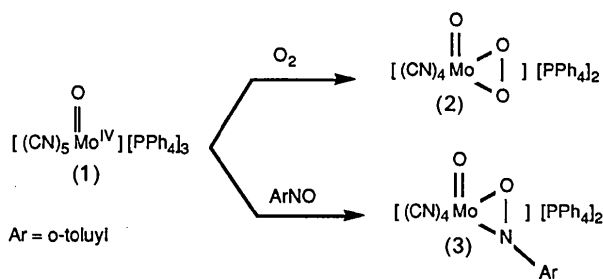
We report here the synthesis and X-ray crystallographic study of compound (3) bearing this type of ligand obtained by the oxidative addition of o-nitrosotoluene onto a cyanooxomolybdate(VI) anion.

Table 1. *Positional and thermal parameters with e.s.d.'s in parentheses*

	x	y	z	B (Å²)
Mo	0·3824 (3)	0·2231 (3)	0·33757 (8)	2·86 (6)*
O(1)	0·250 (2)	0·185 (2)	0·3264 (5)	3·2 (5)
C(1)	0·467 (3)	0·079 (3)	0·346 (1)	5 (1)
N(1)	0·496 (3)	0·003 (3)	0·3483 (9)	6·0 (8)
C(2)	0·349 (3)	0·194 (2)	0·3980 (8)	3·2 (8)
N(2)	0·341 (3)	0·178 (2)	0·4292 (8)	5·7 (8)
C(3)	0·309 (3)	0·364 (3)	0·354 (1)	4·5 (9)
N(3)	0·268 (3)	0·433 (2)	0·3635 (9)	6·2 (9)
C(4)	0·553 (3)	0·279 (3)	0·3671 (9)	4·1 (8)
N(4)	0·634 (3)	0·314 (2)	0·3816 (8)	5·5 (8)
O(2)	0·478 (2)	0·210 (2)	0·2934 (5)	3·8 (5)
N(5)	0·437 (2)	0·306 (2)	0·2913 (6)	2·8 (6)
C(5)	0·349 (3)	0·320 (2)	0·2608 (8)	2·8 (8)
C(6)	0·304 (3)	0·240 (2)	0·2404 (8)	3·1 (7)
C(7)	0·225 (3)	0·254 (2)	0·2105 (9)	3·8 (8)
C(8)	0·191 (3)	0·349 (3)	0·2010 (9)	3·4 (8)
C(9)	0·239 (3)	0·425 (3)	0·2208 (9)	3·2 (8)
C(10)	0·317 (3)	0·415 (3)	0·2500 (9)	3·2 (8)
C(11)	0·377 (4)	0·496 (3)	0·269 (1)	6 (1)
P(1)	0·7378 (8)	0·2422 (7)	0·1997 (2)	3·6 (3)
C(12)	0·8429 (3)	0·2020 (2)	0·1677 (8)	3·0 (7)
C(13)	0·901 (3)	0·269 (3)	0·1447 (9)	3·9 (8)
C(14)	0·989 (3)	0·230 (3)	0·1216 (9)	5·2 (9)
C(15)	1·023 (3)	0·140 (3)	0·123 (1)	5 (1)
C(16)	0·973 (3)	0·072 (3)	0·146 (1)	4·4 (9)
C(17)	0·878 (3)	0·105 (3)	0·167 (1)	4·4 (9)
C(18)	0·628 (3)	0·151 (3)	0·200 (1)	5 (1)
C(19)	0·564 (3)	0·127 (3)	0·1687 (9)	4·3 (9)
C(20)	0·475 (3)	0·056 (3)	0·168 (1)	5 (1)
C(21)	0·451 (4)	0·015 (3)	0·201 (1)	6 (1)
C(22)	0·512 (3)	0·033 (3)	0·2322 (9)	3·5 (8)
C(23)	0·604 (3)	0·099 (3)	0·234 (1)	4·3 (9)
C(24)	0·815 (3)	0·258 (3)	0·2443 (8)	3·0 (7)
C(25)	0·931 (3)	0·243 (2)	0·2496 (8)	3·4 (8)
C(26)	0·992 (3)	0·259 (3)	0·2837 (9)	4·7 (9)
