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(3*R*,3a*S*,6*R*,6a*R*)-3-(1-Nitroethyl)-perhydrofuro[3,2-*b*]furan-3,6-diol

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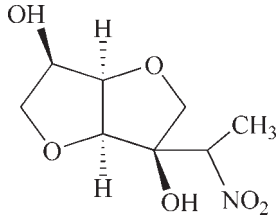
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.049; wR factor = 0.129; data-to-parameter ratio = 6.8.

The molecule of the title compound, $C_8H_{13}NO_6$, a sucrose derivative, consists of two fused tetrahydrofuran rings having the *cis* arrangement at the ring junctions, giving a V-shaped molecule. An intramolecular O—H···O interaction occurs. Intermolecular O—H···O hydrogen bonds help to stabilize the crystal structure.

Related literature

For applications of sucrose and its derivatives, see: Chang *et al.* (2001); Liu *et al.* (2004); Stutz *et al.* (1999).



Experimental

Crystal data

$C_8H_{13}NO_6$
M_r = 219.19
Monoclinic, $P21$
a = 6.959 (4) Å
b = 5.525 (3) Å
c = 12.384 (6) Å
β = 97.077 (7)°
V = 472.5 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 298 K
0.42 × 0.23 × 0.14 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.946, T_{max} = 0.982
2416 measured reflections
935 independent reflections
743 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.129
S = 0.98
932 reflections
137 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1^{i}	0.82	2.06	2.785 (5)	147
O4—H4···O3^{ii}	0.82	2.05	2.777 (4)	147
O4—H4···O1	0.82	2.23	2.655 (4)	113

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge financial support from the National Natural Science Foundation of China (No. 20572103).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2141).

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supporting information

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S1. Comment

Sugar derivatives are an important class of compounds having a broad spectrum of applications in the chemical, biochemical, medicinal (Chang *et al.*, 2001), and pharmaceutical fields,(Liu *et al.*, 2004; Stutz *et al.*, 1999) Here we report a structure of a novel Sugar derivatives. To develop new applications for sucrose and its derivatives, structural modifications of sucrose have been extensively investigated. As a contribution to the sucrose chemistry, we report here the crystal structure of the title compound.

Molecular structure of title compound is shown in Fig.1. Torsion angle C(6)—C(1)—C(2)—C(3) is -120.4. Intermolecular hydrogen bonds links molecules in crystal structure into three-dimensional structure.

S2. Experimental

Nitroethane and a catalytic amount of Et3N were added to a stirring solution of 1,4:3,6-dianhydrofructose in EtOH. The mixture was stirred at room temperature for 4 h, and evaporated under reduced pressure to dryness. The residue was recrystallized with EtOH to give title compound as a white crystal.

S3. Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H = 0.96 Å (methylene) or 0.93 Å (aromatic), 0.82 Å (hydroxyl) and U_{iso}(H) =1.2U_{eq}(C). Because the absolute configuration was established by the structure determination of a compound containing a chiral reference molecule of known absolute configuration, we have merged the Friedels in the refinement.

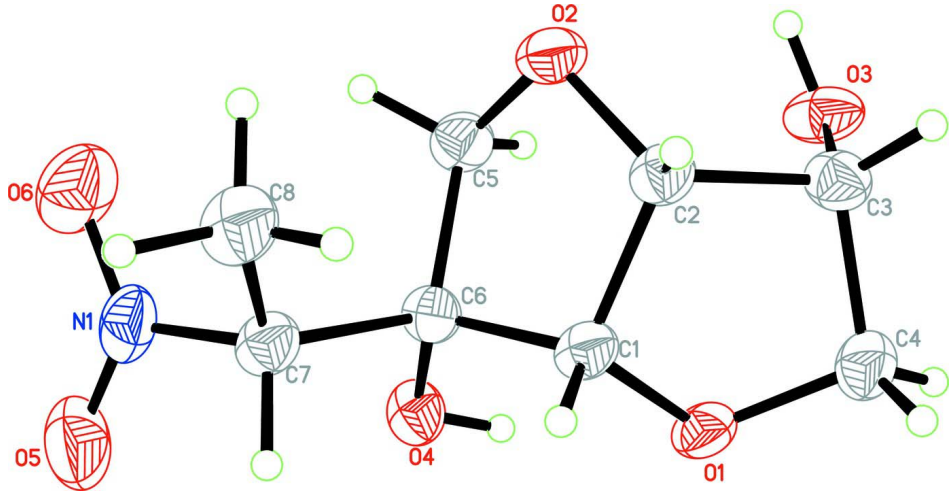

Figure 1

The molecular structure of the compound, with atom labels and 50% probability displacement ellipsoids.

