

atom in the water molecule. The average Ru—O(edta) distance is 2.018 (3) Å and Ru—O(water) is 2.131 (3) Å. Although the Ru—O distances are somewhat longer than those of the corresponding Fe<sup>III</sup> complex [1.98 (3) and 2.07 (3) Å, respectively], the bond angles for each chelate ring of the Ru complex, N(1)—Ru—O(1) [85.0 (1)°], N(1)—Ru—O(3) [82.0 (1)°], N(2)—Ru—O(7) [80.7 (1)°] and N(1)—Ru—N(2) [86.0 (1)°], are larger than those of the corresponding Fe<sup>III</sup> complex [83 (1), 77 (1), 78 (1) and 83 (1)°, respectively (Kennard, 1967)]. The bond angles between the *trans* positions for the Ru complex, O(1)—Ru—O(7) [173.4 (1)°], N(2)—Ru—O(3) [163.0 (1)°] and N(1)—Ru—O(W) [174.9 (1)°] are also closer to 180° than those of the corresponding Fe<sup>III</sup> complex [167 (1), 155 (1) and 168 (1)°, respectively]. These indicate that [Ru(Hedta)(H<sub>2</sub>O)] assumes a more regular octahedral structure than [Fe(Hedta)(H<sub>2</sub>O)].

Intermolecular hydrogen bonds are recognized between the non-coordinated O atoms in edta and the O atom in water, O(2)⋯O(W) ( $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$ ) [2.819 (4) Å], O(4)⋯O(W) ( $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$ )

[3.139 (5) Å] and O(6)⋯O(W) ( $1-x, 1-y, 1-z$ ) [3.020 (5) Å].

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## The Structure of 1,1,1,2,2,2,3,3-Octacarbonyl- $\mu$ -(1-3- $\eta$ -cyclododeca-1,6,9-trien-1-yl-3-ylidene)-2,3- $\mu$ -hydrido-3-[(*As, As, As', As'*-tetraphenyl)methylenebis(arsine)-*As*]-triangulo-triruthenium(0)(3 Ru–Ru)

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**Abstract.** [Ru<sub>3</sub>(C<sub>12</sub>H<sub>15</sub>)(H)(C<sub>25</sub>H<sub>22</sub>As<sub>2</sub>)(CO)<sub>8</sub>], *M<sub>r</sub>* = 1159.85, orthorhombic, *Pca*2<sub>1</sub>, *a* = 30.385 (2), *b* = 10.047 (2), *c* = 14.402 (3) Å, *V* = 4396 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.752, *D<sub>m</sub>* = 1.740 Mg m<sup>-3</sup> (floatation in chloroform and iodomethane),  $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$ ,  $\mu = 24.14 \text{ cm}^{-1}$ , *F*(000) = 2280, *T* = 295 K, *R* = 0.0429 for 3712 observed reflections. The structure contains a triangle of Ru atoms; Ru(1) and Ru(2) are bonded to three CO groups, and Ru(3) to two. The hydride bridges Ru(2) and Ru(3). The C<sub>12</sub>H<sub>15</sub> ligand is bound in the  $\mu_3\text{-}2\eta^1, \eta^3$  mode to the Ru<sub>3</sub> face. Ru–Ru distances: 2.981 (1) Å (H– bridged), 2.775 (1) and

2.777 (1) Å; The dpam [bis(diphenylarsino)methane] ligand is monodentate with an Ru(3)—As distance of 2.431 (2) Å.

**Introduction.** The reaction between Ru<sub>3</sub>(CO)<sub>12</sub> and 1,5,9-cyclododecatriene [CDT] results in the complex Ru<sub>3</sub>( $\mu$ -H)( $\mu_3\text{-}2\eta^1, \eta^3\text{-C}_{12}\text{H}_{15}$ ) (1) in high yield (Bruce, Cairns & Green, 1972). These authors have also examined the substitution reaction of complex (1) with PMe<sub>2</sub>Ph, P(OMe)<sub>3</sub> and P(OCH<sub>3</sub>)<sub>2</sub>OEt. However, it was not possible for them to determine the precise stereochemistry of the products formed. As part of our study on the coordination of Group 15 bidentate ligands to transition metal clusters, we have determined the structure of the title compound Ru<sub>3</sub>( $\mu$ -H)( $\mu_3\text{-}2\eta^1, \eta^3\text{-C}_{12}\text{H}_{15}$ )( $\mu_1\text{-Ph}_2\text{AsCH}_2\text{AsPh}_2$ )-(CO)<sub>8</sub>.

