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# FROM STRUCTURE TOPOLOGY TO CHEMICAL COMPOSITION. XXVIII. TITANIUM SILICATES: JINSHAJIANGITE FROM THE OKTYABR'SKII MASSIF, DONETSK REGION, UKRAINE, A NEW OCCURRENCE

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## ABSTRACT

Here we report electron-microprobe data and unit-cell parameters for jinshajiangite, ideally $NaBaFe^{2+}{}_4Ti_2(Si_2O_7)_2O_2(OH)_2F$, from a new locality: the Oktyabr'skii massif in the coastal area of the Azov Sea, Donetsk region, Ukraine. Chemical analysis by electron microprobe gave $Nb_2O_5$ 1.59, $ZrO_2$ 0.61, $TiO_2$ 17.07, $SiO_2$ 27.60, $Al_2O_3$ 0.08, $Fe_2O_3$ 2.04, FeO 16.42, BaO 9.81, ZnO 0.76, MnO 12.97, CaO 1.82, MgO 0.07, $K_2O$ 2.05, $Na_2O$ 2.51, F 2.48, $H_2O$ 1.92, O = F –1.04, sum 98.76 wt.%; $H_2O$ was determined in accord with the required number of monovalent anions for the Ti-dominant perraultite-type minerals: OH + F = 3 *pfu*; the $Fe^{3+}/Fe^{2+}$ ratio was assigned in accord with Mössbauer-spectroscopy results for jinshajiangite from a different locality. The empirical formula calculated on the basis of 19 (O + F) is $(Na_{0.71}Ca_{0.28}\square_{0.01})_{\Sigma1}(Ba_{0.56}K_{0.38}\square_{0.06})_{\Sigma1}(Fe^{2+}{}_{1.99}Mn_{1.59}Fe^{3+}{}_{0.22}Zn_{0.08}Mg_{0.02}Al_{0.01}\square_{0.09})_{\Sigma4}$ $(Ti_{1.86}Nb_{0.10}Zr_{0.04})_{\Sigma2}(Si_{4.00}O_{14})O_2[(OH)_{1.86}F_{0.14}]_{\Sigma2}F_{1.00}$, $Z = 4$. Unit-cell parameters from the single-crystal data were determined by least-squares refinement of 9807 reflections with $I > 10\sigma I$ and are as follows: $a = 10.726(8)$, $b = 13.834(10)$, $c = 11.065(8)$ Å, $\alpha = 108.172(5)$, $\beta = 99.251(7)$, $\gamma = 90.00(1)°$, $V = 1537.5(3.4)$ Å$^3$, space group $C\bar{1}$ .

*Keywords*: jinshajiangite, perraultite, seidozerite supergroup, TS block, electron-microprobe analysis, unit-cell parameters, Oktyabr'skii massif, Donetsk region, Ukraine.

## INTRODUCTION

Perraultite, ideally $NaBaMn_4Ti_2(Si_2O_7)_2O_2(OH)_2F$, jinshajiangite, ideally $NaBaFe^{2+}{}_4Ti_2(Si_2O_7)_2O_2(OH)_2F$, and bobshannonite, ideally $Na_2KBa(Mn_7Na)Nb_4(Si_2O_7)_4O_4(OH)_4O_2$, are bafertisite-group minerals of the seidozerite supergroup (Sokolova & Cámara 2017). They belong to the structure type of perraultite (Yamnova *et al*. 1998), and we call them perraultite-type minerals (Table 1). The ideal formulae of perraultite and jinshajiangite are in accord with the current nomenclature of the seidozerite supergroup (Sokolova & Cámara 2017). The ideal formula of bobshannonite has been revised by Sokolova *et al*. (2020). The seidozerite-supergroup minerals have structures based on a TS-block (TS = Titanium-Silicate). The TS-block consists of HOH sheets (H = heteropolyhedral, O = octahedral) and is characterized by a planar cell based on translation vectors $\mathbf{t_1}$ and $\mathbf{t_2}$, with $t_1 \sim 5.5$ and $t_2 \sim 7$ Å and $t_1 \wedge t_2$ close to 90°. The seidozerite-supergroup minerals are divided into four groups based on the content of Ti (+ Nb + Zr + $Fe^{3+}$ + Mg + Mn), topology, chemical composition, and stereochemistry of the TS block (Sokolova 2006, 2010, Sokolova & Cámara 2017). In the rinkite, bafertisite, lamprophyllite, and murmanite groups, Ti (+ Nb + Zr + $Fe^{3+}$ + Mg + Mn) = 1, 2, 3, and 4 *apfu* (atoms per formula unit), respectively. Perraultite and perraultite-type minerals jinshajiangite and bobshannonite have basic structures characterized by doubled $t_1$ and $t_2$ unit-cell parameters. The concept of *basic* and *derivative structures* for TS-block minerals was

