

An investigation of two new minerals: rhodesite and mountainite.

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Summary. Rhodesite, from Kimberley, S. Africa, has been examined by electron-microscope, X-ray, and other methods. The acicular crystals are orthorhombic, with a 23·8, b 6·54, c 7·05 Å.; elongation c , cleavage (100); unit-cell contents approximately $(\text{Ca}, \text{Na}_2, \text{K}_2)_8 \text{Si}_{16} \text{O}_{40} \cdot 11 \text{H}_2\text{O}$; optical properties α 1·502, β 1·505, γ 1·515; $\alpha = b$, $\beta = a$, $\gamma = c$. With it occurs a similar but distinct fibrous mineral, which appears to be a new species for which the name *mountainite* is proposed; it is monoclinic, with a 13·51, b 13·10, c 13·51 Å., β 104°; elongation b , cleavage (001); unit-cell contents approximately $(\text{Ca}, \text{Na}_2, \text{K}_2)_{16} \text{Si}_{32} \text{O}_{80} \cdot 24 \text{H}_2\text{O}$; optical properties α 1·504, β 1·510, γ 1·519; $\beta = b$ (elongation). Both rhodesite and mountainite show close similarities to the fibrous zeolites, mountainite in particular resembling thomsonite. The possible structural basis of these similarities is discussed.

E. D. MOUNTAIN (1957) has recently described the new mineral rhodesite, including details of its occurrence, chemical composition (approximately $4(\text{Ca}, \text{Na}_2, \text{K}_2)\text{O} \cdot 10\text{SiO}_2 \cdot 7\text{H}_2\text{O}$), optical properties, and behaviour on dehydration and rehydration. Prof. Mountain very kindly provided several portions of his specimen for crystallographic investigations, and the results of these form the subject of the present paper.

Several pieces of material were examined, weighing in all a few grams. All consisted of matted white fibres. To test whether the material was homogeneous, powder specimens and also several tens of individual fibres were taken from different parts of the specimen, and were examined using optical, X-ray powder and single-crystal, and electron-microscope methods. The results showed that two species were present, similar in appearance and optical properties. One was subsequently identified with rhodesite from its chemical analysis and sign of elongation. The other appears to be a further new species, for which the name *mountainite* is proposed (pronounced with the *ou* as in *house*). Some of the pieces examined consisted almost wholly of one or the other mineral; in others, both were present.¹

¹ The original portion sent by Prof. Mountain happened to consist almost wholly of mountainite. This was wrongly assumed to be rhodesite and described as such at the November 1956 meeting of the Mineralogical Society (Notice of Meeting, No. 95).

Optical data found for the two species in the present investigation are given in table I. The data for rhodesite agree closely with those reported by Mountain.

TABLE I. Optical and unit-cell data for rhodesite and mountainite.

	Rhodesite.	Mountainite.
<i>Optical properties:</i>		
Appearance:	White fibres (length c). Larger and better crystallized than those of mountainite. Laths $500 \times 100 \times 50 \mu$ appeared to be single crystals though subsequent X-ray examination showed considerable angular spread of all reflections	White fibres (length b). Even the smallest visible were polycrystalline, though ones of $400 \times 20 \times 10 \mu$ approximated to single crystals
Cleavage:	One good cleavage (100) in the prism zone	One doubtful cleavage in the prism zone
Extinction:	Parallel: + elongation (γ)	Parallel: \pm elongation (β)
Indices (Na):	α 1.502 β 1.505 γ 1.515	α 1.504* β 1.510 γ 1.519*
Orientation:	$b = \alpha$ $c = \gamma$ (elongation) $a = \beta$ (normal to cleavage)	$b = \beta$ (elongation)
Optic sign and 2V:	Probably + with low 2V	Probably + with moderate or high 2V
<i>Unit-cell data:</i> †		
Crystal system:	Orthorhombic.	Monoclinic.
a	23.8 Å.	13.51 Å.
b	6.54 Å.	13.10 Å.
c	7.05 Å.	13.51 Å.
β	90°	104°
Cell contents:	(Ca,Na ₂ ,K ₂) ₈ Si ₁₆ O ₄₀ · 11H ₂ O	(Ca,Na ₂ ,K ₂) ₁₆ Si ₃₂ O ₈₀ · 24H ₂ O

* True single crystals of mountainite might be expected to have α lower and γ higher than these values.

