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3-Ethoxy-5-phenyl-1*H*-1,2,4-triazole

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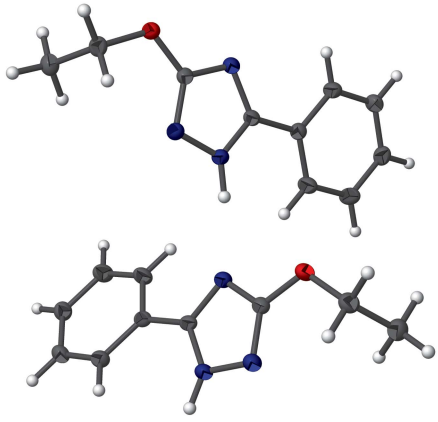
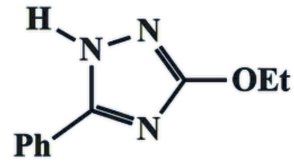
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{10}H_{11}N_3O$, crystallizes in the triclinic space group $P\overline{1}$ with $Z' = 2$. The two independent molecules (*A* and *B*) differ in the orientation of the phenyl rings with respect to the plane of the triazine ring, with an interplanar angle of 11.45 (6)° in molecule *A* and 19.71 (5)° in molecule *B*, in the opposite sense. In the crystal, classical N—H···N hydrogen bonds cross-link the molecules to form chains parallel to the *b* axis. Two additional 'weak' C—H···O hydrogen bonds link the chains to form layers parallel to (101).

3D view



Chemical scheme



Structure description

Cyanoketene *S*,*S*-acetals and cyanoketene *N*,*S*-acetals are important synthetic intermediates (Elgemeie *et al.*, 2015, 2016, 2017, 2018) that have been used as building blocks to assemble a wide range of heterocyclic compounds (Azzam *et al.* 2017*a*,*b*, 2019; Azzam & Elgemeie, 2019); they are also of general interest in medicinal chemistry (Abu-Zaied & Elgemeie, 2017, 2018; Elgemeie *et al.* 2017*c*). Recently, we have reported the synthesis of various antimetabolic agents starting from cyanoketene *N*,*S*-acetals (Elgemeie *et al.* 2006, 2009), cyanoketene *S*,*S*-acetals (Elgemeie *et al.*, 2003*a*, 2017*d*), and cyanoketene *N*,*N*-acetals (Elgemeie *et al.*, 2003*b*). As a part of this programme, the reaction of (*E*)-ethyl 3-benzamido-2-cyano-3-(methylthio)acrylate (**1**) with hydrazine was investigated (Fig. 1). This gave a product whose mass spectrum was not consistent with the proposed pyrazole structure (**3**). Other spectroscopic measurements did not allow us to identify the product unambiguously and therefore the X-ray crystal structure was determined, confirming the exclusive presence of the triazole derivative (**7**) as sole product in the solid state. The formation of (**7**) is assumed to proceed *via* initial addition of the basic N atom of hydrazine to the double bond of (**1**), followed by formation of adduct (**4**) and elimination of ethyl cyanoacetate. From adduct (**4**), the favoured, kinetically and thermodynamically controlled product (**7**) is formed.

