This investigation is a part of a research program supported by the Swedish Natural Science Research Council.

References

Allmann, R. (1975). *Monatsh. Chem.* **106**, 779–793.

Andersson, G. (1954). *Acta Chem. Scand.* **8**, 1599–1606.

Åsbrink, S. (1973). Unpublished.

Åsbrink, S. (1975). *Mater. Res. Bull.* **10**, 861–864.

Åsbrink, S. & Andresen, A. (1980). In preparation.

Åsbrink, S., Friberg, S., Magnéli, A. & Andersson, G. (1959). *Acta Chem. Scand.* **13**, 603.

Åsbrink, S. & Sävborg, Ö. (1980). In preparation.

Brandt, B. G. (1971). *Chem. Commun. Univ. Stockholm*, No. II.

Brauer, G. (1962). *Handbuch der Präparativen Anorganischen Chemie*, II, p. 1109. Stuttgart: Ferdinand Enke Verlag.

Brückner, W., Moldenhauer, W., Thuss, B. & Försterling, G. (1976). *Phys. Status Solidi A*, **35**, K49–K51.

Brückner, W., Wich, H., Terukov, E. I. & Chudnovskii, F. A. (1975). *Fiz. Tverd. Tela* (*Leningrad*), **17**, 2191–2193.

Chudnovskii, F. A., Terukov, E. I. & Khomskii, D. I. (1978). *Solid State Commun.* **25**, 573–577.

Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.

Cruickshank, D. W. J., Pilling, D. E., Bujosa, A., Lovell, F. M. & Truter, M. R. (1961). *Computing Methods and the Phase Problem in X-ray Crystal Analysis*, p. 45. Oxford: Pergamon Press.

Dernier, P. D. (1970). *J. Phys. Chem. Solids*, **31**, 2569–2575.

Dunitz, J. D. & Orgel, L. E. (1960). *Adv. Inorg. Chem. Radiochem.* **2**, 1–60.

Hamilton, W. C. (1955). *Acta Cryst.* **8**, 185–186.

Hodeau, J.-L. & Marezio, M. (1978). *J. Solid State Chem.* **23**, 253–263.

Hong, S.-H. & Åsbrink, S. (1977). Fourth European Crystallographic Meeting, Oxford, PII.85.

Hong, S.-H. & Åsbrink, S. (1980). In preparation.

Hope, H. (1971). *Acta Cryst.* A**27**, 392–393.

Horiuchi, H., Morimoto, N. & Tokonami, M. (1976). *J. Solid State Chem.* **17**, 407–424.

Horiuchi, H., Tokonami, M., Morimoto, N., Nagasawa, K., Bando, Y. & Takada, T. (1971). *Mater. Res. Bull.* **6**, 833–843.

International Tables for X-ray Crystallography (1952). Vol. I. Birmingham: Kynoch Press.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Jennings, L. D. (1968). *Acta Cryst.* A**24**, 472–474.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Kartenko, N. F., Terukov, E. I. & Chudnovskii, F. A. (1976). *Fiz. Tverd. Tela* (*Leningrad*), **18**, 1874–1876.

Kihlborg, L. (1963). *Ark. Kemi*, **21**, 471–495.

Koenig, D. (1971). Unpublished.

Longo, J. M. & Kierkegaard, P. (1970). *Acta Chem. Scand.* **24**, 420–426.

Magnéli, A., Andersson, S., Westman, S., Kihlborg, L., Holmberg, B., Åsbrink, S. & Nordmark, C. (1959). *Studies on the Crystal Chemistry of Titanium, Vanadium and Molybdenum Oxides at Elevated Temperatures. Final Technical Report* 1, *DA*-91-591-*EUC*-935, pp. 23–30.

Miyake, S., Togawa, S. & Hosoya, S. (1964). *Acta Cryst.* **17**, 1083–1084.

Nagasawa, K., Bando, Y. & Takada, T. (1972). *J. Cryst. Growth*, **17**, 143–148.

Olekhnovich, N. M. (1969). *Sov. Phys. Crystallogr.* **14**, 203–206.

