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Crystal structure and Hirshfeld surface analysis of hydronium 3,5-dicarboxybenzenesulfonate trihydrate

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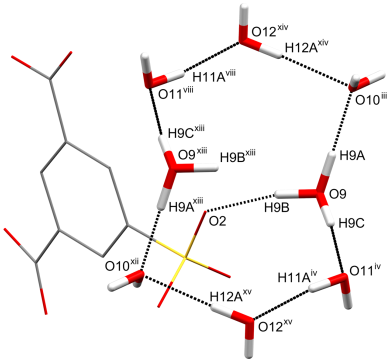
The title compound, hydronium 3,5-dicarboxybenzenesulfonate trihydrate, $H_3O^+ \cdot C_8H_5O_7S^- \cdot 3H_2O$, crystallizes in the triclinic space group $P\overline{1}$ with one molecule in the asymmetric unit. The structure is the pseudopolymorph with an additional three water molecules to known hydronium 3,5-dicarboxybenzenesulfonate. The 3,5-dicarboxybenzenesulfonate (SIP^-) moiety is surrounded by nine molecules, *i.e.* three SIP^- anions, five water molecules and one oxonium ion. The structure, containing a hydrogen-bonded water cluster with eight molecules, forms an $R_8^6(16)$ ring motif through intermolecular hydrogen bonding.



1. Chemical context

3,5-Dicarboxybenzenesulfonic acid (also known as 5-sulfoisophthalic acid, SIPA) has a simple structure with two carboxyl groups and one sulfonic acid group on the benzene ring, and has been used in a wide range of fields, especially in industry. As a sulfonated aromatic dicarboxylic acid, SIPA is a well-known monomer for introducing sulfonic acid into a resin to achieve various functions. For example, SIPA is used as a dyeability modifier for polyesters and polyamides (Ogata *et al.*, 2004; Vouyiouka *et al.*, 2007; Oster *et al.*, 2011; Xiong *et al.*, 2016), and as monomer of proton-exchange membranes for fuel cells (Bai *et al.*, 2009). In addition, SIPA is also reported as a raw material for polymer-type ionic liquids used in antistatic agents (Terada, 2009; Noda, 2010), thermal acid generators for the manufacture of semiconductor devices (Kaur *et al.*, 2017*a*; Kaur *et al.*, 2017*b*; Kaur *et al.*, 2018) and dyes for colour filters (Sakamoto *et al.*, 2014). It is also used in research as a molecular tecton of supramolecular assemblies and metal–organic frameworks due to its *exo*-trianionic structure. The crystal structure of SIPA was already reported as hydronium 3,5-dicarboxybenzenesulfonate ($H_3O^+ \cdot SIP^-$), without additional water molecules (Novozhilova *et al.*, 1989*a*). We have already successfully produced high-purity SIPA that reduced residual sulfuric acid and metal salts on an industrial scale (Inui, 2023). In an effort to produce high-purity SIPA, we discovered and report here the crystal structure of hydronium 3,5-dicarboxybenzenesulfonate trihydrate ($H_3O^+ \cdot SIP^- \cdot 3H_2O$).

2. Structural commentary

The title compound crystallizes in the triclinic space group $P\overline{1}$ with one molecule in the asymmetric unit (Fig. 1). The crystal structure is a pseudopolymorph with three additional water

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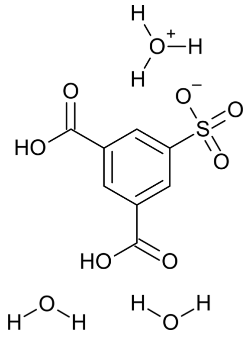

molecules with respect to the known $H_3O^+ \cdot SIP^-$ compound. The arrangement of the two carboxyl groups is mostly planar with respect to benzene ring. The torsion angles C14—C15—C19—O6 and C18—C17—C20—O8 are 3.3 (2) and 11.7 (2)°, respectively.

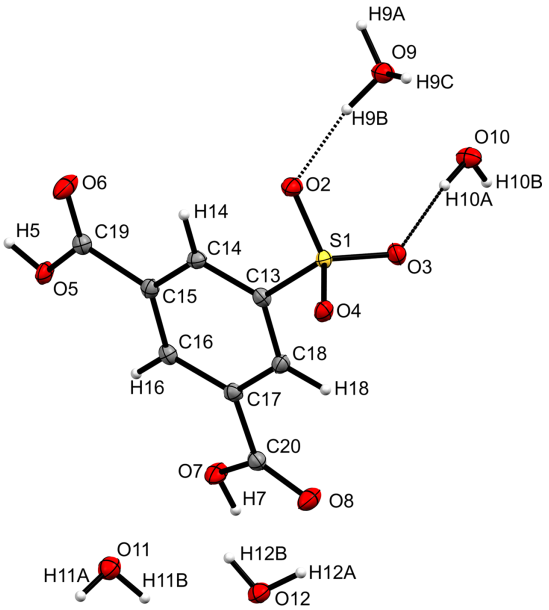

Figure 1
The molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary radius.

