

une coordination peu fréquente, mais elle a été récemment rencontrée dans le niobate lamellaire $\text{K}_4\text{Nb}_6\text{O}_{17}$ (Gasperin & Le Bihan, 1982). Toutes les distances O—O ont été calculées: la plus courte, O(4)—O(5), est de 2,56 Å.

En conclusion, cet oxyde dans lequel la plupart des cations ont un entourage peu fréquent est membre d'une nouvelle famille cristallographique de formule générale $A^+B_2^{4.5+}U_2^{6+}O_{11}$ avec $A = \text{K,Cs}$ et $B = (\text{Nb,Ti})$.

L'examen de la balance des charges montre qu'il n'est pas impossible qu'un alcalino-terreux de grande dimension comme le baryum puisse donner un composé isotype de formule $\text{BaTi}_2\text{U}_2\text{O}_{11}$, et cette synthèse sera tentée. D'autre part, les canaux infinis qui composent la structure incitent à penser que ces niobotitanouranates

pourraient être, sur le site de l'alcalin, le siège d'échanges cationiques.

Références

- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1963). *ORFLS*. Rapport ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.
- CHEVALIER, R. (1973). Thèse d'Etat Paris. CNRS n° A 0 8636.
- GASPERIN, M. & LE BIHAN, M. T. (1982). *J. Solid State Chem.* **43**, 346–353.
- International Tables for X-ray Crystallography*. (1974). Tome IV. Birmingham: Kynoch Press. (Distributeur actuel D. Reidel, Dordrecht.)
- MEULENAER, J. DE & TOMPA, H. (1965). *Acta Cryst.* **19**, 1014–1018.
- ZACHARIASEN, W. H. (1954). *Acta Cryst.* **7**, 795–799.

Acta Cryst. (1986). **C42**, 138–141

GdMg₅: a Complex Structure with a Large Cubic Cell

BY M. L. FORNASINI AND P. MANFRINETTI

Istituto di Chimica Fisica, Università di Genova, Palazzo delle Scienze, Corso Europa, 16132 Genova, Italy

AND K. A. GSCHNEIDNER JR

Ames Laboratory, and Department of Material Sciences and Engineering, Iowa State University, Ames, IA 50011, USA*

(Received 8 July 1985; accepted 24 September 1985)

Abstract. $M_r = 280$, cubic, $F\bar{4}3m$, $a = 22.344(8)$ Å, $V = 11155(7)$ Å³, $D_m(293\text{ K}) = 3.03$, $D_x = 3.03$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 11.5$ mm⁻¹, $F(000) = 9059$, room temperature, $R = 0.048$ for 606 reflections with $F_o > 2\sigma(F_o)$. The final composition was determined to be GdMg_{5.05}. The structure, which is closely related to that of Sm₁₁Cd₄₅, consists of nested polyhedra units A , B , C , D with their centres at $0,0,0$; $\frac{1}{4},\frac{1}{4},\frac{1}{4}$; $\frac{1}{2},\frac{1}{2},\frac{1}{2}$; and $\frac{3}{4},\frac{3}{4},\frac{3}{4}$, respectively. The units A and D , each containing 27 atoms, are typical of the body-centred cubic structure, while the units B and C , each containing 29 atoms, are found in the α -Mn structure. The A unit has two partially occupied Mg positions and another position filled two thirds by Gd and one third by Mg atoms. Cubes plus octahedra (CN 14) and Kasper polyhedra (CN 12, 14, 16) are the most common coordination polyhedra in this structure.

Introduction. In a recent revision of the Gd–Mg phase diagram (Manfrinetti & Gschneidner, 1985) the existence of three phases with cubic symmetry was

confirmed: GdMg (CsCl type), GdMg₂ (MgCu₂ type) and GdMg₃ (BiF₃ type). A fourth phase, which was located by thermal and metallographic analyses, was found to lie close to a composition of 84 at.% Mg and to have a face-centred cubic cell with $a \approx 22.35$ Å. A compound with formula GdMg_{4.5} (82 at.% Mg) and with the same crystallographic characteristics was reported by Kripyakevich & Evdokimenko (1969), but the structure remained unresolved. The present paper is concerned with the structure determination of this phase, in order to complete the study of the Gd–Mg system.

