Table 4. *A comparison of interatomic separations* (Å) *found in iron-group chloride dihydrates*

	Mn	Fe	Co	Ni
Expected*				
Ionic M–Cl	2·61	2·57	2·55	2·53
Covalent M–Cl		2·22	2·31	2·38
Experimental				
M–Cl	2·515	2·488	2·459 (3)†	2·387 (2)
M–Cl′	2·592	2·542	2·487 (3)	2·410 (2)
M–O	2·150	2·074	2·034 (5)	2·089 (5)
Cl–O	3·767	3·713	3·695 (6)	3·542 (5)‡
				3·521 (5)
Cl–Cl	3·992	3·989	3·958 (4)	3·589 (2)
				4·316 (2)
Difference in M–Cl and M–Cl′ separation	0·077	0·054	0·028	0·023

* The radii used to obtain the expected ionic and covalent separations are taken from Pauling (1960); no value is given for the six-coordinated covalent manganese(II) radius.

† The errors in MIC are all similar.

‡ These separations in $NiCl_2.2H_2O$ are believed to be influenced by hydrogen interactions.

oxygen atom is associated with a direction perpendicular to the chain as well as to the 2·921 Å oxygen–oxygen separation.

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Complex Sulphur Compounds. I. The Crystal Structure of $MgGa_2S_4$

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Single monoclinic crystals of $MgGa_2S_4$ are obtained by heating the elements in appropriate amounts *in vacuo* at 1200°C. The space group is $C2/c$, $Z=12$ and the unit-cell dimensions are $a=12·74$, $b=22·54$, $c=6·43$ Å and $\beta=108·8$°. Comparison with analogous $M^{II}M_2^{III}S_4$ compounds and consideration of lattice dimensions lead to a cubic close-packed array of sulphur ions. Initial parameters were derived from a Patterson analysis and the structure was refined by Fourier and least-squares methods using 1310 observed reflexions. Three double layers of anions with Ga in tetrahedral interstices in the first and third double layer and with Mg in octahedral interstices in the second one are succeeded by an empty double layer. The interspacing of the empty layers is larger than that of the filled layers and results in a distortion of the array of sulphur ions.

Introduction

Structures of complex sulphides with composition $M^{II}M_2^{III}S_4$ have been reported by several authors. M^{II} and M^{III} being metals of the first transition period, spinel (Lotgering, 1956; Koerts, 1965; Bouchard & Wold, 1966; Holt, Bouchard & Wold, 1966) or defect NiAs-structure types (Jellinek, 1957) usually occur. We have extended the investigations to compounds in which M^{II} is Mg, Mn, Fe, Co, Ni and M^{III} is Al, Ga, Nb or Ta.

In this article we report the crystal structure of $MgGa_2S_4$ derived from single-crystal X-ray analysis. The remaining compounds, which are only available in the form of very small plate-like crystals, have been analysed by powder methods and will be dealt with in subsequent papers.

Experimental

$MgGa_2S_4$ was obtained by adding the elements in appropriately weighed amounts in an alumina crucible. The crucible was inserted into a quartz tube which was evacuated, sealed and slowly heated to 1200°C. After slowly cooling down to room temperature at a rate of approximately 16°C per hr, single crystals were present in the crucible.

The crystals are cream-coloured needles with a metallic lustre and elongated along [001]. Unit-cell dimensions (Table 1) were derived from an $h0l$ precession photograph (Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å) and from rotation and zero layer Weissenberg photographs about [001] (Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å). The Weissenberg exposure was superposed with aluminum powder lines ($a = 4{\cdot}0489$ Å at 20°C) for calibration purposes. The stated errors in cell edges are estimated standard deviations. The density was measured by the flotation method and corresponded to twelve formulae units per unit cell.

Table 1. *Crystal data of* $MgGa_2S_4$ *at* 20°

$a = 12{\cdot}74 \pm 0{\cdot}02$ Å	$d(\text{exp}) = 3{\cdot}29$ g.cm^{-3}
$b = 22{\cdot}54 \pm 0{\cdot}05$	$d(\text{X-ray}) = 3{\cdot}32$ g.cm^{-3}
$c = 6{\cdot}43 \pm 0{\cdot}02$	$Z = 12$; $F(000) = 1656$
$\beta = 108{\cdot}8 \pm 0{\cdot}2°$	$\mu(\text{Cu } K\alpha) = 247$ cm^{-1}

The absent reflexions hkl for $h+k=2n+1$ and $h0l$ for $(h), l=2n+1$ indicate the possible space groups $C2/c$ and Cc. The former was chosen on grounds of the Patterson analysis and its choice was corroborated by the ultimate refinement of the structure (next section).

Non-integrated equi-inclination Weissenberg photographs (multiple film technique) of the layers $l = 0,1\ldots5$ were taken with copper radiation at room temperature. The intensities of 1619 reflexions, excluding 309 too weak to be observable, were estimated visually with a calibrated intensity strip and reduced to structure factors in the usual way. An absorption correction was applied, assuming a cylindrical cross-section for the crystal used ($r \simeq 0{\cdot}008$ cm, $\mu r = 2{\cdot}0$). In addition, integrated precession photographs of the layers $h0l$ and $h1l$ were recorded with molybdenum radiation. However, attempts to put all measured structure factors on one common scale were not successful. Therefore, only Weissenberg data with separate scale factors for all six layers were used in the least-squares refinement (see next section). The least-squares program (Geise, Romers & Rutten, 1966) was used in a version that refines n scale factors and one overall temperature factor from an $(n+1) \times (n+1)$ matrix. The individual temperature factors of all atoms were kept isotropic and the ultimate scale factors were 0·80, 0·88, 0·81, 1·01, 0·83 and 0·77, respectively.

Structure determination and refinement

The hexagonal or spinel-like structures of most $M^{II}M_2^{III}S_4$ compounds are based upon hexagonal, cubic or mixed closed-packed arrays of sulphur ions (IJdo, 1967). Let us consider an ideally closed-packed cubic array of sulphur atoms with diameter $d = 3{\cdot}75$ Å. The lattice has a cell edge of $d\sqrt{2}$. With the transformation

$$\begin{matrix} \frac{1}{2} & 1 & \frac{1}{2} \\ -\frac{1}{2} & 0 & \frac{1}{2} \\ \frac{1}{2} & -1 & \frac{1}{2} \end{matrix}$$

this unit cell can be transformed into a face-centered monoclinic cell having the same volume with dimensions

$$a' = c' = d\sqrt{3} = 6{\cdot}49 \text{ Å}$$
$$b' = d = 3{\cdot}75 \text{ Å}$$
$$\text{and } \beta' = 109{\cdot}5° .$$

This corresponds to a subcell of the unit cell of $MgGa_2S_4$, since the angles β and β' are nearly the same and $a/a' = 1{\cdot}97$, $b/b' = 6{\cdot}0$ and $c/c' = 0{\cdot}99$. It follows that the true cell contains $12 \times 4 = 48$ anions and 12 formula units in agreement with the result of the experimentally determined density. The presence of the subcell is more or less evident from the intensity distribution of a small set of strong and a large set of weak reflexions. The former set is ruled by the conditions:

$$\begin{matrix} h = \text{even}, & l = \text{even}, & k = 0 & \text{modulo } 12; \\ h = \text{odd}, & l = \text{even}, & k = \pm 1 & \text{modulo } 12; \\ h = \text{even}, & l = \text{odd}, & k = 6 & \text{modulo } 12; \\ h = \text{odd}, & l = \text{odd}, & k = 6 \pm 1 & \text{modulo } 12. \end{matrix}$$

Such a distribution indicates that all or nearly all y parameters are odd multiples of 1/24. By arranging sulphur ions on lines parallel to [010] at positions $y = -3/24$, 1/24, 5/24, ... *etc.* or at positions $y = -1/24$, 3/24, 7/24, ... *etc.* we get separations of $22{\cdot}54/6 = 3{\cdot}76$ Å confirming that b coincides with b'. The cations have to be accommodated in octahedral or tetrahedral interstices and a model study reveals that their y parameters should be odd multiples of 1/24. The Patterson function $P(uvw)$ contains two strong peaks on the line $0v0$ at $v = \frac{1}{6}$ and $\frac{2}{6}$ and three strong ones on the line $0v\frac{1}{2}$ at $v = \frac{1}{12}$, $\frac{3}{12}$ and $\frac{5}{12}$. The peaks at $0\frac{1}{6}0$ and at $0\frac{1}{12}\frac{1}{2}$ are outstanding with heights of 0·4 and 0·6 times the value of the origin peak. The former indicates that all atoms are located approximately on lines parallel to [010] at separations of 3·76 Å. The latter is due to glide plane interactions between atoms on the array $x, y = -3/24$, 1/24, 5/24 *etc.*, z and atoms on the array $x, y = 3/24$, $-1/24$, $-5/24$ *etc.*, $\frac{1}{2}+z$. A very weak peak is observed at $0\frac{1}{2}0$ indicating that exclusively rows of anions are running *ad infinitum*. The rows of cations terminate after the third one. Consideration of the section $v = 0$ and of all remaining peaks in the Patterson function demonstrated that the centrosymmetric space group $C2/c$ should be chosen. It proved possible to postulate a model whose parameters are listed in Table 2. All sulphur atoms are in general positions $8(f)$ with z nearly equal to zero. The gallium ions are also in general positions, but the magnesium ions are on twofold axes in special positions $4(e)$.

