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# 4-Cyclopropyl-1-(6′-deoxy-1′,2′-*O*-isopropylidene-*α*-D-glucofuranosyl)-1*H*-1,2,3-triazole

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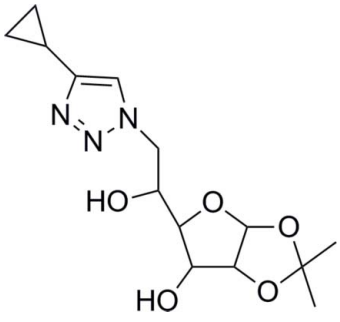
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.004 Å; disorder in main residue; $R$ factor = 0.042; $wR$ factor = 0.113; data-to-parameter ratio = 12.6.

In the title compound, $C_{14}H_{21}N_3O_5$, the tetrahydrofuran ring adopts an envelope conformation with the C atom bearing the substituent as the flap. The pentafuranose ring adopts a twisted conformation about the C—C bond fusing the rings. The dihedral angle between these rings (all atoms), which are *cis* fused, is 72.89 (14)°. The cyclopropane ring is disordered over two orientations in a 0.576 (5):0.424 (5) ratio; the dihedral angles subtended to the triazole ring are 53.3 (11) and 46.6 (9)°, respectively. In the crystal, the molecules are linked by O—H···N and O—H···O hydrogen bonds, generating (001) sheets. A weak C—H···O interaction also occurs.

## Related literature

For further synthetic details, see: Pradere *et al.* (2008). For background to 1,2,3-triazoles, see: Alvarez *et al.* (1994); Genin *et al.* (2000).



## Experimental

### Crystal data

$C_{14}H_{21}N_3O_5$
$M_r$ = 311.34
Orthorhombic, $P2_12_12_1$
$a$ = 8.5905 (3) Å
$b$ = 8.7215 (3) Å
$c$ = 20.7373 (7) Å
$V$ = 1553.68 (9) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.85 mm$^{-1}$
$T$ = 291 K
0.22 × 0.2 × 0.18 mm

### Data collection

Bruker MWPC diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min}$ = 0.835, $T_{max}$ = 0.862
5692 measured reflections
2778 independent reflections
2503 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
$S$ = 1.06
2778 reflections
220 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···N3$^{i}$ | 0.83 (2) | 1.95 (2) | 2.767 (3) | 171 (3) |
| O5—H5···O3$^{ii}$ | 0.82 (3) | 2.02 (3) | 2.821 (3) | 164 (3) |
| C7—H7···O1$^{iii}$ | 0.93 | 2.59 | 3.496 (3) | 165 |

Symmetry codes: (i) $x, y+1, z$; (ii) $x+\frac{1}{2}, -y+\frac{5}{2}, -z+1$; (iii) $x+1, y, z$.

Data collection: *FRAMBO* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

We gratefully acknowledge the financial support of the National Natural Science Foundation of China (grant No. 81172937).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7111).

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# supporting information

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# 4-Cyclopropyl-1-(6′-deoxy-1′,2′-*O*-isopropylidene-*α*-D-glucofuranosyl)-1*H*-1,2,3-triazole

**Qiurong Zhang, Peng He, Guangqiang Zhou, Kang Yu and Hongmin Liu**

## S1. Comment

1,2,3-Triazoles have been shown to have various biological activities, such as anti-HIV (Alvarez *et al.*, 1994) and antibacterial (Genin *et al.*, 2000). $C_{14}H_{21}N3_O5$, the title compound (I), is a new 1,2,3-triazole. The nucleus of the molecule consists of one methylenedioxy rings, one 1,2,3-triazole ring, one cyclopropyl ring and one tetrahydrofuran ring (Fig. 1). The tetrahydrofuran ring fuses with one methylenedioxy ring,having the *cis* arrangement at the ring junctions and giving a V-shaped molecule.

The crystal packing, which features O—H···N hydrogen bonds (Table 1), is shown in Figure 2.

