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Crystal structure and topological features of manganonaujakasite, a mineral with microporous heteropolyhedral framework related to AlPO-25 (ATV)

Sergey M. Aksenov[a,b,*], Nikita V. Chukanov[c], Igor V. Pekov[d,e], Ramiza K. Rastsvetaeva[b], Amy E. Hixon[a]

[a] Department of Civil and Environmental Engineering and Earth, Sciences, University of Notre Dame, Notre Dame, IN, 46556, United States
[b] Institute of Crystallography of Federal Scientific Research Centre, 'Crystallography and Photonics', Russian Academy of Sciences, Leninskii, Prospekt 59, Moscow, 119333, Russian Federation
[c] Institute of Problems of Chemical Physics, Russian Academy of Sciences, Chernogolovka, Moscow Region, 142432, Russian Federation
[d] Faculty of Geology, Moscow State University, Vorobievy Gory, Moscow, 119991, Russian Federation
[e] Vernadsky Institute of Geochemistry and Analytical Chemistry, Russian Academy of Sciences, Kosygina str. 19, 119991, Moscow, Russian Federation

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ABSTRACT

The crystal structure of manganonaujakasite was studied by single crystal X-ray analysis and infrared spectroscopy using a holotype sample with the empirical chemical formula $(Na_{5.96}Ca_{0.01})(Mn_{0.53}Fe_{0.49})Al_{3.95}Si_{8.03}O_{26.00}$. The monoclinic unit cell parameters are: $a = 15.029(1)$ Å, $b = 7.999(1)$ Å, $c = 10.467(1)$ Å, $\beta = 113.551(1)^{\circ}$; $V = 1153.47(15)$ Å^3, space group $C2/m$, $Z = 2$. The microporous crystal structure of manganonaujakasite is similar to that of naujakasite and is based on tetrahedral $[Al_4Si_8O_{26}]$-double layers which are linked *via* isolated $(Mn,Fe)O_6$-octahedra to form a heteropolyhedral framework containing two systems of parallel wide channels. The naujakasite-type framework topologically relates to the **ATV**-type of $AlPO_4$. Both are built from the same *t-kah* [6^3] and *t-lov* [$4^2.6^2$] tiles and are characterized by similar one-dimensional *composite building units* represented by the narsarsukite-type *nsc*-chain. The comparative data for the frameworks of naujakasite- and **ATV**-types are given.

1. Introduction

Zeolites compose an important class of inorganic crystalline materials that have been widely used in technology [1]. The modern topological analysis of zeolite structures [2] allows for prediction of new zeolite types based on the linkage of natural tiles, the smallest tetrahedral clusters, to form a framework structure [3]. However, this approach considers only “classic” zeolites which are tetrahedral frameworks whereas a large family of microporous materials demonstrates that frameworks can be built by TO_4 tetrahedra and MO_6 octahedra (*M* – predominantly transitional metals: Ti, Nb, Zr, Sn, Fe, Mn, etc.) [4–6]. Such heteropolyhedral zeolite-like materials are also characterized by many useful physical and chemical properties [7–9] and attract interest (especially titanium silicates) as ion-exchangers because of their efficient absorption of heavy elements, including toxic and radioactive pollutants (Cs^+, Pb^{2+}, Hg^{2+}, *etc.*), from aqueous solutions [10,11]. Moreover, silicates with heteropolyhedral frameworks are stable at high pressure [12–15] and could be used as a material for immobilization of nuclear waste.

Micropourous heteropolyhedral frameworks could be built by alternating multi-layered silicate packages with the octahedral parts to form 2D zeolites [16]. Among natural compounds [17], such microporous layered silicates have been found, for example, in the structures of rhodesite [18], delhayelite [19], günterblassite [20], naujakasite [21], and related minerals.

Manganonaujakasite, $Na_6(Mn,Fe)[Al_4Si_8O_{26}]$, the Mn-dominant analogue of naujakasite $Na_6Fe[Al_4Si_8O_{26}]$, was discovered by A.P. Khomyakov and co-authors in lovozerite-lomonosovite lujavrites on the Lovozero alkaline massif (Kola Peninsula, Russia). The following unit-cell parameters of a holotype sample were reported: $a = 15.033(3)$, $b = 8.001(1)$, $c = 10.478(2)$ Å, $\beta = 113.51(1)^{\circ}$; space group $C2/m$ [22]. However, there is no further information in the literature about the X-ray diffraction experiment and refinement procedure or data on atomic coordinates, atomic displacement parameters, and geometric characteristics of the structure.

