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Poly[aqua(μ_3-3,5-dinitrosalicylato)calcium(II)]

Wen-Dong Song,[a] Xian-Xia Guo[a] and Chao-Hua Zhang[b]*

[a]College of Science, Guang Dong Ocean University, Zhan Jiang 524088, People's Republic of China, and [b]School of Food Science and Technology, Guang Dong Ocean University, Zhan Jiang 524088, People's Republic of China

Correspondence e-mail: zhangch60@126.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.127
Data-to-parameter ratio = 14.1

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

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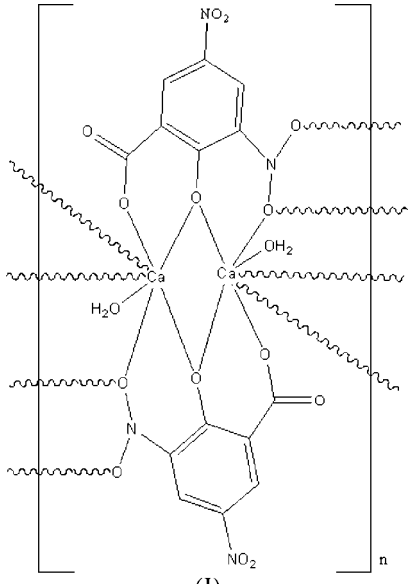
In the title coordination polymer, $[Ca(C_7H_2N_2O_7)(H_2O)]_n$, the Ca^{II} atom is seven-coordinate by six O atoms from four 3,5-dinitrosalicylatate ligands and one water molecule, and displays a distorted pentagonal–bipyramidal geometry. Centrosymmetrically related dinuclear calcium units with a Ca···Ca separation of 3.8665 (8)Å form layers which are self-assembled into a three-dimensional network by intermolecular O—H···O hydrogen bonds.

Comment

Molecular self-assembly of supramolecular architectures has received much attention during recent decades (Kim *et al.*, 2003; Iglesias *et al.*, 2003; Moulton & Zaworotko, 2001). The structures and properties of such systems depend on the coordination and geometric preferences of both the central metals ions and bridging building blocks as well as the influence of weaker non-covalent interactions, such as hydrogen bonds and π–π stacking interactions. 3,5-Dinitrosalicylate, which can act as a multidentale ligand (Vincent *et al.*, 1986), shows versatile binding modes and coordination properties. The crystal structure of a zinc complex based on 3,5-dinitrosalicylate (Song & Xi, 2006) has been reported recently. In this paper we report the crystal structure of the title compound, (I), a new Ca complex obtained by reaction of 3,5-dinitrosalicylic acid with $CaCl_2$.



(I)

As illustrated in Fig. 1, in the asymmetric unit of (I) a Ca metal centre is coordinated by six O atoms from four 3,5-

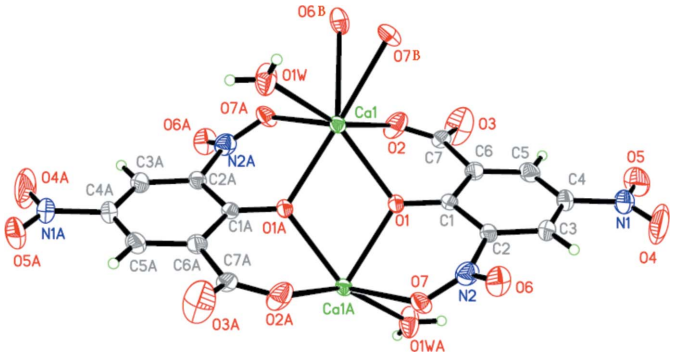


Figure 1
Part of the polymeric structure of (I), showing the atomic numbering scheme. Non-H atoms are shown as 50% probability displacement ellipsoids. [Symmetry codes: (A) $1-x, -y, -z$; (B) $1-x, 1-y, -z$.]

dinitrosalicylate ligands and the O atoms of a water molecule in a distorted pentagonal bipyramidal geometry (Table 1). Centrosymmetrically related dinuclear units with a Ca···Ca separation of 3.8665 (8)Å are assembled into ruffled layers parallel to the *bc* plane. The layers are further linked into a three-dimensional supramolecular network *via* intermolecular O—H···O hydrogen interactions (Table 2 and Fig. 2).

