

OLTHOF-HAZEKAMP, R. (1990). *CRYLSQ. XTAL3.0 User's Manual*, edited by S. R. HALL & J. M. STEWART. Univs. of Western Australia, Australia, and Maryland, USA.

PARTHÉ, E. & HOVESTREYDT, E. (1985). *J. Less-Common Met.* **110**, 307–313.

PERLITZ, H. & WESTGREN, A. (1943). *Ark. Kemi Mineral. Geol.* **16B**(13), 5.

ROSEN, S. & GOEBEL, J. A. (1968). *J. Less-Common Met.* **16**, 285–287.

RYKHAL', R. M. & ZARECHNYUK, O. S. (1977). *Dopov. Akad. Nauk Ukr. RSR Ser. A*(4), pp. 375–377.

RYKHAL', R. M., ZARECHNYUK, O. S. & YARMOLYUK, YA. P. (1972). *Sov. Phys. Crystallogr.* **17**, 453–455.

SCHWARZENBACH, D. (1966). Private communication.

*Acta Cryst.* (1992). **C48**, 232–236

## Structure of Monoclinic Y<sub>4</sub>Ni<sub>6</sub>Al<sub>23</sub>

BY R. E. GLADYSHEVSKII\* AND E. PARTHÉ

*Laboratoire de Cristallographie aux Rayons X, Université de Genève, 24 Quai E. Ansermet, CH-1211 Geneva 4, Switzerland*

(Received 24 May 1991; accepted 6 August 1991)

**Abstract.** Hexanickel tetrayttrium tricosaaluminide,  $M_r = 1328.45$ , monoclinic, new type  $mS66$ ,  $C2/m - i^6c$ ,  $a = 15.836$  (2),  $b = 4.0681$  (7),  $c = 18.311$  (2) Å,  $\beta = 112.97$  (1)°,  $V = 1086.1$  (2) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 4.062$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 16.896$  mm<sup>-1</sup>,  $F(000) = 1246$ ,  $T = 293$  K,  $wR = 0.058$  for 648 contributing unique reflections. The structure of Y<sub>4</sub>Ni<sub>6</sub>Al<sub>23</sub> is closely related to YNiAl<sub>4</sub> and U<sub>4</sub>Ni<sub>5</sub>Al<sub>18</sub>, all having similar coordination polyhedra around Y (U) and Ni atoms. YNiAl<sub>4</sub>-type slabs can be intergrown with another kind of slab to form the U<sub>4</sub>Ni<sub>5</sub>Al<sub>18</sub> structure. In Y<sub>4</sub>Ni<sub>6</sub>Al<sub>23</sub> the same slabs but in a different proportion are intergrown to form packets which are stacked without common atoms.

**Introduction.** In the Y–Ni–Al system eight compounds are known with less than 34 at.% Y (Rykhali' & Zarechnyuk, 1977). For six of them the crystal structures have been determined previously (for details see Bodak & Gladyshevskii, 1985). We report here the determination of the crystal structure of a new Al-rich compound with a composition not mentioned in the phase-diagram paper.

**Experimental.** Single crystals were found in a sample of nominal composition Y<sub>5</sub>Ni<sub>15</sub>Al<sub>80</sub>. It was prepared from Y (99.9%), Ni (99.99%) and Al (99.99%) by arc melting under an argon atmosphere (weight loss 0.4%) and annealed at 773 K for two weeks in a silica tube under a 400 mm Hg argon atmosphere. A needle-shaped single crystal [ $\pm(100)$  0.032,  $\pm(010)$  0.128,  $\pm(001)$  0.016 mm] was mounted on a Philips

