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MACALLISTERITE, $2MgO \cdot 6B_2O_3 \cdot 15H_2O$, FROM SALTA, ARGENTINA[1]

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ABSTRACT

Amber colored crystals of macallisterite as large as 12 mm in maximum dimension occur in nodules with rivadavite in a plug-like deposit of massive borax at the Tincalayu Mine, Salta, Argentina. Morphological measurements showed the presence of the forms: c $\{0001\}$, f $\{10\bar{1}4\}$, e $\{01\bar{1}2\}$ and yielded the axial ratio $a:c=1:3.082$. There is good $\{0001\}$ and $\{01\bar{1}2\}$ cleavage. The specific gravity is 1.867 (meas.), 1.867_3 (calc.). The hardness is $2\frac{1}{2}$. Optically uniaxial (−), $\omega=1.5070$, $\epsilon=1.4635$ (Na light ± 0.0003). Space group, $R\bar{3}c$; $a=11.543$ Å, $c=35.556$ Å (± 0.005 Å); cell volume 4,102.8 Å^3; $Z=6$. Chemical analysis in weight percent: B_2O_3 53.6, FeO 0.25, MnO 0.04, MgO 10.4, H_2O+34.9, H_2O-0.76, total 99.95 yields the formula: $2.04(Mg,Fe,Mn)O \cdot 6B_2O_3 \cdot 15.10H_2O$. D. T. A. shows prominent endothermic peaks with maxima at 230°C and 1025°C and two exothermic peaks at 720°C (minor) and 785°C (major).

INTRODUCTION

Macallisterite, $2MgO \cdot 6B_2O_3 \cdot 15H_2O$, was described as a new mineral by Schaller, Vlisidis and Mrose (1965). It was found as an efflorescent product in the Death Valley region of California. Also in 1965, Ch'u, Han, Chien, Liu and Min described the same compound as an efflorescence from a continental saline lake in China under the name of trigonomagneborite. The identity of these two minerals was pointed out by Fleischer (1965) who properly discredited the name trigonomagneborite, for not only was the manuscript on macallisterite received for publication prior to the other, but the name was approved by the I.M.A. Commission on New Minerals and Mineral Names in 1963.

This mineral was earlier reported from the Death Valley region by Allen and Kramer (1957) and by Erd *et al.*, (1959) although mineralogical descriptions were not carried out. This paper describes the third natural occurrence of macallisterite and the first one in which it is a primary mineral. The synthetic compound has been known for a long time (Rammelsberg, 1840).

OCCURRENCE

The macallisterite specimen used in the present study came to our attention through Dr. Luis Conti who in 1964 collected it at the Tincalayu Mine, Salta, Argentina (Fig. 1). Muessig and Allen (1957) in studying this mine concluded that original borate playa deposits were buried and subsequently folded and faulted forming a plug-like body

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[1] Mineralogical Contribution No. 439, Harvard University.

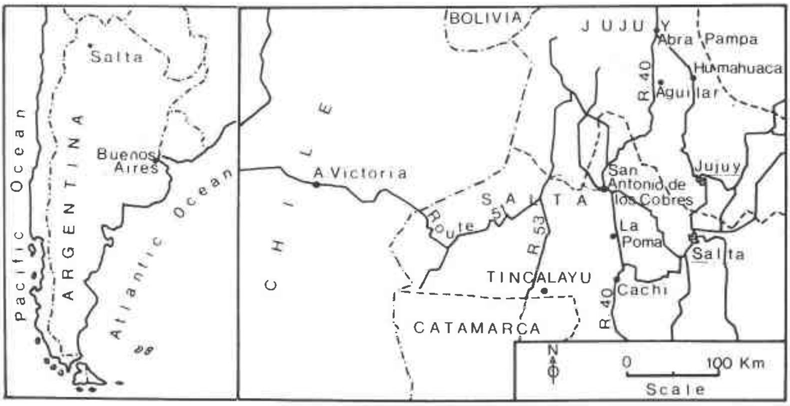

FIG. 1. Location of the Tincalayu Mine, Salta, Argentina.

