

arranged in centrosymmetric pairs. Fig. 1 shows the packing in a stereoscopic *ORTEP* drawing.

The average angles between azide ions and the metal atoms ( $M-N-N$ ) are  $133(15)^\circ$  around Ca and  $114(27)^\circ$  around Cs. Two Cs-N-N angles are below  $90^\circ$ . This results in a rather close contact between Cs and the middle N atoms (Fig. 2). Close contacts of Cs to the central azide N atoms have been observed in several other complex azides (Krischner, Saracoglu & Kratky, 1982).

Bond lengths and angles within the azide groups are as expected (Pringle & Noakes, 1968).

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## Structures of $Ba_8Ga_7$ , $Sr_8Ga_7$ and $Sr_8Al_7$

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**Abstract.**  $Ba_8Ga_7$ ,  $cP60$ ,  $M_r = 1586.8$ , cubic,  $P2_13$ ,  $a = 12.990(6) \text{ \AA}$ ,  $V = 2192 \text{ \AA}^3$ ,  $Z = 4$ ,  $D_x = 4.81 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$ ,  $\mu(\text{Mo } K\alpha) = 23.4 \text{ mm}^{-1}$ ,  $F(000) = 2660$ ,  $R = 0.040$  for 784 reflections.  $Sr_8Ga_7$ ,  $M_r = 1189$ ,  $P2_13$ ,  $a = 12.484(6) \text{ \AA}$ ,  $V = 1946 \text{ \AA}^3$ ,  $D_x = 4.06 \text{ Mg m}^{-3}$ ,  $\mu(\text{Mo } K\alpha) = 32.7 \text{ mm}^{-1}$ ,  $F(000) = 2084$ ,  $R = 0.048$  for 224 reflections.  $Sr_8Al_7$ ,  $M_r = 889.8$ ,  $P2_13$ ,  $a = 12.753(5) \text{ \AA}$ ,  $V = 2074 \text{ \AA}^3$ ,  $D_x = 2.85 \text{ Mg m}^{-3}$ ,  $\mu(\text{Mo } K\alpha) = 21.6 \text{ mm}^{-1}$ ,  $F(000) = 1580$ ,  $R = 0.031$  for 555 reflections. For this last phase a previous structural proposal for equiatomic stoichiometry was revised and the new formula  $Sr_8Al_7$  determined. The three compounds are isotopic and their structure is characterized by the presence of isolated tetrahedral and triangular clusters of Ga (or Al) atoms included in cages of 16 and 13 alkaline-earth atoms, respectively.

**Introduction.** After the structure determination of  $Ba_{10}Ga$  (Fornasini & Merlo, 1979), work was continued on the study of the other alkaline-earth-rich phases present in the Ba–Ga, Sr–Ga (Bruzzone, 1966) and Sr–Al (Bruzzone & Merlo, 1974) systems. This paper deals with the structure determination of the compounds reported as  $Ba_3Ga_2$ ,  $Sr_3Ga_2$  and  $Sr_3Al_2$  in the corresponding phase diagrams. In the case of  $Sr_3Al_2$

a previous structural proposal for equiatomic stoichiometry (Fornasini & Merlo, 1976) is re-examined.

**Experimental.** Metals used: Ba (99.5 wt%) from Fluka, Switzerland, Sr (99wt%) and Ga (99.999wt%) from Koch-Light, England.  $Ba_3Ga_2$  prepared by melting in a sealed tantalum container, and slowly cooling the alloy; single crystals examined by X-ray precession technique; cubic Laue symmetry  $m\bar{3}$  with the systematic absence of  $h00$  reflections with  $h = 2n + 1$  led univocally to the space group  $P2_13$ . A nearly spherical crystal with mean radius 0.08 mm sealed in a thin glass capillary under vacuum, mounted on an Enraf–Nonius CAD-4 automatic diffractometer, graphite-monochromated Mo  $K\alpha$  radiation; lattice constant determined by least-squares over 25 diffractometer-measured high-angle reflections; equivalent  $hkl$ ,  $lhk$  and  $khl$  reflections collected in the  $\omega$  scan mode up to  $2\theta = 55^\circ$ . Intensities corrected for  $L_p$  and absorption, applying both the semi-empirical method of North, Phillips & Mathews (1968) with  $\psi$ -scan data of four top reflections and the spherical correction, the maximum transmission-factor ratio being 2.4; equivalents merged ( $R_{\text{int}} = 0.069$ ), 944 unique reflections obtained, 784 with  $F_o > 2\sigma(F_o)$  used in the refinement. The structure already proposed for SrAl (Fornasini & Merlo, 1976)