Pauling, L. (1947). *J. Am. Chem. Soc.* **69**, 542–553.

Shannon, R. D. & Prewitt, C. T. (1969). *Acta Cryst.* B**25**, 925–946.

Terukov, E. I. & Chudnovskii, F. A. (1974). *Fiz. Tekh. Poluprovodn.* **8**, 1226–1227.

Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Zachariasen, W. H. (1978). *Acta Cryst.* A**34**, S 406.

Acta Cryst. (1980). B**36**, 1339–1343

The Structure Determination of a New Intermetallic Compound K_3Ga_{13}

By Claude Belin

Laboratoire des Acides Minéraux, LA 79, *Université des Sciences et Techniques du Languedoc,*
Place Eugène Bataillon, 34060 *Montpellier CEDEX, France*

(*Received* 31 *October* 1979; *accepted* 6 *February* 1980)

Abstract

The compound is orthorhombic; the unit cell with $a = 6{\cdot}441\ (3)$, $b = 16{\cdot}143\ (3)$, $c = 28{\cdot}404\ (7)$ Å contains eight formula units. Diffraction data with $0 < 2\theta < 50°$ (Mo $K\alpha$ radiation) were collected on a Nonius CAD-4 automatic diffractometer within the octant $\bar{h}kl$. The structure was solved in space group *Cmcm* by direct methods and refined by full-matrix least squares to a final $R(F)$ of 5·6% for 1040 reflections with $I > 3\sigma(I)$.

0567-7408/80/061339-05$01.00

The structure can be described in terms of a packing of two types of polyhedra (11 Ga and 12 Ga atoms) linked to each other in a non-compact netting leaving holes where potassium atoms can sit.

Introduction

The system K–Ga was first investigated (DTA) by Rinck & Feschotte (1961) and Feschotte (1961); the diagram displays two compounds: K_5Ga_8 which melts congruently at 893 K and KGa_4 which decomposes peritectically at 778 K. So far, Bruzzone (1965) has reported MX_4 phases between Group III*a* elements and alkaline-earth metals and more recently (Bruzzone, 1969) with alkaline metals. KGa_4 was found to possess an orthorhombic symmetry with the following parameters $a = 13{\cdot}86$, $b = 16{\cdot}02$ and $c = 6{\cdot}49$ Å while $NaGa_4$ displays a tetragonal $D1_3$ ($BaAl_4$) structure type.

Experimental

The metals used were Alusuisse gallium and Merck potassium; the latter was purified through several fractional recrystallizations.

A 95 mol % gallium mixture was prepared by weighing the elements in a dry box filled with argon. The mixture was then fused and heated up to 773 K in a tantalum tube which had previously been sealed by welding in an argon atmosphere and then allowed to cool slowly to room temperature. The resulting solid metallic mass appeared to contain many thin needles which were then separated from the liquid phase by centrifugation at 353 K in sealed Pyrex tubes; these tubes contained silica wool to retain the crystals.

The crystals were examined under an optical microscope inside a glove bag filled with argon; most of them appeared to be twinned along the elongation axis (*a*); however, a trigonal-prism-shaped crystal was selected and mounted in a 0·2 mm diameter Lindemann-glass capillary using a cyanoacrylate ester adhesive to hold it in position.

Preliminary oscillation and Weissenberg photographs showed the crystal to belong to the orthorhombic system; systematic extinctions suggested the following space groups: *Cmcm*, *Ama*2, *Cmc*2_1. Lattice constants were determined by least-squares refinement of the angular positions of 25 reflections collected and centered on an Enraf–Nonius CAD-4 automatic diffractometer. The crystal data are given in Table 1.

Integrated diffraction intensities were collected at room temperature (293 K) in the range $0 < 2\theta < 50°$ within the $\bar{h}kl$ octant using graphite-monochromatized Mo $K\alpha$ radiation. (The profile analysis of a few low-angle reflections indicated that an ω–2θ scan method was the most appropriate for data collection.)

