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Triazolite, $NaCu_2(N_3C_2H_2)_2(NH_3)_2Cl_3{\cdot}4H_2O$, a new mineral species containing 1,2,4-triazolate anion, from a guano deposit at Pabellón de Pica, Iquique Province, Chile

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ABSTRACT

The new mineral triazolite is found in a guano deposit located on the Pabellón de Pica Mountain, Iquique Province, Tarapacá Region, Chile. Associated minerals are salammoniac, halite, dittmarite, joanneumite, chanabayaite, nitratine, natroxalate and möhnite. Triazolite occurs as deep blue prismatic crystals up to 0.1 mm × 0.15 mm × 0.75 mm and their radial aggregates up to 1.5 mm across. The mineral is brittle. Its Mohs hardness is 2. $D_{calc} = 2.028$ g cm^{-3}. The infrared spectrum shows the presence of 1,2,4-triazolate anion and ammonia and water molecules. Triazolite is optically biaxial (–), $\alpha = 1.582(4)$, $\beta = 1.625(3)$, $\gamma = 1.625(3)$ and $2V_{meas} = 5(3)°$. The chemical composition (electron-microprobe data for Cl, Na, Fe and Cu; H, C, N and S contents were measured by gas chromatography of products of ignition at 1200°C; wt.%) is: Na 4.91, Fe 1.51, Cu 22.06, Cl 19.80, S 1.4, C 7.7, H 4.4, N 24.2, O (calc.) 12.83, total 98.81. The empirical formula is $Na_{1.14}(Cu_{1.86}Fe_{0.14})(Cl_{2.99}S_{0.23})N_{9.23}C_{3.43}H_{23.34}O_{4.29}$. The idealized formula is $NaCu_2(N_3C_2H_2)_2(NH_3)_2Cl_3{\cdot}4H_2O$. Triazolite is a metalorganic ammine complex in which 1,2,4-triazolate anion and ammonia molecule are ligands coordinating Cu^{2+}. The crystal structure was solved by direct methods and refined to $R = 0.0242$ based on 4210 unique reflections with $I > 2\sigma(I)$. Triazolite is orthorhombic, space group $P2_12_12_1$, $a = 19.3575(5)$, $b = 7.15718(19)$, $c = 12.5020(4)$ Å, $V = 1732.09(8)$ Å^3 and $Z = 4$. The eight strongest reflections of the powder X-ray diffraction pattern [d, Å (I,%) (hkl)] are: 10.22 (97) (101), 6.135 (40) (011), 5.696 (17) (301), 5.182 (59) (202), 5.119 (100) (211), 4.854 (19) (400), 3.752 (16) (312, 501), 3.294 (18) (221). Triazolite is named for the presence of 1,2,4-triazolate anion.

Keywords: triazolite, new mineral, organic mineral, 1,2,4-triazolate anion, ammine complex, crystal structure, guano, Pabellón de Pica, Chile.

Introduction

Numerous guano deposits occur in the Atacama Desert, Chile, in a narrow band (69°30' to 70°10'W by 19°30' to 26°S) attached to the Coastal Range composed of Late Paleozoic and Mesozoic igneous

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rocks and stretching along the northern coast of Chile. Most of these deposits are situated on low-lying hills that were formerly islands. Some details of the geological setting and the mining history of guano deposits in the Tarapacá region are published elsewhere (Ericksen, 1981; Pankhurst and Herve, 2007; Appelton and Nothold, 2002; Bojar *et al.*, 2010).

The guano deposit located on Pabellón de Pica Mountain, 1.5 km south of Chanabaya village, Iquique Province, Tarapacá Region, Chile (20°55'S, 70°08'W) has become known in the last decade as an interesting, very unusual mineralogical object being the type locality of several nitrogen-bearing and organic minerals: ammineite, $CuCl_2(NH_3)_2$ (Bojar *et al.*, 2010), joanneumite, $Cu(C_3N_3O_3H_2)_2(NH_3)_2$ (Bojar and Walter, 2012), chanabayaite, $Cu_4(N_3C_2H_2)_4(NH_3)_4Cl_2(Cl,OH)_2 \cdot H_2O$ (Chukanov *et al.*, 2015*a*), shilovite, $Cu(NH_3)_4(NO_3)_2$ (Chukanov *et al.*, 2015*b*), antipinite, $KNa_3Cu_2(C_2O_4)_4$ (Chukanov *et al.*, 2015*c*) and möhnite, $(NH_4)K_2Na(SO_4)_2$ (Chukanov *et al.*, 2015*d*).