Experimental. 5 g of alloy containing 84 at.% Mg prepared from 99.8 at.% pure Gd and >99.9 at.% pure Mg by melting in a sealed Ta crucible and slowly cooling. The density of the bulk material determined pycnometrically using *sym*-dibromoethane as the fluid. A parallelepiped single crystal $0.07 \times 0.12 \times 0.15$ mm examined using an Enraf–Nonius CAD-4 automatic diffractometer and graphite-monochromated Mo $K\alpha$ radiation; the lattice constant determined from powder patterns. Data collection in the ω – θ scan mode up to $\sin\theta/\lambda = 0.65$ Å⁻¹ with intensity variations of <2% for 1295 reflections with $0 \leq h, k, l \leq 16$ and $h > l, k > l$. A

* Operated for the US Department of Energy by Iowa State University under contract No. W-7405-ENG-82. This work was partially supported by the Office of Basic Energy Sciences.

semiempirical absorption correction by azimuthal scans of two top reflections with a ratio of 1.4 between max. and min. transmission factors. A total of 699 independent reflections with $R_{\text{int}} = 0.035$ obtained, of which 606 with $F_o > 2\sigma(F_o)$ used in the refinement. Cubic symmetry, Laue group $m\bar{3}m$, presence of hkl reflections only if h, k, l all odd or all even, possible space groups $F432, F\bar{4}3m$ and $Fm\bar{3}m$.

A suitable starting model was found to be the $\text{Sm}_{11}\text{Cd}_{45}$ structure (space group $F\bar{4}3m$) formed by several rare-earth-cadmium (Fornasini, Chabot & Parthé, 1978) and rare-earth-mercury (Merlo & Fornasini, 1979) compounds. Isotropic refinement of 39 parameters based on F^2 's with *SHELX76* (Sheldrick, 1976), atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974), yielded $R = 0.077$. At this point the thermal factors of Gd in *A* CO, Mg in *A* IT and Mg in *A* OH were abnormally higher than the mean values of the other corresponding atoms (for the particular identification of the atoms see *Discussion*). Taking into account the most probable composition of the phase, ~84 at.% Mg, and the measured density, a greater content of magnesium than 80.4 at.% Mg ($\text{Gd}_{11}\text{Mg}_{45}$) was proposed. Therefore, the 48 atoms of Gd in *A* CO were progressively replaced by Mg, while partial occupancy was given to the Mg sites in *A* IT and *A* OH. The occupancies and thermal factors were alternately fixed and varied in several cycles of refinement of the whole structure using weights $w = 1/[\sigma^2(F_o) + 0.011F_o^2]$. The final results are $R = 0.048$, $wR = 0.076$ with $(\Delta/\sigma)_{\text{max}}$ in the last cycle 0.06, and max. and min. heights in the final difference Fourier synthesis of 3.7 and $-2.9 \text{ e } \text{Å}^{-3}$, respectively. The final composition resulted in 72.7 Gd atoms and 367.2 Mg atoms per unit cell, corresponding to $\text{GdMg}_{5.05 \pm 0.05}$ (83.5 \pm 0.1 at.% Mg). The occupancies, positional and isotropic thermal parameters are listed in Table 1.*

In an attempt to determine the composition range of the GdMg_5 phase, two other alloys, one on the Gd-rich side (82 at.% Mg) and the other on the Mg-rich side (85 at.% Mg), were prepared in the same way. In both cases extra lines due to a second phase were present but GdMg_5 appeared as the predominant phase with a lattice constant ($a = 22.36 \text{ Å}$) slightly larger than the value observed for the single-crystal sample. This fact can be related to the structural features of the compound: in the Gd-rich alloy Gd atoms can replace Mg in the *A* CO position, while in the Mg-rich alloy the partially occupied positions *A* IT and *A* OH can be filled up. In each case an enlargement of the elementary cell is expected. Metallographic analysis of the

