

Antolini, Macrotrigiano, Menabue & Pellacani, 1983; Solans, Ruiz-Ramírez, Martínez, Gasque & Moreno-Esparza, 1992) though longer Cu—N(ar) distances of up to 2.145 (2) Å have been reported (Nardin, Randaccio, Bonomo & Rizzarelli, 1980).

The Cu—O(1) (carboxylate) and Cu—N (amino-acidato) bond lengths in (I) and (II) are slightly longer than those observed in related complexes (Solans, Ruiz-Ramírez, Martínez, Gasque & Briansó, 1988; Antolini, Macrotrigiano, Menabue & Pellacani, 1983).

The Cu—O(W1) (aqua) apical bond length alters from 2.209 (11) in (I) to 2.458 (6) Å in (II). Values of 2.24 (1) and 2.368 (5) Å are reported for aqua(1,10-phenanthroline)(L-phenylalaninato)copper(II) nitrate monohydrate (Solans, Ruiz-Ramírez, Martínez, Gasque & Briansó, 1988) and aqua(aspartato)-(2,2'-bipyridine)copper(II) trihydrate (Antolini, Macrotrigiano, Menabue & Pellacani, 1983), respectively.

The N(5)—Cu—O(1) bond angle of 83.5 (4)° in (I) and the N(4)—Cu—O(1) angle of 86.1 (4)° in (II) correlate with the mean metal–ligand bond lengths in (I) and (II) (2.055 and 2.014 Å, respectively). This correlation is based on the constancy of the O(1)···N(5) and O(1)···N(4) distances (average 2.66 Å). The mean donor–metal distance increases linearly from 2.0 to 2.5 Å as the interionic angle decreases from 84 to 64° (Freeman, 1967).

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## Structural Characterization of $W^4-WCl_4(PMePh_2)_4$

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**Abstract.** Bis[dichlorobis(methyldiphenylphosphine-P)tungsten]( $W^4-W$ ) benzene solvate,  $[W_2Cl_4(C_{13}H_{13}P)_4]$ ,  $M_r = 1388.64$ , orthorhombic, *Pbca*,  $a = 12.2783$  (8),  $b = 21.5387$  (6),  $c = 41.9626$  (2) Å,  $V = 11097$  (8) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.664$  g cm<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71073$  Å,  $\mu = 45.827$  cm<sup>-1</sup>,  $F(000) = 5456$ ,  $T = 173$  K,  $R = 0.0435$  for 4359 unique observed reflec-

tions. The molecule is dinuclear with a  $W^4-W$  distance of 2.2728 (7) Å. The W—Cl distances range from 2.373 (4) to 2.398 (4) Å and the W—P distances from 2.529 (4) to 2.548 (4) Å. The angles between the *trans* Cl ligands are 139.9 (1) and 137.3 (1)° for Cl(1)—W(1)—Cl(2) and Cl(3)—W(2)—Cl(4), respectively. The angles between the *trans* phosphine ligands are 158.2 (1) and 156.0 (1)° for P(1)—W(1)—P(2) and P(3)—W(2)—P(4), respectively.

**Introduction.** While quadruply bonded dimolybdenum compounds have been extensively studied, the

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analogous ditungsten chemistry is more limited (Cotton & Walton, 1993). Owing to the presence of bulky, labile phosphine ligands, the compound W<sup>4</sup>-WCl<sub>4</sub>(PMePh<sub>2</sub>)<sub>4</sub> is an excellent starting material in the synthesis of other W<sub>2</sub>Cl<sub>4</sub>L<sub>4</sub> and W<sub>2</sub>Cl<sub>4</sub>(L-L)<sub>2</sub> complexes, where L = monodentate and L-L = bidentate phosphine ligands (Schrock, Sturgeoﬀ & Sharp, 1983). During the investigation of quadruply bonded ditungsten compounds, crystals of the title complex were obtained. Since there are only two other reported crystal structures of W<sub>2</sub>Cl<sub>4</sub>L<sub>4</sub> complexes, those of W<sup>4</sup>-WCl<sub>4</sub>(PMe<sub>3</sub>)<sub>4</sub> and W<sup>4</sup>-WCl<sub>4</sub>(PBu<sub>3</sub>)<sub>4</sub> (Cotton, Extine, Felthouse, Kolthammer & Lay, 1983; Cotton, Jennings, Price & Vidyasagar, 1990), the crystal structure of W<sup>4</sup>-WCl<sub>4</sub>(PMePh<sub>2</sub>)<sub>4</sub> was investigated and is reported here.

