Discussion. Atomic coordinates, bond lengths and angles are given in Tables 1 and 2.* Corresponding bond lengths and angles of the two crystallographically independent molecules are insignificantly different. The molecular structure, illustrated in Fig. 1, shows the compound to be a dimer of the lithiumamide with terminal THF molecules completing the three-coordinated planar Li-atom environment. The Li-bonded N atom has distorted tetrahedral geometry. Bond lengths and angles are comparable to those found in $[(Me_3Si)_2NLi.OEt_2]_2$ (Lappert, Slade, Singh, Atwood, Rogers & Shakir, 1983). The Si_3N_3 ring has a strongly distorted boat conformation [torsion angles Si(1)—N(1)—Si(2)—N(2)—Si(3)—N(3)—Si(1): −36·7 (2), 1·6 (9), 37·6 (9), −29·7 (10), −15·1 (19), 47·6 (17)°; Bucourt & Hainant, 1965]. This change in conformation from the almost planar $(RR'SiNH)_3$ rings (Clegg, Sheldrick & Stalke, 1984) to an approximate boat conformation may be caused by the anionic character of the N atom. The Si—N bond lengths in the ring involving either this N or the diagonally opposite Si are short [mean 1·697 (3) Å], whereas the middle Si—N distances are normal [mean 1·735 (3) Å]. The H-bonded N atoms have virtually planar geometry [mean N—H 0·75 (7) Å, freely refined].

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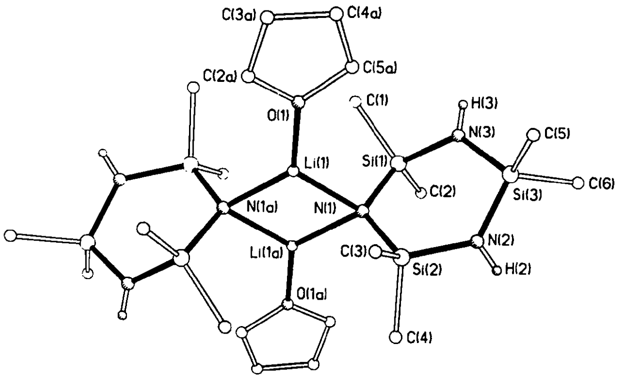

Fig. 1. Molecular structure of the dimer. H atoms of the methyl groups and of the THF molecules are omitted, and the unique atoms plus three symmetry-generated atoms N(1*a*), Li(1*a*) and O(1*a*) are labelled.

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Structure of Decacarbonyl-tetra-μ-hydrido-μ-[methylenebis(diphenylphosphine)-*P,P'*]-*tetrahedro*-tetraruthenium

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Abstract. $[Ru_4(C_{25}H_{22}P_2)(CO)_{10}H_4]$, $M_r = 1072{\cdot}8$, monoclinic, $P2_1/c$, $a = 10{\cdot}887\ (2)$, $b = 19{\cdot}751\ (3)$, $c = 18{\cdot}278\ (2)$ Å, $\beta = 105{\cdot}89\ (1)°$, $V = 3780{\cdot}1\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}89$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 16{\cdot}65$ cm^{-1}, $F(000) = 2088$, $T = 118\ (1)$ K, $R = 0{\cdot}030$ for 5569 observed reflections. The structure consists of a closed tetrahedral metal core involving four hydrido-bridged metal–metal bonds [Ru(1)—Ru(3)

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2·958 (1), Ru(2)—Ru(3) 2·982 (1), Ru(2)—Ru(4) 2·917 (1), Ru(3)—Ru(4) 2·991 (1) Å] and two unsupported metal–metal bonds [Ru(1)—Ru(2) 2·771 (1), Ru(1)—Ru(4) 2·760 (1) Å]. The bis(diphenylphosphino)methane ligand bridges the metal–metal vector Ru(3)—Ru(4).

