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X-RAY CRYSTALLOGRAPHY OF OMPHACITE

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INTRODUCTION

In recent years there has been much petrologic interest in omphacite and other minerals found in eclogites. This report describes the x-ray crystallography of chemically analyzed omphacite number 1725 from the Eiksundsdal Eclogite Complex, Hareidland, Sunmøre, Norway, described by H. H. Schmitt (1963, p. 60).

Schmitt gives a chemical analysis of this sample (No. 1725, Table 2), which is reproduced in Table 1. The analysis is also recast in Table 1, as number of ions per six oxygens. The sample is approximately $\frac{2}{3}$ diopsidic-augite and $\frac{1}{3}$ acmitic-jadite. It should be noted that this omphacite is richer in ferric and ferrous iron than most omphacites.

EXPERIMENTAL PROCEDURES AND RESULTS

Single crystal x-ray photographs were taken of the sample, verifying the space group $C2/c$ with Z equals 4 for omphacite. Indexing was done on the measurements from a FeK_α radiation powder photograph.

The reflection angle 2θ was calculated for all allowed reflections with h, k, and l varying from 0 to 9, 0 to 9, and -5 to $+5$ respectively, using preliminary values of a, b, c and β derived from single crystal photos. The cell constants were refined and the calculation was repeated. Final indexing is shown in Table 2. I was not able to index lines 3 and 6. These line are probably due to the oriented inclusions reported below.

Between ten and forty-two reflections were used in each refinement cycle. Several criteria were used to select which reflections and hkl's were to be used in a given cycle. Refinement I was made by choosing the reflections that had the closest matching calculated and observed 2θ

Table 1. Chemical Analysis and Number of Ions per 6 Oxygens for Omphacite 1725

(Jun Ito, Analyist, see Schmitt, 1963, p. 274)

Oxide	Wt. %	Ion	Number
SiO_2	54.74	Si^{4+}	1.973 ⎫
TiO_2	0.09	Ti^{4+}	0.002 ⎬ 2.000
Al_2O_3	5.61	Al^{3+}	0.238 ⎫
Cr_2O_3	0.01[1]	Cr^{3+}	0.0004 ⎬ 0.336
Fe_2O_3	4.56	Fe^{3+}	0.123 ⎭
FeO	3.83	Fe^{2+}	0.116 ⎫
MnO	0.03	Mn^{2+}	0.0009 ⎪
MgO	10.83	Mg^{2+}	0.582 ⎬ 1.283
NiO	0.01[1]	Ni^{2+}	0.0002 ⎪
SrO	0.05[1]	Sr^{2+}	0.001 ⎪
CaO	15.11	Ca^{2+}	0.583 ⎭
Na_2O	4.56	Na^{1+}	0.325 ⎫
K_2O	0.01	K^{1+}	0.0004 ⎬ 0.325
H_2O^+	0.50	H^{1+}	0.12
H_2O^-	0.20		
	100.23[2]		4.065
		O^{2-}	6.000

[1] Spectrographically determined.

[2] Ga, Pb and V also spectrographically determined.

values. Those *hkl*'s that were recorded as being of high intensity on the single crystal photographs and had a good 2θ match were used in Refinement II. Refinement III used the *hkl*'s recommended by Coleman (1962) as being the best lines to use when calculating the cell constants of clino-pyroxenes. The 10 *hkl*'s used in Refinement IV were selected by sub-jectively choosing reflections from the previous three sets of *hkl*'s while keeping track of the quality of each line on the powder photograph, *i.e.*, the 2θ value, line broadness, and line intensity were considered in choos-ing *hkl*'s. Refinement V is a combination of the 42 best lines from runs I, II, III and IV. The *hkl*'s used in the final refinement are noted in Table 2 by an asterisk after the line number. The values of *a*, *b*, *c* and β ob-tained from each refinement are listed in Table 3. The number of *hkl*'s used in each cycle is also tabulated in Table 3. The values from refine-ment V are adopted for omphacite 1725.

