A NEW OCCURRENCE OF SAMUELSONITE IN THE BURANGA PEGMATITE, RWANDA

ANDRÉ-MATHIEU FRANSOLET, OLEG VON KNORRING and FRANÇOIS FONTAN

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A new occurrence of the rare mineral species, samuelsonite, is reported from the Buranga pegmatite, Rwanda, in close association with gatumbaite and burangaite, as aggregates of bladed crystals imbedded in trolleite and in bertossaite.

Its crystallographic properties, determined both by the rotation and Weissenberg techniques and by the powder method, are: $a = 18.621(3)$, $b = 6.842(1)$, $c = 14.066(2)$Å, $\beta = 112°30'(1')$, $C2/m$, $Z = 2$.$G_{meas.} = 3.24(5)$ and $G_{calc.} = 3.22$. A wet chemical analysis gives: 43.79 P_2O_5, 6.18 Al_2O_3, 0.07 Fe_2O_3, 7.92 FeO, 8.91 MnO, 0.07 MgO, 28.41 CaO, 2.31 BaO, 0.43 Li_2O, 0.35 Na_2O, 1.85 H_2O^+, total 100,29%. Because of some structural similarities with the apatite structure, a few assigments of the infrared absorption bands are tentatively proposed.

A discussion on the crystal chemistry shows that Li resides in the octahedral M sites, whereas Na is sequestred with the excess of Mn and Ca in the X sites of the structure.

Key words: minerals, phosphates, samuelsonite, chemical composition, infrared spectra, crystal structure, pegmatite, Buranga, Rwanda.

André-Mathieu Fransolet, Institut de Minéralogie, Bât. B18, Université de Liège, Sart-Tilman, B - 4000 Liège, Belgium

Oleg von Knorring, Department of Earth Sciences, The University, Leeds LS2 9JT, England

François Fontan, Laboratoire de Minéralogie — U.R.A.67 Université Paul-Sabatier Allées Jules-Guesde, 39 F - 31400 Toulouse, France

Introduction

A re-examination of the phosphate minerals from the Buranga pegmatite, Rwanda (von Knorring 1972) showed that one of the major constituents of the Al-rich phosphate masses is trolleite, the common associated minerals being montebrasite, scorzalite, bertossaite, brazilianite, and apatite *s.l.*. In that paper, von Knorring also recorded a new barium mineral, which has been identified as bjarebyite (von Knorring and Fransolet 1975), and gave the *d*-spacings for two undetermined fibrous phosphates. One of them, occurring as white rosettes, has been described as gatumbaite (von Knorring and Fransolet 1977). The purpose of the present paper is to discuss the results of the mineralogical investigation of the second one, tentatively thought to be another Ba-bearing greenish phosphate (von Knorring and Fransolet 1977) and finally identified as samuelsonite.

Physical and optical properties

In the trolleite masses from Buranga, samuelsonite occurs as fan-like aggregates of bladed crystals. They have irregular contours and can

Table 1: Optical properties.

	Buranga	Palermo No. 1 (Moore et al. 1975)
Sign	+	+
α†	1.648(2)	1.645(2)
β	1.655(2)	1.650(2)
γ	1.667(2)	1.655(2)
$2V_{obs.}$	80—85°	large (70—80°)
$2V_{calc.}$	75°	—
XΛ trace of {001}	31(2)°	—
Extinction	—	22°

† The indices were measured for $\lambda_{Na} = 589nm$.

reach up to 2 cm, particularly in the specimen R.G.M. 9979 provided by the Musée royal de l'Afrique centrale in Tervuren, Belgium. Although the aggregates appear yellow green to pale green in colour, fragments of the pure mineral are colourless and transparent. Some altered grains are partly turbid. It must be noted that the greenish colour is caused by a very thin coating of undetermined olive green impurities in the cleavages and fractures of the mineral. The luster is vitreous on the perfect {001} cleavage, and vitreous to resinous on the irregular fractures. The specific gravity = 3.24(5) was obtained on six selected grains using a Berman balance.

