

The Crystal Structure of Mo_4P_3

STIG RUNDQVIST

Institute of Chemistry, University of Uppsala, Uppsala, Sweden

The crystal structure of Mo_4P_3 has been determined by X-ray single-crystal methods. The space group is *Pnma* and the unit cell dimensions are $a = 12.428 \text{ \AA}$; $b = 3.158 \text{ \AA}$; $c = 20.440 \text{ \AA}$. The cell contains 32 molybdenum and 24 phosphorus atoms situated in 4(c) positions. The structure can be described as a complex packing of triangular prisms of molybdenum atoms enclosing central phosphorus atoms. The structural relationships between Mo_4P_3 and some other transition metal phosphides are discussed.

In a previous paper on molybdenum phosphides,¹ the occurrence of a phase with the approximate composition $\text{MoP}_{0.75}$ was mentioned. This observation has recently been corroborated by Gingerich.² Further work has now led to the elucidation of the crystal structure of this phase, and the chemical formula Mo_4P_3 has been established.

EXPERIMENTAL

Phosphide samples were prepared as described previously.¹ In order to grow single crystals, compacted powder samples of Mo_4P_3 were heated in an argon-filled arc furnace. It was possible to melt the samples without excessive phosphorus losses, and on cooling, needle-shaped crystals of Mo_4P_3 were formed. X-Ray powder photographs of the arc-melted material showed diffraction lines from Mo_4P_3 and Mo_3P , but some additional, fairly strong lines indicated the occurrence of a further molybdenum phosphide. Mo-P samples of various compositions were also heated in evacuated and sealed tubes of transparent silica at temperatures up to 1350°C and quenched. The powder photographs of these samples showed only lines from Mo_3P , Mo_4P_3 , and MoP, which indicates that the new compound is formed at temperatures higher than 1350°C . This compound was never obtained as a single phase, and its composition is therefore unknown at present.

The unit cell dimensions of Mo_4P_3 were determined from powder photographs recorded in a Guinier-type focussing camera with $\text{CrK}\alpha_1$ radiation. Silicon ($a = 5.4305 \text{ \AA}$) was used as the internal calibration standard. The estimated accuracy of the unit cell dimensions is higher than 0.04 %. No variations of cell dimensions with composition were observed.

In the X-ray powder data for $\text{MoP}_{0.75}$ given in Ref. 1, (Table 2), a few weak lines among those listed were found to belong to impurities, probably containing oxygen. In carefully prepared samples, these lines were absent. A revised list of diffraction lines for Mo_4P_3 is given in Table 1.

Table 1. X-Ray powder data for Mo_6P_3 .
(Guinier-type camera, $\text{CrK}\alpha_1$ radiation, $\lambda = 2.28962 \text{ \AA}$.)

