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The Crystal Structure of Nb_5P_3

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The crystal structure of Nb_5P_3 has been determined by X-ray single crystal methods. The symmetry is orthorhombic and the cell dimensions, determined by powder diffraction techniques, are $a = 25.384$ Å, $b = 3.433$ Å, and $c = 11.483$ Å. The unit cell contains 40 niobium atoms and 24 phosphorus atoms. The refinement of the structure is based on the space group *Pnma* with all atoms in 4 *c* positions.

Previous work[1,2] on the Nb–P system has shown that reaction between the component elements at temperatures below 1100°C results only in the formation of the monophosphide or the diphosphide. However, when mixtures of niobium and niobium monophosphide are heated at much higher temperatures, several new intermediate phases are formed.[2] Among these, the two compounds Nb_3P and Nb_7P_4 have been identified and characterized crystallographically.[3,4] In the present paper the occurrence of a third phase, Nb_5P_3, is reported. Nb_5P_3 has been characterized by powder diffraction work and a complete single crystal structure determination. In other studies carried out at this Institute,[5,6] compounds isostructural with Nb_5P_3 have been found in the Hf–As and Nb–As systems, and there is probably also an isostructural compound in the Ta–P system.[7]

EXPERIMENTAL

Preparation. Niobium (Hermann Starck, Berlin, claimed purity 99.3 %) and phosphorus (purity higher than 99 %) powders were thoroughly mixed and pressed into pellets, which were slowly heated to 750°C in evacuated silica tubes. The sintered products were arcmelted in an atmosphere of purified argon. During the preparation some phosphorus was lost.

X-Ray work. The unit cell dimensions were obtained from powder photographs in a Guinier-Hägg focussing camera with monochromatic Cr$K\alpha_1$ radiation ($\lambda = 2.28962$ Å). Silicon ($a = 5.43054$ Å) was used as an internal calibration standard.

From a specimen containing Nb_5P_3 and a small amount of an unidentified phase, a single crystal was chosen and rotated about the *b* axis in a Weissenberg camera. Using Zr-filtered Mo$K\alpha$ radiation, intensity data were recorded using the multiple film technique. Thin iron foils were used as absorbers between successive films. The intensities were estimated visually using an intensity scale, prepared by exposing a reflexion of the same crystal for various numbers of oscillations through the diffraction position.

Table 1. Powder diffraction data for Nb_5P_3. Guinier-Hägg camera with $CrK\alpha_1$ radiation. $I_{calc} = p \times |F_{calc}|^2 \times 10^4$.

h k l	$\sin^2\theta \times 10^4$ obs.	$\sin^2\theta \times 10^4$ calc.	*I* obs.	*I* calc.	*h k l*	$\sin^2\theta \times 10^4$ obs.	$\sin^2\theta \times 10^4$ calc.	*I* obs.	*I* calc.
2 0 0		81.4		0.0	10 0 0	2035.0	2033.9	w−	13.6
1 0 1		119.7		0.1	9 0 2	2045.1	2045.0	w	16.9
2 0 1	180.1	180.7	w−	0.3	2 1 3	2088.0	2087.9	st	119.1
3 0 1	281.5	282.5	w−	0.7	5 0 4	2098.6	2098.7	w+	42.3
4 0 0		325.4		0.0	10 0 1	2133.8	2133.3	w	12.5
0 0 2	396.8	397.5	w−	1.0	3 1 3	2198.7	2189.6	w	29.0
1 0 2		417.9		0.4	8 0 3	2196.4	2196.2	w−	14.3
4 0 1	423.5	424.8	w−	1.8	7 1 1	2207.3	2208.1	w−	15.0
2 0 2		478.9		0.3	6 1 2	2242.1	2241.9	w	27.1
3 0 2		580.6		0.1	6 0 4		2322.4		1.9
5 0 1		607.9		0.6	4 1 3	2331.6	2332.0	st	194.9
4 0 2		723.0		0.2	8 1 0	2413.3	2413.8	m	109.2
6 0 0		732.2		1.0	10 0 2		2431.5		12.4
6 0 1	832.0	831.6	w−	0.7	1 0 5	2505.7	2505.0	st	4.9
5 0 2		906.0		0.3	7 1 2		2506.3		232.6
1 0 3		914.8		1.0	8 1 1	2514.4	2513.2	st	67.2
2 0 3	975.3	975.8	w	4.8	5 1 3		2515.1		121.5
3 0 3		1077.5		0.0	9 0 3	2541.5	2542.0	w−	16.3
7 0 1		1096.0		1.2	11 0 1	2560.5	2560.4	w+	89.3
6 0 2	1128.4	1129.8	w	3.0	2 0 5	2568.6	2566.0	w	41.1
2 1 0		1193.5		0.3	7 0 4	2586.6	2586.8	m	130.1
0 1 1	1210.9	1211.5	w	6.6	3 0 5	2667.3	2667.7	w−	12.0
4 0 3	1219.1	1219.9	w	11.2	1 1 4	2722.0	2722.6	m+	145.7
1 1 1	1231.4	1231.8	w	13.6	6 1 3		2738.8		1.7
2 1 1		1292.8		0.7	2 1 4 [a]	2784.5	2783.6	w	4.0
8 0 0	1302.0	1301.7	w	9.8	4 0 5	2810.2	2810.1	m+	73.0
7 0 2		1394.2		0.4	2 1 2		2811.4		73.8
3 1 1		1394.5		2.2	11 0 2	2858.5	2858.6	w+	0.0
8 0 1	1402.2	1401.1	w−	3.2	9 1 1		2859.0		61.7
5 0 3		1403.0		0.7	3 1 4 [a]	2883.9	2885.3	w−	4.5
4 1 0		1437.5		1.7	8 0 4	2891.9	2891.9	w	42.4
1 1 2	1530.6	1530.0	w−	6.3	10 0 3	2982.1	2928.4	m−	90.4
4 1 1	1536.9	1536.9	w	9.8	12 0 0		2928.9		0.8
0 0 4	1591.3	1590.2	m−	8.3	5 0 5		2993.1		7.5
2 1 2		1591.0		22.2	7 1 3		3003.2		0.2
1 0 4	1610.5	1610.5	w+	19.8	4 1 4	3027.4	3027.7	w	41.8
6 0 3		1626.7		2.9	12 0 1		3028.3		8.9
2 0 4		1671.5		0.3	10 1 0	3145.4	3146.0	w	37.4
3 1 2		1692.7		0.0	9 1 2		3157.1		7.8
8 0 2	1699.0	1699.3	w−	5.9	5 1 4	3210.5	3210.8	w	25.9
5 1 1	1720.1	1720.0	m−	33.8	6 0 5		3216.9		1.5
9 0 1	1747.1	1746.9	m−	25.6	9 0 4		3237.7		5.7
3 0 4		1773.2		0.4	10 1 1 [a]	3243.2	3245.4	w−	7.6
4 1 2	1836.0	1835.1	w−	11.1	8 1 3	3308.8	3308.3	w−	11.3
6 1 0	1844.3	1844.3	m	76.8	12 0 2	3325.2	3326.4	w	39.7
7 0 3		1891.1		0.9	11 0 3		3355.5		1.2
4 0 4	1914.9	1915.6	w+	29.8	6 1 4		3434.5		0.8
6 1 1		1943.7		0.6	7 0 5	3480.8	3481.3	w−	16.8
0 1 3	2006.4	2006.6	w−	7.7	13 0 1		3536.7		0.1
5 1 2		2018.1		0.2	10 1 2		3543.6		0.0
1 1 3	2026.5	2026.9	m−	41.2	0 0 6		3577.9		5.0

Table 1. Continued.

0 1 5	3595.9	3596.8	w−	20.3	14 0 0		3986.5		0.1
1 0 6		3598.2		0.2	8 1 4		4004.0		0.3
1 1 5	3617.5	3617.1	w−	13.3	10 1 3		4040.5		0.4
10 0 4		3624.1		0.5	12 1 0		4041.0		0.9
9 1 3		3654.0		0.1	11 0 4		4051.2		0.0
2 0 6	3660.1	3659.2	w	35.2	5 0 6	4087.8	4085.9	w	1.6
11 1 1		3672.5		8.3	14 0 1		4086.4		33.4
2 1 5	3677.6	3678.1	w−	16.7	5 1 5		4105.2		0.1
7 1 4	3698.8	3698.9	w−	7.0	9 0 5	4136.9	4132.1	w	18.5
3 0 6	3761.5	3760.9	w−	16.1	12 1 1		4140.4		25.5
3 1 5	3779.1	3779.8	w−	16.5	6 0 6		4310.1		1.0
8 0 5		3786.4		1.5	6 1 5		4329.0		2.8
12 0 3 [a]	3822.8	3823.3	w−	9.6	13 0 3	4334.5	4331.8	w−	14.8
13 0 2		3834.9		0.0	9 1 4		4349.8		8.3
4 0 6		3903.3		0.1	14 0 2		4384.0		0.0
4 1 5		3922.2		0.1	12 1 2		4438.5		0.5
11 1 2		3970.7		0.3	0 2 0	4450.5	4448.4	st	304.9

[a] Overlapped by a line belonging to an unidentified phase.

The crystal was in the shape of a parallelepiped, bounded by {100}, {010}, and {001} planes. The dimensions in the *a*, *b*, and *c* directions, respectively, were 0.065, 0.176, and 0.026 mm. An absorption correction was applied to the observed intensities using the above mentioned dimensions of the crystal and the value 115 cm^{-1} for the linear absorption coefficient.

Calculation. The calculations were performed on a CDC 3600 computer using the FORTRAN IV programmes listed in Table 1, Ref. 4.

DETERMINATION OF THE STRUCTURE

Inspection of the Weissenberg films showed that the crystal symmetry was orthorhombic. By use of the information from the Weissenberg films, the reflexions on the powder films were indexed. A least squares refinement of the powder diffraction data gave the following unit cell dimensions and standard deviations: $a = 25.3843 \pm 0.0010$ Å; $b = 3.4329 \pm 0.0002$ Å; $c = 11.4834 \pm 0.0004$ Å. (For programmes used in the calculations see Table 1, Ref. 4.) Powder diffraction data are given in Table 1.

Systematic extinctions were observed for $hk0$ reflexions with $h = 2n+1$, and for $0kl$ reflexions $k+l = 2n+1$, and the most probable space groups are accordingly *Pnma* or $Pn2_1a$. The short *b* axis excludes the 4 *a*, 4 *b*, and 8 *d* positions in *Pnma*. The ratio $F_o(h0l)/F_o(h2l)$ was constant within the errors in the visual estimation of the intensities. This indicates that the atoms are situated in two planes perpendicular to the *b* axis *b*/2 apart, and it seemed reasonable to assume that the atoms should occupy 4 *c* positions in the space group *Pnma*.

Since it was impossible to prepare homogeneous single phase samples of the new compound in amounts sufficient for ordinary chemical analysis, the composition of the compound remained somewhat uncertain. A comparison of the unit cell volume with the unit cell volumes of Nb_7P_4 and NbP indicated a unit cell content of 40 niobium and 24 phosphorus atoms. This composition was subsequently confirmed by the results of the structure analysis.