PW 1100 automatic four-circle diffractometer, Mo  $K\alpha$  radiation with graphite monochromator. The unit-cell parameters were refined from  $2\theta$  values of 26 reflections (Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $16 < 2\theta < 30^\circ$ ) using the program *LATCON* (Schwarzenbach, 1966). 2330 reflections were collected out to  $(\sin\theta/\lambda) = 0.603$  Å<sup>-1</sup> ( $-18 \leq h \leq 18$ ,  $0 \leq k \leq 4$ ,  $0 \leq l \leq 22$  and the anti-reflections) in the  $\omega$ - $2\theta$ -scan mode, yielding 1130 unique reflections ( $R_{\text{int}} = 0.12$ ). Two standard reflections, 020 and 0 $\bar{2}$ 0, were measured with maximum intensity variations 0.9 and 1.3% respectively. Absorption correction was made using the program *LSABS* (Blanc, Schwarzenbach & Flack, 1991) with maximum and minimum transmission factors of 0.7616 and 0.5521. The anomalous-dispersion coefficients were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Systematic absences led to the following possible space groups:  $C2$ ,  $Cm$  and  $C2/m$  (*International Tables for Crystallography*, 1983, Vol. A). The structure was solved in space group  $C2/m$  by the *MULTAN87* program (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987) and confirmed by a structure refinement, based on  $|F|$  values using the program *CRYLSQ* (Olthof-Hazekamp, 1990). 101 variables including anisotropic atomic displacement parameters refined to  $R = 0.073$  and  $wR = 0.058$  [ $w = 1/\sigma^2(|F_{\text{rel}}|)$ ,  $S = 1.537$ ] considering 648 contributing unique reflections with  $|F_{\text{rel}}| > 4\sigma(|F_{\text{rel}}|)$ . The maximum shift/e.s.d. in the last cycle was 0.0001. Final residual electron density  $+5.7(-4.5)$  e Å<sup>-3</sup>. The programs used to refine the structure are all from the *XTAL3.0* system (Hall & Stewart, 1990). The atomic positional parameters were standardized by using the *STRUCTURE TIDY* program (Gelato & Parthé, 1987). The atomic posi-

\* Permanent address: Physics Department, Lvov State University, 50 Ulica Dragomanova, SU-290005 Lvov, USSR.

tional and displacement parameters are given in Table 1 and the interatomic distances in Table 2.\*

**Discussion.** The structure of  $Y_4Ni_6Al_{23}$  is conveniently discussed together with the structures of orthorhombic  $YNiAl_4$  (Rykhali', Zarechnyuk & Yarmolyuk, 1972) and monoclinic  $U_4Ni_5Al_{18}$  (Grin', Rogl, Aksel'rud & Pertlik, 1989). All these structures are shown in Fig. 1 in projections along the shortest cell translation, which is approximately of the same length (4.061 to 4.08 Å). Similar numerical values are observed also for a second, perpendicular unit-cell translation (maximum difference  $\approx 2.5\%$ ). It should be noted that the unit-cell parameters given in Fig. 1 for  $U_4Ni_5Al_{18}$  are different from the originally published values. We have made a transformation to the 'best' monoclinic cell (Parthé & Gelato, 1985), which has a smaller  $c$  value and a smaller  $\beta$  angle ( $\mathbf{a}' = -\mathbf{a}$ ,  $\mathbf{b}' = -\mathbf{b}$ ,  $\mathbf{c}' = \mathbf{a} + \mathbf{c}$ ).

Common to these three structures are the particular coordination polyhedra of two of the constituent elements: Y (U) atoms are at the centres of regular pentagonal  $Ni_2Al_8$  prisms with the five rectangular faces being capped by Al atoms, and Ni atoms are at the centres of trigonal prisms of formula  $Y_2Al_4$  ( $U_2Al_4$ ) and, in the case of the monoclinic structures, also some of formula  $Al_6$ , with both kinds of prisms having the three rectangular faces capped by Al atoms.

The trigonal  $Al_6$  prisms are regular but the mixed ones are deformed due to the different sizes of the Y (U) and Al atoms. The ratio of the number of trigonal  $Y_2Al_4$  ( $U_2Al_4$ ) prisms to the number of all Ni-centred prisms is directly related to the composition of the compound and is equal to  $x/y$  if the composition of the aluminide is written as  $R_xNi_yAl_z$  ( $R = Y$  or  $U$ ). For more details see the Appendix.