Table 1. *Occupancies, positional and isotropic thermal parameters for GdMg_5*

A unit		x	y	z	$B(\text{Å}^2)$
<i>A</i> C 4(a)	Gd	0	0	0	0.89 (8)
<i>A</i> IT 16(e)	Mg	0.0846 (6)	0.0846 (6)	0.0846 (6)	2.3 (4)
<i>A</i> OT 16(e)	Mg	0.9146 (5)	0.9146 (5)	0.9146 (5)	2.6 (4)
<i>A</i> OH 24(f)	Mg	0.1574 (9)	0	0	2.7 (3)
<i>A</i> CO 48(h)	Gd	0.1741 (1)	0.1741 (1)	0.0111 (1)	1.37 (3)
	Mg	0.68(1)%			
B unit					
<i>B</i> C 4(c)	Gd	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	0.60 (6)
<i>B</i> TT 48(h)	Mg	0.2959 (3)	0.2959 (3)	0.3902 (3)	1.4 (1)
<i>B</i> T 16(e)	Mg	0.1621 (4)	0.1621 (4)	0.1621 (4)	1.3 (3)
<i>B</i> CO 48(h)	Mg	0.4343 (3)	0.4343 (3)	0.2624 (4)	1.5 (1)
C unit					
<i>C</i> C 4(b)	Mg	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	3.1 (8)
<i>C</i> TT 48(h)	Mg	0.5457 (3)	0.5457 (3)	0.6416 (4)	1.5 (1)
<i>C</i> T 16(e)	Gd	0.4072 (1)	0.4072 (1)	0.4072 (1)	1.03 (5)
<i>C</i> CO 48(h)	Mg	0.6722 (3)	0.6722 (3)	0.5129 (4)	1.2 (1)
D unit					
<i>D</i> C 4(d)	Mg	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	1.6 (5)
<i>D</i> IT 16(e)	Mg	0.8300 (5)	0.8300 (5)	0.8300 (5)	2.4 (3)
<i>D</i> OT 16(e)	Gd	0.6598 (1)	0.6598 (1)	0.6598 (1)	0.82 (4)
<i>D</i> OH 24(g)	Mg	0.9066 (6)	$\frac{1}{2}$	$\frac{1}{2}$	1.7 (2)
<i>D</i> CO 48(h)	Mg	0.9151 (4)	0.9151 (4)	0.7664 (5)	3.0 (2)

85 at.% Mg alloy shows the presence of ~15% eutectic (91.2 at.% Mg) indicating an upper composition limit of 84 at.% Mg for GdMg_5 .

Discussion. Table 1 shows the atoms grouped according to the nested polyhedra concept, instead of listing them in a sequential order. The same criterion has already been adopted for the structure of $\text{Sm}_{11}\text{Cd}_{45}$ to which GdMg_5 is closely related. We prefer to use here the term 'nested polyhedra unit' introduced by Chabot, Cenual & Parthé (1981) for describing complicated cubic structures, instead of the term 'cluster' used originally in the description of $\text{Sm}_{11}\text{Cd}_{45}$, since in intermetallic compounds all atoms participate in a three-dimensional framework and do not form separate groups.

The structure of GdMg_5 is composed of nested polyhedra units with their centres at 0,0,0; $\frac{1}{4}, \frac{1}{4}, \frac{1}{4}$; $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$ and $\frac{3}{4}, \frac{3}{4}, \frac{3}{4}$ along the body diagonal of the cubic cell, and identified by the letters *A, B, C, D*. The units *A* and *D* in their full occupation are formed by 27 atoms. On going from the centre to the periphery one finds an atom at the centre (*C*), four atoms as an inner tetrahedron (*IT*) and four atoms as an outer tetrahedron (*OT*) giving rise together to a cube, six atoms as an octahedron (*OH*) and finally twelve atoms as a cubo-octahedron (*CO*). This sequence of nested polyhedra is typical of the body-centred cubic structure (b.c.c.). The units *B* and *C* are formed by 29 atoms: an atom at the centre (*C*), twelve atoms as a truncated tetrahedron (*TT*), four atoms which merge from the centre of the hexagonal faces of *TT* as a tetrahedron (*T*), and finally twelve atoms as a cubo-octahedron (*CO*). In this case the sequence of nested polyhedra is typical of the α -Mn structure. The units *A* and *D*, or *B* and *C* differ only in

* Lists of structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42526 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the atomic distribution on the polyhedra sites, and are repeated by the translational vectors of the face-centred lattice, thus generating the whole structure. Pictures of the b.c.c. and α -Mn units are given by Fornasini, Chabot & Parthé (1978) and by Chabot, Cenual & Parthé (1981).

