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# 唐有祺文集

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1951—1952

1951 - 1952

## A Cubic Structure for the Phase Pt<sub>3</sub>Cu\*

Schneider & Esch (1944) proposed for the intermetallic phase with composition about  $Pt_3Cu$  an orthorhombic structure which is derived from a face-centered cubic arrangement. They found that this structure gave satisfactory agreement with X-ray intensities on their powder photographs, and that no other well-defined arrangement of the atoms gave as good agreement. However, the proposed structure for  $Pt_3Cu$  may well not be the right one, in as much as an orthorhombic structure such as this would very probably distort the cubic lattice perceptibly; yet no splitting of the diffraction lines was observed. I have found (Tang, 1950) that the data for this phase, which covers the composition range  $63\% \sim 88\%$  of platinum, are compatible with a cubic structure of the type  $ABC_6$  with

A at 
$$(0,0,0;0,\frac{1}{2},\frac{1}{2};\frac{1}{2},0,\frac{1}{2};\frac{1}{2},\frac{1}{2},0)+0,0,0;$$

B at 
$$+\frac{1}{2}$$
,0,0;

and C at 
$$+\frac{1}{4}$$
,  $\frac{1}{4}$ , 0;  $+0$ ,  $\frac{1}{4}$ ,  $\frac{1}{4}$ ;  $+\frac{1}{4}$ , 0,  $\frac{1}{4}$ ;  $+\frac{1}{4}$ ,  $\frac{1}{4}$ ,  $\frac{1}{2}$ ;

$$+\frac{1}{2}, \frac{1}{4}, \frac{1}{4}; +\frac{1}{4}, \frac{1}{2}, \frac{1}{4},$$

as shown in Fig. 1. The structure factors for the three types of unmixed *hkl* are:

 $F_{hkl} = 4(f_A + f_B + 6f_C)$ , if hkl are all multiples of 4, or all even but not divisible by 4;

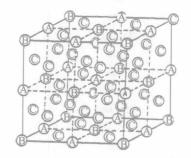


Fig. 1 The cubic structure  $ABC_6 Z=4$ 

 $F_{hkl} = 4(f_A + f_B - 2f_C)$ , if hkl are all even and only one or two of them are divisible by 4;  $F_{hkl} = 4(f_A - f_B)$ , if hkl are all odd.

<sup>\*</sup> Contribution no. 1527 from the Gates and Crellin Laboratories of Chemistry, California Institute of Technology. Acta Crystallographica, 1951, 4, 377.

For the alloy containing 72.5% of platinum A is Pt,B is Cu and C is the statistical atom (Pt<sub>0.8</sub>Cu<sub>0.2</sub>). The cubic structure PtCu(Pt<sub>0.8</sub>Cu<sub>0.2</sub>)<sub>6</sub> gives at least as good agreement with X-ray intensities as Schneider & Esch's structure. The calculated intensities and those observed by Schneider & Esch are given in Table 1.

Table 1 Calculated and observed intensities  $for^{(a)}$   $PtCu(Pt_{0,\,8}\,Cu_{0,\,2}\,)_6$ 

hkl	$\sin \theta$	$PLHF^2 \times 10^{-6}$	I
111	(0, 173)	9	_
200	(0.200)	2	
220	0. 284	2	vvw
311	0. 335	11	υω
222	0.347	350	ms
400	0.402	200	mw
331,402	0.441	ldelw ond 111 keldme	Hodro How 1 H and
224	(0.510)	g earrum 2 automit l	vergennant <del>. They loun</del>
333,511	0.528	rr 1-22 Gay 8 days a sh	registro ve
440	, 0.565	250	S
531	0.592	10	vw
600,442	(0.600)	has statem 2 is littled only	nt mare dis <u>pi</u> erale to con-
620	(0.632)	petitibode 2 gainibas a	n sej dan <del>gp</del> ang sana
335	0,653	phones Holiston	rand and vow the last
622	0.663	410	US .
444	0.691	135	m
551,711	0.713	10	The www
406	(0.720)	2	
624	0.749	4	vvw
731,553	0.768	15	vw
800	0.798	113	mw
733	0.818	6	vw
802,446	0, 830	5	vvw
228,660	0.845	3	vvw
555,157	0.864	16 10 20 10 16 10 20 10 20 20 20 20 20 20 20 20 20 20 20 20 20	l a sid w
662	0.870	626	White bearings at the some
840	0.893	680	vs vs
911,735	0.910	28	W W
842	(0, 917)	7	
664	(0.938)	the fished High and Marian	at within
913	0.952	29	Sept 1 Col vw
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<sup>(</sup>a) P = polarization factor; L = Lorentz factor; H = multiplicity factor, m = moderate; s = strong; v = very; w = weak

It is not particularly surprising that the alloy in question, which corresponds to a maximum in the electric conductivity-composition curve and which gave maximum intensities to superstructure lines (Schneider & Esch, 1944) contains the statistical atom (Pt<sub>0.8</sub>Cu<sub>0.2</sub>). As a matter of fact it is the composition (Pt<sub>0.8</sub>Cu<sub>0.2</sub>)Cu<sub>3</sub> in the cubic phase of nominal composition PtCu<sub>3</sub> which corresponds to a high peak in the conductivity diagram and also gives maximum intensities to superstructure lines (Schneider & Esch, 1944; Johansson & Linde, 1927). The statistical atom (Pt<sub>0.8</sub>Cu<sub>0.2</sub>) may involve some short-range order which is not detectable by ordinary X-ray diffraction methods.