Isolated crystals are tabular on {001} and elongated along [010]. As initially observed on the type mineral from the Palermo No. 1 pegmatite (Moore *et al.* 1975), all the crystals are finely striated parallel to *b* on {001}. No forms were observed, and no twinning was found.

The optical properties, determined both on isolated grains and in thin sections, are compared in Table 1 with the data of Moore *et al.* (1975). The value for 2V = (+)80—85° has been estimated on the frequent sections perpendicular to the optical axis, as previously noted by Moore *et al.* (1975). In thin section, laths with the trace of the {001} cleavage show a negative elongation and, in the optic axial plane, the angle between X and the {001} cleavage is 31(2°). The dispersion $r < v$, is weak but distinct. The optical orientation is: *Y//b* and *O.A.P.//*(010).

Description of the association

Under the polarizing microscope, the material availabe for this study shows samuelsonite occurring as bundles of bladed crystals, colourless in thin section. Samuelsonite clearly corrodes trolleite, bertossaite, and scorzalite. It is accompanied by late apatite with an interstitial texture. Rosettes of gatumbaite, sometimes intimately intergrown with thin laths, or even with small sheafs, of burangaite intensely pleochroic in blue hues (von Knorring *et al.* 1977) appear at the contact of samuelsonite with trolleite. As gatumbaite also occurs within the aggregates of samuelsonite, cross-cutting its bladed crystals it seems justified to propose the following transformation, samuelsonite — apatite → gatumbaite — burangaite, as a step of the late evolution stage observed so far for the Al-rich phosphate associations in the Buranga mine. No bjarebyite has been recognized in this material.

Crystallographic properties

The X-ray diffractometer data obtained on the amount of handpicked samuelsonite selected for the wet chemical analysis are listed in Table 2. Although they are similar to those given by Moore *et al.* (1975), our data, however, show *d*-values which are systematically a little larger. Moreover, a few *d*-values recorded by these authors are missing in our data, while we measured some additional *d*-spacings, for instance, 8.59 Å (200) and 3.419 Å (020). These differences do not seem to be attributable to the diffractometer technique used, which certainly gave reliable resolution of the *d*-values between 3.100 and 3.030 Å.

The cell parameters preliminarily measured on single crystal patterns using the rotation and

Table 2: X-ray powder data of samuelsonite from Buranga.

I/Io	$d_{obs.}$(Å)	*hkl*	d_{calc}(Å)
10	13.01	001	12.995
30	8.59	200	8.602
10	6.172	201	6.168
5	6.059	$\bar{1}11$	6.059
15	4.648	$\bar{4}01$	4.648
15	4.455	$\bar{4}02$	4.456
20	4.396	310	4.395
10	4.299	400	4.301
5	3.882	$\bar{4}03$	3.884
10	3.840	311	3.836
		$\bar{1}13$	3.841
10	3.685	401	3.684
55	3.419	020	3.421
25	3.382	203	3.384
15	3.306	021	3.308
35	3.259	$\bar{4}04$	3.261
20	3.176	$\bar{2}20$	3.179
40	3.100	$\bar{6}02$	3.097
100	3.078	$\bar{3}14$	3.080
40	3.053	$\bar{6}01$	3.057
20	3.030	$\bar{2}22$	3.029
5	2.971	$\bar{6}03$	2.971
20	2.870	$\bar{6}00$	2.867
50	2.803	$\bar{2}05$	2.802
40	2.754	$\bar{4}21$	2.755
		$\bar{2}23$	2.755
40	2.709	222	2.708
65	2.677	420	2.677
50	2.595	403	2.596
		$\bar{3}15$	2.595
5	2.568	$\bar{4}23$	2.567
5	2.507	421	2.507
5	2.439	$\bar{5}15$	2.438
		$\bar{7}13$	2.437
40b	2.316	602	2.316
		$\bar{7}14$	2.320
		710	2.313
5	2.217	404	2.216
		$\bar{3}16$	2.218
10b	2.141	$\bar{3}31$	2.140
5	2.105	$\bar{3}32$	2.104
10	2.070	025	2.069
		621	2.069
10b	2.001	801	2.002
15	1.991		
10	1.977		
10	1.937		
10	1.921		
10	1.902		
10	1.895		
10	1.886		
10	1.854		
10	1.842		
15	1.820		
20	1.711		

b: broad. Diffractometer; monochromatized Fe radiation; *d* values corrected with an internal standard $Pb(NO_3)_2$ ($a = 7.8568$Å).

