

Crystal structure of caesium dihydrogen citrate from laboratory X-ray powder diffraction data and DFT comparison

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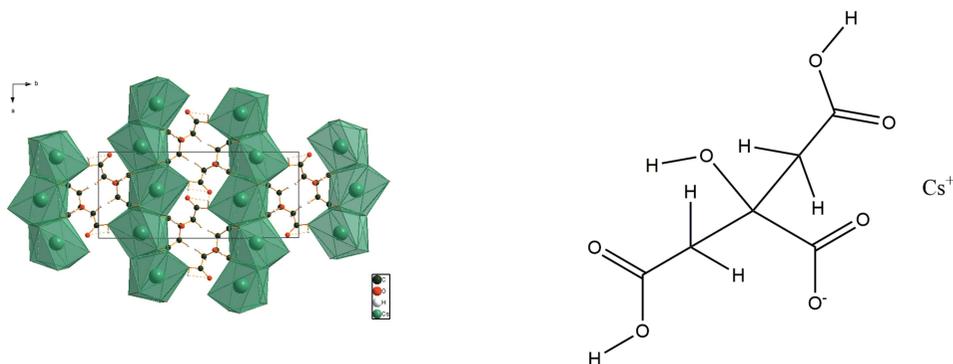
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Supporting information: this article has supporting information at journals.iucr.org/e

The crystal structure of caesium dihydrogen citrate, $\text{Cs}^+\cdot\text{H}_2\text{C}_6\text{H}_5\text{O}_7^-$, has been solved and refined using laboratory X-ray powder diffraction data, and optimized using density functional techniques. The coordination polyhedra of the nine-coordinate Cs^+ cations share edges to form chains along the *a*-axis. These chains are linked by corners along the *c*-axis. The un-ionized carboxylic acid groups form two different types of hydrogen bonds; one forms a helical chain along the *c*-axis, and the other is discrete. The hydroxy group participates in both intra- and intermolecular hydrogen bonds.

1. Chemical context

In the course of a systematic study of the crystal structures of Group 1 (alkali metal) citrate salts to understand the anion's conformational flexibility, ionization, coordination tendencies, and hydrogen bonding, we have determined several new crystal structures. Most of the new structures were solved using powder diffraction data (laboratory and/or synchrotron), but single crystals were used where available. The general trends and conclusions about the 16 new compounds and 12 previously characterized structures are being reported separately (Rammohan & Kaduk, 2017*a*). Ten of the new structures – $\text{NaKHC}_6\text{H}_5\text{O}_7$, $\text{NaK}_2\text{C}_6\text{H}_5\text{O}_7$, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$, $\text{NaH}_2\text{C}_6\text{H}_5\text{O}_7$, $\text{Na}_2\text{HC}_6\text{H}_5\text{O}_7$, $\text{K}_3\text{C}_6\text{H}_5\text{O}_7$, $\text{Rb}_2\text{HC}_6\text{H}_5\text{O}_7$, $\text{Rb}_3\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})$, $\text{Rb}_3\text{C}_6\text{H}_5\text{O}_7$, and $\text{Na}_5\text{H}(\text{C}_6\text{H}_5\text{O}_7)_2$ – have been published recently (Rammohan & Kaduk, 2016*a,b,c,d,e*, 2017*b,c,d,e*; Rammohan *et al.*, 2016), and two additional structures – $\text{KH}_2\text{C}_6\text{H}_5\text{O}_7$ and $\text{KH}_2\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})_2$ – have been communicated to the CSD (Kaduk & Stern, 2016*a,b*).



