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# *catena*-Poly[[diaquacalcium(II)]-bis(*μ*-quinoline-3-carboxylato)]

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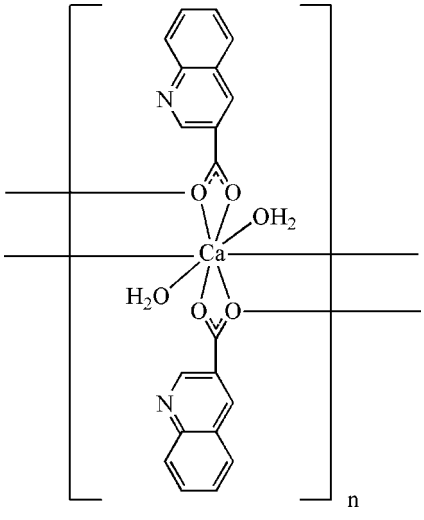
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.035; $wR$ factor = 0.090; data-to-parameter ratio = 13.0.

In the title complex, $[Ca(C_{10}H_6NO_2)_2(H_2O)_2]_n$, the $Ca^{II}$ ion is eight-coordinated by six carboxylate O atoms from four separate quinoline-3-carboxylate ligands, two of which are bidentate chelate and two bridging, and two water molecules in a distorted square-antiprismatic geometry. The bridging groups form a polymeric chain substructure extending along the $c$ axis, the chains being connected by coordinated-water O—H···N and O—H···$O_{carboxylate}$ hydrogen bonds into a three-dimensional framework structure.

## Related literature

For the potential uses and diverse structural types of metal complexes with the quinoline-3-carboxylate ligand, see: Hu *et al.* (2007). For related structures, see: Martell & Smith (1974); Haendler (1986, 1996); Okabe & Koizumi (1997); Okabe & Makino (1998, 1999); Okabe & Muranishi (2002); Odoko *et al.* (2001).




## Experimental

### Crystal data

$[Ca(C_{10}H_6NO_2)_2(H_2O)_2]$
$M_r$ = 420.43
Monoclinic, $P2_1/c$
$a$ = 16.0115 (16) Å
$b$ = 15.3636 (16) Å
$c$ = 7.7962 (8) Å
$\beta$ = 97.928 (1)°
$V$ = 1899.5 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.37 mm$^{-1}$
$T$ = 296 K
0.30 × 0.26 × 0.25 mm

### Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.897, $T_{max}$ = 0.913
9735 measured reflections
3411 independent reflections
2433 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.090
$S$ = 1.02
3411 reflections
262 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2$W$—H4$W$···N2$^{i}$ | 0.88 | 2.01 | 2.880 (3) | 170 |
| O2$W$—H3$W$···O1$^{ii}$ | 0.92 | 1.92 | 2.813 (2) | 163 |
| O1$W$—H2$W$···N1$^{iii}$ | 0.72 | 2.18 | 2.885 (2) | 165 |
| O1$W$—H1$W$···O4$^{iv}$ | 0.84 | 1.94 | 2.785 (2) | 174 |

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2067).

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# supporting information

*Acta Cryst.* (2010). E**66**, m1441–m1442 [https://doi.org/10.1107/S1600536810039401]

