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# Niksergievite, $[Ba_{1.33}Ca_{0.67}Al(CO_3)(OH)_4][Al_2(AlSi_3O_{10})(OH)_2]\cdot nH_2O$, a new phyllosilicate related to the surite-ferrisurite series

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## ABSTRACT

Niksergievite, $[Ba_{1.33}Ca_{0.67}Al(CO_3)(OH)_4][Al_2(AlSi_3O_{10})(OH)_2]\cdot nH_2O$, is a new phyllosilicate closely related to the surite-ferrisurite series. It was found at the –400 m level of the Tekeli Pb-Zn mine, southeast Kazakhstan (44° N, 78° E). The mineral occurs as curved plates 3–5 mm in size forming rosette-like aggregates up to 5 cm across. Associated minerals include calcite, quartz, dolomite, celsian, sphalerite, pyrite, barite, and montmorillonite. Niksergievite is white with a light greenish tint and pearly luster on cleavage planes. The streak is white and the mineral is non-fluorescent. The Mohs hardness is $1–1^1/_2$. The cleavage is perfect (mica-like) on {001}. $D_m = 3.16$ g/cm$^3$ and $D_x = 3.21$ g/cm$^3$. The IR spectrum shows the following peaks (* shoulder): 3665*, 3640, 3405, 1630, 1454, 1105*, 1080*, 1035, 1020*, 980*, 960*, 920*, 876, 835*, 750*, 704, 625*, 560*, 535, 474, and 417 cm$^{-1}$. Optically, the mineral is colorless, non-pleochroic, biaxial (–), $2V = 0–10°$, $\alpha = 1.580(2)$, $\beta = 1.625(2)$, $\gamma = 1.625(2)$, and $X \sim c$. The chemical composition (electron microprobe, $CO_2$ and $H_2O$ by TGA) is $K_2O$ 0.1, CaO 5.1, BaO 27.1, MgO 0.4, FeO 0.2, $Al_2O_3$ 24.8, $SiO_2$ 28.7, $CO_2$ 6.1, and $H_2O$ 8.3, with a total of 100.8 wt%. The empirical formula based on $(Si + Al + Mg + Fe) = 7$ is $(Ba_{1.27}Ca_{0.65}K_{0.02})_{1.92}(Al_{3.49}Si_{3.42}Mg_{0.07}Fe^{2+}_{0.02})_{7.00}O_{10.00}(CO_3)_{0.99}(OH)_{6.20}\cdot 0.20H_2O$. The simplified formula is $(Ba,Ca)_2(Al,Si)_7O_{10}(CO_3)(OH)_6\cdot nH_2O$ and the proposed structural formula is $[Ba_{1.33}Ca_{0.67}Al(CO_3)(OH)_4][Al_2(AlSi_3O_{10})(OH)_2]\cdot nH_2O$. The mineral is monoclinic, $C2/c$, $C2$, or $Cm$, $a$ 5.176(3), $b$ 8.989(3), $c$ 16.166(5) Å, $\beta$ 96.44(6)°, $V$ 747.4(9) Å$^3$, $Z = 2$. The strongest reflections in the X-ray powder diffraction pattern are as follows [$d$ in Å, ($I$) ($hkl$)]: 16.1(40)(001), 4.49(90)(020), 3.68(60)($014,\bar{1}13$), 2.585(100)($130,\bar{2}01, \bar{1}31$), 2.230(90)($\bar{1}34,220$), 2.069(80)(043), 1.692(60)($\bar{3}11,\bar{1}51,240$). It is named in honor of Prof. Nikolai Grigorievich Sergiev (1901–1960) for his contributions to the geology of Kazakhstan.