The coordination polyhedra around the Al atoms are not equal: there are cubes with at maximum five different compositions, some of them more regular and some of them quite deformed, and there are also pentagonal prisms in the two monoclinic structures. All these Al-centred polyhedra are capped by different kinds of atoms.

Since the compounds are rich in Al one might assume that an understanding of the construction of these crystal structures might be found by a thorough analysis of all Al-coordination polyhedra and their connections. However, this approach was not successful. Instead we found it more convenient to base the reasoning on the surprisingly regular

Table 1. Atomic positional and displacement parameters for  $Y_4Ni_6Al_{23}$  with space group  $C2/m$

The equivalent isotropic atomic displacement parameters are expressed as  $U_{eq} = \frac{1}{3} \sum_j U_{ij} a_i^* a_j^* a_i a_j$ . E.s.d.'s are given in parentheses.

	Wyckoff position	x	y	z	$U_{eq} (\text{Å}^2 \times 100)$
Ni(1)	4(i)	0.0082 (2)	0	0.1387 (2)	0.9 (1)
Al(1)	4(i)	0.0820 (6)	0	0.6855 (6)	1.4 (3)
Al(2)	4(i)	0.0894 (6)	0	0.0512 (5)	1.1 (3)
Al(3)	4(i)	0.0901 (6)	0	0.2825 (5)	1.3 (3)
Al(4)	4(i)	0.2011 (6)	0	0.5204 (5)	0.8 (3)
Ni(2)	4(i)	0.2267 (2)	0	0.3988 (2)	0.8 (1)
Al(5)	4(i)	0.2560 (6)	0	0.0451 (5)	0.9 (3)
Y(1)	4(i)	0.2643 (2)	0	0.2186 (2)	0.9 (1)
Al(6)	4(i)	0.2733 (6)	0	0.6757 (5)	0.7 (3)
Al(7)	4(i)	0.3776 (6)	0	0.4057 (5)	1.2 (4)
Al(8)	4(i)	0.4340 (6)	0	0.0591 (5)	0.8 (3)
Al(9)	4(i)	0.4675 (6)	0	0.2188 (5)	1.3 (4)
Y(2)	4(i)	0.5725 (2)	0	0.4069 (2)	0.9 (1)
Al(10)	4(i)	0.6104 (6)	0	0.1626 (5)	1.1 (3)
Ni(3)	4(i)	0.6895 (2)	0	0.0687 (2)	0.9 (1)
Al(11)	4(i)	0.8552 (6)	0	0.1434 (5)	0.8 (3)
Al(12)	2(c)	0	0	$\frac{1}{2}$	1.4 (5)

shapes of the polyhedra around the Y (U) and Ni atoms. In particular, all Al atoms participate in the formation of these polyhedra and the Al sites are determined from the positions of the metal atoms. This may be verified in the three structure drawings of Fig. 1, where the pentagonal and trigonal prisms are emphasized. In order not to overload the drawing, only one half of the prisms are shown, *i.e.* those with their base at height  $\frac{1}{2}$  (that means having their centre at 0). All Al atoms at height  $\frac{1}{2}$  either participate in the formation of prisms centred by Y (U) or Ni atoms at height 0 or cap faces of prisms centred by atoms at height  $\frac{1}{2}$ . In the same way the positions of the Al atoms at height 0 which are related to the former by the  $C$  Bravais lattice translation are determined.

It should be mentioned that similar capped pentagonal and trigonal prisms also occur in ternary aluminides such as  $YNiAl_2$  with an  $MgCuAl_2$ -type structure and  $YNiAl_3$  with its own structure type. The polyhedra, due to the lower Al content, have different occupations of the vertices and/or of the sites capping the rectangular faces (for more details see Gladyshevskii & Parthé, 1992).

