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Garnet-type $\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$

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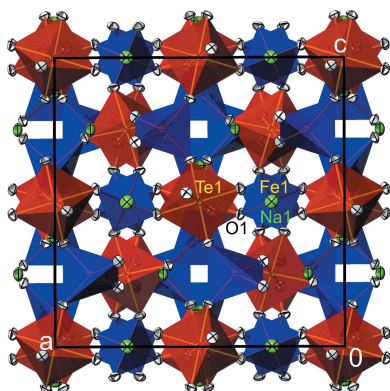
$\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$ or $\text{Na}_3\text{Te}_2\text{Fe}_3\text{O}_{12}$, trisodium ditellurium(VI) triiron(III) dodecaoxide, was obtained in the form of single-crystals under hydrothermal conditions. $\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$ adopts the garnet structure type in space group $Ia\bar{3}d$ and comprises one Na (multiplicity 24, Wyckoff letter *c*, site symmetry 2.22), one Te (16 *a*, $\bar{3}$), one Fe (24 *d*, $\bar{4}$.) and one O atom (96 *h*, 1) in the asymmetric unit. The three-dimensional framework structure is built of $[\text{TeO}_6]$ octahedra and $[\text{FeO}_4]$ tetrahedra by vertex-sharing. The larger Na^+ cations are situated in the interstices of the framework and are eightfold coordinated in the form of a distorted dodecahedron. Quantitative structural comparisons with isotypic $\text{Na}_3\text{Te}_2[(\text{Fe}_{0.5}\text{Al}_{0.5})\text{O}_4]_3$ and $\text{Na}_3\text{Te}_2(\text{GaO}_4)_3$ show a high degree of similarity between the three crystal structures.

1. Chemical context

Layered oxidotellurates(VI) comprising an alkali metal (or ammonium) and a transition metal *M*, such as $(\text{NH}_4)_4(\text{VO}_2)_2\text{Te}_2\text{O}_8(\text{OH})_2 \cdot 2\text{H}_2\text{O}$ (Nagarathinam *et al.*, 2022), $\text{Li}_2\text{Ni}_2\text{TeO}_6$ (Grundish *et al.*, 2019), $\text{Na}_3\text{Ni}_{1.5}\text{TeO}_6$ (Grundish *et al.*, 2020) or $\text{K}_2\text{M}_2\text{TeO}_6$ (*M* = Ni, Mg, Zn, Co, Cu; Masese *et al.*, 2018) are considered to be promising battery materials. In the quest for new representatives of this group of materials comprising K and Fe^{III} , we obtained a phase under hydrothermal conditions with a supposed composition of $\text{K}_{12}\text{Fe}^{\text{III}}_6\text{Te}^{\text{VI}}_4\text{O}_{27} \cdot 3\text{H}_2\text{O}$. However, this phase is not layered but crystallizes in a cubic framework structure with positionally disordered crystal water molecules [*Z* = 4, space group $I\bar{4}3d$, *a* = 14.7307 (12) Å at room temperature; Eder & Weil, 2023], which is closely related to the phase $\text{K}_{12+6x}\text{Fe}_6\text{Te}_{4-x}\text{O}_{27}$ [*x* = 0.222 (4), *Z* = 4, space group $I\bar{4}3d$, *a* = 14.7440 (10) Å at 100 K; Albrecht *et al.*, 2021]. With the intention of synthesizing the possible Na-analogue $\text{Na}_{12}\text{Fe}^{\text{III}}_6\text{Te}^{\text{VI}}_4\text{O}_{27} \cdot 3\text{H}_2\text{O}$, we obtained garnet-type $\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$ instead, and report here its crystal structure and quantitative comparisons with related crystal structures.

2. Structural commentary

The garnet supergroup has the general formula $\{X_3\}[Y_2](Z_3)\varphi_{12}$ and includes all phases, which crystallize isostructurally with garnet, regardless of the type of elements present at the four atomic sites (Grew *et al.*, 2013). The crystal structure of garnet comprises a three-dimensional framework built of $[Y\varphi_6]$ octahedra and $(Z\varphi_4)$ tetrahedra in which each octahedron is joined to six others through vertex-sharing tetrahedra. In turn, each tetrahedron shares its vertices with four octahedra, so that the composition of the framework is $Y_2Z_3\varphi_{12}$. Larger *X* atoms occupy positions in the interstices of



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Table 1

Selected bond lengths (Å) in related garnet-type $\text{Na}_3\text{Te}_2(\text{ZO}_4)_3$ oxidotellurates(VI) and their structure similarity parameters relative to $\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$.

