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Crystal structure of tetraaquabis(5-(4-carboxybenzyloxy)isophthalato)-tricalcium(II) tetrahydrate, $Ca_3(H_2O)_4(C_{16}H_9O_7)_2 \cdot 4H_2O$

Jing Li and Jian-Fang Ma*

Department of Chemistry, Northeast Normal University, Changchun 130024, P. R. China

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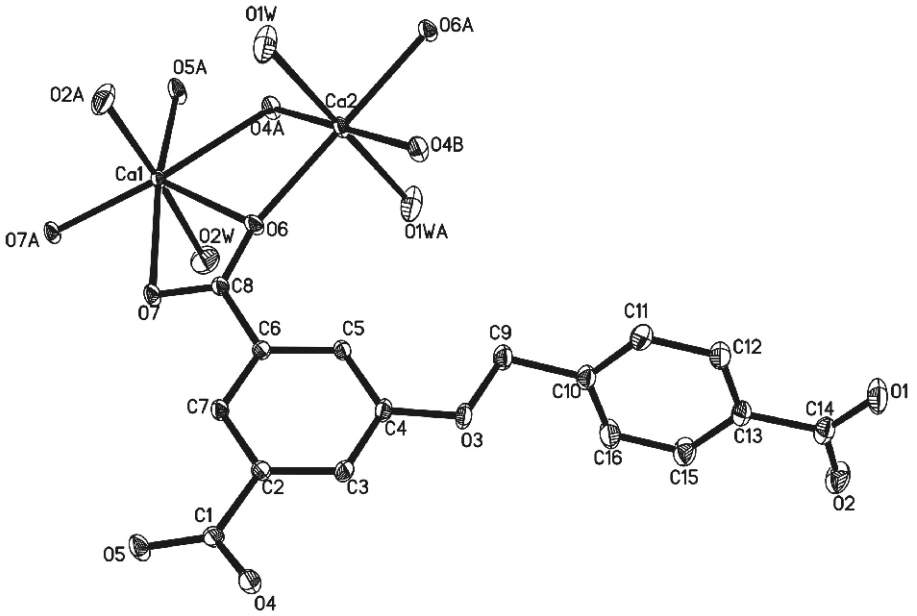

Abstract

$C_{32}H_{34}Ca_3O_{22}$, triclinic, $P\bar{1}$ (no. 2), a = 9.824(5) Å, b = 9.963(5) Å, c = 10.150(5) Å, α = 74.044(5)°, β = 86.361(5)°, γ = 78.328(5)°, V = 935.4 Å^3, Z = 1, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.114, T = 293 K.

Source of material

All reagents and solvents employed were commercially available and used as received without further purification. The compound of 5-(4-carboxybenzyloxy)isophthalic acid (CIA) was synthesized by the procedure reported in the literature [1]. The mixture of CIA (0.05 g, 0.16 mmol), NaOH (0.02 g, 0.48 mmol) , $CaCl_2 \cdot 2H_2O$ (0.0263 g, 0.237 mmol) and 10 mL water was sealed in a 25 mL Teflon-lined stainless reactor and heated at 130 °C for 72 h under autogeneous pressure. With cooling to room temperature, colourless crystals suitable for X-ray diffraction were obtained.

Experimental details

All hydrogen atoms on carbons were generated geometrically with d(C—H) = 0.93-0.97 Å, and U_{iso}(H) = 1.2 U_{eq}(C) for atoms of CH_2 groups and aromatic ring. Hydrogen atoms of the water molecules were not localized.

Discussion

Recently, the rational design and synthesis of novel coordination polymers have attracted intense attention in the field of supra-molecular chemistry and crystal engineering. We have seen a related report [2], it has similarly used CIA as an organic ligand with the different metal ion Cd(II) to form a 3D structure in which the coordination mode of CIA is not the same as that in this paper.

