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Bis(μ-pyridazine-3-carboxylato)-$\kappa^3N,O:O;\kappa^3O:N,O$-bis[triaquacalcium(II)]

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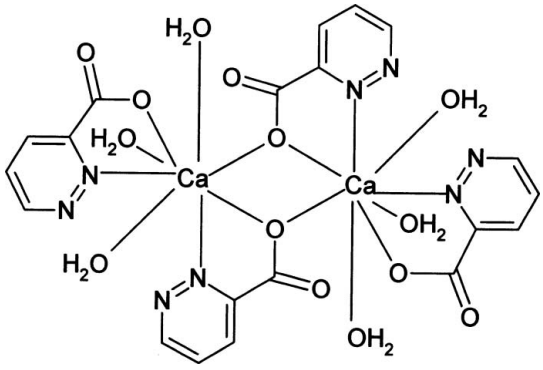
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.102; data-to-parameter ratio = 16.9.

The structure of the title compound, $[Ca_2(C_5H_3N_2O_2)_2(H_2O)_6]$, is composed of centrosymmetric dimeric molecules, in which the Ca^{II} ions are bridged by two carboxylate O atoms of bidentate ligands. Each Ca^{II} ion is coordinated by two carboxylate O atoms, a hetero-ring N atom and three water O atoms. The coordination number of each Ca^{II} ion is eight, forming a distorted decahedron. Coordinated water molecules act as donors linking adjacent dimers into a three-dimensional hydrogen-bonded network. Intra-dimer hydrogen bonds are also present.

Related literature

We have recently determined the structure of the free ligand (Gryz *et al.*, 2003) and a related structure has also been reported (Ptasiewicz-Bąk *et al.*, 1998). The bond lengths and angles in the title compound are typical (Einspahr & Bugg, 1981).



Experimental

Crystal data

$[Ca_2(C_5H_3N_2O_2)_2(H_2O)_6]$
M_r = 680.63
Monoclinic, $P2_1/n$
a = 9.1730 (18) Å
b = 10.539 (2) Å
c = 14.984 (3) Å
β = 99.43 (3)°
V = 1429.0 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.48 mm^{-1}
T = 293 (2) K
0.54 × 0.22 × 0.18 mm

Data collection

Kuma KM4 four-circle diffractometer
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2000)
T_{min} = 0.877, T_{max} = 0.901
4432 measured reflections
4182 independent reflections
3154 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
3 standard reflections
every 200 reflections
intensity decay: 2.5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.102
S = 0.98
4182 reflections
247 parameters
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ca1—O2	2.3662 (14)	Ca1—O1	2.4334 (15)
Ca1—O3	2.3680 (13)	Ca1—N12	2.5853 (14)
Ca1—O21	2.4040 (13)	Ca1—N22	2.6057 (14)
Ca1—O11	2.4318 (11)	Ca1—O11^{i}	2.6158 (11)
O21—Ca1—O11	77.38 (5)	O21—Ca1—N22	63.66 (4)
O2—Ca1—O1	77.35 (6)	N12—Ca1—N22	77.28 (5)
O3—Ca1—O1	78.25 (5)	O2—Ca1—O11^{i}	76.37 (5)
O11—Ca1—N12	65.29 (4)	O3—Ca1—O11^{i}	74.73 (5)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H31···O21^{i}	0.87 (3)	1.89 (3)	2.7458 (18)	166 (3)
O1—H11···O22^{ii}	0.78 (3)	1.92 (3)	2.703 (2)	173 (2)
O1—H12···N21	0.91 (4)	2.00 (4)	2.812 (2)	148 (3)
O1—H12···N22	0.91 (4)	2.43 (3)	2.941 (2)	116 (2)
O3—H32···N11^{iii}	0.81 (3)	2.25 (3)	3.000 (2)	154 (2)
O2—H21···O12^{iv}	0.79 (3)	2.00 (3)	2.725 (2)	152 (3)
O2—H22···O12^{i}	0.87 (3)	1.76 (3)	2.622 (2)	171 (3)
O2—H22···O11^{i}	0.87 (3)	2.61 (3)	3.0861 (18)	115 (2)

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

Data collection: *KM-4 Software* (Kuma Diffraction, 1996); cell refinement: *KM-4 Software*; data reduction: *DATAPROC* (Kuma Diffraction, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1992) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2377).

