Table 2.* The atom-numbering scheme for the molecule is shown in Fig. 1 and Fig. 2 shows part of one infinite column of dimers: both were generated using *ORTEP* (Mallinson & Muir, 1985). Molecular-geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. The mean Hg—Cl bond in $HgCl_2$ is 2·283 (9) Å and the Cl—Hg—Cl angle is 178·6 (4)° (Subramanian & Seff, 1980). The closest non-bonded interactions occur at 3·33 Å (Cl···Cl) and at 3·37 and 3·44 Å (Hg···Cl). The closest Hg···Hg contact is at 4·33 Å.

We thank the SERC for funding towards the purchase of a diffractometer.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51907 (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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Acta Cryst. (1989). C**45**, 1424–1426

Structure of 1,3-μ_2-Acetato-2-(pyridine)-μ_3-sulfido-tris[(diethyl dithiophosphato-*S*,*S'*)-μ_2-sulfido-tungsten(IV)](3 *W*–*W*)

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Abstract. $[W_3S_4(C_2H_3O_2)(C_4H_{10}O_2PS_2)_3(C_5H_5N)]$, $M_r = 1374$, triclinic, $P\bar{1}$, $a = 13{\cdot}939$ (2), $b = 16{\cdot}018$ (3), $c = 9{\cdot}627$ (2) Å, $\alpha = 101{\cdot}08$ (2), $\beta = 105{\cdot}69$ (2), $\gamma = 77{\cdot}64$ (3)°, $V = 2000{\cdot}6$ Å^3, $Z = 2$, $D_x = 2{\cdot}28$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 94{\cdot}5$ cm^{-1}, $F(000) = 1302$, $R = 0{\cdot}050$ for 3896 unique observed reflections $I \geq 8\sigma(I)$. The W atoms are all octahedrally coordinated by two μ_2-S atoms (W—μ_2S av. 2·298 Å), a μ_3-S atom (W—μ_3S av. 2·343 Å) and a chelating $S_2P(OC_2H_5)_2$ terminal ligand [W—S_t (dtp) 2·538 Å]; in addition, W(1) and W(3) are coordinated by a bridging CH_3COO ligand (W—O_b 2·174 Å), while W(2) is coordinated by a C_5H_5N molecule (W—N 2·390 Å). There are three W—W bonds [W(1)—W(2) 2·739, W(1)—W(3) 2·673, W(2)—W(3) 2·750 Å] in the cluster core $[W_3S_4]^{4+}$, which has an incomplete cubane-like structure.

Experimental. Crystals of the title compound were prepared by the method described by Zhan (1989). Crystal dimensions 0·30 × 0·25 × 0·35 mm. Data were collected on a CAD-4 κ-geometry diffractometer using Mo $K\alpha$ radiation at ~296 K. $\omega/2\theta$ scan, scan speed varied from 3 to 5° min^{-1} (in ω), the scan width was $(0{\cdot}60 + 0{\cdot}35\tan\theta)$°. Cell constants were obtained by least-squares analysis of 25 diffraction maxima ($26 \leq 2\theta \leq 27$°). The intensities were corrected for absorption using empirical scan data (maximum and minimum transmission factors 1·07 and, 0·95 respectively) and Lorentz and polarization factors to give a total of 6605 intensities, up to a

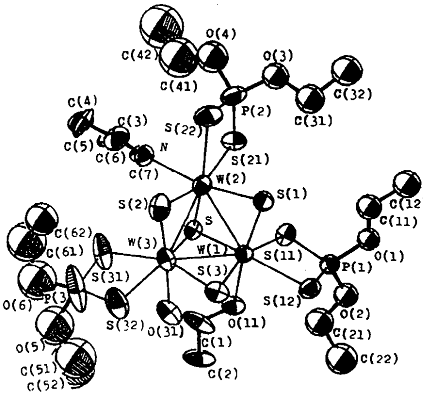

Fig. 1. Drawing of the title compound with thermal ellipsoids.

