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# Molybdophyllite: crystal chemistry, crystal structure, OD character and modular relationships with britvinite

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**ABSTRACT**

A detailed crystal-chemical study of the complex layered silicate molybdophyllite was conducted using single-crystal X-ray diffraction (XRD) methods, supplemented by powder XRD, infrared (IR) and Raman spectroscopic studies, chemical analyses by energy-dispersive spectrometry (EDS) on a scanning electron microscope (SEM), and electron probe microanalysis (EPMA). The results, based on several samples from both Långban and Harstigen, Filipstad, Sweden, show that the crystal structure of molybdophyllite has an order-disorder (OD) character. The latter is especially evident in specimens from Långban which display a complex diffraction pattern characterized by the simultaneous presence of sharp spots, diffuse reflections and continuous streaks. The sharp reflections define the unit cell of the family structure ($a$ = 3.124, $c$ = 41.832 Å, space group $R32$). Two main polytypes (maximum degree of order structures) are indicated by the OD approach: a trigonal one and a monoclinic one; the latter polytype is the most common in the samples that were studied and has space group $C2$, with $a$ = 16.232(6), $b$ = 9.373(2), $c$ = 14.060(3) Å, β = 97.36(4)° and $V$ = 2121.5(10) Å$^3$.

The crystal structure determination [$R_1$ = 0.096], together with the EPMA, IR and Raman data, reveal that molybdophyllite is built up by a regular alternation of complex layers with a composition $\{Mg_9[Si_{10}O_{28}(OH)_8][OPb_4]_2\}^{6+}$ and simple layers with a composition $[(CO_3)_3{\cdot}H_2O]^{6-}$, leading to the ideal crystal-chemical formula $Pb_8Mg_9[Si_{10}O_{28}(OH)_8|O_2|(CO_3)_3]{\cdot}H_2O$ ($Z$ = 2).

This contribution is mainly devoted to the results obtained for molybdophyllite *sensu stricto*, but new data for britvinite [i.e. 'molybdophyllite-18 Å'] are also presented and its modular relationship with molybdophyllite is discussed.

**KEYWORDS:** Molybdophyllite, crystal structure, OD character, electron-microprobe data, britvinite, spectroscopy, modular aspects

## Introduction

MOLYBDOPHYLLITE is a very rare lead magnesium carbonate silicate mineral which was first described by Flink (1901) from the famous metamorphic, carbonate-hosted Fe-Mn(-As-Pb-Sb-Be-B) deposit at Långban, Filipstad District, Värmland, Sweden (Holtstam and Langhof, 1999). The name is derived from the Greek μολυβδος (*molybdos*), meaning lead, and φυλλον (*phyllon*), a leaf. Molybdophyllite has also been found on specimens from the Harstigen deposit, in the same district (Charalampides and Lindqvist, 1988). The species forms colourless to pale yellow, very pale green or pale grey micaceous aggregates, which are flexible and only a little

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more brittle than mica flakes. The aggregates have a perfect cleavage and are always embedded in matrix; free-standing crystals are unknown.

Flink (1901) reported a Pb:Mg:Si ratio close to 1:1:1, and a hexagonal symmetry, on the basis of etching experiments and optical studies. In a later single-crystal study, using Laue photographs, Aminoff (1918) suggested that the mineral was rhombohedral. The only modern published study of molybdophyllite is due to Charalampides and Lindqvist (1988), with a later summary by Charalampides (1994). Electron-probe microanalysis (EPMA), powder X-ray diffraction (PXRD), single-crystal X-ray diffraction (SXRD) and thermogravimetric studies were used to characterize the mineral. In common with previous workers, Charalampides (1994) observed that molybdophyllite formed poor-quality crystals, which are not suitable for single-crystal X-ray diffraction studies. He studied crystals from several samples from Långban in the collection of the Swedish Museum of Natural History, Stockholm and reported approximately hexagonal symmetry with refined unit-cell parameters $a$ = 9.32(2), $c$ = 27.39(7) Å (the space group was not determined) on the basis of an SXRD study of a flake from sample 980568 and refinement of the unit-cell parameters using Guinier-Hägg PXRD data. He also noted that "Slightly higher values, $a$ = 9.47 and $c$ = 27.9 Å respectively, are reported by P.B. Moore (University of Chicago, personal communication, 1987) who also claims that the space group is $P6_322$ (no. 182)." Charalampides (1994) tentatively suggested the ideal formula to be $Pb_9Mg_9Si_9O_{24}(OH)_{24}$, based on the assumption that no other anions such as $CO_3^{2-}$ are present, but noted that the true chemical composition was unknown. Specifically, he pointed out that all analysed molybdophyllites show a lowering of the Pb content (of ~10%) with respect to the ideal formula. He gave $Pb_{7.9}(Mg_{8.8}Mn_{0.1}Al_{0.2})Si_{9.0}O_{24}(OH)_{21.8}$ as a representative formula for most samples. The lower Pb content was assumed to be due to "loss of loosely bound interlayer lead." Charalampides (1994) tabulated EPMA data for sample 191581 ("molybdophyllite, representative of the large majority of samples") and sample 100168 which was extremely Pb depleted ("Pb-depleted molybdophyllite"), but gave a PXRD pattern that was typical of molybdophyllite, and attributed the Pb-depleted character to extreme leaching of lead.

Charalampides (1994) also addressed "molybdophyllite-like minerals" and noted that "According to P.B. Moore (University of Chicago, personal communication, 1987), there exists a monoclinic polymorph of molybdophyllite which was not found among the present samples." No further information was given.

In sample 740945 Charalampides (1994) discovered a new mineral, which is macroscopically very similar to molybdophyllite, but has a Pb:Mg:Si ratio of 3:2:2 according to EPMA data. This was described as "18 Å-molybdophyllite". The hexagonal unit-cell parameters of this mineral were determined to be $a$ = 9.34(2), $c$ = 36.79(0) Å [*sic*] by refinement of PXRD data, indicating an increased layer thickness in comparison to molybdophyllite.

A new triclinic-pseudohexagonal mineral species closely related to molybdophyllite was recently described from Långban and named britvinite (IMA 2006-031; Chukanov *et al*., 2008; Yakubovich *et al*., 2008). Britvinite is visually similar to molybdophyllite and forms colourless to pale yellow platelets that are transparent with a white streak and adamantine lustre. The mineral is sectile and mica-like, with a perfect cleavage on {001}. Britvinite has an empirical formula $Pb_{14.75}Mg_{9.03}Si_{9.73}Al_{0.37}O_{30.76}(BO_3)_{3.51}(CO_3)_{2.18}(OH)_{11.17}$ and a simplified formula $Pb_{15}Mg_9(Si_{10}O_{28})(BO_3)_4(CO_3)_2(OH)_{12}O_2$ (Chukanov *et al*., 2008). It is triclinic, space group $P\bar{1}$, with unit-cell parameters $a$ = 9.341, $b$ = 9.360, $c$ = 18.833 Å, $\alpha$ = 80.37, $\beta$ = 75.82 and $\gamma$ = 59.87º.

Chukanov *et al*. (2008) noted that there was a close relationship between britvinite and the ''18 Å-molybdophyllite'' described by Charalampides (1994) [this mineral was described as "molybdophyllite-18 Å" by Chukanov *et al*. (2008), and this name is used hereafter] and maintained: "It cannot be ruled out that britvinite and molybdophyllite-18 Å are identical or, taking into account the double parameter $c$ in the unit cell of molybdophyllite-18 Å, are polytypic modifications."

We can now definitely prove the identity of ''molybdophyllite-18 Å'' and britvinite by comparing (1) the chemical data collected by Chukanov *et al*. (2008) for britvinite with those collected by Charalampides (1994) and by ourselves for "molybdophyllite-18 Å" from sample 740945 (Table 1), and (2) by comparing the crystallographic data presented by Chukanov *et al*. (2008) with those we have obtained for crystals of "molybdophyllite-18 Å": $a$ = 9.337(2), $b$ = 9.359(2), $c$ = 18.929(4) Å, $\alpha$ = 76.01(3), $\beta$ =

TABLE 1. Chemical composition of molybdophyllite and britvinite (in wt.%).

| Sample | 1<br>M | 2<br>M | 3<br>M | 4<br>M | 5a<br>B | 5b<br>B | 6<br>B | 7<br>M |
|---|---|---|---|---|---|---|---|---|
| number* | 020154 | 19419 | unknown | 191581 | 740945 | 740945 | - | Ideal formula |
| Locality | Långban | Långban | Långban | Långban | Långban | Långban | Långban | |
| Reference | This work | This work | Flink (1901) | Charalampides (1994) | Charalampides (1994) | This work | Chukanov *et al.* (2008) | |
| $SiO_2$ | 19.04 | 19.05 | 18.15 | 18.25 | 12.42 | 13.18 | 12.77 | 20.22 |
| $Al_2O_3$ | 0.22 | 0.39 | 0.46 | 0.32 | 0.35 | 0.47 | 0.41 | |
| MgO | 11.10 | 10.90 | 11.71 | 11.94 | 7.98 | 7.64 | 7.95 | 12.21 |
| MnO | 0.28 | 0.18 | | 0.18 | 0.03 | 0.04 | | |
| $Na_2O$ | 0.23 | 0.78 | 0.82 | | | 0.01 | | |
| $K_2O$ | 0.26 | 0.16 | 0.69 | | | 0.00 | | |
| PbO | 59.32 | 58.90 | 61.09 | 59.46 | 70.92 | 69.87 | 71.92 | 60.10 |
| CaO | | | | 0.01 | 0.01 | | | |
| BaO | | | | 0.25 | 0.13 | | | |
| $H_2O$ | 2.72 | n.d. | 6.32 | n.d. | n.d. | n.d. | 2.2 | 3.03 |
| Cl | | | | 0.00 | 0.07 | | | |
| F | | | | n.d. | 0.05 | | | |
| $CO_2$ | 3.04 | n.d. | n.d. | n.d. | n.d. | n.d. | 2.1 | 4.44 |
| $B_2O_3$ | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 2.67† | |
| Total | 96.21 | 90.36 | 99.24 | 90.41‡ | 91.96 | 91.21 | 100.02 | 100.00 |

* Abbreviations are: M is molybdophyllite, B is britvinite (of Chukanov *et al.*, 2008); the britvinite samples 4 (191581) and 5a (740945) were originally described as "molybdophyllite-18 Å" by Charalampides (1994). n.d. is not determined.

† The $B_2O_3$ content calculated on the basis of the crystal-structure determination.

‡ The total weight loss was estimated to be ~8%.