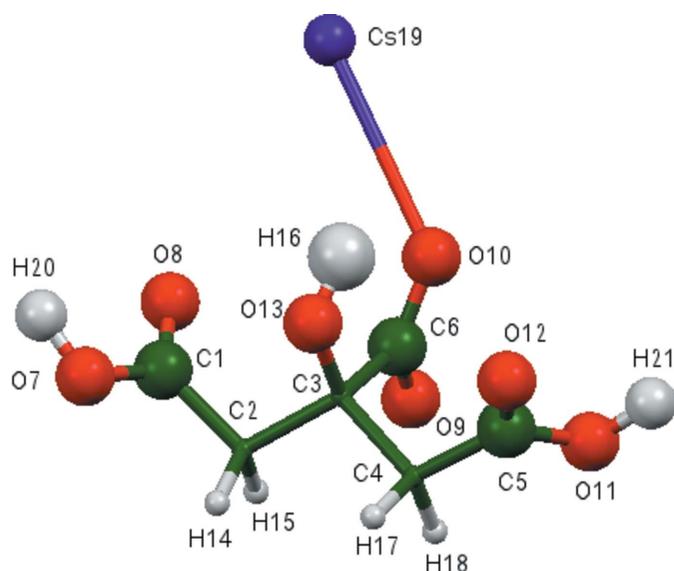


Figure 1
The asymmetric unit, with the atom numbering. The atoms are represented by 50% probability spheroids.

2. Structural commentary

The asymmetric unit of the title compound is shown in Fig. 1. The root-mean-square deviation of the non-hydrogen atoms in the Rietveld-refined and DFT-optimized structures is 0.387 Å (Fig. 2). This agreement is at the upper end of the range of correct structures as discussed by van de Streek & Neumann (2014). Re-starting the Rietveld refinement from the DFT-optimized structure led to higher residuals ($R_{wp} = 0.1287$ and $\chi^2 = 26.43$). Accurate determination of the positions of C and O atoms in the presence of the heavy Cs atoms using X-ray powder data might be expected to be difficult. This discussion uses the DFT-optimized structure. Most of the bond lengths, bond angles, and torsion angles fall within the normal ranges indicated by a *Mercury* Mogul geometry check (Macrae *et al.*, 2008), but the torsion angles involving the central carboxylate and hydroxyl group are flagged as unusual; the central portion of the molecule is less-planar than usual. In the refined structure, the O8—C1 and O10—C6 bonds, as well as the C3—C2—C1 angle, were flagged as unusual. The citrate anion

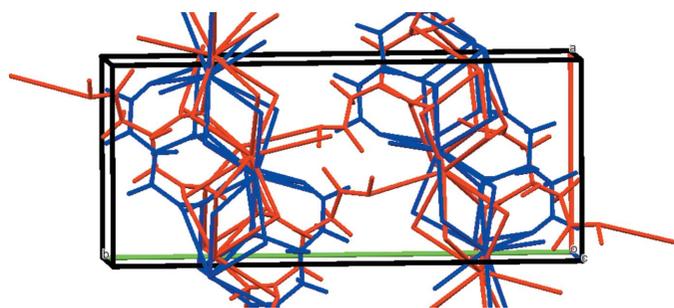


Figure 2
Comparison of the refined and optimized structures of caesium dihydrogen citrate. The refined structure is in red, and the DFT-optimized structure is in blue.

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O11—H21 \cdots O10 ⁱ	1.028	1.575	2.600	174.4
O7—H20 \cdots O8 ⁱⁱ	0.996	1.674	2.637	161.7
O13—H16 \cdots O9 ⁱⁱⁱ	0.979	1.985	2.865	148.4
O13—H16 \cdots O10	0.979	2.149	2.691	113.3

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

occurs in the *trans,trans* conformation, which is one of the two low-energy conformations of an isolated citrate. The central carboxylate O10 and the terminal carboxylate O12 atoms chelate to the Cs⁺ cation. The Mulliken overlap populations and atomic charges indicate that the metal-oxygen bonding is ionic.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) morphology suggests that we might expect a platy morphology for caesium dihydrogen citrate, with {020} as the principal faces. A 4th-order spherical harmonic texture model was included in the refinement. The texture index was 1.183, indicating that preferred orientation was significant for this rotated flat-plate specimen.

3. Supramolecular features

The nine-coordinate Cs⁺ cation (bond-valence sum 0.96) share edges to form chains along the *a* axis (Fig. 3). These chains are linked by corners along the *c* axis. The O7—H20 \cdots O8 hydrogen bonds (Table 1) form a helical chain along the *c* axis, and the O11—H21 \cdots O10 hydrogen bonds are discrete. The Mulliken overlap populations in these hydrogen bonds are 0.064 and 0.095 *e*, respectively. By the correlation in Rammohan & Kaduk (2017*a*), these hydrogen bonds contribute 13.8 and 16.8 kcal mol⁻¹ to the crystal energy. The hydroxy group O13—H16 acts as a donor in two hydrogen bonds. The one to O10 is intramolecular, with a graph-set symbol *S*(5). The one to O9 is intermolecular, with a graph set symbol *S*(7). These hydrogen bonds are weaker, contributing 11.2 and 9.1 kcal mol⁻¹ to the crystal energy.