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Table 1. Atomic coordinates ( $\times 10^4$ ,  $\times 10^5$  for Ru and As) and  $B_{eq}$  values ( $\text{\AA}^2$ )
$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$B_{eq}$
Ru1	-5569 (1)	-14307 (1)	0	3.63 (2)
Ru2	-3418 (1)	-29458 (1)	15461 (1)	3.36 (2)
Ru3	-12167 (1)	-16392 (1)	13269 (1)	3.13 (3)
As1	-17646 (1)	-27145 (1)	23019 (1)	3.35 (4)
As2	-11647 (1)	-39189 (1)	39758 (1)	4.68 (3)
C91	-1652 (4)	-2699 (13)	3617 (8)	4.19 (11)
C12	-2424 (2)	-796 (8)	2819 (6)	4.94 (13)
C13	-2822 (2)	-116 (8)	2739 (6)	6.11 (16)
C14	-3135 (2)	-533 (8)	2093 (6)	6.94 (13)
C15	-3050 (2)	-1629 (8)	1527 (6)	8.61 (18)
C16	-2651 (2)	-2309 (8)	1607 (6)	7.46 (14)
C11	-2338 (2)	-1892 (8)	2253 (6)	3.89 (14)
C22	-1566 (2)	-5393 (7)	1703 (6)	4.46 (13)
C23	-1625 (2)	-6769 (7)	1679 (6)	5.84 (17)
C24	-1990 (2)	-7342 (7)	2109 (6)	5.86 (18)
C25	-2296 (2)	-6538 (7)	2563 (6)	5.91 (13)
C26	-2237 (2)	-5162 (7)	2587 (6)	5.63 (15)
C21	-1872 (2)	-4589 (7)	2157 (6)	3.61 (17)
C32	-939 (3)	-3884 (9)	5907 (7)	7.33 (12)
C33	-767 (3)	-3330 (9)	6717 (7)	7.75 (11)
C34	-655 (3)	-1984 (9)	6742 (7)	8.45 (14)
C35	-715 (3)	-1192 (9)	5957 (7)	8.11 (17)
C36	-887 (3)	-1746 (9)	5146 (7)	5.95 (16)
C31	-999 (3)	-3092 (9)	5121 (7)	4.85 (14)
C42	-1356 (4)	-6667 (10)	4242 (8)	6.96 (18)
C43	-1585 (4)	-7767 (10)	4583 (8)	9.15 (15)
C44	-1961 (4)	-7582 (10)	5125 (8)	9.05 (21)
C45	-2107 (4)	-6298 (10)	5327 (8)	8.54 (23)
C46	-1877 (4)	-5199 (10)	4987 (8)	6.83 (17)
C41	-1502 (4)	-5383 (10)	4444 (8)	5.30 (15)
C1R1	-437 (4)	33 (14)	839 (9)	4.73 (17)
C2R1	-813 (5)	-398 (13)	-949 (11)	5.32 (15)
C3R1	-4 (4)	-1505 (16)	-618 (13)	7.32 (24)
C1R2	-244 (4)	-1865 (13)	2641 (9)	4.51 (23)
C2R2	-345 (4)	-4582 (13)	2138 (9)	4.47 (16)
C3R2	278 (4)	-2906 (11)	1329 (10)	4.47 (26)
C1R3	-1097 (4)	-423 (12)	2324 (9)	4.21 (18)
C2R3	-1614 (4)	-411 (12)	846 (9)	4.36 (19)
O1R1	-355 (3)	915 (10)	1246 (9)	7.29 (22)
O2R1	-968 (4)	224 (10)	-1539 (9)	7.74 (17)
O3R1	337 (4)	-1560 (13)	-943 (11)	9.53 (25)
O1R2	-172 (4)	-1253 (11)	3287 (8)	7.05 (17)
O2R2	-347 (4)	-5676 (10)	2435 (8)	6.81 (21)
O3R2	643 (3)	-2846 (11)	1211 (9)	7.30 (24)
O1R3	-1047 (3)	329 (10)	2907 (7)	6.32 (16)
O2R3	-1864 (3)	326 (9)	521 (8)	6.68 (18)
C1	-479 (3)	-3636 (9)	183 (7)	3.05 (17)
C2	-859 (3)	-3419 (10)	-287 (8)	3.74 (24)
C3	-1209 (4)	-2571 (10)	58 (8)	3.72 (21)
C4	-1576 (4)	-2434 (12)	-689 (10)	4.73 (22)
C5	-1919 (4)	-3548 (13)	-618 (12)	5.50 (18)
C6	-1751 (6)	-4918 (19)	-820 (19)	9.01 (16)
C7	-1574 (5)	-5424 (23)	-1435 (24)	10.33 (21)
C8	-1349 (5)	-6714 (17)	-1547 (16)	7.93 (18)
C9	-855 (7)	-6560 (16)	-1211 (17)	10.88 (25)
C10	-710 (7)	-6496 (17)	-502 (17)	9.13 (16)
C101	-246 (4)	-6069 (11)	-140 (11)	5.21 (27)
C102	-143 (4)	-4547 (11)	-325 (9)	4.55 (23)