was adopted as a starting model and refined to an  $R$  value of 0.052; however, for the atomic position 4(a) labelled Ga(4), which was the same as Al(4) in SrAl, the temperature factor increased to a value 14 times that of the other Ga atoms. The elimination of this atom gave a much better agreement between the observed and calculated structure factors, especially for some low-angle reflections, while no peak corresponding to this position was found in the difference Fourier synthesis. The composition of the crystal was thus established as Ba<sub>8</sub>Ga<sub>7</sub>. Anisotropic refinement, 46 parameters, weight  $w = 1/\sigma^2(F_o)$ ,  $R = 0.040$ ,  $wR = 0.031$ ; maximum shift to error 0.02; maximum and minimum height in final difference Fourier synthesis 2.4 and  $-1.5 \text{ e } \text{Å}^{-3}$ , respectively.

Sr<sub>8</sub>Ga<sub>7</sub> prepared by melting and annealed one month at 923 K. Plate-like single crystal  $0.03 \times 0.09 \times 0.14 \text{ mm}$ ,  $\omega$ - $\theta$  scan mode, 2576 reflections corrected for absorption using  $\psi$ -scan data of a top reflection (maximum transmission-factor ratio 3.2); equivalents merged ( $R_{\text{int}} = 0.24$ ), 847 unique reflections obtained, 224 with  $F_o > 2\sigma(F_o)$  retained in the refinement, starting from the same model of Ba<sub>8</sub>Ga<sub>7</sub>; isotropic refinement of 23 parameters,  $R = 0.048$ ,  $wR = 0.031$ ; maximum shift to error 0.01; maximum and minimum height in final difference map 1.6 and  $-1.6 \text{ e } \text{Å}^{-3}$ , respectively.

At this point it was decided to re-examine the structure already reported for SrAl, since at that time the intensities had been collected photographically, with a precision certainly inferior to that provided today by an automatic diffractometer. The data collection was made on the same plate-like crystal, still in good condition, in the  $\omega$ - $\theta$  scan mode with the option *FLAT*, allowing each reflection to be measured at the appropriate  $\psi$  angle minimizing absorption. 2724 equivalents merged ( $R_{\text{int}} = 0.049$ ), 894 unique reflections obtained, 555 with  $F_o > 2\sigma(F_o)$  employed in the refinement, starting from the old atomic coordinates, but eliminating Al(4); isotropic and then anisotropic refinement of 46 parameters,  $R = 0.031$ ,  $wR = 0.017$ ; maximum shift to error 0.1; no peak corresponding to the Al(4) position on the difference Fourier map, with 1.1 and  $-0.9 \text{ e } \text{Å}^{-3}$  as maximum and minimum height, respectively.

While changing the signs of the positional parameters of the three compounds gave very small improvements for Ba<sub>8</sub>Ga<sub>7</sub> and Sr<sub>8</sub>Ga<sub>7</sub>, the  $R$  value for Sr<sub>8</sub>Al<sub>7</sub> was lowered from 0.045 to 0.031 and this was considered as indicative for the choice of the absolute configuration for all three structures.