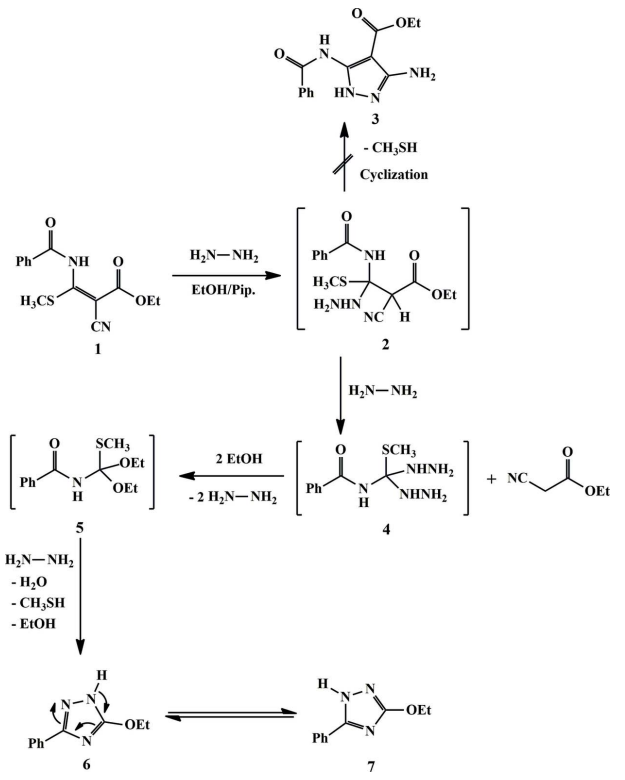


Figure 1
Reaction scheme.

Compound (**7**) crystallizes with two molecules (*A* and *B*) in the asymmetric unit, linked by the hydrogen bond N1—H01···N4′ (Table 1 and Fig. 2). The triazine rings of the two molecules subtend an interplanar angle of 74.75 (4)°. The asymmetric unit was chosen so that the molecules are linked by a hydrogen bond, but the best least-squares fit (r.m.s. deviation 0.057 Å excluding C12, C13, C15, C16) is obtained when one molecule is inverted (Fig. 3). The molecules differ in the orientation of the phenyl ring, whereby the interplanar angle to the triazine ring is 11.45 (6)° in molecule *A* (unprimed atoms) but 19.71 (5)° in molecule *B* (in the opposite sense).

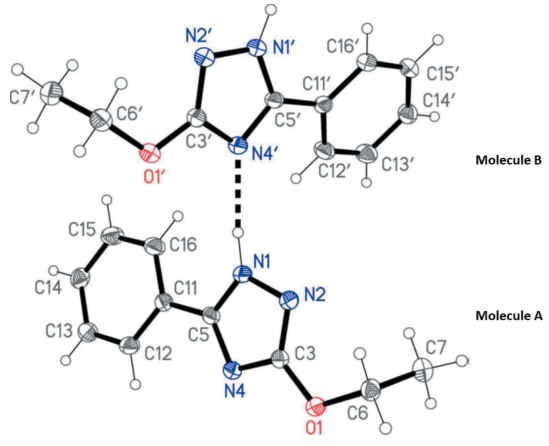


Figure 2
A view of the molecular structures of the two independent molecules of compound (**7**), with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a classical hydrogen bond (Table 1).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H01···N4′	0.94 (2)	1.94 (2)	2.866 (1)	170 (1)
N1′—H01′···N4^{i}	0.90 (2)	2.02 (2)	2.916 (1)	176 (1)
C13—H13···O1′^{ii}	0.95	2.55	3.478 (2)	165
C15′—H15′···N2^{iii}	0.95	2.52	3.463 (2)	172

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

In the crystal, molecules are linked by two classical hydrogen bonds, N1—H01···N4′ (within the asymmetric unit) and N1′—H01···N4 (by *b*-axis translation), to form chains parallel to the *b* axis. Weak intermolecular hydrogen bonds, C13—H13···O1′ and C15′—H15′···N2 (for operators see Table 1) cross-link these chains to form layers parallel to (101) (Table 1 and Fig. 4).

The Cambridge Structural Database (Groom *et al.*, 2016) contains no other example of a 1,2,4-triazine, unsubstituted at N1, with an oxygen substituent at C3. There are eight examples of a 1,2,4-triazine with a phenyl substituent at C5: refcodes DIWZOA (Othman *et al.*, 2014*a*), DOLCAJ (Dolzhenko *et al.*, 2009), HIYTAM (Othman *et al.*, 2014*a*), IBOMAP (two polymorphs of the 3,5-diphenyl derivative; Brough *et al.*, 2011 and Sudheendran *et al.*, 2014), LAGCAX (Carlsen *et al.*, 1991), SISNIS (Buzykin *et al.*, 2006), URELIN (Zhu *et al.*, 2011), XUHBEJ (De Rosa *et al.*, 2014).

