

CRYSTALLOGRAPHIC STUDY OF RASHLEIGHITE, A MEMBER OF THE TURQUOIS GROUP¹

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ABSTRACT

The lattice constants of rashleighite were determined from X-ray diffraction data on polycrystalline material. The mineral has a triclinic cell similar to the turquoise cell and provides an example of the limit of the usefulness of Ito's method to determine the cell constants from powder X-ray diffraction patterns.

The results from structure factor calculations of two model structures for rashleighite confirm that this mineral can be described as a substitutional solid solution of turquoise and chalcociderite.

INTRODUCTION

Minerals of the turquoise group can be described by the general formula $XY_6(PO_4)_4(OH)_8 \cdot 4H_2O$, where X corresponds to Cu, Zn, Ca, or Fe and Y to Al or Fe. Chalcociderite (Schaller, 1912; Graham, 1948), faustite (Erd *et al.*, 1953), and ceruleolacite (Fischer, 1958), are minerals where each site X and Y is occupied by only one of the ions mentioned above. The relationship between these minerals and turquoise is schematized below

Element in X	Element in Y	
	Al	Fe
Cu	turquoise	chalcociderite
Zn	faustite	
Ca	ceruleolacite	

There is also evidence of partial replacement of elements in X and Y , giving different isomorphous series. There is not a standard criterion, however, for differentiating members of these series. For example, minerals where X corresponds to iron and copper, in almost equivalent amounts, are simply called "turquoise" (Hintze, 1933, page 941, analyses numbers 1, 5, 6, 7, 16). There is also evidence of some substitution of PO_4 by AsO_4 in some minerals belonging to this group (see for example Table 1, analyses 6, 7, 9).

A complete series of minerals with a chemical composition intermediate between those of turquoise and chalcociderite is known. The general

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TABLE 1. CHEMICAL ANALYSES OF THE MEMBERS OF THE TURQUOIS-CHALCOSIDERITE SERIES

	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
FeO			0.71			0.32				
CaO				0.54	0.57			0.87		
CuO	9.78	9.00	7.54	7.10	8.81	7.72	7.87	6.82	8.15	8.06
Al ₂ O ₃	37.60	36.50	32.75	18.24	28.01	21.63	20.84	10.45	4.45	
Fe ₂ O ₃		0.21	4.28	2.74	9.58	20.09	21.29	34.26	42.81	48.56
P ₂ O ₅	34.90	34.13	33.72	48.94	34.90	31.59	28.60	33.82	29.93	28.77
As ₂ O ₅						0.48	2.11		0.61	
H ₂ O	17.72	20.12	18.96	17.10	17.13	17.40	16.45	13.70	15.00	14.61
SiO ₂			2.24	1.37		0.16	2.25			
MgO						0.12				
Total	100.00	99.96	100.20	96.03	99.00	99.51	99.41	99.92	100.95	100.00

(1) Ideal turquoise, $\text{CuO} \cdot 3\text{Al}_2\text{O}_3 \cdot 2\text{P}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$

(2) Turquoise, Campbell County, Virginia, USA (Schaller, 1912)

(3) Turquoise, Los Cerrillos, New Mexico, USA (Jung, 1932)

(4) Henwoodite, West-Phoenix Mine, Cornwall, England (Collins, 1876)

(5) Henwoodite, Redruth, Cornwall, England, (Fischer, 1961)

(6) Rashleighite, Castle-an-Dinas, St. Columb Major, Cornwall, England (Russell, 1948) Analyst J.A. Smythe

(7) Rashleighite, Bunny Mine, St. Austell, Cornwall, England (Russell, 1948) Analyst J.A. Smythe

(8) Alumo-chalcosiderite, Schneckenstein, Saxony, Germany (Jahn and Gruner, 1933)

(9) Chalcosiderite, Phoenix Mine, Cornwall, England (Maskelyne, 1875) Analyst W. Flight

