Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# $Zr_3NiSb_7$: a new antimony-enriched $ZrSb_2$ derivative

**V. Romaka,[a]* A. Tkachuk[b] and L. Romaka[a]**

[a]Department of Inorganic Chemistry, Ivan Franko National University of Lviv, Kyryla i Mefodiya Street 6, 79005 Lviv, Ukraine, and [b]Department of Chemistry, University of Alberta, Edmonton, Alberta, Canada T6G 2G2
Correspondence e-mail: romakav@yahoo.com

Received 27 June 2008; accepted 2 July 2008

Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(Sb–Ni) = 0.001 Å; $R$ factor = 0.025; $wR$ factor = 0.054; data-to-parameter ratio = 25.3.

Single crystals of trizirconium nickel heptaantimonide were synthesized from the constituent elements by arc-melting. The compound crystallizes in a unique structure type and belongs to the family of two-layer structures. All crystallographically unique atoms (3 × Zr, 1 × Ni and 7 × Sb) are located at sites with $m$ symmetry. The structure contains '$Zr_2Ni_2Sb_5$' and '$Zr_4Sb_9$' fragments and might be described as a new $ZrSb_2$ derivative with a high Sb content.

## Related literature

The structure of $ZrSb_2$ was described by Garcia & Corbett (1988). For related antimonides, see: Romaka *et al.* (2007); Tkachuk *et al.* (2007). For related literature, see: Emsley (1991).

## Experimental

### *Crystal data*

$Zr_3NiSb_7$
$M_r$ = 1184.62
Orthorhombic, *Pnma*
$a$ = 17.5165 (19) Å
$b$ = 3.9266 (4) Å
$c$ = 14.3968 (15) Å
$V$ = 990.22 (18) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 23.56 mm$^{-1}$
$T$ = 295 (2) K
0.37 × 0.06 × 0.04 mm

### *Data collection*

Bruker SMART 1000 diffractometer
Absorption correction: numerical (*SHELXTL*; Sheldrick, 2008)
$T_{min}$ = 0.057, $T_{max}$ = 0.426
11118 measured reflections
1722 independent reflections
1500 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.043

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.053
$S$ = 1.18
1722 reflections
68 parameters
$\Delta\rho_{max}$ = 2.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −2.89 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Zr1—Sb4$^{i}$ | 2.9620 (6) | Zr3—Sb6$^{v}$ | 2.9975 (8) |
| Zr1—Sb6 | 2.9876 (8) | Zr3—Sb3$^{vi}$ | 3.1616 (9) |
| Zr1—Sb1$^{ii}$ | 3.0669 (8) | Ni1—Sb7 | 2.5728 (10) |
| Zr1—Sb3$^{iii}$ | 3.0720 (7) | Ni1—Sb2$^{iv}$ | 2.5875 (7) |
| Zr1—Sb7$^{iii}$ | 3.0960 (6) | Ni1—Sb1$^{iv}$ | 2.6140 (7) |
| Zr1—Sb5 | 3.1324 (8) | Ni1—Sb4$^{vi}$ | 2.7141 (10) |
| Zr2—Sb6$^{iv}$ | 2.9478 (6) | Sb1—Sb2 | 3.1998 (8) |
| Zr2—Sb4$^{iii}$ | 2.9499 (6) | Sb1—Sb3$^{vii}$ | 3.2645 (6) |
| Zr2—Sb2$^{iv}$ | 2.9604 (6) | Sb1—Sb4$^{viii}$ | 3.2981 (6) |
| Zr2—Sb2$^{ii}$ | 3.0029 (8) | Sb2—Sb2$^{ix}$ | 3.2250 (7) |
| Zr2—Sb5 | 3.0044 (8) | Sb2—Sb4$^{viii}$ | 3.3111 (6) |
| Zr3—Sb1$^{iv}$ | 2.9569 (6) | Sb5—Sb7$^{iii}$ | 3.1380 (6) |
| Zr3—Sb3$^{iv}$ | 2.9944 (7) | Sb5—Sb6$^{iv}$ | 3.1387 (6) |
| Zr3—Sb5$^{iv}$ | 2.9958 (6) | Sb6—Sb7$^{i}$ | 3.1393 (6) |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

The work was in part supported by the Ministry of Ukraine for Education and Science (grant No. 0106U001299).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2182).

## References

Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2000). *SMART* and *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Emsley, J. (1991). *The Elements*, 2nd ed. Oxford: Clarendon Press.
Garcia, E. & Corbett, J. D. (1988). *J. Solid State Chem.* **73**, 440–451.
Romaka, L., Tkachuk, A. & Stadnyk, Yu. (2007). *11th Scientific Conference 'L'viv Chemistry Reading 2007'*, Collected Abstracts, p. H37. L'viv: Publishing Center of Ivan Franko National University.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tkachuk, A., Romaka, L. & Stadnyk, Yu. (2007). *10th International Conference on Crystal Chemistry of Intermetallic Compounds, L'viv (Ukraine)*, Collected Abstracts, p. 71. L'viv: Publishing Center of Ivan Franko National University.

# supplementary materials

*Acta Cryst.* (2008). E**64**, i47 [ **doi:10.1107/S1600536808020278** ]

# $Zr_3NiSb_7$: a new antimony-enriched $ZrSb_2$ derivative

**V. Romaka, A. Tkachuk and L. Romaka**

## Comment

Antimony based intermetallics attract interest due to interesting thermoelectric properties of some phases, *e.g.* antimonides with MgAgAs and $Y_3Au_3Sb_4$ type structures. The investigation of new intermetallic phases is useful for the development of new materials, and the accurate determination of their crystal structures is a basic requirement for a better understanding of the corresponding physical properties.

Investigation of the Zr–Ni–Sb ternary system revealed the presence of several compounds in the Sb-enriched area (Romaka *et al.*, 2007), including the new title antimonide with composition $Zr_{27}Ni_9Sb_{64}$ (in $\%_{at}$). Interatomic distances (Table 1) between Sb atoms are in good agreement with the sum of the atomic radius (Emsley, 1991), whereas the majority of Zr—Sb and all Ni—Sb distances are somewhat shortened. Such shortening may be explained by partial covalent bonding which appears to be significant between Ni—Sb atoms because their contact distances are rather close to the sum of their covalent radii (2.56 Å). As the majority of ternary intermetallics are constructed from the fragments of their most stable binary compounds, the structure analysis of the antimonides $Zr_3NiSb_7$ and the already known $Zr_2NiSb_3$ (Tkachuk *et al.*, 2007) in the Sb-enriched area shows that both can be derived from the binary compound $ZrSb_2$ (Garcia & Corbett, 1988), which crystallizes in the $PbCl_2$ structure type.

$Zr_3NiSb_7$ belongs to the family of two-layer structures. It may be represented as a net of trigonal prisms formed by Sb atoms that are bridged by nickel atoms (Fig. 1a). Such an arrangement is very similar to that in the binary $ZrSb_2$ structure (Fig. 1b). The coordination polyhedra are distorted tri-capped trigonal prisms for the Zr atoms, and distorted octahedra for Ni atoms. In an alternative description, the $Zr_3NiSb_7$ structure contains fragments of the hypothetical "$Zr_2Ni_2Sb_5$" and "$Zr_4Sb_9$" structures (Fig. 2) which are so far unknown for the ternary Zr–Ni–Sb or binary Zr–Sb systems. The main feature of the $Zr_3NiSb_7$ structure is the absence of covalent bonding between antimony atoms in contrast to the $ZrSb_2$ structure. The general conclusion is that the presence of Ni atoms intensifies the interaction between Zr/Ni and Sb and, at the same time, reduces the bonding between Sb atoms. One may speculate that the composition of the $Zr_3NiSb_7$ compound may be the boundary limit of some solid solutions based on $ZrSb_2$. However, the detailed study of the phase equilibria in the Zr–Ni–Sb system did not show a formation of any substitutional or interstitial solid solution. Moreover, the diffraction patterns of $Zr_3NiSb_7$ and $ZrSb_2$ are rather different.

