# Magnesiokoritnigite, $\mathrm{Mg}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$, from the Torrecillas mine, Iquique Province, Chile: the Mg-analogue of koritnigite 

A. R. Kampr ${ }^{1 *}$, B. P. $\mathrm{NaSh}^{2}$, M. Dini ${ }^{3}$ and A. A. Molina Donoso ${ }^{4}$<br>${ }^{1}$ Mineral Sciences Department, Natural History Museum of Los Angeles County, 900 Exposition Boulevard, Los Angeles, CA 90007, USA<br>${ }^{2}$ Department of Geology and Geophysics, University of Utah, Salt Lake City, Utah 84112, USA<br>${ }^{3}$ Pasaje San Agustin 4045, La Serena, Chile<br>${ }^{4}$ Los Algarrobos 2986, Iquique, Chile

[Received 25 August 2013; Accepted 20 October 2013; Associate Editor: S.J. Mills]

## ABSTRACT

The new mineral magnesiokoritnigite (IMA 2013-049), ideally $\mathrm{Mg}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$, was found at the Torrecillas mine, Salar Grande, Iquique Province, Chile, where it occurs as a secondary alteration phase in association with anhydrite, chudobaite, halite, lavendulan, quartz and scorodite. Crystals of magnesiokoritnigite are colourless to pale-pink, thin to thick laths up to 2 mm long. Laths are elongated on [001], flattened on $\{010\}$ and exhibit the forms $\{010\},\{110\},\{1 \overline{1} 0\},\{101\},\{031\}$ and $\{0 \overline{3} 1\}$. The crystals also occur in dense deep-pink intergrowths. Crystals are transparent with a vitreous lustre. The mineral has a white streak, Mohs hardness of $\sim 3$, brittle tenacity, conchoidal fracture and one perfect cleavage on $\{101\}$. The measured and calculated densities are $2.95(3)$ and $2.935 \mathrm{~g} \mathrm{~cm}^{-3}$, respectively. Optically, magnesiokoritnigite is biaxial ( + ) with $\alpha=1.579(1), \beta=1.586(1)$ and $\gamma=1.620(1)$ (measured in white light). The measured 2 V is $50(2)^{\circ}$ and the calculated 2 V is $50^{\circ}$. Dispersion is $\mathrm{r}<\mathrm{v}$, medium. The optical orientation is $Y \approx \mathbf{b} ; Z^{\wedge} \mathbf{c}=36^{\circ}$ in obtuse $\beta$ (note pseudomonoclinic symmetry). The mineral is non-pleochroic. The empirical formula, determined from electron-microprobe analyses, is $\left(\mathrm{Mg}_{0.94} \mathrm{Cu}_{0.03} \mathrm{Mn}_{0.02} \mathrm{Ca}_{0.01}\right)_{\Sigma 1.00} \mathrm{As}_{0.96} \mathrm{O}_{5} \mathrm{H}_{3.19}$. Magnesiokoritnigite is triclinic, $P \overline{1}$, with $a=$ $7.8702(7), b=15.8081(6), c=6.6389(14) \AA, \alpha=90.814(6), \beta=96.193(6), \gamma=90.094(7)^{\circ}, V=$ $821.06(19) \AA^{3}$ and $Z=8$. The eight strongest X-ray powder diffraction lines are $\left[d_{\text {obs }} \AA(I)(h k l)\right]$ : $7.96(100)(020), \quad 4.80(54)(101), \quad 3.791(85)(\overline{2} 10,210, \overline{1} \overline{3} 1, \overline{1} 31), \quad 3.242(56)(0 \overline{1} 2, \overline{2} \overline{2} 1,012)$, $3.157(92)(2 \overline{1} 1, \overline{2} 30,230), 3.021(61)(1 \overline{4} 1,141,2 \overline{2} 1,221), 2.798(41)(0 \overline{3} 2,032)$ and $1.908(43)($ multiple $)$. The structure, refined to $R_{1}=5.74 \%$ for $2360 F_{\mathrm{o}}>4 \sigma F$ reflections, shows magnesiokoritnigite to be isostructural with koritnigite and cobaltkoritnigite.

Keywords: magnesiokoritnigite, new mineral, crystal structure, koritnigite, Torrecillas mine, Chile.

## Introduction

The Torrecillas mine, in the northern Atacama Desert of Chile, is a small, long-inactive arsenic mine consisting of several small pits and a shaft on Torrecillas Hill, overlooking the Pacific

[^0]Ocean. The deposit was probably discovered and first mined during the guano mining boom of the early $19^{\text {th }}$ century. During that period, the Peruvian Government owned and claimed several guano fields along the Chilean coast in Iquique and Tarapacá provinces, including the prolific Huanillos, Chanabaya and Pabellón de Pica deposits, which are not far from the Torrecillas area (Mortimer et al., 1971). The guano mines

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under Peruvian administration, were exploited mainly by English companies; many Chinese coolies laboured in the mines and caves. Torrecillas mine was abandoned probably several years prior to 1950 (Pimentel, 1978).

Our mineralogical investigation of the Torrecillas deposit has shown it to host a remarkable secondary assemblage of rare chlorides, arsenates and arsenites. In addition to the new mineral magnesiokoritnigite, described here, the deposit has yielded the new atacamite-group mineral leverettite, $\mathrm{CoCu}_{3} \mathrm{Cl}_{2}(\mathrm{OH})_{6}$, (Kampf et al., 2013; IMA2013-011), the recently approved new alluaudite-group mineral canutite, $\mathrm{NaMn}_{3}\left[\mathrm{AsO}_{4}\right]_{2}\left[\mathrm{AsO}_{2}(\mathrm{OH})_{2}\right]$ (IMA2013-070), a new mineral of formula $\mathrm{Na}(\mathrm{As}, \mathrm{Sb})_{4}^{3+} \mathrm{O}_{6} \mathrm{Cl}$, currently awaiting approval (IMA2013-112), and several other potentially new secondary arsenates and arsenites, currently under study.

