

# 4-[(4-Hydroxymethyl-2H-1,2,3-triazol-2-yl)methyl]-6-isopropyl-2H-chromen-2-one

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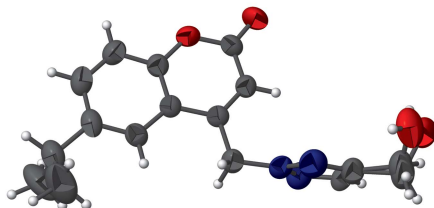
Keywords: crystal structure; chromones; triazole; conformation.

CCDC reference: 1523344

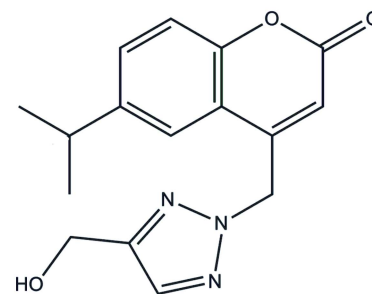
Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

In the title compound, C<sub>16</sub>H<sub>17</sub>N<sub>3</sub>O<sub>3</sub>, the chromene ring is planar, with a maximum deviation of 0.017 (4) Å for the ring O atom. The triazole and the chromene rings, bridged by a methylene C atom, are inclined to one another by 78.3 (2)°. In the crystal, methylene–triazole C–H···N hydrogen bonds lead to the formation of helical supramolecular chains along the *b* axis. The sample was refined as an inversion twin. The terminal methylhydroxy group is disordered over two sets of sites [site occupancy = 0.610 (13) for the major component].

## 3D view



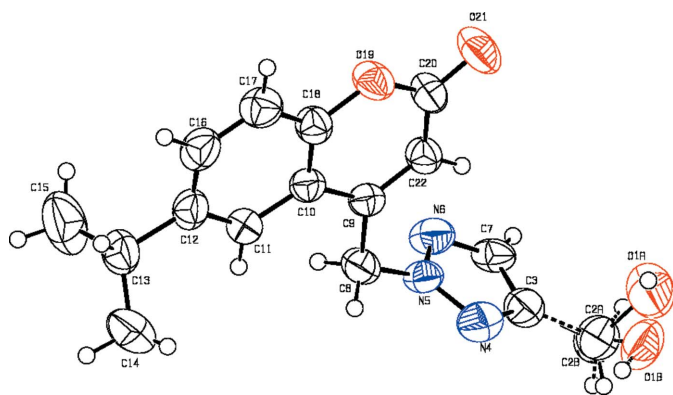
## Chemical scheme



## Structure description

Coumarins and their derivatives form represent an important class of natural and synthetic heterocycles that are often linked to a broad array of biological activities (Gaspar *et al.*, 2015), such as anti-bacterial (Basanagouda *et al.*, 2009), anti-oxidant (Vukovic *et al.*, 2010) and anti-inflammatory (Emmanuel-Giota *et al.*, 2001). These derivatives are also used in the pharmaceutical industry as precursor reagents in the synthesis of a number of synthetic anti-coagulant pharmaceuticals (Bairagi *et al.*, 2012). As part of our ongoing studies of coumarin–triazole derivatives (El-Khatatneh *et al.*, 2016), the title compound was synthesized and its crystal structure is reported herein.

In the molecular structure (Fig. 1), the chromene unit (O19/C9/C10/C18/C20/C22) is planar, with a maximum deviation of 0.017 (4) Å for the ring atom O21. The triazole (N4/N5/N6/C3/C7) and the chromene (O19/C9/C10/C18/C20/C22) rings, bridged *via* a methylene–C8 atom, are inclined to one another by 78.3 (2)°. The intra-ring bond conformation between the chromene and triazole moieties are also characterized by torsion angles of 100.2 (5)° [for N4–N5–C8–C9] and –178.6 (3)° [for C10–C9–C8–N5]. The hydroxymethyl group is not coplanar with the triazole ring, as indicated by