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O4^{i}	0.86 (3)	1.84 (3)	2.6850 (15)	168 (2)
O7—H7···O12^{ii}	0.87 (2)	1.79 (2)	2.6236 (16)	160 (3)
O9—H9A···O10^{iii}	0.94 (3)	1.64 (3)	2.5811 (16)	175 (2)
O9—H9B···O2	0.93 (2)	1.70 (3)	2.6124 (15)	168 (3)
O9—H9C···O11^{iv}	0.91 (2)	1.54 (2)	2.4469 (17)	173 (3)
O10—H10A···O3	0.83 (2)	1.89 (2)	2.7167 (16)	175 (2)
O10—H10B···O4^{v}	0.83 (3)	1.98 (3)	2.7778 (16)	161 (3)
O11—H11A···O12^{vi}	0.89 (3)	1.89 (3)	2.7708 (17)	173 (3)
O11—H11B···O8^{ii}	0.86 (3)	1.84 (3)	2.6834 (16)	168 (3)
O12—H12A···O10^{vii}	0.87 (3)	1.90 (3)	2.7604 (17)	174 (2)
O12—H12B···O6^{viii}	0.83 (3)	1.94 (3)	2.7373 (17)	161 (3)
C18—H18···O3^{vii}	0.95	2.57	3.5008 (19)	167

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

3. Supramolecular features

The 3,5-dicarboxybenzenesulfonate (SIP^-) moiety is surrounded by nine molecules, three SIP^- anions, five water molecules and one hydronium ion, involved in nine hydrogen bonds of eight different kinds (Table 1 and Fig. 2). In the crystal, SIP^- anions are linked by intermolecular C—H···O hydrogen bonds [C18—H18···O3^{vii}; symmetry code: (vii) $-x+1, -y+1, -z+1$], forming an inversion dimer with $R_2^2(10)$ ring motifs (Fig. 3). The sheet structure of SIP^- anions and water molecules is formed by intermolecular hydrogen-bond networks parallel to (101), as shown in Fig. 4. One one-dimensional chain structure is formed by O—H···O hydrogen bonds [O5—H5···O4^{i} and O5^{x}—H5^{x}···O4; symmetry codes: (i) $x, y+1, z$; (x) $x, y-1, z$] and another one-dimensional chain structure is formed by two kinds of O—H···O hydrogen bonds [O7—H7···O12^{ii}, O12^{ii}—H12B^{ii}···O6^{ix}, O7^{xi}—H7^{xi}···O12^{viii} and O12^{viii}—H12B^{viii}···O6; symmetry codes: (ii) $-x+1, -y+2, -z$; (viii) $-x, -y+2, -z+1$; (ix) $x+1, y, z-1$; (xi) $x-1, y, z+1$] due to an intermediate water

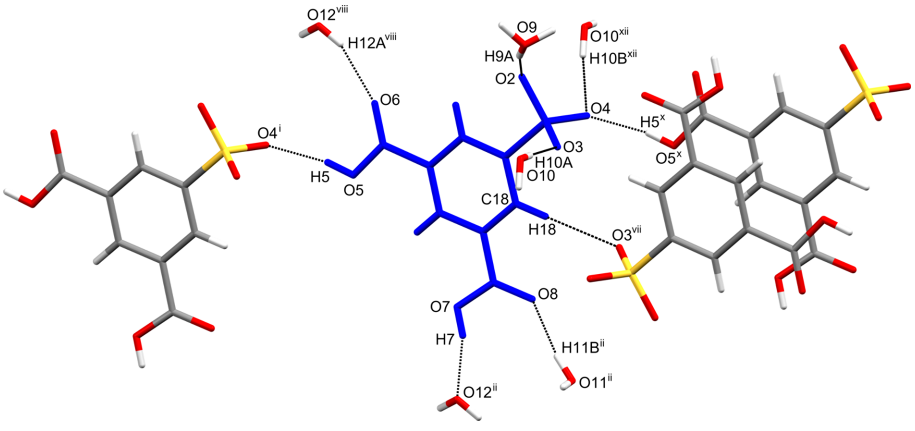

Figure 2
The structure of the SIP^- anion (blue) and the surrounding nine molecules. The intermolecular hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y+2, -z$; (vii) $-x+1, -y+1, -z+1$; (viii) $-x, -y+2, -z+1$; (x) $x, y-1, z$; (xii) $x-1, y, z$.]