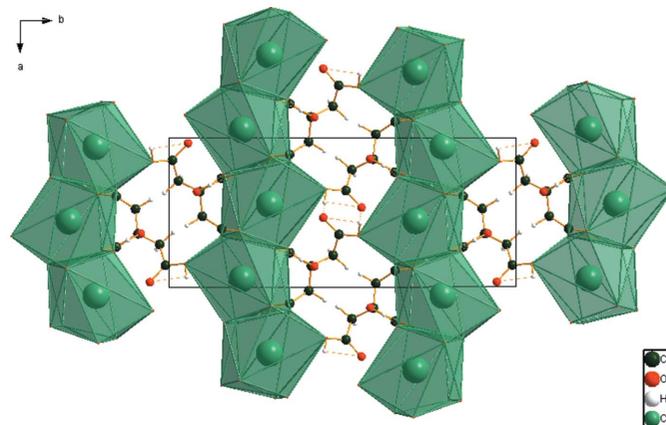
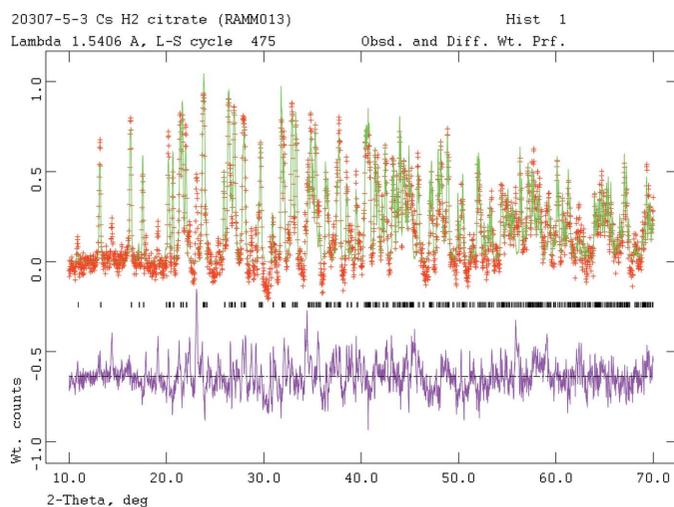


Figure 3
Crystal structure of CsH₂C₆H₅O₇, viewed down the *c*-axis.


Figure 4

Rietveld plot for the refinement of $\text{CsH}_2\text{C}_6\text{H}_5\text{O}_7$. The vertical scale is not the raw counts but the counts multiplied by the least squares weights. This plot emphasizes the fit of the weaker peaks. The red crosses represent the observed data points, and the green line is the calculated pattern. The magenta curve is the difference pattern, plotted at the same scale as the other patterns. The row of black tick marks indicates the reflection positions.

4. Database survey

Details of the comprehensive literature search for citrate structures are presented in Rammohan & Kaduk (2017a). A reduced-cell search of the cell of cesium dihydrogen citrate in the Cambridge Structural Database (Groom *et al.*, 2016) (increasing the default tolerance from 1.5 to 2.0%) yielded 60 hits, but combining the cell search with the elements C, H, Cs, and O only yielded no hits.

5. Synthesis and crystallization

$\text{H}_3\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})$ (2.0766 g, 10.0 mmol) was dissolved in 10 ml deionized water. Cs_2CO_3 (1.6508 g, 5.0 mmol, Sigma–Aldrich) was added to the citric acid solution slowly with stirring. A white precipitate formed in about two minutes, and the colourless solution was evaporated to dryness at ambient conditions.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. The powder pattern (Fig. 4) was indexed using *DICVOL06* (Louer & Boultif, 2007) [M/F(18) = 64/117] on a primitive orthorhombic unit cell having $a = 8.7362$ (2), $b = 20.5351$ (2), $c = 5.1682$ (5) Å, $V = 927.17$ (9) Å³, and $Z = 4$. The peak list from a Le Bail fit in *GSAS* was imported into Endeavour 1.7b (Putz *et al.*, 1999), and used for structure solution. The successful solution used a citrate, a Cs atom, and two oxygen atoms from water molecules. Initial Rietveld refinements moved the oxygens close to the Cs site, so they were deleted from the refinement.