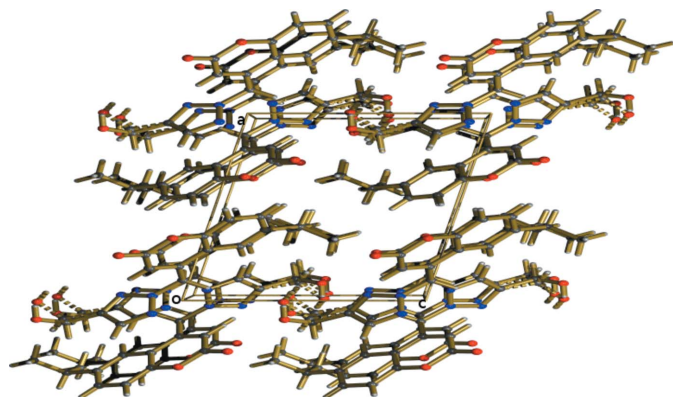
**Figure 1**  
Perspective diagram of the title molecule, showing 50% probability displacement ellipsoids and both components of the disordered hydroxymethyl group.

torsion angle  $C7-C3-C2A-O1A = 77(2)^\circ$ . One methyl unit of the isopropyl group is not co-planar with the chromene ring, as suggested by the  $C11-C12-C13-C14$  torsion angle of  $40.5(8)^\circ$ , while the other methyl group is below, with a  $C11-C12-C13-C15$  torsion angle of  $-88.1(6)^\circ$ .

The crystal features  $C8-H8A \cdots N6$  hydrogen bonds (Table 1), which lead to helical supramolecular chains along the  $b$  axis. The molecular packing exhibits layered stacking when viewed along the  $b$  axis, as shown in Fig. 2.

### Synthesis and crystallization

The general procedure for the synthesis of  $N_2$  coumarin 1,2,3-triazoles has been reported (Shamala *et al.*, 2016). To a solution of propargyl alcohol (0.11 g, 1.9 mmol) in acetone, CuI (10 mol%) and triethylamine (0.19 g, 1.9 mmol) were added. The mixture was stirred at room temperature for 15 min. Then, 4-(azidomethyl)-6-isopropyl-2*H*-chromen-2-one (1.9 mmol) was added and the resulting mixture was stirred until the starting material was consumed as judged by TLC. After the completion of the reaction, the catalyst was filtered through celite and the product was extracted with ether (3.10 ml). The solvent was removed under vacuum. The crude



**Figure 2**  
Packing diagram of the molecule viewed parallel to the  $b$  axis.

**Table 1**  
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C8-H8A \cdots N6^i$	0.97	2.55	3.070 (6)	114

Symmetry code: (i)  $-x, y - \frac{1}{2}, -z$ .

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{17}N_3O_3$
$M_r$	299.32
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	273
$a, b, c$ ( $\text{\AA}$ )	8.715 (5), 7.301 (4), 12.113 (6)
$\beta$ ( $^\circ$ )	101.209 (9)
$V$ ( $\text{\AA}^3$ )	756.0 (7)
$Z$	2
Radiation type	Mo $K\alpha$
$\mu$ ( $\text{mm}^{-1}$ )	0.09
Crystal size (mm)	$0.35 \times 0.25 \times 0.15$
Data collection	
Diffractometer	Bruker MicroStar microfocus rotating anode
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	7069, 2680, 2307
$R_{int}$	0.026
$(\sin \theta/\lambda)_{max}$ ( $\text{\AA}^{-1}$ )	0.594
Refinement	
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.058, 0.173, 1.06
No. of reflections	2680
No. of parameters	214
No. of restraints	38
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$ , $\Delta\rho_{min}$ ( $e \text{\AA}^{-3}$ )	0.25, -0.28
Absolute structure	Flack $x$ determined using 884 quotients $[(I^-)-(I^+)]/[(I^-)+(I^+)]$ (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.4 (6)

Computer programs: APEX2 and SAINT (Bruker, 2009), SHELXS97 (Sheldrick, 2008), Mercury (Macrae *et al.*, 2008), SHELXL2016 (Sheldrick, 2015) and PLATON (Spek, 2009).

product was dried and recrystallized from ethyl acetate to give colourless blocks of the title compound.

