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Structure of Zinc(II), Magnesium(II) and Manganese(II) Bis(phosphoenolpyruvate) Dihydrate

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Abstract. $Zn^{2+}.2(C_3H_4O_6P)^-.2H_2O$, $M_r = 435.47$, triclinic, $P\bar{1}$, $a = 5.184\ (3)$, $b = 5.341\ (5)$, $c = 12.124\ (9)$ Å, $\alpha = 84.69\ (7)$, $\beta = 86.72\ (6)$, $\gamma = 86.36\ (7)°$, $V = 333.1\ (5)$ Å^3, $Z = 1$, $D_m = 2.17$, $D_x = 2.171\ (4)$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 22.2$ cm^{-1}, $F(000) = 220$, $T = 294\ (1)$ K, final $R = 0.0236$ for 1486 non-zero reflections. $Mg^{2+}.2(C_3H_4O_6P)^-.2H_2O$, $M_r = 394.41$, triclinic, $P\bar{1}$, $a = 5.172\ (3)$, $b = 5.333\ (3)$, $c = 12.184\ (6)$ Å, $\alpha = 85.30\ (4)$, $\beta = 86.90\ (4)$, $\gamma = 86.56\ (4)°$, $V = 333.9\ (4)$ Å^3, $Z = 1$, $D_m = 1.91$, $D_x = 1.961\ (3)$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 42.0$ cm^{-1}, $F(000) = 202$, $T = 293\ (1)$ K, final $R = 0.0294$ for 1170 non-zero reflections. $Mn^{2+}.2(C_3H_4O_6P)^-.2H_2O$, $M_r = 425.04$, triclinic, $P\bar{1}$, $a = 5.277\ (3)$, $b = 5.443\ (3)$, $c = 12.090\ (6)$ Å, $\alpha = 83.74\ (4)$, $\beta = 86.35\ (4)$, $\gamma = 85.96\ (4)°$, $V = 343.8\ (4)$ Å^3, $Z = 1$, $D_m = 2.04$, $D_x = 2.053\ (3)$ g cm^{-3}, μ(Cu $K\alpha$) = 111.9 cm^{-1}, $F(000) = 215$, $T = 295\ (1)$ K, final $R = 0.0296$ for 1268 non-zero reflections. All three salts are isomorphous with calcium bis(phosphoenolpyruvate) dihydrate. The M^{2+} ions occupy centers of symmetry and are six coordinate (by two water and four phosphate O atoms). Two terminal O atoms of each phosphate group bridge pairs of M atoms, thereby forming linear chains along **b**. The carboxylic hydroxyl is *trans* planar to the ester O atom in the Mn and Ca crystals but is *syn* planar in the Zn and Mg salts.

Introduction. We are currently investigating the crystal structures of phosphoenolpyruvate (PEP) salts and complexes (Weichsel & Lis, 1990; Lis & Kuczek, 1991) to see how PEP geometries depend on the cations present, on the solvent used during crystallization and on the protonation.

Experimental. Almost colorless crystals of the title compounds were grown from aqueous solutions containing a 1:1 molar ratio mixture of MCl_2 (where M

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$= Zn^{2+}$, Mg^{2+} or Mn^{2+}) and phosphoenolpyruvic acid. D_m measured by flotation in $CCl_4/C_2H_4Br_2$ mixture. Kuma KM4 diffractometer (κ geometry) with graphite monochromator was used for lattice parameters and intensity measurements; ω–2θ-scan technique. After each group of 50 reflections two standards were measured: variation ±4%. The experimental details are summarized in Table 1. Neutral-atom scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV); anomalous dispersion was included for all non-H atoms. The refinement was started by using the published coordinates for heavy atoms of the $Ca(PEP)_2.2H_2O$ crystal (Lis & Kuczek, 1991), on F by *SHELX*76 (Sheldrick, 1976). The H atoms were found from difference maps and refined with constraints d(O—H) = 0.97 and d(C—H) = 1.08 Å. Final refinement was performed using anisotropic thermal parameters (isotropic for H atoms); $w = 1/\sigma^2(F_o)$. Absorption corrections following the *DIFABS* procedure (Walker & Stuart, 1983) were applied for all crystals: minimum and maximum absorption corrections were 0.948 and 1.059, 0.928 and 1.122, 0.849 and 1.267 for Zn, Mg and Mn, respectively. No extinction correction. Final atomic parameters for $Zn(PEP)_2.2H_2O$, $Mg(PEP)_2.2H_2O$ and $Mn(PEP)_2.2H_2O$ are given in Table 2.*