**Experimental.** The compound was prepared essentially by the method described in the literature except that NaBEt<sub>3</sub>H instead of Na/Hg was used as the reducing agent (Schrock, Sturgeoﬀ & Sharp, 1983). X-ray-quality crystals were obtained by layering a THF solution of W<sup>4</sup>-WCl<sub>4</sub>(PMePh<sub>2</sub>)<sub>4</sub> with a benzene/hexanes mixture (2:8) in glass tubes sealed under argon. The quality of the crystals was confirmed by polarized-light microscopy. A green irregularly shaped crystal, 0.20 × 0.20 × 0.10 mm, was mounted on a quartz fiber with silicon grease. Unit-cell dimensions were determined from least-squares analysis of 25 reflections with 16.68 < 2θ < 29.24°. Laue class and the unit-cell dimensions were confirmed with axial photographs. Intensity data were collected to maximum (sin θ)/λ = 0.583 Å<sup>-1</sup> (h = 0 → 13, k = 0 → 23, l = 0 → 45) using the 2θ-ω technique on a CAD-4S diffractometer with graphite-monochromated Mo Kα radiation. Three standard reflections (8,0,12, 396, 6,1,16) measured every hour decreased 0.3% over 159.5 h of data collection and no decay correction was made. Data were corrected for Lorentz and polarization effects, as well as for absorption based on ψ scans using the empirical method of North, Phillips & Mathews (1968); T<sub>min</sub> = 0.8010, T<sub>max</sub> = 0.9982. 7992 unique reflections were measured and 4359 with F<sub>o</sub><sup>2</sup> > 3σ(F<sub>o</sub><sup>2</sup>) were considered observed.

Positions of W, Cl and P atoms were determined by direct methods using SHELXS86 (Sheldrick, 1986) and subjected to alternating full-matrix least-squares refinements and difference Fourier maps employing the Enraf-Nonius (1979) *Structure Determination Package* to reveal the rest of the molecule. wR = [Σw(|F<sub>o</sub> - F<sub>c</sub>||<sup>2</sup>)/Σw|F<sub>o</sub>|<sup>2</sup>]<sup>1/2</sup> was minimized, where w = 1/σ<sup>2</sup>(|F<sub>o</sub>|). All non-H atoms were refined anisotropically. The phenyl groups were refined as rigid bodies (Sheldrick, 1976), geometrically idealized as hexagons with C—C = 1.395 and C—H = 0.98 Å.

However, each C atom was permitted to have a freely varying anisotropic displacement tensor. All phenyl H atoms were required to have the same isotropic thermal parameter. The methyl C atoms in the coordinated PPh<sub>2</sub>Me ligand were assigned H atoms at idealized positions. All H atoms were then refined using a riding model and with the same thermal parameter. A total of 321 variables were used with the final R = 0.0435, wR = 0.0436 and S = 1.494. At convergence, (Δ/σ)<sub>max</sub> = 0.001; (Δρ)<sub>max</sub> = 1.309 and (Δρ)<sub>min</sub> = 0.565 e Å<sup>-3</sup> on the final difference Fourier map. Atomic scattering factors, including anomalous-dispersion corrections, were taken from *International Tables for X-ray Crystallography* (1974, Vol IV).

**Discussion.** The positional and equivalent isotropic displacement parameters are given in Table 1. The structure is depicted in Fig. 1 where the atom-numbering scheme is also defined. Selected important interatomic bond distances and angles are given in Table 2.\*

The presence of the bulky PPh<sub>2</sub>Me phosphine ligands makes W<sup>4</sup>-WCl<sub>4</sub>(PMePh<sub>2</sub>)<sub>4</sub> an excellent starting material in the preparation of other W<sup>4</sup>-W complexes with monodentate and bidentate phosphine ligands *via* substitution reactions. While there are several known compounds with bidentate phosphine ligands, the number of W<sup>4</sup>-W complexes containing monodentate phosphines that have been structurally characterized is limited (Cotton & Walton, 1993). W<sup>4</sup>-WCl<sub>4</sub>(PMe<sub>3</sub>)<sub>4</sub>, reported in 1981, and W<sup>4</sup>-WCl<sub>4</sub>(PBu<sub>3</sub>)<sub>4</sub> are the only other structurally characterized complexes of the type W<sub>2</sub>Cl<sub>4</sub>L<sub>4</sub> (Cotton, Extine, Felthouse, Kolthammer & Lay, 1983; Cotton, Jennings, Price & Vidyasagar, 1990).