## INTRODUCTION

Surite, $Pb(Pb,Ca)(CO_3)_2[(Al,Mg,Fe)_2(Si,Al)_4O_{10}(OH)_2]$, and ferrisurite, $(Pb,Ca)_{2-3}(CO_3)_{1.5-2}(OH,F)_{0.5}[(Fe,Al)_2Si_4O_{10}(OH)_2]\cdot nH_2O$, are rare and insufficiently studied 16 Å phyllosilicates. Since first described as new minerals (Hayase et al. 1978; Kampf et al. 1992) little has been added to our understanding of their chemistry and crystal structure. Studies of hydrothermally synthesized surite-like compounds were carried out by Tsutsumi et al. (1993) and Uehara et al. (1997a); Uehara et al. (1997b) later obtained new data on natural surite. Although all of these authors came to the conclusion that surite and related minerals are intercalates of lead carbonate in a smectite host, the detailed nature of the $Pb\text{-}CO_3$-intercalant remains uncertain. Note that only $Pb^{2+}$-members are known in this family, although other large-radius cations, i.e., $Sr^{2+}$ or $Ba^{2+}$, might substitute for $Pb^{2+}$ in the interlayer. In the present paper we introduce a new barium-bearing mineral closely related to surite. It is named niksergievite in honor of Prof. Nikolai Grigorievich Sergiev (1901–1960), a Soviet geologist, for his contributions to the geology of Kazakhstan. Both the mineral and its name have been approved by the Commission on New Minerals and Mineral Names of the International Mineralogical Association. The type material is deposited in the Geological Museum of the Satpaev Institute of Geological Sciences, Almaty, Kazakhstan.

## APPEARANCE AND PHYSICAL PROPERTIES

The Tekeli Pb-Zn mine (now abandoned) is located in southeast Kazakhstan, 44° N, 78° E, near the village of Tekeli. Its geology has been extensively studied since the early forties (Weitz 1945; Zhukov and Krikunova 1964; Patalakha 1975, 1983). This stratiform deposit is confined to the Tekeli mid-Ordovician suite of dolomitized, calcareous, and siliceous coal-clay schists. The concordant lenticular ore body is 400–850 m along strike and up to 1200 m down dip and consists of intensively metamorphosed dolomites and limestones enriched in galena, sphalerite,

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and pyrite, with minor jamesonite, boulangerite, tetrahedrite, bournonite, pyrrhotite, chalcopyrite, and marcasite.

Niksergievite was found at the –400 m level of the mine in cm-sized quartz-calcite veinlets cross-cutting siliceous dolomitized limestones. Associated minerals include dolomite, celsian, barite, sphalerite, galena, pyrite, and montmorillonite. Niksergievite occurs as curved plates up to 3 mm forming rosette-like aggregates up to 5 cm across. The platelets are translucent, white with a greenish tint (resembling greenish talc), and possess pearly luster on the cleavage plane. The streak is white and the mineral is non-fluorescent with perfect cleavage (mica-like) on {001}. The hardness is 1–1$^1/_2$ and the mineral is fairly brittle. The density measured by sink-float in heavy liquids ($CH_2I_2$-$CHBr_3$) is 3.16(3) g/cm$^3$; calculated using unit cell parameters it is –3.21 g/cm$^3$; and from Gladstone-Dale relationship (Mandarino 1979) it is –3.05 g/cm$^3$. In immersion liquids the mineral is colorless, non-pleochroic, and biaxial (–). The observed $2V$ varies in the range 0–10° due to plate deformation. Optical constants measured using a spindle stage are: $\alpha = 1.580(2)$, $\beta = 1.625(2)$, $\gamma = 1.625(2)$ for $\lambda = 589$ nm. The orientation is $X \sim \mathbf{c}$.

