Crystal structure of 2,5-dihydroxyterephthalic acid from powder diffraction data

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The crystal structure of anhydrous 2,5-dihydroxyterephthalic acid, C₈H₆O₆, was solved and refined using laboratory X-ray powder diffraction data, and optimized using density functional techniques. The published structure of 2,5-dihydroxyterephthalic acid dihydrate was also optimized. The carboxylic acid groups form strong hydrogen bonds, which form centrosymmetric rings with graph set $R_2^2(8)$. These hydrogen bonds link the molecules into chains along [011]. There is an intramolecular O—H⋯O hydrogen bond between the hydroxyl group and the carbonyl group of the carboxylic acid. The hydrogen bonding in the dihydrate is very different. Although the intramolecular hydroxy/carboxylic acid hydrogen bond is present, the water molecule acts as an acceptor to the carboxylic acid and a donor to two other oxygen atoms. The carboxylic acid groups do not interact with each other directly.

1. Chemical context

2,5-Dihydroxyterephthalate (C₈H₄O₆$_2^-$; dhtp) is of current interest as a linker in metal–organic frameworks (MOFs). It can add extra functionality to alter adsorption and catalytic properties. In an attempt to replicate the ionothermal preparation of the Co-dhtp MOF Co₂(dobdc)-ST (Azbell et al., 2022), an unexpected product was obtained, namely anhydrous 2,5-dihydroxyterephthalic acid, C₈H₆O₆, (I).

The crystal structures of three Co-dhtp MOFs have been reported: Cambridge Structural Database refcodes FEGEBEB (Gen, 2017), VOFJIM (Rosnes et al., 2019) and VOFJIM01 (Ayoub et al., 2019). The calculated powder patterns of these three compounds, which have been given the name CPO-27-Co, indicate that they have the same structure (Fig. 1).

2. Structural commentary

Compound (I) crystallizes in the triclinic space group $P\overline{1}$ with half a molecule in the asymmetric unit. The root-mean-square
Cartesian displacements of the non-H atoms in the Rietveld-refined and CRYSTAL17-optimized structures is 0.053 Å (Fig. 2). The good agreement provides strong evidence that the structure is correct (van de Streek & Neumann, 2014). The CRYSTAL17 and VASP-optimized structures are essentially identical (r.m.s. displacement = 0.031 Å). This discussion concentrates on the CRYSTAL17-optimized structure. The full molecule (with atom numbering) is illustrated in Fig. 3 and a view of the packing down the a-axis direction is shown in Fig. 4.

All of the bond distances, angles, and torsion angles fall within the normal ranges indicated by a Mercury Mogul geometry check (Macrae et al., 2020). The plane of the phenyl ring lies approximately on the (98 9) Miller plane. The peak profiles are dominated by anisotropic microstrain broadening: the average microstrain is 8362 ppm.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) morphology suggests that we might expect platy (with {001} as the major faces) morphology for this crystal. A 4th order spherical harmonics preferred orientation model was included in the refinement. The refined texture index was 1.059 (2), indicating that preferred orientation was small for this capillary specimen. In flat plate specimens examined in Bragg–Brentano geometry using Cu radiation, the preferred orientation tended to be higher.

### 3. Supramolecular features

In the extended structure of (I), the carboxylic acid groups form strong O3—H4···O5 hydrogen bonds, which form centrosymmetric loops with graph set $R_2^2(8)$ (Etter, 1990; Bernstein et al., 1995; Shields et al., 2000). These hydrogen bonds link the molecules into chains propagating along [011] (Table 1; Fig. 5). There is an intramolecular O1—H2···O5 hydrogen bond between the hydroxyl group and the carbonyl group of the carboxyl acid. A C—H···O hydrogen bond also contributes to the lattice energy. The Mercury aromatics

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O3—H4···O5 | 1.00 | 1.69  | 2.689 | 174     |
| O1—H2···O5 | 0.99 | 1.68  | 2.567 | 147     |

Symmetry code: (i) $-x+1, -y+2, -z+2$.

