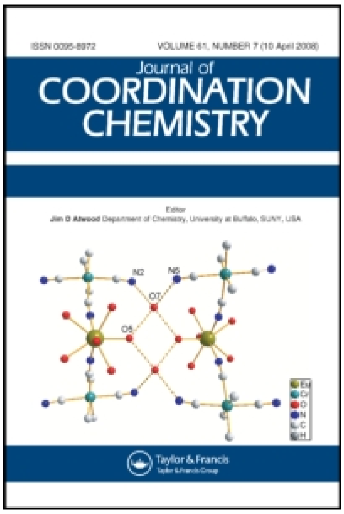

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# Monomeric molecules in the crystal structures of magnesium(II) and zinc(II) complexes with imidazole-4-carboxylate and water ligands

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Magnesium imidazolate: *trans*-diaquabis(imidazole-4-carboxylato-$\kappa^2$N,O)-magnesium(II) $Mg(C_4H_3N_2O_2)_2(H_2O)_2$ crystallizes in the monoclinic system (space group $P2_1/c$). The structure is composed of monomeric molecules, each containing a Mg(II) ion located at the inversion centre and chelated by two imidazole-4-carboxylate ligand molecules *via* their N,O bonding moieties (Mg–N 2.193(2)Å′, Mg–O 2.070(2)Å′) and two water oxygen atoms (Mg–O 2.063(2)Å′. A fairly regular octahedron with water oxygen atoms at the apical positions is formed. The monomers are kept together by hydrogen bonds. Triclinic unit cell (space group $P\bar{1}$) of zinc imidazolate: [*trans*-diaquabis(imidazole-4-carboxylato-$\kappa^2$N,O)-zinc(II)] [monoaquabis(imidazole-4-carboxylato-N,O)-zinc(II)] trihydrate $[Zn(C_4H_3N_2O_2)_2(H_2O)_2]$ $[Zn(C_4H_3N_2O_2)_2(H_2O)]\cdot 3H_2O$ contains two monomeric complex molecules in which the Zn(II) ion is octahedrally coordinated by two *trans* ligand molecules and two water oxygen atoms. The unit cell also contains two monomeric complex molecules in which the Zn(II) ion is pentacoordinate with two ligand molecules and one water oxygen atom. Six solvation water molecules complete the content of the unit cell. Mean bond distances in the pentacoordinated Zn(II) polyhedron are: 2.101(2)Å′ (Zn–O), 2.040(2)Å′ (Zn–N) and 2.002(3)Å′ ($Zn–O_{water}$). An extended network of hydrogen bonds is responsible for the stability of the structure.

*Keywords*: Mg(II) and Zn(II) complexes; Hydrogen bonds; Imidazole-4-carboxylate ligand

## 1. Introduction

Our previous crystal structure studies of Mg(II) and Zn(II) complexes with diazine monocarboxylate ligands [1–5] have shown that all form monomeric molecules and exhibit the same metal coordination mode and geometry: metal ions are chelated by two ligand molecules *via* their N,O bonding moieties consisting of one hetero-ring nitrogen atom and one oxygen atom donated by the nearest carboxylate group. Octahedral coordination is completed by two water oxygen atoms. Since imidazole-4-carboxylate shows the same position of the hetero-ring nitrogen atom and carboxylate group, one may expect, that the structures of the Mg(II) and Zn(II) complexes with this ligand

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will be composed of monomeric molecules, the metal ions will be chelated by the N,O bonding moieties donated by the ligand molecules and the coordination will be octahedral. Therefore, continuing our crystal-chemical studies on divalent metal complexes with heterocycle carboxylate ligands, the crystal structures of Mg(II) and Zn(II) imidazolates have been determined. The results are reported in this article.