C(27)	0·926 (3)	0·297 (3)	0·3104 (9)	4·3 (8)
C(28)	0·816 (3)	0·318 (3)	0·307 (1)	5 (1)
C(29)	0·751 (3)	0·299 (3)	0·275 (1)	6 (1)
C(30)	0·671 (3)	0·356 (2)	0·1874 (9)	3·1 (8)
C(31)	0·739 (3)	0·439 (3)	0·1865 (9)	4·0 (9)
C(32)	0·696 (3)	0·527 (3)	0·176 (1)	5 (1)
C(33)	0·576 (3)	0·537 (3)	0·167 (1)	5 (1)
C(34)	0·507 (4)	0·459 (3)	0·171 (1)	5 (1)
C(35)	0·551 (3)	0·365 (3)	0·1788 (9)	3·9 (9)
P(2)	0·5661 (9)	0·2958 (8)	0·0339 (2)	3·6 (3)*
C(36)	0·615 (3)	0·173 (3)	0·0475 (9)	3·8 (8)
C(37)	0·540 (4)	0·099 (4)	0·042 (1)	7 (1)
C(38)	0·571 (4)	0·001 (4)	0·051 (1)	8 (1)
C(39)	0·676 (4)	−0·009 (3)	0·068 (1)	7 (1)
C(40)	0·755 (4)	0·060 (3)	0·072 (1)	7 (1)
C(41)	0·726 (4)	0·157 (3)	0·063 (1)	6 (1)
C(42)	0·688 (3)	0·373 (3)	0·0407 (9)	3·4 (8)
C(43)	0·764 (4)	0·411 (3)	0·010 (1)	6 (1)
C(44)	0·861 (5)	0·472 (4)	0·012 (1)	10 (2)
C(45)	0·884 (4)	0·500 (4)	0·048 (1)	8 (1)
C(46)	0·810 (4)	0·473 (3)	0·079 (1)	6 (1)
C(47)	0·710 (3)	0·414 (3)	0·074 (1)	4·7 (9)
C(48)	0·509 (3)	0·288 (3)	−0·0138 (8)	3·3 (7)
C(49)	0·374 (4)	0·312 (3)	−0·023 (1)	6 (1)
C(50)	0·347 (4)	0·299 (3)	−0·060 (1)	6 (1)
C(51)	0·421 (3)	0·270 (3)	−0·085 (1)	6 (1)
C(52)	0·532 (3)	0·248 (3)	−0·0769 (9)	5 (1)
C(53)	0·581 (3)	0·252 (3)	−0·042 (1)	6 (1)
C(54)	0·453 (3)	0·337 (3)	0·0631 (9)	3·8 (8)
C(55)	0·417 (4)	0·431 (3)	0·061 (1)	6 (1)
C(56)	0·322 (4)	0·470 (3)	0·079 (1)	6 (1)
C(57)	0·268 (4)	0·408 (3)	0·101 (1)	7 (1)
C(58)	0·297 (4)	0·316 (3)	0·105 (1)	6 (1)
C(59)	0·391 (4)	0·275 (3)	0·0855 (9)	5·2 (9)

* Anisotropically refined atoms, whose thermal parameters are given in the form of the isotropic equivalent displacement parameter defined as: $4/3[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab\cos\gamma B(1,2) + ac\cos\beta B(1,3) + bc\cos\alpha B(2,3)]$.

$$[(CN)_5 Mo^{IV}][PPh_4]_3$$
(1)

Ar = o-toluyl

$$[(CN)_4 Mo \, (O)(O_2)][PPh_4]_2$$
(2)

$$[(CN)_4 Mo \, (O)(N-Ar)][PPh_4]_2$$
(3)