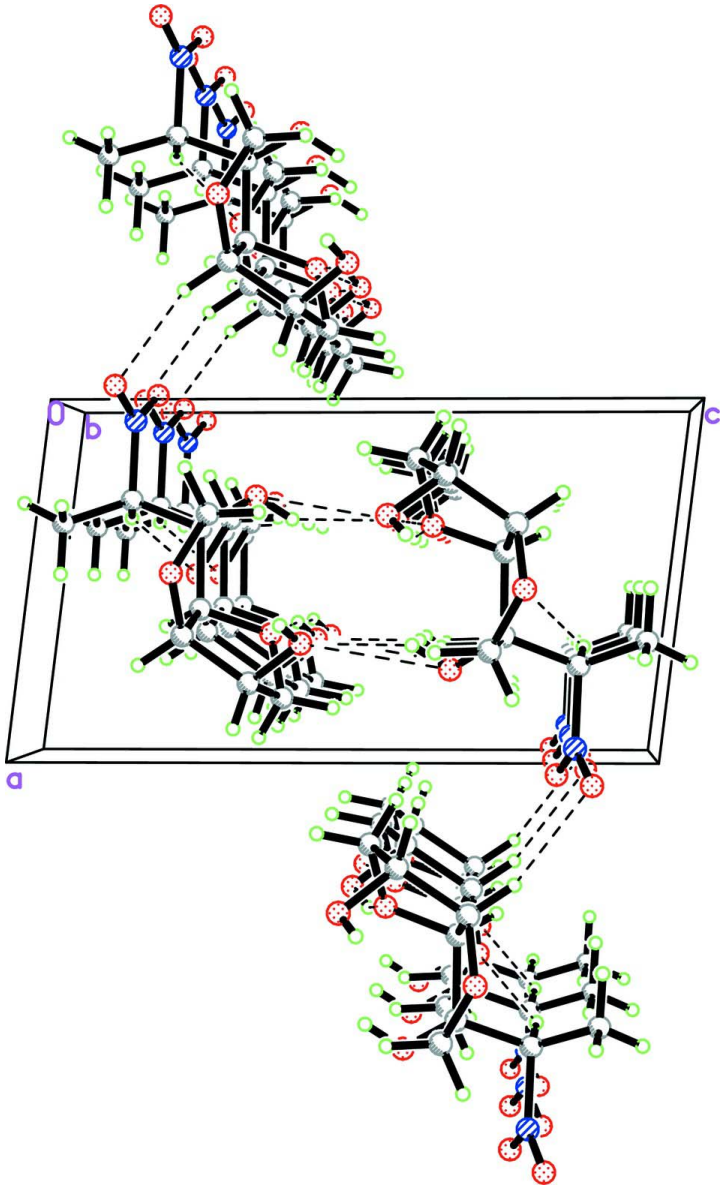

Figure 2

Crystal packing of the title compound, showing a three-dimensional structure, linked by hydrogen bonds(dashed lines).

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$c = 12.384$ (6) Å
$\beta = 97.077$ (7)°
$V = 472.5$ (4) Å^3
$Z = 2$
$F(000) = 232$
$D_x = 1.541$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 895 reflections
$\theta = 3.0$–22.5°

μ = 0.13 mm^{-1}
T = 298 K

Colorless, needlelike
0.42 × 0.23 × 0.14 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.946, T_{max} = 0.982

2416 measured reflections
935 independent reflections
743 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 25.0°, θ_{min} = 1.7°
h = −4→8
k = −6→6
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.129
S = 0.98
932 reflections
137 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.9397 (6)	0.4876 (9)	0.8645 (3)	0.0565 (11)
O1	0.3548 (4)	0.4642 (5)	0.61605 (19)	0.0468 (7)
O2	0.5198 (4)	−0.0131 (5)	0.7607 (2)	0.0485 (7)
O3	0.3353 (5)	−0.0495 (6)	0.55654 (18)	0.0586 (9)
H3	0.3836	−0.1713	0.5865	0.088*
O4	0.7338 (4)	0.5239 (6)	0.6667 (2)	0.0475 (8)
H4	0.6696	0.5192	0.6066	0.071*
O5	1.0171 (6)	0.6770 (9)	0.8438 (3)	0.0869 (13)
O6	1.0297 (6)	0.3057 (9)	0.8937 (3)	0.0867 (12)
C1	0.4230 (5)	0.3930 (7)	0.7261 (3)	0.0402 (10)
H1	0.3753	0.5017	0.7795	0.048*
C2	0.3545 (6)	0.1339 (7)	0.7386 (3)	0.0419 (9)
H2	0.2752	0.1239	0.7986	0.050*
C3	0.2290 (7)	0.0763 (8)	0.6291 (3)	0.0469 (10)