Keller et al. (1979) described the mineral koritnigite, $\mathrm{Zn}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$, from Tsumeb, Namibia, and just two years later Schmetzer et al. (1981) described its Co analogue cobaltkoritnigite, $\mathrm{Co}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$, from the Schwarzenberg District, Erzgebirge, Saxony, Germany. Herein, we describe the third member of the koritnigite group, magnesiokoritnigite, $\mathrm{Mg}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$, named as the Mg analogue,
from the Torrecillas mine, Iquique Province, Chile.

The new mineral and the name have been approved by the International Mineralogical Association (IMA2013-049). One holotype and two cotype specimens are deposited in the Natural History Museum of Los Angeles, Los Angeles, California, USA, under catalogue numbers 64057 (holotype), 64058 and 64059.

## Occurrence

The Torrecillas mine is located on Torrecillas Hill near Salar Grande, Iquique Province, Tarapacá Region, Chile (approximately $20^{\circ} 58^{\prime} 13^{\prime \prime} \mathrm{S}$ $\left.70^{\circ} 8^{\prime} 17^{\prime \prime} \mathrm{W}\right)$. The rare secondary chlorides, arsenates and arsenites have been found at three sites: an upper pit measuring $\sim 8 \mathrm{~m}$ long and 3 m deep, a lower pit $\sim 100 \mathrm{~m}$ from the upper pit and measuring $\sim 5 \mathrm{~m}$ long and 3 m deep, and a mine shaft adjacent to the lower pit and lower on the hill (Fig. 1). Magnesiokoritnigite was first found by one of the authors (AAMD) in early 2012 in the upper pit and this pit has produced the best crystals. Magnesiokoritnigite has also been found in boulders on the hill slope just below the mine shaft.

Four different rock units are exposed on the hill. Rocks of the Coastal Range Batholith


Fig. 1. View looking southwest down Torrecillas Hill towards the Pacific coast. The three principle sites at which unusual secondary minerals have been recovered are labelled. The Pan-American Road can be seen at the base of the hill.
(mainly gabbros) extend from the seashore to the Pan-American Road along the base of the hill. At the foot of the hill is a small area of contact metamorphic rocks in which garnet crystals occur in metamorphosed shales. Higher on the hill, rocks are predominantly andesites and porphyritic lavas of the Jurassic La Negra Formation. The Torrecillas deposit, which the Torrecillas mine exploits, consists of two main veins rich in secondary arsenic and copper minerals that intersect metamorphosed marine shales and lavas. These veins are genetically related to the above-mentioned andesites and porphyritic lavas of the La Negra Formation. More information on the geology and mineralogy of the area is provided by Gutiérrez (1975).

The new mineral is associated with anhydrite, chudobaite, halite, lavendulan, quartz, scorodite, the recently approved new alluaudite-group mineral canutite, $\mathrm{NaMn}_{3}\left[\mathrm{AsO}_{4}\right]_{2}\left[\mathrm{AsO}_{2}(\mathrm{OH})_{2}\right]$ (IMA2013-070), a new mineral of formula $\mathrm{Na}(\mathrm{As}, \mathrm{Sb})_{4}^{3+} \mathrm{O}_{6} \mathrm{Cl}$, currently awaiting approval (IMA2013-112), and at least two other potentially new arsenate minerals, currently under study. The recently approved new alluaudite-group mineral canutite, $\mathrm{NaMn}_{3}\left[\mathrm{AsO}_{4}\right]_{2}\left[\mathrm{AsO}_{2}(\mathrm{OH})_{2}\right]$, (IMA2013-070) has also been found in the upper pit on Torrecillas Hill, but not in direct association with magnesiokoritnigite. The assemblage is interpreted as having formed from the oxidation of native arsenic and other As-bearing primary phases, followed by later alteration by saline fluids derived from evaporating meteoric water under hyperarid conditions (cf. Cameron et al., 2007). The metallic elements thus far


Fig. 2. Pale-pink laths of magnesiokoritnigite. Field of view $=2 \mathrm{~mm}$.


Fig. 3. Colourless laths of magnesiokoritnigite with blue lavendulan. Field of view 2 mm .
identified in the secondary minerals of this deposit include $\mathrm{Na}, \mathrm{Mg}, \mathrm{Al}, \mathrm{Ca}, \mathrm{Mn}, \mathrm{Fe}, \mathrm{Co}$, $\mathrm{Cu}, \mathrm{Ni}$ and Sb .


FIG. 4. Crystal drawing of magnesiokoritnigite (clinographic projection in standard orientation).

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TABLE 1. Electron microprobe analyses of magnesiokoritnigite.

| Constituent | Wt. $\%$ | Range | SD | Norm. wt. $\%$ | Standard |
| :--- | ---: | :---: | :---: | ---: | :---: |
| MgO | 19.88 | $19.27-20.54$ | 0.42 | 20.86 | Diopside |
| CaO | 0.36 | $0.12-0.72$ | 0.18 | 0.38 | Diopside |
| MnO | 0.90 | $0.40-1.46$ | 0.37 | 0.94 | Rhodonite |
| CuO | 1.03 | $0.74-1.24$ | 0.21 | 1.08 | Cu metal |
| $\mathrm{As}_{2} \mathrm{O}_{5}$ | 58.02 | $57.40-58.74$ | 0.48 | 60.88 | Syn. GaAs |
| $\mathrm{H}_{2} \mathrm{O}$ | 15.11 |  |  | 15.86 |  |
| Total | 95.30 |  |  | 100.00 |  |

Table 2. Powder X-ray data for magnesiokoritnigite.

