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# *N*,*N*′-Bis[tris(hydroxymethyl)methyl]propane-1,3-diamine (bis-tris propane)

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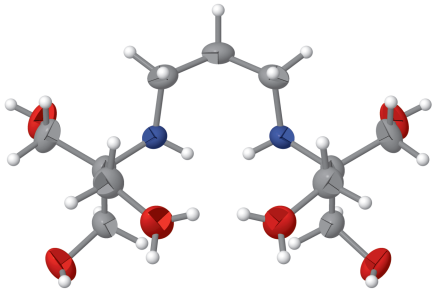
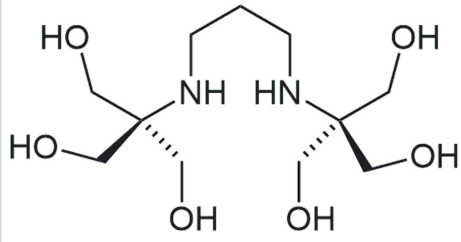
**Structural data:** full structural data are available from iucrdata.iucr.org

The title compound, $C_{11}H_{26}N_2O_6$, used for the preparation of buffer solutions and high-nuclearity coordination complexes, crystallizes with a half molecule in the asymmetric unit. The full molecule is completed through mirror symmetry *m* in the space group *Pnma*. The molecular shape is bent and, as a consequence, some H atoms are disordered to avoid too short H···H intramolecular contacts. Molecules in the crystal are linked *via* O—H···N and N—H···O hydrogen bonds, forming chains along [100], which are further packed through other O—H···O hydrogen bonds between hydroxy groups. The here-reported structure probably represents the less-stable form in a set of polymorphs.

3D view



Chemical scheme




## Structure description

1,3-Bis[tris(hydroxymethyl)methylamino]propane, also known as bis-tris propane or BTP, is a diaminopolyol used in biochemistry and molecular biology for the preparation of buffer solutions in the wide pH range 6.0–9.5. It is readily soluble in water, and can be recrystallized as large plate-shaped single crystals (Fig. 1, inset).

This polydentate molecule can also be used as a ligand for coordination chemistry. Crystal structures based on $Cu^{2+}$ (*e.g.* Milway *et al.*, 2013; Kirillova *et al.*, 2017), $V^{4+}$ (Nachtigall *et al.*, 2017), polyoxidomolybdates (Li *et al.*, 2015) and lanthanides (*e.g.* Yinling *et al.*, 2022) have been reported. It is surprising that the crystal structure of the free ligand BTP has never been published.

BTP crystallizes in a centrosymmetric space group, *Pnma*, with the molecule placed on the mirror plane normal to the unit-cell *b* axis (Fig. 1). Within this structure, the molecule thus belongs to the $C_s$ point group, although it does not display a *trans*-extended geometry, as might be expected. Instead, it adopts a bent-shaped geometry, defined by the *gauche* torsion angle N1—C2—C1—C2$^{i}$ = −70.4 (3)° [symmetry code: (i) $x, \frac{3}{2} - y, z$]. This

