Trisodium Dicalcium Bismuth Hexaoxide

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Key indicators
Single-crystal X-ray study
T = 294 K
Mean |–O| = 0.002 Å
R factor = 0.013
wR factor = 0.032
Data-to-parameter ratio = 15.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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Single crystals of the title compound, Na₃Ca₂BiO₆, were grown from a high-temperature reactive flux solution of Na₂CO₃. Na₃Ca₂BiO₆ crystallizes as an ordered rock-salt structure (space group Fddd), in which the octahedral holes in the oxide array are filled by an ordered 3:2:1 arrangement of Na⁺, Ca²⁺ and Bi⁵⁺ cations. All atoms except for one O atom lie on special positions; site symmetries are as follows: Bi 222, Ca 2, Na 222 and O 2.

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Comment

The most common bismuth oxidation state found in oxides is Bi³⁺ as, for example, in BiNbO₄ (Keve et al., 1973) and Bi₂MoO₆ (Teller et al., 1984). However, some oxides, including NaBiO₃ (Kumada et al., 2000), KBiO₃ (Nguyen et al., 1993), Li₄Sr₂BiO₆, Na₄Sr₂BiO₆, Li₆KBiO₆, Li₆RbBiO₆ and Li₂Ba₅Bi₂O₁₁ (Carlson et al., 1992) contain Bi(V) cations.

Compound (I) also possesses a fully ordered arrangement of Na⁺, Ca²⁺ and Bi⁵⁺ cations (Fig. 1). The metal-oxygen bond distances (Table 1) are normal and the octahedra are close to

Figure 1

The asymmetric unit of (I), expanded to show the metal coordination polyhedra. Displacement ellipsoids are drawn at the 75% probability level. Colour key: Ca yellow, Bi blue, Na green and O red. [Symmetry codes: (i) 4/-x, 4/-y, 4/-z; (ii) x, y, z; (iii) x, y, z; (iv) 4/-x, 4/-y, 4/-z; (v) 4/-x, 4/-y, 4/-z; (vi) 4/-x, 4/-y, 4/-z; (vii) 4/-x, 4/-y, 4/-z; (viii) 4/-x, 4/-y, 4/-z; (ix) x, y, z; (x) x, y, z; (xi) x, y, z; (xii) 4/-x, 4/-y, 4/-z; (xiii) 4/-x, 4/-y, 4/-z; (xiv) 4/-x, 4/-y, 4/-z; (xv) 4/-x, 4/-y, 4/-z; (xvi) 4/-x, 4/-y, 4/-z.]

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regular. The rock-salt-type structure contains edge- and corner-sharing NaO$_6$, CaO$_6$ and BiO$_6$ octahedra (Fig. 2), ordered so that the calcium and bismuth octahedra share an edge.

**Experimental**

Bi$_2$O$_3$ (Alfa Aesar, 99.975%, 2.0 mmol) and CaCO$_3$ (Alfa Aesar, 99.95%, 1.0 mmol) were ground under acetone in an agate mortar until dry. The mixture, along with excess Na$_2$CO$_3$ (Fisher, ACS reagent, 12.5 g), was loaded into an alumina crucible, covered with an alumina lid, and placed into a programmable tube furnace. The system was heated to 1323 K at a rate of 873 K h$^{-1}$ and held at the target temperature for 24 h. It was then cooled slowly to 1073 K at a rate of 15 K h$^{-1}$ and held at that temperature for 1 h, at which point the furnace was shut off and the reaction allowed to cool to room temperature. The excess flux was dissolved in water and yellow transparent crystals of (I) were isolated using sonication and vacuum filtration.

**Crystal data**

Na$_3$Ca$_2$BiO$_6$

V = 1287.1 (3) Å$^3$

Z = 8

Mo $K\alpha$ radiation

$\mu$ = 29.17 mm$^{-1}$

$T$ = 294 (2) K

0.05 x 0.04 x 0.03 mm

**Data collection**

Bruker SMART APEX CCD diffractometer

Absorption correction: multi-scan (SADABS, Bruker, 2003)

$T_{\text{min}}$ = 0.778, $T_{\text{max}}$ = 1.000

(expected range = 0.324-0.417)

5827 measured reflections

499 independent reflections

414 reflections with $I > 2\sigma(I)$

$R_{\text{int}}$ = 0.047

**Refinement**

$R[F^2 > 2\sigma(F^2)]$ = 0.013

$wR[F^2]$ = 0.032

$S$ = 1.08

32 parameters

$\Delta\rho_{\text{max}}$ = 0.92 e Å$^{-3}$

$\Delta\rho_{\text{min}}$ = -0.65 e Å$^{-3}$

**Table 1**

Selected bond lengths (Å).

<table>
<thead>
<tr>
<th></th>
<th>B1–O1$^i$</th>
<th>Na1–O1</th>
<th>Na2–O1$^{iv}$</th>
<th>Ca1–O1$^{iv}$</th>
<th>Na2–O2$^i$</th>
<th>Ca1–O2$^{iv}$</th>
<th>Na1–O2$^i$</th>
<th>Na2–O2$^{iv}$</th>
</tr>
</thead>
<tbody>
<tr>
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<td>2.117 (3)</td>
<td>2.696 (3)</td>
<td>2.4305 (13)</td>
<td>2.3040 (7)</td>
<td>2.458 (3)</td>
<td>2.533 (3)</td>
<td>2.398 (2)</td>
<td>2.458 (3)</td>
</tr>
</tbody>
</table>

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

Data collection: SMART-NT (Bruker, 2003); cell refinement: SAINT-Plus-NT (Bruker, 2003); data reduction: SAINT-Plus-NT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXL (Sheldrick, 2001); software used to prepare material for publication: SHELXL.

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**References**


