# Kolitschite, $Pb[Zn_{0.5},\square_{0.5}]Fe_3(AsO_4)_2(OH)_6$, a new mineral from the Kintore opencut, Broken Hill, New South Wales.

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## ABSTRACT

*Kolitschite, ideally $Pb[Zn_{0.5},\square_{0.5}]Fe_3(AsO_4)_2(OH)_6$, where $\square$ = vacancy, (IMA 2008–063), is a newly defined mineral from the Kintore opencut, Broken Hill, New South Wales, Australia. Kolitschite occurs as steeply pseudorhombohedral crystals to 0.2 mm associated with mimetite, segnitite, carminite, bayldonite and cryptomelane. The main forms observed are {110} (prism) and {001} (pinacoid). Kolitschite crystals are yellowish green with a light yellow streak and are transparent to translucent with an adamantine lustre. Mohs hardness is 4, no cleavage was observed and all crystals are twinned. Kolitschite is monoclinic, C2/c, with $a$ = 25.8898(6), $b$ = 14.8753(2), $c$ = 12.1700(2) Å, $\beta$ = 110.681(1)°, $V$ = 4384.9(2) $Å^3$ and $Z$ = 16. Kolitschite crystals are biaxial (+), with estimated indices of refraction $n$ = 2.0. 2V is ≤ 10°. Orientation: Y = b, with straight extinction and very weak pleochroism: X = green to olive green, Y = green to olive green, Z = yellowish green to apple green. Absorption: X ≈ Y > Z. The simplified formula (on the basis of [As+P+S] = 2 and OH = 6) is: $Pb[Zn_{0.25}\square_{0.75}]Fe_3H_{0.5}(AsO_4)_2(OH)_6$, which requires Zn 2.12, Pb 26.86, Fe 21.72, As 19.42, O 29.03, H 0.85, total 100.00. A solid-solution series extends towards the end-member composition of $Pb[Zn_{0.5}\square_{0.5}]Fe_3(AsO_4)_2(OH)_6$, with the single-crystal structure giving a charge-balanced empirical formula of $Pb[Zn_{0.439}\square_{0.561}]Fe_3H_{0.122}(AsO_4)_2(OH)_6$. The strongest five powder-diffraction lines [d in Å, ($I/I_0$), (hkl)] are: 3.114, (100), (621, 62-3, 04-2); 6.034, (45), (400, 22-1); 2.280, (37), (404, 22-5); 3.719, (31), (62-1, 040); 2.844, (25), (004). Kolitschite is related to members of the alunite supergroup, in particular segnitite by the substitution mechanism $(ZnO_2)(\square(OH)_2)_{-1}$. The mineral is named for Dr Uwe Kolitsch (b. 1966), for his contribution to mineralogy.*

## INTRODUCTION

The Broken Hill mines in far western New South Wales (31°58′ S, 141°28′ E) have made a significant contribution to Australian mineralogy, with over 300 species thus far being discovered (Birch, 1999). The mines have provided the type localities for fourteen species, including bernalite, costibite, kintoreite, mawbyite, marshite, miersite, paradocrasite, raspite, segnitite and willyamite, and the recent new minerals hoganite and paceite (Hibbs *et. al.*, 2002), birchite (Elliott *et al.*, 2008) and IMA2008–013, the $Zn^{2+}$ analogue of rockbridgeite, whose description is in preparation. The Kintore opencut is the type locality for kintoreite, mawbyite and segnitite.

Here, we report the fifteenth type mineral described from Broken Hill. Kolitschite is named for Dr Uwe Kolitsch (b. 1966), Curator of Mineralogy at the Natural History Museum, Vienna, Austria, to recognise his contribution to mineralogy — in particular to the characterisation of new minerals (of which he has been involved in ~30) and crystallography, whilst also recognising his work on alunite-group minerals (Kolitsch *et al.*, 1999a,b,c; Kolitsch & Pring, 2001; Kolitsch *et al.*, 2005). The mineral data and name were approved by the IMA Commission of New Minerals, Nomenclature and Classification (CNMNC) prior to publication (IMA 2008–063). The type specimen is deposited in the collection of Museum Victoria, Melbourne, Australia (M41714).