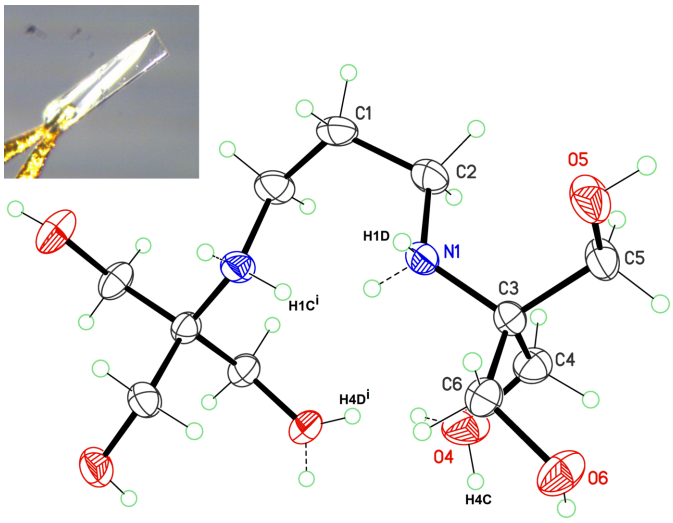



**Figure 1**
Molecular structure of the title compound, with displacement ellipsoids for non-H atoms at the 30% probability level. H atoms with dashed bonds are disordered counterparts for crystallographically equivalent H atoms generated through *m* symmetry ($x$, $\frac{3}{2} - y$, $z$). Non-labelled C, N and O atoms are generated using the same mirror symmetry. The top-left inset shows the single crystal used for data collection. It is *ca*. 0.7 mm long.

shape was previously observed for a $Ca^{2+}$ complex (Liu *et al.*, 2021) or in $Cu^{2+}$ coordination compounds (Milway *et al.*, 2013). The amine H atom is clearly disordered over two equally occupied positions, H1*C* and H1*D*, avoiding a short H···H contact [H1*C*···H1*C*$^{i}$ ≃ 1.4 Å], which would be destabilizing for the molecular structure. In the same way, the H atom for the hydroxy group O4 is disordered over two sites, H4*C* and H4*D*. An alternative would be to refine a non-disordered model in space group $Pn2_1a$, with independent amine H atoms fully occupying sites H1*C* and H1*D* on two independent N atoms, as well as hydroxy H atoms with full occupancy on two independent O4 atoms. Such a model refines well ($R_1$ = 0.039) but is unlikely, for two reasons: (i) convergence is not reached if H atoms are refined with free coordinates, and (ii) intensity statistics show a centric distribution, with, for example, <|*Z* − 1|> = 0.957 (theoretical: 0.968). These disordered H atoms also allow the formation of two intramolecular N—H···N and O—H···O hydrogen bonds, consolidating the bent conformation (Table 1, entries 1 and 2).

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*C*···N1$^{i}$ | 0.86 (2) | 2.24 (3) | 2.970 (3) | 143 (3) |
| O4—H4*D*···O4$^{i}$ | 0.80 (3) | 1.90 (3) | 2.679 (3) | 162 (6) |
| O4—H4*C*···N1$^{ii}$ | 0.82 (3) | 2.09 (3) | 2.905 (2) | 175 (4) |
| N1—H1*D*···O4$^{iii}$ | 0.82 (2) | 2.09 (3) | 2.905 (2) | 169 (3) |
| O6—H6···O5$^{ii}$ | 0.81 (2) | 2.02 (3) | 2.7390 (19) | 148 (3) |
| O5—H5···O6$^{iv}$ | 0.85 (2) | 1.88 (2) | 2.7305 (18) | 176 (3) |

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

The crystal structure is essentially monoperiodic: the molecules form chains along the [100] direction through the intermolecular hydrogen bonds O4—H4*C*···N1$^{ii}$ and N1—H1*D*···O4$^{iii}$ involving amine and hydroxy functional

