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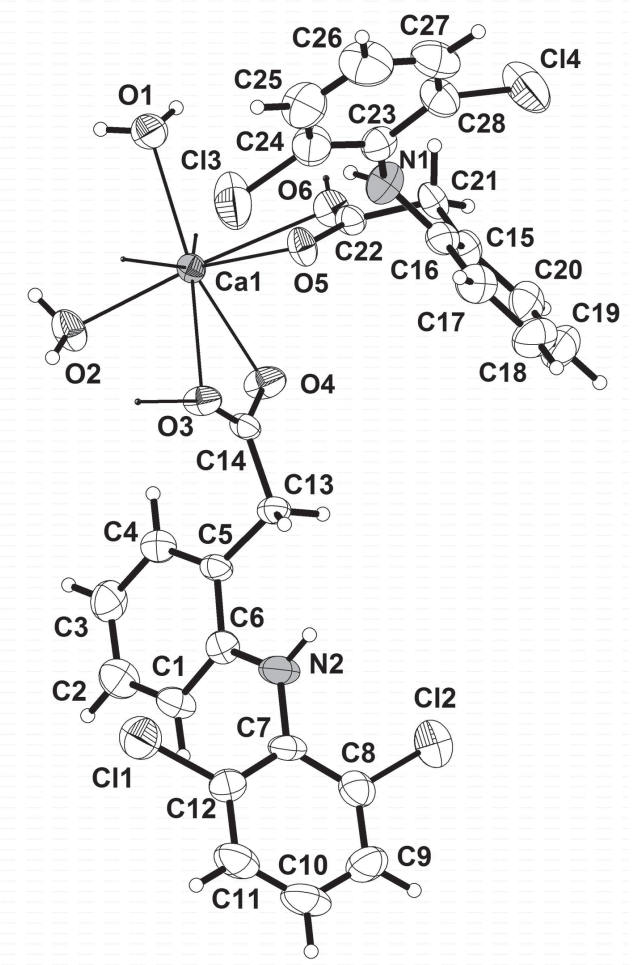
Crystal structure of *catena*-poly[diaqua-bis(μ$_2$-2-(2-((2,6-dichlorophenyl)amino)phenyl)acetato-κ^3*O*,*O*′:*O*′)calcium(II)], $C_{56}H_{48}N_4Ca_2Cl_8O_{12}$



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Abstract

$C_{56}H_{48}N_4Ca_2Cl_8O_{12}$, monoclinic, $P2_1/c$, $a = 16.776(8)$ Å, $b = 24.193(12)$ Å, $c = 7.657(4)$ Å, $\beta = 101.110(7)°$, $V = 3050(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0685$, $wR_{ref}(F^2) = 0.1663$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.32 × 0.22 × 0.19 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.60 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	25.5°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15005, 5585, 0.106
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2669
$N(param)_{refined}$:	372
Programs:	Bruker programs [1], SHELX [2]

Source of material

A mixture of $Ca(OAc)_2{\cdot}H_2O$ (0.1 mmol, 0.0176 g), 2-(2-((2,6-dichlorophenyl)amino)phenyl)acetic acid (0.1 mmol, 891 mg), 4-(1*H*-pyrazol-3-yl)-pyridine (0.1 mmol, 55 mg) and distilled water (10 mL) was heated in a 25 mL steel reactor with a Teflon liner 403 K for 36 h, followed by slow cooling to room temperature. Colorless crystals of the title compound had formed.

Experimental details

Hydrogen atoms were placed in calculated positions and were included using the riding model, with U_{iso}(H) set to $1.2U_{eq}$(C).

Discussion

The design and construction of metal-organic frameworks (MOFs) with well-regulated network structures has been provoked significant interest [3–6]. The organic ligands, which have their differences in the size, the flexibility, the coordination ability, the number of the carboxylate groups, the positions of the carboxylate groups, play crucial roles in the construction of frameworks with interesting properties [7–9].