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supplementary materials

Acta Cryst. (2007). E**63**, m1662-m1663 [**doi:10.1107/S1600536807022544**]

Bis(*μ*-pyridazine-3-carboxylato)-$\kappa^3N,O:O;\kappa^3O:N,O$-bis[triaquacalcium(II)]

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Comment

The structure of compound (1) is composed of discrete, dinuclear centrosymmetric units consisting of two Ca^{II} ions bridged by two carboxylate oxygen atoms each donated by the ligand molecule coordinated to a different Ca^{II} ion. Figure 1 shows the molecular structure of a dimer with atom labelling scheme; Figure 2 illustrates the way the dimers are packed in the crystal. The Ca^{II} ion is coordinated by two pyridazine-3-carboxylate ligands *via* their N,*O* bonding groups. In both, only one carboxylate O atom participates in coordination. Pyridazine rings are planar with r.m.s. of 0.0074 Å and 0.0050Å for the two symmetry unique ligand molecules. The planes of the rings make an angle of 84.0° each to the other. The planes of the carboxylate groups make the angles of 11.6° and 0.2° with the two pyridazine rings to which they are bonded. Bond distances and bond angles within the ligand molecules agree well with those reported earlier for the parent acid (Gryz *et al.*, 2003). The coordinated carboxylate oxygen atom of one ligand molecule acts as bidentate being coordinated to the symmetry related Ca^{II} ion, thus forming two bridges Ca – O11 – Ca^{i} and Ca – $O11^{i}$ – Ca^{i} [symmetry code as in Table 1]. Apart from the two N,*O* bonding groups and the bridging carboxylate O atom, each Ca^{II} ion is also coordinated by three O atoms of water molecules. The coordination number of each Ca^{II} ion is eight and the coordination geometry is represented by a severely distorted decahedron, similar to that observed in a Ca complex with pyrazine-2-carboxylate and water ligands (Ptasiewicz-Bąk & *et al.*, 1998). One of the strongly distorted tetragonal bases (r.m.s 0.2715 Å) is composed of O11, N12, O21 and N22 atoms, the other base (r.m.s. 0.2990 Å) is formed by three water oxygen O (O1, O2, O3) and the bridging carboxylate O atom $O11^{i}$. The bases are almost parallel making an angle of 6.1° but the tetragons are rotated by *ca* 45°, one in respect to the other. The observed Ca—O bond distances and angles are typical for calcium complexes with carboxylate ligands (Einspahr & Bugg, 1981). Coordinated water molecules act as donors in hydrogen bonds to the carboxylate oxygen atoms and hetero-ring nitrogen atoms in adjacent dimers (Fig. 2). Intradimeric hydrogen bonds are also observed. For details – see Table 2.

Experimental

Hot aqueous solutions containing 2 mmol s of pyridazine-3-carboxylic acid and 1 mmol of calcium(II) acetate tetrahydrate, respectively, were mixed and boiled for one hour with constant stirring. After cooling to room temperature, 5 ml of 3 N acetic acid were added. The solution when evaporated to dryness at room temperature, over several days, provided rectangular blocks as single crystals. They were washed with cold ethanol and dried in air.

Refinement

H atoms were refined independently with isotropic displacement parameters.

Figures

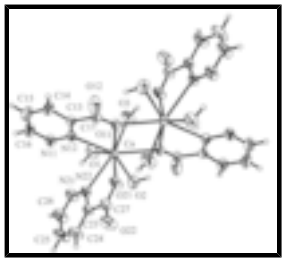

Fig. 1. The molecular structure of (1) with atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabeled atoms are related by the symmetry operator (−*x* + 1, −*y* + 2, −*z* + 1).

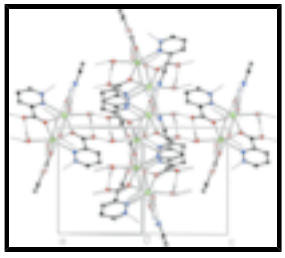

Fig. 2. The packing of (1) with hydrogen bonds shown as dashed lines.