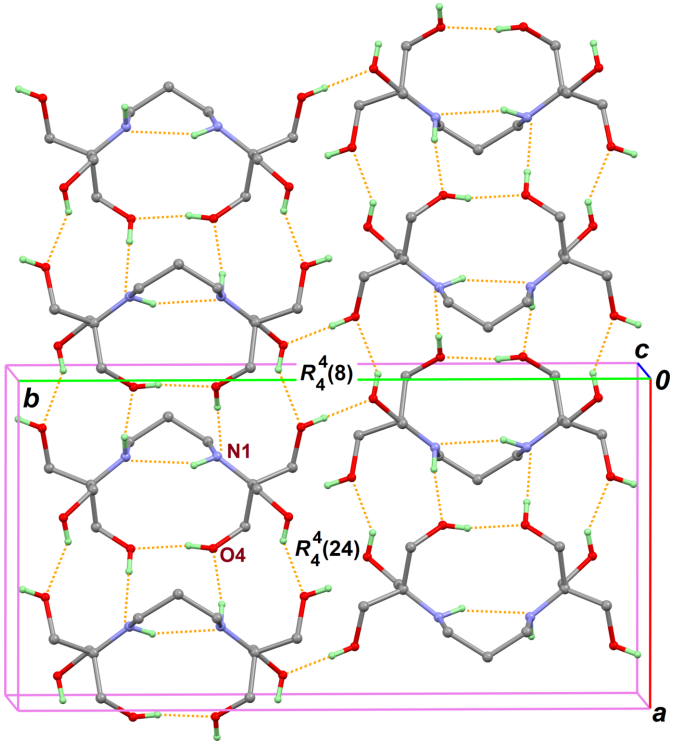



**Figure 2**
Part of the crystal structure, as viewed down unit-cell axis *c*, showing the framework of hydrogen bonds (orange dashed lines). For the sake of clarity, only one position for disordered H atoms bonded to amine N1 and hydroxy O4 groups is retained, and all C-bonded H atoms are omitted.

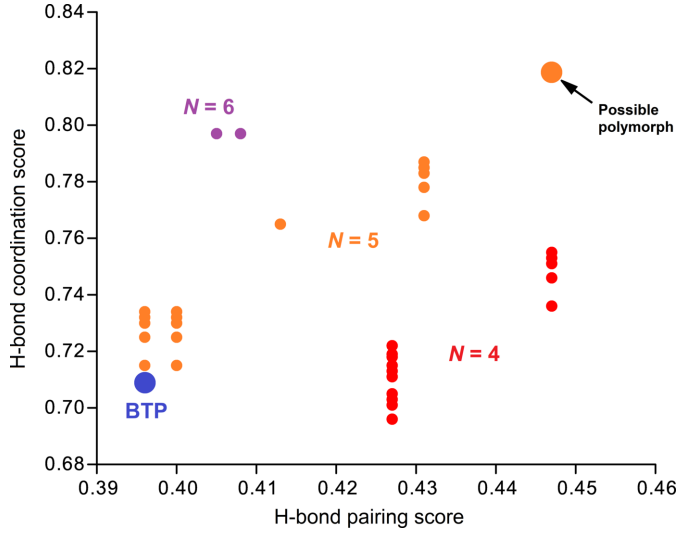



**Figure 3**
Hydrogen-bonding landscape for BTP, calculated with *Mercury* (Macrae *et al.*, 2020). The blue dot on the left-bottom corner corresponds to the here-reported structure, while the orange spot on the upper-right corner is for an hypothetical polymorph. *N* represents the number of intermolecular hydrogen-bond pairs, which are shown with different colours. For the regression analysis, the area under ROC curve was 0.835. Putative hydrogen-bonding networks were built with likelihood > 0.1, for both hydrogen-bond propensity and hydrogen-bond coordination.

groups with disordered H atoms (Table 1, entries 3 and 4). Interchain O—H···O contacts build a network of fused centrosymmetric $R_4^4(8)$ and $R_4^4(24)$ ring motifs, parallel to the monoperiodic chains (see two last entries in Table 1 and Fig. 2).

The unexpected conformation reported herein for BTP could be a consequence of a propensity to polymorphism. The assessment of hydrogen-bond coordination likelihood of BTP was carried out using the hydrogen bond propensity tool available in *Mercury* (Galek *et al.*, 2014; Macrae *et al.*, 2020), with the CSD-6.00 database as a training dataset. The model refined in the $Pn2_1a$ space group was used as a target, since the molecule is disorder-free, and the asymmetric unit includes the complete BTP molecule ($Z' = 1$). The resulting hydrogen-bonding landscape is compelling (Fig. 3): in the map (mean hydrogen-bond pairing propensity, mean hydrogen-bond coordination), the here-reported crystal structure is found at coordinates (0.396, 0.709), while a more stable polymorph is predicted at coordinates (0.447, 0.819). We thus assume that the Ostwald's rule for the formation of polymorphs holds, and that we crystallized the less-stable form of BTP.