† The parameters given are considered the most probable ones, but for mountainite the true value of b may be 6.55 Å. and for rhodesite the cell given may be a pseudo-cell; see Discussion.

Electron-microscope investigation. Fibres of each species were identified from X-ray oscillation photographs and then crushed lightly in water. Drops of the resulting suspensions were allowed to dry on specimen grids coated with formvar films, and examined using a Metropolitan-Vickers EM-3 electron microscope. Some specimens were shadow-cast with gold-palladium alloy.

A typical electron micrograph of *rhodesite* is shown in fig. 1. The

particles were elongated laths, which in many cases had one end pointed. The electron-diffraction patterns (fig. 2) faded quickly in the electron beam, but examination was made possible by use of the specimen screening aperture described by Page and Agar (1954). If the fibre axis is called

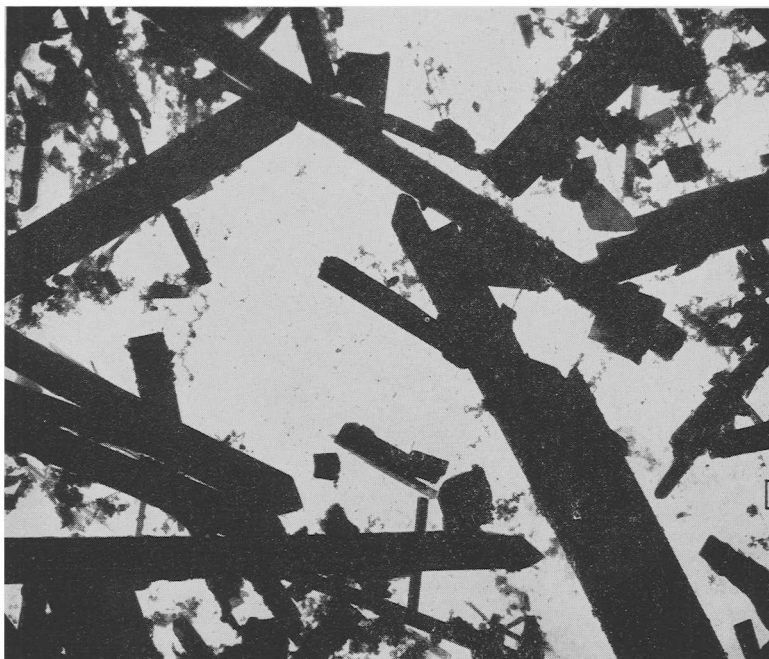


FIG. 1. Electron micrograph of rhodesite, showing laths with elongation c and principal cleavage (100). Some crystals have one end pointed. $\times 4000$.

c , and the principal cleavage (100), the electron-diffraction data give the mean values b 6.57, c 7.00 Å., α 90°. Most patterns were intersected by Laue zones if the crystals were tilted. The relative positions of the reflections in adjacent zones indicated a primitive lattice, and application of a procedure described elsewhere (Gard, 1956) gave a mean value of 25 Å. for a . The zero-order Laue zone was usually nearly central when the specimen grid was normal to the electron beam, as shown in fig. 2*a*, indicating that the a -axis is probably normal to (100), and the unit cell

is orthorhombic. Some of the patterns, one of which is shown in fig. 2*b*, had weak reflections at the face-centres of the typical spot pattern, which suggests that the true unit cell may be A-centred with doubled values for b and c . The intensity of this pattern of weak reflections varied from one crystal to another.