Discussion. The principal interatomic distances, bond angles and torsion angles are summarized in Table 3 and are compared with the values for $Ca(PEP)_2.2H_2O$. The structure of the phosphoenolpyruvate monoanion and the atom-numbering schemes for $Zn(PEP)_2.2H_2O$ and $Mn(PEP)_2.2H_2O$ are shown in Figs. 1(*a*) and 1(*b*) respectively. The anions in the Zn (Fig. 1*a*) and Mg salts differ from those in the Mn (Fig. 1*b*) and Ca salts by virtue of the different orientation of the carboxylic hydroxyl group to the ester O(4) atom. We have noted earlier that the hydroxyl group is in a *trans* planar orientation in PEP salts crystallized from water solution and is *syn* planar when crystallized from alcohol solutions (Weichsel & Lis, 1991). The structures of $Zn(PEP)_2.2H_2O$ and $Mg(PEP)_2.2H_2O$ show that from water solutions the PEP moiety with a *syn* planar conformation may also be isolated.

The enolpyruvate system in $M(PEP)_2.2H_2O$ salts is not quite planar. The angle between the carboxyl plane and that formed by C(1), C(2), C(3) and O(4) ranges from 4.8 (8)° in the Mg crystal to 6.3 (8)° in the Mn salt. The P—O (ester) and the enolic bond

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54569 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0276]

Table 1. *Summary of data collection and structure refinement details*

	M = Zn	M = Mg	M = Mn
Crystal size (mm)	0.4 × 0.2 × 0.1	0.09 × 0.05 × 0.03	0.05 × 0.05 × 0.02
Cell constants; 25 reflections (°)	19 < 2θ < 27	21 < 2θ < 47	26 < 2θ < 51
θ_{max} (°)	30	80	80
Scan range	$-7 \le h \le 1$	$-6 \le h \le 6$	$-6 \le h \le 6$
	$-7 \le k \le 7$	$-6 \le k \le 6$	$-6 \le k \le 6$
	$-17 \le l \le 17$	$-15 \le l \le 15$	$-15 \le l \le 15$
Total data measured	2186	2471	2319
Data with $I > 3\sigma(I)$	1636	2045	1966
Unique data with $I > 3\sigma(I)$	1486	1170	1268
R_{int} (after *DIFABS*)	0.0155	0.0188	0.0290
R; wR	0.0236; 0.0285	0.0294; 0.0324	0.0296; 0.0358
$(\Delta/\sigma)_{max}$ in last cycle	0.06	0.03	0.04
$\Delta\rho$ in final ΔF map (e Å^{-3})	−0.40; +0.34	−0.34; +0.32	−0.37; +0.42

Table 2. *Final atomic parameters for zinc*(II), *magnesium*(II) *and manganese*(II) *bis(phosphoenolpyruvate) dihydrate*

