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Structure of Monoclinic $Y_4Ni_6Al_{23}$

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Abstract. Hexanickel tetrayttrium tricosaaluminide, $M_r = 1328.45$, monoclinic, new type *mS*66, $C2/m - i^{16}c$, $a = 15.836\ (2)$, $b = 4.0681\ (7)$, $c = 18.311\ (2)$ Å, $\beta = 112.97\ (1)°$, $V = 1086.1\ (2)$ Å^3, $Z = 2$, $D_x = 4.062$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 16.896$ mm^{-1}, $F(000) = 1246$, $T = 293$ K, $wR = 0.058$ for 648 contributing unique reflections. The structure of $Y_4Ni_6Al_{23}$ is closely related to $YNiAl_4$ and $U_4Ni_5Al_{18}$, all having similar coordination polyhedra around Y (U) and Ni atoms. $YNiAl_4$-type slabs can be intergrown with another kind of slab to form the $U_4Ni_5Al_{18}$ structure. In $Y_4Ni_6Al_{23}$ 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 (Rykhal' & 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 $Y_5Ni_{15}Al_{80}$. 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 [± (100) 0.032, ± (010) 0.128, ± (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 2θ values of 26 reflections (Mo $K\alpha$, $\lambda = 0.71073$ Å, $16 < 2\theta < 30°$) using the program *LATCON* (Schwarzenbach, 1966). 2330 reflections were collected out to $(\sin\theta/\lambda) = 0.603$ Å^{-1} ($-18 \le h \le 18$, $0 \le k \le 4$, $0 \le l \le 22$ and the anti-reflections) in the ω–2θ-scan mode, yielding 1130 unique reflections ($R_{int} = 0.12$). 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: *C*2, *Cm* and *C*2/*m* (*International Tables for Crystallography*, 1983, Vol. A). The structure was solved in space group *C*2/*m* by the *MULTAN*87 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 $wR = 0.058$ [$w = 1/\sigma^2(|F_{rel}|)$, $S = 1.537$] considering 648 contributing unique reflections with $|F_{rel}| > 4\sigma(|F_{rel}|)$. The maximum shift/e.s.d. in the last cycle was 0.0001. Final residual electron density +5.7 (−4.5) e Å^{-3}. The programs used to refine the structure are all from the *XTAL*3.0 system (Hall & Stewart, 1990). The atomic positional parameters were standardized by using the *STRUCTURE TIDY* program (Gelato & Parthé, 1987). The atomic posi-

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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 $YNiAl_4$ (Rykhal', 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 $\simeq 2.5\%$). 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 β 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 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

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 $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i\cdot\mathbf{a}_j$. E.s.d.'s are given in parentheses.

	Wyckoff position	x	y	z	U_{eq}(Å^2 × 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)