## *catena*-Poly[[diaquacalcium(II)]-bis(*μ*-quinoline-3-carboxylato)]

**Dong-Liang Miao, Shi-Jie Li, Wen-Dong Song, Juan-Hua Liu and Xiao-Fei Li**

### S1. Comment

Design and synthesis of metal-organic complexes have attracted extensive attention in coordination chemistry. Quinoline-2-carboxylic acid, which is a tryptophan metabolite (Martell & Smith, 1974) can be considered as a potential ligand and the crystal structures of a number of metal complexes containing the quinoline-2-carboxylate ligand have been determined, e.g. with $Mn^{II}$ (Haendler, 1996; Okabe & Koizumi, 1997), $Cu^{II}$ (Haendler, 1986), $V^{IV}$ (Okabe & Muranishi, 2002). $Fe^{II}$ and $Co^{II}$ (Okabe & Makino, 1998, 1999), and $Ni^{II}$ (Odoko *et al.*, 2001). However, to the best of our knowledge, there are few crystal structures containing the quinoline-3-carboxylate ligand, one example being the coordination polymer with $Zn^{II}$ (Hu *et al.*,2007). In this paper, we report the synthesis and structure of a new $Ca^{II}$ complex obtained from the reaction of quinoline-3-carboxylic acid with $CaCl_2$ under hydrothermal condition, the title compound $[Ca(C_{20}H_{12}N_2O_4)(H_2O)_2]_n$ (I).

In the title complex molecule the $Ca^{II}$ atom is eight-coordinated by six carboxylate O atoms from four separate quinoline-2-carboxylate ligands (two bidentate chelate and two bridging) and two water O atoms, in a distorted square-antiprismatic environment (Fig. 1). The bridging carboxylate O atoms (O2 and O3) [Ca—O, 2.3877 (16), 2.3829 (16) Å] link separate $Ca^{II}$ centres forming a one-dimensional chain substructure extended along *c* (Fig.2). The chains are inter-connected by coordinated-water O—H···N and O—H···$O_{carboxylate}$ hydrogen bonds (Table 1) giving a three-dimensional framework structure (Fig.3).

### S2. Experimental

A mixture of $CaCl_2$ (0.02 g, 0.2 mmol) and quinoline-3-carboxylic acid (0.04 g, 0.2 mmol) in 12 ml of distilled water was sealed in an autoclave equipped with a Teflon liner (20 ml) and then heated at 394 K for 2 days. Crystals of the title compound were obtained by slow evaporation of the solvent at room temperature.

### S3. Refinement

Water H atoms were located in a difference Fourier map and were allowed to ride on the parent atom, with $U_{iso}$(H) = 1.5$U_{eq}$(O). Other H atoms were placed at calculated positions and were treated as riding on parent atoms with C—H = 0.96 Å and N—H = 0.86 Å and $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C, N).

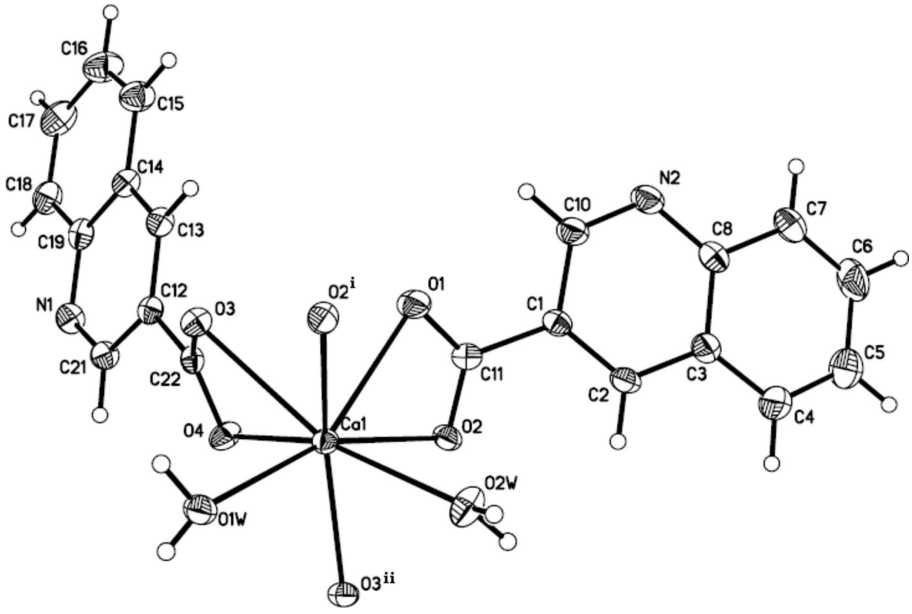


**Figure 1**

The structure of the title compound, showing the atomic numbering scheme. Non-H atoms are shown with 30% probability displacement ellipsoids.