$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}/U_{iso}(Å^2)
Zinc(II)				
Zn	0.5	0.5	0.5	0.0134 (2)
P	0.25407 (10)	1.01819 (8)	0.36652 (4)	0.0133 (3)
O(1)	−0.0170 (4)	1.0950 (3)	0.31941 (13)	0.0208 (6)
O(2)	0.2397 (3)	0.7573 (3)	0.42237 (12)	0.0171 (5)
O(3)	0.3478 (4)	1.2218 (3)	0.42515 (13)	0.0221 (6)
O(4)	0.4388 (4)	0.9730 (3)	0.25752 (12)	0.0215 (6)
O(5)	0.8075 (5)	0.8549 (4)	0.11381 (17)	0.0400 (8)
O(6)	0.8203 (5)	1.2491 (5)	0.03553 (18)	0.0544 (9)
O(7)	0.7943 (4)	0.5571 (3)	0.36575 (12)	0.0183 (6)
C(1)	0.7277 (6)	1.0851 (5)	0.10405 (20)	0.0283 (8)
C(2)	0.5087 (5)	1.1653 (5)	0.18003 (18)	0.0229 (8)
C(3)	0.4032 (6)	1.3968 (5)	0.16865 (21)	0.0332 (9)
H(1)	−0.100 (8)	1.243 (5)	0.350 (4)	0.094 (16)
H(5)	0.965 (7)	0.829 (9)	0.068 (4)	0.156 (24)
H(3)	0.231 (4)	1.454 (6)	0.216 (3)	0.040 (9)
H(31)	0.473 (7)	1.527 (5)	0.102 (2)	0.061 (11)
H(7)	0.720 (8)	0.647 (6)	0.301 (2)	0.083 (14)
H(71)	0.951 (4)	0.629 (5)	0.386 (3)	0.037 (9)
Magnesium(II)				
Mg	0.5	0.5	0.5	0.0106 (3)
P	0.25433 (12)	1.01784 (10)	0.36603 (4)	0.0097 (2)
O(1)	−0.0194 (4)	1.0910 (4)	0.32050 (14)	0.0165 (5)
O(2)	0.2447 (4)	0.7584 (3)	0.42241 (13)	0.0136 (5)
O(3)	0.3506 (4)	1.2249 (3)	0.42313 (13)	0.0166 (5)
O(4)	0.4345 (4)	0.9704 (3)	0.25640 (13)	0.0165 (5)
O(5)	0.8079 (5)	0.8508 (4)	0.11363 (18)	0.0330 (7)
O(6)	0.8158 (6)	1.2435 (5)	0.03445 (19)	0.0487 (8)
O(7)	0.7915 (4)	0.5567 (3)	0.36869 (14)	0.0150 (5)
C(1)	0.7243 (6)	1.0826 (6)	0.10334 (21)	0.0226 (8)
C(2)	0.5053 (6)	1.1627 (5)	0.17871 (19)	0.0184 (8)
C(3)	0.3960 (7)	1.3929 (6)	0.16740 (23)	0.0282 (9)
H(1)	−0.122 (8)	1.245 (5)	0.334 (4)	0.085 (16)
H(5)	0.923 (11)	0.817 (12)	0.050 (4)	0.185 (31)
H(3)	0.219 (5)	1.453 (7)	0.211 (3)	0.060 (13)
H(31)	0.472 (7)	1.516 (5)	0.100 (2)	0.041 (10)
H(7)	0.743 (10)	0.668 (8)	0.305 (3)	0.106 (19)
H(71)	0.942 (4)	0.633 (6)	0.393 (3)	0.040 (11)
Manganese(II)				
Mn	0.5	0.5	0.5	0.0151 (2)
P	0.25153 (11)	1.02691 (11)	0.36243 (5)	0.0148 (2)
O(1)	−0.0134 (4)	1.1097 (4)	0.31393 (18)	0.0241 (6)
O(2)	0.2354 (4)	0.7687 (4)	0.41795 (16)	0.0194 (6)
O(3)	0.3434 (4)	1.2217 (4)	0.42261 (17)	0.0238 (6)
O(4)	0.4368 (4)	0.9866 (4)	0.25314 (17)	0.0241 (6)
O(5)	0.8057 (5)	0.8704 (5)	0.10863 (23)	0.0409 (8)
O(6)	0.8358 (6)	1.2664 (6)	0.03787 (24)	0.0495 (8)
O(7)	0.8048 (4)	0.5676 (4)	0.35848 (17)	0.0210 (6)
C(1)	0.7324 (6)	1.0940 (7)	0.10349 (25)	0.0267 (8)
C(2)	0.5164 (6)	1.1790 (6)	0.17801 (23)	0.0224 (7)
C(3)	0.4198 (7)	1.4099 (7)	0.16779 (28)	0.0327 (8)
H(1)	−0.099 (7)	1.265 (4)	0.332 (4)	0.042 (11)
H(6)	0.982 (9)	1.216 (12)	−0.009 (5)	0.143 (29)
H(3)	0.276 (6)	1.480 (8)	0.226 (3)	0.046 (12)
H(31)	0.488 (7)	1.552 (6)	0.106 (3)	0.039 (11)
H(7)	0.731 (8)	0.667 (9)	0.296 (3)	0.074 (16)
H(71)	0.915 (8)	0.656 (8)	0.400 (4)	0.072 (16)

lengths {[1.615 (2)–1.618 (2) Å] and [1.378 (3)–1.390 (3) Å]} are in agreement with values in other mono-ionized PEP structures. All other bond lengths and angles do not differ significantly from those found in other mono-ionized PEP moieties. The torsion angle P—O(4)—C(2)—C(3), defining the phosphate-group orientation with respect to the enolpyruvate system, ranges from $-14.5\,(9)°$ in the Zn salt to $-17.4\,(7)°$ in the Ca salt. The values of this torsion angle in other crystals of PEP (Weichsel & Lis, 1991) are in the $\pm 90°$ range.