Experimental

The title complex was prepared by the addition of a stoichiometric amount of calcium chloride (20 mmol) to a hot aqueous solution of 3,5-dinitrosalicylic acid (20 mmol). The pH was then adjusted to 7.0 with NaOH (30 mmol). The resulting solution was filtered, and yellow single crystals suitable for X-ray analysis were obtained over several days on slow evaporation of the solvent at room temperature (yield: 58%).

Crystal data

$[Ca(C_7H_2N_2O_7)(H_2O)]$
M_r = 284.20
Monoclinic, $P2_1/c$
a = 12.6381 (4) Å
b = 6.6650 (3) Å
c = 12.4126 (5) Å
β = 109.391 (2)°
V = 986.24 (7) Å^3
Z = 4
D_x = 1.914 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.68 mm^{-1}
T = 293 (2) K
Prism, yellow
0.25 × 0.22 × 0.20 mm

Data collection

Bruker APEX-II area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.849, T_{max} = 0.876
9924 measured reflections
2301 independent reflections
2059 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.7°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.127
S = 1.07
2301 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0756P)^2 + 1.0265P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.76 e Å^{-3}
$\Delta\rho_{min}$ = −0.85 e Å^{-3}

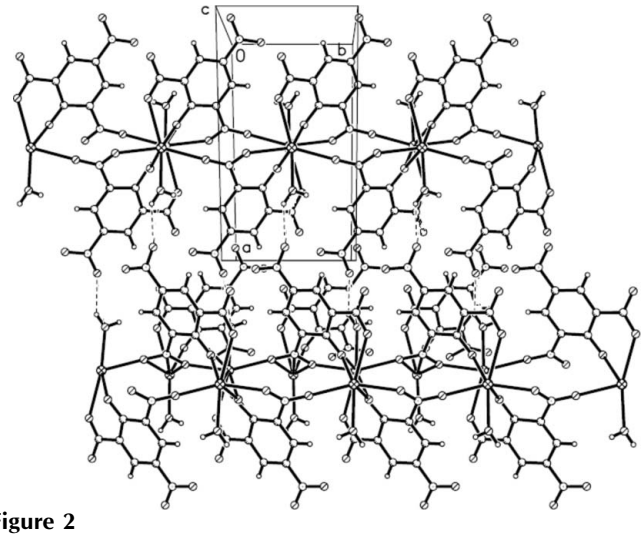


Figure 2
A packing diagram of (I), showing the intermolecular hydrogen bonds as dashed lines.

Table 1
Selected geometric parameters (Å, °).

Ca1—O1^{i}	2.3386 (14)	Ca1—O1*W*	2.408 (2)
Ca1—O6^{ii}	2.3407 (17)	Ca1—O7^{iii}	2.4210 (15)
Ca1—O7^{i}	2.3476 (16)	Ca1—O2	2.5999 (19)
Ca1—O1	2.3662 (15)	Ca1—O6^{iii}	3.017 (2)
O1^{i}—Ca1—O6^{ii}	97.45 (6)	O7^{i}—Ca1—O1	114.64 (6)
O1^{i}—Ca1—O7^{i}	73.49 (5)	O1^{i}—Ca1—O1*W*	87.73 (7)
O6^{ii}—Ca1—O7^{i}	158.05 (7)	O6^{ii}—Ca1—O1*W*	77.42 (7)
O1^{i}—Ca1—O1	69.47 (6)	O7^{i}—Ca1—O1*W*	82.21 (7)
O6^{ii}—Ca1—O1	79.07 (6)	O1—Ca1—O1*W*	144.57 (7)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2*W*···O5^{iv}	0.81	2.19	2.889 (3)	144
O1*W*—H1*W*···O2^{v}	0.82	2.18	2.924 (3)	153

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

H atoms were placed in calculated positions with C—H = 0.93 Å, O—H = 0.82 Å, and refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(O).

Data collection: *SMART* (Bruker,1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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