In this paper we report the results of the crystal structure solution of manganonaujakasite, topological analysis of the framework, and detailed infrared (IR) spectroscopic data on naujakasite-type minerals.

* Corresponding author. Department of Civil and Environmental Engineering and Earth, Sciences, University of Notre Dame, Notre Dame, IN, 46556, United States.
E-mail address: aks.crys@gmail.com (S.M. Aksenov).

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2. Experimental

2.1. Samples

Fragments of a holotype specimen of manganonaujakasite deposited in the systematic collection of the Fersman Mineralogical Museum of the Russian Academy of Sciences, Moscow, Russia (catalogue no. 90284), were used for this study. The mineral forms light blue grains that are irregular in shape and up to 5 mm across. The empirical formula of the sample is $(Na_{5.96}Ca_{0.01})$ $(Mn_{0.53}Fe_{0.49})Al_{3.95}Si_{8.03}O_{26.00}$ [22].

Unaltered naujakasite and partly hydrated naujakasite were used for comparison. Both samples were collected by one of the coauthors (I.V.P.) in 1994 and originate from Hill 435, which is about 1 km southeast of the Tuperssuatsiait Bay in the southern part of the Ilímaussaq alkaline complex and located in Narsaq, Kujalleq municipality, South Greenland. Hydrated naujakasite forms peripheral zones (replacement rims) up to 1 mm thick of naujakasite crystals (0.5 × 1.5 × 2 cm) in porphyry-like naujakasite lujavrite. The inner parts of these crystals are unaltered. A detailed description of this locality and naujakasite-bearing rocks spread at Hill 435 was reported by Petersen & Andersen [23].

2.2. Single crystal X-ray diffraction analysis

Single crystal X-ray diffraction data for manganonaujakasite were collected at room temperature using a Bruker APEX II Quasar diffractometer with graphite-monochromated Mo*Kα* X-ray radiation using a ω-θ scanning mode. Data were integrated using the program SAINT and were then scaled, merged, and corrected for Lorentz, polarization and absorption effects using the SADABS package. The structure determination and refinement were carried out using the Jana2006 program package [24,25]. Atomic scattering factors for neutral atoms together with anomalous dispersion correction were taken from *International Tables for Crystallography* [26]. Illustrations were produced using Jana2006 in combination with DIAMOND [27]. Crystal data, data collection, and structure refinement details are summarized in Table 1. Bond-valence calculations (Table 2) were performed using bond-valence parameters taken from Brown & Altermatt [28] and Brese & O'Keeffe [29].

The final model included coordinates and anisotropic displacement parameters for all atoms. CCDC 1878306 contains supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

2.3. Infrared spectroscopy

In order to obtain infrared (IR) absorption spectra, powdered samples were mixed with dried KBr, pelletized, and analyzed using an ALPHA FTIR spectrometer (Bruker Optics) with a resolution of 4 cm^{-1} and 16 scans (Fig. 1). The IR spectrum of an analogues pellet of pure KBr was used as a reference.

3. Results and discussion

The microporous crystal structure of manganonaujakasite (Fig. 2) is similar to that of naujakasite [21,30] and is based on tetrahedral $[T_{12}O_{26}]$-double layers where $T = Si^{4+}$, Al^{3+}. These double layers are formed by the linking of two single *parent* layers representing the 6^3-type net topology (Fig. 3). Each layer consists of two types of six-membered rings characterized by the different orientations (up = ***u*** and down = ***d***) of TO_4-tetrahedra, which can be written as $(\boldsymbol{u^2d^4})_2(\boldsymbol{ud^2ud^2})$ [17,31]. Double layers are linked *via* isolated MO_6-octahedra

Table 1
Crystal data, data collection information, and structure refinement details for manganonaujakasite.