85.75(3) and $\gamma = 60.10(3)^o$. In fact, whereas these parameters correspond to the reduced cell, this is not true for those presented by Chukanov *et al*. (2008), which have to be transformed via the matrix [1 −1 0 / 1 0 0 / 0 0 1] to give $a = 9.332$, $b1 = 9.341$, $c = 18.833$ Å, $\alpha = 75.82$, $\beta = 85.56$ and $\gamma = 60.17^o$. The agreement between the two sets of parameters is excellent, especially taking into account the fact that the data presented by Chukanov *et al*. (2008) were obtained at low temperature (173 K). Therefore, from now on, we shall refer only to britvinite.

We have also carried out a data collection on a crystal of britvinite from sample 740945, followed by structure determination and refinement which confirmed the general aspects of the structural arrangement obtained by Yakubovich *et al*. (2008) for britvinite, with some distinctions in structural details and in the crystal-chemical interpretation. Our structural study of britvinite will be presented in a future paper, which will include new chemical analyses of the light elements (B, C, O, H) and a detailed discussion of the results and comparison with those obtained by Yakubovich *et al*. (2008). In the present paper, which is primarily devoted to the results obtained in our study of molybdophyllite *sensu stricto*, we discuss only those aspects of britvinite that are useful for a better understanding of the modular relationships between the two minerals.

As the crystal structure of molybdophyllite was undetermined prior to our study, we have studied different "molybdophyllite" samples from both Långban and Harstigen intermittently over the course of the last ten years using a combination of single-crystal X-ray diffraction, spectroscopic and analytical techniques. The results reported here demonstrate that molybdophyllite has an OD (order-disorder) structure, a complex crystal chemistry and interesting modular relationships with britvinite.

## Sample descriptions

The molybdophyllite and britvinite samples that were studied, with the exception of the last two, are from the collection of the Swedish Museum of Natural History.

Specimen 221135 consists of crystal fragments in a test tube, from specimen 221131 and is from Harstigen. Pale yellow molybdophyllite occurs on the surface of a fissure in a Mn-bearing skarn, with tephroite, oxides (mostly jacobsite) and carbonates. The molybdophyllite forms coarse crystals (to 10 mm) which are intergrown with calcite, sahlinite and a subordinate orange mineral (possibly berzeliite).

Specimen 335316 is from Långban and consists of spotty hausmannite ore which contains extremely coarse molybdophyllite crystals (to 3 cm) on a fissure surface.

Specimen 020154 is from Långban and consists of massive manganese ore (hausmannite/braunite) with a slab of muscovite-like molybdophyllite, $1 \times 2$ cm across, in a calcite vein. This specimen was studied by Flink (1901).

Specimen 670857 is from Långban and consists of manganese ore with ~40% hausmannite and ~60% carbonate minerals (barytocalcite and calcite). Colourless pearly britvinite forms crystals up to 3 mm which are irregularly distributed in the carbonate fraction with minor forsterite and phlogopite.

Specimen 740945 is from Långban and is very similar to specimen 670857 but smaller and somewhat richer in britvinite. According to Charalampides (1994), the matrix of specimen 740945 consists primarily of hausmannite and barytocalcite. This sample is known as 'the royal sample', as it once belonged to the Swedish king Gustav VI Adolf.

Specimen H 6590 is in the collection of the Natural History Museum, Vienna (catalogued in 1907) and is labelled as molybdophyllite from "Långbanshytten [*sic*], Wermland, Schweden." Small, silvery white, micaceous masses occur on a matrix of "calcite and hausmannite." An older SEM-EDS analysis of a carbon-coated subsample had shown only Pb, Si and Mg.

Specimen 19419 is in the collection of the Museo di Storia Naturale e del Territorio, Università degli Studi di Pisa (hereafter designated 'Pisa') and is molybdophyllite from Långban. A micaceous mass occurs on a matrix composed of carbonates (mainly dolomite), hausmannite and braunite.

## Optical properties

Optical studies of transparent cleavage flakes from two samples that were studied by SXRD (see below) and a two further samples were made using standard polarized-light microscopy. Britvinite sample 740945 is biaxial negative, with medium–small 2V (roughly 10–20º) and $r < v$ weak. These properties are in agreement with the data reported for type britvinite (Chukanov *et al*., 2008). Molybdophyllite sample 221135 is also biaxial negative, the 2V value is very small (<5–10º), and the optical axis figure has a strongly pseudo-isotropic character. This may explain why Flink (1901) considered molybdophyllite to be uniaxial. Molybdophyllite samples 020154 and 335316 were also pseudo-isotropic, but gave slightly sharper optical axis figures. The 2V value was estimated to be around 5–10º.

None of the samples showed any twinning features, although only those directions that were more or less perpendicular to the cleavage plane were investigated.

## Chemical composition

Microchemical studies confirmed that all samples contain carbonate. For molybdophyllite sample 020154, a partial chemical analysis of the carbonate and water content was done using quantitative absorbents ($H_2O$ was determined by adsorption on magnesium perchlorate, and $CO_2$ on granular asbestos). The analysis gave 3.04% $CO_2$ and 2.72% $H_2O$ (sample mass 0.3782 g). The $H_2O$ content (6.32%) reported by Flink (1901) probably corresponds to a weight loss on heating, and therefore includes both $H_2O$ and $CO_2$. Charalampides (1994) estimated a weight loss of "approximately 8%" on heating 20 mg of molybdophyllite.

Preliminary qualitative chemical analyses, using a Philips XL30 SEM equipped with a DX4 EDAX system, were carried on crystals from the following four samples: 221135, 020154, 740945 and 19419. Quantitative electron-microprobe analyses were performed on crystals from samples 020154, 740945 and 19419, using a JEOL JXA-8600 system, operating in wavelength-dispersive spectrometry (WDS) mode; the voltage was set to 15 kV, the beam current to 20 nA and the beam diameter to 12 μm. We did not succeed in preparing a sample of specimen 221135 that was suitable for EMP analysis. However, EDS analyses of crystals from sample

221135 gave results that were closely comparable with those obtained from sample 020154, which indicates that both specimens have very similar compositions.

Several spot analyses were done on each crystal. The standards used were cerussite for Pb, olivine (Spring Water) for Mg, spessartine for Mn, albite for Na and Si, and microcline for K and Al. Analytical results are given in Table 1. The four analyses presented in that table for molybdophyllite, namely the analysis reported by Flink (1901) (3), that of Charalampides (1994) (4) and the two new analyses obtained in the present work, (1) and (2), are consistent and, together with the information obtained from the structural study point to a crystal-chemical formula that can be written $Pb_8Mg_9[Si_{10}O_{28}(OH)_8|O_2|(CO_3)_3]\cdot H_2O$. The theoretical composition corresponding to this formula is reported in Table 1, column 7; it indicates a minor substitution of silicon by aluminium and of magnesium by manganese and possibly sodium. The potassium content in compositions (1), (2) and (3) and the barium content in composition (4) are discussed below.

The results for britvinite sample 740945 obtained in the present study (composition 5a in Table 1) compare well with those given by Charalampides (1994) for molybdophyllite-18 Å (5b) and by Chukanov *et al*. (2008) for type britvinite (6); these last authors proposed the ideal chemical formula $Pb_{15}Mg_9(Si_{10}O_{28})(BO_3)_4(CO_3)_2(OH)_{12}O_2$.

## Infrared and Raman spectroscopy

Infrared spectroscopic data were collected from samples 020154 (molybdophyllite) and 740945 (britvinite). The spectra, which are shown in Fig. 1, were collected using a Bruker Equinox55 microscope-spectrometer fitted with a mercury cadmium telluride (MCT) detector and a halogen lamp. The area of measurement was quadratic ($100 \times 100$ μm), with the sample lying on a $CaF_2$ plate. The measurements were done perpendicular to the cleavage, at a resolution of 4 $cm^{-1}$, and with 128 cycles in the range from 2000 to 4000 $cm^{-1}$. In both spectra the region <2380 $cm^{-1}$ appears to be affected by detector saturation. In the remaining wavenumber range the absorption bands ($cm^{-1}$) and their tentative assignments are described below (b, broad; s, strong; w, weak; sh, shoulder).

(1) Sample 740945 (britvinite): the multiple bands at 3694 $cm^{-1}$ (s), ~3658 $cm^{-1}$ (s), ~3579 $cm^{-1}$ (s), 3520 and 3495 $cm^{-1}$ (both s) and ~3400–3160 $cm^{-1}$ (very broad shoulder) are all assigned to absorptions due to OH stretching vibrations in hydroxyl groups; the weak band at 2780 $cm^{-1}$ may have the same origin but is left unassigned, as is the band at ~2450 $cm^{-1}$ (s, b); the strong bands >3100 $cm^{-1}$ are very similar to those reported for type britvinite (Chukanov *et al*., 2008) where the IR spectrum has three weak bands at 3685 (sharp), ~3570 (broad) and ~3440 $cm^{-1}$ (very broad). The britvinite spectrum

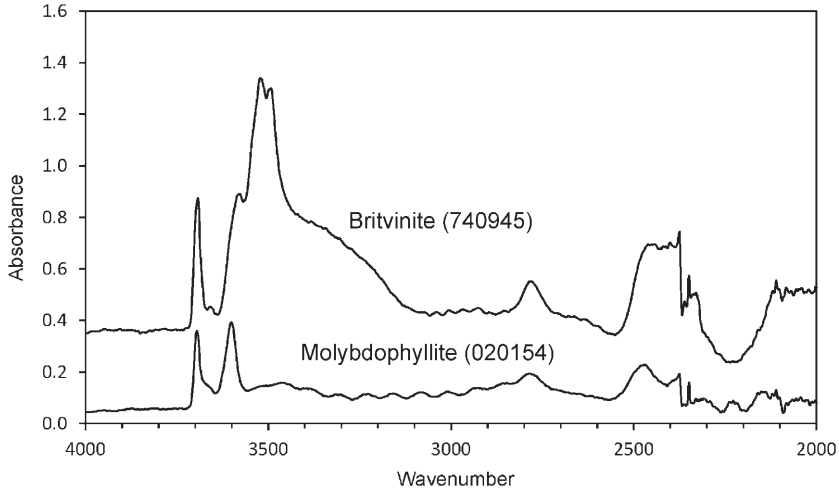


FIG. 1. Infrared spectra collected from single-crystal cleavages of molybdophyllite (sample 020154) and britvinite (sample 740945; offset by 0.2 absorbance units for ease of comparison).

has no other absorption features in the region between ~3200 and 1500 $cm^{-1}$.