Experimental. Tetraphenylphosphonium pentacyano-oxomolybdate(IV) (1·5 g, 1·19 mmol) in dichloromethane (20 ml) was reacted at 243 K with an equimolecular amount of o-nitrosotoluene (0·14 g). The red solution obtained was allowed to reach

Table 2. *Selected bond distances* (Å) *and angles* (°)
with e.s.d.'s in parentheses

Mo—O(1)	1·629 (7)	C(2)—N(2)	1·152 (11)
Mo—C(1)	2·209 (14)	C(3)—N(3)	1·109 (13)
Mo—C(2)	2·262 (10)	C(4)—N(4)	1·136 (12)
Mo—C(3)	2·189 (13)	O(2)—N(5)	1·393 (10)
Mo—C(4)	2·310 (12)	C(1)—N(1)	1·093 (14)
Mo—O(2)	1·968 (6)	Mo—N(5)	2·126 (7)

O(1)—Mo—C(1)	98·1 (4)	O(2)—Mo—N(5)	39·6 (3)
O(1)—Mo—C(2)	89·8 (3)	Mo—C(1)—N(1)	171· (1)
O(1)—Mo—C(3)	89·4 (4)	Mo—C(2)—N(2)	175· (1)
O(1)—Mo—C(4)	166·7 (3)	Mo—C(3)—N(3)	177· (1)
O(1)—Mo—O(2)	107·5 (3)	Mo—C(4)—N(4)	175· (1)
O(1)—Mo—N(5)	105·5 (4)	Mo—O(2)—N(5)	76·3 (5)
C(1)—Mo—C(2)	78·1 (4)	C(2)—Mo—O(2)	151·8 (3)
C(1)—Mo—C(3)	156·2 (4)	C(2)—Mo—N(5)	156·1 (3)
C(1)—Mo—C(4)	82·2 (5)	C(3)—Mo—C(4)	84·5 (4)
C(1)—Mo—O(2)	77·5 (4)	C(3)—Mo—O(2)	121·8 (4)
C(1)—Mo—N(5)	116·7 (4)	C(3)—Mo—N(5)	82·5 (4)
C(2)—Mo—C(3)	79·3 (4)	C(4)—Mo—O(2)	85·7 (3)
C(2)—Mo—C(4)	77·5 (4)	C(4)—Mo—N(5)	85·5 (3)

298 K within 1 h and the product was precipitated with diethyl ether. The red solid obtained (98%) was recrystallized from acetonitrile/diethyl ether (60/40). IR (KBr) 920 cm^{-1} (ν Mo=O). Anal. calc. for $C_{59}H_{47}MoN_5P_2$: C, 69·75, H, 4·63; N, 6·90; P, 6·11; Mo, 9·53. Found: C, 68·99; H, 4·65; N, 6·42; P, 6·01; Mo, 9·53.

Crystal (0·4 × 0·3 × 0·2 mm) grown from acetonitrile–ether solution, sealed in a capillary under inert atmosphere, mounted on a CAD-4 diffractometer (graphite monochromator for Mo $K\alpha$). Unit-cell parameters refined by least squares on $\sin\theta/\lambda$ values for 25 reflections ($16 < \theta < 18°$). Density obtained by flotation: $D_m = 1·16$ (4) g cm^{-3}. Intensities measured using $\omega/2\theta$ scan of 0·91 to 10·06° min^{-1}, over a range of $(0·8 + 0·35\tan\theta)°$, $\theta_{max} = 20°$. Intensities of three standard reflections measured every 10000 s showed a loss of intensity of 10% and a linear correction was applied on the 5208 measured reflections; 926 symmetry-related reflections averaged (agreement factor on $I = 0·042$), and 3972 unique reflections. Finally less than one half of the data set [1742 reflections with $I > 3\sigma(I)$] was kept for structure determination; $0 \le h \le 10$; $0 \le k \le 10$; $-28 \le l \le 27$.

