

become available as observed in [(H₂O)₄Mn]₂[(EDTA)Cd]₂·4nH₂O (Solans, Font-Altaba, Oliva & Herrera, 1983) where Cd is heptacoordinated.

Comparing the distances in the coordination polyhedra of the hydrate ions it appears that the differences between related distances of different polyhedra (Table 4) are essentially related to the different ionic radii, while the differences between the X—O distances for the same metal are determined by the hydrogen bonds in which the coordinated O atoms are involved; thus, the shortest values correspond to the X—O(acetato) and X—O(aqua) distances, the latter when the water coordinating to metal is linked by weak hydrogen bonds to the uncoordinated water, while the largest values correspond to X—O(aqua) when the coordinated water molecule is linked by two strong hydrogen bonds to the acetato O atoms. From the distances excluded it can also be deduced that the different cationic radii produce variations in intrachain distances greater than 3.5 Å, but do not produce differences in interchain distances or intrachain hydrogen bonds.

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Crystal Structures of Ethylenediaminetetraacetato Metal Complexes. III. Hexa-aquamagnesium(II) Aquaethylenediaminetetraacetatocadmium(II) Trihydrate, [(H₂O)₆Mg(C₁₀H₁₂N₂O₈)(H₂O)Cd]_n·3nH₂O

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Abstract. $M_r = 605.1$, triclinic, $P\bar{1}$, $a = 20.594(4)$, $b = 7.755(2)$, $c = 7.619(2)$ Å, $\alpha = 102.25(3)$, $\beta = 102.01(3)$, $\gamma = 82.52(4)^\circ$, $V = 1158(2)$ Å³, $Z = 2$, $D_x = 1.73$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.052$ mm⁻¹, $F(000) = 620$. Final $R = 0.047$ for 4011 observed reflections. The crystal structure consists of discrete ions linked by hydrogen bonds. There are two independent sets of Mg atoms located on inversion centres separated by $\frac{1}{2}a$ and displaying a distorted octahedral coordination. The Cd atom is coordinated to a water and an ethylenediaminetetraacetato molecule, the latter ligand acting in a hexadentate fashion.

Introduction. A systematic study on crystal structures with ethylenediaminetetraacetato (EDTA) as a ligand is being carried out by the Departments of Crystallography and Mineralogy (Faculty of Geology) and Inorganic Chemistry (Faculty of Pharmacy) of the University of Barcelona (preceding papers: Solans, Font-Altaba, Oliva & Herrera, 1983*a,b*).

In general, as can be observed from the literature, the crystal structures of compounds with the formula (H₂O)_nX(EDTA)(H₂O)_mY·pH₂O consist of infinite chains of X and Y atoms, where the EDTA ligand acts as bridge between the two metals (Candlish, Michael,

Neal, Lingafelter & Rose, 1978; Pozhidaev, Polynova, Porai-Koshits & Neronova, 1973; Solans, Font-Altaba, Oliva & Herrera, 1983a). The two exceptions are $\{(H_2O)_4Mn[(EDTA)Cd]_2\}_n \cdot 4nH_2O$ (Solans, Font-Altaba, Oliva & Herrera, 1983b) and $(H_2O)_6Mg(EDTA)(H_2O)Mg \cdot 3H_2O$ (Passer, White & Cheng, 1977), which are double-chain and discrete ions, respectively. Thereby, it was considered interesting to solve the crystal structure with $X = Mg$ and $Y = Cd$, in order to elucidate their configuration.

Experimental. Crystal $0.2 \times 0.4 \times 0.4$ mm, Philips PW1100 diffractometer, Mo $K\alpha$, graphite-crystal monochromator, unit-cell parameters from 25 independent reflections, ω -scan technique, scan width 1.6° , 4031 independent reflections with $\theta \leq 25^\circ$, 4011 with $I \geq 2.5\sigma(I)$, Lp correction, absorption ignored; direct methods [Mg and Cd atoms located with *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), remaining non-H atoms with *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, Van den Hark & Prick, 1980), H atoms not located], anisotropic full matrix (*SHELX*, Sheldrick, 1976), anomalous-scattering factors from *International Tables for X-ray Crystallography* (1974), $R = 0.047$, $R_w = 0.050$, $w = [\sigma^2(F_o) + 0.005|F_o|^2]^{-1}$; Digital VAX-750.

Discussion. The atomic parameters are given in Table 1.*

The relative arrangement of anions and cations in the structure is determined by a complex three-dimensional network of hydrogen bonds (Fig. 1 and Table 2). The $W(5)$ and $W(7)$ hydrate molecules act as acceptors from $(H_2O)_6Mg^{2+}$ ions by two hydrogen bonds and donate protons to $W(6)$ or O atoms of the EDTA ligand, also by two hydrogen bonds. $W(6)$ is only involved in one acceptor and two donor hydrogen bonds.

Each O atom of the EDTA ligand coordinated to the Cd atom is hydrogen-bonded to a water molecule linked to Mg atoms, the exception being O(20), which is hydrogen-bonded to a hydrate molecule [$W(5)$]. The remaining O atoms of the EDTA ligand are involved in two hydrogen bonds with water molecules, but O(14) and O(26) also accept a third hydrogen bond from a hydrate molecule.