The *SHELX76* program was used (Sheldrick, 1976), taking atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Refinements were based on  $F$ . No secondary-extinction corrections. The lattice constants quoted in the *Abstract* for Sr<sub>8</sub>Ga<sub>7</sub> and Sr<sub>8</sub>Al<sub>7</sub> are those already derived from powder patterns (Fornasini & Merlo, 1976), and are very close to the diffractometer-determined values.

**Discussion.** Table 1 gives atomic coordinates and thermal parameters for the three compounds examined.\* Table 2 reports the interatomic distances for the three phases according to the 'gap criterion' (Bruzzone, Fornasini & Merlo, 1970). They have normal values with 6–8% contraction with respect to the sum of the metallic radii for coordination number (CN) 12 as given by Teatum, Gschneidner & Waber (1960).

Fig. 1 shows the projection of the structure of Ba<sub>8</sub>Ga<sub>7</sub> along the edge of the cubic cell. The structure is characterized by the presence of isolated tetrahedral and triangular clusters of Ga atoms. Three Ga(1) and a Ga(3) form a tetrahedron surrounded by a cage of 16 Ba atoms, which are arranged at the vertices of a CN 16 Frank & Kasper polyhedron or Friauf polyhedron (a truncated tetrahedron with each hexagonal face capped). Three Ga(2) atoms form a triangle surrounded by a cage of 13 Ba atoms. There are four tetrahedra and four triangles in the elementary cell, with their centres located according to the Na and Cl positions, respectively, in a very distorted NaCl lattice.

Ga(1) and Ga(3) both have the same coordination polyhedron: a deformed octahedron of Ba with a face enlarged to allow contacts with the three other Ga atoms forming the tetrahedron. This is very similar to

\* Lists of structure factors for all three compounds and anisotropic thermal parameters for Ba<sub>8</sub>Ga<sub>7</sub> and Sr<sub>8</sub>Al<sub>7</sub> have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38530 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic parameters for Ba<sub>8</sub>Ga<sub>7</sub>, Sr<sub>8</sub>Ga<sub>7</sub> and Sr<sub>8</sub>Al<sub>7</sub> with e.s.d.'s in parentheses*

The thermal factors are defined as  $T = \exp[-8\pi^2 10^{-2} U(\sin\theta/\lambda)^2]$ , with  $U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$ .

|                                     | $x$        | $y$        | $z$        | $U_{\text{eq}}$ or $U(\text{Å}^2)$ |
|-------------------------------------|------------|------------|------------|------------------------------------|
| <b>Ba<sub>8</sub>Ga<sub>7</sub></b> |            |            |            |                                    |
| Ba(1)                               | 0.7987 (2) | 0.5579 (1) | 0.0011 (1) | 2.3 (1)*                           |
| Ba(2)                               | 0.5186 (1) | 0.7025 (2) | 0.9387 (1) | 2.0 (1)*                           |
| Ba(3)                               | 0.8148 (1) | 0.8148 (1) | 0.8148 (1) | 2.1 (1)*                           |
| Ba(4)                               | 0.1873 (1) | 0.1873 (1) | 0.1873 (1) | 1.8 (1)*                           |
| Ga(1)                               | 0.7610 (2) | 0.1941 (3) | 0.5760 (3) | 2.1 (2)*                           |
| Ga(2)                               | 0.7467 (2) | 0.6867 (3) | 0.5831 (3) | 2.1 (2)*                           |
| Ga(3)                               | 0.4311 (2) | 0.4311 (2) | 0.4311 (2) | 2.0 (1)*                           |
| <b>Sr<sub>8</sub>Ga<sub>7</sub></b> |            |            |            |                                    |
| Sr(1)                               | 0.8040 (6) | 0.5571 (5) | 0.0035 (5) | 1.8 (2)                            |
| Sr(2)                               | 0.5203 (4) | 0.7021 (6) | 0.9378 (5) | 1.7 (2)                            |
| Sr(3)                               | 0.8141 (6) | 0.8141 (6) | 0.8141 (6) | 2.1 (3)                            |
| Sr(4)                               | 0.1854 (5) | 0.1854 (5) | 0.1854 (5) | 1.2 (3)                            |
| Ga(1)                               | 0.7559 (5) | 0.1946 (7) | 0.5741 (7) | 2.2 (3)                            |
| Ga(2)                               | 0.7516 (5) | 0.6863 (7) | 0.5785 (7) | 2.0 (3)                            |
| Ga(3)                               | 0.4331 (5) | 0.4331 (5) | 0.4331 (5) | 1.4 (3)                            |
| <b>Sr<sub>8</sub>Al<sub>7</sub></b> |            |            |            |                                    |
| Sr(1)                               | 0.8056 (2) | 0.5594 (1) | 0.0035 (1) | 1.9 (1)*                           |
| Sr(2)                               | 0.5204 (1) | 0.6997 (1) | 0.9388 (1) | 2.0 (1)*                           |
| Sr(3)                               | 0.8148 (1) | 0.8148 (1) | 0.8148 (1) | 2.0 (1)*                           |
| Sr(4)                               | 0.1849 (1) | 0.1849 (1) | 0.1849 (1) | 1.8 (1)*                           |
| Al(1)                               | 0.7554 (3) | 0.1940 (4) | 0.5749 (4) | 2.1 (3)*                           |
| Al(2)                               | 0.7504 (3) | 0.6853 (4) | 0.5803 (4) | 1.8 (3)*                           |
| Al(3)                               | 0.4301 (3) | 0.4301 (3) | 0.4301 (3) | 2.1 (2)*                           |

