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Caesium(I) hydrogen phthalate

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Key indicators

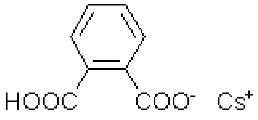
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.027
wR factor = 0.073
Data-to-parameter ratio = 11.6

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

The title compound, $Cs^+\cdot C_8H_5O_4^-$, crystallizes in the orthorhombic space group $Pca2_1$ and is isostructural with $Tl^+\cdot C_8H_5O_4^-$, $K^+\cdot C_8H_5O_4^-$ and $Rb^+\cdot C_8H_5O_4^-$. The Cs^+ cation is surrounded by eight O atoms. The crystal packing is stabilized by intermolecular O—H···O hydrogen bonds.

Comment

Phthalic acid is a small carboxylic acid that has special relevance to environmental chemists and geochemists. It represents common functional groups found in natural organic matter (Salzjimenez, 1994). Binding of the common alkali-metal (Na^+ and K^+) (Emsley, 1991) and alkaline-earth-metal (Mg^{2+} and Ca^{2+}) (Hartwig, 2000) cations to biorelevant ligands is of great current interest, since these metals are abundant in biological systems. In the past, much structural work was performed on phthalate complexes of some metal ions such as $Tl^+\cdot C_8H_5O_4^-$ (Liu & Zhang, 1985), $Rb^+\cdot C_8H_5O_4^-$ (Smith, 1975) and $K^+\cdot C_8H_5O_4^-$ (Okaya, 1965). We report here the single-crystal structure of $Cs^+\cdot C_8H_5O_4^-$, (I). It was found to be orthorhombic with unit-cell dimensions close to those published earlier by Okaya & Pepinsky (1957).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1. The structure is essentially ionic, containing a caesium cation and a hydrogen phthalate ion. The Cs^+ cations are eight-coordinated by the carboxyl O atoms of four anions. The Cs—O distances cover a wide range from 2.962 (4) to 3.529 (8) Å. In the solid state, (I) is isostructural with $Tl^+\cdot C_8H_5O_4^-$, $K^+\cdot C_8H_5O_4^-$ and $Rb^+\cdot C_8H_5O_4^-$.

The crystal packing is stabilized by intermolecular O—H···O hydrogen bonds (Table 2 and Fig. 2).

Experimental

Phthalic anhydride (18.7 mmol) was added to a solution of CsOH (18.7 mmol) in water (5 ml). The mixture was stirred for 2 h at 333 K. The solution was then filtered under reduced pressure and set aside for crystallization. After 7 d, pure white crystals of (I) were collected. Cs^+ was determined by Tas-986 atomic absorption spectrometry at wavelength 852.1 nm. Analysis calculated: C 32.21, H 1.68, Cs 44.59%; found: C 31.63, H 1.62, Cs 44.30%. IR (KBr, cm^{-1}): 3397, 1673, 1561, 1482, 1385, 1290, 1147, 1083, 853, 807, 759. Raman spectra (cm^{-1}): 3064, 1676, 1596, 1482, 1411, 1379, 1150, 1036, 854, 808, 791.

Crystal data

$\text{Cs}^+\cdot\text{C}_8\text{H}_5\text{O}_4^-$
$M_r = 298.03$
Orthorhombic, $Pca2_1$
$a = 10.659\ (5)$ Å
$b = 12.768\ (6)$ Å
$c = 6.556\ (3)$ Å
$V = 892.2\ (7)$ Å^3
$Z = 4$
$D_x = 2.219$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3510 reflections
$\theta = 2.5$–$28.1°$
$\mu = 4.13$ mm^{-1}
$T = 293\ (2)$ K
Prism, colorless
$0.49 \times 0.41 \times 0.28$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.144$, $T_{\max} = 0.315$
4329 measured reflections

1364 independent reflections
1272 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.040$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 15$
$l = -5 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.073$
$S = 1.01$
1364 reflections
118 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.63$ e Å^{-3}
$\Delta\rho_{\min} = -1.03$ e Å^{-3}
Absolute structure: Flack (1983), 498 Friedel pairs
Flack parameter = 0.16 (4)

Table 1
Selected geometric parameters (Å, °).