Synthesis and crystallization

Hydrazine hydrate (1 mmol) was added to a solution of (*E*)-ethyl 3-benzamido-2-cyano-3-(methylthio)acrylate (**1**) (1 mmol) in ethanol (20 ml) containing a few drops of piperidine. The mixture was heated under reflux with continuous stirring for 2 h, then poured onto ice. The solid product was filtered off, dried and recrystallized from ethanol to afford compound (**7**) as colourless crystals (yield 60%, m.p. 393 K). ^{1}H NMR (400 MHz, DMSO): δ 1.37 (*t*, 3H, CH_3), 4.34 (*q*, 2H, CH_2), 7.47–7.93 (*m*, 5H, Ph), 13.72 (*s*, H, NH-triazole). Analysis: calculated for $C_{10}H_{11}N_3O$ (189.21): C, 63.48; H, 5.86; N, 22.21. Found: C, 63.25; H, 5.62; N, 22.44.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

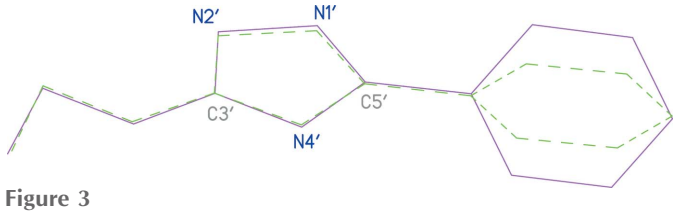


Figure 3
Least-squares fit of all non-hydrogen atoms, except C12, C13, C15 and C16, of inverted molecule *A* (dashed lines) on molecule *B*.

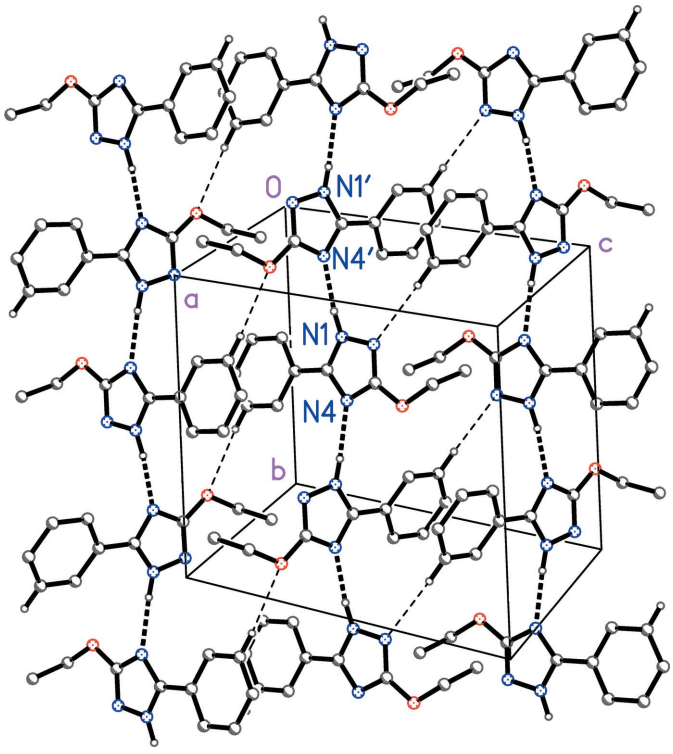


Figure 4
Packing diagram of compound (**7**), viewed perpendicular to plane (101). Classical hydrogen bonds are indicated by thick dashed lines, C—H···*X* interactions by thin dashed lines (see Table 1). Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{11}N_3O$
M_r	189.22
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	8.0664 (4), 10.0476 (5), 12.5229 (5)
α, β, γ (°)	79.554 (4), 81.137 (4), 70.517 (5)
V (Å^3)	936.17 (8)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.74
Crystal size (mm)	0.20 × 0.10 × 0.05
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Atlas Nova
Absorption correction	Multi-scan (*SADABS*; Rigaku OD, 2015)
T_{min}, T_{max}	0.937, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30281, 3894, 3397
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.630
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.090, 1.05
No. of reflections	3894
No. of parameters	263
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.27

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXS97* (Sheldrick, 2008), *SHELXL2017* (Sheldrick, 2015) and *XP* (Siemens, 1994).

