Crystal structure of cesium 3,4,5-trihydroxybenzoate dihydrate, $Cs[C_7H_5O_5] \cdot 2H_2O$

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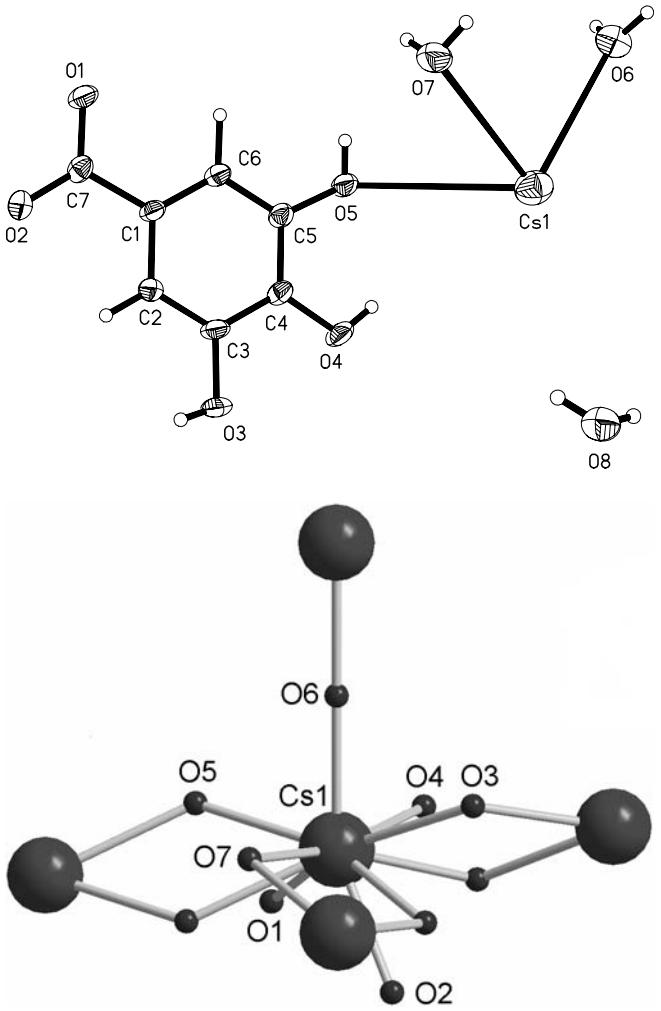

Abstract

$C_7H_9CsO_7$, monoclinic, *C*12/*c*1 (no. 15), a = 17.328(1) Å, b = 9.0329(7) Å, c = 13.190(1) Å, β = 103.957(1)°, V = 2003.6 Å^3, Z = 8, $R_{gt}(F)$ = 0.030, $wR_{ref}(F^2)$ = 0.118, T = 296 K.

Source of material

To the solution of 10 mmol gallic acid in 200 ml bidistilled water, solution of equimolar amount of cesium hydroxide was added dropwise. After reflux for 10 h with continuous stirring, the resulting solution was evaporated to about 20 ml in vacuum and filtered hot. The filtrate was then set aside for crystallization at room temperature. Brown crystals of the title compound suitable for X-ray structure determination were isolated a month later.

Experimental details

The H atoms associated with the aromatic C atoms were placed in idealized positions and included in the refinement in the riding model approximation with U_{iso} fixed at 1.5 U_{eq}(C) (d(C—H) = 0.93 Å). H atoms associated with O atoms were located in Fourier difference maps and refined with O—H bond lengths restrained to 0.82(1) Å and fixed U_{iso} values.

Discussion

Gallic acid (3,4,5-trihydroxybenzoic acid; GA) is a natural plant phenol with well-known antioxidant properties [1], which occurs in many medicinal plants and presents mostly in a conjugated form. It can scavenge peroxyl radicals [2] and has antifungal activity [3], as well. But investigations of its coordination chemistry are relatively rare. Only the structure of a nickel complex has been reported [4], in which GA was used as a reagent, but has no direct interactions with the central cation.

The asymmetric unit of the title crystal structure consists of one Cs atom, one gallic acid anion and crystal water molecules (figure, top). The occupancies of O6 and O8 are both 0.5 according to the symmetry constraints, thus the asymmetric unit contains two water molecules. The water molecule O8 has no interactions with the cesium cation. O3, O5, O6, O7 are on dibridging positions (figure, bottom), among which O3, O5 and O7 form Cs_2O_2 rings with their symmetry-related water molecules and two cesium cations bridged. Two carboxylate oxygen atoms (O1/O2) and a phenolic oxygen atom were involved in coordination leading to a coordination number of ten for the cation. The Cs—O distances cover a wide range from 3.051(4) Å to 3.748(4) Å, which seem to be normal comparing to those previously found in cesium 2,4-dinitrophenoxide monohydrate, 3.120(4) Å to 3.805(5) Å [5]. The Cs–O–Cs angles are 120.1(1)°, 129.58(8)° and 114.42(8)° for O3, O5 and O7, respectively. The crystal structure is assembled into a rather complicated three-dimensional network owing to the μ_2-O atoms and the bridging function of the ligand GA.