Specimens with the new mineral triazolite, $NaCu_2(N_3C_2H_2)_2(NH_3)_2Cl_3 \cdot 4H_2O$, were collected at Pabellón de Pica in 2012 by one of the authors (G.M.). The mineral is named for the presence of 1,2,4-triazolate anion.

The new mineral and its name were approved by the International Mineralogical Association Commission on New Minerals, Nomenclature and Classification (IMA2017-025 Chukanov *et al.*, 2017). The holotype specimen is deposited in the collection of the Fersman Mineralogical Museum of the Russian Academy of Sciences, Moscow, Russia, with the registration number 5037/1.

Occurrence, general appearance and physical properties

Triazolite occurs in the contact zone between altered guano and the host rock, a gabbro which consists of amphibole, plagioclase and minor clinochlore and containing accessory chalcopyrite. Well-shaped prismatic crystals of triazolite up to 0.1 mm × 0.15 mm × 0.75 mm elongated along *b* and their radial aggregates up to 1.5 mm in diameter (Fig. 1) occur in cavities in aggregates of salammoniac (with subordinate halite) near the contact of a guano deposit with the host rock, or grow over the surface of the host rock. Other associated minerals are dittmarite, joanneumite, chanabayaite, nitratine, natroxalate and möhnite. Nitratine is present as an interspersed trace admixture. Joanneumite is observed in association with triazolite only sporadically.

FIG. 1. Aggregates of triazolite crystals on salammoniac (field of view 2.2 mm). Photograph: M. Burkhardt.

The observed forms of triazolite crystals are: pinacoids {001}, {010} and {100} and prism {*hk*0} or {0*kl*}.

The colour of the new mineral is deep blue. The streak is blue. Crystals of triazolite are translucent, with a vitreous lustre. The new mineral is brittle, with a Mohs hardness of 2. Cleavage is perfect on (001) and imperfect on (100) and (010). No parting is observed. The fracture across the perfect cleavage plane is uneven. Triazolite is non-fluorescent in ultraviolet light.

All attempts to measure the density of triazolite by flotation in heavy liquids gave an unsatisfactory, definitely erroneously low value ~1.85 g cm^{-3}. This could be explained by the fact that triazolite crystals, although appearing perfect (Fig. 1), contain many microcracks (Fig. 2). In addition,

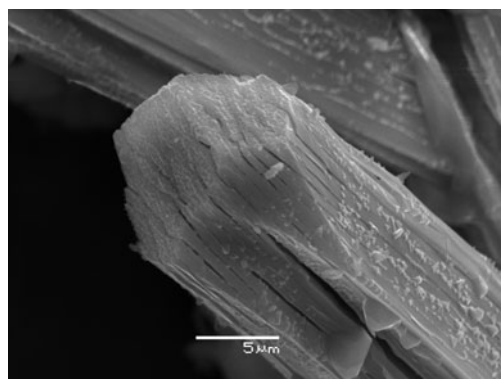

FIG. 2. Scanning electron microscopy image of a triazolite crystal.

micro-inclusions of salammoniac are common in triazolite crystals that could also decrease the bulk measured density. The density calculated using the empirical formula is 2.028 g cm^{-3}.

Triazolite is optically biaxial (–), $\alpha = 1.582(4)$, $\beta = 1.625(3)$, $\gamma = 1.625(3)$, $2V_{meas} = 5(3)°$ and $2V_{calc.} \approx 0°$ ($\lambda = 589$ nm). The dispersion of optical axes is not observed. The orientation is: $X = b$. Pleochroism is distinct: Y (violet) > $X \approx Z$ (greenish-blue).