Table 1. *Summary of crystal data*

Mo $K\alpha$ radiation $\lambda = 0{\cdot}71069$ Å
Molecular formula K_3Ga_{13}
$M_r = 1023{\cdot}67$
Linear absorption coefficient $\mu = 25{\cdot}3$ mm^{-1}
$D_c = 4{\cdot}604$ Mg m^{-3}
Crystal shape: elongated trigonal prism
Crystal dimensions 0·10 × 0·25 mm
Boundary planes $\{00\bar{1}\}$, $\{011\}$, $\{0\bar{1}1\}$, $\{100\}$, $\{\bar{1}00\}$
Crystal volume 0·00119 mm^3
Space group *Cmcm*
$Z = 8$
Cell constants $a = 6{\cdot}441$ (3), $b = 16{\cdot}143$ (3), $c = 28{\cdot}404$ (7) Å
Cell volume 2953·36 Å^3

During data collection, the intensities of three standard reflections were checked after every 50 reflections; a loss of 30, 26 and 45% in intensity was observed due to crystal decomposition and was taken into account in data reduction.

The data were corrected for background and Lorentz–polarization effects. Once the composition of the compound was known, the data were corrected for the effects of absorption by Gaussian integration (Busing & Levy, 1957) with $\mu = 25{\cdot}3$ mm^{-1}. The final data set consisted of 1847 independent reflections of which 1040 with $I > 3\sigma(I)$ were used in refinement.

Structure solution and refinement

The structure was solved by direct methods. The Wilson plot gave a strong indication of centricity, so an attempt was made to solve the structure in the space group *Cmcm* which proved later on to be appropriate.

The output from the Fourier step of *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974) contained 11 peaks of high weight which were attributed to Ga atoms and three peaks of lower weight to K atoms. After a few cycles of positional and isotropic thermal-parameter refinement, $R = \sum||F_o| - |F_c||/\sum|F_o| = 0{\cdot}21$ and a subsequent Fourier map contained no extra peaks. Finally, all atomic positional parameters and anisotropic temperature factors were refined by full-matrix least squares minimizing the function $W(|F_o| - |F_c|)^2$ with $W^{-1} = \sigma^2_{\rm count}(F^2)/4(F)^2 + (0{\cdot}01F)^2$ and using the final data set corrected for absorption. The final agreement factors were $R(F) = 0{\cdot}056$ and $R_w(F) = 0{\cdot}070$; the goodness-of-fit, defined as $[W(|F_o| - |F_c|)^2/(N - M)]^{1/2}$ where N is the number of observations (1040) and M is the number of parameters varied (91), was 1·06.

The data were corrected for isotropic extinction according to Zachariasen (1967) and Coppens & Hamilton (1970) but 30 reflections which appeared to be very seriously affected were excluded from the R calculation. The final difference Fourier map was flat except for some spurious peaks near Ga atoms of height <2·5 e Å^{-3}. In the final cycle of refinement the shifts in atom positional parameters were zero while the shift in temperature factors was <0·004σ. The uncertainties in gallium positions range from 0·0032 to 0·0064 Å while those of potassium range from 0·0048 to 0·010 Å. This fact with the yet relatively high residual (R = 0·056) must arise primarily from the occurrence of some random error in the data set originating from a crystal still slightly twinned and, to a larger extent, from the anisotropy in the decay of intensities shown by the three standard reflections, which was not corrected by the procedure used.

Residual peaks of 2·5 e Å^{-3} could have suggested the presence of some impurities in the crystal (Li atoms for example) but they occur at a distance less than 1·1 Å from gallium centers. The probability of some gallium disorder can be excluded because of small temperature factors and the multiplicity of Ga atoms showed no change on refinement.

Corrections for the anomalous-dispersion effects were included in the neutral-atom scattering factors (Doyle & Turner, 1968).

The crystallographic programs used were *MULTAN* (Main *et al.*, 1974), *DRF* (a modification of the Zalkin Fourier program), *ORFLS* (Busing, Martin & Levy, 1962) for least-squares refinements, *ORFFE* (Busing, Martin & Levy, 1964) for molecular and error functions and *ORTEP* II (Johnson, 1971), the thermal ellipsoid program for molecular plots.