The GdMg₅ structure differs from Sm₁₁Cd₄₅ in that in the *A* unit of GdMg₅ both the inner tetrahedron and the octahedron are defective, while the cubo-octahedron comprises two thirds Gd and one third Mg atoms. Except for these changes, which are responsible for the difference in composition, all other crystallographic features of the two structures are similar.

Table 2. Interatomic distances (Å) in GdMg₅

Gd <i>A</i> C–4Mg	<i>A</i> IT 3.27 (1)	Mg <i>D</i> C–4Mg	<i>D</i> IT 3.10 (1)
–4Mg	<i>A</i> OT 3.31 (1)	–4Gd	<i>D</i> OT 3.49 (1)
–6Mg	<i>A</i> OH 3.52 (1)	–6Mg	<i>D</i> OH 3.50 (1)
Mg <i>A</i> IT–Mg	<i>B</i> T 3.00 (2)	Mg <i>D</i> IT–3Mg	<i>D</i> CO 3.04 (1)
–3Mg	<i>A</i> OH 3.13 (2)	–3Mg	<i>D</i> OH 3.05 (1)
–3Gd(Mg)	<i>A</i> CO 3.27 (1)	–Mg	<i>D</i> C 3.10 (1)
–Gd	<i>A</i> C 3.27 (1)	–Mg	<i>A</i> OT 3.27 (2)
–3Mg	<i>A</i> OT 3.80 (2)	–3Gd	<i>D</i> OT 3.82 (1)
–3Mg	<i>B</i> CO 4.02 (2)	–3Gd(Mg)	<i>A</i> CO 4.05 (1)
Mg <i>A</i> OT–3Mg	<i>A</i> OH 3.14 (2)	Gd <i>D</i> OT–3Mg	<i>D</i> OH 3.21 (1)
–Mg	<i>D</i> IT 3.27 (2)	–3Mg	<i>C</i> CO 3.31 (1)
–Gd	<i>A</i> C 3.31 (1)	–3Mg	<i>D</i> CO 3.36 (1)
–3Mg	<i>D</i> CO 3.31 (2)	–Mg	<i>D</i> C 3.49 (1)
–3Gd(Mg)	<i>A</i> CO 3.54 (1)	–3Mg	<i>C</i> TT 3.63 (1)
–3Mg	<i>A</i> IT 3.80 (2)	–3Mg	<i>D</i> IT 3.82 (1)
Mg <i>A</i> OH–2Mg	<i>A</i> IT 3.13 (2)	Mg <i>D</i> OH–2Mg	<i>C</i> CO 3.05 (1)
–2Mg	<i>B</i> CO 3.13 (2)	–2Mg	<i>D</i> IT 3.05 (1)
–2Mg	<i>A</i> OT 3.14 (2)	–2Gd	<i>D</i> OT 3.21 (1)
–2Mg	<i>D</i> CO 3.18 (1)	–2Gd(Mg)	<i>A</i> CO 3.35 (1)
–Gd	<i>A</i> C 3.52 (1)	–Mg	<i>D</i> C 3.50 (1)
–4Gd(Mg)	<i>A</i> CO 3.92 (1)	–4Mg	<i>D</i> CO 3.71 (1)
Gd(Mg) <i>A</i> CO–2Mg	<i>D</i> CO 3.22 (1)	Mg <i>D</i> CO–Mg	<i>D</i> IT 3.04 (1)
–Mg	<i>A</i> IT 3.27 (1)	–Mg	<i>C</i> TT 3.05 (1)
–Mg	<i>D</i> OH 3.35 (1)	–Mg	<i>A</i> OH 3.18 (1)
–2Mg	<i>B</i> CO 3.35 (1)	–2Gd(Mg)	<i>A</i> CO 3.22 (1)
–Mg	<i>B</i> T 3.40 (1)	–2Mg	<i>C</i> CO 3.29 (1)
–2Mg	<i>C</i> CO 3.48 (1)	–Mg	<i>A</i> OT 3.31 (2)
–Mg	<i>A</i> OT 3.54 (1)	–Gd	<i>D</i> OT 3.36 (1)
–2Mg	<i>B</i> TT 3.57 (1)	–2Mg	<i>B</i> CO 3.45 (1)