	$\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$	$\text{Na}_3\text{Te}_2[(\text{Al,Fe})\text{O}_4]_3$	$\text{Na}_3\text{Te}_2(\text{GaO}_4)_3$
Na1—O1 (4×)	2.4208 (10)	2.396 (3)	2.3907 (17)
Na1—O1 (4×)	2.6226 (10)	2.597 (3)	2.5609 (17)
Te1—O1 (6×)	1.9169 (9)	1.914 (2)	1.9124 (17)
M1—O1 (4×)	1.8680 (9)	1.829 (2)	1.8405 (16)
Degree of lattice distortion, S		0.0064	0.0079
Atomic displacement of O1 ^a (Å)		0.0205	0.0322
Measure of similarity, Δ		0.001	0.002

Note: (a) The three other atomic sites do not show a displacement due to their site symmetries.

the framework and are eightfold coordinated in the form of a distorted dodecahedron (Wells, 1975). In a crystal-chemical sense, the final composition can therefore be expressed as $\{X_3\}^{[\text{8do}]}[Y_2]^{[\text{6ol}]}(Z_3^{[\text{4tl}]})\varphi_{12}$, or as $\{X_3\}^{[\text{8do}]}[Y_2]^{[\text{6ol}]}(Z^{[\text{4tl}]})\varphi_4$. In the title compound, Na takes the X position (multiplicity 24, Wyckoff letter c , site symmetry 2.22), Te the Y position (16 a , $\bar{3}$), Fe the Z position (24 d , $\bar{4}$.) and O the φ position (96 h , 1). The crystal structure of $\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$ is displayed in Fig. 1. Bond-valence sums (Brown, 2002) for all atoms were computed with the parameters of Brese & O’Keeffe (1991). The values (in valence units) of 1.19 for Na, 6.00 for Te, 2.98 for Fe and 2.04 for O are in very good agreement with the expected values of 1, 6, 3 and 2, respectively.

The garnet supergroup includes several chemical classes, which is also reflected by the high number of phases that adopt

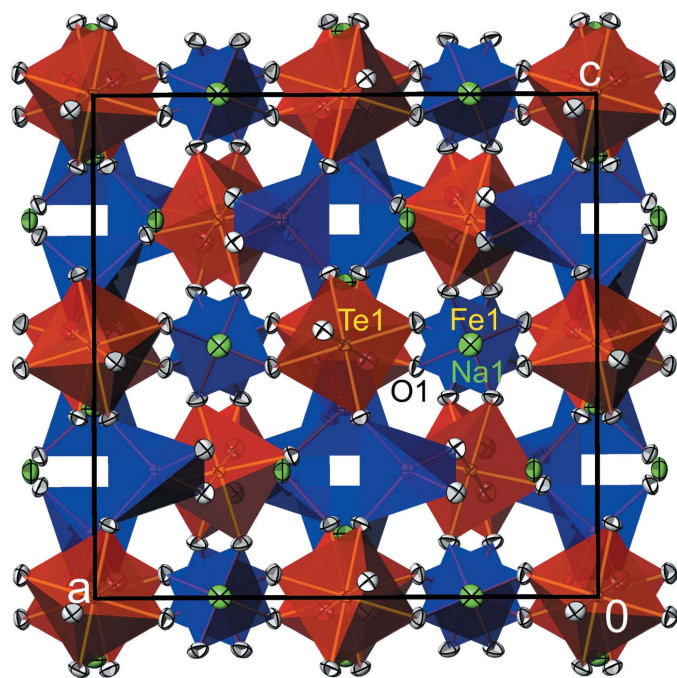


Figure 1

Projection of the garnet-type crystal structure of $\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$ along $[0\bar{1}0]$. Displacement ellipsoids are drawn at the 90% probability level. $[\text{TeO}_6]$ octahedra (red) and (FeO_4) tetrahedra (blue) are given in the polyhedral representation, Na atoms as green ellipsoids and O atoms as white ellipsoids.