Introduction. In recent studies of the chemistry of substituted metal clusters involving bridging phosphine ligands, we noticed a different reactivity of $Ru_4H_4(CO)_{10}$(diphos) [diphos = ethylenebis(diphenylphosphine)] and $Ru_4H_4(CO)_{10}$(dppm) [dppm = methylenebis(diphenylphosphine)] (Bergounhou, Bonnet, Fompeyrine, Lavigne, Lugan & Mansilla, 1986). Typically, the latter complex was shown to experience facile oxidative cleavage of a C—P bond to yield $Ru_4(\mu\text{-}H)_3(CO)_{10}[\mu_3\text{-}\eta^2\text{-}(C_6H_5)PCH_2P(C_6H_5)_2]$, a reaction as yet unobserved for $Ru_4H_4(CO)_{10}$(diphos) (Churchill, Lashewycz, Shapley & Richter, 1980). Since a different behaviour might be related to a different structure of the two complexes, we attempted an X-ray structural analysis of $Ru_4H_4(CO)_{10}$(dppm) in order to confirm the model derived from spectroscopic studies in solution (Bergounhou *et al.*, 1986).

Experimental. The title complex was prepared according to our published procedure (Bergounhou *et al.*, 1986) and crystallized from dichloromethane/ethanol. Selected crystal, 0·08 × 0·24 × 0·35 mm, mounted on an Enraf–Nonius CAD-4F diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, NJET cooling device (Bonnet & Askenazy, 1983). Cell parameters from 25 accurately centred reflections in the range $12 < \theta < 15°$. Data measured using ω–2θ scan, scan width 0·42° below $K\alpha_1$ to 0·42° above $K\alpha_2$. Four standard reflections (0,12,$\bar{1}$, 506, 66$\bar{3}$, 4,10,1) measured every 2 h of exposure time showed no decay. Data collected: *h*, *k*, *l* range 12, 22, ±20; $\theta_{max} = 24{\cdot}0°$. 6993 reflections measured, 1424 unobserved [$I < 3\sigma(I)$]. Data corrected for Lorentz, polarization and absorption effects; distances from crystal faces to centre: 0·046 (001, 00$\bar{1}$), 0·119 (100, $\bar{1}$00), 0·108 (011), 0·133 (0$\bar{1}\bar{1}$), 0·118 (01$\bar{1}$), 0·105 mm (0$\bar{1}$1); crystal volume 0·0067 mm³; $\mu = 16{\cdot}65$ cm⁻¹, Gaussian method (Coppens, Leiserowitz & Rabinovitch, 1965); 4 × 6 × 4 grid; min. and max. transmission factors 0·67 and 0·87. Structure solved by direct methods (Main *et al.*, 1980), Fourier syntheses, and refined by full-matrix least squares based on *F*. Function minimized $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = \sigma_c^2(F_o^2) + (p|F_o^2|)^2$; σ_c based on Poisson counting statistics, *p* an empirical constant (here $p = 0{\cdot}02$). Ibers' library of programs, implemented on the Multics computer of the Centre Interuniversitaire de Calcul de Toulouse. Phenyl rings treated as rigid groups (idealized D_{6h} symmetry, C—C = 1·395 Å with C—H = 0·95 Å; isotropic thermal parameters of H atoms taken 1 Å² greater than those of corresponding carbon atoms).

The four hydride ligands were located on a Fourier difference map and refined using fixed isotropic thermal parameters ($B = 3{\cdot}5$ Å²). Model converged with 5569 unique observations for which $F_o^2 > 3\sigma(F_o^2)$, 305 variables, $R = 0{\cdot}030$, $wR = 0{\cdot}035$, isotropic extinction parameter refined = $0{\cdot}93 \times 10^{-8}$. Max. residual electron density 1·0 e Å⁻³. Highest parameter shift Δ/σ is 2·0 [*x* coordinate of the hydride ligand H(2,4)]. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Table 1. *Final positional parameters and isotropic thermal parameters with e.s.d.'s in parentheses*