## Experimental

A sample with nominal composition $Zr_{30}Ni_{10}Sb_{60}$ was prepared by arc-melting the constituent elements Zr (99.99 wt.%), Ni (99.99 wt.%), and Sb (99.99 wt.%) on a water-cooled copper hearth under a protective Ti-gettered argon atmosphere. 5 wt.% excess of Sb was required to compensate the evaporative loss during arc-melting. The ingot was annealed at 870 K for 720 h in an evacuated silica ampoule, and finally quenched in cold water. A crystal of the title compound suitable for single-crystal X-ray diffraction was extracted directly from the annealed sample. The chemical composition of the crystal

was determined on the basis of an energy dispersive X-ray spectroscopical analysis using a Hitachi S-2700 scanning electron microscope. The result of the analysis is in good aggreement with the composition calculated from the structural refinement: Measured: 24.5 (8) $\%_{at}$ Zr, 11.3 (6) $\%_{at}$ Ni, 64.2 (16) $\%_{at}$ Sb; calculated Zr 27 $\%_{at}$, Ni 9$\%_{at}$, Sb 64 $\%_{at}$.

### Refinement

The highest remaining electron density peak and the deepest hole are located 0.80 Å from Sb1 and 1.78 Å from Ni1, respectively. The structure solution and refinement were also performed in the non-centrosymmetric space group $Pna2_1$, but were less satisfactory and resulted in larger *R* indices and atomic displacement parameters.

### Figures

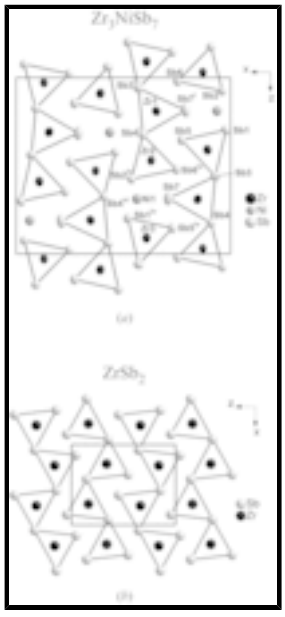


Fig. 1. (*a*). Projection of the $Zr_3NiSb_7$ structure onto the (010) plane with displacement ellipsoids drawn at the 95% probability level. [Symmetry codes: (i) 0.5 - *x*, 1 - *y*, -1/2 + *z*; (iv) 0.5 - *x*, -*y*, 0.5 - *z*; (vi) 1/2 + *x*, *y*, 1.5 - *z*]; (*b*) Projection of the $ZrSb_2$ structure onto the (010) plane.

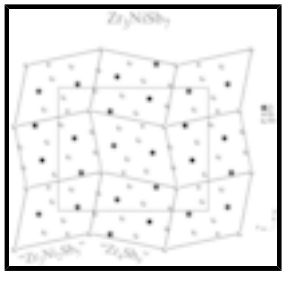


Fig. 2. The stacked "$Zr_2Ni_2Sb_5$" and "$Zr_4Sb_9$" fragments in the $Zr_3NiSb_7$ structure.

### trizirconium nickel heptaantimonide

*Crystal data*

| | |
|---|---|
| $Zr_3NiSb_7$ | $F_{000}$ = 2020 |
| $M_r$ = 1184.62 | $D_x$ = 7.946 Mg m$^{-3}$ |
| Orthorhombic, *Pnma* | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ac 2n | Cell parameters from 4956 reflections |

| | |
|---|---|
| $a$ = 17.5165 (19) Å | θ = 2.3–33.1° |
| $b$ = 3.9266 (4) Å | μ = 23.56 mm$^{-1}$ |
| $c$ = 14.3968 (15) Å | $T$ = 295 (2) K |
| $V$ = 990.22 (18) Å$^3$ | Needle, silver |
| $Z$ = 4 | 0.37 × 0.06 × 0.04 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART 1000 diffractometer | 1722 independent reflections |
| Radiation source: fine-focus sealed tube | 1500 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.044 |
| $T$ = 295(2) K | $\theta_{max}$ = 30.5° |
| φ and ω scans | $\theta_{min}$ = 2.3° |
| Absorption correction: numerical (SHELXTL; Sheldrick, 2008) | $h$ = −25→25 |
| $T_{min}$ = 0.057, $T_{max}$ = 0.426 | $k$ = −5→5 |
| 11118 measured reflections | $l$ = −20→20 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | $w = 1/[\sigma^2(F_o^2) + (0.0223P)^2 + 1.1518P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.024 | $(\Delta/\sigma)_{max}$ = 0.001 |
| $wR(F^2)$ = 0.054 | $\Delta\rho_{max}$ = 2.12 e Å$^{-3}$ |
| $S$ = 1.18 | $\Delta\rho_{min}$ = −2.89 e Å$^{-3}$ |
| 1722 reflections | Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| 68 parameters | Extinction coefficient: 0.00069 (5) |
| Primary atom site location: structure-invariant direct methods | |