In the asymmetric unit there are two crystallographically independent Ca(II) centers with different coordinations. The Ca1 is seven-coordinated by six O atoms from four independent ligands CIA and one water molecule, showing a pentagonal bipyramidal coordination. The bond lengths d(Ca—O) are in the range of 2.236(2)-2.5481(2) Å. Ca2 is six-coordinated by four O atoms from four independent CIA ligands in the equatorial positions and two water molecules in the apical positions of an octahedron. The bond lengths d(Ca—O) are 2.3113(2) and 2.334(2) Å, respectively. Each CIA anion connects five Ca(II) (three Ca1 and two Ca2) atoms to form a layer. The layers are linked by carboxylate groups of CIA anions to generate a three-dimensional framework.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.15 × 0.20 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	5.32 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6869, 4290
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3302
$N(param)_{refined}$:	259
Programs:	SHELXS-97, SHELXL-97 [3]

* Correspondence author (e-mail: majf247nenu@yahoo.com.cn)

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	2*i*	0.6914	–0.7212	0.4730	0.028
H(5)	2*i*	0.6724	–0.3140	0.4696	0.025
H(7)	2*i*	0.5115	–0.4073	0.1579	0.025
H(44A)	2*i*	0.7260	–0.4163	0.6964	0.038
H(44B)	2*i*	0.8646	–0.4173	0.6096	0.038
H(47)	2*i*	0.7362	–0.5355	0.9376	0.039
H(58)	2*i*	0.8681	–0.6569	1.1301	0.038
H(56)	2*i*	1.1974	–0.7404	0.8923	0.047
H(46)	2*i*	1.0688	–0.6150	0.7012	0.049