## S2. Experimental

The title compound (I) was synthesized from 6-azido-6-deoxy-1,2-*O*-isopropylidene-alpha -*D*-glucofuranose, whose starting material was D-glucose. The copper catalyzed reaction of 6-azido-6-deoxy-1,2-*O*-isopropylidene-alpha -*D*-glucofuranose(1 mmol) and cyclopropylacetylene (1.2 mmol) in water/tetrahydrofuran (2 ml:2 ml) was stirred for 3 h at room temperature. The mixture was filtered and evaporated. and the residue extracted with EtOAc (50 ml). The organic layer was washed brine, dried over $Na_2SO_4$ for 6 h, filtered, and the solvent evaporated *in vacuo*. Purification of the residue by column chromatography gave the title compound as white solid.

Colourless prisms were grown by slow evaporation from acetone solution at room temperature for two weeks. mp:389–391k; $R_f$ = 0.30 (petroleum ether/EtOAc, 1:1); $^1$H NMR (400 MHz, DMSO-d$_6$)$\sigma$: 7.69(1*H*, s), 5.86(1*H*, d, *J* = 3.6 Hz), 5.27(1*H*, d, *J* = 4.9 Hz), 5.19(1*H*, d, *J* = 6.4 Hz), 4.47(1*H*, dt, *J* = 10.2, 5.1 Hz), 4.42(1*H*, d, *J* = 3.6 Hz), 4.23(1*H*, d, *J* = 14.0, 8.0 Hz), 4.08–3.93(2*H*, m), 3.75(1*H*, dd, *J* = 8.8, 2.5 Hz), 1.92(1*H*, dq, *J* = 8.4, 5.0 Hz), 1.37(3*H*, s), 1.24(3*H*, s), 0.94–0.82(2*H*, m), 0.79–0.63(2*H*, m); $^{13}$C NMR (100 MHz, DMSO-d$_6$) $\sigma$: 153.61, 126.82, 115.95, 109.77, 89.90, 86.23, 78.05, 71.49, 58.80, 31.88, 31.39, 12.70, 11.72.

## S3. Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H are 0.96 Å (methylene) or 0.93 Å (aromatic), 0.82 Å (hydroxyl)and $U_{iso}$(H) =1.2$U_{eq}$(C).

Attempts to confirm the absolute structure by refinement of the Flack parameter in the presence of 1156 sets of Friedel equivalents led to an inconclusive value of 0.0 (3). Therefore, the absolute configuration was assigned to correspond with that of the known chiral centres in a precursor molecule, which remained unchanged during the synthesis of the title compound.

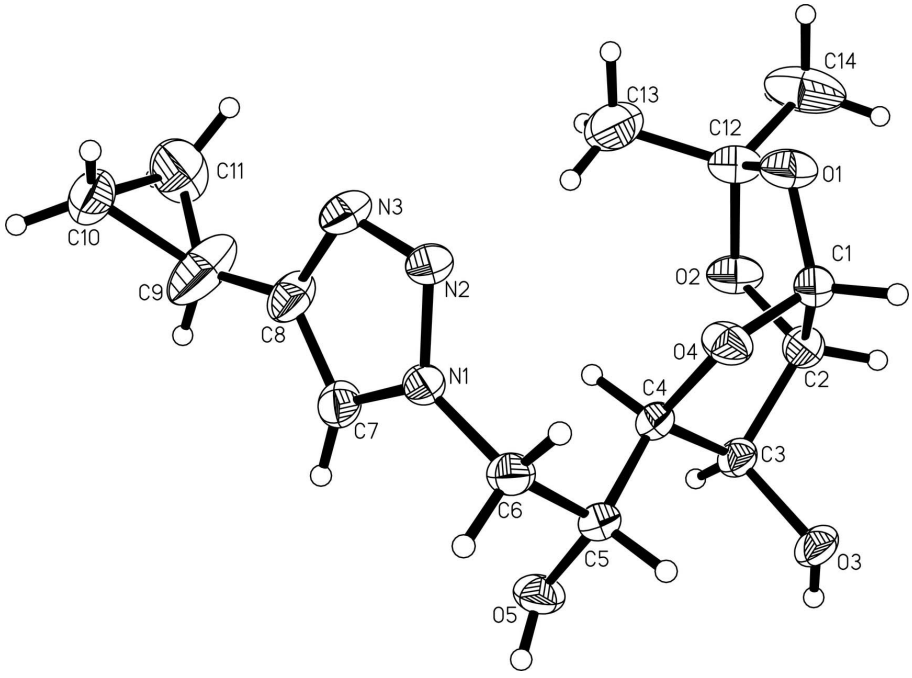


**Figure 1**

The molecular structure of (I) showing 30% probability displacement ellipsoids.