| $I_{\text {obs }}$ | $d_{\text {obs }}$ | $d_{\text {calc }}$ | $I_{\text {calc. }}$ | $h k l$ | $I_{\text {obs }}$ | $d_{\text {obs }}$ | $d_{\text {calc. }}$ | $I_{\text {calc. }}$ | $h k l$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 100 | 7.96 | 7.9032 | 100 | 020 | 15 | 2.332 | 2.3404 | 2 | $\overline{3} 30$ |
| 17 | 7.05 | 7.0211 | 6 | 110 |  |  | 2.3344 | 2 | 330 |
|  |  | 7.0033 | 6 | 110 |  |  | 2.3212 | 3 | $1 \overline{6} 1$ |
| 5 | 5.57 | 5.5692 | 2 | $\overline{1} 20$ | 13 | 2.173 | 2.2969 | 3 | 161 |
|  |  | 5.5514 | 2 | 120 |  |  | 2.1799 | 3 | 140 |
| 17 | 5.07 | 5.0707 | 8 | $\overline{1} 11$ |  |  | 2.1735 | 3 | 340 |
|  |  | 5.0415 | 9 | 111 |  |  | 2.0867 | 2 | $\overline{1} 71$ |
| 54 | 4.803.972 | 4.7960 | 28 | 101 | 19 | 2.050 | 2.0725 | 2 | 171 |
| 12 |  | 3.9516 | 15 | 040 |  |  | 2.0615 | 2 | $2 \overline{4} 2$ |
|  | 3.791 | 3.8004 | 13 | $\overline{2} 10$ |  |  | 2.0471 | 2 | $1 \overline{1} 3$ |
| 85 |  | 3.7947 | 13 | 210 |  |  | 2.0395 | 4 | 113 |
|  |  | 3.7673 | 13 | $\overline{1} \overline{3} 1$ | 43 |  | 1.9561 | 2 | 400 |
|  |  | 3.7316 | 13 | $\overline{1} 31$ |  | 1.908 | 1.9285 | 2 | $1 \overline{3} 3$ |
| 14 | 3.505 | 3.5318 | 4 | $\overline{1} 40$ |  |  | 1.9197 | 3 | $1 \overline{4} 3$ |
|  |  | 3.5228 | $4$ | 140 |  |  | 1.9186 | 2 | $\overline{4} 11$ |
|  |  | 3.4551 | 2 | $\overline{2} 111$ |  |  | 1.9027 | 3 | $\overline{3} \overline{4} 2$ |
|  |  | 3.4479 | 2 | 211 |  |  | 1.8980 | 3 | 143 |
| 56 | 3.242 | 3.2395 | 17 | $0 \overline{1} 2$ | 26 | 1.773 | 1.8918 | 3 | $\overline{3} 42$ |
|  |  | 3.2342 | 3 | $\overline{2} 21$ |  |  | 1.7907 | 2 | $\overline{3} 52$ |
|  |  | 3.2208 | 24 | 012 |  |  | 1.7845 | 2 | $2 \overline{6} 2$ |
| 92 | 3.157 | 3.1578 | 2 | $2 \overline{1} 1$ |  |  | 1.7794 | 2 | $\overline{3} 52$ |
|  |  | 3.1458 | 21 | $\overline{2} 30$ |  |  | 1.7624 | 2 | 262 |
|  |  | 3.1362 | 19 | 230 |  |  | 1.6907 | 2 | $\overline{1} \overline{6} 3$ |
|  |  | 3.0961 | 2 | 112 | 37 | 1.658 | 1.6499 | 8 | 004 |
| 61 | 3.021 | 3.0686 | 10 | $1 \overline{4} 1$ | 16 | 1.5732 | 1.5725 | 2 | 380 |
|  |  | 3.0313 | 10 | 141 |  |  | 1.5567 | 2 | 510 |
|  |  | 2.9893 | 2 | $2 \overline{2} 1$ |  |  | 1.5341 | 2 | 520 |
|  |  | 2.9692 | 2 | 221 | 36 | 1.528 | 1.5331 | 2 | 173 |
|  |  | 2.9343 | 4 | 150 |  |  | 1.5160 | 2 | $\overline{4} 23$ |
|  |  | 2.9278 | 4 | 150 |  |  | 1.5113 | 2 | 173 |
| 41 | 2.798 | 2.8149 | 15 | $0 \overline{3} 2$ | 15 |  | 1.5068 | 2 | $1 \cdot \overline{10} \cdot 1$ |
|  |  | 2.7785 | 12 | 032 |  | 1.460 | 1.4957 | 2 | $1 \cdot 10 \cdot 1$ |
| 17 | 2.672 | 2.6682 | 10 | 202 |  |  | 1.4565 | 2 | 214 |
|  |  | 2.6344 | 3 | 060 |  |  | 1.4163 | 2 | $2 \overline{3} 4$ |
| 12 | 2.527 | 2.5203 | 5 | $\overline{3} 01$ | 30 | 1.403 | 1.4061 | 2 | 234 |
|  |  | 2.4627 | 5 | $\overline{2} 50$ |  |  | 1.4038 | 2 | $\overline{3} \overline{7} 3$ |
| 14 | 2.465 | 2.4550 | 5 | 250 |  |  | 1.3851 | 2 | $\overline{5} \overline{4} 2$ |

$\begin{array}{lllll}10 & 2.402 & 2.3980 & 5 & 202\end{array}$

Only calculated lines with intensities of 2 or greater are shown. Numerous lower-intensity lines also contribute to the observed peaks.

## Physical and optical properties

Crystals of magnesiokoritnigite (Figs 2 and 3) are colourless to pale pink, thin to thick laths up to 2 mm long. The laths are elongated on [001], flattened on $\{010\}$ and exhibit the forms $\{010\}$, $\{110\},\{1 \overline{1} 0\},\{101\},\{031\}$ and $\{0 \overline{3} 1\}$ (Fig. 4). The crystals also occur in dense intergrowths that are deep pink in colour. Crystals are transparent with a vitreous lustre. The mineral has white streak, Mohs hardness of $\sim 3$, brittle tenacity, conchoidal fracture and one perfect cleavage on $\{101\}$. The density measured by flotation in Clerici solution is $2.95(3) \mathrm{g} \mathrm{cm}^{-3}$. The calculated densities are $2.935 \mathrm{~g} \mathrm{~cm}^{-3}$ based on the empirical formula and $2.948 \mathrm{~g} \mathrm{~cm}^{-3}$ based on the ideal formula.

Optically, magnesiokoritnigite is biaxial (+) with $\alpha=1.579(1), \beta=1.586(1)$ and $\gamma=1.620(1)$
(measured in white light). The 2 V measured directly using a spindle stage is $50(2)^{\circ}$ and the calculated 2 V is $50^{\circ}$. Dispersion is $r<v$, moderate. The optical orientation is $Y \approx \mathbf{b} ; Z^{\wedge}$ $\mathbf{c}=36^{\circ}$ in obtuse $\beta$ (note that the mineral exhibits pseudomonoclinic symmetry). The mineral is non-pleochroic.