Table 2. *Initial parameters*

	x	y	z
S(1)	$\frac{1}{8}$	$-\frac{1}{8}$	0
S(2)	$\frac{1}{8}$	$\frac{1}{24}$	0
S(3)	$\frac{1}{8}$	$\frac{5}{24}$	0
S(4)	$\frac{3}{8}$	$\frac{1}{8}$	0
S(5)	$\frac{3}{8}$	$-\frac{1}{24}$	0
S(6)	$\frac{3}{8}$	$-\frac{5}{24}$	0
Ga(1)	0·31	$-\frac{1}{8}$	0·14
Ga(2)	0·31	$\frac{1}{24}$	0·14
Ga(3)	0·31	$\frac{5}{24}$	0·14
Mg(1)	0	$-\frac{1}{8}$	$\frac{1}{4}$
Mg(2)	0	$\frac{1}{24}$	$\frac{1}{4}$
Mg(3)	0	$\frac{5}{24}$	$\frac{1}{4}$

This idealized structure roughly accounts for the strong reflexions and gives a reliability index $R=69\%$. After five cycles of electron density maps R was reduced to 35%. The atoms had slowly shifted from their initial positions and a fair account of the weak reflexions was now obtained. The refinement was continued with the least-squares method in a block-diagonalized version (Geise, Romers & Rutten, 1966; Rutten-Keulemans, 1966) that calculates separate scale factors for the levels $hk0$, $hk1$, ... $hk5$. The reliability index dropped to 19% after five cycles with isotropic individual temperature factors.

A search for possible errors in structure factors indicated that nearly all strong reflexions suffer from extinction. This phenomenon was also clear in a difference electron-density map which showed spherical troughs coinciding with atomic sites. Attempts to correct for extinction according to the method outlined by Zachariasen (1963) gave only little improvement. Intensity measurements of the strong reflexions by the powder method were not successful because the large unit cell gives too many overlapping diffraction lines. Finally we decided to omit 32 strong reflexions in the further refinement.

The difference Fourier synthesis gave no indication of anisotropy of atomic thermal motion. The isotropic refinement was, therefore, continued and after four cycles R decreased to 14·6%. Because the parameter shifts in the final cycle were less than one fourth of their corresponding standard deviations the refinement was stopped at that point. The scattering factors were taken from *International Tables for X-ray Crystallography* (1962) and the structure factors were calculated for the asymmetric unit. A list of observed and calculated structure factors including those suffering from extinction is given in Table 3.

Positional and isotropic thermal parameters as well as their respective standard errors are given in Table 4. The standard deviations calculated from the diagonal elements in the normal equation matrix are too low, because the refinement had not been carried out with a full-matrix program. Moreover, the applied absorption correction has an approximate character, explaining why attempts to correct for extinction were not successful. It is difficult to guess how much larger the standard deviations should be, but a sound estimate based upon our experience in earlier structure determinations (Geise & Romers, 1966) seems to be 30%. Adding this percentage to the standard deviations of the interatomic distances we arrive at errors of 0·007 Å

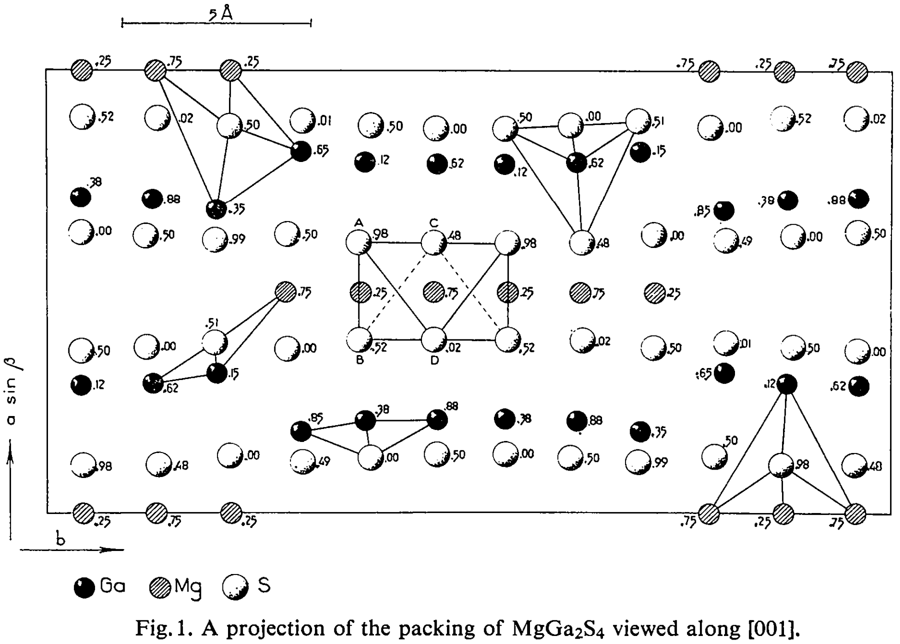

Fig. 1. A projection of the packing of $MgGa_2S_4$ viewed along [001].

in Ga–S and of 0·013 Å in Mg–S in directions parallel to **b** and of 0·0055 Å in directions perpendicular to **b**. The errors in the dimensions of the unit cell are suffiently small to neglect their influence on the standard deviations in bond distances and angles.

Discussion

The projection of the structure perpendicular to [001] shows clearly (Fig. 1) that the packing deviates from the postulated ideal model of Table 2. The anions are,

Table 3. *Structure factors of* MgGa$_2$S$_4$ *calculated for the asymmetric unit*

Structure factors suffering from extinction are listed separately under the heading *extinction*.

