# MACALLISTERITE, 2MgO·6B<sub>2</sub>O<sub>3</sub>·15H<sub>2</sub>O, A NEW HYDROUS MAGNESIUM BORATE MINERAL FROM THE DEATH VALLEY REGION, INYO COUNTY, CALIFORNIA<sup>1</sup>

Waldemar T. Schaller, Angelina C. Vlisidis and Mary E. Mrose, U. S. Geological Survey, Washington, D. C.

#### ABSTRACT

Macallisterite is a new hydrous magnesium borate mineral, 2MgO·6B<sub>2</sub>O<sub>3</sub>·15H<sub>2</sub>O, found in the Death Valley region, California, where it occurs as small pellets of clusters of crystals intimately intermixed with ginorite and sassolite.

The new mineral is optically uniaxial negative,  $\epsilon = 1.459$ ,  $\omega = 1.504$ . Hardness  $2\frac{1}{2}$ , specific gravity 1.868.

Macallisterite is trigonal; the space group is R3c (non-piezoelectric); hexagonal  $a=11.546\pm0.001,\ c=35.562\pm0.005$  Å, c/a=3.080; rhombohedral  $a_{rh}=13.600\pm0.003$  Å,  $\alpha=50^{\circ}14'$ ; volume 4105.7 ų (hex.); cell contents:  $6[2\mathrm{MgO}\cdot 6\mathrm{B}_2\mathrm{O}_3\cdot 15\mathrm{H}_2\mathrm{O}]$ , in the hexagonal unit. Crystals are rhombohedral scalenohedral (32/m); the only forms observed are  $c\{0001\},f\{10\overline{1}4\}$ , and  $e\{01\overline{1}2\}$ . Strongest indexed x-ray lines (in Å) are: 8.715 (100) (01.2); 5.772 (50) (11.0); 4.063 (50) (10.8); 3.351 (50) (10.10); 3.261 (50) (11.9); 4.359 (42) (02.4); 6.645 (35) (10.4).

Two chemical analyses of samples about three-quarters macallisterite gave, after deduction of impurities: MgO 10.06, 10.97;  $B_2O_3$  53.43, 53.17;  $H_2O$  36.51, 35.86; Total 100.00.

The new mineral is named after James F. McAllister, geologist, U. S. Geological Survey, who first detected its presence in the Death Valley region. The compound was first synthesized by Rammelsberg in 1840 and since then by many others.

#### Introduction

The hydrous magnesium borate  $2MgO \cdot 6B_2O_3 \cdot 15H_2O$ , known for more than a hundred years as a synthetic product, was first noted as a natural mineral in October, 1954, when James F. McAllister, geologist, U. S. Geological Survey, after whom the mineral is named,² collected specimens containing small aggregates of minute crystals of the new mineral at the open cut of the Mott prospect in the Furnace Creek Wash area, Death Valley region, California.

At about this same time a small sample of the synthetic compound  $2 \text{MgO} \cdot 6 \text{B}_2 \text{O}_3 \cdot 15 \text{H}_2 \text{O}$ , made by D'Ans and Behrendt (1957), the composition of which was established by their chemical analyses, was received through the kindness of Dr. Robert Kühn of Hannover, Germany. X-ray powder diffraction patterns of our mineral showed that identity with the pattern of the synthetic product was complete. Such identity was later confirmed by determinations of the optical properties of the two samples and by chemical analyses of the California mineral.

<sup>&</sup>lt;sup>1</sup> Publication authorized by the Director, U. S. Geological Survey, Washington, D. C.

<sup>&</sup>lt;sup>2</sup> The name macallisterite has been approved in advance of publication by the "Commission of New Minerals and Mineral Names, I.M.A."