Yield 85%; colourless solid; m.p. 466–468 K. IR (KBr,  $\text{cm}^{-1}$ ): 1720 (lactone  $C=O$ ), 3190 (OH).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.25 (*d*, 6H, 2- $\text{CH}_3$  of *i*-Pr,  $J = 5.2$  Hz), 2.06 (*s*, 1H, OH), 2.95 (*m*, 1H, CH of *i*-Pr,  $J = 5.2$  Hz), 4.93 (*s*, 2H,  $-\text{CH}_2\text{O}-$ ), 5.97 (*s*, 2H,  $-\text{CH}_2\text{N}-$ ), 6.21 (*s*, 1H,  $C_3-H$ ), 7.36 (*d*, 1H,  $C_7-H$ ,  $J = 8.4$  Hz), 7.55 (*d*, 1H,  $C_8-H$ ,  $J_{1,2} = 7.6$  Hz), 7.70 (*s*, 1H,  $C_5-H$ ), 8.38 (*s*, 1H, Tr-H) p.p.m.  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  16.9 (2C), 26.7, 43.3, 49.5, 101.1, 107.9, 109.6, 110.4, 113.7, 124.2, 138.7, 141.0, 142.9, 145.0, 153.0 p.p.m. Analysis calculated for  $C_{16}H_{17}N_3O_3$ : C, 64.20; H, 5.72; N, 14.04%; found: C, 64.08; H, 5.60; N, 14.00%.

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The sample was refined as an inversion twin. The terminal methylhydroxyl group is disor-

dered over two sets of sites (site occupancy = 0.610 (13) for the major component).

### Acknowledgements

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## full crystallographic data

*IUCrData* (2016). **1**, x161989 [https://doi.org/10.1107/S2414314616019891]

## 4-[(4-Hydroxymethyl-2*H*-1,2,3-triazol-2-yl)methyl]-6-isopropyl-2*H*-chromen-2-one

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### 4-[(4-Hydroxymethyl-2*H*-1,2,3-triazol-2-yl)methyl]-6-isopropyl-2*H*-chromen-2-one

#### Crystal data

$C_{16}H_{17}N_3O_3$

$M_r = 299.32$

Monoclinic,  $P2_1$

$a = 8.715$  (5) Å

$b = 7.301$  (4) Å

$c = 12.113$  (6) Å

$\beta = 101.209$  (9)°

$V = 756.0$  (7) Å<sup>3</sup>

$Z = 2$

$F(000) = 316$

$D_x = 1.315$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2680 reflections

$\theta = 1.7$ – $25.0$ °

$\mu = 0.09$  mm<sup>-1</sup>

$T = 273$  K

Block, colourless

$0.35 \times 0.25 \times 0.15$  mm

#### Data collection

Bruker MicroStar microfocus rotating anode diffractometer

Detector resolution: 18.4 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

7069 measured reflections

2680 independent reflections

2307 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\text{max}} = 25.0$ °,  $\theta_{\text{min}} = 1.7$ °

$h = -10 \rightarrow 10$

$k = -8 \rightarrow 8$

$l = -14 \rightarrow 14$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.058$

$wR(F^2) = 0.173$

$S = 1.06$

2680 reflections

214 parameters

38 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.1139P)^2 + 0.0997P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.28$  e Å<sup>-3</sup>

Absolute structure: Flack  $x$  determined using

884 quotients  $[(I^+)-(I^-)]/[(I^+)+(I^-)]$  (Parsons *et al.*, 2013)

Absolute structure parameter: 0.4 (6)