	x	*y*	*z*	*B* (Å²)
Ru(1)	0·77692 (4)	0·11988 (2)	0·77288 (2)	1·35 (1)
Ru(2)	0·99547 (4)	0·12625 (2)	0·89376 (2)	1·22 (1)
Ru(3)	0·99256 (4)	0·03100 (2)	0·76671 (2)	1·14 (1)
Ru(4)	0·98691 (4)	0·18111 (2)	0·74425 (2)	1·24 (1)
P(1)	1·2038 (1)	0·02506 (6)	0·75798 (7)	1·21 (4)
P(2)	1·1957 (1)	0·18355 (6)	0·73365 (7)	1·24 (4)
C(1)	0·6724 (5)	0·1050 (3)	0·6725 (3)	2·2 (2)
O(1)	0·6071 (4)	0·0953 (2)	0·6129 (2)	3·5 (2)
C(2)	0·6610 (5)	0·0818 (3)	0·8227 (3)	1·8 (2)
O(2)	0·5858 (3)	0·0618 (2)	0·8501 (2)	3·1 (2)
C(3)	0·7257 (5)	0·2114 (3)	0·7745 (3)	2·2 (2)
O(3)	0·6873 (4)	0·2651 (2)	0·7742 (2)	3·2 (2)
C(4)	1·1478 (5)	0·1320 (3)	0·9774 (3)	1·8 (2)
O(4)	1·2286 (4)	0·1370 (2)	1·0322 (2)	3·2 (2)
C(5)	0·9134 (5)	0·2015 (3)	0·9233 (3)	2·1 (2)
O(5)	0·8662 (4)	0·2475 (2)	0·9429 (2)	3·2 (2)
C(6)	0·9086 (5)	0·0643 (3)	0·9416 (3)	1·7 (2)
O(6)	0·8581 (3)	0·0263 (2)	0·9708 (2)	2·3 (2)
C(7)	0·9120 (5)	−0·0108 (2)	0·6727 (3)	1·6 (2)
O(7)	0·8562 (4)	−0·0358 (2)	0·6174 (2)	2·6 (2)
C(8)	0·9987 (5)	−0·0503 (3)	0·8232 (3)	1·7 (2)
O(8)	1·0040 (4)	−0·0977 (2)	0·8593 (2)	2·5 (2)
C(9)	0·8821 (5)	0·1974 (3)	0·6451 (3)	2·1 (2)
O(9)	0·8153 (4)	0·2090 (2)	0·5860 (2)	3·3 (2)
C(10)	0·9800 (5)	0·2713 (3)	0·7706 (3)	2·1 (2)
O(10)	0·9816 (4)	0·3278 (2)	0·7890 (2)	3·5 (2)
C(11)	1·2836 (5)	0·1079 (2)	0·7808 (3)	1·3 (2)
C(21)	1·2312 (3)	−0·0024 (2)	0·6680 (1)	1·34 (8)
C(22)	1·3170 (3)	0·0302 (1)	0·6358 (2)	1·75 (9)
C(23)	1·3372 (3)	0·0058 (2)	0·5685 (2)	2·00 (9)
C(24)	1·2715 (3)	−0·0512 (2)	0·5336 (1)	2·03 (10)
C(25)	1·1856 (3)	−0·0838 (1)	0·5659 (2)	2·18 (10)
C(26)	1·1654 (3)	−0·0594 (2)	0·6331 (2)	1·88 (9)
C(31)	1·3101 (3)	−0·0299 (2)	0·8280 (2)	1·48 (8)
C(32)	1·3125 (3)	−0·0225 (2)	0·9041 (2)	2·26 (10)
C(33)	1·3910 (3)	−0·0638 (2)	0·9591 (1)	2·8 (1)
C(34)	1·4672 (3)	−0·1125 (2)	0·9380 (2)	2·7 (1)
C(35)	1·4648 (3)	−0·1200 (3)	0·8619 (2)	2·7 (1)
C(36)	1·3863 (3)	−0·0786 (2)	0·8069 (1)	2·10 (9)
C(41)	1·2983 (3)	0·2531 (1)	0·7807 (2)	1·69 (9)
C(42)	1·4068 (3)	0·2446 (1)	0·8415 (2)	1·95 (9)
C(43)	1·4808 (3)	0·3004 (2)	0·8725 (2)	2·8 (1)
C(44)	1·4465 (3)	0·3647 (1)	0·8428 (2)	3·2 (1)
C(45)	1·3380 (3)	0·3732 (1)	0·7820 (2)	3·0 (1)
C(46)	1·2639 (3)	0·3174 (2)	0·7509 (2)	2·4 (1)
C(51)	1·2234 (3)	0·1855 (2)	0·6387 (1)	1·44 (8)
C(52)	1·3407 (3)	0·2063 (2)	0·6298 (1)	1·77 (9)
C(53)	1·3622 (2)	0·2048 (2)	0·5581 (2)	2·2 (1)
C(54)	1·2664 (3)	0·1826 (2)	0·4955 (1)	2·4 (1)
C(55)	1·1491 (3)	0·1619 (2)	0·5044 (1)	2·4 (1)
C(56)	1·1276 (2)	0·1633 (2)	0·5760 (2)	1·96 (9)
H(1,3)	0·844 (5)	0·032 (3)	0·781 (3)	3·5
H(3,4)	0·995 (5)	0·097 (3)	0·704 (3)	3·5
H(2,4)	1·091 (5)	0·171 (3)	0·842 (3)	3·5
H(2,3)	1·067 (5)	0·066 (3)	0·852 (3)	3·5