The electron-diffraction results, therefore, give the following provi-

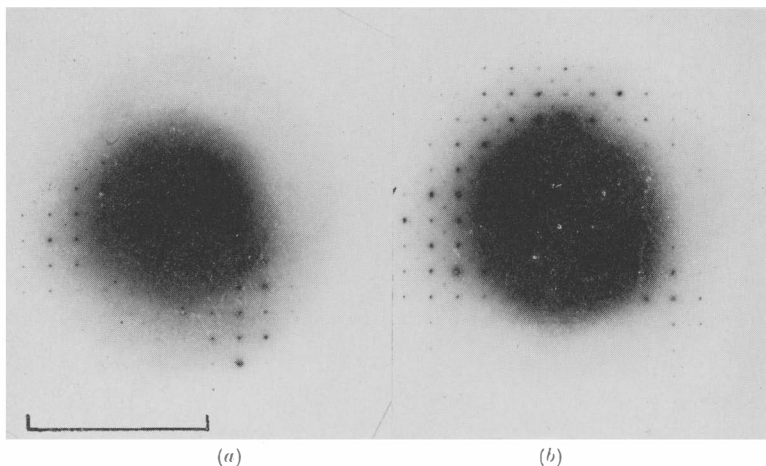


FIG. 2. Electron-diffraction patterns from single crystals of rhodesite with (100) approximately normal to the beam. Zero- and first-order Laue zones are present in each case. (a) Typical pattern with no superlattice reflections. The lattice is primitive. (b) Pattern with weak superlattice reflections giving doubled values of b and c . The scale represents 1 \AA^{-1} .

sional unit-cell data for rhodesite: orthorhombic; a 25, b 6.57, c 7.00 \AA .; elongation c , principal cleavage (100); some indication of a superlattice with doubled values of b and c .

A typical electron micrograph of *mountainite* is shown in fig. 3. The specimen consisted of laths of rectangular outline, with their principal cleavage faces lying on the supporting film. The laths were less elongated than with rhodesite. Similar electron-diffraction patterns, of which fig. 4 is typical, were given by most of the crystals. The patterns did not fade quickly, and the mineral therefore appears to be stable in the electron beam. If the cleavage is called (001) and the prism axis b , the patterns give mean values of a 13.7, b 13.1 \AA , γ 90°. These patterns appeared to be divided into Laue zones parallel to the b -axis. The centres of the zones were too far from the direct electron-beam spot to be located,

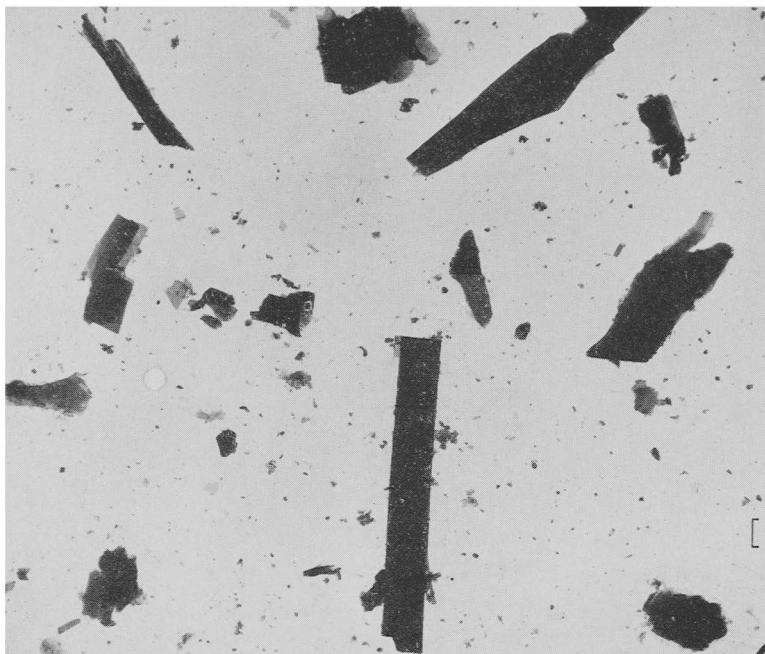


FIG. 3. Electron micrograph of mountainite showing rectangular laths with elongation b and principal cleavage (001). $\times 4000$.

