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A 2′-deoxycytidine long-linker click adduct forming two conformers in the asymmetric unit

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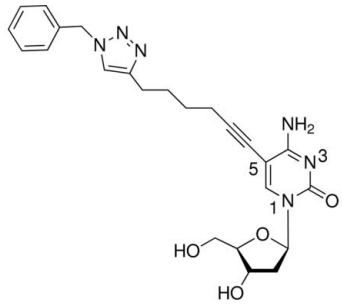
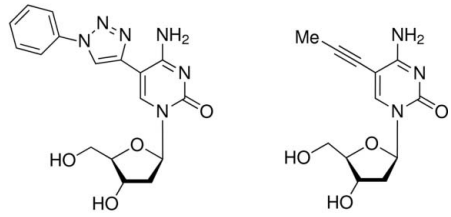
The title compound {systematic name: 4-amino-1-(2-deoxy-*β*-D-*erythro*-pentofuranosyl)-5-[6-(1-benzyl-1*H*-1,2,3-triazol-4-yl)hex-1-ynyl]pyrimidin-2(1*H*)-one}, $C_{24}H_{28}N_6O_4$, shows two conformations in the crystalline state, *viz.* (I-1) and (I-2). The pyrimidine groups and side chains of the two conformers are almost superimposable, while the greatest differences between them are observed for the sugar groups. The N-glycosylic bonds of both conformers adopt similar *anti* conformations, with χ = −168.02 (12)° for conformer (I-1) and χ = −159.08 (12)° for conformer (I-2). The sugar residue of (I-1) shows an *N*-type (C3′-*endo*) conformation, with *P* = 33.1 (2)° and τ_m = 29.5 (1)°, while the conformation of the 2′-deoxyribofuranosyl group of (I-2) is *S*-type (C3′-*exo*), with *P* = 204.5 (2)° and τ_m = 33.8 (1)°. Both conformers participate in hydrogen-bond formation and exhibit identical patterns resulting in three-dimensional networks. Intermolecular hydrogen bonds are formed with neighbouring molecules of different and identical conformations (N—H···N, N—H···O, O—H···N and O—H···O).

Comment

The Cu^I-catalysed Huisgen–Meldal–Sharpless alkyne–azide ‘click’ reaction has emerged as a convenient and effective approach to conjugate two molecules irreversibly under simple reaction conditions (Kolb *et al.*, 2001; Meldal & Tornøe, 2008). This strategy has become particularly attractive for applications in synthetic chemistry (Meldal & Tornøe, 2008), bioconjugation (Wang *et al.*, 2003), drug discovery (Kolb & Sharpless, 2003), molecular diagnostics (Kolb & Sharpless, 2003) and materials science (Moses & Moorhouse, 2007). The ease of click chemistry has inspired researchers to construct a variety of chemically modified nucleosides and oligonucleotide conjugates for medicinal, biological and nanotechnological applications (El-Sagheer & Brown, 2010). Our laboratory and others have reported on the click functionalization of alkynylated 7-deazapurine, 8-aza-7-deazapurine and pyrimidine 2′-deoxyribonucleosides with various reporter groups on the nucleoside and oligonucleotide levels (Gramlich *et al.*, 2008; Seela *et al.*, 2008, 2010). The click chemistry approach has also been extended to the crosslinking of nucleosides and oligonucleotides (Kočalka *et al.*, 2008; Pujari *et al.*, 2010; Xiong & Seela, 2011).



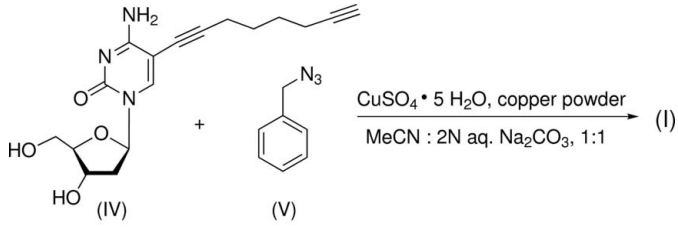
(I)
Systematic numbering



(II) (III)

Scheme 1

Recently, 5-ethynyl-2′-deoxycytidine and phenylazide have been employed as substrates in the click reaction, yielding the click conjugate (II) (see Scheme 1) (Dodd *et al.*, 2010; Andersen *et al.*, 2011), and its solid-state structure was elucidated (Dodd *et al.*, 2010). We used 5-octadiynyl-2′-deoxycytidine, (IV) (Seela *et al.*, 2008), and benzyl azide, (V), as starting materials for the copper(I)-mediated click reaction to afford the title click product 4-amino-1-(2-deoxy-*β*-D-*erythro*-pentofuranosyl)-5-[6-(1-benzyl-1*H*-1,2,3-triazol-4-yl)hex-1-ynyl]pyrimidin-2(1*H*)-one, (I) (see Scheme 2). The synthetic



Scheme 2

procedure for (I) is given in the *Experimental* section. Slow crystallization from hot water gave the click conjugate (I) as colourless crystals. Consequently, we became interested in performing a single-crystal X-ray analysis of (I), which is reported herein. The crystal structure of (I) is compared with

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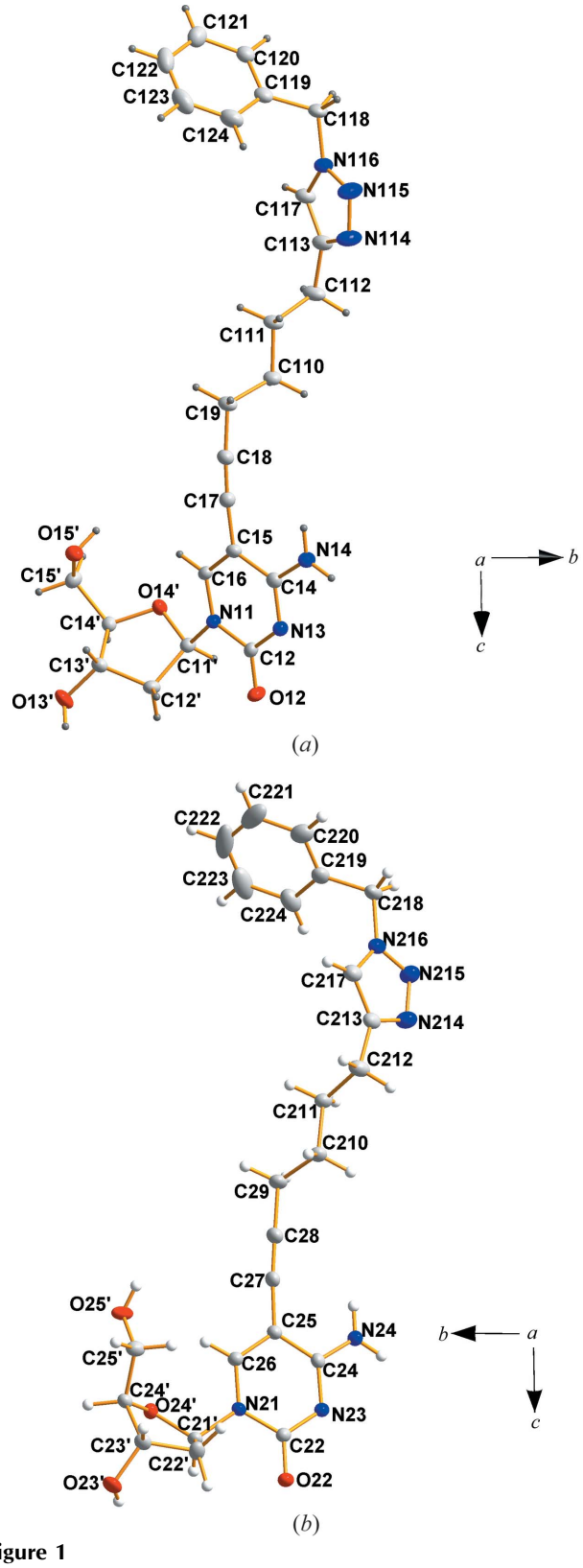

Figure 1
Perspective views of (*a*) conformer (I-1) and (*b*) conformer (I-2), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level.

the two conformers of the click conjugate 5-(1-phenyl-1*H*-1,2,3-triazol-4-yl)-2′-deoxycytidine, (II) (Dodd *et al.*, 2010), and the two conformers of 5-propynyl-2′-deoxycytidine, (III) (Seela *et al.*, 2007).

There are two molecules in the asymmetric unit of (I), denoted (I-1) and (I-2). The three-dimensional structures of conformers (I-1) and (I-2) are shown in Fig. 1, and selected geometric parameters are summarized in Table 1. For the related crystal structures of (II) (Dodd *et al.*, 2010) and (III) (Seela *et al.*, 2007), two conformers were also found in the unit cells. Both nucleoside click conjugates (I) and (II) crystallize in the same space group (monoclinic, $P2_1$) (Dodd *et al.*, 2010), while the space group of (III) is triclinic ($P1$) (Seela *et al.*, 2007).

