

The proposed hydrogen-bonding scheme is supported (a) by the O—O distances in Table 2, and (b) by the observation of peaks in the difference Fourier map attributable to H atoms. Table 2 includes all O—O distances (not including O atoms in the same phosphate group) out to a distance of 3.10 Å. Nearly all of these short contacts may be attributed to O—H...O interactions. As can be seen, O(16) has four oxygen near neighbours in the range 2.738 to 3.003 Å, all of which suggest some degree of interaction between the atoms concerned. It appears that H(19) does not take part in hydrogen bonding; this seems reasonable as the nearest possible acceptor, O(21), is at a distance 3.032 Å from O(20).

We also conducted a ^{31}P solid-state magic-angle-spinning NMR study of the isomorphous compound $\text{Na}_3\text{ZnP}_3\text{O}_{10}\cdot 12\text{H}_2\text{O}$; spectra of the title compound cannot be obtained owing to the paramagnetism of the Mn^{2+} ion. Broad resonances were observed at -6.0 and -19.3 p.p.m. (upfield relative to 85% phosphoric acid solution). The relative intensities of the two peaks show that the terminal P nuclei resonate at lower field than the bridging P, in agreement with previous work (Grimmer & Haubenreisser, 1983). However, the presence of the quadrupolar Na nuclei causes sufficient broadening to make the terminal P nuclei indistinguishable.

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Structure of $\zeta_1\text{-Mn}_{5.11}\text{Ge}_2$

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Abstract. $M_r = 425.97$, trigonal, $P3c1$, $a = 7.198$ (1), $c = 39.227$ (4) Å, $V = 1760.1$ Å³, $D_x = 7.233$ Mg m⁻³, $Z = 18$ (92 Mn and 36 Ge per unit cell), $\text{Mo } K\alpha$, $\lambda = 0.71069$ Å, $\mu = 31.0$ mm⁻¹, $F(000) = 3452$, $T = 295$ K, final $R(F) = 0.0557$, $wR(F) = 0.0400$ for 923

independent reflections. The structure is characterized by two kinds of fundamental layers having three atoms stacked alternately by $c/30$. Additional atoms are placed on three threefold axes. Ge atoms are located every 2.5 layers and Mn atoms are placed at the

remaining fundamental layers and on three threefold axes.

Introduction. There are five intermetallic compounds in the Mn–Ge system: Mn_{3.25}Ge, Mn₅Ge₂, Mn₂Ge, Mn₅Ge₃ and Mn₁₁Ge₈. Mn_{3.25}Ge has high- and low-temperature phases, whose structures are respectively of types Ni₃Sn (*D0*₁₉) (Zwicker, Jahn & Schubert, 1949) and Al₃Ti (*D0*₂₂) (Kádár & Krén, 1971). Mn₅Ge₂ also has high- and low-temperature phases. The low-temperature κ phase belongs to a Mg₅Ga₂-type structure (Ohba, Ueyama, Kitano & Komura, 1984). The crystal structures of χ -Mn₂Ge, η -Mn₅Ge₃ and θ -Mn₁₁Ge₈ have been analyzed to be Ni₂In (*B8*₂) (Ellner, 1980), Mn₅Si₃ (*D8*₈) (Castelliz, 1953) and Cr₁₁Ge₈ types (Israeloff, Völlenknecht & Wittmann, 1974; Ohba, Watanabe & Komura, 1984), respectively.

The high-temperature phase ζ -Mn₅Ge₂ shows anomalous magnetic behaviour (Ohoyama, 1961; Wachtel & Henig, 1969; Yamada, Ohashi & Ohoyama, 1982). The structure of ζ -Mn₅Ge₂ was first reported by Zwicker *et al.* (1949) and they found by X-ray powder diffraction that the structure is closely related to NiAs (*B8*₁) or Ni₂In (*B8*₂) type. Ohoyama (1961) proposed that the structure was hexagonal, $a = 7.18$, $c = 13.08$ Å and had close connections with the Ni₂In (*B8*₂) type structure. Komura & Hirayama (1981) examined the crystal structure and found that the crystal was trigonal, space group *P3c1*, lattice constants $a = 7.185$, $c = 39.17$ Å from single-crystal X-ray diffraction data. The structure was refined by full-matrix least squares to a final *R* value of 0.095. However, a few atoms were found to have negative temperature factors and some others anomalously large ones.