The M^{2+} ions are located at the centers of distorted octahedra formed by two water and four phosphate O atoms. These octahedra are doubly connected in the [010] direction by terminal phosphate O atoms, forming a linear polymer (Fig. 2*a* for the Mg salt and Fig. 2*b* for the Mn salt) with an $M\cdots M$ distance of 5.341 (5), 5.333 (3) and 5.443 (3) Å for Zn, Mg and Mn crystals, respectively. The metal–oxygen bond distances [M—O(2), M—O(3) and M—O(7); see Table 3] decrease in the order Ca > Mn > Zn > Mg. These changes are in accordance with the decrease in the ionic radius of the central ions $Ca^{2+} > Mn^{2+} > Zn^{2+} > Mg^{2+}$.

The hydrogen-bond distances and angles are listed in Table 4: the hydroxyl O(1)—H(1) group is involved (as donor) with a water molecule and pairs of centrosymmetrically related carboxyl groups interact with each other in the usual way irrespective of whether the H atom is bonded to O(5) or O(6) (Fig. 2). Furthermore, the water H(71) atom forms a hydrogen bond with phosphate atom O(2) and the

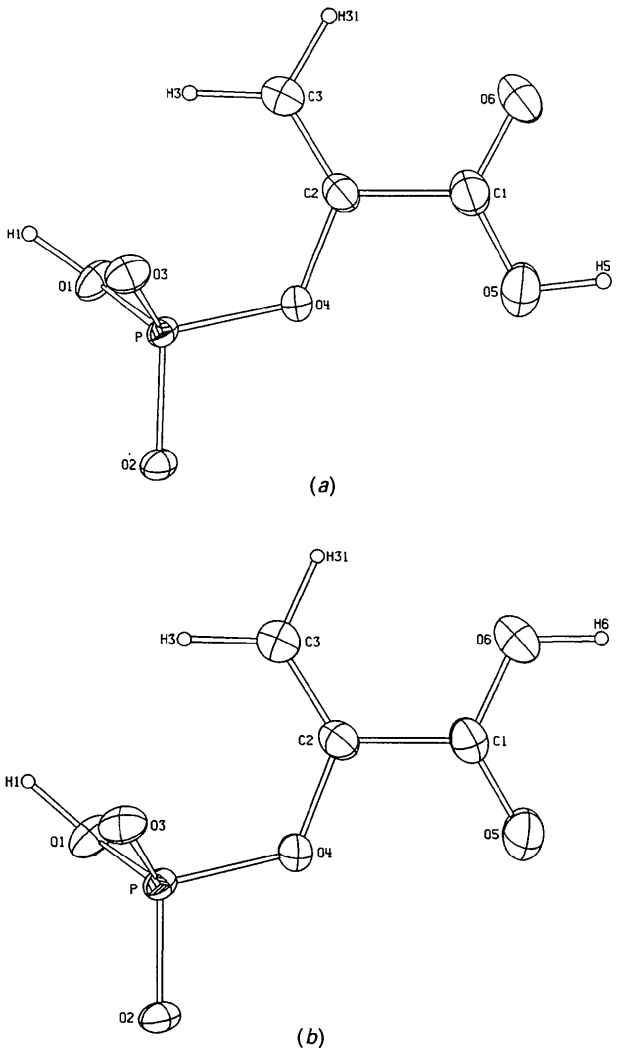

Fig. 1. Molecular geometry and numbering scheme for the phosphoenolpyruvate monoanion in (*a*) zinc(II) and (*b*) manganese(II) bis(phosphoenolpyruvate) dihydrate.