Results and discussion

The final positional and thermal parameters are listed in Table 2,* bond distances and angles are given in Table 3.

Table 2. *Final positional parameters* (×10^4) *for atoms in* K_3Ga_{13}

	N	x	y	z	B_{eq} (Å^2)
Ga(1)	16(*h*)	8004 (5)	8287 (2)	7968 (1)	1·008
Ga(2)	8(*g*)	7099 (7)	9683 (2)	7500	0·953
Ga(3)	4(*c*)	5000	7276 (4)	7500	1·196
Ga(4)	4(*c*)	0	738 (4)	7500	1·017
Ga(5)	8(*f*)	5000	9268 (3)	8288 (2)	1·022
Ga(6)	8(*f*)	5000	7590 (3)	8439 (2)	1·011
Ga(7)	8(*f*)	5000	8429 (3)	9672 (2)	1·002
Ga(8)	16(*h*)	8069 (5)	9508 (2)	9643 (1)	0·943
Ga(9)	8(*f*)	5000	9842 (3)	9105 (2)	1·043
Ga(10)	16(*h*)	2950 (5)	1105 (2)	9534 (1)	1·278
Ga(11)	8(*f*)	5000	7018 (3)	9280 (2)	0·921
K(1)	8(*f*)	5000	1376 (6)	6798 (4)	2·489
K(2)	8(*f*)	0	163 (6)	9611 (3)	1·691
K(3)	8(*f*)	0	7871 (6)	9138 (4)	1·966

N = Number of positions and Wyckoff notation.

Table 3. *Interatomic distances* (Å) *and angles* (°)

