Discussion. The $Sc_{11}Al_2Ge_8$ structure can be considered as a ternary substitution variant of the $Ho_{11}Ge_{10}$ type. The four Sc sites correspond to the Ho sites and the four Ge sites together with the Al site to the Ge sites in $Ho_{11}Ge_{10}$. The projection of half a unit cell of $Sc_{11}Al_2Ge_8$ is shown in Fig. 1. Sets of four Al atoms form a square around the origin. These squares of tightly bonded Al atoms are also found in $YbMo_2Al_4$ (Fornasini & Palenzona, 1976). The interatomic distances of the Al atoms in $Sc_{11}Al_2Ge_8$ are 2·646 Å and in $YbMo_2Al_4$ 2·65 Å, respectively. These distances are 5·5% smaller than the sum of the metallic radii of the Al atoms. Similar short distances are, however, also found in the structures with the $Ho_{11}Ge_{10}$ type and its other substitution variants without Al.

Four ternary compounds which are substitution variants of the $Ho_{11}Ge_{10}$ type have been reported: $Sc_7Cr_{4+x}Si_{10-x}$ $(x = 0·8)$ (Kotur, Bodak & Zavodnik, 1985), both $Sc_7Mn_{4+x}Ge_{10-x}$ $(x = 1·3)$ and $Sc_7Cr_{4+x}Ge_{10-x}$ $(x = 1·2)$ (Kotur, Andrusyak & Zavodnik, 1988) and $Sc_7Re_{4-x}Si_{10+x}$ $(x = 0·65)$ (Zhao, Chabot & Parthé, 1988) for which the site occupations are also given in Table 1. A substitution of the $Ho_{11}Ge_{10}$ structure has been observed on two sites only, that is one $16(n)$ Ho site (marked in Fig. 1 by large shaded circles) and the $8(h)$ Ge site (occupied by Al in $Sc_{11}Al_2Ge_8$). The transition elements substitute essentially on the Ho site; however, Cr and

Mn, to a lesser degree also Re, partially occupy also the $8(h)$ Ge site.

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Structure of $(\mu\text{-}I)_2Os_2(CO)_6I_2$

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Abstract. Hexacarbonyl-1κ^3C,2κ^3C-di-μ-iodo-1κI:-2κI-diiodo-1κI,2κI-diosmium, $[Os_2(CO)_6I_4]$, monoclinic, $C2/m$, $a = 11·082$ (2), $b = 7·740$ (5), $c = 9·711$ (3) Å, $\beta = 107·22°$, $V = 795·6$ Å³, $Z = 2$, $D_x = 4·408$ g cm⁻³, $M_r = 1056·08$, $F(000) = 896$, Mo $K\alpha$, $\lambda = 0·71703$ Å, $\mu = 237$ cm⁻¹, room temperature, $R_f = 0·037$ for 841 reflections. The molecular structure of the title compound consists of two $(CO)_3Os$ units bridged by two iodines. The symmetry around the Os atoms was found to be octahedral.

Introduction. Although there have been reports for the synthesis of $(\mu\text{-}I)_2Os_2(CO)_6I_2$ (Psaro, Ugo, Zanderighi, Besson, Smith & Basset, 1981) by the reac-

tion of $[Os(CO)_3Cl_2]$ with LiI, by the reaction of osmium oxiodide with copper turnings at 403 K under CO pressure for 24 h (Hales & Irving, 1967), and also by dimerization of *cis*-$[Os(CO)_4I_2]$ at 403 K (Moss, Niven & Sutton, 1988), there is no conclusive evidence for the molecular structure of the compound synthesized. Here we report the molecular structure of $(\mu\text{-}I)_2Os_2(CO)_6I_2$. This compound was isolated from the reaction of $(\mu\text{-}H)Os_3(CO)_{11}^-$ with CH_2ClI in the presence of BCl_3.

Experimental. Light yellow, single crystals were obtained from CH_2Cl_2 at 298 K. For X-ray examination and data collection, a suitable crystal of approx-

imate dimensions 0·25 × 0·125 × 0·125 mm was mounted in a glass capillary. Intensity data were collected on a four-circle Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation.