\*  $U_{\text{eq}}$  values.

Table 2. *Interatomic distances (Å) in Ba<sub>8</sub>Ga<sub>7</sub>, Sr<sub>8</sub>Ga<sub>7</sub> and Sr<sub>8</sub>Al<sub>7</sub>*

The e.s.d.'s are < 0.01 Å for Ba<sub>8</sub>Ga<sub>7</sub> and Sr<sub>8</sub>Al<sub>7</sub>, and ≤ 0.01 Å for Sr<sub>8</sub>Ga<sub>7</sub>. The limit for Ba–Ba and Sr–Sr distances is 5 Å, for all other distances 4.4 Å.

| Ba <sub>8</sub> Ga <sub>7</sub> |                   | Sr <sub>8</sub> Ga <sub>7</sub> |                   | Sr <sub>8</sub> Al <sub>7</sub> |                   |
|---------------------------------|-------------------|---------------------------------|-------------------|---------------------------------|-------------------|
| Ba(1)–Ga(2) 3.40                | Ba(2)–Ga(2) 3.43  | Sr(1)–Ga(2) 3.25                | Sr(2)–Ga(2) 3.28  | Sr(1)–Al(2) 3.35                | Sr(2)–Al(2) 3.38  |
| –Ga(1) 3.45                     | –Ga(3) 3.48       | –Ga(1) 3.31                     | –Ga(1) 3.34       | –Al(1) 3.37                     | –Al(1) 3.40       |
| –Ga(2) 3.63                     | –Ga(1) 3.48       | –Ga(3) 3.40                     | –Ga(3) 3.35       | –Al(3) 3.50                     | –Al(3) 3.44       |
| –Ga(3) 3.63                     | –Ga(1) 3.63       | –Ga(2) 3.48                     | –Ga(1) 3.45       | –Al(2) 3.58                     | –Al(1) 3.52       |
| –Ga(2) 3.68                     | –Ga(1) 3.64       | –Ga(2) 3.53                     | –Ga(2) 3.48       | –Al(2) 3.62                     | –Al(2) 3.54       |
| –Ba(3) 4.13                     | –Ga(2) 3.66       | –Sr(3) 3.99                     | –Ga(1) 3.52       | –Sr(3) 4.05                     | –Al(1) 3.61       |
| –Ba(2) 4.16                     | –Ba(3) 4.16       | –Sr(2) 4.02                     | –Sr(1) 4.02       | –Sr(2) 4.12                     | –Sr(3) 4.10       |
| –Ba(2) 4.17                     | –Ba(1) 4.16       | –2Sr(1) 4.04                    | –Sr(3) 4.03       | –2Sr(1) 4.13                    | –Sr(1) 4.12       |
| –Ba(4) 4.27                     | –Ba(1) 4.17       | –Sr(2) 4.06                     | –Sr(1) 4.06       | –Sr(2) 4.14                     | –Sr(1) 4.14       |
| –Ba(2) 4.30                     | –Ba(1) 4.30       | –Sr(2) 4.11                     | –Sr(1) 4.11       | –Sr(2) 4.18                     | –Sr(1) 4.18       |
| –Ba(2) 4.31                     | –Ba(1) 4.31       | –Sr(4) 4.11                     | –Sr(1) 4.14       | –Sr(4) 4.22                     | –Sr(1) 4.26       |
| –2Ba(1) 4.32                    | –Ba(3) 4.42       | –Sr(2) 4.14                     | –Sr(3) 4.22       | –Sr(2) 4.26                     | –Sr(3) 4.33       |
| –Ba(4) 4.39                     | –Ba(4) 4.46       | –Sr(4) 4.20                     | –Sr(4) 4.30       | –Sr(4) 4.29                     | –2Sr(2) 4.38      |