Table 2. Selected bond distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with e.s.d.'s in parentheses

Ru2—Ru1	2.775 (1)	C1R2—Ru2	1.938 (13)
Ru3—Ru1	2.777 (1)	C2R2—Ru2	1.852 (13)
C1R1—Ru1	1.937 (13)	C3R2—Ru2	1.910 (12)
C2R1—Ru1	1.883 (14)	C1—Ru2	2.124 (9)
C3R1—Ru1	1.903 (14)	As1—Ru3	2.431 (2)
C1—Ru1	2.243 (9)	C1R3—Ru3	1.920 (13)
C2—Ru1	2.237 (10)	C2R3—Ru3	1.860 (12)
C3—Ru1	2.289 (11)	C3—Ru3	2.054 (12)
Ru3—Ru2	2.982 (1)		
Ru3—Ru1—Ru2	64.9 (1)	Ru3—Ru2—Ru1	57.6 (1)
C1R1—Ru1—Ru2	82.6 (4)	C1R2—Ru2—Ru1	112.4 (4)
C1R1—Ru1—Ru3	76.4 (4)	C1R2—Ru2—Ru3	88.6 (4)
C2R1—Ru1—Ru2	168.9 (4)	C2R2—Ru2—Ru1	148.8 (4)
C2R1—Ru1—Ru3	104.0 (4)	C2R2—Ru2—Ru3	115.8 (3)
C2R1—Ru1—C1R1	96.4 (6)	C2R2—Ru2—C1R2	97.1 (6)
C3R1—Ru1—Ru2	98.4 (5)	C3R2—Ru2—Ru1	95.1 (4)
C3R1—Ru1—Ru3	163.0 (5)	C3R2—Ru2—Ru3	148.5 (3)
C3R1—Ru1—C1R1	98.9 (6)	C3R2—Ru2—C1R2	88.3 (5)
C3R1—Ru1—C2R1	92.7 (7)	C3R2—Ru2—C2R2	95.7 (5)
C1—Ru1—Ru2	48.7 (2)	C1—Ru2—Ru1	52.5 (2)
C1—Ru1—Ru3	85.5 (3)	C1—Ru2—Ru3	82.6 (3)
C1—Ru1—C1R1	131.0 (5)	C1—Ru2—C1R2	164.9 (5)
C1—Ru1—C2R1	132.2 (5)	C1—Ru2—C2R2	97.7 (5)
C1—Ru1—C3R1	85.6 (5)	C1—Ru2—C3R2	92.8 (5)
C2—Ru1—Ru2	75.8 (3)	Ru2—Ru3—Ru1	57.5 (1)
C2—Ru1—Ru3	76.3 (3)	As1—Ru3—Ru1	157.7 (1)
C2—Ru1—C1R1	150.5 (5)	As1—Ru3—Ru2	110.7 (1)
C2—Ru1—C2R1	100.8 (5)	C1R3—Ru3—Ru1	109.3 (3)
C2—Ru1—C3R1	103.9 (6)	C1R3—Ru3—Ru2	91.8 (3)
C2—Ru1—C1	35.2 (4)	C1R3—Ru3—As1	88.9 (4)
C3—Ru1—Ru2	84.3 (3)	C2R3—Ru3—Ru1	99.3 (4)
C3—Ru1—Ru3	46.6 (3)	C2R3—Ru3—Ru2	155.5 (4)
C3—Ru1—C1R1	121.3 (5)	C2R3—Ru3—As1	93.8 (4)
C3—Ru1—C2R1	86.8 (5)	C2R3—Ru3—C1R3	88.8 (5)
C3—Ru1—C3R1	139.6 (6)	C3—Ru3—Ru1	54.1 (3)
C3—Ru1—C1	66.0 (4)	C3—Ru3—Ru2	83.3 (3)
C3—Ru1—C2	37.3 (4)	C3—Ru3—As1	108.6 (3)
		C3—Ru3—C1R3	162.4 (5)
		C3—Ru3—C2R3	88.8 (5)

