Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

catena-Poly[[aquacalcium(II)]bis(μ-1*H*-imidazole-4-carboxylato)-$\kappa^4N,O:O,O';\kappa^3O,O':O'$]

Wojciech Starosta and Janusz Leciejewicz*

Institute of Nuclear Chemistry and Technology, Dorodna 16, 03-195 Warszawa, Poland

Correspondence e-mail: jlec@orange.ichtj.waw.pl

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.124
Data-to-parameter ratio = 16.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

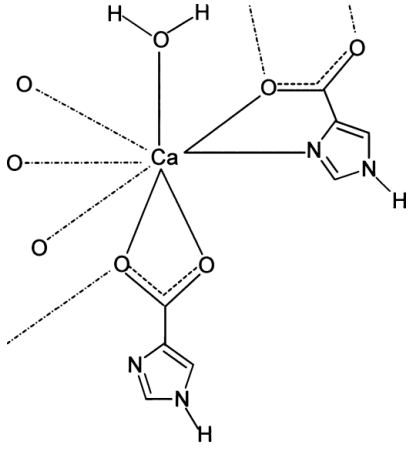
Received 20 August 2006
Accepted 13 September 2006

The structure of the title compound, $[Ca(C_4H_3N_2O_2)_2(H_2O)]_n$, is composed of molecular ribbons in which the Ca^{II} ions are bridged by two symmetry-independent imidazole-4-carboxylate ligands. Each Ca^{II} ion is coordinated by the *N*,*O*-bonding group of one ligand, two carboxylate O atoms of the other ligand, an O atom of the coordinated water molecule and three bridging carboxylate O atoms donated by both ligands. The coordination polyhedron of the Ca^{II} ion (coordination number 8) is a bicapped pentagonal bipyramid with a strongly deformed equatorial plane. The ribbons are held together by a network of hydrogen bonds.

Comment

The structure of compound (I) is composed of molecular ribbons in which Ca^{II} ions are bridged by two symmetry-independent imidazole-4-carboxylate ligands. The bridging pathways operate in two planes. In one, three Ca ions are bridged by atoms belonging to the ligand '1'; the Ca ion is coordinated by the *N*,*O*-bonding group composed of atoms N13 and O11. However, atom O11, acting in bidentate mode, is also bonded to an adjacent Ca ion, while atom O12 atom of this carboxylate group is bonded to a third Ca ion. The other bridging pathway is formed by carboxylate atoms O21 and O22 which coordinate the Ca ion. Atom O22, acting in bidentate mode, is also linked to a fourth Ca ion (for symmetry codes and bond lengths, see Table 1). Molecular ribbons are formed across successive inversion centres propagating in the direction of the *a* axis. Fig. 1 shows the content of the asymmetric unit with the atom labelling scheme; the bridging O atoms are also shown. A single molecular ribbon aligned along the *a* axis is displayed in Fig. 2. One water molecule, O31, is coordinated to the metal ion [Ca—O31 = 2.412 (1) Å]. The calcium ion is coordinated by eight atoms: the hetero-ring N13, the carboxylate O11, two carboxylate O21 and O22, the water O31 and three bridging atoms $O11^{i}$, $O12^{ii}$, $O22^{iii}$. The coordination polyhedron is a pentagonal bipyramid with O31 at the single apex on one side and O21 and O22 forming two apices on the other side of a strongly deformed equatorial pentagonal plane composed of atoms N13, $O11^{i}$, O11, $O22^{iii}$ and $O12^{ii}$. The maximum deviations from the mean equatorial plane are −0.5435 Å (atom O11) and +0.5319 Å (atom $O22^{iii}$); the r.m.s. deviation is 0.3401 Å. The observed Ca—O and Ca—N bonds distances fall in the range commonly observed in Ca complexes with carboxylate ligands (Einspahr & Bugg, 1981). The atoms forming the ligand molecules are coplanar with r.m.s. deviations of 0.0486 and 0.0147 Å, for ligands '1' and '2', respectively. Their planes form an angle of 80.9 (1)°. Bond distances and angles within both ligands agree well with those observed in the structures

of magnesium(II) and zinc(II) complexes with the imidazole-4-carboxylate ligand (Gryz *et al.* 2006). Adjacent ribbons are held together by hydrogen bonds operating between the coordinated water molecules O31 and the carboxylate O atoms as well as between hetero-ring N11 and N21 atoms acting as donors and carboxylate O atoms as acceptors, thus giving rise to weak bonding pathways in the directions of the *b* and *c* axes. An intra-ribbon hydrogen bond with coordinated water O31 as a donor and the non-coordinated hetero-ring N23 acting as an acceptor is also observed. Geometrical details of the hydrogen-bond network are given in Table 2.