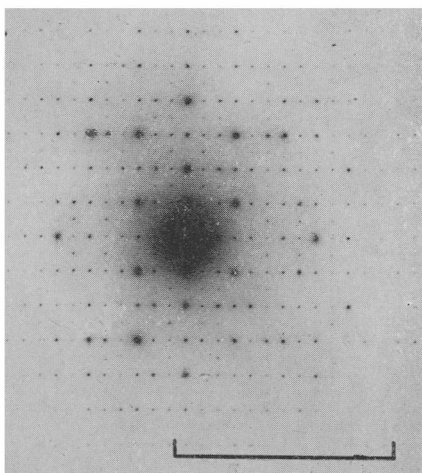


FIG. 4. A typical electron-diffraction pattern from a single crystal of mountainite, with (001) normal to the electron beam. Alternate strong and weak layer-lines are visible for even and odd values of k . The scale represents 1 \AA^{-1} .

even when the crystals were tilted through the maximum angle (*c.* 7°) possible in the tilt block of the specimen stage. This indicated that *b* coincides with *b**, but that the angle between *a* and *a** is much greater than 7°. The unit cell is thus monoclinic, and β exceeds 97°. Two fibrous crystals were lying with sufficient tilt about the fibre axis to give distinct Laue zones, as shown in fig. 5 *a* and *b*, and the relative positions of spots in adjacent zones indicated a primitive lattice. The procedure

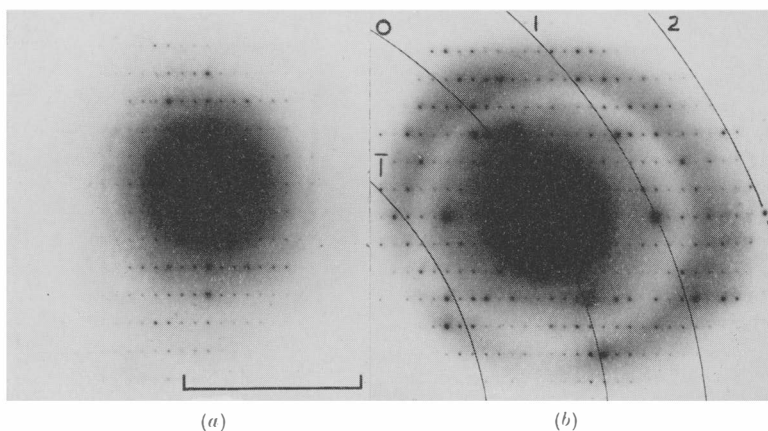


FIG. 5. Electron-diffraction patterns from the same crystal of mountainite: (*a*) with the *c*-axis nearly parallel to the electron beam; the $hk0$ Laue zone is almost central; (*b*) with the *c*-axis inclined at a few degrees to the beam; zero-, first-, and second-order Laue zones are visible and values of *l* are marked. The scale represents 1 \AA^{-1} .

described by Gard (1956) for the estimation of the third-row lattice spacing gave values of 12.8 and 10.9 Å. for *c*. All reflections with *k* odd were weak, and $0k0$ reflections with *k* odd were absent. No other systematic absences were observed. 040, 080, 320, and 360 reflections appeared to be stronger than the others. These reflections fall on a face-centred sub-lattice with *a* 4.6, *b* 6.55 Å., and may be related to the positions of the heaviest, i.e. the calcium and potassium atoms, projected on the (001) plane.

The electron-diffraction results thus give the following provisional unit-cell data for mountainite: monoclinic, *a* 13.7, *b* 13.1, *c* 12 ± 1 Å., $\beta > 97^\circ$; elongation *b*, principal cleavage (001); strong pseudo-halving of *b*.

X-ray investigation. Rotation, oscillation, and equatorial Weissenberg photographs about the prism axis were obtained for both species.

In both cases, even though small crystals ($200 \times 20 \times 10 \mu$) were used, reflections showed considerable angular spread, especially in the sense of rotation around the prism axis. Because of this, Weissenberg photographs were rather indefinite, as were any photographs taken with the crystals mounted about other axes. It was nevertheless found possible to determine the unit cells (or possibly pseudo-cells) from the oscillation and rotation photographs about the prism axes. The following results were obtained:

Rhodesite: orthorhombic; a 23.8, b 6.54, c 7.05 Å.; elongation c , principal cleavage (100).

TABLE II. X-ray powder data for rhodesite and mountainite, and for mountainite after heating at 275° C.