(10) Ideal chalcosiderite, $\text{CuO} \cdot 3\text{Fe}_2\text{O}_3 \cdot 2\text{P}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$

formula for this series is $\text{Cu}(\text{Al}, \text{Fe})_6(\text{PO}_4)_4(\text{OH})_8 \cdot 4\text{H}_2\text{O}$. Minerals such as ferri-turquoise (Robinson, 1942), "henwoodite" (Fisher, 1961), rashleighite (Russell, 1948), and alumo-chalcosiderite (Jahn and Gruner, 1933), have been reported as being intermediate members. Of all the known minerals belonging to this group, only turquoise and chalcosiderite occur as single crystals. Each is triclinic $P\bar{1}$, and of them, only the structure of turquoise has been completely determined (Cid-Dresdner, 1965a). Chalcosiderite seems to be isostructural with turquoise (Graham, 1948; Cid-Dresdner, 1965b).

RASHLEIGHITE

According to Russell (1948) and to our data, rashleighite does not present single crystals suitable for X-ray diffraction work. Pieces as small as 2 to 3 tenths of a millimeter have been shown to consist of

several crystals. Due to this fact there are no crystallographic data available, although Russell made reference to the existence of powder diffraction diagrams which show that it is probably isostructural with turquoise and chalcociderite. A chemical analysis on material obtained from Bunny Mine, St. Austell, Cornwall, cited by Russell (1948) gives the formula $\text{CuO} \cdot 3\frac{1}{2}(\text{Al}, \text{Fe})_2\text{O}_3 \cdot 2\text{P}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$, with molecular ratios $\text{Al}_2\text{O}_3/\text{Fe}_2\text{O}_3 = 0.2044/0.133$. This formula can also be written as $\text{Cu}(\text{Al}, \text{Fe})_6(\text{PO}_4)_4(\text{OH})_8 \cdot 4\text{H}_2\text{O} + \text{H}_2\text{O} + \frac{1}{2}(\text{Al}, \text{Fe})_2\text{O}_3$; the first part of this formula allows us to describe rashleighite as a substitutional structure of turquoise where 2 Al atoms have been replaced by Fe atoms in the unit cell. The extra water molecule and the excess of $(\text{Al}, \text{Fe})_2\text{O}_3$ could be due to hydrated aluminum-iron impurities admixed in the material analyzed, as reported for turquoise (Graham, 1948). Most of turquoise chemical analyses reported the existence of 5 water molecules in the unit cell; however a 3-dimensional crystal structure determination showed that only 4 water molecules belonged to the structure. (Cid-Dresdner, 1965a). If rashleighite is to be considered isostructural with turquoise its only unknown parameters would be the dimensions of the unit cell and the proportion of Al and Fe at each Al site.

We thought that the determination of the lattice constants of rashleighite could be done by Ito's method (Ito, 1950). The limitation of this method for triclinic crystals lies in the difficulty of a correct indexing of the powder diffraction patterns, due to multiple superpositions, and also in the fact that the results are reliable only if the interplanar spacings can be measured to at least 3 significant figures.

The indexing of rashleighite powder diffraction patterns was achieved by comparison with other previously-indexed patterns of turquoise and chalcociderite obtained under the same experimental conditions. The use of a Philips diffractometer provided the required accuracy in the determination of the interplanar spacings from powder diffraction data.

UNIT-CELL DIMENSIONS OF RASHLEIGHITE

Specimens of rashleighite from Castle-an-Dinas, St. Columb Major, Cornwall, and of chalcociderite from Wheal Phoenix, Linkinhorne, Cornwall, were kindly provided by the Department of Mineralogy of the British Museum of Natural History. Specimens of crystalline turquoise from Campbell County, Virginia (Schaller, 1912), already used in the determination of the structure of turquoise (Cid-Dresdner, 1965a), were obtained from Dr. G. Switzer, curator of the U.S. National Museum.

Powder diffraction patterns of turquoise, rashleighite, and chal-

cosiderite were obtained on a Unicam single-crystal X-ray goniometer, using 60.0 and 229.2 mm diameter film-cassettes. The latter was used mainly to improve the precision of the measurements of low-angle reflections. $\text{CoK}\alpha$ radiation was used to avoid fluorescence from iron. The generator was set at 30 kV and 16 mA. Exposure times were 2 hours for the X-ray spectra recorded using the cassette of 60 mm diameter and 24 hours for the spectra recorded on the cassette of large diameter. The X-ray diffraction diagrams obtained displayed the similarity expected for three isomorphous compounds, where the dimensions of rashleighite's unit cell were intermediate between those of turquoise and chalcociderite.