Discussion. Final positional parameters and isotropic thermal parameters are given in Table 1.* Fig. 1 shows a perspective view of the complex. Selected interatomic distances and bond angles are listed in Table 2.

The crystal consists of well separated molecular units of $Ru_4(\mu\text{-}H)_4(CO)_{10}[\mu\text{-}(C_6H_5)_2PCH_2P(C_6H_5)_2]$. The cluster has a closed tetrahedral metal core, as found in the parent complex $Ru_4(\mu\text{-}H)_4(CO)_{12}$ (Wilson, Wu, Love & Bau, 1978). The bridging methylenebis(diphenylphosphine) ligand spans the metal–metal vector Ru(3)–Ru(4); Ru–P vectors are almost coplanar with the cluster face Ru(1)–Ru(3)–Ru(4) [the dihedral angle between the plane defined by P(1), Ru(3) and Ru(4) and the plane defined by Ru(1), Ru(3) and Ru(4) is 176·1 (1)°, and that between the plane defined by P(2), Ru(3) and Ru(4) and the plane defined by Ru(1), Ru(3) and Ru(4) is 176·4 (1)°]. The environment of Ru(3) and Ru(4) is achieved with two terminal CO ligands, while three terminal CO ligands coordinate to Ru(1) and Ru(2), respectively. The Ru–Ru distances fall into two distinct classes. Normal metal–metal separations (average 2·765 Å) are found for Ru(1)–Ru(2) and Ru(1)–Ru(4), while longer metal–metal separations (average 2·962 Å) are found for the four hydrido-bridged bonds Ru(1)–Ru(3), Ru(2)–Ru(4), Ru(2)–Ru(3), Ru(3)–Ru(4), in agreement with related observations on $Ru_4(\mu\text{-}H)_4(CO)_{10}$(diphos) (Churchill *et al.*, 1980). The four hydride ligands were found in reasonable positions in a difference Fourier map and their refinement proved to be satisfactory. All Ru–H bonds fall in the range 1·7–1·9 Å (average 1·77 Å) in agreement with the values determined by