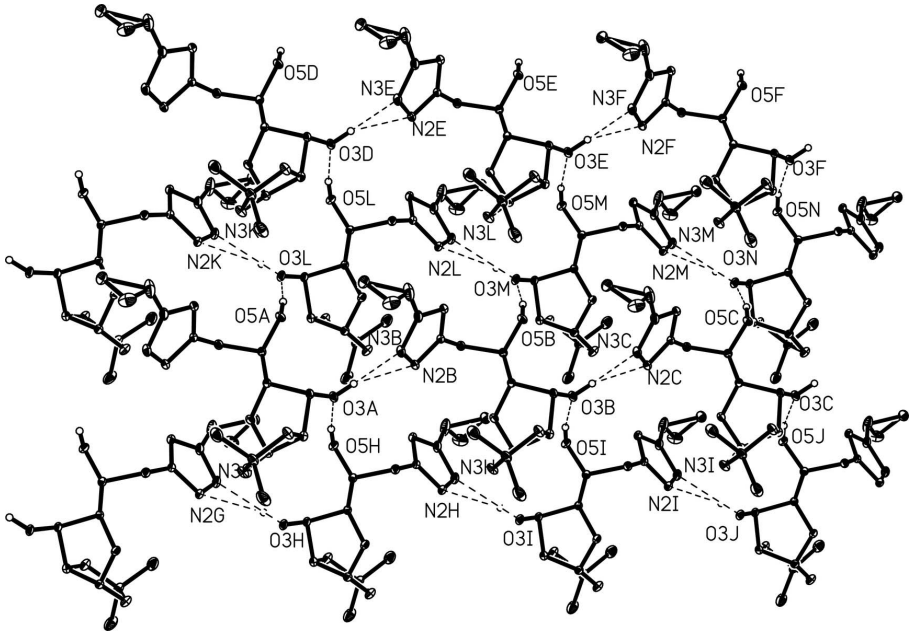


**Figure 2**

Packing diagram for (I).

**4-Cyclopropyl-1-(6′-Deoxy-1′,2′-*O*-isopropylidene-*α*-*D*-glucofuranosyl)-1*H*-1,2,3-triazole**

*Crystal data*

$C_{14}H_{21}N_3O_5$
$M_r = 311.34$
Orthorhombic, $P2_12_12_1$
$a = 8.5905$ (3) Å
$b = 8.7215$ (3) Å
$c = 20.7373$ (7) Å
$V = 1553.68$ (9) Å$^3$
$Z = 4$
$F(000) = 664$
$D_x = 1.331$ Mg m$^{-3}$
Melting point = 389–391 K
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 2697 reflections
$\theta = 4.3$–$67.0°$
$\mu = 0.85$ mm$^{-1}$
$T = 291$ K
PRISMATIC, colourless
0.22 × 0.2 × 0.18 mm

*Data collection*

Bruker MWPC diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm$^{-1}$
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.835$, $T_{max} = 0.862$
5692 measured reflections
2778 independent reflections
2503 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 67.1°$, $\theta_{min} = 4.3°$
$h = -10 \rightarrow 6$
$k = -6 \rightarrow 10$
$l = -24 \rightarrow 22$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.113$
$S = 1.06$
2778 reflections
220 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.1063P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0020 (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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | −0.0805 (2) | 0.9329 (2) | 0.39252 (9) | 0.0556 (5) | |
| O2 | 0.0625 (2) | 1.0684 (2) | 0.31968 (8) | 0.0541 (5) | |
| O3 | 0.2108 (2) | 1.32136 (18) | 0.44356 (9) | 0.0487 (4) | |