Unit-cell parameters were obtained by least-squares refinement of the angular settings from 25 reflections which lie in a 2θ range of 24–30°. Intensity data were collected in the ω–2θ mode, in the range $4 < 2\theta < 55°$ with the following collected indices: $-14 < h < 14$, $0 < k < 10$, $-12 < l < 12$. Three standard reflections, chosen from reflections used to determine the unit-cell parameters, were measured every three hours, showing 0·7% decay. Data were corrected for Lorentz–polarization factors, intensity decay (correction factors, 0·996 min., 1·00 max.) and absorption (ψ scans, transmission factors, 38·46 min., 99·66 max., 70·82% av.). Averaging yielded 982 reflections of which 841 with $I > 3\sigma(I)$ were used in the structure solution and refinement.

The structure was solved by a combination of direct methods (*MULTAN*11/82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and difference Fourier techniques, and refined by full-matrix least squares. Full-matrix least-squares refinement which minimizes $w(|F_o| - |F_c|)^2$, $w = [\sigma(I)^2 + 0·03(I)^2]^{-1/2}$, was carried out by using anisotropic thermal parameters for all atoms. Final $R_f = 0·037$, $R_{wf} = 0·053$ for 841 reflections, $S = 1·190$, $(\Delta/\sigma)_{max} = 0·01$. The final difference Fourier synthesis was featureless except in the immediate vicinity of Os, $(\Delta\rho)_{max} = 3·40$, $(\Delta\rho)_{min} = -2·39$ e Å^{-3}. Other computers programs used: B. A. Frenz & Associates, Inc. (1982) *SDP*. Atomic scattering factors: for non-H atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV) and for H atoms from Stewart, Davidson & Simpson (1965). All calculations were performed on a PDP11/44 minicomputer.
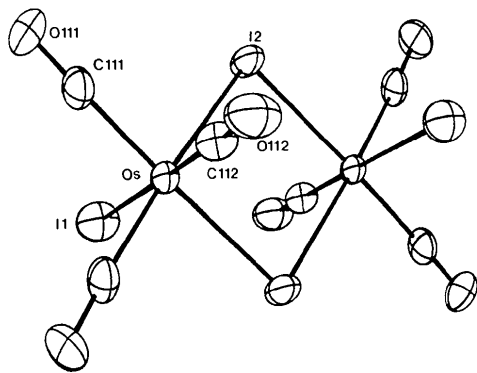

Fig. 1. *ORTEP* drawing of $(\mu\text{-I})_2Os_2(CO)_6I_2$ showing 50% thermal ellipsoid probability for the non-H atoms and atomic labeling scheme.

Table 1. *Positional parameters and their e.s.d.'s for* $(\mu\text{-I})_2Os_2(CO)_6I_2$

	x	y	z	$B(Å^2)$
Os(1)	0·43535 (4)	0·000	0·28207 (4)	1·973 (9)
I(1)	0·67748 (8)	0·000	0·2690 (1)	3·64 (2)
I(2)	0·500	0·2391 (1)	0·500	3·30 (2)
C(111)	0·4055 (7)	0·1816 (16)	0·1433 (9)	2·6 (2)
O(111)	0·3915 (7)	0·3004 (13)	0·0648 (8)	4·2 (2)
C(112)	0·2589 (12)	0·000	0·2854 (14)	2·9 (3)
O(112)	0·1598 (8)	0·000	0·2826 (12)	3·8 (2)

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

Table 2. *Bond distances* (Å) *and angles* (°) *and their e.s.d.'s for* $(\mu\text{-I})_2Os_2(CO)_6I_2$

Os(1)—I(1)	2·723 (1)	Os(1)—C(112)	1·965 (14)
Os(1)—I(2)	2·7411 (8)	C(111)—O(110)	1·174 (14)
Os(1)—C(111)	1·906 (11)	C(112)—O(112)	1·090 (17)
I(1)—Os(1)—I(2)	90·00 (2)	I(2)—Os(1)—C(112)	91·3 (3)
I(1)—Os(1)—C(111)	86·2 (3)	C(111)—Os(1)—C(112)	92·7 (4)
I(1)—Os(1)—C(112)	178·4 (4)	Os(1)—C(111)—O(111)	175·7 (8)
I(2)—Os(1)—C(111)	90·0 (4)	Os(1)—C(112)—O(112)	177·7 (12)

Discussion. The molecular structure of $(\mu\text{-I})_2Os_2(CO)_6I_2$ is shown in Fig. 1. Table 1 lists the final atomic coordinates.* Selected bond distances and bond angles are given in Table 2.