## X-RAY AND ELECTRON DIFFRACTION

Several attempts to collect data were undertaken using micro fragments of niksergievite plates and a Bruker PLATFORM CCD diffractometer with Mo$K\alpha$ radiation as well as a synchrotron radiation source (Argonne National Laboratory). No good quality single crystals were found, probably due to severe curvature of the crystal plates. Electron diffraction studies were performed with JEM-100CX and EMR-100M transmission electron microscopes. Selected-area electron diffraction patterns obtained from {001} sections (Fig. 1) and oblique textures (Figs. 2, 3) of niksergievite samples revealed monoclinic symmetry, space group $C2/c$, $C2$, or $Cm$, $a = 5.17$, $b = 8.97$, $c = 16.05$ Å, $\beta = 96.2°$. An X-ray powder diffraction pattern (Table 1) was obtained with a Debye-Scherrer 114.6 mm camera using Mn-filtered Fe$K\alpha$ radiation at 30 kV and 40 mA. The exact position of the 16.1 Å reflection was measured with a DRON-2.0 powder diffractometer using Cu$K\alpha$ radiation at 35 kV and 20 mA. Cell parameters refined from powder data are: $a = 5.176(3)$, $b = 8.989(3)$, and $c = 16.166(5)$ Å, $\beta = 96.44(6)°$, $V = 747.4(9)$ Å$^3$, and $Z = 2$. Note that platelets of niksergievite are sometimes coated with a minute film of a swelling montmorillonite-like mineral. In these cases the latter phase is revealed on patterns of textured samples as a broad 15 Å shoulder on the 16.1 Å reflection. After one-day saturation in ethylene glycol this shoulder disappears from the pattern, probably due to interlayer swelling and a corresponding $d$-shift to 16 Å.

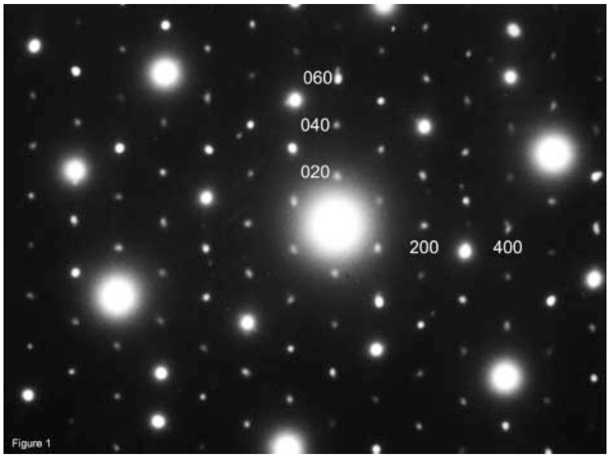

**FIGURE 1.** Selected area electron diffraction pattern of niksergievite, {001} plane.

## INFRARED ABSORPTION SPECTRUM

An infrared spectrum of the mineral was obtained from a powdered sample embedded in a KBr-pellet using a Specord-85 scanning IR-spectrophotometer. As it can be seen from Figure 4, the spectrum of niksergievite is similar to those of surite and ferrisurite (Hayase et al. 1978; Kampf et al. 1992), with a noticeable difference compared to ferrisurite in the principal hydroxyl-stretching region (3800–3000 cm$^{-1}$). Sharp absorp-

**TABLE 1.** Powder X-ray diffraction data for niksergievite*

| $I_{obs}$ | $d_{obs}$ (Å) | $d_{calc}$ (Å) | $h$ | $k$ | $l$ | $I_{obs}$ | $d_{obs}$ (Å) | $d_{calc}$ (Å) | $h$ | $k$ | $l$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 16.1† | 16.064 | 0 | 0 | 1 | 9 | 2.230{ | 2.234 | 1 | 3 | $\bar{4}$ |
| 1 | 7.99 | 8.032 | 0 | 0 | 2 | | | 2.232 | 2 | 2 | 0 |
| 3 | 5.35 | 5.355 | 0 | 0 | 3 | 3 | 2.166 | 2.164 | 0 | 4 | 2 |
| 9 | 4.49 | 4.495 | 0 | 2 | 0 | 8 | 2.069 | 2.072 | 0 | 4 | 3 |
| 0.5 | 4.21 | 4.197 | 1 | 1 | 1 | 0.5 | 2.011 | 2.008 | 0 | 0 | 8 |
| 1 | 4.01 | 4.016 | 0 | 0 | 4 | 4 | 1.963 | 1.965 | 1 | 1 | 7 |
| 6 | 3.68 | 3.750 | 1 | 1 | $\underline{2}$ | | | 1.963 | 1 | 3 | 5 |
| | | 3.606 | 1 | 1 | 3 | | | 1.961 | 0 | 4 | $\underline{4}$ |
| 4 | 3.21 | 3.213 | 0 | 0 | 5 | 1 | 1.915 | 1.916 | 1 | 3 | 6 |
| 5 | 2.845 | 2.851 | 1 | 1 | 4 | 2 | 1.766 | 1.768 | 1 | 1 | $\underline{8}$ |
| 3 | 2.679 | 2.677 | 0 | 0 | 6 | | | 1.768 | 1 | 3 | $\underline{7}$ |
| 10 | 2.585 | 2.589 | 1 | 3 | $\underline{0}$ | 6 | 1.692 | 1.694 | 3 | 1 | $\underline{1}$ |
| | | 2.585 | 2 | 0 | $\underline{1}$ | | | 1.694 | 1 | 5 | 1 |
| | | 2.579 | 1 | 3 | 1 | | | 1.692 | 2 | 4 | 0 |
| 0.5 | 2.375 | 2.373 | 2 | 0 | 2 | 1 | 1.637 | 1.638 | 2 | 2 | $\underline{6}$ |
| 1 | 2.296 | 2.295 | 0 | 0 | 7 | | | 1.635 | 1 | 5 | 3 |
| 1 | 2.244 | 2.247 | 0 | 4 | $\underline{0}$ | | | | | | |
| | | 2.241 | 2 | 2 | 1 | | | | | | |