Table 1

*Hydrogen-bond geometry (Å, °) for (I).*
analysed. The hydrogen bonding in the dihydrate DUSJUX (Cheng et al., 2010) is very different (Table 2; Fig. 6). Although the intramolecular hydroxy–carboxylic acid O—H···O hydrogen bond is present, the water molecule acts as an acceptor to the carboxylic acid and a donor to two other oxygen atoms. The carboxylic acid groups do not interact with each other directly.

The CRYSTAL17 (Dovesi et al., 2018) calculations suggest that DUSJUX is 28.5 kcal mol⁻¹ lower in energy than the sum of anhydrous 2,5-dihydroxyterephthalic acid and two water molecules. The corresponding VASP (Kresse & Furthmüller, 1996) calculations indicate that DUSJUX is 114.0 kcal mol⁻¹ more stable. As chemists, we would like to attribute the ‘extra’ energy to the formation of additional hydrogen bonds.

Rammohan & Kaduk (2018) developed (for citrates using earlier versions of CRYSTAL) a correlation between the energy of an O—H···O hydrogen bond and the Mulliken overlap population between the H and the O acceptor: $E = 54.7(\text{overlap})^{1/2}$. Using this correlation to estimate the energies of the individual hydrogen bonds, we calculate that DUSJUX is 59.6 kcal mol⁻¹ lower in energy than the sum of the anhydrous molecule and two water molecules – a value between the two DFT calculations. While the disagreements indicate that the absolute energy calculated for a hydrogen bond may be more uncertain than we would like, the Mulliken overlap population is certainly a guide to whether a hydrogen bond is stronger or weaker than another, and to whether a (geometrically possible) hydrogen bond is real or not.

### 4. Database survey

A connectivity search in the Cambridge Structural Database [CSD version 5.43 June 2022 (Groom et al., 2016); ConQuest 2022.2.0 (Bruno et al., 2002)] of a 2,5-dihydroxyterephthalate fragment with the elements C, H, and O only yielded the structure of the dihydrate (Cheng et al., 2010; DUSJUX), as well as two esters. The dihydrate was also obtained accidentally during the synthesis of metal–organic coordination polymers. Removing the chemistry limitation yielded 249 entries, many of which are metal–organic frameworks. A search of the powder pattern against the Powder Diffraction File (Gates-Rector & Blanton, 2019) yielded no hits. Not even the usual accidental matches were obtained; this pattern evidently occupies a very different portion of ‘diffraction space’.

### 5. Synthesis and crystallization

Cobalt(II) chloride hexahydrate (1.78 g, 7.50 mmol) and 2,5-dihydroxyterephthalic acid (1.00 g, 5.05 mmol) were crushed together with mortar and pestle and added to a 10 ml round-bottom flask. The flask was connected to a Schlenk line and placed in a glass bowl of sand on top of a hot plate. The hot plate was heated to 443 K for approximately 18 h and the round-bottom flask was under vacuum. After being removed from the heat and allowed to cool, the remaining solid was transferred to a Pyrex container with acetonitrile (50 ml) and placed in a vacuum oven at 343 K for 24 h. After removal from the oven, the solution was decanted and replaced with acetonitrile (50 ml). This wash procedure was done a total of three times. The remaining solid was then added to 100 ml of methanol at 343 K for 24 h and decanted, this wash was done two times. The remaining solid was then added to a vacuum oven at 423 K for 24 h. The remaining solid was then added to a scintillation vial wrapped with Parafilm for storage.

### 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. A portion of the sample was blended with 11.51% < 1 micron diamond powder (Alfa

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### Table 2

| $D—H···A$ | $D—H$ | $H···A$ | $D···A$ | $D—H···A$ |
|-----------|--------|--------|--------|-----------|
| O2—H2···O4 | 1.07   | 1.43   | 2.500  | 178       |
| O1—H1···O3¹ | 1.01   | 1.64   | 2.562  | 149       |
| O4—H4···O3² | 0.99   | 1.78   | 2.736  | 161       |
| O4—H5···O1⁰⁰ | 0.99   | 1.82   | 2.794  | 169       |

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

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Figure 6

The hydrogen bonds in the structure of 2,5-dihydroxyterephthalic acid dihydrate DUSJUX. Image generated using Mercury (Macrae et al., 2020).