The polyhedra arrangements of the three structures, presented in Fig. 1, show common features. In particular, it is possible to recognize two-dimensional segments, cut parallel to the (001) planes of the two monoclinic structures – approximately 9.5 Å thick in the case of  $U_4Ni_5Al_{18}$  and 6.3 Å thick in  $Y_4Ni_6Al_{23}$  – which are built up like the  $YNiAl_4$  structure. These slabs are constructed of pentagonal ( $Ni_2Al_8$ ) and trigonal ( $Y_2Al_4$ ) prisms, oriented in such a way that all Y—Ni bonds are in the slab plane. The structure of  $YNiAl_4$  itself can be described as an intergrowth of this unique kind of slab (approximately 3.3 Å

\* Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54582 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances in  $\text{Y}_4\text{Ni}_6\text{Al}_{23}$  (up to 4.0 Å for Y and 3.6 Å for Ni, Al atoms)*

E.s.d.'s are given in parentheses.

Y(1)—2Al(6)	3.024 (8)	Al(3)—Ni(2)	2.371 (8)
2Al(10)	3.030 (6)	Ni(1)	2.439 (9)
2Al(11)	3.107 (9)	2Al(9)	2.738 (8)
Al(5)	3.128 (1)	2Al(6)	2.848 (9)
2Al(1)	3.132 (7)	Al(1)	3.004 (16)
Al(7)	3.187 (9)	2Al(10)	3.099 (11)
Al(9)	3.217 (11)	2Y(2)	3.147 (9)
Al(2)	3.231 (8)	Y(1)	3.390 (12)
2Ni(3)	3.246 (3)	Al(11)	3.596 (11)
Al(3)	3.390 (12)	Al(4)—Ni(2)	2.413 (11)
Ni(2)	3.575 (6)	2Ni(2)	2.513 (5)
Ni(1)	3.736 (5)	Al(6)	2.619 (11)
Y(2)—2Al(4)	3.049 (6)	2Al(4)	2.828 (10)
Al(7)	3.078 (10)	2Al(7)	2.974 (11)
2Al(1)	3.124 (7)	2Y(2)	3.049 (6)
2Al(12)	3.145 (3)	Al(12)	3.058 (9)
2Al(3)	3.147 (9)	Y(2)	3.302 (9)
Al(9)	3.187 (9)	Al(5)—2Ni(3)	2.406 (6)
Al(7)	3.208 (10)	Ni(3)	2.547 (11)
2Ni(2)	3.225 (4)	2Al(5)	2.580 (8)
Al(4)	3.302 (9)	Al(2)	2.684 (14)
Al(6)	3.335 (11)	Al(8)	2.729 (14)
Ni(2)	3.734 (4)	2Al(11)	2.763 (7)
Ni(1)—Al(2)	2.415 (12)	Y(1)	3.128 (10)
Al(3)	2.439 (9)	Al(11)	3.204 (11)
Al(11)	2.458 (11)	2Al(8)	3.515 (9)
2Al(8)	2.511 (5)	Al(6)—2Ni(2)	2.449 (6)
2Al(10)	2.529 (6)	Al(4)	2.619 (11)
2Al(9)	2.728 (8)	Al(10)	2.811 (11)
Al(2)	3.207 (9)	2Al(3)	2.848 (9)
Ni(2)—Al(7)	2.344 (10)	2Y(1)	3.024 (8)
Al(3)	2.371 (8)	2Al(7)	3.048 (8)
Al(4)	2.413 (11)	Al(1)	3.104 (15)
2Al(6)	2.449 (6)	Y(2)	3.335 (11)
2Al(4)	2.513 (5)	Al(7)—Ni(2)	2.344 (10)
2Y(2)	3.225 (4)	2Al(1)	2.857 (11)
Y(1)	3.575 (6)	2Al(12)	2.875 (5)
Ni(3)—Al(8)	2.394 (8)	2Al(4)	2.974 (11)
2Al(5)	2.406 (6)	2Al(6)	3.048 (8)
Al(11)	2.440 (9)	Y(2)	3.078 (10)
Al(10)	2.492 (12)	Y(1)	3.187 (9)
2Al(2)	2.521 (6)	Y(2)	3.208 (10)
Al(5)	2.547 (11)	Al(8)—Ni(3)	2.394 (8)
2Y(1)	3.246 (3)	2Ni(1)	2.511 (5)
Al(1)—2Al(7)	2.857 (11)	Al(10)	2.694 (11)
Al(11)	2.894 (13)	Al(5)	2.729 (14)
2Al(9)	2.983 (12)	Al(9)	2.761 (14)
Al(3)	3.004 (16)	2Al(2)	2.787 (9)
Al(6)	3.104 (15)	2Al(11)	3.095 (12)
2Y(2)	3.124 (7)	2Al(2)	3.239 (11)
Al(12)	3.127 (10)	2Al(5)	3.515 (9)
2Y(1)	3.132 (7)	Al(8)	3.546 (16)
Al(2)—Ni(1)	2.415 (12)	Al(9)—2Al(11)	2.700 (7)
2Ni(3)	2.521 (6)	2Ni(1)	2.728 (8)
Al(5)	2.684 (14)	2Al(3)	2.738 (8)
Al(2)	2.719 (11)	Al(8)	2.761 (14)
2Al(8)	2.787 (9)	Al(10)	2.827 (15)
2Al(10)	2.807 (9)	2Al(1)	2.983 (12)
Ni(1)	3.207 (9)	Y(2)	3.187 (9)
Y(1)	3.231 (8)	Y(1)	3.217 (11)
2Al(8)	3.239 (11)		