* 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* $Y_4Ni_6Al_{23}$ *(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)	
2Al(10)	3.030 (6)	
2Al(11)	3.107 (9)	
Al(5)	3.128 (1)	
2Al(1)	3.132 (7)	
Al(7)	3.187 (9)	
Al(9)	3.217 (11)	
Al(2)	3.231 (8)	
2Ni(3)	3.246 (3)	
Al(3)	3.390 (12)	
Ni(2)	3.575 (6)	
Ni(1)	3.736 (5)	
Y(2)—2Al(4)	3.049 (6)	
Al(7)	3.078 (10)	
2Al(1)	3.124 (7)	
2Al(12)	3.145 (3)	
2Al(3)	3.147 (9)	
Al(9)	3.187 (9)	
Al(7)	3.208 (10)	
2Ni(2)	3.225 (4)	
Al(4)	3.302 (9)	
Al(6)	3.335 (11)	
Ni(2)	3.734 (4)	
Ni(1)— Al(2)	2.415 (12)	
Al(3)	2.439 (9)	
Al(11)	2.458 (11)	
2Al(8)	2.511 (5)	
2Al(10)	2.529 (6)	
2Al(9)	2.728 (8)	
Al(2)	3.207 (9)	
Ni(2)— Al(7)	2.344 (10)	
Al(3)	2.371 (8)	
Al(4)	2.413 (11)	
2Al(6)	2.449 (6)	
2Al(4)	2.513 (5)	
2Y(2)	3.225 (4)	
Y(1)	3.575 (6)	
Ni(3)— Al(8)	2.394 (8)	
2Al(5)	2.406 (6)	
Al(11)	2.440 (9)	
Al(10)	2.492 (12)	
2Al(2)	2.521 (6)	
Al(5)	2.547 (11)	
2Y(1)	3.246 (3)	
Al(1)—2Al(7)	2.857 (11)	
Al(11)	2.894 (13)	
2Al(9)	2.983 (12)	
Al(3)	3.004 (16)	
Al(6)	3.104 (15)	
2Y(2)	3.124 (7)	
Al(12)	3.127 (10)	
2Y(1)	3.132 (7)	
Al(2)— Ni(1)	2.415 (12)	
2Ni(3)	2.521 (6)	
Al(5)	2.684 (14)	
Al(2)	2.719 (11)	
2Al(8)	2.787 (9)	
2Al(10)	2.807 (9)	
Ni(1)	3.207 (9)	
Y(1)	3.231 (8)	
2Al(8)	3.239 (11)	
Al(3)— Ni(2)	2.371 (8)	
Ni(1)	2.439 (9)	
2Al(9)	2.738 (8)	
2Al(6)	2.848 (9)	
Al(1)	3.004 (16)	
2Al(10)	3.099 (11)	
2Y(2)	3.147 (9)	
Y(1)	3.390 (12)	
Al(11)	3.596 (11)	
Al(4)— Ni(2)	2.413 (11)	
2Ni(2)	2.513 (5)	
Al(6)	2.619 (11)	
2Al(4)	2.828 (10)	
2Al(7)	2.974 (11)	
2Y(2)	3.049 (6)	
Al(12)	3.058 (9)	
Y(2)	3.302 (9)	
Al(5)—2Ni(3)	2.406 (6)	
Ni(3)	2.547 (11)	
2Al(5)	2.580 (8)	
Al(2)	2.684 (14)	
Al(8)	2.729 (14)	
2Al(11)	2.763 (7)	
Y(1)	3.128 (10)	
Al(11)	3.204 (11)	
2Al(8)	3.515 (9)	
Al(6)—2Ni(2)	2.449 (6)	
Al(4)	2.619 (11)	
Al(10)	2.811 (11)	
2Al(3)	2.848 (9)	
2Y(1)	3.024 (8)	
2Al(7)	3.048 (8)	
Al(1)	3.104 (15)	
Y(2)	3.335 (11)	
Al(7)— Ni(2)	2.344 (10)	
2Al(1)	2.857 (11)	
2Al(12)	2.875 (5)	
2Al(4)	2.974 (11)	
2Al(6)	3.048 (8)	
Y(2)	3.078 (10)	
Y(1)	3.187 (9)	
Y(2)	3.208 (10)	
Al(8)— Ni(3)	2.394 (8)	
2Ni(1)	2.511 (5)	
Al(10)	2.694 (11)	
Al(5)	2.729 (14)	
Al(9)	2.761 (14)	
2Al(2)	2.787 (9)	
2Al(11)	3.095 (12)	
2Al(2)	3.239 (11)	
2Al(5)	3.515 (9)	
Al(8)	3.546 (16)	
Al(9)—2Al(11)	2.700 (7)	
2Ni(1)	2.728 (8)	
2Al(3)	2.738 (8)	
Al(8)	2.761 (14)	
Al(10)	2.827 (15)	
2Al(1)	2.983 (12)	
Y(2)	3.187 (9)	
Y(1)	3.217 (11)	

Table 2 (*cont.*)

Al(10)— Ni(3)	2.492 (12)	
2Ni(1)	2.529 (6)	
Al(8)	2.694 (11)	
2Al(2)	2.807 (9)	
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)	
Al(12)—4Al(7)	2.875 (5)	
2Al(4)	3.058 (9)	
2Al(1)	3.127 (10)	
4Y(2)	3.145 (3)	

thick), parallel to the (001) plane, with consecutive slabs being rotated by 180°. In the unit cell there are two such intergrown slabs of formula $Y_2Ni_2Al_8$.

In the structure $U_4Ni_5Al_{18}$ the 9.5 Å thick fragments consist of three $YNiAl_4$-type slabs. This slab packet is further intergrown with a new kind of slab of formula $U_2Ni_4Al_{12}$, about 4.7 Å thick, where half of the Ni atoms are at the centres of Al_6 prisms. The composition of this compound can thus be written as $3U_2Ni_2Al_8 + U_2Ni_4Al_{12} = 2U_4Ni_5Al_{18}$. As calculated in the Appendix, in this structure the ratio of the number of Ni-centred trigonal U_2Al_4 prisms to the number of Ni-centred trigonal Al_6 prisms is 4:1. Of the four trigonal U_2Al_4 prisms in one formula unit three are in the $YNiAl_4$-type slab packet and the forth is in the other kind of slab.

In the structure $Y_4Ni_6Al_{23}$ the 6.3 Å thick fragments consist of two $YNiAl_4$-type slabs. This double slab packet is intergrown on each side with two differently oriented slabs of the same kind as in $U_4Ni_5Al_{18}$, with the same stoichiometry. As stated above, in these slabs half of the Ni atoms are at the centres of 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: $2Y_2Ni_2Al_8 + 2Y_2Ni_4Al_{12} + Al_6 = 2Y_4Ni_6Al_{23}$. As shown in the Appendix, in this structure the ratio of the number of Ni-centred trigonal Y_2Al_4 prisms to the number of Ni-centred trigonal Al_6 prisms is 4:2. Of the four trigonal Y_2Al_4 prisms in one formula unit two are in the $YNiAl_4$-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 $c_o \simeq 2c_m \sin\beta$.