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full crystallographic data

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Crystal data

$C_{10}H_{11}N_3O$
M_r = 189.22
Triclinic, $P\overline{1}$
a = 8.0664 (4) Å
b = 10.0476 (5) Å
c = 12.5229 (5) Å
α = 79.554 (4)°
β = 81.137 (4)°
γ = 70.517 (5)°
V = 936.17 (8) Å^3
Z = 4
$F(000)$ = 400
D_x = 1.343 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 16098 reflections
θ = 3.6–76.1°
μ = 0.74 mm^{-1}
T = 100 K
Lath, colourless
0.20 × 0.10 × 0.05 mm

Data collection

Oxford Diffraction Xcalibur Atlas Nova diffractometer
Radiation source: micro-focus sealed X-ray tube
Detector resolution: 10.3543 pixels mm^{-1}
ω–scan
Absorption correction: multi-scan (SADABS; Rigaku OD, 2015)
T_{min} = 0.937, T_{max} = 1.000
30281 measured reflections
3894 independent reflections
3397 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 76.3°, θ_{min} = 3.6°
h = −10→10
k = −12→11
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.090
S = 1.05
3894 reflections
263 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.2414P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.
Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
7.6131 (0.0016) x + 0.4132 (0.0052) y - 0.6162 (0.0069) z = 2.2732 (0.0040)
* 0.0019 (0.0006) N1 * 0.0006 (0.0006) N2 * -0.0028 (0.0006) C3 * 0.0038 (0.0006) N4 * -0.0035 (0.0006) C5
Rms deviation of fitted atoms = 0.0028
6.9734 (0.0022) x - 1.5826 (0.0053) y - 1.2344 (0.0061) z = 0.9885 (0.0034)
Angle to previous plane (with approximate esd) = 11.449 (0.057)
* 0.0008 (0.0008) C11 * -0.0011 (0.0009) C12 * 0.0011 (0.0009) C13 * -0.0008 (0.0009) C14 * 0.0004 (0.0009) C15 * -0.0004 (0.0009) C16
Rms deviation of fitted atoms = 0.0008
3.3871 (0.0042) x + 1.0816 (0.0053) y + 11.8627 (0.0024) z = 4.1527 (0.0012)
Angle to previous plane (with approximate esd) = 77.006 (0.042)
* 0.0001 (0.0006) N1′ * -0.0006 (0.0006) N2′ * 0.0009 (0.0007) C3′ * -0.0008 (0.0006) N4′ * 0.0004 (0.0006) C5′
Rms deviation of fitted atoms = 0.0006
3.7369 (0.0034) x + 4.3701 (0.0047) y + 11.6356 (0.0025) z = 4.0750 (0.0012)
Angle to previous plane (with approximate esd) = 19.712 (0.046)
* -0.0014 (0.0008) C11′ * 0.0001 (0.0008) C12′ * 0.0008 (0.0009) C13′ * -0.0005 (0.0009) C14′ * -0.0008 (0.0009) C15′ * 0.0017 (0.0008) C16′
Rms deviation of fitted atoms = 0.0010