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{4}{3}\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}(Å^2)
W(1)	0·03379 (8)	0·26630 (6)	0·1104 (1)	2·80 (2)
W(2)	0·21831 (8)	0·17927 (7)	0·0702 (1)	3·14 (2)
W(3)	0·20122 (9)	0·30711 (7)	0·3023 (1)	3·70 (2)
S	0·1564 (5)	0·3265 (4)	0·0568 (6)	3·2 (1)
S(1)	0·0806 (5)	0·1194 (4)	0·0615 (7)	3·5 (1)
S(2)	0·0694 (6)	0·2674 (4)	0·3585 (7)	4·0 (2)
S(3)	0·2997 (6)	0·1720 (5)	0·3111 (8)	4·7 (2)
S(11)	−0·0626 (5)	0·2875 (5)	−0·1448 (7)	4·1 (2)
S(12)	−0·1425 (5)	0·2437 (4)	0·1078 (7)	3·9 (2)
S(21)	0·1862 (6)	0·1594 (5)	−0·2051 (7)	4·4 (2)
S(22)	0·3087 (6)	0·0237 (5)	0·0128 (9)	5·3 (2)
S(31)	0·2679 (7)	0·3414 (6)	0·5781 (8)	6·1 (2)
S(32)	0·3357 (7)	0·3940 (6)	0·3241 (9)	7·3 (2)
P(1)	−0·1841 (5)	0·2624 (5)	−0·0985 (8)	3·7 (2)
P(2)	0·2627 (6)	0·0392 (6)	−0·1954 (9)	5·1 (2)
P(3)	0·3599 (9)	0·4056 (8)	0·539 (1)	10·7 (3)
O(11)	−0·029 (1)	0·400 (1)	0·161 (2)	4·0 (4)
O(31)	0·110 (2)	0·437 (1)	0·327 (2)	5·5 (5)
N	0·372 (2)	0·215 (1)	0·051 (2)	4·1 (5)
C(1)	0·024 (3)	0·455 (2)	0·255 (3)	6·2 (9)
C(2)	−0·038 (3)	0·546 (2)	0·279 (3)	6·3 (9)
C(3)	0·370 (2)	0·269 (2)	−0·040 (3)	4·4 (7)
C(4)	0·456 (2)	0·290 (2)	−0·052 (3)	5·0 (8)
C(5)	0·548 (2)	0·252 (2)	0·030 (4)	5·7 (8)
C(6)	0·551 (2)	0·196 (2)	0·120 (4)	6·1 (9)
C(7)	0·462 (2)	0·175 (2)	0·126 (4)	5·6 (9)
O(1)	−0·230 (1)	0·185 (1)	−0·205 (2)	4·6 (4)*
O(2)	−0·279 (1)	0·336 (1)	−0·132 (2)	5·3 (4)*
O(3)	0·202 (2)	−0·031 (1)	−0·293 (2)	6·5 (5)*
O(4)	0·355 (2)	0·016 (2)	−0·276 (3)	8·7 (7)*
O(5)	0·353 (3)	0·506 (2)	0·612 (4)	14 (1)*
O(6)	0·459 (3)	0·397 (2)	0·627 (4)	13 (1)*
C(11)	−0·170 (2)	0·099 (2)	−0·205 (3)	5·6 (7)*
C(12)	−0·223 (3)	0·041 (2)	−0·331 (4)	6·8 (9)
C(21)	−0·271 (3)	0·428 (3)	−0·076 (4)	8 (1)*
C(22)	−0·374 (4)	0·474 (3)	−0·045 (5)	10 (1)*
C(31)	0·105 (3)	−0·040 (2)	−0·272 (4)	7·1 (9)*
C(32)	0·064 (3)	−0·108 (3)	−0·397 (4)	8 (1)*
C(41)	0·418 (5)	0·077 (4)	−0·285 (7)	15 (2)*
C(42)	0·495 (6)	0·055 (5)	−0·340 (8)	18 (3)*
C(51)	0·259 (5)	0·559 (4)	0·617 (7)	16 (2)*
C(52)	0·242 (7)	0·637 (6)	0·55 (1)	22 (4)*
C(61)	.0·531 (5)	0·347 (4)	0·570 (6)	13 (2)*
C(62)	0·535 (4)	0·258 (3)	0·565 (5)	9 (1)*