The W<sup>4</sup>-W distance in W<sup>4</sup>-WCl<sub>4</sub>(PMePh<sub>2</sub>)<sub>4</sub> is 2.2728 (7) Å, slightly longer than the distances of 2.262 (1) and 2.267 (1) Å observed in W<sup>4</sup>-WCl<sub>4</sub>(PMe<sub>3</sub>)<sub>4</sub> and W<sup>4</sup>-WCl<sub>4</sub>(PBu<sub>3</sub>)<sub>4</sub>, respectively. The average W—Cl bond distances for all of these compounds are in the range 2.39–2.40 Å. The W—P bond distance for W<sup>4</sup>-WCl<sub>4</sub>(PMePh<sub>2</sub>)<sub>4</sub> is 2.54 Å. This distance is only 0.03 Å longer than in W<sup>4</sup>-WCl<sub>4</sub>(PMe<sub>3</sub>)<sub>4</sub> and 0.01 Å longer than in W<sup>4</sup>-WCl<sub>4</sub>(PBu<sub>3</sub>)<sub>4</sub>. For the complexes W<sup>4</sup>-WCl<sub>4</sub>(PMePh<sub>2</sub>)<sub>4</sub>, W<sup>4</sup>-WCl<sub>4</sub>(PMe<sub>3</sub>)<sub>4</sub> and W<sup>4</sup>-WCl<sub>4</sub>(PBu<sub>3</sub>)<sub>4</sub>, the average W—W—Cl angles are 110.7, 111.6 and 108.8° and the W—W—P angles are 101.3, 101.1 and 103.0°, respectively. Only small changes in

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and complete bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55861 (41 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1033]

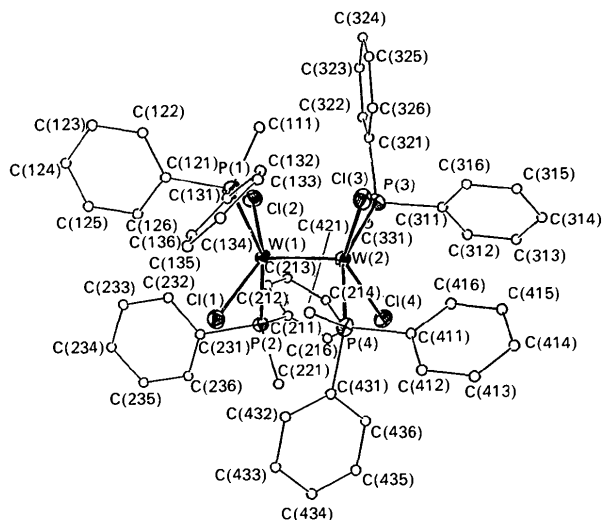
Table 1. Positional and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for  $W^4\text{-WCl}_4(\text{PMePh}_2)_4$ 

$$B_{\text{eq}} = (1/3)[a^2a^{*2}B_{11} + b^2b^{*2}B_{22} + c^2c^{*2}B_{33} + 2ab(\cos\gamma)a^*b^*B_{12} + 2ac(\cos\beta)a^*c^*B_{13} + 2bc(\cos\alpha)b^*c^*B_{23}]$$