## OCCURRENCE

The type specimen of kolitschite was taken from the footwall of number 3 ore lens between 230 and 240 metres (above sea level) near the base of the Kintore opencut at its southern end (shown in Fig. 7 of Birch & van der Heyden, 1997). The sample was collected by Bill Birch and Dermot Henry (Museum Victoria) during a Mineralogical Society of Victoria field trip in March 1991.

Kolitschite occurs on a piece of siliceous and ferruginous ore. Mimetite forms a significant portion of the matrix, occurring as massive patches as well as linings to a number of irregular cavities up to 2 cm across. These linings generally show mimetite crystal faces. Other arsenates occur within the

**Table 1: Electron microprobe analyses for kolitschite.**

| Constituent | Wt.% | Range | Ideal* (as oxides) | Probe Standard |
|---|---|---|---|---|
| Pb | 26.06 | 25.11–26.61 | 28.35 | $PbSO_4$ (Mα) |
| Fe | 21.63 | 21.07–22.22 | 30.42 | Hematite (Kα) |
| Zn | 2.19 | 1.77–2.52 | 5.17 | Sphalerite (Lα) |
| Cu | 0.27 | 0.12–0.43 | | Chalcopyrite (Kα) |
| Al | 0.11 | 0.10–0.18 | | $MgAl_2O_4$ (Kα) |
| As | 19.46 | 18.70–20.17 | 29.19 | GaAs (Lα) |
| S | 0.25 | 0.15–0.60 | | $PbSO_4$ (Kα) |
| P | 0.03 | 0.02–0.04 | | Apatite (Kα) |
| O* | 30.42 | 30.05–30.70 | 6.86 | $PbSO_4$ (Kα) |
| Total | 100.42 | | 100.00 | |

*Based on the endmember formula $Pb[Zn_{0.5},\square_{0.5}]Fe_3(AsO_4)_2(OH)_6$ ; Fe as $Fe_2O_3$ and O as $H_2O$