–2Mg	<i>A</i> OH 3.92 (1)	–2Mg	<i>D</i> OH 3.71 (1)
–Mg	<i>D</i> IT 4.05 (1)		
Gd <i>B</i> C–4Mg	<i>B</i> T 3.40 (1)	Mg <i>C</i> C–12Mg	<i>C</i> TT 3.48 (1)
–12Mg	<i>B</i> TT 3.45 (1)	–4Gd	<i>C</i> T 3.59 (1)
Mg <i>B</i> TT–Mg	<i>B</i> TT 2.90 (1)	Mg <i>C</i> TT–Mg	<i>C</i> TT 2.89 (1)
–Mg	<i>C</i> CO 2.92 (1)	–2Mg	<i>C</i> CO 3.00 (1)
–2Mg	<i>B</i> TT 2.98 (1)	–2Mg	<i>C</i> TT 3.03 (1)
–2Mg	<i>B</i> CO 3.33 (1)	–Mg	<i>D</i> CO 3.05 (1)
–2Mg	<i>B</i> T 3.34 (1)	–2Mg	<i>B</i> CO 3.32 (1)
–Gd	<i>B</i> C 3.45 (1)	–2Gd	<i>C</i> T 3.45 (1)
–Gd	<i>C</i> T 3.54 (1)	–Mg	<i>C</i> C 3.48 (1)
–2Gd(Mg)	<i>A</i> CO 3.57 (1)	–Gd	<i>D</i> OT 3.63 (1)
Mg <i>B</i> T–Mg	<i>A</i> IT 3.00 (2)	Gd <i>C</i> T–3Mg	<i>B</i> CO 3.35 (1)
–6Mg	<i>B</i> TT 3.34 (1)	–3Mg	<i>C</i> CO 3.45 (1)
–3Gd(Mg)	<i>A</i> CO 3.40 (1)	–6Mg	<i>C</i> TT 3.45 (1)
–Gd	<i>B</i> C 3.40 (1)	–3Mg	<i>B</i> TT 3.54 (1)
–3Mg	<i>B</i> CO 3.78 (1)	–Mg	<i>C</i> C 3.59 (1)
Mg <i>B</i> CO–Mg	<i>A</i> OH 3.13 (2)	Mg <i>C</i> CO–Mg	<i>B</i> TT 2.92 (1)
–2Mg	<i>C</i> CO 3.30 (1)	–2Mg	<i>C</i> TT 3.00 (1)
–2Mg	<i>C</i> TT 3.32 (1)	–Mg	<i>D</i> OH 3.05 (1)
–2Mg	<i>B</i> TT 3.33 (1)	–2Mg	<i>D</i> CO 3.29 (1)
–Gd	<i>C</i> T 3.35 (1)	–2Mg	<i>B</i> CO 3.30 (1)
2Gd(Mg)	<i>A</i> CO 3.35 (1)	–Gd	<i>D</i> OT 3.31 (1)
2Mg	<i>D</i> CO 3.45 (1)	–Gd	<i>C</i> T 3.45 (1)
Mg	<i>B</i> T 3.78 (1)	–2Gd(Mg)	<i>A</i> CO 3.48 (1)
Mg	<i>A</i> IT 4.02 (2)		
Mg	<i>B</i> CO 4.15 (1)		

On the other hand, partial occupancy or replacement in complex cubic structures belonging to the same space group, $F\bar{4}3m$, and containing from 392 to 448 atoms in the elementary cell are common. For instance, Pt₃Zn₁₀ (Johansson & Westman, 1970) presents five cases where Pt and Zn are distributed on the same site set and an octahedral site set is completely vacant; Mg₆Pd (Samson, 1972) contains a 16(*e*) position filled one half by Mg and one half by Pd atoms; in Sn₁₁Cu₄₁ (Booth, Brandon, Brizard, Chieh & Pearson, 1977) an inner tetrahedron site is occupied by about $\frac{3}{4}$ of the Cu atoms.