Table 2 (cont.)

Al(10)—Ni(3)	2.492 (12)	Al(12)—4Al(7)	2.875 (5)
2Ni(1)	2.529 (6)	2Al(4)	3.058 (9)
Al(8)	2.694 (11)	2Al(1)	3.127 (10)
2Al(2)	2.807 (9)	4Y(2)	3.145 (3)
Al(6)	2.811 (11)		
Al(9)	3.827 (15)		
2Y(1)	3.030 (6)		
2Al(3)	3.099 (11)		
Al(11)—Ni(3)	2.440 (9)		
Ni(1)	2.458 (11)		
2Al(9)	2.700 (7)		
2Al(5)	2.763 (7)		
Al(1)	2.894 (13)		
2Al(8)	3.095 (12)		
2Y(1)	3.107 (9)		
Al(5)	3.204 (11)		
Al(3)	3.596 (11)		

thick), parallel to the (001) plane, with consecutive slabs being rotated by  $180^\circ$ . In the unit cell there are two such intergrown slabs of formula  $\text{Y}_2\text{Ni}_2\text{Al}_8$ .

In the structure  $\text{U}_4\text{Ni}_5\text{Al}_{18}$  the 9.5 Å thick fragments consist of three  $\text{YNiAl}_4$ -type slabs. This slab packet is further intergrown with a new kind of slab of formula  $\text{U}_2\text{Ni}_4\text{Al}_{12}$ , about 4.7 Å thick, where half of the Ni atoms are at the centres of  $\text{Al}_6$  prisms. The composition of this compound can thus be written as  $3\text{U}_2\text{Ni}_2\text{Al}_8 + \text{U}_2\text{Ni}_4\text{Al}_{12} = 2\text{U}_4\text{Ni}_5\text{Al}_{18}$ . As calculated in the Appendix, in this structure the ratio of the number of Ni-centred trigonal  $\text{U}_2\text{Al}_4$  prisms to the number of Ni-centred trigonal  $\text{Al}_6$  prisms is 4:1. Of the four trigonal  $\text{U}_2\text{Al}_4$  prisms in one formula unit three are in the  $\text{YNiAl}_4$ -type slab packet and the fourth is in the other kind of slab.