#### 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.

**Refinement.** Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C2A	0.432 (4)	−0.285 (8)	−0.109 (3)	0.080 (3)	0.610 (13)
H2A1	0.474773	−0.398344	−0.075005	0.096*	0.610 (13)
H2A2	0.399222	−0.304324	−0.189741	0.096*	0.610 (13)
O1A	0.5459 (9)	−0.1448 (13)	−0.0887 (7)	0.109 (4)	0.610 (13)
H1A	0.567622	−0.123296	−0.021052	0.164*	0.610 (13)
C2B	0.445 (6)	−0.277 (14)	−0.090 (6)	0.080 (3)	0.390 (13)
H2B1	0.430161	−0.396496	−0.126013	0.096*	0.390 (13)
H2B2	0.467401	−0.191108	−0.145752	0.096*	0.390 (13)
O1B	0.5778 (11)	−0.2871 (17)	−0.0017 (10)	0.089 (4)	0.390 (13)
H1B	0.560748	−0.359343	0.046267	0.133*	0.390 (13)
O19	0.0897 (4)	0.2890 (4)	0.3404 (2)	0.0600 (8)	
C11	−0.1689 (4)	−0.1100 (6)	0.2962 (4)	0.0508 (10)	
H11	−0.191640	−0.210542	0.248665	0.061*	
C9	0.0411 (4)	−0.0058 (5)	0.1920 (3)	0.0446 (9)	
C10	−0.0518 (5)	0.0111 (5)	0.2791 (3)	0.0453 (9)	
C18	−0.0219 (5)	0.1603 (6)	0.3514 (4)	0.0509 (10)	
C20	0.1754 (5)	0.2769 (6)	0.2573 (4)	0.0550 (11)	
C22	0.1485 (5)	0.1217 (6)	0.1844 (3)	0.0488 (10)	
H22	0.208492	0.108718	0.129199	0.059*	
C17	−0.1015 (6)	0.1867 (7)	0.4379 (4)	0.0612 (12)	
H17	−0.078051	0.286037	0.486358	0.073*	
N5	0.1126 (4)	−0.1759 (5)	0.0339 (3)	0.0497 (8)	
C12	−0.2519 (5)	−0.0858 (7)	0.3810 (4)	0.0562 (11)	
C8	0.0117 (5)	−0.1697 (6)	0.1159 (4)	0.0576 (11)	
H8A	0.027464	−0.279960	0.161356	0.069*	
H8B	−0.096445	−0.168051	0.076627	0.069*	
O21	0.2705 (4)	0.3975 (6)	0.2539 (3)	0.0804 (11)	
C7	0.1952 (5)	−0.0965 (6)	−0.1125 (3)	0.0530 (10)	
H7	0.204076	−0.038826	−0.179462	0.064*	
N4	0.2440 (5)	−0.2750 (7)	0.0358 (4)	0.0734 (12)	
C16	−0.2155 (6)	0.0646 (7)	0.4515 (4)	0.0642 (12)	
H16	−0.269929	0.082688	0.509450	0.077*	
N6	0.0842 (5)	−0.0676 (5)	−0.0562 (3)	0.0628 (10)	
C3	0.2948 (6)	−0.2225 (7)	−0.0586 (4)	0.0636 (12)	
C13	−0.3833 (5)	−0.2128 (8)	0.3976 (5)	0.0683 (13)	
H13	−0.392494	−0.200590	0.476651	0.082*	
C14	−0.3496 (9)	−0.4077 (10)	0.3806 (8)	0.121 (3)	
H14A	−0.429774	−0.482233	0.401963	0.182*	
H14B	−0.250109	−0.439262	0.425926	0.182*	
H14C	−0.347001	−0.428504	0.302684	0.182*	
C15	−0.5364 (6)	−0.1510 (13)	0.3291 (7)	0.120 (3)	
H15A	−0.542993	−0.187489	0.252200	0.180*	
H15B	−0.543668	−0.020020	0.333103	0.180*	
H15C	−0.620528	−0.205715	0.358126	0.180*	