In order to obtain infrared (IR) absorption spectra (Fig. 3), powdered samples were mixed with dried KBr, pelletized and analysed using an ALPHA FTIR spectrometer (Bruker Optics) with a resolution of 4 cm^{-1}. Sixteen scans were obtained. The IR spectrum of an analogous pellet of pure KBr was used as a reference.

The assignment of absorption bands made in accordance with Grinshtein *et al*. (1970), Nakamoto (2008, 2009), Chukanov *et al*. (2015*a*) and Chukanov and Chervonnyi (2016) is as follows. Strong bands in the range 3100–3500 cm^{-1} correspond to N–H and O–H stretching vibrations. The bands in the range 2800–3100 cm^{-1} correspond to C–H stretching vibrations of the 1,2,4–triazolate anion, possibly combined with N–H stretching vibrations. Bands in the range 1620–1660 cm^{-1} are due to degenerate bending vibrations of NH_3 and bending vibrations of H_2O molecules. Strong bands in the range 1095–1510 cm^{-1} correspond to in-plane stretching and mixed vibrations of the 1,2,4-triazolate ring and to symmetrical bending vibrations of NH_3 molecules. The bands in the range 990–1062 cm^{-1} are assigned to in-plane bending vibrations of C–H bonds. The band at 887 cm^{-1} corresponds to in-plane bending vibrations of the 1,2,4-triazolate ring. Bands below 800 cm^{-1} are due

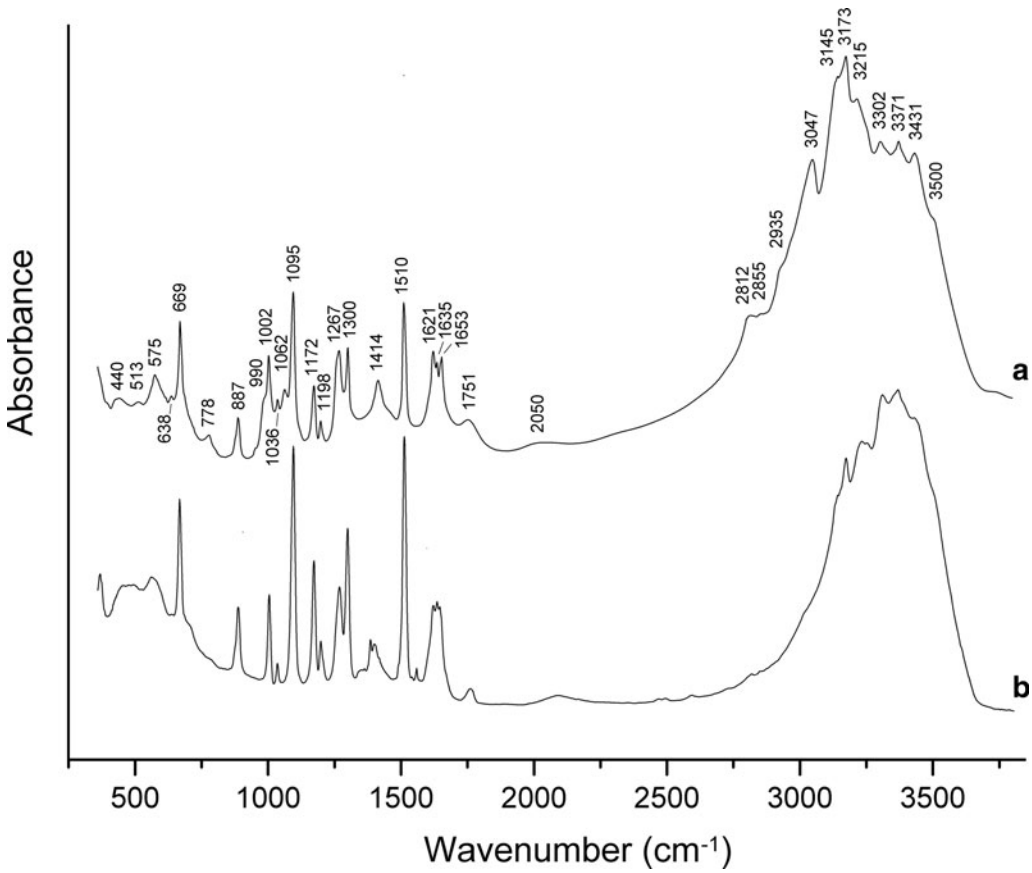

FIG. 3. Powder IR absorption spectra of (*a*) triazolite and (*b*) chanabayaite.

to out-of-plane vibrations of the 1,2,4-triazolate anion, rocking vibrations of NH_3 and librational vibrations of H_2O molecules.