| | | | | | |
|---|---|---|---|---|---|
| O4 | 0.12445 (19) | 0.99779 (18) | 0.46166 (8) | 0.0470 (4) | |
| O5 | 0.5249 (2) | 1.1144 (2) | 0.44763 (9) | 0.0517 (4) | |
| N1 | 0.4448 (2) | 0.7779 (2) | 0.46218 (9) | 0.0395 (4) | |
| N2 | 0.3396 (2) | 0.6662 (2) | 0.45796 (11) | 0.0461 (5) | |
| N3 | 0.3860 (3) | 0.5760 (2) | 0.41084 (11) | 0.0536 (5) | |
| C1 | −0.0064 (3) | 1.0493 (3) | 0.42728 (12) | 0.0427 (5) | |
| H1 | −0.0800 | 1.1009 | 0.4561 | 0.051* | |
| C2 | 0.0544 (3) | 1.1602 (3) | 0.37614 (12) | 0.0458 (5) | |
| H2 | −0.0119 | 1.2508 | 0.3708 | 0.055* | |
| C3 | 0.2187 (3) | 1.1997 (3) | 0.39848 (11) | 0.0398 (5) | |
| H3A | 0.2871 | 1.2245 | 0.3621 | 0.048* | |
| C4 | 0.2639 (2) | 1.0486 (2) | 0.43038 (11) | 0.0378 (5) | |
| H4 | 0.2922 | 0.9745 | 0.3968 | 0.045* | |
| C5 | 0.3927 (2) | 1.0555 (2) | 0.47975 (11) | 0.0401 (5) | |
| H5A | 0.3625 | 1.1273 | 0.5139 | 0.048* | |
| C6 | 0.4246 (3) | 0.8990 (3) | 0.51006 (11) | 0.0427 (5) | |
| H6A | 0.5179 | 0.9056 | 0.5363 | 0.051* | |
| H6B | 0.3387 | 0.8723 | 0.5382 | 0.051* | |
| C7 | 0.5573 (3) | 0.7605 (3) | 0.41813 (13) | 0.0486 (6) | |
| H7 | 0.6429 | 0.8238 | 0.4115 | 0.058* | |
| C8 | 0.5199 (4) | 0.6308 (3) | 0.38513 (14) | 0.0560 (7) | |
| C9 | 0.6279 (7) | 0.5539 (8) | 0.3376 (4) | 0.0575 (13) | 0.576 (5) |
| H9 | 0.7207 | 0.6118 | 0.3247 | 0.069* | 0.576 (5) |
| C10 | 0.5544 (9) | 0.4602 (11) | 0.2880 (4) | 0.0937 (19) | 0.576 (5) |
| H10A | 0.4419 | 0.4512 | 0.2890 | 0.112* | 0.576 (5) |
| H10B | 0.5990 | 0.4632 | 0.2450 | 0.112* | 0.576 (5) |
| C11 | 0.6422 (10) | 0.3805 (10) | 0.3380 (4) | 0.076 (2) | 0.576 (5) |
| H11A | 0.7418 | 0.3361 | 0.3262 | 0.091* | 0.576 (5) |
| H11B | 0.5842 | 0.3241 | 0.3703 | 0.091* | 0.576 (5) |
| C12 | −0.0353 (3) | 0.9372 (3) | 0.32677 (12) | 0.0537 (6) | |
| C13 | 0.0573 (5) | 0.7939 (4) | 0.31160 (18) | 0.0822 (10) | |
| H13A | 0.1480 | 0.7901 | 0.3386 | 0.123* | |
| H13B | 0.0886 | 0.7957 | 0.2672 | 0.123* | |
| H13C | −0.0060 | 0.7051 | 0.3194 | 0.123* | |
| C14 | −0.1795 (4) | 0.9543 (5) | 0.28580 (19) | 0.0976 (14) | |
| H14A | −0.2461 | 0.8672 | 0.2922 | 0.146* | |
| H14B | −0.1503 | 0.9606 | 0.2412 | 0.146* | |
| H14C | −0.2338 | 1.0460 | 0.2980 | 0.146* | |
| C9A | 0.5637 (10) | 0.5566 (12) | 0.3225 (5) | 0.0575 (13) | 0.424 (5) |
| H9A | 0.5328 | 0.6124 | 0.2835 | 0.069* | 0.424 (5) |
| C10A | 0.7242 (11) | 0.4953 (13) | 0.3224 (5) | 0.0937 (19) | 0.424 (5) |
| H10C | 0.7798 | 0.4917 | 0.3631 | 0.112* | 0.424 (5) |
| H10D | 0.7883 | 0.5136 | 0.2847 | 0.112* | 0.424 (5) |
| C11A | 0.5835 (14) | 0.3960 (15) | 0.3149 (7) | 0.076 (2) | 0.424 (5) |
| H11C | 0.5634 | 0.3524 | 0.2727 | 0.091* | 0.424 (5) |
| H11D | 0.5550 | 0.3307 | 0.3509 | 0.091* | 0.424 (5) |
| H5 | 0.591 (3) | 1.141 (4) | 0.4739 (12) | 0.059 (9)* | |
| H3 | 0.266 (3) | 1.393 (3) | 0.4302 (14) | 0.058 (8)* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0553 (10) | 0.0571 (10) | 0.0543 (9) | −0.0242 (9) | 0.0018 (8) | 0.0009 (8) |