the garnet structure type. A search in the ICSD (version 2022-1; Zagorac *et al.*, 2019), using the garnet structure type in space group $Ia\bar{3}d$ and with Si on the Z position as search field revealed about 420 entries, and with atoms other than Si on the Z position about 350 entries. With Te on the Y position, only five phases were found, including the mineral yafsoanite [ideally $\text{Ca}_3\text{Te}_2(\text{ZnO}_4)_3$, Jarosch & Zemann, 1989; Mills *et al.*, 2010], the Li-conducting $\text{Nd}_3(\text{Te}_{2-x}\text{Sb}_x)(\text{Li}_{3+x}\text{O}_4)_3$ ($x = 0.05, 0.10$) (O’Callaghan *et al.*, 2008), $\text{Na}_3\text{Te}_2[(\text{Fe}_{0.5}\text{Al}_{0.5})\text{O}_4]_3$ (Wedel & Sugiyama, 1999) and $\text{Na}_3\text{Te}_2(\text{GaO}_4)_3$ (Frau *et al.*, 2008). The latter two phases comprise Na on the X position and, with respect to the title compound, therefore are the chemically most related compounds. A comparison of relevant bond lengths in the three garnets, together with structural similarity parameters, as revealed by the program *compstru* (de la Flor *et al.*, 2016) available at the Bilbao Crystallographic Server (Aroyo *et al.*, 2006), is given in Table 1. The cations occupying the Z site apparently influence the two Na—O bond lengths in the crystal structures, although the ionic radii (Shannon, 1976) of Z do not directly correlate with this behaviour. The title compound with $Z = \text{Fe}$ (ionic radius 0.49 Å) has the longest Na—O bonds, followed by the mixed-occupied compound with $Z = (\text{Fe,Al})$ (averaged ionic radius 0.44 Å) and the compound with $Z = \text{Ga}$ (ionic radius 0.47 Å). On the other hand, the Te—O bond lengths in the three garnet structures are virtually identical.

An X-ray powder diffraction pattern of $\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$ has been deposited with the ICDD (PDF 00-048-0300; Gates-Rector & Blanton, 2019) without giving atomic coordinates for the O-atom site or displacement parameters for the atoms. The corresponding unit-cell parameter $a = 12.5257(1)$ Å determined from room-temperature powder X-ray measurement data is in very good agreement with the one from single-crystal data (Table 2). In the context of investigating the magnetic ordering of Fe^{III} on the Z sites, neutron powder data recorded at room temperature were also reported for $\text{Na}_3\text{Te}_2(\text{FeO}_4)_3$ (Plakhtii *et al.*, 1977).

3. Synthesis and crystallization

The solid educts $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, TeO_2 , H_6TeO_6 and NaOH were weighed in the molar ratios 2:1:2:15 and placed into a Teflon container (inner volume *ca* 5 ml). The container was filled to about 2/3 of its volume with water, closed with a

Table 2
Experimental details.

Crystal data	
Chemical formula	Na ₃ Te ₂ Fe ₃ O ₁₂
<i>M_r</i>	683.72
Crystal system, space group	Cubic, <i>Ia</i> $\bar{3}$ <i>d</i>
Temperature (K)	296
<i>a</i> (Å)	12.5276 (9)
<i>V</i> (Å ³)	1966.1 (4)
<i>Z</i>	8
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	10.39
Crystal size (mm)	0.06 × 0.06 × 0.06
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.677, 0.748
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	42303, 569, 446
<i>R_{int}</i>	0.060
(sin θ /λ) _{max} (Å ⁻¹)	0.934
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.017, 0.041, 1.16
No. of reflections	569
No. of parameters	18
Δρ _{max} , Δρ _{min} (e Å ⁻³)	1.25, -0.68

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *ATOMS for Windows* (Dowty, 2006) and *pubCIF* (Westrip, 2010).

Teflon lid and embedded into a steel autoclave. The hydrothermal experiment was conducted at 473 K for five days. The solid product was filtered off, washed with water and ethanol and dried in air. It consisted of light-brown microcrystalline material and a few amber-coloured cuboid crystals of Na₃Te₂(FeO₄)₃, as well as a very few small yellowish platy crystals of an unknown phase. Preliminary single-crystal measurements of the latter indicated a unit cell with hexagonal metrics (*a* = 5.252, *c* = 15.724 Å) and obvious twinning, which has precluded a structure solution so far. Similar metrics were found for Na₂GeTeO₆ (Woodward *et al.*, 1998). The powder X-ray diffraction pattern of the bulk revealed Na₃Te₂(FeO₄)₃ as a side product and the unknown phase (assuming a close relation with Na₂GeTeO₆) as the main phase, in an approximate mass ratio of 0.15:0.85.