The broad bands at 1751 and 2050 cm^{-1} correspond to acid groups which may arise e.g. as a result of partial protonation of triazolate anions in accordance with the dynamic equilibrium $C_2N_3H_2^- + H_2O \leftrightarrow C_2N_3H_3 + OH^-$.

Chemical data

Chemical analyses (four for EMPA) were carried out using an electron microprobe (EDS mode, 20 kV, 600 pA, beam rastered on an area 10 µm × 10 µm in order to minimize unstable sample damage) for Na, Cu, Fe and Cl. The standards used are: albite for Na, metallic iron for Fe, $CuFeS_2$ for Cu and NaCl for Cl. Attempts to use the wavelength dispersive mode, with higher beam current, were unsuccessful because of the instability of the mineral. H, N, C and S were analysed by gas chromatography of products of ignition in oxygen at 1200°C with a Vario Micro cubeanalyser (Elementar GmbH, Germany). Structural data and chemical tests show the absence of CO_3^{2-} anions. All carbon belongs to the triazolate anion. Analytical data are given in Table 1.

The empirical formula calculated on the basis of Cu + Fe = 2 apfu [because occupancy of the (Cu,Fe) sites is 100%] is $Na_{1.14}(Cu_{1.86}Fe_{0.14})\Sigma_{2.0}N_{9.23}C_{3.43}H_{23.34}O_{4.29}(Cl_{2.99}S_{0.23})\Sigma_{3.22}$. Taking into account that the presence of organic minerals indicates mineral formation under reducing conditions, admixed sulfur is considered as S^{2-}.

TABLE 1. Chemical composition of triazolite*.

Constituent	Content, wt.%	Range
Na	4.91	4.26–5.48
Fe	1.51	1.39–1.76
Cu	22.06	20.57–23.94
Cl	19.80	18.98–19.72
S	1.4 ± 0.2	
C	7.7 ± 1.5	
H	4.4 ± 0.7	
N	24.2 ± 2.4	
O	12.83**	
Total	98.81	

*Contents of other elements with an atomic number > 6 are below detection limits; **calculated from the idealized formula.

The idealized formula is $NaCu_2(N_3C_2H_2)_2(NH_3)_2Cl_3 \cdot 4H_2O$ ($Z = 4$; see description of the crystal structure), which requires Na 4.61, Cu 25.48, Cl 21.33, N 22.48, C 9.63, H 3.64, O 12.83, total 100.00 wt.%.

Reactions of triazolite solution in 20% HCl with potassium hexacyanoferrate(II) and potassium hexacyanoferrate(III) show that all the Cu in the mineral is bivalent. Triazolite dissolves in dilute hydrochloric and nitric acids without gas evolution. It is insoluble in water, but crystals of triazolite become dull after exposure to warm water. Heating in a closed tube gives a white sublimate of NH_4Cl. Melting is accompanied by the release of water and an additional portion of sublimate. Stronger heating results in reduction to metallic Cu.

X-ray diffraction and crystal structure

Powder X-ray diffraction data (Table 2) were collected using a Stoe IPDS II image plate diffractometer (Mo*K*α radiation, Gandolfi mode). Orthorhombic unit-cell parameters refined from the powder data are: $a = 19.319(5)$, $b = 7.149(2)$, $c = 12.519(4)$ Å and $V = 1729(1)$ Å^3.

