Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Crystal growth and redetermination of strontium nitride iodide, Sr_2NI

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Received 8 August 2007; accepted 10 August 2007

Key indicators: single-crystal X-ray study; T = 150 K; mean σ() = 0.000 Å; R factor = 0.061; wR factor = 0.192; data-to-parameter ratio = 18.3.

Single crystals of Sr_2NI have been grown for the first time. Redetermination from single-crystal X-ray diffraction data confirms the anti-α-$NaFeO_2$ structure, previously found from powder diffraction data [Bowman, Smith & Gregory (2006). *J. Solid State Chem.* **179**, 130–139]. Iodide ions occupy octahedral voids between layers of edge-sharing NSr_6 octahedra.

Related literature

For related literature, see: Andersson (1970); Brese & O'Keeffe (1990); Bowman *et al.* (2001, 2005, 2006); Ehrlich & Deissmann (1958); Ehrlich *et al.* (1964, 1971); Hadenfeldt & Herdejürgen (1987, 1988); Emons *et al.* (1964, 1968); Jack *et al.* (2005); Nicklow *et al.* (2001); Reckeweg & DiSalvo (2002); Rietschel & Baernighausen (1969); Sebel & Wagner (2004); Wagner (2002).

Experimental

Crystal data

$INSr_2$
M_r = 316.15
Trigonal, $R\bar{3}m$
a = 4.0049 (6) Å
c = 23.055 (7) Å
V = 320.24 (12) Å^3
Z = 3
Mo $K\alpha$ radiation
μ = 31.99 mm^{-1}
T = 150 (2) K
0.14 × 0.04 × 0.04 mm

Data collection

Bruker SMART1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*TWINABS*; Bruker, 2007)
T_{min} = 0.175, T_{max} = 0.278
682 measured reflections
165 independent reflections
163 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.192
S = 1.21
165 reflections
9 parameters
$\Delta\rho_{max}$ = 1.77 e Å^{-3}
$\Delta\rho_{min}$ = −1.76 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* and *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ATOMS* (Dowty, 1998); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004) and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MG2033).

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supplementary materials

Acta Cryst. (2007). E**63**, i177 [**doi:10.1107/S1600536807039797**]

Crystal growth and redetermination of strontium nitride iodide, Sr_2NI

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Comment

Sr_2NI belongs to the A_2NX (A = Ca—Ba, *X* = F—I) family of compounds which were originally synthesized several decades ago (Ehrlich & Deissmann, 1958; Ehrlich *et al.*, 1964; Emons *et al.*, 1964; Emons *et al.*, 1968; Andersson, 1970; Ehrlich *et al.*, 1971). It has only been recently that the structures and properties of these compounds have been revealed (Hadenfeldt & Herdejürgen, 1987; Hadenfeldt & Herdejürgen, 1988; Bowman *et al.*, 2001; Nicklow *et al.*, 2001; Reckeweg & DiSalvo, 2002; Wagner, 2002; Sebel & Wagner, 2004; Bowman *et al.*, 2005; Jack *et al.*, 2005; Bowman *et al.*, 2006). With the exception of Ca_2NX (*X* = Cl, Br, I) (Hadenfeldt & Herdejürgen, 1987; Hadenfeldt & Herdejürgen, 1988), single-crystal determinations have been restricted to the lighter halide (*X* = F) members. These single-crystal determinations have often shown the structures to exhibit important differences to those refined from powder data. We recently investigated the structure of Sr_2NI using powder X-ray diffraction (Bowman *et al.*, 2006). Now the successful and unprecedented growth of single crystals of a strontium nitride halide (Sr_2NX) phase has allowed the single-crystal structure of Sr_2NI to be determined precisely for the first time. Importantly, this study has allowed an accurate determination of the Sr position within the rhombohedral cell and describes well defined thermal parameters which are elongated only slightly along the *c* direction for Sr and I and very close to isotropic for N.

The data (obtained at 150 K) show that Sr_2NI crystallizes in space group *R*-3 m (No. 166). The structure consists of $[NSr_2]^+$ slabs in which N is coordinated octahedrally to six Sr atoms. The layers of the edge-sharing NSr_6 octahedra lie parallel to the *ab* plane stacked along the c-direction. The iodide ion occupies the octahedral voids between these positively charged N—Sr layers. This creates alternating edge-sharing layers of NSr_6 and ISr_6 octahedra in a cubic close packed (*CCP*) arrangement (Fig. 1, Fig. 2).