#### OCCURRENCE

The first macallisterite collected occurred as a very minor constituent of an efflorescent layer between colemanite and priceite-bearing shale and a surface crust of powdery clay on a steep hillside at the Mott colemanite prospect, near the head of Twenty Mule Team Canyon, northwest of, and downstream from, a diversion channel in SW<sup>1</sup>/<sub>4</sub> sec. 9, T. 26N., R. 2 E, SBM, in the Furnace Creek Wash area, Death Valley region, Inyo County, California. The prospect was opened because of the borate minerals present, essentially colemanite and priceite. The macallisterite occurred very sparingly in a pale yellowish-brown matrix of sassolite and clay with closely associated small white pellets of ginorite (2CaO ·7B<sub>2</sub>O<sub>3</sub> · 8H<sub>2</sub>O), as described by Allen and Kramer (1957).

In its occurrences macallisterite is closely associated with other boron minerals, particularly ginorite and sassolite. Many of the snow-white pellets, on sight believed to be pure macallisterite, were found to be intimate mixtures with ginorite. Occasionally, small glassy clusters of sugary crystals not more than a millimeter across are encountered.

Most of the new mineral is in small rounded pellets generally a millimeter or two in size, though occasionally clusters of pellets extend for a centimeter or so. The white pellets, and some may be called snow-white, stand out prominently from their tawny colored matrix of sassolite and clay.

Macallisterite is the "unidentified mineral" of Allen and Kramer (1957) and the "magnesium borate of uncertain identity" mentioned by Erd et al., (1959, p. 913). It is the eighth boron mineral from the weathered colemanite and priceite veins in the altered olivine basalts of the area. Other boron minerals are ulexite, hydroboracite, gowerite, nobleite, ginorite, meyerhofferite and sassolite. The list is not yet complete, for Erd, McAllister and Vlisidis (1961, p. 561) mention as an additional mineral "an undescribed calcium borate."

## PHYSICAL AND OPTICAL PROPERTIES

The minute crystals of macallisterite, mostly much less than a millimeter across, are colorless (unless stained by clay) and occasionally show a rhombohedral development in their faces. The natural crystals are larger than the closely associated crystals of ginorite but smaller than the associated plates of sassolite.

Both natural and synthetic crystals are optically uniaxial negative. Because the synthetic crystals are so much larger and better developed than the natural ones, they were used for the accurate measurements of the indices of refraction and of the specific gravity. The results obtained by Joseph J. Fahey, using checked oils of immersion, are as follows:

```
D'Ans and Behrendt synthetic material, \epsilon=1.459, \ \omega=1.504
Schaller synthetic material, \epsilon=1.458, \ \omega=1.504
```

The values  $\epsilon = 1.459$  and  $\omega = 1.504$  are taken for macallisterite. Nikolaev and Chelishcheva (1940) obtained  $\epsilon = 1.463$  and  $\omega = 1.508$ , for their synthetic material.

Very similar values for the natural mineral were obtained:

Allen 
$$\epsilon = 1.465, \ \omega = 1.505$$
  
Schaller  $\epsilon = 1.464, \ \omega = 1.507$ 

The specific gravity of the synthetic material, as determined by the pycnometer method described by Fahey (1961), gave him a value of 1.868. Chelishcheva (1940) gave 1.86. The hardness is about  $2\frac{1}{2}$ .

## MORPHOLOGY

On all the available specimens of macallisterite, the mineral occurs as very fine aggregates of minute, colorless crystals, the largest observed crystal attaining a diameter less than 0.2 mm. At best, macallisterite crystals are poorly developed. Under the binocular microscope with magnification  $200\times$  a number of euhedral crystals were observed. These showed the same rhombohedral development noted on crystals of  $2\text{MgO} \cdot 6\text{B}_2\text{O}_3 \cdot 15\text{H}_2\text{O}$  from Schaller's borate experiments. The synthetic crystals of macallisterite were chosen for morphological and single-crystal x-ray study because they were larger than any observed crystals of natural macallisterite and had well-developed crystal faces (Fig. 1).

Four well-formed crystals of synthetic macallisterite were measured goniometrically; all faces gave fair or good signals. The crystals are tabular on  $\{0001\}$ . Only three forms were observed:  $c\{0001\}$ ,  $f\{10\overline{1}4\}$ , and  $e\{01\overline{1}2\}$ . No evidence was found of less than holohedral symmetry (rhombohedral scalenohedral  $\overline{3}2/m$ ), although general forms are not present. The absence of any positive piezoelectric response to crystals of synthetic macallisterite by the Giebe-Scheibe apparatus supports the assumption for the crystal class  $\overline{3}2/m$ .