(2) Sample 020154 (molybdophyllite): the bands, at 3695 $cm^{-1}$ (s) and ~3663 $cm^{-1}$ (sh) are very similar to those in the spectrum of sample 740945 and point to practically identical OH configurations and O···O bond lengths; the band at ~3601 $cm^{-1}$ (s) reflects a shift towards higher wavenumbers in the spectrum of sample 020154 in comparison to that of sample 740945 (this is also observed in the Raman spectra which are discussed in more detail below). An indistinct, broad and weak bump seems to be located at roughly 3450 $cm^{-1}$ (s) although this is tentative considering the rough background of the spectrum in this region. The two bands at ~2780 (w) and ~2472 $cm^{-1}$ (s, b), which are similar to those in the spectrum of sample 740945, are not assigned and may be artefacts.

The main difference between the infrared spectra is the presence of additional absorption bands due to OH groups in the region between ~3560 and ~3170 $cm^{-1}$ in the britvinite spectrum (sample 740945), which are absent in the spectrum of molybdophyllite (sample 020154). Due to detector saturation in the wavenumber region <2380 $cm^{-1}$, it is not possible make meaningful comparisons regarding the presence or absence of carbonate and borate groups.

Single-crystal laser-Raman spectroscopy was used to investigate the anionic components of samples 020154 (molybdophyllite) and 740945 (britvinite). The spectra were collected using a Renishaw MicroRaman Imaging system (M1000) interfaced with a Leica DMLM optical microscope at a laser excitation wavelength of 633 nm (and also at 488 nm on sample 740945) using unpolarized laser light in 180º backscatter geometry at a resolution of ±2 $cm^{-1}$ with a minimum lateral resolution of ~2 μm in random sample orientation. All of the fragments studied were stable under the laser beam.

The relatively poor spectrum of the molybdophyllite (sample 020154; Fig. 2, bottom) has one strong and sharp band and one weak band in the OH stretching region, at frequencies (3696 and ~3600 $cm^{-1}$) which suggest weak hydrogen bonds, with O···O distances 3.0–3.2 Å (Libowitzky, 1999). The bands in the region between ~800 and 1100 $cm^{-1}$ are assigned to vibrations of the $CO_3$ and $SiO_4$ groups.

The 633 nm spectrum of britvinite (sample 740945; Fig. 2, top) is very similar to the spectrum recorded using the 488 nm laser, but of better quality. Therefore, the following discussion, which will also serve as a basis for a future detailed structural paper on britvinite, focuses on the 633 nm spectrum. In the

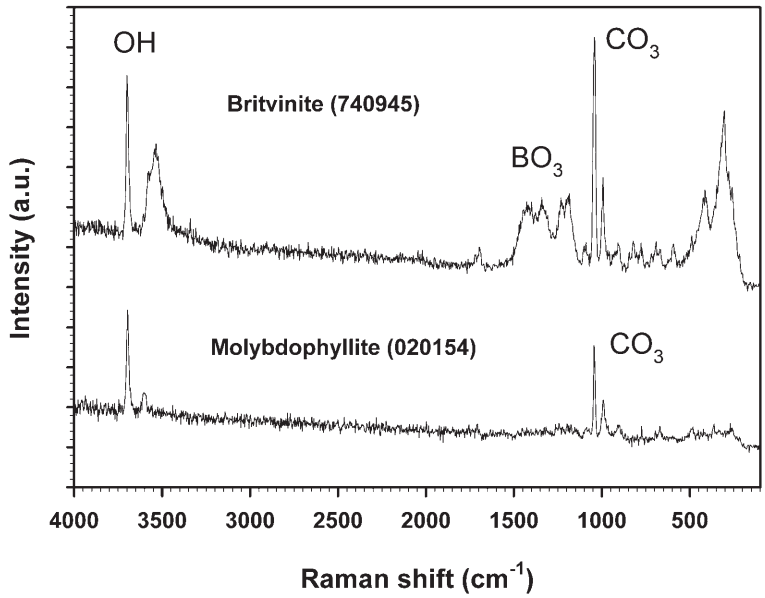


FIG. 2. Single-crystal laser-Raman spectra of molybdophyllite (sample 020154) and britvinite (sample 740945; offset for ease of comparison).

OH-stretching region there are two strong bands, a sharp one at 3697 $cm^{-1}$ (very similar to the one observed for sample 020154) and a broad one, consisting of at least two components, at ~3543 $cm^{-1}$ (stronger by comparison to sample 020154 and shifted towards lower wavenumbers). The frequencies of the two bands correspond to weak hydrogen bonds, with inferred O···O distances similar to those for sample 020154 (~2.9–3.2 Å). These bands can be assigned to bonds involving the OH groups within the brucite layer. Although a very weak band at ~1696 $cm^{-1}$ seems to indicate the presence of molecular water, no water was detected in the structure refinement. As the H atoms belonging to the OH groups were not located in the structural studies, we refrain from tentative band assignments related to the hydrogen bonding. In britvinite, three bands were reported in the OH stretching region of the IR spectrum (Chukanov *et al*., 2008) at 3685 (sharp), ~3570 (broad) and ~3440 $cm^{-1}$ (very broad). The positions of the first two bands show only fair agreement with those in sample 740945; the third band is not present in the Raman spectrum, although a very weakly pronounced slope towards the broad peak at ~3543 $cm^{-1}$ might be considered to be equivalent to this third band.

There is clear evidence of the presence of $CO_3$ and $BO_3$ groups in sample 740945. The region between 900 and 1500 $cm^{-1}$ has overlapping bands due to vibrations of $SiO_4$, $CO_3$ and $BO_3$ groups. Further vibrational bands due to polymerized $SiO_4$ groups are present in the region between ~1000 and 400 $cm^{-1}$. Vibrations of the $MgO_6$ octahedra, Si–O–Si bending vibrations and lattice modes are expected to produce bands in the region <500 $cm^{-1}$. Bands in the range between 1150 and 1500 $cm^{-1}$ are absent in the spectrum of sample 020154 and this indirectly confirms the structural differences between molybdophyllite and britvinite. Due to the triclinic symmetry of britvinite, the closeness of the bands and their overlap, no detailed assignment was attempted. The IR spectrum reported for type material (Chukanov *et al*., 2008) has similar bands. These authors provide a detailed assignment, according to which the broad band at ~1418 $cm^{-1}$ is due to the stretching vibration of the $CO_3$ groups; the bands at ~1349, ~1230 and ~1195 $cm^{-1}$ are assigned to stretching vibration of the $BO_3$ groups; and five partly overlapping bands in the region between 1100 and 900 $cm^{-1}$ are assigned to stretching vibrations of the $SiO_4$ groups. With respect to a hypothetical water content in type britvinite, Chukanov *et al*. (2008) state that "There are no absorption bands in the region of 1500–1700 $cm^{-1}$, which testifies to the absence of water molecules in the mineral."

In conclusion, the IR and Raman data qualitatively confirm the reported anionic components of both of the structurally studied molybdophyllite and britvinite samples. However, as pointed out by N.V. Chukanov (personal communications as a referee, 2011), the IR spectra of some other molybdophyllite samples contain weak bands due to $BO_3$ (these are much weaker than that in IR spectra of britvinite). The IR spectra of different britvinite samples from Långban, as well as britvinite from the Kombat mine (Namibia) are somewhat different in the relative intensities of the vibrational bands of the $CO_3$, $BO_3$ and OH groups. Moreover, in some IR spectra, the band at 3440 $cm^{-1}$ is absent. Thus, our results do not allow a generalization concerning both species.

## Crystal structure determination and refinement

Selected cleavage fragments from all samples were studied using a Bruker-Nonius APEX II diffractometer equipped with a CCD area detector, using Mo$K\alpha$ radiation ($\lambda$ = 0.71073 Å). The molybdophyllite samples 335316, 020154 and H 6590 invariably showed poor crystal quality, irrespective of the size of the individual fragments. The patterns were typically characterized by severe streaking (indicating stacking disorder), subparallel intergrowth and poorly defined reflections. In most cases it was impossible to determine a unit cell. Crystal fragments with distinctly better quality were encountered in sample 221135 (from Harstigen), even though streaking was still present. The intensity of the streaks was between 20 and 50% of the intensity of the immediately adjacent reflection spots. These fragments all indicated a *C*-centred monoclinic unit cell with *a* ~16.25 Å, *b* ~9.37 Å, *c* ~14.0 Å and $\beta$ ~97º.

The britvinite sample 670857 also yielded fragments of poor quality, but sample 740945 provided fragments of better quality, albeit affected by streaking. They clearly and consistently showed triclinic-pseudomonoclinic symmetry (see below).

Using the Weissenberg film method, a suitable fragment from molybdophyllite specimen 19419 was investigated by collecting various

Weissenberg layers (the *h0l* pattern is figured in the Appendix). The whole pattern is characterized by the simultaneous presence of rows of sharp spots and rows of diffuse reflections or continuous streaking, both rows running parallel to *c**. The set of sharp spots defines a rhombohedral cell with *a* ~3.1 Å, *c* ~42 Å, and displays space group symmetry *R*32. Those aspects point to an OD character of molybdophyllite, as discussed in the Appendix.

Intensity data from a suitable fragment of sample 221135 (molybdophyllite) and sample 740945 (britvinite) were collected, processed with the Nonius program suite *DENZO-SMN* and corrected for Lorentz, polarization and background effects. An initial standard absorption correction using the multi-scan method (Otwinowski *et al*., 2003) did not produce satisfactory results, so a semi-empirical absorption correction was made using *XEMP* (Sheldrick, 1988) for both datasets.

### *Molybdophyllite (sample 221135)*

The intensity statistics for sample 221135 suggested a lack of a centre of symmetry, and internal *R*-values suggested either a primitive triclinic or a *C*-centred monoclinic unit cell. The crystal structure was successfully solved in space group *P*1 using direct methods (*SHELXS-97*; Sheldrick, 2008) but strong correlations between similar atoms suggested the possibility of higher symmetry. A subsequent symmetry analysis of the refined structure model using *PLATON* (Spek, 2003, 2009) clearly pointed to space group *C*2. Alternate trial solutions in the centrosymmetric space groups $P\bar{1}$ and *C*2/*m* gave physically impossible results. Final atom coordinates and anisotropic displacement parameters were refined in space group *C*2 by full-matrix least-squares techniques on $F^2$ using *SHELXL-97* (Sheldrick, 2008).

The first stage of refinement in *C*2 led to an agreement factor $R(F) = 0.123$ [for 2996 unique reflections with $F_o > 4\sigma(F_o)$]. The Pb, Mg, Si cations, as well as most of the oxygen atoms had been correctly located, but the carbonate groups were not yet clearly defined and large residual peaks were found in the difference Fourier synthesis.

The large residual peaks were located at $z = 0.34$ and $z = 0.44$, at the levels of the Pb atoms, and were stacked with respect to the positions of each Pb atom according to a hexagonal two-dimensional net with translation periods $a = b = 3.1$ Å. This peculiar distribution was easily explained and the refinement procedure satisfactorily completed once an OD approach was applied. The OD character of molybdophyllite is particularly evident in specimens from Långban, which display a complex diffraction pattern characterized by the simultaneous presence of sharp spots, diffuse reflections and continuous streaks. This OD character is also manifest in specimens from Harstigen, although there is a lower degree of disorder.