With the atoms in two planes perpendicular to the b axis and $b/2$ apart, the Patterson sections $P(u0w)$ and $P(u\frac{1}{2}w)$ should contain all relevant maxima. These sections were calculated from the intensities of the $h0l$ and $h1l$ layer lines. An analysis of the Harker peaks gave a reasonable arrangement for 40 niobium atoms placed in 10 fourfold positions. From space considerations 6 fourfold positions for the phosphorus atoms were obtained. An electron density calculation confirmed the structure proposal.

The refinement of the structure was performed with a full-matrix least squares program using 785 $h0l$ and 916 $h1l$ reflexions. Atomic scattering factor values were taken from *International Tables*, III.[8] Since extinction effects were rather marked, a correction was applied according to Zachariasen:[9]

$$F_{o,corr} \approx F_o\{1 + c \cdot \beta(2\theta) \cdot I_o\}$$

The following β-function was used:

$$\beta(2\theta) = \frac{(1 + q \cdot \cos^4 2\theta) \cdot \mathrm{Lp} \cdot 2}{(1 + q \cdot \cos^2 2\theta) \cdot \sin 2\theta} \frac{\mathrm{d}A^*}{\mathrm{d}\mu}(2\theta)$$

where A^* denotes the absorption for the reflexion, μ the linear absorption coefficient, and where q is 1 for an unpolarized incident beam. The value of c was 1.5×10^{-4} with I_o on an absolute scale.

The reflexions were weighted according to a formula, $w = 1/(a + |F_o| + c|F_o|^2 + d|F_o|^3)$, suggested by Cruickshank *et al.*[10] The constants were finally given the values: $a = 250$; $c = 0.010$; and $d = 0.0005$.

The 50 parameters refined were one scale factor for each of the $h0l$ and $h1l$ sets of reflexions, 32 positional parameters and 16 isotropic temperature factors. The final R-value (defined as $R = \sum||F_o| - |F_c||/|F_o|$) with all 1701 reflexions included was 0.114. The last shifts for all parameters were less than 3 % of the standard deviations.

Table 2. Final structural data for Nb_5P_3. Space group *Pnma* (No. 62). $z = 8$. All atoms in position 4 c with $y = 1/4$. $a = 25.384$ Å; $b = 3.433$ Å; $c = 11.483$ Å; $U = 1000.7$ Å^3. Standard deviations within parentheses.

Atom	x	z	B (Å^2)
Nb(1)	0.10135 (7)	0.56855 (16)	0.147 (18)
Nb(2)	0.12423 (7)	0.28862 (17)	0.213 (19)
Nb(3)	0.13736 (7)	0.84442 (17)	0.254 (20)
Nb(4)	0.22586 (7)	0.62056 (15)	0.152 (18)
Nb(5)	0.26009 (7)	0.34465 (16)	0.174 (18)
Nb(6)	0.32294 (7)	0.95971 (15)	0.119 (17)
Nb(7)	0.34834 (7)	0.55675 (15)	0.138 (18)
Nb(8)	0.45455 (7)	0.69458 (15)	0.128 (18)
Nb(9)	0.46426 (7)	0.41586 (16)	0.190 (19)
Nb(10)	0.49917 (7)	0.10817 (16)	0.168 (19)
P(1)	0.04901 (21)	0.7489 (5)	0.22 (6)
P(2)	0.07718 (23)	0.0524 (5)	0.33 (6)
P(3)	0.21960 (22)	0.0001 (5)	0.35 (6)
P(4)	0.30268 (20)	0.7462 (5)	0.17 (6)
P(5)	0.34161 (21)	0.1883 (5)	0.28 (6)
P(6)	0.42732 (21)	0.9191 (5)	0.28 (6)

Table 3. Interatomic distances for Nb_5P_3 (in Ångström units). Distances shorter than 4.0 Å are listed. Standard deviations within parentheses.

Nb(1) –		Nb(6) –		P(1) –	
1 P(1)	2.460 (6)	1 P(4)	2.504 (6)	1 Nb(1)	2.460 (6)
2 P(6)	2.534 (4)	1 P(3)	2.664 (6)	1 Nb(8)	2.485 (5)
2 P(5)	2.634 (4)	1 P(5)	2.669 (6)	1 Nb(3)	2.497 (6)
2 Nb(6)	2.864 (2)	1 P(6)	2.691 (6)	2 Nb(9)	2.596 (4)
2 Nb(10)	3.109 (2)	2 Nb(4)	2.809 (2)	2 Nb(10)	2.656 (4)
1 Nb(4)	3.216 (2)	2 Nb(1)	2.864 (2)	2 P(5)	3.337 (6)
1 Nb(2)	3.266 (3)	2 Nb(2)	2.934 (2)	2 P(1)	3.433 (0)
1 Nb(10)	3.294 (2)	2 Nb(5)	3.022 (2)	1 P(2)	3.558 (8)
1 Nb(3)	3.297 (3)	2 Nb(6)	3.433 (0)	1 P(6)	3.642 (8)
2 Nb(1)	3.433 (0)				
Nb(2) –		Nb(7) –		P(2) –	
2 P(4)	2.574 (4)	1 P(4)	2.466 (6)	2 Nb(8)	2.502 (4)
2 P(6)	2.628 (4)	2 P(3)	2.519 (4)	2 Nb(9)	2.551 (5)
2 Nb(8)	2.848 (2)	2 P(2)	2.554 (4)	2 Nb(7)	2.554 (4)
2 Nb(6)	2.934 (2)	2 Nb(3)	3.004 (2)	1 Nb(3)	2.834 (6)
1 P(2)	2.964 (6)	1 Nb(8)	3.126 (2)	1 Nb(9)	2.890 (6)
2 Nb(7)	3.243 (2)	1 Nb(4)	3.194 (2)	1 Nb(2)	2.964 (6)
1 Nb(1)	3.266 (3)	2 Nb(2)	3.243 (2)	2 P(2)	3.433 (0)
1 Nb(10)	3.389 (2)	1 Nb(5)	3.310 (2)	1 P(1)	3.558 (8)
2 Nb(2)	3.433 (0)	1 Nb(9)	3.358 (2)	1 P(3)	3.665 (8)
1 Nb(5)	3.508 (2)	2 Nb(7)	3.433 (0)		
Nb(3) –		Nb(8) –		P(3) –	
1 P(1)	2.497 (6)	1 P(1)	2.485 (5)	2 Nb(7)	2.519 (4)
2 P(5)	2.538 (4)	2 P(2)	2.502 (4)	2 Nb(5)	2.530 (5)
1 P(3)	2.748 (6)	1 P(6)	2.670 (6)	2 Nb(4)	2.603 (5)
1 P(2)	2.834 (6)	2 Nb(2)	2.848 (2)	1 Nb(6)	2.664 (6)
2 Nb(7)	3.004 (2)	2 Nb(9)	2.967 (2)	1 Nb(3)	2.748 (6)
2 Nb(5)	3.118 (2)	2 Nb(10)	3.075 (2)	2 P(4)	3.355 (7)
2 Nb(9)	3.205 (2)	1 Nb(7)	3.126 (2)	2 P(3)	3.433 (0)
1 Nb(1)	3.297 (3)	1 Nb(9)	3.210 (3)	1 P(4)	3.597 (8)
1 Nb(4)	3.414 (3)	2 Nb(8)	3.433 (0)	1 P(2)	3.665 (8)
2 Nb(3)	3.433 (0)	1 P(4)	3.901 (5)	1 P(5)	3.777 (8)
Nb(4) –		Nb(9) –		P(4) –	
1 P(4)	2.426 (5)	2 P(2)	2.551 (5)	1 Nb(4)	2.426 (5)
2 P(5)	2.547 (4)	2 P(1)	2.596 (4)	1 Nb(7)	2.466 (6)
2 P(3)	2.603 (5)	1 P(2)	2.890 (6)	1 Nb(6)	2.504 (6)
2 Nb(6)	2.809 (2)	2 Nb(8)	2.967 (2)	2 Nb(2)	2.574 (4)
2 Nb(5)	3.114 (2)	2 Nb(9)	3.158 (3)	2 Nb(5)	2.600 (4)
1 Nb(7)	3.194 (2)	2 Nb(3)	3.205 (2)	2 P(3)	3.355 (7)
1 Nb(1)	3.216 (2)	1 Nb(8)	3.210 (3)	2 P(4)	3.433 (0)
1 Nb(5)	3.286 (3)	1 Nb(7)	3.358 (2)	1 P(3)	3.597 (8)
1 Nb(3)	3.414 (3)	2 Nb(9)	3.433 (0)	1 P(6)	3.736 (8)
2 Nb(4)	3.433 (0)	1 Nb(10)	3.643 (3)	1 Nb(8)	3.901 (5)
Nb(5) –		Nb(10) –		P(5) –	
2 P(3)	2.530 (5)	2 P(6)	2.555 (4)	2 Nb(3)	2.538 (4)
2 P(4)	2.600 (4)	2 P(1)	2.656 (4)	2 Nb(3)	2.547 (4)
1 P(5)	2.738 (6)	1 P(6)	2.835 (6)	2 Nb(1)	2.634 (4)
2 Nb(6)	3.022 (2)	2 Nb(10)	3.020 (3)	1 Nb(6)	2.669 (6)
2 Nb(4)	3.114 (2)	2 Nb(8)	3.075 (2)	1 Nb(5)	2.738 (6)
2 Nb(3)	3.118 (2)	2 Nb(1)	3.109 (2)	2 P(1)	3.337 (6)
1 Nb(4)	3.286 (3)	1 Nb(1)	3.294 (2)	2 P(5)	3.433 (0)
1 Nb(4)	3.286 (3)	1 Nb(1)	3.294 (2)	2 P(5)	3.433 (0)
1 Nb(7)	3.310 (2)	1 Nb(2)	3.389 (2)	1 P(3)	3.777 (8)
2 Nb(5)	3.433 (0)	2 Nb(10)	3.433 (0)	1 P(6)	3.782 (8)
1 Nb(2)	3.508 (2)	1 Nb(9)	3.643 (3)		

Table 3. Continued.

P(6)–		P(6)–		P(6)–	
2 Nb(1)	2.534 (4)	1 Nb(8)	2.670 (6)	2 P(6)	3.433 (0)
2 Nb(10)	2.555 (4)	1 Nb(6)	2.691 (6)	1 P(1)	3.642 (8)
2 Nb(2)	2.628 (4)	1 Nb(10)	2.835 (6)	1 P(4)	3.736 (8)
				1 P(5)	3.782 (8)

A check on the refinement was made by comparing a difference-Fourier synthesis and an F_o-synthesis for the asymmetric part of the unit cell. The largest positive and negative parts of the difference map were evenly distributed and were less than 20 % of the phosphorus peaks in the F_o-synthesis.

