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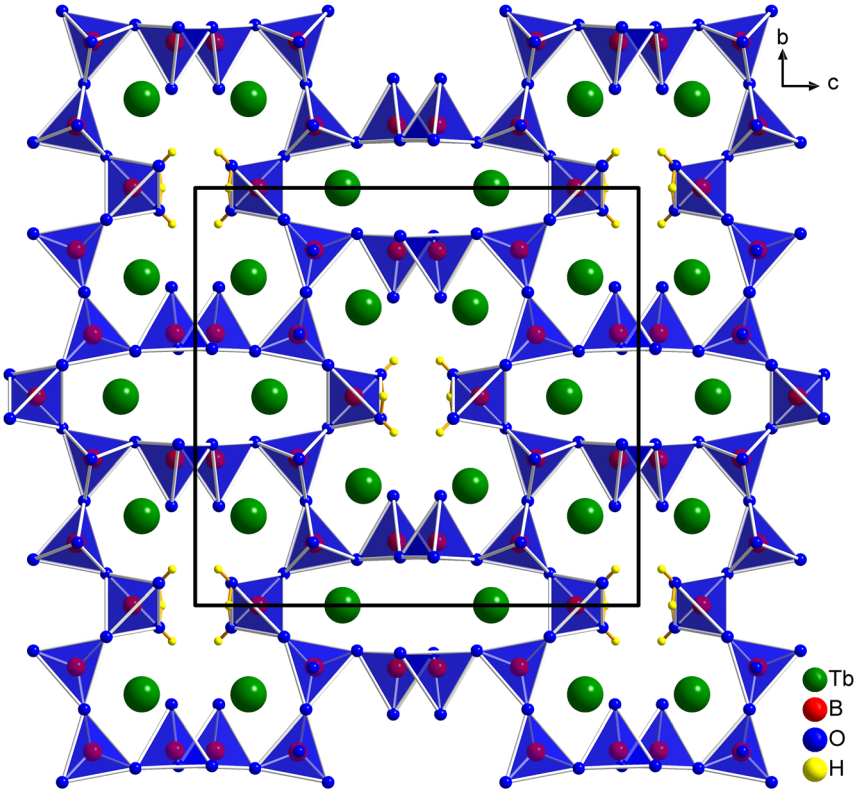
The crystal structure of $Tb_3B_5O_{11}(OH)_2$



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Abstract

$Tb_3B_5O_{11}(OH)_2$, orthorhombic, *Pnna* (no. 52), a = 4.4471(1) Å, b = 13.0934(4) Å, c = 13.9055(3) Å, V = 809.68(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0260, $wR_{ref}(F^2)$ = 0.0556, T = 301(1) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.02 × 0.02 × 0.02 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	26.0 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Quest Photon III C14, φ and ω
θ_{max}, completeness:	35.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,730, 1786, 0.053
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1169
$N(param)_{refined}$:	101
Programs:	SHELX [1, 2], Olex2 [3], Diamond [4]

1 Source of material

$Tb_3B_5O_{11}(OH)_2$ was obtained as a side phase during related experiments, investigating the system Tb_4O_7–CuO–B_2O_3. Small colorless crystals were found within the blue main product, which was obtained after the following procedure. Tb_4O_7 (0.06 mmol, Auer–Remy KG, 99.9 %), CuO (0.11 mmol, ChemPur, 99 %), and B_2O_3 (0.46 mmol, Alfa

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Tb1	0.24656 (7)	0.78626 (2)	0.12047 (2)	0.00698 (7)
Tb2	0.250000	0.500000	0.16760 (2)	0.00493 (7)
B1	0.7597 (16)	0.6513 (4)	0.0458 (3)	0.0055 (8)*
B2	0.7346 (17)	0.6483 (4)	0.2313 (4)	0.0061 (9)
B3	0.750000	0.500000	0.3579 (5)	0.0050 (11)
O1	0.7504 (10)	0.7617 (3)	0.0534 (2)	0.0059 (6)
O2	0.0805 (9)	0.6150 (3)	0.0377 (3)	0.0081 (7)
O3	0.6292 (9)	0.6092 (3)	0.1351 (3)	0.0074 (7)
O4	0.0526 (9)	0.6499 (3)	0.2343 (3)	0.0066 (7)
O5	0.5833 (9)	0.5757 (3)	0.2997 (3)	0.0064 (7)
O6	0.5897 (12)	0.750000	0.250000	0.0058 (9)
O7	0.5222 (10)	0.4472 (3)	0.4179 (3)	0.0101 (8)
H7A[a]	0.42 (5)	0.499 (11)	0.424 (17)	0.10 (10)*
H7B[a]	0.40 (2)	0.414 (10)	0.448 (9)	0.01 (4)*

[a]Occupancy: 0.5.

Aesar, 99.9 %) were ground in an agate mortar. The mixture was placed in a platinum capsule and centered in an 14/8 assembly of a modified Walker type multianvil apparatus [5–7]. 11 GPa of pressure were built up within 267 min. Subsequently, the temperature was raised to 700 °C within 10 min and kept constant for 30 min. The temperature was then incrementally lowered to 200 °C within 75 min, where the reaction was quenched. Finally, the pressure was released within 800 min and the sample was recovered from the platinum capsule.

2 Experimental details

Refinements of the crystal structure were carried out in space group *Pnna* (no. 52). Bond valence sums indicated protonation of the O7 atom. As a direct consequence of the symmetry of the crystal structure, a split-site H7a and H7b with occupation factors 0.5 each is necessary for a reasonable description of the hydrogen atoms. Interatomic OH-distances were fixed at 0.83 Å using DFIX records. Two reflections were omitted from the refinement due to their high deviation.

3 Comment

Terbium is the second lanthanoid displaying a structure type recently reported for $Nd_3B_5O_{11}(OH)_2$ [8]. The crystal structure consists of a three-dimensional framework of corner-sharing [BO_4] tetrahedra forming corrugated 10- and 14-membered rings in the *bc*-plane, and eight-membered rings in the *ac*-plane. Different coordination numbers are observed for the two crystallographically different Tb-sites (Tb1: *CN* = 10; Tb2: *CN* = 8). The H7-split-site can be interpreted as a hydroxide-group, forming a hydrogen-bond to another O7 atom, acting as the acceptor.

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