## Synthesis and crystallization

BTP, coming from a commercial supplier (Sigma-Aldrich), was recrystallized from a saturated water solution, at room temperature. Single crystals were obtained after a few days.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were visible in difference maps, and were refined with free coordinates and isotropic displacement parameters. O—H and N—H bond lengths were restrained to 0.85 (2) and 0.90 (2) Å, respectively. H atoms bonded to N1 and O4 are disordered over two positions (H1*C*/H1*D* and H4*C*/H4*D*), and their occupancies were fixed to 1/2.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{11}H_{26}N_2O_6$ |
| $M_r$ | 282.34 |
| Crystal system, space group | Orthorhombic, *Pnma* |
| Temperature (K) | 296 |
| $a, b, c$ (Å) | 10.7262 (3), 20.5189 (5), 6.4624 (3) |
| $V$ (Å$^3$) | 1422.31 (8) |
| $Z$ | 4 |
| Radiation type | Ag $K\alpha$, $\lambda$ = 0.56083 Å |
| $\mu$ (mm$^{-1}$) | 0.07 |
| Crystal size (mm) | 0.68 × 0.15 × 0.05 |
| Data collection | |
| Diffractometer | Stoe Stadivari |
| Absorption correction | Multi-scan (*LANA*; Stoe, 2025) |
| $T_{min}$, $T_{max}$ | 0.960, 0.997 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 68162, 1942, 1392 |
| $R_{int}$ | 0.058 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.682 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.047, 0.140, 1.08 |
| No. of reflections | 1942 |
| No. of parameters | 150 |
| No. of restraints | 6 |
| H-atom treatment | All H-atom parameters refined |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.25, −0.22 |

Computer programs: *X-AREA Pilatus3-SV*, *X-AREA Recipe*, *X-AREA Integrate* and *LANA* (Stoe, 2025), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2025/1* (Sheldrick, 2015*b*), *XP* in *SHELXTL-Plus* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

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## References

Galek, P. T. A., Chisholm, J. A., Pidcock, E. & Wood, P. A. (2014). *Acta Cryst.* B**70**, 91–105.
Kirillova, M. V., Santos, C. I. M., André, V., Fernandes, T. A., Dias, S. S. P. & Kirillov, A. M. (2017). *Inorg. Chem. Front.* **4**, 968–977.
Li, H., Shao, B., Li, Y., Chen, L. & Zhao, J. (2015). *Inorg. Chem. Commun.* **61**, 68–72.
Liu, K.-T., Chuang, J.-Y., Jeng, R.-J. & Leung, M. (2021). *ACS Omega* **6**, 27279–27287.
Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
Milway, V. A., Tuna, F., Farrell, A. R., Sharp, L. E., Parsons, S. & Murrie, M. (2013). *Angew. Chem. Int. Ed.* **52**, 1949–1952.
Nachtigall, O., Hagenbach, A., Wiecko, J., Lentz, D., Abram, U. & Spandl, J. (2017). *Dalton Trans.* **46**, 509–516.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Stoe (2025). *X-AREA Pilatus3-SV*, *X-AREA recipe*, *X-AREA Integrate* and *LANA*. Stoe & Cie, Darmstadt, Germany.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Yinling, H., Jia, J., Yao, Z., Shengtao, L., Xinchao, W., Xiaomeng, H. & Xiaoqiang, H. (2022). *Chin. J. Inorg. Chem.* **38**, 2267–2274.