H3A	0.1108	−0.0111	0.6407	0.056*
C4	0.1837 (6)	0.3261 (9)	0.5835 (3)	0.0512 (11)
H4A	0.1558	0.3209	0.5049	0.061*
H4B	0.0732	0.3949	0.6133	0.061*
C5	0.6784 (6)	0.1117 (8)	0.7223 (4)	0.0463 (10)
H5A	0.6822	0.0785	0.6456	0.056*
H5B	0.8001	0.0608	0.7625	0.056*
C6	0.6445 (5)	0.3804 (7)	0.7401 (3)	0.0390 (9)
C7	0.7219 (5)	0.4776 (9)	0.8547 (3)	0.0454 (10)
H7	0.6749	0.6440	0.8596	0.055*
C8	0.6571 (6)	0.3377 (10)	0.9479 (3)	0.0553 (12)
H8A	0.7002	0.1730	0.9450	0.083*
H8B	0.5184	0.3413	0.9429	0.083*
H8C	0.7115	0.4094	1.0154	0.083*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.056 (2)	0.072 (3)	0.0384 (18)	0.008 (2)	−0.0049 (16)	−0.008 (2)
O1	0.0563 (16)	0.0408 (16)	0.0409 (14)	0.0072 (14)	−0.0031 (12)	0.0095 (13)
O2	0.0592 (17)	0.0359 (16)	0.0496 (15)	0.0079 (14)	0.0034 (13)	0.0026 (14)
O3	0.099 (2)	0.0404 (17)	0.0355 (14)	0.0131 (17)	0.0040 (15)	0.0019 (14)
O4	0.0544 (16)	0.0548 (19)	0.0330 (13)	−0.0059 (15)	0.0037 (12)	−0.0004 (13)
O5	0.072 (2)	0.102 (3)	0.081 (3)	−0.026 (3)	−0.012 (2)	0.000 (2)
O6	0.069 (2)	0.099 (3)	0.090 (3)	0.029 (2)	0.001 (2)	−0.007 (3)
C1	0.051 (2)	0.039 (2)	0.0301 (18)	0.010 (2)	0.0039 (16)	0.0002 (16)
C2	0.047 (2)	0.043 (2)	0.0353 (19)	0.004 (2)	0.0038 (16)	0.0008 (18)
C3	0.057 (2)	0.042 (2)	0.041 (2)	−0.003 (2)	0.0034 (18)	0.0019 (18)
C4	0.052 (3)	0.054 (3)	0.044 (2)	0.003 (2)	−0.0067 (18)	−0.001 (2)
C5	0.051 (2)	0.042 (2)	0.046 (2)	0.007 (2)	0.0061 (18)	−0.0064 (19)
C6	0.050 (2)	0.038 (2)	0.0289 (19)	−0.0013 (18)	0.0054 (16)	−0.0012 (15)
C7	0.052 (2)	0.048 (2)	0.0360 (18)	0.008 (2)	0.0031 (16)	−0.002 (2)
C8	0.070 (3)	0.064 (3)	0.033 (2)	0.006 (3)	0.0090 (18)	0.0003 (19)

Geometric parameters (Å, º)

N1—O6	1.215 (6)	C2—H2	0.9800
N1—O5	1.219 (6)	C3—C4	1.509 (6)
N1—C7	1.506 (5)	C3—H3A	0.9800
O1—C4	1.430 (5)	C4—H4A	0.9700
O1—C1	1.442 (4)	C4—H4B	0.9700
O2—C2	1.408 (5)	C5—C6	1.523 (6)
O2—C5	1.431 (5)	C5—H5A	0.9700
O3—C3	1.415 (5)	C5—H5B	0.9700
O3—H3	0.8200	C6—C7	1.550 (5)
O4—C6	1.407 (5)	C7—C8	1.504 (6)
O4—H4	0.8200	C7—H7	0.9800
C1—C2	1.523 (6)	C8—H8A	0.9600