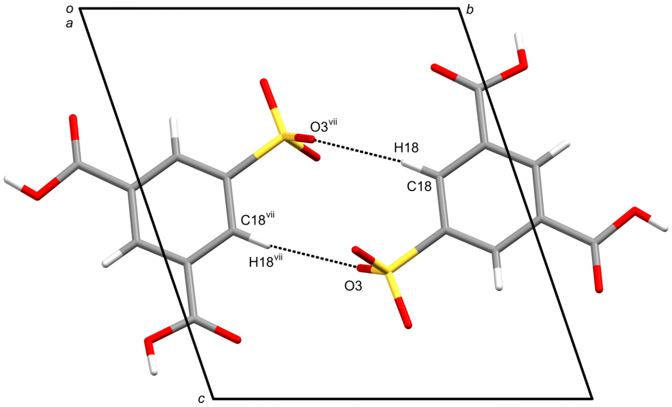

Figure 3
The centrosymmetric dimeric structure of the $H_3O^+\cdot SIP^-\cdot 3H_2O$. The intermolecular C18—H18···O3 hydrogen bonds are shown as dashed lines. Solvated water molecules have been omitted for clarity. [Symmetry code: (vii) $-x + 1, -y + 1, -z + 1$.]

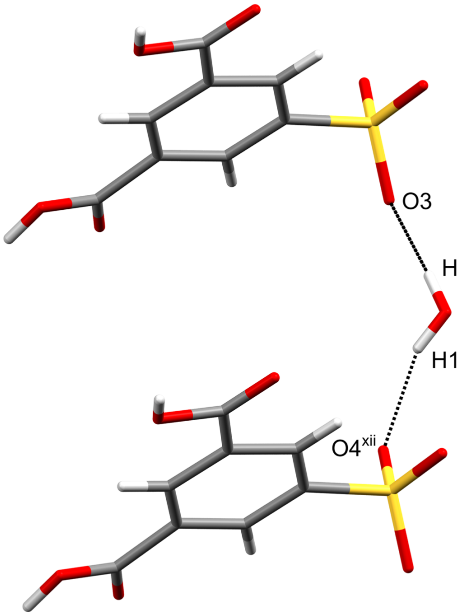

Figure 5
One-dimensional chain structure between SIP^- anions and water molecules along the *a* axis. The intermolecular O10—H10*A*···O3 and O10—H10*B*···O4$^{\rm xii}$ hydrogen bonds are shown as dashed lines. Water molecules not involved in the interactions have been omitted for clarity. [Symmetry code: (xii) $x - 1, y, z$.]

molecule. Fig. 5 shows the one-dimensional chain structure formed by two intermolecular O—H···O hydrogen bonds [O10—H10*A*···O3 and O10—H10*B*···O4$^{\rm xii}$; symmetry code: (xii) $x - 1, y, z$] between a water molecule and SIP^- anions along the *a* axis. In the crystal, the SIP^- anions and water molecules containing atoms O10 and O12 are linked by intermolecular O—H···O hydrogen bonds, forming a three-dimensional network structure. Furthermore, focusing on the water molecules and the hydronium ion, the structure containing a hydrogen-bonded water cluster with eight molecules forms an $R_8^6(16)$ ring motif through intermolecular O—H···O hydrogen bonding [O9—H9*A*···O10$^{\rm iii}$, O9—H9*C*···O11$^{\rm iv}$, O11$^{\rm iv}$—H11*A*$^{\rm iv}$···O12$^{\rm xv}$, O12$^{\rm xv}$—H12*A*$^{\rm xv}$···O10$^{\rm xii}$, O9$^{\rm xiii}$—H9*A*$^{\rm xiii}$···O10$^{\rm xii}$, O9$^{\rm xiii}$—H9*C*$^{\rm xiii}$···O11$^{\rm viii}$, O11$^{\rm viii}$—O11*A*$^{\rm viii}$···O12$^{\rm xiv}$ and O12$^{\rm xiv}$—O12*A*$^{\rm xiv}$···O10$^{\rm iii}$; symmetry codes: (iii) $-x + 1, -y + 1, -z + 2$; (iv) $x, y - 1, z + 1$; (viii) $-x, -y + 2, -z + 1$; (xii) $x - 1, y, z$;