Kifune & Komura (1986) observed ζ -Mn₅Ge₂ by transmission electron microscopy. They found the parallel intergrowth of two types of structures in electron micrographs. Selected-area electron diffraction patterns of the two structures showed that they have different lattice constants along the *c* axis; *i.e.* one is 39 Å and the other 13 Å in the hexagonal cell. We call the former ζ_1 and the latter ζ_2 hereafter in this paper.

Mixed crystals of ζ_1 and ζ_2 have double peaks in the magnetization measurement against temperature (Yamada, Sakai, Usami & Ohoyama, 1986). The samples which show no such double peaks in magnetization have been obtained by a precise control of the concentration. Such samples have been found by X-ray examination to be pure ζ_1 -Mn_{5.11}Ge₂. In this paper, the structure determination of ζ_1 -Mn_{5.11}Ge₂ is reported using such a sample.

Experimental. A tiny fragment of an irregular shape, maximum dimension 0.09 mm, was selected for single-crystal work from an ingot of pure ζ_1 -Mn_{5.11}Ge₂. Laue photographs were taken in order to ascertain that the

fragment was a single crystal and they showed Laue symmetry $\bar{3}m$. Lattice constants estimated from interplanar spacings are $a = 7.198$ (1) and $c = 39.227$ (4) Å using 25 reflections ($19^\circ < 2\theta < 41^\circ$). Rigaku automated four-circle diffractometer (AFC-5), graphite-monochromated Mo *K* α radiation, 3729 measured reflections with $|F_o| \geq 3\sigma(|F_o|)$, $-9 \leq h \leq 9$, $-9 \leq k \leq 9$, $0 \leq l \leq 50$, $2^\circ \leq 2\theta \leq 50^\circ$, averaged to 923 independent reflections; $R_{\text{int}} = 0.0644$. ω scan was employed because of long *c* axis, scan range $(1.5 + 0.5 \tan\theta)^\circ$, scan rate 2° min^{-1} . Backgrounds measured at the beginning and end of each scan range for 5 s. *Lp* and spherical absorption corrections ($\mu r = 1.1$) applied. Systematic absences indicate space group *P3c1* or *P3̄c1*.

An initial structure was constructed by comparison with the structure of Mn₅Ge₃. The direct method was also applied for the determination of the structure. The framework obtained by the latter method is similar to that obtained by the former. Several atoms were added to the initial structure model and atomic positions were corrected to reduce the residual peaks in the Fourier synthesis and the difference Fourier maps. Centrosymmetric space group *P3̄c1* was ruled out in the course of this procedure.

Full-matrix least-squares refinement using *RADIEL* (Coppens, Guru Row, Leung, Stevens, Becker & Yang, 1979); variable parameters: isotropic temperature factors, scale factor, positional parameters and isotropic extinction correction parameter. Scattering factors and anomalous-dispersion correction f' and f'' for Mn and Ge atoms from *International Tables for X-ray Crystallography* (1974). Final *R* value is $R(F) = 0.0557$, $wR(F) = 0.0400$, $w = 1/\sigma^2(|F_o|)$, $S = 1.3123$, $g = 0.104 \times 10^{-4}$ ($y_{\text{min}} = 0.66$). $(\Delta/\sigma)_{\text{max}} = 0.28$ and $\Delta\rho$ excursions $\leq 13.5 \text{ e} \text{ \AA}^{-3}$. Determination of the polar direction has not been attempted. Calculations carried out with HITAC M-200H computer at the Information Processing Center of Hiroshima University.

Discussion. In a previous work (Komura & Hirayama, 1981), the *R* value could not be reduced sufficiently and a few atoms had negative temperature factors and some others anomalously large ones. The reason for these difficulties may be the inappropriate choice of the crystal specimen; that is, the sample used in the previous work was a mixture of ζ_1 and ζ_2 , so the intensities of reflections with $l = 3n$ should be the sum of ζ_1 and ζ_2 .