The mineral is slowly soluble in concentrated HCl or $\mathrm{HNO}_{3}$ and very slowly soluble in concentrated $\mathrm{H}_{2} \mathrm{SO}_{4}$.

## Composition

Quantitative analyses (9) were performed at the University of Utah using a Cameca SX-50 electron microprobe (EMPA) with four wave-length-dispersive spectrometers. Analytical conditions were 15 kV accelerating voltage, 20

Table 3. Data collection and structure-refinement details for magnesiokoritnigite.
Diffractometer
X-ray radiation/power
Temperature
Structural formula
Space group
Unit-cell dimensions

$V$
$Z$
Density (for formula above)
Absorption coefficient
$F(000)$
Crystal size ( $\mu \mathrm{m})$
$\theta$ range
Index ranges
Reflections collected $/$ unique
Reflections with $F_{\mathrm{o}}>4 \sigma(F)$
Completeness to $\theta=25.03^{\circ}$
Max. and min. transmission
Refinement method
Parameters refined
GoF
Final $R$ indices $\left[F_{\mathrm{o}}>4 \sigma(F)\right]$
$R$ indices $($ all data)
Largest diff. peak/hole

Rigaku R-Axis Rapid II
$\operatorname{MoK} \alpha(\lambda=0.71075 \AA) / 50 \mathrm{kV}, 40 \mathrm{~mA}$
293(2) K
$\mathrm{Mg}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$
$P \overline{1}$
$a=7.8702(7) \AA$
$b=15.8081(6) \AA$
$c=6.6389(14) \AA$
$\alpha=90.814(6)^{\circ}$
$\beta=96.193(6)^{\circ}$
$\gamma=90.094(7)^{\circ}$
821.06(19) $\AA^{3}$

8
$2.948 \mathrm{~g} \mathrm{~cm}^{-3}$
$8.330 \mathrm{~mm}^{-1}$
704
$150 \times 80 \times 10$
3.09 to $25.03^{\circ}$
$-9 \leqslant h \leqslant 8,-18 \leqslant k \leqslant 18,-7 \leqslant l \leqslant 7$
$10865 / 2877 ; R_{\text {int }}=0.106$
2360
99.4\%
0.921 and 0.368

Full-matrix least-squares on $F^{2}$
253
1.059
$R_{1}=0.0574, w R_{2}=0.1218$
$R_{1}=0.0695, w R_{2}=0.1294$
$+1.127 /-1.237 \mathrm{e} \mathrm{A}^{-3}$

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Table 4. Atom coordinates and displacement parameters $\left(\AA^{2}\right)$ for magnesiokoritnigite.