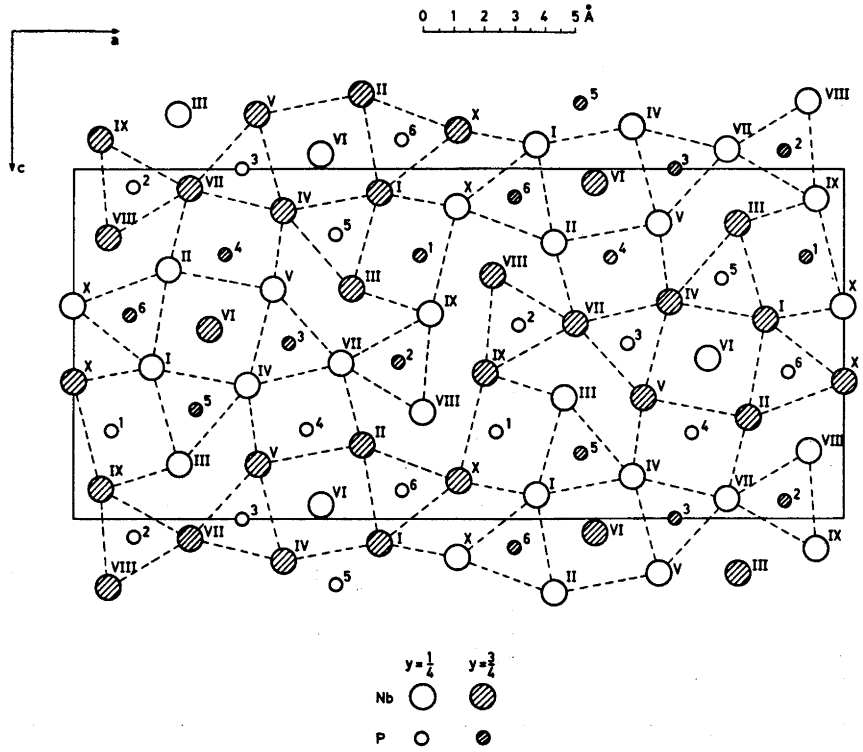

Fig. 1. The crystal structure of Nb_5P_3 viewed along the *b* axis.

Positional and thermal parameters with their standard deviations are given in Table 2. Table 3 gives the interatomic distances and standard deviations in Nb_5P_3. In Table 4 a comparison is made between the observed (absorption and extinction corrected) and the calculated structure factors.

Table 4. Observed and calculated structure factors.