Fig. 2 shows an overlay of conformers (I-1) and (I-2), indicating that the pyrimidine groups and side chains of the two conformers are almost superimposable, while the greatest differences between them are observed for the sugar groups. Some interesting structural features of the side chains are: (i) the angle between the triazole group and the benzyl ring; (ii) the angle formed by the triple-bonded C7 and C8 atoms with adjacent atom C9; (iii) the angle of inclination of the side chain with respect to the pyrimidine ring plane; (iv) the planarity of the nucleobase. These will be discussed in turn.

The N—C—C angle connecting the methylene group (C118 or C218), the triazole group (N116 or N216) and the phenyl C atom (C119 or C219) is almost identical in both conformers [112.59 (13)° for (I-1) and 112.87 (13)° for (I-2)].

In conformer (I-2), the triple-bonded C27 and C28 atoms, together with adjacent atom C29, form an almost linear entity [C27—C28—C29 = 179.29 (18)°]. For the propynyl groups of conformers (III-1) and (III-2), comparable angles were observed [179.3 (3) and 178.7 (3)°; Seela *et al.*, 2007]. However, for conformer (I-1), this unit is slightly bent [C17—C18—C19 = 173.49 (16)°]. The lengths of the C7—C8 triple bond in the two conformers [1.194 (2) Å for (I-1) and 1.190 (2) Å for (I-2)] are comparable.

The heterocyclic skeletons of (I-1) and (I-2) are nearly planar; the r.m.s. deviations of the ring atoms (N1/C2/N3/C4/C5/C6) from their calculated least-squares planes are 0.0205 and 0.0272 Å, respectively.

The triple-bonded C17 atom of conformer (I-1) almost lies within the pyrimidine ring plane (0.4° inclination), while the triple-bonded C27 atom of conformer (I-2) is slightly displaced from the pyrimidine ring plane (2.7° inclination).

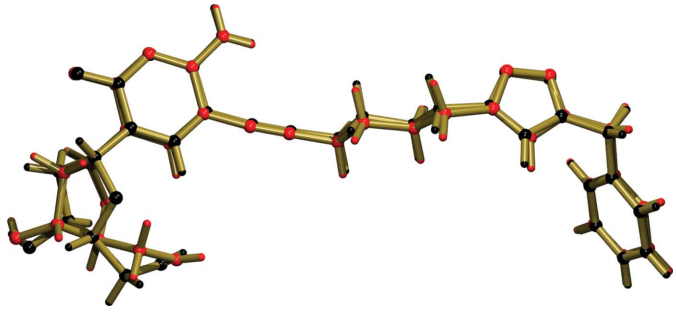

Figure 2
Overlay of conformers (I-1) (darker atoms) and (I-2) (lighter atoms) (black and red, respectively, in the electronic version of the paper).

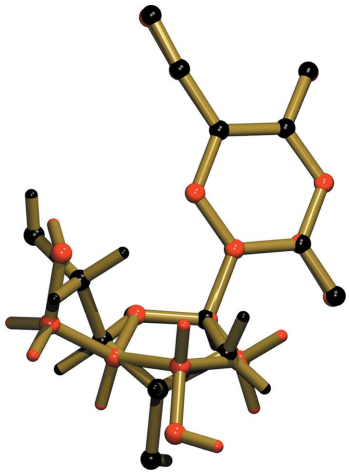

Figure 3
A comparison of the sugar groups of conformers (I-1) and (I-2). The shading is as for Fig. 2.

The orientation of the pyrimidine group relative to the sugar residue (*syn*/*anti*) is defined by the torsion angle χ (O4′—C1′—N1—C2) (IUPAC–IUB Joint Commission on Biochemical Nomenclature, 1983), and usually adopts a conformation in the *anti* range. Indeed, the two conformers of (I) show glycosylic bond torsion angles of $\chi = -168.02\ (12)^\circ$ for (I-1) and $-159.08\ (12)^\circ$ for (I-2), corresponding to *anti* conformations. The conformers of the closely related click compound (II) adopt *anti* conformations within the same range [$\chi = -165.6\ (3)^\circ$ for (II-1) and $-165.2\ (4)^\circ$ for (II-2); Dodd *et al.*, 2010]. A similar torsion angle was also found for conformer (III-2) of 5-propynyl-2′-deoxycytidine, with $\chi = -156.4\ (2)^\circ$, while conformer (III-1) shows a torsion angle of $\chi = -135.0\ (2)^\circ$ around the glycosylic bond (Seela *et al.*, 2007).

The length of the glycosylic N1—C1′ bond is 1.4939 (19) Å for (I-1) and 1.4948 (19) Å for (I-2), which are in the same range as the bond lengths observed for the two conformers of (II) [1.495 (5) Å for (II-1) and 1.484 (5) Å for (II-2); Dodd *et al.*, 2010] and for conformer (III-2) [1.490 (2) Å; Seela *et al.*, 2007], while a shorter glycosylic bond was found for conformer (III-1) [1.475 (2) Å; Seela *et al.*, 2007].

The most pronounced difference between conformers (I-1) and (I-2) is the conformation of the sugar group (Fig. 3). The 2′-deoxyribofuranosyl group of conformer (I-1) shows an *N*-type conformation, with a pseudorotational phase angle $P = 33.1\ (2)^\circ$ and a maximum amplitude $\tau_m = 29.5\ (1)^\circ$, referring to a major C3′-*endo* sugar pucker (C3′-*endo*-C4′-*exo*, ${}^3T_4$). Surprisingly, conformer (I-2) exhibits an *S*-type sugar pucker instead of the *N*-type conformation found for (I-1). The pseudorotational phase angle for (I-2) is $P = 204.5\ (2)^\circ$ and the maximum amplitude is $\tau_m = 33.8\ (1)^\circ$, which corresponds to a major C3′-*exo* sugar pucker (C3′-*exo*-C4′-*endo*, ${}_3T^4$). It is interesting to note that this phenomenon was also observed for the two conformers of the closely related click compound (II). Conformer (II-1) adopts an *S*-type sugar pucker with a major C3′-*exo* conformation [$P = 205.6\ (4)^\circ$, $\tau_m = 37.6\ (3)^\circ$, C3′-*exo*-C4′-*endo*, ${}_3T^4$], while conformer (II-2) shows an *N*-type sugar pucker with a major C3′-*endo* envelope conformation [$P = 18.6\ (4)^\circ$, $\tau_m = 34.7\ (3)^\circ$, ${}^3E$; Dodd *et al.*, 2010]. In contrast, this kind of observation was not made in the case of the two conformers of 5-propynyl-2′-deoxycytidine, (III): for (III-1) and (III-2), similar *S*-type sugar puckers were found (Seela *et al.*, 2007).

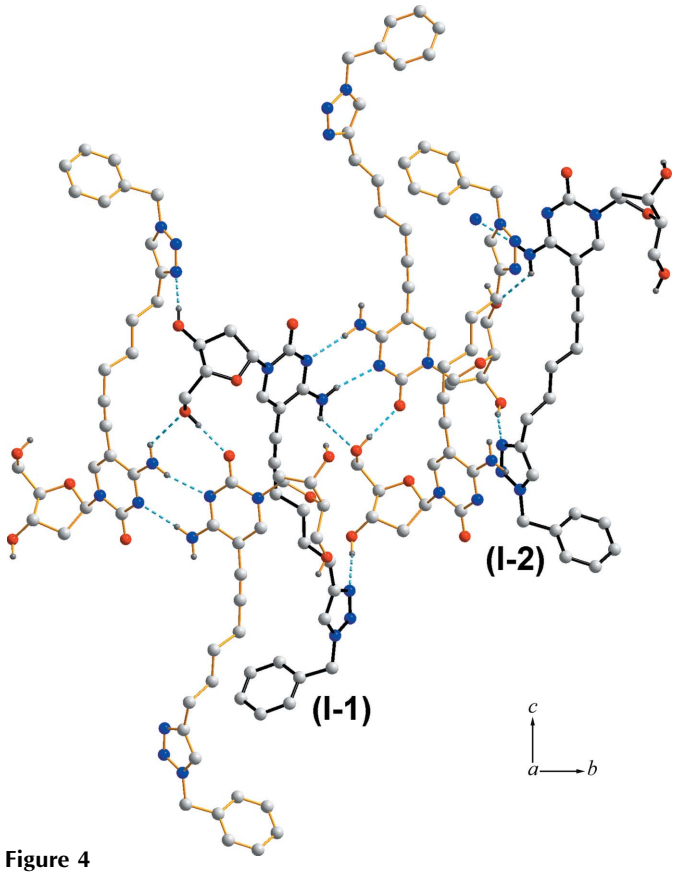

Figure 4
The crystal packing of (I), showing the intermolecular hydrogen-bonding network (parallel to the *bc* plane).