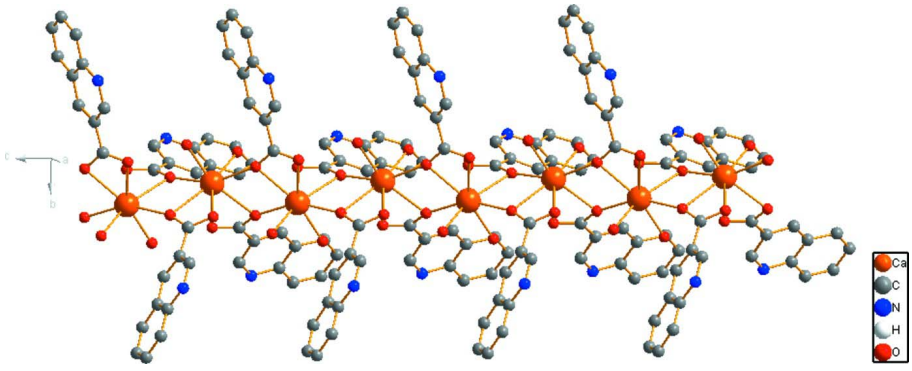


**Figure 2**

The one-dimensional chain substructure of (I) extending along *c*.

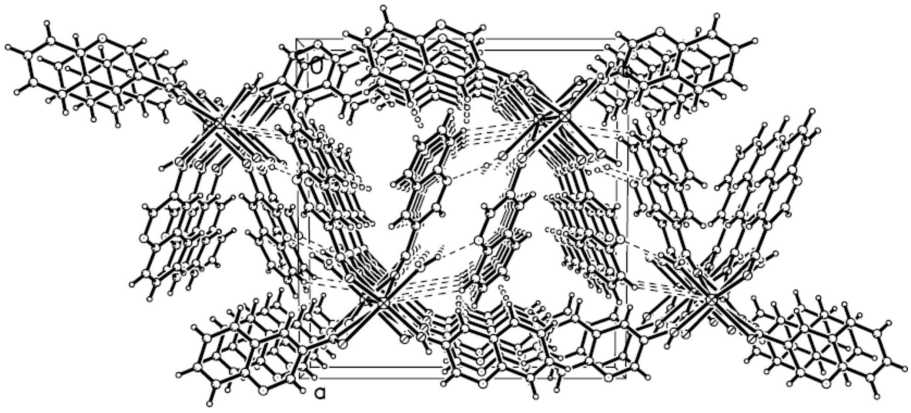

**Figure 3**

The three-dimensional hydrogen-bonded structure of (I).

**catena-Poly[[diaquacalcium(II)]-bis(μ-quinoline-3-carboxylato)]**

*Crystal data*

$[Ca(C_{10}H_6NO_2)_2(H_2O)_2]$
$M_r = 420.43$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 16.0115\ (16)$ Å
$b = 15.3636\ (16)$ Å
$c = 7.7962\ (8)$ Å
$\beta = 97.928\ (1)°$
$V = 1899.5\ (3)$ Å$^3$
$Z = 4$
$F(000) = 872$
$D_x = 1.470$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3600 reflections
$\theta = 1.4–25.0°$
$\mu = 0.37$ mm$^{-1}$
$T = 296$ K
Block, colorless
0.30 × 0.26 × 0.25 mm