**Table 2: X-ray powder diffraction data for kolitschite**

| *h* | *k* | *l* | $I_{obs}$ | $d_{obs}$ | $d_{calc}$ |
|---|---|---|---|---|---|
| 4 | 0 | 0 | **45** | 6.034 | 6.052 |
| 2 | 2 | -1 | | | 6.030 |
| 0 | 0 | 2 | 9 | 5.695 | 5.686 |
| 3 | 1 | 1 | 2 | 5.324 | 5.320 |
| 3 | 1 | -2 | | | 5.318 |
| 2 | 2 | 1 | 2 | 5.140 | 5.144 |
| 4 | 2 | -1 | 5 | 4.832 | 4.832 |
| 2 | 0 | 2 | 3 | 4.564 | 4.562 |
| 6 | 0 | 0 | 5 | 4.037 | 4.035 |
| 6 | 2 | -1 | **31** | 3.719 | 3.732 |
| 0 | 4 | 0 | | | 3.717 |
| 2 | 2 | -3 | 11 | 3.556 | 3.556 |
| 4 | 2 | -3 | 2 | 3.447 | 3.448 |
| 7 | 1 | 0 | 2 | 3.363 | 3.368 |
| 5 | 3 | -2 | | | 3.360 |
| 8 | 0 | -2 | 9 | 3.163 | 3.179 |
| 2 | 4 | -2 | | | 3.165 |
| 4 | 4 | 0 | | | 3.167 |
| 6 | 2 | 1 | **100** | 3.114 | 3.120 |
| 6 | 2 | -3 | | | 3.119 |
| 0 | 4 | -2 | | | 3.111 |
| 4 | 4 | -2 | 9 | 3.010 | 3.015 |
| 8 | 2 | -1 | 2 | 2.956 | 2.956 |
| 0 | 0 | 4 | **25** | 2.844 | 2.843 |
| 8 | 2 | -3 | 5 | 2.724 | 2.721 |
| 7 | 1 | -4 | 2 | 2.678 | 2.675 |
| 4 | 2 | 3 | 2 | 2.619 | 2.618 |
| 8 | 0 | -4 | 21 | 2.569 | 2.577 |
| 4 | 4 | 2 | | | 2.572 |
| 8 | 2 | 1 | 3 | 2.534 | 2.535 |
| 5 | 5 | 0 | | | 2.534 |
| 10 | 0 | 0 | 2 | 2.411 | 2.421 |
| 8 | 4 | -2 | | | 2.416 |
| 2 | 6 | -1 | | | 2.410 |
| 2 | 6 | 1 | 2 | 2.334 | 2.341 |
| 4 | 0 | 4 | **37** | 2.280 | 2.281 |
| 2 | 2 | -5 | | | 2.279 |
| 6 | 2 | 3 | 18 | 2.259 | 2.262 |
| 0 | 4 | -4 | | | 2.258 |
| 6 | 6 | -1 | 3 | 2.144 | 2.149 |
| 8 | 2 | -5 | | | 2.144 |
| 10 | 4 | -2 | 3 | 2.115 | 2.120 |
| 2 | 6 | -3 | | | 2.114 |
| 12 | 2 | -3 | 3 | 2.039 | 2.038 |
| 6 | 6 | 1 | 15 | 2.008 | 2.010 |
| 6 | 6 | -3 | | | 2.010 |
| 9 | 5 | -3 | 9 | 1.997 | 1.995 |
| 8 | 2 | 3 | 2 | 1.968 | 1.966 |
| 10 | 4 | -4 | 1 | 1.943 | 1.947 |
| 5 | 7 | 0 | | | 1.945 |
| 12 | 4 | -2 | 10 | 1.864 | 1.866 |
| 0 | 8 | 0 | 9 | 1.856 | 1.859 |
| 12 | 2 | 1 | 3 | 1.820 | 1.818 |
| 2 | 0 | 6 | 3 | 1.775 | 1.779 |
| 4 | 4 | -6 | | | 1.778 |
| 14 | 2 | -1 | 2 | 1.755 | 1.756 |
| 10 | 4 | 2 | | | 1.753 |
| 2 | 6 | -5 | 4 | 1.720 | 1.722 |
| 6 | 2 | 5 | 11 | 1.690 | 1.690 |
| 6 | 2 | -7 | | | 1.690 |
| 0 | 4 | -6 | | | 1.689 |
| 2 | 2 | -7 | 5 | 1.650 | 1.649 |
| 10 | 2 | -7 | 4 | 1.607 | 1.607 |
| 8 | 4 | 4 | | | 1.606 |
| 8 | 8 | -2 | | | 1.605 |
| 8 | 6 | 3 | 2 | 1.577 | 1.574 |
| 0 | 8 | -4 | 7 | 1.554 | 1.556 |
| 8 | 8 | -4 | 19 | 1.508 | 1.508 |
| 16 | 4 | -4 | 5 | 1.460 | 1.462 |
| 14 | 4 | -6 | 3 | 1.460 | 1.460 |
| 10 | 8 | -4 | 6 | 1.440 | 1.442 |
| 5 | 9 | -4 | 3 | 1.440 | 1.441 |
| 0 | 0 | 8 | 3 | 1.421 | 1.422 |
| 14 | 4 | 2 | 4 | 1.401 | 1.401 |
| 8 | 0 | 6 | | | 1.399 |
| 12 | 8 | -4 | 4 | 1.364 | 1.367 |
| 6 | 10 | -3 | | | 1.365 |
| 14 | 6 | 1 | 4 | 1.360 | 1.360 |
| 16 | 4 | -6 | | | 1.360 |
| 14 | 0 | -8 | | | 1.360 |
| 16 | 6 | -3 | 4 | 1.355 | 1.354 |
| 12 | 6 | -7 | 2 | 1.325 | 1.323 |
| 15 | 7 | -1 | 2 | 1.321 | 1.319 |
| 2 | 2 | -9 | 2 | 1.286 | 1.286 |
| 2 | 10 | -5 | 4 | 1.265 | 1.263 |
| 4 | 8 | 6 | 3 | 1.233 | 1.234 |
| 16 | 8 | -2 | 2 | 1.216 | 1.217 |
| 18 | 0 | -8 | | | 1.215 |
| 18 | 6 | -1 | | | 1.215 |
| 10 | 10 | -5 | 4 | 1.206 | 1.206 |
| 8 | 8 | -8 | 3 | 1.170 | 1.171 |
| 4 | 12 | 2 | | | 1.170 |
| 2 | 6 | -9 | 3 | 1.154 | 1.155 |
| 4 | 12 | -4 | 2 | 1.148 | 1.146 |
| 22 | 2 | -1 | 2 | 1.121 | 1.121 |
| 16 | 0 | -10 | | | 1.119 |
| 10 | 12 | -2 | 2 | 1.118 | 1.117 |