*Special details*

**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.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| Zr1 | 0.34664 (4) | 0.2500 | 0.19076 (5) | 0.00788 (14) |
| Zr2 | 0.37045 (4) | 0.2500 | 0.47072 (5) | 0.00721 (13) |
| Zr3 | 0.39237 (4) | 0.2500 | 0.90990 (5) | 0.00782 (14) |
| Ni1 | 0.43680 (5) | 0.2500 | 0.68908 (6) | 0.00903 (18) |
| Sb1 | 0.02147 (3) | 0.2500 | 0.29766 (3) | 0.00871 (11) |
| Sb2 | 0.03748 (2) | 0.2500 | 0.07626 (3) | 0.00790 (10) |
| Sb3 | 0.07123 (3) | 0.2500 | 0.56057 (3) | 0.00957 (11) |
| Sb4 | 0.09131 (3) | 0.2500 | 0.82504 (3) | 0.00823 (10) |
| Sb5 | 0.22833 (3) | 0.2500 | 0.35390 (3) | 0.00918 (11) |
| Sb6 | 0.24792 (2) | 0.2500 | 0.02153 (3) | 0.00875 (11) |
| Sb7 | 0.28995 (3) | 0.2500 | 0.68532 (4) | 0.01218 (11) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Zr1 | 0.0068 (3) | 0.0083 (3) | 0.0085 (3) | 0.000 | −0.0007 (2) | 0.000 |
| Zr2 | 0.0054 (3) | 0.0077 (3) | 0.0085 (3) | 0.000 | 0.0002 (2) | 0.000 |
| Zr3 | 0.0064 (3) | 0.0078 (3) | 0.0092 (3) | 0.000 | 0.0004 (2) | 0.000 |
| Ni1 | 0.0085 (4) | 0.0092 (5) | 0.0094 (4) | 0.000 | 0.0000 (3) | 0.000 |
| Sb1 | 0.0081 (2) | 0.0089 (2) | 0.0092 (2) | 0.000 | −0.00158 (15) | 0.000 |
| Sb2 | 0.0069 (2) | 0.0084 (2) | 0.0084 (2) | 0.000 | 0.00046 (15) | 0.000 |
| Sb3 | 0.0104 (2) | 0.0100 (2) | 0.0083 (2) | 0.000 | 0.00042 (16) | 0.000 |
| Sb4 | 0.0082 (2) | 0.0086 (2) | 0.0079 (2) | 0.000 | 0.00022 (15) | 0.000 |
| Sb5 | 0.0069 (2) | 0.0104 (2) | 0.0102 (2) | 0.000 | 0.00005 (15) | 0.000 |
| Sb6 | 0.0069 (2) | 0.0100 (2) | 0.0094 (2) | 0.000 | −0.00051 (15) | 0.000 |
| Sb7 | 0.0076 (2) | 0.0100 (3) | 0.0189 (3) | 0.000 | 0.00142 (16) | 0.000 |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Zr1—Sb4$^{i}$ | 2.9620 (6) | Sb2—Ni1$^{ii}$ | 2.5876 (7) |
| Zr1—Sb4$^{ii}$ | 2.9620 (6) | Sb2—Zr2$^{ii}$ | 2.9604 (6) |
| Zr1—Sb6 | 2.9876 (8) | Sb2—Zr2$^{i}$ | 2.9604 (6) |
| Zr1—Sb1$^{iii}$ | 3.0669 (8) | Sb2—Zr2$^{viii}$ | 3.0030 (8) |
| Zr1—Sb3$^{ii}$ | 3.0720 (7) | Sb2—Sb2$^{xi}$ | 3.2250 (7) |
| Zr1—Sb3$^{i}$ | 3.0720 (7) | Sb2—Sb2$^{xii}$ | 3.2250 (7) |
| Zr1—Sb7$^{ii}$ | 3.0960 (6) | Sb2—Sb4$^{x}$ | 3.3111 (6) |
| Zr1—Sb7$^{i}$ | 3.0960 (6) | Sb2—Sb4$^{ix}$ | 3.3111 (6) |
| Zr1—Sb5 | 3.1324 (8) | Sb3—Zr3$^{ii}$ | 2.9943 (7) |
| Zr2—Sb6$^{iv}$ | 2.9478 (6) | Sb3—Zr3$^{i}$ | 2.9943 (7) |
| Zr2—Sb6$^{v}$ | 2.9478 (6) | Sb3—Zr1$^{v}$ | 3.0720 (7) |
| Zr2—Sb4$^{ii}$ | 2.9499 (6) | Sb3—Zr1$^{iv}$ | 3.0720 (7) |
| Zr2—Sb4$^{i}$ | 2.9499 (6) | Sb3—Zr3$^{xiii}$ | 3.1617 (9) |
| Zr2—Sb2$^{v}$ | 2.9604 (6) | Sb3—Sb1$^{ix}$ | 3.2645 (6) |
| Zr2—Sb2$^{iv}$ | 2.9604 (6) | Sb3—Sb1$^{x}$ | 3.2645 (6) |
| Zr2—Sb2$^{iii}$ | 3.0029 (8) | Sb4—Ni1$^{xiii}$ | 2.7141 (10) |

| | | | |
|---|---|---|---|
| Zr2—Sb5 | 3.0044 (8) | Sb4—Zr2$^{v}$ | 2.9499 (6) |
| Zr2—Sb7 | 3.3962 (9) | Sb4—Zr2$^{iv}$ | 2.9499 (6) |
| Zr3—Sb1$^{iv}$ | 2.9569 (6) | Sb4—Zr1$^{iv}$ | 2.9619 (6) |
| Zr3—Sb1$^{v}$ | 2.9569 (6) | Sb4—Zr1$^{v}$ | 2.9619 (6) |
| Zr3—Sb3$^{v}$ | 2.9944 (7) | Sb4—Sb1$^{x}$ | 3.2981 (6) |
| Zr3—Sb3$^{iv}$ | 2.9944 (7) | Sb4—Sb1$^{ix}$ | 3.2981 (6) |
| Zr3—Sb5$^{iv}$ | 2.9958 (6) | Sb4—Sb2$^{x}$ | 3.3111 (6) |
| Zr3—Sb5$^{v}$ | 2.9958 (6) | Sb4—Sb2$^{ix}$ | 3.3110 (6) |
| Zr3—Sb6$^{vi}$ | 2.9975 (8) | Sb5—Zr3$^{ii}$ | 2.9958 (6) |
| Zr3—Sb3$^{vii}$ | 3.1616 (9) | Sb5—Zr3$^{i}$ | 2.9958 (6) |
| Zr3—Ni1 | 3.2730 (12) | Sb5—Sb7$^{ii}$ | 3.1380 (6) |
| Ni1—Sb7 | 2.5728 (10) | Sb5—Sb7$^{i}$ | 3.1380 (6) |
| Ni1—Sb2$^{iv}$ | 2.5875 (7) | Sb5—Sb6$^{v}$ | 3.1387 (6) |
| Ni1—Sb2$^{v}$ | 2.5875 (7) | Sb5—Sb6$^{iv}$ | 3.1387 (6) |
| Ni1—Sb1$^{v}$ | 2.6140 (7) | Sb6—Zr2$^{ii}$ | 2.9478 (6) |
| Ni1—Sb1$^{iv}$ | 2.6140 (7) | Sb6—Zr2$^{i}$ | 2.9478 (6) |
| Ni1—Sb4$^{vii}$ | 2.7141 (10) | Sb6—Zr3$^{xiv}$ | 2.9974 (8) |
| Sb1—Ni1$^{ii}$ | 2.6139 (7) | Sb6—Sb5$^{ii}$ | 3.1388 (6) |
| Sb1—Ni1$^{i}$ | 2.6139 (7) | Sb6—Sb5$^{i}$ | 3.1388 (6) |
| Sb1—Zr3$^{i}$ | 2.9570 (6) | Sb6—Sb7$^{i}$ | 3.1393 (6) |
| Sb1—Zr3$^{ii}$ | 2.9570 (6) | Sb6—Sb7$^{ii}$ | 3.1393 (6) |
| Sb1—Zr1$^{viii}$ | 3.0669 (8) | Sb7—Zr1$^{iv}$ | 3.0960 (6) |
| Sb1—Sb2 | 3.1998 (8) | Sb7—Zr1$^{v}$ | 3.0960 (6) |
| Sb1—Sb3$^{ix}$ | 3.2645 (6) | Sb7—Sb5$^{v}$ | 3.1380 (6) |
| Sb1—Sb3$^{x}$ | 3.2645 (6) | Sb7—Sb5$^{iv}$ | 3.1380 (6) |
| Sb1—Sb4$^{x}$ | 3.2981 (6) | Sb7—Sb6$^{iv}$ | 3.1393 (6) |
| Sb1—Sb4$^{ix}$ | 3.2981 (6) | Sb7—Sb6$^{v}$ | 3.1393 (6) |
| Sb2—Ni1$^{i}$ | 2.5876 (7) | | |
| Sb4$^{i}$—Zr1—Sb4$^{ii}$ | 83.03 (2) | Ni1$^{i}$—Sb2—Zr2$^{viii}$ | 108.12 (2) |
| Sb4$^{i}$—Zr1—Sb6 | 138.128 (11) | Ni1$^{ii}$—Sb2—Zr2$^{viii}$ | 108.12 (2) |
| Sb4$^{ii}$—Zr1—Sb6 | 138.128 (11) | Zr2$^{ii}$—Sb2—Zr2$^{viii}$ | 114.528 (17) |
| Sb4$^{i}$—Zr1—Sb1$^{iii}$ | 66.303 (16) | Zr2$^{i}$—Sb2—Zr2$^{viii}$ | 114.528 (17) |
| Sb4$^{ii}$—Zr1—Sb1$^{iii}$ | 66.303 (16) | Ni1$^{i}$—Sb2—Sb1 | 52.409 (19) |
| Sb6—Zr1—Sb1$^{iii}$ | 128.48 (3) | Ni1$^{ii}$—Sb2—Sb1 | 52.409 (19) |
| Sb4$^{i}$—Zr1—Sb3$^{ii}$ | 130.54 (3) | Zr2$^{ii}$—Sb2—Sb1 | 123.991 (17) |
| Sb4$^{ii}$—Zr1—Sb3$^{ii}$ | 78.623 (15) | Zr2$^{i}$—Sb2—Sb1 | 123.991 (17) |
| Sb6—Zr1—Sb3$^{ii}$ | 76.910 (19) | Zr2$^{viii}$—Sb2—Sb1 | 97.986 (19) |
| Sb1$^{iii}$—Zr1—Sb3$^{ii}$ | 64.250 (16) | Ni1$^{i}$—Sb2—Sb2$^{xi}$ | 163.78 (3) |
| Sb4$^{i}$—Zr1—Sb3$^{i}$ | 78.623 (15) | Ni1$^{ii}$—Sb2—Sb2$^{xi}$ | 92.085 (17) |
| Sb4$^{ii}$—Zr1—Sb3$^{i}$ | 130.54 (3) | Zr2$^{ii}$—Sb2—Sb2$^{xi}$ | 57.900 (17) |

