Canadian Mineralogist
Vol. 23, pp. 53-56 (1985)

X-RAY POWDER-DIFFRACTION DATA AND CELL PARAMETERS FOR COPIAPITE-GROUP MINERALS

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ABSTRACT

The chemical formula of the copiapite group is $(A^{2+}_{1-3x}A'^{3+}_{2x}\square_x)Fe^{3+}_4(SO_4)_6(OH)_2\cdot20H_2O$, where the A sites are completely filled by divalent cations (Ca, Cu, Fe, Mg or Zn) for $x = 0$, or two-thirds filled by trivalent cations (Al or Fe) for $x = 1/3$. Powder X-ray-diffraction data now available have been re-indexed in light of the results of crystal-structure determinations in the literature. The cell parameters of these copiapite-group minerals have been refined by least-squares analysis. Data on magnesiocopiapite are tabulated. Challantite is discredited as a synonym of ferricopiapite.

Keywords: copiapite, ferricopiapite, magnesiocopiapite, challantite, cell parameters, powder-diffraction data.

SOMMAIRE

Dans la formule chimique des minéraux du groupe de la copiapite, $(A^{2+}_{1-3x}A'^{3+}_{2x}\square_x)Fe^{3+}_4(SO_4)_6(OH)_2\cdot20H_2O$, la position A se trouve remplie complètement par les cations divalents (Ca, Cu, Fe, Mg ou Zn) pour $x = 0$ ou au deux-tiers par les cations trivalents (Al ou Fe) pour $x = 1/3$. Les données de diffraction X (méthode des poudres) disponibles à présent ont été revues à la lumière des structures cristallines publiées récemment. Les paramètres réticulaires des membres de ce groupe ont été affinés par la méthode des moindres carrés. On présente les données pour la magnésiocopiapite. La challantite est discréditée (= ferricopiapite).

(Traduit par la Rédaction)

Mots-clés: copiapite, ferricopiapite, magnésiocopiapite, challantite, paramètres réticulaires, méthode des poudres.

INTRODUCTION

The copiapite group is represented by four mineral species in the Group Data Book of the Mineral Powder Diffraction File (Bayliss *et al.* 1983): aluminocopiapite PDF 20–659, cuprocopiapite PDF 19–394, calciocopiapite PDF 27–77, and zincocopiapite PDF 33–1472. Additional species of the copiapite group were listed by Fleischer (1983): copiapite, ferricopiapite and magnesiocopiapite.

A comparison between the powder X-ray-diffraction data of the four copiapite species in the Mineral Powder Diffraction File shows that similar values of d commonly are attributed different indices, although the unit-cell parameters are similar. Secondly, for the same reflections, the intensities of the four copiapite minerals are not correlatable with the F_{obs} values of either magnesiocopiapite of Süsse (1972) or ferricopiapite of Fanfani *et al.* (1973).

Therefore, the powder-diffraction data in the literature have been re-indexed, and unit-cell parameters have been refined by least-squares analysis. In addition, powder-diffraction data with a precision higher than those presently available for magnesiocopiapite were collected.

CHEMICAL FORMULAE

The chemical formulae of the copiapite group proposed by Fleischer (1983) are $A^{2+}Fe^{3+}_4(SO_4)_6(OH)_2\cdot20H_2O$, in which A^{2+} is Ca, Cu, Fe, Mg or Zn, and $A^{3+}Fe^{3+}_4(SO_4)_6O(OH)\cdot20H_2O$, in which A^{3+} is Al or Fe. Zodrow (1980) has had problems with these chemical formulae, as he states: "whether the excess of sulfate is a characterizing feature of copiapite samples from the Sydney Coalfield [Nova Scotia], or a problem of mathematical manipulation in the prediction process, is not known at this juncture . . ."

In contrast to the full occupancy of the A site, Fanfani *et al.* (1973) have determined by chemical analysis that the A site of a specimen of ferricopiapite has an occupancy of 0.69, with 0.43 Fe^{3+}, 0.23 Al^{3+} and 0.03 Zn^{2+}. This partial occupancy is supported by a crystal-structure analysis with an R value of 0.074, in which the A site has a temperature factor (3.54) greater than that of other sites (Fe 2.4, S 2.0) and contains the lighter atom Al.