h	k	l	F_c	F_o
+0	+4	+0	-4.5	-5.8
	+6		+8.1	+9.2
	+8		-6.0	-7.2
	+10		-1.1	-2.8
	+14		-12.4	-15.2
	+16		+10.1	+10.1
	+18		-10.8	-10.5
	+20		+4.3	+5.9
	+22		+7.9	+7.8
	+26		+13.4	+10.9
	+28		-5.3	-4.8
+1	+1		-5.7	-6.0
	+3		+7.0	+8.9
	+5		-5.5	-8.5
	+7		+4.0	+5.4
	+11		-16.6	-19.5
	+13		+22.1	+20.7
	+15		-5.3	-5.8
	+23		+15.2	+11.3
	+25		-15.9	-11.9
+2	+2		-6.6	-7.1
	+4		+5.4	+4.8
	+6		-6.1	-7.6
	+8		+5.0	+3.8
	+10		-2.9	-4.1
	+12		+32.7	+29.5
	+14		+12.2	+15.0
	+16		-7.3	-7.6
	+18		+8.9	+9.1
	+22		+5.4	+3.1
	+24		-12.8	-11.6
	+26		-7.9	-6.6
	+28		+4.0	+3.6
+3	+3		-20.5	-18.1
	+5		+14.3	+14.0
	+7		-9.3	-11.1
	+9		+8.2	+9.5
	+11		-20.2	-19.2
	+15		+13.5	+15.5
	+17		-5.3	-6.0
	+25		+25.2	+18.5
	+27		-2.5	-2.5
+4	+0		-30.2	-26.6
	+2		+13.6	+11.8
	+4		-3.8	-4.6
	+8		+9.3	+9.3
	+10		-12.1	-10.7
	+12		+11.6	+15.6
	+14		-5.6	-5.4
	+20		-6.9	-6.6
	+24		-3.5	-2.5
	+26		-2.4	-2.7
+5	+1		-38.9	-31.3
	+3		+10.4	+11.5
	+5		-6.5	-8.1
	+11		+28.7	+24.4
	+13		+26.5	+21.9
	+15		-10.4	-9.7
	+17		+6.3	+6.9
	+23		-14.8	-12.5
	+25		-9.1	-8.5
	+27		+5.2	+5.4
+6	+0		+17.0	+16.2
	+2		-16.0	-14.8
	+4		+7.7	+9.6
	+8		-9.6	-9.1
	+10		+14.5	+13.2
	+12		-10.2	-9.6
	+14		+5.8	+6.0
	+20		+4.4	+4.5
	+22		-6.1	-5.9
	+24		+1.7	+3.0
+7	+1		+28.1	+25.4
	+3		-8.0	-8.4
	+5		+5.7	+5.6
	+9		+8.0	+8.2
	+11		-27.4	-22.2
	+13		-12.6	-11.9
	+15		+3.9	+4.8
	+19		-2.7	-3.0
	+21		-4.2	-2.6
	+23		+14.6	+13.2
	+25		+1.7	+2.6
+8	+0		+13.6	+17.5
	+2		+6.3	+6.6
	+6		-6.1	-8.3
	+8		+6.6	+7.6
	+10		-10.6	-9.1
	+12		-10.7	-10.9
	+16		-6.5	-5.6
	+18		+10.1	+9.3
	+20		-5.3	-5.2
	+22		+8.4	+8.1
	+24		+4.0	+3.8
+9	+1		-7.3	-9.0
	+3		-6.7	-7.7
	+5		+10.9	+10.9
	+7		-9.2	-10.3
	+11		-1.4	-3.4
	+13		+12.1	+10.9
	+15		+6.2	+6.2
	+17		-6.6	-5.6
	+19		+5.8	+4.2
	+21		+2.5	+2.0
	+23		+4.0	+4.5
+10	+0		+42.9	+39.0
	+2		+2.3	+3.4
	+4		-7.3	-9.1
	+6		+4.1	+5.9
	+8		+2.4	+3.4
	+10		-3.8	-4.6
	+12		-30.3	-24.0
	+14		-2.6	-3.0
	+16		+8.5	+8.8
	+18		-7.0	-6.9
+11	+1		+17.0	+18.4
	+3		+13.0	+14.6
	+5		-15.9	-15.8
	+7		+13.5	+13.7
	+9		-7.7	-7.0
	+11		-16.9	-17.3
	+13		-10.4	-9.9
	+15		-10.6	-10.1
	+17		+8.1	+7.5
	+19		-4.6	-4.5
+12	+0		-31.7	-32.6
	+2		-8.5	-8.2
	+4		+5.4	+6.8
	[illegible]		-6.4	-7.0
	+10		+5.3	+6.0
	+12		+24.2	+24.0
	+14		+8.5	+9.1
+13	+1		+3.7	+4.7
	+3		-10.4	-10.5
	+5		+6.2	+6.4
	+7		-5.6	-6.1
	+9		+6.4	+7.3
	+11		+5.0	+5.5
	+13		-8.8	-10.1
	+15		+7.1	+8.9
+14	+0		+3.4	+5.0
	+2		+11.2	+12.3
	+6		-3.7	-4.0
	+8		+7.2	+8.4
	+10		-9.8	-10.7
-15	+1	+1	-5.9	-7.6
	+3		+2.6	+4.2
	+7		+6.8	+9.3
	+9		+1.4	+2.0
-14	+2		+4.0	+5.3
	+4		-7.4	-8.5
	+6		-15.9	-18.0
	+8		-5.5	-6.7
	+10		-1.2	-2.0
	+12		+5.3	+5.6
-13	+1		-4.5	-5.4
	+3		+7.3	+8.7
	+5		-6.9	-9.0
	+7		-3.2	-5.0
	+9		+5.2	+6.6
	+11		-3.4	-4.2
	+13		-5.7	-5.6
	+17		+5.8	+6.7
-12	+2		-9.6	-11.4
	+4		+14.7	+15.2
	+6		+8.2	+9.4
	+8		+11.2	+11.6
	+10		-7.0	-9.1
	+16		-9.9	-12.5
	+18		-6.1	-6.2
-11	+1		+2.8	+3.6
	+3		-3.2	-3.6
	+5		+12.1	+13.9
	+7		+10.6	+10.6
	+9		-6.5	-7.7
	+11		+4.6	+5.7
	+17		-9.1	-9.9
	+19		-4.8	-5.8
	+21		+5.1	+5.0
-10	+2		+7.4	+9.0
	+4		-15.0	-15.1
	+6		-1.7	-3.6
	+8		-10.6	-11.5
	+10		+3.2	+3.6
	+14		-7.4	-8.6
	+16		+8.8	+8.5
	+18		+2.0	+2.6
	+20		+2.5	+3.0
	+22		-1.2	-1.5
	+24		+1.8	+1.6
-9	+1		-9.2	-8.8
	+3		+8.8	+10.8
	+5		-11.2	-15.0
	+7		-22.1	-19.0
	+9		+12.5	+11.6
	+11		-6.6	-6.3
	+13		+3.5	+3.6
	+19		+17.9	+14.0
	+21		-5.8	-5.6
	+25		+1.5	+2.0
-8	+6		+26.3	+20.8
	+8		+8.3	+9.6
	+10		-2.4	-2.6
	+12		+3.9	+3.6
	+16		+4.0	+4.9
	+18		-14.5	-12.8
	+20		-8.6	-8.0
-7	+1		+11.8	+11.9
	+3		-6.9	-9.4
	+5		-35.7	-29.5
	+7		-15.5	-12.0
	+9		-9.2	-9.4
	+11		+6.9	+7.3
	+13		-7.6	-8.2
	+17		+31.0	+25.4
	+21		+4.5	+5.5
	+25		+2.3	+2.4
-6	+2		-3.5	-3.1
	+4		+8.2	+9.0
	+10		+5.8	+6.1
	+12		-8.1	-7.2
	+14		+6.1	+7.3
	+20		+8.7	+8.7
	+22		-6.4	-5.7
	+24		+5.3	+4.7
	+26		-2.5	-2.0
-5	+1		-5.4	-6.4
	+5		+23.8	+19.7
	+7		+42.3	+31.8
	+13		+6.1	+5.6
	+15		-5.4	-2.6
	+17		-2.6	-3.6
	+25		-3.6	-3.1
	+27		+2.0	+1.5
-4	+2		+3.1	+3.7
	+4		-11.2	-11.6
	+6		+19.0	+16.2
	+8		-3.1	-3.1
	+10		-7.4	-8.7
	+12		+10.6	+10.3
	+14		-8.6	-10.2
	+16		+8.7	+9.0
	+18		-8.4	-8.2
	+20		-2.5	-3.5
	+22		+7.7	+6.4
	+24		-7.8	-6.0
	+26		+4.4	+4.2
-3	+1		-10.1	-13.9
	+3		+20.1	+19.4
	+5		-39.6	-27.6
	+9		+20.7	+19.7
	+11		-11.1	-13.6
	+13		+2.8	+3.5
	+15		-4.2	-3.1
	+17		+7.7	+8.2
	+21		-7.0	-7.0
	+25		+4.5	+4.4
-2	+4		+2.3	+4.0
	+8		+7.7	+9.9
	+10		-4.0	-5.4
	+12		+3.1	+3.3
	+14		-3.2	-3.5
	+16		-2.4	-2.0
	+18		-27.9	-21.4
	+20		-9.1	-8.8
-1	+3		-11.8	-16.0
	+5		+34.0	+29.0
	+7		+32.2	+27.1
	+9		-16.2	-17.0
	+11		+11.0	+13.6
	+17		-17.4	-15.1
	+19		-16.1	-14.6
	+21		+9.7	+9.2
	+23		-5.2	-4.4
	+25		-4.0	-3.7
+0	+4		+3.9	+6.5
	+10		+3.9	+5.3
	+12		-8.0	-10.5
	+14		+3.8	+4.3
	+18		+28.1	+22.3
	+20		+5.6	+5.0
	+22		-5.4	-5.8
	+24		+6.0	+5.0
+1	+1		-4.1	-4.7
	+3		+8.6	+11.0
	+5		-36.4	-30.0
	+7		-19.1	-19.6
	+9		+6.6	+9.0
	+11		-4.2	-6.5
	+15		-3.9	-3.6
	+17		+26.3	+21.7
	+19		+2.5	+3.6
	+23		+1.8	+3.1
	+25		+2.7	+2.6
+2	+2		+1.8	+1.4
	+4		-4.6	-5.1
	+6		-5.5	-15.3
	+8		+5.6	+6.0
	+10		-8.1	-10.2
	+12		+13.9	+14.8
	+14		-5.8	-6.2
	+16		+10.4	+11.6
	+20		-5.3	-5.0
	+22		+9.0	+7.9
	+24		-11.1	-9.2
	+28		-7.8	-6.5
+3	+1		-12.5	-10.7
	+3		+10.0	+11.6
	+5		-18.2	-19.8
	+7		-4.9	-5.3
	+9		+12.4	+12.1
	+11		-11.1	-12.2
	+13		+5.4	+6.2
	+17		+15.6	+15.1
	+19		-4.3	-3.6
	+21		-6.9	-5.8
	+25		+4.6	+4.2
	+27		-2.7	-1.6
+4	+2		+6.0	+7.8
	+4		-9.4	-10.5
	+8		-9.4	-10.4
	+10		+9.2	+10.8
	+12		-8.0	-9.4
	+16		+8.4	+9.0
	+18		+33.7	+24.9
	+20		+7.3	+8.2
	+22		-7.1	-6.7
	+24		+6.6	+6.5
+5	+1		+17.6	+17.8
	+3		-14.5	-15.9
	+7		+7.5	+8.9
	+9		-18.1	-16.3
	+11		+15.2	+15.1
	+13		-6.5	-6.3
	+15		+4.1	+3.6
	+19		-4.3	-4.8
	+21		+10.4	+8.6