| | | | |
|---|---|---|---|
| Sb6—Zr1—Sb3$^{i}$ | 76.911 (19) | Zr2$^{i}$—Sb2—Sb2$^{xi}$ | 106.03 (2) |
| Sb1$^{iii}$—Zr1—Sb3$^{i}$ | 64.250 (16) | Zr2$^{viii}$—Sb2—Sb2$^{xi}$ | 56.628 (16) |
| Sb3$^{ii}$—Zr1—Sb3$^{i}$ | 79.45 (2) | Sb1—Sb2—Sb2$^{xi}$ | 129.982 (17) |
| Sb4$^{i}$—Zr1—Sb7$^{ii}$ | 136.09 (3) | Ni1$^{i}$—Sb2—Sb2$^{xii}$ | 92.085 (17) |
| Sb4$^{ii}$—Zr1—Sb7$^{ii}$ | 83.092 (15) | Ni1$^{ii}$—Sb2—Sb2$^{xii}$ | 163.78 (3) |
| Sb6—Zr1—Sb7$^{ii}$ | 62.103 (16) | Zr2$^{ii}$—Sb2—Sb2$^{xii}$ | 106.03 (2) |
| Sb1$^{iii}$—Zr1—Sb7$^{ii}$ | 140.627 (10) | Zr2$^{i}$—Sb2—Sb2$^{xii}$ | 57.900 (16) |
| Sb3$^{ii}$—Zr1—Sb7$^{ii}$ | 86.630 (15) | Zr2$^{viii}$—Sb2—Sb2$^{xii}$ | 56.628 (16) |
| Sb3$^{i}$—Zr1—Sb7$^{ii}$ | 138.75 (3) | Sb1—Sb2—Sb2$^{xii}$ | 129.982 (17) |
| Sb4$^{i}$—Zr1—Sb7$^{i}$ | 83.092 (15) | Sb2$^{xi}$—Sb2—Sb2$^{xii}$ | 75.00 (2) |
| Sb4$^{ii}$—Zr1—Sb7$^{i}$ | 136.09 (3) | Ni1$^{i}$—Sb2—Sb4$^{x}$ | 107.40 (3) |
| Sb6—Zr1—Sb7$^{i}$ | 62.103 (16) | Ni1$^{ii}$—Sb2—Sb4$^{x}$ | 53.08 (2) |
| Sb1$^{iii}$—Zr1—Sb7$^{i}$ | 140.627 (10) | Zr2$^{ii}$—Sb2—Sb4$^{x}$ | 101.431 (12) |
| Sb3$^{ii}$—Zr1—Sb7$^{i}$ | 138.75 (3) | Zr2$^{i}$—Sb2—Sb4$^{x}$ | 169.95 (2) |
| Sb3$^{i}$—Zr1—Sb7$^{i}$ | 86.630 (15) | Zr2$^{viii}$—Sb2—Sb4$^{x}$ | 55.446 (14) |
| Sb7$^{ii}$—Zr1—Sb7$^{i}$ | 78.71 (2) | Sb1—Sb2—Sb4$^{x}$ | 60.840 (12) |
| Sb4$^{i}$—Zr1—Sb5 | 75.722 (19) | Sb2$^{xi}$—Sb2—Sb4$^{x}$ | 69.745 (15) |
| Sb4$^{ii}$—Zr1—Sb5 | 75.722 (19) | Sb2$^{xii}$—Sb2—Sb4$^{x}$ | 112.07 (2) |
| Sb6—Zr1—Sb5 | 103.21 (2) | Ni1$^{i}$—Sb2—Sb4$^{ix}$ | 53.08 (2) |
| Sb1$^{iii}$—Zr1—Sb5 | 128.31 (3) | Ni1$^{ii}$—Sb2—Sb4$^{ix}$ | 107.40 (3) |
| Sb3$^{ii}$—Zr1—Sb5 | 140.115 (11) | Zr2$^{ii}$—Sb2—Sb4$^{ix}$ | 169.95 (2) |
| Sb3$^{i}$—Zr1—Sb5 | 140.115 (11) | Zr2$^{i}$—Sb2—Sb4$^{ix}$ | 101.431 (12) |
| Sb7$^{ii}$—Zr1—Sb5 | 60.504 (16) | Zr2$^{viii}$—Sb2—Sb4$^{ix}$ | 55.446 (14) |
| Sb7$^{i}$—Zr1—Sb5 | 60.504 (16) | Sb1—Sb2—Sb4$^{ix}$ | 60.840 (12) |
| Sb6$^{iv}$—Zr2—Sb6$^{v}$ | 83.52 (2) | Sb2$^{xi}$—Sb2—Sb4$^{ix}$ | 112.07 (2) |
| Sb6$^{iv}$—Zr2—Sb4$^{ii}$ | 83.851 (14) | Sb2$^{xii}$—Sb2—Sb4$^{ix}$ | 69.745 (15) |
| Sb6$^{v}$—Zr2—Sb4$^{ii}$ | 141.21 (3) | Sb4$^{x}$—Sb2—Sb4$^{ix}$ | 72.732 (15) |
| Sb6$^{iv}$—Zr2—Sb4$^{i}$ | 141.21 (3) | Zr3$^{ii}$—Sb3—Zr3$^{i}$ | 81.94 (2) |
| Sb6$^{v}$—Zr2—Sb4$^{i}$ | 83.851 (14) | Zr3$^{ii}$—Sb3—Zr1$^{v}$ | 139.58 (2) |
| Sb4$^{ii}$—Zr2—Sb4$^{i}$ | 83.45 (2) | Zr3$^{i}$—Sb3—Zr1$^{v}$ | 85.597 (17) |
| Sb6$^{iv}$—Zr2—Sb2$^{v}$ | 134.23 (3) | Zr3$^{ii}$—Sb3—Zr1$^{iv}$ | 85.597 (16) |
| Sb6$^{v}$—Zr2—Sb2$^{v}$ | 79.287 (15) | Zr3$^{i}$—Sb3—Zr1$^{iv}$ | 139.58 (2) |
| Sb4$^{ii}$—Zr2—Sb2$^{v}$ | 133.05 (3) | Zr1$^{v}$—Sb3—Zr1$^{iv}$ | 79.45 (2) |
| Sb4$^{i}$—Zr2—Sb2$^{v}$ | 78.456 (15) | Zr3$^{ii}$—Sb3—Zr3$^{xiii}$ | 107.962 (18) |
| Sb6$^{iv}$—Zr2—Sb2$^{iv}$ | 79.287 (15) | Zr3$^{i}$—Sb3—Zr3$^{xiii}$ | 107.962 (18) |
| Sb6$^{v}$—Zr2—Sb2$^{iv}$ | 134.23 (3) | Zr1$^{v}$—Sb3—Zr3$^{xiii}$ | 112.458 (19) |
| Sb4$^{ii}$—Zr2—Sb2$^{iv}$ | 78.456 (15) | Zr1$^{iv}$—Sb3—Zr3$^{xiii}$ | 112.458 (19) |
| Sb4$^{i}$—Zr2—Sb2$^{iv}$ | 133.05 (3) | Zr3$^{ii}$—Sb3—Sb1$^{ix}$ | 162.39 (2) |
| Sb2$^{v}$—Zr2—Sb2$^{iv}$ | 83.09 (2) | Zr3$^{i}$—Sb3—Sb1$^{ix}$ | 99.468 (13) |
| Sb6$^{iv}$—Zr2—Sb2$^{iii}$ | 137.839 (11) | Zr1$^{v}$—Sb3—Sb1$^{ix}$ | 57.799 (16) |
| Sb6$^{v}$—Zr2—Sb2$^{iii}$ | 137.839 (11) | Zr1$^{iv}$—Sb3—Sb1$^{ix}$ | 103.64 (2) |