| O2 | 0.0620 (11) | 0.0563 (10) | 0.0439 (8) | −0.0223 (9) | −0.0041 (8) | 0.0044 (8) |
| O3 | 0.0526 (9) | 0.0281 (7) | 0.0655 (11) | −0.0076 (7) | 0.0121 (8) | −0.0079 (7) |
| O4 | 0.0394 (8) | 0.0445 (8) | 0.0573 (9) | −0.0097 (7) | −0.0010 (7) | 0.0133 (7) |
| O5 | 0.0420 (9) | 0.0476 (9) | 0.0654 (11) | −0.0149 (7) | −0.0044 (8) | 0.0029 (8) |
| N1 | 0.0358 (9) | 0.0308 (8) | 0.0521 (10) | −0.0021 (7) | −0.0022 (8) | 0.0047 (8) |
| N2 | 0.0431 (10) | 0.0332 (9) | 0.0621 (11) | −0.0064 (8) | 0.0011 (9) | 0.0018 (9) |
| N3 | 0.0657 (13) | 0.0322 (9) | 0.0628 (12) | −0.0073 (10) | 0.0019 (10) | −0.0037 (9) |
| C1 | 0.0357 (10) | 0.0382 (11) | 0.0542 (12) | −0.0002 (9) | 0.0036 (10) | −0.0022 (10) |
| C2 | 0.0435 (12) | 0.0362 (11) | 0.0579 (14) | 0.0003 (10) | −0.0021 (10) | 0.0052 (10) |
| C3 | 0.0414 (11) | 0.0291 (10) | 0.0488 (12) | −0.0028 (9) | 0.0066 (10) | −0.0005 (9) |
| C4 | 0.0369 (10) | 0.0283 (10) | 0.0483 (11) | −0.0003 (8) | 0.0021 (9) | −0.0038 (9) |
| C5 | 0.0384 (11) | 0.0320 (10) | 0.0498 (11) | −0.0040 (9) | −0.0006 (9) | −0.0061 (9) |
| C6 | 0.0415 (12) | 0.0397 (11) | 0.0470 (11) | −0.0034 (9) | −0.0059 (9) | 0.0011 (10) |
| C7 | 0.0426 (12) | 0.0357 (11) | 0.0676 (15) | −0.0003 (10) | 0.0094 (11) | 0.0072 (10) |
| C8 | 0.0673 (17) | 0.0349 (11) | 0.0658 (15) | 0.0006 (11) | 0.0148 (13) | 0.0022 (11) |
| C9 | 0.049 (4) | 0.0495 (16) | 0.074 (4) | 0.001 (3) | 0.006 (3) | −0.003 (2) |
| C10 | 0.074 (3) | 0.127 (5) | 0.080 (3) | 0.012 (4) | 0.000 (3) | −0.046 (4) |
| C11 | 0.089 (6) | 0.047 (2) | 0.091 (6) | 0.017 (4) | 0.022 (4) | −0.001 (4) |
| C12 | 0.0585 (15) | 0.0525 (14) | 0.0502 (12) | −0.0192 (12) | −0.0062 (12) | 0.0014 (12) |
| C13 | 0.091 (2) | 0.0624 (19) | 0.093 (2) | −0.0184 (18) | 0.019 (2) | −0.0190 (18) |
| C14 | 0.092 (3) | 0.107 (3) | 0.093 (2) | −0.047 (2) | −0.042 (2) | 0.038 (2) |
| C9A | 0.049 (4) | 0.0495 (16) | 0.074 (4) | 0.001 (3) | 0.006 (3) | −0.003 (2) |
| C10A | 0.074 (3) | 0.127 (5) | 0.080 (3) | 0.012 (4) | 0.000 (3) | −0.046 (4) |
| C11A | 0.089 (6) | 0.047 (2) | 0.091 (6) | 0.017 (4) | 0.022 (4) | −0.001 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.399 (3) | C7—C8 | 1.361 (4) |
| O1—C12 | 1.418 (3) | C8—C9 | 1.511 (8) |
| O2—C2 | 1.420 (3) | C8—C9A | 1.499 (12) |
| O2—C12 | 1.428 (3) | C9—H9 | 0.9800 |
| O3—C3 | 1.416 (3) | C9—C10 | 1.459 (10) |
| O3—H3 | 0.829 (18) | C9—C11 | 1.517 (11) |
| O4—C1 | 1.405 (3) | C10—H10A | 0.9700 |
| O4—C4 | 1.432 (3) | C10—H10B | 0.9700 |
| O5—C5 | 1.413 (3) | C10—C11 | 1.458 (11) |
| O5—H5 | 0.821 (18) | C11—H11A | 0.9700 |
| N1—N2 | 1.332 (3) | C11—H11B | 0.9700 |
| N1—C6 | 1.460 (3) | C12—C13 | 1.514 (4) |
| N1—C7 | 1.338 (3) | C12—C14 | 1.509 (4) |
| N2—N3 | 1.316 (3) | C13—H13A | 0.9600 |
| N3—C8 | 1.355 (4) | C13—H13B | 0.9600 |
| C1—H1 | 0.9800 | C13—H13C | 0.9600 |
| C1—C2 | 1.527 (3) | C14—H14A | 0.9600 |