composed essentially of massive borax. In addition to borax and kernite several rare and unusual borates are present, three of which have been found only at this locality. These are ezcurrite, $2Na_2O \cdot 5B_2O_3 \cdot 7H_2O$; rivadavite, $3Na_2O \cdot MgO \cdot 12B_2O_3 \cdot 22H_2O$, and ameghinite, $Na_2O \cdot 3B_2O_3$ $4H_2O$ described respectively by Muessig and Allen (1957), Hurlbut and Aristarain (1967) and Aristarain and Hurlbut (1967). All these rare borates occur in nodular masses embedded in the massive borax. Large

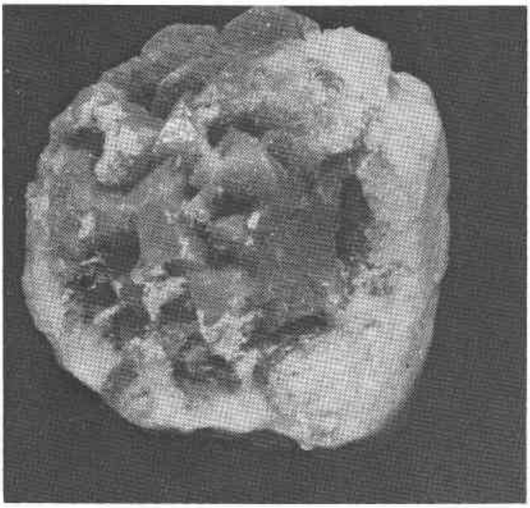

FIG. 2. Nodule composed of macallisterite crystals surrounded by rivadavite, from Argentina. ×1.2.

crystals of macallisterite occupy the core of a nodular mass of rivadavite (see Fig. 2), and include small amounts of this mineral.

The presence of macallisterite in this association is of particular interest, for, unlike the U.S.A. and Chinese occurrences, it here appears as a hypogene mineral. In Death Valley it was found as an efflorescence derived from colemanite and priceite veins in an altered basalt and is intimately mixed with ginorite and sassolite. The boron minerals ulexite, hydroboracite, gowerite, nobleite and meyerhofferite as well as gypsum, thenardite and limonite have also been described in the same association (Erd *et al.*, 1959; Schaller *et al.*, 1965). In the reported occurrence in China, macallisterite is associated with hydroboracite and hungchaoite.

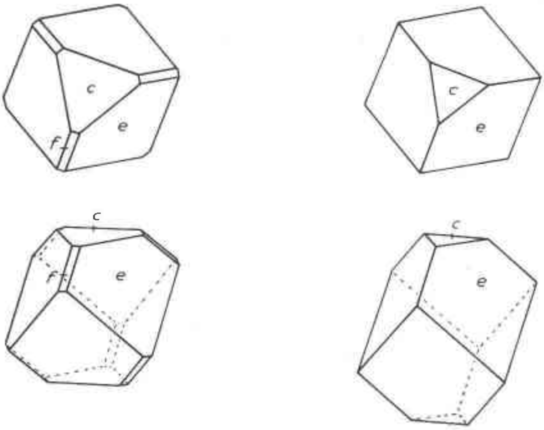

FIG. 3. Crystal drawings of macallisterite from Argentina.

MORPHOLOGY

The macallisterite from Death Valley was found in aggregates of minute crystals less than 0.2 mm in diameter. In contrast the Argentine mineral occurs in relatively large crystals, some as much as 12 millimeters in maximum dimension. They are randomly intergrown in a nodular mass of rivadavite 8 centimeters in diameter (see Fig. 2). In addition, the faces of the large crystals are encrusted with a second generation of well formed but small (0.5 mm) macallisterite crystals. The latter were used for optical goniometric work and single-crystal X-ray study.