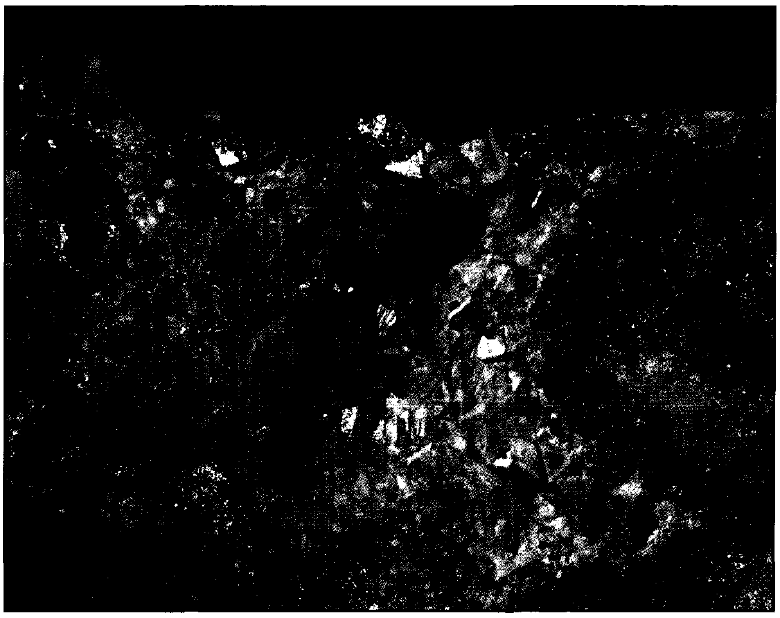

**Figure 1: Yellowish green, steeply pseudorhombohedral crystals of kolitschite. Field of view 5 mm across. Photo: B. Heally. Specimen: Museum Victoria collection, M41714.**

**Figure 2: Backscattered SEM image of a portion of a kolitschite crystal showing Zn distribution. Crystal is ~150 microns across. Specimen: Museum Victoria collection, M41714.**

cavities, growing on the mimetite. These are dominated by various forms of yellow to brown segnitite crystals, ranging from granular yellow aggregates to crusts showing acute rhombohedral crystal faces and to yellowish microcrystals scattered on the mimetite. Carminite prisms, bayldonite crusts and cryptomelane coatings are also present. The yellowish kolitschite crystals occupy several small cavities up to about 5 mm across.

## APPEARANCE AND PROPERTIES

Kolitschite occurs as steeply pseudorhombohedral crystals to 0.2 mm (Figure 1), which are visually indistinguishable from some examples of segnitite, with the main forms observed being {110} (prism) and {001} (pinacoid). Kolitschite crystals are yellowish green with a light-yellow streak and are transparent to translucent with an adamantine lustre. The Mohs hardness is 4 (estimated from its relationship to other Pb-dominant alunite-type minerals) and no cleavage was observed. Kolitschite crystals are brittle with an uneven fracture. All crystals examined were twinned by 120° rotations about the pseudohexagonal $c_h$ axis (see Grey *et al.*, 2008). The calculated density is 4.674 g/cm$^3$ (for $Pb[Zn_{0.25}\square_{0.75}]Fe_3H_{0.5}(AsO_4)_2(OH)_6$ and crystals sink in Clerici solution (density = 4.2 g/cm$^3$).

Kolitschite crystals are biaxial (+) [pseudo-uniaxial (+)]. Refractive indices are >1.8, using the highest RI liquids available. The calculated average refractive index for white light based on the Gladstone–Dale compatibility (Mandarino, 1981) is $n$ = 2.0. 2V is very small and hard to measure but is ≤ 10°. Orientation: Y = $b$, with extinction parallel to $b$. In transmitted light crystals show very weak pleochroism, from X = green to olive green, Y = green to olive green, Z = yellowish green to apple green. Absorption: X ≈ Y > Z. Kolitschite crystals show bluish interference colours.