Ito's method for a triclinic crystal consists of the determination of a reciprocal unit cell using any 3 non-coplanar reciprocal vectors σ_{h00} , σ_{0k0} , and σ_{00l} , which conform to the relation

$$\sigma_{hkl}^2 = \frac{1}{d_{hkl}^2} = \frac{4 \sin^2 \theta_{hkl}}{\lambda^2} = Q_{hkl} \quad (1)$$

The relation of Q_{hkl} to the reciprocal lattice constants is

$$\begin{aligned} Q_{hkl} &= h^2 a^{*2} + k^2 b^{*2} + l^2 c^{*2} + 2hka^*b^* \cos \gamma^* \\ &\quad + 2hla^*c^* \cos \beta^* + 2klb^*c^* \cos \alpha^* \\ Q_{100} &= \sigma_{100}^2 = a^{*2} \\ Q_{010} &= \sigma_{010}^2 = b^{*2} \\ Q_{001} &= \sigma_{001}^2 = c^{*2} \end{aligned} \quad (2)$$

As the values of Q_{hkl} are calculated from experimental data, the edge-lengths a^* , b^* , c^* of a reciprocal unit cell can then be calculated. For the determination of the angles of the reciprocal cell, use is made of the relation

$$\cos \gamma^* = \frac{Q_{hk0} - Q_{h\bar{k}0}}{4hka^*b^*}$$

and two other similar formulae obtained for $\cos \alpha^*$ and $\cos \beta^*$ from relation (2).

Once a reciprocal unit cell has been determined, the reduced cell, either direct or reciprocal, can be calculated (Buerger, 1957 and 1960). In the case of rashleighite, the unit cell chosen—very similar to those of turquoise and chalcociderite—was already the reduced cell.

The unit cell constants obtained from film-measurements were: $a = 7.48$; $b = 7.70$; $c = 10.00$ Å; $\alpha = 68^\circ 3'$; $\beta = 70^\circ 36'$; $\gamma = 65^\circ 42'$.

Almost all the reflections of the powder diffraction pattern obtained could be indexed using these parameters. There was however some uncertainty with respect to the angles, due to the fact that the accuracy

of measurements depended on the presence of several pairs of symmetric reflections such as $h0l$, $\bar{h}0l$, and similar pairs for $hk0$ and $0kl$, all correctly indexed.

In order to refine the lattice parameters obtained, new powder patterns of rashleighite were recorded on a Philips diffractometer using $\text{CoK}\alpha$ and also $\text{CuK}\alpha$ radiation. The alignment of the instrument was carefully checked for the Cu tube, and for the Co tube the results were corrected using CaF_2 as standard. The results thus obtained were compared to the calculated "powder diagram" obtained from the preliminary lattice constants and the DFST4 program (Onken, 1964). This allowed us to check some reflections with doubtful indexes and to know how many planes were contributing to each maximum.

The three reciprocal axes were uniquely defined with these data. For the angles we found more useful, instead of relation (3), the equivalent equation

$$\cos \gamma^* = \frac{Q_{hkl} - (h^2 a^{*2} + k^2 b^{*2})}{2hka^*b^*} \quad (4)$$

and two similar ones for $\cos \alpha^*$ and $\cos \beta^*$, also obtained from equation (2).

The final lattice parameters of rashleighite have been listed in Table 2 together with the lattice constants of turquoise and chalcociderite. The unit cell is an all-acute-reduced cell (Balashov, 1956; Buerger, 1957). The calculated density is 3.07 g/cm^3 , which com-