## 2. Experimental

The title compound **1** was obtained by adding 2 mmol of imidazole-4-carboxylic acid (ALDRICH) dissolved in 50 mL of boiling, doubly distilled water to 20 mL of hot aqueous solution containing 1 mmol of magnesium acetate tetrahydrate. The mixture was boiled with stirring for 1 h, cooled and then left for crystallization at room temperature. After few days, pale yellow, well formed single crystal blocks deposited in the mother liquid. They were washed with cold water and dried in the air.

The title compound **2** was synthesized by reacting 1 mmol of zinc acetate tetrahydrate dissolved in 50 mL of hot water and 50 mL of hot aqueous solution containing 2 mmol of imidazole-4-carboxylic acid (ALDRICH). The mixture was boiled with stiring for 1 h and then left for crystallization at room temperature. Colourless single crystals appeared in the mother liquid after a couple of days. They were washed with cold water and dried in air. The dimensions of crystals used for data collection are given in table 1.

X-ray intensity data were measured at room temperature using KUMA KM4 four circle diffractometer operating in $\omega$–$2\theta$ mode. Unit cell parameters were obtained by least-squares fit to 25 reflections ($15^\circ < 2\theta < 30^\circ$). Reflections were processed using profile analysis and corrected for Lorentz factor and polarization effects. An analytical absorption correction was adopted [6]. Non-hydrogen atoms were located by direct methods and hydrogen atoms then found by successive Fourier syntheses. Final refinement on $F^2$ by full-matrix least squares was performed on positional parameters of all atoms, anisotropic temperature factors of all non H-atoms and isotropic temperature factors of hydrogen atoms. A weighting scheme was used: $w = 1/[\sigma^2(F_o^2) + (A * P)^2 + B * P]$, where $P = [\max(F_o^2, 0) + 2F_c^2]/3$. The $A$ and $B$ parameters, as well as other experimental details are listed in table 1. All calculations were carried out using the SHELX97 program package [8]. Crystal data and details of the structure determination are given in table 1 and the determined bond lengths and bond angles in tables 2 and 3.

## 3. Results and discussion

Figure 1 shows the monomeric molecule of **1** with atom labelling; figure 2 shows the alignment of monomers in the unit cell. The bond distances and bond angles within the coordination octahedron around the Mg(II) ion are listed in table 2. The Mg(II) ion situated in the inversion centre is coordinated *trans* by two ligand molecules *via* their N,O bonding moieties [Mg–N3 2.194(2)Å′, Mg–O1 2.070(1)Å′]. Two, related by this inversion centre water oxygen atoms [Mg–O3 2.063(2)Å′] form the apices of the coordination octahedron. The bond lengths within the

Table 1. Crystal Data and Structure Refinement for $Mg(C_4H_3N_2O_2)_2(H_2O)_2$ and $[Zn(C_4H_3N_2O_2)_2(H_2O)_2][Zn(C_4H_3N_2O_2)_2(H_2O)] \cdot 3H_2O$.