The symmetry of the molecule could be described as two Os(CO)$_3$I units bridged by two iodine ligands. All equatorial ligand atoms (C111, O111, I2) lie in a mirror plane of the molecule. The molecule has crystallographic C_{2h} symmetry, with the two halves of the dimer related by a C_2 axis which passes through the bridging iodine ligands.

Terminal Os—I and bridge Os—μ-I distances are 2·743 (1) and 2·7411 (8) Å, respectively. Previously determined osmium–iodine distances are Os—I 2·772 (3) Å for Os$_3$(CO)$_{12}$I$_2$ (Cook, Smart & Woodward, 1977); Os—μ-I 2·749 (5) Å for [Os$_4$(CO)$_{12}$H$_3$I]$^-$ (Johnson, Lewis, Raithby, Sheldrick, Wong & McPartlin, 1978); Os—I 2·740 (5) Å for [Os$_5$(CO)$_{15}$I]$^-$ (Rivera, Sheldrick & Hursthouse, 1978); Os—μ-I 2·753 (2) and 2·736 (2) Å for Os$_8$(CO)$_{22}$I (Johnson, Lewis, Nelson, Vargas, Braga & McPartlin, 1983). The Os—Os distance in $(\mu\text{-I}_2)Os_2(CO)_6I_2$ is 4·0462 (6) Å, a nonbonded distance.

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Structure Cristalline du Phosphite Acide de Fe^{III}, Fe(HPO₃H)₃

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Abstract. Fe(HPO₃H)₃, $M_r = 298 \cdot 8$, monoclinic, Cc, $a = 13 \cdot 619 \, (4)$, $b = 6 \cdot 422 \, (1)$, $c = 9 \cdot 699 \, (3)$ Å, $\beta = 106 \cdot 27 \, (2)°$, $V = 814 \cdot 3 \, (7)$ Å³, $Z = 4$, $D_m = 2 \cdot 507$ (by picnometry), $D_x = 2 \cdot 438$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71069$ Å, $\mu = 2 \cdot 21$ mm^{-1}, $F(000) = 428$, room temperature. Final $R = 0 \cdot 051$, $wR = 0 \cdot 035$ for 642 independent reflections. Fe(HPO₃H)₃ has been studied by single-crystal X-ray analysis with an automatic diffractometer. The structure is built up of a three-dimensional network of FeO₆ octahedra and HPO₃H^{-} tetrahedra. Three hydrogenphosphite anions are hydrogen bonded and constitute an (HPO₃H)$_3^{3-}$ anion.

Introduction. Cette étude structurale s'inscrit dans le cadre de l'étude générale des phosphites acides réalisée au laboratoire (Larbot, Durand & Cot, 1984; Loukili, Durand, Rafiq & Cot, 1988; Tijani, Durand & Cot, 1988). Une précédente étude nous a permis de décrire la structure du phosphite acide de cuivre divalent Cu(HPO₃H)₂ et de montrer la présence de groupements (HPO₃H)$_4^{4-}$ obtenus par mise en jeu de liaisons hydrogènes entre quatre anions HPO₃H^{-} (Sghyar, Durand, Cot & Rafiq, 1990).

La structure cristalline de Fe(HPO₃H)₃ est un nouvel exemple d'arrangement dans cette famille de sels acides de métaux de transition 3*d*.

Partie expérimentale. Cristaux de Fe(HPO₃H)₃ obtenus par évaporation lente à 363 K d'une solution contenant de l'acide phosphoreux et de l'oxyde ferrique dans un rapport molaire de 4 pour 1. Cristaux précipitant sous forme de bâtonnets parallélépipédiques (0,015 × 0,015 × 0,120 mm). Etudes préliminaires radiocristallographiques en chambre de Weissenberg (symétrie monoclinique C2/c ou Cc). Paramètres de maille cristalline affinés par moindres carrés à partir de 25 réflexions ($10 < \theta < 20°$) optimisées sur diffractomètre Enraf–Nonius CAD-4; mesures d'intensités effectuées au minimum d'absorption avec balayage ω–θ; $0 \leq l \leq 11$, $0 \leq k \leq 7$, $-14 \leq h \leq 14$; $\theta < 25°$; largeur de balayage 1·20°; trois réflexions contrôles (013, 204, 020) mesurées toutes les heures; pas de variation d'intensité supér-