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TABLE 1. IDEAL STRUCTURAL FORMULAE OF THE PERRAULTITE-TYPE MINERALS (STRUCTURE TYPE B1BG)*

| Mineral | Ideal structural formula** | | | | | | | | *a* (Å) α(°) | *b* (Å) β(°) | *c* (Å) γ(°) | Space group | *Z* | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $A^P$ | $B^P$ | $M^O{}_4$ | $M^H{}_2$ | $(Si_2O_7)_2$ | $(X^O{}_M)_2$ | $(X^O{}_A)_2$ | $X^P{}_M$ | | | | | | |
| Perraultite | Ba | Na | $Mn_4$ | $Ti_2$ | $(Si_2O_7)_2$ | $O_2$ | $(OH)_2$ | F | 10.731<br>90 | 13.841<br>95.06 | 20.845<br>90 | *C*2 | 8 | (1,2) |
| Jinshajiangite | Ba | Na | $Fe^{2+}{}_4$ | $Ti_2$ | $(Si_2O_7)_2$ | $O_2$ | $(OH)_2$ | F | 10.720<br>108.222 | 13.823<br>99.28 | 11.044<br>89.989 | $C\bar{1}$ | 4 | (3,4) |
| | $A^P{}_2$ | $B^P{}_2$ | $M^O{}_8$ | $M^H{}_4$ | $(Si_2O_7)_4$ | $(X^O{}_M)_4$ | $(X^O{}_A)_4$ | $(X^P{}_M)_2$ | | | | | | |
| Bobshannonite | K<br>Ba | $Na_2$ | $(Mn_7Na)$ | $Nb_4$ | $(Si_2O_7)_4$ | $O_4$ | $(OH)_4$ | $O_2$ | 10.831<br>108.145 | 13.903<br>99.215 | 11.149<br>90.007 | $C\bar{1}$ | 4 | (5,4) |

* Structure type (B1BG: B – basic, BG – bafertisite group) and ideal structural formulae of perraultite and jinshajiangite are from Sokolova & Cámara (2017); ideal structural formula of bobshannonite is from Sokolova *et al.* (2019); formulae are per $(Si_2O_7)_2$, for bobshannonite: per $(Si_2O_7)_4$.

** Atoms' labelling: $M^H$ = cations of the H sheet, $M^O$ = cations of the O sheet, $A^P$ and $B^P$ = cations at the peripheral (*P*) sites; $X^O{}_M$ = common anions for three $M^O$ octahedra of the O sheet and one $M^H$ octahedron of the H sheet; $X^O{}_A$ = common anions for three $M^O$ octahedra of the O sheet; $X^P{}_M$ = bridging anion between two $M^H$ octahedra (Sokolova 2006). References (first description of a new mineral, the most recent structure work): (1) Chao (1991); (2) Yamnova *et al.* (1998); (3) Hong & Fu (1982); (4) Sokolova *et al.* (2020); (5) Sokolova *et al.* (2015).

introduced by Sokolova & Cámara (2013). A *basic structure* has the following four characteristics: (1) there is only one type of TS block, (2) the two H sheets of the TS block are identical, (3) there is only one type of **I** block or it is absent, (4) there is only one type of self-linkage of TS blocks. Basic structures obey the general structural principles of Sokolova (2006). A *derivative structure* has one or more of the three following characteristics: (1) there is more than one type of TS block, (2) there is more than one type of **I** block, (3) there is more than one type of self-linkage of TS blocks. A derivative structure is related to two or more basic structures of the same group: it can be derived by adding these structures *via* sharing the central O sheet of the TS blocks of adjacent structural fragments which represent basic structures. Selected structure information for the perraultite-type minerals is given in Table 1. This table does not list surkhobite (Rastsvetaeva *et al.* 2008), as it is a doubtful mineral species (for an explanation, see Sokolova *et al.* 2020).