The Sr—N distance is in excellent agreement with that found in the binary subnitride Sr_2N (2.6118 (3) Å) (Brese & O'Keeffe, 1990). Further, the Sr—I bond length is also in close agreement with data for SrI_2 (3.3382–3.4142 Å) (Rietschel & Baernighausen, 1969).

Experimental

The title Sr_2NI crystals were prepared by reaction of SrI_2 (Aldrich, 99.99+%) with distrontium subnitride, Sr_2N. Sr_2N powder was prepared by the reaction of cleaned Sr metal (Alfa, 99%) with dried nitrogen at 793 K. Due to the air sensitivity of the reactants and products involved, all manipulations were carried out in glove boxes (either recirculating nitrogen-filled or evacuable argon-filled). Stoichiometric ratios of the reactants were thoroughly mixed and ground together, then pressed to form a pellet (*ca* 1 g). The pellet was placed in a molybdenum foil liner and transferred to a stainless steel crucible, which was subsequently welded shut under an argon atmosphere. The sealed crucibles were heated in a tube furnace (1023 K, 5 d) under flowing argon to prevent oxidation of the steel crucibles. The furnace was cooled slowly (20 K h^{-1}). The crucibles were opened in an N_2-filled glove-box. Orange irregular crystals were observed on the pellet surface. Crystals were selected in a recirculating N_2-filled glove-box under an optical microscope and placed under RS3000 perfluoropolyether (Riedel de Haën) on a microscope slide prior to mounting on the diffractometer. The moisture-free viscous perfluoropolyether protects the crystals from atmospheric oxygen and moisture without interfering with the diffraction experiment.

Refinement

Due to twinning present in the crystal the data were integrated using 2 orientation matrices, related by the twin law (−1 0 0, 1 1 0, 0 0 − 1), giving 682 reflections, 388 have contributions from only one component − 197 only belong to the first component, 186 only to second and 303 have contributions from both components. The fraction of the twin component was refined to 0.183 (6).

Figures

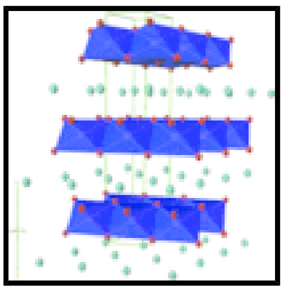

Fig. 1. Polyhedral representation of the structure of Sr_2NI, showing $[Sr_2N]^+$ layers of edge-sharing NSr_6 octahedra alternating with layers of I^-.

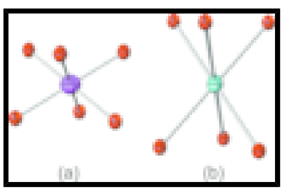

Fig. 2. *ORTEP*type plot of the local coordination of Sr to N and I. Both anions are coordinated in a distorted octahedral geometry. (Ellipsoids are drawn at the 90% probability level).

Distrontium nitride iodide

Crystal data

$INSr_2$	$Z = 3$
$M_r = 316.15$	$F_{000} = 408$
Trigonal, $R\bar{3}m$	$D_x = 4.918$ Mg m^{-3}
Hall symbol: -R 3 2"	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 4.0049$ (6) Å	Cell parameters from 340 reflections
$b = 4.0049$ (6) Å	$\theta = 5.3$–$27.2°$
$c = 23.055$ (7) Å	$\mu = 31.99$ mm^{-1}
$\alpha = 90°$	$T = 150$ (2) K
$\beta = 90°$	Needle, orange
$\gamma = 120°$	0.14 × 0.04 × 0.04 mm
$V = 320.24$ (12) Å^3	

Data collection

Bruker SMART1000 CCD area-detector	165 independent reflections

diffractometer	
Radiation source: sealed tube	163 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.032$
$T = 150(2)$ K	$\theta_{max} = 24.9°$
ω scans	$\theta_{min} = 5.3°$
Absorption correction: multi-scan (TWINABS; Bruker, 2007)	$h = -4 \rightarrow 4$
$T_{min} = 0.175$, $T_{max} = 0.278$	$k = -4 \rightarrow 4$
682 measured reflections	$l = -26 \rightarrow 26$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.061$	$w = 1/[\sigma^2(F_o^2) + (0.1578P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.192$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.21$	$\Delta\rho_{max} = 1.77$ e Å^{-3}
165 reflections	$\Delta\rho_{min} = -1.76$ e Å^{-3}
9 parameters	Extinction correction: none

Special details

Experimental. Data were integrated using 2 orientation matrices, related by the twin law (−1 0 0, 1 1 0, 0 0 − 1). This gave a total of 682 reflections; 379 of these have contributions from only one component, 197 only belong to component 1, 186 only to 2 and 294 have contributions from both components.