The γ torsion angle (O5′—C5′—C4′—C3′) characterizes the orientation of the exocyclic 5′-hydroxy group relative to the 2′-deoxyribose ring. Conformers (I-1) and (I-2) display different conformations about the C4′—C5′ bond. For (I-1), the torsion angle γ is $60.40\ (17)^\circ$, corresponding to a synclinal (+*sc*; *gauche*, *gauche*) conformation, while in (I-2) the C4′—C5′ bond adopts an antiperiplanar (+*ap*; *gauche*, *trans*) orientation with $\gamma = 174.40\ (12)^\circ$. In the case of click compound (II), conformer (II-2) shows a similar torsion angle with $\gamma = 169.9\ (3)^\circ$ (+*ap*; *gauche*, *trans*), while in conformer (II-1) the C5′-hydroxy group was disordered (Dodd *et al.*, 2010).

In the crystal structure of nucleoside click conjugate (I), conformers (I-1) and (I-2) are linked into an infinite three-dimensional network by several intermolecular hydrogen bonds (Table 2 and Fig. 4). The two conformers exhibit identical hydrogen-bond patterns, and hydrogen bonds are formed with neighbouring molecules of different and identical conformations. The amino group of each conformer acts as a hydrogen-bond donor. Amino group N4—H4*A* of one conformer acts as donor to atom N3 of the pyrimidine group of the other conformer (N14—H14*A*···N23^{i} and N24—H24*A*···N13^{v}; see Table 2 for symmetry codes and geometry). The other amino group, N4—H4*B*, functions as a hydrogen-

bond donor to atom O5′ of the exocyclic sugar hydroxy group of a neighbouring molecule of identical conformation (N14—H14*B*···O15′^{ii} and N24—H24*B*···O25′^{i}). The 5′-hydroxy group is also an H-atom donor, and atom O2 attached to the nucleobase of the other conformer acts as the acceptor site (O15′—H15*C*···O22^{iv} and O25′—H25*C*···O12^{vi}). Apart from the nucleobase and the sugar group, the side chains of the two conformers participitate in hydrogen bonding as well. Atom N14 of the triazole ring functions as a hydrogen-bond acceptor and hydroxy group O3′—H3*C* of the same conformer acts as donor (O13′—H13*C*···N114^{iii} and O23′—H23*C*···N214^{v}).

Experimental

For the synthesis of (I), copper(II) sulfate pentahydrate (7.5% in water; 12.5 mg, 0.05 mmol) and copper powder (32.0 mg, 0.5 mmol) were added to a solution of (IV) (166.5 mg, 0.5 mmol) and benzyl azide, (V) (133 mg, 1.0 mmol), in a mixture of acetonitrile and a 2 *N* solution of aqueous Na_2CO_3 (1:1 *v*/*v*, 10 ml). The reaction mixture was stirred vigorously in the dark at room temperature for 16 h. After completion of the reaction [monitored by thin-layer chromatography (TLC)], the solvent was evaporated under reduced pressure and the residue was applied to a flash chromatography (FC) column (silica gel, column 8 × 3 cm, eluted with CH_2Cl_2/MeOH, 90:10 *v*/*v*). The solvent was evaporated under reduced pressure and the residue was washed with MeOH/H_2O (10:90 *v*/*v*) to afford (I) as a colourless foam (yield 130 mg, 56%). TLC (silica gel, CH_2Cl_2/MeOH, 90:10 *v*/*v*): R_F 0.4; UV (MeOH, λ_{max}, nm): 260 (ε, dm^{-3} mol^{-1} cm^{-1} 160 200), 297.5 (7 400). ^{1}H NMR (300 MHz, DMSO-d_6): δ 1.53–1.60 (*m*, 2H, CH_2), 1.64–1.71 (*m*, 2H, CH_2), 1.95–2.01 (*m*, 1H, H_α—C2′), 2.10–2.14 (*m*, 1H, H_β—C2′), 2.42 (*t*, *J* = 7.2 Hz, 2H, CH_2), 2.63 (*t*, *J* = 7.2 Hz, 2H, CH_2), 3.55–3.60 (*m*, 2H, 2 × H—C5′), 3.76–3.78 (*m*, 1H, H—C4′), 4.17–4.21 (*m*, 1H, H—C3′), 5.10 (*t*, *J* = 5.1 Hz, 1H, HO—C5′), 5.22 (*d*, *J* = 4.2 Hz, 1H, HO—C3′), 5.53 (*s*, 2H, NCH_2), 6.11 (*t*, *J* = 6.6 Hz, 1H, H—C1′), 6.73 (*br s*, 1H, NH), 7.26–7.37 (*m*, 5H, arom. H), 7.67 (*br s*, 1H, NH), 7.90 (*s*, 1H, H5-triazole), 8.08 (*s*, 1H, H—C6). ^{13}C NMR (75.48 MHz, DMSO-d_6): δ 18.8 (CH_2), 24.5 (CH_2), 27.6 (CH_2), 28.3 (CH_2), 40.7 (C2′), 52.7 (CH_2), 61.0 (C5′), 70.1 (C3′), 72.1 (CC), 85.2 (C1′), 87.4 (C4′), 90.4 (CC), 95.4 (C5), 122.0 (triazole CH), 127.8 (arom. C), 128.0 (arom. C), 128.7 (arom. C), 136.3 (triazole C), 143.6 (C6), 147.0 (arom. C), 153.5 (C2), 164.4 (C4). Analysis calculated for $C_{24}H_{28}N_6O_4$: C 62.06, H 6.08, N 18.09%; found: C 61.45, H 5.89, N 17.89%.

Slow crystallization from hot water afforded (I) as colourless crystals (m.p. 446 K). For the diffraction experiment, a single crystal was mounted on a MiTeGen Micro-Mountsfibre in a thin smear of oil.

Crystal data

$C_{24}H_{28}N_6O_4$
M_r = 464.52
Monoclinic, $P2_1$
a = 12.525 (6) Å
b = 15.051 (7) Å
c = 12.719 (6) Å
β = 106.241 (10)°
V = 2302 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 130 K
0.17 × 0.15 × 0.14 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.701, T_{max} = 0.746
131640 measured reflections
11402 independent reflections
10254 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Table 1
Selected geometric parameters (Å, °) for conformers (I-1) and (I-2).

Bond or angle	(I-1), *X* = 1	(I-2), *X* = 2
N*X*1—C*X*1′	1.4939 (19)	1.4948 (19)
C*X*5—C*X*7	1.431 (2)	1.435 (2)
C*X*7—C*X*8	1.194 (2)	1.190 (2)
C*X*12—C*X*13	1.501 (2)	1.504 (2)
N*X*16—C*X*18	1.467 (2)	1.476 (2)
C*X*8—C*X*7—C*X*5	174.89 (16)	174.68 (16)
C*X*7—C*X*8—C*X*9	173.49 (16)	179.29 (18)
N*X*14—N*X*15—N*X*16	106.71 (14)	106.73 (14)
N*X*16—C*X*18—C*X*19	112.59 (13)	112.87 (13)
C*X*11—C*X*12—C*X*13—N*X*14	76.6 (2)	−79.34 (19)
N*X*15—N*X*16—C*X*18—C*X*19	−119.04 (17)	129.99 (17)
C*X*2—N*X*1—C*X*1′—O*X*4′	−168.02 (12)	−159.08 (12)
C*X*3′—C*X*4′—C*X*5′—O*X*5′	60.40 (17)	174.40 (12)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N14—H14*A*···N23^{i}	0.88	2.14	2.978 (2)	158
N14—H14*B*···O15′^{ii}	0.88	2.45	3.1038 (19)	131
O13′—H13*C*···N114^{iii}	0.84	1.98	2.815 (2)	173
O15′—H15*C*···O22^{iv}	0.84	1.95	2.7813 (18)	173
N24—H24*A*···N13^{v}	0.88	2.07	2.944 (2)	173
N24—H24*B*···O25′^{i}	0.88	2.36	2.9839 (19)	128
O23′—H23*C*···N214^{v}	0.84	2.02	2.833 (2)	164
O25′—H25*C*···O12^{vi}	0.84	1.84	2.6483 (17)	161

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

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.093
S = 1.02
11402 reflections
617 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: established by known chemical absolute configuration

The known configuration of the parent molecule was used to define the enantiomer employed in the refined model. In the absence of suitable anomalous scattering, Friedel equivalents could not be used to determine the absolute structure. Refinement of the Flack (1983) parameter led to an inconclusive value for this parameter [−0.3 (5)]. Further confirmation of the configuration was sought using the Hooft analysis. The absolute structure parameter *y* (Hooft *et al.*, 2008) was calculated using *PLATON* (Spek, 2009). The resulting Hooft analysis parameters were *P*2(true) = 1.000, *P*3(true) = 0.987, *P*3(false) = 0.000 and *y* = 0.04 (16) calculated for 5342 Bijvoet pairs (95% coverage), indicating that the known absolute configuration used for the analysis is correct.

