

Table 2. Bond lengths (Å) and angles (°)

Ni—O(1)	1.959 (2)	Ni—O(2)	1.966 (3)
Ni—N(1)	1.996 (3)	Ni—N(2)	2.004 (3)
Ni—Cl(1)	2.558 (1)	O(1)—C(1)	1.249 (5)
O(2)—C(7)	1.254 (4)	N(1)—C(5)	1.496 (4)
N(1)—C(6)	1.474 (6)	N(2)—C(2)	1.476 (5)
N(2)—C(3)	1.488 (3)	N(3)—C(1)	1.297 (5)
N(4)—C(7)	1.297 (6)	C(1)—C(2)	1.514 (4)
C(3)—C(4)	1.506 (5)	C(4)—C(5)	1.505 (6)
C(6)—C(7)	1.521 (4)	Cl(2)—O(4)	1.408 (5)
Cl(2)—O(5)	1.405 (4)	Cl(2)—O(6)	1.388 (8)
Cl(2)—O(7)	1.395 (5)		
O(1)—Ni—O(2)	92.5 (1)	O(1)—Ni—N(1)	165.6 (1)
O(2)—Ni—N(1)	83.5 (1)	O(1)—Ni—N(2)	84.3 (1)
O(2)—Ni—N(2)	162.9 (1)	N(1)—Ni—N(2)	95.4 (1)
O(1)—Ni—Cl(1)	99.3 (1)	O(2)—Ni—Cl(1)	95.4 (1)
N(1)—Ni—Cl(1)	94.8 (1)	N(2)—Ni—Cl(1)	101.6 (1)
Ni—O(1)—C(1)	113.5 (2)	Ni—O(2)—C(7)	112.3 (2)
Ni—N(1)—C(5)	114.0 (2)	Ni—N(1)—C(6)	106.4 (2)
C(5)—N(1)—C(6)	112.4 (3)	Ni—N(2)—C(2)	106.7 (2)
Ni—N(2)—C(3)	113.2 (3)	C(2)—N(2)—C(3)	115.0 (3)
O(1)—C(1)—N(3)	122.8 (3)	O(1)—C(1)—C(2)	118.0 (3)
N(3)—C(1)—C(2)	119.2 (4)	N(2)—C(2)—C(1)	107.7 (3)
N(2)—C(3)—C(4)	111.4 (3)	C(3)—C(4)—C(5)	116.3 (3)
N(1)—C(5)—C(4)	112.2 (3)	N(1)—C(6)—C(7)	109.3 (3)
O(2)—C(7)—N(4)	121.8 (3)	O(2)—C(7)—C(6)	118.8 (4)
N(4)—C(7)—C(6)	119.3 (3)	O(4)—Cl(2)—O(5)	108.6 (3)
O(4)—Cl(2)—O(6)	111.1 (4)	O(5)—Cl(2)—O(6)	111.9 (3)
O(4)—Cl(2)—O(7)	105.3 (3)	O(5)—Cl(2)—O(7)	111.1 (3)
O(6)—Cl(2)—O(7)	108.6 (4)		

$S = 1.44$ ,  $(\Delta/\sigma)_{\max} = 0.038$ ,  $\Delta\rho_{\max} = 0.55$ ,  $\Delta\rho_{\min} = -0.51 \text{ e } \text{Å}^{-3}$ . Scattering factors were taken from Cromer & Waber (1974). All calculations were performed on a MicroVAXII computer system using *SHELXTL-Plus* program (Sheldrick, 1986). Atomic parameters are given in Table 1, bond distances and angles in Table 2.\* A molecular drawing of the

\* 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 51752 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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### A Third Modification of $[(\text{Ph}_3\text{P})_2\text{N}][\text{H}_3\text{Ru}_4(\text{CO})_{12}]$

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**Abstract.** Bis(triphenylphosphoranylidene)ammonium tri- $\mu$ -hydrido-tetrahydro-tetrakis(tricarbonylruthenate),

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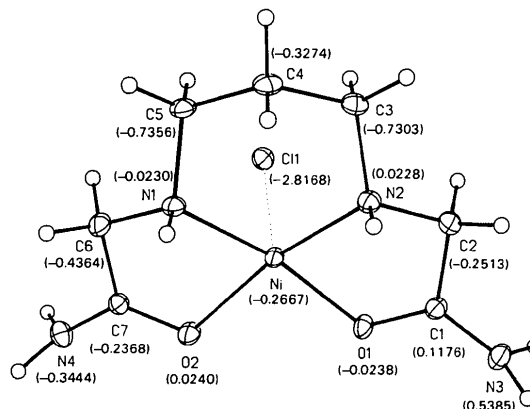


Fig. 1. The molecular drawing of  $[\text{NiCl}(L-1,3,1)]^+$  with the deviations of atoms (Å) from the  $\text{N}_2\text{O}_2$  plane (the atom positions below the plane are indicated by negative signs).

structure and the atomic numbering system are given in Fig. 1.

