deficiency of one-third of eighteen, namely six, represents the six negative charges on the complex anion.

This structure, like that of the 6-heteropoly compounds, involves octahedral coordination of oxygen atoms around the central ion, whereas the structure of the upper limiting series of 12-compounds and the structure recently published by Dawson (1953) for the more highly condensed 18-anions or doubled 9-anions is based on tetrahedral coordination of the central ion.

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The Crystal Structure of Rhodium Silicide, RhSi

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Rhodium–silicon melts containing between 50 and 75 atomic % silicon when quenched give only two phases: RhSi and Si. The RhSi has the FeSi structure, *B*20 of the *Strukturbericht*, with space group $P2_13$ and $a_0 = 4{\cdot}675 \pm 0{\cdot}001$ Å. Four rhodium atoms and four silicon atoms are in positions $(x, x, x;\ x+\frac{1}{2}, \frac{1}{2}-x, \bar{x};\ \circlearrowleft)$. The parameters, $x_{\mathrm{Rh}} = 0{\cdot}144 \pm 0{\cdot}003$ and $x_{\mathrm{Si}} = 0{\cdot}840 \pm 0{\cdot}007$, were determined by trial and error.

There is a real difference between the rhodium parameter in RhSi and the Fe parameter in FeSi but the uncertainty in the Si parameter is such as not to rule out the assignment of bond numbers, as done by Pauling & Soldate in the FeSi case.

Introduction

The melts discussed in this paper were prepared by B. T. Matthias and E. Corenzwit as part of a program of investigation of superconductivity in intermetallic compounds (Matthias, 1952, 1953). Mixtures of rhodium and silicon were put into magnesia crucibles and heated by induction in a helium atmosphere to about 1600±200°C. for a few minutes. The temperature of the melts dropped rapidly when heating was discontinued. Melts with 50, 56, 60, 67 and 75 atomic % silicon were prepared. Matthias found that none of these contained material that was superconducting above 1·3° K.*

Determination of the structure

Interpretation of powder photographs of the five melts indicated that two phases (silicon and one with a primitive cubic unit) existed in the melts. The photograph of the 50% melt showed in addition faint lines from a phase which was richer in rhodium. A comparison of the powder pattern of the primitive cubic unit material with tabulated diffraction patterns (Frevel, 1942) suggested isomorphism with the FeSi structure (Phragmén, 1923; Wever & Moeller, 1930; Borèn, 1933; Pauling & Soldate, 1948) and single-crystal work subsequently confirmed this.

A fragment of dimensions 0·08 × 0·08 × 0·55 mm. which was very nearly a single crystal was aligned by the Hendershot (1937) method. Weissenberg photographs were taken using Mo $K\alpha$ radiation. The longest exposure given the zero level was 18 hr. Three simultaneous photographs were taken. The relative intensities of the $(hk0)$ reflections were estimated visually by comparison with a carefully prepared intensity scale.

The symmetry of the X-ray photographs is that of T_h–$m3$ and reflections of the type $(h00)$, h odd, are absent. The space group uniquely indicated therefore is T^4–$P2_13$. The cubic unit cell has $a_0 = 4{\cdot}675 \pm 0{\cdot}001$ Å as measured from the powder diffraction photographs of the 50 atomic % melt. If one assumes four molecules of RhSi per unit cell, the calculated density is 8·5 g.cm.$^{-3}$, in good agreement with the pycnometrically determined density of 8·3 g.cm.$^{-3}$.

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* Private communication.

Table 1. *Comparison of calculated with observed* (*hk*0) *reflection amplitudes*