All H atoms were found in a difference Fourier synthesis. In order to maximize the data/parameter ratio, H atoms were placed in geometrically idealized positions, with C—H = 0.95 (aromatic), 0.99 (methylene) or 1.00 Å (methine) and N—H = 0.88 Å, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,N)$. The hydroxy groups were refined as groups allowed to rotate but not tip, with O—H = 0.84 Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *PLATON* (Spek, 2009) and *DIAMOND* (Brandenburg, 2004); software used to prepare material for publication: *SHELXTL* and *PLATON*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SF3166). Services for accessing these data are described at the back of the journal.

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A 2′-deoxycytidine long-linker click adduct forming two conformers in the asymmetric unit

Frank Seela, Hai Xiong, Simone Budow, Henning Eickmeier and Hans Reuter

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *DIAMOND* (Brandenburg, 2004); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

4-amino-1-(2-deoxy-*β*-D-*erythro*- pentofuranosyl)-5-[6-(1-benzyl-1*H*-1,2,3-triazol-4-yl)hex-1-ynyl]pyrimidin-2(1*H*)-one

Crystal data

$C_{24}H_{28}N_6O_4$
M_r = 464.52
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 12.525 (6) Å
b = 15.051 (7) Å
c = 12.719 (6) Å
β = 106.241 (10)°
V = 2302 (2) Å^3
Z = 4
F(000) = 984
D_x = 1.340 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9838 reflections
θ = 3.0–27.0°
μ = 0.09 mm^{-1}
T = 130 K
Block, colourless
0.17 × 0.15 × 0.14 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.701, T_{max} = 0.746
131640 measured reflections
11402 independent reflections
10254 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 28.5°, θ_{min} = 2.7°
h = −16→16
k = −20→18
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.093
S = 1.02
11402 reflections
617 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

$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.4036P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Absolute structure: established by known chemical absolute configuration

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}
N11	1.19723 (10)	0.02052 (8)	0.73958 (9)	0.0192 (2)
C12	1.16252 (13)	0.08492 (10)	0.80209 (11)	0.0208 (3)
O12	1.22008 (9)	0.09527 (8)	0.89846 (8)	0.0273 (2)
N13	1.07127 (10)	0.13429 (9)	0.75600 (10)	0.0216 (3)
C14	1.01605 (11)	0.12271 (10)	0.65053 (11)	0.0194 (3)
N14	0.93230 (10)	0.17812 (9)	0.60623 (10)	0.0229 (3)
H14A	0.9151	0.2205	0.6462	0.027*
H14B	0.8944	0.1722	0.5370	0.027*
C15	1.04534 (12)	0.05313 (10)	0.58620 (11)	0.0196 (3)
C16	1.13899 (12)	0.00555 (10)	0.63403 (11)	0.0193 (3)
H16A	1.1637	−0.0386	0.5929	0.023*
C17	0.98271 (12)	0.04093 (10)	0.47435 (12)	0.0214 (3)
C18	0.92644 (12)	0.03687 (10)	0.38166 (12)	0.0225 (3)
C19	0.86530 (13)	0.03996 (11)	0.26513 (12)	0.0254 (3)
H19A	0.7845	0.0425	0.2574	0.030*
H19B	0.8804	−0.0149	0.2288	0.030*
C110	0.89909 (12)	0.12079 (11)	0.20904 (11)	0.0224 (3)
H11A	0.8826	0.1758	0.2443	0.027*
H11B	0.9801	0.1189	0.2177	0.027*
C111	0.83665 (13)	0.12228 (12)	0.08773 (12)	0.0253 (3)
H11C	0.7563	0.1300	0.0798	0.030*
H11D	0.8466	0.0643	0.0550	0.030*
C112	0.87527 (13)	0.19611 (12)	0.02446 (12)	0.0298 (4)
H11E	0.8836	0.2521	0.0668	0.036*
H11F	0.9487	0.1804	0.0148	0.036*
C113	0.79327 (13)	0.20951 (12)	−0.08570 (12)	0.0267 (3)
N114	0.69668 (13)	0.25342 (12)	−0.09505 (11)	0.0376 (4)
N115	0.63685 (12)	0.25435 (12)	−0.19830 (11)	0.0368 (4)
N116	0.69484 (11)	0.21017 (10)	−0.25471 (10)	0.0253 (3)
C117	0.79240 (12)	0.18058 (11)	−0.18774 (12)	0.0253 (3)
H11I	0.8481	0.1470	−0.2074	0.030*

C118	0.65018 (14)	0.19993 (12)	−0.37363 (12)	0.0279 (3)
H11J	0.7021	0.2277	−0.4100	0.033*
H11K	0.5783	0.2317	−0.3983	0.033*
C119	0.63291 (12)	0.10393 (11)	−0.40827 (12)	0.0242 (3)
C120	0.66719 (12)	0.07418 (11)	−0.49726 (12)	0.0251 (3)
H12A	0.7062	0.1135	−0.5319	0.030*
C121	0.64535 (13)	−0.01177 (12)	−0.53606 (15)	0.0312 (4)
H12B	0.6677	−0.0308	−0.5979	0.037*
C122	0.59076 (14)	−0.06999 (12)	−0.48435 (17)	0.0371 (4)
H12C	0.5758	−0.1291	−0.5103	0.045*
C123	0.55833 (15)	−0.04153 (13)	−0.39496 (17)	0.0393 (4)
H12D	0.5219	−0.0818	−0.3589	0.047*
C124	0.57800 (14)	0.04476 (13)	−0.35705 (14)	0.0331 (4)
H12E	0.5541	0.0637	−0.2961	0.040*
C11′	1.30738 (12)	−0.02174 (10)	0.79117 (11)	0.0204 (3)
H11L	1.3640	0.0255	0.8205	0.025*
C12′	1.30546 (13)	−0.08702 (11)	0.88286 (11)	0.0236 (3)
H12F	1.3691	−0.0764	0.9482	0.028*
H12G	1.2355	−0.0814	0.9040	0.028*
C13′	1.31391 (12)	−0.17846 (10)	0.83401 (11)	0.0216 (3)
H13B	1.2381	−0.2038	0.8014	0.026*
O13′	1.38126 (11)	−0.23978 (8)	0.90800 (10)	0.0345 (3)
H13C	1.3633	−0.2397	0.9669	0.052*
C14′	1.36991 (11)	−0.15873 (9)	0.74467 (11)	0.0188 (3)
H14C	1.4523	−0.1597	0.7782	0.023*
O14′	1.33761 (9)	−0.06994 (7)	0.70830 (8)	0.0217 (2)
C15′	1.34184 (12)	−0.22150 (11)	0.64767 (12)	0.0242 (3)
H15A	1.3845	−0.2037	0.5963	0.029*
H15B	1.3657	−0.2823	0.6738	0.029*
O15′	1.22664 (9)	−0.22320 (8)	0.59030 (9)	0.0253 (2)
H15C	1.2130	−0.1839	0.5414	0.038*
N21	1.15939 (10)	0.98960 (8)	1.27399 (9)	0.0192 (2)
C22	1.14665 (12)	0.91193 (10)	1.33046 (11)	0.0193 (3)
O22	1.19943 (9)	0.90592 (7)	1.42849 (8)	0.0238 (2)
N23	1.07767 (10)	0.84732 (8)	1.27555 (10)	0.0207 (2)
C24	1.02777 (11)	0.85573 (10)	1.16816 (11)	0.0187 (3)
N24	0.95577 (11)	0.79325 (9)	1.11906 (10)	0.0234 (3)
H24A	0.9421	0.7480	1.1571	0.028*
H24B	0.9219	0.7972	1.0486	0.028*
C25	1.04833 (12)	0.93082 (10)	1.10604 (11)	0.0197 (3)
C26	1.11298 (12)	0.99686 (10)	1.16438 (11)	0.0201 (3)
H26A	1.1258	1.0490	1.1276	0.024*
C27	0.99763 (13)	0.93556 (10)	0.99007 (12)	0.0222 (3)
C28	0.95145 (13)	0.93315 (11)	0.89467 (12)	0.0252 (3)
C29	0.89363 (14)	0.92914 (11)	0.77695 (12)	0.0278 (3)
H29A	0.9031	0.9868	0.7431	0.033*
H29B	0.8131	0.9206	0.7676	0.033*
C210	0.93524 (12)	0.85453 (11)	0.71602 (12)	0.0229 (3)