	+25		-5.4	-5.1
+6	+2		-8.8	-10.6
	+4		+15.2	+15.2
	+6		+50.6	+44.5
	+8		+16.7	+15.9
	+10		-5.4	-6.2
	+14		+6.4	+7.3
	+16		-9.2	-9.4
	+18		-28.6	-22.5
	+20		-11.5	-10.0
+7	+1		-9.8	-11.6
	+3		+13.9	+15.4
	+5		-18.4	-18.5
	+7		-32.8	-30.0
	+9		+14.0	+14.2
	+11		-6.4	-7.3
	+15		-4.5	-5.0
	+19		+24.9	+21.4
	+21		-4.9	-5.0
+8	+2		+7.2	+9.6
	+4		-17.8	-17.5
	+6		+4.0	+7.3
	+8		-12.0	-12.2
	+10		-4.9	-6.3
	+12		+10.1	+10.2
	+14		-12.2	-10.3
	+16		+10.6	+9.0
	+20		+5.2	+2.4
	+22		+7.4	+7.0
+9	+1		-12.9	-14.2
	+3		+5.1	+6.4
	+5		+23.2	+25.7
	+7		+29.4	+27.0
	+9		+5.7	+3.6
	+11		-8.6	-9.2
	+13		+9.5	+8.8
	+15		-4.0	-5.0
	+17		-10.4	-11.1
	+19		-21.2	-21.9
+10	+2		-5.9	-5.0
	+4		+11.6	+11.8
	+6		-40.9	-34.1
	+8		+6.6	+7.6
	+14		+4.5	+4.0
	+16		-5.4	-6.1
	+18		+19.3	+13.0
	+20		+2.1	+2.1
+11	+1		+11.5	+12.2
	+3		-6.1	-8.2
	+5		-24.6	-20.0
	+7		-11.0	-11.5
	+9		-8.5	-9.6
	+11		+7.5	+8.5
	+15		-7.5	-7.2
	+17		+25.4	+21.1
+12	+6		+1.5	+3.4
	+8		+3.3	+3.3
+13	+1		-3.5	-4.5
	+3		+2.5	+3.4
	+5		-3.5	-5.2
	+7		-8.0	-9.2
	+9		+5.4	+7.4
	+11		-2.7	-3.8
	+15		+1.6	+2.7
+14	+2		+5.2	+7.0
	+4		-9.7	-12.4
	+6		+0.9	+1.9
	+8		-6.0	-7.1
-16	+0	+2	+0.5	+1.7
	+2		-5.8	-9.0
	+4		+3.0	+4.5
-15	+1		-4.3	-5.5
	+3		+4.3	+5.1
	+5		-3.2	-3.8
	+7		+2.6	+3.2
-14	0		+3.2	+4.6
	+2		-1.3	-3.2
	+4		+1.7	+2.2
	+8		-1.6	-2.0
	+10		+4.6	+6.7
	+12		-2.7	-4.4
-13	+1		-19.7	-21.1
	+3		-5.8	-5.3
	+5		+7.3	+8.2
	+7		-7.8	-8.6
	+9		+2.7	+2.3
	+11		+24.8	+33.4
	+13		+6.8	+10.9
	+15		+4.7	+5.5
	+17		-2.4	-2.26
-12	+0		-40.2	-29.6
	+2		+12.2	+12.7
	+8		+5.5	+3.8
	+10		-10.6	-11.0
	+12		+27.8	+26.9
	+14		-3.1	-3.8
	+16		-2.7	-1.8
	+18		+2.4	+2.0
-11	+1		+26.3	+22.7
	+5		-6.0	-6.0
	+7		+7.3	+7.8
	+9		-3.0	-2.9
	+11		-12.7	-14.4
	+13		-27.2	-29.6
	+17		+2.6	+5.8
-10	+0		-4.8	-6.1
	+2		-14.3	-13.0
	+6		+4.8	+5.3
	+8		-8.3	-6.8
	+10		+13.1	+13.7
	+12		+4.6	+6.5
	+14		+7.3	+8.6
	+16		+6.7	+8.3
-9	+1		-23.2	-18.4
	+3		+18.1	+15.6
	+5		-11.7	-14.5
	+7		+8.9	+11.4
	+9		-10.6	-10.6
	+11		+3.4	+3.0
	+13		+27.5	+26.3
	+15		-11.5	-12.0
	+17		+3.5	+2.6
-8	+2		+11.6	+10.4
	+4		-6.0	-7.7
	+8		+3.6	+4.3
	+10		-7.0	-9.2
	+14		-12.8	-12.4
	+16		+4.3	+2.9
	+22		+5.1	+5.5
	+24		+18.4	+22.4
-7	+3		-15.7	-15.4
	+5		+14.5	+14.9
	+7		-10.1	-10.0
	+9		+8.2	+7.8
	+11		+2.6	+3.0
	+15		+13.0	+10.5
	+17		-8.3	-7.7
-6	+2		-4.0	-4.5
	+4		+6.7	+7.0
	+6		-6.1	-7.2
	+10		+3.4	+4.1
	+14		+6.6	+7.5
	+16		-9.5	-9.1
	+18		+9.6	+8.2
	+22		-4.2	-4.6
	+24		-17.5	-15.0
	+26		-5.3	-6.4
-5	+1		-5.7	-6.3
	+3		+6.0	+5.4
	+5		-6.1	-5.7
	+7		+4.5	+4.6
	+9		-3.0	-3.4
	+11		+13.6	+13.6
	+13		-8.3	-9.9
	+15		-4.9	-4.3
	+17		+4.1	+3.7
	+23		-11.1	-10.3
	+25		+8.3	+6.8
	+27		+2.8	+3.3
-4	+0		-9.5	-17.1
	+4		-2.9	-3.2
	+6		+8.1	+10.1
	+8		-5.2	-6.4
	+10		+9.5	+10.4
	+12		+0.9	+7.1
	+14		-5.3	-4.3
	+16		+8.9	+9.2
	+18		-12.6	-11.7
	+22		-9.7	-8.5
	+26		+2.4	+2.2
-3	+1		-35.1	-20.7
	+3		+13.7	+13.7
	+5		-10.9	-11.3
	+7		+5.8	+6.0
	+9		-8.2	-9.1
	+11		+33.7	+30.6
	+13		+16.5	+15.3
	+15		-8.9	-9.1
	+17		+6.1	+6.8
	+23		-17.6	-12.1
	+25		-2.0	-2.0
	+27		+3.6	+3.0
-2	+4		+1.3	+2.0
	+6		-3.1	-3.9
	+10		+1.0	+2.7
	+12		+37.1	+30.9
	+14		+3.6	+3.0
	+18		+4.7	+4.3
	+22		-2.6	-2.6
	+24		-12.6	-10.5
	+26		-5.9	-3.0
	+28		+2.7	+2.3
-1	+1		+36.3	+29.2
	+3		-16.0	-19.6
	+5		+14.0	+17.0
	+7		-5.8	-7.0
	+9		+5.7	+6.3
	+11		-23.5	-26.0
	+13		-27.9	-25.7
	+15		+13.7	+13.2
	+17		-9.3	-9.2
	+23		+10.4	+9.9
	+25		+10.0	+7.8
	+27		-6.2	-5.5
+0	+2		+7.9	+9.6
	+4		-4.7	-6.5
	+8		+3.5	+3.6
	+10		-6.0	-7.1
	+12		-28.5	-30.5
	+14		-7.9	-9.0
	+22		+5.4	+4.5
	+24		+11.9	+8.9
	+26		+5.7	+4.5
+1	+3		+17.7	+17.8
	+5		-11.1	-12.2
	+7		+6.6	+8.2
	+9		-9.0	-10.3
	+11		+20.6	+22.2
	+13		+48.7	+38.3
	+15		-10.0	-10.5
	+17		+3.0	+4.3
	+19		+2.7	+3.6
	+25		-25.3	-22.1
+2	+0		+36.3	+30.6
	+2		-7.9	-8.6
	+4		-0.9	-1.7
	+6		+8.5	+10.1
	+8		-10.8	-11.3
	+10		+11.6	+11.9
	+12		-23.6	-22.0
	+16		+9.0	+9.2
	+18		-12.4	-9.9
	+20		+8.7	+6.9
	+22		-4.6	-3.6
	+24		+7.8	+6.5
	+26		+4.1	+3.4
+3	+1		+28.7	+24.4
	+3		+5.0	+5.7
	+5		-11.0	-10.8
	+7		+11.3	+10.5
	+9		-4.4	-4.9
	+11		-11.1	-11.8
	+13		-30.0	-24.0
	+15		-3.0	-3.0
	+17		+5.1	+4.3
	+19		-5.8	-5.7
	+21		+1.8	+2.6
	+25		+17.5	+15.7
+4	+2		+2.6	+2.7
	+6		-4.6	-4.9
	+8		+4.4	+4.1
	+14		+6.8	+7.5
	+16		-6.3	-7.4
	+18		+6.4	+5.7
	+20		-3.1	-2.6
	+22		-4.4	-4.5
	+24		-25.5	-23.9
	+26		-9.4	-9.1
+5	+1		-16.7	-17.6
	+3		-11.8	-14.0
	+5		+14.5	+13.9
	+7		-13.1	-12.8
	+9		+4.6	+3.7
	+11		+31.2	+31.3
	+15		+9.5	+7.4
	+17		-5.5	-4.9
	+19		+4.6	+5.7
	+23		-22.4	-21.0
	+25		+7.6	+7.9
+6	+0		+19.2	+17.4
	+2		+9.6	+11.1
	+8		+5.4	+3.0
	+12		-14.7	-15.5
	+14		-10.5	-8.9
	+20		-3.0	-2.7
	+22		-2.1	-2.5
	+24		+7.7	+8.4
+7	+1		-21.8	-24.1
	+3		+11.4	+12.2
	+5		-8.0	-8.1
	+7		+5.6	+5.3
	+9		-2.7	-3.0
	+11		+7.8	+9.9
	+13		+23.5	+20.9
	+15		-8.8	-7.7
	+17		+3.3	+3.5
	+19		-0.1	-2.2
	+21		-4.1	-2.4
+8	+0		+4.0	+5.3
	+2		-16.5	-16.2
	+4		+6.5	+7.4
	+8		-10.6	-10.4
	+10		+15.7	+13.7
	+14		+6.3	+6.4
	+20		+7.3	+7.8
	+22		-6.1	-6.6
+9	+1		+12.3	+13.4
	+3		-2.9	-4.2
	+11		-12.6	-14.9
	+13		-6.7	-6.5
	+15		+3.9	+4.9
	+17		-2.6	-3.2
	+19		-2.0	-2.0
+10	+0		+6.1	+9.3
	+2		+13.6	+14.0
	+4		-8.2	-8.2
	+8		+8.5	+8.5
	+10		-13.0	-15.6
	+12		-5.7	-6.6
	+14		-6.5	-7.2
	+16		+3.7	+4.0
+11	+1		-4.8	-6.1
	+3		+3.6	+4.2
	+5		-1.7	-1.7
	+9		-5.5	-5.9
	+11		+7.2	+7.8
	+15		-1.0	-0.9
+12	+0		-14.8	-16.4
	+2		-4.7	-5.4
	+6		+3.0	+5.6
	+8		-4.4	-5.5
	+10		+3.1	+5.7
	+12		+11.1	+12.1
+13	+1		+3.1	+7.5
	+3		+2.8	+3.6
	+5		-7.3	-8.6
	+7		+6.3	+8.9
-16	+2	+3	-2.8	-2.7
	+4		+8.2	+8.7
-15	+1		+7.3	+7.4
	+3		-9.1	-9.5
	+5		-3.4	-3.4
	+7		+4.4	+3.9
	+9		-9.6	-8.9
	+11		+4.5	[illegible]
-14	+2		+3.9	+4.4
	+4		-2.4	-3.0
	+6		-27.7	-32.2