Bis(μ-pyridazine-3-carboxylato)-κ^3*N*,*O*:*O*;κ^3*O*:*N*,*O*-bis[triaquacalcium(II)]

Crystal data

$[Ca_2(C_5H_3N_2O_2)_2(H_2O)_6]$	F_{000} = 704
M_r = 680.63	D_x = 1.582 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 25 reflections
a = 9.1730 (18) Å	θ = 6–15°
b = 10.539 (2) Å	μ = 0.48 mm^{-1}
c = 14.984 (3) Å	*T* = 293 (2) K
β = 99.43 (3)°	Rectangular block, colourless
V = 1429.0 (5) Å^3	0.54 × 0.22 × 0.18 mm
Z = 2	

Data collection

Kuma KM4 four-circle diffractometer	R_{int} = 0.017
Radiation source: fine-focus sealed tube	θ_{max} = 30.1°
Monochromator: graphite	θ_{min} = 2.4°
T = 293(2) K	*h* = 0→12
profile data from θ/2θ scans	*k* = −14→0
Absorption correction: analytical (CrysAlis RED; Oxford Diffraction, 2000)	*l* = −21→20
T_{min} = 0.877, T_{max} = 0.901	3 standard reflections
4432 measured reflections	every 200 reflections
4182 independent reflections	intensity decay: 2.5%

3154 reflections with $I > 2\sigma(I)$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	Only H-atom coordinates refined
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.4166P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{max} = 0.001$
4182 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
247 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ca1	0.65075 (3)	0.88232 (3)	0.575164 (18)	0.02345 (8)
O11	0.39476 (11)	0.95185 (10)	0.55635 (7)	0.0282 (2)
N12	0.49408 (13)	0.77300 (12)	0.68154 (8)	0.0277 (2)
O3	0.67401 (17)	1.08313 (13)	0.64699 (10)	0.0474 (3)
O2	0.87173 (14)	0.87577 (15)	0.51303 (10)	0.0434 (3)
O1	0.83709 (16)	0.83982 (14)	0.70706 (9)	0.0473 (3)
O21	0.53629 (13)	0.76031 (11)	0.44708 (8)	0.0391 (3)
C17	0.30113 (15)	0.89606 (14)	0.59370 (10)	0.0279 (3)
O12	0.16530 (13)	0.90909 (17)	0.57563 (12)	0.0590 (4)
N22	0.73182 (16)	0.64577 (12)	0.57056 (9)	0.0327 (3)
N11	0.54731 (15)	0.68921 (14)	0.74637 (10)	0.0362 (3)
C13	0.35352 (16)	0.80508 (14)	0.66984 (9)	0.0270 (3)
C23	0.68131 (18)	0.58150 (14)	0.49646 (10)	0.0317 (3)
O22	0.5225 (2)	0.59002 (14)	0.35821 (10)	0.0611 (4)
N21	0.8315 (2)	0.59353 (15)	0.63436 (11)	0.0501 (4)
C27	0.56975 (18)	0.64986 (15)	0.42743 (11)	0.0341 (3)
C14	0.2554 (2)	0.7596 (2)	0.72419 (13)	0.0436 (4)