(I)

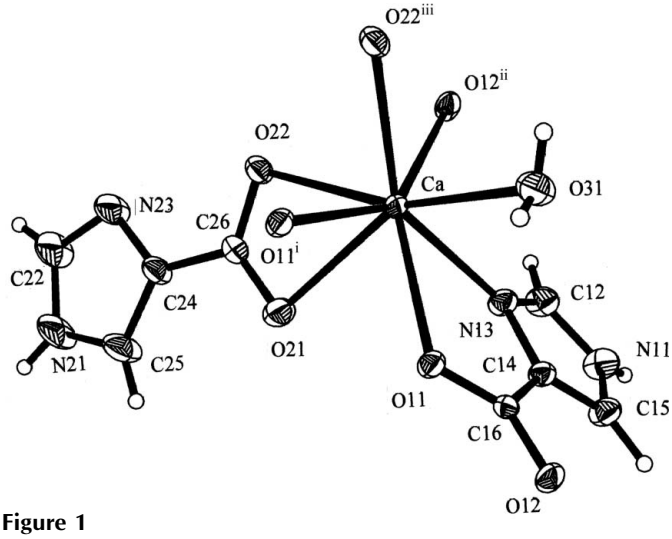


Figure 1
The asymmetric unit of compound (I), together with additional atoms to complete the Ca coordination, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes as in Table 1.

Experimental

Calcium(II) oxide was added in small portions to 50 ml of a hot aqueous solution containing 2 mmol of imidazole-4-carboxylic acid (Aldrich) until a small amount of the oxide remained undissolved. After boiling for 2 h with constant stirring the solution was filtered and left to crystallize at room temperature. After several days, single crystals in the form of colourless blocks deposited in the mother liquid. They were washed with cold water and ethanol, and dried in air.

Crystal data

$[Ca(C_4H_3N_2O_2)_2(H_2O)]$
M_r = 280.26
Monoclinic, $P2_1/c$
a = 6.2930 (13) Å
b = 19.784 (4) Å
c = 8.7620 (18) Å
β = 99.24 (3)°
V = 1076.7 (4) Å^3
Z = 4
D_x = 1.729 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 293 (2) K
Rectangular blocks, colourless
0.40 × 0.29 × 0.10 mm

Data collection

Kuma KM-4 four-circle diffractometer
$\omega/2\theta$ scan
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2000)
T_{min} = 0.692, T_{max} = 0.871
3406 measured reflections
3147 independent reflections
2592 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 30.1°
3 standard reflections
every 200 reflections
intensity decay: 2.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.124
S = 1.06
3147 reflections
195 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0857P)^2 + 0.4634P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.97 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ca1—O11^{i}	2.3836 (13)	Ca1—N13	2.5082 (16)
Ca1—O31	2.4120 (15)	Ca1—O11	2.5117 (13)
Ca1—O12^{ii}	2.4522 (14)	Ca1—O21	2.5273 (15)
Ca1—O22^{iii}	2.4646 (14)	Ca1—O22	2.5297 (13)
O11^{i}—Ca1—O31	94.42 (5)	O31—Ca1—N13	81.37 (5)
O31—Ca1—O12^{ii}	96.82 (5)	O12^{ii}—Ca1—N13	77.89 (5)
O11^{i}—Ca1—O22^{iii}	73.75 (4)	O11^{i}—Ca1—O11	72.90 (5)
O31—Ca1—O22^{iii}	76.69 (5)	O31—Ca1—O11	69.75 (5)
O12^{ii}—Ca1—O22^{iii}	75.62 (5)	N13—Ca1—O11	66.35 (5)

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N21—H21···O12^{iv}	0.88 (4)	1.95 (4)	2.825 (2)	175 (3)
O31—H31···O21^{i}	0.85 (4)	2.01 (4)	2.844 (2)	168 (3)
N11—H11···O22^{v}	0.86 (3)	2.29 (3)	2.995 (2)	140 (3)
O31—H32···N23^{iii}	0.79 (4)	1.95 (4)	2.743 (2)	178 (4)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (iii) $-x, -y+1, -z+1$; (iv) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x, -y+1, -z$.

H atoms were refined independently with isotropic displacement parameters [C—H = 0.88 (3)–0.96 (3) Å].

Data collection: *KM-4 Software* (Kuma, 1996); cell refinement: *KM-4 Software*; data reduction: *DATAPROC* (Kuma, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997);

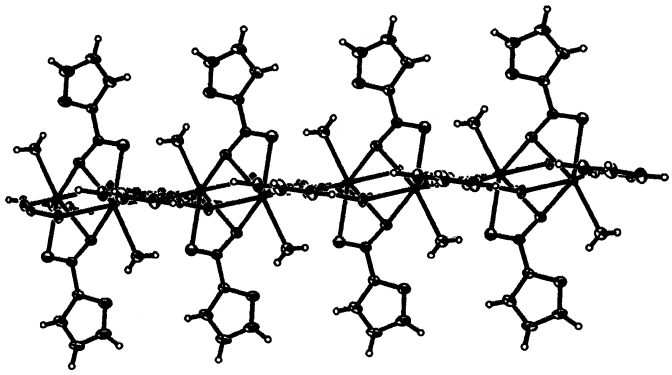


Figure 2
A single molecular ribbon of compound (I), propagating in the direction of the *a* axis as viewed along the *c* axis.

program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1992); software used to prepare material for publication: *SHELXL97*.

References

Einspahr, H. & Bugg, C. E. (1981). *Acta Cryst.* B**37**, 1044–1052.
Gryz, M., Starosta, W. & Leciejewicz, J. (2006). *J. Coord. Chem.* In the press.
Kuma (1996). *KM-4 Software*. Kuma Diffraction, Wroclaw, Poland.
Kuma (2001). *DATAPROC*. Version 10.0.7. Kuma Diffraction, Wroclaw, Poland.
Oxford Diffraction (2000). *CrysAlis RED*. Version 1.69. Oxford Diffraction Ltd., Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1992). *XP*. Version 4.3 Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.