*Data collection*

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.897$, $T_{max} = 0.913$
9735 measured reflections
3411 independent reflections
2433 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 25.2°$, $\theta_{min} = 1.9°$
$h = -19\rightarrow16$
$k = -18\rightarrow18$
$l = -9\rightarrow9$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.090$
$S = 1.02$
3411 reflections
262 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 0.6204P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Ca1 | 0.24944 (3) | 0.78080 (3) | 0.25589 (6) | 0.02366 (14) |
| C1 | 0.45779 (14) | 0.63295 (14) | 0.1201 (3) | 0.0284 (5) |
| N2 | 0.58144 (13) | 0.54488 (14) | 0.1998 (3) | 0.0420 (6) |
| C2 | 0.48749 (15) | 0.65988 (16) | −0.0265 (3) | 0.0349 (6) |
| H2 | 0.4562 | 0.6989 | −0.1008 | 0.042* |
| C10 | 0.50824 (16) | 0.57576 (17) | 0.2313 (3) | 0.0394 (6) |
| H10 | 0.4887 | 0.5587 | 0.3331 | 0.047* |
| C11 | 0.37390 (15) | 0.66290 (14) | 0.1614 (3) | 0.0278 (5) |
| C8 | 0.61064 (15) | 0.57047 (16) | 0.0508 (3) | 0.0360 (6) |
| C3 | 0.56531 (16) | 0.62891 (17) | −0.0660 (3) | 0.0374 (6) |
| C7 | 0.68710 (17) | 0.53586 (18) | 0.0112 (4) | 0.0471 (7) |
| H7 | 0.7182 | 0.4980 | 0.0885 | 0.057* |
| C4 | 0.59749 (19) | 0.6509 (2) | −0.2199 (4) | 0.0626 (9) |
| H4 | 0.5687 | 0.6903 | −0.2970 | 0.075* |
| C6 | 0.71558 (19) | 0.5574 (2) | −0.1385 (4) | 0.0634 (9) |
| H6 | 0.7659 | 0.5337 | −0.1638 | 0.076* |
| C5 | 0.6704 (2) | 0.6145 (3) | −0.2557 (5) | 0.0743 (11) |
| H5 | 0.6904 | 0.6279 | −0.3590 | 0.089* |
| N1 | 0.00972 (12) | 0.44091 (13) | 0.2997 (3) | 0.0352 (5) |
| C22 | 0.15016 (13) | 0.63803 (14) | 0.3447 (3) | 0.0244 (5) |
| C12 | 0.11785 (14) | 0.54892 (15) | 0.3760 (3) | 0.0272 (5) |
| C21 | 0.04213 (15) | 0.51849 (15) | 0.2802 (3) | 0.0323 (6) |
| H21 | 0.0134 | 0.5556 | 0.1982 | 0.039* |
| C13 | 0.16177 (15) | 0.49416 (16) | 0.4924 (3) | 0.0319 (6) |
| H13 | 0.2113 | 0.5129 | 0.5586 | 0.038* |
| C18 | 0.02177 (17) | 0.30129 (17) | 0.4363 (4) | 0.0423 (7) |
| H18 | −0.0301 | 0.2856 | 0.3752 | 0.051* |
| C14 | 0.13216 (16) | 0.40902 (15) | 0.5126 (3) | 0.0327 (6) |
| C19 | 0.05460 (16) | 0.38521 (15) | 0.4152 (3) | 0.0329 (6) |
| C17 | 0.0653 (2) | 0.24367 (19) | 0.5447 (4) | 0.0543 (8) |
| H17 | 0.0433 | 0.1884 | 0.5569 | 0.065* |
| C15 | 0.17626 (18) | 0.34683 (18) | 0.6242 (4) | 0.0470 (7) |
| H15 | 0.2280 | 0.3612 | 0.6876 | 0.056* |
| C16 | 0.1433 (2) | 0.26606 (19) | 0.6392 (4) | 0.0579 (9) |
| H16 | 0.1727 | 0.2254 | 0.7126 | 0.069* |

