(Mruz, Pecharskii, Bodak & Bruskov, 1987) or CeNiSi₂ (Dörrscheidt, Savelsberg, Stöhr & Schäfer, 1982; Chabot, Parthé & Steinmetz, 1986; François, Venturini, Malaman & Roques, 1990; Venturini, François, Malaman & Roques, 1990).

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La₂Al₃Ge₄ and Isotypes of the Orthorhombic Ba₂Cd₃Bi₄ Type, a Slab-Stacking Variant of the HfFe₂Si₂ Type

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Abstract. La₂Al₃Ge₄, $M_r = 649\cdot125$, orthorhombic, Ba₂Cd₃Bi₄ type, $oS36$, (64) $Cmca - f^2e^2a$, $a = 6\cdot146$ (2), $b = 15\cdot194$ (4), $c = 8\cdot037$ (2) Å, $V = 750\cdot4$ (2) Å³, $Z = 4$, $D_x = 5\cdot745$ Mg m⁻³, $\lambda(Mo\ K\alpha) = 0\cdot71073$ Å, $\mu = 28\cdot318$ mm⁻¹, $F(000) = 1124$, $T = 300$ K, $wR = 0\cdot036$ for 472 contributing unique reflections. Isotypes are found for $R_2Al_3Ge_4$ with $R = Ce$, Pr, Nd, Sm, Gd, Tb, Dy, Ho and Er. The Ba₂Cd₃Bi₄ type together with a branch, called the Hf₂Ni₃Si₄ type, can be considered as a slab-stacking variant of the HfFe₂Si₂ type. The change of stoichiometry from HfFe₂Si₂ to Hf₂Ni₃Si₄ is caused by the coalescing of two different Fe sites at the stacking border.

Introduction. The La-deficient part (< 35 at.% La) of the La–Al–Ge ternary phase diagram was investigated by Muravyeva & Zarechnyuk (1970). Three crystal structures of the five compounds reported had been known: LaAl₂Ge₂ has the CaAl₂Si₂ type (*anti*-Ce₂O₂S type) (Zarechnyuk, Muravyeva & Gladyshevskii, 1970), LaAl₂₋ₓGeₓ (0·2 ≤ x ≤ 0·5) crystallizes with the AlB₂ type (Raman & Steinfink, 1967) and LaAlGe with the α-ThSi₂ type (or an ordered

variant of this type) (Raman & Steinfink, 1967). The structure of the latter phase has been confirmed by Zhao & Parthé (1990). The structure of the fourth compound with nominal composition 'LaAlGe₃' has only recently been resolved and its composition was shown to be $La_2Al_{1+x}Ge_{6-x}$ ($x = 0.6$) (Zhao, Cenzual & Parthé, 1991). Here we report on the structure determination of the remaining compound with nominal composition 'LaAlGe₂'.

Experimental. A sample of nominal composition $La_2Al_3Ge_5$ was prepared by arc melting under an argon atmosphere (La, Al and Ge: 99·99%). The weight loss was 0·5%. The sample was annealed at 773 K for two months in an argon-filled silica tube. A single crystal with irregular shape (mean radius 0·024 mm) obtained from the annealed sample 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 24 reflections (Mo $K\alpha$; $20 < 2\theta < 37°$) using the program *LATCON* (Schwarzenbach, 1966). 1350 reflections were collected to $(\sin\theta/\lambda) = 0.703$ Å^{-1} ($0 \leq h \leq 8, 0 \leq k \leq 21$, $0 \leq l \leq 11$ and the anti-reflections) in the ω–2θ scan mode, yielding 652 unique reflections ($R_{int} = 0.025$). Two standard reflections, 221 and 200, were measured with maximum intensity variations 0·9 and 0·7%, respectively. Absorption correction was made using *ABSORB* (Davenport, Spadaccini & Stewart, 1990) with max. and min. transmission factors 0·3788 and 0·3646. 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: *C2cb* (= *Aba2*) and *Cmca* (*International Tables for Crystallography*, 1983, Vol. A). The structure was solved in the space group *Cmca* by *MULTAN87* (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987) and confirmed by the least-squares refinement, based on |F| values, using *CRYLSQ* (Olthof-Hazekamp, 1990). 28 variables including anisotropic atomic displacement parameters refined to $R = 0.040$ and $wR = 0.036$ [$w = 1/\sigma^2(|F_{rel}|), S = 2.527$] considering 472 contributing unique reflections with $|F_{rel}| > 3\sigma(|F_{rel}|)$. Secondary-extinction correction parameter (Gaussian distribution of mosaic spread) was refined to $G = 0.021$ (2). The max. shift/e.s.d. in the last cycle was 0·0001.* Final residual electron density +3·0 (−3·0) e Å^{-3}. The programs used to refine the structure are all from the