Starred atoms were refined isotropically.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

W(1)—W(2)	2·7393 (6)	S(12)—P(1)	1·975 (4)
W(1)—W(3)	2·6728 (6)	S(21)—P(2)	1·997 (4)
W(1)—S	2·343 (2)	S(22)—P(2)	1·979 (5)
W(1)—S(1)	2·295 (3)	S(31)—P(3)	1·953 (5)
W(1)—S(2)	2·301 (3)	S(32)—P(3)	1·986 (5)
W(1)—S(11)	2·509 (2)	P(1)—O(1)	1·584 (7)
W(1)—S(12)	2·551 (3)	P(1)—O(2)	1·583 (4)
W(1)—O(11)	2·151 (6)	P(2)—O(3)	1·56 (1)
W(2)—W(3)	2·7497 (6)	P(2)—O(4)	1·62 (1)
W(2)—S	2·346 (2)	P(3)—O(5)	1·62 (2)
W(2)—S(1)	2·293 (3)	P(3)—O(6)	1·40 (2)
W(2)—S(3)	2·303 (3)	O(11)—C(1)	1·32 (1)
W(2)—S(21)	2·535 (3)	O(31)—C(1)	1·22 (2)
W(2)—S(22)	2·569 (3)	N—C(3)	1·34 (1)
W(2)—N	2·390 (8)	N—C(7)	1·36 (1)
W(3)—S	2·340 (2)	C(1)—C(2)	1·54 (2)
W(3)—S(2)	2·290 (3)	C(3)—C(4)	1·36 (1)
W(3)—S(3)	2·304 (4)	C(4)—C(5)	1·39 (2)
W(3)—S(31)	2·557 (3)	C(5)—C(6)	1·35 (2)
W(3)—S(32)	2·507 (3)	C(6)—C(7)	1·36 (2)
W(3)—O(31)	2·197 (9)	O—C(av.) (in OEt)	1·43 (0·06)*
S(11)—P(1)	1·999 (4)	C—C(av.) (in OEt)	1·44 (0·09)*

Table 2 (*cont.*)