Crystal data	
Chemical formula	$Na_6\{(Mn_{0.53}Fe_{0.47})[Al_4Si_8O_{26}]\}$
M_r	941.9
Crystal system, space group	Monoclinic, *C2/m*
Temperature (K)	293
a, *b*, *c* (Å)	15.029(1), 7.999(1), 10.467(1)
β (°)	113.551(1)
V (Å^3)	1153.47(15)
Z	2
F(000)	927
D_x (Mg m^{-3})	2.712
Radiation type	Mo K_α
μ (mm^{-1})	1.42
Crystal size, mm	0.03 × 0.14 × 0.15
Data collection	
Diffractometer	Bruker Apex II Quasar
Scan method	ω-θ-scans
No. of measured, independent and observed reflections	7330, 1527, 1480
R_{int}	0.023
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.686
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.021, 0.065, 1.30
No. of reflections	1527
No. of parameters	113
$\Delta\rho_{max}$, $\Delta\rho_{max}$ (e Å^{-3})	0.26, −0.35

($M = Mn^{2+}$, Fe^{2+}) to form a heteropolyhedral framework. The main chemical feature of manganonaujakasite, distinguishing it from naujakasite, is the predominance of manganese over iron in the MO_6-polyhedron. The MO_6 polyhedra are characterized by high distortion with the elongated distances to apical vertexes, so that the coordination of the *M*-site is [4 + 2] tetragonal bipyramid.

The heteropolyhedral frameworks of manganonaujakasite and naujakasite contain two systems of parallel, wide channels running along *b* (Fig. 2). Channel **I** is delimited by eight-membered rings formed by six TO_4 tetrahedra and two MO_6 polyhedra with an effective channel width (*e.c.w.*) of ~7.24 × 1.58 Å [calculated according to McCusker et al. [32] by subtracting the ionic diameter of O^{2-} (2.7 Å) from the longest and shortest O···O distances across the channel]. Channel **II** is delimited by six-membered rings formed by six TO_4 tetrahedra with an *e.c.w.* of ~2.66 × 0.78 Å. Both channels are occupied by Na atoms. Na1 and Na2 sites are characterized by the typical coordination environments (*i.e.*, CNs are 6 and 7 for Na1 and Na2, respectively), while Na3 is placed in the center of square pyramid. The low CN of Na^+ at Na3 is unusual and is observed in a limited group of inorganic compounds [33–36].

Following the rules developed for the description of ordered microporous and mesoporous materials with inorganic hosts approved by International Zeolite Association, the crystal chemical formula should be written in the following order: |*guest composition*| [*host composition*] $_h\{$*dimensionality of the host* $\boldsymbol{D}^h\}$ $_p\{$*dimensionality of the pore system* $\boldsymbol{D}^p$ – *shape of the pore* $n_i^{m_i}$ – *direction of the channel* [*uvw*]$\}$ (*symmetry*) [32,37]. Consequently, the general crystal chemical formula of manganonaujakasite and related compounds (*i.e.*, naujakasite and products of its hydration) can be written as follows ($Z = 4$)

$$|^{[7]}A^+|\,[M^{[4+2]}T_{12}^{[4]}O_{26}]\,{}_h\{3\}\,{}_p\left\{\begin{matrix}1[3^35^16^{4/2}][010](6\text{−ring})\\1[3^35^16^38^{2/2}][010](8\text{−ring})\end{matrix}\right\}(C2/m),$$

indicating that (1) the guests are monovalent A^+ cations

Table 2
Bond valence calculation for manganonaujakasite.

Site	O1	O2	O3	O4	O5	O6	O7	O8	V_c
M				$0.43^{\times 2\rightarrow}$			$0.08^{\times 2\rightarrow}$		1.88
*T*1	$0.91_{\times 2\downarrow}$		1.00	1.15	0.97				4.03
*T*2		$0.86_{\times 2\downarrow}$	0.84			0.88		0.84	3.42
*T*3					0.83	0.87	$0.85_{\times 2\downarrow}$	0.85	3.40
Na1	0.04		$0.18^{\times 2\rightarrow}$	$0.27^{\times 2\rightarrow}$			0.08		1.02
Na2		0.17			$0.12^{\times 2\rightarrow}$			$(0.20 + 0.04)^{\times 2\rightarrow}$	0.89
Na3	0.28				$0.16^{\times 2\rightarrow}$	$0.21^{\times 2\rightarrow}$			1.02
V_a	2.14	1.89	2.02	1.85	2.08	1.96	1.86	1.93	

Note: In mixed sites, the bond-valence contribution of each cation has been weighted according to its occupancy.