Rhodesite.				Mountainite.				Mountainite (heated). Observed.		
Observed.		Calc.		Observed.		Calc.				
d (Å.)	I .	hkl .	d (Å.)	d (Å.)	I .	hkl .	d (Å.)	d (Å.)	I .	
11.8	s	200	11.9	13.1	s	001	13.11	11.3	m	
7.1	vw/d	{	7.05	6.6	vs	{	201	6.59	6.4	vw
6.7			101				6.76	200		
6.56	s	010	6.54	5.9	vw	{	002	6.55	5.73	m
6.28	w	110	6.32				021	5.87		
5.95	m	400	5.95	5.4	vw	{	201	5.40	4.34	vw
5.25	vw	301	5.27				202	5.38		
5.04	w	310	5.04	4.67	s	{	221	4.65	4.06	vw
4.80	w	011	4.79				221	4.17		
4.42	m	410	4.42	3.74	w	{	321	3.72	3.9	vw/d
4.11	vw	311	4.11				023	3.66		
3.99	vw	{	3.97	3.36	mw	{	123	3.35	3.04	w
3.87	vvw	510	3.85				203	3.31		
3.76	vvw	411	3.73	3.30	w	{	204	3.29	2.86	w
3.51	vvw	002	3.53				402	3.29		
3.39	w	610	3.39	3.28	mw	{	040	3.28	2.75	w
3.27	vw	020	3.27				004	3.28		
3.07	vs	112	3.08	3.04	vw	{	124	3.02	2.89	s
3.02	s	{	3.02				420	2.95		
2.98	m	{	2.98	2.94	vvs	{	422	2.95	2.86	w
			021				2.97	224		
2.89	s	{	2.87	2.80	ms	{	223	2.95	2.76	w
			312				2.89	240		
2.78	s	{	2.78	2.75	ms	{	801	2.74	2.75	w
			711				2.77	412		

TABLE II (cont.).

Rhodesite.		Mountainite.		Mountainite (heated).		Rhodesite.		Mountainite.	
<i>d</i> (Å.)	<i>I</i> .	<i>d</i> (Å.)	<i>I</i> .	<i>d</i> (Å.)	<i>I</i> .	<i>d</i> (Å.)	<i>I</i> .	<i>d</i> (Å.)	<i>I</i> .
2.72	vvw					1.488	vw	1.495	vvw
2.67	vvw					1.453	vw/b	1.463	mw
2.63	vvw	2.65	w			1.415	w	1.436	vw
2.53	w	2.54	w			1.373	vvw/d	1.391	vw
2.49	vvw					1.368	mw	1.363	vvw
2.44	vw	2.42	vw			1.343	vw	1.342	vvw
2.25	vvw	2.32	mw			1.320	vw	1.323	w
2.20	vvw	2.23	vw			1.300	} vw/d		
2.15	w					1.291			
2.10	vvw	2.11	mw/d			1.275	w		
2.05	w					1.266	vvw	1.261	w
1.967	vw	1.967	ms			1.246	vw		
1.924	vw					1.231	vw		
1.890	vw	1.882	vw			1.222	vvw	1.226	vw
1.861	ms			1.86	vvw	1.206	vw		
1.840	vw					1.193	vvw		
1.815	vw	1.820	w			1.181	vvw	1.187	vw
1.771	vw/d			1.76	vvw	1.162	mw/b	1.162	vvw
1.756	ms					1.154	vvw	1.151	w
1.724	vvw	1.719	m			1.133	vvw	1.130	vvw
1.697	m					1.119	vvw	1.112	vw
1.695	m					1.108	vvw		
1.675	vw	1.678	vw			1.086	vvw	1.091	vw
1.650	vvw					1.067	vvw	1.074	vw
1.640	vvw	1.639	w			1.054	vvw	1.038	vvw
1.603	w					1.034	vvw	1.018	vvw
1.542	mw	1.578	vw			1.021	vw	1.011	vvw
1.522	vw							0.974	vw
1.501	vw							0.956	vvw

Mountainite: monoclinic; a 13.51, b 6.55, c 13.51 Å.; β 104°; elongation b . No low-order $h0l$ reflections were detected.