|  | $x / a$ | $y / b$ | $z / c$ | $U_{\text {eq }}$ | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Mg 1 | 0.0964(4) | 0.06260(19) | 0.3760 (4) | 0.0195(6) | $0.0194(15)$ | 0.0231(17) | $0.0160(15)$ | $0.0025(11)$ | 0.0021(12) | $-0.0005(12)$ |
| Mg2 | 0.1403(3) | $0.45809(19)$ | 0.3656(4) | 0.0189(6) | $0.0144(15)$ | 0.0273(18) | $0.0157(15)$ | 0.0023(12) | $0.0049(11)$ | -0.0012(12) |
| Mg3 | 0.4050(4) | 0.5627(2) | 0.1291(4) | 0.0222(7) | $0.0190(16)$ | 0.0302(18) | $0.0178(16)$ | 0.0027(12) | $0.0033(12)$ | -0.0002(12) |
| Mg4 | 0.3591(4) | 0.95876(19) | 0.1303(4) | 0.0209(7) | $0.0177(15)$ | 0.0304(18) | $0.0148(15)$ | 0.0020 (12) | 0.0027(12) | $0.0009(12)$ |
| As1 | 0.08007(11) | 0.10410(6) | 0.88440(13) | 0.0213(3) | $0.0192(5)$ | $0.0265(6)$ | 0.0182(5) | 0.0022(4) | 0.0014(4) | -0.0007(4) |
| As2 | 0.17790(11) | 0.41844(6) | 0.86145(13) | 0.0216(3) | 0.0188(5) | 0.0262(6) | 0.0198(5) | $0.0015(4)$ | 0.0022(4) | -0.0005(4) |
| As3 | $0.42138(11)$ | 0.60426(6) | 0.62370 (13) | 0.0217(3) | 0.0197(5) | 0.0265(6) | 0.0189(5) | 0.0024(4) | 0.0019(4) | -0.0008(4) |
| As4 | $0.32086(11)$ | 0.91790(6) | $0.63236(13)$ | 0.0213(3) | 0.0185(5) | 0.0271(6) | 0.0183(5) | $0.0031(4)$ | 0.0024(4) | -0.0005(4) |
| O1 | 0.0573(8) | 0.0446(4) | 0.6732(8) | $0.0229(14)$ | 0.030 (4) | 0.026(4) | 0.013(3) | 0.000(2) | 0.005(3) | -0.002(3) |
| O2 | 0.1700(7) | $0.0535(4)$ | 0.0880(9) | $0.0230(14)$ | 0.021(3) | 0.025(4) | 0.022(3) | 0.006(3) | 0.000(3) | 0.007(3) |
| OW3 | 0.2159(8) | 0.1774(4) | 0.4400(9) | 0.0283(15) | 0.022 (3) | 0.031(4) | $0.032(4)$ | 0.004(3) | 0.002 (3) | -0.008(3) |
| O4 | 0.3313(7) | 0.5531(4) | 0.4153(9) | $0.0224(14)$ | 0.023(3) | 0.026(4) | 0.018(3) | -0.001(2) | 0.000 (3) | -0.003(3) |
| O5 | 0.0851(7) | 0.4748(4) | 0.6680(9) | $0.0237(14)$ | $0.021(3)$ | 0.029(4) | 0.020(3) | $0.005(3)$ | -0.001(3) | $0.005(3)$ |
| OH6 | 0.3737(8) | $0.3695(4)$ | 0.4260(9) | $0.0259(15)$ | $0.021(3)$ | 0.036(4) | 0.020(3) | 0.004(3) | $0.000(3)$ | -0.007(3) |
| OW7 | 0.0326 (8) | 0.3390 (4) | 0.3590 (10) | $0.0328(16)$ | $0.027(4)$ | 0.034(4) | $0.038(4)$ | $0.001(3)$ | $0.005(3)$ | -0.007(3) |
| O8 | 0.1962(7) | 0.4773(4) | 0.0757(9) | $0.0219(14)$ | 0.024(3) | 0.021(3) | 0.021(3) | 0.000(2) | 0.004(3) | -0.004(3) |
| OW9 | 0.2870(8) | 0.6774(4) | 0.0746(9) | $0.0267(15)$ | 0.020 (3) | 0.027(4) | 0.034(4) | -0.002(3) | 0.003(3) | 0.003(3) |
| O10 | 0.3041 (8) | $0.9769(4)$ | 0.4227(9) | $0.0248(14)$ | $0.025(3)$ | 0.030(4) | 0.020(3) | 0.007(3) | 0.002(3) | 0.000(3) |
| OW11 | 0.4673(8) | 0.8402(4) | 0.1327(10) | $0.0333(16)$ | 0.023(4) | 0.036(4) | 0.039(4) | 0.002(3) | -0.003(3) | 0.007(3) |
| OH12 | 0.1241 (7) | 0.8696(4) | 0.0633(9) | $0.0239(14)$ | $0.017(3)$ | $0.032(4)$ | 0.023(3) | 0.003(3) | 0.001(3) | 0.004(3) |
| O13 | $0.1895(8)$ | $0.1915(4)$ | 0.8410(9) | $0.0269(15)$ | 0.024(3) | 0.027(4) | 0.032(4) | 0.004(3) | $0.011(3)$ | 0.001(3) |
| OH14 | 0.0481(8) | $0.3337(4)$ | 0.8955(10) | $0.0284(15)$ | 0.020 (3) | 0.030(4) | 0.036(4) | $0.005(3)$ | 0.011(3) | -0.007(3) |
| O15 | 0.3623 (7) | 0.3750(4) | 0.8053(9) | $0.0237(14)$ | $0.021(3)$ | 0.030(4) | 0.021(3) | $0.005(3)$ | 0.003(3) | -0.002(3) |
| O16 | 0.4426(8) | 0.5444(4) | 0.8300(9) | $0.0238(14)$ | 0.029(4) | 0.023(4) | 0.019(3) | 0.004(2) | 0.000(3) | -0.001(3) |
| O17 | $0.3145(8)$ | 0.6921(4) | $0.6734(10)$ | $0.0299(15)$ | $0.031(4)$ | 0.025(4) | $0.034(4)$ | 0.002(3) | 0.006 (3) | 0.004(3) |
| O18 | 0.1364(8) | 0.8750(4) | 0.6871(9) | $0.0257(14)$ | 0.024 (3) | 0.029(4) | 0.025(4) | -0.003(3) | 0.006(3) | -0.002(3) |
| O19 | 0.4137(7) | 0.9737(4) | 0.8315(8) | $0.0229(14)$ | 0.020 (3) | 0.034(4) | 0.014(3) | 0.000(3) | -0.002(2) | -0.009(3) |
| OH20 | 0.4481(8) | 0.8328(4) | 0.5923(10) | $0.0293(15)$ | 0.021(3) | 0.025(4) | 0.043(4) | 0.004(3) | 0.009(3) | 0.006(3) |

nA beam current and a beam diameter of $20 \mu \mathrm{~m}$. Counting times were 20 s on peak and background. Wavelength scans were performed across the $\mathrm{As} L \alpha_{1}$ and $\mathrm{Mg} K \alpha_{1,2}$ peaks in order to establish that there was no peak shift for As $L \alpha_{1}$ between the GaAs standard and magnesiokoritnigite, and to aid in the selection of appropriate background positions due to the proximity of the two analytical peaks. No other elements were detected by energy dispersive spectroscopy. Other likely elements were sought by EMPA. $\mathrm{Co}, \mathrm{Ga}$ and Sb were detected, but were below the limits of analytical significance. Raw X-ray intensities were corrected for matrix effects with a $\phi(\rho z)$ algorithm (Pouchou and Pichoir, 1991).

Because insufficient material was available for a direct determination of $\mathrm{H}_{2} \mathrm{O}$, the amount of water was calculated on the basis of ( $\mathrm{Mg}+\mathrm{Ca}+$ $\mathrm{Mn}+\mathrm{Cu})=1$, charge balance and 5 O atoms p.f.u., as determined by the crystal structure analysis. Analytical data and standards are given in Table 1. Note that magnesiokoritnigite crystals did not take a good polish, to which is attributed the low analytical total; consequently, we also provide the analytical values normalized to $100 \%$.

The empirical formula is $\left(\mathrm{Mg}_{0.94} \mathrm{Cu}_{0.03}\right.$ $\left.\mathrm{Mn}_{0.02} \mathrm{Ca}_{0.01}\right)_{\Sigma 1.00} \mathrm{As}_{0.96} \mathrm{O}_{5} \mathrm{H}_{3.19}$. The simplified structural formula is $\mathrm{Mg}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$, which requires MgO 22.12, $\mathrm{As}_{2} \mathrm{O}_{5} 63.06, \mathrm{H}_{2} \mathrm{O}$ 14.83, total $100 \mathrm{wt} . \%$. The Gladstone-Dale compatibility index $1-\left(\mathrm{K}_{\mathrm{P}} / \mathrm{K}_{\mathrm{C}}\right)$ is -0.020 for the empirical formula and -0.026 for the ideal formula, in both cases in the range of excellent compatibility (Mandarino, 2007).