| Compound | $Mg(C_4H_3N_2O_2)_2(H_2O)_2$ | $Zn_2(C_4H_3N_2O_2)_4(H_2O)_3 \cdot 3H_2O$ |
|---|---|---|
| Shape | Rectangular blocks | Rectangular blocks |
| Colour | Pale yellow | Colourless |
| Empirical formula | $C_8H_{10}N_4O_6Mg$ | $C_{16}H_{24}N_8O_{14}Zn_2$ |
| Formula weight | 282.51 | 683.17 |
| Temperature (K) | 293(2) | |
| Wavelength (Mo-Kα) (Å) | 0.71073 | |
| Crystal system | Monoclinic | Triclinic |
| Space group | $P2_1/c$ | $P(\bar{1})$ |
| Unit cell dimensions (Å, °, Å$^3$) | $a = 6.748(1)$, $\alpha = 90$ | $a = 8.024(2)$, $\alpha = 77.98(3)$ |
| | $b = 10.258(2)$, $\beta = 102.83(3)$ | $b = 11.585(2)$, $\beta = 81.18(3)$ |
| | $c = 7.337(1)$, $\gamma = 90$ | $c = 13.889(3)$, $\gamma = 88.69(3)$ |
| | $V = 591.81$ | $V = 1248.27$ |
| $Z$ | 4 | 2 |
| Calculated density (g cm$^{-3}$) | 1.585 | 1.818 |
| $\mu$ (Mo-Kα) (mm$^{-1}$) | 0.18 | 2.01 |
| $F(000)$ | 292.0 | 696.0 |
| Crystal size (mm$^3$) | 0.11 × 0.13 × 0.18 | 0.07 × 0.10 × 0.21 |
| $\theta_{min}$, $\theta_{max}$ (°) | 2.52–30.06 | 2.52–28.43 |
| Index range | $-9 \le h \le 9$, $-17 \le k \le 0$, $0 \le l \le 10$ | $-10 \le h \le 10$, $-15 \le k \le 0$, $-17 \le l \le 17$ |
| Total data | 1715 | 6097 |
| Observed data $[I > 4\sigma(I)]$ | 875 | 3677 |
| $R_{int}$ | 0.0125 | 0.0316 |
| No. of parameters | 108 | 445 |
| Goodness-of-fit on $F^2$ | 0.981 | 1.010 |
| Final $R_1$ $[I > 4\sigma(I)]$ | 0.0371 | 0.0297 |
| Final $wR_2$ index | 0.1284 | 0.0984 |
| Absorption correction | Analytical [6] | |
| Min. and max. transmission | 0.97138, 0.997790 | 0.77045, 0.84885 |
| Extinction correction | None | |
| Largest diff. peak and hole (e Å$^{-3}$) | 0.36, −0.19 | 0.63, −0.56 |
| Weight parameters ($A$, $B$) | 0.0764, 0.00 | 0.0558, 0.00 |
| Mean shift/esd | 0.000 | 0.000 |

Table 2. Selected interatomic distances (Å) and bond angles (°) for $Mg(C_4H_3N_2O_2)_2(H_2O)_2$.

| Mg(II) coordination | | | |
|---|---|---|---|
| Mg–O1 | 2.070(1) | O1–Mg–N3 | 78.43(6) |
| Mg–N3 | 2.193(2) | O1–Mg–O3 | 89.14(6) |
| Mg–O3 | 2.063(6) | N3–Mg–O3 | 89.38(6) |

| Hydrogen bond system | | |
|---|---|---|
| D–H···A | d(D–H···A) | ∠(D–H–A) |
| O3–H31···O2$^{I}$ | 2.747(2) | 171(2) |
| O3–H32···O2$^{II}$ | 2.728(2) | 170(3) |
| N1–H1···O2$^{III}$ | 2.991(3) | 149(4) |

Symmetry code: $^{I}x, -x+1/2; z+1/2$; $^{II}-x, y-1/2, -z+1/2$; $^{III}x+1, -y+1/2, z+1/2$.

octahedron are typical for Mg(II) complexes with carboxylate ligands. The imidazole ring is planar [r.m.s. 0.0021(1)Å′], the carboxylic group C6O1O2 deviates by 2.7(1)°. Monomeric molecules have been also reported in the structures of a number of Mg(II) complexes with monocarboxylate ligands, such as

Table 3. Selected interatomic distances (Å) and bond angles (°) for $[Zn(C_4H_3N_2O_2)_2(H_2O)_2][Zn(C_4H_3N_2O_2)_2(H_2O)] \cdot 3H_2O$.