* 114.6 mm Debye-Scherrer camera, Fe$K\alpha$ radiation, Mn-filter. Not corrected for shrinkage. Calculated $d$ values generated using cell parameters refined from powder data: $a = 5.176(3)$, $b = 8.989(3)$, $c = 16.166(5)$ Å, $\beta = 96.44(6)°$, $V = 747.4(9)$ Å$^3$, $Z = 2$.

† This $d$ value measured using a powder diffractometer.

**TABLE 2.** Chemical composition of niksergievite (wt%)

| Analyses<br>wt% | 1 | 2 | 3 | 4 | 5 | 6 | 7 | Average |
|---|---|---|---|---|---|---|---|---|
| $K_2O$ | 0.1 | 0.1 | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 | 0.1 |
| CaO | 6.0 | 5.1 | 4.8 | 4.8 | 4.9 | 5.3 | 4.6 | 5.1 |
| BaO | 31.2 | 29.4 | 25.5 | 25.3 | 25.5 | 28.9 | 24.0 | 27.1 |
| MgO | 0.3 | 0.6 | 0.3 | 0.3 | 0.4 | 0.3 | 0.3 | 0.4 |
| FeO | 0.1 | 0.2 | 0.4 | 0.4 | 0.2 | 0.1 | 0.3 | 0.2 |
| $Al_2O_3$ | 23.2 | 24.0 | 25.0 | 26.1 | 25.4 | 24.5 | 25.8 | 24.8 |
| $SiO_2$ | 26.2 | 26.1 | 30.1 | 30.0 | 30.5 | 27.2 | 31.0 | 28.7 |
| $CO_2$ | 6.1 | 6.1 | 6.1 | 6.1 | 6.1 | 6.1 | 6.1 | 6.1 |
| $H_2O$ | 8.3 | 8.3 | 8.3 | 8.3 | 8.3 | 8.3 | 8.3 | 8.3 |
| Total | 101.5 | 99.9 | 100.7 | 101.4 | 101.4 | 100.9 | 100.5 | 100.8 |
| **Formula contents based on (Si + Al + Mg + Fe) = 7** | | | | | | | | |
| K | 0.02 | 0.02 | 0.03 | 0.01 | 0.01 | 0.03 | 0.01 | 0.02 |
| Ca | 0.83 | 0.69 | 0.60 | 0.58 | 0.60 | 0.70 | 0.56 | 0.65 |
| Ba | 1.58 | 1.45 | 1.16 | 1.13 | 1.14 | 1.40 | 1.06 | 1.27 |
| Mg | 0.06 | 0.11 | 0.05 | 0.05 | 0.07 | 0.06 | 0.05 | 0.07 |
| Fe | 0.01 | 0.02 | 0.04 | 0.04 | 0.02 | 0.01 | 0.03 | 0.02 |
| Al | 3.54 | 3.57 | 3.42 | 3.50 | 3.42 | 3.57 | 3.43 | 3.49 |
| Si | 3.39 | 3.30 | 3.49 | 3.41 | 3.49 | 3.36 | 3.49 | 3.42 |
| C | 1.08 | 1.05 | 0.97 | 0.95 | 0.95 | 1.03 | 0.94 | 0.99 |
| H | 7.17 | 6.99 | 6.42 | 6.30 | 6.33 | 6.85 | 6.24 | 6.60 |