TABLE 2. X-RAY POWDER DATA FOR OMPHACITE 1725
$a=9.662$ Å; $b=8.819$ Å; $c=5.228$ Å; $\beta=106.55°$

h k l	d calculated (Å)	d observed (Å)	Intensity	Line No.
1 1 0	6.387	6.4	3	1*
0 2 0	4.409	4.4	5	2
– – –	—	3.8	1	3
0 2 1	3.310	3.33	1	4*
2 2 0	3.193	3.19	5	5*
– – –	—	3.12	1	6
2 2 $\bar{1}$	2.971	2.976	10	7*
3 1 0	2.914	2.915	5	8*
3 1 $\bar{1}$	2.878	2.880	5	9*
1 3 $\bar{1}$	2.451	2.549	4	10
2 0 $\bar{2}$	2.526	2.521	4	11*
2 2 1	2.481	2.484	5	12*
3 1 1	2.263	2.268	3.5	13*
2 2 $\bar{2}$	2.192			
1 1 2	2.191	2.192	3	14
3 3 0	2.129	2.127	7	15
3 3 $\bar{1}$	2.115	2.118	7	16
4 2 $\bar{1}$	2.091	2.088	2	17*
0 4 1	2.018	2.019	7	18
2 4 0	1.991	1.987	1	19
1 3 $\bar{2}$	1.953	1.958	1	20*
2 4 $\bar{1}$	1.933	1.934	1	21*
5 1 $\bar{1}$	1.881	1.879	1	22*
3 3 1	1.834			
4 2 $\bar{2}$	1.829	1.833	1	23*
5 1 0	1.813			
3 3 $\bar{2}$	1.807	1.810	2	24
2 4 1	1.777	1.778	1	25*
4 2 1	1.749	1.749	1	26*
1 5 0	1.733	1.733	4	27*
2 4 $\bar{2}$	1.661			
0 4 2	1.655	1.660	3	28
2 2 $\bar{3}$	1.616			
4 4 $\bar{1}$	1.616	1.624	2	29
5 3 $\bar{1}$	1.611	1.609	3	30*

* Signifies that line was used in the final refinement.

TABLE 2—(*continued*)

h k l	d calculated (Å)	d observed (Å)	Intensity	Line No.
4 4 0	1.597	1.597	2	31*
5 3 0	1.567	1.566	1	32
6 0 0	1.544 ⎫			
1 1 3	1.543 ⎭	1.545	1	33*
3 5 0	1.531	1.531	1	34*
6 0 $\bar{2}$	1.522	1.523	1	35*
5 3 $\bar{2}$	1.502 ⎫			
4 0 2	1.501 ⎭	1.502	2	36*
2 4 2	1.473 ⎫			
0 6 0	1.470 ⎭	1.472	2	37
4 4 1	1.442 ⎫			
6 2 $\bar{2}$	1.439 ⎭	1.441	1	38
4 2 2	1.421	1.421	1	39*
0 6 1	1.410	1.412	1	40*
5 3 1	1.403 ⎫			
2 6 0	1.401 ⎭	1.402	8	41*
2 6 $\bar{1}$	1.380	1.378	1	42
7 1 $\bar{1}$	1.364	1.363	1	43
7 1 $\bar{2}$	1.323	1.324	3	44*
2 6 1	1.320 ⎫			
5 3 $\bar{3}$	1.312 ⎭	1.316	1	45
7 1 0	1.309 ⎫			
6 2 1	1.308 ⎭	1.308	2	46
4 4 $\bar{3}$	1.288	1.290	1	47
5 5 0	1.277 ⎫			
2 6 $\bar{2}$	1.270 ⎭	1.273	3	48
4 0 $\bar{4}$	1.263	1.262	1	49*
4 6 $\bar{1}$	1.250	1.250	1	50
1 5 $\bar{3}$	1.236	1.234	2	51*
1 7 $\bar{1}$	1.222	1.225	1	52*
3 3 3	1.204	1.204	2	53

TABLE 2—*(continued)*

h k l	d calculated (Å)	d observed (Å)	Intensity	Line No.
4 6 1	1.164 ⎫			
6 4 1	1.163 ⎬	1.163	1	54
8 2 1̄	1.163 ⎭			
8 0 0	1.158	1.156	1	55*
8 2 2̄	1.147	1.150	1	56
1 7 2̄	1.135	1.137	1	57
1 7 2	1.101	1.101	1	58
4 6 3̄	1.078	1.079	1	59
7 5 2̄	1.066	1.066 ⎫ 1.066 ⎭	4	60
7 5 0	1.058 ⎫	1.058 ⎫	5	61
8 4 1̄	1.058 ⎭	1.058 ⎭		
4 6 2	1.050	1.050	1	62*
1 5 4̄	1.042	1.043 ⎫ 1.043 ⎭	1	63
3 1 5̄	1.037	1.037 ⎫ 1.036 ⎭	1	64*
2 6 3	1.031 ⎫	1.029 ⎫	1	65
5 3 3	1.026 ⎭	1.028 ⎭		
9 1 0	1.022	1.022 ⎫	1	66*
8 0 4̄	1.005	1.005 ⎭ 1.005		
5 5 4̄	0.9979 ⎫	0.9973	1	67*
3 3 4	0.9974 ⎭			
4 8 0	0.9953	0.9951	1	68*
1 5 4	0.9904 ⎫	0.9903 ⎫	1	69
6 6 3̄	0.9903 ⎬	0.9904 ⎭		
4 0 4	0.9900 ⎭			
8 2 4̄	0.9799	0.9785	1	70
6 2 3	0.9756	0.9755 ⎫ 0.9759 ⎭	2	71*