Single-crystal X-ray studies were carried out using a Bruker Kappa X8 APEX CCD diffractometer (Institute of Mineralogy and Crystallography, University of Vienna), Mo*K*α-radiation (λ = 0.71073 Å). A full sphere of three-dimensional data was collected at $T = 200$ K. Triazolite is orthorhombic, space group $P2_12_12_1$, $a = 19.3575(5)$, $b = 7.15718(19)$, $c = 12.5020(4)$ Å, $V = 1732.09(8)$ Å^3 and $Z = 4$. The crystal structure was solved by direct methods and refined with the use of *SHELX* software (Sheldrick, 2008) to the final $R = 0.0242$ for 4210 unique reflections with $I > 2\sigma(I)$.

A detailed crystal-structure description is given by Zubkova *et al.* (2016). The crystal structure of triazolite (Fig. 4) contains zig-zag chains of the corner-sharing CuN_4Cl_2 octahedra (with N atoms belonging to four 1,2,4-triazole rings) running along the *b* axis and isolated $Cu^{2+}N_4Cl_2$ octahedra (with two N atoms belonging to two 1,2,4-triazole rings and two N atoms of ammonia molecules NH_3) connected with the chains *via* 1,2,4-triazolate anions $C_2N_3H_2^-$. $Na(H_2O)_6$ octahedra share edges to form columns parallel to the *b* axis (Fig. 5). The system of hydrogen bonds formed by H_2O and NH_3 molecules connects isolated Cu-centred octahedra with each other and with the columns of $Na(H_2O)_6$ octahedra as well as the octahedral chains built by Cu and Na polyhedra.

TABLE 2. Powder X-ray diffraction data of triazolite.

I_{obs}	d_{obs}, Å	I_{calc}*	d_{calc}, Å**	*h k l*
97	10.22	81	10.502	101
7	9.71	12	9.679	200
40	6.135	41	6.211	011
17	5.696	20	5.734	301
59	5.182	100	5.251	202
100	5.119	47	5.227	211
19	4.854	34	4.839	400
2	4.599	10	4.575	112
8	4.475	1, 3	4.513, 4.475	401, 311
4	4.156	5, 1	4.234, 4.074	212, 103
16	3.752	25, 2	3.803, 3.698	312, 501
1	3.499	2	3.519	120
3	3.413	3	3.440	021
13	3.358	22, 2	3.375, 3.375	213, 412
4	3.343	4	3.357	220
18	3.294	7	3.242	221
10	3.072	15, 1	3.106, 3.086	022, 104
12	3.024	5	3.036	321
10	2.971	4, 6	2.990, 2.974	512, 204
10	2.863	18, 4, 10	2.889, 2.867, 2.863	413, 602, 611
10	2.847	8, 10	2.837, 2.833	503, 114
4	2.796	3, 3, 3	2.813, 2.804, 2.798	304, 421, 322
4	2.747	3	2.747	214
6	2.707	9, 14, 17	2.715, 2.700, 2.689	023, 701, 123
17	2.644	11	2.626	404
14	2.599	5, 35	2.618, 2.614	314, 422
14	2.561	2	2.572	521
4	2.468	1	2.502	323
2	2.431	4	2.423	522
3	2.395	2, 8	2.403, 2.396	613, 620
3	2.378	4, 4	2.384, 2.367	712, 130
1	2.337	2	2.326	131
9	2.285	7, 30	2.304, 2.287	703, 224
6	2.267	17	2.278	231
12	2.242	1, 5	2.255, 2.237	811, 622
18	2.202	7, 1	2.211, 2.193	324, 713
6	2.158	3	2.155	721
1	2.105	3, 6	2.109, 2.100	431, 505
4	2.069	1, 2	2.070, 2.065	033, 722
4	2.051	6	2.037	206
4	1.997	2, 1	2.008, 2.001	813, 016
2	1.970	1, 1	1.976, 1.956	605, 912
5	1.908	16, 4	1.909, 1.905	822, 615
5	1.895	14, 2	1.902, 1.896	624, 034
4	1.854	5, 1, 6	1.849, 1.849, 1.848	10.0.2, 416, 10.1.1
2	1.825	1	1.819	334
1	1.796	3, 3	1.801, 1.793	026, 724
2	1.779	2	1.782	140
1	1.753	9	1.750	606
3	1.709	1, 1, 4	1.713, 1.705, 1.703	142, 10.1.3, 10.2.0
4	1.682	5, 1	1.687, 1.686	426, 923
3	1.676	4	1.678	440
1	1.605	3	1.611	541

