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## Key indicators

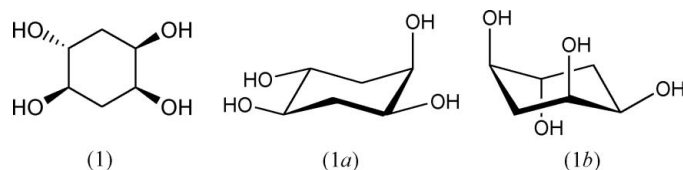
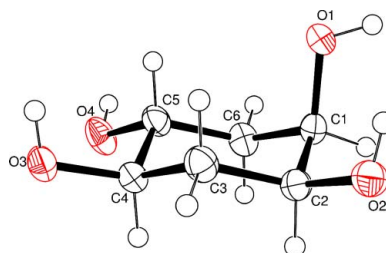
Single-crystal X-ray study  
 $T = 296$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.029  
 $wR$  factor = 0.081  
Data-to-parameter ratio = 8.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(1*R*\*,2*S*\*,4*S*\*,5*S*\*)-Cyclohexane-1,2,4,5-tetrol**The title compound,  $\text{C}_6\text{H}_{12}\text{O}_4$ , exists in a chair form, with three of the four OH groups equatorially disposed. All four hydroxy groups participate in extensive intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonding.

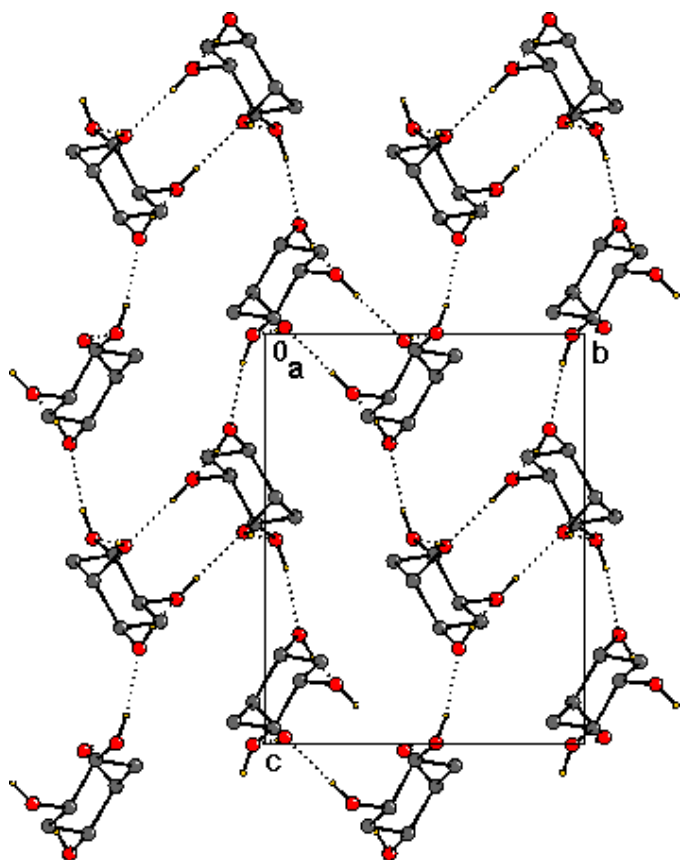
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## Comment

The title compound, (1), is one of the five possible geometrical isomers of 1,2,4,5-cyclohexanetetrol. Compound (1) can be conveniently prepared from 1,4-cyclohexadiene *via* a selective epoxidation–hydrolysis–osmylation strategy (McCasland *et al.*, 1963) and is the only readily obtainable configurational isomer of 1,2,4,5-cyclohexanetetrol capable of existing in two energetically different conformational isomers, (1*a*) and (1*b*). While conformer (1*a*) has three of the four hydroxy groups equatorial and is capable of solely intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonding, conformer (1*b*), with two *syn*-diaxial hydroxy groups, can be stabilized through an intramolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond (Girling *et al.*, 1974; Panagioto-poulos *et al.*, 1974; James *et al.*, 1978).Experimentally, the hydroxy groups in (1) were found to adopt the spatial disposition present in (1*a*) (Fig. 1). Molecules of (1) pack in a herringbone-type arrangement in the non-centrosymmetric space group  $P2_12_12_1$  (Fig. 2). Each tetrol molecule is linked to six nearest neighbors by intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds (Table 2). The puckering parameters (Cremer & Pople, 1975) for the cyclohexane ring [ $q_2 = 0.026$  (2) Å,  $q_3 = -0.582$  (2) Å,  $\varphi_2 = -17$  (4)°,  $Q_T = 0.585$  (2) Å and  $\theta_2 = 177.2$  (2)°] describe a slightly distorted chair conformation. The total puckering amplitude  $Q_T$  is only**Figure 1**  
View of (1), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.