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
Ca1	0.31716(6)	0.23340(4)	0.32629(13)	0.0275(3)
Cl1	0.59735(10)	0.37297(7)	1.2470(2)	0.0635(5)
Cl2	0.60015(11)	0.53917(7)	0.7758(2)	0.0739(6)
Cl3	0.05177(12)	0.27575(8)	0.6306(3)	0.0852(7)
Cl4	−0.09075(11)	0.41390(9)	0.1081(2)	0.0862(7)
N1	0.0511(3)	0.35264(19)	0.3265(6)	0.0465(13)
H1	0.0755	0.3239	0.2971	0.056*
N2	0.5660(3)	0.42473(19)	0.8782(6)	0.0502(14)
H2	0.5185	0.4342	0.8227	0.060*
O1	0.2301(2)	0.16215(16)	0.1639(5)	0.0498(11)
H1A	0.2498	0.1302	0.1941	0.075*
H1B	0.2276	0.1667	0.0524	0.075*
O2	0.4218(2)	0.17453(15)	0.4898(4)	0.0473(11)
H2A	0.4120	0.1408	0.4594	0.071*
H2B	0.4222	0.1779	0.6011	0.071*
O3	0.3765(2)	0.29091(15)	0.5873(4)	0.0375(10)
O4	0.4172(2)	0.30954(15)	0.3395(5)	0.0468(11)
O5	0.2108(2)	0.30595(14)	0.3095(5)	0.0415(10)
O6	0.2511(2)	0.29626(15)	0.0551(5)	0.0419(10)
C1	0.6811(3)	0.3602(3)	0.8970(7)	0.0467(16)
H1C	0.7131	0.3832	0.9793	0.056*
C2	0.7120(4)	0.3112(3)	0.8482(8)	0.0508(17)
H2C	0.7648	0.3009	0.8991	0.061*
C3	0.6661(4)	0.2776(3)	0.7263(8)	0.0511(17)
H3	0.6864	0.2437	0.6980	0.061*
C4	0.5889(4)	0.2943(2)	0.6446(7)	0.0433(15)
H4	0.5591	0.2721	0.5563	0.052*
C5	0.5546(3)	0.3434(2)	0.6906(6)	0.0321(13)
C6	0.6015(3)	0.3754(2)	0.8225(7)	0.0364(14)
C7	0.6049(3)	0.4584(2)	1.0187(7)	0.0391(14)
C8	0.6234(3)	0.5132(3)	0.9907(8)	0.0468(16)
C9	0.6597(4)	0.5473(3)	1.1273(9)	0.0598(19)
H9	0.6712	0.5840	1.1049	0.072*
C10	0.6787(4)	0.5264(3)	1.2967(9)	0.066(2)
H10	0.7050	0.5488	1.3888	0.079*
C11	0.6594(4)	0.4729(3)	1.3326(8)	0.0596(19)
H11	0.6711	0.4595	1.4486	0.071*
C12	0.6225(3)	0.4393(2)	1.1951(8)	0.0423(15)
C13	0.4734(3)	0.3641(2)	0.5961(6)	0.0335(13)
H13A	0.4815	0.3918	0.5096	0.040*
H13B	0.4460	0.3818	0.6818	0.040*
C14	0.4197(3)	0.3188(2)	0.5021(7)	0.0298(13)
C15	0.1400(3)	0.4124(2)	0.1981(7)	0.0382(14)
C16	0.0887(3)	0.4039(2)	0.3199(7)	0.0377(14)
C17	0.0753(4)	0.4467(3)	0.4332(8)	0.0531(17)
H17	0.0419	0.4411	0.5154	0.064*
C18	0.1122(4)	0.4976(3)	0.4225(9)	0.065(2)
H18	0.1034	0.5259	0.4987	0.078*
C19	0.1613(4)	0.5070(3)	0.3018(10)	0.067(2)
H19	0.1851	0.5414	0.2950	0.080*
C20	0.1749(3)	0.4645(2)	0.1902(8)	0.0524(17)
H20	0.2081	0.4708	0.1080	0.063*
C21	0.1556(3)	0.3681(2)	0.0723(6)	0.0382(14)
H21A	0.1796	0.3853	−0.0197	0.046*
H21B	0.1036	0.3529	0.0149	0.046*
C22	0.2097(3)	0.3207(2)	0.1498(7)	0.0319(13)
C23	−0.0230(4)	0.3452(2)	0.3773(8)	0.0419(15)
C24	−0.0330(4)	0.3102(3)	0.5155(9)	0.0564(18)
C25	−0.1069(5)	0.3013(3)	0.5665(10)	0.069(2)
H25	−0.1108	0.2767	0.6579	0.083*
C26	−0.1739(5)	0.3291(3)	0.4806(10)	0.076(2)
H26	−0.2235	0.3240	0.5152	0.092*
C27	−0.1683(4)	0.3649(3)	0.3422(9)	0.065(2)
H27	−0.2136	0.3844	0.2853	0.078*
C28	−0.0943(4)	0.3711(3)	0.2900(8)	0.0519(17)

N-containing auxiliary ligands are known to be ideal connectors for the propagation of coordination networks [10].

The asymmetric unit of the title structure contain one Ca(II), two anionic ligands and two water molecules. The calcium atom is eight-coordinated [CdO_8] by six oxygen atoms from several ligands (*cf*. the figure). The Ca—O bond lengths range from 2.322(4) to 2.639(4) Å, respectively. The Ca—O bond lengths of the water ligands are 2.439(4) Å and 2.414(4) Å. The bond angles of O—Ca—O are in the range of 50.57(11)°–156.20(12)°. The hydrogen bonding between carboxylate oxygen atoms and the NH group of the organic ligands are: (d(N···O): 2.933(6) Å, and the (d(N···Cl): 3.451(5) Å.

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