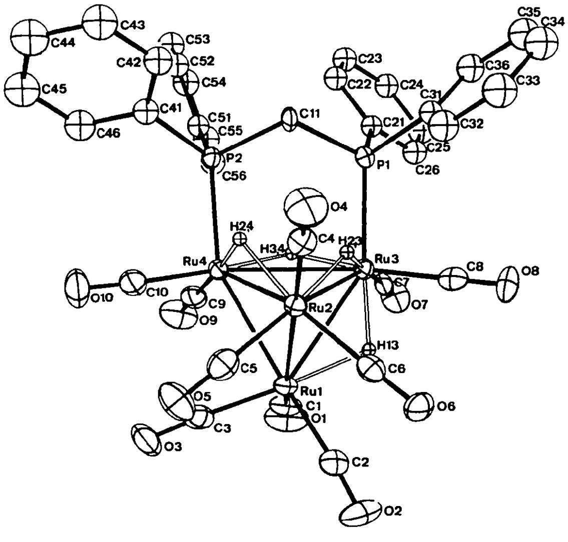

Fig. 1. Perspective view of the cluster complex $Ru_4(\mu\text{-}H)_4(CO)_{10}[\mu\text{-}(C_6H_5)_2PCH_2P(C_6H_5)_2]$ [*ORTEP* (Johnson, 1965) diagram, with thermal ellipsoids shown at 60% probability level; hydride ligands are represented by dummy ellipsoids].