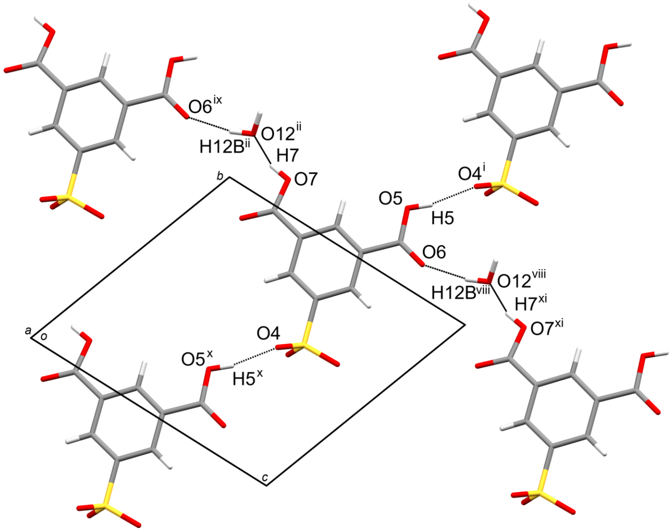

Figure 4
Two-dimensional sheet structure between SIP^- anions and water molecules parallel to (101). The intermolecular O5—H5···O4$^{\rm i}$, O5$^{\rm x}$—H5$^{\rm x}$···O4, O7—H7···O12$^{\rm ii}$, O12$^{\rm ii}$—H12B$^{\rm ii}$···O6$^{\rm ix}$, O7$^{\rm xi}$—H7$^{\rm xi}$···O12$^{\rm viii}$ and O12$^{\rm viii}$—H12B$^{\rm viii}$···O6 hydrogen bonds are shown as dashed lines. Water molecules not involved in the interactions have been omitted for clarity. [Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y + 2, -z$; (viii) $-x, -y + 2, -z + 1$; (ix) $x + 1, y, z - 1$; (x) $x, y - 1, z$; (xi) $x - 1, y, z + 1$.]

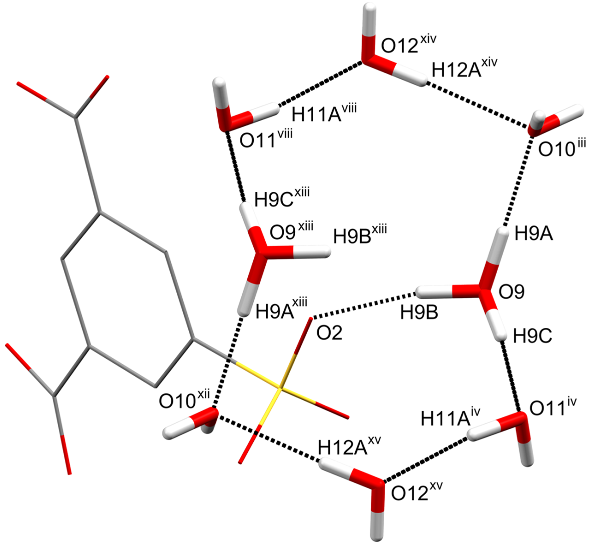

Figure 6
The $R_8^6(16)$ ring motif formed by intermolecular O—H···O hydrogen bonds involving eight water molecules. The intermolecular O—H···O hydrogen bonds are shown as dashed lines. The SIP^- anions in the asymmetric unit are shown in wireframe style and the H atoms have been omitted. The intermolecular O9—H9*B*···O2 hydrogen bond is also shown as a dashed line. [Symmetry codes: (iii) $-x + 1, -y + 1, -z + 2$; (iv) $x, y - 1, z + 1$; (viii) $-x, -y + 2, -z + 1$; (xii) $x - 1, y, z$; (xiii) $-x, -y + 1, -z + 2$; (xiv) $x, y, z + 1$; (xv) $-x, -y + 1, -z + 1$.]

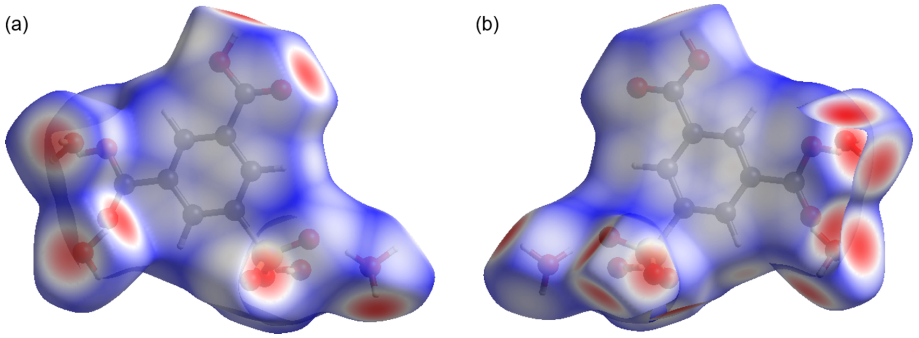

Figure 7
Hirshfeld surface mapped over $d_{\rm norm}$ for the title compound, showing (*a*) a front view and (*b*) a back view. Red spots indicate short O—H⋯O contacts.