**Related literature.** Crystal structures of many copper(II) diazadiamide complexes have been determined and reported (Hong, Lee, Lee, Chao & Chung, 1987; Lu, Shan, Chao & Chung, 1987).

Support of this study by the National Science Council is gratefully acknowledged.

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Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )

$U_{\text{eq}}$  is the equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	$x$	$y$	$z$	$U_{\text{eq}}$
Ru(1)	6699.3 (3)	1437.8 (1)	2043.6 (3)	37 (1)
Ru(2)	4221.2 (3)	1426.3 (1)	2134.6 (3)	38 (1)
Ru(3)	5014.7 (3)	1182.2 (1)	526.7 (3)	42 (1)
Ru(4)	5044.8 (4)	2143.3 (1)	1142.3 (3)	45 (1)
H(1)	5618 (55)	1428 (22)	2729 (45)	117 (24)
H(2)	6446 (42)	2012 (17)	1756 (34)	68 (16)
H(3)	5235 (56)	1781 (22)	250 (43)	116 (23)
C(1)	7698 (4)	1661 (2)	3145 (4)	50 (2)
O(1)	8294 (4)	1776 (2)	3796 (3)	80 (2)
C(2)	7809 (5)	1451 (2)	1244 (4)	52 (2)
O(2)	8512 (4)	1461 (2)	801 (3)	84 (2)
C(3)	6909 (4)	799 (2)	2252 (4)	48 (2)
O(3)	7057 (3)	415 (1)	2387 (3)	75 (2)
C(4)	4013 (4)	828 (2)	2589 (4)	52 (2)
O(4)	3883 (4)	470 (2)	2886 (3)	81 (2)
C(5)	3899 (4)	1731 (2)	3208 (4)	54 (2)
O(5)	3679 (4)	1890 (2)	3872 (3)	88 (2)
C(6)	2735 (5)	1457 (2)	1391 (4)	50 (2)
O(6)	1797 (3)	1467 (1)	987 (3)	72 (2)
C(7)	3632 (5)	1182 (2)	-372 (4)	70 (2)
O(7)	2815 (4)	1179 (2)	-921 (3)	117 (3)
C(8)	4787 (5)	569 (2)	850 (4)	61 (2)
O(8)	4629 (4)	184 (1)	990 (3)	91 (2)
C(9)	6032 (6)	1026 (2)	-310 (4)	65 (2)
O(9)	6617 (5)	906 (2)	-803 (4)	108 (2)
C(10)	5881 (5)	2574 (2)	515 (5)	68 (2)
O(10)	6381 (5)	2844 (2)	180 (4)	108 (2)
C(11)	3551 (5)	2286 (2)	480 (4)	61 (2)
O(11)	2641 (4)	2379 (2)	79 (3)	93 (2)
C(12)	4815 (5)	2497 (2)	2184 (5)	60 (2)
O(12)	4703 (4)	2725 (2)	2796 (3)	88 (2)
P(1)	1188 (1)	1159.5 (4)	6269 (1)	31 (1)
P(2)	-644 (1)	769.6 (4)	7216 (1)	32 (1)
N	561 (3)	816 (1)	6864 (3)	35 (1)
C(21)	756 (4)	1062 (2)	5043 (3)	38 (2)
C(22)	1168 (5)	1349 (2)	4417 (3)	55 (2)
C(23)	912 (5)	1249 (2)	3483 (4)	68 (2)
C(24)	217 (6)	874 (2)	3170 (4)	67 (2)
C(25)	-189 (5)	597 (2)	3783 (4)	57 (2)
C(26)	88 (4)	682 (2)	4723 (3)	44 (2)
C(31)	2729 (3)	1045 (2)	6520 (3)	34 (1)
C(32)	3134 (4)	645 (2)	6989 (4)	49 (2)
C(33)	4301 (4)	534 (2)	7110 (4)	62 (2)
C(34)	5067 (5)	817 (2)	6777 (5)	68 (2)
C(35)	4672 (5)	1216 (2)	6327 (5)	80 (3)
C(36)	3511 (5)	1331 (2)	6198 (5)	66 (2)
C(41)	947 (4)	1757 (1)	6481 (3)	34 (1)
C(42)	-12 (4)	1983 (2)	5992 (4)	49 (2)
C(43)	-301 (5)	2418 (2)	6241 (4)	66 (2)
C(44)	381 (6)	2640 (2)	6967 (5)	73 (3)
C(45)	1346 (6)	2425 (2)	7448 (4)	70 (2)
C(46)	1642 (4)	1984 (2)	7213 (4)	52 (2)
C(51)	-1838 (4)	642 (2)	6279 (3)	35 (1)
C(52)	-2303 (4)	996 (2)	5680 (3)	47 (2)
C(53)	-3130 (5)	894 (2)	4910 (4)	61 (2)
C(54)	-3495 (5)	447 (3)	4731 (4)	68 (2)
C(55)	-3017 (5)	94 (2)	5320 (4)	61 (2)
C(56)	-2199 (4)	190 (2)	6090 (3)	45 (2)
C(61)	-503 (4)	294 (2)	8020 (3)	34 (1)
C(62)	588 (4)	107 (2)	8351 (3)	40 (2)
C(63)	694 (5)	-260 (2)	8962 (3)	50 (2)
C(64)	-261 (5)	-443 (2)	9245 (3)	53 (2)
C(65)	-1348 (5)	-256 (2)	8934 (3)	53 (2)
C(66)	-1474 (4)	115 (2)	8329 (3)	44 (2)
C(71)	-1006 (4)	1274 (2)	7831 (3)	35 (1)
C(72)	-2088 (5)	1474 (2)	7703 (4)	62 (2)
C(73)	-2280 (6)	1861 (2)	8213 (5)	84 (3)
C(74)	-1397 (7)	2039 (2)	8848 (4)	72 (3)
C(75)	-319 (6)	1835 (2)	8997 (4)	74 (3)
C(76)	-126 (5)	1456 (2)	8500 (4)	56 (2)