h	k	l	$\|F_o\|$	$\|F_c\|$
0	0	4	260.5	203.2
0	0	6	155.5	158.3
0	0	8	217.7	207.8
0	0	10	94.9	74.6
0	0	14	43.2	39.5
0	0	16	46.8	42.4
0	0	20	122.0	123.6
0	0	22	125.3	118.7
0	0	26	90.2	95.8
0	0	30	38.9	53.6
1	0	3	55.1	51.1
1	0	4	309.7	222.3
1	0	5	120.6	110.7
1	0	6	26.7	23.2
1	0	7	41.6	36.5
1	0	8	193.7	173.0
1	0	9	183.1	175.2
1	0	11	58.9	52.3
1	0	13	92.5	72.6
1	0	14	250.8	226.4
1	0	15	47.3	36.3
1	0	16	44.4	42.5
1	0	17	48.6	47.5
1	0	18	249.1	234.1
1	0	21	109.9	103.8
1	0	22	59.5	50.2
1	0	27	56.2	65.0
1	0	29	80.7	100.2
1	0	30	33.7	35.0
2	0	3	108.9	109.8
2	0	4	37.6	27.7
2	0	5	401.4	320.7
2	0	6	406.1	296.6
2	0	7	158.6	148.3
2	0	8	163.4	155.8
2	0	10	266.4	250.2
2	0	11	181.4	179.6
2	0	13	156.7	135.6
2	0	15	112.3	93.4
2	0	17	74.0	74.9
2	0	18	101.4	94.5
2	0	19	61.3	60.4
2	0	24	58.7	47.6
2	0	25	74.0	83.1
3	0	2	17.9	13.4
3	0	4	36.1	32.5
3	0	5	172.8	173.2
3	0	6	231.2	200.5
3	0	7	268.7	242.5
3	0	8	197.0	186.3
3	0	9	50.0	43.0
3	0	10	180.0	149.9
3	0	11	31.0	27.7
3	0	13	141.4	126.5
3	0	14	76.9	69.0
3	0	15	82.7	72.5
3	0	17	120.3	114.7
3	0	19	143.5	141.0
3	0	22	131.7	122.8
3	0	23	92.8	107.0
3	0	24	53.5	37.1
3	0	25	68.1	67.6
3	0	26	57.2	49.2
4	0	2	29.0	22.7
4	0	3	176.6	167.3
4	0	4	312.8	273.0
4	0	5	599.6	415.5
4	0	6	23.5	19.3
4	0	7	60.6	51.6
4	0	8	421.3	334.2
4	0	9	101.7	86.8
4	0	10	191.7	180.9
4	0	11	176.5	156.4
4	0	14	179.7	160.5
4	0	25	49.4	50.1
4	0	26	69.1	79.5
4	0	27	56.0	59.1
4	0	29	40.0	37.8
5	0	1	36.9	38.6
5	0	2	30.2	27.4
5	0	3	47.3	41.4
5	0	4	311.7	284.8
5	0	5	142.8	137.2
5	0	6	63.1	63.6
5	0	7	252.7	227.3
5	0	8	30.0	21.0
5	0	9	59.3	52.1
5	0	10	270.1	227.0
5	0	11	107.2	97.6
5	0	12	107.0	104.3
5	0	13	259.7	252.9
5	0	14	140.0	126.4
5	0	15	133.2	150.2
5	0	17	69.5	63.4
5	0	18	53.1	46.7
5	0	19	61.6	49.0
5	0	21	48.3	35.7
5	0	24	56.1	54.4
6	0	0	58.2	69.6
6	0	1	42.0	42.1
6	0	2	80.2	86.7
6	0	3	82.0	85.0
6	0	4	67.1	69.7
6	0	5	64.3	60.3
6	0	6	60.5	50.7
6	0	7	235.5	228.4
6	0	8	37.7	30.8
6	0	11	87.9	77.8
6	0	12	120.3	106.0
6	0	13	52.7	45.5
6	0	14	192.7	171.1
6	0	16	45.1	40.2
6	0	17	46.7	44.0
6	0	19	70.1	64.2
6	0	20	67.5	71.1
6	0	22	66.8	56.7
6	0	25	97.9	103.1
7	0	1	46.3	54.9
7	0	2	23.8	22.6
7	0	3	46.5	48.5
7	0	4	623.7	570.4
7	0	5	201.2	204.7
7	0	6	48.6	46.6
7	0	7	61.2	55.5
7	0	8	53.7	42.4
7	0	9	193.2	191.1
7	0	11	103.2	102.0
7	0	13	109.2	94.2
7	0	15	41.1	32.2
7	0	17	49.4	41.9
7	0	18	75.8	78.9
7	0	20	73.8	66.8
7	0	22	125.8	122.5
7	0	27	44.5	37.3
7	0	29	39.5	57.2
7	0	30	41.1	49.9
8	0	0	155.1	221.8
8	0	1	76.8	90.1
8	0	2	101.8	121.5
8	0	3	177.4	189.0
8	0	4	332.7	325.5
8	0	5	64.8	60.9
8	0	6	121.6	111.9
8	0	7	143.0	137.6
8	0	8	94.8	98.5
8	0	9	42.2	35.2
8	0	10	44.1	35.5
8	0	11	74.4	62.9
8	0	13	225.4	192.9
8	0	14	130.8	132.4
8	0	15	138.0	137.7
8	0	16	139.1	110.0
8	0	18	175.2	175.7
8	0	19	96.0	89.8
8	0	21	73.0	63.0
8	0	22	132.3	128.9
8	0	25	63.6	49.6
8	0	29	39.3	41.1
9	0	1	197.6	253.5
9	0	2	174.1	205.9
9	0	3	170.1	202.2
9	0	4	120.6	119.7
9	0	5	203.2	215.1
9	0	6	115.7	104.3
9	0	7	278.8	266.0
9	0	9	161.1	150.4
9	0	10	87.2	80.6
9	0	12	170.3	153.8
9	0	13	112.2	106.7
9	0	14	243.7	204.6
9	0	15	148.8	136.4
9	0	16	101.1	82.5
9	0	17	112.9	84.7
9	0	18	88.0	77.1
9	0	20	78.1	70.5
9	0	21	89.8	91.5
9	0	23	68.8	74.0
10	0	0	183.1	260.9
10	0	1	133.8	176.9
10	0	2	145.4	176.3
10	0	3	394.5	475.4
10	0	4	32.5	33.8
10	0	6	201.9	209.3
10	0	8	181.7	176.1
10	0	9	159.4	160.3
10	0	10	103.4	97.0
10	0	11	160.3	148.1
10	0	13	60.1	47.4
10	0	14	57.3	49.0
10	0	17	63.6	51.5
10	0	18	73.3	71.6
10	0	20	92.0	90.5
10	0	23	59.5	57.1
10	0	27	92.1	106.3
10	0	28	104.6	122.4
10	0	30	44.1	49.9
11	0	1	386.6	472.5
11	0	3	46.3	53.8
11	0	5	30.0	27.2
11	0	6	154.6	160.0
11	0	7	64.4	65.6
11	0	8	128.4	118.8
11	0	9	248.7	231.2
11	0	11	106.5	100.6
11	0	13	97.9	81.7
11	0	14	107.9	97.2
11	0	15	44.8	38.9
11	0	17	83.4	71.3
11	0	19	71.5	62.8
11	0	21	110.3	82.8
11	0	23	94.5	92.3
11	0	24	31.0	31.4
11	0	25	51.5	54.8
11	0	27	54.7	69.1
12	0	0	60.4	63.2
12	0	1	118.5	149.2
12	0	2	248.0	315.1
12	0	3	129.5	155.2
12	0	4	131.8	141.1
12	0	5	206.1	205.4
12	0	8	30.7	20.8
12	0	9	286.5	294.0
12	0	10	171.2	163.4
12	0	11	87.9	79.3
12	0	13	109.1	91.0
12	0	14	123.4	118.5
12	0	17	109.0	97.1
12	0	18	131.8	126.9
12	0	19	67.7	61.9
12	0	23	78.9	79.7
12	0	24	80.8	79.8
12	0	27	58.1	62.3
13	0	3	162.2	192.2
13	0	4	46.7	49.0
13	0	5	232.8	225.0
13	0	8	92.1	93.0
13	0	9	261.9	268.6
13	0	10	94.9	87.7
13	0	11	250.6	236.7
13	0	12	114.3	94.8
13	0	13	152.5	152.0
13	0	14	76.8	56.9
13	0	16	86.6	78.6
13	0	19	165.7	160.1
13	0	20	101.2	99.4
13	0	25	60.6	66.4
13	0	26	57.7	53.9
14	0	1	207.4	209.0
14	0	3	81.7	91.0
14	0	4	123.8	137.3
14	0	5	109.3	121.2
14	0	6	54.1	46.8
14	0	8	183.0	200.3
14	0	9	112.4	103.7
14	0	10	36.2	27.7
14	0	11	166.3	144.8
14	0	13	49.9	42.9
14	0	14	42.0	36.7
14	0	16	63.5	51.8
14	0	17	61.1	56.5
14	0	18	173.3	172.3
14	0	23	72.9	73.1
15	0	2	156.8	201.2
15	0	3	24.2	27.3
15	0	4	162.9	177.1
15	0	5	24.6	23.6
15	0	6	95.6	98.9
15	0	7	65.1	71.1
15	0	8	65.6	64.4
15	0	9	173.0	160.6
15	0	10	100.0	91.7
15	0	11	173.4	151.9
15	0	13	30.2	29.6
15	0	14	97.1	76.8
15	0	15	43.8	33.3
15	0	16	103.7	90.1
15	0	17	111.7	105.0
15	0	18	60.7	54.9
15	0	20	54.9	59.8
16	0	0	24.2	27.3
16	0	1	58.2	67.6
16	0	2	181.3	223.4
16	0	3	151.0	168.4
16	0	4	27.3	29.8
16	0	5	119.7	117.8
16	0	6	92.7	94.2
16	0	7	189.4	195.9
16	0	8	124.3	122.7
16	0	11	171.2	175.9
16	0	14	95.8	77.2
16	0	16	220.1	194.1
16	0	17	89.7	77.3
16	0	19	71.7	67.0
16	0	20	80.1	78.7
16	0	22	57.6	51.4
17	0	1	250.2	336.8
17	0	2	295.1	319.9
17	0	3	39.8	44.3
17	0	4	120.7	151.0
17	0	5	113.2	122.8
17	0	6	85.4	94.9
17	0	7	151.9	165.3
17	0	9	73.8	69.3
17	0	10	47.0	45.1
17	0	11	94.6	236.7
17	0	13	121.2	109.0
17	0	14	108.9	96.4
17	0	16	85.0	62.2
17	0	19	64.6	64.6
17	0	20	98.0	84.7
17	0	23	59.2	48.3
17	0	24	84.9	91.9
18	0	0	91.2	109.6
18	0	2	51.0	54.5
18	0	3	63.7	69.1
18	0	5	255.1	273.1
18	0	6	60.5	57.7
18	0	7	39.6	44.0
18	0	8	81.0	90.5
18	0	9	107.2	103.8
18	0	10	188.3	191.9
18	0	12	188.1	166.3
18	0	14	116.2	103.4
18	0	15	96.8	84.9
18	0	16	157.9	141.5
18	0	18	54.2	47.0
18	0	21	127.0	120.7
18	0	23	70.4	63.9
18	0	24	49.7	76.8
19	0	1	85.0	95.7
19	0	2	48.9	53.0
19	0	3	138.4	158.8
19	0	4	28.2	22.9
19	0	5	159.6	183.3
19	0	6	320.8	346.4
19	0	7	119.1	115.6
19	0	8	32.7	30.4
19	0	9	107.4	77.5
19	0	11	80.6	68.8
19	0	13	170.1	146.8
19	0	14	149.9	124.5
19	0	15	84.5	76.9
19	0	23	92.0	101.6
19	0	28	35.1	51.2
20	0	0	66.9	72.3
20	0	1	98.1	111.3
20	0	2	56.9	66.8
20	0	3	115.5	135.3
20	0	4	42.5	37.2
20	0	5	106.7	121.1
20	0	6	86.7	91.4
20	0	7	29.6	25.4
20	0	8	102.8	95.0
20	0	9	51.2	49.0
20	0	10	65.0	52.3
20	0	11	175.8	166.5
20	0	12	145.6	138.4
20	0	14	48.6	35.6
20	0	15	177.8	163.4
20	0	16	149.9	133.2
20	0	17	85.8	72.0
20	0	20	77.2	73.4
20	0	22	79.8	88.4
20	0	23	47.9	41.5
20	0	24	135.6	154.1
21	0	1	40.7	44.6
21	0	2	166.8	183.7
21	0	3	177.0	240.9
21	0	4	141.8	174.5
21	0	5	196.5	220.1
21	0	6	191.0	242.2
21	0	9	121.0	115.7
21	0	10	116.1	124.5
21	0	11	39.2	40.4
21	0	13	43.1	40.8
21	0	14	59.6	47.8
21	0	17	151.3	130.2
21	0	18	78.4	68.8
21	0	25	53.6	59.3
21	0	29	45.5	72.2
22	0	0	246.3	311.9
22	0	1	102.8	137.2
22	0	4	122.1	133.6
22	0	7	118.9	125.0
22	0	8	101.0	103.2
22	0	9	30.2	32.2
22	0	10	64.5	58.0
22	0	13	70.7	73.2
22	0	14	114.2	93.6
22	0	15	80.7	67.2
22	0	17	75.3	66.0
22	0	21	61.0	48.6
22	0	22	51.9	53.3
22	0	23	58.3	58.8
22	0	25	46.0	46.4
22	0	26	94.5	96.5
23	0	1	108.9	117.4
23	0	2	135.7	160.5
23	0	3	80.7	95.1
23	0	5	82.9	83.7
23	0	8	217.4	236.0
23	0	11	206.8	196.4
23	0	13	93.9	79.0
23	0	15	127.9	116.1
23	0	16	110.1	94.9
23	0	18	81.4	78.0
23	0	19	135.3	130.5
23	0	22	73.4	71.4
24	0	0	90.1	88.2
24	0	2	141.8	159.9
24	0	4	50.9	51.2
24	0	5	65.8	63.3
24	0	7	115.4	126.6
24	0	9	60.8	60.6
24	0	10	192.6	194.8
24	0	11	110.7	100.6
24	0	13	66.0	52.1
24	0	14	51.7	48.1
24	0	15	61.5	55.4
24	0	16	99.1	91.1
24	0	18	61.7	58.0
24	0	19	108.1	102.9
24	0	20	77.8	72.6
24	0	21	88.3	85.6
25	0	1	125.1	158.1
25	0	2	71.6	85.5
25	0	3	73.4	75.0
25	0	4	56.1	57.1
25	0	5	71.9	76.5
25	0	6	46.8	45.2
25	0	7	100.6	103.7
25	0	8	87.3	90.8
25	0	9	176.8	165.8
25	0	10	100.8	92.1
25	0	11	79.2	71.0
25	0	14	79.5	89.4
25	0	15	54.2	41.1
25	0	16	84.5	83.0
25	0	19	86.2	82.7
25	0	20	85.8	93.1
25	0	25	46.9	63.6
26	0	0	162.3	197.2
26	0	1	102.8	113.1
26	0	4	99.1	98.8
26	0	6	162.7	168.7
26	0	7	54.0	56.3
26	0	9	136.0	147.6
26	0	10	142.1	139.4
26	0	11	109.5	98.2
26	0	13	90.0	77.6
26	0	14	62.4	48.2
26	0	16	94.4	90.0
26	0	17	78.2	85.7
27	0	2	148.5	167.2
27	0	3	144.3	157.4
27	0	5	105.5	101.7
27	0	7	185.4	203.6
27	0	8	123.2	118.6
27	0	9	57.5	51.4
27	0	13	56.2	46.0
27	0	14	63.0	61.7
27	0	15	112.7	103.0
27	0	16	88.1	76.6
27	0	18	57.0	43.9
27	0	20	77.8	78.7
27	0	23	71.1	72.9
27	0	25	43.4	46.5
27	0	26	53.3	61.5
27	0	28	45.4	59.9
28	0	0	130.3	139.9
28	0	1	131.4	140.4
28	0	2	264.5	270.0
28	0	3	74.6	75.7
28	0	4	113.5	119.3
28	0	6	83.7	83.9
28	0	7	176.2	202.3
28	0	8	65.8	65.2
28	0	9	91.3	83.0
28	0	11	166.5	168.6
28	0	13	90.4	86.3
28	0	15	88.1	75.0
28	0	16	74.5	64.5
28	0	21	111.0	112.8
28	0	25	51.4	49.0
28	0	26	57.0	70.7
29	0	3	200.5	244.0
29	0	4	114.9	116.6
29	0	5	49.6	45.0
29	0	6	69.7	64.3
29	0	7	131.6	137.1
29	0	9	40.7	36.0
29	0	13	103.4	105.9
29	0	14	64.2	54.8
29	0	17	113.3	113.7
29	0	22	67.0	70.4
30	0	0	103.2	99.8
30	0	1	47.7	52.4
30	0	3	70.3	61.0
30	0	4	191.5	202.9
30	0	6	94.4	89.0
30	0	8	53.2	53.3
30	0	11	154.4	148.1
30	0	15	162.7	173.0
30	0	16	84.0	77.6
30	0	17	73.1	66.0
30	0	21	94.8	88.3
30	0	22	72.4	68.2
31	0	1	116.4	115.8
31	0	2	81.0	75.8
31	0	3	197.7	225.3
31	0	4	50.7	53.0
31	0	6	77.6	71.4
31	0	8	111.4	109.5
31	0	9	42.1	43.9
31	0	10	74.2	72.2
31	0	13	63.5	58.8
31	0	14	72.8	81.4
31	0	15	84.4	82.7
31	0	17	66.1	66.0
31	0	19	78.6	80.9
31	0	23	105.4	119.9
32	0	1	71.2	71.3
32	0	2	68.0	65.3
32	0	3	215.4	230.5
32	0	5	107.8	103.0
32	0	6	112.9	110.3
32	0	10	144.9	154.5
32	0	13	86.0	85.9
32	0	15	57.0	47.6
32	0	18	77.4	80.1
32	0	19	67.1	58.2
33	0	1	37.5	31.6
33	0	2	64.1	63.5
33	0	3	81.2	80.8
33	0	5	156.7	158.1
33	0	8	130.4	123.3
33	0	9	69.0	75.0
33	0	11	98.0	104.1
33	0	14	61.4	48.2
33	0	16	74.1	58.2
33	0	17	71.4	72.1
33	0	19	56.5	50.8
33	0	21	55.7	52.1
33	0	24	39.8	50.9
33	0	26	43.0	31.8
34	0	0	48.8	42.0
34	0	1	42.9	33.0
34	0	2	77.5	72.6
34	0	8	48.1	48.5
34	0	9	76.9	71.6

Table 4. Continued.