Table 2 reports the interatomic distances in GdMg₅ up to $d/\sum r = 1.3$, where *d* is the observed distance and $\sum r$ the appropriate sum of the metallic radii of the two atoms, taken from Teatum, Gschneidner & Waber (1968). According to this limit, which corresponds to the first significant gap in the distance distribution, the coordination polyhedra can be determined. With respect to the Sm₁₁Cd₄₅ structure, a different distance distribution occurs for GdMg₅, and consequently some polyhedra are varied.

In the units *A* and *D* (b.c.c.) 14 neighbours form a cube plus an octahedron around Gd *A* C, Mg *A* IT, Mg *A* OT, Mg *D* C and Mg *D* IT, while a cube plus a defective octahedron formed by 13 atoms surround Mg *A* OH, Mg *D* OH and Mg *D* CO. Analogous but distorted polyhedra are also found around Gd(Mg) *A* CO, where a vertex of the cube is replaced by a pair of atoms achieving CN 15, and around Gd *D* OT, where a vertex of the cube is replaced by three atoms achieving CN 16.

In the units *B* and *C* (α -Mn) the atoms are surrounded by Kasper polyhedra. The CN 16 Kasper polyhedron is found for Gd *B* C, Mg *C* C and Gd *C* T; the CN 14 Kasper polyhedron is found for Mg *B* T; the CN 12 icosahedron is found for Mg *B* TT, Mg *C* TT and Mg *C* CO. Finally, Mg *B* CO is coordinated with 15 atoms forming a distorted CN 14 Kasper polyhedron with the addition of another atom.

Interestingly, similar coordinations are present also in the other Gd–Mg phases: cube plus octahedron is found in both GdMg (CsCl type) and in GdMg₃ (BiF₃ type), while CN 16 and CN 12 Kasper polyhedra surround the Gd and Mg atoms, respectively, in the Laves phase GdMg₂ with MgCu₂ structure.

References

- BOOTH, M. H., BRANDON, J. K., BRIZARD, R. Y., CHIEH, C. & PEARSON, W. B. (1977). *Acta Cryst.* **B33**, 30–36.
 CHABOT, B., CENZUAL, K. & PARTHÉ, E. (1981). *Acta Cryst.* **A37**, 6–11.
 FORNASINI, M. L., CHABOT, B. & PARTHÉ, E. (1978). *Acta Cryst.* **B34**, 2093–2099.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
 JOHANSSON, A. & WESTMAN, S. (1970). *Acta Chem. Scand.* **24**, 3471–3479.

KRIPYAKEVICH, P. I. & EVDOKIMENKO, V. I. (1969). *Visn. L'viv. Derzh. Univ. Ser. Khim.* **11**, 3–7.
 MANFRINETTI, P. & GSCHNEIDNER, K. A. JR (1985). To be published.
 MERLO, F. & FORNASINI, M. L. (1979). *J. Less-Common Met.* **64**, 221–231.

SAMSON, S. (1972). *Acta Cryst.* **B28**, 936–945.
 SHELDRIK, G. M. (1976). *SHELX76*. A program for crystal structure determination. Univ. of Cambridge, England.
 TEATUM, E., GSCHNEIDNER, K. A. JR & WABER, J. (1968). Report LA-4003. National Technical Information Service, Springfield, VA 22151, USA.