In order to compare these chemical formulae, the chemical composition of six theoretical end-members was calculated (Table 1). The chemical composition of six minerals and three synthetic compounds is also listed in Table 1 for comparison. The values of the ratio Fe_2O_3/SO_3 for these chemical compositions are in agreement with an A site that is partly occupied, so that the formula is $(A^{2+}_{1-3x}A'^{3+}_{2x}\square_x)Fe^{3+}_4(SO_4)_6(OH)_2\cdot20H_2O$.

METHODS AND RESULTS

If the approximate cell-parameter of a cubic mineral is known, then the powder-diffraction data

TABLE 1. CHEMICAL COMPOSITIONS OF MINERALS IN THE COPIAPITE GROUP

Theoretical Formula or Mineral Name	PDF Number or Reference	FeO	MgO	Al_2O_3	Fe_2O_3	SO_3	H_2O	$\frac{Fe_2O_3}{SO_3}$
		wt. %						
$MgFe_4(SO_4)_6(OH)_2 \cdot 20H_2O$			3.3		26.2	39.4	31.1	
$AlFe_4(SO_4)_6O(OH) \cdot 20H_2O$				4.2	26.2	39.4	30.2	
$Al_{2/3}Fe_4(SO_4)_6(OH)_2 \cdot 20H_2O$				2.8	26.4	39.6	31.2	
$FeFe_4(SO_4)_6(OH)_2 \cdot 20H_2O$		5.7			25.6	38.4	30.3	
$Fe_{2/3}Fe_4(SO_4)_6(OH)_2 \cdot 20H_2O$					30.3	39.0	30.7	0.777
$Fe_5(SO_4)_6O(OH) \cdot 20H_2O$					32.0	38.5	29.6	0.831
Aluminocopiapite	20-659		1.97	3.17	23.58	39.90	30.90	
Ferricopiapite	Specimen 3, Berry(1947)				29.98	39.68	30.45	0.756
Ferricopiapite	Specimen 4, Berry(1947)				30.10	38.91	30.74	0.774
Ferricopiapite, synthetic	16-897				30.3	39.0	30.7	0.777
Ferricopiapite≡Challantite	29-714				30.29	38.82	31.07	0.780
Ferricopiapite, synthetic	Posnjak & Merwin(1922)				30.75	39.28	29.97	0.783
Ferricopiapite	Specimen 5, Berry(1947)				30.24	38.62	30.75	0.783
Ferricopiapite	Specimen 2, Berry(1947)				30.68	38.72	30.60	0.792
Ferricopiapite, synthetic	28-497				30.1	37.7	32.2	0.798

will index automatically to produce the best parameter by least-squares refinement. However, if approximate cell-parameters of a triclinic mineral with a large unit-cell are known, then an attempt to index automatically the powder X-ray-diffraction data will generally lead to wrongly assigned indices and hence incorrect unit-cell parameters by least-squares refinement. Therefore, the indices of a triclinic mineral with large unit-cell parameters must be assigned based upon the F_{obs} values derived from a single-crystal investigation.

With the cell parameters and triclinic space-group $P\bar{1}$ (#2) of Süsse (1972) for reference, the indices of a few reflections with large d values were assigned based upon the strongest F_{obs} values. The cell parameters were refined with the least-squares programme of Appleman & Evans (1973) to calculate new unit-cell parameters, and then the indices of the reflections with the next highest d values were asssigned based upon the strongest F_{obs} values. Further least-squares cycles were run with the new unit-cell parameters, and then additional indices were assigned. This process was repeated until all the reflections were assigned with indices that have strong F_{obs} values, and until all the strong F_{obs} values were assigned to reflections. In addition the agreement between d_{obs} and d_{calc} was found to have improved. Finally, the assigned indices were checked by comparison with the I_{calc} values obtained from the programme of H. Langhof (Physikalische Chemie Institut, Darmstadt) with the crystal-structure parameters of both Süsse (1972) and Fanfani *et al.* (1973).

The cell parameters determined from powder X-ray-diffraction data in the literature are given in Table 2. Some of the data in the literature (*e.g.*, Srebrodolsky 1970) were not used to refine unit-cell parameters because there are a small number of reflections with only three-figure accuracy available to determine six variables (a, b, c, α, β and γ).