In the structure  $\text{Y}_4\text{Ni}_6\text{Al}_{23}$  the 6.3 Å thick fragments consist of two  $\text{YNiAl}_4$ -type slabs. This double slab packet is intergrown on each side with two differently oriented slabs of the same kind as in  $\text{U}_4\text{Ni}_5\text{Al}_{18}$ , with the same stoichiometry. As stated above, in these slabs half of the Ni atoms are at the centres of  $\text{Al}_6$  prisms. The total slab packets, consisting of four intergrown slabs, are stacked perpendicular to the (001) plane with only Al—Al bonds available for the interaction between the packets. The composition of the ternary compound can be obtained as follows:  $2\text{Y}_2\text{Ni}_2\text{Al}_8 + 2\text{Y}_2\text{Ni}_4\text{Al}_{12} + \text{Al}_6 = 2\text{Y}_4\text{Ni}_6\text{Al}_{23}$ . As shown in the Appendix, in this structure the ratio of the number of Ni-centred trigonal  $\text{Y}_2\text{Al}_4$  prisms to the number of Ni-centred trigonal  $\text{Al}_6$  prisms is 4:2. Of the four trigonal  $\text{Y}_2\text{Al}_4$  prisms in one formula unit two are in the  $\text{YNiAl}_4$ -type slab packet and two are in the two slabs of the other kind.

It should be mentioned that the two monoclinic structures can also be described with face-centred pseudo-orthorhombic unit cells, where  $a_o = b_m$ ,  $b_o = a_m$  and  $c_o = 2c_m \sin\beta$ .