Cs1—O3^{i}	2.962 (4)	Cs1—O3	3.186 (8)
$\text{Cs1—O1}^{\text{ii}}$	3.092 (4)	$\text{Cs1—O3}^{\text{ii}}$	3.243 (4)
$\text{Cs1—O4}^{\text{iii}}$	3.157 (5)	Cs1—O1	3.427 (5)
$\text{Cs1—O4}^{\text{ii}}$	3.157 (4)	$\text{Cs1—O3}^{\text{iii}}$	3.529 (8)
$\text{O3}^{\text{i}}\text{—Cs1—O1}^{\text{ii}}$	88.75 (14)	$\text{O4}^{\text{iii}}\text{—Cs1—O3}$	129.06 (11)
$\text{O3}^{\text{i}}\text{—Cs1—O4}^{\text{iii}}$	108.09 (14)	$\text{O4}^{\text{ii}}\text{—Cs1—O3}$	85.41 (11)
$\text{O1}^{\text{ii}}\text{—Cs1—O4}^{\text{iii}}$	93.05 (12)	$\text{O3}^{\text{i}}\text{—Cs1—O3}^{\text{ii}}$	152.59 (10)
$\text{O3}^{\text{i}}\text{—Cs1—O4}^{\text{ii}}$	118.55 (11)	$\text{O1}^{\text{ii}}\text{—Cs1—O3}^{\text{ii}}$	65.84 (13)
$\text{O1}^{\text{ii}}\text{—Cs1—O4}^{\text{ii}}$	59.00 (12)	$\text{O4}^{\text{iii}}\text{—Cs1—O3}^{\text{ii}}$	84.47 (14)
$\text{O4}^{\text{iii}}\text{—Cs1—O4}^{\text{ii}}$	122.97 (9)	$\text{O4}^{\text{ii}}\text{—Cs1—O3}^{\text{ii}}$	39.84 (12)
$\text{O3}^{\text{i}}\text{—Cs1—O3}$	87.57 (16)	$\text{O3—Cs1—O3}^{\text{ii}}$	103.54 (17)
$\text{O1}^{\text{ii}}\text{—Cs1—O3}$	136.54 (11)		

Symmetry codes: (i) $\frac{5}{2} - x, y, \frac{1}{2} + z$; (ii) $2 - x, 2 - y, \frac{1}{2} + z$; (iii) $x, y, 1 + z$.

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

$D\text{—H}\cdots A$	D—H	H$\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
$\text{O2—H2}\cdots\text{O4}^{\text{iv}}$	0.82	1.71	2.52 (1)	173 (6)

Symmetry code: (iv) $\frac{3}{2} - x, y, \frac{1}{2} + z$.

H atoms were placed in calculated positions (O—H = 0.82, C—H = 0.93 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{O})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

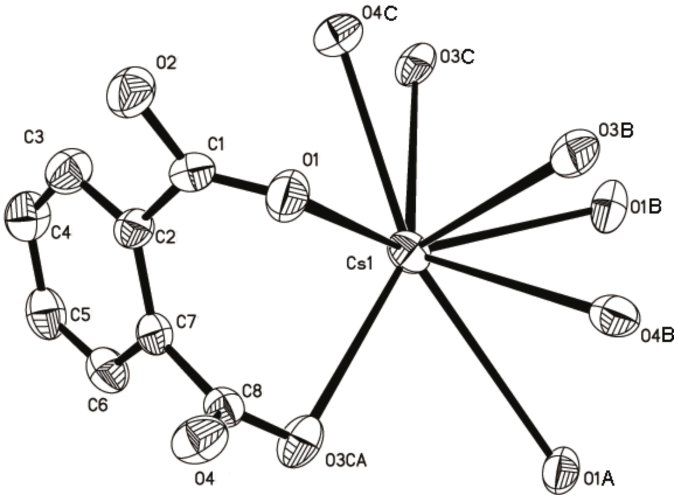


Figure 1
The eight-coordinated caesium cation of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. Atoms marked with an A, B or C are at the symmetry positions $(2 - x, 2 - y, z + \frac{1}{2})$, $(x, y, z + 1)$ or $(\frac{5}{2} - x, y, z+1/2)$, respectively.

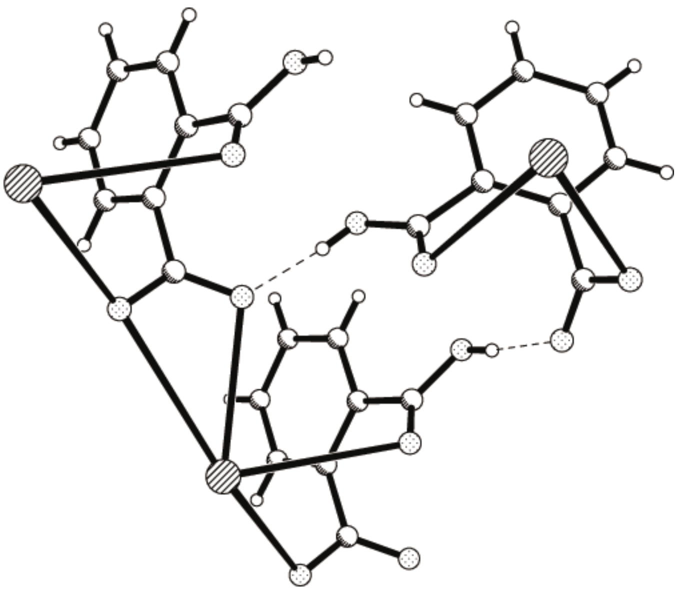


Figure 2
Intermolecular O—H$\cdots$O hydrogen bonds (dashed lines) in (I).

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