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

Ru–Ru (normal)			
Ru(1)–Ru(2)	2·7712 (7)	Ru(1)–Ru(4)	2·7600 (7)
	Average = 2·766		
Ru–Ru (H-bridged)			
Ru(1)–Ru(3)	2·9581 (6)	Ru(2)–Ru(4)	2·9173 (5)
Ru(2)–Ru(3)	2·9821 (5)	Ru(3)–Ru(4)	2·9913 (5)
	Average = 2·962		
Ru–P		P–C	
Ru(3)–P(1)	2·351 (1)	P(1)–C(11)	1·846 (4)
Ru(4)–P(2)	2·334 (1)	P(2)–C(11)	1·855 (5)
Ru–C		C–O	
Ru(1)–C(1)	1·899 (6)	C(1)–O(1)	1·143 (6)
Ru(1)–C(2)	1·902 (5)	C(2)–O(2)	1·141 (6)
Ru(1)–C(3)	1·895 (6)	C(3)–O(3)	1·138 (6)
Ru(2)–C(4)	1·927 (5)	C(4)–O(4)	1·142 (6)
Ru(2)–C(5)	1·888 (6)	C(5)–O(5)	1·148 (6)
Ru(2)–C(6)	1·901 (5)	C(6)–O(6)	1·144 (6)
Ru(3)–C(7)	1·892 (5)	C(7)–O(7)	1·140 (6)
Ru(3)–C(8)	1·900 (5)	C(8)–O(8)	1·138 (6)
Ru(4)–C(9)	1·886 (6)	C(9)–O(9)	1·149 (6)
Ru(4)–C(10)	1·853 (5)	C(10)–O(10)	1·165 (6)
Ru–H			
Ru(1)–H(1,3)	1·88 (6)	Ru(3)–H(1,3)	1·70 (6)
Ru(2)–H(2,3)	1·72 (6)	Ru(3)–H(2,3)	1·69 (5)
Ru(2)–H(2,4)	1·81 (5)	Ru(4)–H(2,4)	1·85 (5)
Ru(3)–H(3,4)	1·74 (6)	Ru(4)–H(3,4)	1·83 (5)
	Average = 1·77		
Ru–Ru–Ru			
Ru(2)–Ru(1)–Ru(3)	62·63 (2)	Ru(1)–Ru(3)–Ru(2)	55·62 (2)
Ru(2)–Ru(1)–Ru(4)	63·66 (2)	Ru(1)–Ru(3)–Ru(4)	55·28 (1)
Ru(3)–Ru(1)–Ru(4)	62·97 (2)	Ru(2)–Ru(3)–Ru(4)	58·47 (1)
Ru(1)–Ru(2)–Ru(3)	61·75 (2)	Ru(1)–Ru(4)–Ru(2)	58·36 (1)
Ru(1)–Ru(2)–Ru(4)	57·98 (2)	Ru(1)–Ru(4)–Ru(3)	61·75 (2)
Ru(3)–Ru(2)–Ru(4)	60·92 (2)	Ru(2)–Ru(4)–Ru(3)	60·61 (1)
Ru–Ru–CO and Ru–Ru–P			
Ru(2)–Ru(1)–C(1)	159·1 (2)	Ru(1)–Ru(3)–P(1)	146·45 (4)
Ru(3)–Ru(1)–C(1)	98·2 (2)	Ru(2)–Ru(3)–P(1)	106·40 (3)
Ru(4)–Ru(1)–C(1)	101·1 (2)	Ru(4)–Ru(3)–P(1)	91·37 (4)
Ru(2)–Ru(1)–C(2)	100·0 (2)	Ru(1)–Ru(3)–C(7)	96·8 (2)
Ru(3)–Ru(1)–C(2)	114·9 (2)	Ru(2)–Ru(3)–C(7)	152·4 (2)
Ru(4)–Ru(1)–C(2)	163·1 (1)	Ru(4)–Ru(3)–C(7)	108·7 (2)
Ru(2)–Ru(1)–C(3)	97·8 (2)	Ru(1)–Ru(3)–C(8)	112·9 (2)
Ru(3)–Ru(1)–C(3)	143·8 (2)	Ru(2)–Ru(3)–C(8)	96·8 (2)
Ru(4)–Ru(1)–C(3)	81·2 (2)	Ru(4)–Ru(3)–C(8)	155·3 (2)
Ru(1)–Ru(2)–C(4)	179·2 (4)	Ru(1)–Ru(4)–P(2)	154·27 (4)
Ru(3)–Ru(2)–C(4)	118·6 (2)	Ru(2)–Ru(4)–P(2)	107·66 (4)
Ru(4)–Ru(2)–C(4)	121·4 (2)	Ru(3)–Ru(4)–P(2)	92·75 (3)
Ru(1)–Ru(2)–C(5)	83·9 (2)	Ru(1)–Ru(4)–C(9)	86·7 (2)
Ru(3)–Ru(2)–C(5)	144·5 (2)	Ru(2)–Ru(4)–C(9)	145·0 (2)
Ru(4)–Ru(2)–C(5)	94·5 (2)	Ru(3)–Ru(4)–C(9)	106·5 (2)
Ru(1)–Ru(2)–C(6)	84·9 (2)	Ru(1)–Ru(4)–C(10)	106·1 (2)
Ru(3)–Ru(2)–C(6)	92·9 (2)	Ru(2)–Ru(4)–C(10)	96·1 (2)
Ru(4)–Ru(2)–C(6)	141·1 (2)	Ru(3)–Ru(4)–C(10)	156·7 (2)
CO–Ru–CO and CO–Ru–P			
C(1)–Ru(1)–C(2)	95·8 (2)	C(7)–Ru(3)–P(1)	97·8 (2)
C(1)–Ru(1)–C(3)	93·6 (2)	C(8)–Ru(3)–P(1)	96·1 (2)
C(2)–Ru(1)–C(3)	97·7 (2)	C(7)–Ru(3)–C(8)	93·7 (2)
C(4)–Ru(2)–C(5)	95·6 (2)	C(9)–Ru(4)–P(2)	105·3 (2)
C(4)–Ru(2)–C(6)	95·8 (2)	C(10)–Ru(4)–P(2)	96·4 (2)
C(5)–Ru(2)–C(6)	92·4 (2)	C(9)–Ru(4)–C(10)	91·7 (2)
Ru–C–O			
Ru(1)–C(1)–O(1)	178·2 (5)	Ru(2)–C(6)–O(6)	178·8 (4)
Ru(1)–C(2)–O(2)	175·6 (5)	Ru(3)–C(7)–O(7)	175·5 (5)
Ru(1)–C(3)–O(3)	175·8 (5)	Ru(3)–C(8)–O(8)	177·7 (4)
Ru(2)–C(4)–O(4)	172·0 (4)	Ru(4)–C(9)–O(9)	177·2 (5)
Ru(2)–C(5)–O(5)	178·2 (5)	Ru(4)–C(10)–O(10)	176·8 (5)
Ru–P–C and C–P–C			
Ru(3)–P(1)–C(11)	110·4 (1)	C(11)–P(1)–C(21)	109·6 (1)
Ru(3)–P(1)–C(21)	118·6 (1)	C(11)–P(1)–C(31)	101·5 (2)

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* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42912 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

neutron diffraction for closely related cluster complexes (Lugan, Savariault, Lavigne & Bonnet, 1983, and references cited therein).