h	k	l	$\|F_o\|$	$\|F_c\|$
34	0	10	48.3	52.2
34	0	11	70.4	65.8
34	0	13	89.5	98.6
34	0	14	95.2	83.4
34	0	16	122.0	117.0
34	0	17	55.2	47.6
34	0	18	77.2	68.1
35	0	1	76.2	74.0
35	0	2	112.6	105.0
35	0	3	56.4	60.9
35	0	6	89.1	82.5
35	0	7	98.5	99.1
35	0	8	65.8	66.7
35	0	9	47.6	51.4
35	0	13	132.4	122.7
35	0	19	63.7	53.9
35	0	20	54.1	57.8
35	0	22	58.2	70.2
36	0	0	102.7	101.8
36	0	3	40.6	44.8
36	0	4	115.2	102.6
36	0	5	41.8	38.8
36	0	7	87.3	88.9
36	0	8	226.0	230.3
36	0	9	101.4	104.2
36	0	11	119.9	115.1
36	0	14	89.0	88.6
36	0	18	50.9	39.1
36	0	21	51.1	51.5
37	0	1	45.7	44.5
37	0	2	58.2	51.3
37	0	3	44.0	37.2
37	0	4	71.3	68.7
37	0	5	97.0	85.5
37	0	6	124.0	112.2
37	0	7	88.8	91.3
37	0	10	144.9	151.0
37	0	11	87.8	99.0
37	0	13	56.5	47.5
37	0	17	66.0	63.6
37	0	23	50.2	45.0
37	0	25	57.0	66.1
38	0	0	48.9	47.4
38	0	1	177.8	177.5
38	0	3	56.0	56.2
38	0	4	42.7	41.9
38	0	7	141.9	148.9
38	0	8	51.2	49.6
38	0	17	83.8	85.3
38	0	19	93.1	96.2
38	0	22	46.4	41.6
38	0	24	53.9	73.0
39	0	1	168.2	168.1
39	0	2	130.6	133.0
39	0	3	83.8	78.3
39	0	5	49.3	50.5
39	0	6	69.2	67.3
39	0	8	49.3	42.5
39	0	10	48.3	55.1
39	0	13	65.1	44.4
39	0	17	70.1	49.8
39	0	24	56.1	62.7
39	0	25	50.2	67.5
40	0	0	202.0	208.6
40	0	1	67.0	64.7
40	0	3	74.1	62.7
40	0	4	58.7	69.2
40	0	6	147.6	157.5
40	0	9	106.8	105.5
40	0	11	52.7	69.0
40	0	12	154.2	167.7
40	0	14	63.3	53.0
40	0	16	137.7	129.3
40	0	18	78.4	79.8
40	0	19	65.1	62.3
40	0	22	50.4	36.6
41	0	1	75.1	69.1
41	0	2	122.4	121.5
41	0	4	61.7	59.5
41	0	5	118.8	119.4
41	0	6	110.5	104.3
41	0	7	74.4	73.3
41	0	8	63.1	51.5
41	0	9	51.4	47.5
41	0	10	55.1	44.7
41	0	13	54.3	55.4
41	0	17	50.1	42.4
41	0	20	96.9	106.8
41	0	21	64.0	65.6
42	0	1	50.2	49.7
42	0	2	122.9	121.7
42	0	5	49.0	34.1
42	0	6	62.0	67.9
42	0	7	50.3	46.5
42	0	17	70.3	67.9
42	0	20	48.3	63.1
42	0	24	40.7	50.1
43	0	2	48.5	36.8
43	0	3	61.0	51.8
43	0	4	121.9	122.4
43	0	5	125.5	121.5
43	0	6	47.4	36.3
43	0	8	51.6	36.1
43	0	10	53.0	45.2
43	0	13	54.3	47.6
44	0	2	61.5	58.9
44	0	4	76.4	67.0
44	0	5	104.2	90.7
44	0	6	74.1	71.9
44	0	7	54.2	45.4
44	0	8	95.6	87.4
44	0	9	60.9	59.7
44	0	10	64.1	64.8
44	0	14	136.1	136.7
44	0	15	64.8	70.4
44	0	16	53.1	41.0
45	0	2	47.0	51.4
45	0	3	57.9	50.9
45	0	4	64.2	62.1
45	0	5	114.8	114.8
45	0	10	56.3	54.4
45	0	13	86.8	82.0
45	0	15	94.8	102.1
45	0	23	43.9	55.7
46	0	0	77.0	75.6
46	0	1	55.7	50.8
46	0	3	73.8	76.2
46	0	5	106.3	104.5
46	0	6	48.8	46.0
46	0	9	66.4	61.9
46	0	16	56.2	56.6
46	0	20	92.4	95.1
47	0	4	74.9	49.5
47	0	6	54.9	54.1
47	0	8	114.0	118.8
47	0	13	73.4	76.0
47	0	15	57.6	51.9
47	0	19	53.3	57.1
48	0	1	104.8	97.1
48	0	3	86.3	99.2
48	0	6	72.1	72.0
48	0	9	61.5	63.0
48	0	10	132.0	141.0
48	0	16	56.9	53.9
48	0	20	43.9	49.7
48	0	22	29.9	35.7
49	0	1	97.9	93.3
49	0	2	135.9	141.0
49	0	3	51.8	43.0
49	0	5	74.1	57.8
49	0	8	49.9	56.0
49	0	15	50.3	44.4
49	0	17	52.6	62.0
49	0	18	59.0	56.0
50	0	0	131.1	128.0
50	0	3	97.2	89.7
50	0	4	49.1	47.3
50	0	5	65.4	59.9
50	0	9	95.5	97.5
50	0	15	98.4	101.8
50	0	19	38.2	48.0
50	0	22	33.4	35.4
51	0	3	54.9	55.0
51	0	7	49.5	45.8
51	0	8	76.6	78.7
51	0	10	65.2	59.6
51	0	12	64.1	62.8
51	0	13	63.1	71.7
51	0	16	52.5	49.3
51	0	18	51.4	58.7
52	0	2	95.5	98.6
52	0	4	54.9	54.6
52	0	6	67.2	66.3
52	0	11	90.8	96.5
52	0	14	51.0	44.2
53	0	2	64.7	64.1
53	0	3	85.1	83.2
53	0	7	98.5	109.4
53	0	8	54.5	62.8
53	0	9	97.6	94.5
53	0	10	61.3	54.4
53	0	16	42.3	45.4
53	0	17	39.4	25.4
54	0	4	77.1	81.0
54	0	11	72.2	69.0
54	0	11	61.7	61.3
55	0	3	51.2	51.2
55	0	4	72.5	57.0
55	0	5	53.5	58.3
55	0	6	53.6	54.5
55	0	10	46.3	49.9
55	0	13	53.1	47.4
56	0	0	62.0	71.7
56	0	10	60.0	62.1
57	0	1	91.9	91.6
57	0	4	79.9	76.9
57	0	6	71.8	74.5
57	0	8	64.6	56.2
57	0	13	77.8	87.8
58	0	0	121.4	126.0
58	0	4	86.7	99.8
58	0	15	44.5	51.9
59	0	1	55.9	52.9
59	0	2	55.8	58.4
59	0	6	77.4	73.6
59	0	9	93.1	93.9
60	0	1	63.2	67.1
60	0	2	44.5	46.6
60	0	6	72.1	78.0
60	0	7	64.8	64.8
60	0	13	38.9	23.1
61	0	4	56.9	62.9
61	0	6	55.7	54.0
61	0	7	57.6	57.8
61	0	8	62.7	40.9
61	0	12	54.2	62.7
62	0	0	47.3	53.1
62	0	1	43.8	40.8
62	0	9	70.1	75.8
62	0	10	36.7	22.3
62	0	12	41.8	43.0
63	0	10	38.9	35.7
64	0	1	52.2	58.0
64	0	2	52.1	55.2
64	0	6	39.6	27.2
64	0	9	48.4	49.9
65	0	1	51.9	45.4
65	0	2	44.2	31.0
65	0	4	36.9	42.9
65	0	9	56.7	70.8
66	0	0	79.3	90.3
66	0	2	43.9	34.9
67	0	3	35.7	39.0
67	0	4	55.7	42.3
68	0	2	33.3	40.0
0	1	3	151.9	138.8
0	1	5	241.5	225.2
0	1	7	173.0	159.9
0	1	9	179.0	153.3
0	1	11	77.0	141.5
0	1	13	113.6	94.9
0	1	15	209.2	200.6
0	1	17	141.7	133.3
1	1	2	76.2	88.7
1	1	3	256.9	226.9
1	1	4	621.0	426.5
1	1	5	133.2	129.1
1	1	6	20.1	13.4
1	1	7	138.5	132.2
1	1	9	63.0	48.3
1	1	10	141.6	120.6
1	1	11	77.6	62.3
1	1	12	108.9	115.8
1	1	13	50.1	33.6
1	1	14	125.1	100.7
1	1	15	126.4	109.0
1	1	16	62.7	53.9
1	1	17	64.9	53.8
1	1	18	93.9	77.8
1	1	20	105.6	91.4
1	1	21	104.5	96.2
1	1	22	135.3	119.3
1	1	23	65.4	62.4
1	1	27	52.7	50.3
1	1	29	34.7	29.3
2	1	1	31.6	29.1
2	1	2	156.6	166.7
2	1	3	372.9	386.0
2	1	4	79.0	70.4
2	1	5	155.5	144.5
2	1	6	68.6	53.0
2	1	7	267.3	234.4
2	1	8	140.5	119.6
2	1	9	79.7	63.2
2	1	11	140.6	140.9
2	1	12	36.0	31.5
2	1	14	171.7	156.2
2	1	15	134.0	108.5
2	1	16	67.8	47.9
2	1	18	100.6	82.1
2	1	19	72.7	51.5
2	1	21	73.5	53.1
2	1	22	119.2	111.4
2	1	25	48.4	45.6
2	1	29	56.3	43.4
3	1	1	145.3	52.5
3	1	3	166.2	190.4
3	1	4	76.8	74.7
3	1	5	139.1	143.6
3	1	6	122.0	105.6
3	1	7	128.8	363.1
3	1	8	36.7	20.9
3	1	9	71.0	47.8
3	1	10	192.2	153.4
3	1	11	109.2	98.2
3	1	12	188.7	184.4
3	1	14	123.5	98.2
3	1	15	152.0	136.0
3	1	17	126.2	97.0
3	1	19	48.7	40.8
3	1	24	76.3	70.8
3	1	25	45.6	40.9
3	1	28	56.2	71.1
3	1	30	43.0	47.4
4	1	0	64.0	65.3
4	1	1	89.8	110.9
4	1	2	106.6	101.7
4	1	3	486.3	493.7
4	1	4	204.9	228.9
4	1	5	22.1	10.7
4	1	6	107.6	87.6
4	1	7	73.7	57.7
4	1	8	91.4	72.4
4	1	9	48.5	33.6
4	1	10	316.5	317.4
4	1	11	320.3	309.5
4	1	12	76.2	63.1
4	1	13	83.4	66.4
4	1	14	103.2	85.3
4	1	15	109.3	89.5
4	1	16	41.9	28.5
4	1	17	109.5	102.7
4	1	18	76.2	67.1
4	1	20	62.2	55.5
4	1	21	92.8	89.9
4	1	25	80.9	84.9
4	1	26	55.7	55.4
4	1	28	53.6	62.1
5	1	1	152.6	205.6
5	1	2	16.1	14.9
5	1	3	313.9	389.8
5	1	4	152.6	180.0
5	1	6	32.3	23.6
5	1	7	186.5	186.3
5	1	8	133.9	119.3
5	1	9	99.9	85.5
5	1	10	146.7	129.8
5	1	11	99.5	79.3
5	1	15	107.9	81.5
5	1	17	134.8	115.1
5	1	18	123.4	106.5
5	1	19	65.3	61.4
5	1	20	47.0	34.7
5	1	22	101.0	94.6
5	1	23	63.2	52.5
5	1	25	129.9	141.8
5	1	26	69.1	76.6
5	1	27	50.3	57.2
5	1	28	55.5	43.2
5	1	29	66.8	78.7
6	1	0	241.5	438.2
6	1	1	28.1	28.1
6	1	2	143.8	184.1
6	1	3	62.5	64.7
6	1	4	32.4	31.7
6	1	5	64.0	58.7
6	1	7	352.2	329.9
6	1	8	297.3	252.6
6	1	9	294.3	235.0
6	1	10	48.4	40.5
6	1	11	170.3	170.6
6	1	12	99.8	69.3
6	1	13	88.0	75.3
6	1	14	122.5	117.7
6	1	16	169.0	159.8
6	1	17	151.9	146.4
6	1	18	127.3	113.8
6	1	19	101.5	92.7
6	1	20	52.7	40.8
6	1	22	53.6	49.7
6	1	25	45.5	50.9
6	1	26	57.6	52.5
6	1	27	41.0	40.5
6	1	31	33.4	32.4
7	1	1	98.2	137.0
7	1	2	409.7	539.3
7	1	3	22.8	16.8
7	1	4	92.3	93.8
7	1	5	39.8	30.1
7	1	6	60.2	55.8
7	1	7	64.5	59.9
7	1	9	65.9	53.4
7	1	11	231.7	180.8
7	1	12	108.9	89.1
7	1	14	41.8	40.6
7	1	16	67.3	64.0
7	1	18	47.9	42.1
7	1	19	75.3	69.0
7	1	20	161.8	149.8
7	1	21	88.3	98.0
7	1	27	52.1	61.2
7	1	29	46.7	49.9
8	1	0	275.7	522.6
8	1	1	194.2	290.0
8	1	2	220.7	303.9
8	1	3	101.4	118.7
8	1	4	23.8	19.6
8	1	5	78.9	80.6
8	1	6	194.4	192.2
8	1	8	258.9	235.8
8	1	10	122.4	117.3
8	1	11	187.5	193.4
8	1	12	132.6	128.3
8	1	13	48.6	42.5
8	1	14	39.6	30.4
8	1	16	178.4	182.9
8	1	17	46.8	59.6
8	1	19	113.3	99.9
8	1	20	53.0	35.0
8	1	21	50.0	41.9
8	1	22	48.0	35.6
8	1	23	47.7	48.0
9	1	1	181.3	277.8
9	1	2	82.9	98.9
9	1	3	16.8	13.0
9	1	4	96.2	101.8
9	1	5	25.6	14.3
9	1	6	83.5	130.9
9	1	8	86.1	87.2
9	1	10	144.0	123.0
9	1	12	76.1	70.3
9	1	13	70.8	52.4
9	1	14	130.7	131.1
9	1	15	46.3	32.8
9	1	16	60.8	56.7
9	1	17	64.8	64.6
9	1	18	161.2	149.1
9	1	20	50.4	36.4
9	1	22	78.2	66.7
9	1	23	71.7	64.7
9	1	29	49.1	44.9
9	1	30	73.3	100.4
10	1	0	194.1	305.0
10	1	1	76.5	97.7
10	1	3	20.6	22.4
10	1	4	60.5	58.8
10	1	5	45.6	40.6
10	1	6	51.2	52.1
10	1	8	75.8	73.9
10	1	11	172.9	151.3
10	1	12	162.2	159.4
10	1	15	130.8	137.1
10	1	17	72.5	62.1
10	1	18	70.8	57.9
10	1	21	101.4	99.8
10	1	23	53.3	54.4
10	1	24	75.9	73.3
10	1	28	37.2	45.2
11	1	1	76.9	102.0
11	1	2	22.8	19.4
11	1	3	22.5	19.6
11	1	4	63.0	63.9
11	1	5	22.0	17.1
11	1	6	470.4	462.0
11	1	7	256.2	227.3
11	1	8	159.5	150.1
11	1	9	49.7	63.6
11	1	10	122.3	98.8
11	1	11	55.9	49.8
11	1	12	134.6	123.2
11	1	14	127.2	102.2
11	1	16	67.2	65.8
11	1	17	63.5	59.5
11	1	18	78.4	73.1
11	1	19	90.2	85.6
11	1	22	48.2	38.1
11	1	23	63.1	49.8
12	1	0	39.0	46.7
12	1	1	126.7	178.7
12	1	2	27.2	25.2
12	1	3	87.0	86.1
12	1	4	20.9	18.0
12	1	5	274.0	307.5
12	1	6	55.6	49.4
12	1	7	39.3	30.3
12	1	8	345.0	275.8
12	1	9	109.7	106.3
12	1	12	56.8	40.5
12	1	13	112.9	109.7
12	1	14	144.3	129.6
12	1	15	140.3	133.0
12	1	16	67.7	51.3
12	1	21	138.2	147.3
12	1	22	48.2	33.3
12	1	24	98.0	110.9
12	1	25	93.0	101.5
12	1	26	50.0	41.8
12	1	27	39.9	37.4
13	1	1	42.6	48.5
13	1	2	33.8	38.6
13	1	3	49.7	54.1
13	1	4	293.4	348.4
13	1	5	121.7	127.0
13	1	7	138.2	137.0
13	1	8	30.2	27.5
13	1	9	134.4	137.7
13	1	10	153.3	123.4
13	1	11	85.7	82.8
13	1	13	319.9	282.7
13	1	14	46.3	42.5
13	1	16	78.4	67.0
13	1	21	48.4	40.4
13	1	22	48.2	35.1
13	1	23	81.0	71.6
13	1	24	46.4	41.7
13	1	26	54.1	52.1
14	1	0	85.7	105.3
14	1	1	42.1	46.4
14	1	2	74.2	90.4
14	1	3	39.9	40.7
14	1	4	31.1	30.7
14	1	5	188.0	185.6
14	1	6	75.1	76.7
14	1	7	61.5	52.2
14	1	8	88.1	79.2
14	1	9	30.1	20.4
14	1	10	182.2	197.2
14	1	11	180.2	180.4
14	1	14	77.3	62.6
14	1	15	181.1	178.6
14	1	16	152.8	145.5
14	1	18	52.4	51.9
14	1	20	76.6	69.3
14	1	25	49.8	61.5
14	1	26	49.2	44.2
14	1	27	59.1	67.1
14	1	31	71.2	105.3
15	1	2	333.0	438.6
15	1	3	213.7	281.5
15	1	5	87.9	96.5
15	1	5	191.2	198.0
15	1	6	112.2	114.1
15	1	7	31.9	31.6
15	1	9	57.2	57.6
15	1	10	35.4	28.5
15	1	11	167.1	154.6
15	1	12	94.4	90.9
15	1	13	111.9	110.3
15	1	14	99.3	95.8
15	1	15	65.6	53.1
15	1	17	116.7	106.1
15	1	18	72.9	104.9
15	1	20	109.4	97.1
15	1	23	47.5	49.4
15	1	24	89.9	92.3
15	1	28	39.1	39.3
16	1	0	113.8	149.6
16	1	1	122.9	168.5
16	1	2	50.0	52.9
16	1	3	51.4	105.5
16	1	4	47.6	50.7
16	1	5	24.5	19.5
16	1	6	271.7	285.4
16	1	7	111.9	111.1
16	1	8	143.4	138.8
16	1	10	52.5	40.7
16	1	11	95.7	91.4
16	1	13	51.3	180.7
16	1	15	75.0	62.9
16	1	16	182.7	167.4
16	1	17	61.5	59.0
16	1	19	140.0	138.7
16	1	20	118.7	105.5
16	1	21	128.1	137.5
16	1	26	91.3	95.5
16	1	27	43.3	53.2
16	1	28	48.3	50.1
17	1	1	204.3	284.4
17	1	2	63.3	62.6
17	1	3	59.7	60.5
17	1	5	203.6	200.9
17	1	6	45.3	41.7
17	1	7	138.5	137.8
17	1	8	307.5	324.3