The only crystal forms found were the same as those described by Schaller *et al.*, (1965) on synthetic crystals. They are: *c* $\{0001\}$, *e* $\{01\bar{1}2\}$, and *f* $\{10\bar{1}4\}$. The first two forms are always present with faces of

TABLE 1. MACALLISTERITE ANGLE TABLE

Hexagonal: Hexagonal-Scalenohedral $\bar{3}2/m$

$a:c=1:3.082$ $p_0:r_0=3.559:1$

$\alpha=50°18'$ $\lambda=112°59'$

	Form	ϕ	$\rho=C$	A_1	A_2
c	0001	—	0°00′	90°00′	90°00′
f	$10\bar{1}4$	30°00′	41°40′	54°51′	90°00′
e	$01\bar{1}2$	−30°00′	60°40′	90°00′	40°58′

$\{01\bar{1}2\}$ dominant. The form $\{10\bar{1}4\}$ has unequal development and in some crystals appears only as line faces; it was not observed on the large crystals. The common habits are illustrated in Figure 3. The crystallographic constants calculated from measurements made on ten crystals are given in Table 1.

PHYSICAL AND OPTICAL PROPERTIES

Macallisterite has good $\{0001\}$ and $\{01\bar{1}2\}$ cleavages. Ch'u *et al.*, (1965) report a cleavage but not its direction. The Argentine mineral is brittle with hardness of $2\frac{1}{2}$; the specific gravity, determined with the Berman balance using large transparent fragments (approx. 50 mg) is 1.867 ± 0.005. These values agree with those measured by Schaller *et al.*, (1965) on synthetic crystals.

The large macallisterite crystals are a light amber color but in thin section are colorless and transparent. The color of the large crystals results from a brown stain, probably due to iron oxide, that is present along some cleavage cracks. The small second generation crystals are nearly colorless.

Macallisterite is uniaxial negative. The refractive indices of the

TABLE 2. REFRACTIVE INDICES OF MACALLISTERITE

	Natural				Synthetic			
	1	2	3	4	5	6	7	8
ϵ	1.4635	1.465	1.464	1.4638	1.463	1.459	1.458	1.4638
ω	1.5070	1.505	1.507	1.5070	1.508	1.504	1.504	1.5070

1) This study, Na light, ±0.0003 from Salta, Argentina; 2) Schaller *in* Allen and Kramer (1957) from Death Valley, U.S.A.; 3) Schaller *in* Schaller *et al.*, (1965); 4) Ch'u *et al.*, (1965) from a continental lake in China; 5) Nikolaev and Chelishcheva (1940); 6) and 7) Fahey *in* Schaller *et al.*, (1965) and 8) Ch'u *et al.*, (1965).

mineral from Argentina are in close agreement with those reported in the literature. See Table 2 for a comparison of refractive indices of natural and synthetic material.

X-RAY STUDY

The extinctions in precession photographs lead to the space groups $R\bar{3}c$ or $R3c$, but the form development and lack of either a piezoelectric or pyroelectric response indicate the space group $R\bar{3}c$ as suggested by Schaller *et al.*, (1965). The unit-cell dimensions $a = 11.543$, $c = 35.56$ Å of the Argentine mineral agree well with theirs as shown in Table 3.

TABLE 3. CRYSTALLOGRAPHIC DATA FOR MACALLISTERITE AND SYNTHETIC EQUIVALENT

	This Study	Schaller *et al.* (1965)		Ch'u *et al.*[1] (1965)
	Macallisterite (Argentina)	Macallisterite (U.S.A.)	Synthetic	Synthetic
a Å	11.543} ±	11.546±0.001	11.549±0.002	11.6±0.1
c Å	35.556} 0.005	35.562±0.005	35.567±0.008	35.3±0.3
c/a	3.080	3.080	3.080	3.043
a_{rh} Å	13.597	13.600	13.602	13.5
α	50°14′	50°14′	50°14′	50°42′
Cell volume Å^3	4102.8	4105.7	4108.4	4141.89
Z (hexagonal)	6	6	6	12
Specific gravity calc.	1.867_3	1.866	1.864	1.85
meas.	1.867	—	1.868_2	

[1] c/a not given in their paper. Z for $MgO \cdot 3B_2O_3 \cdot 7.5H_2O$.