#### SINGLE-CRYSTAL X-RAY DATA

Two crystals of synthetic macallisterite, about 0.4 mm. in diameter, were investigated by the Buerger precession method and yielded these crystallographic data: possible space groups,  $R\overline{3}c$  and R3c; hexagonal  $a=11.55\pm0.01$  Å,  $c=35.55\pm0.03$ . If the crystal class is taken to be scalenohedral  $(\overline{3}2/m)$  as is indicated by the form development and the absence of piezoelectric effects, the space group is established as  $R\overline{3}c$ . The

unit-cell constants for natural macallisterite were derived by least-squares analysis of the measured x-ray powder data of the mineral (Evans  $et\ al.$ , 1963). The unit-cell parameters for macallisterite and synthetic  $2\text{MgO} \cdot 6\text{B}_2\text{O}_3 \cdot 15\text{H}_2\text{O}$  obtained with the least-squares refinement technique of Evans  $et\ al.$  (1963), together with their standard errors, are compared in Table 1.

### X-RAY POWDER DATA

X-ray powder diffraction patterns were prepared from crystals of

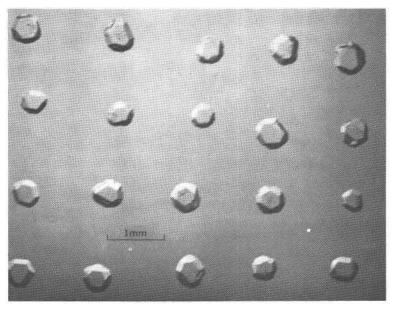


Fig. 1. Some of the larger well-formed crystals of synthetic macallisterite,  $2MgO \cdot 6B_2O_3 \cdot 15H_2O$ .  $15 \times$ . Schaller Photo 1686.

2MgO·6B<sub>2</sub>O<sub>3</sub>·15H<sub>2</sub>O synthesized by Schaller and by D'Ans and Behrendt (1957) for comparison with those from macallisterite selected from the various specimens collected in the Furnace Creek Wash area. All powder patterns of synthetic 2MgO·6B<sub>2</sub>O<sub>3</sub>·15H<sub>2</sub>O are identical. Except for the presence of small amounts of ginorite as impurity, all powder patterns of macallisterite are identical to those of the synthetic preparations. It was impossible to obtain an impurity-free x-ray powder pattern of macallisterite; a pattern prepared from very carefully handpicked, coarsely crystallized macallisterite (film no. 17448) showed the strongest line of ginorite present as a weak line with intensity 3. The

x-ray powder patterns taken of material from other samples of macallisterite contain the lines of gypsum, ginorite, and sassolite in varying amounts.

Indexed x-ray powder data for macallisterite and synthetic 2MgO  $\cdot 6B_2O_3 \cdot 15H_2O$  are given in Table 2. Powder data by Lehmann and Papenfuss (1959) for their synthetic crystals of MgO  $\cdot 3B_2O_3 \cdot 7.5H_2O$ 

Table 1. Crystallographic Data Compared for Synthetic  $2MgO \cdot 6B_2O_3 \cdot 15H_2O$  and Macallisterite

	Synthetic 2MgO+6B <sub>2</sub> O <sub>3</sub> +15H <sub>2</sub> O <sup>1</sup>	Macallisterite <sup>1</sup>
Symmetry	Trigonal	Trigonal
a	$11.549 \pm 0.002 \text{ Å}$	11.546±0.001 Å
C	$35.567 \pm 0.008$	$35.562 \pm 0.005$
c/a	3.080	3.080
$a_{\tau h}$	$13.602 \pm 0.004 \text{ Å}$	$13.600 \pm 0.003 \text{ Å}$
α	50°14′	50°14′
Space Group	R3c (non-piezoelectric)	$R\overline{3}c$ (non-piezoelectric)
Cell Volume	4108 4 ų	4105.7 ų
Vol/O-atom	19.6 ų	19.6 ų
Cell Contents (hex.)	6[2MgO 6B <sub>2</sub> O <sub>3</sub> · 15H <sub>2</sub> O]	6[2MgO · 6B <sub>2</sub> O <sub>3</sub> · 15H <sub>2</sub> O]
Specific Gravity	,	
(calc.)	1.864	1.866
(meas.)	1.8682	
Mean meas. n	$1.489^{3}$	1.489
Mean calc. n	1.483	1.485;4 1.4845