# full crystallographic data

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## *N*,*N*′-Bis[tris(hydroxymethyl)methyl]propane-1,3-diamine (bis-tris propane)

**Ismael Angel-Nieto, Rosa Elena Arroyo-Carmona, Aarón Pérez-Benítez and Sylvain Bernès**

### *N*,*N*′-Bis[tris(hydroxymethyl)methyl]propane-1,3-diamine

*Crystal data*

$C_{11}H_{26}N_2O_6$
$M_r$ = 282.34
Orthorhombic, *Pnma*
$a$ = 10.7262 (3) Å
$b$ = 20.5189 (5) Å
$c$ = 6.4624 (3) Å
$V$ = 1422.31 (8) Å$^3$
$Z$ = 4
$F$(000) = 616
$D_x$ = 1.319 Mg m$^{-3}$
Melting point: 437 K
Ag $K\alpha$ radiation, $\lambda$ = 0.56083 Å
Cell parameters from 34115 reflections
$\theta$ = 2.9–26.7°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 296 K
Plate, colourless
0.68 × 0.15 × 0.05 mm

*Data collection*

Stoe Stadivari diffractometer
Radiation source: Sealed X-ray tube, Axo Astix-f Microfocus source
Graded multilayer mirror monochromator
Detector resolution: 5.81 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*LANA*; Stoe, 2025)
$T_{min}$ = 0.960, $T_{max}$ = 0.997
68162 measured reflections
1942 independent reflections
1392 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.058
$\theta_{max}$ = 22.5°, $\theta_{min}$ = 3.0°
$h$ = −14→12
$k$ = −28→28
$l$ = −8→8

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.140
$S$ = 1.08
1942 reflections
150 parameters
6 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.2802P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.1413 (2) | 0.750000 | 0.6466 (4) | 0.0518 (6) | |
| H1A | 0.064 (3) | 0.750000 | 0.554 (5) | 0.053 (7)* | |
| H1B | 0.104 (3) | 0.750000 | 0.788 (5) | 0.071 (9)* | |
| C2 | 0.2155 (2) | 0.68773 (9) | 0.6187 (3) | 0.0546 (5) | |