Table 4. Continued.

h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$
17	1	9	151.6	126.7	23	1	15	120.3	101.7	30	1	10	96.2	97.4	38	1	4	65.6	53.8	47	1	21	41.6	57.7
17	1	12	126.6	129.5	23	1	16	97.9	79.6	30	1	11	96.9	78.6	38	1	5	97.0	86.5	47	1	23	28.9	62.6
17	1	15	79.1	76.6	23	1	17	151.3	138.7	30	1	12	103.0	99.2	38	1	6	98.6	100.6	48	1	0	139.2	125.6
17	1	16	86.4	79.7	23	1	18	65.5	59.8	30	1	13	47.0	33.2	38	1	8	73.9	67.4	48	1	5	61.8	65.8
17	1	19	48.4	44.4	23	1	19	76.7	67.1	30	1	15	66.0	66.0	38	1	9	71.7	67.2	48	1	8	54.9	46.2
17	1	23	53.0	57.6	23	1	20	91.4	91.1	30	1	19	75.2	58.1	38	1	10	69.3	59.4	48	1	11	49.3	42.6
17	1	25	46.5	45.9	23	1	22	47.8	48.8	30	1	24	72.3	79.9	38	1	11	86.1	78.6	48	1	12	73.9	77.1
17	1	29	52.1	50.7	23	1	23	46.2	49.9	30	1	26	55.3	59.3	38	1	12	49.3	41.6	48	1	16	73.4	71.6
18	1	0	102.7	121.1	23	1	24	99.7	90.3	31	1	4	41.2	42.0	38	1	14	50.8	59.4	48	1	17	69.7	72.8
18	1	2	133.9	149.3	24	1	0	199.0	292.3	31	1	6	38.5	33.9	38	1	15	91.3	87.1	48	1	18	62.4	58.4
18	1	3	86.1	85.7	24	1	1	140.3	191.5	31	1	8	91.6	95.4	38	1	17	50.4	45.1	48	1	22	53.6	47.0
18	1	4	31.1	25.2	24	1	2	34.3	32.9	31	1	10	84.4	81.2	38	1	20	70.1	73.7	49	1	1	63.6	53.4
18	1	5	97.9	99.9	24	1	3	192.3	260.0	31	1	11	126.5	111.6	38	1	25	33.1	26.9	49	1	4	70.6	60.3
18	1	6	42.9	37.9	24	1	4	132.7	140.2	31	1	14	69.3	56.1	39	1	1	44.9	37.3	49	1	5	102.4	99.6
18	1	7	43.6	30.1	24	1	5	73.6	75.7	31	1	15	50.1	34.4	39	1	2	42.5	38.4	49	1	12	77.2	72.1
18	1	8	119.0	104.2	24	1	6	32.4	29.6	31	1	16	94.9	95.5	39	1	3	123.2	118.1	49	1	13	85.4	92.9
18	1	9	36.6	25.2	24	1	7	95.7	97.5	31	1	18	132.2	133.8	39	1	4	45.9	44.1	49	1	18	63.5	76.3
18	1	10	182.6	183.2	24	1	8	47.4	41.1	31	1	19	88.7	99.0	39	1	5	60.1	58.6	49	1	21	40.0	28.3
18	1	12	118.4	118.6	24	1	9	45.7	42.1	31	1	25	48.8	31.9	39	1	7	48.0	47.2	50	1	0	108.7	106.5
18	1	13	77.1	69.7	24	1	10	94.8	96.1	32	1	0	151.0	171.6	39	1	8	78.4	87.0	50	1	1	63.8	61.1
18	1	16	89.9	72.8	24	1	12	232.9	222.8	32	1	1	165.9	204.8	39	1	9	52.5	48.9	50	1	2	53.9	52.5
18	1	17	87.1	79.2	24	1	13	100.8	84.1	32	1	2	115.1	113.8	39	1	13	71.4	83.1	50	1	6	111.8	101.8
18	1	21	77.5	72.7	24	1	14	116.5	111.5	32	1	3	41.5	38.6	39	1	15	83.4	80.2	50	1	20	38.3	45.8
18	1	26	58.0	62.4	24	1	17	55.1	54.5	32	1	4	89.9	82.0	39	1	17	56.0	53.8	51	1	3	85.8	83.0
18	1	27	37.9	45.3	24	1	19	112.6	108.2	32	1	6	68.7	73.8	39	1	22	55.6	57.9	51	1	4	48.4	49.4
18	1	28	57.0	72.0	24	1	21	48.8	42.1	32	1	8	41.4	33.3	39	1	23	55.6	57.8	51	1	5	68.8	65.4
18	1	30	40.0	40.3	24	1	22	81.1	78.0	32	1	9	95.7	86.3	40	1	1	70.0	73.5	51	1	6	145.3	135.5
19	1	1	150.8	207.5	24	1	23	46.0	43.6	32	1	10	169.0	173.0	40	1	2	66.8	70.5	51	1	17	43.6	43.1
19	1	2	169.2	223.7	24	1	26	36.0	37.8	32	1	11	108.7	100.4	40	1	3	142.5	133.5	51	1	20	40.3	43.2
19	1	3	149.0	163.4	25	1	1	144.2	151.9	32	1	12	79.5	81.6	40	1	5	128.3	120.8	52	1	2	53.9	51.9
19	1	4	93.5	94.9	25	1	2	75.3	85.9	32	1	13	53.7	52.0	40	1	11	99.1	99.4	52	1	7	70.6	75.8
19	1	7	172.5	175.9	25	1	3	55.0	65.0	32	1	18	111.5	110.8	40	1	15	76.9	58.3	52	1	8	63.9	64.7
19	1	8	33.1	27.4	25	1	4	69.2	69.8	32	1	19	76.9	76.9	40	1	17	96.9	95.3	52	1	12	85.8	93.7
19	1	9	160.9	153.6	25	1	5	146.9	167.0	32	1	22	63.4	54.2	40	1	22	50.6	44.9	52	1	13	60.4	58.1
19	1	11	85.9	77.4	25	1	6	140.2	153.3	32	1	23	44.8	50.4	41	1	1	58.0	44.2	52	1	14	106.9	113.3
19	1	12	115.8	107.8	25	1	7	49.7	45.1	32	1	26	42.6	40.9	41	1	2	44.0	32.8	52	1	15	56.2	56.1
19	1	13	42.1	35.6	25	1	8	134.1	157.3	33	1	1	100.0	105.1	41	1	5	101.8	87.3	52	1	17	57.8	55.2
19	1	14	71.9	73.6	25	1	9	76.2	67.1	33	1	2	47.9	47.8	41	1	7	52.0	45.6	53	1	3	74.2	61.4
19	1	16	111.7	96.2	25	1	10	119.9	120.1	33	1	3	37.9	37.1	41	1	8	133.5	130.7	53	1	4	101.4	102.9
19	1	18	48.3	45.5	25	1	11	86.7	72.9	33	1	4	167.0	180.9	41	1	10	59.7	53.9	53	1	7	50.8	50.1
19	1	19	51.8	42.9	25	1	12	115.6	118.1	33	1	5	58.8	56.2	41	1	12	79.5	84.6	53	1	12	45.5	39.3
19	1	20	49.0	49.1	25	1	14	216.2	214.1	33	1	7	121.7	125.9	41	1	13	64.5	61.8	53	1	15	61.7	68.5
19	1	21	110.4	105.2	25	1	16	69.6	56.0	33	1	8	155.6	154.2	41	1	15	67.4	71.9	53	1	16	59.5	67.2
19	1	22	59.1	62.4	25	1	17	76.8	71.1	33	1	9	43.3	31.6	41	1	17	69.6	67.8	53	1	17	47.4	51.0
19	1	28	52.2	55.5	25	1	18	81.2	66.3	33	1	10	75.9	72.3	41	1	21	71.3	78.4	53	1	19	38.7	46.2
20	1	0	102.0	114.3	25	1	26	49.9	52.3	33	1	11	51.3	52.0	42	1	1	144.5	33.7	54	1	1	105.0	106.7
20	1	1	84.9	104.5	25	1	28	31.7	31.6	33	1	14	133.1	126.7	42	1	3	122.5	102.7	54	1	3	73.8	73.0
20	1	2	34.6	30.1	25	1	29	24.5	26.6	33	1	16	73.2	61.1	42	1	4	70.0	61.5	54	1	4	63.2	63.2
20	1	3	148.0	174.6	25	1	30	36.6	60 1	33	1	18	93.5	97.6	42	1	6	46.3	33.2	54	1	10	46.1	32.5
20	1	4	25.4	19.5	26	1	0	101.0	125.1	33	1	20	111.3	119.8	42	1	8	58.4	54.7	54	1	14	45.4	42.9
20	1	5	242.2	243.2	26	1	1	67.4	85.5	33	1	24	59.5	58.7	42	1	9	135.5	129.2	54	1	17	45.1	18.8
20	1	6	45.8	44.3	26	1	2	45.7	47.6	34	1	0	165.1	190.2	42	1	10	127.7	121.5	55	1	7	57.2	50.6
20	1	7	116.6	113.6	26	1	3	111.8	136.9	34	1	1	144.0	162.0	42	1	11	49.6	39.4	55	1	9	78.1	73.8
20	1	8	35.9	34.1	26	1	4	115.0	143.7	34	1	2	57.8	54.3	42	1	12	61.5	40.3	55	1	10	77.0	78.8
20	1	9	152.1	154.8	26	1	6	137.5	150.3	34	1	3	38.0	38.2	42	1	13	70.1	52.7	55	1	11	44.4	42.0
20	1	10	56.8	54.3	26	1	6	37.1	33.3	34	1	4	153.5	167.3	42	1	14	56.6	55.7	55	1	16	39.4	46.3
20	1	11	161.9	163.1	26	1	11	44.3	33.2	34	1	5	107.3	99.3	42	1	15	82.3	73.4	55	1	18	36.6	39.8
