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Poly[di-μ_3-aqua-μ_4-terephthalato-dicaesium]

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Key indicators

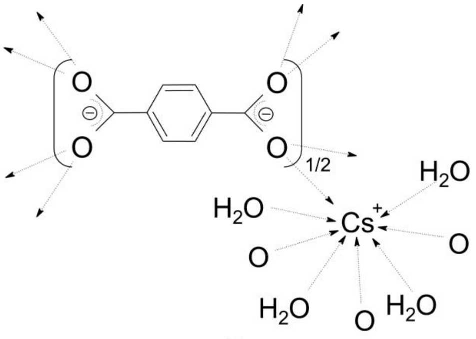
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.008 Å
R factor = 0.033
wR factor = 0.082
Data-to-parameter ratio = 17.0

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

Colourless platelets of dicaesium terephthalate dihydrate, $[Cs_2(C_8O_4H_4)(H_2O)_2]_n$, were obtained from an aqueous solution of Cs_2CO_3, NaOH and terephthalic acid in demineralized water and subsequent evaporation of the solvent at room temperature. Caesium is coordinated by four O atoms from four water molecules and four O atoms from four different centrosymmetric terephthalates. The metal centres are connected by the water molecules, forming a double layer parallel to (011). These double layers are linked by the terephthalate dianions, forming a three-dimensional framework structure.

Comment

The dianion of terephthalic acid (benzene-1,4-dicarboxylic acid) is a versatile and well established ligand for the construction of coordination polymers. Surprisingly, little is known about the structural chemistry of simple terephthalate salts of monopositive cations. Kaduk (2000) investigated the crystal structures of dilithium, disodium and dipotassium terephthalate, which crystallized as anhydrous salts. This mainly confirmed earlier work by Ebara and Furuyama (Furuyama & Ebara, 1967; Ebara & Furuyama, 1973), who also investigated the crystal structure of anhydrous dirubidium terephthalate (Furuyama & Ebara, 1967). Just recently, we were able to synthesize and characterize the crystal structure of dirubidium terephthalate dihydrate (Stein & Ruschewitz, 2006), which crystallizes in a crystal structure isotypic with that of the title compound, (I).



(I)

From an aqueous solution containing Cs_2CO_3, NaOH and terephthalic acid, single crystals of (I) were obtained by slow evaporation of the solvent. The crystal structure contains distorted CsO_8 square antiprisms (Fig. 1). Four of the eight O atoms are from four different but symmetry-equivalent centrosymmetric terephthalate anions [Cs—O = 3.027 (4)–

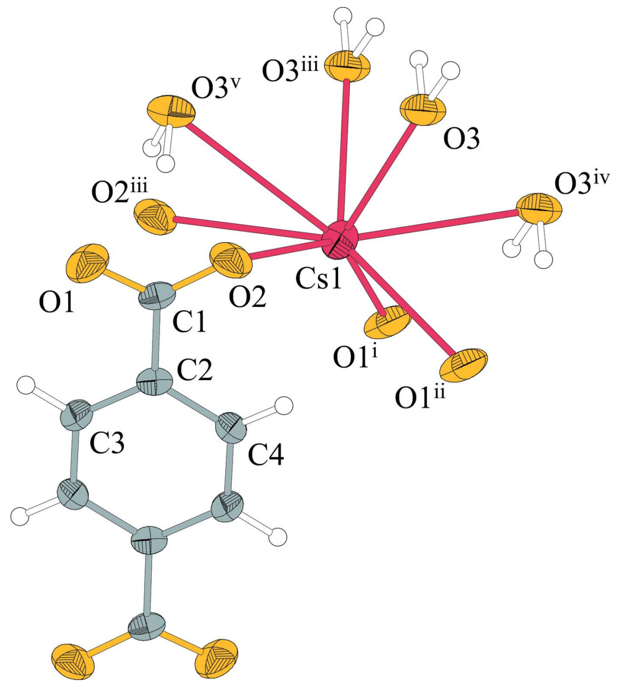


Figure 1
The asymmetric unit of (I), expanded to show the complete terephthalate and the Cs coordination. Unlabelled atoms in the terephthalate are related to labelled atoms by $(2 - x, 1 - y, 1 - z)$. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (ii) $x, \frac{3}{2} - y, -\frac{1}{2} + z$; (iii) $x, -1 + y, z$; (iv) $1 - x, 1 - y, -z$; (v) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$.]