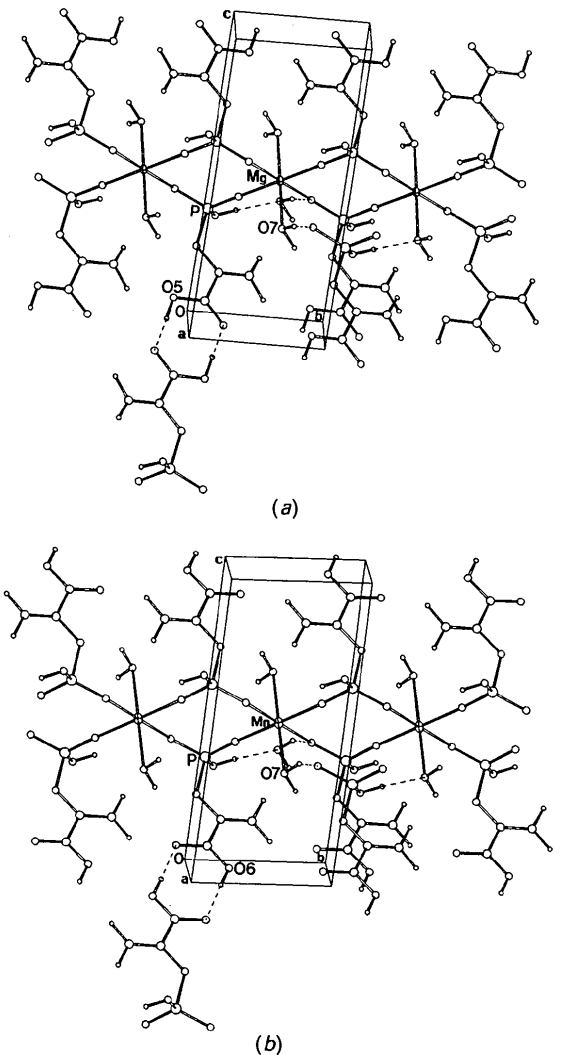

Fig. 2. The packing arrangement in (*a*) the $Mg(PEP)_2.2H_2O$ crystal and (*b*) the $Mn(PEP)_2.2H_2O$ crystal. Dashed lines show strong hydrogen bonds.

Table 3. *Principal interatomic distances* (Å), *angles* (°) *and torsion angles* (°) *in* $M(C_3H_4O_6P)_2.2H_2O$

	M = Zn	M = Mg	M = Mn	M = Ca
P—O(1)	1.564 (2)	1.564 (2)	1.568 (2)	1.573 (3)
P—O(2)	1.496 (2)	1.496 (2)	1.495 (2)	1.499 (2)
P—O(3)	1.474 (2)	1.477 (2)	1.474 (2)	1.477 (2)
P—O(4)	1.615 (2)	1.615 (2)	1.618 (2)	1.616 (2)
O(4)—C(2)	1.378 (3)	1.390 (3)	1.380 (3)	1.383 (4)
O(5)—C(1)	1.269 (3)	1.283 (3)	1.247 (4)	1.237 (4)
O(6)—C(1)	1.249 (3)	1.246 (4)	1.289 (4)	1.296 (4)
C(1)—C(2)	1.487 (3)	1.484 (4)	1.487 (4)	1.486 (5)
C(2)—C(3)	1.318 (3)	1.320 (4)	1.319 (4)	1.320 (5)
M—O(2)	2.065 (2)	2.059 (2)	2.155 (2)	2.326 (3)
M—O(3')	2.033 (2)	2.016 (2)	2.106 (2)	2.259 (3)
M—O(7)	2.180 (2)	2.154 (2)	2.290 (2)	2.451 (3)
O(1)—P—O(2)	107.0 (1)	107.4 (1)	107.5 (2)	107.9 (2)
O(1)—P—O(3)	111.1 (1)	111.3 (1)	111.0 (2)	111.4 (2)
O(1)—P—O(4)	103.9 (1)	103.5 (1)	103.9 (2)	104.0 (2)
O(2)—P—O(3)	120.7 (1)	120.5 (1)	120.3 (2)	119.9 (2)
O(2)—P—O(4)	102.3 (1)	102.3 (1)	102.5 (2)	102.4 (2)
O(3)—P—O(4)	110.3 (1)	110.3 (1)	110.1 (2)	109.8 (2)
P—O(4)—C(2)	123.1 (2)	123.3 (3)	123.3 (2)	124.0 (3)
O(5)—C(1)—O(6)	124.7 (3)	124.1 (3)	124.4 (3)	124.1 (4)
O(5)—C(1)—C(2)	117.6 (3)	117.6 (3)	120.3 (3)	121.4 (3)
O(6)—C(1)—C(2)	117.7 (3)	118.3 (3)	115.3 (3)	114.5 (3)
O(4)—C(2)—C(1)	112.1 (2)	112.4 (3)	111.0 (3)	110.7 (3)
O(4)—C(2)—C(3)	127.3 (3)	126.6 (3)	127.2 (3)	126.7 (4)
C(1)—C(2)—C(3)	120.6 (3)	121.0 (3)	121.7 (3)	122.5 (4)
O(2)—*M*—O(3')	89.2 (1)	89.3 (1)	89.0 (1)	87.0 (1)
O(2)—*M*—O(7)	92.4 (1)	91.7 (1)	91.7 (1)	89.2 (1)
O(3')—*M*—O(7)	91.9 (1)	91.3 (1)	92.8 (1)	93.8 (1)
P—O(2)—*M*	134.9 (1)	136.8 (1)	134.7 (2)	133.4 (2)
P—O(3)—*M*^{ii}	176.1 (2)	177.2 (2)	175.6 (2)	175.0 (2)
O(1)—P—O(4)—C(2)	69.2 (7)	70.4 (4)	71.2 (5)	74.1 (5)
O(2)—P—O(4)—C(2)	−179.6 (6)	−178.0 (4)	−176.9 (4)	−173.6 (5)
O(3)—P—O(4)—C(2)	−50.0 (7)	−48.7 (5)	−47.8 (5)	−45.2 (5)
P—O(4)—C(2)—C(1)	165.9 (8)	164.5 (5)	165.7 (5)	163.8 (5)
P—O(4)—C(2)—C(3)	−14.5 (9)	−16.6 (6)	−15.4 (6)	−17.4 (7)
C(3)—C(2)—C(1)—O(5)	−174.7 (9)	−174.7 (6)	−173.8 (7)	−174.7 (7)
C(3)—C(2)—C(1)—O(6)	5.5 (10)	5.5 (7)	7.7 (7)	5.6 (8)
O(4)—C(2)—C(1)—O(5)	5.0 (9)	4.2 (6)	5.1 (6)	4.2 (7)
O(4)—C(2)—C(1)—O(6)	−174.8 (9)	−175.6 (6)	−173.4 (6)	−175.5 (7)