| | | | | | |
|---|---|---|---|---|---|
| H2A | 0.291 (2) | 0.6900 (11) | 0.706 (4) | 0.080 (7)* | |
| H2B | 0.168 (2) | 0.6504 (12) | 0.675 (4) | 0.075 (6)* | |
| N1 | 0.25043 (14) | 0.67764 (6) | 0.4019 (2) | 0.0429 (3) | |
| H1C | 0.275 (3) | 0.7150 (13) | 0.357 (5) | 0.043 (9)* | 0.5 |
| H1D | 0.188 (3) | 0.6744 (16) | 0.329 (5) | 0.033 (8)* | 0.5 |
| C3 | 0.33326 (13) | 0.62212 (7) | 0.3552 (2) | 0.0398 (3) | |
| C4 | 0.46486 (15) | 0.63221 (9) | 0.4434 (3) | 0.0510 (4) | |
| H4A | 0.5140 (18) | 0.5938 (10) | 0.417 (3) | 0.050 (5)* | |
| H4B | 0.4579 (17) | 0.6395 (9) | 0.596 (3) | 0.050 (5)* | |
| O4 | 0.52710 (14) | 0.68472 (8) | 0.3514 (3) | 0.0724 (5) | |
| H4C | 0.592 (3) | 0.6809 (18) | 0.284 (6) | 0.048 (10)* | 0.5 |
| H4D | 0.519 (6) | 0.7230 (15) | 0.375 (9) | 0.12 (3)* | 0.5 |
| C5 | 0.28593 (15) | 0.55783 (8) | 0.4441 (3) | 0.0501 (4) | |
| H5A | 0.343 (2) | 0.5226 (10) | 0.405 (3) | 0.059 (5)* | |
| H5B | 0.293 (2) | 0.5591 (10) | 0.591 (4) | 0.062 (6)* | |
| O5 | 0.16180 (11) | 0.54508 (7) | 0.3811 (2) | 0.0631 (4) | |
| H5 | 0.136 (3) | 0.5082 (12) | 0.423 (5) | 0.103 (9)* | |
| C6 | 0.33686 (17) | 0.62006 (8) | 0.1183 (3) | 0.0479 (4) | |
| H6A | 0.2525 (18) | 0.6105 (9) | 0.072 (3) | 0.051 (5)* | |
| H6B | 0.3643 (18) | 0.6628 (10) | 0.065 (3) | 0.058 (5)* | |
| O6 | 0.41686 (13) | 0.57189 (6) | 0.0346 (2) | 0.0616 (4) | |
| H6 | 0.490 (2) | 0.5798 (15) | 0.052 (5) | 0.104 (10)* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0506 (12) | 0.0636 (15) | 0.0410 (12) | 0.000 | 0.0170 (11) | 0.000 |
| C2 | 0.0696 (11) | 0.0542 (10) | 0.0400 (9) | 0.0025 (9) | 0.0154 (8) | 0.0084 (7) |
| N1 | 0.0535 (8) | 0.0364 (6) | 0.0388 (7) | 0.0008 (6) | 0.0116 (6) | 0.0046 (5) |
| C3 | 0.0404 (7) | 0.0332 (7) | 0.0458 (8) | −0.0037 (6) | 0.0032 (6) | 0.0031 (6) |
| C4 | 0.0406 (8) | 0.0500 (9) | 0.0625 (11) | −0.0065 (7) | 0.0037 (8) | −0.0049 (8) |
| O4 | 0.0602 (8) | 0.0573 (8) | 0.0998 (12) | −0.0234 (7) | 0.0339 (8) | −0.0183 (8) |
| C5 | 0.0407 (7) | 0.0407 (8) | 0.0690 (12) | −0.0048 (6) | −0.0056 (8) | 0.0159 (8) |
| O5 | 0.0443 (6) | 0.0509 (7) | 0.0941 (11) | −0.0121 (5) | −0.0080 (6) | 0.0221 (7) |
| C6 | 0.0555 (9) | 0.0386 (8) | 0.0496 (9) | 0.0020 (7) | 0.0062 (8) | −0.0051 (7) |
| O6 | 0.0526 (7) | 0.0546 (7) | 0.0776 (9) | 0.0017 (6) | 0.0052 (7) | −0.0260 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2$^{i}$ | 1.515 (2) | C4—O4 | 1.400 (2) |
| C1—C2 | 1.515 (2) | C4—H4A | 0.96 (2) |
| C1—H1A | 1.02 (3) | C4—H4B | 1.00 (2) |
| C1—H1B | 1.00 (3) | O4—H4C | 0.82 (3) |
| C2—N1 | 1.465 (2) | O4—H4D | 0.80 (3) |
| C2—H2A | 0.99 (3) | C5—O5 | 1.417 (2) |
| C2—H2B | 0.99 (3) | C5—H5A | 0.98 (2) |
| N1—C3 | 1.4759 (19) | C5—H5B | 0.95 (2) |
| N1—H1C | 0.86 (2) | O5—H5 | 0.85 (2) |