h	k	l	$\sin^2\theta_{\text{obs}} \times 10^4$	$\sin^2\theta_{\text{calc}} \times 10^4$	I_{obs}	$p F^2 \times 10^{-3}$	h	k	l	$\sin^2\theta_{\text{obs}} \times 10^4$	$\sin^2\theta_{\text{calc}} \times 10^4$	I_{obs}	$p F^2 \times 10^{-3}$
1	0	1	116	116.3	w-	2	5	0	3	2403.7	2403.7	st	935
0	0	2		125.5		0	2	1	5	2439	2437.5	w-	59
1	0	2		210.4		0	4	0	6	2487.0	2487.0	m	314
2	0	0	338	339.4	w	9	1	1	6	2528.0	2528.0	m	419
1	0	3		367.3		0	3	1	4	2578.7	2579.4	st+	1577
2	0	1		370.8		0	5	0	4	2625	2623.2		34
2	0	2		464.9		3	1	0	9	2625	2625.8	w-	8
0	0	4		502.0		4	4	1	0	2671	2671.5	w	100
1	0	4		586.8		6	4	1	1	2702.2	2702.9	m	337
2	0	3		621.7		1	3	0	8		2771.2		2
3	0	1	795.2	795.0	w+	33	2	1	6		2782.5		0
2	0	4	843	841.3	w-	15	4	1	2	2796.5	2797.0	m	315
1	0	5		869.1		2	0	1	7	2849.8	2850.9	st	1216
3	0	2	888	889.1	w-	11	3	1	5	2861.4	2861.7	m	662
3	0	3	1047.0	1046.0	w+	51	2	0	9	2879.5	2880.3	st	1459
2	0	5	1124.0	1123.6	m-	80	4	0	7		2894.7		12
0	0	6	1129	1129.3	w	18	5	0	5	2904	2905.5	w	167
1	0	6	1214.7	1214.2	m	196	1	1	7	2936.1	2935.8	m-	280
3	0	4		1265.6		1	4	1	3		2953.9		10
0	1	1	1346.0	1345.3	w	41	6	0	0		3054.7		4
4	0	0		1357.7		8	6	0	1	3087	3086.1	w-	44
4	0	1		1389.1		4	0	0	10		3136.9		4
1	1	1	1430.8	1430.2	w	55	4	1	4		3173.5		723
2	0	6	1468.9	1468.7	m	266	6	0	2	3176	3180.2	st	461
4	0	2	1483	1483.2	w-	36	2	1	7		3190.3		75
1	1	2		1524.2		5	3	1	6		3206.8		1
3	0	5		1547.8		2	1	0	10	3221	3221.8	w	91
0	1	3	1596	1596.2	w-	49	5	0	6	3249	3250.6	w	89
1	0	7		1621.9		15	3	0	9	3304.5	3304.6	m+	741
4	0	3	1640.2	1640.0	m	467	6	0	3	3336.8	3337.1	st	997
2	1	0		1653.0		6	4	0	8		3365.3		3
1	1	3		1681.1		50	1	1	8	3405.7	3406.4	m	491
2	1	1	1683.5	1684.6	st	508	4	1	5		3455.8		30
2	1	2	1779.3	1778.7	m	391	5	1	1		3466.6		1
4	0	4	1860	1859.6	w-	33	2	0	10	3475	3476.3	w	80
2	0	7	1877	1876.4	w	145	6	0	4	3559	3556.6	w+	142
3	0	6		1892.9		15	5	1	2	3559	3560.6		162
1	1	4	1900.9	1900.7	w	87	3	1	7	3614.7	3614.5	m	609
2	1	3	1936.2	1935.6	w	98	5	0	7	3660	3658.3	m	38
0	0	8		2007.6		15	2	1	8	3660	3660.8		478
1	0	8		2092.5		215	5	1	3		3717.5		9
0	1	5	2098.2	2098.1	st	705	4	1	6	3800	3800.8	w-	71
3	1	1	2108.9	2108.9	st	746	6	0	5		3838.9		3
4	0	5		2141.9		24	0	1	9	3851	3854.8	w--	18
5	0	1		2152.7		185	1	0	11		3880.6		19
2	1	4	2154.7	2155.2	st	783	4	0	9		3896.6		12
1	1	5		2183.0		1	3	0	10		3900.5		7
3	1	2	2202.6	2203.0	w+	171	5	1	4	3937.4	3937.1	st	1108
5	0	2	2248	2246.8	w-	23	1	1	9		3939.7		1
3	0	7		2300.7		12	3	1	8		4085.1		6
2	0	8	2347.1	2347.0	w	167	5	0	8		4128.9		0
3	1	3	2359.5	2359.9	m+	620	2	0	11		4135.1		1

It proved difficult to find single crystals of Mo_4P_3 suitable for recording accurate intensities. The crystals obtained from the melts were needle-shaped with the needle-axis coinciding with the crystallographic b -axis. Weissenberg photographs showed that the "crystals" actually consisted of several smaller crystallites with their b -axes in very nearly parallel orientation, but with their a - (and c -) axes more or less randomly oriented. Eventually, a very small specimen was selected for the structure determination. It consisted of one large and a few much smaller crystallites, making it possible to make a clear distinction on the Weissenberg photographs between reflexions belonging to the large crystallite and those of the other fragments. The intensities were recorded in an ordinary Weissenberg camera with zirconium-filtered MoK radiation. The multiple-film technique was used with thin iron foils placed between successive films, and the intensities were estimated visually. Due to the minute size of the reflexion spots on the films, the intensity measurement was very difficult and the accuracy was accordingly much lower than that normally attained for similar types of crystals.