| | | | |
|---|---|---|---|
| Sb4$^{ii}$—Zr2—Sb2$^{iii}$ | 67.583 (16) | Zr3$^{xiii}$—Sb3—Sb1$^{ix}$ | 54.764 (13) |
| Sb4$^{i}$—Zr2—Sb2$^{iii}$ | 67.583 (16) | Zr3$^{ii}$—Sb3—Sb1$^{x}$ | 99.468 (13) |
| Sb2$^{v}$—Zr2—Sb2$^{iii}$ | 65.474 (17) | Zr3$^{i}$—Sb3—Sb1$^{x}$ | 162.39 (2) |
| Sb2$^{iv}$—Zr2—Sb2$^{iii}$ | 65.474 (17) | Zr1$^{v}$—Sb3—Sb1$^{x}$ | 103.64 (2) |
| Sb6$^{iv}$—Zr2—Sb5 | 63.641 (17) | Zr1$^{iv}$—Sb3—Sb1$^{x}$ | 57.799 (15) |
| Sb6$^{v}$—Zr2—Sb5 | 63.641 (17) | Zr3$^{xiii}$—Sb3—Sb1$^{x}$ | 54.764 (13) |
| Sb4$^{ii}$—Zr2—Sb5 | 77.886 (19) | Sb1$^{ix}$—Sb3—Sb1$^{x}$ | 73.942 (15) |
| Sb4$^{i}$—Zr2—Sb5 | 77.886 (19) | Ni1$^{xiii}$—Sb4—Zr2$^{v}$ | 106.24 (2) |
| Sb2$^{v}$—Zr2—Sb5 | 137.621 (11) | Ni1$^{xiii}$—Sb4—Zr2$^{iv}$ | 106.24 (2) |
| Sb2$^{iv}$—Zr2—Sb5 | 137.621 (12) | Zr2$^{v}$—Sb4—Zr2$^{iv}$ | 83.45 (2) |
| Sb2$^{iii}$—Zr2—Sb5 | 132.94 (3) | Ni1$^{xiii}$—Sb4—Zr1$^{iv}$ | 108.49 (2) |
| Sb6$^{iv}$—Zr2—Sb7 | 58.813 (15) | Zr2$^{v}$—Sb4—Zr1$^{iv}$ | 145.27 (2) |
| Sb6$^{v}$—Zr2—Sb7 | 58.813 (15) | Zr2$^{iv}$—Sb4—Zr1$^{iv}$ | 86.535 (17) |
| Sb4$^{ii}$—Zr2—Sb7 | 137.823 (12) | Ni1$^{xiii}$—Sb4—Zr1$^{v}$ | 108.49 (2) |
| Sb4$^{i}$—Zr2—Sb7 | 137.823 (12) | Zr2$^{v}$—Sb4—Zr1$^{v}$ | 86.535 (17) |
| Sb2$^{v}$—Zr2—Sb7 | 76.068 (18) | Zr2$^{iv}$—Sb4—Zr1$^{v}$ | 145.27 (2) |
| Sb2$^{iv}$—Zr2—Sb7 | 76.068 (18) | Zr1$^{iv}$—Sb4—Zr1$^{v}$ | 83.03 (2) |
| Sb2$^{iii}$—Zr2—Sb7 | 127.55 (2) | Ni1$^{xiii}$—Sb4—Sb1$^{x}$ | 50.403 (16) |
| Sb5—Zr2—Sb7 | 99.51 (2) | Zr2$^{v}$—Sb4—Sb1$^{x}$ | 155.92 (2) |
| Sb1$^{iv}$—Zr3—Sb1$^{v}$ | 83.21 (2) | Zr2$^{iv}$—Sb4—Sb1$^{x}$ | 96.928 (13) |
| Sb1$^{iv}$—Zr3—Sb3$^{v}$ | 136.27 (3) | Zr1$^{iv}$—Sb4—Sb1$^{x}$ | 58.375 (16) |
| Sb1$^{v}$—Zr3—Sb3$^{v}$ | 81.481 (15) | Zr1$^{v}$—Sb4—Sb1$^{x}$ | 105.36 (2) |
| Sb1$^{iv}$—Zr3—Sb3$^{iv}$ | 81.481 (16) | Ni1$^{xiii}$—Sb4—Sb1$^{ix}$ | 50.403 (16) |
| Sb1$^{v}$—Zr3—Sb3$^{iv}$ | 136.27 (3) | Zr2$^{v}$—Sb4—Sb1$^{ix}$ | 96.928 (13) |
| Sb3$^{v}$—Zr3—Sb3$^{iv}$ | 81.94 (2) | Zr2$^{iv}$—Sb4—Sb1$^{ix}$ | 155.92 (2) |
| Sb1$^{iv}$—Zr3—Sb5$^{iv}$ | 77.172 (16) | Zr1$^{iv}$—Sb4—Sb1$^{ix}$ | 105.36 (2) |
| Sb1$^{v}$—Zr3—Sb5$^{iv}$ | 130.41 (3) | Zr1$^{v}$—Sb4—Sb1$^{ix}$ | 58.375 (16) |
| Sb3$^{v}$—Zr3—Sb5$^{iv}$ | 140.80 (3) | Sb1$^{x}$—Sb4—Sb1$^{ix}$ | 73.066 (15) |
| Sb3$^{iv}$—Zr3—Sb5$^{iv}$ | 85.155 (14) | Ni1$^{xiii}$—Sb4—Sb2$^{x}$ | 49.662 (16) |
| Sb1$^{iv}$—Zr3—Sb5$^{v}$ | 130.41 (3) | Zr2$^{v}$—Sb4—Sb2$^{x}$ | 104.14 (2) |
| Sb1$^{v}$—Zr3—Sb5$^{v}$ | 77.172 (15) | Zr2$^{iv}$—Sb4—Sb2$^{x}$ | 56.972 (16) |
| Sb3$^{v}$—Zr3—Sb5$^{v}$ | 85.155 (14) | Zr1$^{iv}$—Sb4—Sb2$^{x}$ | 97.880 (13) |
| Sb3$^{iv}$—Zr3—Sb5$^{v}$ | 140.80 (3) | Zr1$^{v}$—Sb4—Sb2$^{x}$ | 157.39 (2) |
| Sb5$^{iv}$—Zr3—Sb5$^{v}$ | 81.89 (2) | Sb1$^{x}$—Sb4—Sb2$^{x}$ | 57.912 (14) |
| Sb1$^{iv}$—Zr3—Sb6$^{vi}$ | 136.371 (13) | Sb1$^{ix}$—Sb4—Sb2$^{x}$ | 100.063 (17) |
| Sb1$^{v}$—Zr3—Sb6$^{vi}$ | 136.371 (13) | Ni1$^{xiii}$—Sb4—Sb2$^{ix}$ | 49.662 (16) |
| Sb3$^{v}$—Zr3—Sb6$^{vi}$ | 77.956 (19) | Zr2$^{v}$—Sb4—Sb2$^{ix}$ | 56.972 (16) |
| Sb3$^{iv}$—Zr3—Sb6$^{vi}$ | 77.956 (19) | Zr2$^{iv}$—Sb4—Sb2$^{ix}$ | 104.14 (2) |
| Sb5$^{iv}$—Zr3—Sb6$^{vi}$ | 63.164 (17) | Zr1$^{iv}$—Sb4—Sb2$^{ix}$ | 157.39 (2) |
| Sb5$^{v}$—Zr3—Sb6$^{vi}$ | 63.164 (17) | Zr1$^{v}$—Sb4—Sb2$^{ix}$ | 97.880 (13) |
| Sb1$^{iv}$—Zr3—Sb3$^{vii}$ | 64.388 (16) | Sb1$^{x}$—Sb4—Sb2$^{ix}$ | 100.063 (17) |