Table 3 (*cont.*)

h	k	l	F_c	F_o
	+8		-5.2	-5.8
	+10		+3.6	+3.2
	+14		-2.6	-3.1
-13	+1		-12.3	-12.7
	+3		+8.5	+8.8
	+5		+16.3	+17.6
	+7		+14.1	+15.5
	+9		+9.7	+9.7
	+11		-9.6	-7.9
	+13		+7.2	+6.1
	+15		-3.9	-3.0
	+17		-11.1	-9.6
-12	+2		-2.8	-4.3
	+6		+30.6	+30.1
	+10		-7.6	-8.1
	+12		+6.6	+6.9
	+16		-1.1	-2.3
	+18		-15.3	-15.5
-11	+1		+5.1	+4.8
	+7		-11.5	-11.8
	+9		-2.0	-3.1
	+11		+4.5	+4.9
	+13		-3.6	-4.4
	+17		-4.1	-4.9
	+19		+11.5	+12.3
-10	+2		-3.7	-3.5
	+4		+9.1	+9.8
	+6		+16.3	+17.5
	+8		+4.2	+4.7
	+10		+3.1	+2.3
	+12		-7.4	-7.4
	+14		+6.0	+6.7
	+16		-9.4	-8.6
	+18		-9.1	-8.6
	+20		-1.5	-1.5
	+22		-5.2	-4.5
-9	+1		+6.6	+7.9
	+3		-10.3	-15.0
	+5		+24.7	+23.6
	+7		+18.8	+18.8
	+9		-9.1	-10.9
	+11		+6.2	+8.2
	+15		+5.6	+5.7
	+17		-16.8	-13.3
	+19		-6.1	-5.9
	+21		+4.3	+3.9
-8	+2		+6.5	+7.8
	+4		-11.6	-12.6
	+6		+6.0	+7.9
	+8		-8.8	-11.2
	+10		+4.1	+6.0
	+14		-4.1	-3.2
	+16		+6.5	+6.6
	+18		-1.4	-2.6
	+20		+2.1	+2.1
-7	+1		-5.2	-5.9
	+3		+6.5	+7.6
	+5		-26.7	-24.9
	+7		-27.3	-23.5
	+9		+11.1	+12.7
	+11		-7.7	-9.3
	+17		+16.1	+14.6
	+19		+14.1	+11.6
	+21		-7.5	-6.0
	+23		+4.4	+3.8
	+25		+3.3	+2.2
-6	+2		-1.9	-2.3
	+4		+7.1	+8.1
	+6		-4.7	-10.7
	+8		+5.2	+6.9
	+14		+3.1	+3.4
	+16		-2.4	-2.3
	+18		+3.1	+2.1
	+22		+1.3	+1.4
	+24		-3.0	-2.1
-5	+1		+8.8	+9.9
	+3		-11.0	-12.9
	+5		+23.9	+22.8
	+7		+38.1	+30.9
	+9		-12.8	-14.2
	+11		+6.2	+7.1
	+17		-3.0	-3.2
	+19		-27.8	-16.9
	+21		+4.5	+3.5
	+25		-2.4	-1.6
-4	+2		-3.3	-3.9
	+4		+2.1	+2.9
	+6		-43.6	-34.8
	+8		-7.3	-10.5
	+10		+5.1	+6.9
	+12		-9.9	-12.2
	+14		+6.0	+7.0
	+16		-4.5	-4.8
	+18		+22.2	+18.4
	+20		+9.4	+8.8
	+22		-5.7	-4.0
	+24		+7.2	+5.8
	+26		-2.1	-1.5
-3	+1		+2.9	+3.3
	+5		+11.0	+15.9
	+7		-4.3	-8.3
	+11		+3.0	+4.3
	+17		-14.7	-13.7
	+19		+10.4	+9.6
-2	+2		+0.6	+5.0
	+8		+6.1	+9.1
	+10		-7.0	-10.2
	+12		+8.5	+11.7
	+14		-3.8	-3.5
	+16		-4.7	-6.0
	+20		-11.9	-11.1
	+22		+6.4	+5.4
	+24		-5.6	-4.1
	+26		+1.5	+1.2
-1	+1		-10.1	-10.7
	+3		+5.7	+8.5
	+5		+1.6	+5.0
	+7		-22.7	-20.0
	+9		+9.6	+12.9
	+11		-7.7	-10.4
	+13		+3.3	+3.5
	+17		-15.1	-14.9
	+19		+23.8	+19.3
	+21		-4.1	-3.7
+0	+2		+4.2	+6.0
	+4		-4.9	-7.7
	+6		-20.2	-20.7
	+8		-10.3	-14.4
	+10		+5.7	+7.7
	+12		-4.1	-7.1
	+18		+11.5	+10.0
	+20		+7.0	+6.9
	+22		-3.5	-3.3
	+24		+2.9	+2.5
+1	+1		+10.9	+13.0
	+3		-13.0	-16.0
	+5		+33.1	+30.7
	+7		+47.5	+39.7
	+9		-15.4	-17.2
	+11		+8.3	+10.6
	+17		-7.9	-9.2
	+19		-32.6	-20.6
	+21		+5.8	+4.9
	+25		-3.0	-1.9
+2	+2		-9.1	-11.1
	+4		+15.4	+19.1
	+6		-25.1	-31.6
	+8		+8.1	+10.4
	+12		-3.6	-3.5
	+14		+10.6	+11.4
	+16		-8.2	-8.7
	+18		+12.2	+10.3
	+26		-5.3	-3.9
+3	+3		+3.5	+4.8
	+5		-29.3	-30.3
	+7		-25.3	-25.6
	+9		+6.1	+7.9
	+11		-2.6	-4.9
	+13		-4.5	-4.9
	+17		+20.3	+16.4
	+19		+10.9	+9.5
	+21		-4.8	-3.7
	+23		+2.8	+2.0
	+25		+3.6	+2.7
+4	+2		+8.2	+9.2
	+4		-14.0	-16.8
	+6		+15.7	+17.6
	+8		-8.3	-10.0
	+10		+3.0	+3.5
	+14		-6.5	-5.6
	+16		+9.2	+8.9
	+18		-5.8	-5.9
	+24		-2.1	-1.4
+5	+3		-3.9	-5.0
	+5		+21.9	+21.5
	+7		+12.2	+13.5
	+13		+2.7	+2.2
	+15		+5.0	+3.6
	+17		-17.5	-14.1
	+19		-1.3	-2.6
+6	+4		+3.9	+4.3
	+6		+10.0	+12.4
	+10		+4.3	+5.6
	+12		-7.7	-8.7
	+14		+4.2	+3.9
	+16		-6.0	-5.5
	+18		-5.3	-3.5
	+22		-5.4	-5.0
+7	+1		+11.6	+10.1
	+3		-6.0	-5.8
	+7		-6.1	-5.5
	+9		-8.5	-8.2
	+11		+9.7	+8.6
	+13		-6.4	-5.9
	+17		-3.4	-3.3
	+19		+7.5	+6.9
	+21		+5.5	+5.1
+8	+2		-5.8	-5.5
	+4		+6.7	+6.9
	+6		+32.4	+27.8
	+8		+4.7	+4.9
	+10		-7.0	-7.0
	+12		+4.2	+4.1
	+14		+2.7	+2.0
	+16		-6.9	-5.4
	+18		-17.2	-14.0
+9	+1		-13.5	-12.1
	+3		+9.0	+9.1
	+5		+14.0	+14.0
	+7		+10.1	+9.4
	+9		+10.4	+10.6
	+11		-10.7	-9.3
	+13		+7.4	+5.9
	+15		-3.8	-2.9
	+17		-10.9	-9.6
+10	+2		+6.5	+6.0
	+4		-8.5	-7.8
	+6		-22.5	-18.9
	+8		-9.3	-8.7
	+10		+2.6	+2.1
	+12		+2.9	+2.5
	+14		-6.5	-5.9
+11	+1		+3.3	+3.1
	+3		-5.7	-6.3
	+5		+1.2	+1.6
	+7		+7.5	+8.3
	+9		-5.7	-6.5
	+11		+1.6	+1.4
+12	+2		-4.1	-4.7
	+4		+10.1	+9.5
-16	+0	+4	+15.0	+17.6
	+2		+5.4	+6.1
-15	+1		+3.2	+4.3
	+3		-7.0	-6.9
	+5		+5.5	+5.2
	+7		-4.3	-3.5
	+9		+6.1	+5.3
-14	+0		-8.2	-9.9
	+2		-8.9	-9.8
	+4		+6.7	+7.6
	+8		-5.5	-5.6
	+10		+8.1	+7.8
	+12		+6.8	+6.2
-13	+1		-7.8	-9.7
	+3		+4.5	+5.3
	+5		-4.1	-4.4
	+7		+1.5	+1.7
	+11		+7.1	+7.2
	+13		+5.2	+5.6
	+15		-5.4	-5.0
-12	+2		+7.0	+7.4
	+4		-2.5	-3.0
	+6		+1.2	+2.0
	+8		+3.2	+2.7
	+10		-6.0	-6.2
	+12		-1.6	-2.2
	+14		-3.5	-3.9
	+16		+1.9	+2.2
	+18		-2.6	-3.1
-11	+1		+9.5	+9.8
	+3		-7.2	-6.5
	+5		+4.7	+4.5
	+7		-3.7	-3.1
	+13		-13.3	-10.9
	+15		+5.3	+4.3
-10	+0		-22.3	-17.6
	+10		-4.4	-4.4
	+12		+16.3	+15.2
	+14		+5.6	+5.3
	+18		+3.8	+3.8
-9	+1		+21.7	+18.8
	+3		+3.2	+3.4
	+5		-2.7	-3.4
	+7		+4.2	+4.8
	+9		-2.2	-2.3
	+11		-26.4	-20.5
	+13		-7.3	-7.4
	+15		-2.3	-2.7
	+19		-2.9	-2.8
	+21		+1.4	+1.4
-8	+2		-5.5	-5.7
	+6		+3.4	+3.3
	+8		-3.7	-3.4
	+10		+4.2	+4.8
	+14		-2.6	-3.1
	+16		+5.3	+5.6
	+18		-4.8	-4.9
	+20		+3.0	+2.8
	+22		+2.6	+2.4
-7	+1		-27.0	-20.5
	+3		+4.2	+4.5
	+11		+6.5	+7.5
	+13		+32.7	+22.3
	+15		-4.0	-4.4
	+17		+1.1	+2.0
	+19		+3.1	+3.0
	+21		-3.0	-2.0
	+23		+3.0	+2.3
-6	+2		+3.7	+4.2