All computations were performed on a PDP 11/44 using the *SDP* package (Frenz, 1978). The structure was solved *via* standard heavy-atom procedures and completed by Fourier methods. H atoms introduced (with $B_{eq} = 6$ Å^2) at idealized positions in the calculation before last refinement cycles but not refined. Full-matrix least-squares refinement included anisotropic thermal parameters for Mo and P atoms only and minimized the function $w(F_o - F_c)^2$, $w = 1/\sigma^2$. Final refinement with $R = 0·085$ ($wR = 0·124$), $S = 4·62$, $(\Delta/\sigma)_{max} = 0·12$; a final difference synthesis did not reveal any peak of density $> 0·45$ e Å^{-3}. These relatively high values of the R factors can be attributed (i) to the poor quality of the diffraction pattern: diffraction angle limited to 20° and great

number of non-significant measurements; (ii) to the isotropic model used for the thermal motion of all but three atoms [Mo, P(1) and P(2)].

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). No correction for absorption or secondary extinction.

Discussion. Final positional and thermal parameters are presented in Table 1.* Relevant bond angles and bond distances found are indicated in Table 2. The seven-coordinate metal structure with ν^2 bonding of the ONR ($R = $ CH$_3$Ph) group is shown in Fig. 1.

The geometry about the Mo atom is pentagonal bipyramidal with the O and N atoms of the ONR group and the C atoms C(1), C(2) and C(4) of the ligand occupying the equatorial positions. Some important structural parameters are comparable to those found in the structure of oxo[N-phenyl-hydroxylamine-O,N)pyridine-2,6-dicarboxylato] ($=$ hmpa)molybdenum(VI), hereafter named compound (4) (Liebeskind *et al.*, 1978): the phenyl ring of the R group does not lie in the equatorial plane and the torsional angle Mo—O(2)—N(5)—C(5) is 110·8 (6)° [111·4 (4)° in compound (4)]; the N atom of the molybdooxaziridium ring displays bond angles consistent with sp^3 hydridization, *e.g.* O(2)—N(5)—C(5) is 112·5 (7)° [112·6 (4)° in compound (4)] and the N—O distance is 1·39 (1) Å [1·416 (7) Å in compound (4)], which is consistent with a single-bond distance.

It is also interesting to compare the structure of the title compound to those of compound (2) and

---

* Tables of anisotropic thermal parameters, bond angles, bond distances, H-atom coordinates and lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52815 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
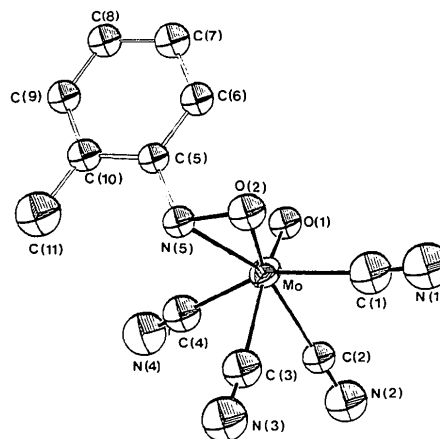

Fig. 1. View of the anion (3) and the atomic numbering scheme.

Table 3. *Structural comparisons with oxo-peroxocyanomolybdates*(VI)

	Mo distances (Å) to				
Compound	Equatorial plane	Oxygen oxo	Ligand *trans*/oxo	Carbon *trans*/peroxo	Carbon *cis*
(2)	0·270 (1)	1·694 (3)	2·197 (4)	2·167 (5)	2·175 (4)–2·291 (4)
(3)	0·268 (6)	1·629 (7)	2·19 (1)	2·26 (1)	2·21 (1)–2·31 (1)
(5)	0·213 (4)	1·638 (7)	2·162 (6)*	2·082 (8)	2·191 (1)–2·173 (9)

* O atom linked to hmpa ligand.

compound (5): [Mo(O)(O₂)(CN)₃hmpa] [PPh₃CH₂Ph] (Arzoumanian *et al.*, 1988).