The spacings of the powder photograph are similar to those of both synthetic and natural material presented by Schaller *et al.*, (1965), Ch'u *et al.*, (1965), and to those calculated from θ values given by Lehmann and Papenfuss (1959). However, the spacings of Ch'u *et al.*, appear to be indexed incorrectly.

Although the spacings agree with those previously published for macallisterite, the intensities are somewhat different. The strongest lines for a routinely prepared sample of the Argentine mineral are (in Å): 4.062 (100), 3.263 (56), 8.74 (44), 6.66 (41), 3.355 (36). When mixed with an equal amount of glass to avoid preferred orientation due to cleavage, the strongest lines are (in Å): 3.263 (100), 8.74 (55), 3.355 (47), 4.062 (46), 1.702 (42). The intensities given are taken from diffractometer charts reproduced in Figure 4.

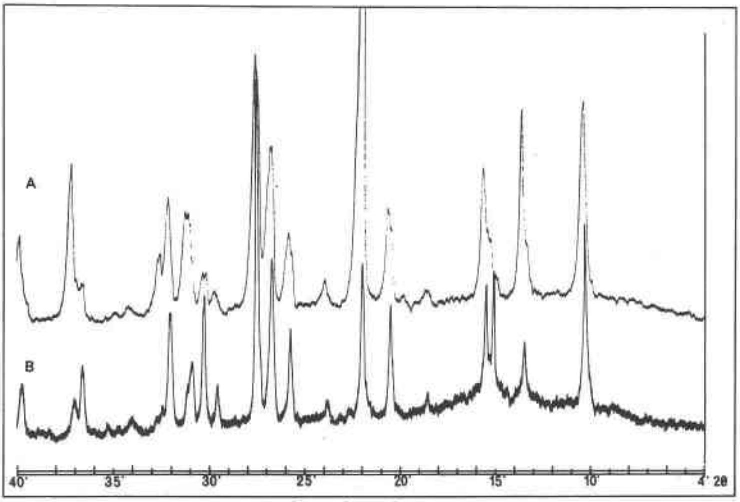

FIG. 4. Diffractometer charts of macallisterite. A. Macallisterite alone. B. Macallisterite with 50 percent glass. Cu radiation, Ni Filter.

CHEMISTRY

The two chemical analyses given by Schaller *et al.*, (1965) were made on samples containing 25 and 30 weight percent of associated minerals as impurities. The large crystals from Argentina presented an opportunity

TABLE 4. CHEMICAL ANALYSES OF MACALLISTERITE

	1	2	3	4	5
B_2O_3	53.6	53.43	53.17	53.12	54.355
FeO	0.25	—	—	—	—
MnO	0.04	—	—	—	—
MgO	10.4	10.06	10.97	10.43	10.490
CaO	—	—	—	0.83	—
$H_2O(+)$	34.9	36.51	35.86	35.53	35.155
$H_2O(-)$	0.76	—	—	—	—
Total	99.95	100.00	100.00	99.91	100.00

1. Present Study. Salta, Argentina. Jun Ito, Analyst. A spectrographic analysis made by Jarrell-Ash Co. indicates faint traces of Na, K, Si; very faint traces of Ca, Cr; and very, very faint traces of Sr, Ag, Al, Cu.

2. and 3. Schaller *et al.*, (1965). Death Valley, U. S. A. Recalculated to 100 percent after deducting impurities.