C16	0.4569 (2)	0.6429 (2)	0.79794 (14)	0.0474 (4)
C24	0.7276 (3)	0.45936 (19)	0.48169 (15)	0.0575 (6)
C15	0.3101 (2)	0.6764 (2)	0.79061 (15)	0.0544 (5)
C26	0.8765 (3)	0.4766 (2)	0.62310 (16)	0.0648 (7)
C25	0.8276 (4)	0.4049 (2)	0.54776 (18)	0.0713 (8)
H31	0.610 (3)	1.141 (3)	0.6249 (19)	0.062 (7)*
H11	0.885 (3)	0.862 (2)	0.7526 (18)	0.049 (6)*
H14	0.152 (3)	0.789 (2)	0.7140 (16)	0.056 (7)*
H15	0.253 (3)	0.649 (3)	0.830 (2)	0.073 (8)*
H16	0.504 (3)	0.586 (2)	0.8425 (17)	0.057 (7)*
H23	0.687 (3)	0.418 (3)	0.427 (2)	0.078 (9)*
H25	0.955 (4)	0.446 (3)	0.674 (2)	0.090 (10)*
H24	0.870 (3)	0.324 (3)	0.5399 (19)	0.070 (8)*
H12	0.853 (3)	0.755 (3)	0.706 (2)	0.090 (10)*
H32	0.734 (3)	1.108 (2)	0.6894 (18)	0.056 (7)*
H21	0.949 (3)	0.873 (3)	0.546 (2)	0.071 (9)*
H22	0.869 (3)	0.949 (3)	0.4853 (17)	0.056 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ca1	0.02048 (13)	0.02388 (13)	0.02488 (13)	−0.00055 (9)	0.00039 (9)	−0.00032 (9)
O11	0.0232 (4)	0.0304 (5)	0.0308 (5)	0.0006 (4)	0.0035 (4)	0.0058 (4)
N12	0.0272 (6)	0.0278 (6)	0.0273 (6)	0.0009 (4)	0.0023 (4)	0.0029 (4)
O3	0.0530 (8)	0.0348 (6)	0.0445 (7)	0.0109 (6)	−0.0219 (6)	−0.0133 (5)
O2	0.0257 (5)	0.0553 (8)	0.0487 (7)	0.0012 (5)	0.0045 (5)	0.0161 (6)
O1	0.0538 (8)	0.0407 (7)	0.0384 (6)	−0.0004 (6)	−0.0195 (6)	−0.0027 (5)
O21	0.0415 (6)	0.0282 (5)	0.0409 (6)	0.0060 (5)	−0.0129 (5)	−0.0040 (5)
C17	0.0230 (6)	0.0284 (6)	0.0315 (7)	0.0002 (5)	0.0018 (5)	0.0024 (5)
O12	0.0218 (5)	0.0739 (10)	0.0796 (11)	0.0013 (6)	0.0035 (6)	0.0423 (8)
N22	0.0407 (7)	0.0270 (6)	0.0273 (6)	0.0034 (5)	−0.0042 (5)	0.0013 (5)
N11	0.0355 (7)	0.0360 (7)	0.0360 (7)	0.0059 (5)	0.0023 (5)	0.0098 (5)
C13	0.0284 (6)	0.0258 (6)	0.0266 (6)	0.0005 (5)	0.0044 (5)	0.0010 (5)
C23	0.0396 (8)	0.0247 (6)	0.0284 (7)	0.0011 (6)	−0.0013 (6)	−0.0006 (5)
O22	0.0825 (11)	0.0446 (7)	0.0432 (7)	0.0131 (7)	−0.0288 (7)	−0.0143 (6)
N21	0.0691 (11)	0.0380 (8)	0.0348 (7)	0.0142 (7)	−0.0166 (7)	0.0015 (6)
C27	0.0384 (8)	0.0281 (7)	0.0315 (7)	0.0013 (6)	−0.0068 (6)	−0.0013 (6)
C14	0.0347 (8)	0.0509 (10)	0.0488 (10)	0.0073 (7)	0.0172 (7)	0.0159 (8)
C16	0.0504 (10)	0.0490 (10)	0.0437 (9)	0.0084 (8)	0.0104 (8)	0.0213 (8)
C24	0.0833 (15)	0.0335 (9)	0.0472 (10)	0.0159 (10)	−0.0143 (10)	−0.0117 (8)
C15	0.0524 (11)	0.0641 (13)	0.0522 (11)	0.0086 (10)	0.0250 (9)	0.0265 (10)
C26	0.0882 (17)	0.0422 (10)	0.0527 (12)	0.0239 (11)	−0.0219 (12)	0.0050 (9)
C25	0.103 (2)	0.0350 (10)	0.0650 (14)	0.0299 (12)	−0.0179 (13)	−0.0060 (9)

Geometric parameters (Å, °)

Ca1—O2	2.3662 (14)	C17—O12	1.2385 (19)
Ca1—O3	2.3680 (13)	C17—C13	1.508 (2)
Ca1—O21	2.4040 (13)	N22—C23	1.3183 (19)