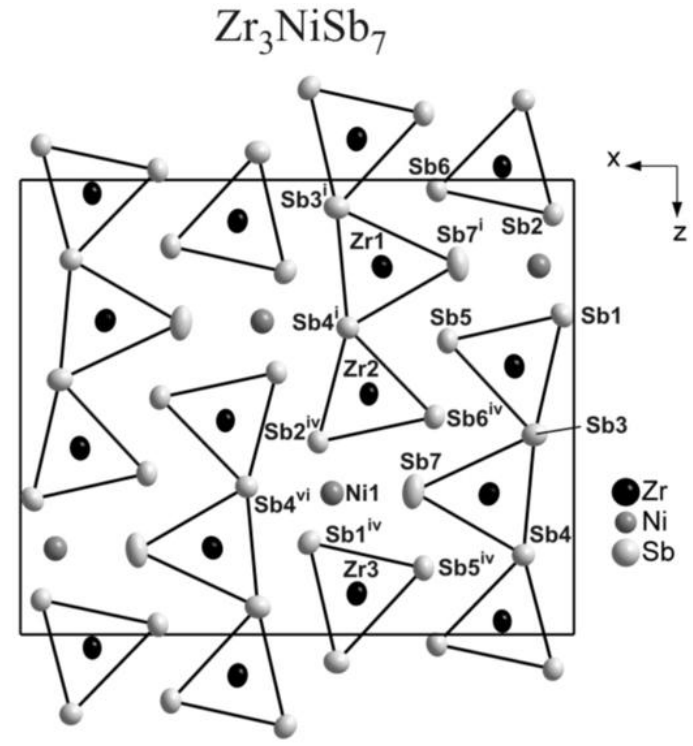
| | | | |
|---|---|---|---|
| Sb1$^{v}$—Zr3—Sb3$^{vii}$ | 64.388 (16) | Sb1$^{ix}$—Sb4—Sb2$^{ix}$ | 57.912 (14) |
| Sb3$^{v}$—Zr3—Sb3$^{vii}$ | 72.038 (18) | Sb2$^{x}$—Sb4—Sb2$^{ix}$ | 72.734 (15) |
| Sb3$^{iv}$—Zr3—Sb3$^{vii}$ | 72.038 (18) | Zr3$^{ii}$—Sb5—Zr3$^{i}$ | 81.89 (2) |
| Sb5$^{iv}$—Zr3—Sb3$^{vii}$ | 137.351 (12) | Zr3$^{ii}$—Sb5—Zr2 | 115.73 (2) |
| Sb5$^{v}$—Zr3—Sb3$^{vii}$ | 137.351 (12) | Zr3$^{i}$—Sb5—Zr2 | 115.73 (2) |
| Sb6$^{vi}$—Zr3—Sb3$^{vii}$ | 139.85 (3) | Zr3$^{ii}$—Sb5—Zr1 | 131.970 (16) |
| Sb1$^{iv}$—Zr3—Ni1 | 49.295 (15) | Zr3$^{i}$—Sb5—Zr1 | 131.970 (16) |
| Sb1$^{v}$—Zr3—Ni1 | 49.295 (15) | Zr2—Sb5—Zr1 | 82.62 (2) |
| Sb3$^{v}$—Zr3—Ni1 | 130.769 (18) | Zr3$^{ii}$—Sb5—Sb7$^{ii}$ | 74.105 (16) |
| Sb3$^{iv}$—Zr3—Ni1 | 130.769 (18) | Zr3$^{i}$—Sb5—Sb7$^{ii}$ | 123.11 (2) |
| Sb5$^{iv}$—Zr3—Ni1 | 84.63 (2) | Zr2—Sb5—Sb7$^{ii}$ | 121.163 (17) |
| Sb5$^{v}$—Zr3—Ni1 | 84.63 (2) | Zr1—Sb5—Sb7$^{ii}$ | 59.174 (15) |
| Sb6$^{vi}$—Zr3—Ni1 | 136.18 (3) | Zr3$^{ii}$—Sb5—Sb7$^{i}$ | 123.11 (2) |
| Sb3$^{vii}$—Zr3—Ni1 | 83.97 (2) | Zr3$^{i}$—Sb5—Sb7$^{i}$ | 74.105 (16) |
| Sb7—Ni1—Sb2$^{iv}$ | 99.26 (3) | Zr2—Sb5—Sb7$^{i}$ | 121.163 (17) |
| Sb7—Ni1—Sb2$^{v}$ | 99.26 (3) | Zr1—Sb5—Sb7$^{i}$ | 59.174 (15) |
| Sb2$^{iv}$—Ni1—Sb2$^{v}$ | 98.71 (3) | Sb7$^{ii}$—Sb5—Sb7$^{i}$ | 77.460 (18) |
| Sb7—Ni1—Sb1$^{v}$ | 106.99 (3) | Zr3$^{ii}$—Sb5—Sb6$^{v}$ | 107.25 (2) |
| Sb2$^{iv}$—Ni1—Sb1$^{v}$ | 153.71 (4) | Zr3$^{i}$—Sb5—Sb6$^{v}$ | 58.444 (16) |
| Sb2$^{v}$—Ni1—Sb1$^{v}$ | 75.925 (16) | Zr2—Sb5—Sb6$^{v}$ | 57.301 (14) |
| Sb7—Ni1—Sb1$^{iv}$ | 106.99 (3) | Zr1—Sb5—Sb6$^{v}$ | 119.266 (16) |
| Sb2$^{iv}$—Ni1—Sb1$^{iv}$ | 75.925 (16) | Sb7$^{ii}$—Sb5—Sb6$^{v}$ | 178.23 (2) |
| Sb2$^{v}$—Ni1—Sb1$^{iv}$ | 153.71 (4) | Sb7$^{i}$—Sb5—Sb6$^{v}$ | 102.523 (11) |
| Sb1$^{v}$—Ni1—Sb1$^{iv}$ | 97.37 (3) | Zr3$^{ii}$—Sb5—Sb6$^{iv}$ | 58.444 (16) |
| Sb7—Ni1—Sb4$^{vii}$ | 174.50 (4) | Zr3$^{i}$—Sb5—Sb6$^{iv}$ | 107.25 (2) |
| Sb2$^{iv}$—Ni1—Sb4$^{vii}$ | 77.26 (2) | Zr2—Sb5—Sb6$^{iv}$ | 57.302 (14) |
| Sb2$^{v}$—Ni1—Sb4$^{vii}$ | 77.26 (2) | Zr1—Sb5—Sb6$^{iv}$ | 119.266 (16) |
| Sb1$^{v}$—Ni1—Sb4$^{vii}$ | 76.46 (2) | Sb7$^{ii}$—Sb5—Sb6$^{iv}$ | 102.523 (11) |
| Sb1$^{iv}$—Ni1—Sb4$^{vii}$ | 76.46 (2) | Sb7$^{i}$—Sb5—Sb6$^{iv}$ | 178.23 (2) |
| Sb7—Ni1—Zr3 | 77.45 (3) | Sb6$^{v}$—Sb5—Sb6$^{iv}$ | 77.438 (18) |
| Sb2$^{iv}$—Ni1—Zr3 | 130.623 (17) | Zr2$^{ii}$—Sb6—Zr2$^{i}$ | 83.52 (2) |
| Sb2$^{v}$—Ni1—Zr3 | 130.623 (17) | Zr2$^{ii}$—Sb6—Zr1 | 127.554 (17) |
| Sb1$^{v}$—Ni1—Zr3 | 59.04 (2) | Zr2$^{i}$—Sb6—Zr1 | 127.554 (17) |
| Sb1$^{iv}$—Ni1—Zr3 | 59.04 (2) | Zr2$^{ii}$—Sb6—Zr3$^{xiv}$ | 117.435 (19) |
| Sb4$^{vii}$—Ni1—Zr3 | 108.05 (3) | Zr2$^{i}$—Sb6—Zr3$^{xiv}$ | 117.435 (19) |
| Ni1$^{ii}$—Sb1—Ni1$^{i}$ | 97.37 (3) | Zr1—Sb6—Zr3$^{xiv}$ | 87.06 (2) |
| Ni1$^{ii}$—Sb1—Zr3$^{i}$ | 133.06 (3) | Zr2$^{ii}$—Sb6—Sb5$^{ii}$ | 59.058 (16) |
| Ni1$^{i}$—Sb1—Zr3$^{i}$ | 71.66 (2) | Zr2$^{i}$—Sb6—Sb5$^{ii}$ | 108.60 (2) |
| Ni1$^{ii}$—Sb1—Zr3$^{ii}$ | 71.66 (2) | Zr1—Sb6—Sb5$^{ii}$ | 123.387 (17) |
| Ni1$^{i}$—Sb1—Zr3$^{ii}$ | 133.06 (3) | Zr3$^{xiv}$—Sb6—Sb5$^{ii}$ | 58.391 (14) |
| Zr3$^{i}$—Sb1—Zr3$^{ii}$ | 83.21 (2) | Zr2$^{ii}$—Sb6—Sb5$^{i}$ | 108.60 (2) |

