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catena-Poly[[diaquacalcium(II)]-μ_3-terephthalato-μ_2-aqua] at 150 K

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The title compound, $[Ca(C_8H_4O_4)(H_2O)_3]_n$, has been previously studied by single-crystal X-ray diffraction at both room temperature [Matsuzaki & Iitaka (1972). *Acta Cryst.* B**28**, 1977–1981] and 173 K [Groeneman & Atwood (1999). *Cryst. Eng.* **2**, 241–249.]. We present here a redetermination of the compound at 150 K, which results in a considerably improved precision and the location of H atoms in the strong O—H···O hydrogen bonds.

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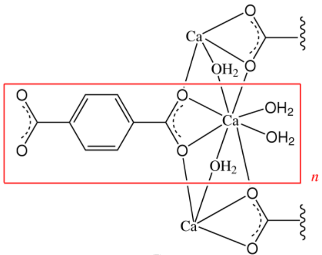
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.143
Data-to-parameter ratio = 14.5

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

Comment

The title compound, (I), calcium benzene-1,4-dicarboxylate (terephthalate) trihydrate ($C_8H_{10}CaO_7$, Ca[TA]·$3H_2O$), has been previously studied at both room temperature (Matsuzaki & Iitaka, 1972) and 173 K (Groeneman & Atwood, 1999) using single-crystal X-ray diffraction, without the location of H atoms involved in the O—H···O hydrogen bonds in either case.



(I)

We have redetermined this structure at 150 K (Fig. 1), finding the same one-dimensional coordination polymeric structure linked into a three-dimensional array by a network of hydrogen bonds (Fig. 2). Bond lengths were found to be similar to those in the earlier determinations (Table 1); however, we have successfully located the H atoms involved in the strong O—H···O hydrogen bonds (Table 2) and vastly improved the precision of the structure [$R1$ ($F^2 > 2\sigma(F^2)$ = 0.054 at 150 K, compared with $R1 = 0.110$ at 173 K and $R1 = 0.135$ at room temperature].

Experimental

Terephthalic acid and $CaCO_3$ were refluxed together in a 1:1 ratio in H_2O for 18 h. Colourless X-ray-quality crystals of the title compound were grown by the slow cooling of this solution. IR (KBr, ν_{max}, cm^{-1}): 3419 (br, OH), 1634 (aromatic C=C), 1574 (asymmetric CO_2^-), 1505 (aromatic C=C), 1402 and 1389 (symmetric CO_2^-), 1312, 1155, 1103 and 1026 (C—O), 826 and 755 (aromatic C—H), 559, 515.

Crystal data

$[Ca(C_8H_4O_4)(H_2O)_3]$
$M_r = 258.24$
Monoclinic, $P2_1/c$
$a = 7.0455\ (15)$ Å
$b = 21.623\ (4)$ Å
$c = 6.5622\ (13)$ Å
$\beta = 92.831\ (3)^\circ$
$V = 998.5\ (3)$ Å^3
$Z = 4$

$D_x = 1.718$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2161 reflections
$\theta = 2.9$–27.9°
$\mu = 0.65$ mm^{-1}
$T = 150\ (2)$ K
Column, colourless
$0.78 \times 0.06 \times 0.05$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
Narrow frame ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.632$, $T_{max} = 0.968$
8282 measured reflections

2362 independent reflections
1751 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 28.7^\circ$
$h = -8 \rightarrow 9$
$k = -29 \rightarrow 28$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.143$
$S = 1.05$
2362 reflections
163 parameters
H atoms refined by a mixture of constrained and independent refinement

$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2 + 0.4161P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.40$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 1
Selected interatomic distances (Å).

Ca1—O2	2.339 (2)	Ca1—O1^{i}	2.479 (2)
Ca1—O1^{ii}	2.357 (2)	Ca1—O5	2.498 (2)
Ca1—O6	2.368 (2)	Ca1—O2^{i}	2.526 (2)
Ca1—O7	2.380 (3)	Ca1—O5^{v}	2.633 (2)

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O5—H5$A\cdots$O4^{vi}	0.80 (2)	1.93 (2)	2.717 (3)	172 (4)
O5—H5$B\cdots$O3^{vii}	0.80 (2)	1.97 (2)	2.755 (3)	167 (4)
O6—H6$B\cdots$O3^{vii}	0.81 (2)	1.93 (2)	2.737 (3)	173 (4)
O6—H6$A\cdots$O4^{viii}	0.80 (2)	1.98 (2)	2.754 (3)	162 (4)
O7—H7$A\cdots$O4^{ix}	0.80 (2)	2.13 (2)	2.914 (3)	165 (4)
O7—H7$B\cdots$O3^{x}	0.81 (2)	2.12 (3)	2.898 (3)	164 (4)

Symmetry codes: (vi) $-x, y - \frac{1}{2}, \frac{3}{2} - z$; (vii) $-x, 2 - y, 1 - z$; (viii) $-x, 2 - y, 2 - z$; (ix) $1 - x, 2 - y, 2 - z$; (x) $1 - x, 2 - y, 1 - z$.

Aromatic H atoms were placed in calculated positions and refined as riding, with a C—H distance of 0.95 Å, while the coordinates of the water H atoms were located in a difference Fourier map and refined using geometric restraints. U_{iso} values were set to $1.2U_{eq}$ for aryl H atoms and $1.5U_{eq}$ for water H atoms. The one residual peak greater than 1 e Å^{-3} is 1.08 Å from atom Ca1.

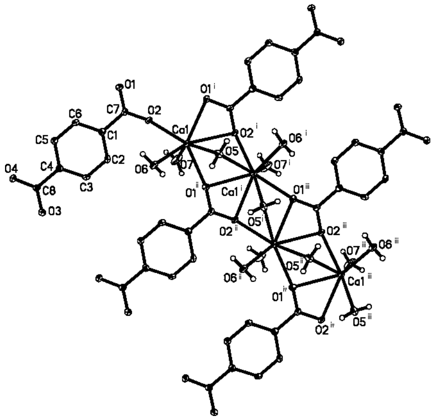


Figure 1
View of (I), showing the atom-labelling scheme and the one-dimensional array formed by the bridging terephthalate anions and water molecules. Displacement ellipsoids are drawn at the 50% probability level. Aromatic H atoms have been omitted for clarity, and water H atoms are represented by circles of arbitrary radii. [Symmetry codes: (i) $x, \frac{3}{2} - y, z - \frac{1}{2}$; (ii) $x, y, z - 1$; (iii) $x, \frac{3}{2} - y, z - \frac{3}{2}$; (iv) $x, y, z - 2$.]

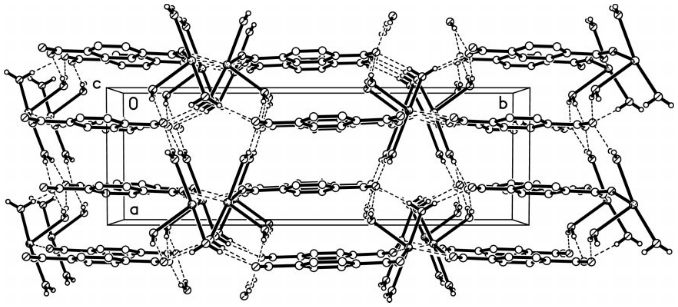


Figure 2
Packing plot of (I), viewed along the c axis, showing the three-dimensional structure produced by the linking of one-dimensional coordination polymers by O—H$\cdots$O hydrogen bonding. Hydrogen bonds are represented by dashed lines.

Data collection: *SMART* (Siemens, 1994); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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