A detailed description of the OD character of molybdophyllite is presented in the Appendix. In this section we only discuss the difficulties we met during the refinement process.

Molybdophyllite can be described in terms of equivalent layers (OD layers) with trigonal symmetry ($a_1 = a_2 = 9.373$ Å; width of the layer $c_0 = c\sin\beta = 13.944$ Å), which may build up according to three distinct stacking vectors. Distinct ways of stacking neighbouring layers allow the existence of a series of both disordered and ordered sequences (polytypes), constituting a family of OD structures.

The various possible disordered and ordered structures display diffraction patterns with common features [*family reflections* are reflections which present the same position and intensities in all the structures of the family; these are reflections with $h = 3n$ and $k = 3n$ in the present case, with reference to the basic vectors of the single layer] and they are distinguished by the position and intensities of the other reflections. The family reflections are always sharp and define the unit cell of the family structure ($a = 3.124$, $c = 41.832$ Å, space group *R*32). The other reflections [*characteristic reflections*] can be more or less diffuse, sometimes appearing as continuous streaks along *c**, if the three stacking sequences follow each other in a random fashion (this is the case for the majority of the molybdophyllite samples from Långban). If there is a single ordered stacking sequence (a polytype), reflections corresponding to $h$ and $k$ different from $3n$ will be sharp and correspond to the particular structure with its specific periodicities and space-group symmetry.

Therefore, the family reflections receive intensity contributions from all of the possible ordered or disordered sequences present in the crystal under study. For this reason and also because the family reflections are always sharp, distinct scaling factors for the two types of reflections are necessary. By trial and error we found that the

best results (with the lowest $R$ values and lowest residual maxima in the difference-Fourier map) were obtained by rescaling the intensities of the family reflections by a factor 0.55, i.e. those reflections which present indices $h = 3n$ and $k = 3n$, also in the reference frame of the monoclinic structure under study.

The refinement was concluded by examining the new difference-Fourier synthesis, which had relatively low residual peaks and allowed the positions of all the atoms of the two carbonate groups, placed on the plane at $z = 0.5$, together with a water molecule, to be located, and by carrying out additional least-squares refinement cycles. The $R_1$ value, obtained for a model with anisotropic refinement applied only to the Pb and Si atoms (no significant improvement was obtained for an additional anisotropic treatment of the Mg atoms), was 0.096 for 2996 reflections with $F_o > 4\sigma(F_o)$. The correction of the intensity dataset for OD character not only reduced the $R(F)$ values, but also led to an improvement of the goodness-of-fit value from 1.526 to 1.063, and a decrease of the Flack parameter from 0.14(4) to 0.04(4). The OD character of the measured crystal is reflected by fairly large uncertainties in the metal–O bond lengths (0.03 to 0.07) and refined C–O bond lengths, which range from 1.08(10) to 1.50(11) Å.

Information on crystal data and details of the structure refinement is presented in Table 2. The final positional and displacement parameters are listed in Tables 3 and 4, respectively. Selected bond lengths are listed in Table 5 and the results of bond valence calculations are presented in Table 6.

TABLE 2. Crystallographic data and refinement parameters for molybdophyllite (sample 221135, from Harstigen).

| | |
|---|---|
| **Crystal data** | |
| Simplified empirical formula | $Pb_8Mg_9[Si_{10}O_{28}(OH)_8\|O_2\|(CO_3)_3]\cdot H_2O$ |
| Crystal system | monoclinic |
| Space group | $C2$ |
| Unit-cell parameters $a$, $b$, $c$ (Å) | 16.232(6), 9.373(2), 14.060(3) |
| β (º) | 97.36(4) |
| Unit-cell volume (Å$^3$) | 2121.5(10) |
| $Z$ | 2 |
| Calculated density (g cm$^{-3}$) | 4.652 |
| Absorption coefficient (mm$^{-1}$) | 32.183 |
| Crystal dimensions (mm) | $0.02 \times 0.13 \times 0.15$ |
| **Data collection** | |
| Diffractometer | Nonius KappaCCD |
| Temperature (K) | 293 |
| Radiation, wavelength (Å) | Mo$K\alpha$, 0.71073 |
| θ range for data collection (º) | 2.51–30.06 |
| $h$, $k$, $l$ ranges | −16/22, −13/9, −17/14 |
| Axes, frame width (º), time per frame (s) | φ, ω, 1, 370 |
| Total reflections collected | 4215 |
| Unique reflections ($R_{int}$) | 3164 (0.0833) |
| Unique reflections $F > 4\sigma(F)$ | 2996 |
| Data completeness to $\theta_{max}$ (%) | 72.5 |
| Absorption correction method | *XEMP* (Sheldrick, 1988) |
| **Structure refinement** | |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Weighting coefficients $a$, $b$ | 0.142, 616.8878 |
| Flack parameter | 0.04(4) |
| Data/restraints/parameters | 3164/1/204 |
| $R_1$ [$F > 4\sigma(F)$], $wR_2$ [$F > 4\sigma(F)$] | 0.0960, 0.2542 |
| $R_1$ all, $wR_2$ all | 0.1018, 0.2678 |
| Goodness-of-fit on $F^2$ | 1.063 |
| Largest difference peak and hole (e$^-$ Å$^{-3}$) | 5.0, −6.4 |

*Britvinite (sample 740945)*

Intensity statistics for sample 740945 suggested the presence of a centre of symmetry. The $R_{int}$ values clearly indicated triclinic symmetry. The cell metrics: $a$ = 9.337(2), $b$ = 9.359(2), $c$ = 18.929(4) Å, α = 76.01(3), β = 85.75(3), γ = 60.10(3)º, are strongly pseudomonoclinic, conforming to a *C*-centred monoclinic pseudocell with $a$ ~16.19, $b$ ~9.35, $c$ ~19.66 Å, α ~89.6, β ~111.1, γ ~90.2º. The crystal structure was successfully solved in space group $P\bar{1}$ using direct

TABLE 3. Fractional atom coordinates and isotropic displacement parameters (Å$^2$) for molybdophyllite (sample 221135).*

| Atom | *x*/*a* | *y*/*b* | *z*/*c* | $U_{eq}/U_{iso}$ |
|---|---|---|---|---|
| Pb1 | 0.53822(11) | 0.76414(16) | 0.34453(15) | 0.0255(5) |
| Pb2 | 0.26645(11) | 1.24371(17) | 0.34476(15) | 0.0262(5) |
| Pb3 | 0.32704(16) | 0.6395(3) | 0.44455(16) | 0.0400(6) |
| Pb4 | 0.64230(11) | 0.41228(17) | 0.34460(15) | 0.0259(5) |
| Si1 | 0.6339(8) | 0.9912(12) | 0.1982(11) | 0.025(3) |
| Si2 | 0.4646(9) | 1.1385(14) | 0.1896(11) | 0.027(3) |
| Si3 | 0.2997(8) | 0.6403(12) | 0.1894(9) | 0.019(2) |
| Si4 | 0.3087(9) | 0.9607(13) | 0.1978(11) | 0.023(3) |
| Si5 | 0.4568(8) | 1.4634(12) | 0.1973(11) | 0.023(3) |
| Mg1 | 0.5 | 1.308(2) | 0.0 | 0.019(4) |
| Mg2 | 0.6644(9) | 0.8048(16) | 0.0057(13) | 0.019(3) |
| Mg3 | 0.5 | 0.972(2) | 0.0 | 0.024(4) |
| Mg4 | 0.1670(10) | 0.9737(16) | 0.0091(13) | 0.022(3) |
| Mg5 | 0.6663(9) | 1.1407(17) | –0.0034(12) | 0.021(3) |
| Mg6 | 0.5 | 0.639(2) | 0.0 | 0.019(4) |
| O1 | 0.454(2) | 0.811(3) | 0.073(3) | 0.023(6) |
| O2 | 0.2832(16) | 0.641(3) | 0.074(2) | 0.013(5) |
| O3 | 0.7890(17) | 0.811(3) | 0.072(2) | 0.016(5) |
| O4 | 0.3813(17) | 1.304(3) | –0.078(2) | 0.015(5) |
| O5 | 0.390(2) | 1.050(4) | 0.239(3) | 0.031(7) |
| O6 | 0.552(2) | 1.075(4) | 0.237(3) | 0.031(8) |
| O7 | 0.621(2) | 0.979(3) | 0.080(3) | 0.025(6) |
| O8 | 0.461(2) | 1.306(3) | 0.231(3) | 0.025(7) |
| O9 | 0.4560(19) | 1.137(4) | 0.078(3) | 0.025(6) |
| O10 | 0.4494(19) | 1.469(3) | 0.078(3) | 0.022(6) |
| O11 | 0.6212(18) | 0.642(3) | 0.072(2) | 0.021(6) |
| O12 | 0.296(3) | 0.971(5) | 0.081(4) | 0.043(10) |
| O13 | 0.323(2) | 0.794(4) | 0.232(3) | 0.033(8) |
| O14 | 0.6542(19) | 0.642(4) | 0.393(3) | 0.023(6) |
| O15 | 0.714(2) | 1.092(3) | 0.230(3) | 0.024(6) |
| O16 | 0.240(2) | 1.023(4) | 0.257(3) | 0.026(7) |
| O17 | 0.372(2) | 0.528(4) | 0.230(3) | 0.032(7) |
| O18 | 0.532(2) | 1.542(3) | 0.254(3) | 0.023(6) |
| O19 | 0.645(2) | 0.852(4) | 0.257(3) | 0.034(8) |
| OW20 | 0.5 | 1.140(5) | 0.5 | 0.021(8) |
| OC1 | 0.5 | 0.625(5) | 0.5 | 0.027(9) |
| OC4 | 0.604(3) | 0.898(5) | 0.500(4) | 0.045(10) |
| OC3 | 0.335(3) | 1.103(5) | 0.505(4) | 0.052(12) |
| OC2 | 0.069(3) | –0.090(5) | 0.502(4) | 0.044(10) |
| OC5 | 0.243(4) | 0.398(7) | 0.496(5) | 0.061(13) |
| C1 | 0.0 | –0.025(12) | 0.5 | 0.06(3) |
| C2 | 0.167(6) | 0.488(11) | 0.502(8) | 0.07(2) |

* Oxygen atoms of the hydroxyl groups are O1, O3, O4 and O11. The oxygen atoms of the $H_2O$ molecule and the $CO_3$ groups are denoted OW and OC, respectively. The $U_{eq}$ values are according to Fischer and Tillmanns (1988).

TABLE 4. Anisotropic displacement parameters (Å$^2$) for molybdophyllite (sample 221135).