In order to calculate the cell constants of a clinopyroxene, I recommend using the following 17 reflections: 020, 220, 22̄1, 310, 311̄, 131̄, 202̄, 221, 311, 330, 331̄, 421̄, 041, 150, 531̄, 440, and 712̄. These reflec-

TABLE 3. UNIT CELL CONSTANTS FOR OMPHACITE 1725

Refinement Number[1]	I	II	III	IV	V[2]
a (Å)	9.659 ±0.002	9.667 ±0.011	9.659 ±0.014	9.671 ±0.005	9.662 ±0.003
b (Å)	8.817 ±0.002	8.812 ±0.014	8.812 ±0.010	8.822 ±0.004	8.819 ±0.004
c (Å)	5.225 ±0.001	5.254 ±0.010	5.243 ±0.032	5.224 ±0.001	5.228 ±0.002
β (Å)	106.57 ±0.02	106.69 ±0.16	106.59 ±0.19	106.54 ±0.02	106.55 ±0.02
Number of reflections used in refinement	31	13	11	19	42

[1] See text for the meaning of refinement number.

[2] Adopted values for omphacite 1725, Z equals 4.

tions are strong, unambiguous and are not interfered with by a silicon standard, however, 041 and 71$\bar{2}$ are often hard to find on a diffractometer pattern.

Detailed microscopic study of omphacite 1725 reveals the presence of mineral inclusions. They are oriented parallel to the c axis of the omphacite, are unevenly distributed but average about 1% of the sample. They range in size from sub-microscopic to 0.02 mm×0.004 mm×0.004 mm with several distinctly larger ones measuring about 0.15 mm×0.03 mm ×0.03 mm. The inclusions have parallel extinction, moderate birefringence, and low relief; they may be an orthopyroxene. They are responsible for line broadening on all the x-ray photos.

The optical constants were determined using the immersion method with Na-light and the Leitz-Jelly refractometer. The density was measured using the Berman micro-balance. The physical properties of omphacite 1725 are: $\alpha = 1.688$, $\beta = 1.693$, $\gamma = 1.709$ (all ±0.002); $2V = 56°$ (calc.); $D = 3.394 \pm 0.007$ gm/cm³.

of the Geophysical Laboratory. The program for the calculation of 2θ was obtained from C. Klein.

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ELECTRON PROBE ANALYSES OF COPPER IN MENEGHINITE

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Meneghinite was first described by Bechi (1852) from the Bottino Mine, near Tuscany, Italy. Based on chemical analyses and x-ray diffraction its space-group has been given as $D_{2h}^{16} = Pbnm$ with $a = 11.36$, $b = 24.04$, $c = 8.26$ Å, and the cell content, as $Pb_{26}Cu_2Sb_{14}S_{48}$ (Berry and Moddle, 1941). Two copper-free meneghinite formulas have been suggested, $Pb_4Sb_2S_7$ (von Rath, 1867) and $Pb_{13}Sb_7S_{23}$ (Palache *et al.*, 1938). Most analyses indicated an excess of Pb, and it seemed doubtful if the Cu (and much smaller amounts of Fe) really belonged in the structure. Ramdohr (1960) still quotes both possibilities.

In order to establish the distribution of Cu, two crystals of meneghinite from Bottino (specimen no. RH1407 in the Mineralogical Department of the Swedish Natural History Museum in Stockholm) were analyzed with an ARL electron microprobe x-ray analyzer. The quantitative results are shown in Table 1, and the distribution of Pb, Sb and Cu is demonstrated semi-quantitatively by beam scanning pictures, Fig. 1. The probe analysis agrees fairly well with the formula $Pb_{26}Cu_2Sb_{14}S_{48}$. The deviation as well as the low total, 95.6 per cent (no other elements were found in concentration above < 0.1 per cent), is probably due to insufficiently known mass absorption and average atomic no. corrections (Ziebold and Ogilvie, 1963). The standards used were pure Sb-metal, galena (PbS), and chemically analysed bornite (Cu_5FeS_4).

It is immediately clear from Fig. 1 that although Pb, Sb and Cu are homogeneously distributed in the meneghinite, this mineral contains minute exsolved particles of a heavier mineral free from Cu and Sb. Probe analyses show this mineral to be galena (PbS). It is obvious that