**Experimental.** The compound is one of the three products formed from the reaction of Ru<sub>3</sub>( $\mu$ -H)( $\mu_3$ -2 $\eta^1$ , $\eta^3$ -C<sub>12</sub>H<sub>15</sub>) and the arsine ligand in refluxing THF. Red crystals suitable for X-ray analysis were grown from a dichloromethane/methanol mixture by solvent diffusion. Weissenberg and Buerger precession photographs yielded approximate cell dimensions and showed an orthorhombic system with space group *Pca*<sub>21</sub> from systematic absences  $0kl$ ,  $l = 2n + 1$ ;  $h0l$ ,  $h = 2n + 1$ . Data collection on crystal  $0.5 \times 0.4 \times 0.2$  mm was obtained with Syntex P<sub>21</sub> diffractometer using  $2\theta/\theta$  mode on octant of the sphere ( $0 < h < 31$ ,  $0 < k < 11$ ,  $0 < l < 15$ ) out to the  $2\theta$  limit

of  $52^\circ$ . A variable scan speed of  $5.0$ – $29.3^\circ \text{ min}^{-1}$  was used. Lattice parameters refined using 25 reflections in the range  $25 < 2\theta < 35^\circ$ . Standard reflection ( $20\bar{1}$ ) checked every 25 reflections: no significant deviation. The data were corrected for Lorentz and polarization effects. 4530 reflections were collected, 4479 unique ( $R_{int} = 0.0435$ ), of which 3721 observed reflections with  $I > 2.5\sigma(I)$  were used for refinement of the structure. The three heavy atoms of Ru were first located through *SHELXS86* (Sheldrick, 1986) and were then used to initiate the starting phase model for the difference method to reveal the other atoms through *SHELX76* (Sheldrick, 1976). Coefficients of scattering factors for C, H, N and O inlaid in *SHELX76*; coefficients of scattering factors and their anomalous-scattering factors for Ru and As from *International Tables for X-ray Crystallography* (1974, Vol. IV). Structure determination and refinement performed on IBM 4361/4381. Refinement by blocked full-matrix least squares based on  $F$  with anisotropic thermal factors for all the non-H atoms, isotropic for H atoms (block 1: 371 scale, positional and thermal parameters of Ru, dpam and CO atoms; block 2: 388 scale, positional and thermal parameters of Ru, dpam and CDT atoms). H atoms (except metal-bonded hydride) were fixed (C—H  $0.96 \text{ \AA}$ ) but not refined. In order to

correct the observed structure factors for absorption effects, program *ABSORB* (Ugozzoli, 1987) was used (transmission factors min.—max.: 0.7451–1.2486). The final agreement factors were  $R = 0.0432$ ,  $wR = 0.0458$ . For completeness, the opposite enantiomorph was also refined and convergence was attained at  $R = 0.0429$  and  $wR = 0.0455$ . The ratio of  $wR$  factors (1.006) is greater than the value of 1.002 required for the Hamilton (1965)  $R$ -factor ratio at a probability level of 99.5%. The preferred chirality was chosen as the one having the lower  $R$  value. Weights for the observed structure factors calculated according to  $w = 1/[\sigma^2(F) + 0.000073F^2]$  for which  $S = 1.48$ ,  $\Delta/\sigma_{\max} = 0.649$ ,  $\Delta\rho_{\max} = 0.845$  and  $\Delta\rho_{\min} = -0.531 \text{ e } \text{Å}^{-3}$ . Atomic coordinates displayed in Table 1 and selected bond distances and bond angles in Table 2.\* Geometrical calculations performed with *XANADU* (Roberts & Sheldrick, 1979) and illustrations drawn with *PLUTO* (Motherwell & Clegg 1978).