Symmetry code: (i) $x, y-1, z$; (ii) $x, 1+y, z$.

Table 4. *Hydrogen-bonding data in* $M(C_3H_4O_6P)_2.2H_2O$ *crystals* (Å, °)

O—H···O	M	O···O	H···O	O—H···O
O(1)—H(1)···O(7^{i})	Zn	2.693 (3)	1.75 (3)	162 (4)
	Mg	2.712 (3)	1.77 (3)	161 (4)
	Mn	2.704 (3)	1.75 (3)	169 (3)
	Ca	2.734 (3)	1.79 (4)	163 (5)
O(5)—H(5)···O(6^{ii})	Zn	2.638 (3)	1.69 (5)	163 (4)
	Mg	2.636 (3)	1.69 (5)	164 (5)
O(6)—H(6)···O(5^{ii})	Mn	2.633 (4)	1.67 (6)	169 (6)
	Ca	2.641 (5)	1.69 (5)	167 (5)
O(7)—H(71)···O(2^{iii})	Zn	2.753 (3)	1.78 (2)	179 (3)
	Mg	2.768 (3)	1.80 (3)	173 (3)
	Mn	2.758 (3)	1.87 (4)	150 (4)
	Ca	2.741 (3)	1.83 (4)	155 (4)
O(7)—H(7)···O(4)	Zn	3.044 (4)	2.24 (4)	139 (3)
	Mg	3.072 (4)	2.27 (5)	140 (4)
	Mn	3.107 (4)	2.29 (5)	142 (4)
	Ca	3.213 (4)	2.51 (5)	129 (4)
O(7)—H(7)···O(5)	Zn	3.310 (4)	2.46 (3)	146 (3)
	Mg	3.364 (4)	2.47 (5)	153 (4)
	Mn	3.279 (4)	2.43 (4)	146 (4)
	Ca	3.211 (4)	2.29 (4)	158 (4)

Symmetry code: (i) $x-1, y+1, z$; (ii) $2-x, 2-y, -z$; (iii) $1+x, y, z$.

second [H(7)] water atom is utilized in weak bifurcated hydrogen bonds. There are only small differences between the analogous strong hydrogen bonded oxygen–oxygen distances in the four structures. The largest differences are observed in the weak bifurcated hydrogen bonds.

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Structure of [(*S*)-Alaninato]tetraamminecobalt(III) Sulfate

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Abstract. $[Co(C_3H_6NO_2)(NH_3)_4]SO_4$, $M_r = 311.20$, monoclinic, $P2_1$, $a = 9.769$ (1), $b = 9.080$ (1), $c = 12.474$ (2) Å, $\beta = 100.98$ (8)°, $V = 1086.2$ (2) Å^3, $Z = 4$, $D_x = 1.90$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.78$ mm^{-1}, $F(000) = 648.0$, $T = 293$ K, final $R = 0.035$, $wR = 0.044$ for 2194 unique observed re-

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