20	1	12	40.5	48.2	26	1	12	73.9	66.9	34	1	7	103.3	102.1	42	1	16	66.5	62.3	56	1	1	91.3	90.3
20	1	13	124.2	128.1	26	1	13	73.4	64.0	34	1	9	81.2	87.2	42	1	17	116.5	113.4	56	1	1	79.7	82.8
20	1	14	47.1	35.5	26	1	15	92.6	75.9	34	1	10	119.1	109.0	42	1	19	56.6	57.7	56	1	3	57.3	44.4
20	1	15	48.7	41.6	26	1	16	91.7	83.7	34	1	12	67.6	57.4	43	1	2	151.3	151.5	56	1	4	66.0	63.0
20	1	17	117.0	117.2	26	1	17	85.1	82.3	34	1	14	68.8	68.8	43	1	5	79.0	88.3	56	1	7	45.9	51.0
20	1	18	73.1	61.6	26	1	19	55.9	42.9	34	1	17	67.8	57.2	43	1	9	90.1	86.1	56	1	9	98.7	103.7
20	1	21	86.9	89.2	26	1	22	72.9	71.7	34	1	21	74.5	77.1	43	1	11	49.8	37.0	56	1	11	66.8	53.8
20	1	23	46.9	33.3	26	1	24	50.4	64.8	34	1	23	45.6	51.8	43	1	12	77.7	59.6	56	1	13	43.4	40.1
20	1	27	58.6	66.9	26	1	26	68.3	86.9	34	1	25	57.1	68.9	43	1	13	106.8	68.5	56	1	19	31.3	30.5
20	1	29	28.9	47.1	26	1	28	32.5	41.4	34	1	28	37.1	68.3	43	1	17	58.5	63.9	57	1	1	85.2	90.2
21	1	1	91.1	115.1	27	1	1	105.7	118.5	35	1	1	75.8	80.1	43	1	19	43.4	44.3	57	1	4	74.6	73.0
21	1	2	87.4	96.7	27	1	3	98.4	110.2	35	1	2	41.6	43.3	44	1	0	118.7	123.8	57	1	7	45.0	33.6
21	1	3	162.1	192.5	27	1	4	35.3	28.5	35	1	3	57.8	51.7	44	1	1	129.4	131.4	57	1	14	41.2	37.6
21	1	4	57.8	62.8	27	1	5	132.5	145.0	35	1	4	99.3	93.1	44	1	3	49.0	36.7	58	1	2	66.2	59.8
21	1	5	46.9	49.6	27	1	6	37.6	33.0	35	1	6	93.5	94.9	44	1	4	61.6	46.3	58	1	6	44.4	33.1
21	1	6	30.9	23.2	27	1	7	36.6	34.6	35	1	9	67.1	62.3	44	1	5	94.5	83.4	58	1	12	55.8	55.9
21	1	9	244.9	264.6	27	1	8	95.3	98.5	35	1	10	81.4	75.7	44	1	6	47.4	55.0	58	1	13	46.9	45.1
21	1	11	140.2	140.3	27	1	11	99.4	96.5	35	1	11	51.1	77.0	44	1	7	50.8	47.4	58	1	16	36.9	13.7
21	1	12	113.2	110.6	27	1	13	91.9	77.8	35	1	13	163.2	164.1	44	1	8	86.1	75.3	59	1	4	44.0	45.6
21	1	13	76.1	71.3	27	1	16	85.2	77.2	35	1	14	131.3	126.1	44	1	10	112.0	112.3	59	1	8	57.8	52.6
21	1	14	76.3	61.7	27	1	17	53.3	49.2	35	1	17	57.2	45.3	44	1	15	84.9	79.2	59	1	9	43.8	39.0
21	1	16	84.3	71.8	27	1	19	81.4	76.1	35	1	18	58.8	53.8	44	1	19	58.2	57.6	59	1	10	60.8	59.9
21	1	17	63.9	48.9	27	1	20	76.4	70.0	35	1	20	57.9	52.0	44	1	24	46.1	13.2	59	1	17	26.2	9.4
21	1	19	87.6	88.8	27	1	22	98.8	117.9	35	1	24	39.0	38.8	45	1	1	93.4	91.0	60	1	3	59.3	52.5
21	1	21	54.7	51.5	27	1	25	67.4	72.6	35	1	26	40.9	45.5	45	1	2	85.9	91.4	60	1	4	60.9	44.4
21	1	22	76.2	69.0	27	1	26	48.2	58.4	36	1	0	73.3	68.6	45	1	3	68.3	53.4	60	1	6	49.5	48.8
21	1	23	46.7	46.6	27	1	29	21.8	35.3	36	1	1	52.8	53.3	45	1	4	57.7	46.6	60	1	7	51.0	56.8
21	1	29	38.6	37.9	28	1	0	59.7	57.2	36	1	2	136.2	137.4	45	1	7	54.0	53.2	60	1	11	73.1	80.6
22	1	0	74.5	78.2	28	1	3	54.5	53.8	36	1	3	126.9	138.6	45	1	8	69.1	63.8	60	1	13	56.2	46.3
22	1	1	143.5	181.7	28	1	6	47.9	41.8	36	1	4	126.9	108.5	45	1	9	108.4	100.2	60	1	15	65.1	66.3
22	1	2	111.6	143.5	28	1	7	195.3	227.1	36	1	5	156.1	155.8	45	1	10	112.3	114.1	61	1	1	42.4	35.8
22	1	3	63.1	70.6	28	1	8	206.8	215.3	36	1	6	131.7	142.0	45	1	13	113.3	108.0	61	1	3	100.7	111.4
22	1	4	50.6	51.3	28	1	13	112.5	111.6	36	1	7	43.2	38.4	45	1	17	48.9	45.0	61	1	6	50.1	43.4
22	1	5	101.7	100.7	28	1	14	63.1	52.3	36	1	8	58.1	53.7	45	1	18	58.1	49.9	61	1	7	79.3	91.1
22	1	6	140.4	148.7	28	1	15	83.8	77.2	36	1	9	77.0	73.3	45	1	19	46.3	55.7	62	1	3	45.6	43.0
22	1	7	161.6	171.6	28	1	16	78.0	56.3	36	1	10	85.7	88.4	45	1	22	62.0	77.5	62	1	11	54.9	67.8
22	1	8	71.5	66.3	28	1	17	66.9	51.1	36	1	12	68.9	66.6	45	1	23	45.1	34.3	63	1	1	39.7	35.7
22	1	9	70.3	70.2	28	1	24	81.6	85.1	36	1	15	110.1	108.0	46	1	1	46.4	68.3	63	1	5	68.3	74.2
22	1	11	39.6	28.8	29	1	1	67.9	79.9	36	1	23	55.3	64.7	46	1	3	50.1	43.9	63	1	7	45.4	42.5
22	1	12	93.4	93.8	29	1	2	96.0	97.5	37	1	1	43.2	36.9	46	1	4	62.9	65.9	63	1	8	72.5	82.7
22	1	13	43.2	43.3	29	1	3	58.9	58.2	37	1	2	75.4	74.3	46	1	5	146.9	141.5	63	1	12	64.2	56.2
22	1	18	117.6	121.5	29	1	4	84.1	92.7	37	1	3	62.0	58.4	46	1	6	94.7	88.6	63	1	14	34.0	15.7
22	1	19	70.1	61.2	29	1	5	36.0	28.0	37	1	4	81.2	77.9	46	1	8	107.8	99.6	64	1	1	40.6	33.2
22	1	20	99.6	98.6	29	1	6	244.9	285.2	37	1	5	109.6	108.5	46	1	9	55.2	48.9	64	1	3	58.4	63.2
22	1	23	46.5	56.7	29	1	10	153.3	155.2	37	1	6	102.9	108.5	46	1	12	84.9	73.1	64	1	4	51.3	36.2
22	1	26	43.9	51.4	29	1	11	77.3	77.5	37	1	9	70.8	72.1	46	1	15	48.7	39.7	64	1	11	40.3	30.5
22	1	27	65.0	80.4	29	1	12	108.7	108.2	37	1	10	79.9	88.8	46	1	17	45.1	34.7	65	1	2	100.5	126.6
23	1	2	106.6	132.4	29	1	15	55.3	42.7	37	1	12	79.8	80.0	46	1	19	55.1	66.2	65	1	6	38.2	36.1
23	1	3	30.9	32.4	29	1	16	122.2	107.3	37	1	13	49.9	48.0	46	1	24	30.2	15.5	66	1	1	38.9	40.2
23	1	4	182.2	247.2	29	1	20	49.2	45.1	37	1	14	50.7	30.7	47	1	1	87.5	85.9	66	1	2	36.6	30.4
23	1	5	88.0	85.4	29	1	21	47.9	44.7	37	1	15	83.5	84.5	47	1	2	113.8	104.2	66	1	4	37.6	35.9
23	1	6	85.7	92.8	29	1	23	44.0	45.9	37	1	16	73.3	57.8	47	1	3	50.5	35.7	66	1	10	34.3	38.8
23	1	7	40.4	32.9	30	1	0	181.1	217.6	37	1	19	97.0	89.3	47	1	4	74.0	71.0	67	1	1	34.6	38.5
23	1	8	68.0	63.2	30	1	1	59.1	65.5	37	1	20	46.1	38.5	47	1	5	56.5	57.6	67	1	7	37.2	24.5
23	1	10	40.7	32.9	30	1	3	74.5	72.8	37	1	23	83.5	93.2	47	1	7	59.8	55.9	69	1	1	36.0	34.1
23	1	12	46.8	35.4	30	1	4	36.0	39.1	37	1	24	49.2	40.9	47	1	8	73.9	72.3	69	1	2	26.8	23.2
23	1	13	92.1	87.9	30	1	5	56.8	45.4	38	1	1	80.7	76.8	47	1	9	55.2	58.0	69	1	4	42.8	26.1
23	1	14	77.6	68.8	30	1	9	50.6	45.9	38	1	2	64.4	60.6	47	1	14	75.4	71.4	69	1	6	41.6	62.6
																				69	1	7	31.8	17.7