(xiii) $-x, -y + 1, -z + 2$; (xiv) $x, y, z + 1$; (xv) $-x, -y + 1, -z + 1$] (Fig. 6). The water cluster is surrounded by eight SIP$^-$ anions and the water clusters do not interact with each other.

To visualize the intermolecular interactions in the crystal of the title compound, a Hirshfeld surface (HS) analysis (Spackman & Jayatilaka, 2009) was carried out using *CrystalExplorer* (Version 21.5; Spackman *et al.*, 2021). The HS mapped over $d_{\rm norm}$ shows several red spots, which mostly correspond to short O—H⋯O contacts between neighbouring molecules (Fig. 7). The percentage contributions of the intermolecular interactions to the total HS were quantified by two-dimensional fingerprint plots (McKinnon *et al.*, 2007). The fingerprint plots of $d_{\rm i}$ *versus* $d_{\rm e}$ shown in Fig. 8 reveal that the most significant contributions arise from O⋯H/H⋯O (55.9%) and H⋯H (26.4%) contacts. Smaller contributions are observed for C⋯C (6.4%), O⋯O (4.2%), O⋯C/C⋯O (4.0%) and C⋯H/H⋯C (3.1%) interactions.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 6.00, update August 2025; Groom *et al.*, 2016) using *ConQuest* (Bruno *et al.*, 2002) for compounds containing the the 1-sulfonato-3,5-dicarboxylatobenzene skeleton gave 151 hits with combinations of localized carboxylate and localized sulfonate. There are four combinations of notations, two kinds of carboxylates (localized: two C=O double bonds and two C—O single bond; delocalized: four delocalized carbon–oxygen bonds) and two kinds of sulfonates (localized: two S=O double bonds and one S—O single bond; delocalized: one S=O double bond and two delocalized S—O bonds). The survey for the combination of localized/delocalized carboxylates/sulfonate gave 151 hits of localized carboxylates and localized sulfonate (see above), 218 hits of delocalized carboxylates and localized sulfonate, two hits of localized carboxylates and delocalized sulfonate, and 31 hits of delocalized carboxylates and delocalized sulfonate. To refine the search for 'organic' structures gave 15 hits from only a combination of delocalized carboxylates and delocalized sulfonate. The carboxylates are protonated in 14 structures and only the crystal structure of the potassium salt of SIPA is partially deprotonated (CSD refcode KIBJUA; Novozhilova *et al.*, 1989*b*). All of 15 structures contain anionic sulfonate structures and the counter-cations are six hits of protonated pyridinium, one hit of methyl pyridinium, four hits of diprotonated secondary ammonium, two hits of protonated imidazolium, one hit of potassium and one hit of oxonium. The oxonium compound was reported previously, *i.e.* $H_3O^+{\cdot}SIP^-$ (JEJLOY; Novozhilova *et al.*, 1989*a*).

5. Synthesis and crystallization

$H_3O^+{\cdot}SIP^-$ (Konishi Chemical Ind. Co. Ltd, 163 g, 0.59 mol) was suspended in water (61 ml) and stirred at room temperature for 3 h. During stirring, the crystal was transformed from $H_3O^+{\cdot}SIP^-$ to $H_3O^+{\cdot}SIP^-{\cdot}3H_2O$ due to solvation. After that, the suspension was filtered off and the solids were collected as the seed crystals of $H_3O^+{\cdot}SIP^-{\cdot}3H_2O$ (73 g, 0.23 mol). To prepare the supersaturated solution of SIPA, a suspension of $H_3O^+{\cdot}SIP^-$ (0.25 *M*, 143 ml) was completely dissolved at 329 K and then cooled to room temperature. A small amount of the seed crystals of $H_3O^+{\cdot}SIP^-{\cdot}3H_2O$ was