Table 2

Experimental details.

	Powder data
Crystal data	
Chemical formula	$\text{Cs}^+ \cdot \text{H}_2\text{C}_6\text{H}_5\text{O}_7^-$
M_r	323.97
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	300
a, b, c (Å)	8.7362 (2), 20.53510 (16), 5.1682 (5)
V (Å ³)	927.17 (9)
Z	4
Radiation type	$K\alpha_1, K\alpha_2, \lambda = 1.540629, 1.544451$ Å
Specimen shape, size (mm)	Flat sheet, 24 × 24
Data collection	
Diffractometer	Bruker D2 Phaser
Specimen mounting	Standard holder
Data collection mode	Reflection
Scan method	Step
	$2\theta_{\min} = 5.042$ $2\theta_{\max} = 70.050$ $2\theta_{\text{step}} = 0.020$
2θ values (°)	
Refinement	
R factors and goodness of fit	$R_p = 0.068, R_{wp} = 0.089,$ $R_{\text{exp}} = 0.026, R(F^2) = 0.171,$ $\chi^2 = 11.765$
No. of parameters	57
No. of restraints	29
H-atom treatment	Only H-atom displacement parameters refined

The same symmetry and lattice parameters were used for the DFT calculation. Computer programs: *DIFFRAC.Measurement* (Bruker, 2009), *GSAS* (Larson & Von Dreele, 2004), *DIAMOND* (Crystal Impact, 2015) and *publicIF* (Westrip, 2010).

Pseudo-Voigt profile coefficients were as parameterized in Thompson *et al.* (1987) with profile coefficients for Simpson's rule integration of the pseudo-Voigt function according to Howard (1982). The asymmetry correction of Finger *et al.* (1994) was applied, and microstrain broadening by Stephens (1999). The structure was refined by the Rietveld method using *GSAS/EXPGUI* (Larson & Von Dreele, 2004; Toby, 2001).

All C–C and C–O bond lengths were restrained. The C–C bonds were restrained at 1.54 (1) Å, and the C3–O13 bond at 1.42 (2) Å. The C–O bonds in the carboxylate groups were restrained at 1.26 (2) Å. All angles were also restrained; the restraints were 109 (3)° for the angles around tetrahedral carbon atoms, and 120 (3)° for the angles in the planar carboxylate groups. The restraints contributed 3.0% to the final χ^2 . The hydrogen atoms were included at fixed positions, which were recalculated during the course of the refinement using *Materials Studio* (Dassault Systèmes, 2014).

7. DFT calculations

A density functional geometry optimization (fixed experimental unit cell) was carried out using *CRYSTAL09* (Dovesi *et al.*, 2005). The basis sets for the C, H, and O atoms were those of Gatti *et al.* (1994), and the basis set for Cs was that of Prencipe (1990). The calculation used 8 k -points and the B3LYP functional, and took about 59 h on a 2.4 GHz PC. U_{iso}

were assigned to the optimized fractional coordinates based on the U_{iso} from the refined structure.

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supporting information

Acta Cryst. (2017). E73, 133-136 [https://doi.org/10.1107/S2056989017000135]

Crystal structure of caesium dihydrogen citrate from laboratory X-ray powder diffraction data and DFT comparison

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Computing details

Data collection: *DIFFRAC.Measurement* (Bruker, 2009) for RAMM013_publ. Program(s) used to refine structure: *GSAS* (Larson & Von Dreele, 2004) for RAMM013_publ. Molecular graphics: *DIAMOND* (Crystal Impact, 2015) for RAMM013_publ. Software used to prepare material for publication: *publCIF* (Westrip, 2010) for RAMM013_publ.

(RAMM013_publ) cesium dihydrogen citrate

Crystal data

$\text{Cs}^+\cdot\text{C}_6\text{H}_7\text{O}_7^-$

$M_r = 323.97$

Orthorhombic, *Pna2*₁

Hall symbol: P 2c -2n

$a = 8.7362$ (2) Å

$b = 20.53510$ (16) Å

$c = 5.1682$ (5) Å

$V = 927.17$ (9) Å³

$Z = 4$

$D_x = 2.321$ Mg m⁻³

$K\alpha_1, K\alpha_2$ radiation, $\lambda = 1.540629, 1.544451$ Å

$T = 300$ K

white

flat_sheet, 24 × 24 mm

Specimen preparation: Prepared at 295 K

Data collection

Bruker D2 Phaser

diffractometer

Specimen mounting: standard holder

Data collection mode: reflection

Scan method: step

$2\theta_{\min} = 5.042^\circ, 2\theta_{\max} = 70.050^\circ, 2\theta_{\text{step}} = 0.020^\circ$