	+4		+3.7	+5.3
	+6		-3.0	-4.5
	+10		-5.7	-8.2
	+16		-5.7	-6.2
	+18		+4.6	+4.8
	+20		-2.5	-2.4
	+22		+3.5	+3.2
-5	+1		+37.0	+24.2
	+3		-16.6	-15.4
	+5		+10.8	+11.6
	+7		-7.1	-8.4
	+9		+8.8	+10.4
	+11		-14.0	-13.9
	+13		-37.4	-25.4
	+15		+10.4	+10.3
	+17		-3.5	-3.1
-4	+6		-3.3	-5.2
	+8		+5.1	+6.3
	+10		-1.9	-2.3
	+14		+6.9	+8.2
	+16		-3.7	-4.6
	+18		+4.4	+4.1
	+20		-4.4	-3.6
	+24		-15.0	-11.9
-3	+1		-19.9	-17.6
	+3		+7.9	+10.3
	+5		-3.6	-5.5
	+9		-3.7	-5.6
	+11		+17.4	+17.9
	+13		+11.7	+11.7
	+15		-7.4	-8.1
	+17		+3.9	+3.8
	+19		+2.8	+2.8
	+23		-10.2	-9.2
	+25		-2.8	-2.9
-2	+2		-2.3	-3.2
	+6		+4.2	+6.5
	+8		-3.8	-5.4
	+10		+3.7	+4.6
	+12		-32.5	-24.1
	+14		-3.3	-4.8
	+16		+4.6	+6.1
	+18		-6.5	-6.7
	+24		+11.3	+9.1
-1	+1		+13.4	+12.9
	+3		-0.8	-2.7
	+5		-1.9	-4.1
	+7		+2.7	+3.1
	+9		+2.7	+3.3
	+11		-20.3	-19.2
	+23		+13.6	+10.3
	+25		-4.2	-4.1
+0	+0		+2.2	+9.0
	+2		-3.2	-4.8
	+4		+3.2	+5.0
	+6		-5.6	-8.2
	+10		-3.9	-4.7
	+12		-2.0	-4.8
	+14		+5.3	+6.7
	+16		-6.6	-7.8
	+18		+9.3	+9.1
	+22		+6.3	+4.8
+1	+1		+13.2	+13.7
	+3		-11.8	-11.8
	+5		+12.5	+13.0
	+7		-8.9	-9.2
	+9		+5.2	+6.7
	+11		-15.6	-15.1
	+13		-3.2	-3.3
	+15		+9.4	+8.4
	+17		-7.4	-5.9
	+23		+9.8	+8.9
+2	+0		+48.4	+38.5
	+2		+10.1	+11.0
	+4		-6.6	-8.6
	+6		+2.3	+2.3
	+8		+5.3	+5.8
	+10		-10.6	-11.2
	+12		-34.7	-28.3
	+14		-7.0	-8.4
	+16		+5.2	+5.9
	+18		-4.0	-4.6
	+20		-2.7	-3.1
	+22		+7.7	+7.3
+3	+1		-5.8	-6.6
	+3		+11.9	+13.7
	+5		-13.0	-14.2
	+7		+8.8	+9.5
	+9		-5.9	-6.7
	+13		+6.2	+7.1
	+15		-10.1	-9.3
	+17		+7.6	+6.9
+4	+0		-33.2	-30.3
	+2		-13.9	-15.1
	+4		+6.2	+6.7
	+8		-8.4	-3.8
	+10		+11.1	+10.6
	+12		+25.9	+22.8
	+14		+10.7	+10.0
	+16		-3.6	-3.1
	+20		+5.3	+4.8
	+22		-6.3	-5.5
+5	+1		+20.4	+22.8
	+3		-7.5	-10.1
	+5		+3.3	+4.7
	+7		-2.1	-3.3
	+9		+4.8	+5.4
	+11		-6.4	-7.3
	+13		-22.5	-21.8
	+15		+4.0	+4.9
	+19		-1.5	-1.6
+6	+0		-10.6	-14.8
	+2		+10.8	+11.2
	+6		-3.2	-6.3
	+8		+8.9	+9.0
	+10		-10.3	-9.4
	+12		+6.6	+9.0
	+14		-4.2	-4.0
	+16		-5.9	-5.1
	+18		+8.5	+8.2
+7	+1		-20.4	-21.2
	+3		-6.7	-7.3
	+5		+12.5	+13.3
	+7		-12.1	-11.
	+9		+5.0	+4.5
	+11		+14.2	+14.8
	+13		+18.0	+17.9
	+15		+4.7	+4.2
	+17		-6.0	-5.4
+8	+0		+34.1	+30.9
	+2		-5.0	-5.3
	+8		-2.1	-2.3
	+10		+4.6	+5.1
	+12		-24.3	-23.4
+9	+1		+11.7	+13.7
	+3		+6.9	+8.1
	+5		-8.7	-9.3
	+7		+8.2	+9.4
	+9		-2.4	-2.4
	+11		-18.9	-20.5
+10	+0		+1.3	+1.2
	+2		-4.3	-6.5
	+8		-2.7	-3.8
+11	+1		+6.3	+8.4
-15	+1	+5	+3.5	+4.0
	+3		-2.0	-2.2
	+5		-16.3	-16.5
-14	+4		+9.0	+8.5
	+6		-23.0	-21.9
	+8		+6.0	+6.1
-13	+1		-2.4	-1.9
	+5		+11.7	+12.2
	+7		+19.6	+19.7
	+9		-2.6	-1.5
	+13		+3.1	+3.5
-12	+4		-7.5	-6.6
	+6		-8.3	-9.0
	+8		-6.3	-6.0
	+10		-3.5	-3.3
	+12		+3.6	+3.1
	+14		-4.0	-4.0
	+16		+4.8	+5.0
-11	+1		-8.1	-7.3
	+3		+10.9	+9.7
	+5		-4.9	-4.5
	+7		-14.9	-10.7
	+9		+11.2	+8.8
	+11		-5.3	-4.7
	+13		+3.3	+3.0
	+15		-4.2	-4.0
	+17		-4.5	-4.2
-10	+8		+6.3	+6.4
	+10		-4.1	-4.4
	+12		+3.1	+2.0
	+16		-1.9	-1.5
	+18		-23.3	-19.7
-9	+1		+6.0	+3.9
	+3		-6.8	-8.4
	+5		-3.3	-4.8
	+7		-4.5	-5.9
	+9		-8.1	-8.1
	+11		+5.6	+5.5
	+15		+3.2	+2.7
	+19		+4.3	+4.8
-8	+10		+6.2	+6.8
	+12		-6.6	-6.5
	+18		+18.9	+13.9
	+20		+3.6	+4.5
-7	+5		-3.5	-3.3
	+7		+7.5	+7.6
	+17		+9.0	+8.9
	+19		-10.1	-9.7
-6	+4		-2.9	-2.2
	+6		-9.3	-11.3
	+8		+1.9	+2.4
	+10		-4.0	-3.4
	+12		+6.7	+6.8
	+16		+6.5	+5.9
	+18		+4.7	+4.5
	+22		+4.9	+4.8
-5	+1		-8.4	-7.6
	+3		+9.3	+8.6
	+5		-24.6	-18.2
	+7		-18.4	-15.2
	+9		+10.1	+10.7
	+11		-8.0	-7.7
	+15		-2.9	-2.2
	+17		+17.4	+13.6
	+19		+5.9	+5.5
	+21		-5.5	-5.4
-4	+4		-2.9	-2.8
	+6		-23.9	-19.5
	+16		+3.6	+3.1
	+18		+12.3	+11.3
-3	+1		+6.7	+7.9
	+3		-6.3	-7.4
	+5		+18.9	+16.8
	+7		+20.5	+18.3
	+9		-9.6	-9.5
	[illegible]		+8.0	+8.4
	+17		-10.5	-10.2
	+19		-11.3	-9.9
	+21		+6.3	+4.9
	+23		-3.8	-4.1
-2	+2		-5.0	-4.5
	+4		+6.4	+6.4
	+6		+17.7	+17.3
	+8		+5.6	+5.7
	+12		-3.4	-3.4
	+14		+3.9	+3.3
	+16		-5.3	-4.4
	+18		-10.9	-9.5
	+20		-3.8	-3.9
-1	+1		-2.8	-4.0
	+3		+5.9	+6.6
	+5		-19.5	-17.0
	+7		-29.9	-21.0
	+9		+5.5	+6.8
	+11		-1.5	-3.4
	+19		+22.5	+17.6
+0	+2		+4.5	+5.3
	+4		-6.0	-7.8
	+6		+26.6	+21.4
	+10		-4.0	-4.2
	+12		+8.9	+10.1
	+14		-7.9	-9.1
	+16		+4.3	+4.2
	+18		-13.5	-13.3
	+20		-3.7	-3.5
	+22		+5.7	+6.7
+1	+1		-12.3	-13.4
	+3		+5.5	+6.6
	+5		+10.7	+10.7
	+7		+15.5	+14.9
	+9		+6.0	+6.9
	+11		-9.2	-8.2
	+13		+7.7	+7.9
	+17		-3.7	-4.5
	+19		-12.5	-12.6
	+21		-3.5	-3.3
+2	+8		-3.5	-4.8
	+10		+2.9	+4.1
	+12		-2.8	-3.9
	+16		+2.8	+2.6
	+18		+31.1	+23.5
	+20		+7.0	+7.5
+3	+1		+11.1	+13.3
	+3		-5.7	-7.7
	+5		-14.1	-16.0
	+7		+2.1	+4.2
	+9		-8.5	-9.9
	+11		+7.9	+8.2
	+13		-6.0	-6.4
	+17		+18.6	+18.2
	+19		-9.0	-9.6
+4	+2		-4.4	-5.8
	+4		+7.0	+8.8
	+6		+4.9	+5.1
	+8		+8.9	+10.5
	+14		+4.9	+4.9
	+18		-4.5	-5.6
+5	+1		-2.6	-3.2
	+3		+3.7	+5.0
	+5		-12.8	-15.8
	+7		-18.3	-18.4
	+9		+5.7	+6.3
	+11		-2.2	-2.6
	+17		+3.8	+5.5
+6	+2		+7.1	+8.7
	+4		-13.1	-15.3
	+6		+7.5	+8.6
	+8		-8.2	-9.9
	+10		+2.3	+3.3
	+14		-7.5	-8.6
+7	+1		-2.4	-2.5
	+5		+9.8	+13.0
	+7		+5.6	+7.4
+8	+2		-5.7	-7.5
	+4		+8.9	+12.0
	+6		+3.9	+7.3
	+8		+6.1	+8.2