The electronic computers BESK and FACIT EDB were used in the numerical computations for the following purposes: corrections for Lorentz and polarisation factors,³ Fourier series summations,⁴ structure factor calculations,⁴ least squares refinement⁵ and calculation of interatomic distances.⁴ In the calculations of structure factors, atomic scattering factors, corrected for anomalous dispersion by addition of the real part of the dispersion correction, were interpolated from the values given in Ref.6.

DETERMINATION AND REFINEMENT OF THE Mo_4P_3 STRUCTURE

The Weissenberg photographs of the Mo_4P_3 crystals showed that the symmetry is orthorhombic. The systematic extinctions, $hk0$ for h odd and $0kl$ for $k + l$ odd, indicate $Pnma$ or $Pn2_1a$ as possible space groups. The very short b axis (3.158 Å) together with the fact that the ratio $F_0(h0l)/F_0(h2l)$ was found to be nearly constant, indicates strongly that the symmetry is $Pnma$ with all atoms in $4(c)$ positions. The unit cell volume suggests a cell content of eight formula units.

The above arguments make it probable that there are eight molybdenum atoms and six phosphorus atoms in the asymmetric part of the cell.

The Patterson projection $P(xz)$ was evaluated. In spite of the extensive overlap of the peaks it was possible to derive a set of eight molybdenum positions consistent with the Patterson projection. By introducing the restriction that all Mo—Mo distances should exceed 2.5 Å, the mutual disposition of the atoms in the b direction could be derived. Working with the assumption that Mo—P and P—P distances should exceed 2 Å and 3 Å, respectively, it was also possible to derive probable positions for the six phosphorus atoms. This structure proposal was further confirmed by analysis of the Patterson sections $P(x0z)$ and $P(x\frac{1}{2}z)$, and more accurate atomic positions were obtained from an electron density projection on (010).

The structure was finally refined by the least squares method. For this purpose 255 observed $F(h0l)$ - and 270 observed $F(h1l)$ -values were used. Two positional parameters and one isotropic temperature factor for each atom together with two scale factors, one for the $h0l$ and one for the $h1l$ set of reflexions, were varied. The programme used minimizes the function $w(F_0 - |F_c|)^2$. The weighting factor w was set equal to $1/F_0^2$ for $F_0 \geq 80$ and to $1/16 F_0^2$ for $F_0 < 80$. After the final refinement cycle, the R -value was 0.12 and the shifts in all parameters were less than 5% of the calculated standard deviations.

The individual temperature factors obtained from the least squares refinement were not very different from zero and in most cases negative values were returned. This effect cannot be ascribed to the influence of absorption, since the crystal size is much too small. Extinction effects may possibly constitute one source of error, but in the author's opinion the negative temperature factor values are most probably related to a systematic error in the intensity measurement; the extremely small size of the diffraction spots on the films makes the intensity estimation of the strong low-angle reflexions very uncertain. Since each spot covers only a moderate number of grains in the photographic emulsion, strong reflexions are liable to be underestimated even if the multiple-film technique is used. These arguments are presented to indicate that no great significance should be attached to the values actually obtained for the temperature factors. On the other hand, there seems to be no reasonable doubt about the essential correctness of the structure determination as regards the positional parameters of the atoms. The least squares refinement converged satisfactorily, and a final difference synthesis did not reveal any spurious peaks or other abnormal features.