Jinshajiangite from the Jinshajiang River, Sichuan Province, China, was described as a new mineral by Hong & Fu (1982). Chao (1991) described perraultite from Mont Saint-Hilaire, Québec, Canada, and suggested that perraultite is isotypic with jinshajiangite. Yamnova *et al.* (1998) solved the crystal structure of perraultite from the Oktyabr'skii (formerly Mariupol'skii) massif in the coastal area of the Azov Sea (Donetsk region, Ukraine): monoclinic, space group *C*2 (Table 1). A description of perraultite from the Oktyabr'skii massif was given by Pekov *et al.* (1999). Sokolova *et al.* (2009) refined the crystal structure of jinshajiangite from Norra Kärr, Jönköping province, Sweden, in space group *C*2/*m*. Lykova *et al.* (2010) reported a chemical composition for jinshajiangite from the Gremyakha-Vyrmes alkaline complex, Kola Peninsula, Russia. Sokolova *et al.* (2015) described bobshannonite, a Nb-analogue of perraultite (Table 1). Sokolova *et al.* (2015) refined the structure of bobshannonite in the unconventional space group $C\bar{1}$ to make comparison with the TS-block structures more straightforward: the TS-block structures are characterized by two minimal translations, $t_1 \sim 5.4$ and $t_2 \sim 7$ Å. Following the work of Sokolova *et al.* (2015), Cámara *et al.* (2016) refined the crystal structure of jinshajiangite from the Verkhnee Espe deposit, Kazakhstan, in space group $C\bar{1}$. Jin *et al.* (2018) studied jinshajiangite from the type locality, Jinshajiang river area in Sichuan Province, China, by TEM (transmission electron microscopy) and refined its crystal structure in space group $P\bar{1}$, with $c \sim 11$ Å compared to $c \sim 21$ Å in all previous refinements of jinshajiangite. They stated that the basic topology of the new $P\bar{1}$ structure is analogous to the previously proposed $C\bar{1}$ structure of Cámara *et al.* (2016). Jin *et al.* (2018) suggested that all minerals with the perraultite-type structure (jinshajiangite itself plus perraultite, surkhobite, and bobshannonite) should have the same $P\bar{1}$ structure as jinshajiangite with $c \sim 11$ Å instead of the previously reported $c \sim 21$ Å (*cf.* perraultite, Table 1) and that all previously proposed monoclinic space groups were pseudosymmetry generated by nanoscale polysynthetic twinning on the (001) composition plane. Following Jin *et al.* (2018),

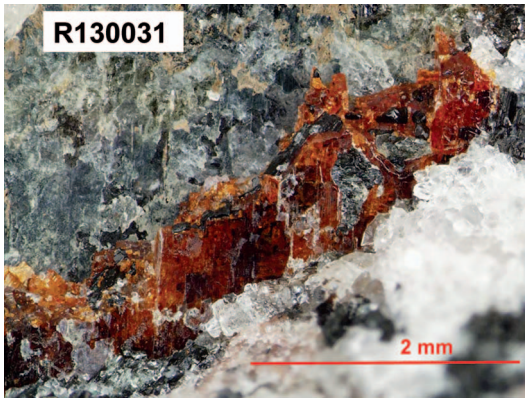


FIG. 1. Prismatic reddish-orange crystals of jinshajiangite in matrix, sample R130031, photograph courtesy of the RRUFF database.

Sokolova *et al*. (2020) refined the crystal structures of jinshajiangite, surkhobite, and bobshannonite in space group $C\bar{1}$ (with $c \sim 11$ Å) to facilitate comparison with the TS-block structures (Table 1).

Perraultite is a Mn-analogue of jinshajiangite: Mn > $Fe^{2+}$ in the O sheet. In jinshajiangite and perraultite the two $A^P$ sites in the **I** (Intermediate) block give Ba > K, ideally Ba *apfu* (atoms per formula unit). Bobshannonite is a Nb-analogue of perraultite: $Nb^{5+}$ > $Ti^{4+}$ in the H sheet. In bobshannonite, Ba:K = 1:1 and Ba and K are ordered at the two $A^P$ sites in the **I** block.

We wanted to refine the crystal structure of perraultite from the Oktyabr'skii massif and obtained several grains of the "perraultite" sample R130031, which is the property of the RRUFF Database, University of Arizona, Tucson, USA. However, electron-microprobe results showed that all available grains had chemical compositions corresponding to jinshajiangite. Here, we report electron-microprobe data and unit-cell parameters for jinshajiangite from a new locality: the Oktyabr'skii massif in the coastal area of the Azov Sea, Donetsk region, Ukraine.