## CHEMICAL COMPOSITION

Chemical analyses (10) were carried out by means of an electron microprobe (WDS mode, 15 kV, 20 nA, 10 μm defocused beam; JEOL Superprobe JXA-8900R) at CSIRO Minerals, Melbourne, Australia. The low voltage used, together with the use of a light-element detector, allowed the sample to be analyzed directly for oxygen. The results as well as the standards used are shown in Table 1.

Some zoning, as shown by slight variations in back-scatter contrast (Figure 2), was found to be due to small variations (1–3 wt %) in zinc content over regions of the order of tens of micrometers. The empirical formula (based on [As+P+S] = 2), but without charge balance involving H atoms, is $Pb_{0.94}Zn_{0.25}Cu_{0.03}(Fe_{2.89}Al_{0.03})_{\Sigma 2.92}[(As_{1.94}S_{0.06}P_{0.01})O_8]O_{6.16}$. With $OH^-$ = 6, the charge-balanced empirical formula is: $Pb_{0.94}Zn_{0.25}Cu_{0.03}(Fe_{2.89}Al_{0.03})_{\Sigma 2.92}H_{0.76}[(As_{1.94}S_{0.06}P_{0.01})O_8](OH)_6$. The simplified formula is: $Pb[Zn_{0.25}\square_{0.75}]Fe_3H_{0.5}(AsO_4)_2(OH)_6$ which requires Zn 2.12, Pb 26.86, Fe 21.72, As 19.42, O 29.03, H 0.85, total 100.00.

It is evident that in kolitschite a solid-solution series extends towards the end-member composition of $Pb[Zn_{0.5}\square_{0.5}]Fe_3(AsO_4)_2(OH)_6$, with the single-crystal structure giving a charge-balanced empirical formula of $Pb[Zn_{0.439}\square_{0.561}]Fe_3H_{0.122}(AsO_4)_2(OH)_6$.

## X-RAY CRYSTALLOGRAPHY

The crystal structure of kolitschite has been solved and the full description is reported in Grey *et al.* (2008). The general features of the kolitschite structure match those of the rhombohedral alunite-type structure ($R$-3$m$, $a$ ~ 7 and $c$ ~ 17 Å). Kolitschite, however, is monoclinic, $C2/c$, with

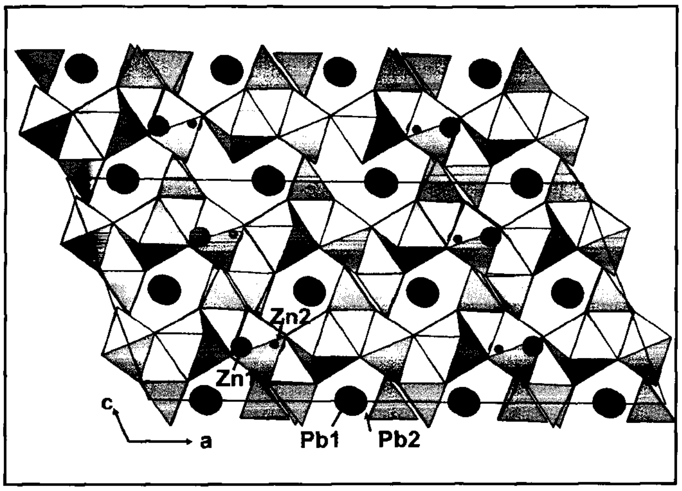


**Figure 3: Projection of the structure of kolitschite along [010]. The Pb and Zn atoms are labelled, with the $AsO_4$ tetrahedron (blue) and $FeO_6$ octahedron (yellow).**

$a = 25.8898(6)$, $b = 14.8753(2)$, $c = 12.1700(2)$ Å, $\beta = 110.681(1)°$, $V = 4384.9(2)$ Å$^3$ and $Z = 16$. The a:b:c: ratio is 1.741:1:0.818. The structure of kolitschite involves rhombohedral stacking of hexagonal tungsten–bronze-type sheets of corner-connected octahedra that share their apical anions with $AsO_4$ tetrahedra on either side of the layer of octahedra, with Pb atoms located between the layers (Figure 3). The structure differs from those of other alunite-type minerals in having zinc atoms ordered in trigonal bipyramidal sites in half of the available six-membered rings of the layers of octahedra. These sites are unoccupied in previously reported alunite-type structures. Ordered displacements of the Pb atoms occur in response to the Zn–□ ordering (Figure 3), which also make the kolitschite structure unique.