**Experimental.** A brown needle  $0.6 \times 0.15 \times 0.1$  mm was mounted in a glass capillary. 8685 profile-fitted intensities were registered on a Stoe-Siemens four-circle diffractometer using monochromated Mo  $K\alpha$  radiation ( $2\theta_{\text{max}} 50^\circ$ ). An absorption correction based on  $\psi$  scans was applied, with resulting transmission factors 0.85–0.91. Three check reflections showed no significant intensity variation. Merging equivalents gave 8525 unique reflections ( $R_{\text{int}} 0.014$ , index range after merging  $h -13$  to  $13$ ,  $k 0$  to  $34$ ,  $l 0$  to  $17$ ), 6634 of which with  $F > 4\sigma(F)$  were used for all calculations (program *SHELX*, locally modified by its author Professor G. M. Sheldrick). Cell constants were refined from  $2\theta$  values of 48 reflections in the range  $20$ – $23^\circ$ .

The structure was solved by the heavy-atom method and subjected to full-matrix refinement on  $F$  (all non-H atoms anisotropic, cluster H identified in difference syntheses and refined freely, other H using riding model). The final  $R$  value was 0.039, with  $wR$  0.037. The weighting scheme was  $w^{-1} = \sigma^2(F) + 0.0002F^2$ . 616 parameters,  $S$  1.4, max.  $\Delta/\sigma$  0.001, max.  $\Delta\rho$   $0.45 \text{ e \AA}^{-3}$ .

Final atomic coordinates are given in Table 1,\* with selected bond lengths in the caption for Fig. 1

**Related literature.** For a discussion of the four known structure types for  $[(\text{Ph}_3\text{P})_2\text{N}][\text{H}_3\text{M}_4(\text{CO})_{12}]$  ( $M = \text{Ru}$ ,  $\text{Os}$ ), see McPartlin & Nelson (1986). The modification with  $M = \text{Os}$  corresponding to the title compound was, however, not subjected to a complete structure determination, only the cell constants being reported.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51737 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

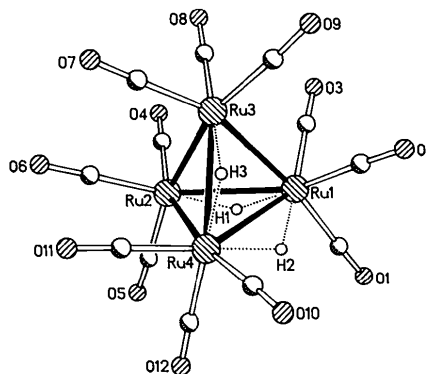


Fig. 1. The anion of the title compound, showing the atom-numbering scheme. Radii are arbitrary. Selected bond lengths ( $\text{\AA}$ ): Ru(1)–Ru(2) 2.915 (1), Ru(1)–Ru(3) 2.796 (1), Ru(1)–Ru(4) 2.955 (1), Ru(2)–Ru(3) 2.773 (1), Ru(2)–Ru(4) 2.799 (1), Ru(3)–Ru(4) 2.921 (1), Ru–H 1.71–1.81 (e.s.d.'s 0.04–0.07), Ru–C 1.867 (6)–1.929 (5).

reflections. The anion has approximate twofold symmetry [the twofold axis passes through H(2) and the midpoints of Ru(1)–Ru(4) and Ru(2)–Ru(3)]. The hydride-bridged Ru–Ru bond lengths are 2.915, 2.921, 2.955  $\text{\AA}$ , the unbridged bond lengths 2.773, 2.796, 2.799 (1)  $\text{\AA}$ .