Table 1. Data collection and handling.

Crystal:	brown block, size 0.27 × 0.30 × 0.39 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	37.11 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX2 CCD, φ/ω
$2\theta_{max}$:	50.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4851, 1780
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{ob4s} > 2\,\sigma(I_{obs})$, 1666
$N(param)_{refined}$:	164
Programs:	SHELXS-97 [6], SHELXL-97 [7], DIAMOND [8]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(2)	8*f*		0.4053	0.6249	0.9664	0.027
H(6)	8*f*		0.2577	0.9413	0.8165	0.026
H(3)	8*f*		0.423(4)	0.629(2)	1.146(5)	0.080
H(4)	8*f*		0.283(1)	0.951(9)	1.157(5)	0.080
H(5)	8*f*		0.212(3)	1.110(8)	0.928(3)	0.080
H(6A)	8*f*		–0.044(4)	1.52(2)	1.01(1)	0.080
H(6B)	8*f*		0.021(8)	1.582(8)	1.00(1)	0.080
H(7A)	8*f*		0.066(3)	1.108(7)	0.847(4)	0.080
H(7B)	8*f*		–0.004(2)	1.101(8)	0.864(4)	0.080
H(8A)	8*f*		0.013(4)	0.533(4)	0.194(3)	0.080

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}
Cs(1)	8*f*	0.11084(2)	1.22823(4)	1.11682(2)	0.0433(3)	0.0502(3)	0.0405(3)	–0.0065(1)	0.0090(2)	0.0055(1)
C(1)	8*f*	0.3316(2)	0.7739(4)	0.8751(3)	0.025(2)	0.021(2)	0.013(2)	–0.000(1)	0.001(2)	–0.000(1)
C(2)	8*f*	0.3752(2)	0.7097(4)	0.9682(3)	0.027(2)	0.021(2)	0.018(2)	0.005(1)	0.004(2)	0.001(1)
C(3)	8*f*	0.3729(3)	0.7738(4)	1.0629(3)	0.030(2)	0.026(2)	0.014(2)	0.002(1)	0.001(2)	0.005(1)
C(4)	8*f*	0.3292(2)	0.9016(4)	1.0670(2)	0.027(2)	0.022(2)	0.016(2)	–0.002(1)	0.005(1)	–0.003(1)
C(5)	8*f*	0.2851(2)	0.9640(4)	0.9734(3)	0.030(2)	0.019(2)	0.019(2)	0.002(1)	0.007(1)	0.001(1)
C(6)	8*f*	0.2869(2)	0.8995(4)	0.8784(2)	0.027(2)	0.023(2)	0.015(2)	0.000(1)	0.003(1)	0.002(1)
C(7)	8*f*	0.3359(2)	0.7003(4)	0.7741(3)	0.030(2)	0.018(2)	0.020(2)	–0.003(2)	0.007(2)	–0.003(1)
O(1)	8*f*	0.2915(2)	0.7515(3)	0.6898(2)	0.042(2)	0.023(1)	0.015(1)	0.002(1)	0.001(1)	0.0006(9)
O(2)	8*f*	0.3819(2)	0.5945(3)	0.7760(2)	0.040(2)	0.030(2)	0.024(1)	0.010(1)	0.007(1)	–0.005(1)
O(3)	8*f*	0.4153(2)	0.7160(3)	1.1564(2)	0.051(2)	0.038(2)	0.015(1)	0.017(1)	–0.001(1)	0.004(1)
O(4)	8*f*	0.3302(2)	0.9620(3)	1.1621(2)	0.053(2)	0.029(1)	0.014(1)	0.009(1)	0.007(1)	–0.003(1)
O(5)	8*f*	0.2425(2)	1.0868(3)	0.9835(2)	0.049(2)	0.025(1)	0.018(1)	0.014(1)	0.006(1)	0.002(1)
O(6)	4*b*	0	½	0	0.042(2)	0.037(1)	0.037(1)	0.015(2)	0.014(1)	0.012(2)
O(7)	8*f*	0.0410(2)	1.0678(4)	0.8847(2)	0.040(2)	0.060(2)	0.034(2)	0.014(2)	0.011(1)	0.017(1)
O(8)	4*e*	0	0.552(3)	¼	0.041(2)	0.041(1)	0.037(2)	0.018(2)	0.016(1)	0.018(1)

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