| | | | |
|---|---|---|---|
| Ni1$^{ii}$—Sb1—Zr1$^{viii}$ | 108.17 (2) | Zr2$^{i}$—Sb6—Sb5$^{i}$ | 59.058 (16) |
| Ni1$^{i}$—Sb1—Zr1$^{viii}$ | 108.17 (2) | Zr1—Sb6—Sb5$^{i}$ | 123.387 (17) |
| Zr3$^{i}$—Sb1—Zr1$^{viii}$ | 118.681 (19) | Zr3$^{xiv}$—Sb6—Sb5$^{i}$ | 58.391 (14) |
| Zr3$^{ii}$—Sb1—Zr1$^{viii}$ | 118.681 (19) | Sb5$^{ii}$—Sb6—Sb5$^{i}$ | 77.437 (18) |
| Ni1$^{ii}$—Sb1—Sb2 | 51.665 (19) | Zr2$^{ii}$—Sb6—Sb7$^{i}$ | 117.02 (2) |
| Ni1$^{i}$—Sb1—Sb2 | 51.665 (19) | Zr2$^{i}$—Sb6—Sb7$^{i}$ | 67.743 (16) |
| Zr3$^{i}$—Sb1—Sb2 | 119.976 (18) | Zr1—Sb6—Sb7$^{i}$ | 60.643 (16) |
| Zr3$^{ii}$—Sb1—Sb2 | 119.976 (18) | Zr3$^{xiv}$—Sb6—Sb7$^{i}$ | 125.528 (15) |
| Zr1$^{viii}$—Sb1—Sb2 | 98.147 (19) | Sb5$^{ii}$—Sb6—Sb7$^{i}$ | 175.37 (2) |
| Ni1$^{ii}$—Sb1—Sb3$^{ix}$ | 164.74 (2) | Sb5$^{i}$—Sb6—Sb7$^{i}$ | 102.379 (11) |
| Ni1$^{i}$—Sb1—Sb3$^{ix}$ | 93.514 (18) | Zr2$^{ii}$—Sb6—Sb7$^{ii}$ | 67.743 (16) |
| Zr3$^{i}$—Sb1—Sb3$^{ix}$ | 60.848 (17) | Zr2$^{i}$—Sb6—Sb7$^{ii}$ | 117.02 (2) |
| Zr3$^{ii}$—Sb1—Sb3$^{ix}$ | 108.15 (2) | Zr1—Sb6—Sb7$^{ii}$ | 60.643 (16) |
| Zr1$^{viii}$—Sb1—Sb3$^{ix}$ | 57.951 (14) | Zr3$^{xiv}$—Sb6—Sb7$^{ii}$ | 125.528 (15) |
| Sb2—Sb1—Sb3$^{ix}$ | 131.792 (14) | Sb5$^{ii}$—Sb6—Sb7$^{ii}$ | 102.379 (11) |
| Ni1$^{ii}$—Sb1—Sb3$^{x}$ | 93.514 (18) | Sb5$^{i}$—Sb6—Sb7$^{ii}$ | 175.37 (2) |
| Ni1$^{i}$—Sb1—Sb3$^{x}$ | 164.74 (2) | Sb7$^{i}$—Sb6—Sb7$^{ii}$ | 77.422 (18) |
| Zr3$^{i}$—Sb1—Sb3$^{x}$ | 108.15 (2) | Ni1—Sb7—Zr1$^{iv}$ | 140.543 (10) |
| Zr3$^{ii}$—Sb1—Sb3$^{x}$ | 60.848 (17) | Ni1—Sb7—Zr1$^{v}$ | 140.543 (10) |
| Zr1$^{viii}$—Sb1—Sb3$^{x}$ | 57.951 (14) | Zr1$^{iv}$—Sb7—Zr1$^{v}$ | 78.71 (2) |
| Sb2—Sb1—Sb3$^{x}$ | 131.792 (13) | Ni1—Sb7—Sb5$^{v}$ | 94.92 (2) |
| Sb3$^{ix}$—Sb1—Sb3$^{x}$ | 73.942 (16) | Zr1$^{iv}$—Sb7—Sb5$^{v}$ | 107.36 (2) |
| Ni1$^{ii}$—Sb1—Sb4$^{x}$ | 53.14 (2) | Zr1$^{v}$—Sb7—Sb5$^{v}$ | 60.321 (16) |
| Ni1$^{i}$—Sb1—Sb4$^{x}$ | 107.12 (3) | Ni1—Sb7—Sb5$^{iv}$ | 94.92 (2) |
| Zr3$^{i}$—Sb1—Sb4$^{x}$ | 173.544 (17) | Zr1$^{iv}$—Sb7—Sb5$^{iv}$ | 60.321 (16) |
| Zr3$^{ii}$—Sb1—Sb4$^{x}$ | 101.722 (12) | Zr1$^{v}$—Sb7—Sb5$^{iv}$ | 107.36 (2) |
| Zr1$^{viii}$—Sb1—Sb4$^{x}$ | 55.321 (14) | Sb5$^{v}$—Sb7—Sb5$^{iv}$ | 77.460 (18) |
| Sb2—Sb1—Sb4$^{x}$ | 61.249 (13) | Ni1—Sb7—Sb6$^{iv}$ | 103.12 (2) |
| Sb3$^{ix}$—Sb1—Sb4$^{x}$ | 113.263 (18) | Zr1$^{iv}$—Sb7—Sb6$^{iv}$ | 57.253 (16) |
| Sb3$^{x}$—Sb1—Sb4$^{x}$ | 71.272 (14) | Zr1$^{v}$—Sb7—Sb6$^{iv}$ | 104.61 (2) |
| Ni1$^{ii}$—Sb1—Sb4$^{ix}$ | 107.12 (3) | Sb5$^{v}$—Sb7—Sb6$^{iv}$ | 161.93 (2) |
| Ni1$^{i}$—Sb1—Sb4$^{ix}$ | 53.14 (2) | Sb5$^{iv}$—Sb7—Sb6$^{iv}$ | 99.677 (12) |
| Zr3$^{i}$—Sb1—Sb4$^{ix}$ | 101.722 (12) | Ni1—Sb7—Sb6$^{v}$ | 103.12 (2) |
| Zr3$^{ii}$—Sb1—Sb4$^{ix}$ | 173.544 (17) | Zr1$^{iv}$—Sb7—Sb6$^{v}$ | 104.61 (2) |
| Zr1$^{viii}$—Sb1—Sb4$^{ix}$ | 55.321 (14) | Zr1$^{v}$—Sb7—Sb6$^{v}$ | 57.253 (16) |
| Sb2—Sb1—Sb4$^{ix}$ | 61.249 (13) | Sb5$^{v}$—Sb7—Sb6$^{v}$ | 99.677 (12) |
| Sb3$^{ix}$—Sb1—Sb4$^{ix}$ | 71.272 (14) | Sb5$^{iv}$—Sb7—Sb6$^{v}$ | 161.93 (2) |
| Sb3$^{x}$—Sb1—Sb4$^{ix}$ | 113.263 (18) | Sb6$^{iv}$—Sb7—Sb6$^{v}$ | 77.422 (18) |
| Sb4$^{x}$—Sb1—Sb4$^{ix}$ | 73.066 (15) | Ni1—Sb7—Zr2 | 66.67 (2) |
| Ni1$^{i}$—Sb2—Ni1$^{ii}$ | 98.71 (3) | Zr1$^{iv}$—Sb7—Zr2 | 110.11 (2) |
| Ni1$^{i}$—Sb2—Zr2$^{ii}$ | 136.93 (3) | Zr1$^{v}$—Sb7—Zr2 | 110.11 (2) |