X-ray powder data (table II) were obtained for each mineral using Cu- $K\alpha$ radiation with cameras of 6 cm. and 11.46 cm. diameter. In each case the low-angle reflections were indexed from direct superposition of powder and single-crystal photographs. Indices and calculated spacings given in table II relate to the unit cells given above, except that for mountainite a doubled value of b (13.10 Å.) was assumed on the basis of the electron-diffraction result. No powder lines were observed with either mineral that could not be accounted for on the single-crystal photographs; the powder data in each case, therefore, relate to substantially pure material.

Chemical analyses and unit-cell contents. Chemical analyses were kindly

made by Dr. R. A. Chalmers of specimens of each mineral, whose purity had been checked optically and by X-ray powder photographs. Table III gives the results for rhodesite (col. 2) and for mountainite (col. 3). The mean of Mountain's two analyses of rhodesite is also included (col. 1).

Specific gravities of the two specimens analysed in this investigation were determined by suspension in bromoform-benzene mixtures, care

TABLE III. Chemical analyses and unit-cell contents for rhodesite and mountainite.

	1.	2.	3.		4.	5.
SiO ₂	61.83	61.6	58.5	Si	16.3	32.2
Al ₂ O ₃	0.29	nil	nil	Al	—	—
FeO	0.25	nil	n.d.	Fe	—	n.d.
CaO	14.90	15.1	13.4	Ca	4.3	7.9
MgO	0.08	nil	0.2	Mg	—	0.2
Na ₂ O	4.93	5.2	7.9	Na	2.7	8.4
K ₂ O	5.28	6.0	6.0	K	2.0	4.3
H ₂ O	12.50	12.3*	13.4	H ₂ O	10.9	24.7
	100.06	100.2	99.4	O (not as H ₂ O)	39.2	79.8
Sp. Gr.	2.36	2.36	2.36	—	—	—

* Total loss on ignition; may include a little CO₂.

1. Rhodesite. E. D. Mountain (1957). Mean of two analyses.

2 and 3. Rhodesite and Mountainite. Analyses of present specimens by Dr. R. A. Chalmers.

4. Atomic cell-contents calculated from analysis 2.

5. Atomic cell-contents calculated from analysis 3.

being taken to remove air by preliminary evacuation. From the unit-cell, analytical, and specific gravity data, atomic cell-contents were calculated for rhodesite (col. 4) and mountainite (col. 5). For this purpose the unit-cell dimensions found using X-rays were used, except that for mountainite a doubled value of *b* (13.10 Å.) was assumed in view of the electron-diffraction evidence.

For rhodesite the unit-cell contents approximate to 16 Si, 8 (Ca + Na₂ + K₂), 11 H₂O, and 40 O (not as H₂O). For mountainite they approximate to 32 Si, 16 (Ca + Na₂ + K₂), 24 H₂O, and 80 O (not as H₂O).

Dehydration. Some preliminary crystallographic observations have been made on crystals of each mineral after heating at various temperatures. With rhodesite no change was detected in crystals heated at 500° C. On ignition the crystals melted to a glass which gave a weak X-ray powder pattern of pseudo-wollastonite (α-CaSiO₃). With moun-

tainite no change was detected in a crystal heated at 160° C. Crystals heated at 275° C. gave a weak pattern showing only slight preferred orientation. X-ray powder data for material so heated are given in table II. A crystal heated at 400° C. gave a weak and diffuse pattern