TABLE 2. A COMPARISON OF THE FINAL LATTICE CONSTANTS OF RASHLEIGHITE WITH THOSE OF TURQUOIS AND CHALCOSIDERITE

	Turquoise	Rashleighite	Chalcociderite
$a \text{\AA} $	7.424	7.49	7.67
$b \text{\AA} $	7.629	7.68	7.81
$c \text{\AA} $	9.910	10.0	10.20
α	$68^\circ 36'$	$68^\circ 36' \pm 11' (+)$	$67^\circ 31'$
β	$69^\circ 43'$	$69^\circ 48' \pm 20' (+)$	$69^\circ 6'$
γ	$65^\circ 5'$	$65^\circ 15' \pm 15' (+)$	$64^\circ 48'$
$V \text{\AA}^3 $	461.4	472	498
$\rho_{\text{cal}} \text{g/cm}^3 $	2.90	3.07	3.26
$\rho_{\text{ob}} \text{g/cm}^3 $	2.84	3.02	3.22

(+) Average of five values

pare well with the experimental values of 3.00 g/cm^3 and 3.02 g/cm^3 for the specimens from Castle-an-Dinas and Bunny Mine, respectively, determined by Russell (1948).

The indexed powder diffraction pattern of rashleighite is given in Table 3. Experimental conditions were: $\text{CoK}\alpha$ radiation, 30 kV, 16 mA (350 W normal focus tube), divergence slit 1° , receiving slit 0.2° mm, scatter slit 1° , Fe-filter.

DISCUSSION

Measurements by diffractometer gave values of a^* , b^* , c^* with errors less than 5×10^{-7} reciprocal lattice units, which yields values of V^* , V , a , b , c , with only three significant figures. Care must be taken in the appropriate rounding of numbers in intermediate steps of the calculation; otherwise, the length of the cell-edges might differ in as much as $1\text{-}2\text{\AA}$ and the cell volume in $5\text{-}10\text{\AA}^3$ (Scarborough, 1962). This is the explanation for the fact that the values for chalcosiderite

TABLE 3. DIFFRACTOMETER POWDER DIAGRAM OF RASHLEIGHITE

hkl	d_{obs}	d_{cal}	I/I_1	hkl	d_{obs}	d_{cal}	I/I_1
001	8.976	9.044	17	200	3.306	3.306	69
010	6.706	6.724	27	113	3.304	3.304	
100	6.706	6.661	27	221	3.184	3.167	12
011	6.223	6.220	33	022	3.110	3.111	16
111	6.223	6.197	33	222	3.100	3.100	
101	6.030	6.031	14	013	3.065	3.062	16
110	5.762	5.780	17	112	3.054	3.054	
$\bar{1}01$	4.828	4.837	51	202		3.015	
01 $\bar{1}$	4.828	4.831	51	003	3.014	3.014	28
112	4.626	4.633	11	103	3.014	3.014	
002	4.523	4.521	13	112	3.005	3.005	
$\bar{1}11$	4.131	4.142	9	123	2.925	2.919	100
110	4.131	4.080	9	121	2.915	2.915	
$\bar{1}11$	3.736	3.742	15	021	2.908	2.910	72
211	3.736	3.728	15	201	2.899	2.899	
$\bar{1}11$	3.697	3.697	100	220	2.890	2.890	
210	3.456	3.460	66	203	2.535	2.533	39
021	3.456	3.457	66	131	2.533	2.533	
102	3.397	3.391	2	114	2.486	2.492	11
012		3.371		211	2.479	2.479	
020	3.365	3.362	32	121	2.476	2.476	
201		3.361		022	2.418	2.416	17
				202		2.419	
				113		2.406	

cell-edges in Table 3 are slightly different from previously published ones (Cid-Dresdner, 1965b).

RELATIONSHIP BETWEEN THE STRUCTURE OF RASHLEIGHITE AND TURQUOIS

The difference in volume of the unit cells of chalcociderite and turquoise is $\Delta V_{\text{Ch-T}} = 36\text{\AA}^3$, whereas the corresponding difference between rashleighite and turquoise is $\Delta V_{\text{R-T}} = 11\text{\AA}^3$. If this difference in volume were due to an isomorphous substitution of Al by Fe in the turquoise structure, one would expect that $\Delta V_{\text{R-T}} = \frac{1}{3}V_{\text{Ch-T}}$, which is close to the value obtained.