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

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

	x	y	z	U_{iso}*/U_{eq}
I1	0.0000	0.0000	0.0000	0.0134 (11)
Sr1	0.0000	0.0000	0.22397 (11)	0.0095 (10)
N1	0.0000	0.0000	0.5000	0.019 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0117 (11)	0.0117 (11)	0.017 (2)	0.0059 (5)	0.000	0.000
Sr1	0.0085 (11)	0.0085 (11)	0.0117 (17)	0.0042 (5)	0.000	0.000
N1	0.019 (7)	0.019 (7)	0.019 (18)	0.009 (4)	0.000	0.000

Geometric parameters (Å, °)

I1—Sr1^{i}	3.421 (2)	Sr1—Sr1^{iii}	3.511 (4)
I1—Sr1^{ii}	3.421 (2)	Sr1—Sr1^{i}	3.511 (4)
I1—Sr1^{iii}	3.421 (2)	Sr1—Sr1^{vi}	3.511 (4)
I1—Sr1^{iv}	3.421 (2)	Sr1—Sr1^{x}	4.0049 (6)
I1—Sr1^{v}	3.421 (2)	Sr1—Sr1^{xi}	4.0049 (6)
I1—Sr1^{vi}	3.421 (2)	Sr1—Sr1^{xii}	4.0049 (6)
Sr1—N1^{ii}	2.6631 (13)	N1—Sr1^{xiii}	2.6631 (13)
Sr1—N1^{iv}	2.6631 (13)	N1—Sr1^{vii}	2.6631 (13)
Sr1—N1^{v}	2.6631 (13)	N1—Sr1^{xiv}	2.6631 (13)
Sr1—I1^{vii}	3.421 (2)	N1—Sr1^{viii}	2.6631 (13)
Sr1—I1^{viii}	3.421 (2)	N1—Sr1^{ix}	2.6631 (13)
Sr1—I1^{ix}	3.421 (2)	N1—Sr1^{xv}	2.6631 (13)
Sr1^{i}—I1—Sr1^{ii}	180.00 (6)	I1^{ix}—Sr1—Sr1^{vi}	96.29 (3)
Sr1^{i}—I1—Sr1^{iii}	71.65 (5)	Sr1^{iii}—Sr1—Sr1^{vi}	69.54 (9)
Sr1^{ii}—I1—Sr1^{iii}	108.35 (5)	Sr1^{i}—Sr1—Sr1^{vi}	69.54 (9)
Sr1^{i}—I1—Sr1^{iv}	108.35 (5)	N1^{ii}—Sr1—Sr1^{x}	41.24 (3)
Sr1^{ii}—I1—Sr1^{iv}	71.65 (5)	N1^{iv}—Sr1—Sr1^{x}	138.76 (3)
Sr1^{iii}—I1—Sr1^{iv}	180.00 (6)	N1^{v}—Sr1—Sr1^{x}	90.0
Sr1^{i}—I1—Sr1^{v}	108.35 (5)	I1^{vii}—Sr1—Sr1^{x}	125.83 (2)
Sr1^{ii}—I1—Sr1^{v}	71.65 (5)	I1^{viii}—Sr1—Sr1^{x}	54.17 (2)
Sr1^{iii}—I1—Sr1^{v}	108.35 (5)	I1^{ix}—Sr1—Sr1^{x}	90.0
Sr1^{iv}—I1—Sr1^{v}	71.65 (5)	Sr1^{iii}—Sr1—Sr1^{x}	55.23 (4)
Sr1^{i}—I1—Sr1^{vi}	71.65 (5)	Sr1^{i}—Sr1—Sr1^{x}	124.77 (4)
Sr1^{ii}—I1—Sr1^{vi}	108.35 (5)	Sr1^{vi}—Sr1—Sr1^{x}	90.0
Sr1^{iii}—I1—Sr1^{vi}	71.65 (5)	N1^{ii}—Sr1—Sr1^{xi}	138.76 (3)
Sr1^{iv}—I1—Sr1^{vi}	108.35 (5)	N1^{iv}—Sr1—Sr1^{xi}	41.24 (3)
Sr1^{v}—I1—Sr1^{vi}	180.00 (6)	N1^{v}—Sr1—Sr1^{xi}	90.0
N1^{ii}—Sr1—N1^{iv}	97.52 (6)	I1^{vii}—Sr1—Sr1^{xi}	54.17 (2)
N1^{ii}—Sr1—N1^{v}	97.52 (6)	I1^{viii}—Sr1—Sr1^{xi}	125.83 (2)
N1^{iv}—Sr1—N1^{v}	97.52 (6)	I1^{ix}—Sr1—Sr1^{xi}	90.0
N1^{ii}—Sr1—I1^{vii}	162.27 (8)	Sr1^{iii}—Sr1—Sr1^{xi}	124.77 (4)
N1^{iv}—Sr1—I1^{vii}	94.14 (2)	Sr1^{i}—Sr1—Sr1^{xi}	55.23 (4)
N1^{v}—Sr1—I1^{vii}	94.14 (2)	Sr1^{vi}—Sr1—Sr1^{xi}	90.0
N1^{ii}—Sr1—I1^{viii}	94.14 (2)	Sr1^{x}—Sr1—Sr1^{xi}	180.00 (15)
N1^{iv}—Sr1—I1^{viii}	162.27 (8)	N1^{ii}—Sr1—Sr1^{xii}	41.24 (3)
N1^{v}—Sr1—I1^{viii}	94.14 (2)	N1^{iv}—Sr1—Sr1^{xii}	90.0
I1^{vii}—Sr1—I1^{viii}	71.65 (5)	N1^{v}—Sr1—Sr1^{xii}	138.76 (3)
N1^{ii}—Sr1—I1^{ix}	94.14 (2)	I1^{vii}—Sr1—Sr1^{xii}	125.83 (2)