X-ray powder-diffraction data (Table 2) were collected using a 114.6-mm-diameter Gandolfi camera with Ni-filtered Cu*Kα* radiation at the Department of Geology, National Museum of Nature and Science, Japan. Data were calibrated with a quartz standard (reference material NIST SRM #1878a) on the crystal of kolitschite. Unit-cell parameters refined from the powder data are $a = 25.888(9)$, $b = 14.869(5)$, $c = 12.161(4)$ Å, $\beta = 110.75(2)°$, $V = 4377(1)$ Å$^3$ with Z = 16, which are in good agreement with those obtained from the single-crystal study.

## PARAGENESIS

The Broken Hill arsenate suite, which includes kolitschite, has crystallized from solutions rich in Pb, Fe and As derived from the breakdown of the primary ore of 3 Lens, one of the Pb lodes at Broken Hill. This primary ore consists mainly of galena and sphalerite, with minor chalcopyrite and arsenopyrite–löllingite in a quartz-rich gangue. Within the paragenetic sequence for arsenate suites (see Plimer, 1999 and Birch & van der Heyden, 1997) kolitschite probably forms at the same stage as segnitite and carminite immediately after mimetite.

## IDENTIFICATION OF KOLITSCHITE

Kolitschite is related to members of the alunite supergroup, in particular segnitite by the substitution mechanism $(ZnO_2)(\square(OH)_2)_{-1}$ and by lowering symmetry (Table 3). Kolitschite shows many similar properties, including colour, which make it difficult to distinguish from segnitite. Some chemical information in addition to X-ray diffraction (either powder or single crystal) is likely to be needed to unambiguously determine the presence of kolitschite over segnitite. From the chemical data it is evident that a solid solution exists, however it has not been determined how much $Zn^{2+}$ is required to stabilise the structure of kolitschite. Recently, Grey *et al.* (2009) noted the presence of zinc occupying a similar trigonal bipyramidal site in the crystal structure of kintoreite from the 280 m RL level of Kintore, where up to 3.82 wt % Zn was observed. Here, the incorporation of Zn into the structure did not change it from having rhombohedral symmetry, therefore we recommend that some structural information be required before labelling specimens of zinc-bearing segnitite as kolitschite.

**Table 3: Comparative data for kolitschite and segnitite.**

| | Kolitschite | Segnitite[1,2] |
|---|---|---|
| Ideal formula | $Pb[Zn_{0.5}\square_{0.5}]Fe_3(AsO_4)_2(OH)_6$ | $PbFe_3H(AsO_4)_2(OH)_6$ |
| Space group | C2/c | *R*-3*m* |
| a, b | 25.8898(6), 14.8753(2) | ~7.3 |
| c | 12.1700(2) | ~17.1 |
| β | 110.681(1) | |
| V | 4384.9 | ~805 |
| Z | 16 | 3 |
| 5 strongest lines in the powder pattern | 3.114(100), 6.034(45), 2.280 (37), 3.719(31), 2.844(25) | 3.092 (100), 5.966 (50), 3.678 (40), 2.283 (30), 1.992 (30) |
| $D_{meas}$ $D_{calc}$ | >4.2, 4.648 | >4.2, 4.77 |
| Colour | Yellowish green | Greenish brown to yellowish brown, dark brown, green |
| Optical class | Biaxial (+), 2V ≤ 10° | Uniaxial (-) |
| Refractive indices | $n_{calc} = 2.0$ | $\omega = 1.975(5)$, $\varepsilon = 1.955(5)$ |

[1]Birch et al., 1992; [2]Mills, 2007

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