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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supporting information

Acta Cryst. (2023). E79, 328-330 [https://doi.org/10.1107/S2056989023002293]

Garnet-type Na₃Te₂(FeO₄)₃

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Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINTE* (Bruker, 2016); data reduction: *SAINTE* (Bruker, 2016); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *ATOMS for Windows* (Dowty, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

Trisodium ditellurium(VI) triiron(III) dodecaoxide

Crystal data

Na₃Te₂Fe₃O₁₂
M_r = 683.72
 Cubic, *Ia3d*
a = 12.5276 (9) Å
V = 1966.1 (4) Å³
Z = 8
F(000) = 2488
D_x = 4.620 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å
 Cell parameters from 6128 reflections
 θ = 4.0–41.1°
 μ = 10.39 mm⁻¹
T = 296 K
 Cube, amber
 0.06 × 0.06 × 0.06 mm

Data collection

Bruker APEXII CCD
 diffractometer
 ω- and φ-scans
 Absorption correction: multi-scan
 (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.677, *T_{max}* = 0.748
 42303 measured reflections

569 independent reflections
 446 reflections with *I* > 2σ(*I*)
R_{int} = 0.060
 θ_{max} = 41.6°, θ_{min} = 4.0°
h = -23→23
k = -23→23
l = -23→23

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2σ(*F*²)] = 0.017
wR(*F*²) = 0.041
S = 1.16
 569 reflections
 18 parameters
 0 restraints

w = 1/[σ²(*F_o*²) + (0.0169*P*)² + 1.9053*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 1.25 e Å⁻³
 Δρ_{min} = -0.68 e Å⁻³
 Extinction correction: *SHELXL-2019/2*
 (Sheldrick 2015b),
*F_c** = *kF_c*[1 + 0.001*xF_c*²λ³/sin(2θ)]^{-1/4}
 Extinction coefficient: 0.00158 (6)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Na1	0.250000	0.375000	0.500000	0.01227 (18)
Te1	0.500000	0.500000	0.500000	0.00480 (5)
Fe1	0.250000	0.625000	0.500000	0.00637 (7)
O1	0.35650 (7)	0.53021 (8)	0.45633 (8)	0.00976 (16)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0150 (3)	0.0068 (4)	0.0150 (3)	0.000	0.0012 (4)	0.000
Te1	0.00480 (5)	0.00480 (5)	0.00480 (5)	0.00038 (3)	0.00038 (3)	0.00038 (3)
Fe1	0.00652 (9)	0.00605 (14)	0.00652 (9)	0.000	0.000	0.000
O1	0.0068 (4)	0.0106 (4)	0.0119 (4)	0.0023 (3)	-0.0013 (3)	-0.0006 (3)

Geometric parameters (\AA , $^\circ$)