extinction

h	k	l	F_c	F_o
+0	+12	+0	-107.9	-61.6
	+24		+36.2	+24.6
+2	+0		-45.2	-22.2
+3	+1		+53.6	+30.8
	+13		-48.8	-34.1
-6	+6	+1	-100.2	-49.9
	+18		+47.3	+29.6
-5	+19		-31.5	-20.7
-3	+7		-62.5	-38.4
	+19		+40.8	+27.3
-2	+6		+67.9	+40.5
+0			-61.9	-38.2
+4			-69.8	-47.6
-8	+0	+2	+60.8	+29.2
	+12		-44.5	-34.1
-6	+0		-72.6	-27.7
	+12		+49.2	+34.5
-2	+0		-56.1	-23.9
+0			+53.0	+29.0
+1	+1		-50.7	-32.1
+4	+0		-105.3	-56.9
	+12		+71.5	+49.3
-2	+6	+3	+113.3	+61.7
	+18		-54.5	-31.0
-6	+0	+4	+72.6	+47.6
	+12		-51.1	-28.6
-4	+0		-54.7	-29.0
	+12		+39.3	+27.3
-2	+0		+47.9	+28.3
-10	+6	+5	+42.2	+28.1
-8			-37.2	-23.5
+2			-59.2	-32.1

indeed, in close-packed layers parallel to (100) at $x=\frac{1}{8}$, $\frac{3}{8}$, $\frac{5}{8}$ and $\frac{7}{8}$, but the individual atoms have x parameters deviating significantly from these values. Consequently the layers are deformed like corrugated sheet-iron and the sulphur atoms are on curved lines.