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## Structure of Dibromotetrakis(dimethyl sulfoxide)osmium(II)

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**Abstract.**  $[\text{OsBr}_2(\text{C}_2\text{H}_6\text{OS})_4]$ ,  $M_r = 662.52$ , tetragonal,  $I4/m$ ,  $a = 9.178$  (2),  $c = 11.126$  (2) Å,  $V = 937.2$  (4) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.35$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 114.66$  cm<sup>-1</sup>,  $F(000) = 628$ ,  $T = 296$  K,  $R = 0.024$  for 425 unique reflections. Crystals were prepared by warming a solution of bis(tetra-*n*-butylammonium) hexabromoosmate(IV) and dimethyl sulfoxide. The molecule has  $4/m$  symmetry with the Os atom at the  $4/m$  intersection, S and O atoms on the symmetry plane, and the *trans*-Br atoms on the fourfold axis. All Me<sub>2</sub>SO ligands are S-bonded to the Os atom. Principal distances and angles are Os–Br = 2.555 (1), Os–S = 2.351 (2), S–O = 1.487 (6), S–C = 1.784 (6) Å, Os–S–C = 115.8 (2), Os–S–O = 113.5 (2), O–S–C = 105.2 (3) and C–S–C' = 99.8 (5)°.

**Experimental.** Preparation by dissolution of 50 mg of bis(tetra-*n*-butylammonium) hexabromoosmate(IV) in 2 ml of dimethyl sulfoxide and heating at 323 K for two weeks. Color of solution changed from wine-red to orange, solution cooled slowly to 253 K, pale-yellow crystals separated by filtration. Crystal used for data collection 0.17 × 0.22 × 0.30 mm, pale-yellow fragment cut from larger tetragonal dipyramid. Rigaku AFC-5S diffractometer, graphite-monochromated Mo  $K\alpha$  radiation,  $\omega$ - $2\theta$  scans, scan speed 6° min<sup>-1</sup>, maximum of three scan repetitions to obtain  $\sigma F/F < 0.10$ . Lattice parameters from least-squares fit of 25 strong reflections in  $2\theta$  range 49–50°. 441 reflections measured ( $h, k$  0 to 10,  $l$  0 to 13), 16 reflections considered unobserved with  $[I < 3\sigma(I)]$ , data set composed of 425 unique reflections,  $(\sin\theta/\lambda)_{\text{max}} = 0.60$  Å<sup>-1</sup>. Three standard reflections (101, 011, 110)

changed by -1.2, -1.1 and 0.5%, respectively; no decay correction applied. Data corrected for Lorentz, polarization and absorption (empirical  $\psi$  scan correction, three reflections, transmission range 0.43–1.00). Systematic absences indicated space group  $I4$ ,  $I\bar{4}$ , or  $I4/m$ ; structure solved in  $I\bar{4}$  and later converted to  $I4/m$  when the plane of symmetry normal to the fourfold axis became apparent. Direct methods used to locate the Os, Br and S atomic sites, C and O positions from difference Fourier synthesis. Full-matrix least-squares refinement was performed to minimize  $\sum w(|F_o| - |F_c|)^2$  where  $w = [1/\sigma^2(|F_o|)]$ . Methyl H atomic sites located from difference Fourier synthesis but failed to refine to reasonable positions. H atoms geometrically optimized from difference Fourier peak positions and fixed (C–H = 0.95 Å, tetrahedral H–C–H and H–C–S angles), all H atom B's set at  $1.2 \times B_{\text{eq}}$  of associated C atom. Regeneration of H positions and refinement to convergence repeated until shifts became insignificant. Final refinement performed with 27 variables including all non-H positional and anisotropic thermal parameters, and one scale factor. Convergence yielded  $R = 0.024$ ,  $wR = 0.034$ ,  $S = 1.14$  and  $(\Delta/\sigma)_{\text{max}} = 0.01$ . Final difference synthesis produced  $(\Delta\rho)_{\text{max}} = 1.33$  and  $(\Delta\rho)_{\text{min}} = -0.69$  e Å<sup>-3</sup>, located near S(1) and Os(1), respectively. Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974). Positional and thermal parameters are listed in Table 1, selected interatomic distances and angles are given in Table 2.†

† Lists of structure factors, H-atom coordinates, intermolecular distances, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51742 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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