H21A	1.0126	0.8670	0.7153	0.028*
H21B	0.9346	0.7973	0.7542	0.028*
C211	0.86056 (12)	0.84814 (11)	0.59896 (12)	0.0227 (3)
H21C	0.8561	0.9075	0.5644	0.027*
H21D	0.7847	0.8317	0.6012	0.027*
C212	0.89938 (13)	0.78100 (12)	0.52732 (12)	0.0279 (3)
H21E	0.9660	0.8044	0.5093	0.033*
H21F	0.9205	0.7249	0.5685	0.033*
C213	0.80968 (13)	0.76259 (11)	0.42318 (12)	0.0240 (3)
N214	0.72444 (12)	0.70628 (11)	0.42188 (11)	0.0337 (3)
N215	0.65498 (13)	0.70303 (11)	0.32241 (12)	0.0349 (3)
N216	0.69496 (12)	0.75791 (9)	0.26042 (10)	0.0284 (3)
C217	0.79071 (14)	0.79621 (11)	0.31940 (13)	0.0285 (3)
H21I	0.8353	0.8376	0.2943	0.034*
C218	0.63533 (16)	0.76621 (12)	0.14307 (13)	0.0342 (4)
H21J	0.5635	0.7344	0.1284	0.041*
H21K	0.6797	0.7373	0.0994	0.041*
C219	0.61358 (13)	0.86145 (11)	0.10654 (13)	0.0269 (3)
C220	0.64184 (15)	0.89096 (14)	0.01480 (14)	0.0367 (4)
H22A	0.6794	0.8527	−0.0225	0.044*
C221	0.61445 (18)	0.97797 (17)	−0.02258 (19)	0.0551 (7)
H22B	0.6322	0.9984	−0.0863	0.066*
C222	0.56172 (19)	1.03401 (15)	0.0332 (2)	0.0620 (8)
H22C	0.5434	1.0929	0.0077	0.074*
C223	0.53583 (17)	1.00482 (15)	0.1251 (2)	0.0534 (6)
H22D	0.5011	1.0441	0.1639	0.064*
C224	0.55993 (14)	0.91844 (13)	0.16177 (16)	0.0361 (4)
H22E	0.5399	0.8981	0.2244	0.043*
C21′	1.22335 (12)	1.06459 (10)	1.33939 (11)	0.0206 (3)
H21L	1.2032	1.0697	1.4099	0.025*
C22′	1.34956 (13)	1.05382 (11)	1.36430 (13)	0.0261 (3)
H22F	1.3687	1.0099	1.3145	0.031*
H22G	1.3827	1.0346	1.4410	0.031*
C23′	1.38992 (12)	1.14726 (11)	1.34498 (12)	0.0228 (3)
H23B	1.4556	1.1423	1.3153	0.027*
O23′	1.41749 (9)	1.20147 (8)	1.43948 (9)	0.0286 (2)
H23C	1.3713	1.1931	1.4753	0.043*
C24′	1.29041 (11)	1.18405 (10)	1.25610 (11)	0.0196 (3)
H24C	1.2873	1.2500	1.2648	0.023*
O24′	1.19385 (8)	1.14436 (7)	1.27905 (8)	0.0196 (2)
C25′	1.29629 (12)	1.16318 (11)	1.14141 (12)	0.0241 (3)
H25A	1.3608	1.1939	1.1273	0.029*
H25B	1.3060	1.0984	1.1337	0.029*
O25′	1.19608 (9)	1.19192 (8)	1.06436 (8)	0.0286 (2)
H25C	1.1898	1.1670	1.0038	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0228 (6)	0.0176 (6)	0.0162 (5)	0.0019 (5)	0.0036 (4)	−0.0007 (4)
C12	0.0275 (7)	0.0163 (7)	0.0179 (6)	−0.0003 (6)	0.0055 (5)	−0.0020 (5)
O12	0.0330 (6)	0.0261 (6)	0.0183 (5)	0.0047 (5)	−0.0003 (4)	−0.0059 (4)
N13	0.0269 (6)	0.0188 (6)	0.0184 (6)	0.0024 (5)	0.0052 (5)	−0.0010 (5)
C14	0.0199 (6)	0.0195 (7)	0.0193 (6)	−0.0014 (5)	0.0061 (5)	0.0016 (5)
N14	0.0241 (6)	0.0241 (7)	0.0191 (6)	0.0043 (5)	0.0039 (5)	−0.0009 (5)
C15	0.0238 (7)	0.0194 (7)	0.0153 (6)	−0.0011 (5)	0.0049 (5)	0.0008 (5)
C16	0.0255 (7)	0.0169 (7)	0.0149 (6)	−0.0014 (5)	0.0047 (5)	−0.0021 (5)
C17	0.0238 (7)	0.0200 (7)	0.0202 (7)	0.0006 (6)	0.0059 (5)	−0.0003 (6)
C18	0.0252 (7)	0.0220 (8)	0.0189 (7)	0.0002 (6)	0.0040 (5)	0.0020 (6)
C19	0.0271 (7)	0.0281 (8)	0.0170 (6)	−0.0032 (6)	−0.0004 (5)	0.0028 (6)
C110	0.0217 (7)	0.0263 (8)	0.0163 (6)	−0.0002 (6)	0.0007 (5)	0.0006 (5)
C111	0.0235 (7)	0.0324 (9)	0.0165 (6)	−0.0023 (6)	−0.0003 (5)	0.0033 (6)
C112	0.0275 (7)	0.0402 (10)	0.0182 (7)	−0.0069 (7)	0.0005 (6)	0.0055 (7)
C113	0.0271 (7)	0.0327 (9)	0.0184 (7)	−0.0048 (7)	0.0031 (6)	0.0029 (6)
N114	0.0373 (8)	0.0523 (10)	0.0200 (6)	0.0081 (7)	0.0027 (6)	−0.0041 (6)
N115	0.0339 (7)	0.0500 (10)	0.0228 (7)	0.0118 (7)	0.0020 (6)	−0.0065 (6)
N116	0.0283 (6)	0.0286 (7)	0.0170 (6)	0.0024 (5)	0.0029 (5)	−0.0009 (5)
C117	0.0244 (7)	0.0317 (9)	0.0179 (7)	0.0010 (6)	0.0029 (5)	0.0037 (6)
C118	0.0334 (8)	0.0299 (9)	0.0154 (6)	0.0027 (7)	−0.0011 (6)	0.0024 (6)
C119	0.0221 (7)	0.0282 (8)	0.0188 (7)	−0.0001 (6)	−0.0003 (5)	0.0045 (6)
C120	0.0231 (7)	0.0277 (8)	0.0222 (7)	0.0014 (6)	0.0026 (6)	0.0034 (6)
C121	0.0269 (8)	0.0296 (9)	0.0343 (8)	0.0059 (7)	0.0038 (7)	−0.0027 (7)
C122	0.0271 (8)	0.0242 (9)	0.0518 (11)	0.0004 (7)	−0.0027 (7)	0.0029 (8)
C123	0.0275 (8)	0.0362 (10)	0.0508 (11)	−0.0056 (7)	0.0052 (8)	0.0176 (9)
C124	0.0295 (8)	0.0429 (11)	0.0266 (8)	−0.0020 (7)	0.0077 (6)	0.0084 (7)
C11′	0.0215 (6)	0.0205 (7)	0.0176 (6)	0.0019 (5)	0.0028 (5)	−0.0018 (5)
C12′	0.0301 (7)	0.0243 (8)	0.0153 (6)	0.0030 (6)	0.0047 (5)	0.0003 (6)
C13′	0.0255 (7)	0.0209 (7)	0.0177 (6)	−0.0006 (6)	0.0049 (5)	0.0027 (5)
O13′	0.0516 (7)	0.0269 (6)	0.0276 (6)	0.0125 (6)	0.0154 (5)	0.0130 (5)
C14′	0.0188 (6)	0.0174 (7)	0.0184 (6)	0.0006 (5)	0.0022 (5)	0.0004 (5)
O14′	0.0283 (5)	0.0179 (5)	0.0203 (5)	0.0041 (4)	0.0089 (4)	0.0023 (4)
C15′	0.0264 (7)	0.0231 (8)	0.0224 (7)	0.0028 (6)	0.0057 (6)	−0.0037 (6)
O15′	0.0274 (5)	0.0248 (6)	0.0203 (5)	−0.0036 (4)	0.0009 (4)	0.0007 (4)
N21	0.0229 (6)	0.0171 (6)	0.0150 (5)	−0.0019 (5)	0.0011 (4)	−0.0018 (4)
C22	0.0234 (7)	0.0177 (7)	0.0162 (6)	0.0018 (5)	0.0045 (5)	0.0000 (5)
O22	0.0315 (5)	0.0204 (5)	0.0160 (5)	−0.0001 (4)	0.0010 (4)	0.0006 (4)
N23	0.0271 (6)	0.0177 (6)	0.0161 (5)	−0.0008 (5)	0.0042 (5)	−0.0002 (5)
C24	0.0213 (6)	0.0158 (7)	0.0186 (6)	0.0009 (5)	0.0047 (5)	−0.0020 (5)
N24	0.0296 (6)	0.0195 (6)	0.0183 (6)	−0.0067 (5)	0.0021 (5)	0.0010 (5)
C25	0.0234 (7)	0.0185 (7)	0.0161 (6)	−0.0011 (5)	0.0036 (5)	−0.0014 (5)
C26	0.0232 (6)	0.0195 (7)	0.0164 (6)	−0.0011 (5)	0.0036 (5)	0.0003 (5)
C27	0.0271 (7)	0.0174 (7)	0.0202 (7)	−0.0046 (6)	0.0035 (5)	−0.0013 (5)
C28	0.0304 (8)	0.0226 (8)	0.0206 (7)	−0.0044 (6)	0.0039 (6)	−0.0017 (6)
C29	0.0349 (8)	0.0267 (8)	0.0167 (7)	0.0005 (7)	−0.0014 (6)	−0.0007 (6)