 $<sup>^1</sup>$  Unit-cell constants obtained by least-squares analysis of powder data, using for the initial cycle the preliminary cell data from measurements of single-crystal precession photographs of synthetic  $2 MgO \cdot 6 B_2 O_3 \cdot 15 H_2 O$ .

 $(=2 {\rm MgO} \cdot 6B_2O_3 \cdot 15H_2O)$  are compared in Table 2 to the data of the present study. The evidence in Table 2 clearly demonstrates that the mineral macallisterite and the synthetic crystals of  $2 {\rm MgO} \cdot 6B_2O_3 \cdot 15H_2O$  are the same compound.

## CHEMICAL PROPERTIES

As it was not possible to obtain a sizable quantity of the uncontaminated mineral, the chemical properties were determined on synthetic material. The compound is readily soluble in acids but only slightly soluble in water at room temperature ( $\pm 25^{\circ}$ C). The solubility of one gram in

<sup>&</sup>lt;sup>2</sup> Determined by the pycnometer method by Joseph J. Fahey.

<sup>&</sup>lt;sup>3</sup> Average of three sets of measurements (see text).

<sup>&</sup>lt;sup>4</sup> Calculated for analysis sample C.

<sup>&</sup>lt;sup>5</sup> Calculated for analysis sample B.

Table 2. Indexed X-ray Powder Data Compared for Macallisterite and Synthetic  $2MgO\cdot 6B_2O_3\cdot 15H_2O$ 

	Macallisterite Synthetic 2MgO						6B <sub>2</sub> O <sub>3</sub> - 15H <sub>2</sub>	0	
hkl	Present study						T ob	mann and	
	area,	e Creek Was Death Valley n, California	7	Schaller Synthetic			Lehmann and Papenfuss (1959) <sup>1</sup>		
	desle. <sup>2</sup> (Å)	d <sub>meas, 3</sub> (Å)	15	d <sub>calc</sub> ² (Å)	d <sub>meas.4</sub> (Å)	I <sub>0</sub>	$\theta/2$	d <sub>meas</sub> (kX)	
00.3	11.854			11.856					
01.2	8,716	8.715	100	8.718	8.716	100			
10.4	6.644	6.645	35	6.645	6.647	25	6.6	6.71	m
00.6	5.927	5,926	4	5.928	5.929	4			
01.5	5.796	01020		5.797		6.500			
11.0	5.773	5.772	50	5:774	5.779	35	7.6	5.83	st
11.3	5.190	3.772	50	5.191	0 /	25.80	516,550		
02.1	4.951			4.952					
20.2	4.813	4.815	4	4.814	4-818	3	9.1	4.87	55
02.4	4.358	4.359	42	4.359	4.357	30	10.2	4.35	st
11.6	4.136	4.138	2	4.136	1.001				
10.8	4.130	4.130	2	4.150					
01.8	4.062	4.063	50	4.063	4.062	35	10.9	4.08	st
00.9	3.951	4.003	30	3.952	1,002	200	1013		
21.0	3.779			3.780					
21.1	3.758	3-758	6	3.759	3.761	3	11.8	3.77	55
02.7	3:563	3.562	2	3.564	0.101		4.000		
21.4	3.478	3.480	21	3.479	3.476	18	12.7	3.51	m
	3.351	3.460	50	3.351	3.351	42	13.1	3.40	S
10.10		3.331	50	3.334	0,001		10.1		-
30.0	3 - 333			3.323					
20_8	3.322	2 261	50	3.323	3.261	35	13:5	3.30	55
11.9	3,261	3.261	50	3.209	3,201		15.5	0.00	- 397
03.3	3.209			3.077					
01.11	3.076	3.033	6	3.033	3.033	6	14.6	3.06	SS
21.7	3.032	2.964	6	2.964	2.962	4	11:0	1	-
00.12	2,963	2.904	0	2 904	2.902				
30 .6	2 005	2-903	21D	2.906	2.904	18	15.2	2.94	m
03.6	2.905	2.903	2110	2.900	2.701	100	10000		
20.10	2.000			2.898					
02 - 10	2.898	2-887	9	2.896	2.890	-6			
22.0	2.887	2.805	50	2.805	2.897	50	15.9	2.81	st
22 - 3	2.805	4 :003	50	2.741	4.007	9595	3434.60	DOTES!	ii.
31.2	2.740	2 627	3	2.637	2.636	3	16.9	2.65	BS
11.12	2,636	2.637	3	2.590	4,000	95	10.7		-
21.10	2,590			2.584	2.582	2			
31.5	2.584			4.304	4.562				