| | | | |
|---|---|---|---|
| C2—H2 | 0.9800 | C14—H14B | 0.9600 |
| C2—C3 | 1.525 (3) | C14—H14C | 0.9600 |
| C3—H3A | 0.9800 | C9A—H9A | 0.9800 |
| C3—C4 | 1.525 (3) | C9A—C10A | 1.479 (12) |
| C4—H4 | 0.9800 | C9A—C11A | 1.420 (17) |
| C4—C5 | 1.509 (3) | C10A—H10C | 0.9700 |
| C5—H5A | 0.9800 | C10A—H10D | 0.9700 |
| C5—C6 | 1.528 (3) | C10A—C11A | 1.495 (18) |
| C6—H6A | 0.9700 | C11A—H11C | 0.9700 |
| C6—H6B | 0.9700 | C11A—H11D | 0.9700 |
| C7—H7 | 0.9300 | | |

| | | | |
|---|---|---|---|
| C1—O1—C12 | 110.60 (18) | C8—C9—H9 | 116.7 |
| C2—O2—C12 | 109.78 (18) | C8—C9—C11 | 119.2 (6) |
| C3—O3—H3 | 108 (2) | C10—C9—C8 | 116.3 (5) |
| C1—O4—C4 | 109.89 (16) | C10—C9—H9 | 116.7 |
| C5—O5—H5 | 110 (2) | C10—C9—C11 | 58.6 (5) |
| N2—N1—C6 | 119.59 (19) | C11—C9—H9 | 116.7 |
| N2—N1—C7 | 111.23 (19) | C9—C10—H10A | 117.5 |
| C7—N1—C6 | 129.17 (19) | C9—C10—H10B | 117.5 |
| N3—N2—N1 | 106.30 (19) | H10A—C10—H10B | 114.6 |
| N2—N3—C8 | 109.8 (2) | C11—C10—C9 | 62.7 (5) |
| O1—C1—O4 | 113.18 (19) | C11—C10—H10A | 117.5 |
| O1—C1—H1 | 110.7 | C11—C10—H10B | 117.5 |
| O1—C1—C2 | 104.92 (19) | C9—C11—H11A | 117.9 |
| O4—C1—H1 | 110.7 | C9—C11—H11B | 117.9 |
| O4—C1—C2 | 106.35 (17) | C10—C11—C9 | 58.7 (5) |
| C2—C1—H1 | 110.7 | C10—C11—H11A | 117.9 |
| O2—C2—C1 | 103.44 (18) | C10—C11—H11B | 117.9 |
| O2—C2—H2 | 112.9 | H11A—C11—H11B | 115.1 |
| O2—C2—C3 | 109.4 (2) | O1—C12—O2 | 106.32 (19) |
| C1—C2—H2 | 112.9 | O1—C12—C13 | 108.8 (3) |
| C3—C2—C1 | 104.40 (18) | O1—C12—C14 | 108.6 (3) |
| C3—C2—H2 | 112.9 | O2—C12—C13 | 109.3 (2) |
| O3—C3—C2 | 109.04 (18) | O2—C12—C14 | 110.2 (3) |
| O3—C3—H3A | 111.8 | C14—C12—C13 | 113.3 (3) |
| O3—C3—C4 | 111.92 (19) | C12—C13—H13A | 109.5 |
| C2—C3—H3A | 111.8 | C12—C13—H13B | 109.5 |
| C2—C3—C4 | 99.90 (17) | C12—C13—H13C | 109.5 |
| C4—C3—H3A | 111.8 | H13A—C13—H13B | 109.5 |
| O4—C4—C3 | 104.53 (17) | H13A—C13—H13C | 109.5 |
| O4—C4—H4 | 109.0 | H13B—C13—H13C | 109.5 |
| O4—C4—C5 | 108.58 (18) | C12—C14—H14A | 109.5 |
| C3—C4—H4 | 109.0 | C12—C14—H14B | 109.5 |
| C5—C4—C3 | 116.50 (18) | C12—C14—H14C | 109.5 |
| C5—C4—H4 | 109.0 | H14A—C14—H14B | 109.5 |
| O5—C5—C4 | 106.52 (18) | H14A—C14—H14C | 109.5 |
| O5—C5—H5A | 108.7 | H14B—C14—H14C | 109.5 |