(continued)

TABLE 2. (*contd.*)

I_{obs}	d_{obs}, Å	I_{calc}*	d_{calc}, Å**	*h k l*
1	1.572	1, 2	1.576, 1.573	10.2.3, 833
2	1.549	3, 1, 2	1.553, 1.553, 1.548	044, 641, 932
2	1.512	3, 2	1.518, 1.510	642, 344
2	1.490	2, 1, 1	1.495, 1.492, 1.487	10.2.4, 741, 408
1	1.474	4, 2, 1	1.479, 1.477, 1.471	13.0.1, 11.2.3, 12.2.0
2	1.439	2	1.439	840
2	1.431	5	1.433	12.0.4
1	1.400	2, 1	1.405, 1.396	835, 337
1	1.355	2, 2, 1	1.354, 1.354, 1.354	146, 744, 053
2	1.346	4	1.344	246

*Only reflections with $I_{calc} \geq 1$ are taken into account; **calculated with the unit-cell parameters obtained from single-crystal data.

Discussion

Usually nitrogen is present in minerals as a NO_3^- anion or NH_4^+ cation. There are only five minerals containing the neutral ammonia molecule NH_3^0 as a species-defining component: ammineite, joanneumite, shilovite, chanabayaite and triazolite. All these species have been discovered at Pabellón de Pica, and in all these minerals ammonia molecules coordinate Cu^{2+}. Note also that chanabayaite and triazolite are currently the only known minerals containing N–N bonds, even though the chemical properties and crystal chemistry of numerous synthetic triazole and triazolate complexes have been investigated in detail.

A review of metal coordination compounds with 1,2,4-triazole derivatives as ligands is given by Haasnoot (2000). In the 5-membered 1,2,4-triazole ring all C–N and N–N bonds are conjugated and have fractional bond orders between 1 and 2. As a result, C–N and N–N bond lengths are rather short (typically, between 1.30 and 1.38 Å). Deprotonated 1,2,4-triazole (= triazolate anion) is known as an active acyl transfer catalyst suitable for the aminolysis

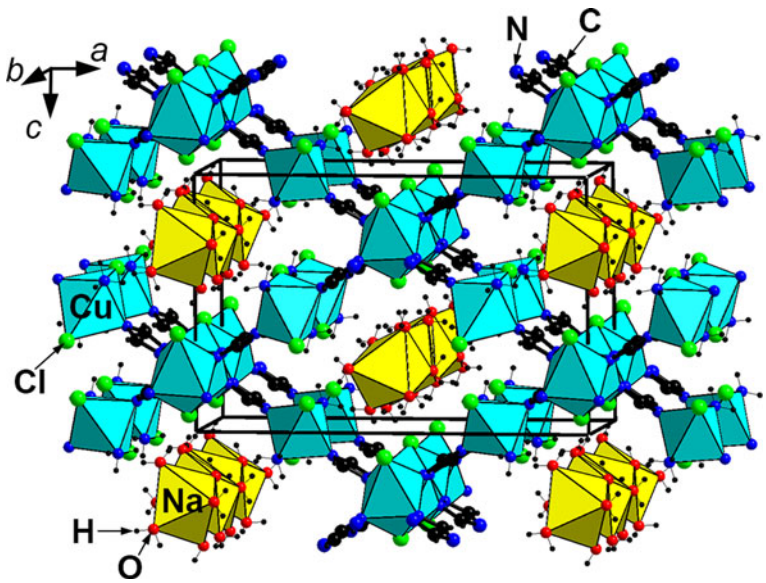

FIG. 4. The crystal structure of triazolite.