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Figure 5

The hydrogen bonds in the structure of anhydrous 2,5-dihydroxyterephthalic acid. Image generated using Mercury (Macrae et al., 2020).
Research communications

Table 3
Experimental details.

|                  | (I)                               | DUSJUX (DFT)                      |
|------------------|-----------------------------------|-----------------------------------|
| Crystal data     |                                   |                                   |
| Chemical formula | C₈H₆O₆                            | C₈H₆O₆·2(H₂O)                     |
| M₂               | 198.08                            | --                                |
| Crystal system, space group | Triclinic, Pt            | Monoclinic, P2₁/c                  |
| Temperature (K)  | 302                               | --                                |
| a, b, c (Å)      | 4.2947 (5), 5.6089 (5), 8.2331 (19) | 5.18830, 17.54500, 5.49900         |
| α, β, γ (°)      | 93.612 (4), 102.219 (4), 96.7621 (14) | 90, 103.03, 90                    |
| V (Å³)           | 191.69 (1)                        | 487.68                            |
| Z                | 1                                 | 2                                 |
| Radiation type   | Mo Kα, λ = 0.70932, 0.71361 Å     | --                                |
| Specimen shape, size (mm) | Cylinder, 12 × 0.7 | --                                |

Data collection

|                  |                                   |                                   |
| Diffactometer    | PANalytical Empyrean              |                                   |
| Specimen mounting| Glass capillary                  |                                   |
| Data collection mode | Transmission                   |                                   |
| Data collection method | Step                           |                                   |
| θ values (°)     | 2θmin = 1.002 2θmax = 49.991 2θstep = 0.008 |                                   |

Refinement

| R factors and goodness of fit | Rf = 0.034, Rwp = 0.042, Rexp = 0.019, χ² = 5.148 |
| No. of parameters            | 53                                               |
| No. of restraints            | 18                                               |
| (Δ/σ)max                    | 2.635                                            |

The same symmetry and lattice parameters were used for the DFT calculations as for the powder diffraction study for (I). Computer program: GSAS-II (Toby & Von Dreele, 2013).

Aesar) internal standard in a mortar and pestle until the color was uniform. The X-ray powder diffraction pattern was measured from a 0.7 mm diameter static capillary specimen on a PANalytical Empyrean diffractometer using Mo Kα radiation. The pattern was measured from 1.0–50.0° 2θ in 0.0083560° steps, counting for 4 sec/step.

After correcting the peak positions using the known diamond peak positions, the pattern was indexed using JADE Pro (MDI, 2022) on a primitive triclinic cell with a = 4.26420, b = 5.58601, c = 8.17902 Å, α = 93.53, β = 12.13, γ = 96.78° and V = 188 Å³. Since the volume corresponds to one molecule of 2,5-dihydroxyterephthalic acid, the space group was assumed to be Pt, with half a molecule in the asymmetric unit. A reduced cell search of the CSD yielded no hits. Preliminary indexing attempts using the default peak list from a pattern collected using Cu radiation were unsuccessful (monoclinic cells with no reasonable structures), until closer examination of the pattern revealed that the peak at 21.6° (9.7° Mo) was actually a doublet, and that there was an additional peak at 22.0° (9.9° Mo). Including these two additional peaks yielded the triclinic cell.

The 2,5-dihydroxyterephthalic acid molecule was extracted from the DUSJUX structure using Materials Studio (Dassault Systèmes, 2021), and saved as a .mol2 file. The crystal structure was solved using Monte Carlo simulated annealing techniques as implemented in EXP02014 (Altomare et al., 2013), using a whole molecule as the fragment. Since the molecule occupies a center of symmetry, the two halves overlapped partially. The overlapping atoms were averaged manually using Materials Studio to obtain the asymmetric unit.