| | | | |
|---|---|---|---|
| Zn1 coordination | | | |
| Zn1–O11 | 2.146(20) | O11–Zn1–N13 | 78.93(9) |
| Zn1–N13 | 2.048(2) | N13–Zn1–O21 | 98.74(8) |
| Zn1–O21 | 2.157(2) | O21–Zn1–N23 | 79.33(9) |
| Zn1–N23 | 2.040(2) | N23–Zn1–O11 | 103.05(9) |
| Zn1–O15 | 2.204(3) | O15–Zn1–O16 | 177.51(9) |
| Zn1–O16 | 2.154(3) | O15–Zn1–O11 | 90.34(9) |
| | | O15–Zn1–N13 | 86.74(10) |
| | | O16–Zn1–O21 | 87.85(9) |
| | | O16–Zn1–N23 | 89.96(11) |
| Zn2 coordination | | | |
| Zn2–O31 | 2.104(2) | O31–Zn2–N33 | 80.91(9) |
| Zn2–N33 | 2.032(2) | N33–Zn2–O41 | 89.46(9) |
| Zn2–O41 | 2.109(2) | O41–Zn2–N43 | 79.83(9) |
| Zn2–N43 | 2.048(3) | N43–Zn2–O31 | 98.50(9) |
| Zn2–O33 | 2.002(3) | O33–Zn2–O31 | 107.92(10) |
| | | O33–Zn2–N33 | 105.78(10) |
| | | O33–Zn2–O41 | 97.01(10) |
| | | O33–Zn2–N43 | 99.95(10) |
| Hydrogen bond system | | | |
| D–H⋯A | d(D–H⋯A) | ∠(D–H–A) | |
| O15–H151⋯O32 | 2.760(3) | 166(5) | |
| O15–H152⋯O51$^{\mathrm{II}}$ | 2.660(4) | 172(4) | |
| O16–H161⋯O53 | 2.724(4) | 172(4) | |
| O16–H162⋯O21$^{\mathrm{V}}$ | 2.756(3) | 172(5) | |
| O33–H331⋯O52 | 2.602(4) | 168(4) | |
| O33–H332⋯O22$^{\mathrm{I}}$ | 2.662(3) | 173(4) | |
| O51–H511⋯O32$^{\mathrm{VI}}$ | 2.931(4) | 168(5) | |
| O51–H512⋯O11$^{\mathrm{V}}$ | 2.737(3) | 172(6) | |
| O52–H521⋯O41$^{\mathrm{III}}$ | 2.774(4) | 176(10) | |
| O52–H522⋯O12 | 2.685(4) | 163(8) | |
| O53–H531⋯O15$^{\mathrm{III}}$ | 2.874(4) | 154(8) | |
| O53–H532⋯O42$^{\mathrm{IV}}$ | 2.805(4) | 166(5) | |
| N11–H11⋯O42$^{\mathrm{VII}}$ | 2.801(3) | 152(4) | |
| N21–H21⋯O32$^{\mathrm{IX}}$ | 2.951(3) | 172(6) | |
| N31–H31⋯O22$^{\mathrm{VI}}$ | 2.733(3) | 161(3) | |
| N41–H41⋯O12$^{\mathrm{VIII}}$ | 2.751(3) | 164(4) | |

Symmetry code: $^{\mathrm{I}}x, y, z+1$; $^{\mathrm{II}}x, y+1, z$; $^{\mathrm{III}}x-1, y, z$; $^{\mathrm{IV}}x-1, y, z-1$; $^{\mathrm{V}}-x+1, -y+1, -z+1$; $^{\mathrm{VI}}-x+2, -y+1, -z+1$; $^{\mathrm{VII}}-x+2, -y+1, -z+2$; $^{\mathrm{VIII}}-x+1, -y+2, -z+2$; $^{\mathrm{IX}}-x+1, -y+2, -z+1$.

*cis*-bisaquodi(pyridine-2-carboxylato-N,O)-magnesium(II) dihydrate [8], *cis*-bisaquodi (3-aminopyrazine-2-carboxylato-N,O)-magnesium(II) hydrate [2] and *trans*-bisaquodi (pyridazine-3-carboxylato-N,O)-magnesium(II) dihydrate [5].