<sup>1</sup> Camera diameter 57.4 mm; Ni-filtered Cu radiation,  $\theta/2$  corresponds to Bragg  $\theta$ ; d<sub>meas</sub>. obtained by present authors from  $\theta/2$  values; intensity notations: sst=very strong, st=strong, m=medium, s=weak, ss=very weak.

 $<sup>^2</sup>$  Interplanar spacings (d<sub>eale.</sub>) calculated from refined cell parameters given in Table 1. All possible lines listed down to d=2.500 Å; only values corresponding to observed lines listed below d=2.500 Å.

<sup>&</sup>lt;sup>3</sup> Film no. 17448. A line with intensity 3 at d=7.110 Å, attributed to ginorite as an impurity, omitted from tabulated data. Camera diameter 114.59 mm; Ni-filtered Cu radiation,  $\lambda(\text{CuK}\alpha)$ =1.5418 Å; film shrinkage negligible; D=diffuse. Lower limit of 20 measurable, approximately 7° (12.6 Å).

<sup>&</sup>lt;sup>4</sup> Film no. 18481, camera diameter 114.59 mm; Ni-filtered Cu radiation,  $\lambda(\text{CuK}\alpha) = 1.5418 \text{ Å}$ ; film measurements corrected for expansion. Lower limit of  $2\theta$  measurable, approximately 7° (12.6 Å).

<sup>&</sup>lt;sup>5</sup> Estimated visually by comparison with a calibrated intensity strip.

Table 2—(continued)

	Macallisterite Synthetic 2MgO⋅6B₂O₃⋅15H₂O										
hk1	Present study										
	Furnace Creek Wash area, Death Valley region, California			Schaller Synthetic			Lehmann and Papenfuss (1959) <sup>1</sup>				
	d <sub>cate</sub> ,² (Â)	d <sub>meas</sub> , <sup>3</sup> (Å)	Is	d <sub>cate</sub> , <sup>2</sup> (Å)	d <sub>mens.</sub> 4 (Å)	I	θ/2	d <sub>meas</sub> (kX)	I		
3.9 0.14)	2.548			2.548							
01.14) 00.15	2.462 2.371	2,462	18	2.462 2.371	2 463	13	18.2 18.9	2.47	m		
0.14	2.265	2.265	25	2,265	2.266	18	19.7	2.29	st		
2.4	2,221	2.222	6	2.222	2.222	6		- 1-18-01	145		
4.8	2.178	2.178	13	2.179	2.177	11	20.6	2.19	m		
1.3	2.146	2.146	2	2.146	2.145	3					
1.11	2.105	2.106	18	2.105	2.107	13	21.4	2.11	st		
2.7	2.091	2.090	3	2.091	2.091	2					
2.12	2.068	2.067	2	2.068			1				
0.10	2.045	2.042	4	2.046	2.044	2	22.0	2.06	55		
2.16	2.031			2.031			22.6	2.01	SS		
		1.985	6		1.986	3					
		1.926	25		1.927	25	23.5	1.933	st		
		1.870	6		1.869	4	24.1	1.888	S		
		1.829	4D		1.824	3	24.8	1.838	S		
		1.781	2								
		1.757	3		1.757	2	25.9	1.765	S		
		1.738	1		4 (00						
		1.700	15		1.699	13	26.7	1.716	m		
		1.665 1.613	8		1.664	11	27.3	1.681	m		
		1.600	2		1.613	9	28.4	1.621	m		
TI III		1.546	1		1.602	1	20. 6	1 561			
		1.526	2		1.546 1.526	3	29.6 30.2	1.561 1.533	SS		
		1.502	2		1.501	3	30.2	1.514	SS		
		1.465	2		1.465	1	31.6	1.471	SS		
		1.437	2		1.435	2	32.3	1.443	SS		
		1.434	2			~	53.0		.,3		
		1.408	3D		1.407	3	33.0	1.415	S		
		1.375	1				34.2	1.372	S		
		1.355	2		1.357	2					
		1.338	1		1.335	1					
		1.322	4		1.321	4	35.5	1.328	m		
							Plus ten a	dditional lir	es all		
- 1							with I=ss	or s.			