C1—C6	1.531 (5)	C8—H8B	0.9600
C1—H1	0.9800	C8—H8C	0.9600
C2—C3	1.552 (5)		

O6—N1—O5	123.2 (4)	O1—C4—H4B	110.8
O6—N1—C7	118.1 (5)	C3—C4—H4B	110.8
O5—N1—C7	118.7 (4)	H4A—C4—H4B	108.9
C4—O1—C1	106.6 (3)	O2—C5—C6	106.4 (3)
C2—O2—C5	107.6 (3)	O2—C5—H5A	110.5
C3—O3—H3	109.5	C6—C5—H5A	110.5
C6—O4—H4	109.5	O2—C5—H5B	110.5
O1—C1—C2	106.4 (3)	C6—C5—H5B	110.5
O1—C1—C6	109.2 (3)	H5A—C5—H5B	108.6
C2—C1—C6	105.6 (3)	O4—C6—C5	111.5 (3)
O1—C1—H1	111.8	O4—C6—C1	114.8 (3)
C2—C1—H1	111.8	C5—C6—C1	101.5 (3)
C6—C1—H1	111.8	O4—C6—C7	105.3 (3)
O2—C2—C1	107.7 (3)	C5—C6—C7	115.3 (3)
O2—C2—C3	114.2 (3)	C1—C6—C7	108.6 (3)
C1—C2—C3	104.7 (3)	C8—C7—N1	110.5 (3)
O2—C2—H2	110.0	C8—C7—C6	114.9 (4)
C1—C2—H2	110.0	N1—C7—C6	108.6 (3)
C3—C2—H2	110.0	C8—C7—H7	107.5
O3—C3—C4	108.2 (3)	N1—C7—H7	107.5
O3—C3—C2	111.9 (3)	C6—C7—H7	107.5
C4—C3—C2	102.0 (3)	C7—C8—H8A	109.5
O3—C3—H3A	111.4	C7—C8—H8B	109.5
C4—C3—H3A	111.4	H8A—C8—H8B	109.5
C2—C3—H3A	111.4	C7—C8—H8C	109.5
O1—C4—C3	104.7 (3)	H8A—C8—H8C	109.5
O1—C4—H4A	110.8	H8B—C8—H8C	109.5
C3—C4—H4A	110.8		

C4—O1—C1—C2	27.7 (4)	O2—C5—C6—C7	85.8 (4)
C4—O1—C1—C6	141.2 (3)	O1—C1—C6—O4	24.1 (4)
C5—O2—C2—C1	−22.0 (4)	C2—C1—C6—O4	138.1 (3)
C5—O2—C2—C3	93.8 (4)	O1—C1—C6—C5	−96.3 (3)
O1—C1—C2—O2	117.5 (3)	C2—C1—C6—C5	17.7 (3)
C6—C1—C2—O2	1.5 (4)	O1—C1—C6—C7	141.7 (3)
O1—C1—C2—C3	−4.4 (4)	C2—C1—C6—C7	−104.3 (4)
C6—C1—C2—C3	−120.4 (3)	O6—N1—C7—C8	40.8 (5)
O2—C2—C3—O3	−20.6 (5)	O5—N1—C7—C8	−138.7 (4)
C1—C2—C3—O3	96.9 (4)	O6—N1—C7—C6	−86.2 (4)
O2—C2—C3—C4	−136.1 (4)	O5—N1—C7—C6	94.4 (5)
C1—C2—C3—C4	−18.6 (4)	O4—C6—C7—C8	−175.5 (3)
C1—O1—C4—C3	−40.5 (4)	C5—C6—C7—C8	−52.1 (5)
O3—C3—C4—O1	−82.4 (4)	C1—C6—C7—C8	61.0 (5)
C2—C3—C4—O1	35.8 (4)	O4—C6—C7—N1	−51.2 (4)

C2—O2—C5—C6	34.2 (4)	C5—C6—C7—N1	72.2 (4)
O2—C5—C6—O4	−154.1 (3)	C1—C6—C7—N1	−174.7 (3)
O2—C5—C6—C1	−31.3 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1^{i}	0.82	2.06	2.785 (5)	147
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