C210	0.0229 (7)	0.0247 (8)	0.0179 (6)	−0.0004 (6)	0.0003 (5)	0.0001 (6)
C211	0.0248 (7)	0.0244 (8)	0.0166 (6)	0.0026 (6)	0.0021 (5)	0.0001 (6)
C212	0.0259 (7)	0.0337 (9)	0.0214 (7)	0.0050 (6)	0.0022 (6)	−0.0045 (6)
C213	0.0276 (7)	0.0226 (8)	0.0210 (7)	0.0058 (6)	0.0053 (6)	−0.0031 (6)
N214	0.0368 (7)	0.0370 (8)	0.0232 (6)	−0.0024 (7)	0.0015 (5)	0.0039 (6)
N215	0.0396 (8)	0.0334 (8)	0.0255 (7)	−0.0044 (7)	−0.0011 (6)	0.0045 (6)
N216	0.0379 (7)	0.0246 (7)	0.0190 (6)	0.0036 (6)	0.0021 (5)	−0.0013 (5)
C217	0.0346 (8)	0.0272 (8)	0.0229 (7)	0.0011 (7)	0.0068 (6)	−0.0036 (6)
C218	0.0515 (10)	0.0253 (9)	0.0187 (7)	0.0057 (8)	−0.0020 (7)	−0.0032 (6)
C219	0.0268 (7)	0.0261 (8)	0.0219 (7)	0.0008 (6)	−0.0027 (6)	−0.0020 (6)
C220	0.0326 (8)	0.0438 (11)	0.0287 (8)	−0.0056 (8)	0.0002 (7)	0.0023 (8)
C221	0.0465 (12)	0.0545 (15)	0.0483 (12)	−0.0243 (11)	−0.0129 (10)	0.0228 (11)
C222	0.0403 (11)	0.0269 (10)	0.0904 (19)	−0.0060 (9)	−0.0288 (12)	0.0112 (12)
C223	0.0314 (9)	0.0343 (11)	0.0795 (16)	0.0094 (8)	−0.0093 (10)	−0.0150 (11)
C224	0.0287 (8)	0.0336 (10)	0.0416 (10)	0.0020 (7)	0.0026 (7)	−0.0100 (8)
C21′	0.0246 (7)	0.0185 (7)	0.0155 (6)	−0.0016 (5)	0.0002 (5)	−0.0008 (5)
C22′	0.0239 (7)	0.0216 (8)	0.0263 (7)	0.0008 (6)	−0.0036 (6)	0.0021 (6)
C23′	0.0206 (6)	0.0233 (8)	0.0210 (7)	−0.0001 (6)	0.0000 (5)	−0.0018 (6)
O23′	0.0293 (6)	0.0290 (6)	0.0215 (5)	−0.0052 (5)	−0.0025 (4)	−0.0068 (5)
C24′	0.0217 (6)	0.0179 (7)	0.0173 (6)	−0.0010 (5)	0.0024 (5)	−0.0024 (5)
O24′	0.0195 (4)	0.0170 (5)	0.0198 (5)	0.0000 (4)	0.0014 (4)	0.0003 (4)
C25′	0.0239 (7)	0.0285 (8)	0.0189 (6)	0.0041 (6)	0.0047 (5)	−0.0007 (6)
O25′	0.0327 (6)	0.0352 (7)	0.0146 (5)	0.0130 (5)	0.0011 (4)	−0.0013 (4)

Geometric parameters (Å, º)

N11—C16	1.3554 (18)	N21—C26	1.3564 (18)
N11—C12	1.3979 (19)	N21—C22	1.4043 (19)
N11—C11′	1.4939 (19)	N21—C21′	1.4948 (19)
C12—O12	1.2459 (18)	C22—O22	1.2405 (18)
C12—N13	1.352 (2)	C22—N23	1.3573 (19)
N13—C14	1.3376 (19)	N23—C24	1.3397 (19)
C14—N14	1.3351 (19)	C24—N24	1.331 (2)
C14—C15	1.438 (2)	C24—C25	1.443 (2)
N14—H14A	0.8800	N24—H24A	0.8800
N14—H14B	0.8800	N24—H24B	0.8800
C15—C16	1.364 (2)	C25—C26	1.363 (2)
C15—C17	1.431 (2)	C25—C27	1.435 (2)
C16—H16A	0.9500	C26—H26A	0.9500
C17—C18	1.194 (2)	C27—C28	1.190 (2)
C18—C19	1.466 (2)	C28—C29	1.471 (2)
C19—C110	1.529 (2)	C29—C210	1.536 (2)
C19—H19A	0.9900	C29—H29A	0.9900
C19—H19B	0.9900	C29—H29B	0.9900
C110—C111	1.522 (2)	C210—C211	1.524 (2)
C110—H11A	0.9900	C210—H21A	0.9900
C110—H11B	0.9900	C210—H21B	0.9900
C111—C112	1.528 (2)	C211—C212	1.528 (2)

C111—H11C	0.9900	C211—H21C	0.9900
C111—H11D	0.9900	C211—H21D	0.9900
C112—C113	1.501 (2)	C212—C213	1.504 (2)
C112—H11E	0.9900	C212—H21E	0.9900
C112—H11F	0.9900	C212—H21F	0.9900
C113—N114	1.354 (2)	C213—N214	1.360 (2)
C113—C117	1.366 (2)	C213—C217	1.371 (2)
N114—N115	1.318 (2)	N214—N215	1.321 (2)
N115—N116	1.333 (2)	N215—N216	1.333 (2)
N116—C117	1.354 (2)	N216—C217	1.353 (2)
N116—C118	1.467 (2)	N216—C218	1.476 (2)
C117—H11I	0.9500	C217—H21I	0.9500
C118—C119	1.508 (2)	C218—C219	1.508 (3)
C118—H11J	0.9900	C218—H21J	0.9900
C118—H11K	0.9900	C218—H21K	0.9900
C119—C120	1.392 (2)	C219—C220	1.384 (2)
C119—C124	1.393 (2)	C219—C224	1.395 (2)
C120—C121	1.384 (3)	C220—C221	1.403 (3)
C120—H12A	0.9500	C220—H22A	0.9500
C121—C122	1.385 (3)	C221—C222	1.383 (4)
C121—H12B	0.9500	C221—H22B	0.9500
C122—C123	1.378 (3)	C222—C223	1.370 (4)
C122—H12C	0.9500	C222—H22C	0.9500
C123—C124	1.383 (3)	C223—C224	1.386 (3)
C123—H12D	0.9500	C223—H22D	0.9500
C124—H12E	0.9500	C224—H22E	0.9500
C11′—O14′	1.4154 (18)	C21′—O24′	1.4172 (18)
C11′—C12′	1.530 (2)	C21′—C22′	1.531 (2)
C11′—H11L	1.0000	C21′—H21L	1.0000
C12′—C13′	1.526 (2)	C22′—C23′	1.537 (2)
C12′—H12F	0.9900	C22′—H22F	0.9900
C12′—H12G	0.9900	C22′—H22G	0.9900
C13′—O13′	1.4159 (19)	C23′—O23′	1.4133 (19)
C13′—C14′	1.522 (2)	C23′—C24′	1.533 (2)
C13′—H13B	1.0000	C23′—H23B	1.0000
O13′—H13C	0.8400	O23′—H23C	0.8400
C14′—O14′	1.4349 (18)	C24′—O24′	1.4499 (18)
C14′—C15′	1.515 (2)	C24′—C25′	1.514 (2)
C14′—H14C	1.0000	C24′—H24C	1.0000
C15′—O15′	1.4234 (19)	C25′—O25′	1.4265 (18)
C15′—H15A	0.9900	C25′—H25A	0.9900
C15′—H15B	0.9900	C25′—H25B	0.9900
O15′—H15C	0.8400	O25′—H25C	0.8400
C16—N11—C12	120.78 (12)	C26—N21—C22	121.02 (12)
C16—N11—C11′	122.64 (12)	C26—N21—C21′	121.33 (12)
C12—N11—C11′	116.19 (11)	C22—N21—C21′	117.63 (11)
O12—C12—N13	122.98 (13)	O22—C22—N23	123.26 (13)