Table 3. Unit-cell data.

	Buranga	Palermo No. 1 (Moore and Araki, 1977)
a(Å)	18.621(3)	18.495(10)
b(Å)	6.842(1)	6.805(4)
c(Å)	14.066(2)	14.000(8)
β	112°30′(1′)	112.75(6)°
V($Å^3$)	1655.6(3)	1624.9
a:*b*:*c*(1)	2.722:1:2.056	2.718:1:2.057
Space group	*C2/m*	*C2/m*
Z	2	2
$D_{meas.}$	3.24(5)	3.353
$D_{calc.}$	3.22	3.355

(1) calculated from the unit-cell parameters.

Weissenberg methods were refined from the powder data. These calculated cell dimensions, compared with the data of Moore and Araki (1977) in Table 3, show that the unit-cell of the Buranga samuelsonite is decidedly larger than that of the type samuelsonite from the Palermo No. 1 pegmatite. The interpretation of the Weissenberg patterns results in the determination of the space group *C2/m, C2* or *Cm* and we adopt *C2/m* in Table 3, in accordance with Moore and Araki (1977) who solved the crystal structure.

Infrared absorption spectrum

An infrared spectrum has been recorded between 200 cm^{-1} and 4000 cm^{-1} with a Beckman IR 4250 spectrophotometer following the conventional pressed-pellet technique. A 2.5 mg sample of samuelsonite was carefully ground and mixed with 0.6 g KBr, and the mixture pressed as to give a disc of 20 mm diameter for the domain between 200 cm^{-1} and 1400 cm^{-1} (Fig. 1a). For the range 1500—4000 cm^{-1}, 6 mg were mixed with the same amount of KBr (Fig. 1b). The observed absorption band frequencies are directly indicated on the spectra.

In the complex part of the spectrum shown in Figure 1a, there is little doubt about the assignment of the strong absorption bands between 950 cm^{-1} and 1100 cm^{-1} to the antisymmetric vibrations of the PO_4 anion. It seems also reasonable to attribute the band at 975 cm^{-1} to the υ_1 vibration, and those at 1015 cm^{-1}, 1050 cm^{-1} and 1097 cm^{-1} to the υ_3 vibrations of PO_4, respectively. The shape of these bands in a relatively narrow domain suggests that the PO_4 tetrahedra are moderately distorted, what is in agreement with the P-O distances measured by Moore and Araki (1977). Additionally, if we take the similarities between particular sections of the samuelsonite atomic arrangement and the apatite structure into account, as reported by these authors, it appears that the four frequencies measured on the samuelsonite spectrum are very close to the ones already known for the υ_1—υ_3 vibrations of the PO_4 anion in the minerals with an apatite structure (Table 17 *in* Ross, 1974). By extending further our comparison, we are inclined to consider the strong absorption bands occurring at 567 cm^{-1}, 598 cm^{-1} and 618 cm^{-1} as the υ_2 PO_4 bending, again with both their frequencies and relative intensities quite similar to that of apatite. Although both the PO_4 bending and AlO_6 stretching vibrations contribute to the absorption in this 650—500 cm^{-1} region (Povarennykh, 1978), we speculate that the doublet at 662—683 cm^{-1}