The monomers with Mg(II) ions in the inversion centres at 0, 0, 0 and 0, 1/2, 1/2 form molecular sheets in which they are held together by hydrogen bonds of 2.728(2)Å′ and 2.747(2)Å′ (see figure 2). The coordinated water molecules O3 act as donors, the acceptors are carboxylate oxygen atoms O2 of monomers belonging to the same sheet. In addition, protonated imidazole ring nitrogen atoms act as donors in rather weak bonds (2.991(2)Å′) to carboxylate oxygen atoms O2 in the monomers forming adjacent sheets. Table 2 lists the geometrical parameters of the observed hydrogen bonds.

The structure of **2** contains two types of monomeric molecules with different zinc coordination modes. Figure 3 shows both types with atom labelling scheme. In the Type 1 monomer the metal ion is coordinated by two N,O bonding moieties belonging to different *trans* oriented ligand molecules and two water oxygen atoms which form the

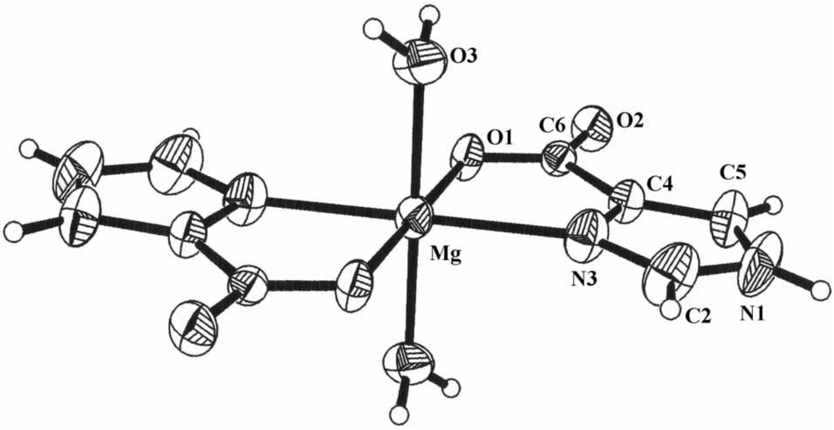

Figure 1. A view of a $Mg(C_4H_3N_2O_2)_2(H_2O)_2$ molecule with atomic labelling. Displacement ellipsoids are drawn at the 50% probability level.

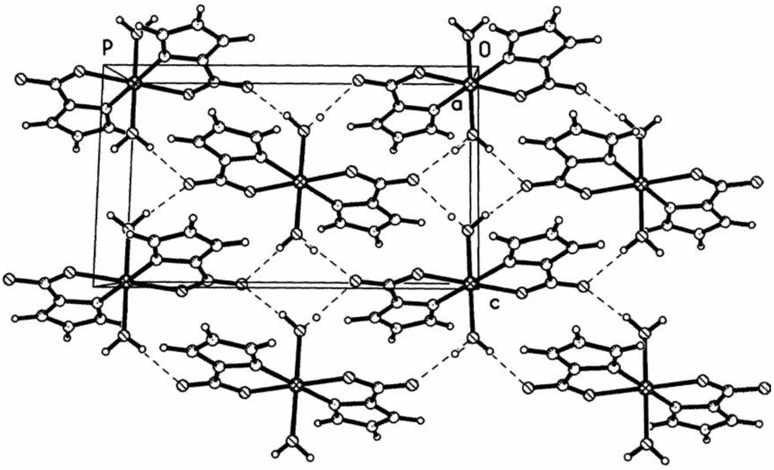

Figure 2. The alignment of $Mg(C_4H_3N_2O_2)_2(H_2O)_2$ molecules in the unit cell. For clarity only half of the molecules with Mg(II) ions at (0, 0, 0) and at (1/2, 1/2, 0) are shown. Hydrogen bonds are marked by dashed lines.