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Pb1 | 0.0295(8) | 0.0232(8) | 0.0226(12) | 0.0029(6) | −0.0014(7) | 0.0004(6) |
| Pb2 | 0.0323(8) | 0.0229(8) | 0.0240(12) | 0.0017(6) | 0.0049(7) | −0.0013(6) |
| Pb3 | 0.0588(14) | 0.0505(13) | 0.0091(13) | 0.0014(8) | −0.0019(10) | −0.0003(10) |
| Pb4 | 0.0327(8) | 0.0215(8) | 0.0224(12) | −0.0032(6) | −0.0006(7) | 0.0013(6) |
| Si1 | 0.033(6) | 0.013(5) | 0.031(9) | 0.002(5) | 0.008(6) | −0.007(4) |
| Si2 | 0.040(7) | 0.020(5) | 0.020(8) | −0.003(5) | 0.006(6) | 0.004(5) |
| Si3 | 0.037(6) | 0.012(4) | 0.007(7) | −0.004(4) | −0.002(5) | 0.004(4) |
| Si4 | 0.037(7) | 0.019(5) | 0.015(8) | −0.009(4) | 0.006(6) | 0.000(4) |
| Si5 | 0.030(6) | 0.009(4) | 0.027(9) | −0.005(4) | −0.007(5) | 0.005(4) |

methods (*SHELXS-97*; Sheldrick, 2008) and refined by full-matrix least-squares techniques on $F^2$ using *SHELXL-97* (Sheldrick, 2008). The main structural features confirm the results obtained by Yakubovich *et al*. (2008) in their structural investigation on type britvinite, but with some differences in structural detail and crystal-chemical interpretation. In fact, Yakubovich *et al*. (2008) gave a crystal-chemical formula of $Pb_{14}[Mg_9Si_{10}O_{28}(OH)_6][(BO_3)_4(CO_3)_2(OH)_6F_2]$ for type britvinite, whereas our results indicate a fluorine-free crystal-chemical formula of $Pb_{15}[Mg_9Si_{10}O_{28}(OH)_8][(BO_3)_3(CO_3)_3(OH)_3O_3]$.

## Description of the structure of molybdophyllite

The asymmetric unit of molybdophyllite (sample 221135) contains four Pb, five Si, six Mg, two C and 25 O atoms, one of which belongs to a water molecule (OW), five of which belong to two different carbonate groups (OC1, OC2, OC3, OC4 and OC5), and four to hydroxyl groups (O1, O3, O4, O11). The derived formula is $Pb_8Mg_9[Si_{10}O_{28}(OH)_8|O_2|(CO_3)_3]\cdot H_2O$.

The structure normal to (001) is illustrated in Fig. 3; in the figure various elements are represented: (1) the infinite brucite-type layer of edge-sharing $MgO_6$ octahedra; (2) the infinite layers with composition $[Si_5O_{14}]$ which are placed on both sides of the octahedral layer; (3) the $[OPb_4]$ groups, centred on the O14 oxygen atom which coordinates four $Pb^{2+}$ cations located at the corners of a tetrahedron (the three basal $Pb^{2+}$ cations Pb1, Pb2 and Pb4 and the apical $Pb^{2+}$ cation Pb3 are represented by large green and blue spheres, respectively); (4) the $CO_3$ groups and the water molecules $H_2O$ (small yellow circles), located at $z$ = 0.5. The average bond lengths of the coordination polyhedra (Table 5) show reasonable to good agreement with expected values (cf. Baur, 1981; Krivovichev, 2009).

The twofold axis in the structure parallel to $b$ passes through the alignment of water molecules. As well as the upward-pointing $[OPb_4]$ tetrahedra, illustrated in Fig. 3, symmetrical downward-pointing tetrahedra are also present; only the apical $Pb^{2+}$ (Pb3) is indicated in Fig. 3. The sequence of the various structural elements along $c$ is illustrated in Fig. 4.

### *Brucite-type layer with composition $Mg_9O_{10}(OH)_8$*

Among the six crystallographically independent $Mg^{2+}$ cations, three (Mg1, Mg3, Mg6) are located in special positions and follow each other along the twofold axis, three (Mg2, Mg4, Mg5) are in general positions. The mean Mg−O bond length in the six $MgO_6$ octahedra is 2.10 Å (range 2.07−2.12 Å), which closely corresponds to the sum of the effective ionic radii of $^{VI}Mg^{2+}$ and $^{IV}O^{2-}$, which is 2.098 Å (Shannon, 1976), and is similar to the Mg−O bond distance in brucite. Ten oxygen anions, five on each side of the layer, correspond to the connection points with the apical corners of the tetrahedral layers; the eight hydroxyl anions (four on each side) correspond to the free corners of the octahedral layer. Very minor substitution of Mg by Mn (and possibly by Na) is suggested by the chemical data.

### *Tetrahedral layer, with composition $Si_5O_{14}$*

The tetrahedral layer, illustrated in Figs 3 and 4, may be obtained from a continuous tetrahedral layer (a characteristic feature of the mica-like sheet silicates, with composition $Si_6O_{15}$) by

TABLE 5. Selected bond distances (Å) for the coordination polyhedra in molybdophyllite (sample 221135). The bond angles (°) in the [$OPb_4$] tetrahedron are also given.

| | | | | | |
|---|---|---|---|---|---|
| Pb1–O14 | 2.23(3) | Pb2–O14 | 2.24(3) | Pb4–O14 | 2.26(3) |
| –O19 | 2.40(4) | –O19 | 2.42(4) | –O16 | 2.37(3) |
| –O18 | 2.43(3) | –O16 | 2.42(4) | –O18 | 2.40(3) |
| –OC4 | 2.63(5) | –OC5 | 2.64(7) | –OC2 | 2.64(5) |
| –OC1 | 2.69(2) | –OC3 | 2.73(6) | –OC5 | 2.73(7) |
| [ –O6]* | [3.30(4)] | [ –O5]* | [3.21(4)] | [ –O8]* | [3.32(4)] |
| **<Pb1–O>** | **2.48** | **<Pb2–O>** | **2.49** | **<Pb4–O>** | **2.48** |
| Pb3–O14 | 2.27(4) | | | Pb1–O14–Pb2 | 112.2(1.4) |
| –OC4 | 2.74(5) | | | Pb1–O14–Pb4 | 111.2(1.4) |
| –OC2 | 2.77(5) | | | Pb1–O14–Pb3 | 108.1(1.3) |
| –OC5 | 2.78(6) | | | Pb2–O14–Pb4 | 111.0(1.3) |
| –OC1 | 2.819(4) | | | Pb2–O14–Pb3 | 107.4(1.4) |
| –OC5 | 2.83(5) | | | Pb4–O14–Pb3 | 106.7(1.3) |
| –OC3 | 2.85(6) | | | | |
| [ –O13]* | [3.31(4)] | | | | |
| [ –O15]* | [3.35(4)] | | | | |
| [ –O17]* | [3.36(4)] | | | | |
| **<Pb3–O>** | **2.72** | | | | |
| Si1–O19 | 1.54(4) | Si2–O9 | 1.55(4) | Si3–O13 | 1.59(4) |
| –O15 | 1.62(4) | –O6 | 1.60(4) | –O2 | 1.61(3) |
| –O7 | 1.65(4) | –O8 | 1.68(4) | –O17 | 1.62(4) |
| –O6 | 1.70(4) | –O5 | 1.69(4) | –O15 | 1.63(3) |
| **<Si1–O>** | **1.63** | **<Si2–O>** | **1.63** | **<Si3–O>** | **1.61** |
| Si4–O16 | 1.59(4) | Si5–O18 | 1.55(4) | | |
| –O5 | 1.61(4) | –O8 | 1.55(4) | | |
| –O12 | 1.63(6) | –O17 | 1.63(4) | | |
| –O13 | 1.64(4) | –O10 | 1.66(4) | | |
| **<Si4–O>** | **1.62** | **<Si5–O>** | **1.60** | | |
| Mg1–O4 (×2) | 2.09(3) | Mg3–O1 (×2) | 2.02(4) | Mg6–O11 (×2) | 2.10(3) |
| –O10 (×2) | 2.09(3) | –O9 (×2) | 2.07(4) | –O1 (×2) | 2.10(4) |
| –O9 (×2) | 2.12(4) | –O7 (×2) | 2.13(4) | –O10 (×2) | 2.16(3) |
| **<Mg1–O>** | **2.10** | **<Mg3–O>** | **2.07** | **<Mg6–O>** | **2.12** |
| Mg2–O11 | 1.97(3) | Mg4–O11 | 2.00(3) | Mg5–O3 | 2.04(3) |
| –O1 | 2.09(4) | –O4 | 2.06(3) | –O2 | 2.06(3) |
| –O7 | 2.11(4) | –O3 | 2.09(3) | –O12 | 2.07(5) |
| –O3 | 2.12(3) | –O10 | 2.12(4) | –O7 | 2.11(4) |
| –O12 | 2.13(5) | –O2 | 2.17(3) | –O4 | 2.11(3) |
| –O2 | 2.14(3) | –O12 | 2.20(5) | –O9 | 2.12(4) |
| **<Mg2–O>** | **2.09** | **<Mg4–O>** | **2.11** | **<Mg5–O>** | **2.09** |
| C1–OC2 (×2) | 1.28(7) | C2–OC3 | 1.08(10) | | |
| –OC1 | 1.40(12) | –OC4 | 1.33(10 | | |
| **<C1–O>** | **1.32** | –OC5 | 1.50(11) | | |
| | | **<C2–O>** | **1.30** | | |

* Not considered in the calculation of the average bond length. However, these long distances have been included in the calculation of the bond-valence sums of the Pb atoms, as suggested by Krivovichev and Brown (2001).