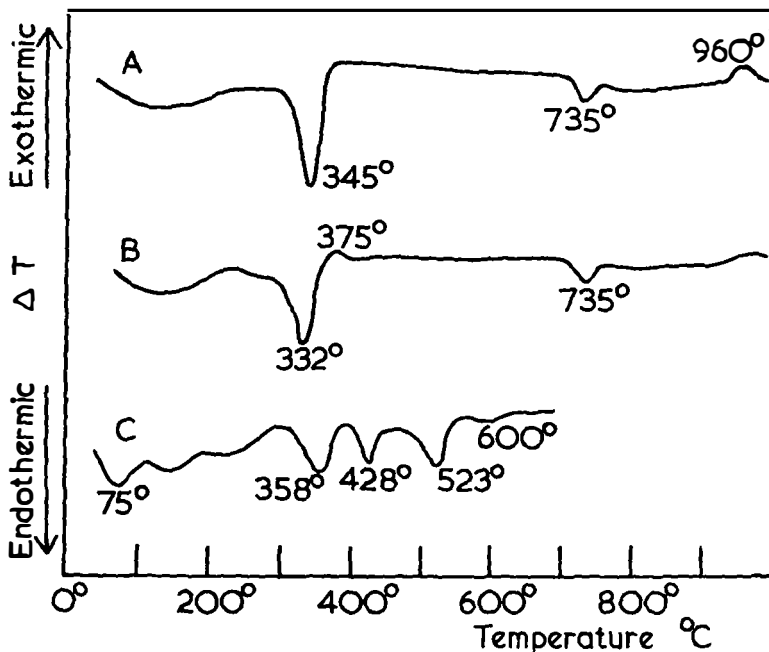


FIG. 6. Differential thermal analysis curves for A, rhodesite; B, mountainite; C, thomsonite. Curves A and B determined by Dr. R. C. Mackenzie (Macaulay Institute for Soil Research, Aberdeen); heating rate 10 deg./min. Curve C from M. Koizumi (1953); heating rate 2-3 deg./min.

similar to that obtained at 275° C.; one heated at 1000° C. melted to a glass that gave a weak pattern of β -CaSiO₃ with indications of much amorphous material.

D.T.A. curves for both minerals (fig. 6) were kindly determined by Dr. R. C. Mackenzie (Macaulay Institute for Soil Research, Aberdeen). One for thomsonite is included for comparison.

Discussion

The status of rhodesite and mountainite as mineral species. The present results show that rhodesite and mountainite are species distinct from each other. Although similar in composition and in optical properties,

there are significant differences in the latter, especially in the signs of elongation and in the indices for light vibrating parallel to the elongation. It is uncertain whether the differences between the chemical analyses are diagnostic because of the possibilities of variable composition in both cases. The two minerals are quite distinct crystallographically, and can readily be distinguished by X-ray powder photographs, or by single-crystal X-ray or electron-diffraction patterns.

Neither mineral could be correlated with any previously known species, excepting of course that described by Mountain. Rhodesite is therefore confirmed as a new species, and mountainite is shown to be an additional one.

Unit-cell data. For rhodesite, the electron-diffraction and X-ray results are in substantial agreement, and the X-ray values can be accepted as the more accurate. Electron diffraction gives indication of a superlattice for some crystals. The variable intensity of the superlattice pattern suggests that the degree of order may vary from crystal to crystal, or that it may be affected by exposure to the electron beam.

For mountainite, the X-ray results establish a pseudo-cell (a 13.51, b 6.55, c 13.51 Å., β 104°) with certainty, but there is some doubt as to the true cell and space-group because of three discrepancies between X-ray and electron-diffraction results, viz.: the electron-diffraction data show intermediate weak layer-lines corresponding to a doubled value (13.10 Å.) for b ; the X-ray data give a precise value for c , but electron-diffraction suggests an indefinite and lower value; and low-angle $h0l$ reflections cannot be detected using X-rays but are not particularly weak on the electron-diffraction patterns.

These differences could be explained in either of two ways. It could be assumed that the crystals are unaltered in the electron microscope, except possibly for lattice shrinkage in the c -direction, and the discrepancies attributed to the fact that the electron-diffraction method is the more sensitive for detection of weak reflections, but less accurate than X-ray diffraction for determination of lattice parameters. In this case the true unit cell is monoclinic with a 13.51, b 13.10, c 13.51 Å., β 104°, and the true space-group $P2_1$ or $P2_1/m$. This is perhaps the more likely explanation. At the same time, it is possible that more profound alteration occurs in the electron microscope, causing not only shrinkage along c but also doubling of b and alteration in space-group. In this case the X-ray results give the true cell for unaltered material and the true space-group is possibly $P2/a$.