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C2A	0.086 (6)	0.071 (6)	0.097 (12)	-0.005 (5)	0.050 (5)	-0.015 (11)
O1A	0.109 (6)	0.109 (7)	0.125 (7)	-0.029 (5)	0.063 (5)	-0.052 (5)
C2B	0.086 (6)	0.071 (6)	0.097 (12)	-0.005 (5)	0.050 (5)	-0.015 (11)
O1B	0.069 (6)	0.093 (8)	0.110 (8)	0.029 (6)	0.033 (6)	0.033 (6)
O19	0.0690 (18)	0.0526 (18)	0.0603 (17)	-0.0160 (15)	0.0173 (15)	-0.0159 (14)
C11	0.048 (2)	0.045 (2)	0.060 (2)	-0.0010 (19)	0.0126 (18)	0.0015 (19)
C9	0.047 (2)	0.0389 (19)	0.047 (2)	0.0028 (17)	0.0077 (16)	0.0007 (16)
C10	0.048 (2)	0.041 (2)	0.048 (2)	0.0023 (16)	0.0110 (16)	0.0018 (17)
C18	0.052 (2)	0.051 (2)	0.051 (2)	-0.0022 (19)	0.0131 (17)	0.0017 (19)
C20	0.053 (2)	0.055 (3)	0.058 (2)	-0.014 (2)	0.0144 (19)	-0.003 (2)
C22	0.052 (2)	0.048 (2)	0.049 (2)	-0.0036 (19)	0.0154 (17)	0.0005 (18)
C17	0.071 (3)	0.060 (3)	0.055 (2)	0.002 (2)	0.019 (2)	-0.006 (2)
N5	0.0589 (19)	0.0377 (17)	0.0539 (19)	0.0035 (16)	0.0139 (15)	-0.0042 (15)
C12	0.049 (2)	0.059 (3)	0.063 (2)	0.007 (2)	0.0184 (18)	0.015 (2)
C8	0.066 (2)	0.046 (2)	0.067 (3)	-0.011 (2)	0.028 (2)	-0.009 (2)
O21	0.083 (2)	0.073 (2)	0.092 (2)	-0.039 (2)	0.0307 (19)	-0.023 (2)
C7	0.080 (3)	0.039 (2)	0.045 (2)	0.001 (2)	0.0247 (19)	0.0076 (18)
N4	0.080 (3)	0.059 (3)	0.084 (3)	0.003 (2)	0.023 (2)	-0.008 (2)
C16	0.065 (3)	0.068 (3)	0.065 (3)	0.011 (2)	0.027 (2)	0.006 (2)
N6	0.077 (2)	0.048 (2)	0.066 (2)	0.0081 (19)	0.0191 (18)	0.0050 (18)
C3	0.070 (3)	0.055 (3)	0.072 (3)	-0.006 (2)	0.030 (2)	-0.009 (2)
C13	0.058 (3)	0.071 (3)	0.080 (3)	-0.002 (2)	0.024 (2)	0.015 (3)
C14	0.119 (6)	0.065 (4)	0.199 (8)	-0.025 (4)	0.079 (6)	0.000 (5)
C15	0.063 (3)	0.134 (7)	0.153 (7)	-0.020 (4)	-0.002 (4)	0.047 (6)

*Geometric parameters (Å, °)*

C2Aa—O1A	1.41 (4)	C17—C16	1.368 (7)
C2Aa—C3	1.52 (5)	C17—H17	0.9300
C2Aa—H2A1	0.9700	N5—N6	1.331 (5)
C2Aa—H2A2	0.9700	N5—N4	1.351 (5)
O1Aa—H1A	0.8200	N5—C8	1.450 (5)
C2Bb—O1B	1.42 (4)	C12—C16	1.389 (7)
C2Bb—C3	1.49 (8)	C12—C13	1.517 (6)
C2Bb—H2B1	0.9700	C8—H8A	0.9700
C2Bb—H2B2	0.9700	C8—H8B	0.9700
O1Bb—H1B	0.8200	C7—N6	1.305 (6)
O19—C20	1.368 (5)	C7—C3	1.343 (7)
O19—C18	1.377 (5)	C7—H7	0.9300
C11—C12	1.378 (6)	N4—C3	1.359 (7)
C11—C10	1.396 (5)	C16—H16	0.9300
C11—H11	0.9300	C13—C14	1.476 (10)
C9—C22	1.335 (6)	C13—C15	1.497 (8)
C9—C10	1.454 (5)	C13—H13	0.9800
C9—C8	1.502 (6)	C14—H14A	0.9600