The final structure data obtained for Mo_4P_3 are as follows: Space group $Pnma$, $a = 12.428 \text{ \AA}$; $b = 3.158 \text{ \AA}$; $c = 20.440 \text{ \AA}$; $Z = 8$. All atoms are situated in $4(c)$ positions with the parameter values

Atom	x	$\sigma(x)$	z	$\sigma(z)$	$B \text{ \AA}^2$	$\sigma(B) \text{ \AA}^2$
Mo _I	0.1939	0.0004	0.2262	0.0002	0.00	0.07
Mo _{II}	0.3843	0.0004	0.8518	0.0002	-0.11	0.06
Mo _{III}	0.1218	0.0004	0.6891	0.0002	-0.05	0.07
Mo _{IV}	0.2980	0.0004	0.0835	0.0002	-0.09	0.07
Mo _V	0.4007	0.0004	0.5269	0.0002	-0.10	0.06
Mo _{VI}	0.0952	0.0004	0.4665	0.0002	-0.02	0.07
Mo _{VII}	0.2977	0.0004	0.3863	0.0002	-0.07	0.07
Mo _{VIII}	0.0095	0.0004	0.8272	0.0002	-0.09	0.07
P _I	0.2464	0.0011	0.9730	0.0007	-0.06	0.20
P _{II}	0.4824	0.0013	0.4186	0.0007	0.21	0.24
P _{III}	0.3206	0.0012	0.6371	0.0007	0.10	0.22
P _{IV}	0.0382	0.0013	0.5805	0.0007	0.27	0.25
P _V	0.0155	0.0010	0.2745	0.0006	-0.33	0.18
P _{VI}	0.1994	0.0010	0.7941	0.0006	-0.23	0.19

Interatomic distances are given in Table 2. A list of observed and calculated structure factors can be obtained from this Institute on request.

DESCRIPTION OF THE Mo_4P_3 STRUCTURE

A projection of the structure on (010) is shown in Fig. 1. The coordination about the phosphorus atoms is summarized in Table 3. There are no P—P distances shorter than 3.1 \AA in the structure, and the immediate environment about the phosphorus atoms, accordingly, consists of molybdenum atoms only. The phosphorus atom P_{II} is surrounded by six molybdenum atoms arranged at the corners of a triangular prism. The remaining phosphorus atoms have seven molybdenum neighbours, six of which take up a triangular prismatic arrangement while the seventh is situated outside one of the quadrilateral faces of the prism. The general structural architecture can be described in terms of a complex packing of six different types of PMo_6 triangular prisms. The packing of these prisms is illustrated in Fig. 1. The triangular faces of the

Table 2. Interatomic distances in Mo_4P_8 . (Since all atoms are situated in the same type of crystallographic position, the distances between crystallographically non-equivalent atoms are listed only once in order to reduce the size of the table. The distances are given in Å units, and only those shorter than 3.9 Å are listed).