Refinement

Least-squares matrix: full

$R_p = 0.068$

$R_{\text{wp}} = 0.089$

$R_{\text{exp}} = 0.026$

$R(F^2) = 0.17055$

3217 data points

Profile function: CW Profile function number 4

with 18 terms Pseudovoigt profile coefficients

as parameterized in P. Thompson, D.E. Cox &

J.B. Hastings (1987). *J. Appl. Cryst.*,20,79-83.

Asymmetry correction of L.W. Finger, D.E. Cox

& A. P. Jephcoat (1994). *J. Appl.*

Cryst.,27,892-900. Microstrain broadening by

P.W. Stephens, (1999). *J. Appl.*

Cryst.,32,281-289. #1(GU) = 1.718 #2(GV) =

0.000 #3(GW) = 4.751 #4(GP) = 0.000 #5(LX)

= 2.847 #6(pte) = 0.00 #7(trns) = 1.83 #8(shft)

= 5.2787 #9(sfec) = 0.00 #10(S/L) = 0.0315

#11(H/L) = 0.0005 #12(eta) = 0.9000 #13(S400)

= 1.7E-04 #14(S040) = 5.1E-06 #15(S004) =

1.4E-02 #16(S220) = -4.1E-05 #17(S202) =

5.1E-02 #18(S022) = 4.5E-04 Peak tails are

ignored where the intensity is below 0.0100

times the peak Aniso. broadening axis 0.0 0.0

1.0

57 parameters
 29 restraints
 Only H-atom displacement parameters refined
 Weighting scheme based on measured s.u.'s
 $(\Delta/\sigma)_{\max} = 0.05$

Background function: GSAS Background
 function number 1 with 6 terms. Shifted
 Chebyshev function of 1st kind 1: 1098.70 2:
 -707.295 3: 219.700 4: -87.7806 5: 41.2782 6:
 -44.6612

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1876 (17)	0.0459 (9)	0.281 (8)	0.065 (4)*
C2	0.347 (2)	0.0446 (9)	0.166 (6)	0.009 (9)*
C3	0.4423 (18)	0.0965 (6)	0.304 (5)	0.009 (9)*
C4	0.609 (2)	0.0896 (10)	0.212 (7)	0.009 (9)*
C5	0.706 (2)	0.1464 (9)	0.317 (6)	0.065 (4)*
C6	0.380 (3)	0.1665 (7)	0.241 (6)	0.065 (4)*
O7	0.130 (2)	-0.0065 (11)	0.333 (9)	0.065 (4)*
O8	0.107 (2)	0.0874 (9)	0.223 (13)	0.065 (4)*
O9	0.371 (5)	0.1862 (12)	0.010 (7)	0.065 (4)*
O10	0.351 (4)	0.2037 (12)	0.418 (7)	0.065 (4)*
O11	0.716 (3)	0.1978 (11)	0.185 (7)	0.065 (4)*
O12	0.730 (3)	0.1503 (12)	0.552 (7)	0.065 (4)*
O13	0.436 (3)	0.0847 (9)	0.577 (5)	0.065 (4)*
H14	0.39911	-0.00564	0.19584	0.012 (11)*
H15	0.34027	0.05603	-0.04959	0.012 (11)*
H16	0.31496	0.11870	0.64320	0.085 (6)*
H17	0.65810	0.04163	0.28558	0.012 (11)*
H18	0.61212	0.09008	-0.00850	0.012 (11)*
Cs19	0.0454 (3)	0.20017 (14)	0.7594	0.0505 (15)*
H20	0.06940	-0.05097	0.56860	0.05*
H21	0.67528	0.24300	0.25240	0.05*

Geometric parameters (\AA , $^\circ$)