| | | | | |
|---|---|---|---|---|
| O3 | 0.18843 (9) | 0.67991 (10) | 0.47024 (19) | 0.0292 (4) |
| O4 | 0.14006 (10) | 0.66737 (10) | 0.1932 (2) | 0.0335 (4) |
| O1 | 0.36040 (11) | 0.66234 (11) | 0.3145 (2) | 0.0405 (4) |
| O2 | 0.32086 (9) | 0.69201 (10) | 0.0404 (2) | 0.0312 (4) |
| O1W | 0.15496 (10) | 0.88329 (10) | 0.3557 (2) | 0.0360 (4) |
| H1W | 0.1487 | 0.8714 | 0.4588 | 0.054* |
| H2W | 0.1156 | 0.9051 | 0.3282 | 0.054* |
| O2W | 0.34752 (10) | 0.88362 (11) | 0.1592 (2) | 0.0414 (5) |
| H3W | 0.3577 | 0.8785 | 0.0459 | 0.062* |
| H4W | 0.3751 | 0.9313 | 0.1964 | 0.062* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ca1 | 0.0245 (3) | 0.0257 (2) | 0.0204 (3) | −0.0003 (2) | 0.00171 (18) | 0.00021 (19) |
| C1 | 0.0283 (13) | 0.0289 (13) | 0.0266 (13) | 0.0030 (10) | −0.0005 (10) | −0.0037 (10) |
| N2 | 0.0387 (13) | 0.0447 (13) | 0.0421 (14) | 0.0147 (11) | 0.0040 (10) | 0.0051 (11) |
| C2 | 0.0309 (14) | 0.0390 (14) | 0.0332 (15) | 0.0070 (11) | −0.0014 (11) | 0.0048 (12) |
| C10 | 0.0383 (16) | 0.0442 (16) | 0.0357 (15) | 0.0123 (12) | 0.0049 (12) | 0.0048 (12) |
| C11 | 0.0328 (14) | 0.0238 (12) | 0.0261 (14) | 0.0012 (10) | 0.0009 (11) | −0.0020 (10) |
| C8 | 0.0306 (15) | 0.0365 (14) | 0.0398 (16) | 0.0041 (11) | 0.0015 (12) | −0.0056 (12) |
| C3 | 0.0332 (15) | 0.0424 (15) | 0.0370 (15) | 0.0036 (12) | 0.0057 (12) | 0.0014 (12) |
| C7 | 0.0353 (16) | 0.0483 (17) | 0.058 (2) | 0.0118 (13) | 0.0067 (14) | −0.0031 (14) |
| C4 | 0.051 (2) | 0.086 (2) | 0.056 (2) | 0.0174 (17) | 0.0216 (16) | 0.0237 (18) |
| C6 | 0.0412 (19) | 0.077 (2) | 0.076 (2) | 0.0149 (17) | 0.0228 (17) | −0.006 (2) |
| C5 | 0.057 (2) | 0.111 (3) | 0.062 (2) | 0.017 (2) | 0.0312 (18) | 0.017 (2) |
| N1 | 0.0315 (12) | 0.0328 (12) | 0.0405 (13) | −0.0059 (9) | 0.0020 (10) | −0.0042 (10) |
| C22 | 0.0215 (12) | 0.0274 (12) | 0.0248 (13) | −0.0006 (10) | 0.0051 (10) | −0.0045 (10) |
| C12 | 0.0283 (13) | 0.0287 (12) | 0.0250 (13) | −0.0030 (10) | 0.0052 (10) | −0.0035 (10) |
| C21 | 0.0309 (14) | 0.0326 (14) | 0.0326 (14) | −0.0038 (11) | 0.0016 (11) | −0.0008 (11) |
| C13 | 0.0314 (14) | 0.0352 (14) | 0.0291 (14) | −0.0056 (11) | 0.0040 (11) | −0.0029 (11) |
| C18 | 0.0436 (17) | 0.0392 (15) | 0.0453 (18) | −0.0111 (13) | 0.0105 (13) | −0.0021 (13) |
| C14 | 0.0363 (15) | 0.0330 (14) | 0.0293 (14) | −0.0026 (11) | 0.0061 (11) | 0.0002 (11) |
| C19 | 0.0351 (15) | 0.0317 (14) | 0.0336 (15) | −0.0060 (11) | 0.0107 (11) | −0.0065 (11) |
| C17 | 0.072 (2) | 0.0379 (16) | 0.055 (2) | −0.0167 (15) | 0.0147 (17) | 0.0054 (14) |
| C15 | 0.0496 (18) | 0.0432 (16) | 0.0461 (18) | −0.0063 (14) | −0.0005 (14) | 0.0062 (13) |
| C16 | 0.073 (2) | 0.0407 (17) | 0.058 (2) | −0.0011 (16) | 0.0035 (17) | 0.0175 (15) |
| O3 | 0.0333 (9) | 0.0287 (9) | 0.0246 (9) | −0.0058 (7) | 0.0010 (7) | −0.0030 (7) |
| O4 | 0.0404 (10) | 0.0370 (10) | 0.0225 (9) | −0.0079 (8) | 0.0019 (7) | 0.0020 (7) |
| O1 | 0.0460 (11) | 0.0530 (11) | 0.0231 (10) | 0.0156 (9) | 0.0070 (8) | 0.0015 (8) |
| O2 | 0.0275 (9) | 0.0386 (10) | 0.0263 (10) | 0.0074 (7) | 0.0000 (7) | 0.0015 (7) |
| O1W | 0.0361 (10) | 0.0431 (10) | 0.0285 (10) | 0.0110 (8) | 0.0035 (8) | 0.0025 (8) |
| O2W | 0.0474 (12) | 0.0481 (11) | 0.0292 (10) | −0.0211 (9) | 0.0070 (8) | −0.0055 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Ca1—O3$^{i}$ | 2.3829 (16) | C5—H5 | 0.9300 |
| Ca1—O1W | 2.3871 (16) | N1—C21 | 1.317 (3) |