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.30398 (12)	0.33959 (10)	0.29129 (7)	0.0186 (2)
H01	0.309 (2)	0.2538 (18)	0.2700 (13)	0.033 (4)*
N2	0.31227 (12)	0.34480 (10)	0.39915 (7)	0.0189 (2)
C3	0.30430 (14)	0.47855 (12)	0.39607 (9)	0.0173 (2)
N4	0.29269 (12)	0.55939 (10)	0.29610 (7)	0.0184 (2)
C5	0.29156 (14)	0.46660 (12)	0.23169 (9)	0.0176 (2)
C6	0.31423 (15)	0.44416 (12)	0.58656 (9)	0.0205 (2)
H6A	0.367766	0.477794	0.638132	0.025*
H6B	0.390314	0.346689	0.574971	0.025*
C7	0.13224 (15)	0.44054 (13)	0.63501 (9)	0.0245 (2)
H7A	0.055441	0.537604	0.643488	0.037*
H7B	0.140288	0.381170	0.706524	0.037*
H7C	0.082810	0.400500	0.586489	0.037*
C11	0.27486 (14)	0.49708 (12)	0.11405 (9)	0.0191 (2)
C12	0.29281 (16)	0.62379 (13)	0.05453 (9)	0.0236 (2)
H12	0.314085	0.691340	0.090316	0.028*
C13	0.27955 (17)	0.65127 (13)	−0.05738 (10)	0.0259 (3)
H13	0.292318	0.737445	−0.097986	0.031*
C14	0.24777 (16)	0.55318 (13)	−0.10967 (9)	0.0251 (3)
H14	0.238324	0.572415	−0.185999	0.030*
C15	0.22973 (18)	0.42703 (14)	−0.05082 (10)	0.0286 (3)
H15	0.208292	0.359830	−0.086908	0.034*
C16	0.24296 (17)	0.39880 (13)	0.06086 (10)	0.0256 (3)
H16	0.230276	0.312403	0.101083	0.031*

O1	0.30570 (10)	0.53820 (8)	0.48320 (6)	0.01994 (18)
N1′	0.34778 (13)	−0.16318 (10)	0.26565 (8)	0.0196 (2)
H01′	0.325 (2)	−0.2465 (18)	0.2761 (12)	0.030 (4)*
N2′	0.51220 (13)	−0.15849 (10)	0.21821 (8)	0.0208 (2)
C3′	0.49143 (15)	−0.02159 (12)	0.21179 (9)	0.0196 (2)
N4′	0.33042 (13)	0.06147 (10)	0.25005 (7)	0.0197 (2)
C5′	0.24252 (15)	−0.03317 (12)	0.28387 (8)	0.0185 (2)
C6′	0.78367 (15)	−0.05107 (13)	0.12871 (10)	0.0240 (2)
H6′1	0.808590	−0.147414	0.171531	0.029*
H6′2	0.879139	−0.012801	0.135839	0.029*
C7′	0.78071 (18)	−0.06106 (14)	0.01023 (10)	0.0293 (3)
H7′1	0.686086	−0.098850	0.003246	0.044*
H7′2	0.894582	−0.124760	−0.017045	0.044*
H7′3	0.759557	0.033952	−0.032440	0.044*
C11′	0.05810 (15)	−0.00021 (12)	0.33152 (8)	0.0192 (2)
C12′	−0.05513 (16)	0.13969 (12)	0.31547 (10)	0.0238 (2)
H12′	−0.012250	0.212809	0.274291	0.029*
C13′	−0.23009 (16)	0.17204 (13)	0.35957 (10)	0.0263 (3)
H13′	−0.306704	0.267400	0.348484	0.032*
C14′	−0.29414 (16)	0.06579 (13)	0.41993 (10)	0.0248 (3)
H14′	−0.414184	0.088374	0.449939	0.030*
C15′	−0.18179 (16)	−0.07356 (13)	0.43617 (10)	0.0248 (2)
H15′	−0.225243	−0.146391	0.477316	0.030*
C16′	−0.00612 (16)	−0.10682 (12)	0.39246 (9)	0.0227 (2)
H16′	0.070371	−0.202176	0.404035	0.027*
O1′	0.61578 (11)	0.04083 (9)	0.17153 (7)	0.02334 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0236 (5)	0.0149 (5)	0.0175 (4)	−0.0067 (4)	−0.0006 (3)	−0.0030 (3)
N2	0.0221 (4)	0.0173 (5)	0.0173 (4)	−0.0064 (4)	−0.0009 (3)	−0.0029 (3)
C3	0.0169 (5)	0.0172 (5)	0.0179 (5)	−0.0059 (4)	0.0001 (4)	−0.0032 (4)
N4	0.0201 (4)	0.0162 (4)	0.0189 (4)	−0.0065 (4)	−0.0003 (3)	−0.0023 (3)
C5	0.0170 (5)	0.0151 (5)	0.0201 (5)	−0.0053 (4)	0.0003 (4)	−0.0025 (4)
C6	0.0223 (5)	0.0222 (6)	0.0169 (5)	−0.0072 (4)	−0.0023 (4)	−0.0017 (4)
C7	0.0237 (6)	0.0266 (6)	0.0224 (5)	−0.0088 (5)	0.0002 (4)	−0.0018 (4)
C11	0.0196 (5)	0.0177 (5)	0.0191 (5)	−0.0053 (4)	−0.0002 (4)	−0.0028 (4)
C12	0.0315 (6)	0.0178 (5)	0.0221 (5)	−0.0088 (5)	−0.0018 (4)	−0.0030 (4)
C13	0.0345 (6)	0.0185 (6)	0.0225 (6)	−0.0083 (5)	−0.0006 (5)	0.0008 (4)
C14	0.0310 (6)	0.0242 (6)	0.0180 (5)	−0.0065 (5)	−0.0024 (4)	−0.0016 (4)
C15	0.0426 (7)	0.0235 (6)	0.0232 (6)	−0.0133 (5)	−0.0065 (5)	−0.0040 (5)
C16	0.0364 (6)	0.0206 (6)	0.0224 (6)	−0.0132 (5)	−0.0038 (5)	−0.0008 (4)
O1	0.0254 (4)	0.0186 (4)	0.0173 (4)	−0.0088 (3)	−0.0012 (3)	−0.0034 (3)
N1′	0.0228 (5)	0.0154 (5)	0.0210 (4)	−0.0077 (4)	0.0003 (3)	−0.0028 (3)
N2′	0.0231 (5)	0.0182 (5)	0.0216 (4)	−0.0083 (4)	0.0004 (4)	−0.0030 (4)
C3′	0.0246 (5)	0.0181 (5)	0.0176 (5)	−0.0091 (4)	−0.0010 (4)	−0.0025 (4)
N4′	0.0249 (5)	0.0160 (5)	0.0188 (4)	−0.0079 (4)	−0.0006 (3)	−0.0026 (3)