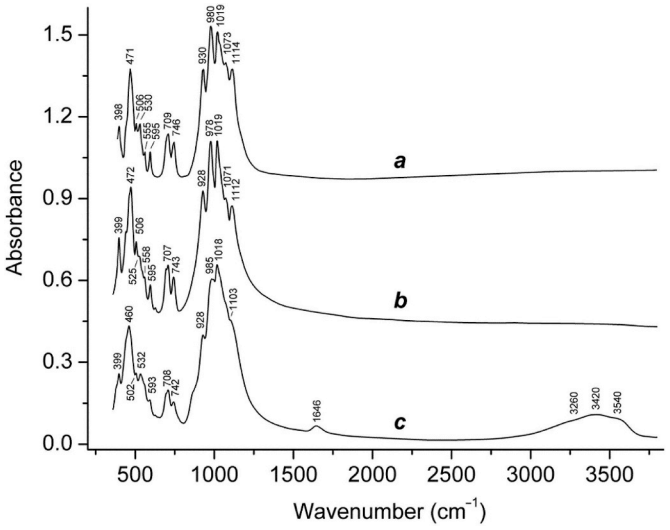

Fig. 1. Powder IR absorption spectra of (a) manganonaujakasite, (b) unaltered naujakasite, and (c) partly hydrated naujakasite.

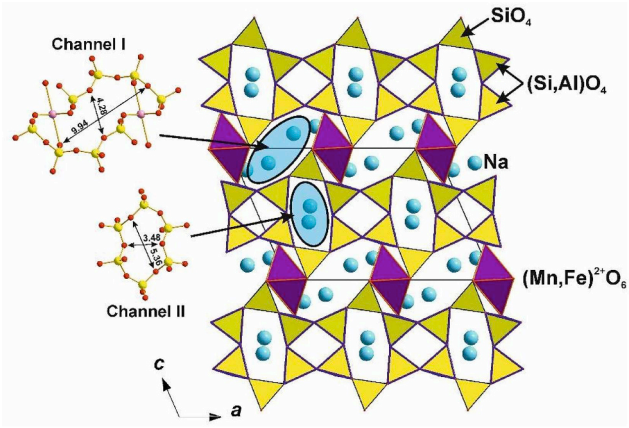

Fig. 2. The general view of the microporous crystal structure of manganonaujakasite.

(predominantly Na^+) and (2) the 3D host structure consists of two different unidimensional channels (both running along [010] direction) with the topology of $[3^35^16^{4/2}]$ [6-membered ring (6R) pore opening] and $[3^35^16^38^{2/2}]$ [8-membered ring (8R) pore opening], respectively.

Topological analysis of the naujakasite-type framework was performed based on a natural tiling analysis of the 3D cation nets [38,39] using the *ToposPro* software [2]. The framework of the naujakasite-type structure is characterized by the tile sequence $[6^3]_2[4^2.6^2][3^3.5.6^4][3^3.5.6^3.8^2]$ (Fig. 4) and topologically related to the **ATV**-type framework of $AlPO_4$ (AlPO-25, Fig. 5) [40] and its gallium-phosphate analogue [41]. The structure of AlPO-25 represents a tetrahedral framework (with tetrahedra occupied by aluminum and phosphorus) formed by the condensation of naujakasite-type double layers. The **ATV** is characterized by the following set of natural tilings: $[6^3]_3[4^2.6^2][6^5][6^4.8^2]$.

Both naujakasite- and **ATV**-type frameworks are built by the same *t-kah* $[6^3]$ and *t-lov* $[4^2.6^2]$ tiles and are characterized by similar one-dimensional *composite building units* (CBU) represented by the narsarsukite-type *nsc*-chain [42] or unbranched dreier double chain $\{\boldsymbol{uB}, 2^1_\infty\}$ $[T_8O_{20}]$, in accordance with the Liebau classification [43]. The calculated structural complexity parameter [44] for the naujakasite-type framework ($I_{G,\text{total}}$ = 136.131 bits/unit cell) is similar to that of **ATV** ($I_{G,\text{total}}$ = 102.117 bits/unit cell) [45]. The insertion of MO_6 octahedra into the tetrahedral framework between naujakasite-type double silicate layers leads to an increase of the framework density (FD); FDs are 22.54 $(M+T)/1000$ Å^3 and 19.9 $T/1000$ Å^3 for naujakasite and **ATV**, respectively. The comparative data for the frameworks of naujakasite and **ATV** are given in Table 3.

