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Key indicators

Single-crystal X-ray study
 $T = 93$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.043
 wR factor = 0.108
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

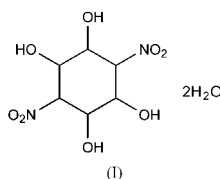
2,3,5,6-Tetrahydroxy-1,4-dinitrocyclohexane dihydrate

The title compound, $\text{C}_6\text{H}_{10}\text{N}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$, involves a cyclohexane with four hydroxyl and two nitro group substituents. In the crystal form reported here, the molecules lie across crystallographic inversion centres. The crystal structure contains an extensive network of hydrogen bonding between the hydroxyl and nitro groups and the water molecules. The water molecule is involved in every hydrogen bond.

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Comment

The title compound, (I), involves a cyclohexane ring with four hydroxyl and two nitro substituents; it was synthesized as a possible intermediate in a search for new energetic propellant ingredients. A crystal structure containing this same title molecule has been reported, but in that crystal structure it is complexed to an organic solvent, thiolan-1-one (Dodge *et al.*, 1972).



In the crystal structure reported here, the title compound co-crystallizes with two symmetry-equivalent water molecules. Both crystal structures contain the same epimer of 2,3,5,6-tetrahydroxy-1,4-dinitrocyclohexane. Nielsen (1961) studied five of the six isomers of dinitrocyclohexane, including the two epimers of 1,4-dinitrocyclohexane. He concluded that the favored epimer was the one in which both nitro groups are in the equatorial position, because of hindrance by axial H atoms. The two nitro groups in the title compound are also both in the equatorial position. The molecule sits on a center of symmetry, with two of its hydroxyl groups (O1 and its centrosymmetric equivalent) in the axial position and the other two in the equatorial position (Fig. 1).

The title compound contains four strong $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds between the hydroxyl and nitro groups and the water molecules, with $\text{H}\cdots\text{O}$ distances ranging from 1.89 (2) to 2.12 (2) Å. This leads to a rather extensive network of hydrogen bonding throughout the crystal. The molecules pack in nearly planar sheets, parallel to the bc plane, and linked to one another through hydrogen bonds, as shown in Fig. 2.

Experimental

The crystals were supplied by Clifford Coon of the Lawrence Livermore National Laboratory.

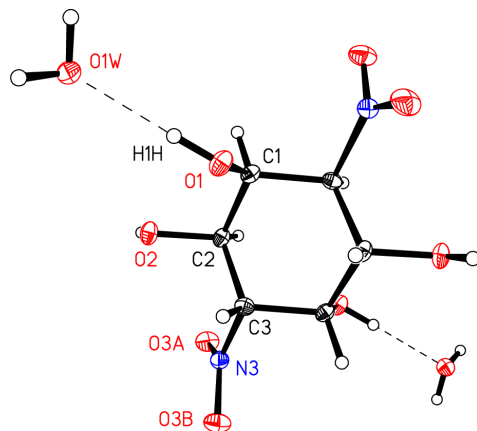


Figure 1
The structure of the title compound (the asymmetric unit and its inversion-equivalent), with 50% probability displacement ellipsoids.

Crystal data

$C_6H_{10}N_2O_8 \cdot 2H_2O$	$Z = 1$
$M_r = 274.19$	$D_x = 1.674 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 4.7309 (5) \text{ \AA}$	Cell parameters from 1494 reflections
$b = 7.8835 (9) \text{ \AA}$	$\theta = 2.5\text{--}28.2^\circ$
$c = 8.0592 (9) \text{ \AA}$	$\mu = 0.16 \text{ mm}^{-1}$
$\alpha = 65.180 (2)^\circ$	$T = 93 (2) \text{ K}$
$\beta = 86.143 (2)^\circ$	Prism, colorless
$\gamma = 85.995 (2)^\circ$	$0.60 \times 0.12 \times 0.08 \text{ mm}$
$V = 271.91 (5) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	1288 independent reflections
φ and ω scans	1075 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SABABS; Bruker, 2001)	$R_{\text{int}} = 0.020$
$T_{\text{min}} = 0.830$, $T_{\text{max}} = 0.981$	$\theta_{\text{max}} = 28.2^\circ$
2086 measured reflections	$h = -6 \rightarrow 5$
	$k = -10 \rightarrow 10$
	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.05P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.108$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.47 \text{ e \AA}^{-3}$
1288 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
94 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1H \cdots O1W	0.87 (2)	1.89 (2)	2.7549 (16)	178 (2)
O2—H2H \cdots O1W ⁱ	0.80 (2)	1.95 (2)	2.7478 (16)	175 (2)
O1W—H1W \cdots O2 ⁱⁱ	0.83 (2)	1.93 (2)	2.7450 (16)	169 (2)
O1W—H2W \cdots O3A ⁱⁱⁱ	0.82 (2)	2.12 (2)	2.9299 (17)	168 (2)

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

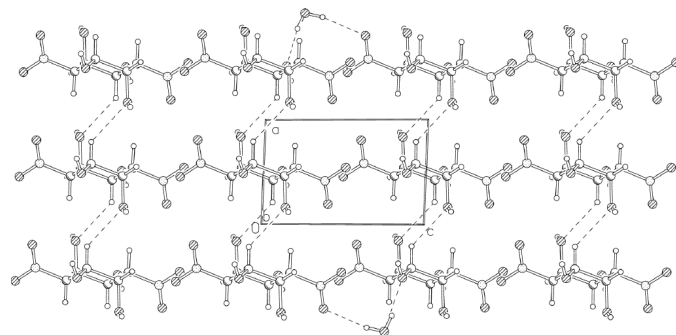


Figure 2

A packing diagram of the title compound, viewed down the b axis. The compound packs in nearly planar stacks, which are connected together by hydrogen bonding.

The hydroxyl and water H atoms were located in a difference Fourier map and their positional parameters were refined, with $U_{\text{iso}}(\text{H})$ displacement parameters set at $1.5U_{\text{eq}}(\text{O})$. The range of O—H distances is 0.80 (2)–0.87 (2) \AA . The C-bound H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 1.00 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The ratio of maximum and minimum residual density (2.01) is significantly greater than 1.0. The top four peaks in the difference map occur at the centers of the C1—C2, C3—N3, C1—C3(1-x, 1-y, -z) and C2—C3 bonds, respectively. The fifth peak, only about 1.15 times the minimum residual, is located 0.8 \AA from O2. No reasonable disorder model for the structure can be proposed from these peaks. As a result of the large fraction of weak data at higher angles, the completeness is slightly low (96%).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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