75 cc or  $1\frac{1}{3}$  parts in 100 parts of water, reported by Rammelsberg (1840), was confirmed. Accurate determinations under controlled conditions may increase this amount slightly. One and a third grams of coarsely ground synthetic material took three days at room temperature, with occasional shaking during the day, to dissolve completely in 100 cc. of water; whereas finely ground material (<100 mesh) completely dissolved in one day under the same conditions.

At about 50°-70° C. the solubility increases somewhat. The solution remains clear until heated to about 80° C. At this temperature a cloud-like precipitate forms which remains insoluble on further heating but redissolves on cooling. This reaction can be repeated many times on the same sample.

A solution containing an excess of undissolved synthetic macallisterite at room temperature will, in time, yield prismatic crystals of synthetic inderite and disc-like aggregates of synthetic kurnakovite; these crystal-

Table 3. Loss of Water of Synthetic Macallisterite at Increasing Temperatures. Half-gram Sample was Held at Each Stated Temperature for 16 Hours

emperature (° C.)	Per cent H <sub>2</sub> O lost	Total per cent H <sub>2</sub> O lost
105	0.00	0.00
115	0.00	0.00
130	5.10	5.10
140	17.52	22.62
150	2.96	25.58
180	2.12	27.70
200	1.20	28.90
280	3.48	32.38
280	0.34	32.72
400	2.00	34.72
400	0.14	34.86
450	0.22	35.08
450	0.16	35.24
	35.24	35.24 (Calc. 35.15

lize on the surface of undissolved macallisterite as well as on the walls of the container for a vertical distance of several centimeters.

The loss of water at increasing temperatures is shown in Table 3. At temperatures 280°, 400°, and 450° C., the sample was held an additional 16 hour period. The water seems to be given off in fairly definite steps. None is lost at either 105° or at 115° C. In this respect macallisterite differs materially from both inderite and kurnakovite (dimorphs of 2MgO·3B<sub>2</sub>O<sub>3</sub>·15H<sub>2</sub>O), which lose much water below 100° C. When a temperature of 130° C. has been reached by the macallisterite, a little more than two molecules of water are lost. At 140° C. half the total water has been drive off. The loss of small amounts of water then continues with rising temperatures. When 450° C. has been reached, all the water (35.24 per cent) has been driven off.

Heated in a closed tube, the crystals become opaque, give off water, but do not fuse. Heated in the blowpipe flame, they fuse with difficulty to an opaque enamel, giving off greenish flashes.

## CHEMICAL COMPOSITION

It was hoped that by careful selection of pellets a nearly pure sample of macallisterite could be obtained. This was not feasible. The two samples (B and C) from one specimen were only about three-quarters macallisterite.

