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X-ray diffraction data for melanovanadite

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Abstract

The strongest reflections from the holotype specimen of melanovanadite, $Ca_2V_4^{4+}V_6^{5+}O_{25} \cdot 7H_2O$, are 8.40Å(100), 4.21Å(55) and 2.97Å(50). The triclinic unit cell was refined to be *a* 6.357(2)Å, *b* 16.868(4)Å, *c* 6.274(2)Å, α 90.04(2)°, β 101.60(2)°, and γ 93.20(3)° in space group *A*1. The D calc. of the chemical formula of $Ca_2V_4^{4+}V_6^{5+}O_{25} \cdot 7H_2O$ is 2.86 compared to the D meas. of 2.90 and 2.83.

Introduction

In a review of the literature for the Mineral Powder Diffraction File by Bayliss et al. (1980), powder X-ray diffraction data could not be found for the mineral species melanovanadite, $Ca_2V_4^{4+}V_6^{5+}O_{25} \cdot nH_2O$. The holotype specimen of melanovanadite from Minasragra (or Minas Ragra), near Cerro de Pasco, Peru was investigated by Barnes and Qurashi (1952) with precession photographs to determine the triclinic unit cell with *a* 6.36Å, *b* 16.86Å, *c* 6.27Å, α 90°0′, β 101°50′, and γ 93°10′. With the number of water molecules (n) of seven as indicated by their volume calculations and the number of formula units per unit cell (Z) of one, the calculated specific gravity is 2.860. However this specific gravity is significantly different from the specific gravity of 3.477 measured by Lindgren et al. (1922).

The objective of this paper is to collect powder X-ray diffraction data for melanovanadite from the holotype specimen, to calculate the unit cell of melanovanadite in order to check the unit cell of Barnes and Qurashi (1952), and to check the number of water molecules in the chemical formula of melanovanadite by comparison of the measured density and calculated density.

Methods and results

A portion of the Harvard Mineralogical Museum holotype specimen number 119652 of melanovanadite was obtained. The same holotype specimen from Minasragra, Peru was also studied by Barnes and Qurashi (1952) and Lindgren et al. (1922). A topotype specimen of melanovanadite from Minasragra, Peru (American Museum specimen number 19310) also contained sherwoodite, $Ca_9Al_2V_4^{4+}V_{24}^{5+}O_{80} \cdot 56H_2O$. Melanovanadite specimens (American Museum numbers 28897, 28898 and 28899) from Jackpole mine, Valencia County, New Mexico, U.S.A. were found to have poor crystallinity by powder X-ray diffraction. A further specimen of melanovanadite was obtained from the Jo Dandy mine (Harvard Mineralogical Museum specimen number 92477).

Nine precession photographs (zero, one and two levels along all three principal axes) were taken of specimen 92447 using Mo$K\alpha$ radiation. These precession photographs confirmed the triclinic unit cell of Barnes and Qurashi (1952), and revealed a strong *pseudo-A* centered lattice, because only a few very weak reflections with $(k + l) = 2n + 1$ were noted. Two precession photographs (0*kl*) and (*hk*0) were taken before and immediately after a melanovanadite crystal was held under vacuum at ambient temperature for 62 days, and these photographs showed no observable change in either the intensity or the position of diffraction spots. Therefore the water content in melanovanadite is more strongly bound than absorbed water.

Both long exposure and short exposure Guinier photographs of melanovanadite holotype specimen 119652 were taken with Cu$K\alpha_1$ radiation (λ = 1.5405Å). The reflection intensities of the weak reflections were estimated visually, whereas the medium and strong reflections were measured with a densitometer. Because precession photographs showed a strong *pseudo-A* centered lattice, the powder X-ray diffraction data was indexed based upon space group *A*1 ($k + l = 2n$) starting with the triclinic unit cell of Barnes and Qurashi (1952) and refined by least-squares analysis with the programme of Appleman et al. (1972) to produce a triclinic unit cell of *a* 6.357(2)Å, *b* 16.868(4)Å, *c* 6.274(2)Å, α 90.04°(2), β 101.60°(2), and γ 93.20°(3). This unit cell is similar to but more accurate than the unit cell measured by Barnes and Qurashi (1952). The values of *hkl*, d_{calc}, d_{obs} and I/I_1 are given in Table 1 for melanovanadite holotype specimen 119652.

Least-squares analysis of Gandolfi powder data from specimen 92477 gives a triclinic unit cell (space group *A*1) with *a* 6.36(1)Å, *b* 16.90(2)Å, *c* 6.300(9)Å, α 90.47°(11), β 101.42°(15), and γ 93.20°(11). The slight difference between

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Table 1. Powder X-ray diffraction data for melanovanadite

hkl	d_{calc}	d_{obs}	I/I_1	hkl	d_{calc}	d_{obs}	I/I_1
020	8.42	8.40	100	171	2.0204	2.0209	5
120	4.870	4.875	4	033	1.9320	1.9327	10
111	4.653	4.649	8	271	1.9316		
040	4.210	4.208	55	033	1.9169	1.9168	2
031	4.170	4.169	30	302	1.9051	1.9052	2
031	4.120	4.120	40	213	1.8780	1.8780	35
111	3.842	3.842	12	262	1.8769		
131	3.321	3.343	30	322	1.8764		
131	3.184	3.182	40	242	1.8337	1.8322	35
200	3.108	3.109	45	113	1.8333		
211	3.009	3.005	40	311	1.8318		
102	3.006			340	1.8185	1.8193	30
220	2.972	2.974	50	271	1.8051	1.8038	1
051	2.939	2.941	15	091	1.7961	1.7966	35
022	2.898	2.903	10	242	1.7716	1.7710	5
122	2.845	2.848	6	153	1.7713		
060	2.807	2.807	3	053	1.7598	1.7591	2
160	2.615	2.614	40	331	1.7322	1.7303	5
231	2.613			342	1.7071	1.7079	25
122	2.470	2.473	50	191	1.6656	1.6649	5
071	2.231	2.231	15	153	1.6331	1.6322	2
171	2.129	2.128	2	313	1.6172	1.6163	3
171	2.102	2.100	10	182	1.5992	1.5996	1
251	2.098			351	1.5877	1.5869	10
062	2.085	2.087	5	411	1.5857		
300	2.072	2.070	20	073	1.5692	1.5685	1
013	2.037	2.035	2	282	1.5682		
162	2.035						

the two sets of triclinic unit cell values is attributed to minor atomic substitutions in the chemical formula.

Measured density values were determined for two melanovanadite crystals from specimen 92477 by the sink-float method using successive addition of acetone (density of 0.786 gm/cc) to methelyene iodide (density of 3.32 gm/cc). The two crystals gave measured density values of 2.90 and 2.83 gm/cc, which are in agreement with the calculated value of 2.860 gm/cc for the ideal formula, $Ca_2V_4^{4+}V_6^{5+}O_{25} \cdot 7H_2O$. Thus the measured density given by Lindgren et al. (1922) was apparently determined on dehydrated material.

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