Table 1. *Atomic positional and displacement parameters for* $La_2Al_3Ge_4$ *with space group Cmca with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	Wyckoff position	x	y	z	U_{eq}(Å² × 100)
La	8(f)	0	0·11389 (7)	0·4170 (1)	0·66 (5)
Ge(1)	8(f)	0	0·1873 (1)	0·0531 (2)	0·8 (1)
Al(1)	8(e)	¼	0·2842 (1)	¼	1·6 (3)
Ge(2)	8(e)	¼	0·4512 (1)	¼	0·9 (1)
Al(2)	4(a)	0	0	0	1·6 (5)

Table 2. *Interatomic distances up to* 4·0 Å *in* La_2Al_3-Ge_4 *with e.s.d.'s in parentheses*

La— Ge(1)	3·130 (2)	Ge(1)—2Al(1)	2·652 (3)
2Ge(2)	3·205 (2)	Al(2)	2·877 (2)
Ge(1)	3·213 (2)	2Al(1)	2·913 (2)
2Ge(2)	3·241 (1)	La	3·130 (2)
2Ge(1)	3·278 (1)	La	3·213 (2)
2Al(1)	3·295 (4)	2La	3·278 (1)
2Al(1)	3·453 (3)	2Ge(2)	3·566 (2)
2Al(2)	3·589 (1)	2Ge(1)	3·716 (2)
La	3·709 (2)		
Al(2)	3·772 (1)		
Al(1)— Ge(2)	2·538 (6)	Ge(2)— Al(1)	2·538 (6)
2Ge(2)	2·652 (3)	2Al(1)	2·6360 (7)
2Ge(1)	2·913 (2)	2Ge(2)	3·073 (1)
2Al(1)	3·073 (1)	2La	3·205 (2)
2La	3·295 (4)	2La	3·241 (1)
2La	3·453 (3)	2Ge(1)	3·566 (2)
2Ge(2)	3·985 (4)	2Al(1)	3·985 (4)
Al(2)—4Ge(2)	2·6360 (7)		
2Ge(1)	2·877 (2)		
4La	3·589 (1)		
2La	3·772 (1)		

XTAL3·0 system (Hall & Stewart, 1990). The atomic positional parameters were standardized by using the *STRUCTURE TIDY* program (Gelato & Parthé, 1987). The atomic positional and displacement parameters are given in Table 1 and the interatomic distances up to 4·0 Å in Table 2.

Isotypic compounds. The nine isotypic $R_2Al_3Ge_4$ compounds listed in the *Abstract* and in Table 3 were also prepared by the arc-melting method under an argon atmosphere. Our efforts to synthesize R_2Al_3-Ge₄, where $R = $ Eu, Tm, Yb, Lu, Y or Sc, were not successful. The samples were annealed at 773 K for two weeks in an argon-filled silica tube. The observed powder patterns were compared with simulated powder diagrams calculated using the program *LAZY PULVERIX* (Yvon, Jeitschko & Parthé, 1977). The unit-cell parameters for the $R_2Al_3Ge_4$ phases isotypic with $La_2Al_3Ge_4$ were refined from Guinier film data of the annealed samples. The films were measured with a film reader using an internal silicon standard ($a = 5.4308$ Å). The program *LATCON* (Schwarzenbach, 1966) was used for the refinement.

Table 3. *Unit-cell parameters for* La$_2$Al$_3$Ge$_4$ *and its isotypes with the* Ba$_2$Cd$_3$Bi$_4$ *type and the compounds of the* Hf$_2$Ni$_3$Si$_4$ *structure-type branch, with e.s.d.'s in parentheses*

All germanides are from this work.