Rietveld refinement was carried out using GSAS-II (Toby & Von Dreele, 2013). All non-H bond distances and angles were subjected to restraints, based on a Mercury Mogul geometry check (Sykes et al., 2011; Bruno et al., 2004). A planar restraint was applied to the benzene ring. The Mogul average and standard deviation for each quantity were used as the restraint parameters. The restraints contributed 1.9% to the final χ². The hydrogen atoms were included in calculated positions, which were recalculated during the refinement using Materials Studio (Dassault Systèmes, 2021). The Uiso of the heavy atoms were grouped by chemical similarity. The Uiso for the H atoms were fixed at 1.3× the Uiso of the heavy atoms to which they are attached. The peak profiles were described using the generalized microstrain model. The background was modeled using a four-term shifted Chebyshev polynomial, along with a peak at 12.05° to model the scattering from the glass capillary and any amorphous component. The final refinements yielded the residuals reported in Table 1. The largest errors in the...
difference plot (Fig. 7) are small, and are in the shapes of the peaks.

The crystal structure (as well as that of DUSJUX and an isolated water molecule) was optimized using VASP (Kresse & Furthmüller, 1996) (fixed experimental unit cells) through the MedeA graphical interface (Materials Design, 2016). The calculations were carried out on 16 2.4 GHz processors (each with 4 Gb RAM) of a 64-processor HP ProLiant DL580 Generation 7 Linux cluster at North Central College. The calculations used the GGA-PBE functional, a plane wave cutoff energy of 400.0 eV, and a \( k \)-point spacing of 0.5 Å leading to a 4 \( \times \) 3 \( \times \) 2 mesh. The structures were also optimized (fixed experimental cells) and population analyses were carried out using CRYSTAL17 (Dovesi et al., 2018). The basis sets for the H, C, N, and O atoms in the calculations were those of Gatti et al. (1994). The calculations were run on a 3.5 GHz PC using 8 \( k \)-points and the B3LYP functional.

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Crystal structure of 2,5-dihydroxyterephthalic acid from powder diffraction data

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Computing details

2,5-Dihydroxybenzene-1,4-dicarboxylic acid (I)

Crystal data

C₈H₆O₆

Mr = 198.08

Triclinic, P1

Hall symbol: -P 1

a = 4.2947 (5) Å

b = 5.6089 (5) Å

c = 8.2331 (19) Å

α = 93.612 (4)°

β = 102.219 (4)°

γ = 96.7621 (14)°

V = 191.69 (1) Å³

Z = 1

Dx = 1.716 Mg m⁻³

T = 302 K

cylinder, 12 × 0.7 mm

Data collection

PANalytical Empyrean
diffractometer

Data collection mode: transmission

Scan method: step

Specimen mounting: glass capillary

Refinement

18 restraints

Preferred orientation correction: Simple spherical harmonic correction Order = 4

Coefficients: 0:0:C(2,-2) = 0.246 (11);

0:0:C(2,-1) = -0.018 (11); 0:0:C(2,0) = -0.313 (16); 0:0:C(2,1) = 0.217 (13); 0:0:C(2,2) = -0.192 (9);

0:0:C(4,-4) = -0.146 (17); 0:0:C(4,-3) = 0.073 (19); 0:0:C(4,-2) = -0.052 (16);

0:0:C(4,-1) = 0.083 (18); 0:0:C(4,0) = -0.058 (17); 0:0:C(4,1) = -0.006 (18); 0:0:C(4,2) = -0.196 (23);