The complex conductivity and X-ray data for the other alloys between 63% and 88% of platinum in the cubic phase of nominal composition PtCuPt<sub>6</sub> are understandable in terms of cubic structures as described by the formula

$$(Pt_{1-x}Cu_x)(Pt_yCu_{1-y})(Pt_{1-z}Cu_z)_6$$

Where x, y and z are less than 0. 3, and 100(7-x+y-6z)/8 is the atomic percentage of platinum in an alloy.

In view of the fact that there is no detectable splitting of the cubic lines and of the satisfactory agreement with X-ray intensities given by the cubic structure PtCu(Pt<sub>0.8</sub> Cu<sub>0.2</sub>)<sub>6</sub>, this structure is probably the correct one for the annealed Pt—Cu alloy containing 72.5% of platinum, and the cubic phase covering the entire range of  $63\% \sim 88\%$  of platinum is probably similarly based upon the cubic structure  $ABC_6$ .

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# Some X-ray Measurements on Single Crystals of Hamster Carbonmonoxy-hemoglobin\*

The interpretation of X-ray photographs obtained from stationary crystals having large unit cells was first described by Crowfoot & Schmidt (1945) in their study of a derivative of tobacco mosaic virus. More recently the method was used by Carlisle & Dornberger (1948) in the examination of crystals of bushy stunt virus. In the present investigation this method was applied to the analysis of small-angle rotation photographs of hamster carbonmonoxy-hemoglobin.



Fig. 1 The crystalline habit of hamster carbonmonoxy-hemoglobin

Crystals of carbonmonoxy-hemoglobin were prepared from citrated hamster blood. The red cells were separated, washed, and hemolysed as described by Drabkin (1946), and the resulting hemoglobin solution was saturated with carbon monoxide. Crystallization was carried out at  ${}^{4}\text{C}$ . by dialysis against a solution of half-saturated (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution adjusted to pH=6. 5. Within a few days prismatic crystals about 0.5mm. across were obtained; their appearance suggested orthorhombic sym-

metry (Fig. 1). Selected wet crystals were sealed in thin capillary tubes and photographed at  $4^{\circ}$ C. with Cu K radiation, in a cylindrical camera of 5cm, radius. The crystal was oscillated through an angle of 1,  $0\sim1$ ,  $5^{\circ}$ . The photographs were measured and interpreted by a modification of the method of Crowfoot & Schmidt adapted to cylindrical films. The calculations are analogous to those described by Carlisle & Dornberger.

The analysis of about twenty X-ray photographs gave the following crystallographic

<sup>\*</sup> Contribution No. 1568 from the Gates and Crellin Laboratories. From Acta Crystallographica, 1951, 4:564.

### information:

Laue symmetry:  $D_{2h}$ -mmm.

Unit cell dimensions: a=123, b=88, c=60 A.

Lattice type: Primitive.

Space group: the following planes were in position to reflect

(h00) Observed: 600, 800, 10, 0, 0, 12, 0, 0, 14, 0, 0, 16, 0, 0;

Absent: 700,900,11,0,0,13,0,0,15,0,0.

(0k0) Observed: 060,080;

Absent: 070,090.

(001) Observed: 004:

Absent: 005,006.

Probable space group derived from the systematic absences and from the stereochemical nature of proteins:  $P2_12_12_1$ .

It is interesting to observe that Boyes-Watson, Davidson & Perutz (1947) have described a form of horse methemoglobin which shows absences characteristic of the space group  $P2_12_12_1$  and which has cell dimensions a=122,b=82,4,c=63,7 A. From this similarity of space group and dimensions it might be inferred that there is some similarity between the arrangement of the molecules in crystals of this form of horse methemoglobin and in crystals of hamster carbonmonoxy-hemoglobin.

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# X-ray Observations on Single Crystals of Carbon-monoxy-hemoglobin from Human Fetal Blood\*

### Introduction

A crystallographic difference between adult and fetal human hemoglobin was described by Haurowitz(1). Kendrew and Perutz(2) have used X-ray diffraction techniques to demonstrate a difference in the crystal form and probably in the molecular composition of adult and fetal hemoglobin of sheep. Although Perutz (3) has published the results of X-ray measurements of adult human carbonmonoxy-hemoglobin (carbonylhemoglobin), no comparable information from crystals of human fetal hemoglobin has appeared.