Ca1—O11	2.4318 (11)	N22—N21	1.3294 (19)
Ca1—O1	2.4334 (15)	N11—C16	1.317 (2)
Ca1—N12	2.5853 (14)	C13—C14	1.394 (2)
Ca1—N22	2.6057 (14)	C23—C24	1.385 (2)
Ca1—O11^{i}	2.6158 (11)	C23—C27	1.513 (2)
Ca1—Ca1^{i}	4.1029 (11)	O22—C27	1.230 (2)
Ca1—H22	2.68 (3)	N21—C26	1.319 (3)
O11—C17	1.2466 (17)	C14—C15	1.359 (3)
O11—Ca1^{i}	2.6158 (11)	C14—H14	0.99 (3)
N12—C13	1.3166 (19)	C16—C15	1.379 (3)
N12—N11	1.3440 (18)	C16—H16	0.95 (3)
O3—H31	0.87 (3)	C24—C25	1.361 (3)
O3—H32	0.81 (3)	C24—H23	0.95 (3)
O2—H21	0.79 (3)	C15—H15	0.90 (3)
O2—H22	0.87 (3)	C26—C25	1.371 (3)
O1—H11	0.78 (3)	C26—H25	1.01 (3)
O1—H12	0.91 (4)	C25—H24	0.96 (3)
O21—C27	1.251 (2)		
O2—Ca1—O3	100.70 (6)	C13—N12—N11	119.79 (13)
O2—Ca1—O21	87.74 (5)	C13—N12—Ca1	115.76 (9)
O3—Ca1—O21	146.71 (4)	N11—N12—Ca1	124.45 (10)
O2—Ca1—O11	146.76 (4)	Ca1—O3—H31	116.4 (18)
O3—Ca1—O11	78.27 (5)	Ca1—O3—H32	129.8 (18)
O21—Ca1—O11	77.38 (5)	H31—O3—H32	114 (2)
O2—Ca1—O1	77.35 (6)	Ca1—O2—H21	119 (2)
O3—Ca1—O1	78.25 (5)	Ca1—O2—H22	101.5 (17)
O21—Ca1—O1	134.97 (5)	H21—O2—H22	107 (3)
O11—Ca1—O1	133.32 (5)	Ca1—O1—H11	150.3 (18)
O2—Ca1—N12	145.91 (5)	Ca1—O1—H12	105 (2)
O3—Ca1—N12	97.81 (5)	H11—O1—H12	104 (3)
O21—Ca1—N12	92.27 (5)	C27—O21—Ca1	126.41 (10)
O11—Ca1—N12	65.29 (4)	O12—C17—O11	126.30 (14)
O1—Ca1—N12	78.74 (5)	O12—C17—C13	114.87 (13)
O2—Ca1—N22	72.25 (5)	O11—C17—C13	118.82 (12)
O3—Ca1—N22	149.57 (5)	C23—N22—N21	119.89 (14)
O21—Ca1—N22	63.66 (4)	C23—N22—Ca1	116.83 (10)
O11—Ca1—N22	124.03 (4)	N21—N22—Ca1	122.89 (10)
O1—Ca1—N22	71.33 (5)	C16—N11—N12	118.60 (14)
N12—Ca1—N22	77.28 (5)	N12—C13—C14	123.08 (14)
O2—Ca1—O11^{i}	76.37 (5)	N12—C13—C17	116.61 (12)
O3—Ca1—O11^{i}	74.73 (5)	C14—C13—C17	120.31 (13)
O21—Ca1—O11^{i}	76.17 (4)	N22—C23—C24	122.52 (15)
O11—Ca1—O11^{i}	71.30 (4)	N22—C23—C27	115.86 (13)
O1—Ca1—O11^{i}	137.64 (5)	C24—C23—C27	121.62 (15)
N12—Ca1—O11^{i}	136.55 (4)	C26—N21—N22	119.19 (16)
N22—Ca1—O11^{i}	129.07 (4)	O22—C27—O21	127.52 (15)

O2—Ca1—Ca1^{i}	110.21 (4)	O22—C27—C23	115.95 (15)
O3—Ca1—Ca1^{i}	73.24 (4)	O21—C27—C23	116.53 (13)
O21—Ca1—Ca1^{i}	73.63 (3)	C15—C14—C13	116.98 (16)
O11—Ca1—Ca1^{i}	37.15 (3)	C15—C14—H14	123.2 (14)
O1—Ca1—Ca1^{i}	151.38 (4)	C13—C14—H14	119.8 (14)
N12—Ca1—Ca1^{i}	102.42 (3)	N11—C16—C15	123.98 (17)
N22—Ca1—Ca1^{i}	137.18 (3)	N11—C16—H16	112.9 (15)
O11^{i}—Ca1—Ca1^{i}	34.15 (2)	C15—C16—H16	123.1 (15)
O2—Ca1—H22	18.6 (6)	C25—C24—C23	117.60 (18)
O3—Ca1—H22	88.6 (6)	C25—C24—H23	123.5 (19)
O21—Ca1—H22	90.2 (6)	C23—C24—H23	118.9 (19)
O11—Ca1—H22	130.5 (6)	C14—C15—C16	117.52 (17)
O1—Ca1—H22	88.5 (6)	C14—C15—H15	120.4 (19)
N12—Ca1—H22	164.1 (6)	C16—C15—H15	122.0 (18)
N22—Ca1—H22	89.8 (6)	N21—C26—C25	123.58 (19)
O11^{i}—Ca1—H22	59.1 (6)	N21—C26—H25	113.6 (19)
Ca1^{i}—Ca1—H22	93.3 (6)	C25—C26—H25	122.7 (19)
C17—O11—Ca1	121.94 (9)	C24—C25—C26	117.19 (19)
C17—O11—Ca1^{i}	128.07 (9)	C24—C25—H24	121.8 (17)
Ca1—O11—Ca1^{i}	108.70 (4)	C26—C25—H24	120.7 (17)