0:0:C(4,3) = 0.071 (16); 0:0:C(4,4) = -1.08 (15)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|     | x     | y     | z     | Ueq  |
|-----|------|------|------|------|
| C10 | 0.492(2) | 0.6406(16) | 0.6392(11) | 0.0323(10)* |
| C6  | 0.6818(18) | 0.4506(18) | 0.6571(9) | 0.0323(10)* |
| C7  | 0.6946(18) | 0.3106(14) | 0.5107(12) | 0.0323(10)* |
| C9  | 0.477(2) | 0.7892(16) | 0.7918(9) | 0.0553(15)* |
| O1  | 0.8599(12) | 0.4082(9) | 0.8120(6) | 0.0323(10)* |
Geometric parameters (Å, º)

| Bond                  | Distance (Å) | Angle (º) |
|-----------------------|--------------|-----------|
| C10—C6                | 1.411 (5)    |           |
| C10—C7i               | 1.384 (5)    |           |
| C10—C9                | 1.482 (6)    |           |
| C6—C10                | 1.411 (5)    |           |
| C6—C7                 | 1.412 (6)    |           |
| C6—C1—C1i             | 1.5529       |           |
| C6—C1—C1 iii          | 1.5529       |           |
| C6—C1—C1 ii           | 1.5529       |           |
| C6—C1—C1 iv           | 1.5529       |           |
| C6—C10—C7i            | 124.3 (7)    |           |
| C6—C10—C9             | 118.1 (9)    |           |
| C10—C7—C6             | 117.4 (6)    |           |
| C10—C6—O1             | 1.391 (5)    |           |
| C10—C9—O3             | 1.365 (5)    |           |
| C9—O3—H4              | 1.004 (6)    |           |
| C9—O5—C10             | 1.277 (5)    |           |
| C6—C10—C7i            | 124.3 (7)    |           |
| C6—C10—C9             | 118.1 (9)    |           |
| C7—C10—C6             | 117.4 (6)    |           |
| C7—C1—C1i             | 1.5529       |           |
| C7—C1—C1 iii          | 1.5529       |           |
| C7—C1—C1 ii           | 1.5529       |           |
| C7—C1—C1 iv           | 1.5529       |           |

Symmetry code: (i) −x+1, −y+1, −z+1.

Crystal data

| Parameter          | Value          |
|--------------------|----------------|
| C                  | Mr = 12.01     |
| Cubic, Fd3m        | V = 46.12 (1) Å³ |
| Hall symbol: F 4vw 2vw | Dx = 3.459 Mg m⁻³ |
| a = 3.58625 (11) Å | T = 302 K      |

Refinement

Preferred orientation correction: March-Dollase correction coef. = 1.000 axis = [0, 0, 1]

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

| Atom | x    | y    | z    | Uiso* |
|------|------|------|------|-------|
| C1   | 0.12500 | 0.12500 | 0.12500 | 0.0159* |

Geometric parameters (Å, º)

| Bond                  | Distance (Å) | Angle (º) |
|-----------------------|--------------|-----------|
| C1—C1i                | 1.5529       |           |
| C1—C1 iii             | 1.5529       |           |
| C1—C1 ii              | 1.5529       |           |
| C1—C1 iv              | 1.5529       |           |
C1—C1—C1\textsuperscript{i} 109.471 C1—C1—C1\textsuperscript{iv} 109.471
C1—C1—C1\textsuperscript{ii} 109.471 C1\textsuperscript{ii}—C1—C1\textsuperscript{iv} 109.471
C1\textsuperscript{i}—C1—C1\textsuperscript{iii} 109.471 C1\textsuperscript{iii}—C1—C1\textsuperscript{iv} 109.471

Symmetry codes: (i) x+1/4, y+1/4, −z; (ii) −z, x+1/4, y+1/4; (iii) y+1/4, −z, x+1/4; (iv) −x, −y, −z.