|                                 | –2Ba(2) 4.51      |                                 | –2Sr(2) 4.34      |                                 | –Sr(4) 4.42       |
| Ba(3)–3Ga(1) 3.53               | Ba(4)–3Ga(1) 3.57 | Sr(3)–3Ga(1) 3.36               | Sr(4)–3Ga(2) 3.39 | Sr(3)–3Al(1) 3.44               | Sr(4)–3Al(2) 3.48 |
| –3Ga(2) 3.55                    | –3Ga(2) 3.62      | –3Ga(2) 3.44                    | –3Ga(1) 3.47      | –3Al(2) 3.51                    | –3Al(1) 3.55      |
| –3Ba(1) 4.13                    | –3Ba(1) 4.27      | –3Sr(1) 3.99                    | –3Sr(1) 4.11      | –3Sr(1) 4.10                    | –3Sr(1) 4.22      |
| –3Ba(2) 4.16                    | –3Ba(1) 4.39      | –3Sr(2) 4.03                    | –3Sr(1) 4.20      | –3Sr(2) 4.10                    | –3Sr(1) 4.29      |
| –3Ba(2) 4.42                    | –3Ba(2) 4.46      | –3Sr(2) 4.22                    | –3Sr(2) 4.30      | –3Sr(2) 4.33                    | –3Sr(2) 4.42      |
| Ga(1)–2Ga(1) 2.68               | Ga(2)–2Ga(2) 2.63 | Ga(1)–2Ga(1) 2.67               | Ga(2)–2Ga(2) 2.67 | Al(1)–2Al(1) 2.72               | Al(2)–2Al(2) 2.68 |
| –Ga(3) 2.75                     | –Ba(1) 3.40       | –Ga(3) 2.73                     | –Sr(1) 3.25       | –Al(3) 2.73                     | –Sr(1) 3.35       |
| –Ba(1) 3.45                     | –Ba(2) 3.43       | –Sr(1) 3.31                     | –Sr(2) 3.28       | –Sr(1) 3.37                     | –Sr(1) 3.38       |
| –Ba(2) 3.48                     | –Ba(3) 3.55       | –Sr(2) 3.34                     | –Sr(2) 3.28       | –Sr(2) 3.40                     | –Sr(2) 3.38       |
| –Ba(3) 3.53                     | –Ba(4) 3.62       | –Sr(3) 3.36                     | –Sr(4) 3.39       | –Sr(3) 3.44                     | –Sr(4) 3.48       |
| –Ba(4) 3.57                     | –Ba(1) 3.63       | –Sr(2) 3.45                     | –Sr(3) 3.44       | –Sr(2) 3.52                     | –Sr(3) 3.51       |
| –Ba(2) 3.63                     | –Ba(2) 3.66       | –Sr(4) 3.47                     | –Sr(2) 3.48       | –Sr(4) 3.55                     | –Sr(2) 3.54       |
| –Ba(2) 3.64                     | –Ba(1) 3.68       | –Sr(2) 3.52                     | –Sr(1) 3.48       | –Sr(2) 3.61                     | –Sr(1) 3.58       |
|                                 |                   |                                 | –Sr(1) 3.53       |                                 | –Sr(1) 3.62       |
| Ga(3)–3Ga(1) 2.75               | Ga(3)–3Ga(1) 2.73 | Ga(3)–3Ga(1) 2.73               | Al(3)–3Al(1) 2.73 | Al(3)–3Al(1) 2.73               |                   |
| –3Ba(2) 3.48                    | –3Sr(2) 3.35      | –3Sr(2) 3.35                    | –3Sr(2) 3.44      | –3Sr(2) 3.44                    |                   |
| –3Ba(1) 3.63                    | –3Sr(1) 3.40      | –3Sr(1) 3.40                    | –3Sr(1) 3.50      | –3Sr(1) 3.50                    |                   |