DESCRIPTION AND DISCUSSION OF THE Nb_5P_3 STRUCTURE

A projection of the structure on (010) is shown in Fig. 1. The phosphorus atoms are situated in triangular prisms of niobium atoms with one to three additional niobium atoms outside the quadrilateral faces of the prisms. (The prisms are indicated by broken lines in Fig. 1.) The triangular faces of the prisms are parallel to the *b* axis for the phosphorus atoms P(1) and P(4), and perpendicular to the *b* axis for the remaining phosphorus atoms. The shortest P – P distance exceeds 3.3 Å. The coordination in Nb_5P_3 is summarized in Table 5. The niobium atoms have 14 to 16 close neighbours, and the phosphorus atoms 7 to 9 close neighbours.

Table 5. The coordination in Nb_5P_3. Neighbours within 3.0 Å are listed for the phosphorus atoms and within 3.7 Å for the niobium atoms.

Niobium atom	1	2	3	4	5	6	7	8	9	10
Nb-neighbours	10	11	10	10	11	10	10	10	11	11
P-neighbours	5	5	5	5	5	4	5	4	5	5
Phosphorus atom	1		2		3	4	5		6	
Nb-neighbours	7		9		8	7	8		9	
P-neighbours	–		–		–	–	–		–	

The coordination about the metal atoms in the metal-rich niobium phosphides varies almost linearly with composition for non-metal neighbours from on average 3.0 in Nb_3P through 4.7 in Nb_7P_4 to 4.8 in Nb_5P_3, and varies for metal neighbours from 11.3 (Nb_3P) through 10.6 (Nb_7P_4) to 10.4 (Nb_5P_3). The sum of the average metal coordination numbers is of the order of 15 for Nb_7P_4 and Nb_5P_3. Such a high average coordination number is not common in transition metal phosphides and seems to occur only for metals of group IV and group V.

In Ref. 11 the geometrical aspects of the packing of atoms, denoted A, in the corners of a trigonal prism around a central atom, C, with additional atoms, B, outside the quadrilateral faces of the prism are discussed. The calculated values of Table 6, where a comparison is made with Nb_3P and Nb_7P_4, refer to the assumptions made in Ref. 11 using a radius for the niobium atoms of 1.47 Å (the Goldschmidt metal radius for 12-coordination) and a radius for the phosphorus atoms of 1.10 Å (the tetrahedral covalent radius). From the values in Table 6 it is evident that the distribution of interatomic distances is

Table 6. Average interatomic distances d_{AA}, d_{AB}, d_{AC}, and d_{BC} in Nb_3P,[3,17] Nb_7P_4,[4] and Nb_5P_3.

Phosphide	$d_{AA}/2\ R_{Me}$ obs.	calc.	$d_{AB}/2\ R_{Me}$ obs.	calc.	$d_{AC}/(R_{Me}+R_X)$ obs.	calc.	$d_{BC}/(R_{Me}+R_X)$ obs.	calc.
Nb_3P	1.13		1.02		1.00		1.06	
Nb_7P_4	1.13	1.14	1.02	1.00	1.00	1.00	1.06	1.05
Nb_5P_3	1.13		1.02		1.00		1.06	

closely similar in the three phosphides and in good accord with the size-factor principle.

Nb_5P_3 is structurally very closely related to a large number of metal-rich phases formed between transition metals and the non-metals phosphorus, arsenic, sulphur, and selenium. Characteristic features, common for all these structures, are the triangular prismatic metal atom coordination about the non-metal atoms, and the dense packing of the metal atoms.

In Nb_5P_3, the atom denoted Nb(6) is surrounded by eight niobium atoms situated in the corners of a slightly distorted cube. Similar "islands" of body-centered cubic metal atom arrangement occur also in phosphide, arsenide, sulphide, and selenide representatives of the Nb_7P_4,[4] Ta_2P,[12] Nb_4As_3,[13] and $Nb_{21}S_8$[14] structure types. Franzen and coworkers, who have thoroughly explored the chalcogenides in question, suggest [15] that the occurrence of elements of the b. c. c. coordination in these compounds might be correlated with the tendency for the elemental metals to form the b. c. c. structure. The transition temperature from h. c. p. to b. c. c. is very much lower for titanium and zirconium than for hafnium. According to Franzen *et al.*,[15] the greater tendency for titanium and zirconium to form the b. c. c. structure would explain why Ti_2S, Ti_2Se, Zr_2S, and Zr_2Se crystallize in the Ta_2P structure type, while Hf_2S and Hf_2Se crystallize in a different structure where no b. c. c. structure elements are discernible. The situation becomes more complex when the structures of phosphides and arsenides are also taken into account. Hf_2P [16] and Hf_2As [5] both crystallize in the Ta_2P structure type. Hf_5As_3 is isostructural with Nb_5P_3, and there is also evidence for the occurrence of an Nb_7P_4 type compound in the Hf–As system.[5] The crystallography of the phosphides and arsenides of titanium and zirconium has not yet been thoroughly explored, but from the information available it appears that phases containing b. c. c. structure elements do not occur more abundantly for these metals than for hafnium.

As a general observation concerning the transition metal phosphides, arsenides, sulphides, and selenides one might say that structures exhibiting b. c. c. structure elements are confined to the compounds formed with the titanium and vanadium group metals. Chromium, molybdenum, and tungsten, all of which crystallize in the b. c. c. structure, apparently do not form such compounds.

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