TABLE 6. Bond valence balance in molybdophyllite (sample 221135).*

| | Mg1 | Mg2 | Mg3 | Mg4 | Mg5 | Mg6 | Si1 | Si2 | Si3 | Si4 | Si5 | Pb1 | Pb2 | Pb3 | Pb4 | C1 | C2 | Σc† Σc′ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| O1 | | .343 | .416 ×2↓ | | | .334 ×2↓ | | | | | | | | | | | | 1.093 |
| O2 | | .296 | | .276 | .368 | | | | 1.045 | | | | | | | | | 1.985 |
| O3 | | .319 | | .345 | .387 | | | | | | | | | | | | | 1.051 |
| O4 | .343 ×2↓ | | | .368 | .321 | | | | | | | | | | | | | 1.032 |
| O5 | | | | | | | | .846 | | 1.030 | | | .078 | | | | | 1.954 |
| O6 | | | | | | | .822 | 1.07 | | | | .065 | | | | | | 1.957 |
| O7 | | .320 | .304 ×2↓ | | .327 | | .936 | | | | | | | | | | | 1.887 |
| O8 | | | | | | | | .870 | | | 1.221 | | | | .063 | | | 2.154 |
| O9 | .315 ×2↓ | | .358 ×2↓ | | .311 | | | 1.208 | | | | | | | | | | 2.192 |
| O10 | .338 ×2↓ | | | .318 | | .285 ×2↓ | | | | | .896 | | | | | | | 1.837 |
| O11 | | .414 | | .379 | | .337 ×2↓ | | | | | | | | | | | | 1.130 |
| O12 | | .309 | | .255 | .364 | | | | | .973 | | | | | | | | 1.901 |
| O13 | | | | | | | | | 1.100 | .951 | | | | .064 | | | | 2.115 |
| O14 | | | | | | | | | | | | .575 | .573 | .536 | .548 | | | 2.232 |
| O15 | | | | | | | .999 | | .972 | | | | | .059 | | | | 2.030 |
| O16 | | | | | | | | | | 1.111 | | | .392 | | .438 | | | 1.941 |
| O17 | | | | | | | | | 1.005 | | .992 | | | .058 | | | | 2.055 |
| O18 | | | | | | | | | | | 1.212 | .382 | | | .412 | | | 2.006 |
| O19 | | | | | | | 1.269 | | | | | .411 | .394 | | | | | 2.074 |
| OC1 | | | | | | | | | | | | .230 ×2→ | | .174 ×2→ | | 1.333 | | 2.141 |
| OC2 | | | | | | | | | | | | | | .191 | .250 | 1.333 ×2↓ | | 1.774 1.904 |
| OC3 | | | | | | | | | | | | | .209 | .169 | | | 1.333 | 1.711 1.841 |
| OC4 | | | | | | | | | | | | .260 | | .204 | | | 1.333 | 1.797 1.927 |
| OC5 | | | | | | | | | | | | | .250 | .187 .164 | .210 | | 1.333 | 2.144 |
| Σa | 1.992 | 2.001 | 2.156 | 1.941 | 2.078 | 1.912 | 4.026 | 3.994 | 4.122 | 4.065 | 4.321 | 1.923 | 1.896 | 1.806 | 1.921 | 4.00 | 4.00 | |

* The symbol ×2↓ indicates a double contribution to the bond-valence sum of the cation; the symbol ×2→ indicates a double contribution to the bond-valence sum of the anion.

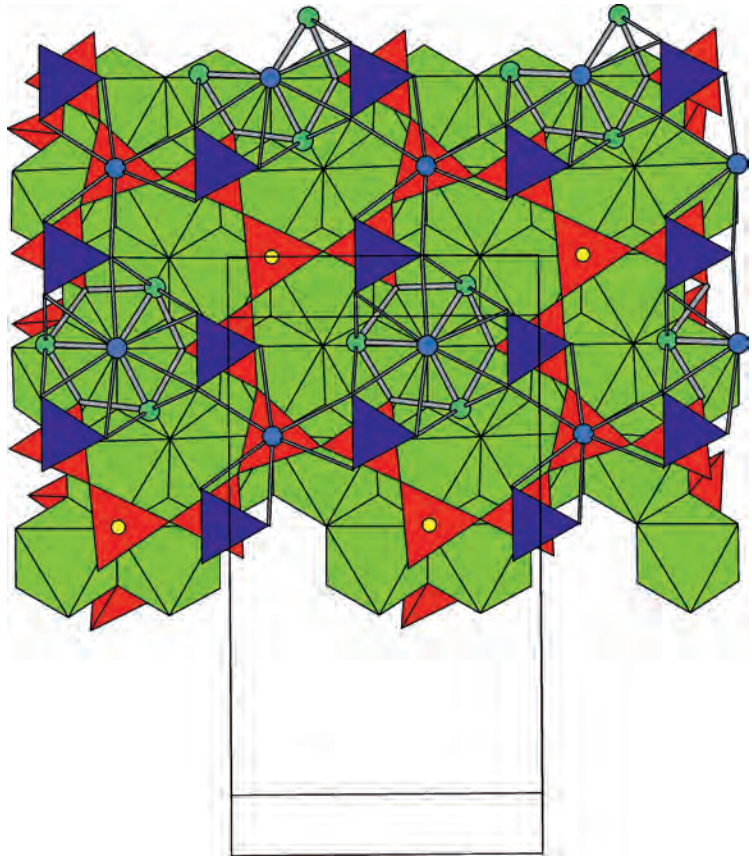

FIG. 3. The structure of molybdophyllite as seen along the direction normal to the (001) plane (*a* vertical, *b* horizontal). Green octahedra are $MgO_6$, red tetrahedra are $SiO_4$, blue triangles are $CO_3$, greenish spheres indicate Pb1, Pb2 and Pb4, blue spheres indicate Pb3 and yellow spheres show the position of the oxygen atom in $H_2O$.

omitting a tetrahedron in the connection point of three six-membered rings, to produce a layer with the composition $Si_5O_{14}$. Among the nine basal oxygen atoms, six are shared by two silicon atoms and three (O16, O18, O19) are 'free', (i.e. they are bonded to only one silicon atom). The average bond lengths in the $SiO_4$ tetrahedra range from 1.60 to 1.63 Å. The small amounts of Al detected in our analyses are assumed to substitute for Si (as was proposed for britvinite; see Yakubovich *et al*., 2008), although a minor Al-for-Mg substitution in the brucite-like layer cannot be excluded.

The linkage of the $SiO_4$ tetrahedra in molybdophyllite results in a layer composed of twelve-membered rings. A topologically identical layer was reported in the structure of britvinite by Yakubovich *et al*. (2008), and has been found in our own data (in preparation). Yakubovich *et al*. (2008) observed that similar layers occur in the structure of zeophyllite, $Ca_{13}[Si_5O_{14}]_2(F,OH)_{10}{\cdot}6H_2O$ (Merlino, 1972), but that they are somewhat modified because, whereas in britvinite and in molybdophyllite all the apical corners point in the same direction, in zeophyllite the tetrahedra point in opposite directions in a 3:1 ratio. The same authors also noted that twelve-membered layers, with tetrahedra pointing in opposite directions in a 3:2 ratio, occur in the structures of the orthorhombic and hexagonal modifications of the synthetic compound $Rb_6Si_{10}O_{23}$, giving rise, through condensation, to frameworks characterized by wide channels delimited by twelve-membered rings, which host the $Rb^+$ cations (Schichl *et al*., 1977; Lapshin *et al*., 2006).

The tetrahedral layer may be also described as being built up by infinite chains with repeats after

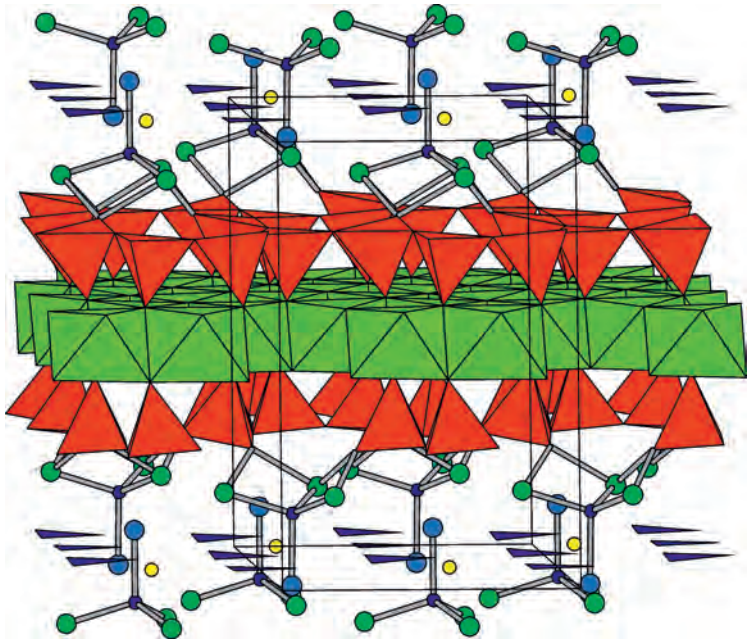

FIG. 4. The structure of molybdophyllite along [100], with a slight rotation to produce a more instructive illustration. Green octahedra are $MgO_6$, red tetrahedra are $SiO_4$, blue triangles are $CO_3$, greenish spheres indicate Pb1, Pb2 and Pb4, blue spheres indicate Pb3 and yellow spheres show the position of the oxygen atom in $H_2O$. The central oxygen atom of the $OPb_4$ groups is represented as a small blue sphere.

four tetrahedra [Si2 to Si4; *viererketten*, according to the terminology of Liebau (1985)], which run along *b* and are interconnected through additional Si1 tetrahedra. The *viererketten* in the structure of molybdophyllite are similar in their shape and periodicity to that in gageite and especially in balangeroite (Ferraris *et al*., 1987): in their structures the chains are grasped on both sides by $3 \times 1$ ribbons of octahedra, centred on $Mn^{2+}$ and $Mg^{2+}$, respectively. In molybdophyllite the tetrahedral layers are connected to both sides of the brucite sheet and related by the twofold axis passing through Mg1, Mg3, Mg6 cations.

### *[$OPb_4$] tetrahedral groups*

The O14 oxygen atom is the only one that is not linked to a silicon or carbon atom. It is strongly bonded to four Pb atoms in a regular tetrahedral coordination (bond distances and angles are given in Table 5). The three basal $Pb^{2+}$ cations (Pb1, Pb2, Pb4) have quite similar coordination, with three short bonds to O14 and two 'free' oxygen atoms of the tetrahedral sheet, and two longer bonds to two oxygen atoms of two different carbonate groups. All the five bonds are at one side, with a lone pair of electrons (a characteristic feature of the $Pb^{2+}$ cation) on the opposite side. The average Pb–O bond length in the Pb1-, Pb2- and Pb4-centred polyhedra (neglecting all Pb–O distances >3.2 Å) is fairly constant at 2.48, 2.49 and 2.48 Å, respectively.

In contrast, the apical Pb3 cation is characterized by an average Pb–O bond length of 2.72 Å, and the Pb3–O polyhedron has a highly asymmetric bond-length distribution; it is linked to O14 by one very short bond of 2.27(4) Å and to oxygen atoms from three different carbonate groups by six long bonds, in the fairly narrow range 2.74(5) to 2.85(6) Å. All of the bonds are on one side, leaving the other side for the lone electron pair. Thus, all the Pb atoms in molybdophyllite show stereochemical activity.