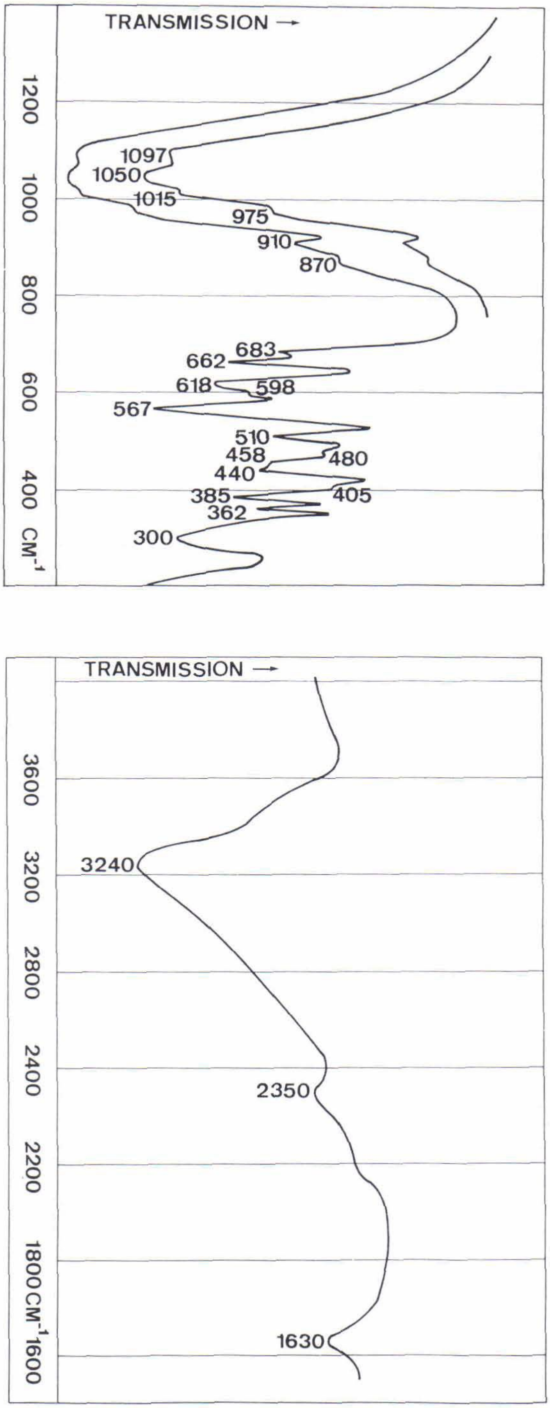

Fig. 1. Infrared spectra of the Buranga samuelsonite.
1a) 200—1300 cm^{-1} domain (2.5 mg of material diluted in 0.6 gr KBr).
1b) 1500—4000 cm^{-1} domain (6 mg diluted in 0.6 gr KBr).

could be due to the stretching vibrations of the octahedral Al.

From the 1500—4000 cm^{-1} region (Fig. 2b), it is virtually impossible to identify of O-H stretching vibration of the hydroxyl groups because they are certainly screened by the stretching vibrations of H_2O. This strong and large absorption band seems to indicate by its frequency at 3240 cm^{-1}, that H_2O is rather strongly bonded in the structure. However, the weak shoulder observed towards the higher frequencies, around 3400 cm^{-1}, is probably caused by a second type of H_2O, less strongly bonded. The two other bands at 1630 cm^{-1} and 2350 cm^{-1} can be attributed to the bending vibration of the water molecules, and to an harmonic or a combination vibration, respectively.

Table 4: Chemical analyses of samuelsonite

1. Wet-chemical analysis performed by O.v.K.; (*) the sum includes 0.28 wt.% insoluble.
2. Wet-chemical analysis performed by J.-M. Speetjens.
3. »Find 1»: electron microprobe analysis by A.J. Irving (Moore *et al.* 1975).
4. »Find 2»; wet chemical analysis by J. Ito (Moore *et al.* 1975).
5. Theoretical composition for the charge balanced formula $(Ba_{0.5}\square_{0.5})(Ca_{0.5}\square_{0.5})_2(Mn_{0.45}Fe_{0.30}Na_{0.25})_4Ca_8Al_2(PO_4)_{10}(OH)_2$ (Moore and Araki, 1977).

tr. :traces; n.d.: not determined.
R = Mn/(Mn + Fe^{2+}).