Ga(1)–1Ga(1)	2·571 (6)
Ga(1)–1Ga(6)	2·609 (4)
Ga(1)–1Ga(1)	2·656 (5)
Ga(1)–1Ga(5)	2·662 (4)
Ga(1)–1Ga(2)	2·680 (4)
Ga(1)–1Ga(3)	2·858 (5)
Ga(1)–1K(1)	3·407 (9)
Ga(1)–1K(3)	3·626 (9)
Ga(1)–1K(2)	3·763 (9)
Ga(2)–1Ga(4)	2·529 (6)
Ga(2)–2Ga(1)	2·680 (4)
Ga(2)–2Ga(5)	2·699 (4)
Ga(2)–1Ga(2)	2·705 (9)
Ga(2)–2K(1)	3·644 (9)
Ga(2)–2K(2)	3·748 (8)
Ga(3)–1Ga(4)	2·482 (9)
Ga(3)–2Ga(6)	2·716 (5)
Ga(3)–4Ga(1)	2·858 (5)
Ga(3)–4K(1)	4·056 (7)
Ga(4)–1Ga(3)	2·482 (9)
Ga(4)–2Ga(2)	2·529 (6)
Ga(4)–2K(2)	3·289 (9)
Ga(4)–4K(1)	3·925 (6)
Ga(5)–1Ga(9)	2·500 (6)
Ga(5)–2Ga(1)	2·662 (4)
Ga(5)–2Ga(2)	2·699 (4)
Ga(5)–1Ga(6)	2·744 (6)
Ga(5)–1K(1)	3·412 (10)
Ga(5)–2K(2)	3·647 (5)
Ga(6)–1Ga(11)	2·560 (6)
Ga(6)–2Ga(1)	2·609 (4)
Ga(6)–1Ga(3)	2·716 (5)
Ga(6)–1Ga(5)	2·744 (6)
Ga(6)–2K(3)	3·809 (6)
Ga(6)–2K(1)	3·829 (6)
Ga(6)–1K(2)	3·948 (10)
Ga(7)–1Ga(11)	2·536 (6)
Ga(7)–2Ga(8)	2·636 (4)
Ga(7)–2Ga(10)	2·718 (5)
Ga(7)–1Ga(9)	2·792 (6)
Ga(7)–2K(3)	3·673 (5)
Ga(7)–1K(3)	3·979 (10)
Ga(8)–1Ga(8)	2·487 (6)
Ga(8)–1Ga(9)	2·556 (4)
Ga(8)–1Ga(8)	2·576 (5)
Ga(8)–1Ga(10)	2·621 (4)
Ga(8)–1Ga(7)	2·636 (4)
Ga(8)–1Ga(10)	2·678 (4)
Ga(8)–1K(3)	3·254 (9)
Ga(8)–1K(2)	3·355 (9)
Ga(9)–1Ga(5)	2·500 (6)
Ga(9)–2Ga(8)	2·556 (4)
Ga(9)–2Ga(10)	2·718 (5)
Ga(9)–1Ga(7)	2·792 (6)
Ga(9)–2K(2)	3·552 (4)
Ga(9)–1K(1)	3·567 (11)
Ga(10)–1Ga(11)	2·511 (4)
Ga(10)–1Ga(8)	2·621 (4)
Ga(10)–1Ga(10)	2·641 (6)
Ga(10)–1Ga(8)	2·678 (4)
Ga(10)–1Ga(9)	2·718 (5)
Ga(10)–1Ga(7)	2·718 (5)
Ga(10)–1K(3)	3·338 (9)
Ga(10)–1K(2)	3·578 (8)
Ga(11)–2Ga(10)	2·511 (4)
Ga(11)–1Ga(7)	2·536 (6)
Ga(11)–1Ga(6)	2·560 (6)
Ga(11)–2K(3)	3·526 (4)
Ga(11)–1K(2)	3·548 (10)
K(1)–2Ga(1)	3·407 (9)
K(1)–1Ga(5)	3·412 (10)
K(1)–1Ga(9)	3·567 (11)
K(1)–1K(3)	3·950 (14)
K(1)–2Ga(2)	3·644 (9)
K(1)–2Ga(6)	3·829 (6)
K(1)–2Ga(4)	3·925 (6)
K(1)–2K(2)	3·945 (8)
K(1)–1K(1)	3·987 (22)
K(2)–1Ga(4)	3·289 (9)
K(2)–2Ga(8)	3·355 (9)
K(2)–1Ga(11)	3·548 (10)
K(2)–2Ga(9)	3·552 (4)
K(2)–2Ga(10)	3·578 (8)
K(2)–2Ga(5)	3·647 (5)
K(2)–2Ga(2)	3·748 (8)
K(2)–2Ga(1)	3·763 (9)
K(2)–2K(1)	3·945 (8)
K(2)–1Ga(6)	3·948 (10)
K(2)–1K(3)	3·990 (14)
K(3)–2Ga(8)	3·254 (9)
K(3)–2Ga(10)	3·339 (9)
K(3)–2Ga(11)	3·526 (4)
K(3)–1K(1)	3·590 (14)
K(3)–2Ga(1)	3·626 (9)
K(3)–2Ga(7)	3·673 (5)
K(3)–2Ga(6)	3·809 (6)
K(3)–1Ga(7)	3·979 (10)
K(3)–1K(2)	3·990 (13)
Ga(2)–Ga(4)–Ga(2)	95·3 (3)
Ga(2)–Ga(4)–Ga(3)	132·4 (1)
Ga(3)–Ga(4)–Ga(2)	132·4 (1)
K(1)–Ga(4)–K(1)	149·6 (3)
K(1)–Ga(4)–K(1)	61·0 (3)
K(2)–Ga(4)–K(2)	147·2 (4)
K(2)–Ga(4)–K(1)	124·1 (1)
K(2)–Ga(4)–K(1)	65·6 (2)
Ga(6)–Ga(11)–Ga(10)	118·7 (1)
Ga(10)–Ga(11)–Ga(10)	98·4 (2)
Ga(6)–Ga(11)–Ga(7)	94·9 (2)
Ga(7)–Ga(11)–Ga(10)	113·6 (1)
K(3)–Ga(11)–K(3)	131·9 (3)
K(3)–Ga(11)–K(2)	105·6 (2)
Ga(9)–Ga(5)–Ga(6)	109·7 (2)
Ga(5)–Ga(6)–Ga(11)	120·1 (2)
Ga(9)–Ga(7)–Ga(11)	118·7 (2)
Ga(5)–Ga(9)–Ga(7)	103·5 (2)
Ga(7)–Ga(10)–Ga(11)	116·3 (2)
Ga(11)–Ga(7)–Ga(10)	127·8 (2)