$N1^{iv}—Sr1—I1^{ix}$	94.14 (2)	$I1^{viii}—Sr1—Sr1^{xii}$	90.0
$N1^{v}—Sr1—I1^{ix}$	162.27 (8)	$I1^{ix}—Sr1—Sr1^{xii}$	54.17 (2)
$I1^{vii}—Sr1—I1^{ix}$	71.65 (5)	$Sr1^{iii}—Sr1—Sr1^{xii}$	90.0
$I1^{viii}—Sr1—I1^{ix}$	71.65 (5)	$Sr1^{i}—Sr1—Sr1^{xii}$	124.77 (4)
$N1^{ii}—Sr1—Sr1^{iii}$	48.76 (3)	$Sr1^{vi}—Sr1—Sr1^{xii}$	55.23 (4)
$N1^{iv}—Sr1—Sr1^{iii}$	101.45 (10)	$Sr1^{x}—Sr1—Sr1^{xii}$	60.0
$N1^{v}—Sr1—Sr1^{iii}$	48.76 (3)	$Sr1^{xi}—Sr1—Sr1^{xii}$	120.0
$I1^{vii}—Sr1—Sr1^{iii}$	141.004 (11)	$Sr1^{xiii}—N1—Sr1^{vii}$	180.0
$I1^{viii}—Sr1—Sr1^{iii}$	96.29 (3)	$Sr1^{xiii}—N1—Sr1^{xiv}$	97.52 (6)
$I1^{ix}—Sr1—Sr1^{iii}$	141.003 (11)	$Sr1^{vii}—N1—Sr1^{xiv}$	82.48 (6)
$N1^{ii}—Sr1—Sr1^{i}$	101.45 (10)	$Sr1^{xiii}—N1—Sr1^{viii}$	82.48 (6)
$N1^{iv}—Sr1—Sr1^{i}$	48.76 (3)	$Sr1^{vii}—N1—Sr1^{viii}$	97.52 (6)
$N1^{v}—Sr1—Sr1^{i}$	48.76 (3)	$Sr1^{xiv}—N1—Sr1^{viii}$	180.0
$I1^{vii}—Sr1—Sr1^{i}$	96.29 (3)	$Sr1^{xiii}—N1—Sr1^{ix}$	82.48 (6)
$I1^{viii}—Sr1—Sr1^{i}$	141.004 (11)	$Sr1^{vii}—N1—Sr1^{ix}$	97.52 (6)
$I1^{ix}—Sr1—Sr1^{i}$	141.003 (11)	$Sr1^{xiv}—N1—Sr1^{ix}$	82.48 (6)
$Sr1^{iii}—Sr1—Sr1^{i}$	69.54 (9)	$Sr1^{viii}—N1—Sr1^{ix}$	97.52 (6)
$N1^{ii}—Sr1—Sr1^{vi}$	48.76 (3)	$Sr1^{xiii}—N1—Sr1^{xv}$	97.52 (6)
$N1^{iv}—Sr1—Sr1^{vi}$	48.76 (3)	$Sr1^{vii}—N1—Sr1^{xv}$	82.48 (6)
$N1^{v}—Sr1—Sr1^{vi}$	101.45 (10)	$Sr1^{xiv}—N1—Sr1^{xv}$	97.52 (6)
$I1^{vii}—Sr1—Sr1^{vi}$	141.003 (11)	$Sr1^{viii}—N1—Sr1^{xv}$	82.48 (6)
$I1^{viii}—Sr1—Sr1^{vi}$	141.003 (11)	$Sr1^{ix}—N1—Sr1^{xv}$	180.00 (10)