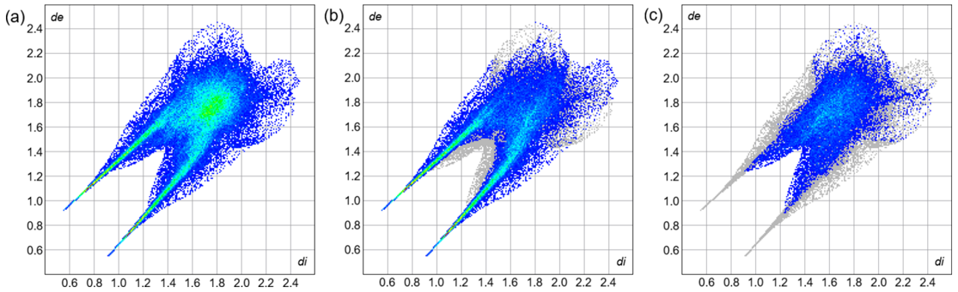

Figure 8
Two-dimensional fingerprint plots for the title compound. (*a*) Full fingerprint plot showing the overall distribution of $d_{\rm i}$ and $d_{\rm e}$. Fingerprint plots highlighting the (*b*) O⋯H/H⋯O contacts and (*c*) H⋯H contacts.

added to the supersaturated solution of SIPA, which was then sealed to prevent dehydration. After storing at room temperature for several days, colourless crystals suitable for X-ray analysis were obtained.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were placed in geometrically calculated positions (C—H = 0.95 Å) and refined as part of a riding model with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The O-bound H atoms H5, H7, H9*A*, H9*B*, H10*A*, H10*B*, H11*A*, H11*B*, H12*A* and H12*B* were located in a difference Fourier map and refined freely. Atom H9*C* was located in the difference Fourier map but was refined with a distance restraint of O—H = 0.84 ± 0.02 Å.

Table 2
Experimental details.

Crystal data	
Chemical formula	$H_3O^+ \cdot C_8H_5O_7S^- \cdot 3H_2O$
M_r	318.25
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, *b*, *c* (Å)	7.0692 (2), 9.4888 (2), 10.5484 (2)
α, β, γ (°)	70.482 (2), 77.103 (2), 85.021 (2)
V (Å^3)	650.03 (3)
Z	2
Radiation type	Cu *K*α
μ (mm^{-1})	2.78
Crystal size (mm)	0.29 × 0.08 × 0.03
Data collection	
Diffractometer	Rigaku XtaLAB Synergy Dualflex HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)
$T_{\rm min}$, $T_{\rm max}$	0.682, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7050, 2534, 2395
$R_{\rm int}$	0.026
$(\sin \theta/\lambda)_{\rm max}$ (Å^{-1})	0.632
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.078, 1.08
No. of reflections	2534
No. of parameters	225
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$, $\Delta\rho_{\rm min}$ (e Å^{-3})	0.31, −0.55

Computer programs: *CrysAlis PRO*; Rigaku OD, 2023), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

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supporting information

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Crystal structure and Hirshfeld surface analysis of hydronium 3,5-dicarboxybenzenesulfonate trihydrate

Kishin Inui, Yukiyasu Kashiwagi and Tomonori Mitsuru

Computing details

Hydronium 3,5-dicarboxybenzenesulfonate trihydrate

Crystal data

$H_3O^+\cdot C_8H_5O_7S^-\cdot 3H_2O$	$Z = 2$
$M_r = 318.25$	$F(000) = 332$
Triclinic, $P\overline{1}$	$D_x = 1.626$ Mg m^{-3}
$a = 7.0692$ (2) Å	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$b = 9.4888$ (2) Å	Cell parameters from 4896 reflections
$c = 10.5484$ (2) Å	$\theta = 4.5$–$76.6°$
$\alpha = 70.482$ (2)°	$\mu = 2.78$ mm^{-1}
$\beta = 77.103$ (2)°	$T = 100$ K
$\gamma = 85.021$ (2)°	Block, colourless
$V = 650.03$ (3) Å^3	0.29 × 0.08 × 0.03 mm