Composition	a (Å)	b (Å)	c (Å)	V (Å³)	b/a	b/c
Ba₂Cd₃Bi₄	7·037 (1)	17·438 (2)	9·267 (1)	1137·2 (4)	2·478	1·882
La₂Al₃Ge₄	6·143 (2)	15·193 (3)	8·035 (2)	750·4 (2)	2·473	1·891
Ce₂Al₃Ge₄	6·0935 (8)	15·061 (2)	7·974 (1)	731·8 (1)	2·472	1·889
Pr₂Al₃Ge₄	6·056 (2)	15·000 (6)	7·920 (5)	719·5 (4)	2·477	1·894
Nd₂Al₃Ge₄	6·042 (2)	14·965 (6)	7·897 (5)	714·1 (4)	2·477	1·895
Sm₂Al₃Ge₄	6·007 (2)	14·921 (6)	7·820 (4)	700·9 (4)	2·484	1·908
Gd₂Al₃Ge₄	5·948 (4)	14·875 (7)	7·762 (5)	686·7 (5)	2·501	1·916
Tb₂Al₃Ge₄	5·914 (3)	14·834 (7)	7·720 (4)	677·2 (4)	2·508	1·922
Dy₂Al₃Ge₄	5·881 (2)	14·800 (4)	7·690 (2)	669·4 (2)	2·517	1·925
Ho₂Al₃Ge₄	5·865 (4)	14·765 (9)	7·657 (6)	663·2 (6)	2·517	1·928
Er₂Al₃Ge₄	5·838 (3)	14·747 (9)	7·637 (3)	657·6 (4)	2·526	1·931
Hf₂Co₃Si₄*	5·206 (2)	13·761 (9)	6·778 (7)	486 (1)	2·643	2·030
Zr₂Ni₃Si₄	5·209 (3)	13·821 (9)	6·905 (4)	497·1 (9)	2·653	2·002
Hf₂Ni₃Si₄	5·1746 (2)	13·643 (1)	6·8492 (3)	483·53 (8)	2·637	1·992

* The unit-cell parameters of low-symmetry Hf$_2$Co$_3$Si$_4$ (Yarmolyuk & Aksel'rud, 1984) are not given in the standard setting *Pbcn* but in setting *Pbna* in order to simplify the comparison with the other listed compounds having space groups *Cmca*.

Discussion. A search in the literature for structures of the same stoichiometry, same space group and same Wyckoff sequence as La$_2$Al$_3$Ge$_4$ led to Ba$_2$Cd$_3$Bi$_4$ (Cordier, Woll & Schäfer, 1982) and Hf$_2$Ni$_3$Si$_4$ (Yarmolyuk, Pecharskii & Aksel'rud, 1988). The bismuthide and the silicide have related structures which can be considered as different branches of a common structure type. They differ in the values of their b/a and b/c ratios (see Table 3), in the heteronuclear coordination of the large cation, *i.e.* 5Ni + 7Si for Hf in Hf$_2$Ni$_3$Si$_4$ and 6Cd + 8Bi for Ba in Ba$_2$Cd$_3$Bi$_4$, and in the formation of Si—Si chains (Si—Si 2·587 Å) in Hf$_2$Ni$_3$Si$_4$ while corresponding Bi—Bi contacts (Bi—Bi 3·519 Å) in Ba$_2$Cd$_3$Bi$_4$ are even larger than the sum of the metallic radii.

The La$_2$Al$_3$Ge$_4$ structure can be classified as being isotypic to the Ba$_2$Cd$_3$Bi$_4$ branch of this structure type.

A projection of the La$_2$Al$_3$Ge$_4$ structure along the a axis is shown in the left-hand-side drawing of Fig. 1. The structure can be considered as a slab-stacking variant of the HfFe$_2$Si$_2$ type (Yarmolyuk, Lysenko & Gladyshevskii, 1976) shown on the right-hand side of Fig. 1 in a projection along the [001] direction. On the drawing of the HfFe$_2$Si$_2$ structure, two approximately 7·5 Å thick two-dimensional slabs are indicated. The slab borders, perpendicular to the [100] direction of HfFe$_2$Si$_2$, are not flat but corrugated. These same slabs are stacked in the La$_2$Al$_3$Ge$_4$ structure along the [010] direction in such a way that every second slab is shifted by ½a. The two Fe-atom sites close to the slab border coalesce into one Al(2) site in La$_2$Al$_3$Ge$_4$. This is the reason for the change of stoichiometry from HfFe$_2$Si$_2$ to Hf$_2$Ni$_3$Si$_4$ (or

La$_2$Al$_3$Ge$_4$) and for a change of coordination of some of the atoms as compared to those of the corresponding atoms in the HfFe$_2$Si$_2$ type.

The coordination figures of the Al(1) and Ge(1) atoms in La$_2$Al$_3$Ge$_4$ are identical to those of the Fe (at height ¼ and ¾) and Si atoms (at height 0 and ½) in HfFe$_2$Si$_2$. Each Al(1) (or Fe) atom has five close Ge (or Si) neighbours forming a trigonal bipyramid. The Ge(1) (or Si at height 0 and ½) are themselves at the centres of deformed trigonal prisms each consisting of five Al (or Fe) and one La (or Hf) atom. The Ge(2) atoms are coordinated each by four La and three Al atoms while the Si atoms are surrounded by a deformed monocapped square antiprism formed by 4Hf + 4Fe + 1Si.