### *Layer of carbonate groups and water molecules*

The distribution of the carbonate groups and water molecules in the layer located at $z = 0.5$ is illustrated in Fig. 5. The carbon atom C1, the oxygen atom OC1, and the oxygen atom of the water molecule (OW20) are located in special

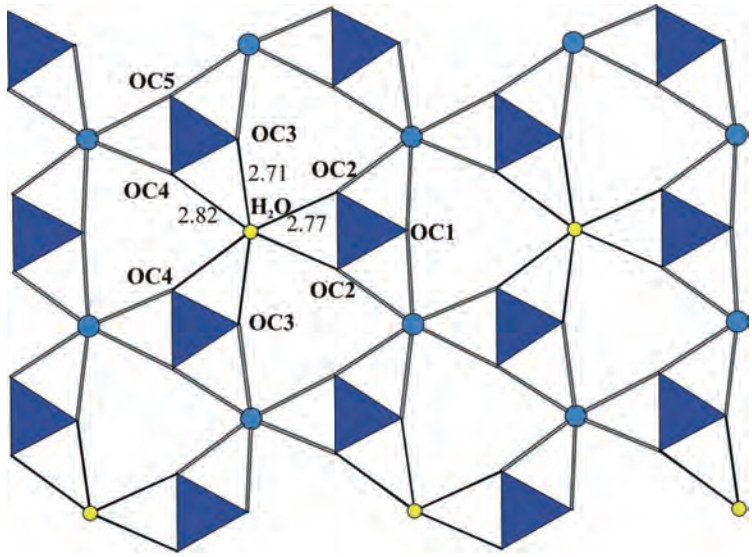


FIG. 5. Layer of carbonate groups (blue triangles) and water molecules (yellow) at $z = 0.5$. The bonds of the oxygen atoms belonging to the carbonate groups with the Pb atoms (blue) and the OW water molecule (yellow) are indicated (with bond distances in Å).

positions on the twofold axis. In the two $CO_3$ groups, the values for the average C–O bond lengths are similar at 1.32 and 1.30 Å, although these values have large statistical uncertainties.

We have already noted that the various EMP analyses of molybdophyllite indicate the limited presence of $K^+$ (compositions (1), (2), (3) in Table 1) or $Ba^{2+}$ (composition (4) in Table 1). A possible location for these cations is as partial substituents for $H_2O$, possibly in positions displaced from the plane of the carbonate groups, namely at $z$ ~0.465 and $z$ ~0.535. In this way the cations may form six bonds of length 2.8 Å on one side and three long bonds (3.3 to 3.4 Å) on the other side.

### *Bond-valence sums and the hydrogen-bond system*

The bond-valence sums for the various oxygen atoms in the structure (only the oxygen atom belonging to the OW20 water molecule, which is not linked to any cation in the structure, is omitted) are reported in Table 6. Calculations were carried out following Brese and O'Keefe (1991), apart from the contributions from the Pb–O bonds, which were evaluated using the parameters suggested by Krivovichev and Brown (2001): $r_0 = 1.963$ Å and $b = 0.49$. Krivovichev (1999) noted that $O^{2-}$ anions encapsulated by $Pb^{2+}$ cations in $OPb_4$ clusters "consistently show bond-valence sums that are higher than expected." The bond-valence sum calculated using the parameters of Brese and O'Keefe (1991) for O14, the central anion of the $OPb_4$ clusters in molybdophyllite, was very large at 2.765 valence units (vu).

It is useful to note that a more general approach to the problem of the deviations of calculated bond-valence sums from stoichiometric valence values has been introduced by Liebau *et al.* (2009). They maintain that two distinct types of valence exist, the stoichiometric valence and the structural valence; in the large majority of inorganic structures they have closely similar values, but they may differ in some materials, for example in compounds containing lanthanide atoms (Liebau and Wang, 2008) or atoms such as lead with a lone electron pair (Liebau, 2000; Wang and Liebau, 2007).

In Table 6, the ideal value of 1.333 vu has been assumed for the valence bond contributions of C–O bonds in the two carbonate groups: the uncertainties in the C–O bond lengths dissuaded us from deriving the valence bond contributions on the basis of experimental bond-length values.

Bond-valence sums that are considerably different from 2.00 vu were obtained for O1, O3, O4 and O11 atoms, which have bond valence

sums close to 1 vu: these therefore are oxygen atoms which belong to hydroxyl anions of the brucite-type layer, and are each linked only to three Mg cations.

Of the five independent oxygen atoms of the carbonate groups, two (OC1 and OC5) are linked to four Pb atoms and have bond valence sums slightly higher than 2 vu, whereas the other three (OC2, OC3, OC4) are linked to only two Pb atoms and present valence bond sums that are lower than 2 vu. These three atoms have relatively short distances to the oxygen atom of the water molecule (OW20–OC2 = 2.77 Å, OW20–OC3 = 2.71 Å, OW20–OC4 = 2.82 Å), which is indicative of hydrogen bonding. Six hydrogen bond schemes are possible by assuming reasonable OC···OW20···OC angles; they are illustrated in Fig. 6. Therefore, each oxygen atom is engaged in hydrogen bonding with frequency 2/3; by assuming, on the basis of the average distance of the three atoms from the water molecule (2.77 Å) a hydrogen bond strength of 0.20 (Ferraris and Ivaldi, 1988), each of them receives a contribution of 0.13 vu, which is sufficient to produce a satisfactory bond-valence sum.

## Modularity in molybdophyllite

The crystal-chemical formula indicated by the results of the structural study is $Pb_8Mg_9[Si_{10}O_{28}(OH)_8|O_2|(CO_3)_3]\cdot H_2O$. As illustrated in Figs 3–5, the structure is built up from the following structural layers: a brucite-type layer, a ($Si_5O_{14}$) tetrahedral sheet, a layer of ($OPb_4$) tetrahedral groups and a layer of carbonate groups and water molecules. The brucite-type layer is sandwiched between two tetrahedral sheets to build a three-layer mica-like TOT (tetrahedral–octahedral–tetrahedral) packet. A difference from the true mica packets is that the tetrahedral sheets are incomplete, due to the regular removal of one in every six tetrahedra, as illustrated in Fig. 3. Britvinite is only remotely related to the heterophyllosilicates (e.g. Ferraris and Gula, 2005), in which a row of Ti–O polyhedra periodically substitutes for a row of disilicate tetrahedra in the tetrahedral T sheet.

The ($OPb_4$) groups in britvinite are only anchored to the free basal vertices of the tetrahedral sheets, thus giving rise to a complex five-layer module with a composition $\{Mg_9[Si_{10}O_{28}(OH)_8](OPb_4)_2\}$. This module has been reported in the crystal structure of type britvinite by Chukanov *et al*. (2008) and Yakubovich *et al*. (2008), and in the results of this study. If the parameters of the reduced cell of britvinite ($a$ = 9.337, $b$ = 9.359, $c$ = 18.929 Å, α = 76.01, β = 85.73, γ = 60.10º, and space group $P\bar{1}$) are transformed using the matrix [1 1 0 / −1 1 0 / 0 −1 1] we obtain the cell parameters $a$ = 16.183, $b$ = 9.3362, $c$ = 18.980 Å, α = 94.64, β = 104.16, γ = 89.84º, and space group $C\bar{1}$, which point to close metrical relationships with molybdophyllite. According to the definition of Makovicky (1997) the minerals molybdophyllite and britvinite belong to a *merotypic series*: in it a structural module is constantly present in the various members of the series and an inter-module layer, which varies both in thickness and in composition, characterizes each distinct member. Various merotypic series have been described in complex sulfides, oxides, hydroxides, and in oxysalts, especially silicates (Ferraris *et al.*, 2008). In the series described in this study the

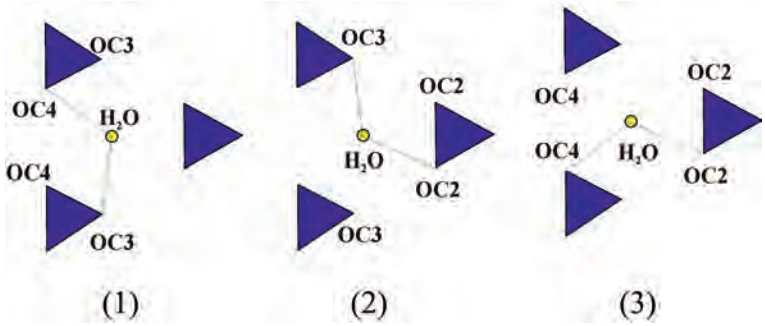


FIG. 6. The three possible schemes for hydrogen bonding in molybdophyllite: (1) OC4···HOH···OC3 and its symmetry-equivalent scheme (angle 119.2º); (2) OC3···HOH···OC2 and its symmetry-equivalent scheme (angle 121.4º); (3) OC4···HOH···OC2 and its symmetry-equivalent scheme (angle 119.4º).

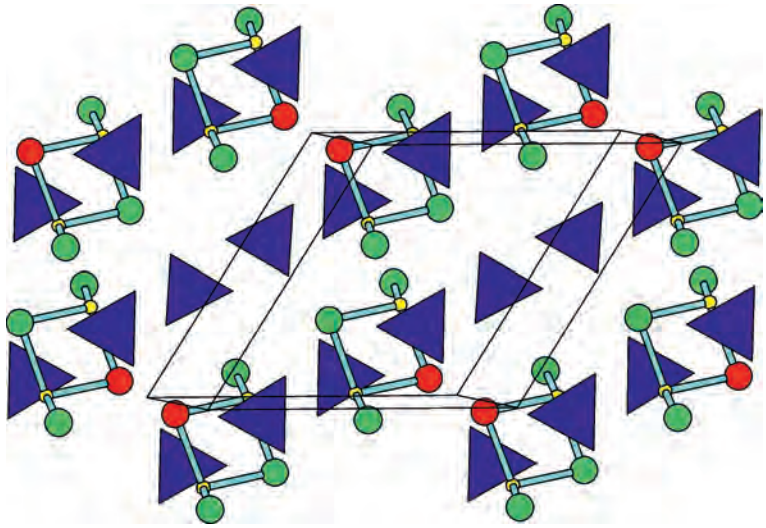

FIG. 7. The structure of the module $\{[(OH)_3OPb_7][(BO_3)_3|(CO_3)_3]\}^{6-}$ in britvinite, perpendicular to (001). The oxygen atoms in the $[(OH)_3OPb_7]$ groups are shown as small yellow circles, and the lead atoms are large circles with green and red colours indicating full or half occupancies. The $CO_3$/$BO_3$ groups are represented by blue triangles.

common module is $\{Mg_9[Si_{10}O_{28}(OH)_8][OPb_4]_2\}^{6+}$ and the second alternating module, which is different in the two members of the series, is $[(CO_3)_3{\cdot}H_2O]^{6-}$ in molybdophyllite and $\{[(OH)_3OPb_7][(BO_3)_3|(CO_3)_3]\}^{6-}$ in britvinite. This last module is illustrated in Fig. 7 as seen down the direction normal to the *ab* plane.