The cation is located on an inversion centre and is very close to regular octahedral geometry. The average Mg—O bond length and O—Mg—O bond angle are 2.80 Å and 91.3° , values similar to those obtained in $(H_2O)_6Mg(EDTA)(H_2O)Mg \cdot 3H_2O$ (Passer *et al.*, 1977) or $[(H_2O)_4Mg(EDTA)Zn]_n \cdot 2nH_2O$ (Solans *et al.*, 1983a).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38247 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters (Å^2)

$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B_{eq}
MgA	5000	5000	5000	1.6 (1)
W(1)A	4027 (6)	4908 (17)	3550 (18)	2.5 (3)
W(2)A	4665 (7)	5387 (19)	7514 (18)	2.7 (5)
W(3)A	4930 (7)	7679 (16)	5249 (18)	2.8 (5)
W(4)	7662 (7)	770 (16)	-1352 (18)	2.2 (4)
W(5)	5577 (5)	413 (16)	7465 (16)	2.1 (3)
W(6)	1203 (7)	376 (18)	2487 (19)	3.4 (5)
W(7)	555 (7)	7300 (18)	832 (18)	2.9 (5)
MgB	10000	5000	5000	1.8 (1)
W(1)B	9872 (6)	7726 (19)	5313 (20)	2.0 (4)
W(2)B	9237 (5)	4873 (18)	2636 (18)	2.9 (4)
W(3)B	9316 (6)	5168 (20)	6655 (21)	2.7 (5)
Cd	7437 (1)	8268 (1)	619 (1)	1.42 (4)
N(8)	6865 (4)	8885 (11)	3129 (11)	1.9 (3)
C(9)	7135 (5)	10478 (14)	4426 (15)	2.0 (4)
C(10)	7213 (6)	11892 (14)	3355 (16)	2.9 (5)
N(11)	7736 (4)	11179 (10)	2203 (11)	1.6 (3)
O(12)	6310 (3)	7269 (10)	-284 (10)	2.1 (3)
C(13)	5908 (5)	6905 (15)	745 (15)	2.4 (4)
O(14)	5321 (3)	7479 (9)	496 (10)	1.9 (3)
C(15)	6149 (4)	9115 (12)	2590 (15)	1.8 (4)
O(16)	8056 (4)	6855 (12)	2994 (11)	2.6 (4)
C(17)	7741 (5)	6650 (13)	4230 (13)	1.6 (3)
O(18)	7983 (3)	5729 (9)	5409 (10)	2.0 (3)
C(19)	7051 (4)	7384 (11)	4126 (11)	1.0 (3)
O(20)	6887 (3)	10323 (8)	-1153 (8)	1.3 (2)
C(21)	7077 (4)	11872 (11)	-807 (10)	1.0 (3)
O(22)	6963 (4)	12969 (9)	-1779 (11)	2.2 (3)
C(23)	7665 (5)	12298 (15)	871 (14)	2.3 (4)
O(24)	8542 (4)	8750 (11)	411 (11)	2.8 (4)
C(25)	8841 (5)	9659 (13)	1991 (12)	1.8 (4)
O(26)	9484 (3)	9522 (9)	2461 (9)	1.9 (3)
C(27)	8444 (5)	11004 (15)	3139 (14)	1.2 (5)

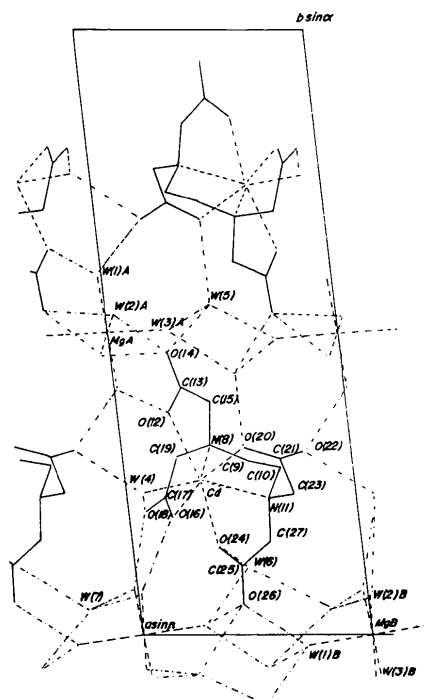


Fig. 1. Projection of the unit-cell contents down the c axis.