| | | | |
|---|---|---|---|
| Ca1—O2$^{ii}$ | 2.3877 (16) | N1—C19 | 1.371 (3) |
| Ca1—O2W | 2.4202 (17) | C22—O4 | 1.254 (3) |
| Ca1—O4 | 2.4712 (16) | C22—O3 | 1.258 (3) |
| Ca1—O1 | 2.5404 (17) | C22—C12 | 1.495 (3) |
| Ca1—O2 | 2.5538 (16) | C12—C13 | 1.360 (3) |
| Ca1—O3 | 2.5689 (16) | C12—C21 | 1.413 (3) |
| Ca1—H1W | 2.7828 | C21—H21 | 0.9300 |
| C1—C2 | 1.362 (3) | C13—C14 | 1.407 (3) |
| C1—C10 | 1.408 (3) | C13—H13 | 0.9300 |
| C1—C11 | 1.496 (3) | C18—C17 | 1.350 (4) |
| N2—C10 | 1.318 (3) | C18—C19 | 1.410 (3) |
| N2—C8 | 1.368 (3) | C18—H18 | 0.9300 |
| C2—C3 | 1.407 (3) | C14—C19 | 1.412 (3) |
| C2—H2 | 0.9300 | C14—C15 | 1.414 (4) |
| C10—H10 | 0.9300 | C17—C16 | 1.402 (4) |
| C11—O1 | 1.242 (3) | C17—H17 | 0.9300 |
| C11—O2 | 1.261 (3) | C15—C16 | 1.360 (4) |
| C8—C3 | 1.407 (3) | C15—H15 | 0.9300 |
| C8—C7 | 1.408 (3) | C16—H16 | 0.9300 |
| C3—C4 | 1.410 (4) | O3—Ca1$^{ii}$ | 2.3829 (16) |
| C7—C6 | 1.352 (4) | O2—Ca1$^{i}$ | 2.3877 (16) |
| C7—H7 | 0.9300 | O1W—H1W | 0.8432 |
| C4—C5 | 1.357 (4) | O1W—H2W | 0.7195 |
| C4—H4 | 0.9300 | O2W—H3W | 0.9231 |
| C6—C5 | 1.395 (4) | O2W—H4W | 0.8836 |
| C6—H6 | 0.9300 | | |