(I_DFT)

Crystal data

\( \text{C}_8\text{H}_6\text{O}_6 \)

\( M_r = 198.08 \)

Triclinic, \( P\bar{1} \)

\( a = 4.2647 \text{ Å} \) \( c = 8.1976 \text{ Å} \)

\( \alpha = 93.6590^\circ \)

\( \beta = 102.1730^\circ \)

\( \gamma = 96.7840^\circ \)

\( Z = 1 \)

Data collection

\( h = \rightarrow \)

\( l = \rightarrow \)

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

|      | \( x \)   | \( y \)   | \( z \)   | \( U_{	ext{iso}} \) or \( U_{	ext{eq}} \) |
|------|-----------|-----------|-----------|------------------|
| C10  | 0.50725   | 0.65165   | 0.64068   | 0.06414*         |
| C6   | 0.68695   | 0.46350   | 0.65571   | 0.06414*         |
| C7   | 0.69097   | 0.31393   | 0.51546   | 0.06414*         |
| C9   | 0.48403   | 0.80691   | 0.79069   | 0.01062*         |
| O1   | 0.87656   | 0.42423   | 0.80458   | 0.01062*         |
| O3   | 0.28942   | 0.97462   | 0.76830   | 0.01062*         |
| O5   | 0.65017   | 0.78063   | 0.93109   | 0.01062*         |
| H8   | 0.84185   | 0.17135   | 0.52928   | 0.08339*         |
| H2   | 0.83782   | 0.54288   | 0.88999   | 0.01381*         |
| H4   | 0.30280   | 1.07255   | 0.87663   | 0.01381*         |

Bond lengths (Å)

|      | C10—C6   | 1.370     | C7—H8     | 1.079     |
|------|-----------|-----------|-----------|-----------|
| C10—C7\textsuperscript{i} | 1.415     | C9—O3     | 1.320     |
| C10—C9  | 1.487     | C9—O5     | 1.245     |
| C6—C7   | 1.382     | O1—H2     | 0.986     |
| C6—O1   | 1.361     | O3—H4     | 1.000     |
| C7—C10\textsuperscript{i} | 1.415     | H4—O3     | 1.000     |

Symmetry code: (i) −x+1, −y+1, −z+1.

Hydrogen-bond geometry (Å, °)

|      | \( D—H \) | \( H···A \) | \( D···A \) | \( D—H···A \) |
|------|-----------|-----------|-----------|---------------|
| O3—H4···O5\textsuperscript{ii} | 1.00      | 1.69      | 2.689      | 174           |
| O1—H2···O5  | 0.99      | 1.68      | 2.567      | 147           |

Symmetry code: (ii) −x+1, −y+2, −z+2.
Crystal data

C₈H₆O₆·2(H₂O)  
Monoclinic, P2₁/c  
c = 5.49900 Å  
β = 103.03°  
V = 487.68 Å³  
Z = 2

Data collection

h = →  
k = →  
l = →

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|    | x   | y   | z   | Biso/*Beq |
|----|-----|-----|-----|-----------|
| O1 | 0.02829 | 0.35100 | 0.84164 |
| H1 | -0.08229 | 0.31915 | 0.93268 |
| O2 | 0.41842 | 0.59430 | 0.63866 |
| H2 | 0.51266 | 0.64044 | 0.56603 |
| O3 | 0.28123 | 0.68492 | 0.87357 |
| C1 | 0.01145 | 0.42401 | 0.92007 |
| C2 | 0.14692 | 0.48106 | 0.82449 |
| H3 | 0.26253 | 0.46707 | 0.68792 |
| C3 | 0.28585 | 0.61663 | 0.80175 |
| C4 | 0.13841 | 0.55682 | 0.90233 |
| O4 | 0.64658 | 0.70011 | 0.46632 |
| H4 | 0.52537 | 0.74263 | 0.39890 |
| H5 | 0.75145 | 0.68680 | 0.34282 |

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|-------|-------|---------|
| O2—H2···O4 | 1.07 | 1.43 | 2.500 | 178 |
| O1—H1···O3¹ | 1.01 | 1.64 | 2.562 | 149 |
| O4—H4···O3¹ | 0.99 | 1.78 | 2.736 | 161 |
| O4—H5···O1³i | 0.99 | 1.82 | 2.794 | 169 |

Symmetry codes: (i) -x, -y+1, -z+2; (ii) x, -y+3/2, z-1/2; (iii) -x+1, -y+1, -z+1.