	x	y	z	$B_{\text{eq}}$
W(1)	0.06386 (5)	0.15662 (3)	0.08365 (1)	1.27 (1)
W(2)	0.13009 (5)	0.19408 (3)	0.13046 (1)	1.35 (1)
Cl(1)	0.1792 (3)	0.0765 (2)	0.06295 (8)	1.86 (8)
Cl(2)	-0.0999 (3)	0.2104 (2)	0.07051 (9)	1.97 (8)
Cl(3)	0.0590 (3)	0.1384 (2)	0.17540 (8)	2.04 (8)
Cl(4)	0.2572 (3)	0.2751 (2)	0.12127 (8)	1.91 (8)
P(1)	-0.0641 (3)	0.0733 (2)	0.10410 (9)	1.56 (8)
P(2)	0.1678 (3)	0.2225 (2)	0.04336 (9)	1.50 (8)
P(3)	0.0032 (3)	0.2855 (2)	0.14079 (9)	1.96 (9)
P(4)	0.2964 (3)	0.1267 (2)	0.13876 (9)	1.64 (8)
C(1)	-0.259 (1)	0.0467 (5)	0.2527 (2)	4.0 (5)
C(2)	-0.357 (1)	0.0197 (5)	0.2627 (2)	3.5 (5)
C(3)	-0.410 (1)	-0.0230 (5)	0.2431 (2)	3.9 (5)
C(4)	-0.365 (1)	-0.0387 (5)	0.2135 (2)	3.9 (5)
C(5)	-0.268 (1)	-0.0117 (5)	0.2036 (2)	4.4 (5)
C(6)	-0.215 (1)	0.0310 (5)	0.2232 (2)	4.2 (5)
C(111)	-0.175 (1)	0.0994 (8)	0.1299 (4)	2.8 (4)
C(121)	-0.1373 (8)	0.0350 (5)	0.0712 (2)	2.4 (4)
C(122)	-0.2297 (8)	-0.0007 (5)	0.0774 (2)	2.3 (4)
C(123)	-0.2804 (8)	-0.0330 (5)	0.0527 (2)	3.0 (4)
C(124)	-0.2386 (8)	-0.0296 (5)	0.0218 (2)	2.3 (4)
C(125)	-0.1461 (8)	0.0062 (5)	0.0156 (2)	3.0 (4)
C(126)	-0.0955 (8)	0.0385 (5)	0.0403 (2)	2.6 (4)
C(131)	-0.0075 (7)	0.0048 (4)	0.1238 (2)	2.0 (3)
C(132)	-0.0213 (7)	-0.0058 (4)	0.1563 (2)	2.1 (4)
C(133)	0.0247 (7)	-0.0582 (4)	0.1705 (2)	2.5 (4)
C(134)	0.0843 (7)	-0.1001 (4)	0.1521 (2)	2.5 (4)
C(135)	0.0980 (7)	-0.0896 (4)	0.1195 (2)	2.2 (4)
C(136)	0.0521 (7)	-0.0371 (4)	0.1053 (2)	2.1 (4)
C(211)	0.1406 (7)	0.3049 (4)	0.0382 (2)	1.8 (3)
C(212)	0.0444 (7)	0.3230 (4)	0.0232 (2)	1.9 (3)
C(213)	0.0204 (7)	0.3859 (4)	0.0195 (2)	2.8 (4)
C(214)	0.0925 (7)	0.4307 (4)	0.0309 (2)	3.1 (4)
C(215)	0.1888 (7)	0.4126 (4)	0.0460 (2)	3.1 (4)
C(216)	0.2128 (7)	0.3497 (4)	0.0497 (2)	2.1 (4)
C(221)	0.315 (1)	0.2166 (6)	0.0454 (3)	1.4 (3)
C(231)	0.1382 (6)	0.1941 (5)	0.0027 (2)	1.9 (3)
C(232)	0.0409 (6)	0.1627 (5)	-0.0032 (2)	2.2 (4)
C(233)	0.0169 (6)	0.1422 (5)	-0.0340 (2)	2.3 (4)
C(234)	0.0903 (6)	0.1530 (5)	-0.0588 (2)	2.4 (4)
C(235)	0.1877 (6)	0.1844 (5)	-0.0528 (2)	2.8 (4)
C(236)	0.2116 (6)	0.2050 (5)	-0.0220 (2)	2.4 (4)
C(311)	0.063 (1)	0.3236 (5)	0.1761 (2)	2.8 (4)
C(312)	0.123 (1)	0.3784 (5)	0.1726 (2)	3.7 (4)
C(313)	0.171 (1)	0.4061 (5)	0.1991 (2)	3.9 (5)
C(314)	0.160 (1)	0.3788 (5)	0.2291 (2)	3.9 (5)
C(315)	0.101 (1)	0.3239 (5)	0.2326 (2)	5.3 (6)
C(316)	0.053 (1)	0.2963 (5)	0.2061 (2)	4.8 (5)
C(321)	-0.1407 (9)	0.2799 (5)	0.1520 (2)	3.4 (4)
C(322)	-0.2174 (9)	0.3213 (5)	0.1397 (2)	3.6 (4)
C(323)	-0.3256 (9)	0.3183 (5)	0.1499 (2)	4.7 (5)
C(324)	-0.3570 (9)	0.2739 (5)	0.1723 (2)	4.8 (6)
C(325)	-0.2803 (9)	0.2324 (5)	0.1845 (2)	4.2 (5)
C(326)	-0.1721 (9)	0.2354 (5)	0.1743 (2)	3.3 (4)
C(331)	0.009 (1)	0.3469 (8)	0.1102 (4)	3.0 (4)
C(411)	0.3610 (7)	0.1475 (4)	0.1768 (2)	1.6 (3)
C(412)	0.4520 (7)	0.1135 (4)	0.1864 (2)	3.5 (4)
C(413)	0.5029 (7)	0.1269 (4)	0.2153 (2)	3.6 (4)
C(414)	0.4627 (7)	0.1744 (4)	0.2346 (2)	3.9 (5)
C(415)	0.3717 (7)	0.2084 (4)	0.2250 (2)	3.8 (5)
C(416)	0.3208 (7)	0.1949 (4)	0.1961 (2)	3.1 (4)
C(421)	0.270 (1)	0.0441 (7)	0.1438 (3)	2.7 (4)
C(431)	0.4142 (7)	0.1309 (4)	0.1123 (2)	1.9 (3)
C(432)	0.4356 (7)	0.0839 (4)	0.0902 (2)	2.7 (4)
C(433)	0.5259 (7)	0.0883 (4)	0.0701 (2)	3.0 (4)
C(434)	0.5948 (7)	0.1397 (4)	0.0721 (2)	3.3 (4)
C(435)	0.5734 (7)	0.1867 (4)	0.0941 (2)	3.3 (4)
C(436)	0.4831 (7)	0.1822 (4)	0.1142 (2)	2.2 (3)