	Buranga		Palermo No. 1		
	1	2	3	4	5
P_2O_5	42.76	43.79	44.0	43.10	42.86
Al_2O_3	6.37	6.18	6.2	4.80	6.16
Fe_2O_3	1.48	0.07	—	nil	—
FeO	7.26	7.92	6.5	11.30	5.21
MnO	8.81	8.91	7.9	7.20	7.71
MgO	—	0.07	—	0.12	—
CaO	26.96	28.41	29.6	28.30	30.48
SrO	—	—	0.3	0.44	—
BaO	2.65	2.31	4.8	2.04	4.63
Li_2O	0.42	0.43	—	—	—
Na_2O	0.44	0.35	1.4	0.21	1.87
K_2O	0.07	tr	—	0.02	—
H_2O^+	1.88	1.85	n.d.	3.00	1.09
H_2O	0.03	—	—	—	—
Total	99.41*	100.29	100.7	100.53	100.01
Cation numbers calculated on the basis of 20.00 PO_4 per unit cell.					
Al^{3+}	4.15	3.93	3.92	3.10	4.00
Fe^{3+}	0.62	0.03	—	—	—
Fe^{2+}	3.35	3.57	2.92	5.18	2.40
Mn^{2+}	4.12	4.07	3.59	3.34	3.60
Mg^{2+}	—	0.06	—	0.10	—
Ca^{2+}	15.96	16.42	17.03	16.62	18.00
Sr^{2+}	—	—	0.09	0.14	—
Ba^{2+}	0.57	0.49	1.01	0.44	1.00
Li^+	0.93	0.93	—	—	—
Na^+	0.47	0.36	1.46	0.22	2.00
K^+	0.05	—	—	0.01	—
H^+	6.93	6.66	?	10.97	4.00
R	0.55	0.53	0.55	0.39	0.60

Chemical composition

In 1973 one of us (O.v.K.) performed a wet chemical analysis on this yellow green mineral but the results were never published. They are shown in Table 4, column 1.

Although the physical and optical features, as well as the crystallographic properties of the mineral described in this paper (Tables 1, 2 and 3), corroborate the identification of samuelsonite, some discrepancies arise between the chemical results of O.v.K. and those given by Moore *et al.* (1975), i.e. the electron microprobe data, and a wet chemical analysis of Jun Ito (see Table 4). On the contrary of the samuelsonite from the Palermo pegmatite, the mineral from Buranga seems to contain significant amounts of Fe_2O_3, and of Li_2O; its H_2O content in wt% is also different from the result obtained by J. Ito.

As the material available at the outset of our reinvestigation only displayed small amounts of samuelsonite dispersed within trolleite, and intimately associated with scorzalite, and burangaite, a purification of the mineral of handpicking was unsuccessful. Moreover, as ferric iron and lithium could not be checked with the electron microprobe, a wet-chemical analysis still appears as the best way to solve the problem.

In the meantime, specimens from the mineral collection of the Museum at Tervuren provided us with new fresh material. A qualitative spectrographical analysis confirm P, Ca, Al, Fe, and Mn as major elements, Ba, Li, Na and Mg as minor elements, Pb, Ga, Ti, Cu and Ag as trace elements. A wet-chemical analysis was performed on 25 mg of selected transparent grains, and the Fe content was determined on another separated amount of 25 mg following the method by Wilson (1960). The Penfield method served to determine the water content on 50 mg.

The new results are also given in Table 4, column 2 and are very similar to those obtained some years before. Both the Li_2O and H_2O contents are virtually identical in the two analyses. The new data also show Fe_2O_3 is not constant; the Fe_2O_3 content obtained by O.v.K. was likely caused by undetermined impurities or alteration products coating the cleavages and fractures of the mineral (see before).

Nevertheless, in order to compare our data with those of Moore *et al.* (1975), a small transparent fragment of the fresh material was polished for electron microprobe analyses, using the full automated equipment Camebax SX50 (operating conditions: accelerating voltage 15 kV, sample current 20 nA). The following standards were used: graftonite from Kabira for P, Fe, Mn and Ca; albite for Al, and Na; barite for Ba. The results of these partial analyses are given in Table 5.

Table 5: Electron microprobe analyses of the Buranga samuelsonite.
1 to 5: The five point-analysis on one crystal performed by F. Autefage with the Camebax SX50.
6: Mean values, $\bar{x}$ in wt%, and the standard deviation of $\bar{x}$, s, given in parentheses.
7: Relative standard derivation of $\bar{x}$, s/$\bar{x}$, multiplied by 100.