Acta Cryst. (1986). **C42**, 141–143

Calcium and Strontium Dichloride Hexahydrates by Neutron Diffraction*

BY P. A. AGRON AND W. R. BUSING

Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831, USA

(Received 1 May 1985; accepted 21 August 1985)

Abstract. $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$, isotypic, trigonal, $P321$, $Z = 1$, unit cells from X-ray $\text{Cu K}\alpha_1$, $\lambda_x = 1.54051$, $\lambda_N = 1.077 \text{ \AA}$, room temperature. $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$, $M_r = 219.08$, $a = 7.8759 (2)$, $c = 3.9545 (2) \text{ \AA}$, $V = 212.43 (6) \text{ \AA}^3$, $D_m = 1.71$, $D_x = 1.7126 \text{ g cm}^{-3}$, $\mu = 2.566 \text{ cm}^{-1}$, $F(000) = 14.00 \text{ fm}$, $R(F) = 0.0287$ for 304 unique reflections. $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$, $M_r = 266.62$, $a = 7.9596 (3)$, $c = 4.1243 (2) \text{ \AA}$, $V = 226.29 \text{ \AA}^3$, $D_m = 1.93$, $D_x = 1.9565 \text{ g cm}^{-3}$, $\mu = 2.430 \text{ cm}^{-1}$, $F(000) = 16.12 \text{ fm}$, $R(F) = 0.0356$ for 325 unique reflections. Nine water molecules are coordinated to each metal ion. Three of these adjoin only one cation and six are shared between two such ions. These water molecules are hydrogen-bonded to the Cl^- ions so that each anion is surrounded by six H atoms in a distorted octahedral arrangement.

Introduction. These single-crystal neutron diffraction studies are part of a program to locate the H atoms precisely and to investigate the role of water in simple salt hydrates.

Experimental. Lattice parameters from least-squares refinement based on X-ray diffractometer Bragg angles for 12 high-angle reflections ($2\theta = 120$ to 130°); $\text{Cu K}\alpha_1$ radiation, $\lambda = 1.54051 \text{ \AA}$. Results agree with reported values (Herrmann, 1930, 1931; Jensen, 1940; Leclaire & Borel, 1977; English & Nassimbeni, 1984).

Crystals for neutron diffraction obtained by slow growth at room temperature in desiccator over concentrated sulfuric acid. Selected crystals mounted on quartz fibers and sealed in thin quartz envelopes (Agron & Busing, 1985). Reported D_m taken from published

values (*CRC Handbook of Chemistry and Physics*, 1984).

Neutron diffraction intensities measured (Busing, Smith, Peterson & Levy, 1964) to $(\sin\theta/\lambda)_{\text{max}} = 0.71 \text{ \AA}^{-1}$, corrected for absorption (Busing & Levy, 1957). No significant variation of standard reflections observed.

Least-squares structure refinement started with reported X-ray structure of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ (Jensen, 1940) with H atoms located on expected hydrogen bonds to Cl^- ions (Agron & Busing, 1969). Refinements based on F^2 with weights $w = 1/[\sigma_c^2 + (0.03 F^2)^2]$, where σ_c is the standard error of F^2 based on counting statistics. Variables included scale factors, coordinates, anisotropic temperature-factor coefficients, and extinction parameters. Anisotropic extinction (Coppens & Hamilton, 1970) used for the calcium compound; isotropic extinction (Zachariasen, 1967) sufficed for the strontium salt. Neutron scattering factors: Ca, 4.90; Sr, 7.02; Cl, 9.579; O, 5.805; H, -3.741 fm (Koester & Yelon, 1982). Equivalent reflections, corrected for extinction, combined for final least-squares adjustment. Table 1 summarizes experiments and refinements.†

Computer programs: lattice-parameter refinement (Busing, Ellison, Levy, King & Roseberry, 1968; Busing, 1970); absorption corrections, *ORABS* (Wehe, Busing & Levy, 1962); structure-factor least squares, *XFLS* (Busing, Martin & Levy, 1962); function and error calculation, *ORFFE* (Busing, Martin & Levy, 1964); and crystal-structure illustration, *ORTEP* (Johnson, 1976).

† Lists of anisotropic temperature-factor coefficients and observed and calculated squares of neutron diffraction structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42520 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Research sponsored by the Division of Materials Sciences, Office of Basic Energy Sciences, US Department of Energy, under contract DE-ACO5-84OR21400 with Martin Marietta Energy Systems, Inc.