Table 2. Selected intermolecular and intramolecular bond lengths (Å) and angles (°)

MgA-W(1)A	2.079 (12)	MgB-W(1)B	2.065 (17)
MgA-W(2)A	2.118 (15)	MgB-W(2)B	2.124 (14)
MgA-W(3)A	2.034 (15)	MgB-W(3)B	2.053 (13)
W(1)A-MgA-W(2)A	91.1 (5)	W(1)B-MgB-W(2)B	88.2 (7)
W(1)A-MgA-W(3)A	91.0 (5)	W(1)B-MgB-W(3)B	88.3 (7)
W(2)A-MgA-W(3)A	88.4 (5)	W(2)B-MgB-W(3)B	91.6 (6)
Cd-W(4)	2.246 (9)	Cd-O(16)	2.370 (9)
Cd-N(8)	2.382 (9)	Cd-O(12)	2.457 (7)
Cd-N(11)	2.414 (7)	Cd-O(20)	2.336 (6)
		Cd-O(24)	2.396 (9)
N(8)-Cd-W(4)	132.8 (5)	O(12)-Cd-W(4)	81.6 (4)
N(8)-Cd-N(11)	76.2 (3)	O(12)-Cd-O(16)	109.6 (3)
N(8)-Cd-O(16)	70.6 (3)	O(12)-Cd-O(20)	78.4 (2)
N(8)-Cd-O(12)	68.3 (3)	O(12)-Cd-O(24)	159.9 (30)
N(8)-Cd-O(20)	102.4 (3)	O(16)-Cd-W(4)	87.6 (4)
N(8)-Cd-O(24)	131.7 (5)	O(16)-Cd-O(20)	165.1 (2)
N(11)-Cd-W(4)	148.6 (4)	O(16)-Cd-O(24)	80.4 (3)
N(11)-Cd-O(12)	126.8 (2)	O(20)-Cd-W(4)	106.3 (4)
N(11)-Cd-O(16)	93.6 (3)	O(2)-Cd-O(24)	96.0 (3)
N(11)-Cd-O(20)	71.8 (2)	O(24)-Cd-W(4)	81.5 (4)
N(11)-Cd-O(24)	67.8 (3)		
W(1)A ⁱ ...O(12)	2.707 (12)	W(6) ^{iv} ...W(1)B	3.086 (14)
W(1)A ⁱⁱ ...O(22)	2.759 (12)	W(6) ^v ...O(24)	2.612 (13)
W(2)A ⁱⁱⁱ ...O(14)	2.711 (15)	W(7) ^{vi} ...O(26)	2.935 (14)
W(2)A ^{iv} ...O(14)	2.938 (15)	W(7) ^{vii} ...W(6)	2.813 (14)
W(3)A ^v ...W(5)	2.721 (15)	W(1)B ^{viii} ...O(26)	2.752 (15)
W(3)A ^{vi} ...W(5)	2.748 (15)	W(1)B ^{ix} ...O(26)	2.730 (15)
W(4) ^{vii} ...O(18)	2.677 (13)	W(2)B ^x ...O(16)	2.734 (15)
W(4) ^{viii} ...O(22)	2.686 (13)	W(2)B ^{xi} ...W(7)	2.898 (15)
W(5) ^{ix} ...O(20)	2.682 (15)	W(3)B ^{xii} ...O(18)	2.721 (13)
W(5) ^x ...O(14)	2.799 (15)	W(3)B ^{xiii} ...W(7)	2.934 (14)

Symmetry code: (i) $1-x, 1-y, -z$; (ii) $1-x, 2-y, -z$; (iii) $x, y, z-1$; (iv) $1-x, 1-y, 1-z$; (v) $x, y-1, z$; (vi) $x, y, z+1$; (vii) $x, 1+y, z$; (viii) $x, 1+y, z-1$; (ix) $1+x, y, z$; (x) x, y, z ; (xi) $2-x, 2-y, 1-z$.

The role of each water molecule linked to an Mg^{2+} in the molecular packing is different. The water molecules of cation B donate protons with the same geometry as those observed in the cation of the previously mentioned Mg-Mg crystal structure (Passer *et al.*, 1977), while in cation A, W(3)A acts as donor to two hydrate molecules and W(1)A to a coordinated O and a non-coordinated O of the EDTA ligand.

The geometry of the anion is more complex. The Cd atom is heptacoordinated by four O atoms and two N

atoms of the EDTA ligand, the coordination being completed by a water molecule. These coordinating atoms lie at the vertices of a pentagonal bipyramid. This coordination is similar to those obtained in other heptacoordinated EDTA complexes (Richards, Pedersen, Silverton & Hoard, 1964; Lind, Hamor, Hamor & Hoard, 1964; Stezowski, Countryman & Hoard, 1973; Passer *et al.*, 1977; Solans *et al.*, 1983b) which changes from the monocapped trigonal prism to the pentagonal bipyramid.

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Monopotassium Salt of 3-Hydroxy-2-methyl-4-nitro-2H-1,2,6-thiadiazine 1,1-Dioxide, $\text{C}_4\text{H}_4\text{N}_3\text{O}_5\text{S}^- \cdot \text{K}^+$

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Abstract. $M_r = 245.27$, triclinic, $P\bar{1}$, $a = 8.933$ (6), $b = 7.645$ (5), $c = 7.168$ (7) Å, $\alpha = 110.7$ (1), $\beta = 106.3$ (1), $\gamma = 70.3$ (1)°, $V = 424.0$ (5) Å³, $Z = 2$, $D_x = 1.92$ Mg m⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu =$