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NEW DATA ON SARMIENTITE

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Sarmientite, $\text{Fe}_2(\text{AsO}_4)(\text{SO}_4)\text{OH} \cdot 5\text{H}_2\text{O}$, was discovered by V. Angelelli in the locality of La Alcaparrosa, Department Barreal, San Juan province, Argentina, and described by Angelelli and Gordon (1941) who performed the optical and morphological study.

While studying some arsenates and sulfates, a specimen of sarmientite from the original locality was investigated at our laboratory; the present note records the results.

On optical examination the sample appeared to be homogeneous and free of impurities formed by very small crystals up to 20 μm long.

Before the wet analysis was undertaken, a spectrochemical analysis revealed, in addition to the major elements: Ca, Cu (0.1%); Al, Mn (0.01–0.1%) and Si, Mg (0.001%).

The quantitative chemical analysis of the specimen now studied agrees with that given by Angelelli and Gordon (Table 1).

In the electron microscope, crystals showing a habit similar to that described by Angelelli and Gordon are seen (Fig. 1). After long exposure to the electron beam, the crystals alter visibly and crack.

X-RAY DIFFRACTION DATA

After a very long search, three crystals permitting single crystal work were found. Rotation, Weissenberg and precession methods were employed to determine the dimensions of the unit cell. These values, slightly modified after data derived from X-ray powder patterns, are

TABLE 1. CHEMICAL ANALYSES OF SARMIENTITE
Wt. %

	Analysis in Angelelli and Gordon	Present study
As ₂ O ₅	22.68	23.00
SO ₃	18.28	19.10
Fe ₂ O ₃	36.57	33.63
FeO	n.d.	1.43
CaO	0.27	n.d.
H ₂ O	22.86	22.60
Total	100.66	99.76

$a=6.55$, $b=18.55$, $c=9.70$ Å, $\beta=97^{\circ}39'$, monoclinic $P2_1/c$, $a:b:c=0.353:1:0.529$, cell volume 1168.08 Å³, $Z=4$. The Weissenberg photographs were taken with c the rotation axis; precession photographs were obtained with c and a the precessing axes. Exposures times were 200–300 hours (Cu/Ni).

The spacings and intensities given in Table 2 were derived from diffractometer charts. The calculated d values were obtained and indexed by means of a Bull G.E. 625 computer.

Extinctions in the single crystal photographs lead to space group

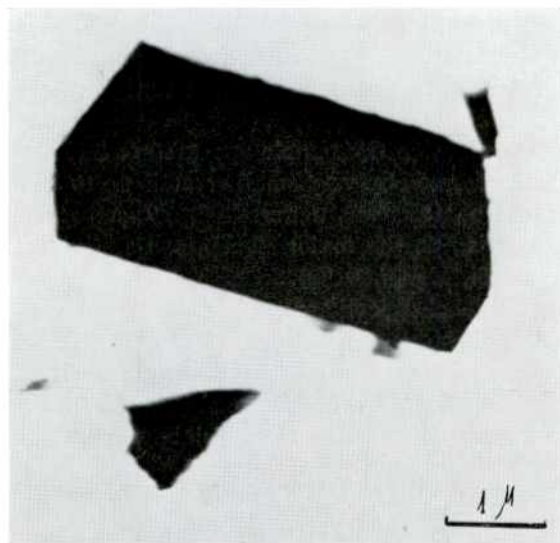


FIG. 1. Electron micrograph of a sarmientite crystal.

TABLE 2
X-RAY POWDER DATA FOR SARMIENTITE

<i>hkl</i>	<i>d</i> (obs) ^a Å	<i>d</i> (calc) ^b Å	<i>I</i>	<i>hkl</i>	<i>d</i> (obs) ^a Å	<i>d</i> (calc) ^b Å	<i>I</i>
020	9.29	9.275	100	052	2.93	2.937	7
011	8.53	8.535	22	211		2.921	
021	6.68	6.675	9	202		2.873	
110	6.13	6.127	35	230	2.87	2.874	16
120	5.31	5.318	15	142	2.84	2.863	10
031	5.21	5.201	27	212		2.839	
121	4.87	4.885	38	033		2.845	
040	4.64	4.637	90	231	2.792	2.847	4 ^b
012		4.653		160		2.791	
130	4.47	4.477	18	222		2.734	
121	4.26	4.453	80	133	2.726	2.727	18
022		4.268		161		2.723	
112		4.036		32		2.669	
140	3.76	3.774	9	231	2.668	2.669	18
122		3.777		240		2.659	
112	3.56	3.570	10	062		2.600	
141	3.43	3.424	40	152	2.60		
132		3.437		232	2.606		
042	3.33	3.337	35	202	2.526	2.538	6
200	3.24	3.246	9	212		2.514	
150	3.22	3.221	8	241	2.492	2.494	7
201		3.207		133		2.500	
132	3.13	3.136	20	242		2.442	
151		3.117		250	2.428		
220		3.06		3.064	70	213	2.426
				251			

^a Values obtained using $\text{CoK}\alpha = 1.7889 \text{ \AA}$, Fe filter. Diffractometer.

^b The calculated *d* values were obtained by means of a Bull G.E. 625 computer.

$P2_1/c$. The morphology (Angelelli and Gordon, 1941) suggests that the crystals belong to class $2/m$.

The calculated density is 2.58 for a unit cell containing four formula units of $\text{Fe}_2(\text{AsO}_4)(\text{SO}_4)\text{OH} \cdot 5\text{H}_2\text{O}$. The density measured by Angelelli and Gordon is 2.58.