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

Fig. 1

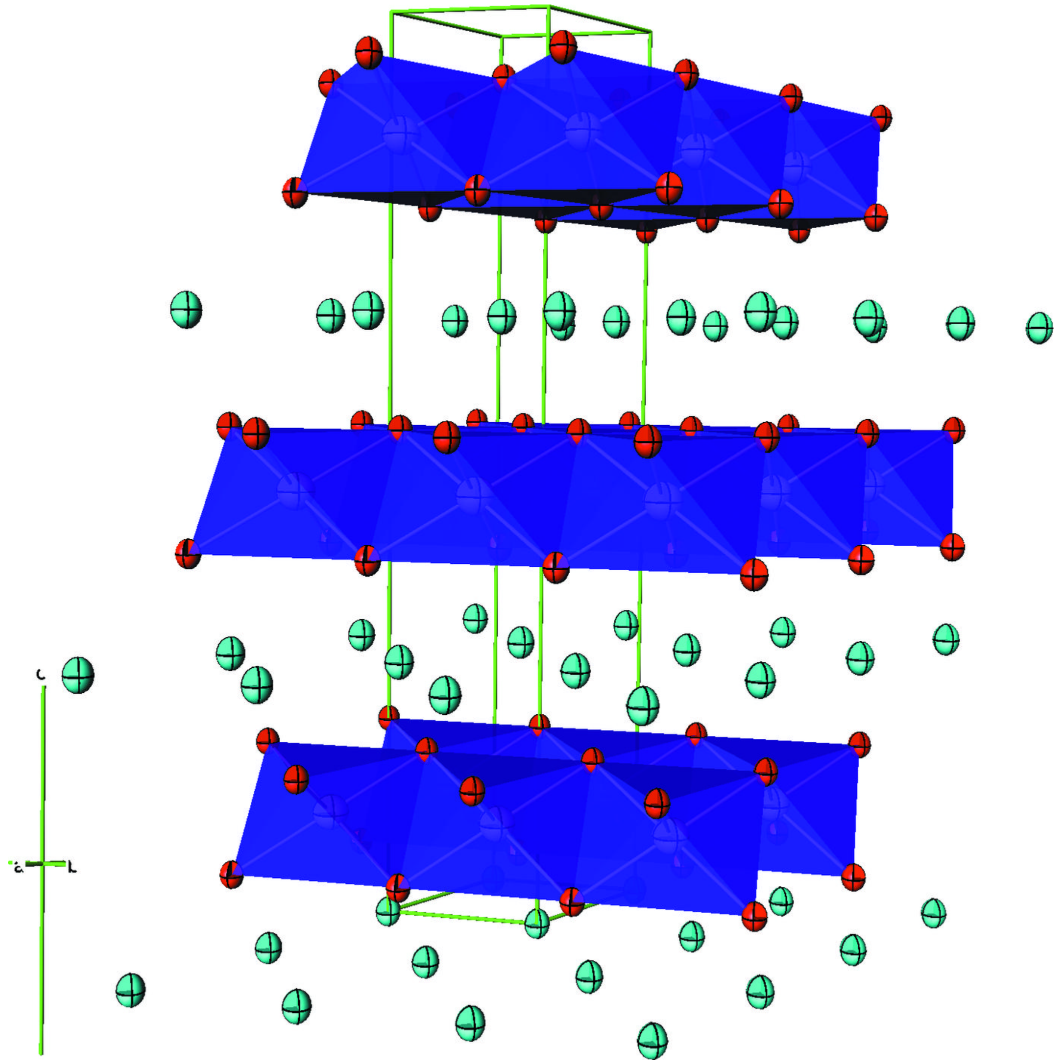

Fig. 2