Data collection

Rigaku XtaLAB Synergy Dualflex HyPix diffractometer	$T_{\min} = 0.682$, $T_{\max} = 1.000$
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source	7050 measured reflections
Mirror monochromator	2534 independent reflections
Detector resolution: 10.0000 pixels mm^{-1}	2395 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.026$
Absorption correction: multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)	$\theta_{\max} = 76.9°$, $\theta_{\min} = 4.6°$
	$h = -5 \rightarrow 8$
	$k = -11 \rightarrow 11$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2 + 0.2593P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.078$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.08$	$\Delta\rho_{\max} = 0.31$ e Å^{-3}
2534 reflections	$\Delta\rho_{\min} = -0.55$ e Å^{-3}
225 parameters	
1 restraint	
Primary atom site location: dual	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.22880 (5)	0.57553 (3)	0.67370 (3)	0.01079 (11)
O2	0.13855 (15)	0.58112 (11)	0.81082 (10)	0.0159 (2)
O3	0.42016 (14)	0.50675 (11)	0.66474 (11)	0.0156 (2)
O4	0.09963 (14)	0.50963 (11)	0.61847 (10)	0.0137 (2)
O5	0.19654 (15)	1.28201 (11)	0.51791 (11)	0.0158 (2)
H5	0.164 (3)	1.345 (3)	0.562 (2)	0.034 (6)*
O6	0.05838 (17)	1.11710 (12)	0.71919 (11)	0.0237 (3)
O7	0.55512 (16)	1.12016 (11)	0.14810 (11)	0.0171 (2)
H7	0.637 (3)	1.132 (3)	0.070 (3)	0.039 (6)*
O8	0.54887 (16)	0.88310 (12)	0.15135 (11)	0.0198 (2)
C13	0.25703 (19)	0.76498 (15)	0.56670 (15)	0.0112 (3)
C14	0.18601 (19)	0.87815 (16)	0.62074 (14)	0.0121 (3)
H14	0.116744	0.854872	0.713265	0.015*
C15	0.21747 (19)	1.02717 (15)	0.53748 (15)	0.0121 (3)
C16	0.3168 (2)	1.06126 (15)	0.40202 (15)	0.0123 (3)
H16	0.338316	1.162595	0.346005	0.015*
C17	0.38506 (19)	0.94608 (16)	0.34825 (15)	0.0124 (3)
C18	0.3562 (2)	0.79706 (16)	0.43054 (15)	0.0126 (3)
H18	0.403445	0.718560	0.394306	0.015*
C19	0.1473 (2)	1.14599 (16)	0.60156 (15)	0.0130 (3)
C20	0.5028 (2)	0.97893 (16)	0.20580 (15)	0.0134 (3)
O9	0.30252 (15)	0.41647 (12)	1.01119 (12)	0.0174 (2)
H9A	0.285 (3)	0.448 (3)	1.089 (3)	0.041 (6)*
H9B	0.247 (3)	0.486 (3)	0.944 (3)	0.043 (6)*
H9C	0.251 (4)	0.326 (2)	1.028 (3)	0.073 (9)*
O12	0.16413 (16)	0.79716 (13)	0.07030 (12)	0.0180 (2)
H12A	0.206 (3)	0.710 (3)	0.115 (3)	0.041 (6)*
H12B	0.118 (4)	0.836 (3)	0.130 (3)	0.050 (7)*
O11	0.16454 (17)	1.17783 (12)	0.03829 (12)	0.0188 (2)
H11A	0.057 (4)	1.193 (3)	0.005 (2)	0.037 (6)*
H11B	0.247 (4)	1.147 (3)	−0.020 (3)	0.051 (7)*
O10	0.72986 (17)	0.48737 (12)	0.78303 (11)	0.0175 (2)
H10A	0.631 (3)	0.496 (2)	0.750 (2)	0.031 (6)*
H10B	0.828 (4)	0.510 (3)	0.721 (3)	0.054 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01341 (18)	0.00786 (17)	0.01068 (18)	0.00055 (12)	−0.00252 (13)	−0.00262 (13)

O2	0.0221 (5)	0.0127 (5)	0.0113 (5)	0.0005 (4)	−0.0021 (4)	−0.0030 (4)
O3	0.0157 (5)	0.0137 (5)	0.0172 (5)	0.0026 (4)	−0.0054 (4)	−0.0042 (4)
O4	0.0159 (5)	0.0103 (5)	0.0163 (5)	−0.0004 (4)	−0.0040 (4)	−0.0057 (4)
O5	0.0222 (5)	0.0085 (5)	0.0159 (5)	0.0017 (4)	−0.0015 (4)	−0.0050 (4)
O6	0.0359 (6)	0.0151 (5)	0.0165 (6)	−0.0013 (5)	0.0059 (5)	−0.0073 (4)
O7	0.0215 (5)	0.0134 (5)	0.0123 (5)	−0.0020 (4)	0.0032 (4)	−0.0028 (4)
O8	0.0259 (6)	0.0165 (5)	0.0156 (5)	−0.0011 (4)	0.0023 (4)	−0.0075 (4)
C13	0.0115 (6)	0.0098 (7)	0.0125 (7)	−0.0004 (5)	−0.0044 (5)	−0.0025 (5)
C14	0.0122 (6)	0.0135 (7)	0.0104 (7)	−0.0004 (5)	−0.0019 (5)	−0.0039 (5)
C15	0.0109 (6)	0.0123 (7)	0.0142 (7)	0.0005 (5)	−0.0036 (5)	−0.0054 (6)
C16	0.0118 (6)	0.0106 (7)	0.0139 (7)	−0.0001 (5)	−0.0037 (5)	−0.0026 (5)
C17	0.0116 (6)	0.0140 (7)	0.0120 (7)	0.0000 (5)	−0.0036 (5)	−0.0043 (6)
C18	0.0130 (6)	0.0125 (7)	0.0139 (7)	0.0016 (5)	−0.0034 (5)	−0.0064 (5)
C19	0.0128 (6)	0.0120 (7)	0.0140 (7)	0.0004 (5)	−0.0032 (5)	−0.0038 (6)
C20	0.0132 (6)	0.0132 (7)	0.0137 (7)	0.0012 (5)	−0.0043 (5)	−0.0036 (6)
O9	0.0215 (5)	0.0167 (5)	0.0139 (5)	−0.0001 (4)	−0.0049 (4)	−0.0042 (4)
O12	0.0206 (5)	0.0188 (6)	0.0137 (5)	0.0015 (4)	−0.0002 (4)	−0.0066 (5)
O11	0.0186 (6)	0.0196 (6)	0.0176 (6)	0.0004 (4)	−0.0012 (5)	−0.0072 (5)
O10	0.0142 (5)	0.0235 (6)	0.0140 (6)	−0.0023 (4)	−0.0025 (5)	−0.0051 (4)