In all cases the Mo atom is out of the equatorial plane (see Table 3). However, the scattering of the Mo—C_{cis} distances does not allow us to distinguish any *trans* effect on the Mo—L distances due to the oxygen 'oxo' atom.

Crystals of the title compound are composed of individual entities: one [Mo(CN)₄(O)(ONPhCH₃)]²⁻ anion for two [PPh₄]⁺ cations which are packed without any remarkable features. Bond distances and angles found in the [PPh₄]⁺ cations are given in the deposited material.

The general character of the reaction given in the scheme as well as the use of these compounds, considered as metaloxoaziridines, in the synthesis of allylic amines or organic aziridines is presently under investigation.

References

Arzoumanian, H., Petrignani, J. F., Pierrot, M., Ridouane, F. & Sanchez, J. (1988). *Inorg. Chem.* 27, 3377–3381.

Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.

Hughes, M. N. (1981). *The Inorganic Chemistry of Biological Processes*, 2nd ed. New York: John Wiley.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Liebeskind, L. S., Sharpless K. B., Wilson, R. D. & Ibers, J. A. (1978). *J. Am. Chem. Soc.* 100, 7061–7063.

Muccigrosso, D. A., Jacobson, S. E., Apgar, P. A. & Mares, F. (1978). *J. Am. Chem. Soc.* 100, 7063–1965.

Murphy, W. R., Takenchi, K., Barley, M. H. & Meyer, T. H. (1986). *Inorg. Chem.* 25, 1041–1053.

Sellmann, D., Seubert, B., Moll, M. & Knoch, F. (1988). *Angew. Chem. Int. Ed. Engl.* 27, 1164–1165.

Ullmann's Encyklopädie der Technischen Chemie (1981). Vol. 6, 4th ed., p.172. Weinheim: Verlag Chemie.

Wieghardt, K. (1984). *Adv. Inorg. Bioinorg. Mech.* 3, 213–239, and references therein.

Wieghardt, K. & Holzbach, W. (1979). *Angew. Chem. Int. Ed. Engl.* 18, 549–549.

Wieghardt, K. & Quilitzsch, U. (1981). *Z. Naturforsch.* 36, 683–686.

μ-(η⁵:η⁵-Pentamethylcyclopentadienyl)-bis[(η⁵-pentamethylcyclopentadienyl-ruthenium(II)] Hexafluorophosphate (1) and [2(η⁵)-Cyclopentadienyl]-μ-[1(η⁵):2(η⁵)-pentamethylcyclopentadienyl]-[1(η⁵)-pentamethylcyclopentadienyl]-osmium(II)ruthenium(II) Hexafluorophosphate (2)

By Paavo O. Lumme* and Urho Turpeinen

Department of Inorganic Chemistry, University of Helsinki, Vuorik. 20, SF-00100 Helsinki, Finland

and A. R. Kudinov and M. I. Rybinskaya

A. N. Nesmeyanov Institute of Organoelement Compounds of the USSR Academy of Sciences, Vavilov St 28, 117813 Moscow, USSR

Abstract. (1) [Ru₂(C₁₀H₁₅)₃]PF₆, $M_r = 752·80$, monoclinic, Cm, $a = 18·855$ (4), $b = 8·792$ (3), $c = 10·494$ (3) Å, $\beta = 113·64$ (2)°, $V = 1593·7$ (8) Å³, $Z = 2$, $D_m = 1·567$, $D_x = 1·569$ g cm⁻³, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu = 10·33$ cm⁻¹, $F(000) = 764$, $T = 295$ (1) K, $R = 0·062$ for 1442 unique reflections with

* To whom all correspondence should be addressed.