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# Rietveld refinement of the mixed boracite $Fe_{1.59}Zn_{1.41}B_7O_{13}Br$

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Key indicators: powder X-ray study; $T$ = 300 K; mean $\sigma$(O–B) = 0.014 Å; disorder in main residue; $R$ factor = 0.018; $wR$ factor = 0.025; data-to-parameter ratio = 22.1.

The structural characterization of the new iron–zinc heptaborate bromide with composition $Fe_{1.59}Zn_{1.41}B_7O_{13}Br$, prepared by chemical transport is reported. A rigid-body model with constrained generalized coordinates was defined in order to hold the positions of the B atoms at reasonable interatomic distances that typically would reach unacceptable values because of the weak scattering power of boron. There are three independent sites for the B atoms of which two are tetrahedrally coordinated. The bond-valence sum around the third B atom, located on a threefold rotation axis, was calculated considering two cases of coordination of boron with oxygens: trigonal-planar and tetrahedral. The contribution of the fourth O atom to the bond-valence sum was found to be only 0.06 v.u., indicating the presence of a very weak bond in the right position to have a distorted tetrahedral coordination in favour of the trigonal-planar coordination for the third B atom. X-ray fluorescence (XRF) was used to determinate the Fe/Zn ratio.

## Related literature

The method of preparation was based on Schmid (1965). For related structures, see: Mao *et al.* (1991); Dowty & Clark (1972, 1973); Mendoza-Alvarez *et al.* (1985); Schindler & Hawthorne (1998); Knorr *et al.* (2007). For properties and potential applications of boracites, see: Campa-Molina *et al.* (1994, 2002); Dana (1951); Mathews *et al.* (1997); Smart & Moore (1992). For bond-valence parameters for oxides, see: Brese & O'Keeffe (1991).

## Experimental

### Crystal data

$Fe_{1.59}Zn_{1.41}B_7O_{13}Br$
$M_r$ = 544.65
Trigonal, $R3c$
$a$ = 8.6081 (1) Å
$c$ = 21.0703 (3) Å
$V$ = 1352.12 (3) Å$^3$
$Z$ = 6
Cu $K\alpha$ radiation
$T$ = 300 K
Specimen shape: irregular
20 × 20 × 0.2 mm
Specimen prepared at 1173 K
Particle morphology: irregular, pale pink

### Data collection

Bruker D8 Advance diffractometer
Specimen mounting: packed powder sample container
Specimen mounted in reflection mode
Scan method: step
$2\theta_{min}$ = 8.1, $2\theta_{max}$ = 110.0°
Increment in $2\theta$ = 0.02°

### Refinement

$R_p$ = 0.018
$R_{wp}$ = 0.025
$R_{exp}$ = 0.014
$R_B$ = 0.06
$S$ = 1.89
Profile function: pseudo-Voigt modified by Thompson *et al.* (1987)
397 reflections
18 parameters

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Zn—Br | 2.680 (3) | B1—O4$^{vii}$ | 1.451 (13) |
| Zn—Br$^{i}$ | 3.412 (1) | B1—O5$^{vi}$ | 1.49 (3) |
| Zn—O2$^{ii}$ | 2.130 (4) | B2—O1 | 1.566 (3) |
| Zn—O3$^{iii}$ | 2.081 (7) | B2—O3$^{viii}$ | 1.452 (8) |
| Zn—O4$^{iv}$ | 2.035 (4) | B2—O4 | 1.463 (18) |
| Zn—O5$^{v}$ | 2.012 (7) | B2—O5 | 1.453 (17) |
| B1—O2$^{vi}$ | 1.506 (14) | B3—O2 | 1.397 (14) |
| B1—O3$^{vii}$ | 1.48 (2) | B3—O1 | 2.38 (3) |
| O2—B3—O2$^{vii}$ | 119.9 (9) | | |

Symmetry codes: (i) $-y+\frac{1}{3}, -x+\frac{2}{3}, z+\frac{1}{6}$; (ii) $-x+y+\frac{1}{3}, y+\frac{2}{3}, z+\frac{1}{6}$; (iii) $-y+\frac{2}{3}, x-y+\frac{1}{3}, z+\frac{1}{3}$; (iv) $x, x-y, z+\frac{1}{2}$; (v) $x-\frac{1}{3}, y+\frac{1}{3}, z+\frac{1}{3}$; (vi) $-y-\frac{1}{3}, -x+\frac{1}{3}, z-\frac{1}{6}$; (vii) $-y, x-y, z$; (viii) $-x+y, -x, z$.

Data collection: *DIFFRAC/AT* (Siemens, 1993); cell refinement: *FULLPROF* (Rodríguez-Carvajal, 2006; Rodriguez & Rodriguez-Carvajal, 1997, a strongly modified version of that described by Wiles & Young, 1981); data reduction: *FULLPROF*; method used to solve structure: coordinates were taken from an isotypic compound (Mao *et al.*, 1991); program(s) used to refine structure: *FULLPROF*; software used to prepare material for publication: *DIAMOND*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2119).