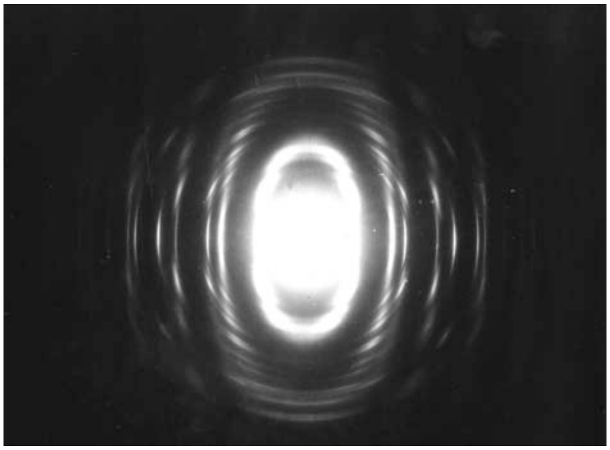

**FIGURE 2.** Oblique texture electron diffraction pattern of niksergievite. Substrate tilted 60° from normal.

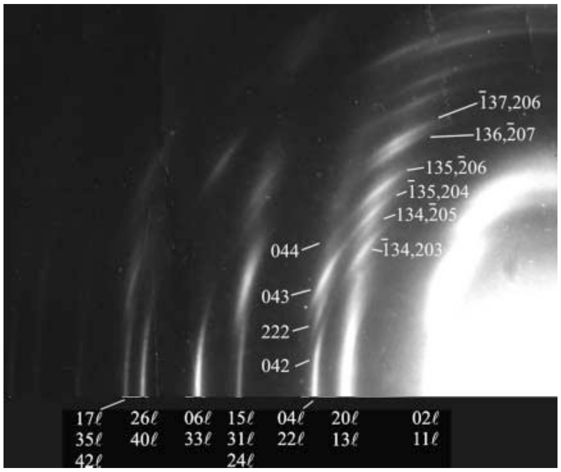

**FIGURE 3.** Interpretation of oblique texture pattern shown on Figure 2.

tion bands at 1454, 876, and 704 are attributed to $CO_3^{2-}$ and correlated with data for Pb-minerals. Note the measurable band shift (23–25 $cm^{-1}$) in the niksergievite spectrum to the long-wavelength region when compared to the surite and ferrisurite spectra. This can be explained by differences in atomic number of the intercalant cation, i.e., $Ba^{2+}$ in niksergievite compared to $Pb^{2+}$ in surite and ferrisurite.

## CHEMICAL COMPOSITION

Seven electron microprobe analyses were carried out with a Link AN-10000 EDS system using 20 kV acceleration voltage and 1 nA beam current, and microcline, grossular, barite, hematite, and spinel as standards (Table 2). No other elements with atomic number greater than 11 were detected, and no B, N, and F were detected with a windowless Link ISIS EDS detector. Minor Fe was recalculated as $Fe^{2+}$. $CO_2$ and $H_2O$ were determined by thermogravimetric analysis using a vacuum thermograph M-10 at the Institute of Chemical Physics, Russian Academy of Sciences. Two samples weighing ~10 mg each were analyzed by heating in a vacuum to 1000 °C at a constant rate of 4°/min, yielding 14.0 and 14.8 wt% total weight loss, respectively. Three stages of weight loss were observed (average of two samples): 1.55 wt% to 200 °C, 6.75 wt% between 500 and 700 °C, and 6.1 wt% up to 1000 °C. The IR spectrum of the sample annealed at 700 °C shows that it is dehydrated, but strong absorption bands at 1450, 880, and 700 $cm^{-1}$ corresponding to $CO_3^{2-}$ suggest that the third stage (6.1 wt%) represents loss of $CO_2$. The sample annealed at 1000 °C does not contain measurable amounts of water and carbonate, as shown by the IR spectrum.