This result still raised the question whether or not rashleighite could be an ordered substitutional structure of turquoise, one in which Fe could occupy only certain Al sites in the turquoise structure. Figure 1 is a representation by means of polyhedra, of one asymmetric unit of the turquoise structure, viewed along the a axis. The aluminum positions are labeled Al₁, Al₂, and Al₃. Sites Al₁ and Al₂ have similar environments: each is in the center of very distorted oxygen octahedra with an unusually short O—O distance at the shared edge and with angles deviating as much as 14° from ideal values. The Al₃ site is different; the coordination polyhedron is quite regular, the maximum angular deviation from ideal values being less than 4° (Cid-Dresdner, 1965a). If one assumes an ordered substitution, a replacement of one Al by one Fe atom in the Al₃ site would cause the least distortion in the structure.

Two different turquoise-like structures were then proposed for rashleighite. One was identical with that of turquoise with the exception that the Al₃ site was occupied by Fe. For the second structure we assumed a statistical distribution of one Fe atom on the three Al sites, so that the atomic scattering factor of Al was replaced by one calculated as

$$f_{\text{Al, Fe}} = \frac{1}{3}f_{\text{Fe}} + \frac{2}{3}f_{\text{Al}}$$

In order to test the hypothesis that Fe "replaces" Al in the Al₃ position, rough structure-factor calculations were done with both model structures and were then compared to the observed values obtained from the diffractometer pattern of rashleighite. For those maxima which originate from the sum of the intensities of the X-ray beams diffracted from more than one crystallographic plane, an average structure factor was calculated as

$$|F_{\text{ca.1}}|_{\text{av}} = \left(\sum_{H=1}^n F_H^2 \right)^{1/2}$$

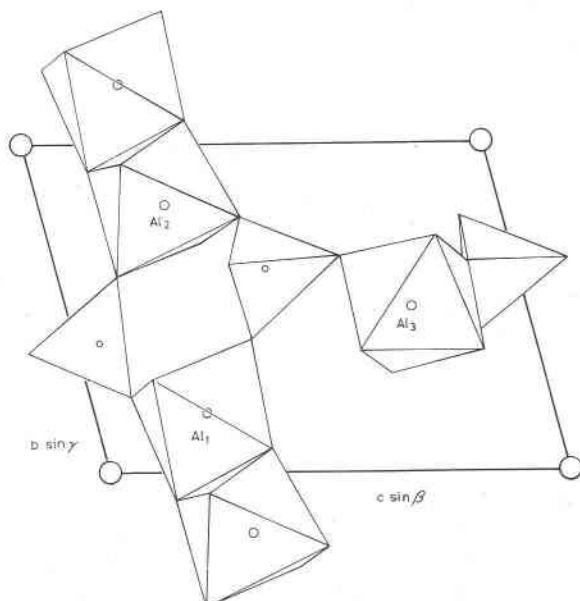


FIG. 1. One asymmetric unit of the structure of turquoise, represented by means of polyhedra, viewed along the *a* axis. The three aluminum sites are labelled Al₁, Al₂, and Al₃.

The integrated intensities were obtained by measuring the areas under the diffraction peaks with a planimeter. These values were then corrected for Lorenz and polarization effects.

The results of both structure factor calculations are listed in Table 4 and, although approximate, show significant differences. The discrepancy index obtained for a structure with a statistical distribution of the Fe on the three Al sites is 19 percent, whereas that obtained assuming a selective replacement of Al₃ by Fe is 31 percent.

In our opinion this result suggests that rashleighite has a turquoise-like structure in which the Al sites are statistically occupied by Al or by Fe atoms, in a proportion 2:1. In other words rashleighite can be considered as an homogenous solid solution of turquoise and chalcociderite.

THE TURQUOIS-CHALCOSIDERITE SERIES

The curve of Figure 2 represents the Al₂O₃ and Fe₂O₃ contents of the minerals belonging to the series, as obtained from different chemical analyses given in Table 1. The points conform perfectly to the theoretical line determined by the formulae $\text{CuO} \cdot 3\text{Al}_2\text{O}_3 \cdot 2\text{P}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$

for ideal turquoise and $\text{CuO} \cdot 3\text{Fe}_2\text{O}_3 \cdot 2\text{P}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$ for ideal chalcosiderite as end points, with the exception of Collins "henwoodite". The analyses of "henwoodite" (Table 1, Nos. 4 and 5) suggest that either the material studied by Collins is different from that studied by Fischer, or that Collins' chemical analysis is not accurate.