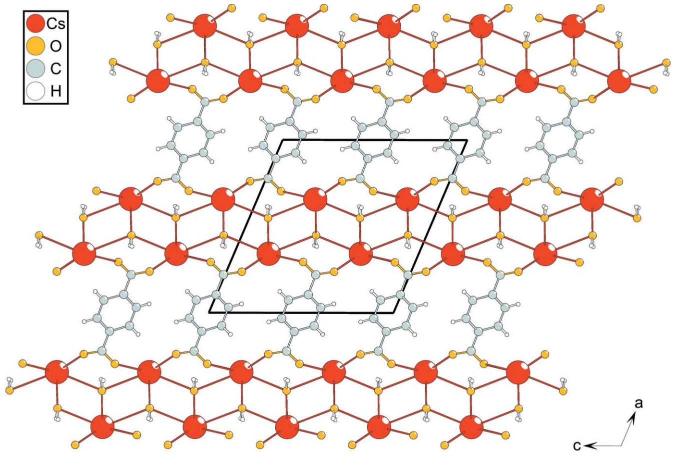


Figure 2
View of the crystal structure of (I) in a projection along [010].

3.229 (4) Å]. The coordination is completed by four water molecules [Cs—O = 3.163 (4)–3.436 (5) Å]. Each terephthalate anion connects eight caesium ions and each water molecule four caesium ions so that the caesium ions and water molecules form a double layer parallel to (011). The shortest Cs···Cs distance within the double layers is 4.1724 (7) Å. These layers are linked by the terephthalate anions along [100], forming a three-dimensional framework structure (Fig. 2).

Experimental

Terephthalic acid (0.17 g, 1.0 mmol) was suspended in 5 ml of deionized water and dissolved by the addition of 1.0 *M* NaOH, until a pH of 7 was reached. A solution of caesium carbonate (0.33 g, 1.0 mmol) in 5 ml of deionized water was then added. The beaker with the solution was sealed with a perforated foil and the solvent was allowed to evaporate at room temperature. After a few days colourless platelets of (I) were obtained. X-ray powder patterns revealed that the sample is not a single phase; accordingly, no yield was determined.

Crystal data

$[Cs_2(C_8H_4O_4)(H_2O)_2]$
$M_r = 232.98$
Monoclinic, $P2_1/c$
$a = 12.729$ (4) Å
$b = 4.1724$ (7) Å
$c = 12.498$ (4) Å
$\beta = 113.10$ (3)°
$V = 610.6$ (3) Å^3
$Z = 4$
$D_x = 2.535$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 5.98$ mm^{-1}
$T = 295$ (2) K
Plate, colourless
0.4 × 0.3 × 0.07 mm

Data collection

Stoe IPDS-I diffractometer
φ scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 2001); crystal shape optimized by *X-SHAPE* (Stoe & Cie, 1999)
$T_{min} = 0.270$, $T_{max} = 0.637$
6555 measured reflections
1431 independent reflections
1088 reflections with $I > 2\sigma(I)$
$R_{int} = 0.088$
$\theta_{max} = 28.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.082$
$S = 0.96$
1431 reflections
84 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.82$ e Å^{-3}
$\Delta\rho_{min} = -1.38$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.013 (1)

Table 1
Selected bond lengths (Å).

Cs1—O1^{i}	3.027 (4)	Cs1—O3^{v}	3.436 (5)
Cs1—O1^{ii}	3.030 (4)	O1—C1	1.244 (6)
Cs1—O2^{iii}	3.125 (4)	O2—C1	1.275 (7)
Cs1—O3	3.163 (4)	C1—C2	1.505 (6)
Cs1—O2	3.229 (4)	C2—C3	1.385 (7)
Cs1—O3^{iii}	3.285 (4)	C2—C4	1.408 (7)
Cs1—O3^{iv}	3.301 (5)	C3—C4^{vi}	1.387 (7)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O2^{v}	0.96 (2)	1.85 (3)	2.772 (6)	160 (7)
O3—H2···O2^{vii}	0.96 (2)	2.18 (6)	3.021 (6)	146 (8)
O3—H2···O1^{vii}	0.96 (2)	2.42 (4)	3.314 (7)	155 (8)

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

H3 and H4 of the terephthalate anion were placed at calculated positions and refined as riding, with C—H = 0.93 Å and $U_{\rm iso}$(H) refined freely. The H atoms of the water molecule were located in difference Fourier maps and refined isotropically with a restrained O—H distance of 0.96 (2) Å. The deepest hole in the final difference Fourier map was 2.15 Å from the Cs atom.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2001); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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