The $\{[(OH)_3OPb_7][(BO_3)_3|(CO_3)_3]\}^{6-}$ module in britvinite has two centrosymmetrically related sheets of carbonate/borate groups with a layer of composition $[(OH)_3OPb_7]$ in between. The carbonate/borate sheets are closely similar to those in molybdophyllite, which explains the substitution features of the two second modules in the merotypic series. So far only two members are known in this series, but additional natural or synthetic members cannot be excluded. Moreover, it is possible that domains with the structure types of molybdophyllite and britvinite coexist in one crystal, which could be one of the causes of the structural disorder in britvinite and would lead to non-stoichiometric formulae. The ideal formula of molybdophyllite, $Pb_8Mg_9[Si_{10}O_{28}(OH)_8|O_2|(CO_3)_3]{\cdot}H_2O$, derived from our crystal-structure determination of sample 221135, has a Pb:Mg:Si ratio of 8:9:10. This ratio is in good agreement with those reported by Charalampides (1994) (representative formula $Pb_{7.9}(Mg_{8.8}Mn_{0.1}Al_{0.2})Si_{9.0}O_{24}(OH)_{21.8}$) and Flink (1901) (~1:1:1). It also agrees fairly well with the ratios derived from our EPMA data (Table 1), recalculated assuming a total of 27 (Si + Al + Mg + Mn + Na + Pb) cations and assuming that the $H_2O$ and $CO_2$ contents determined for specimen 020154 are also valid for specimen 19419:

Specimen 020154: $Pb_{8.21}K_{0.17}(Mg_{8.51}Mn_{0.12}Na_{0.23})_{\Sigma 8.86}[(Si_{9.79}Al_{0.13})_{\Sigma 9.92}O_{28}(OH)_8|O_2|(CO_3)_{2.14}(OH)_{1.35}]$

Specimen 19419: $Pb_{8.04}K_{0.10}(Mg_{8.23}Mn_{0.08}Na_{0.76})_{\Sigma 9.07}[(Si_{9.65}Al_{0.23})_{\Sigma 9.88}O_{28}(OH)_8|O_2|(CO_3)_{2.10}(OH)_{0.64}]{\cdot}0.28H_2O$

The density calculated on the basis of XRD data for the ideal crystal-chemical formula of molybdophyllite, 4.652 g $cm^{-3}$, is somewhat lower than the density measured by Flink (1901) of 4.717 g $cm^{-3}$.

The OD character and related polytypic features of molybdophyllite have only been outlined in the foregoing discussion; they are more extensively discussed in the Appendix.

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_exptl_crystal_size_max           ?
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_exptl_crystal_density_method     'not measured'
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_exptl_special_details
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_diffrn_radiation_monochromator   graphite
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_diffrn_reflns_number             4215
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_diffrn_reflns_limit_k_min        -13
_diffrn_reflns_limit_k_max        9
_diffrn_reflns_limit_l_min        -17
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_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        ?
_computing_cell_refinement        ?
_computing_data_reduction         ?
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_refine_special_details
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
```

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_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      SHELXL
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_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_abs_structure_details
 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack    0.04(4)
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_refine_ls_goodness_of_fit_ref    1.063
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Pb2 Pb 0.26646(11) 1.24370(17) 0.34477(15) 0.0262(5) Uani 1 1 d . . .
Pb4 Pb 0.64230(11) 0.41228(17) 0.34460(15) 0.0259(5) Uani 1 1 d . . .
Pb3 Pb 0.32704(16) 0.6395(3) 0.44455(16) 0.0400(6) Uani 1 1 d . . .
Si1 Si 0.6340(8) 0.9913(13) 0.1983(11) 0.025(3) Uani 1 1 d . . .
Si2 Si 0.4646(9) 1.1388(15) 0.1896(11) 0.026(3) Uani 1 1 d . . .
Si3 Si 0.2997(8) 0.6405(12) 0.1894(9) 0.019(2) Uani 1 1 d . . .
Si4 Si 0.3087(9) 0.9608(13) 0.1978(11) 0.023(3) Uani 1 1 d . . .
Si5 Si 0.4568(8) 1.4635(12) 0.1973(11) 0.023(3) Uani 1 1 d . . .
Mg1 Mg 0.5000 1.308(2) 0.0000 0.019(4) Uiso 1 2 d S . .
Mg2 Mg 0.6645(9) 0.8049(16) 0.0057(13) 0.019(3) Uiso 1 1 d . . .
Mg3 Mg 0.5000 0.972(2) 0.0000 0.024(4) Uiso 1 2 d S . .
Mg4 Mg 0.1670(10) 0.9738(17) 0.0091(13) 0.022(3) Uiso 1 1 d . . .
Mg5 Mg 0.6663(9) 1.1409(18) -0.0034(12) 0.021(3) Uiso 1 1 d . . .
Mg6 Mg 0.5000 0.639(2) 0.0000 0.019(4) Uiso 1 2 d S . .
OW O 0.5000 1.140(5) 0.5000 0.020(8) Uiso 1 2 d S . .
O1 O 0.454(2) 0.811(3) 0.073(3) 0.023(6) Uiso 1 1 d . . .
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O5 O 0.391(2) 1.050(4) 0.239(3) 0.031(7) Uiso 1 1 d . . .
O6 O 0.552(2) 1.075(4) 0.237(3) 0.031(8) Uiso 1 1 d . . .
O7 O 0.621(2) 0.980(3) 0.080(3) 0.025(6) Uiso 1 1 d . . .
O8 O 0.461(2) 1.306(3) 0.231(3) 0.025(7) Uiso 1 1 d . . .
O9 O 0.4559(19) 1.137(4) 0.078(3) 0.025(6) Uiso 1 1 d . . .
O10 O 0.4494(19) 1.469(3) 0.078(3) 0.022(6) Uiso 1 1 d . . .
O11 O 0.6212(18) 0.642(3) 0.072(2) 0.021(6) Uiso 1 1 d . . .
O12 O 0.296(3) 0.971(5) 0.081(4) 0.043(10) Uiso 1 1 d . . .
O13 O 0.323(2) 0.794(4) 0.232(3) 0.033(8) Uiso 1 1 d . . .
O14 O 0.6543(19) 0.642(4) 0.393(3) 0.023(6) Uiso 1 1 d . . .
O15 O 0.714(2) 1.092(3) 0.230(3) 0.024(6) Uiso 1 1 d . . .
O16 O 0.240(2) 1.023(4) 0.257(3) 0.026(7) Uiso 1 1 d . . .
O17 O 0.372(2) 0.528(4) 0.230(3) 0.031(7) Uiso 1 1 d . . .
O18 O 0.532(2) 1.542(3) 0.254(3) 0.023(6) Uiso 1 1 d . . .
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Pb4 0.0327(8) 0.0215(8) 0.0223(12) -0.0032(6) -0.0006(7) 0.0013(6)
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_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Pb1 O6 3.30(4) . ?
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Pb2 Si1 3.622(15) 3_455 ?
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Pb2 O15 3.69(3) 3_455 ?
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Pb4 OC2 2.64(5) 3 ?
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Pb4 Si5 3.462(14) 1_545 ?
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O1 O11 2.74(5) 2_655 ?
O1 O7 2.81(5) 2_655 ?
O1 O12 2.99(5) . ?
O1 O9 3.05(5) . ?
O1 O7 3.12(5) . ?
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O3 O9 3.16(4) 3_545 ?
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O4 O9 2.84(5) . ?
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O13 O17 2.62(5) . ?
O13 Mg2 3.37(5) 2_655 ?
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O14 Pb3 2.27(4) 2_656 ?
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O14 O16 2.74(5) 3_545 ?
O14 O18 2.77(5) 1_545 ?
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O14 OC5 3.07(8) 3 ?
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Pb1 O18 O11 99.9(12) 1_565 1_565 ?
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Mg1 O18 Mg4 42.8(4) . 3 ?
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Pb4 O18 Mg4 138.8(11) 1_565 4 ?
Pb1 O18 Mg4 121.0(11) 1_565 4 ?
O8 O18 Mg4 60.8(12) . 4 ?
O17 O18 Mg4 45.7(12) 1_565 4 ?
O10 O18 Mg4 15.5(7) . 4 ?
O14 O18 Mg4 167.2(13) 1_565 4 ?
O11 O18 Mg4 75.3(9) 1_565 4 ?
Mg6 O18 Mg4 42.1(5) 1_565 4 ?
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Pb1 O19 Pb2 100.7(16) . 3_545 ?
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Pb1 O19 O15 137.5(18) . . ?
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Pb2 O19 O6 146(2) 3_545 . ?
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O6 O19 O7 62.3(15) . . ?
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O6 O19 O14 129.7(19) . . ?
O7 O19 O14 160(2) . . ?
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O6 O19 Mg2 96.8(17) . . ?
O7 O19 Mg2 35.7(9) . . ?
O14 O19 Mg2 126.2(15) . . ?
O11 O19 Mg2 32.7(7) . . ?

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O6 O19 Mg6 92.0(16) . . ?
O7 O19 Mg6 57.9(11) . . ?
O14 O19 Mg6 102.5(13) . . ?
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O14 O19 Mg5 167.5(15) . . ?
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Mg3 O19 Mg5 41.4(5) . . ?
Mg6 O19 Mg5 73.1(8) . . ?
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OC2 OC1 Pb1 105.8(13) 4_556 2_656 ?
OC2 OC1 Pb1 125.2(15) 3 2_656 ?
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OC2 OC1 Pb1 105.8(13) 3 . ?
Pb1 OC1 Pb1 121.9(19) 2_656 . ?
C1 OC1 Pb3 92.8(10) 3 . ?
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OC2 OC1 Pb3 120.8(19) 3 . ?
Pb1 OC1 Pb3 82.9(5) 2_656 . ?
Pb1 OC1 Pb3 94.4(6) . . ?
C1 OC1 Pb3 92.8(10) 3 2_656 ?
OC2 OC1 Pb3 120.8(19) 4_556 2_656 ?
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Pb1 OC1 Pb3 94.4(6) 2_656 2_656 ?
Pb1 OC1 Pb3 82.9(5) . 2_656 ?
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Pb1 OC1 O14 44.9(7) . . ?
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Pb3 OC1 O14 44.9(7) 2_656 . ?
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OC4 OC1 O14 115.8(17) 2_656 . ?
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OC2 OC1 O14 68.1(13) 4_556 2_656 ?
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OC4 OC1 O14 58.5(12) 2_656 2_656 ?
O14 OC1 O14 174(2) . 2_656 ?
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Pb3 OC4 Pb1 77.9(11) 2_656 2_656 ?
OW OC4 Pb1 81.6(11) . 2_656 ?
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