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ca(1)	2*i*	0.49216(4)	0.12032(4)	0.11653(4)	0.0299(2)	0.0142(2)	0.0139(2)	–0.0031(2)	–0.0056(2)	–0.0031(2)
Ca(2)	1*f*	½	0	½	0.0364(4)	0.0152(3)	0.0125(3)	–0.0059(2)	–0.0039(2)	–0.0026(2)
C(1)	2*i*	0.5512(2)	–0.6870(2)	0.2469(2)	0.031(1)	0.0151(9)	0.0186(9)	–0.0040(8)	–0.0022(8)	–0.0031(8)
C(2)	2*i*	0.5939(2)	–0.5804(2)	0.3078(2)	0.029(1)	0.0154(9)	0.0167(9)	–0.0045(8)	–0.0016(8)	–0.0031(8)
C(3)	2*i*	0.6653(2)	–0.6247(2)	0.4301(2)	0.033(1)	0.0145(9)	0.021(1)	–0.0026(8)	–0.0066(8)	–0.0005(8)
C(4)	2*i*	0.6982(2)	–0.5244(2)	0.4889(2)	0.030(1)	0.021(1)	0.0182(9)	–0.0028(9)	–0.0077(8)	–0.0009(8)
C(5)	2*i*	0.6545(2)	–0.3810(2)	0.4281(2)	0.029(1)	0.0182(9)	0.0171(9)	–0.0041(8)	–0.0055(8)	–0.0052(8)
C(6)	2*i*	0.5834(2)	–0.3372(2)	0.3041(2)	0.026(1)	0.0142(9)	0.0160(9)	–0.0034(8)	–0.0019(8)	–0.0023(7)
C(7)	2*i*	0.5555(2)	–0.4367(2)	0.2424(2)	0.030(1)	0.0175(9)	0.0146(9)	–0.0047(8)	–0.0041(8)	–0.0020(7)
C(8)	2*i*	0.5347(2)	–0.1823(2)	0.2408(2)	0.026(1)	0.0163(9)	0.0158(9)	–0.0031(8)	–0.0030(8)	–0.0038(8)
C(9)	2*i*	0.8087(3)	–0.4769(2)	0.6725(2)	0.042(1)	0.027(1)	0.028(1)	–0.005(1)	–0.015(1)	–0.0061(9)
C(10)	2*i*	0.8900(2)	–0.5606(2)	0.7988(2)	0.034(1)	0.027(1)	0.027(1)	–0.004(1)	–0.0120(9)	–0.0057(9)
C(11)	2*i*	0.8294(2)	–0.5750(2)	0.9283(2)	0.028(1)	0.035(1)	0.033(1)	–0.000(1)	–0.0080(9)	–0.007(1)
C(12)	2*i*	0.9086(2)	–0.6487(3)	1.0436(2)	0.031(1)	0.038(1)	0.024(1)	–0.004(1)	–0.0031(9)	–0.004(1)
C(13)	2*i*	1.0471(2)	–0.7103(2)	1.0321(2)	0.028(1)	0.031(1)	0.024(1)	–0.0050(9)	–0.0075(9)	–0.0039(9)
C(14)	2*i*	1.1340(3)	–0.7857(3)	1.1570(2)	0.032(1)	0.032(1)	0.030(1)	–0.005(1)	–0.0105(9)	–0.002(1)
C(15)	2*i*	1.1053(3)	–0.6979(3)	0.9020(2)	0.026(1)	0.054(2)	0.031(1)	0.003(1)	–0.004(1)	–0.008(1)
C(16)	2*i*	1.0279(3)	–0.6234(3)	0.7875(2)	0.037(1)	0.057(2)	0.023(1)	–0.001(1)	–0.004(1)	–0.008(1)
O(1)	2*i*	1.0766(2)	–0.8000(2)	1.2721(2)	0.042(1)	0.068(1)	0.0241(8)	–0.0016(9)	–0.0090(7)	–0.0009(9)
O(2)	2*i*	1.2610(2)	–0.8319(2)	1.1408(2)	0.028(1)	0.058(1)	0.042(1)	0.0035(8)	–0.0109(8)	–0.0036(9)
O(3)	2*i*	0.7708(2)	–0.5780(2)	0.6096(2)	0.051(1)	0.0207(8)	0.0269(8)	–0.0027(7)	–0.0231(7)	–0.0024(6)
O(4)	2*i*	0.5459(2)	–0.8108(1)	0.3239(1)	0.050(1)	0.0151(7)	0.0194(7)	–0.0106(7)	–0.0061(6)	–0.0011(6)
O(5)	2*i*	0.5201(2)	–0.6512(2)	0.1237(1)	0.067(1)	0.0187(7)	0.0195(7)	–0.0118(8)	–0.0135(7)	–0.0018(6)
O(6)	2*i*	0.5206(2)	–0.0961(1)	0.3139(1)	0.053(1)	0.0147(7)	0.0189(7)	–0.0016(7)	–0.0076(7)	–0.0066(6)
O(7)	2*i*	0.5080(2)	–0.1376(1)	0.1146(1)	0.048(1)	0.0169(7)	0.0133(7)	–0.0045(7)	–0.0086(6)	–0.0007(6)
O(1W)	2*i*	0.2597(2)	0.0722(2)	0.4744(2)	0.037(1)	0.072(1)	0.0334(9)	–0.007(1)	–0.0096(8)	0.0043(9)
O(2W)	2*i*	0.7320(2)	0.0658(2)	0.0787(2)	0.034(1)	0.058(1)	0.057(1)	–0.0040(9)	–0.0012(9)	–0.016(1)
O(3W)	2*i*	–0.0905(2)	0.0035(3)	0.2964(2)	0.044(1)	0.086(2)	0.052(1)	–0.015(1)	–0.002(1)	0.004(1)
O(4W)	2*i*	0.8233(3)	–0.8727(2)	0.7853(2)	0.078(2)	0.048(1)	0.084(2)	–0.017(1)	0.003(1)	–0.008(1)

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References

1. He, Y.-H.; Feng, Y.-L.; Lan, Y.-Z.; Wen, Y.-H.:Syntheses, Structures, and Photoluminescence of Four d^{10} Metal-Organic Frameworks Constructed from 3,5-Bis-oxyacetate-benzoic Acid. Cryst. Growth Des. **8** (2008) 3586-3594.
2. Zhang, X.; Yang, J.-X.; Cheng, J.-K.; Sun, M.-L.; Yao, Y.-G.: A new open framework material based on designed semi-rigid T-shaped tricarboxylate ligand. Inorg. Chem. Commun. **14** (2011) 986-989.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.