All the minerals of the turquoise-chalcosiderite series have very similar X-ray diffraction powder patterns and at least three of the members (turquoise, chalcosiderite, and rashleighite), have very similar unit cells. Even when in the reported member of the series there is a general tendency of the Fe atoms to conform to integer numbers which might suggest an ordered substitution of Al by Fe, the results found for rashleighite and the curve of Figure 2 indicate that the Al sites in the turquoise structure are tolerant to disorder between Al and Fe atoms in any proportion. Thus, rashleighite, "henwoodite", and alumo-chalcosiderite can be considered as solid solutions between turquoise and chalcosiderite.

TABLE 4. STRUCTURE FACTOR CALCULATIONS FOR TWO POSSIBLE MODELS OF RASHLEIGHITE BASED ON THE TURQUOISE STRUCTURE

(Model A assumes that the iron is statistically distributed on the three Al sites. Model B assumes that Al₃ has been replaced by Fe)

hkl	F _{obs}	F _{cal} _{av}		hkl	F _{obs}	F _{cal} _{av}	
		Model A	Model B			Model A	Model B
001	51	60	75	221	127	72	81
010	88	88	159	022	153	128	129
100				222			
011	103	170	231	013	152	120	147
111				112			
101	71	110	42	202			
110	81	65	75	00	230	225	309
101				103			
011	168	158	81	112			
112	82	70	81	123	404	495	618
002	91	68	84	121			
111	81	73	99	021	345	222	300
110				201			
111	120	108	114	220			
211				203	298	155	105
111	355	398	417	131			
210	272	298	333	114	158	170	198
021				211			
012	197	218	192	121			
020				022	242	250	273
201				202			
200	293	302	387	113			
113						R _{hkl} = 19%	R _{hkl} = 31%

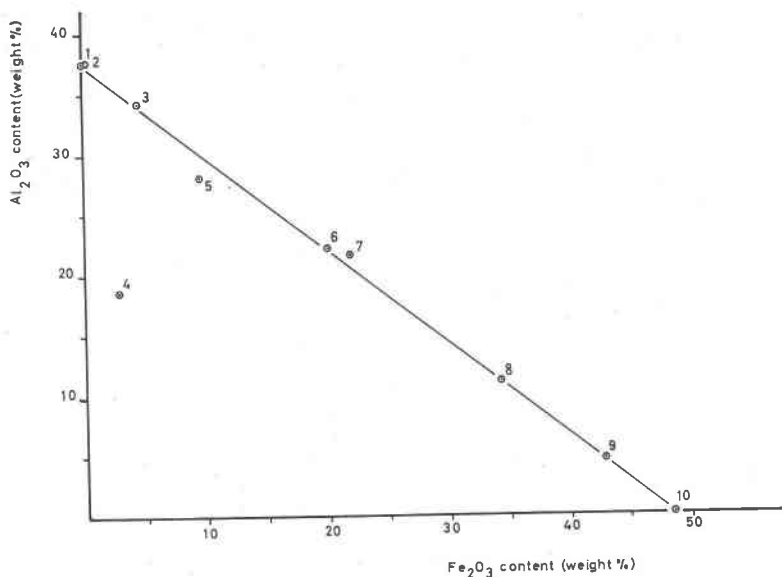


FIG. 2. Graphical representation of the relationship between Al_2O_3 and Fe_2O_3 contents in the turquoise-chalcosiderite series. The corresponding chemical analyses of all the samples represented in the graph are listed with the same numbers in Table 1.

The name of "henwoodite" was not approved by the Commission on New Minerals and Minerals Names I.M.A. [*Amer. Mineral.* 51, 1279 (1966)] on the basis that the similarity of turquoise and "henwoodite" X-ray powder patterns, and the chemical analysis of henwoodite, indicated that this was a solid solution of turquoise and chalcosiderite. By the same criteria the names of rashleighite and alumo-chalcosiderite should not be used any longer.

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