C5′	0.0257 (5)	0.0145 (5)	0.0159 (5)	−0.0070 (4)	−0.0030 (4)	−0.0016 (4)
C6′	0.0232 (5)	0.0223 (6)	0.0267 (6)	−0.0088 (5)	0.0002 (4)	−0.0033 (4)
C7′	0.0347 (6)	0.0254 (6)	0.0260 (6)	−0.0094 (5)	0.0032 (5)	−0.0043 (5)
C11′	0.0239 (5)	0.0176 (5)	0.0170 (5)	−0.0074 (4)	−0.0026 (4)	−0.0030 (4)
C12′	0.0269 (6)	0.0173 (6)	0.0267 (6)	−0.0081 (5)	−0.0023 (4)	0.0000 (4)
C13′	0.0256 (6)	0.0186 (6)	0.0321 (6)	−0.0045 (5)	−0.0026 (5)	−0.0016 (5)
C14′	0.0231 (5)	0.0244 (6)	0.0257 (6)	−0.0073 (5)	0.0000 (4)	−0.0031 (5)
C15′	0.0291 (6)	0.0205 (6)	0.0247 (6)	−0.0107 (5)	0.0014 (4)	−0.0008 (4)
C16′	0.0273 (6)	0.0164 (5)	0.0232 (5)	−0.0063 (4)	−0.0011 (4)	−0.0017 (4)
O1′	0.0261 (4)	0.0192 (4)	0.0264 (4)	−0.0113 (3)	0.0036 (3)	−0.0049 (3)

Geometric parameters (Å, º)