	1	2	3	4	5	6	7
P_2O_5	42.19	41.86	40.87	40.87	41.12	41.38(0.61)	1.47
Al_2O_3	5.97	6.02	5.93	5.93	5.81	5.93(0.08)	1.35
FeO	7.57	7.36	7.63	7.84	7.79	7.64(0.19)	2.49
MnO	8.75	8.72	8.82	8.19	7.59	8.41(0.52)	6.18
CaO	25.67	25.60	24.98	25.09	24.72	25.21(0.41)	1.62
BaO	0.89	1.16	2.03	3.75	4.85	2.54(1.71)	67.32
Na_2O	0.48	0.49	0.28	0.20	0.10	0.31(0.17)	55.84

Cation numbers on the basis of 20.00 PO_4 per unit cell (for column 6): Al = 3.99; Fe^{2+} = 3.65; Mn = 4.07; Ca = 15.42; Ba = 0.57; Na = 0.34; Mn/(Mn + Fe) = 0.53.

Discussion of the results

The new wet-chemical analysis (Table 4) allows to calculate the cation numbers on the basis of 20 $[PO_4]^{3-}$ in the unit-cell, and the distribution of the cations, as proposed by Moore *et al.* (1975), results in the following structural formula for the mineral from Buranga:

PO_4 $:20.00$
Ca $:16.00$
Al $:3.93\ Al + 0.03\ Fe^{3+} \sim 4.00$
M(1) + M(2) $:3.57\ Fe^{2+} + 0.93\ Li + 0.06\ Mg + 3.44\ Mn^{2+} = 8.00$
X(2) $:0.63\ Mn^{2+} + 0.42\ Ca + 0.36\ Na + 2.59\ \square = 4.00$
X(1) $:0.49\ Ba + 1.51\ \square = 2.00$
OH $:2.30\ OH^- + 1.70\ H_2O = 4.00$
$+0{,}48H_2O$

With Z = 2, this formula gives a calculated specific gravity of 3.26 g/cm³ in good agreement with the measured specific gravity. By using the revised constant published by Mandarino (1981) for the Gladstone-Dale relationship, we calculated $n = 1.635$ and a compatibility index $(1\text{-}K_p/K_c) = -0.034$, which ranges the category »excellent» (Mandarino, 1981).

The relevant fragments of the samuelsonite structure, as defined by Moore and Araki (1977), are: the double column $[Ca_4(PO_4)_{10}]$, which is the common element with the apatite structure permitting some reasonable assignments of the complex infrared spectrum, and the cluster $[Ca_4Al_2(OH)_2]$ constituted by Ca(1), Ca(2), and the $[Al(OH)O_4]$ octahedral chain. Consequently, the available amounts of Al and Ca atoms on the basis of $20(PO_4)$ in the cell must reach at least 4 and 16, respectively, to build up a valid formula. A comparison with the results obtained by Moore *et al.* (1975) effectively shows that the Al, Ca and P contents are varying in a short range only.

On the other hand, the cationic contents in the other sites of the structure display very distinct variations.

While the Mn/(Mn + Fe) ratio in the Buranga samuelsonite averages 0.53, and is virtually identical to the ratio obtained for the mineral of the Palermo mine (»Find 1»), it becomes 0.39 for the mineral called »Find 2» in the same mine (Moore *et al.* 1975). Marked variations of this ratio are known to occur in other phosphate minerals occurring as late products in granitic pegmatites.

The variations of the Li, Ba and H_2O contents are more puzzling.

In the proposed formula for the Buranga mineral, the small amount of Li (0.93 atom per cell) is reasonally located in the octahedral M(1) + M(2) sites. Subsequently, the excess of Mn^{2+} and the amount of Na reside in the X(2) site, normally populated by Ca. Nevertheless, it can be seen that the large amount of vacant X(2) positions (2.59), as well as the 1.51 empty X(1) positions, are in agreement with the description of Moore and Araki (1977, who reported that X(1) and X(2) must be at most half-filled for steric grounds.