The structure is characterized by a relatively complex packing of two types of gallium polyhedra where the K atoms occupy some vacant holes (Fig. 1); of eleven Ga atoms, three occur at general positions (1,

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35144 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

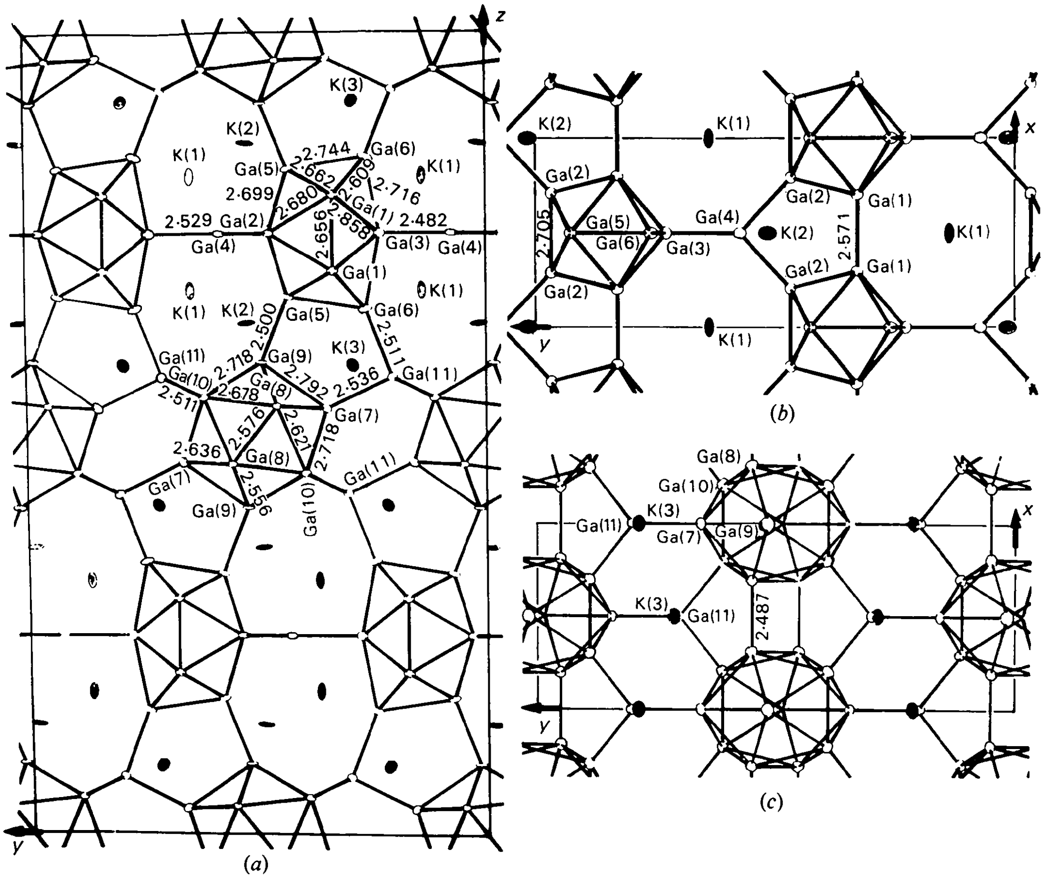

Fig. 1. (*a*) Projection of crystal packing down the *x* axis. (*b*) Section between $z = 0{\cdot}63$ and $z = 0{\cdot}87$ showing type (*A*) polyhedra interconnections. (*c*) Section between $z = 0{\cdot}38$ and $z = 0{\cdot}62$ showing type (*B*) polyhedra interconnections.