The final parameters obtained are listed in Table 1. The structure obtained (Fig. 1) is thus more accurate than that reported previously. The structure is constructed by 30 fundamental layers of two kinds, shown in Fig. 1(a), stacked alternately along the *c* direction by about *c*/30. Ge layers are placed on every five layers. Mn and Ge atoms placed on the threefold axes are

represented by open and filled circles, respectively, in Fig. 1(b). Ge atoms on the threefold axes are located almost every five layers, so that it is found that distances between Ge atoms on the threefold axes and fundamental layers are approximately 2.5 layers. This arrangement produces strong intensity for 0,0,12.

Table 1. Atomic parameters for ζ_1 -Mn_{5,11}Ge₂

		x	y	z	B ₀ (Å ²)
Mn(1)	6(d)	0.3263 (16)	0.0278 (17)	0.0	0.92 (17)
Mn(2)	6(d)	-0.0292 (13)	-0.3626 (13)	0.1679 (3)	0.03 (12)
Mn(3)	6(d)	0.3364 (17)	0.0333 (16)	0.3349 (3)	0.68 (16)
Mn(4)	6(d)	0.6055 (14)	-0.0549 (14)	0.0325 (3)	0.42 (13)
Mn(5)	6(d)	0.0412 (8)	-0.6524 (7)	0.1999 (1)	1.05 (8)
Mn(6)	6(d)	0.6097 (14)	-0.0589 (15)	0.3651 (3)	1.18 (15)
Ge(7)	6(d)	0.3230 (6)	0.0064 (11)	0.0653 (1)	0.82 (4)
Ge(8)	6(d)	-0.0026 (13)	-0.3482 (10)	0.2316 (2)	0.80 (10)
Ge(9)	6(d)	0.3390 (13)	-0.0067 (12)	0.3991 (2)	0.91 (13)
Mn(10)	6(d)	0.6639 (8)	0.0566 (8)	0.0987 (1)	1.54 (9)
Mn(11)	6(d)	-0.0385 (16)	-0.6797 (15)	0.2671 (3)	0.95 (15)
Mn(12)	6(d)	0.6912 (15)	0.0285 (15)	0.4319 (3)	0.71 (15)
Mn(13)	6(d)	0.3053 (18)	-0.0334 (18)	0.1321 (2)	0.79 (18)
Mn(14)	6(d)	0.0231 (7)	-0.3223 (8)	0.2973 (1)	0.76 (8)
Mn(15)	6(d)	0.3281 (20)	-0.0222 (20)	0.4648 (3)	1.71 (21)
Mn(16)	2(a)	0.0	0.0	0.0359 (4)	1.92 (29)
Mn(17)	2(a)	0.0	0.0	0.0988 (3)	0.12 (17)
Mn(18)	2(a)	0.0	0.0	0.2359 (4)	1.83 (22)
Mn(19)	2(a)	0.0	0.0	0.3761 (4)	0.75 (17)
Ge(20)	2(a)	0.0	0.0	0.1626 (4)	1.78 (22)
Ge(21)	2(a)	0.0	0.0	0.3077 (3)	1.02 (13)
Ge(22)	2(a)	0.0	0.0	0.4678 (3)	0.49 (14)
Ge(23)	2(b)			0.4906 (3)	0.74 (17)
Ge(24)	2(b)			0.1585 (3)	0.89 (18)
Ge(25)	2(b)			0.3238 (3)	1.11 (18)
Mn(26)	2(b)			0.0884 (4)	1.30 (29)
Mn(27)	2(b)			0.2545 (5)	1.63 (31)
Mn(28)	2(b)			0.4234 (5)	1.06 (28)
Mn(29)	2(c)			0.0412 (4)	0.88 (26)
Mn(30)	2(c)			0.2069 (4)	0.32 (23)
Mn(31)	2(c)			0.3748 (4)	0.04 (20)
Ge(32)	2(c)			0.1410 (3)	1.01 (19)
Ge(33)	2(c)			0.3087 (3)	0.91 (19)
Ge(34)	2(c)			0.4730 (3)	0.88 (18)