Copiapite-group specimens were obtained as follows: specimen number 16783, American Museum of Natural History, New York, from Knoxville, Napa County, California; David Shannon Minerals specimen from the Denver mine, Santa Cruz County, Arizona, and specimen number H8307, Natural History Museum, Vienna, Austria from Knoxville, California. A diffractometer trace was obtained with Fe$K\alpha_1$ radiation (1.93597 Å) and a 0002 graphite monochromator from 4° to 60° 2θ at a scanning speed of 0.125°/min with 1° incidence slits and a 2-cm-long specimen holder. The unit-cell parameters of these three specimens are given in Table 2.

A semi-quantitative analysis performed using a Kevex solid-state analyzer showed specimen 16738 and that from the Denver mine to have major Al, Mg, Fe and S, whereas specimen H8307 contains major Mg, Fe and S, and minor Cr and Al, and is thus magnesiocopiapite. Since the indexed powder-diffraction data of specimen H8307 are significantly better than the unindexed data on magnesiocopiapite of Kravtsov (1971), the hkl, d_{calc}, d_{obs}, relative intensities (I/I_1) and I_{calc} from the crystal-structure parameters of Süsse (1972) are presented in Table 3. Agreement between I/I_1 and I_{calc} is generally good, except for the first two reflections at low 2θ, where only part of the X-ray beam is intersected by the specimen.

DISCUSSION

The alphabetical chemical index for the Inorganic Phases of the Powder Diffraction File showed three unindexed patterns (PDF 16–897, 28–497 and 29–714) that appear to belong to the copiapite group. The chemical composition of the synthetic compounds PDF 16–897 (Walter-Lévy & Quéméneur 1963) and 28–497 (Margulis *et al.* 1973) calculated from their formulae are presented in Table 1. These patterns were indexed, and the refined unit-cell parameters are given in Table 2. Since the chemical compositions are similar to the theoretical chemical composition for ferricopiapite, the unit-cell param-

TABLE 2. UNIT-CELL PARAMETERS FOR MINERALS IN THE COPIAPITE GROUP

Mineral Species Name	*a*	*b*	*c*	α	β	γ	Reference	PDF Number
Magnesiocopiapite	7.35(2)	18.84(2)	7.39(2)	91.7(2)	102.6(2)	99.0(2)	Kravtsov (1971)	—
Magnesiocopiapite	7.341(4)	18.824(7)	7.395(4)	91.23(4)	102.26(4)	98.92(4)	David Shannon Minerals	—
Magnesiocopiapite	7.351(2)	18.805(6)	7.394(2)	91.32(3)	102.17(3)	98.79(3)	H8307 Natural History Museum Vienna	—
Magnesiocopiapite	7.335(2)	18.782(4)	7.377(2)	91.37(2)	102.21(2)	98.80(2)	16783 American Museum New York	—
Aluminocopiapite	7.30(1)	18.80(2)	7.31(1)	91.5(2)	102.3(2)	98.7(1)	Jolly & Foster (1967)	20-659
Calciocopiapite	7.44(3)	18.79(4)	7.22(3)	85.3(3)	104.7(3)	102.2(3)	Kashkai & Aliev (1960)	27-77
Copiapite	7.337(6)	18.76(1)	7.379(6)	91.46(7)	102.18(6)	98.95(6)	Schmetzer & Medenbach (1983)	—
Zincocopiapite	7.33(1)	18.72(2)	7.35(1)	91.5(2)	102.1(2)	98.7(2)	Tu *et al.* (1964)	33-1472
Ferricopiapite	7.37(2)	18.38(3)	7.31(2)	93.9(1)	102.3(2)	98.7(3)	Margulis *et al.* (1973)	28-497
Ferricopiapite	7.394(5)	18.36(2)	7.324(8)	93.8(1)	102.2(1)	98.9(1)	Ramusino & Giuseppetti (1973)	29-714
Ferricopiapite	7.380(2)	18.352(5)	7.319(2)	94.00(1)	102.12(1)	98.54(1)	Walter-Lévy & Quéméneur (1963)	16-897
Ferricopiapite	7.378(7)	18.30(2)	7.327(8)	93.87(8)	102.23(7)	98.96(6)	Kubisz (1964) synthetic	—
Ferricopiapite	7.413(8)	18.23(2)	7.32(1)	93.73(8)	102.11(7)	98.97(7)	Kubisz (1964) Alcaparossa	—
Ferricopiapite	7.29(2)	18.23(4)	7.28(2)	93.5(2)	102.1(2)	98.2(2)	Kravtsov (1971)	—
Ferricopiapite	7.41(1)	18.20(2)	7.309(8)	93.77(8)	102.11(8)	99.13(7)	Kubisz (1964) Kopalnia Staszic	—
Cuprocopiapite	7.31(1)	18.15(2)	7.25(1)	92.5(2)	102.3(2)	100.4(2)	Escobar & Gifford (1961)	19-394

a, *b* and *c* in Ångströms, α, β and γ in degrees.

eters are similar to those of ferricopiapite, and also the intensities of the reflections are similar to the I_{calc} values derived from the crystal-structure parameters of Fanfani *et al.* (1973), these two synthetic compounds are considered to be ferricopiapite.