**Discussion.** The structure analysis confirms the precise stereochemistry of the cluster complex. The structure of the discrete cluster (Fig. 1) reveals the presence of the *triangulo*-Ru<sub>3</sub> found in many species. The C<sub>12</sub>H<sub>15</sub> ligand remains attached to the Ru<sub>3</sub> face by the same  $\mu_3\text{-}2\eta^1, \eta^3$  interaction found in the

parent hydrido complex (Bruce, Cairns, Cox, Green, Smith & Woodward, 1970; Cox & Woodward, 1971). The same mode of attachment has been observed in Ru<sub>3</sub>Au<sub>3</sub>( $\mu_3\text{-}2\eta^1, \eta^3\text{-C}_{12}\text{H}_{15}$ )(CO)<sub>8</sub>(PPh<sub>3</sub>)<sub>3</sub> (Bruce, Shawkataly & Nicholson, 1984).

The Ru<sub>3</sub> triangle has two short edges [2.775 (1) and 2.777 (1) Å] and one long edge [2.981 (1) Å]; the latter links the two metal atoms Ru(2) and Ru(3) which are  $\eta^1$ -bonded to the C<sub>12</sub> ring and these atoms are also bridged by the hydrido ligand. Ru(3) is also bound to the As atom of the dpam ligand. The analogous separations in Ru<sub>3</sub>( $\mu_3\text{-}2\eta^1, \eta^3\text{-C}_{12}\text{H}_{15}$ )(CO)<sub>9</sub> are 2.779 (4), 2.775 (4) and 2.929 (4) Å (Cox & Woodward, 1971). The longer Ru(2)—Ru(3) edge in the present complex presumably results from the presence of the As atom at Ru(3); this trend in the lengthening of the Ru—Ru bond when bonded to a Group 15 ligand has also been observed in Ru<sub>3</sub>(CO)<sub>12-n</sub>L<sub>n</sub> complexes ( $n = 1, 3$ , and  $L = \text{Group 15 ligand}$ ).

The Ru—As distance 2.431 (2) Å is similar to that in Ru<sub>3</sub>(CO)<sub>11</sub>(AsPh<sub>3</sub>) [2.464 (1) Å] (Bruce, Liddell, Hughes, Skelton & White, 1988), Ru<sub>3</sub>(CO)<sub>9</sub>(AsMe<sub>2</sub>Ph)<sub>3</sub> [av. 2.445 (1) Å] (Bruce, Liddell, Shawkataly, Hughes, Skelton & White, 1988), and also Ru<sub>3</sub>( $\mu_2\text{-dpam}$ )(CO)<sub>10</sub> [av. 2.426 (1) Å] (Teoh, Fun & Shawkataly, 1990).

\* Lists of structure factors, anisotropic thermal parameters, full bond lengths, bond angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53175 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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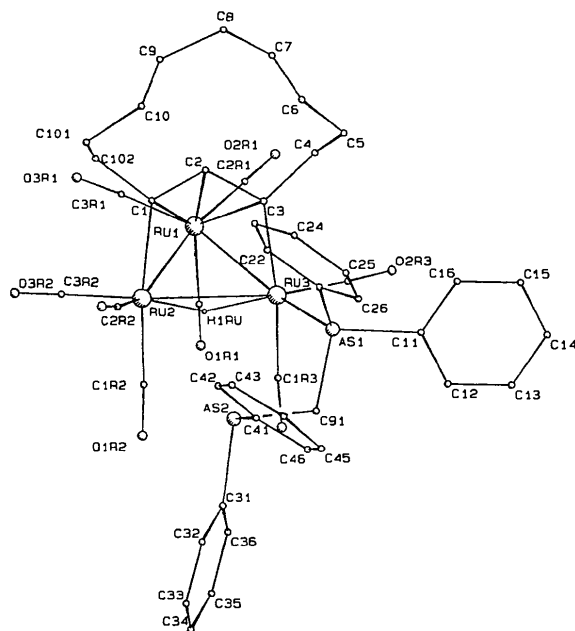


Fig. 1. A perspective view of the molecule.

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