C10—C18	1.390 (6)	C14—H14B	0.9600
C18—C17	1.378 (6)	C14—H14C	0.9600
C20—O21	1.215 (5)	C15—H15A	0.9600
C20—C22	1.428 (6)	C15—H15B	0.9600
C22—H22	0.9300	C15—H15C	0.9600
O1Aa—C2Aa—C3	107 (4)	C11—C12—C13	122.7 (5)
O1Aa—C2Aa—H2A1	110.3	C16—C12—C13	119.3 (4)
C3—C2Aa—H2A1	110.3	N5—C8—C9	113.1 (3)
O1Aa—C2Aa—H2A2	110.3	N5—C8—H8A	109.0
C3—C2Aa—H2A2	110.3	C9—C8—H8A	109.0
H2A1a—C2Aa—H2A2	108.5	N5—C8—H8B	109.0
C2Aa—O1Aa—H1A	109.5	C9—C8—H8B	109.0
O1Bb—C2Bb—C3	117 (5)	H8A—C8—H8B	107.8
O1Bb—C2Bb—H2B1	108.1	N6—C7—C3	109.3 (4)
C3—C2Bb—H2B1	108.1	N6—C7—H7	125.3
O1Bb—C2Bb—H2B2	108.1	C3—C7—H7	125.3
C3—C2Bb—H2B2	108.1	N5—N4—C3	104.4 (4)
H2B1b—C2Bb—H2B2	107.3	C17—C16—C12	121.7 (4)
C2Bb—O1Bb—H1B	109.5	C17—C16—H16	119.1
C20—O19—C18	121.6 (3)	C12—C16—H16	119.1
C12—C11—C10	122.3 (4)	C7—N6—N5	107.1 (4)
C12—C11—H11	118.9	C7—C3—N4	108.5 (4)
C10—C11—H11	118.9	C7—C3—C2B	125 (3)
C22—C9—C10	119.2 (4)	N4—C3—C2B	126 (3)
C22—C9—C8	123.5 (4)	C7—C3—C2A	120 (2)
C10—C9—C8	117.4 (3)	N4—C3—C2A	132 (2)
C18—C10—C11	117.1 (4)	C14—C13—C15	113.2 (7)
C18—C10—C9	117.5 (4)	C14—C13—C12	113.3 (5)
C11—C10—C9	125.5 (4)	C15—C13—C12	110.9 (5)
O19—C18—C17	116.6 (4)	C14—C13—H13	106.3
O19—C18—C10	121.5 (4)	C15—C13—H13	106.3
C17—C18—C10	121.9 (4)	C12—C13—H13	106.3
O21—C20—O19	116.8 (4)	C13—C14—H14A	109.5
O21—C20—C22	125.8 (4)	C13—C14—H14B	109.5
O19—C20—C22	117.3 (3)	H14A—C14—H14B	109.5
C9—C22—C20	122.9 (4)	C13—C14—H14C	109.5
C9—C22—H22	118.5	H14A—C14—H14C	109.5
C20—C22—H22	118.5	H14B—C14—H14C	109.5
C16—C17—C18	119.0 (5)	C13—C15—H15A	109.5
C16—C17—H17	120.5	C13—C15—H15B	109.5
C18—C17—H17	120.5	H15A—C15—H15B	109.5
N6—N5—N4	110.7 (4)	C13—C15—H15C	109.5
N6—N5—C8	120.1 (3)	H15A—C15—H15C	109.5
N4—N5—C8	129.1 (4)	H15B—C15—H15C	109.5
C11—C12—C16	118.0 (4)		

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*Hydrogen-bond geometry (Å, °)*

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<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C8—H8 <i>A</i> ···N6 <sup>i</sup>	0.97	2.55	3.070 (6)	114

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Symmetry code: (i)  $-x, y-1/2, -z$ .