Noticeably, each hydride ligand is coplanar, within experimental error, with one face of the metal tetrahedron. The orientations of carbonyl ligands clearly reflect the presence of hydrides.

Finally, all structural features of $Ru_4(\mu\text{-}H)_4(CO)_{10}$-($\mu$-dppm) are analogous to those of $Ru_4(\mu\text{-}H)_4(CO)_{10}$-(diphos). Thus, the different chemical behaviour of the two complexes cannot be related to any structural difference.

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Structure of (2,2-Biphenylylene)(1-pyrrolidinecarbodithioato)iodine(III) Chloroform Solvate

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Abstract. $C_{17}H_{16}INS_2.CHCl_3$, $M_r = 544{\cdot}7$, triclinic, $P\bar{1}$, $a = 12{\cdot}082\,(3)$, $b = 9{\cdot}859\,(4)$, $c = 9{\cdot}638\,(3)$ Å, $\alpha = 108{\cdot}46\,(3)$, $\beta = 108{\cdot}29\,(3)$, $\gamma = 89{\cdot}56\,(3)°$, $V = 1028{\cdot}6$ Å^3, $Z = 2$, $D_m = 1{\cdot}765$, $D_x = 1{\cdot}758$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 2{\cdot}15$ mm^{-1}, $F(000) = 536$, $T = 298$ K, final $R = 0{\cdot}060$ for 1875 independent non-zero reflections. The coordination around each I atom is planar tetragonal comprising two normal bonds to C_{ar} atoms [C—I 2·11 (2), 2·13 (1) Å] and two secondary bonds to S atoms [S···I 3·053 (4), 3·162 (4) Å] of different ligands. Two centrosymmetrically related formula units form a dimer, bis-(2,2′-biphenylylene)-μ-bis(1-pyrrolidinecarbodithioato-*S,S′*)-diiodine(III). Different dimers are held together by van der Waals forces.

Introduction. The structural study of the title compound (BPPI hereafter) is the first of a series of systematic structure determinations of diaryliodinanes with an I—S bond, aiming at a better understanding of bonding in this class of hypervalent iodine compounds. The structures of various classes of hypervalent iodine compounds have revealed interesting features (Koser, 1983), yet none of them refers to an I—S bond. The formation of secondary bonds (Alcock, 1972) is a common characteristic of almost all of these structures.

Experimental. Yellow crystals, 0·40 × 0·22 × 0·20 mm, m.p. 414–415 K. D_m measured by flotation in 1,2-dibromobutane/carbon tetrachloride. Computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. Cell parameters and standard deviations by least-squares analysis of measured θ angles of 80 strong reflections with $\theta = 6$–15°. Three-dimensional data, graphite-monochromated Mo $K\alpha$, ω-scan mode. Intensity statistics indicated $P\bar{1}$. Three standard reflections exhibited same percentage linear decrease in intensity (12%). 2117 measured reflections, $\theta = 3$–25°, index range $h = 0 \to 13$, $k = -11 \to 11$, $l = -11 \to 9$, 1875 with $I > 2\sigma(I)$, $R_{int} = 0{\cdot}104$ from merging 586 symmetry-equivalent reflections. Correction for intensity drop, no absorption correction. I atom located by Patterson synthesis, remaining non-H atoms by Fourier synthesis. Full-matrix least squares using *F*, *XRAY*72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). H atoms at calculated positions (C—H 1·1 Å) with

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