## Description of the Sample

Jinshajiangite was found in sample R130031, which is the property of the RRUFF Database, University of Arizona, Tucson, USA; the sample was given to to the RRUFF database as a sample of perraultite. The sample is from Dmitrievka village, the Oktyabr'skii massif in the coastal area of the Azov Sea, Donetsk region, Ukraine, and came from the mineral collection of Don Doell [sample #DD 13-05], Canada. Jinshajiangite forms reddish brown prismatic crystals which exhibit good cleavage (Fig. 1).

## Chemical Composition

Table 2 gives six chemical analyses of jinshajiangite: analyses 1–5 are taken from the literature (references are given in the footnote), and analysis 6 for jinshajiangite from the Oktyabr'skii massif (sample R130031) is from this work. Analysis 7 is for a different sample of perraultite from the same locality: Oktyabr'skii massif, Donetsk region, Ukraine, and it is taken from Pekov *et al*. (1999).

### *Electron-microprobe analysis of jinshajiangite*

Electron-microprobe analysis was done on several grains of sample R130031 with a Cameca SX-100 electron-microprobe operating in wavelength-dispersion mode with an accelerating voltage of 15 kV, a specimen current of 20 nA, a beam size of 5 μm, and count times on peak and background of 20 and 10 s, respectively (except for fluorine: 30 and 15 s). The following standards were used: F: fluoro-riebeckite; Na: albite; Nb, Ba: $Ba_2NaNb_5O_{15}$; Si, Ca: diopside; Fe: fayalite; Mn: spessartine; K: orthoclase; Ti: titanite; Zn: gahnite; Zr: zircon; Mg: forsterite; Sr: $SrTiO_3$; Al: andalusite. Tantalum, Sr and Cs were sought but not detected. Data were reduced using the φ(ρZ) procedure of Pouchou & Pichoir (1985). The chemical composition of jinshajiangite from the Oktyabr'skii massif, Donetsk region, Ukraine, is given in Table 2 (analysis 6) and is the mean of 10 determinations on the grain shown in Figure 2. $H_2O$ was determined in accord with the required number of monovalent anions for the Ti-dominant perraultite-type minerals: OH + F = 3 *pfu*. $Fe^{3+}/Fe^{2+}$ was assigned in accord with our Mössbauer-spectroscopy results for jinshajiangite from Norra Kärr (Table 2, analysis 2, $Fe^{3+}/Fe^{2+} = 0.10$), which is close to the Mössbauer-spectroscopy results for jinshajiangite from Jinshajiang River, China (Table 2, analysis 5, $Fe^{3+}/Fe^{2+} = 0.07$). The empirical formula calculated on the basis of 19 (O + F) is $(Na_{0.71}Ca_{0.28}\square_{0.01})_{\Sigma1}(Ba_{0.56}K_{0.38}\square_{0.06})_{\Sigma1}(Fe^{2+}_{1.99}Mn_{1.59}Fe^{3+}_{0.22}Zn_{0.08}Mg_{0.02}Al_{0.01}\square_{0.09})_{\Sigma4}(Ti_{1.86}Nb_{0.10}Zr_{0.04})_{\Sigma2}(Si_{4.00}O_{14})O_2[(OH)_{1.86}F_{0.14}]_{\Sigma2}F_{1.00}$, ideally $NaBaFe^{2+}{}_4Ti_2(Si_2O_7)_2O_2(OH)_2F$, $Z = 4$.

## Data Collection and Unit-Cell Parameters

Single-crystal X-ray data for jinshajiangite were collected with a Bruker D8 QUEST ECO CMOS diffractometer equipped with a fine-focus tube (Mo$K\alpha$) and a PHOTON 50 detector from a crystal with following dimensions: 0.08 × 0.04 × 0.02 mm. Unit-cell parameters from the single-crystal data were determined by least-squares refinement of 9807 reflections with $I > 10\sigma I$ and are as follows: $a$ = 10.726(8), $b$ = 13.834(10), $c$ = 11.065(8) Å, α =