C1—C2	1.509 (10)	O9—Cs19 ⁱⁱ	3.07 (3)
C1—O7	1.218 (18)	O10—C6	1.217 (19)
C1—O8	1.148 (17)	O10—O9	2.15 (2)
C2—C1	1.509 (10)	O10—Cs19	3.20 (4)
C2—C3	1.532 (10)	O10—Cs19 ⁱⁱⁱ	3.14 (4)
C3—C2	1.532 (10)	O11—C5	1.260 (19)
C3—C4	1.537 (10)	O11—O12	2.14 (3)
C3—C6	1.569 (9)	O11—Cs19 ^{iv}	3.62 (3)
C3—O13	1.429 (11)	O11—Cs19 ⁱⁱ	3.38 (3)
C4—C3	1.537 (10)	O11—Cs19 ⁱⁱⁱ	3.93 (3)
C4—C5	1.542 (10)	O12—C5	1.24 (2)
C5—C4	1.542 (10)	O12—O11	2.14 (3)
C5—O11	1.260 (19)	O12—Cs19 ^v	3.13 (3)
C5—O12	1.24 (2)	O12—Cs19 ⁱⁱⁱ	3.63 (3)
C6—C3	1.569 (9)	O13—C3	1.429 (11)
C6—O9	1.267 (19)	Cs19—O8	3.65 (5)

C6—O10	1.217 (19)	Cs19—O8 ^{vi}	3.37 (5)
O7—C1	1.218 (18)	Cs19—O9 ^{vi}	3.14 (4)
O7—O8	2.02 (2)	Cs19—O9 ^{vii}	3.07 (3)
O8—C1	1.148 (17)	Cs19—O10	3.20 (4)
O8—O7	2.02 (2)	Cs19—O10 ^{viii}	3.14 (4)
O8—Cs19 ⁱ	3.37 (5)	Cs19—O11 ^{ix}	3.62 (3)
O8—Cs19	3.65 (5)	Cs19—O11 ^{viii}	3.93 (3)
O9—C6	1.267 (19)	Cs19—O11 ^{vii}	3.38 (3)
O9—O10	2.15 (2)	Cs19—O12 ^x	3.13 (3)
O9—Cs19 ⁱ	3.14 (4)	Cs19—O12 ^{viii}	3.63 (3)
C2—C1—O7	116.8 (10)	C5—O11—Cs19 ⁱⁱ	147.2 (15)
C2—C1—O8	118.6 (10)	C5—O12—Cs19 ^v	120.3 (19)
O7—C1—O8	117.4 (10)	C3—O13—H16	114.5 (15)
C1—C2—C3	107.9 (8)	O8 ^{vi} —Cs19—O9 ^{vi}	60.0 (7)
C2—C3—C4	108.0 (8)	O8 ^{vi} —Cs19—O9 ^{vii}	107.5 (11)
C2—C3—C6	110.6 (8)	O8 ^{vi} —Cs19—O10	106.0 (7)
C2—C3—O13	108.6 (9)	O8 ^{vi} —Cs19—O10 ^{viii}	156.2 (7)
C4—C3—C6	110.2 (9)	O8 ^{vi} —Cs19—O11 ^{vii}	83.9 (9)
C4—C3—O13	109.1 (9)	O8 ^{vi} —Cs19—O12 ^x	99.1 (6)
C6—C3—O13	110.3 (8)	O9 ^{vi} —Cs19—O9 ^{vii}	110.2 (11)
C3—C4—C5	110.1 (9)	O9 ^{vi} —Cs19—O10	58.2 (5)
C4—C5—O11	118.7 (9)	O9 ^{vi} —Cs19—O10 ^{viii}	141.1 (6)
C4—C5—O12	119.1 (9)	O9 ^{vi} —Cs19—O11 ^{vii}	52.3 (7)
O11—C5—O12	118.0 (10)	O9 ^{vi} —Cs19—O12 ^x	155.4 (7)
C3—C6—O9	120.8 (8)	O9 ^{vii} —Cs19—O10	129.0 (7)
C3—C6—O10	119.5 (8)	O9 ^{vii} —Cs19—O10 ^{viii}	59.5 (5)
O9—C6—O10	119.5 (8)	O9 ^{vii} —Cs19—O11 ^{vii}	58.4 (7)
C1—O8—Cs19 ⁱ	142 (2)	O9 ^{vii} —Cs19—O12 ^x	87.4 (8)
C6—O9—Cs19 ⁱ	119 (3)	O10—Cs19—O10 ^{viii}	97.2 (9)
C6—O9—Cs19 ⁱⁱ	127 (3)	O10—Cs19—O11 ^{vii}	88.6 (7)
Cs19 ⁱ —O9—Cs19 ⁱⁱ	101.9 (7)	O10—Cs19—O12 ^x	123.6 (10)
C6—O10—Cs19	125 (3)	O10 ^{viii} —Cs19—O11 ^{vii}	102.4 (6)
C6—O10—Cs19 ⁱⁱⁱ	135 (3)	O10 ^{viii} —Cs19—O12 ^x	62.5 (7)
Cs19—O10—Cs19 ⁱⁱⁱ	98.9 (9)	O11 ^{vii} —Cs19—O12 ^x	144.4 (7)