The cause of deformation must be sought in the distribution of cations over the available tetrahedral and octahedral sites. Starting with the latter it can be seen that the magnesium atoms at $z=0{\cdot}25$ and at $z=0{\cdot}75$ are exactly on straight lines (twofold axes). Each Mg is coordinated octahedrally by six S in such a way that three octahedra share edges, *i.e.* *AB* or *CD* perpendicular to [010].

These octahedra at $z=0{\cdot}25$ and at $z=0{\cdot}75$ form infinite slabs running parallel to [001] by sharing edges of the kind *AD* and *BC* (Fig. 1). *AD* and *BC* are related by the symmetry operation of the glide plane. The same kind of infinite zigzag ribbons of oxygen octahedra have been found in $Na_3Fe_5O_9$ (Romers, Rooymans & de Graaff, 1967). The slabs of octahedra in $MgGa_2S_4$ are positioned at $x=0$, $y=0$ and at $x=\frac{1}{2}$, $y=\frac{1}{2}$ in accordance with the centring of the (001) plane. The dimensions of the separate octahedra are shown in Fig. 2. The Mg–S distances are within the range of 2·52–2·71 Å, indicating that the octahedra are slightly distorted. The average value (2·59 Å) agrees with the value observed in the rocksalt-type structure of MgS (2·60 Å).

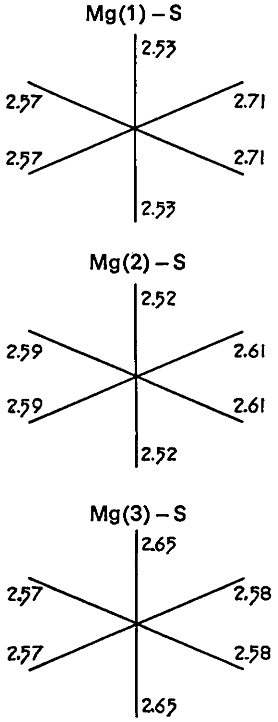

Fig. 2. The coordination of Mg by S.

The gallium ions are located in a number of tetrahedral interstices. Their arrangement is more distorted than the alignment of the magnesium ions (Fig. 1). The Ga–S distance and S–Ga–S angles are shown in Fig. 3. The mean value of Ga–S distances (2·29 Å) agrees excellently with the mean Ga–S distances (2·28Å) found by Goodyear & Steigmann (1963) in α-Ga_2S_3.

The coordination of the anions, the four types being indicated in Fig. 1, is quite irregular and varies between

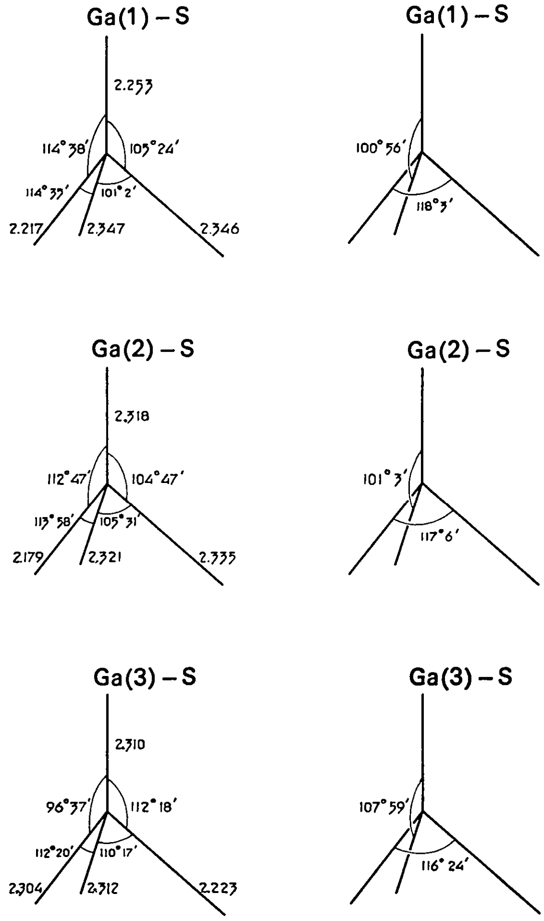

Fig. 3. The coordination of Ga by S.

Table 4. *Positional parameters (fractions of cell edges), their standard deviations (in* 10^4 Å) *and isotropic temperature parameters and corresponding standard deviations* (Å^2)

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$	B	$\sigma(B)$
S(1)	0·1091	45	−0·1319	44	−0·0158	58	1·59	0·65
S(2)	0·1085	49	0·0439	43	−0·0162	61	1·71	0·71
S(3)	0·1283	45	0·2162	41	−0·0021	55	1·50	0·64
S(4)	0·3742	45	0·1179	41	0·0002	56	1·51	0·64
S(5)	0·3665	45	−0·0409	40	0·0009	58	1·47	0·67
S(6)	0·3844	50	−0·1981	46	0·1223	60	1·86	0·69
Ga(1)	0·2921	23	−0·1249	21	0·1180	28	2·07	0·35
Ga(2)	0·2887	24	0·0402	21	0·1188	29	2·10	0·37
Ga(3)	0·3163	22	0·2003	22	0·1476	26	1·95	0·34
Mg(1)	0	—	−0·1298	93	0·2500	—	2·05	1·52
Mg(2)	0	—	0·0436	88	0·2500	—	1·99	1·57
Mg(3)	0	—	0·2180	85	0·2500	—	1·72	1·40

trigonal configurations SGa_3 and $SMgGa_2$ and irregular tetrahedral arrangements SMg_2Ga_2 and SMg_3Ga.

Comparison of Fig. 1 of this paper with Fig. 2 of Goodyear & Steigmann's publication on the structure of α-Ga_2S_3 demonstrates that the stacking of sulphur atoms is similar in the two compounds. In α-Ga_2S_3 the third layer of sulphur atoms is identical with the first one, giving rise to a wurtzite type lattice with four cation vacancies per cell. In $MgGa_2S_4$ double the number of sulphur layers is required to define the period in the direction [100]. Starting with the slab of octahedra at $x=0$, $y=0$ one notices the following arrangement of atom layers parallel to (100):

$$x=-\tfrac{1}{8},\ 0,\ \tfrac{1}{8},\ 0{\cdot}3,\ \tfrac{3}{8},\ \tfrac{5}{8},\ 0{\cdot}7,\ \tfrac{7}{8},\ 1,\ \tfrac{9}{8}$$

S Mg S Ga S S Ga S Mg S

Three filled double layers of anions are succeeded by an empty double layer. The mutual distance in the empty double layer (3·3 Å) is larger than in the filled layers (3·0 Å), an effect which can be readily explained in terms of attractive and repulsive electrostatic forces. The same type of empty and filled layers also occurs in $MgAl_2S_4$ and $MnAl_2S_4$. In this structure, however, the stacking sequence is not disrupted in directions perpendicular to the layers, explaining why these compounds have rhombohedral (hexagonal) symmetry. The *C* centring in $MgGa_2S_4$ destroys this hexagonal layer symmetry. Why gallium causes this less symmetric coordination in this class of compounds is not clear and will be an object of further investigations.

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The Crystal Structure of $Ho_{11}Ge_{10}$[*]

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The crystal structure of a compound in the holmium–germanium system near 1:1 stoichiometry has been determined by single-crystal X-ray diffraction techniques. The composition established is $Ho_{11}Ge_{10}$. The unit cell is body-centered tetragonal, and contains four $Ho_{11}Ge_{10}$ units. Lattice constants are $a=10{\cdot}79$, $c=16{\cdot}23$ Å; the calculated density is 8·93 $g.cm^{-3}$. The space group as deduced from the structure analysis is *I*4/*mmm*.

Germanium atoms in the structure are of three types: (1) isolated atoms; (2) pairs of atoms (bond lengths 2·54 and 2·96 Å); and (3) a square cluster of four atoms (bond length 2·58 Å). Infinite chains of Ge atoms found in CrB-type HoGe do not occur in $Ho_{11}Ge_{10}$. Coordination numbers of Ge atoms are either 8 or 9; of Ho, 15–17.

Introduction

During a recent investigation of rare-earth monogermanide compounds (Tharp, Smith & Johnson, 1966), three different structure-types were encountered. Two of these, FeB- and CrB-types, were similar to phases observed in the rare-earth monosilicide series (Gladyshevskii & Kripyakevich, 1964). The third, a complex, body-centered tetragonal phase of unknown structure-type, occurred for the eight rare-earth elements, gadolinium through lutetium. This phase was consistently obtained from preparations whose initial composition was equiatomic; however, composition of the crystals was not definitely established as 1:1. The

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