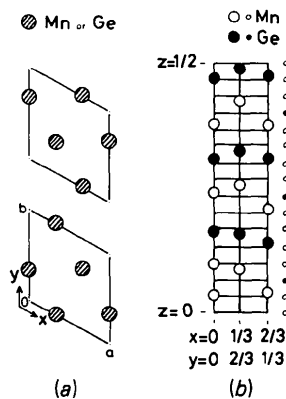


Fig. 1. Structure of ζ_1 -Mn_{5,11}Ge₂. (a) Arrangement of atoms in the fundamental layers. (b) Projection of the structure along [010]. Half of the unit cell is drawn. Fundamental layers and threefold axes are represented by horizontal and vertical lines, respectively. Atoms on threefold axes are represented by large open and filled circles. The kinds of atoms in the fundamental layers are shown by small open and filled circles beside the figure.

Table 2. Coordination numbers and interatomic distances (Å) for ζ_1 -Mn_{5,11}Ge₂

	Coordination numbers	Interatomic distances	
		min.	max.
Mn(1)	13 (9 Mn, 4 Ge)	2.564 (9)	2.889 (17)
Mn(2)	12 (8 Mn, 4 Ge)	2.429 (10)	2.833 (21)
Mn(3)	13 (9 Mn, 4 Ge)	2.497 (10)	2.919 (15)
Mn(4)	12 (8 Mn, 4 Ge)	2.577 (12)	3.022 (10)
Mn(5)	13 (8 Mn, 5 Ge)	2.542 (11)	3.074 (13)
Mn(6)	12 (8 Mn, 4 Ge)	2.540 (16)	3.178 (12)
Ge(7)	12 (12 Mn)	2.533 (9)	3.097 (7)
Ge(8)	11 (11 Mn)	2.506 (15)	3.035 (12)
Ge(9)	11 (11 Mn)	2.518 (12)	3.178 (12)
Mn(10)	12 (8 Mn, 4 Ge)	2.533 (9)	3.097 (7)
Mn(11)	13 (8 Mn, 5 Ge)	2.633 (10)	3.208 (16)
Mn(12)	13 (8 Mn, 5 Ge)	2.612 (8)	3.200 (16)
Mn(13)	13 (9 Mn, 4 Ge)	2.492 (15)	2.906 (16)
Mn(14)	13 (9 Mn, 4 Ge)	2.441 (6)	3.307 (11)
Mn(15)	12 (8 Mn, 4 Ge)	2.449 (17)	2.880 (23)
Mn(16)	11 (7 Mn, 4 Ge)	2.468 (20)	2.672 (20)
Mn(17)	11 (7 Mn, 4 Ge)	2.468 (20)	2.667 (15)
Mn(18)	14 (9 Mn, 5 Ge)	2.516 (9)	3.307 (11)
Mn(19)	13 (9 Mn, 4 Ge)	2.625 (11)	3.199 (15)
Ge(20)	11 (11 Mn)	2.503 (19)	3.016 (20)
Ge(21)	11 (11 Mn)	2.441 (6)	2.926 (14)
Ge(22)	10 (10 Mn)	2.449 (17)	2.724 (14)
Ge(23)	10 (10 Mn)	2.474 (16)	2.651 (13)
Ge(24)	10 (10 Mn)	2.492 (15)	2.749 (21)
Ge(25)	10 (10 Mn)	2.499 (8)	2.718 (22)
Mn(26)	13 (9 Mn, 4 Ge)	2.643 (10)	2.954 (16)
Mn(27)	13 (9 Mn, 4 Ge)	2.530 (12)	3.074 (13)
Mn(28)	13 (9 Mn, 4 Ge)	2.518 (12)	3.026 (18)
Mn(29)	13 (9 Mn, 4 Ge)	2.594 (7)	3.003 (14)
Mn(30)	13 (9 Mn, 4 Ge)	2.531 (9)	3.208 (16)
Mn(31)	13 (9 Mn, 4 Ge)	2.586 (9)	3.200 (16)
Ge(32)	10 (10 Mn)	2.429 (10)	2.644 (9)
Ge(33)	10 (10 Mn)	2.497 (10)	2.717 (14)
Ge(34)	10 (10 Mn)	2.521 (10)	2.798 (14)

The arrangement of atoms at two successive fundamental layers resembles the arrangement at the layer $z = \frac{1}{4}$ or $\frac{3}{4}$ in the structure of Mn₅Si₃ (D8₈). They are separated into two layers in the structure of ζ_1 . Atoms on two threefold axes, $\frac{1}{3}z$ and $\frac{2}{3}z$, in Mn₅Si₃ are placed regularly at $z = 0$ and $\frac{1}{2}$. On the other hand, the positions of atoms on three threefold axes in ζ_1 -Mn_{5,11}Ge₂ are not so regular.