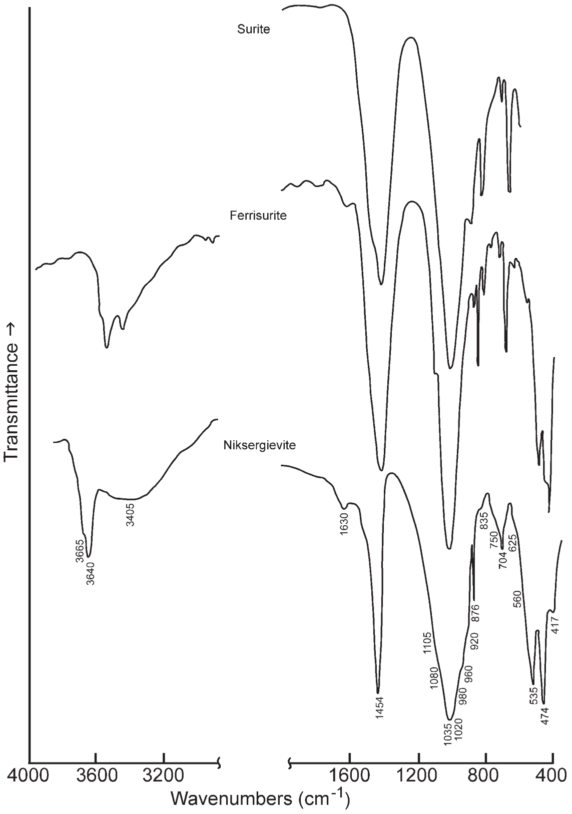

**FIGURE 4.** Infrared absorption spectra of niksergievite, surite (Hayase et al. 1978), and ferrisurite (Kampf et al. 1992).

Because of such uncertainty along with the absence of direct structural data, the chemical formula of niksergievite was derived on the basis of the measured density. Calculations based on (Si + Al + Mg + Fe) = 6 as required by a smectite-Ba-carbonate intercalate model yielded an unacceptable calculated density value of 2.75 $g/cm^3$. The best compatibility among $D_m$, $D_x$, and the density calculated using the Gladsone-Dale relationship was achieved for (Si + Al + Mg + Fe) = 7, resulting in the empirical formula (average of 7 analyses):

$$(Ba_{1.27}Ca_{0.65}K_{0.02})_{1.92}(Al_{3.49}Si_{3.42}Mg_{0.07}Fe^{2+}_{0.02})_{7.00}O_{10.00}(CO_3)_{0.99}(OH)_{6.20}\cdot 0.20H_2O$$

The simplified formula is $(Ba,Ca)_2(Al,Si)_7O_{10}(CO_3)(OH)_6\cdot nH_2O$. Niksergievite is relatively stable in acids—it does not dissolved in cool 10% HCl and $HClO_4$ after 1 hour.