Reasons for the lack of single crystal X-ray diffraction data for fetal hemoglobin have become evident during the course of the present investigation. Although well-formed crystals similar to those described by Haurowitz can be obtained from cord blood of infants without great difficulty, the X-ray data which can be obtained from them are meager. Even when maintained at temperatures near 0°C., the crystals appeared to be very unstable and to undergo rapid degenerative changes during X-ray photography. Indeed, only one satisfactory photograph could be obtained from an individual crystal, a circumstance which placed a severe limitation upon the determination of its crystallographic symmetry and dimensions. Repeated attempts to obtain optical figures from the crystals were unsuccessful.

In spite of the difficulties inherent in the material, it has been possible to obtain sat-

<sup>\*</sup> 本文为作者与 Hams H. Zinsser, Fellow of the John Simon Guggenheim Memorial Foundation 合作而成. 原载: Arch. Biochem. Biophys., 1951, 34:81.

isfactory measurements of repetition distances in two directions in the crystals, and from an interpretation of the photographs to derive a possible unit cell. Some confirmation of this unit is provided by determinations of the density of the crystals and of their salt and water contents.

### Experimental Methods and Results a banqueta and

Crystals of carbonmonoxy-hemoglobin were prepared from cord blood of full-term and premature infants. The red cells were washed and hemolyzed essentially by the method of Haurowitz (1). The resulting hemoglobin solution was saturated with carbon monoxide and dialyzed against a comparatively large volume of saturated solution of ammonium sulfate buffered at pH 6.96 by addition of potassium phosphate<sup>①</sup>. The crystallization, photography, and density determinations were carried out at about 4°C. Within 10 days, crystals about 0.5mm. across were generally obtained. The majority of the crystals appeared to be tiny dodecahedra (Fig. 1a) but many of them had the habit of square prisms (Fig. 1b). Between crossed Nicol prisms the crystals extinguished sharply parallel and perpendicular to the prism axis. This observation and the general appearance of the crystals suggested possible orthorhombic or tetragonal symmetry.

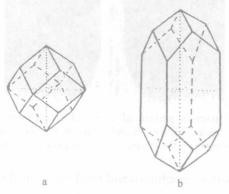


Fig. 1 Characteristic habits of carbonmonoxy-hemoglobin crystals obtained from fetal blood

Crystals were mounted and sealed in thin-walled glass capillaries as described by Perutz(4). Diffraction patterns were obtained with unfiltered Cu—K radiation. Exposures were made for 10~15 hr. at about 15 ma, and 35 kv. A crystal was mounted so that it could be rotated about the prism axis, and an attempt was made to determine the presence of a possible fourfold axis of symmetry by taking photographs along two directions

 $<sup>\</sup>bigcirc$  To 1 l. of saturated (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> was added 50 ml. of buffer (47, 2g. K<sub>2</sub>HPO<sub>4</sub> and 26, 5 g. KH<sub>2</sub>PO<sub>4</sub>l.).

at 90° to one another. Unfortunately, after the first exposure, we did not succeed in obtaining a satisfactory pattern at 90° to it because of the degenerative changes mentioned above.

A typical photograph taken with the X-ray beam perpendicular to the prism axis (c-axis) is shown in Fig. 2. In all photographs the crystal remained stationary; the patterns were interpreted in a manner already described by Crowfoot and Schmidt (5). The difference in the vertical diameters of two concentric circles shown in Fig. 2 gave 198 A. as one of the repetition distances perpendicular to the c-axis. Similar measurements on a photograph taken with the X-ray beam approximately parallel to the c-axis gave 101 A. as the length of the c-axis. Examination of other photographs indicated that spacings of about 198 A. in directions perpendicular to the c-axis made 90° angles with one another and that they were shorter than spacings at a 45° angles to them by a factor of approximately  $\sqrt{2}$ . These observations are compatible with a tetragonal unit of structure having approximate dimensions a=198 A., c=101A.

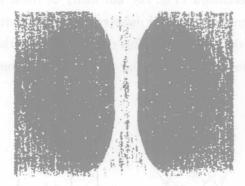


Fig. 2 X-ray diffraction pattern of a crystal of carbonmonoxy-hemoglobin from fetal blood, made with Cu—K radiation, in a cylindrical camera (radius, 5.0 cm.), c-axis vertical; the crystal was stationary throughout the exposure

The percentages of salt, hemoglobin, and total water in the crystals were determined by the method of Drabkin(8). Concordant values for the density of the crystals were obtained by flotation in bromobenzene—xylene and in copper sulfate solution, and also by comparing the rate of fall of the crystals in the mother liquor with that of selected fragments of plastic of known density and dimensions. These data are collected in Table I. They were used to calculate the number of molecules of hemoglobin (mol. wt. 66,000) per unit cell as follows:

Volume of unit cell= $(198 \text{ A})^2(101 \text{ A.})=3,960,000\times10^{-24} \text{ cm}^3$ . Weight of unit cell= $(3,960,000)(1.214)\times10^{-24}=4,807,000\times10^{-24} \text{ g.}$ Weight of Hb per unit cell× $(4,807,000)(36.2)=1,740,000\times10^{-24} \text{ g.}$