## X-ray crystallography and structure refinement

Both powder and single-crystal X-ray studies were carried out using a Rigaku R-Axis Rapid II curved imaging plate microdiffractometer, with monochromatized $\mathrm{Mo} K \alpha$ radiation. For the powder-diffraction study, a Gandolfi-like motion on the $\varphi$ and $\omega$ axes was used to randomize the sample and observed $d$ spacings and intensities were derived by profile fitting using JADE 2010 software (Materials Data, Inc.). The powder data presented in Table 2 show good agreement with the pattern calculated from the structure determination. Unit-cell parameters refined from the powder data using JADE 2010 with wholepattern fitting are $a=7.868(7), b=15.838(7)$, $c=6.626(7) \AA, \alpha=91.064(18), \beta=96.394(17)$, $\gamma=90.02(3)^{\circ}$ and $V=820.4(12) \AA^{3}$.


| Mgl-O1 | 2.053(6) | Mg2-O8 | 2.047(6) | Mg3-O4 | 2.053(6) | Mg4-O10 | $2.050(7)$ | $\mathrm{H}_{\text {donor }} \cdots \mathrm{H}_{\text {acceptor }}$ |  | $\mathrm{O} \cdot \mathrm{OW} \cdot \mathrm{O}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| $\mathrm{Mgl}-\mathrm{O} 2$ | $2.060(7)$ | Mg2-O5 | 2.061(7) | Mg3-O16 | $2.055(7)$ | Mg4-OW11 | $2.060(7)$ | OW3 ...OH20 | 2.682(9) |  |
| Mgl-OW3 | 2.061(7) | Mg2-OW7 | 2.061(7) | Mg3-OW9 | $2.057(7)$ | Mg4-O19 | 2.071(6) | OW3 $\cdots$ O13 | $2.698(9)$ | 105.3(2) |
| Mgl-O1 | $2.082(7)$ | Mg2-O5 | $2.112(7)$ | Mg3-O15 | $2.079(7)$ | Mg4-O19 | 2.091(6) | OW7...O17 | $2.760(9)$ |  |
| Mg1-O18 | 2.087(7) | Mg2-O4 | 2.121(6) | Mg3-O16 | $2.081(7)$ | Mg4-O2 | 2.114(7) | OW7...OW3 | $2.962(9)$ | 108.7(2) |
| Mg1-O10 | 2.124(7) | Mg2-OH6 | $2.315(7)$ | Mg3-O8 | $2.122(6)$ | $\mathrm{Mg} 4-\mathrm{OH} 12$ | 2.324(7) | OW9...OH14 | $2.672(9)$ |  |
| $<\mathrm{Mgl}-\mathrm{O}>$ | 2.078 | < Mg2-O> | 2.120 | $<\mathrm{Mg} 3-\mathrm{O}>$ | 2.075 | < Mg4-O> | 2.118 | $\begin{aligned} & \text { OW9…O17 } \\ & \text { OW11…O13 } \end{aligned}$ | $\begin{aligned} & 2.709(9) \\ & 2.736(9) \end{aligned}$ | 105.3(2) |
| As1-O1 | 1.670(6) | As2-O5 | $1.676(6)$ | As3-O16 | 1.669(6) | As4-O19 | 1.678(6) | OW11...OW9 | $2.937(9)$ | 107.8(2) |
| As1-O2 | 1.671(6) | As2-O8 | 1.682(6) | As3-O17 | 1.672(6) | As4-O18 | 1.679(6) | OH6 $\cdots$ O15 | 2.530(8) |  |
| As1-O13 | 1.672(6) | As2-O15 | 1.682(6) | As3-O4 | 1.681(6) | As4-O10 | 1.680(6) | OH12 ...O18 | $2.513(9)$ |  |
| As1-OH12 | 1.730(6) | As2-OH14 | $1.715(6)$ | As3-OH6 | $1.732(6)$ | As4-OH20 | 1.714(6) | OH14...O13 | $2.549(9)$ |  |
| <As1-O> | 1.686 | <As1-O> | 1.689 | <As1-O> | 1.689 | <As1-O> | 1.688 | OH20...O17 | $2.547(9)$ |  |

Table 6. Bond-valence analysis for magnesiokoritnigite. Values are expressed in valence units.

|  | O1 | O2 | OW3 | O4 | O5 | OH6 | OW7 | O8 | OW9 | O 10 | OW11 | OH12 | O13 | OH14 | O15 | O16 | O17 | O18 | O19 | OH20 | $\Sigma_{\text {c }}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Mg1 | $\begin{aligned} & 0.38 \\ & 0.35 \end{aligned}$ | 0.37 | 0.37 |  |  |  |  |  |  | 0.31 |  |  |  |  |  |  |  | 0.34 |  |  | 2.12 |
| Mg 2 |  |  |  | 0.31 | $\begin{aligned} & 0.37 \\ & 0.32 \end{aligned}$ | 0.19 | 0.37 | 0.38 |  |  |  |  |  |  |  |  |  |  |  |  | 1.95 |
| Mg3 |  |  |  | 0.38 |  |  |  | 0.31 | 0.37 |  |  |  |  |  | 0.35 | $\begin{aligned} & 0.38 \\ & 0.35 \end{aligned}$ |  |  |  |  | 2.14 |
| Mg4 |  | 0.32 |  |  |  |  |  |  |  | 0.38 | 0.37 | 0.18 |  |  |  |  |  |  | $\begin{aligned} & 0.36 \\ & 0.34 \end{aligned}$ |  | 1.96 |
| As1 | 1.30 | 1.30 |  |  |  |  |  |  |  |  |  | 1.11 | 1.29 |  |  |  |  |  |  |  | 4.99 |
| As2 |  |  |  |  | 1.28 |  |  | 1.26 |  |  |  |  |  | 1.15 | 1.26 |  |  |  |  |  | 4.95 |
| As3 |  |  |  | 1.26 |  | 1.10 |  |  |  |  |  |  |  |  |  | 1.30 | 1.29 |  |  |  | 4.96 |
| As4 |  |  |  |  |  |  |  |  |  | 1.27 |  |  |  |  |  |  |  | 1.27 | 1.27 | 1.15 | 4.96 |
| H3a |  |  | 0.80 |  |  |  |  |  |  |  |  |  | 0.20 |  |  |  |  |  |  |  | 1.00 |
| H3b |  |  | 0.79 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  | 0.21 | 1.00 |
| H6 |  |  |  |  |  | 0.71 |  |  |  |  |  |  |  |  | 0.29 |  |  |  |  |  | 1.00 |
| H7a |  |  |  |  |  |  | 0.81 |  |  |  |  |  |  |  |  |  | 0.19 |  |  |  | 1.00 |
| H7b |  |  | 0.12 |  |  |  | 0.88 |  |  |  |  |  |  |  |  |  |  |  |  |  | 1.00 |
| H9a |  |  |  |  |  |  |  |  | 0.80 |  |  |  |  |  |  |  | 0.20 |  |  |  | 1.00 |
| H9b |  |  |  |  |  |  |  |  | 0.79 |  |  |  |  | 0.21 |  |  |  |  |  |  | 1.00 |
| H11a |  |  |  |  |  |  |  |  |  |  | 0.81 |  | 0.19 |  |  |  |  |  |  |  | 1.00 |
| H11b |  |  |  |  |  |  |  |  | 0.13 |  | 0.87 |  |  |  |  |  |  |  |  |  | 1.00 |
| H12 |  |  |  |  |  |  |  |  |  |  |  | 0.69 |  |  |  |  |  | 0.31 |  |  | 1.00 |
| H14 |  |  |  |  |  |  |  |  |  |  |  |  | 0.27 | 0.73 |  |  |  |  |  |  | 1.00 |
| H20 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  | 0.27 |  |  | 0.73 | 1.00 |
| $\Sigma_{\text {a }}$ | 2.03 | 1.99 | 2.08 | 1.95 | 1.97 | 2.00 | 2.06 | 1.96 | 2.09 | 1.96 | 2.05 | 1.98 | 1.95 | 2.09 | 1.90 | 2.03 | 1.95 | 1.92 | 1.97 | 2.09 |  |