Interatomic distances and coordination numbers are listed in Table 2.* An anomalously short distance, 2.21 Å, in the previous work has been removed here. Coordination numbers of Mn range from 11 to 14. On the other hand, Ge atoms are surrounded by only 10 to 12 Mn atoms. It is interesting to consider the number of Mn atoms which are located around an Mn atom. Mn atoms in the fundamental layers are surrounded by eight or nine Mn atoms. Mn atoms on the threefold axes with $\frac{1}{3}z$ and $\frac{2}{3}z$ have nine Mn atoms around them. The number of coordinated Mn atoms on 00z is seven or nine. The difference arises from the number of atoms on the three threefold axes, *i.e.* there are fourteen atoms located on the threefold axis through 00z and twelve atoms on the other threefold axes. The difference in the

* Lists of structure factors and interatomic distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43253 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

numbers on the threefold axes leads to the deviation from stoichiometry of the compound. The chemical formula can be described as Mn_{5.11}Ge₂ instead of Mn₅Ge₂ according to the results of the refinement. This deviation sensitively affects the magnetic properties as shown by Yamada *et al.* (1986).

The structure is fairly complex with long periodicity along *c*. The structure is not directly related to Mn₅Si₂ (Shoemaker & Shoemaker, 1976), contrary to the relation between Mn₅Ge₃ and Mn₅Si₃.

The separation of a single crystal of ζ_2 -Mn₅Ge₂ is now being attempted in order to perform a structure determination.

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Refinement of the Structure of Copper(II) Chromate from Single-Crystal Data

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Abstract. CuCrO₄, $M_r = 179.54$, orthorhombic, *Cmcm*, $a = 5.433$ (1), $b = 8.968$ (3), $c = 5.890$ (3) Å, $V = 287.0$ (2) Å³, $Z = 4$, D_m (pycnometrically) = 4.04, $D_x = 4.155$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 109.5$ cm⁻¹, $F(000) = 340$, $T = 293$ K, $R = 0.039$ with 556 unique observed reflections. The structure is built up by chains of edge-linked tetragonally distorted CuO₆ octahedra, which are connected by slightly irregular CrO₄ tetrahedra sharing their O atoms with the octahedra. Cu–O(equatorial) = 1.965 (2), Cu–O(axial) = 2.400 (3), mean Cr–O = 1.665 Å. These bond lengths are significantly different from those found previously from powder data.

Introduction. Compounds *ABO*₄ have been shown to exist in numerous structures, many of which are polymorphous at atmospheric pressure and also adopt different structures at higher pressure. These compounds range from those with silica-like structures, with tetrahedral coordination of *A* and *B*, to oxide salts

containing well defined anions *BO*₄ and exhibiting high coordination number of *A* (Wells, 1984).

CuCrO₄ is isomorphous with CrVO₄, and its structure has been determined from powder data by Brandt (1943). Owing to the limited data – strong reflections at small 2θ angles such as 110, 020 are not mentioned – the previous work cannot be considered as very precise. Since mixed oxides containing chromium in various oxidation states and other transition metals are of importance in heterogeneous catalysis, a refinement of the crystal structure of CuCrO₄ using diffractometer data was undertaken.

Experimental. Dark red twinned preferentially needle-shaped crystals of CuCrO₄, with a metallic lustre, were obtained by heating an aqueous solution of CrO₃ with an excess of Cu₂(OH)₂CO₃ in a sealed glass tube at 470 K for ~20 h. Structure determination with an irregularly shaped tabular single crystal 0.30 × 0.06 × 0.02 mm, cut from a twin. Preliminary precession and