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H31···O21^{i}	0.87 (3)	1.89 (3)	2.7458 (18)	166 (3)
O1—H11···O22^{ii}	0.78 (3)	1.92 (3)	2.703 (2)	173 (2)
O1—H12···N21	0.91 (4)	2.00 (4)	2.812 (2)	148 (3)
O1—H12···N22	0.91 (4)	2.43 (3)	2.941 (2)	116 (2)
O3—H32···N11^{iii}	0.81 (3)	2.25 (3)	3.000 (2)	154 (2)
O2—H21···O12^{iv}	0.79 (3)	2.00 (3)	2.725 (2)	152 (3)
O2—H22···O12^{i}	0.87 (3)	1.76 (3)	2.622 (2)	171 (3)
O2—H22···O11^{i}	0.87 (3)	2.61 (3)	3.0861 (18)	115 (2)

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

Fig. 1

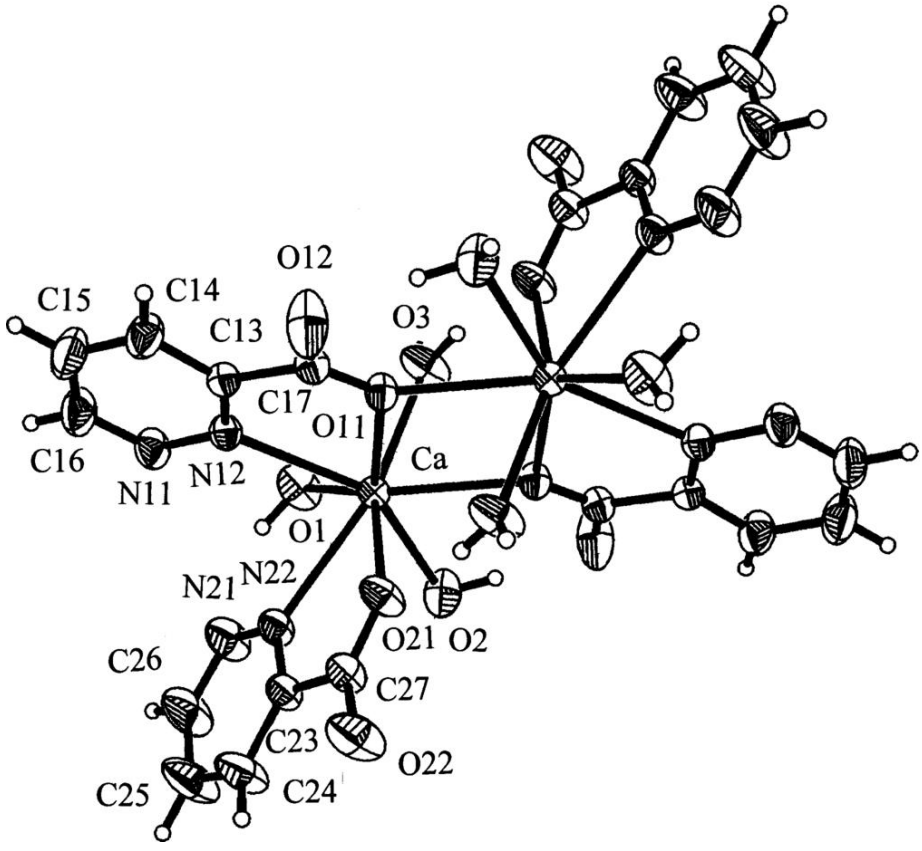
O12
C15
C14
C13
O3
C17
O11
C16
N11
N12
Ca
O1
N21
N22
O21
O2
C26
C27
C23
O22
C25
C24

Fig. 2