(*hk*0)	$\|F_o\|$	F_c
110	62·4	54·0
120	73·5	59·0
130	58·6	−48·7
140	14·0	−14·1
150	15·8	−14·0
160	20·5	−23·0
170	24·2	27·3
180	< 15·8	13·0
190	< 15·8	− 1·7
1,10,0	< 15·8	5·3
1,11,0	< 14·0	− 9·3
1,12,0	< 11·2	5·6
200	53·0	−51·1
210	146	−123
220	13·0	11·7
230	34·4	−39·4
240	19·1	17·7
250	73·4	73·2
260	14·0	12·8
270	< 14·0	− 7·4
280	< 15·8	− 3·7
290	20·5	−27·2
2,10,0	< 15·8	5·1
2,11,0	< 14·0	− 8·8
310	18·6	24·0
320	64·1	−58·0
330	25·1	−32·6
340	17·7	15·1
350	< 12·1	− 3·7
360	23·2	28·8
370	< 15·0	13·5
380	< 15·8	−15·6
390	< 15·8	− 2·0
3,10,0	< 15·0	− 7·3
3,11,0	< 13·0	− 4·4
3,12,0	< 9·1	7·5
400	89·2	−89·5
410	46·4	44·6
430	14·0	16·4
440	49·2	60·6
450	30·6	−31·3
460	30·2	−29·2
470	< 15·0	− 4·1
480	< 15·8	6·9
490	< 15·8	6·0
4,10,0	< 15·0	14·1
4,11,0	< 12·0	4·3
510	24·2	−31·4
520	12·1	−15·8
530	30·7	36·6
540	< 13·0	7·4
550	< 14·0	10·4
560	< 15·0	5·3
570	18·6	−23·0
580	< 15·8	− 2·2
590	< 15·8	1·9
5,10,0	< 14·0	− 0·5
5,11,0	< 11·0	9·0
600	48·3	48·6
610	25·1	34·8
630	15·8	−14·6
650	27·9	−27·1
660	22·3	17·8
670	< 15·8	1·6
680	< 15·8	7·1
690	< 15·0	11·3
6,10,0	< 13·0	− 9·3
6,11,0	< 9·3	− 3·5
710	< 14·0	− 2·2
720	32·6	32·0
730	< 15·0	3·4
740	< 15·8	−11·0
750	< 15·8	− 1·4
760	< 15·8	−16·4
770	< 15·8	− 0·8
780	< 15·0	10·7
790	< 13·0	1·3
7,10,0	< 10·0	4·3
800	14·9	17·5
810	27·9	−26·1
830	< 15·8	−10·0
850	20·5	21·2
870	< 15·0	− 2·6
880	< 13·0	4·4
890	< 11·0	− 8·7
10,0,0	17·7	14·0

Notes: (1) All (*hk*0) amplitudes with *h* odd are imaginary.
(2) All (*hk*0) amplitudes with *h* even are real.
(3) When *h* and *k* are both even, $F_{hk0} = F_{kh0}$.
(4) Of the total number of (*hk*0) reflections possibly observable, all those that were observed are listed. A representative number of those too weak to be observed is also listed.

The rhodium and silicon atoms occupy positions $4(a)$, $(x, x, x;\ \frac{1}{2}+x,\ \frac{1}{2}-x,\ \bar{x};\ \circlearrowright)$ (see *International Tables for X-ray Crystallography*, 1952). The parameters were determined by trial and error. Discrepancy factors ($R = \Sigma\|\|F_o\|-\|F_c\|\|\div\Sigma\|F_o\|$) for the (*hk*0) reflections were calculated for several combinations of parameters, as shown in Fig. 1. The minimum of 11·0% occurs for $x_{\text{Rh}} = 0{\cdot}144$ and $x_{\text{Si}} = 0{\cdot}840$. This value of the discrepancy factor compares very well with the 10·5% obtained from the data for FeSi (Pauling & Soldate, 1948). Based on Fig. 1, the conservative limits of error of $\pm 0{\cdot}003$ for rhodium and $\pm 0{\cdot}007$ for silicon were assigned.

The comparison of observed and calculated structure amplitudes is shown in Table 1. A temperature factor $\exp\left[-0{\cdot}83\times10^{-16}(\sin\theta/\lambda)^2\right]$ was applied to the calculated structure-factor values. The value of the linear absorption coefficient for RhSi (Mo $K\alpha$ radiation) is 188 cm.$^{-1}$. This means that for the observable smallest-angle reflections the relative error due to absorption is about 20% as compared with the observable highest-angle reflections. Thus the important corrections would be only on the smaller-angle reflections which are few in number. Absorption corrections were not made.