Mo_I	— Mo_I	: 3.158 (2)	Mo_{VI}	— Mo_{VI}	: 3.158 (2)
	— Mo_{II}	: 3.166 (2)		— Mo_{VI}	: 3.156 (2)
	— Mo_{III}	: 2.883 (2)		— Mo_{VII}	: 3.004
	— Mo_{IV}	: 3.191		— P_I	: 2.528 (2)
	— Mo_{VIII}	: 3.175 (2)		— P_{IV}	: 2.436
	— Mo_{VIII}	: 3.518		— P_{IV}	: 2.482 (2)
	— P_{III}	: 2.418 (2)	Mo_{VII}	— Mo_{VII}	: 3.158 (2)
	— P_V	: 2.428		— Mo_{VIII}	: 3.113 (2)
	— P_{VI}	: 2.485 (2)		— P_I	: 2.436 (2)
Mo_{II}	— Mo_{II}	: 3.158 (2)		— P_{II}	: 2.388
	— Mo_{III}	: 3.067		— P_{VI}	: 2.460 (2)
	— Mo_{VI}	: 2.839 (2)	Mo_{VIII}	— Mo_{VIII}	: 3.158 (2)
	— Mo_{VII}	: 2.848 (2)		— P_{II}	: 2.448 (2)
	— P_I	: 3.013		— P_{III}	: 2.458
	— P_{IV}	: 2.361		— P_V	: 2.630 (2)
	— P_V	: 2.557 (2)		— P_{VI}	: 2.456
	— P_{VI}	: 2.583	P_I	— P_I	: 3.158 (2)
Mo_{III}	— Mo_{III}	: 3.158 (2)		— P_{II}	: 3.44 (2)
	— Mo_{IV}	: 2.855 (2)		— P_{III}	: 3.80 (2)
	— Mo_{VIII}	: 3.149		— P_{IV}	: 3.79
	— P_{III}	: 2.691		— P_{IV}	: 3.81 (2)
	— P_{IV}	: 2.451		— P_{VI}	: 3.70
	— P_V	: 2.440 (2)	P_{II}	— P_{II}	: 3.158 (2)
	— P_{VI}	: 2.352		— P_{II}	: 3.71 (2)
Mo_{IV}	— Mo_{IV}	: 3.158 (2)		— P_{III}	: 3.13 (2)
	— Mo_V	: 3.151 (2)		— P_{VI}	: 3.75 (2)
	— Mo_{VI}	: 3.158 (2)	P_{III}	— P_{III}	: 3.158 (2)
	— P_I	: 2.348		— P_{IV}	: 3.70
	— P_{III}	: 2.422 (2)		— P_V	: 3.81 (2)
	— P_{IV}	: 2.577 (2)		— P_{VI}	: 3.55
Mo_V	— Mo_V	: 3.158 (2)	P_{IV}	— P_{IV}	: 3.158 (2)
	— Mo_V	: 3.130 (2)		— P_{IV}	: 3.77 (2)
	— Mo_{VII}	: 3.145		— P_V	: 3.42 (2)
	— Mo_{VIII}	: 3.275	P_V	— P_V	: 3.158 (2)
	— P_I	: 2.655 (2)		— P_{VI}	: 3.40 (2)
	— P_{II}	: 2.419 (2)		— P_{VI}	: 3.90 (2)
	— P_{II}	: 2.435	P_{VI}	— P_{VI}	: 3.158 (2)
	— P_{III}	: 2.462			

P_I and P_V prisms are parallel with (010), while the triangular faces of the remaining prisms are perpendicular to this plane. As seen in Fig. 1, the seventh molybdenum neighbour to P_I and P_{III} — P_{VI} belongs to an adjacent prism.

The coordination about the molybdenum atoms is summarized in Table 4. Each molybdenum atom is surrounded by 8 or 9 molybdenum atoms at distances between 2.84 and 3.28 Å (distances to further neighbours exceed 3.52 Å) and 4–6 phosphorus neighbours at distances between 2.35 and

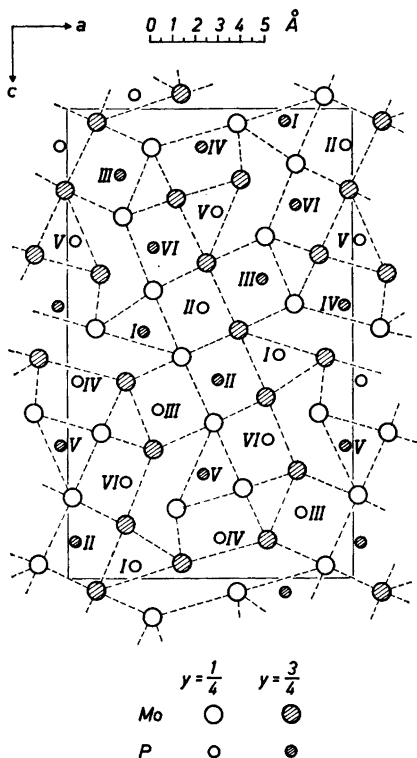


Fig. 1. The crystal structure of Mo_4P_3 projected on (010). The triangular PMo_3 prisms are indicated by broken lines. The Roman numerals refer to the six crystallographically non-equivalent phosphorus atom sites.