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| O5—C5—C6 | 111.98 (18) | C8—C9A—H9A | 115.6 |
| C4—C5—H5A | 108.7 | C10A—C9A—C8 | 113.0 (7) |
| C4—C5—C6 | 112.02 (17) | C10A—C9A—H9A | 115.6 |
| C6—C5—H5A | 108.7 | C11A—C9A—C8 | 123.5 (10) |
| N1—C6—C5 | 112.82 (18) | C11A—C9A—H9A | 115.6 |
| N1—C6—H6A | 109.0 | C11A—C9A—C10A | 62.1 (8) |
| N1—C6—H6B | 109.0 | C9A—C10A—H10C | 118.1 |
| C5—C6—H6A | 109.0 | C9A—C10A—H10D | 118.1 |
| C5—C6—H6B | 109.0 | C9A—C10A—C11A | 57.0 (7) |
| H6A—C6—H6B | 107.8 | H10C—C10A—H10D | 115.3 |
| N1—C7—H7 | 127.3 | C11A—C10A—H10C | 118.1 |
| N1—C7—C8 | 105.5 (2) | C11A—C10A—H10D | 118.1 |
| C8—C7—H7 | 127.3 | C9A—C11A—C10A | 60.9 (7) |
| N3—C8—C7 | 107.2 (2) | C9A—C11A—H11C | 117.7 |
| N3—C8—C9 | 128.4 (3) | C9A—C11A—H11D | 117.7 |
| N3—C8—C9A | 113.7 (4) | C10A—C11A—H11C | 117.7 |
| C7—C8—C9 | 123.5 (4) | C10A—C11A—H11D | 117.7 |
| C7—C8—C9A | 137.3 (4) | H11C—C11A—H11D | 114.8 |
| C9A—C8—C9 | 24.4 (3) | | |