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| O3$^{i}$—Ca1—O1W | 86.63 (5) | O1—C11—C1 | 119.0 (2) |
| O3$^{i}$—Ca1—O2$^{ii}$ | 154.94 (6) | O2—C11—C1 | 118.7 (2) |
| O1W—Ca1—O2$^{ii}$ | 79.95 (6) | N2—C8—C3 | 121.8 (2) |
| O3$^{i}$—Ca1—O2W | 75.13 (6) | N2—C8—C7 | 119.1 (2) |
| O1W—Ca1—O2W | 97.97 (6) | C3—C8—C7 | 119.1 (3) |
| O2$^{ii}$—Ca1—O2W | 85.81 (5) | C2—C3—C8 | 117.8 (2) |
| O3$^{i}$—Ca1—O4 | 78.80 (5) | C2—C3—C4 | 122.9 (2) |
| O1W—Ca1—O4 | 93.79 (6) | C8—C3—C4 | 119.1 (2) |
| O2$^{ii}$—Ca1—O4 | 122.87 (5) | C6—C7—C8 | 120.3 (3) |
| O2W—Ca1—O4 | 150.61 (6) | C6—C7—H7 | 119.9 |
| O3$^{i}$—Ca1—O1 | 122.42 (5) | C8—C7—H7 | 119.9 |
| O1W—Ca1—O1 | 150.78 (6) | C5—C4—C3 | 120.1 (3) |
| O2$^{ii}$—Ca1—O1 | 74.02 (6) | C5—C4—H4 | 119.9 |
| O2W—Ca1—O1 | 93.21 (6) | C3—C4—H4 | 119.9 |
| O4—Ca1—O1 | 89.39 (6) | C7—C6—C5 | 120.9 (3) |
| O3$^{i}$—Ca1—O2 | 71.54 (5) | C7—C6—H6 | 119.6 |
| O1W—Ca1—O2 | 158.17 (6) | C5—C6—H6 | 119.6 |
| O2$^{ii}$—Ca1—O2 | 120.27 (6) | C4—C5—C6 | 120.5 (3) |
| O2W—Ca1—O2 | 76.99 (6) | C4—C5—H5 | 119.8 |
| O4—Ca1—O2 | 82.10 (5) | C6—C5—H5 | 119.8 |
| O1—Ca1—O2 | 50.97 (5) | C21—N1—C19 | 117.5 (2) |