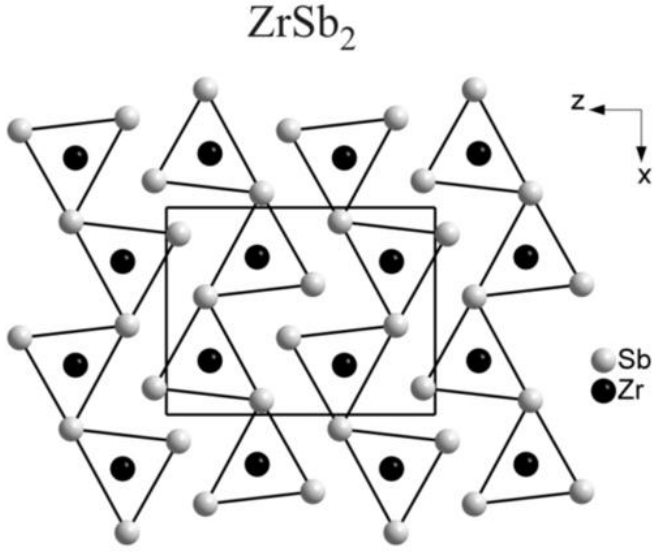
| | | | |
|---|---|---|---|
| Ni1$^{ii}$—Sb2—Zr2$^{ii}$ | 73.99 (2) | Sb5$^{v}$—Sb7—Zr2 | 138.240 (11) |
| Ni1$^{i}$—Sb2—Zr2$^{i}$ | 73.99 (2) | Sb5$^{iv}$—Sb7—Zr2 | 138.240 (11) |
| Ni1$^{ii}$—Sb2—Zr2$^{i}$ | 136.93 (3) | Sb6$^{iv}$—Sb7—Zr2 | 53.446 (14) |
| Zr2$^{ii}$—Sb2—Zr2$^{i}$ | 83.09 (2) | Sb6$^{v}$—Sb7—Zr2 | 53.446 (14) |

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

**Fig. 1**


Zr3NiSb7
x
z
Sb6
Sb3i
Zr1
Sb7i
Sb2
Sb4i
Sb5
Sb1
Zr2
Sb2iv
Sb6iv
Sb3
Sb7
Sb4vi
Ni1
Sb1iv
Sb4
Zr3
Sb5iv
Zr
Ni
Sb

(*a*)


ZrSb2
z
x
Sb
Zr

(*b*)

**Fig. 2**