Analysis of impure		C	ation of per of impuritie nalyzed san	S	Macall	isterite	Percentage of impurities in	
	mple			Remain- der	Recalc. 100%	analyzed sample		
$\mathrm{B_{2}O_{3}}$	50.52		10.39	-	40.13	53.43	Gypsum 0.51	
CaO	2.56	0.17	2.39	_			Ginorite 15.85	
MgO	7.56	-	-	-	7.56	10.06	R <sub>2</sub> O <sub>3</sub> 1.32	
$R_2O_3$	1.32	-		1.32	-	-	Insol. 7.32	
$SO_3$	0.24	0.24	-	_	10000	-	Σ Imp. 25.00	
Insol.	7.32		_	7.32	-	-	Macall. 75,11	
$\mathrm{H_{2}O}$	30.59	0.10	3.07		27.42	36.51	100.11	
	-		-		-			
Total	100.11	0.51	15.85	8.64	75.11	100.00		

TABLE 4. MACALLISTERITE-SAMPLE B

Selected pellets were first hand-picked from a small specimen. These pellets were then divided into two groups, largely on the basis of color. Sample C weighing 2.8 grams was very fine grained. No individual crystals could be observed with a 10× hand lens. The pellets were very similar in appearance to the pellets of ginorite described by Allen and Kramer (1957). Sample B weighing 3.7 grams was much coarser, though still very fine grained, and was more grayish and dirty looking than sample C. After hand picking, both samples were subjected to supersonic treatment, using acetone, which loosened some but not all of the gangue. Both samples were crushed to pass 48 mesh and the small amount of resulting dust was not removed.

The analyses of material from each of the two groups (analyzed samples B and C), with their interpretation, are given in Tables 4 and 5. In deducting the admixed impurities present in the samples analyzed (Tables 4 and 5), all the SO<sub>3</sub> is allocated to gypsum, the presence of

which, in other samples, was confirmed by x-ray patterns. The remaining CaO is calculated as ginorite, though other calcium borates (ulexite, gowerite, etc.) may be present in very small amounts. A little Na<sub>2</sub>O is present in all samples, either as ulexite or more likely as dehydrated mirabilite. Qualitative tests indicated the absence of carbonates and chlorides. Only a faint trace of phosphate was obtained (animal bones?). The iron and other  $R_2O_3$  elements are largely due to admixed clay. Both

Analysis of		0	ation of per f impurities alyzed samp		Macall	isterite	Percentage of impurities in analyzed	
	pure mple	Gypsum	Ginorite	R <sub>2</sub> O <sub>3</sub> + Insol.	Remain- der	Recalc. 100%	samp	ole
$B_2O_3$	55.77	-	18.56		37.21	53.17	Gypsum	0.51
CaO	4.44	0.17	4.27	-	-	400	Ginorite	28.32
MgO	7.68	=	_	200	7.68	10.97	$R_2O_3$	0.36
$R_2O_3$	0.36	-		0.36			Insol.	1.00
SO <sub>3</sub>	0.24	0.24	-		_	2000	Σ Imp.	30.19
Insol.	1.00	95	_	1.00	_		Macall.	69.99
$\mathrm{H_{2}O}$	30.69	0.10	5.49	-	25.10	35.86		100.18
	_	-						
Total	100.18	0.51	28.32	1.36	69.99	100.00		

TABLE 5. MACALLISTERITE—SAMPLE C

samples contain minute fragments of altered volcanic rock which with clay furnish the  $\rm Fe_2O_3, Al_2O_3,$  and silica reported.

#### SYNTHESIS

Macallisterite is readily synthesized. In addition to the method commonly used, it can be obtained by several other procedures. The usual method is to dissolve either MgO, Mg(OH)<sub>2</sub>, or MgCO<sub>3</sub> in a hot water solution of boric acid and to let the solution stand. Kurnakova (1953) in her study of kaliborite obtained 2MgO·6B<sub>2</sub>O<sub>3</sub>·15H<sub>2</sub>O. We obtained good crystals by dissolving inderite or kurnakovite in boric acid solution; by adding ammonia to the remaining mother liquor from which a first crop of macallisterite crystals had been removed; and by adding magnesium hydroxide and boric acid in the solid state to a jar of cold water and

<sup>&</sup>lt;sup>1</sup> Our thanks are due Dr. Robert Kühn of Hannover, Germany, who called our attention to some of the earlier syntheses of this compound.

shaking vigorously and frequently for a day or so. The first new product formed is an amorphous-appearing, very fine grained precipitate in which clear crystals of  $2MgO \cdot 6B_2O_3 \cdot 15H_2O$  form and grow (Fig. 2).