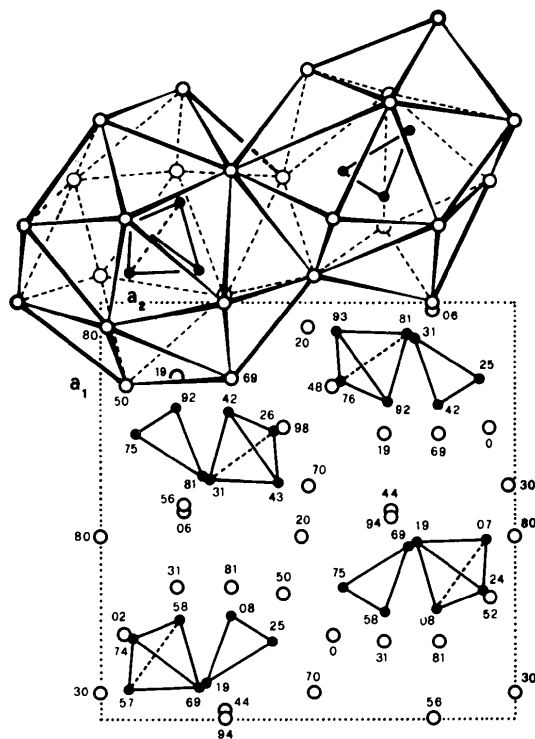


Fig. 1. Projection of the structure of Ba<sub>8</sub>Ga<sub>7</sub> along the edge of the cubic cell. Open circles: Ba; full circles: Ga. The tetrahedra and triangles of Ga are indicated. In the upper part the Ba cages surrounding each tetrahedron (16-vertex polyhedron) and each triangle (13-vertex polyhedron) are drawn.

the environment around Ge in the KGe phase (Busmann, 1961), a structure also containing isolated Ge<sub>4</sub> tetrahedra, each included in a cage of 16 K atoms. Ga(2) is coordinated with seven Ba and two Ga(2) atoms forming a tricapped irregular trigonal prism with one Ga(2) at a vertex of the prism and the other centring a face.

The characteristic bond geometries of the Ga sublattice are partially similar to those found in some polyanionic compounds. Actually, besides the cited KGe, isolated tetrahedral groups of the more electronegative element occur in phases crystallizing in the structure types NaSi, NaGe, NaPb, BaSi<sub>2</sub>. These compounds have been described on the basis of an ionic-covalent model by Zintl, Klemm and Busmann, as reported and extensively used by Schäfer, Eisenmann & Müller (1973). Applying this hypothesis to Ba<sub>8</sub>Ga<sub>7</sub>, the Ga(1) and Ga(3) atoms should carry a formal charge of –2, and Ga(2) should carry a formal charge of –3. The resulting formal ionic formulation Ba<sub>8</sub><sup>16+</sup>Ga<sub>4</sub><sup>8–</sup>Ga<sub>3</sub><sup>9–</sup> shows a not-exact charge balance: the Ba atoms provide 16 electrons instead of the required 17.