It will be noticed in Table 1 that for several of the

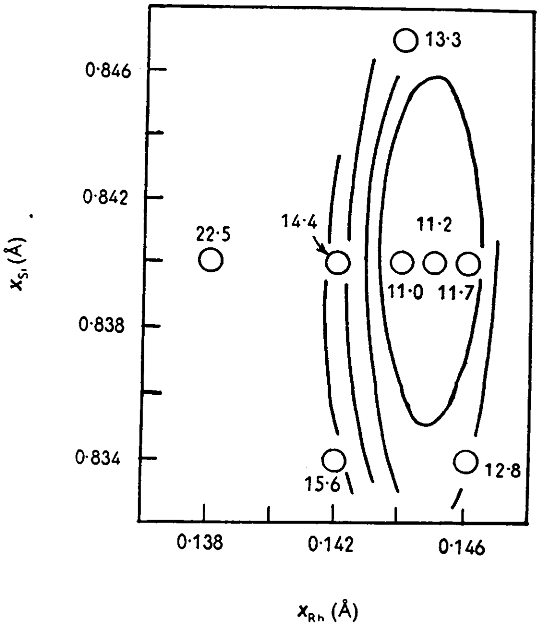

Fig. 1. Discrepancy factors for trial pairs of rhodium and silicon parameters.

intense low-angle reflections, which should be most affected by absorption, the observed value of the reflection amplitude is greater than that of the calculated. The estimated intensities of the most intense spots had to be multiplied by large film factors to put them on the same basis as the weak ones. Since there is apt to be error in the determination of the film factor as well as in the estimated intensities, the largest errors are in the intense low-angle $|F_o|$ values. Therefore it is not surprising that the discrepancies for these low-angle reflections are as given in Table 1. The overall agreement between calculated and observed amplitudes confirms the correctness of the structure as reported.

Table 2. *Interatomic distances between nearest neighbors in* RhSi

Atom	Number of equivalent neighbors	Neighboring atom	Distance (Å)
Rh	1	Si	2·46±0·08
Rh	3	Si	2·44±0·05
Rh	3	Si	2·57±0·05
Rh	6	Rh	2·87±0·01
Si	6	Si	2·90±0·02

The important interatomic distances are given in Table 2.

This work has shown that there is a real difference between the rhodium parameter in RhSi and that of iron (0·1370±0.0020) in the FeSi structure (see Fig. 1).

We wish to thank J. Andrus for taking the powder diffraction photographs used in this study.

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The Crystal Structure of Creatine Monohydrate

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Creatine monohydrate crystallizes in the monoclinic system, space group $P2_1/c$, $a = 12{\cdot}50$, $b = 5{\cdot}01$, $c = 12{\cdot}16$ Å, $\beta = 109°$. The crystal structure was solved through the calculation of the Patterson projection along [010] and trial structure factors, and the atomic positions have been refined by Fourier series and least-squares calculations. The molecular structure found corresponds to that of a zwitter ion; the atoms of the substituted guanidine group lie nearly in one plane which is inclined at 84½° to a second plane containing the carboxyl group. The molecules are held in layers in the crystal by a very complete system of hydrogen bonds.

Introduction

Creatine plays an important role in the metabolism of proteins and is therefore of considerable biological interest. As there are no accurate data on the type of substituted guanidine group present in this molecule, we considered it worth while to study its structure in some detail.

Experimental

Creatine crystallizes as the monohydrate in well formed monoclinic prisms, elongated along [010] and showing {001}, {100} and {011} dominating. The crystals are described by Groth (1906–19), who gives references to measurements by several authors. Allowing for a change of nomenclature (a and c reversed), the quoted axial ratio, $b{:}c$, varying from 2·39 to 2·44, agrees well with the X-ray data; $a{:}c$ generally differs widely but is based on doubtful measurements on small faces. The correct relation was derived by Fletcher (1887), who gives

$$a{:}b{:}c \text{ (goniometric)} = 2{\cdot}48{:}1{:}2{\cdot}40$$
$$(\text{cf. } a{:}b{:}c \text{ (X-ray data)} = 2{\cdot}49{:}1{:}2{\cdot}43)\,.$$

The preliminary crystallographic data are as follows:

$$a = 12{\cdot}50\pm0{\cdot}05,\quad b = 5{\cdot}01\pm0{\cdot}02,\quad c = 12{\cdot}16\pm0{\cdot}04 \text{ Å},$$
$$\beta = 109° \text{ (goniometric)};$$
$$\text{space group, } P2_1/c;\ n = 4;$$