TABLE 2. CHEMICAL ANALYSIS* (wt.%) AND FORMULA UNIT (*apfu*) FOR JINSHAJIANGITE

| Mineral | Jinshajiangite | | | | | | Perraultite |
|---|---|---|---|---|---|---|---|
| Locality | (1) Jinshajiang River, China Holotype | (2) Norra Kärr, Sweden | (3) Verkhnee Espe deposit, Kazakhstan | (4) Gremyakha-Vyrmes Complex, Russia | (5) Jinshajiang River, China | (6) Oktyabr'skii massif, Ukraine | (7) Oktyabr'skii massif, Ukraine |
| Oxide | | | | | | | |
| $Ta_2O_5$ | 0.07 | n.d. | n.d. | n.a. | n.d. | n.d. | 0.11 |
| $Nb_2O_5$ | 1.03 | 0.12 | 2.74 | 0.93 | 1.59 | 1.59 | 1.24 |
| $ZrO_2$ | 0.70 | 0.51 | 0.22 | 0.2 | 0.76 | 0.61 | 1.09 |
| $TiO_2$ | 15.90 | 18.36 | 16.24 | 18.19 | 17.06 | 17.07 | 17.83 |
| $SiO_2$ | 27.10 | 27.56 | 27.41 | 27.45 | 26.74 | 27.60 | 27.72 |
| $Ce_2O_3$ | 0.30 | n.a. | n.a. | n.a. | n.d. | n.a. | n.a. |
| $Al_2O_3$ | 0.36 | n.d. | 0.07 | 0.16 | n.d. | 0.08 | 0.03 |
| $Fe_2O_3$ | 1.64 | 2.89 | 2.34 | n.a. | 1.16 | 2.04 | 0.75 |
| FeO | 19.07 | 23.42 | 19.00 | 29.27 | 15.07 | 16.42 | 12.06 |
| BaO | 9.80 | 10.24 | 10.46 | 10.01 | 10.24 | 9.81 | 10.64 |
| SrO | 0.08 | n.d. | n.d. | n.a. | n.d. | n.d. | 0.05 |
| ZnO | 0 | n.d. | 0.10 | 0.12 | n.d. | 0.76 | 1.02 |
| MnO | 12.98 | 5.13 | 10.19 | 2.42 | 14.76 | 12.97 | 19.28 |
| CaO | 2.94 | 2.52 | 1.45 | 2.84 | 2.09 | 1.82 | 1.48. |
| MgO | 0.28 | 0.44 | 0.19 | 0.19 | 0.05 | 0.07 | 0.04 |
| $Cs_2O$ | 0 | 0.03 | n.d. | n.a. | n.d. | n.d. | n.d |
| $K_2O$ | 2.31 | 1.95 | 1.92 | 2.40 | 2.19 | 2.05 | 1.67 |
| $Na_2O$ | 3.15 | 2.27 | 2.74 | 2.03 | 2.33 | 2.51 | 2.76 |
| F | 2.66 | 2.33 | 2.31 | 1.60 | 2.31 | 2.48 | 2.18 |
| $H_2O$ | 0.33 | 2.01 | 1.96 | 2.35** | 2.00 | 1.92 | 1.79 |
| O = F | –1.12 | –0.98 | –0.99 | –0.67 | –0.97 | –1.04 | –0.92 |
| Total | 99.58 | 98.80 | 98.39 | 99.49 | 97.38 | 98.76 | 99.80 |

TABLE 2. CONTINUED.