Geometric parameters (Å, º)

S1—O2	1.4614 (10)	C13—C14	1.3849 (19)
S1—O3	1.4477 (10)	C13—C18	1.394 (2)
S1—O4	1.4643 (10)	C14—C15	1.399 (2)
S1—C13	1.7752 (14)	C15—C16	1.386 (2)
O5—C19	1.3212 (18)	C15—C19	1.4956 (19)
O6—C19	1.2100 (18)	C16—C17	1.396 (2)
O7—C20	1.3205 (18)	C17—C18	1.395 (2)
O8—C20	1.2153 (18)	C17—C20	1.490 (2)
O2—S1—O4	111.58 (6)	C16—C15—C19	121.91 (13)
O2—S1—C13	105.41 (6)	C15—C16—C17	119.75 (13)
O3—S1—O2	113.95 (6)	C16—C17—C20	120.93 (13)
O3—S1—O4	112.28 (6)	C18—C17—C16	120.38 (13)
O3—S1—C13	107.03 (6)	C18—C17—C20	118.52 (12)
O4—S1—C13	105.90 (6)	C13—C18—C17	119.04 (13)
C14—C13—S1	119.65 (11)	O5—C19—C15	113.05 (12)
C14—C13—C18	121.17 (13)	O6—C19—O5	124.70 (13)
C18—C13—S1	119.14 (10)	O6—C19—C15	122.23 (13)
C13—C14—C15	119.23 (13)	O7—C20—C17	113.15 (12)
C14—C15—C19	117.63 (13)	O8—C20—O7	124.28 (13)
C16—C15—C14	120.42 (13)	O8—C20—C17	122.54 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O4$^{\rm i}$	0.86 (3)	1.84 (3)	2.6850 (15)	168 (2)

O7—H7···O12$^{\text{ii}}$	0.87 (2)	1.79 (2)	2.6236 (16)	160 (3)
O9—H9*A*···O10$^{\text{iii}}$	0.94 (3)	1.64 (3)	2.5811 (16)	175 (2)
O9—H9*B*···O2	0.93 (2)	1.70 (3)	2.6124 (15)	168 (3)
O9—H9*C*···O11$^{\text{iv}}$	0.91 (2)	1.54 (2)	2.4469 (17)	173 (3)
O10—H10*A*···O3	0.83 (2)	1.89 (2)	2.7167 (16)	175 (2)
O10—H10*B*···O4$^{\text{v}}$	0.83 (3)	1.98 (3)	2.7778 (16)	161 (3)
O11—H11*A*···O12$^{\text{vi}}$	0.89 (3)	1.89 (3)	2.7708 (17)	173 (3)
O11—H11*B*···O8$^{\text{ii}}$	0.86 (3)	1.84 (3)	2.6834 (16)	168 (3)
O12—H12*A*···O10$^{\text{vii}}$	0.87 (3)	1.90 (3)	2.7604 (17)	174 (2)
O12—H12*B*···O6$^{\text{viii}}$	0.83 (3)	1.94 (3)	2.7373 (17)	161 (3)
C18—H18···O3$^{\text{vii}}$	0.95	2.57	3.5008 (19)	167

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1, −*y*+2, −*z*; (iii) −*x*+1, −*y*+1, −*z*+2; (iv) *x*, *y*−1, *z*+1; (v) *x*+1, *y*, *z*; (vi) −*x*, −*y*+2, −*z*; (vii) −*x*+1, −*y*+1, −*z*+1; (viii) −*x*, −*y*+2, −*z*+1.