The coordination figure of the Al(2) atom in La$_2$Al$_3$Ge$_4$ is a deformed icosahedron formed by 6La + 6Ge atoms. The Ge coordination of the Al(2) atom is a stretched octahedron. A similar coordination figure is also found for the Al atoms in U$_2$Cu$_3$Al (Blažina & Ban, 1980) with the Mg$_2$Cu$_3$Si type (Witte, 1939), a ternary-substitution variant of the MgZn$_2$ type, in which the Cu octahedron is strongly deformed.

The compounds Ba$_2$Cd$_3$Bi$_4$ and La$_2$Al$_3$Ge$_4$ are not valence compounds. 12 and 16 additional electrons would be needed to complete the Bi and Ge octets respectively; however, only 10 and 15 electrons respectively could at most be provided by the partner elements. With the number of valence electrons being insufficient to complete the octets, one can expect that a polyanionic valence compound with homonuclear anion bonds is formed. In the two structures under discussion there are no Bi—Bi or Ge—Ge bonds, the main bonds being essentially the Cd—Bi
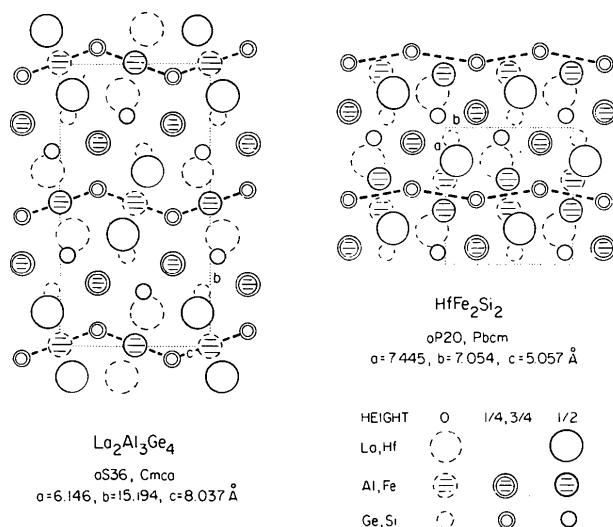

HfFe$_2$Si$_2$

oP20, Pbcm

a = 7·445, b = 7·054, c = 5·057 Å

HEIGHT	0	1/4,3/4	1/2
La,Hf			
Al,Fe			
Ge,Si			

La$_2$Al$_3$Ge$_4$

oS36, Cmca

a = 6·146, b = 15·194, c = 8·037 Å

Fig. 1. Projections of La$_2$Al$_3$Ge$_4$ along [100] and of HfFe$_2$Si$_2$ along [001]. The corrugated slab borders are indicated by heavy broken lines.

and Al—Ge bonds, respectively. However, as mentioned above, in the other structure-type branch, characterized by the $Hf_2Ni_3Si_4$ structure, there are Si—Si chains along the [100] direction.

As can be seen from Table 3 the unit-cell parameters and cell volumes of the isotypic $R_2Al_3Ge_4$ compounds decrease smoothly with increasing atomic number of the lanthanide elements. This is to be expected from the lanthanide contraction.

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Structure of 1,3,5,7-Tetrafluorocyclotetraazathiene at 103 K

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Abstract. $N_4F_4S_4$, $M_r = 260.26$, tetragonal, $P\bar{4}2_1c$, $a = 9.090$ (3), $c = 4.199$ (3) Å, $V = 346.9$ (5) Å³, $Z = 2$, $D_x = 2.49$ Mg m⁻³, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 1.34$ mm⁻¹, $F(000) = 256$, $T = 103$ (3) K, $R = 0.0385$, $wR = 0.0384$ (inverted direction $R = 0.052$ and $wR = 0.038$) based on 1384 unique reflections with $F \geq 3\sigma(F)$. The extended low-temperature data set yielded precise positional and thermal parameters. The bond lengths for N—S are 1.655 (2) and 1.544 (2) Å, the S—F distance is 1.616 (1) Å. The angles are: S—N—S 123.3 (1), N—S—N 111.7 (1), F—S—N 91.6 (1) and F—S=N 106.2 (1)°.

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Introduction. The present structure investigation was performed as part of a detailed study of S—N bonding. An electron density study of $S_4N_4H_4$ (Gregson, Klebe & Fuess, 1988) revealed the distribution of density between the atoms and in the lone-pair regions. Furthermore, the influence of hydrogen bonds on the electron density was stated. Whereas the S—N bonds in $S_4N_4H_4$ are almost equal in length, considerable differences were reported for $S_4N_4F_4$ which, therefore, seemed to be a good candidate for a comparative study. The electron densities were, however, not conclusive and we therefore report here the results of a precise low-temperature X-ray structure refinement.

Experimental. The title compound has been prepared by fluorination of S_4N_4 by AgF_2 in CCl_4 as solvent as described by Glemser, Schröder & Haeseler