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| O1—C1—C2—O2 | −21.9 (2) | C2—O2—C12—O1 | −11.0 (3) |
| O1—C1—C2—C3 | −136.45 (19) | C2—O2—C12—C13 | −128.3 (3) |
| O2—C2—C3—O3 | 164.93 (18) | C2—O2—C12—C14 | 106.5 (3) |
| O2—C2—C3—C4 | −77.6 (2) | C2—C3—C4—O4 | −38.2 (2) |
| O3—C3—C4—O4 | 77.1 (2) | C2—C3—C4—C5 | −158.00 (19) |
| O3—C3—C4—C5 | −42.7 (3) | C3—C4—C5—O5 | −59.1 (2) |
| O4—C1—C2—O2 | 98.2 (2) | C3—C4—C5—C6 | 178.16 (19) |
| O4—C1—C2—C3 | −16.3 (2) | C4—O4—C1—O1 | 106.0 (2) |
| O4—C4—C5—O5 | −176.68 (17) | C4—O4—C1—C2 | −8.7 (2) |
| O4—C4—C5—C6 | 60.6 (2) | C4—C5—C6—N1 | 50.0 (2) |
| O5—C5—C6—N1 | −69.6 (2) | C6—N1—N2—N3 | 179.2 (2) |
| N1—N2—N3—C8 | −0.2 (3) | C6—N1—C7—C8 | −179.0 (2) |
| N1—C7—C8—N3 | 0.1 (3) | C7—N1—N2—N3 | 0.3 (3) |
| N1—C7—C8—C9 | −170.2 (4) | C7—N1—C6—C5 | 65.6 (3) |
| N1—C7—C8—C9A | 163.2 (6) | C7—C8—C9—C10 | −155.2 (6) |
| N2—N1—C6—C5 | −113.0 (2) | C7—C8—C9—C11 | 137.6 (6) |
| N2—N1—C7—C8 | −0.2 (3) | C7—C8—C9A—C10A | 70.7 (11) |
| N2—N3—C8—C7 | 0.1 (3) | C7—C8—C9A—C11A | 141.4 (9) |
| N2—N3—C8—C9 | 169.7 (4) | C8—C9—C10—C11 | −109.7 (7) |
| N2—N3—C8—C9A | −167.5 (5) | C8—C9—C11—C10 | 104.7 (7) |
| N3—C8—C9—C10 | 36.6 (9) | C8—C9A—C10A—C11A | 116.9 (11) |
| N3—C8—C9—C11 | −30.5 (8) | C8—C9A—C11A—C10A | −100.3 (9) |
| N3—C8—C9A—C10A | −127.0 (8) | C9—C8—C9A—C10A | 5.6 (10) |
| N3—C8—C9A—C11A | −56.2 (10) | C9—C8—C9A—C11A | 76.4 (16) |
| C1—O1—C12—O2 | −4.1 (3) | C12—O1—C1—O4 | −99.3 (2) |
| C1—O1—C12—C13 | 113.5 (2) | C12—O1—C1—C2 | 16.3 (3) |
| C1—O1—C12—C14 | −122.7 (3) | C12—O2—C2—C1 | 20.2 (3) |
| C1—O4—C4—C3 | 30.3 (2) | C12—O2—C2—C3 | 131.0 (2) |

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|---|---|---|---|
| C1—O4—C4—C5 | 155.31 (18) | C9A—C8—C9—C10 | −22.7 (12) |
| C1—C2—C3—O3 | −84.9 (2) | C9A—C8—C9—C11 | −89.8 (16) |
| C1—C2—C3—C4 | 32.6 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···N3$^{i}$ | 0.83 (2) | 1.95 (2) | 2.767 (3) | 171 (3) |
| O5—H5···O3$^{ii}$ | 0.82 (3) | 2.02 (3) | 2.821 (3) | 164 (3) |
| C7—H7···O1$^{iii}$ | 0.93 | 2.59 | 3.496 (3) | 165 |

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*+1/2, −*y*+5/2, −*z*+1; (iii) *x*+1, *y*, *z*.