**Figure 2**

The molecular packing of (1), viewed along the *a* axis. H atoms bonded to C atoms have been omitted for clarity. Dotted lines indicate hydrogen bonds.

slightly smaller than that for an ideal chair (0.63 Å).  $\varphi_2$  is close to 0°, which corresponds to a boat conformation. Therefore the cyclohexane ring is distorted from an ideal chair conformation and is flattened at C6, allowing the C1–C6–C5 angle to increase to 112.86 (13)°, while the other internal ring angles remain close to the tetrahedral values. The flattening of the cyclohexane ring at C6 can be ascribed to the non-bonding (1,3-diaxial) interaction between the atom O1 and H atoms bonded to atoms C3 and C5.

## Experimental

Compound (1) was prepared by a modification of the procedure described by McCasland *et al.* (1963). 1,4-Cyclohexadiene (0.5 ml, 5.3 mmol) in dichloromethane (3 ml) was treated with *m*-chloroperbenzoic acid (70% purity, 1.4 g) in dichloromethane (5 ml) at 273 K. The monoepoxide thus obtained was heated with a 0.2 *M* aqueous solution of Na<sub>2</sub>CO<sub>3</sub> (5 ml) at 368 K to obtain *trans*-4-cyclohexene-1,2-diol (0.36 g) in 70% yield (Michaud & Viala, 1999). The diol (0.20 g, 1.8 mmol), upon *cis*-dihydroxylation with catalytic osmium tetroxide (0.5 mol%) and *N*-methylmorpholine-*N*-oxide (50% solution in water, 0.40 ml) in 4:1 acetone–water (0.5 ml), gave the tetrol (1) (0.21 g) in 80% yield. Suitable crystals of (1) were obtained by slow evaporation of its solution in 1:2 dry ethyl acetate–methanol.

## Crystal data

C<sub>6</sub>H<sub>12</sub>O<sub>4</sub>  
*M<sub>r</sub>* = 148.16  
 Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 6.756 (2) Å  
*b* = 8.783 (3) Å  
*c* = 11.271 (4) Å  
*V* = 668.8 (4) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.471 Mg m<sup>−3</sup>

Mo *K*α radiation  
 Cell parameters from 700 reflections  
 $\theta$  = 2.9–27.0°  
 $\mu$  = 0.12 mm<sup>−1</sup>  
*T* = 296 (2) K  
 Block, colorless  
 0.40 × 0.35 × 0.30 mm

## Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min}$  = 0.922,  $T_{\max}$  = 0.964  
 5158 measured reflections

814 independent reflections  
 800 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}}$  = 0.016  
 $\theta_{\text{max}}$  = 26.4°  
 $h$  = −8 → 8  
 $k$  = −10 → 10  
 $l$  = −14 → 13

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)]$  = 0.029  
 $wR(F^2)$  = 0.081  
 $S$  = 1.18  
 814 reflections  
 95 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.064P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}}$  = 0.002  
 $\Delta\rho_{\text{max}}$  = 0.23 e Å<sup>−3</sup>  
 $\Delta\rho_{\text{min}}$  = −0.16 e Å<sup>−3</sup>

**Table 1**

Selected bond angles (°).

C1–C2–C3	111.04 (13)	C4–C5–C6	109.96 (13)
C1–C6–C5	112.86 (13)	C5–C4–C3	109.68 (12)
C4–C3–C2	111.01 (13)	C6–C1–C2	109.83 (12)

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1O...O4 <sup>i</sup>	0.82	1.89	2.705 (2)	172
O2–H2O...O3 <sup>i</sup>	0.82	2.01	2.765 (2)	153
O3–H3O...O1 <sup>ii</sup>	0.82	1.92	2.742 (2)	176
O4–H4O...O2 <sup>iii</sup>	0.82	1.94	2.752 (2)	169

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $+x + \frac{1}{2}, -y + \frac{3}{2}, -z + 1$ ; (iii)  $-x + \frac{3}{2}, -y + 2, +z + \frac{1}{2}$

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances in the range 0.97–0.98 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ , and O–H distances fixed at 0.82 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ . Though (1) is obtained in a racemic form through synthesis, its chiral structure in the solid state appears to have resulted from a spontaneous resolution during crystallization. However, owing to the absence of any heavy atom ( $Z > \text{Si}$ ) in (1), the absolute configuration could not be refined. Friedel pairs (539) were averaged prior to merging of data in *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>; the reported value of  $R_{\text{int}}$  corresponds to subsequent merging of equivalent reflections in this space group.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998); program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and CAMERON (Watkin *et*

*al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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## References

- Altomare, A., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Bruker (1998). *SMART* (Version 6.028) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Girling, R. L. & Jeffrey, G. A. (1974). *Acta Cryst.* **B30**, 327–333.
- James, V. J., Stevens, J. D. & Moore, F. H. (1978). *Acta Cryst.* **B34**, 188–193.
- McCasland, G. E., Furuta, S., Johnson, L. F. & Shoolery, J. N. (1963). *J. Org. Chem.* **28**, 894–900.
- Michaud, S. & Viala, J. (1999). *Tetrahedron*, **55**, 3019–3024.
- Panagiotopoulos, N. C., Jeffrey, G. A., La Placa, S. J. & Hamilton, W. C. (1974). *Acta Cryst.* **B30**, 1421–1430.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.