O12—C12—N11	117.38 (13)	O22—C22—N21	118.03 (13)
N13—C12—N11	119.62 (13)	N23—C22—N21	118.70 (12)
C14—N13—C12	120.03 (12)	C24—N23—C22	120.34 (13)
N14—C14—N13	117.82 (13)	N24—C24—N23	118.24 (13)
N14—C14—C15	120.66 (13)	N24—C24—C25	119.85 (13)
N13—C14—C15	121.52 (13)	N23—C24—C25	121.90 (13)
C14—N14—H14A	120.0	C24—N24—H24A	120.0
C14—N14—H14B	120.0	C24—N24—H24B	120.0
H14A—N14—H14B	120.0	H24A—N24—H24B	120.0
C16—C15—C17	122.68 (13)	C26—C25—C27	123.22 (14)
C16—C15—C14	116.94 (13)	C26—C25—C24	116.29 (13)
C17—C15—C14	120.19 (13)	C27—C25—C24	120.42 (13)
N11—C16—C15	120.80 (13)	N21—C26—C25	121.29 (14)
N11—C16—H16A	119.6	N21—C26—H26A	119.4
C15—C16—H16A	119.6	C25—C26—H26A	119.4
C18—C17—C15	174.89 (16)	C28—C27—C25	174.68 (16)
C17—C18—C19	173.49 (16)	C27—C28—C29	179.29 (18)
C18—C19—C110	111.24 (13)	C28—C29—C210	113.81 (14)
C18—C19—H19A	109.4	C28—C29—H29A	108.8
C110—C19—H19A	109.4	C210—C29—H29A	108.8
C18—C19—H19B	109.4	C28—C29—H29B	108.8
C110—C19—H19B	109.4	C210—C29—H29B	108.8
H19A—C19—H19B	108.0	H29A—C29—H29B	107.7
C111—C110—C19	110.72 (12)	C211—C210—C29	109.69 (12)
C111—C110—H11A	109.5	C211—C210—H21A	109.7
C19—C110—H11A	109.5	C29—C210—H21A	109.7
C111—C110—H11B	109.5	C211—C210—H21B	109.7
C19—C110—H11B	109.5	C29—C210—H21B	109.7
H11A—C110—H11B	108.1	H21A—C210—H21B	108.2
C110—C111—C112	113.32 (13)	C210—C211—C212	114.43 (13)
C110—C111—H11C	108.9	C210—C211—H21C	108.7
C112—C111—H11C	108.9	C212—C211—H21C	108.7
C110—C111—H11D	108.9	C210—C211—H21D	108.7
C112—C111—H11D	108.9	C212—C211—H21D	108.7
H11C—C111—H11D	107.7	H21C—C211—H21D	107.6
C113—C112—C111	110.77 (13)	C213—C212—C211	111.29 (13)
C113—C112—H11E	109.5	C213—C212—H21E	109.4
C111—C112—H11E	109.5	C211—C212—H21E	109.4
C113—C112—H11F	109.5	C213—C212—H21F	109.4
C111—C112—H11F	109.5	C211—C212—H21F	109.4
H11E—C112—H11F	108.1	H21E—C212—H21F	108.0
N114—C113—C117	107.61 (14)	N214—C213—C217	107.12 (14)
N114—C113—C112	120.58 (14)	N214—C213—C212	121.08 (14)
C117—C113—C112	131.72 (16)	C217—C213—C212	131.74 (16)
N115—N114—C113	109.75 (13)	N215—N214—C213	109.84 (14)
N114—N115—N116	106.71 (14)	N214—N215—N216	106.73 (14)
N115—N116—C117	110.93 (13)	N215—N216—C217	110.95 (13)
N115—N116—C118	119.97 (13)	N215—N216—C218	118.89 (14)

C117—N116—C118	129.10 (14)	C217—N216—C218	130.11 (15)
N116—C117—C113	105.00 (14)	N216—C217—C213	105.35 (15)
N116—C117—H11I	127.5	N216—C217—H21I	127.3
C113—C117—H11I	127.5	C213—C217—H21I	127.3
N116—C118—C119	112.59 (13)	N216—C218—C219	112.87 (13)
N116—C118—H11J	109.1	N216—C218—H21J	109.0
C119—C118—H11J	109.1	C219—C218—H21J	109.0
N116—C118—H11K	109.1	N216—C218—H21K	109.0
C119—C118—H11K	109.1	C219—C218—H21K	109.0
H11J—C118—H11K	107.8	H21J—C218—H21K	107.8
C120—C119—C124	118.64 (16)	C220—C219—C224	120.02 (18)
C120—C119—C118	119.61 (14)	C220—C219—C218	119.87 (16)
C124—C119—C118	121.66 (15)	C224—C219—C218	120.03 (16)
C121—C120—C119	121.02 (15)	C219—C220—C221	119.3 (2)
C121—C120—H12A	119.5	C219—C220—H22A	120.3
C119—C120—H12A	119.5	C221—C220—H22A	120.3
C120—C121—C122	119.76 (17)	C222—C221—C220	120.1 (2)
C120—C121—H12B	120.1	C222—C221—H22B	119.9
C122—C121—H12B	120.1	C220—C221—H22B	119.9
C123—C122—C121	119.58 (18)	C223—C222—C221	120.2 (2)
C123—C122—H12C	120.2	C223—C222—H22C	119.9
C121—C122—H12C	120.2	C221—C222—H22C	119.9
C122—C123—C124	120.96 (17)	C222—C223—C224	120.5 (2)
C122—C123—H12D	119.5	C222—C223—H22D	119.8
C124—C123—H12D	119.5	C224—C223—H22D	119.8
C123—C124—C119	120.02 (17)	C223—C224—C219	119.8 (2)
C123—C124—H12E	120.0	C223—C224—H22E	120.1
C119—C124—H12E	120.0	C219—C224—H22E	120.1
O14′—C11′—N11	107.32 (11)	O24′—C21′—N21	108.68 (11)
O14′—C11′—C12′	107.63 (12)	O24′—C21′—C22′	107.42 (12)
N11—C11′—C12′	113.50 (12)	N21—C21′—C22′	113.38 (12)
O14′—C11′—H11L	109.4	O24′—C21′—H21L	109.1
N11—C11′—H11L	109.4	N21—C21′—H21L	109.1
C12′—C11′—H11L	109.4	C22′—C21′—H21L	109.1
C13′—C12′—C11′	104.55 (12)	C21′—C22′—C23′	103.69 (12)
C13′—C12′—H12F	110.8	C21′—C22′—H22F	111.0
C11′—C12′—H12F	110.8	C23′—C22′—H22F	111.0
C13′—C12′—H12G	110.8	C21′—C22′—H22G	111.0
C11′—C12′—H12G	110.8	C23′—C22′—H22G	111.0
H12F—C12′—H12G	108.9	H22F—C22′—H22G	109.0
O13′—C13′—C14′	108.06 (12)	O23′—C23′—C24′	111.93 (13)
O13′—C13′—C12′	114.43 (13)	O23′—C23′—C22′	113.90 (13)
C14′—C13′—C12′	103.11 (12)	C24′—C23′—C22′	102.26 (12)
O13′—C13′—H13B	110.3	O23′—C23′—H23B	109.5
C14′—C13′—H13B	110.3	C24′—C23′—H23B	109.5
C12′—C13′—H13B	110.3	C22′—C23′—H23B	109.5
C13′—O13′—H13C	109.5	C23′—O23′—H23C	109.5
O14′—C14′—C15′	109.76 (12)	O24′—C24′—C25′	112.12 (11)