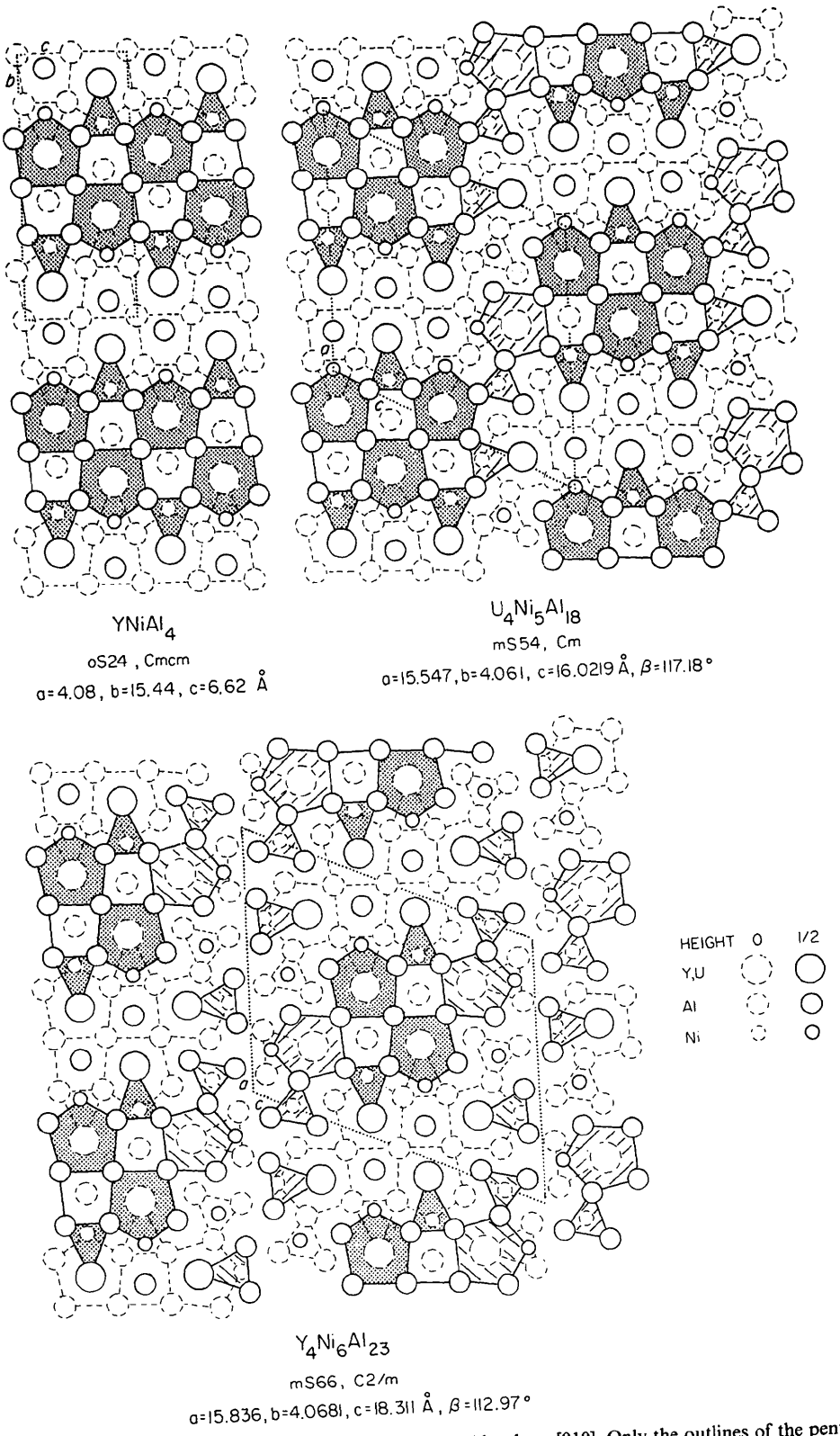

Fig. 1. Projections of $YNiAl_4$ along [100] and of $U_4Ni_5Al_{18}$ and $Y_4Ni_6Al_{23}$ along [010]. Only the outlines of the pentagonal and trigonal prisms are indicated. The base planes at height $\frac{1}{2}$ for the prisms in the $YNiAl_4$-type slabs have been shaded. Those at height $\frac{1}{2}$ for the other kind of slab have been marked by stripes. In the case of the $Y_4Ni_6Al_{23}$ 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

$$R_x^{[(2,8)p;0]}Ni_y^{[6p;0]}Al_z.$$

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

$$R_x^{[(2,8)p;0]}Ni_y^{[(2x/y,6-2x/y)p;0]}Al_z.$$

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 $YNiAl_4$, 4:1 in $U_4Ni_5Al_{18}$ and 4:2 in $Y_4Ni_6Al_{23}$.

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Structure of a Thallium(I) Transition-Metal Carbonyl Salt $Tl[Fe(CO)_3(NO)]$

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

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