$\mathrm{Mg}^{2+}-\mathrm{O}$ and $\mathrm{As}^{5+}-\mathrm{O}$ bond strengths from Brown and Altermatt (1985); hydrogen-bond strengths based on $\mathrm{O} \cdots \mathrm{O}$ bond lengths from Brown and Altermatt (1985).

The Rigaku CrystalClear software package was used for processing structure data, including the application of an empirical multi-scan absorption correction using $A B S C O R$ (Higashi, 2001). The structure was solved by direct methods using SIR2004 (Burla et al., 2005), after which atom positions were transformed to correspond to those reported for the structure of koritnigite (Keller et al., 1980). SHELXL-2013 (Sheldrick, 2008) was used for the refinement of the structure. A high degree of mosaicity, particularly in the [010] direction, made necessary the use of a rather small crystal, which provided a limited data set with a relatively large $R_{\text {int }}$. The resulting data did not permit the determination of H atom positions.

Data collection and refinement details are given in Table 3, atom coordinates and displacement parameters in Table 4, selected bond distances in Table 5 and a bond-valence analysis in Table 6.

## Description of the structure

The structure is considered isostructural with those of koritnigite (Keller et al., 1980) and cobaltkoritnigite (Zettler et al., 1979). It contains 4 Mg sites, 4 As sites and 20 O sites. The structure consists of zig-zag edge-sharing chains of $\mathrm{MgO}_{5}\left(\mathrm{H}_{2} \mathrm{O}\right)$ octahedra running parallel to [101] that are joined by corner-sharing with $\mathrm{AsO}_{3}(\mathrm{OH})$ tetrahedra to form sheets parallel to $\{010\}$. Two


Fig. 5. The crystal structure of magnesiokoritnigite viewed down [101] with [010] vertical. Unit cell shown by dashed line. Hydrogen bonds shown as solid lines.

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Fig. 6. One polyhedral sheet in the structure of magnesiokoritnigite viewed down [010] with [101] horizontal. Unit cell shown by dashed line.
of the four vertices of each of the $\mathrm{AsO}_{3}(\mathrm{OH})$ tetrahedra are O atoms of Mg octahedra, while the other two are unlinked, except by hydrogen bonding. The sheets are linked in the [010] direction only by hydrogen bonds (Figs 5 and 6). The Mg 1 and Mg 4 octahedra form one set of chains linked into a sheet centred at 0 along $b$ and the Mg 2 and Mg 3 octahedra form another set of chains linked into a sheet centred at $1 / 2$ along $b$.

The assignment of O sites as $\mathrm{O}, \mathrm{OH}$ or $\mathrm{H}_{2} \mathrm{O}$ (OW) was straightforward based upon bondvalence considerations. Each of the four arsenate groups has one significantly longer As-O bond, which corresponds to an unshared tetrahedral vertex; the corresponding O atoms must correspond to OH groups because they cannot otherwise achieve bond-valence balance. The hydrogen bond scheme proposed also takes into consideration the $\mathrm{O} \cdot \mathrm{OW} \cdot \mathrm{O}$ angles for potential hydrogen bonds. In the proposed scheme, $\mathrm{O}-\mathrm{O}$ bond lengths corresponding to proposed hydrogen bonds fall in the range $2.51-2.96 \AA, \mathrm{O} \cdot \mathrm{OW} \cdot \mathrm{O}$ angles are in the range $105-109^{\circ}$, and all O atoms have resulting bond-valence sums in the range $1.92-2.09 \mathrm{vu}$. This is the same hydrogen bonding scheme proposed for koritnigite by Keller et al. (1980).