**TABLE 3.** Comparative characteristics of niksergievite, surite, and ferrisurite

| | Niksergievite | Surite* | Surite† | Ferrisurite |
|---|---|---|---|---|
| Reference | This paper | Hayase et al. (1978) | Kampf et al. (1992) | Kampf et al. (1992) |
| Chemical formula | $[Ba_{1.33}Ca_{0.67}Al(CO_3)(OH)_4][Al_2(AlSi_3O_{10})(OH)_2]\cdot nH_2O$ | $Pb(Pb,Ca)_{1.17}(CO_3)_2(Al,Fe,Mg)_2(Si,Al)_4O_{10}(OH)_2$ | $(Pb,Ca)_{2-3}(CO_3)_{1.5-2}(OH,F)_{0.5-1}[(Al,Fe^{3+})_2(Si,Al)_4O_{10}(OH)_2]\cdot nH_2O$ | $(Pb,Ca)_{2-3}(CO_3)_{1.5-2}(OH,F)_{0.5-1}[(Fe^{3+},Al)_2(Si,Al)_4O_{10}(OH)_2]\cdot nH_2O$ |
| Space Group | $C2/m$, $C2$, or $Cm$ | $P2_1$ | $P2_1$ | $P2_1$ or $P2_1/m$ |
| $a$ (Å) | 5.176 | 5.22 | 5.241 | 5.241 |
| $b$ (Å) | 8.989 | 8.97 | 8.95 | 9.076 |
| $c$ (Å) | 16.166 | 16.3 | 16.20 | 16.23 |
| β | 96.44 | 96.1 | 90.0 | 90.03 |
| $V$ (Å$^3$) | 747.4 | 759 | 760 | 772 |
| $Z$ | 2 | 2 | 2 | 2 |
| Cleavage | Perfect {001} | Perfect {001} | | Perfect {001}‡ |
| $D_m$ (g/cm$^3$) | 3.16 | 4.0 | | 4.0 |
| $D_x$ (g/cm$^3$) | 3.21 | 3.91 | 3.97 | 3.89 |
| Optical Sign | ( − ) | | | ( + ) |
| 2$V$ | 0–10 (meas) | | | 76 (calc) |
| $a$ | 1.580 | 1.693 | | 1.757 |
| $b$ | 1.625 | | | 1.763 |
| $γ$ | 1.625 | 1.738 | | 1.773 |
| IR-absorption bands related to $CO_3^{2-}$ | 1454<br>876<br>704 | 1430<br>840<br>690 | | 1422<br>853<br>691 |

* Original data by Hayase et al. (1978).

† Cell parameters of surite recalculated by Kampf et al. (1992).

‡ Private communication by A.R. Kampf; in the original paper given as {010}.

## DISCUSSION

As can be seen from our data, niksergievite is a close relative of surite and ferrisurite (Table 3). Nevertheless, differences in lattice centering (the centered cell of niksergievite is contrary to the primitive one of surite-ferrisurite) and chemical composition (i.e., significant relative excess Al in the niksergievite composition) do not allow us to draw a direct analogy among these minerals. As a consequence, the niksergievite structure cannot be expressed as an intercalate of a basic Ba-Ca carbonate interlayer in smectite like the Pb-Ca-carbonate/smectite model accepted for surite (Hayase et al. 1978; Kampf et al. 1992; Uehara et al. 1997a, 1997b). The statistical analysis of formula amounts shows a strong positive correlation between Ca and Ba, with Ba = 2Ca, $R = 0.97$, and SD = 0.05. The correlation between (Ca + Ba) and (Al + Mg + Fe) is also positive, (Al + Mg + Fe) ~ 3 + 0.2(Ba + Ca), $R = 0.72$, and SD = 0.06. This leads to a possible interpretation of the niksergievite structure as an intercalate of a basic Ba-Ca-Al-carbonate layer (with stoichiometric atomic ratio Ba:Ca = 2) between 2:1 smectite-like layers. If this is correct the structural formula of the mineral may be represented by $[Ba_{1.33}Ca_{0.67}Al(CO_3)(OH)_4][Al_2(AlSi_3O_{10})(OH)_2]\cdot nH_2O$, with some deviations due to the substitution of Mg for Al in the silicate interlayer, as well as probable stacking errors. Thus the composition of the carbonate layer in niksergievite is different from the Al-free cerussite-like layer in surite and ferrisurite (Hayase et al. 1978; Kampf et al. 1992; Uehara et al. 1997a, 1997b). Dresserite, $Ba_2Al_4(CO_3)_4(OH)_8\cdot 3H_2O$ (Jambor et al. 1969), and its Ca-, Sr-, and Pb-relatives are the nearest known Al-bearing compounds but they have inverse cation stoichiometry ($M^{2+}/Al = 1/2$). The nature of the carbonate layer, $[Ba_{1.33}Ca_{0.67}Al(CO_3)(OH)_4]^+$, remains uncertain with no direct analogues among natural and synthetic compounds.

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