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2,3,5,6-Tetrafluorohydroquinone dihydrate

Paweł Rusak and Maria Gdaniec*

Faculty of Chemistry, Adam Mickiewicz University, 60-780 Poznań, Poland

Correspondence e-mail: magdan@amu.edu.pl

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Key indicators

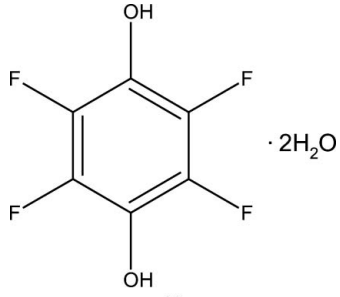
Single-crystal X-ray study
T = 130 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.080
Data-to-parameter ratio = 9.3

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

2,3,5,6-Tetrafluoro-1,4-dihydroxybenzene easily crystallises as a dihydrate, $C_6H_2F_4O_2\cdot 2H_2O$. The molecule exhibits approximate C_{2v} symmetry. The crystal packing is dominated by O—H···O hydrogen bonds.

Comment

The structure of 2,3,5,6-tetrafluoro-1,4-dihydroxybenzene dihydrate, (I), was determined in the course of our studies on molecular complexes formed by fluorinated phenols and polyphenols. The crystal structure of the anhydrous form of (I) has been determined previously (Thalladi *et al.*, 1999). The hydrated form precipitated from many solvents which were not carefully dried, including benzene and toluene. The crystals of (I) were not stable and when exposed to air slowly decomposed, losing water. X-ray powder diffraction studies confirmed that, on decomposition, (I) was transformed completely to the known anhydrous monoclinic form.



(I)

The endocyclic bond angles at C atoms with attached OH groups are 4° smaller than those at C atoms with F substituents, and the C—O bonds are 0.01Å longer than the C—F bonds (Table 1).

Hydroquinone molecules related by a glide plane are arranged into offset stacks running along the [100] direction. Their mean planes form a dihedral angle of 1.99 (3)°; however, there is not much overlap between the aromatic rings. Centroids of the benzene rings of adjacent molecules related by the symmetry operation $(\frac{1}{2}+x, y, \frac{1}{2}-z)$ are 4.11Å apart. Atoms F2 and F6 belonging to adjacent molecules of the stack are situated directly above and below the aromatic ring centroid at separations of 3.22 and 3.21Å.

The phenolic OH groups and water molecules are involved in O—H···O hydrogen bonds linking molecules into a three-dimensional network (Fig. 2*a*). The water molecules act as double donors and double acceptors, whereas the OH groups are single donors and single acceptors (Table 2 and Fig. 2). The longest hydrogen bonds are those between water molecules and phenol OH groups (Table 2).

Experimental

Anhydrous 2,3,5,6-tetrafluorohydroquinone was purchased from Aldrich. The single crystal used for this study was obtained by recrystallization from benzene containing water.

Crystal data

$C_6H_2F_4O_2\cdot 2H_2O$
$M_r = 218.11$
Orthorhombic, *Pbca*
$a = 7.6277\ (9)$ Å
$b = 11.9718\ (9)$ Å
$c = 17.5010\ (13)$ Å
$V = 1598.1\ (3)$ Å^3
$Z = 8$
$D_x = 1.813$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3879 reflections
$\theta = 2$–25°
$\mu = 0.21$ mm^{-1}
$T = 130\ (2)$ K
Needle, colourless
$0.6 \times 0.15 \times 0.05$ mm

Data collection

Kuma KM-4-CCD κ-geometry diffractometer
ω scans
Absorption correction: none
7711 measured reflections
1400 independent reflections
1077 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 25.0^\circ$
$h = -9 \rightarrow 5$
$k = -14 \rightarrow 13$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.080$
$S = 1.00$
1400 reflections
151 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.364 (2)	O4—C4	1.367 (2)
F2—C2	1.348 (2)	F5—C5	1.352 (2)
F3—C3	1.353 (2)	F6—C6	1.350 (2)
C6—C1—C2	117.36 (17)	C5—C4—C3	117.12 (17)
C3—C2—C1	121.32 (17)	C6—C5—C4	121.69 (17)
C2—C3—C4	121.40 (17)	C5—C6—C1	121.07 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1W	0.85 (2)	1.79 (2)	2.6229 (19)	165 (2)
O4—H4···O2W	0.80 (3)	1.96 (3)	2.721 (2)	159 (3)
O1W—H1W1···O2W^{i}	0.85 (3)	1.90 (3)	2.735 (2)	166 (2)
O1W—H2W1···O4^{ii}	0.90 (3)	2.01 (3)	2.846 (2)	154 (3)
O2W—H2W2···O1W^{iii}	0.93 (3)	1.86 (3)	2.778 (2)	170 (2)
O2W—H1W2···O1^{iv}	0.83 (3)	2.05 (3)	2.835 (2)	156 (3)

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

All H atoms were located in electron-density difference maps and their positional and displacement parameters were refined freely.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: Stereochemical Workstation Operation Manual (Siemens, 1989) and *MERCURY* (Version 1.4; Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97*.

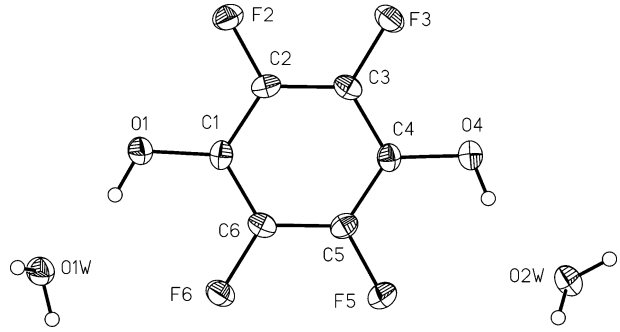


Figure 1
View of the asymmetric unit, shown with 50% displacement ellipsoids.

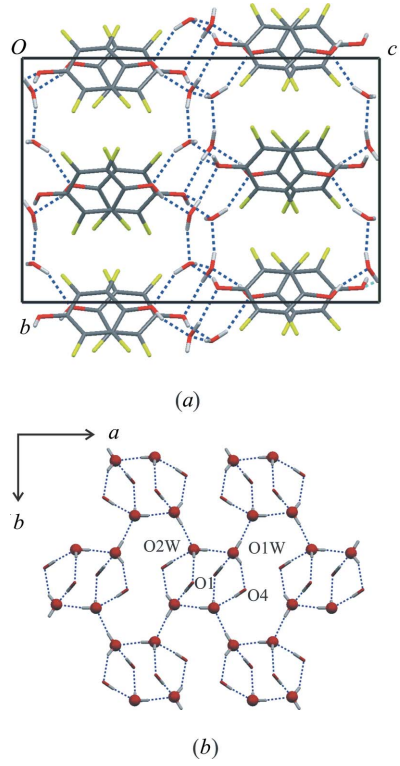


Figure 2
The crystal structure of (I), showing (*a*) the packing of the molecules viewed down the *a* axis, with hydrogen bonds shown as dashed lines, and (*b*) the hydrogen-bond network formed by hydroxyl groups and water molecules viewed down the *c* axis.

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