In our experiments minute crystals of macallisterite appeared in a few weeks in the prepared solutions. In one experiment, minute crystals formed overnight, but generally it takes several weeks to form usable crystals. In other experiments, crystals two millimeters across took four years to form. The crystals grown by D'Ans and Behrendt (1957) are

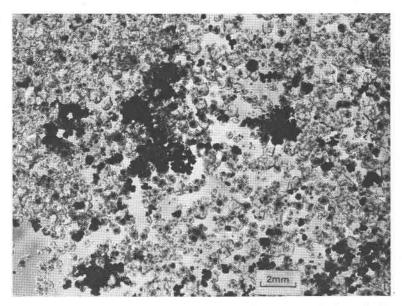


Fig. 2. Grouping of many crystals of synthetic macallisterite in the fine-grained amorphous appearing precipitate. 4½ ×. Schaller Photo 1564.

very minute; most are about 0.004 millimeter thick, the maximum found being 0.08 millimeter.

Attempts to produce crystals of macallisterite by evaporation at room temperature of a water solution (1.3 grams in 100 cc.) were not successful; the resultant product repeatedly was an amorphous glass of index of refraction 1.467. The mean index of refraction of macallisterite is 1.489. However, if boric acid is added to the solution, a mixture of crystals of macallisterite and sassolite is obtained.

#### REFERENCES

ALLEN, R. D. AND HENRY KRAMER (1957) Ginorite and sassolite from Death Valley, California. Am. Mineral. 42, 56-61.

- Chelishcheva, A. G. (1940) Heating curves and specific weights of synthetic borates. *Akad. Nauk SSSR Doklady*, **28**, 508-511.
- D'Ans, Jean and Karl-Heinz Behrendt (1957) Über die Existenzbedingungen einiger Magnesium Borate. Kali Steinsalz, 2, 121-137.
- ERD, R. C., J. F. McAllister and Hy Almond (1959) Gowerite, a new hydrous calcium borate, from the Death Valley region, California. *Am. Mineral.* **44**, 911–919.
- J. F. McAllister and A. C. Vlisidis (1961) Noblette, another new hydrous calcium borate from the Death Valley region, California. Am. Mineral. 46, 560-571.
- Evans, Howard T., Jr., Daniel E. Appleman and David S. Handwerker (1963) The The least squares refinement of crystal unit cells with powder diffraction data by an automatic computer indexing method (abs. E-1). *Program and Abstracts. Am. Crystal. Assoc. Meet.*, Cambridge, Mass., 42.
- FAHEY, J. J. (1961) A method for determining the specific gravity of sand and ground rock or minerals. U. S. Geol. Survey Prof. Paper 424-C, C372-373.
- Kurnakova, A. G. (1953) Ternary and quaternary systems containing boric acid. *Akad. Nauk Latviiskoi SSR*, *Riga*, Khimiya Boratov (Chemistry of the borates) 45–66, (In Russian).
- Lehmann, Hans-Albert and Hans-Jürgen Papenfuss (1959) Über ein wasserhaltiges Magnesiumhexaborat der Formel MgB<sub>6</sub>O<sub>10</sub>·SH<sub>2</sub>O. Zeit. anorg. allg. Chemie, **301**, 228–232.
- Nikolaev, A. V. and A. G. Chelishcheva (1940) The 25° isotherm of the systems: CaO+B<sub>2</sub>O<sub>3</sub>+H<sub>2</sub>O and MgO+B<sub>2</sub>O<sub>3</sub>+H<sub>2</sub>O. Akad. Nauk SSSR Doklady, 28, 127-130.
- RAMMELSBERG, CARL (1840) Ueber die chemische Zusammensetzung des Boracits, so wie diejenige der Verbindungen der Borsäure mit der Talkerde überhaupt. Ann. Physik Chemie (Poggendorff), 49, 445–460.
- Manuscript received, September 25, 1964; accepted for publication, January 26, 1965.