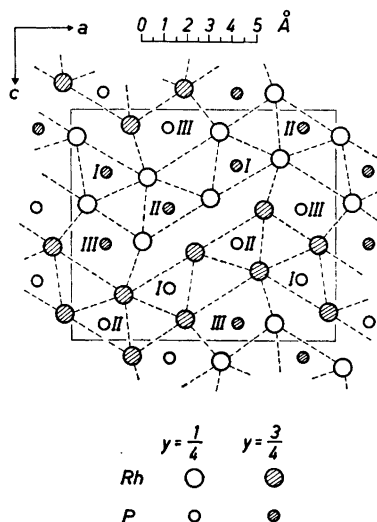


Fig. 2. The crystal structure of Rh_4P_3 projected on (010). The triangular PRh_3 prisms are indicated by broken lines. The Roman numerals refer to the three crystallographically non-equivalent phosphorus atom sites.

Table 3. The coordination about the phosphorus atoms in Mo_4P_3 . N_{Me} = number of metal neighbours within 3.0 Å. d_{av} = average distance in Å. R_{Me} = Goldschmidt metal radius for 12-coordination. r_{P} = tetrahedral covalent radius for phosphorus.

Central atom	N_{Me}	d_{av}	$d_{\text{av}} - (R_{\text{Me}} + r_{\text{P}})$
P _I	7	2.51	+0.01
P _{II}	6	2.43	-0.07
P _{III}	7	2.47	-0.03
P _{IV}	7	2.48	-0.02
P _V	7	2.53	+0.03
P _{VI}	7	2.47	-0.03

2.69 Å (further neighbours at 3.01 Å or more). The mean Mo—Mo distance (average of 34 non-equivalent distances) is 3.08 Å, which exceeds the Goldschmidt CN 12 metal diameter of 2.80 Å by 10 %. The mean Mo—P distance

Table 4. The coordination about the molybdenum atoms in Mo_4P_3 . N_{Me} = number of metal neighbours within 3.5 Å. N_{P} = number of phosphorus neighbours within 3.0 Å. (Further notations are explained in Table 3.)

Central atom	N_{Me}	d_{av}	$d_{\text{av}}/2R_{\text{Me}}$	N_{P}	d_{av}	$d_{\text{av}} - (R_{\text{Me}} + r_{\text{P}})$
Mo _I	9	3.11	1.11	5	2.45	-0.06
Mo _{II}	9	3.01	1.08	4	2.51	+0.01
Mo _{III}	8	3.00	1.07	5	2.47	-0.03
Mo _{IV}	9	3.09	1.10	5	2.47	-0.03
Mo _V	8	3.16	1.13	6	2.51	+0.01
Mo _{VI}	9	3.07	1.10	5	2.49	-0.01
Mo _{VII}	8	3.05	1.09	5	2.44	-0.06
Mo _{VIII}	8	3.16	1.13	6	2.51	+0.01

Table 5. The coordination about the phosphorus atoms in Rh_4P_3 . (The notations are explained in Table 3.)

Central atom	N_{Me}	d_{av}	$d_{\text{av}} - (R_{\text{Me}} + r_{\text{P}})$
P _I	7	2.43	-0.01
P _{II}	7	2.41	-0.03
P _{III}	6	2.38	-0.06

Table 6. The coordination about the rhodium atoms in Rh_4P_3 . (The notations are explained in Table 4.)

Central atom	N_{Me}	d_{av}	$d_{\text{av}}/2R_{\text{Me}}$	N_{P}	d_{av}	$d_{\text{av}} - (R_{\text{Me}} + r_{\text{P}})$
Rh _I	8	3.01	1.12	5	2.44	0.00
Rh _{II}	8	2.99	1.12	5	2.40	-0.04
Rh _{III}	7	3.03	1.13	5	2.38	-0.06
Rh _{IV}	7	2.99	1.12	5	2.42	-0.02

(average of 41 non-equivalent distances) is 2.48 Å, which is 0.02 Å smaller than the sum of the metal radius for molybdenum, 1.40 Å, and the tetrahedral covalent radius for phosphorus, 1.10 Å. These distances can be compared with those in MoP in which each molybdenum atom is surrounded by eight molybdenum neighbours at an average distance of 3.215 Å and six phosphorus neighbours at 2.451 Å.