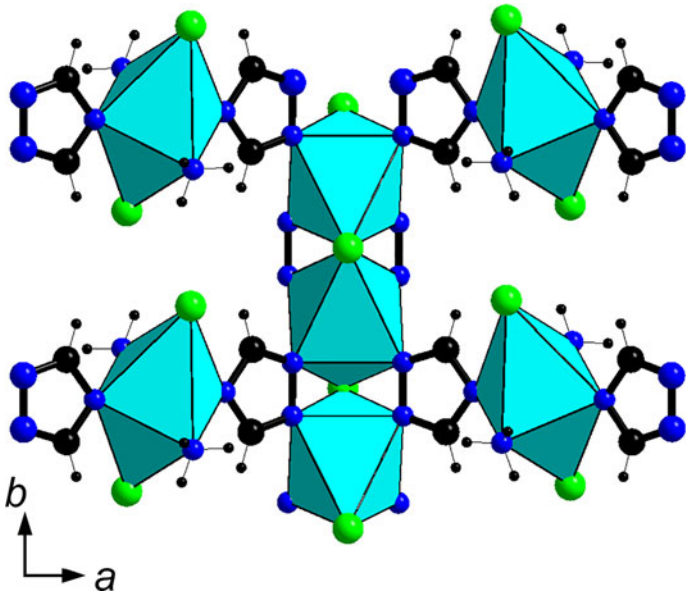

FIG. 5. A fragment of the triazolite structure showing mutual alignment of the zig-zag chain of Cu-centred octahedra and isolated Cu-centred octahedra connected *via* 1,2,4-triazolate anions. For better clarity, only the connection on the upper side of the chain is shown.

TABLE 3. Comparative data for triazolite and chanabayaite.

Mineral	Triazolite	Chanabayaite
Formula	$NaCu_2(N_3C_2H_2)_2(NH_3)_2Cl_3 \cdot 4H_2O$	$Cu_4(N_3C_2H_2)_4(NH_3)_4Cl_2(Cl,OH)_2 \cdot H_2O$
Crystal system	Orthorhombic	Orthorhombic
Space group	$P2_12_12_1$	*Imma*
a, Å	19.3575	19.484
b, Å	7.15718	7.2136
c, Å	12.5020	11.999
V, Å^3	1732.09	1686.5
Z	4	2
Strongest lines	10.22 (97)	10.19 (100)
of the powder	6.135 (40)	6.189 (40)
X-ray diffraction	5.696 (17)	5.729 (23)
pattern:	5.182 (59)	5.216 (75)
d, Å (*I*, %)	5.119 (100)	4.964 (20)
	4.854 (19)	2.830 (20)
	3.752 (16)	2.611 (24)
	3.294 (18)	
	2.644 (17)	
	2.202 (18)	
Optical data	Biaxial (–)	Biaxial (–)
	$\alpha = 1.582$	$\alpha = 1.561$
	$\beta = 1.625$	$\beta = 1.615$
	$\gamma = 1.625$	$\gamma = 1.620$
	$2V \approx 0°$	$2V = 25°$
Density, g cm^{-3}		1.48 (meas.)
	2.028 (calc.)	1.464 (calc.)
Sources	This work	Chukanov *et al*. (2015*a*)

and transesterification of esters (Yang and Birman, 2009). Some transition elements (copper, zinc, iron, etc.) show high affinity for the triazolate ligand (Hasnoot, 2000). Usually triazolate complexes contain additional ligands that stabilize their crystal structures. In triazolite such ligands are NH_3^0 and Cl^-.

Triazolite is a supergene mineral formed in the contact zone between an altered guano deposit and chalcopyrite-bearing gabbro. Guano was the source of organic matter and nitrogen. Oxidized chalcopyrite was the source of Cu. The high affinity of Cu for N as a ligand, including N in the 1,2,4-triazolate anion and NH_3, promoted the formation of triazolite.

Triazolite is related structurally to chanabayaite (in part of the topology of the main structural unit containing zig-zag chains of the corner-sharing Cu-centred octahedra connected with isolated Cu-centred octahedra *via* 1,2,4-triazolate anions), which is a product of triazolite alteration as the result of dehydration and leaching of Na and a part of Cl (Zubkova *et al*., 2016). Comparative data for triazolite and chanabayaite are given in Table 3.

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