Na1—O1 ⁱ	2.4208 (10)	Te1—O1 ^{viii}	1.9169 (9)
Na1—O1 ⁱⁱ	2.4208 (10)	Te1—O1 ^{ix}	1.9169 (9)
Na1—O1 ⁱⁱⁱ	2.4208 (10)	Te1—O1 ^x	1.9169 (9)
Na1—O1	2.4208 (10)	Te1—O1 ^{vii}	1.9169 (9)
Na1—O1 ^{iv}	2.6226 (10)	Te1—O1 ^{xi}	1.9169 (9)
Na1—O1 ^v	2.6226 (10)	Fe1—O1 ^{xii}	1.8680 (9)
Na1—O1 ^{vi}	2.6226 (10)	Fe1—O1 ⁱⁱ	1.8680 (9)
Na1—O1 ^{vii}	2.6226 (10)	Fe1—O1 ^{xiii}	1.8680 (9)
Te1—O1	1.9169 (9)	Fe1—O1	1.8680 (9)
O1 ⁱ —Na1—O1 ⁱⁱ	153.42 (4)	O1—Te1—O1 ^{viii}	91.50 (4)
O1 ⁱ —Na1—O1 ⁱⁱⁱ	73.12 (4)	O1—Te1—O1 ^{ix}	88.50 (4)
O1 ⁱⁱ —Na1—O1 ⁱⁱⁱ	113.33 (4)	O1 ^{viii} —Te1—O1 ^{ix}	180.0
O1 ⁱ —Na1—O1	113.33 (4)	O1—Te1—O1 ^x	91.50 (4)
O1 ⁱⁱ —Na1—O1	73.12 (4)	O1 ^{viii} —Te1—O1 ^x	88.50 (4)
O1 ⁱⁱⁱ —Na1—O1	153.42 (4)	O1 ^{ix} —Te1—O1 ^x	91.50 (4)
O1 ⁱ —Na1—O1 ^{iv}	125.56 (2)	O1—Te1—O1 ^{vii}	88.50 (4)
O1 ⁱⁱ —Na1—O1 ^{iv}	77.63 (3)	O1 ^{viii} —Te1—O1 ^{vii}	91.50 (4)
O1 ⁱⁱⁱ —Na1—O1 ^{iv}	63.92 (4)	O1 ^{ix} —Te1—O1 ^{vii}	88.50 (4)
O1—Na1—O1 ^{iv}	94.14 (3)	O1 ^x —Te1—O1 ^{vii}	180.0
O1 ⁱ —Na1—O1 ^v	94.14 (3)	O1—Te1—O1 ^{xi}	180.0
O1 ⁱⁱ —Na1—O1 ^v	63.92 (4)	O1 ^{viii} —Te1—O1 ^{xi}	88.50 (4)
O1 ⁱⁱⁱ —Na1—O1 ^v	77.63 (3)	O1 ^{ix} —Te1—O1 ^{xi}	91.50 (4)
O1—Na1—O1 ^v	125.56 (2)	O1 ^x —Te1—O1 ^{xi}	88.50 (4)

O1 ^{iv} —Na1—O1 ^v	106.99 (4)	O1 ^{vii} —Te1—O1 ^{xi}	91.50 (4)
O1 ⁱ —Na1—O1 ^{vi}	63.92 (4)	O1 ^{xii} —Fe1—O1 ⁱⁱ	113.83 (3)
O1 ⁱⁱ —Na1—O1 ^{vi}	94.14 (3)	O1 ^{xii} —Fe1—O1 ^{xiii}	101.06 (6)
O1 ⁱⁱⁱ —Na1—O1 ^{vi}	125.56 (2)	O1 ⁱⁱ —Fe1—O1 ^{xiii}	113.83 (3)
O1—Na1—O1 ^{vi}	77.63 (3)	O1 ^{xii} —Fe1—O1	113.83 (3)
O1 ^{iv} —Na1—O1 ^{vi}	169.86 (4)	O1 ⁱⁱ —Fe1—O1	101.06 (6)
O1 ^v —Na1—O1 ^{vi}	73.94 (4)	O1 ^{xiii} —Fe1—O1	113.83 (3)
O1 ⁱ —Na1—O1 ^{vii}	77.63 (3)	Fe1—O1—Te1	135.38 (5)
O1 ⁱⁱ —Na1—O1 ^{vii}	125.56 (2)	Fe1—O1—Na1	92.91 (4)
O1 ⁱⁱⁱ —Na1—O1 ^{vii}	94.14 (3)	Te1—O1—Na1	107.08 (4)
O1—Na1—O1 ^{vii}	63.92 (4)	Fe1—O1—Na1 ^{ix}	116.33 (4)
O1 ^{iv} —Na1—O1 ^{vii}	73.94 (4)	Te1—O1—Na1 ^{ix}	99.78 (4)
O1 ^v —Na1—O1 ^{vii}	169.86 (4)	Na1—O1—Na1 ^{ix}	98.95 (3)
O1 ^{vi} —Na1—O1 ^{vii}	106.99 (4)		

Symmetry codes: (i) $-z+3/4, -y+3/4, -x+3/4$; (ii) $-x+1/2, y, -z+1$; (iii) $z-1/4, -y+3/4, x+1/4$; (iv) $y-1/4, -x+3/4, z+1/4$; (v) $-z+1/2, x, -y+1$; (vi) $-y+3/4, -x+3/4, -z+3/4$; (vii) z, x, y ; (viii) $-y+1, -z+1, -x+1$; (ix) y, z, x ; (x) $-z+1, -x+1, -y+1$; (xi) $-x+1, -y+1, -z+1$; (xii) $-z+3/4, -y+5/4, x+1/4$; (xiii) $z-1/4, -y+5/4, -x+3/4$.