THE STRUCTURAL RELATIONSHIPS BETWEEN Mo_4P_3 AND OTHER PHOSPHIDES

In a recent survey of transition metal phosphides⁷ some general structural features of metal-rich phosphides were presented. It was pointed out that the P—P distances are always greater than 2.9 Å, and the number of metal neighbours to phosphorus is 6—10. The arrangement of the metal neighbours about phosphorus is frequently triangular prismatic with 0—3 additional neighbours lying outside the quadrilateral faces of the prism. Mo_4P_3 is evidently

a typical representative of the phases conforming to the above mentioned structural principles.

Among the structures of transition metal mono-phosphides there are four types, which can be described as being composed of PMe_6 triangular prisms. These types are NiAs (VP), MnP (CrP, MnP, FeP, CoP, WP, RuP), NbAs (NbP, TaP), and WC (MoP). The Mo_4P_3 structure bears resemblances to all four of these types. For instance, a block of prisms stacked in the same way as in NiAs (or in MnP) can be distinguished in Fig. 1 near the middle of the unit cell. The block is composed of three pairs of prisms (the P_{II} , P_{III} , and P_{VI} prisms) and extends infinitely in the [010] direction.

The structure⁸ of Rh_4P_3 is closely related to that of Mo_4P_3 . The compound Rh_4P_3 crystallizes with the same space group symmetry as Mo_4P_3 and the atoms are situated in the same type of crystallographic position. The unit cell content, however, is only 4 formula units. A projection of the Rh_4P_3 structure is shown in Fig. 2, where the triangular prismatic metal environment about phosphorus is indicated. The structure contains WC-type corrugated layers, which are roughly parallel with (001) and are composed of P_I and P_{II} prisms. Pairs of P_{III} prisms form NiAs-type columns extending along [010].

The coordination about the atoms in Rh_4P_3 is summarized in Tables 5 and 6. (As in the case of Mo_4P_3 , only metal-metal distances shorter than 3.5 Å and metal-phosphorus distances shorter than 3.0 Å are taken into account). Each rhodium atom has five phosphorus neighbours and 7–8 rhodium neighbours. The average Rh–Rh distance is 3.01 Å, which exceeds the metal diameter, 2.68 Å, by 12 %. The average Rh–P distance of 2.41 Å is 0.03 Å shorter than the radius sum. The coordination about the metal atoms is evidently higher in Mo_4P_3 than in Rh_4P_3 . This may be associated with the difference in metal radii, the molybdenum radius being somewhat larger than that of rhodium. The structural differences between Mo_4P_3 and Rh_4P_3 may therefore arise in consequence of the general tendency to good space-filling in the two structures.

Acknowledgements. The author wishes to thank Professor G. Hägg for his encouraging interest.

The author is indebted to Mr. B. Sellberg for valuable work in the preparation of crystals and in the preliminary X-ray investigations.

The work has been supported by the *Swedish Natural Science Research Council*. Facilities for use of the electronic computers BESK and FACIT were granted by the Computer Division of the *National Swedish Office for Administrative Rationalization and Economy*.

REFERENCES

1. Rundqvist, S. and Lundström, T. *Acta Chem. Scand.* **17** (1963) 37.
2. Gingerich, K. A. *J. Phys. Chem.* **68** (1964) 2514.
3. Lundberg, B. Programme available at BESK.
4. Liminga, R. and Olovsson, I. *Acta Polytechn. Scand.* (1964) Ma 10.
5. Åsbrink, S. and Brändén, C.-I. *IUCr World List of Cryst. Computer Programs*, No. 6023 FACIT (1962).
6. *Int. Tables for X-Ray Crystallography*, Vol. III, The Kynoch Press, Birmingham (1962).
7. Rundqvist, S. *Arkiv Kemi* **20** (1962) 67.
8. Rundqvist, S. and Hede A. *Acta Chem. Scand.* **14** (1960) 893.

Received November 24, 1964.

Acta Chem. Scand. **19** (1965) No. 2