apices of a fairly regular octahedron. The relevant Zn–O and Zn–N bond distances and bond angles are listed in table 2. They are typical for Zn complexes with azole and azine carboxylate ligands. The imidazole rings of both ligands denoted as 1 and 2 are planar with r.m.s of 0.0012(1)Å′ and 0.0035(2)Å′, respectively. The plane of the carboxylic group C16O11O12 deviates from the the hetero-ring plane 1 by 1.3(1)°, the plane of the C26O21O22 group by 2.7(1)°. The planes of the imidazole rings 1 and 2 make a dihedral angle of 26.8(1)° to each other. The symmetry independent Zn2 ion which forms the center of the Type 2 monomer shows pentacoordination (see figure 3). Two ligand molecules denoted as 3 and 4 coordinate their N,O bonding moieties. A single water oxygen atom (O33) completes the number of coordinated atoms to five. The imidazole rings of the ligand molecules 3 and 4 are planar with r.m.s of 0.0021(1)Å′

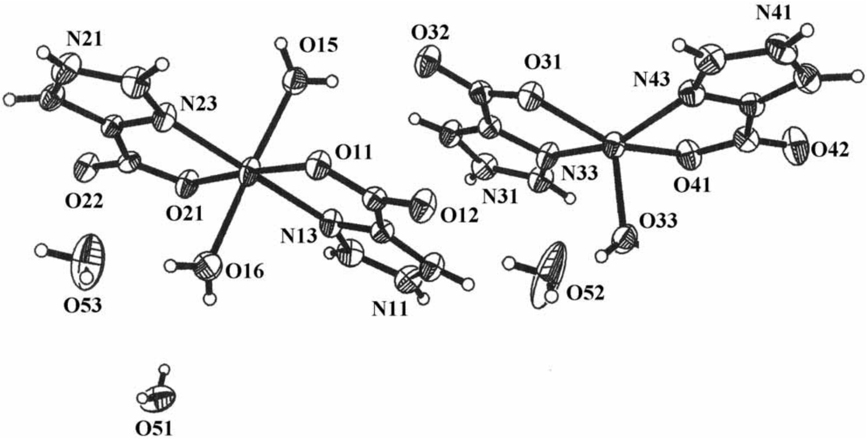

Figure 3. Molecules forming the $[Zn(C_4H_3N_2O_2)_2(H_2O)_2][Zn(C_4H_3N_2O_2)_2(H_2O)] \cdot 3H_2O$ showing the atom labelling scheme. Displacement ellipsoids are at the 50% probability level.

and 0.0015(1)Å′, respectively. The corresponding carboxylic groups deviate from the ring plane by 3.4(1)° in the case of ligand 3 and by 4.3(1)° in the case of ligand 4. The coordination polyhedron around the Zn2 ion is a distorted pyramid with nearly planar [r.m.s. 0.0062(1)Å′] base composed of O31, N33, O41 and N43 atoms (see figure 3). An inspection of table 3 shows that the O31–Zn2–N33, O31–Zn2–N43, N43–Zn2–O41 and O41–Zn2–N33 bond angles do not deviate much from 90°, so that the base approaches a square configuration. The Zn2 and the O33 atoms are above the mean plane by 0.458(1)Å′ and 2.450(1)Å′, respectively.

Pentacoordination is fairly common among Zn(II) complexes [9]. A distorted trigonal bipyramid has been reported as the coordination polyhedron in the structures of a number of complexes with large organic ligands [10, 11]. The coordination mode and the geometry of Zn(II) complex molecules results from such factors as lattice forces produced by specific modes of molecular packing in the crystal, the steric effects due to the size and shape of the ligand molecules and the mutual repulsion of electronegative donor atoms. It seems that the first two factors show negligible influence on the coordination mode of the Type 2 molecule since the packing is rather loose and the size of complex molecule is small, so that the lack of the second apical water molecule may be attributed to a polarization of the 3d electron shell which is too small to enforce the typical pentacoordination geometry a more or less distorted trigonal bipyramid.