Relations between rhodesite, mountainite, and the zeolites. Mountain

drew attention to the close similarity of rhodesite in many of its properties to the fibrous zeolites, and showed that this similarity extended to ready loss and recovery of the greater part of the water. A similar resemblance exists with mountainite, which shows a close crystallographic relation to thomsonite and gonnardite (M. H. Hey, 1932; H. Meixner, M. H. Hey, and A. A. Moss, 1956). Resemblances include the fibrous habit; the optical properties; the fibre-repeat or pseudo-repeat distance; the approximate values and equality of the other two axes; the number of oxygen atoms (80) and of water molecules (24) in the unit cell; and the apparent ability of Na, K, and Ca to deputize for each other in cation

TABLE IV. Correlation of the unit-cell parameters (Å.) for rhodesite and mountainite.

Rhodesite.	Mountainite.
<i>c</i> 14.1 (7.05)	<i>b</i> 13.10 (6.55)
<i>b</i> 13.1 (6.54)	<i>a</i> 13.51
<i>a</i> 23.8	<i>c</i> 13.51

positions. Differences include the absence of alumina in mountainite; the crystal system, which is orthorhombic in thomsonite; the number of cations in the cell (over 20 in mountainite, usually near 12 in thomsonite); and the greater effect of heating at 275° C. with mountainite. Thomsonite suffers only minor lattice shrinkage at this temperature (Hey, 1932).

Although no such close crystallographic relationship has been traced between rhodesite and any particular zeolite, the general similarity to the fibrous zeolites is apparent. There is also a marked resemblance to mountainite in the unit-cell contents, which are just half as great, except for a small difference in water contents. The unit-cell parameters may also be related. They are correlated in table IV with respect to their fibre axes and cleavage planes. The true cell of rhodesite is assumed to have doubled values for *b* and *c* (in accordance with the super-lattice reflections found in some of the electron-diffraction patterns), and the pseudo-cell parameters are shown in brackets.

The parameters are comparable in the cleavage planes of the two minerals, while the other parameter is roughly twice as great for rhodesite as for mountainite. The degree of order in rhodesite has been shown to be variable, and it is possible that the structures of the two minerals are closely related, but have slight distortions and differences of atomic contents that necessitate the selection of rather different axes and parameters.

In the absence of more complete structural studies, only a tentative explanation is possible of the similarity of rhodesite and mountainite to

the fibrous zeolites. In the latter there are three-dimensional anionic frameworks of empirical composition $(\text{Si,Al})\text{O}_2$, on which a negative charge arises from replacement of silicon by aluminium. Rhodesite and mountainite may possibly have incomplete anionic frameworks, derived from those of fibrous zeolites by the absence of one-fifth of the tetrahedral atoms. Such frameworks would bear a negative charge, even though no aluminium were present, because some of its oxygen atoms would be linked to only one silicon atom. The empty tetrahedral sites probably occur in regular and not random positions, because the unit cell of mountainite differs from that of thomsonite in symmetry as well as in size. The reduction in the number of tetrahedral atoms apparently makes room for an increased number of cations.

Dehydration. The present results show that the unit cell of rhodesite is substantially unaltered after heating at 500°C ., while Mountain's results show that all the water is lost, to a large extent reversibly, by 275°C . Mountainite is less stable to heat, loss of the water in this case being accompanied or followed by collapse of the structure. Both kinds of behaviour are known among zeolites.

The differential thermal analysis curves (fig. 5) of the two minerals are closely similar to each other, but differ markedly from that of thomsonite. The endothermic peaks at $332\text{--}45^\circ\text{C}$. are probably caused by dehydration, and those at 735°C . by melting. The first endothermic peaks differ slightly in shape and in position for the two minerals. There is a slight exothermic bulge at 375°C . with mountainite, but not with rhodesite; it may be associated with the structural collapse already mentioned. Mountainite shows only a slight exothermic bulge at 960°C ., but with rhodesite there is a distinct peak. This possibly corresponds with the transition from β - to α - CaSiO_3 as the X-ray results show that this probably occurs below 1000°C . in the partly devitrified glass formed from rhodesite. As both minerals melt well below this temperature, the difference between them in this respect must be attributed to the minor differences in composition and not to the differences in structure.

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