Table 2. Selected interatomic bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) for  $W^4\text{-WCl}_4(\text{PMePh}_2)_4$ 

W(1)—W(2)	2.2728 (7)	P(1)—C(131)	1.828 (9)
W(1)—Cl(1)	2.394 (4)	P(2)—C(211)	1.818 (9)
W(1)—Cl(2)	2.384 (4)	P(2)—C(221)	1.81 (1)
W(1)—P(1)	2.534 (4)	P(2)—C(231)	1.846 (9)
W(1)—P(2)	2.548 (4)	P(3)—C(311)	1.85 (1)
W(2)—Cl(3)	2.398 (4)	P(3)—C(321)	1.83 (1)
W(2)—Cl(4)	2.373 (4)	P(3)—C(331)	1.84 (2)
W(2)—P(3)	2.548 (4)	P(4)—C(411)	1.84 (1)
W(2)—P(4)	2.529 (4)	P(4)—C(421)	1.82 (2)
P(1)—C(111)	1.82 (2)	P(4)—C(431)	1.83 (1)
P(1)—C(121)	1.841 (9)		
W(2)—W(1)—Cl(1)	110.96 (9)	Cl(3)—W(2)—P(4)	84.2 (1)
W(2)—W(1)—Cl(2)	109.19 (9)	Cl(4)—W(2)—P(3)	82.0 (1)
W(2)—W(1)—P(1)	100.40 (9)	Cl(4)—W(2)—P(4)	85.1 (1)
W(2)—W(1)—P(2)	101.31 (9)	P(3)—W(2)—P(4)	156.0 (1)
Cl(1)—W(1)—Cl(2)	139.9 (1)	Cl(111)—P(1)—C(121)	102.7 (6)
Cl(1)—W(1)—P(1)	88.8 (1)	Cl(111)—P(1)—C(131)	105.3 (6)
Cl(1)—W(1)—P(2)	82.2 (1)	Cl(121)—P(1)—C(131)	99.3 (4)
Cl(2)—W(1)—P(1)	84.3 (1)	Cl(211)—P(2)—C(221)	104.9 (5)
Cl(2)—W(1)—P(2)	89.9 (1)	Cl(211)—P(2)—C(231)	100.2 (5)
P(1)—W(1)—P(2)	158.2 (1)	Cl(221)—P(2)—C(231)	102.5 (5)
W(1)—W(2)—Cl(3)	111.80 (9)	Cl(311)—P(3)—C(321)	102.2 (5)
W(1)—W(2)—Cl(4)	110.85 (8)	Cl(311)—P(3)—C(331)	103.0 (6)
W(1)—W(2)—P(3)	101.70 (9)	Cl(321)—P(3)—C(331)	105.2 (6)
W(1)—W(2)—P(4)	101.75 (9)	Cl(411)—P(4)—C(421)	102.3 (6)
Cl(3)—W(2)—Cl(4)	137.3 (1)	Cl(411)—P(4)—C(431)	100.1 (4)
Cl(3)—W(2)—P(3)	91.7 (1)	Cl(421)—P(4)—C(431)	105.0 (6)

Fig. 1. An ORTEP (Johnson, 1965) drawing of the  $W^4\text{-WCl}_4(\text{PMePh}_2)_4$  molecule. W, P and Cl atoms are represented by displacement ellipsoids at the 50% level. The C atoms are drawn with arbitrary radii and the H atoms are omitted for clarity.