Figure 4 shows the packing of the monomers. Pentacoordinated and hexacoordinated Zn(II) monomers form molecular sheets aligned approximately along the (001) plane. The monomers forming a sheet and belonging to adjacent sheets interact through an extended system of hydrogen bonds with $D\text{–}H \cdots A$ bond distances in the range from 2.602(3) to 2.951(3)Å′. Coordinated water molecules, solvation water molecules as well as protonated imidazole ring nitrogen atoms act as donors, the acceptors are mostly the carboxylate oxygen atoms. The detailed geometrical parameters are listed in table 3.

The coordination of the Zn(II) ion by a N,O bonding moiety is also observed in the structures of its complexes with azine carboxylate ligands such as *cis*-bisaquadi(pyridine-3-carboxylato-N,O)-zinc(II) tetrahydrate [12], *trans*-bisaquadi

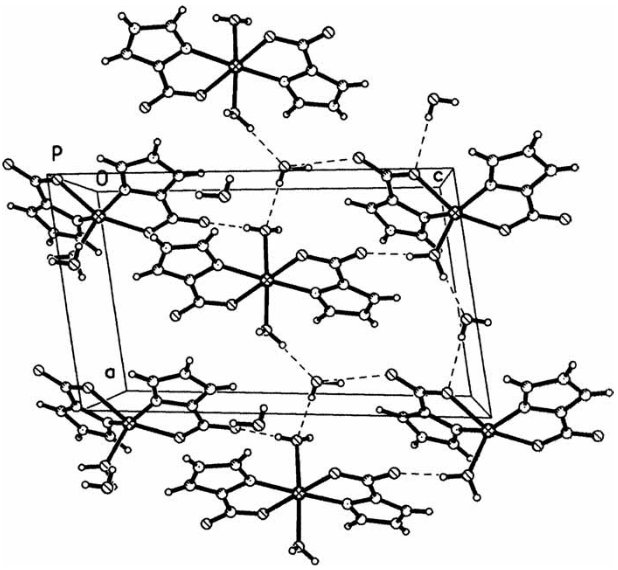

Figure 4. Packing diagram of $[Zn(C_4H_3N_2O_2)_2(H_2O)_2][Zn(C_4H_3N_2O_2)_2(H_2O)] \cdot 3H_2O$. Hydrogen bonds are indicated by dashed lines.

(pyrazine-2-carboxylato-N,O)-zinc(II) [1], triclinc *trans*-bisaquadi(pyridazine-3-carboxylato-N,O)-zinc(II) tetrahydrate [3] and monoclinic *trans*-bisaquodi(pyridazine-3-carboxylato-N,O)-zinc(II) [4]. In all of them the zinc ion is coordinated octahedrally.

The results of the present study support an analysis that Mg(II) shows distinct preference for octahedral coordination, while the Zn(II) ion tends to aquire coordination numbers 4, 5, and 6 with the same probability [9]. An unexpected feature of the structure compound **2** is a simultaneous occurrence of hexa-and pentacoordinated complex molecules, the latter with a rarely observed square pyramidal coordination geometry instead of a trigonal bipyramid. The other result is the confirmation of a regularity frequently observed in the structures of metal complexes with azine carboxylate ligands – the coordination via a N,O bonding moiety when the hetero-ring nitrogen atom and the carboxylate group are close to each other.

Only one structure of a divalent metal complex with the 4-IMC ligand has been previously reported: bis(imidazole-4-carboxylate-N,O)copper(II) which contains monomeric molecules with copper ion chelated by the N,O bonding moieties of two ligand molecules in a planar tetracoordination mode [13]. Catenated molecular pattern has been recently discovered in the structure of a calcium(II) complex with Ca(II) ions bridged by two symmetry independent 4-IMC ligand molecules. Ca(II) ion is octacoordinated in a bicapped pentagonal bipyramid with a strongly deformed equatorial plane [14].

## Supplementary material

Crystallographic data (excluding structure factors) for the structures reported in this article have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication Nos CCDC 6000590 and 600591. Copies of the available material can be obtained, free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-(0)1223-336033 or Email: teched@chemcrys.cam.ac.uk).

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