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## Structural Redetermination of the ThNi Phase

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**Abstract.**  $M_r = 290.8$ , orthorhombic,  $Pnma$ ,  $a = 14.146$  (6),  $b = 4.286$  (3),  $c = 5.702$  (3) Å,  $V = 345.7$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 11.17$  Mg m<sup>-3</sup>,  $F(000) = 944$ , room temperature,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu(\text{Mo } K\alpha) = 138$  mm<sup>-1</sup>, final  $R = 0.049$  for 306 observed reflections. Contrary to the report of Florio, Baenziger & Rundle [*Acta Cryst.* (1956), **9**, 367–372], ThNi was found to crystallize in a stacking variant of the CrB–FeB types with trigonal-prismatic coordination of the Ni atoms, and recognized to be isotypic with the SrAg phase, having stacking code  $(hc)_2$ .

**Introduction.** The crystal structure of the orthorhombic ThNi was described by Florio, Baenziger & Rundle (1956). During a study on the structures of phases formed by the alkaline-earth elements with Cu and Ag (Merlo & Fornasini, 1981), it was noted that ThNi had the same space group and number of atoms per cell, and very similar lattice-constant ratios as SrAg, a stacking variant of the CrB–FeB types, whereas atomic positions and coordination were different. A sample of ThNi was then prepared and the observed powder intensities were compared with the values calculated both for the original proposition by Florio *et al.* (1956) and for the SrAg model. As the latter gave a significantly better agreement, a single-crystal refinement was undertaken and this paper reports the results obtained.

**Experimental.** Metals supplied by Koch-Light: Th 99.8 and Ni 99.999 wt % pure; the sample prepared by melting under vacuum the two elements in an alumina crucible in an induction furnace, and then annealed in an evacuated silica tube at 1073 K for 20 d. The alloy (brittle, well crystallized, air-resistant) checked by micrographic examination proved to be a

homogeneous phase. A plate-like single crystal with dimensions 0.08 × 0.07 × 0.03 mm isolated and mounted on a Enraf–Nonius CAD-4 automatic diffractometer; lattice constants determined by least-squares refinement of the angular values of 25 diffractometer-measured reflections. Systematic extinctions confirmed the space group  $Pnma$  and reflections from three octants of reciprocal lattice were collected; graphite-monochromated Mo  $K\alpha$  radiation,  $\omega$ – $\theta$  scan mode up to  $2\theta = 60^\circ$ . Intensities processed for Lp and absorption effects, spherical ( $\mu r = 3.45$ ) and semi-empirical correction based on the  $\psi$ -scan data of two top reflections (North, Phillips & Mathews, 1968), maximum transmission-factor ratio 12.5; 555 unique reflections ( $R_{\text{int}} = 0.084$ ), 306 with  $F_o > 2\sigma(F_o)$  used in the structure refinement.

The parameters of SrAg (Merlo & Fornasini, 1981) were assumed as starting values for a full-matrix least-squares refinement (on  $F$ ) in  $Pnma$  with *SHELX76* (Sheldrick, 1976), taking atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Isotropic cycles,  $R = 0.071$ ; anisotropic refinement over 25 parameters with weights  $w = 1/\sigma^2(F_o)$ ,  $R = 0.049$ ,  $wR = 0.039$ , maximum shift to e.s.d. in the last cycle 0.3; maximum and minimum height in final difference Fourier map 7.4 and  $-4.2$  e Å<sup>-3</sup>, respectively. No correction for secondary extinction.

**Discussion.** The final atomic parameters are listed in Table 1.\* Table 2 gives the interatomic distances in ThNi. The Th–Ni contacts seem to play an important

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38531 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.