8, 10), two at the special position $0,y,\frac{1}{4}$ (3, 4), one at $x,y,\frac{1}{4}$ (2) and five at $0,y,z$ (5, 7, 9, 6, 11) while all K atoms occur at the special position $0,y,z$.

Polyhedron (*A*) contains 11 Ga atoms (1, 2, 3, 5, 6). Crystal symmetry reproduces Ga(1) three times [through perpendicular (100) and (004) symmetry planes]; Ga(2) is reproduced once with respect to (100), Ga(5) and Ga(10) are reproduced once with respect to (004). Ga(2), Ga(3) and Ga(4) lie in the plane (004). Polyhedron (*A*) is linked along the *x* axis to two similar adjacent polyhedra in the lower and upper cells through four direct Ga(1)–Ga(1) and two bridged Ga(2)–Ga(4)–Ga(2) bonds. Polyhedron (*B*) is an icosahedron similar to those found in boron structures; it contains 12 Ga atoms (7, 8, 9, 10) arranged around inversion centers; in the same way as (*A*), it is linked to its lower and upper homologues through four direct Ga(8)–Ga(8) and four bridged Ga(10)–Ga(11)–Ga(10) bonds. Polyhedra (*A*) and (*B*) are linked in the plane (100) through Ga(5)–Ga(9) direct bonding and Ga(6)–Ga(11)–Ga(7) bridged bonding, approximately constituting zig-zag chains.

Moreover, each chain so defined is linked to its parallel homologues produced by *C* centering through double-bridged bonds involving Ga(4) and Ga(11) as centers.

The packing of Ga atoms is not compact but leaves holes in which K atoms can lie; these atoms lie in parallel planes (100) surrounding polyhedron (*A*) Ga atoms. Ga(1), (2), (5), (7), (8), (9), (10) have six coordinating Ga at less than 3·5 Å, Ga(3) has seven while Ga(6) has five coordinating Ga atoms; Ga atoms with the lowest coordination numbers are Ga(4), coordination number 3, and Ga(11), coordination number 4. In fact, these atoms occupy key positions that ensure the linkage between zig-zag chains. Ga(4) is surrounded by one Ga(3) and two Ga(2) atoms with a nearly threefold arrangement. The nine coordination is re-established by six K atoms at distances less than 4·0 Å. Ga(11) is coordinated to four neighboring Ga atoms arranged nearly tetrahedrally (98·4, 113·6, 118·7, 94·9°); the coordination is supplemented by the insertion of three K atoms at distances less than 3·6 Å and two K atoms at 4·563 Å that account for the non-equality of gallium coordination angles.

On the other hand, one must stress the relatively short gallium–potassium distances K(3)–Ga(8) = 3·254, K(2)–Ga(4) = 3·289, K(3)–Ga(10) = 3·339 and K(2)–Ga(8) = 3·355 Å compared to an expected metal–metal distance of about 3·5 Å.

The author wishes to express his thanks to Dr P. de la Bretèque (Alusuisse France SA) for the gift of a large quantity of gallium and to Drs J. Potier and J. Rozière for helpful discussions.

References

BRUZZONE, G. (1965). *Acta Cryst.* **18**, 1081–1082.

BRUZZONE, G. (1969). *Acta Cryst.* B**25**, 1206–1207.

BUSING, W. R. & LEVY, H. A. (1957). *Acta Cryst.* **10**, 180–182.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee.

COPPENS, P. & HAMILTON, W. C. (1970). *Acta Cryst.* A**26**, 71–83.

DOYLE, P. A. & TURNER, P. S. (1968). *Acta Cryst.* A**24**, 390–397.

FESCHOTTE, P. (1961). *Ann. Chim.* (*Paris*), **6**, 1029–1070.

JOHNSON, C. K. (1971). *ORTEP* II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univ. of York, England.

RINCK, E. & FESCHOTTE, P. (1961). *C. R. Acad. Sci.* **252**, 3592–3594.

ZACHARIASEN, W. H. (1967). *Acta Cryst.* **23**, 558–564.