The IR spectrum of manganonaujakasite is similar to that of naujakasite (Fig. 1), which confirms the above conclusion that these minerals are isostructural. The assignment of absorption bands of these minerals was made in accordance with Chukanov & Chervonnyi [46]. Si—O stretching vibrations are observed in the range 900–1200 cm^{-1}, O—Si—O bending vibrations are observed in the range 700–800 cm^{-1}, and peaks in the range 450–600 cm^{-1} correspond to Si—O—Si bending and (Mn,Fe)—O stretching vibrations. Peaks below 450 cm^{-1} correspond to mixed lattice modes. In the IR spectrum of naujakasite the intensity of the 525–530 cm^{-1} band is significantly reduced with respect to the same band in manganonaujakasite. Based on this observation, the assignment of this band to stretching vibrations of the shortest Mn—O bond is most likely. In the IR spectrum of hydrated naujakasite additional bands of O—H stretching and H—O—H bending vibrations are observed (in the range 3200–3600 and at 1646 cm^{-1}, respectively). These bands can be assigned to both water molecules and hydronium ions.

The hydronium ions and water molecules most likely substitute for Na^+ in Na1 and Na3 sites which fill channel **I**, because its wide effective channel width creates the prerequisites for ion-exchange by the following schemes:

$$2Na^+ + O^{2-} \rightarrow Na^+ + H_3O^+ + O^{2-} \quad (1)$$

$$2Na^+ + O^{2-} \rightarrow Na^+ + H_2O^0 + OH^- \quad (2)$$

However, the Na1—O and Na3—O distances do not support the possibility such direct substitution. Therefore, the processes and products of naujakasite hydration requires further study and are the topic of current work.