O14′—C14′—C13′	105.97 (11)	O24′—C24′—C23′	104.76 (12)
C15′—C14′—C13′	115.83 (13)	C25′—C24′—C23′	112.77 (12)
O14′—C14′—H14C	108.4	O24′—C24′—H24C	109.0
C15′—C14′—H14C	108.4	C25′—C24′—H24C	109.0
C13′—C14′—H14C	108.4	C23′—C24′—H24C	109.0
C11′—O14′—C14′	110.32 (11)	C21′—O24′—C24′	110.62 (11)
O15′—C15′—C14′	113.56 (12)	O25′—C25′—C24′	109.18 (12)
O15′—C15′—H15A	108.9	O25′—C25′—H25A	109.8
C14′—C15′—H15A	108.9	C24′—C25′—H25A	109.8
O15′—C15′—H15B	108.9	O25′—C25′—H25B	109.8
C14′—C15′—H15B	108.9	C24′—C25′—H25B	109.8
H15A—C15′—H15B	107.7	H25A—C25′—H25B	108.3
C15′—O15′—H15C	109.5	C25′—O25′—H25C	109.5

C16—N11—C12—O12	−179.89 (13)	C26—N21—C22—O22	173.38 (13)
C11′—N11—C12—O12	−6.95 (19)	C21′—N21—C22—O22	−8.12 (19)
C16—N11—C12—N13	−1.6 (2)	C26—N21—C22—N23	−6.6 (2)
C11′—N11—C12—N13	171.38 (13)	C21′—N21—C22—N23	171.94 (12)
O12—C12—N13—C14	176.84 (14)	O22—C22—N23—C24	−176.29 (13)
N11—C12—N13—C14	−1.4 (2)	N21—C22—N23—C24	3.7 (2)
C12—N13—C14—N14	−174.35 (14)	C22—N23—C24—N24	−176.10 (13)
C12—N13—C14—C15	5.6 (2)	C22—N23—C24—C25	2.6 (2)
N14—C14—C15—C16	173.20 (14)	N24—C24—C25—C26	172.65 (14)
N13—C14—C15—C16	−6.7 (2)	N23—C24—C25—C26	−6.0 (2)
N14—C14—C15—C17	−1.8 (2)	N24—C24—C25—C27	−4.4 (2)
N13—C14—C15—C17	178.29 (13)	N23—C24—C25—C27	176.95 (13)
C12—N11—C16—C15	0.2 (2)	C22—N21—C26—C25	3.0 (2)
C11′—N11—C16—C15	−172.27 (13)	C21′—N21—C26—C25	−175.46 (13)
C17—C15—C16—N11	178.57 (13)	C27—C25—C26—N21	−179.96 (14)
C14—C15—C16—N11	3.7 (2)	C24—C25—C26—N21	3.1 (2)
C18—C19—C110—C111	178.95 (13)	C28—C29—C210—C211	172.54 (13)
C19—C110—C111—C112	−174.08 (14)	C29—C210—C211—C212	175.28 (14)
C110—C111—C112—C113	−165.41 (14)	C210—C211—C212—C213	166.65 (14)
C111—C112—C113—N114	76.6 (2)	C211—C212—C213—N214	−79.34 (19)
C111—C112—C113—C117	−99.3 (2)	C211—C212—C213—C217	97.4 (2)
C117—C113—N114—N115	−1.1 (2)	C217—C213—N214—N215	0.66 (19)
C112—C113—N114—N115	−177.90 (16)	C212—C213—N214—N215	178.13 (14)
C113—N114—N115—N116	0.6 (2)	C213—N214—N215—N216	−0.69 (19)
N114—N115—N116—C117	0.0 (2)	N214—N215—N216—C217	0.47 (19)
N114—N115—N116—C118	−179.63 (15)	N214—N215—N216—C218	178.43 (15)
N115—N116—C117—C113	−0.64 (19)	N215—N216—C217—C213	−0.07 (18)
C118—N116—C117—C113	178.96 (15)	C218—N216—C217—C213	−177.73 (15)
N114—C113—C117—N116	1.01 (19)	N214—C213—C217—N216	−0.35 (17)
C112—C113—C117—N116	177.36 (17)	C212—C213—C217—N216	−177.45 (16)
N115—N116—C118—C119	−119.04 (17)	N215—N216—C218—C219	129.99 (17)
C117—N116—C118—C119	61.4 (2)	C217—N216—C218—C219	−52.5 (2)
N116—C118—C119—C120	−134.96 (15)	N216—C218—C219—C220	129.66 (17)
N116—C118—C119—C124	48.5 (2)	N216—C218—C219—C224	−53.6 (2)

C124—C119—C120—C121	1.4 (2)	C224—C219—C220—C221	−1.0 (2)
C118—C119—C120—C121	−175.27 (14)	C218—C219—C220—C221	175.76 (16)
C119—C120—C121—C122	−1.5 (2)	C219—C220—C221—C222	1.3 (3)
C120—C121—C122—C123	0.3 (2)	C220—C221—C222—C223	−0.1 (3)
C121—C122—C123—C124	1.0 (3)	C221—C222—C223—C224	−1.4 (3)
C122—C123—C124—C119	−1.0 (3)	C222—C223—C224—C219	1.7 (3)
C120—C119—C124—C123	−0.1 (2)	C220—C219—C224—C223	−0.5 (3)
C118—C119—C124—C123	176.44 (15)	C218—C219—C224—C223	−177.23 (16)
C16—N11—C11′—O14′	4.77 (18)	C26—N21—C21′—O24′	19.41 (18)
C12—N11—C11′—O14′	−168.02 (12)	C22—N21—C21′—O24′	−159.08 (12)
C16—N11—C11′—C12′	−114.01 (15)	C26—N21—C21′—C22′	−99.96 (16)
C12—N11—C11′—C12′	73.20 (16)	C22—N21—C21′—C22′	81.54 (15)
O14′—C11′—C12′—C13′	−11.18 (15)	O24′—C21′—C22′—C23′	17.34 (15)
N11—C11′—C12′—C13′	107.42 (13)	N21—C21′—C22′—C23′	137.43 (12)
C11′—C12′—C13′—O13′	141.30 (13)	C21′—C22′—C23′—O23′	90.74 (14)
C11′—C12′—C13′—C14′	24.20 (15)	C21′—C22′—C23′—C24′	−30.22 (15)
O13′—C13′—C14′—O14′	−150.80 (12)	O23′—C23′—C24′—O24′	−89.40 (14)
C12′—C13′—C14′—O14′	−29.29 (14)	C22′—C23′—C24′—O24′	32.91 (14)
O13′—C13′—C14′—C15′	87.26 (15)	O23′—C23′—C24′—C25′	148.40 (13)
C12′—C13′—C14′—C15′	−151.23 (13)	C22′—C23′—C24′—C25′	−89.29 (15)
N11—C11′—O14′—C14′	−130.15 (11)	N21—C21′—O24′—C24′	−119.28 (12)
C12′—C11′—O14′—C14′	−7.65 (15)	C22′—C21′—O24′—C24′	3.75 (14)
C15′—C14′—O14′—C11′	149.31 (12)	C25′—C24′—O24′—C21′	99.14 (13)
C13′—C14′—O14′—C11′	23.56 (14)	C23′—C24′—O24′—C21′	−23.48 (14)
O14′—C14′—C15′—O15′	−59.51 (16)	O24′—C24′—C25′—O25′	56.44 (17)
C13′—C14′—C15′—O15′	60.40 (17)	C23′—C24′—C25′—O25′	174.40 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N14—H14*A*···N23^{i}	0.88	2.14	2.978 (2)	158
N14—H14*B*···O15′^{ii}	0.88	2.45	3.1038 (19)	131
O13′—H13*C*···N114^{iii}	0.84	1.98	2.815 (2)	173
O15′—H15*C*···O22^{iv}	0.84	1.95	2.7813 (18)	173
N24—H24*A*···N13^{v}	0.88	2.07	2.944 (2)	173
N24—H24*B*···O25′^{i}	0.88	2.36	2.9839 (19)	128
O23′—H23*C*···N214^{v}	0.84	2.02	2.833 (2)	164
O25′—H25*C*···O12^{vi}	0.84	1.84	2.6483 (17)	161

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

Selected geometric parameters for conformers (I-1) (X = 1) and (I-2) (X = 2) (Å, °)

NX1—CX1′	1.4939 (19)	1.4948 (19)
CX5—CX7	1.431 (2)	1.435 (2)
CX7—CX8	1.194 (2)	1.190 (2)
CX12—CX13	1.501 (2)	1.504 (2)
NX16—CX18	1.467 (2)	1.476 (2)

CX8—CX7—CX5	174.89 (16)	174.68 (16)
CX7—CX8—CX9	173.49 (16)	179.29 (18)
NX14—NX15—NX16	106.71 (14)	106.73 (14)
NX16—CX18—CX19	112.59 (13)	112.87 (13)
CX11—CX12—CX13—NX14	76.6 (2)	-79.34 (19)
NX15—NX16—CX18—CX19	-119.04 (17)	129.99 (17)
CX2—NX1—CX1′—OX4′	-168.02 (12)	-159.08 (12)
CX3′—CX4′—CX5′—OX5′	60.40 (17)	174.40 (12)