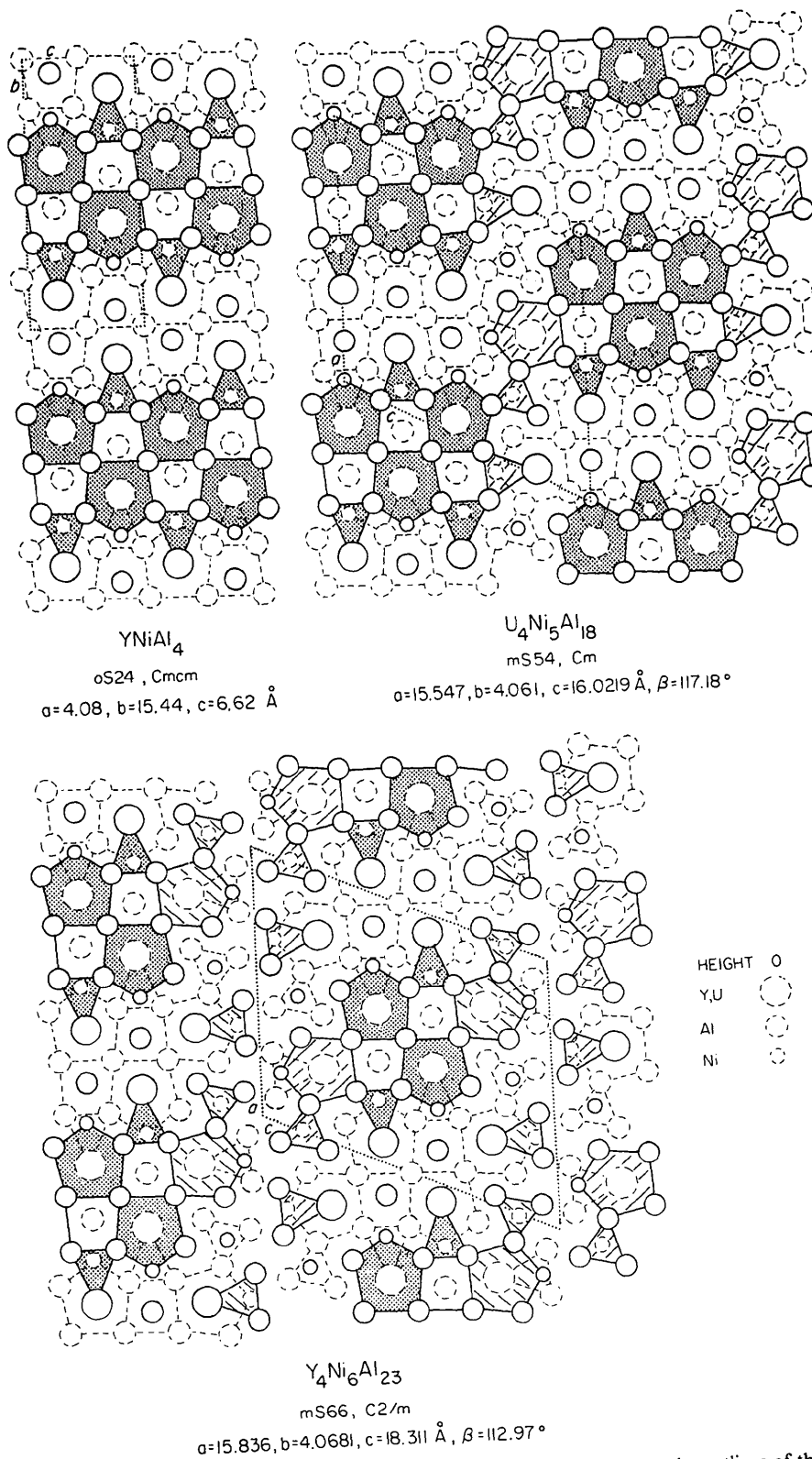


Fig. 1. Projections of  $\text{YNiAl}_4$  along  $[100]$  and of  $\text{U}_4\text{Ni}_5\text{Al}_{18}$  and  $\text{Y}_4\text{Ni}_6\text{Al}_{23}$  along  $[010]$ . Only the outlines of the pentagonal and trigonal prisms are indicated. The base planes at height  $\frac{1}{2}$  for the prisms in the  $\text{YNiAl}_4$ -type slabs have been shaded. Those at height  $\frac{1}{2}$  for the other kind of slab have been marked by stripes. In the case of the  $\text{Y}_4\text{Ni}_6\text{Al}_{23}$  structure the different orientation of the stripes is supposed to indicate the different orientation of the slabs.

## APPENDIX

## Correlation between atom coordinations and compound stoichiometry

The crystal-chemical formulae of ternary compounds  $R_xT_yM_z$  can be written in a general form (Lima-de-Faria, Hellner, Makovicky, Liebau & Parthé, 1990) as

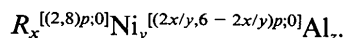
$$R_x^{[r_T, r_M; r_R]} T_y^{[t_R, t_M; t_T]} M_z^{[m_R, m_T; m_M]}$$

Coordination symbols are added to the symbols of the chemical elements as trailing superscripts and placed between square brackets. Symbols before a semicolon refer to the heteronuclear coordination ( $r_T, r_M$  for  $R$ ;  $t_R, t_M$  for  $T$  and  $m_R, m_T$  for  $M$ ) and those after it to the homonuclear coordination ( $r_R, t_T$  and  $m_M$ ). The form of a coordination polyhedron can be further indicated by a small letter, as for example  $p$  for a prism, where the number (or the sum of the numbers) before the letter indicates the number of the vertices.

The simplified crystal-chemical formulae of the particular ternary Ni aluminides of interest here, taking into account the known coordination polyhedra of the  $R$  ( $= Y$  or  $U$ ) and Ni atoms, can be written as



Based on the simple fact that the number of bonds extended from all atoms  $i$  to the atoms  $ii$  must be equal to the number of bonds extended from all atoms  $ii$  to the atoms  $i$  it is possible to calculate the average values of  $t_R$  and  $t_{Al}$  and to rewrite the crystal-chemical formulae as



From this result it is possible to calculate that the ratio of the number of Ni-centred trigonal prisms of composition  $R_2Al_4$  to the total number of Ni-centred trigonal prisms (compositions  $R_2Al_4$  and  $Al_6$ ) is

equal to  $x/y$ ; that means the ratio of the number of  $R_2Al_4$  prisms to the number of  $Al_6$  prisms (normalized to one formula unit) is 1:0 in YNiAl<sub>4</sub>, 4:1 in U<sub>4</sub>Ni<sub>5</sub>Al<sub>18</sub> and 4:2 in Y<sub>4</sub>Ni<sub>6</sub>Al<sub>23</sub>.