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| O3$^{i}$—Ca1—O3 | 128.11 (6) | O4—C22—O3 | 122.3 (2) |
| O1W—Ca1—O3 | 82.58 (5) | O4—C22—C12 | 118.7 (2) |
| O2$^{ii}$—Ca1—O3 | 71.20 (5) | O3—C22—C12 | 119.0 (2) |
| O2W—Ca1—O3 | 156.61 (6) | C13—C12—C21 | 118.4 (2) |
| O4—Ca1—O3 | 51.72 (5) | C13—C12—C22 | 121.1 (2) |
| O1—Ca1—O3 | 76.70 (5) | C21—C12—C22 | 120.5 (2) |
| O2—Ca1—O3 | 110.53 (5) | N1—C21—C12 | 124.2 (2) |
| O3$^{i}$—Ca1—Ca1$^{i}$ | 37.49 (4) | N1—C21—H21 | 117.9 |
| O1W—Ca1—Ca1$^{i}$ | 123.88 (4) | C12—C21—H21 | 117.9 |
| O2$^{ii}$—Ca1—Ca1$^{i}$ | 151.60 (4) | C12—C13—C14 | 119.9 (2) |
| O2W—Ca1—Ca1$^{i}$ | 76.36 (4) | C12—C13—H13 | 120.1 |
| O4—Ca1—Ca1$^{i}$ | 74.71 (4) | C14—C13—H13 | 120.1 |
| O1—Ca1—Ca1$^{i}$ | 84.96 (4) | C17—C18—C19 | 120.3 (3) |
| O2—Ca1—Ca1$^{i}$ | 34.37 (4) | C17—C18—H18 | 119.9 |
| O3—Ca1—Ca1$^{i}$ | 122.74 (4) | C19—C18—H18 | 119.9 |
| O3$^{i}$—Ca1—Ca1$^{ii}$ | 155.93 (4) | C13—C14—C19 | 117.7 (2) |
| O1W—Ca1—Ca1$^{ii}$ | 75.75 (4) | C13—C14—C15 | 123.3 (2) |
| O2$^{ii}$—Ca1—Ca1$^{ii}$ | 37.14 (4) | C19—C14—C15 | 118.9 (2) |
| O2W—Ca1—Ca1$^{ii}$ | 122.94 (4) | N1—C19—C18 | 118.5 (2) |
| O4—Ca1—Ca1$^{ii}$ | 86.04 (4) | N1—C19—C14 | 122.3 (2) |
| O1—Ca1—Ca1$^{ii}$ | 75.51 (4) | C18—C19—C14 | 119.2 (2) |
| O2—Ca1—Ca1$^{ii}$ | 124.99 (4) | C18—C17—C16 | 120.9 (3) |
| O3—Ca1—Ca1$^{ii}$ | 34.37 (3) | C18—C17—H17 | 119.5 |
| Ca1$^{i}$—Ca1—Ca1$^{ii}$ | 152.71 (2) | C16—C17—H17 | 119.5 |
| O3$^{i}$—Ca1—H1W | 102.1 | C16—C15—C14 | 120.2 (3) |
| O1W—Ca1—H1W | 16.6 | C16—C15—H15 | 119.9 |
| O2$^{ii}$—Ca1—H1W | 68.0 | C14—C15—H15 | 119.9 |
| O2W—Ca1—H1W | 107.6 | C15—C16—C17 | 120.5 (3) |
| O4—Ca1—H1W | 90.8 | C15—C16—H16 | 119.8 |
| O1—Ca1—H1W | 134.6 | C17—C16—H16 | 119.8 |
| O2—Ca1—H1W | 171.3 | C22—O3—Ca1$^{ii}$ | 161.76 (15) |
| O3—Ca1—H1W | 68.3 | C22—O3—Ca1 | 89.47 (13) |
| Ca1$^{i}$—Ca1—H1W | 138.4 | Ca1$^{ii}$—O3—Ca1 | 108.15 (6) |
| Ca1$^{ii}$—Ca1—H1W | 59.2 | C22—O4—Ca1 | 94.08 (13) |
| C2—C1—C10 | 118.0 (2) | C11—O1—Ca1 | 91.84 (14) |
| C2—C1—C11 | 121.0 (2) | C11—O2—Ca1$^{i}$ | 160.71 (15) |
| C10—C1—C11 | 121.0 (2) | C11—O2—Ca1 | 90.76 (13) |
| C10—N2—C8 | 118.1 (2) | Ca1$^{i}$—O2—Ca1 | 108.49 (6) |
| C1—C2—C3 | 120.2 (2) | Ca1—O1W—H1W | 109.4 |
| C1—C2—H2 | 119.9 | Ca1—O1W—H2W | 141.1 |
| C3—C2—H2 | 119.9 | H1W—O1W—H2W | 99.8 |
| N2—C10—C1 | 124.0 (2) | Ca1—O2W—H3W | 116.8 |
| N2—C10—H10 | 118.0 | Ca1—O2W—H4W | 139.0 |
| C1—C10—H10 | 118.0 | H3W—O2W—H4W | 103.8 |
| O1—C11—O2 | 122.2 (2) | | |

Symmetry codes: (i) $x$, $-y+3/2$, $z-1/2$; (ii) $x$, $-y+3/2$, $z+1/2$.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2*W*—H4*W*···N2$^{iii}$ | 0.88 | 2.01 | 2.880 (3) | 170 |
| O2*W*—H3*W*···O1$^{i}$ | 0.92 | 1.92 | 2.813 (2) | 163 |
| O1*W*—H2*W*···N1$^{iv}$ | 0.72 | 2.18 | 2.885 (2) | 165 |
| O1*W*—H1*W*···O4$^{ii}$ | 0.84 | 1.94 | 2.785 (2) | 174 |

Symmetry codes: (i) $x, -y+3/2, z-1/2$; (ii) $x, -y+3/2, z+1/2$; (iii) $-x+1, y+1/2, -z+1/2$; (iv) $-x, y+1/2, -z+1/2$.