| Mineral | Jinshajiangite | | | | | | Perraultite |
|---|---|---|---|---|---|---|---|
| Locality | (1) Jinshajiang River, China Holotype | (2) Norra Kärr, Sweden | (3) Verkhnee Espe deposit, Kazakhstan | (4) Gremyakha-Vyrmes Complex, Russia | (5) Jinshajiang River, China | (6) Oktyabr'skii massif, Ukraine | (7) Oktyabr'skii massif, Ukraine |
| Formula unit calculated on a basis of 19 (O + F) | | | | | | | |
| Ta | | | | | | | |
| Nb | 0.07 | 0.01 | 0.18 | 0.06 | 0.11 | 0.10 | 0.08 |
| Zr | 0.05 | 0.04 | 0.02 | 0.01 | 0.06 | 0.04 | 0.08 |
| Ti | 1.79 | 1.99 | 1.79 | 1.98 | 1.90 | 1.86 | 1.94 |
| Si | 4.06 | 3.97 | 4.01 | 3.97 | 3.96 | 4.00 | 4.02 |
| $Ce^{3+}$ | 0.02 | | | | | | |
| $Fe^{3+}$ | 0.19 | 0.31 | 0.26 | | 0.13 | 0.22 | 0.08 |
| Al | 0.06 | | 0.01 | 0.03 | | 0.01 | 0.01 |
| Ba | 0.58 | 0.58 | 0.60 | 0.57 | 0.59 | 0.56 | 0.60 |
| Sr | 0.01 | | | | | | |
| Zn | | | 0.01 | 0.01 | | 0.08 | |
| $Fe^{2+}$ | 2.39 | 2.83 | 2.32 | 3.54 | 1.87 | 1.99 | 1.46 |
| Mn | 1.65 | 0.63 | 1.26 | 0.30 | 1.85 | 1.59 | 2.37 |
| Ca | 0.47 | 0.39 | 0.23 | 0.44 | 0.33 | 0.28 | 0.23 |
| Mg | 0.06 | 0.10 | 0.04 | 0.04 | 0.01 | 0.02 | 0.01 |
| K | 0.44 | 0.36 | 0.36 | 0.44 | 0.41 | 0.38 | 0.31 |
| Na | 0.91 | 0.64 | 0.78 | 0.57 | 0.67 | 0.71 | 0.78 |
| F | 1.26 | 1.06 | 1.09 | 0.73 | 1.08 | 1.14 | 1.00 |
| OH | 0.33 | 1.94 | 1.91 | 2.27 | 1.97 | 1.86 | 1.73 |
| ΣCat | 12.75 | 11.83 | 11.87 | 11.95 | 11.89 | 11.86 | 11.97 |

* (1) wet chemistry; (2–7) electron-microprobe analysis; $H_2O$: (1) TGA; (7) Penfield method; (2,3,4,6) calculated in accord with required number of monovalent anions: OH + F = 3 *pfu* per $(Si_2O_7)_2$; see next note for (4). $Fe^{2+}/Fe^{3+}$: (1) wet chemistry; (2,5) Mössbauer spectroscopy; (3,6) assigned in accord with (2); (7) wet chemistry.
** There is no data for $H_2O$ in (4) so we calculated $H_2O = 2.35$ wt.% in accord with required number of monovalent anions: OH + F = 3 *pfu* per $(Si_2O_7)_2$ (this work).
Abbreviations: n.r. = not reported; n.a. = not analyzed; n.d. = not detected. References: (1) Hong & Fu (1982); (2) Sokolova *et al.* (2009); (3) Cámara *et al.* (2016); (4) analysis 9, Lykova *et al.* (2010); (5) Jin *et al.* (2018); (6) this work; (7) Pekov *et al.* (1999).

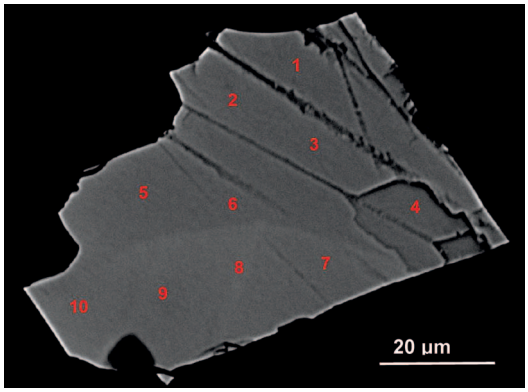


FIG. 2. BSE image of the jinshajiangite crystal; the 10 analytical points are labeled in red.

108.172(5), β = 99.251(7), γ = 90.00(1)°, $V$ = 1537.5(3.4) Å$^3$, space group $C\bar{1}$. The values of the $a$ and $b$ unit-cell parameters of $Fe^{2+}$-dominant jinshajiangite from the Oktyabr'skii massif (this work) are close to those of jinshajiangite from Norra Kärr, Sweden (Sokolova *et al*. 2020, Table 1) and smaller than those of Mn-dominant perraultite (Yamnova *et al*. 1998, Table 1) and Mn-dominant bobshannonite (Sokolova *et al*. 2020, Table 1). The values of the $a$ and $b$ unit-cell parameters correlate with the ionic radius of the dominant cation in the O sheet: $^{[6]}Fe^{2+}$ and $^{[6]}Mn^{2+}$ have $r$ = 0.78 Å and $r$ = 0.83 Å, respectively (Shannon 1976).

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