W(2)—W(1)—W(3)	61·06 (2)	S(32)—W(3)—O(31)	81·5 (3)
S—W(1)—S(1)	105·91 (9)	W(1)—S—W(2)	71·48 (7)
S—W(1)—S(2)	108·75 (9)	W(1)—S—W(3)	69·61 (7)
S—W(1)—S(11)	84·31 (8)	W(2)—S—W(3)	71·86 (7)
S—W(1)—S(12)	157·82 (8)	W(1)—S(1)—W(2)	73·21 (7)
S—W(1)—O(11)	82·2 (2)	W(1)—S(2)—W(3)	71·21 (8)
S(1)—W(1)—S(2)	94·0 (1)	W(2)—S(3)—W(3)	73·29 (8)
S(1)—W(1)—S(11)	96·09 (9)	W(1)—S(11)—P(1)	88·4 (1)
S(1)—W(1)—S(12)	89·08 (9)	W(1)—S(12)—P(1)	87·7 (1)
S(1)—W(1)—O(11)	172·9 (2)	W(2)—S(21)—P(2)	87·7 (1)
S(2)—W(1)—S(11)	160·6 (1)	W(2)—S(22)—P(2)	87·1 (1)
S(2)—W(1)—S(12)	85·89 (9)	W(3)—S(31)—P(3)	87·8 (2)
S(2)—W(1)—O(11)	84·3 (2)	W(3)—S(32)—P(3)	88·5 (2)
S(11)—W(1)—S(12)	77·79 (9)	S(11)—P(1)—S(12)	106·1 (2)
S(11)—W(1)—O(11)	83·4 (2)	S(11)—P(1)—O(1)	113·5 (3)
S(12)—W(1)—O(11)	82·9 (2)	S(11)—P(1)—O(2)	112·4 (3)
W(1)—W(2)—W(3)	58·28 (8)	S(12)—P(1)—O(1)	113·2 (3)
S—W(2)—S(1)	105·70 (9)	S(12)—P(1)—O(2)	113·3 (3)
S—W(2)—S(3)	105·4 (1)	O(1)—P(1)—O(2)	98·4 (5)
S—W(2)—S(21)	86·40 (8)	S(21)—P(2)—S(22)	107·4 (2)
S—W(2)—S(22)	160·65 (9)	S(21)—P(2)—O(3)	113·3 (4)
S—W(2)—N	83·7 (2)	S(21)—P(2)—O(4)	114·2 (4)
S(1)—W(2)—S(3)	98·63 (9)	S(22)—P(2)—O(3)	113·8 (4)
S(1)—W(2)—S(21)	92·65 (9)	S(22)—P(2)—O(4)	112·4 (4)
S(1)—W(2)—S(22)	86·3 (1)	O(3)—P(2)—O(4)	95·8 (5)
S(1)—W(2)—N	168·5 (2)	S(31)—P(3)—S(32)	106·5 (3)
S(3)—W(2)—S(21)	160·7 (2)	S(31)—P(3)—O(5)	116·5 (7)
S(3)—W(2)—S(22)	87·3 (2)	S(31)—P(3)—O(6)	117·3 (7)
S(3)—W(2)—N	85·0 (2)	S(32)—P(3)—O(5)	110·9 (6)
S(21)—W(2)—S(22)	77·8 (1)	S(32)—P(3)—O(6)	120·2 (8)
S(21)—W(2)—N	81·1 (2)	O(5)—P(3)—O(6)	84·2 (8)
S(22)—W(2)—N	82·9 (2)	W(1)—O(11)—C(1)	121·6 (8)
W(1)—W(3)—W(2)	60·66 (1)	W(3)—O(31)—C(1)	122·7 (8)
S—W(3)—S(2)	109·3 (1)	W(2)—N—C(3)	120·3 (7)
S—W(3)—S(3)	105·5 (1)	W(2)—N—C(7)	119·8 (8)
S—W(3)—S(31)	158·4 (1)	C(3)—N—C(7)	120 (1)
S—W(3)—S(32)	84·56 (9)	O(11)—C(1)—O(31)	126 (1)
S—W(3)—O(31)	82·5 (2)	O(11)—C(1)—C(2)	113 (1)
S(2)—W(3)—S(3)	94·0 (1)	O(31)—C(1)—C(2)	121 (1)
S(2)—W(3)—S(31)	85·2 (2)	N—C(3)—C(4)	122 (2)
S(2)—W(3)—S(32)	157·6 (1)	C(3)—C(4)—C(5)	119 (1)
S(2)—W(3)—O(31)	83·0 (2)	C(4)—C(5)—C(6)	120 (2)
S(3)—W(3)—S(31)	88·8 (1)	C(5)—C(6)—C(7)	119 (1)
S(3)—W(3)—S(32)	99·3 (1)	N—C(7)—C(6)	121 (1)
S(3)—W(3)—O(31)	172·0 (2)	P—O—C(av.) (in OEt)	120·8 (2·2)*
S(31)—W(3)—S(32)	77·2 (2)	O—C—C(av.) (in OEt)	112 (6)*
S(31)—W(3)—O(31)	83·7 (2)		

* Standard error $\sigma = [(\sum x^2 - n\bar{x}^2)/n]$.

maximum 2θ of 50° ($0 < h < 15$, $-17 < k < 18$, $-11 < l < 10$). Max. $(\sin\theta)/\lambda = 0{\cdot}59$ Å^{-1}, 3896 reflections with $I \geq 8\sigma(I)$ were used in the refinement. Three standard reflections were measured periodically, but no significant variation in intensity was observed.