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the structures of  $W_2Cl_4L_4$  complexes are observed when changing from a small monodentate phosphine such as  $\text{PMe}_3$  to bulkier ones, such as  $\text{PPh}_2\text{Me}$  or  $\text{PBU}_3$ , as expected from the crystal structures of  $\text{Mo}_2\text{Cl}_4L_4$  complexes (Cotton, Extine, Felthouse, Kolthammer & Lay, 1981; Cotton, Daniels, Powell, Kahaian, Smith & Vogel, 1988).

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## Structure of 2-Methyl-5,6,7-triphenyl-6,7-dihydropyrazolo[2,3-*a*]pyrimidine

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**Abstract.** C<sub>25</sub>H<sub>21</sub>N<sub>3</sub>, *M<sub>r</sub>* = 363.46, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 9.245 (2), *b* = 23.502 (5), *c* = 9.340 (2) Å, β = 103.50 (3)°, *V* = 1973.3 (2) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.220 (2) g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.068 cm<sup>-1</sup>, *F*(000) = 768, *T* = 292 K, *R* = 0.091 for 1442 unique observed reflections. The dihydropyrimidine ring adopts a distorted sofa conformation. The aryl substituents on the saturated C atoms have an axial orientation.

**Introduction.** Dihydro derivatives of pyrazolo[1,5-*a*]pyrimidine have high physiological activity, the most important being cardiovascular activity (Tsuda, Mishina, Obata, Inui & Nakamura, 1986). Though chemical and physiological characteristics of these compounds are directly defined by the conformation of their molecules, relevant data on the spatial structure of the dihydropyrazolo[2,3-*a*]pyrimidines are not available. The present paper is a continuation of our investigation of the molecular and crystal structures of dihydropyrazolopyrimidines containing a bridgehead nitrogen (Orlov, Desenko, Potekhin & Struchkov, 1988) and is devoted to an X-ray diffraction study of 2-methyl-5,6,7-triphenyl-6,7-dihydropyrazolo[2,3-*a*]pyrimidine (I).

**Experimental.** The title compound was prepared by the method described by Orlov, Quiroga, Kolos & Desenko, (1988). Crystals of (I) as pale-yellow plates of approximate dimensions 0.10 × 0.15 × 0.20 mm,

suitable for an X-ray study, were grown from a 2-propanol solution by slow evaporation of the solvent. A Siemens P3/PC diffractometer with graphite-monochromated Mo *K*α radiation was used. The unit-cell parameters were determined by least-squares fit of setting angles of 9 automatically centred reflections (24 < θ < 26°). The θ/2θ scan technique with a variable scan speed (2.0 to 30.0° min<sup>-1</sup>) was used. Two check reflections were monitored after every 50 measurements and they showed no systematic variation in intensity. 2775 reflections up to 2θ = 56.0° were measured including the check and equivalent reflections, with 0 < *h* < 12, 0 < *k* < 26, -11 < *l* < 11. Of 2640 unique reflections (*R*<sub>int</sub> = 0.023), 2467 reflections had *I* > 2σ(*I*). Only *Lp* corrections were applied.

The structure was solved by direct methods using the *SHELXTL-Plus* (Siemens, 1990) programs. After non-H atoms were refined anisotropically, positions of all H atoms were calculated but were not refined further. The function minimized was Σ*w*(*F<sub>o</sub>* - *F<sub>c</sub>*)<sup>2</sup>, where *w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*) + 0.0003*F<sub>o</sub>*<sup>2</sup>]. Four strong reflections with (*F<sub>o</sub>* - *F<sub>c</sub>*)/σ > 4.0 were excluded from the final refinement cycles, giving 1442 reflections with *F<sub>o</sub>* > 5σ(*F*). The final cycle of the full-matrix least-squares refinement gave *R* = 0.091, *wR* = 0.087 and *S* = 2.56 [*S* = (n - m)<sup>-1</sup>Σ(*F<sub>o</sub>*<sup>2</sup> - *F<sub>c</sub>*<sup>2</sup>)/σ<sup>2</sup>, n = 1442, m = 253]. The high *R* value is due to the low quality of the crystal. For all parameters the final shift/σ ratio was smaller than 0.061, the highest peak in the final