A tiny crystal of samuelsonite from Palermo mine (U.S.N.M. 128.071) and labelled »Find 1», being thus considered as type material by Moore *et al.* (1975), was sacrificed for a qualitative check of Li by atomic absorption spectroscopy. Although the rather small available amount of the mineral prevents from obtaining accurate results, the presence of Li was detected, and its content was roughly estimated to be around 0.5 wt.%. This observation forces us to conclude that the presence of a significant content of Li was not previously reported for the type mineral from

Palermo.

The Ba variations were already mentioned in the two occurrences in the Palermo pegmatite (Table 4). In the case of Buranga, marked variations in the Ba contents were also detected thanks to the electron microprobe analyses, however, no zoning was seen. Although the microprobe results were obtained from five point-analyses only (Table 5), the average contents of the analysed elements are in fair agreement with the wet-chemical data. However, the CaO contents are rather low and one of the MnO values appears to be an analytical error. If the relative standard deviation of $\bar{x}$, $s/\bar{x}$, is taken into account (Table 5), the values indicated for $\bar{x}_{P2O5}$, $\bar{x}_{A12O3}$, $\bar{x}_{FeO}$, and even for $\bar{x}_{CaO}$, may be considered as the normal analytical uncertainties of these element determinations by electron microprobe. The values of $s/\bar{x}$ noticed for $\bar{x}_{BaO}$ and $\bar{x}_{Na2O}$, higher than the above analytical uncertainty (Table 5), reflect the non-uniform distribution of both these components within the analysed fragment of samuelsonite. As it can be seen from Table 5, the variations of the Ba contents are very sensitive. The range 0.89—4.85 wt.% BaO gives an upper value, corresponding to 1.09 atom Ba in the unit-cell, which is virtually identical to the theoretical value considered by Moore and Araki (1977) for the ideal formula (Table 4). Additionally, from the few chemical data obtained, the increase of Ba appears to be accompanied by a decrease of Na (Table 5).

Although our interpretation of the chemical results is constructed on cation numbers calculated on the basis of 20(PO_4) per unit cell and that Moore *et al.* (1975) computed the cell contents based on 84 oxygen, we cannot agree with the limited and, however, plausible replacement of [PO_4] tetrahedra by [H_4O_4] as suggested by these authors to explain a relatively high water content for samuelsonite »Find 2» in Palermo (Table IV). However Moore and Araki (1977) did not envisage the role of H_2O molecules in the crystal structure determination. They only specify the position of the OH groups in the shared corners of the Al octahedra. In order to balance the charges in our chemical formula, we are forced to consider 1.70 H_2O occurring in the sites theoretically populated by the 4 OH, and acting thus far as ligands of the Al-centered octahedra. Nevertheless, an excess of about 0.5 H_2O per unit cell subsists. Although it could be reasonably suggested that these additional water molecules could stabilize the structure of samuelsonite, in which the large X(1) and X(2) sites are partly vacant, the infrared analysis, however, did not provide us with valid arguments to locate H_2O — or H_3O^+ — in these particular sites.

As a conclusion, the comparison of our data, with those given for the two occurrences of samuelsonite in the Palermo mine, does not provide us with anything relevant. The discrepancy between the physical properties, which clearly appears from the Tables 1 and 3, cannot be reasonably justified so far. The chemical composition of this mineral is complex, and very flexible, as we have reported it here.

According to our chemical data, it seems that the role of Na^+ in the octahedral sites has been over-estimated by Moore and Araki (1977). We are of the opinion that the too low electron density distribution for M(1) and M(2), observed by these authors, is caused by Li^+. Consequently, Na^+ must reside in the X(2) site with Ca. Moreover, the calculation of the site occupancy for the Buranga samuelsonite leads us to consider a small amount of Mn^{2+}, which must also populate the X(2) sites. It must be pointed out that similarly an excess of Mn^{2+} is also known to coexist with Na^+ and a low amount of Ca^{2+} in the X(1) sites of the alluaudites occurring in the lithium-rich pegmatite of Buranga (Moore, 1971; Fransolet, 1975).

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