N1—C5	1.3372 (14)	N1′—C5′	1.3372 (14)
N1—N2	1.3739 (13)	N1′—N2′	1.3795 (13)
N1—H01	0.935 (17)	N1′—H01′	0.898 (17)
N2—C3	1.3176 (15)	N2′—C3′	1.3179 (15)
C3—O1	1.3399 (13)	C3′—O1′	1.3428 (14)
C3—N4	1.3610 (14)	C3′—N4′	1.3576 (15)
N4—C5	1.3418 (14)	N4′—C5′	1.3407 (15)
C5—C11	1.4670 (15)	C5′—C11′	1.4656 (15)
C6—O1	1.4537 (13)	C6′—O1′	1.4477 (14)
C6—C7	1.5092 (15)	C6′—C7′	1.5095 (17)
C6—H6A	0.9900	C6′—H6′1	0.9900
C6—H6B	0.9900	C6′—H6′2	0.9900
C7—H7A	0.9800	C7′—H7′1	0.9800
C7—H7B	0.9800	C7′—H7′2	0.9800
C7—H7C	0.9800	C7′—H7′3	0.9800
C11—C12	1.3940 (16)	C11′—C12′	1.3958 (16)
C11—C16	1.3949 (17)	C11′—C16′	1.3964 (16)
C12—C13	1.3921 (16)	C12′—C13′	1.3861 (17)
C12—H12	0.9500	C12′—H12′	0.9500
C13—C14	1.3857 (18)	C13′—C14′	1.3903 (17)
C13—H13	0.9500	C13′—H13′	0.9500
C14—C15	1.3864 (17)	C14′—C15′	1.3893 (17)
C14—H14	0.9500	C14′—H14′	0.9500
C15—C16	1.3892 (16)	C15′—C16′	1.3896 (17)
C15—H15	0.9500	C15′—H15′	0.9500
C16—H16	0.9500	C16′—H16′	0.9500
C5—N1—N2	110.54 (9)	C5′—N1′—N2′	110.63 (9)
C5—N1—H01	130.1 (10)	C5′—N1′—H01′	130.4 (10)
N2—N1—H01	119.4 (10)	N2′—N1′—H01′	118.8 (10)
C3—N2—N1	101.40 (9)	C3′—N2′—N1′	100.98 (9)
N2—C3—O1	124.72 (10)	N2′—C3′—O1′	125.44 (10)
N2—C3—N4	116.09 (10)	N2′—C3′—N4′	116.40 (10)
O1—C3—N4	119.19 (10)	O1′—C3′—N4′	118.16 (10)
C5—N4—C3	102.22 (9)	C5′—N4′—C3′	102.35 (9)