The powder X-ray-diffraction data (PDF 29–714) of challantite, which was first described by Ramusino & Giuseppetti (1973), may be indexed using the cell parameters of ferricopiapite. The chemical composition (Table 1), refined unit-cell parameters (Table 2) and I/I_1 are similar to those of ferricopiapite. Challantite is thus identical with ferricopiapite and therefore should be discredited. This nomenclature proposal (number 84, submitted by Atencio & Bayliss in 1984) has been approved by the I.M.A.

Fanfani *et al.* (1973) showed that Al may occupy the *A* site, with an average Al–O bond length of 1.93 Å, whereas Süsse (1972) showed that Mg may occupy the *A* site, with an average Mg–O bond length of 2.07 Å. These average bond-lengths will increase to allow occupancy by Ca (calciocopiapite), since the atomic radius of Ca is larger than that of Mg. Some specimens of copiapite contain large quantities of Al, so that Al must occupy the *A* site and substitute for Fe. Similarly, some specimens of copiapite (Zodrow 1980) contain large quantities of Mg, so that Mg must occupy the *A* site and substitute for Fe. The Fe-site average Fe–O bond length in the ferricopiapite of Fanfani *et al.* (1973) is 2.00 Å, compared to 2.01 Å for the magnesiocopiapite of Süsse (1972).

The occupancy of the *A* site and Fe^{3+} site by Mg and Al is difficult to determine by crystal-structure analysis, because Mg and Al have similar atomic-scattering factors. The bond lengths available are insufficient to predict the occupancy of the *A* and Fe sites. The octahedral *A* site is surrounded by six H_2O, whereas the octahedral Fe site is surrounded by three O, two H_2O and one OH. On the basis of the principle of electrostatic valence, cations of lower charge (Ca^{2+}, Cu^{2+}, Fe^{2+}, Mg^{2+} or Zn^{2+}) preferentially occupy the *A* site compared to cations of higher charge (Al^{3+} or Fe^{3+}). Therefore, some specimens of the copiapite group that contain significant amounts of both Mg^{2+} and Al^{3+}, like specimen 16783 and that from the Denver mine, are called aluminian magnesiocopiapite.

The I_{calc} values determined from the crystal-structure parameters of magnesiocopiapite by Süsse (1972) are significantly different from the I_{calc} values determined from the crystal-structure parameters of ferricopiapite by Fanfani *et al.* (1973), so that ferricopiapite can be differentiated from magnesiocopiapite by powder-diffraction data. The reflection intensities of zincocopiapite and aluminocopiapite are similar to the I_{calc} values from magnesiocopiapite of Süsse (1972). The unit-cell data