Symmetry codes: (i) $x, y, z-1$; (ii) $x+1/2, -y+1/2, z-1$; (iii) $x+1/2, -y+1/2, z$; (iv) $x+1, y, z-1$; (v) $x+1, y, z$; (vi) $x, y, z+1$; (vii) $x-1/2, -y+1/2, z+1$; (viii) $x-1/2, -y+1/2, z$; (ix) $x-1, y, z+1$; (x) $x-1, y, z$.

(ramm013_DFT)

Crystal data

CsH₂C₆H₅O₇

$M_r = 323.97$

Orthorhombic, $Pna2_1$

Hall symbol: P 2c -2n

$a = 8.7362 \text{ \AA}$

$b = 20.5351 \text{ \AA}$

$c = 5.1682 \text{ \AA}$

$V = 927.17 \text{ \AA}^3$

$Z = 4$

$D_x = 2.321 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.5418 \text{ \AA}$

$T = 300 \text{ K}$

Data collection

Density functional calculation

 $k = \rightarrow$ $h = \rightarrow$ $l = \rightarrow$ *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.14729	0.01007	0.32268	0.06500*
C2	0.28682	0.02722	0.16639	0.00900*
C3	0.35878	0.08945	0.28492	0.00900*
C4	0.52514	0.09664	0.18015	0.00900*
C5	0.60947	0.15212	0.30828	0.06500*
C6	0.26991	0.15158	0.20135	0.06500*
O7	0.15730	-0.04600	0.44742	0.06500*
O8	0.03401	0.04586	0.34061	0.06500*
O9	0.23818	0.15901	-0.03279	0.06500*
O10	0.24301	0.19347	0.37889	0.06500*
O11	0.62642	0.20442	0.15627	0.06500*
O12	0.65699	0.15090	0.53096	0.06500*
O13	0.36027	0.08089	0.55685	0.06500*
H14	0.36802	-0.01307	0.17323	0.01200*
H15	0.25479	0.03604	-0.03477	0.01200*
H16	0.31496	0.11870	0.64320	0.08500*
H17	0.58685	0.05170	0.22176	0.01200*
H18	0.52243	0.10375	-0.02890	0.01200*
Cs19	-0.05606	0.21080	0.74122	0.05030*
H20	0.06940	-0.05097	0.56860	0.05000*
H21	0.67528	0.24300	0.25240	0.05000*

Bond lengths (\AA)

C1—C2	1.504	C4—H17	1.090
C1—O7	1.322	C4—H18	1.091
C1—O8	1.354	C5—O11	1.339
C2—C3	1.550	C5—O12	1.224
C2—H14	1.090	C6—O9	1.251
C2—H15	1.092	C6—O10	1.279
C3—C4	1.558	O7—H20 ⁱ	0.996
C3—C6	1.555	O11—H21	1.028
C3—O13	1.416	O13—H16	0.979
C4—C5	1.510	H20—O7 ⁱⁱ	0.996

Symmetry codes: (i) $x-1/2, -y+1/2, z$; (ii) $x+1/2, -y+1/2, z$.*Hydrogen-bond geometry ($\text{\AA}, ^\circ$)*

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O11—H21 ⁱⁱ ···O10 ⁱⁱ	1.028	1.575	2.600	174.4
O7—H20 ⁱⁱⁱ ···O8 ⁱⁱⁱ	0.996	1.674	2.637	161.7

O13—H16···O9 ^{iv}	0.979	1.985	2.865	148.4
O13—H16···O10	0.979	2.149	2.691	113.3

Symmetry codes: (ii) $x+1/2, -y+1/2, z$; (iii) $-x, -y, z+1/2$; (iv) $x, y, z+1$.