N1—C5—N4	109.75 (9)	N1′—C5′—N4′	109.64 (10)
N1—C5—C11	123.83 (10)	N1′—C5′—C11′	124.66 (10)
N4—C5—C11	126.40 (10)	N4′—C5′—C11′	125.69 (10)
O1—C6—C7	110.87 (9)	O1′—C6′—C7′	110.59 (10)
O1—C6—H6A	109.5	O1′—C6′—H6′1	109.5
C7—C6—H6A	109.5	C7′—C6′—H6′1	109.5
O1—C6—H6B	109.5	O1′—C6′—H6′2	109.5
C7—C6—H6B	109.5	C7′—C6′—H6′2	109.5
H6A—C6—H6B	108.1	H6′1—C6′—H6′2	108.1
C6—C7—H7A	109.5	C6′—C7′—H7′1	109.5
C6—C7—H7B	109.5	C6′—C7′—H7′2	109.5
H7A—C7—H7B	109.5	H7′1—C7′—H7′2	109.5
C6—C7—H7C	109.5	C6′—C7′—H7′3	109.5
H7A—C7—H7C	109.5	H7′1—C7′—H7′3	109.5
H7B—C7—H7C	109.5	H7′2—C7′—H7′3	109.5
C12—C11—C16	119.61 (10)	C12′—C11′—C16′	119.52 (11)
C12—C11—C5	120.15 (10)	C12′—C11′—C5′	119.52 (10)
C16—C11—C5	120.24 (10)	C16′—C11′—C5′	120.96 (10)
C13—C12—C11	119.94 (11)	C13′—C12′—C11′	120.06 (11)
C13—C12—H12	120.0	C13′—C12′—H12′	120.0
C11—C12—H12	120.0	C11′—C12′—H12′	120.0
C14—C13—C12	120.13 (11)	C12′—C13′—C14′	120.41 (11)
C14—C13—H13	119.9	C12′—C13′—H13′	119.8
C12—C13—H13	119.9	C14′—C13′—H13′	119.8
C13—C14—C15	120.14 (11)	C15′—C14′—C13′	119.69 (11)
C13—C14—H14	119.9	C15′—C14′—H14′	120.2
C15—C14—H14	119.9	C13′—C14′—H14′	120.2
C14—C15—C16	120.06 (12)	C14′—C15′—C16′	120.25 (11)
C14—C15—H15	120.0	C14′—C15′—H15′	119.9
C16—C15—H15	120.0	C16′—C15′—H15′	119.9
C15—C16—C11	120.12 (11)	C15′—C16′—C11′	120.06 (11)
C15—C16—H16	119.9	C15′—C16′—H16′	120.0
C11—C16—H16	119.9	C11′—C16′—H16′	120.0
C3—O1—C6	115.05 (9)	C3′—O1′—C6′	116.23 (9)
C5—N1—N2—C3	−0.12 (11)	C5′—N1′—N2′—C3′	0.07 (12)
N1—N2—C3—O1	179.17 (10)	N1′—N2′—C3′—O1′	179.42 (10)
N1—N2—C3—N4	−0.36 (12)	N1′—N2′—C3′—N4′	−0.15 (12)
N2—C3—N4—C5	0.68 (12)	N2′—C3′—N4′—C5′	0.17 (13)
O1—C3—N4—C5	−178.88 (9)	O1′—C3′—N4′—C5′	−179.43 (10)
N2—N1—C5—N4	0.55 (12)	N2′—N1′—C5′—N4′	0.03 (12)
N2—N1—C5—C11	−178.03 (9)	N2′—N1′—C5′—C11′	178.84 (10)
C3—N4—C5—N1	−0.70 (12)	C3′—N4′—C5′—N1′	−0.11 (12)
C3—N4—C5—C11	177.83 (10)	C3′—N4′—C5′—C11′	−178.90 (10)
N1—C5—C11—C12	−169.05 (11)	N1′—C5′—C11′—C12′	−159.54 (11)
N4—C5—C11—C12	12.60 (17)	N4′—C5′—C11′—C12′	19.07 (17)
N1—C5—C11—C16	10.20 (17)	N1′—C5′—C11′—C16′	20.26 (17)
N4—C5—C11—C16	−168.14 (11)	N4′—C5′—C11′—C16′	−161.13 (11)

C16—C11—C12—C13	−0.25 (18)	C16′—C11′—C12′—C13′	−0.19 (17)
C5—C11—C12—C13	179.02 (10)	C5′—C11′—C12′—C13′	179.61 (11)
C11—C12—C13—C14	0.28 (18)	C11′—C12′—C13′—C14′	−0.02 (19)
C12—C13—C14—C15	−0.25 (19)	C12′—C13′—C14′—C15′	0.09 (19)
C13—C14—C15—C16	0.2 (2)	C13′—C14′—C15′—C16′	0.07 (18)
C14—C15—C16—C11	−0.2 (2)	C14′—C15′—C16′—C11′	−0.28 (18)
C12—C11—C16—C15	0.18 (18)	C12′—C11′—C16′—C15′	0.34 (17)
C5—C11—C16—C15	−179.08 (11)	C5′—C11′—C16′—C15′	−179.46 (10)
N2—C3—O1—C6	−0.91 (15)	N2′—C3′—O1′—C6′	2.20 (16)
N4—C3—O1—C6	178.61 (9)	N4′—C3′—O1′—C6′	−178.24 (9)
C7—C6—O1—C3	−84.57 (11)	C7′—C6′—O1′—C3′	87.32 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H01···N4′	0.94 (2)	1.94 (2)	2.866 (1)	170 (1)
N1′—H01′···N4^{i}	0.90 (2)	2.02 (2)	2.916 (1)	176 (1)
C13—H13···O1′^{ii}	0.95	2.55	3.478 (2)	165
C15′—H15′···N2^{iii}	0.95	2.52	3.463 (2)	172

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