We wish to acknowledge the help of Ms Birgitta Künzler for the preparation of the drawings and we would like to thank Dr Karin Cenzual for useful comments. Thanks are also due to Mr Jing-Tai Zhao for technical assistance and discussions. This study was supported by the Swiss National Science Foundation under contract 20-28490.90 and the Alfred and Hilde Freissler Fond.

## References

- BLANC, E., SCHWARZENBACH, D. & FLACK, H. D. (1991). *J. Appl. Cryst.* **24**, 1035–1041.
- BODAK, O. I. & GLADYSHEVSKII, E. I. (1985). *Ternary Systems Containing Rare Earth Metals*, pp. 68–69. Lvov: Vysycha Shkola Publishing House. (In Russian.)
- DEBAERDEMAEKER, T., GERMAIN, G., MAIN, P., TATE, C. & WOOLFSON, M. M. (1987). *MULTAN87. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- GELATO, L. M. & PARTHÉ, E. (1987). *J. Appl. Cryst.* **20**, 139–143.
- GLADYSHEVSKII, R. E. & PARTHÉ, E. (1992). *Acta Cryst.* **C48**, 229–232.
- GRIN', YU. N., ROGL, P., AKSEL'RUD, L. G. & PERTLIK, F. (1989). *Z. Kristallogr.* **188**, 271–277.
- HALL, S. R. & STEWART, J. M. (1990). Editors. *XTAL3.0 User's Manual*. Univs. of Western Australia, Australia, and Maryland, USA.
- LIMA-DE-FARIA, J., HELLNER, E., MAKOVICKY, E., LIEBAU, F. & PARTHÉ, E. (1990). *Acta Cryst.* **A46**, 1–11.
- OLTHOF-HAZEKAMP, R. (1990). *CRYLSQ. XTAL3.0 User's Manual*, edited by S. R. HALL & J. M. STEWART. Univs. of Western Australia, Australia, and Maryland, USA.
- PARTHÉ, E. & GELATO, L. M. (1985). *Acta Cryst.* **A41**, 142–151.
- RYKHAL', R. M. & ZARECHNYUK, O. S. (1977). *Dopov. Akad. Nauk Ukr. RSR Ser. A(4)*, pp. 376–380.
- RYKHAL', R. M., ZARECHNYUK, O. S. & YARMOLYUK, YA. P. (1972). *Sov. Phys. Crystallogr.* **17**, 453–455.
- SCHWARZENBACH, D. (1966). Private communication.

*Acta Cryst.* (1992). **C48**, 236–239

Structure of a Thallium(I) Transition-Metal Carbonyl Salt Tl[Fe(CO)<sub>3</sub>(NO)]

BY LUCY M. CLARKSON, WILLIAM CLEGG,\* DAVID C. R. HOCKLESS AND NICHOLAS C. NORMAN

*Department of Chemistry, The University, Newcastle upon Tyne NE1 7RU, England*

(Received 14 June 1991; accepted 29 August 1991)

**Abstract.** Thallium tricarbonylnitrosoiron,  $M_r = 374.25$ , cubic,  $P2_13$ ,  $a = 8.853(1) \text{ \AA}$ ,  $V = 693.8 \text{ \AA}^3$ ,  $Z = 4$ ,  $D_x = 3.582 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$ ,  $\mu = 254.8 \text{ cm}^{-1}$ ,  $F(000) = 656$ ,  $T = 240 \text{ K}$ ,  $R = 0.0197$  for 420 unique observed reflections. Discrete  $\text{Tl}^+$  cations are well separated by  $[\text{Fe}(\text{CO})_3(\text{NO})]^-$  anions

\* Author to whom correspondence should be addressed.