TABLE 3. POWDER X-RAY-DIFFRACTION DATA FOR MAGNESIOCOPIAPITE H8307

hkl	d_{calc}Å	d_{obs}Å	I/I_1	I_{calc}	*hkl*	d_{calc}Å	d_{obs}Å	I/I_1	I_{calc}
010	18.55	18.57	90	350	$\bar{1}41$	3.052	3.052	25	20
020	9.28	9.29	100	154	$\bar{1}60$	3.019	3.021	6	6
001	7.217	7.219	10	15	$2\bar{1}1$	2.976	2.978	7	7
100	7.089	7.093	4	5	$0\bar{4}2$	2.931	2.931	15	7
$0\bar{1}1$	6.861	6.872	10	11	$1\bar{2}2$	2.924			8
011	6.600	6.604	5	7	$\bar{1}42$	2.849	2.848	7	7
110	6.292	6.290	20	21	211	2.836	2.834	8	6
030	6.184	6.192	45	44	$2\bar{3}1$	2.833			4
$\bar{1}01$	5.716	5.710	10	10	$1\bar{3}2$	2.803	2.805	2	5
$\bar{1}11$	5.598	5.600	80	90	$\bar{1}\bar{4}2$	2.782	2.783	9	9
$\bar{1}\bar{1}1$	5.337	5.343	25	28	$1\bar{6}1$	2.750	2.750	4	4
$0\bar{3}1$	4.835	4.826	7	1	122	2.736	2.734	8	8
$\bar{1}\bar{2}1$	4.692	4.697	20	25	$\bar{2}41$	2.681	2.680	10	5
031	4.569	4.574	8	9	$\bar{2}\bar{2}2$	2.668	2.668	10	11
130	4.327	4.319	12	8	$\bar{1}70$	2.626	2.626	12	9
111	4.311			7	132	2.562	2.562	6	4
$\bar{1}40$	4.206	4.208	40	45	$\bar{2}\bar{3}2$	2.514	2.514	6	8
131	4.030	4.033	30	33	$\bar{1}71$	2.487	2.486	10	10
121	3.897	3.898	15	19	$1\bar{5}2$	2.459	2.457	1	4
041	3.804	3.803	10	8	$0\bar{1}3$	2.403	2.403	5	5
$\bar{1}41$	3.762	3.762	7	7	$\bar{3}20$	2.384	2.383	4	7
$\bar{2}10$	3.590	3.588	50	32	300	2.363	2.363	6	6
$0\bar{1}2$	3.580			20	$\bar{3}\bar{1}1$	2.343	2.345	2	1
200	3.545	3.543	10	12	023	2.297	2.293	6	5
$1\bar{4}1$	3.518	3.519	1	14	202	2.292			5
012	3.505	3.506	40	33	$\bar{1}81$	2.216	2.217	4	4
$\bar{1}\bar{4}1$	3.461	3.462	20	18	$1\bar{8}1$	2.198	2.197	4	2
$\bar{2}\bar{1}1$	3.356	3.359	7	6	$\bar{2}\bar{5}2$	2.177	2.176	3	5
$\bar{2}30$	3.316	3.317	7	5	$\bar{3}\bar{3}2$	2.161	2.162	2	0
022	3.300	3.301	7	4	$1\bar{2}3$	2.133	2.134	4	3
$\bar{1}51$	3.240	3.231	20	11	$\bar{2}80$	2.103	2.102	6	4
231	3.232			6	$\bar{2}81$	2.062	2.061	14	4
051	3.226			8	090	2.061			6
220	3.146	3.144	8	5	$\bar{2}72$	2.031	2.030	5	1
$\bar{2}\bar{2}1$	3.139			5	$2\bar{6}2$	1.978	1.978	3	4

in Table 2 are listed in the order of decreasing values of *b* , since the numerous specimens of magnesiocopiapite and ferricopiapite each have similar values of *b*. Although the unit-cell parameters show a trend, this trend is not sufficient to differentiate between the seven mineral species in the copiapite group.

From unindexed powder-diffraction data and qualitative signals for Fe and S, Schmetzer & Medenbach (1983) described a second occurrence of challantite. Their powder data were indexed on a copiapite-group unit cell. The cell parameters, listed in Table 2, are similar to the unit-cell parameters of magnesiocopiapite. Secondly, the intensities of the reflections are similar to the I_{calc} for copiapite with the crystal-structure parameters of Süsse (1972) rather than to the I_{calc} for ferricopiapite with the crystal-structure parameters of Fanfani *et al.* (1973). Therefore, this second occurrence of challantite is not ferricopiapite, but is possibly copiapite.

Powder X-ray-diffraction and chemical data were given for copiapite by Kravtsov (1971). An attempt to index the data on a copiapite-group unit cell was unsuccessful; subsequently, the mineral was identified as roemerite, $Fe^{2+}Fe_2^{3+}(SO_4)_4 \cdot 14H_2O$.

ACKNOWLEDGEMENTS

The Natural History Museum in Vienna, The American Museum in New York, and David Shannon Minerals in Arizona provided specimens. The Natural Sciences and Engineering Research Council of Canada and the Sao Paulo Research Agency FAPESP (Proc. 83/2016-4) gave financial support. Drs. R. Hypolito, J.V. Valarelli, H.H.G.J. Ulbrich, T.R. Fairchild, E.E. Sano, H.G. Fernandes, A. Zular and E.H. Nickel gave advice. Dr. J.J. Szymánski and the referees provided excellent critical reviews.

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Received February 6, 1984, revised manuscript accepted July 31, 1984.