The Mg coordinations all include 5 O atoms shared with $\mathrm{AsO}_{3}(\mathrm{OH})$ tetrahedra and one
unshared $\mathrm{H}_{2} \mathrm{O}$ vertex. The Mg 1 and Mg 3 coordinations are $\mathrm{MgO}_{5}\left(\mathrm{H}_{2} \mathrm{O}\right)$, while the Mg 2 and Mg 4 octahedra include one arsenate OH ligand and, therefore, are $\mathrm{MgO}_{4}(\mathrm{OH})\left(\mathrm{H}_{2} \mathrm{O}\right)$. The $\mathrm{Mg} 2-\mathrm{OH} 6(2.315 \AA)$ and $\mathrm{Mg} 4-\mathrm{OH} 12(2.324 \AA)$ bonds are significantly longer than the other $\mathrm{Mg}-\mathrm{O}$ bonds. These bonds are even longer in the koritnigite structure, with a $\mathrm{Zn} 2-\mathrm{OH} 6$ bond length of $2.487 \AA$ and a $\mathrm{Zn} 4-\mathrm{OH} 12$ bond length of $2.495 \AA$. The cobaltkoritnigite is somewhat different in this respect. Although the atom positions and labels for the cobaltkoritnigite structure are generally analogous to the other structures, it is the Co1 and Co3 coordinations in the cobaltkoritnigite structure that include one arsenate OH ligand; however, the bond lengths $\mathrm{Co} 1-\mathrm{OH} 18(2.186 \AA)$ and $\mathrm{Co} 3-\mathrm{OH} 15(2.188 \AA)$ are not appreciably longer than those for the other $\mathrm{Co}-\mathrm{O}$ bonds. Nevertheless, the bond topologies of all three structures are essentially identical, except that the directions of the $06 \cdots \mathrm{O} 15$ and O12 $\cdots$ O18 hydrogen bonds are reversed in the cobaltkoritnigite structure; that is, instead of the $\mathrm{OH} 6 \cdots \mathrm{O} 15$ and $\mathrm{OH} 12 \cdots \mathrm{O} 18$ hydrogen bonds in the structures of koritnigite and magnesiokoritnigite, there are $\mathrm{OH} 15 \cdots \mathrm{O} 6$ and $\mathrm{OH} 18 \cdots \mathrm{O} 12$ hydrogen bonds in the structure of cobaltkoritnigite.

## MAGNESIOKORITNIGITE: THE Mg-ANALOGUE OF KORITNIGITE

TABLE 7. Comparative data for koritnigite, magnesiokoritnigite and cobaltkoritnigite.

|  | Koritnigite* | Magnesiokoritnigite | Cobaltkoritnigite ${ }^{\S}$ |
| :--- | :--- | :--- | :--- |
| Ideal formula | $\mathrm{Zn}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$ | $\mathrm{Mg}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$ | $\mathrm{Co}\left(\mathrm{AsO}_{3} \mathrm{OH}\right) \cdot \mathrm{H}_{2} \mathrm{O}$ |
| Space group | $P \overline{1}$ | $P \overline{1}$ | $P \overline{1}$ |
| $a(\AA)$ | $7.8702(7)$ | 7.95 |  |
| $b(\AA \mathrm{~A})$ | $15.829(5)$ | $15.8081(6)$ | 15.83 |
| $c(\AA)$ | $6.6389(14)$ | 6.67 |  |
| $\alpha\left({ }^{\circ}\right)$ | $90.814(6)$ | 90.9 |  |
| $\beta\left({ }^{\circ}\right)$ | $90.86(2)$ | $96.193(6)$ | 96.6 |
| $\gamma\left({ }^{\circ}\right)$ | $90.094(7)$ | 90.0 |  |
| $V\left(\mathrm{~A}^{3}\right)$ | $96.56(2)$ | $821.06(19)$ | 834 |
| $Z$ | $90.05(2)$ | 8 | 8 |
| Density calc. $\left(\mathrm{g} \mathrm{cm}^{-3}\right)$ | 8 | 2.935 | 3.46 |
| Optical character | 3.54 | biaxial $(+)$ | biaxial $(+)$ |
| $\alpha$ | biaxial $(+)$ | $1.579(1)$ | $1.646(2)$ |
| $\beta$ | $1.632(5)$ | $1.586(1)$ | $1.668(2)$ |
| $\gamma$ | $1.652(3)$ | $1.620(1)$ | $1.705(5)$ |
| $2 \mathrm{~V}\left({ }^{\circ}\right)$ | $50(2)$ | $78(2)$ |  |

* Keller et al. (1979). ${ }^{\text {§ }}$ Schmetzer et al. (1981).

Comparative crystallographic, density and optical data for koritnigite, magnesiokoritnigite and cobaltkoritnigite are given in Table 7. It is worth noting that, in spite of the generally comparable cell parameters, the density and indices of refraction for magnesiokoritnigite are dramatically smaller than those of for koritnigite and cobaltkoritnigite. Obviously, this is due to the much lower atomic number of Mg .

## Acknowledgements

Reviewers Peter Williams, Nikita Chukanov and Igor Pekov and Editorial Board Member Stuart Mills are thanked for their constructive comments on the manuscript. A portion of this study was funded by the John Jago Trelawney Endowment to the Mineral Sciences Department of the Natural History Museum of Los Angeles County.

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[^0]:    * E-mail: akampf@nhm.org

    DOI: 10.1180/minmag.2013.077.8.03

[^1]:    $* R_{\text {int }}=\Sigma \mid F_{\mathrm{o}}^{2}-F_{\mathrm{o}}^{2}($ mean $)\left|/ \Sigma\left[F_{\mathrm{o}}^{2}\right] . \mathrm{GoF}=S=\left\{\Sigma\left[w\left(F_{\mathrm{o}}^{2}-F_{\mathrm{c}}^{2}\right)^{2}\right] /(n-p)\right\}^{1 / 2} . R_{1}=\Sigma\right|\left|F_{\mathrm{o}}\right|-\left|F_{\mathrm{c}}\right||/ \Sigma| F_{\mathrm{o}} \mid$.
    $w R_{2}=\left\{\Sigma\left[w\left(F_{\mathrm{o}}^{2}-F_{\mathrm{c}}^{2}\right)^{2}\right] / \Sigma\left[w\left(F_{\mathrm{o}}^{2}\right)^{2}\right]\right\}^{1 / 2} ;$
    $w=1 /\left[\sigma^{2}\left(F_{\mathrm{o}}^{2}\right)+(a P)^{2}+b P\right]$ where $a$ is $0.0279, b$ is 8.5788 and $P$ is $\left[2 F_{\mathrm{c}}^{2}+\operatorname{Max}\left(F_{\mathrm{o}}^{2}, 0\right)\right] / 3$.