4. Ch'u *et al.*, (1965), China.

5. Composition of $2MgO \cdot 6B_2O_3 \cdot 15H_2O$.

TABLE 5. MOLECULAR PROPORTIONS OF MACALLISTERITE

Oxide	Analysis 1, Table 3, recalculated to 100%	Molecular proportions	Molecular proportions	$B_2O_3=6$
B_2O_3	54.038	0.7760	6	6.000
FeO	0.252	0.0035	0.027	2.043
MnO	0.040	0.0006	0.005	
MgO	10.485	0.2600	2.011	
H_2O	35.185	1.9530	15.101	15.101
Total	100.00			

for a chemical analysis to be made on the clean natural mineral. Such an analysis, made by Dr. Jun Ito, on material selected to avoid the iron stain is reported in Table 4 (Column 1) with the other available analyses

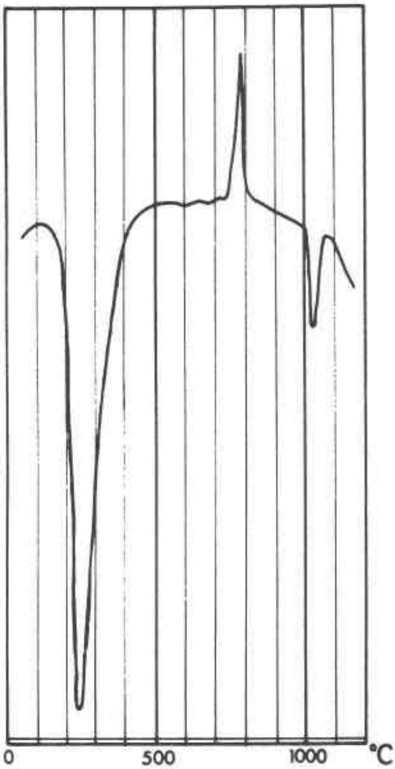

FIG. 5. DTA. curve of macallisterite. Rate of heating 25°C/min; reference junction 0°C; thermocouple Pt/Pt+13% Rh, reference material Al_2O_3.

of macallisterite. In Table 5 are given the molecular proportions derived from Ito's analysis recalculated to 100 percent disregarding $H_2O(-)$. With $B_2O_3=6$ the proportions yield the following empirical formula: $2.04(Mg, Fe, Mn)O \cdot 6B_2O_3 \cdot 15.10H_2O$. This agrees closely with, $2MgO \cdot 6B_2O_3 \cdot 15H_2O$, the proposed formula for the mineral.

A differential thermal analysis yielded a curve reproduced in Figure 5. The curve is characterized by three prominent peaks, two endothermic and one exothermic. The positions of these peaks are given in Table 6, with values obtained by other workers.

The first endothermic peak results from the complete loss of water. Schaller *et al.*, (1965) report that the water is lost between 130° and 450°C in fairly definite steps but these steps are not shown in our curve. The compound $MgO \cdot 3B_2O_3 \cdot 5H_2O$ (Lehmann and Papenfuss, 1959) was not observed during heating experiments. On heating to 500°C, the sample becomes amorphous with a refractive index of 1.505. After maintaining the sample at a temperature of 800°C for 10 minutes or more and on both rapid and slow cooling, X-ray diffraction shows a crystalline phase, probably $MgO \cdot 2B_2O_3$ as described by Grigor'ev and Nekrasov (1966). The major exothermic peak at 785°C is interpreted as resulting from its formation. The mean index of refraction of this phase is 1.547 and the spacings of the most intense X-ray reflections are (in Å): 3.92 (100); 5.08 (50); 4.40 (70); 3.40 (65); 2.81 (60).

TABLE 6. SUMMARY OF DTA PEAKS OF MACALLISTERITE

Sample	Endothermic peaks, °C		Exothermic peaks, °C	
	1	2	1	2
A. Synthetic	160–205		690	
B. Chinese	150–229	1105–1130		760–834
C. Argentine	160–450	980–1060	720	740–800
	Max. 230	Max. 1025		Max. 785

A. Chelishcheva (1940)
B. Ch'u *et al* (1965b)
C. This Study.

The endothermic peak with maximum at 1030°C results from the fusion of the sample. When this product is quenched in air a green glass is formed, with index of refraction, 1.545. On slow cooling the crystalline compound, $MgO \cdot B_2O_3$, forms together with a glass of B_2O_3, identified respectively by X-ray powder photographs and index of refraction. Lehmann and Papenfuss (1959) obtained the same products on heating $MgO \cdot 3B_2O_3 \cdot 5H_2O$ to 700°C.

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