| | | | |
|---|---|---|---|
| N1—H1D | 0.82 (2) | C6—O6 | 1.416 (2) |
| C3—C5 | 1.526 (2) | C6—H6A | 0.973 (19) |
| C3—C6 | 1.532 (2) | C6—H6B | 0.99 (2) |
| C3—C4 | 1.536 (2) | O6—H6 | 0.81 (2) |
| | | | |
| C2$^{i}$—C1—C2 | 114.9 (2) | O4—C4—C3 | 112.62 (16) |
| C2$^{i}$—C1—H1A | 111.0 (7) | O4—C4—H4A | 107.1 (11) |
| C2—C1—H1A | 111.0 (7) | C3—C4—H4A | 109.2 (11) |
| C2$^{i}$—C1—H1B | 108.6 (8) | O4—C4—H4B | 109.7 (11) |
| C2—C1—H1B | 108.6 (8) | C3—C4—H4B | 108.5 (11) |
| H1A—C1—H1B | 102 (2) | H4A—C4—H4B | 109.6 (16) |
| N1—C2—C1 | 111.53 (16) | C4—O4—H4C | 124 (3) |
| N1—C2—H2A | 109.8 (15) | C4—O4—H4D | 128 (5) |
| C1—C2—H2A | 108.9 (14) | H4C—O4—H4D | 106 (5) |
| N1—C2—H2B | 111.9 (14) | O5—C5—C3 | 111.35 (14) |
| C1—C2—H2B | 109.7 (13) | O5—C5—H5A | 111.9 (12) |
| H2A—C2—H2B | 104.7 (19) | C3—C5—H5A | 109.6 (12) |
| C2—N1—C3 | 117.32 (14) | O5—C5—H5B | 111.2 (14) |
| C2—N1—H1C | 106 (2) | C3—C5—H5B | 109.1 (13) |
| C3—N1—H1C | 116 (2) | H5A—C5—H5B | 103.4 (18) |
| C2—N1—H1D | 110 (2) | C5—O5—H5 | 112 (2) |
| C3—N1—H1D | 108 (2) | O6—C6—C3 | 114.61 (15) |
| H1C—N1—H1D | 98 (3) | O6—C6—H6A | 107.8 (11) |
| N1—C3—C5 | 112.95 (13) | C3—C6—H6A | 106.8 (11) |
| N1—C3—C6 | 103.95 (12) | O6—C6—H6B | 107.8 (12) |
| C5—C3—C6 | 111.15 (14) | C3—C6—H6B | 109.4 (12) |
| N1—C3—C4 | 111.92 (13) | H6A—C6—H6B | 110.4 (16) |
| C5—C3—C4 | 106.41 (13) | C6—O6—H6 | 113 (2) |
| C6—C3—C4 | 110.56 (14) | | |
| | | | |
| C2$^{i}$—C1—C2—N1 | −70.4 (3) | C6—C3—C4—O4 | 50.48 (19) |
| C1—C2—N1—C3 | 174.27 (15) | N1—C3—C5—O5 | 53.8 (2) |
| C2—N1—C3—C5 | 52.1 (2) | C6—C3—C5—O5 | −62.6 (2) |
| C2—N1—C3—C6 | 172.74 (15) | C4—C3—C5—O5 | 176.99 (16) |
| C2—N1—C3—C4 | −67.92 (19) | N1—C3—C6—O6 | 176.97 (13) |
| N1—C3—C4—O4 | −64.88 (19) | C5—C3—C6—O6 | −61.23 (19) |
| C5—C3—C4—O4 | 171.30 (16) | C4—C3—C6—O6 | 56.72 (18) |

Symmetry code: (i) $x$, $-y+3/2$, $z$.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*C*···N1$^{i}$ | 0.86 (2) | 2.24 (3) | 2.970 (3) | 143 (3) |
| O4—H4*D*···O4$^{i}$ | 0.80 (3) | 1.90 (3) | 2.679 (3) | 162 (6) |
| O4—H4*C*···N1$^{ii}$ | 0.82 (3) | 2.09 (3) | 2.905 (2) | 175 (4) |
| N1—H1*D*···O4$^{iii}$ | 0.82 (2) | 2.09 (3) | 2.905 (2) | 169 (3) |

| | | | | |
|---|---|---|---|---|
| O6—H6···O5$^{ii}$ | 0.81 (2) | 2.02 (3) | 2.7390 (19) | 148 (3) |
| O5—H5···O6$^{iv}$ | 0.85 (2) | 1.88 (2) | 2.7305 (18) | 176 (3) |

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