The structure was solved by direct methods using *MULTAN*11/82 (Main *et al.*, 1982). Seven cluster-core atoms were located in the E map. The remaining non-H atoms were located in the succeeding difference Fourier syntheses (H atoms were placed in geometrically calculated positions with C—H 0·95 Å, but not included in the refinement). The structure was refined by full-matrix least-squares techniques with anisotropic thermal parameters for 25 non-H atoms (307 variables). Final $R = 0{\cdot}050$, $wR = 0{\cdot}071$ and $S = 7{\cdot}35$, the function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = 4F_o^2/\sigma^2(F_o^2)$, $\sigma^2(F_o^2) = [\sigma_o^2(F_o^2) + (0{\cdot}04F_o^2)^2]$ where $\sigma_o^2(F_o^2)$ is the standard deviation based on counting statistics. $(\Delta/\sigma)_{\max} = 0{\cdot}78$. In final difference electron density synthesis max. height less than 2·1 e Å^{-3}. All calculations were performed on a VAX 785 computer using *SDP* (Frenz, 1978), the scattering factors were taken from Cromer & Waber (1974). *ORTEP*II (Johnson, 1976) was used to produce the molecular configuration shown in Fig. 1. The atom coordinates and equivalent isotropic thermal parameters are listed in Table 1, important bond lengths and bond angles are given in Table 2.*

Related literature. A derivative of the title compound, [$(W_3CuS_4)(I)\{S_2P(OC_2H_5)_2\}_3(\mu_2\text{-}OOCCH_3)(C_5H_5N)$], has been reported (Zhan, Zheng, Wu & Lu, 1989).

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* Lists of H-atom coordinates, full bond lengths and angles, structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51891 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Rhodium–Allene Complex, Chloro(1,2-cyclononadiene)bis(triphenylphosphine)rhodium(I)

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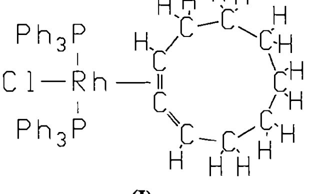
Abstract. [$RhCl(C_9H_{14})(C_{18}H_{15}P)_2$], $M_r = 785{\cdot}2$, monoclinic, $P2_1/n$, $a = 12{\cdot}066\,(5)$, $b = 17{\cdot}280\,(8)$, $c = 18{\cdot}330\,(8)$ Å, $\beta = 97{\cdot}59\,(5)^\circ$, $V = 3788\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}38$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 6{\cdot}27$ cm^{-1}, $F(000) = 1624$, $T = 293$ K, $R = 0{\cdot}059$ for 3287 reflections with $F_o^2 > 3\sigma(F_o^2)$. The Rh atom is bonded to the cyclic allene by one of the two adjacent C═C units. The coordinated C═C group makes a 74 (2)° angle with the Cl—P—P—Rh plane and is lengthened to 1·45 (4) Å while the other C═C bond is 1·26 (1) Å. The C═C═C bond angle is 146 (2)°, and the Rh—C distances are 2·15 (2) and 2·041 (9) Å where the shorter distance is to the central C of the allene unit.

Experimental. Title compound (I) obtained by the reaction of [$RhCl(PPh_3)_3$] with 1,2-cyclononadiene in benzene. Data crystal obtained by slow diffusion of *n*-hexane into a benzene solution. Orange crystal (0·2 × 0·2 × 0·3 mm) mounted under argon in a capillary tube. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using θ–2θ scans of 4–16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $28 < 2\theta < 32^\circ$. Absorption correction based on ψ scans varied from 0·94 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·59 Å^{-1}, $0 < h < 14$, $0 < k < 20$, $-21 < l < 21$. Three standard reflections ($4,\bar{4},\overline{12}$ $\bar{4},\bar{6},10$, $2,2,12$) indicated no crystal decomposition over 64 h



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