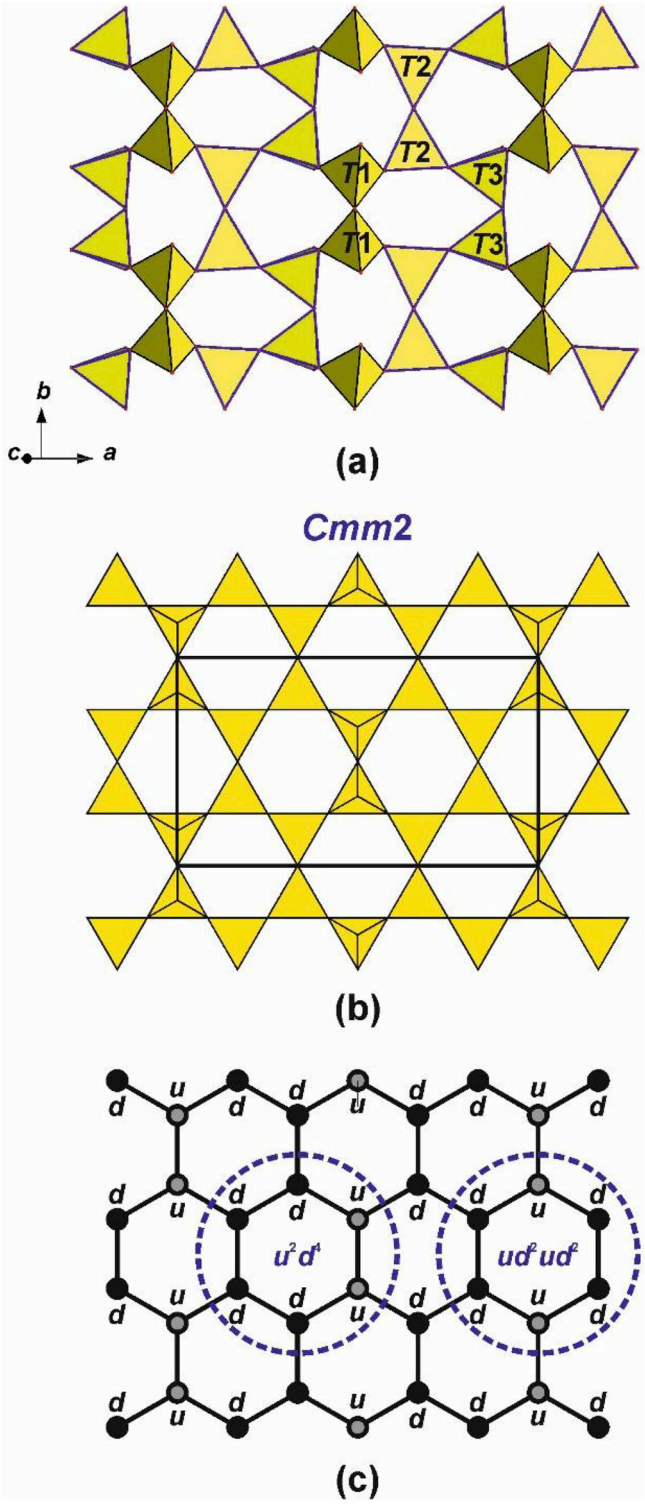

Fig. 3. A single *parent* layer of naujakasite-type double layer (a), the idealized view (b) and 2D graph showing the different orientation of the tetrahedra (c).

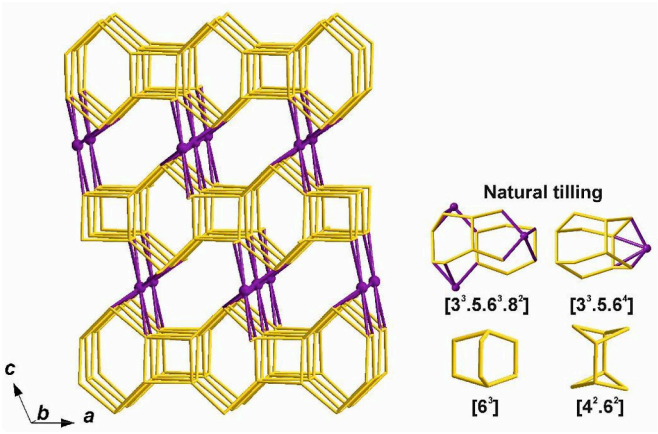

Fig. 4. Topological features of the naujakasite-type framework.

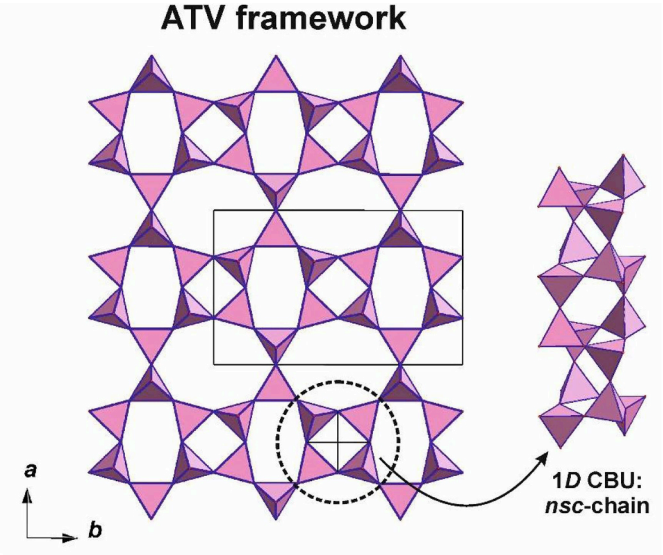

Fig. 5. General view of the $AlPO_4$ crystal structure.

Table 3
Comparative data for naujakasite- and **ATV**-type frameworks.

Parameter	Compound (type of framework)	
	Manganonaujakasite (naujakasite)	AlPO-25 (ATV)
Unit cell parameters (Å, °)	a = 15.029 b = 7.999; β = 113.551 c = 10.467	a = 8.408 b = 15.203 c = 9.449
Volume (Å^3)	1153.47	1207.84
Space group	*C2/m*	*Cmme*
FD [(T + M)/1000 Å^3)	22.54	19.9
ecw_{max} (Å)	7.24 × 1.58	4.9 × 3.0
Natural tiles	$[6^3]_2[4^2.6^2][3^3.5.6^4]$ $[3^3.5.6^3.8^2]$	$[6^3]_3[4^2.6^2][6^5]$ $[6^4.8^2]$
CBU	*nsc*-chain	*nsc*-chain
v (atoms)	39	36
I_G (bits/atom)	3.491	2.837
I_G (bits/unit cell)	136.131	102.117

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