INFRARED SPECTRUM

The infrared absorption spectrum shown in Figure 2 was obtained in a Perkin Elmer model 337 grating spectrophotometer. A sample of 1.5 mg was mixed and ground with 210 mg of KBr.

Hydroxyl and water bands appear in the regions near 3.0 and 6.1 μm respectively.

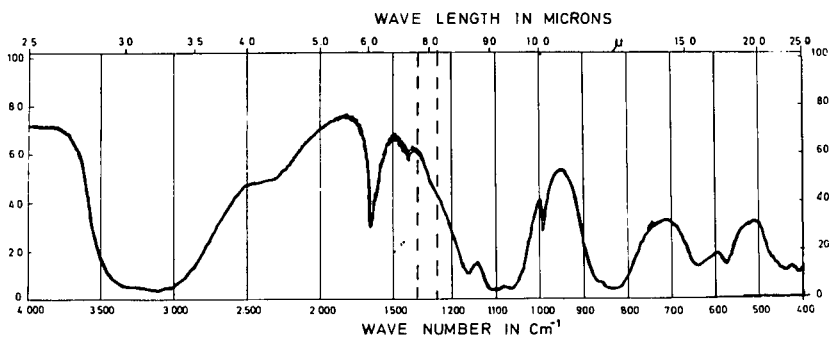


FIG. 2. Infrared absorption spectrum of sarmientite.

The following bands correspond to the vibrations of the sulfate ion, according to Adler and Kerr (1965); three intense bands in the 8- to 10- μ m region which correspond to the τ_3 vibrations, a weak band slightly above 10 μ m which represents the ν_1 vibration; two bands in the 15- to 18- μ m region are attributed to the ν_4 mode, and a band at 22 μ m, to the ν_2 mode. The bands in the 11- to 13- μ region are attributed to vibrations of the arsenate ions.

THERMAL PROPERTIES

The weight loss of sarmientite on heating was determined by means of a thermogravimetric balance. A 204 mg sample, was heated from room temperature to 980°C, at 5°C/min. The resulting curve is shown in Figure 3. The loss in weight between 200 and 685°C is 22.95 percent;

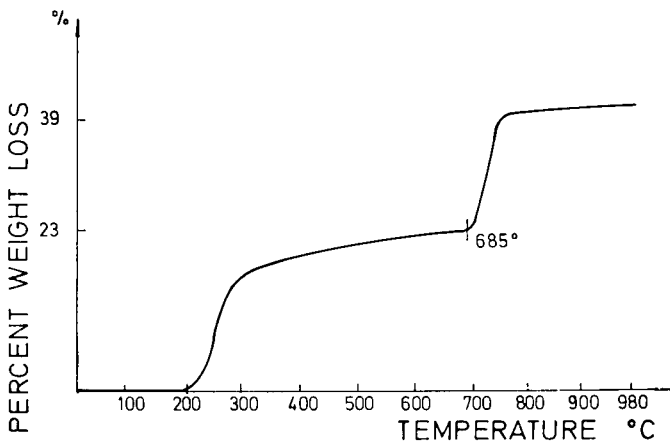


FIG. 3. Thermogravimetric curve of sarmientite.

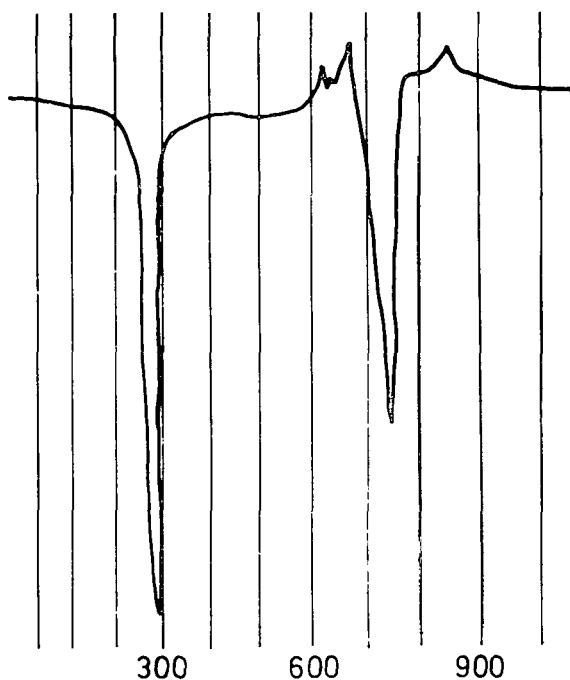


FIG. 4. Differential thermal analysis curve of sarmientite, temperature in °C.

most of this loss occurs between 200 and 300°C, and represents the water content; it agrees closely with the water determination in the chemical analyses (Table 1). A loss of weight of 16.4 percent occurred between 685 and 980°C; it agrees reasonably well with the SO_3 determination in the chemical analyses.

A differential thermal analysis of sarmientite yielded the curve of Figure 4 in which are shown major endothermic peaks at 200–300°C (loss of water) and at 700–800°C (loss of SO_3) and small exothermic peaks at 600–700°C and 850°C which probably represent recrystallization processes.

When heated at 300°C for one hour sarmientite yields a buff colored product, amorphous to X-rays. A sample held at 650°C for two hours lost all the SO_3 ; a reddish-brown, crystalline material, remained. The spacings in Å and the relative intensities of the strongest lines, as obtained from a diffractometer chart are: 3.04 (100), 3.13 (50), 3.26 (50), 2.99 (30), 2.49 (30), 3.68 (30), 3.53 (30). The line at 3.68 Å belongs to hematite; the spacings of the remaining lines correspond to those of strong lines in the angelellite pattern (Weber, 1959).

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