IUCrData

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4-[(4-Hydroxymethyl-2*H*-1,2,3-triazol-2-yl)-methyl]-6,8-dimethyl-2*H*-chromen-2-one. Corrigendum

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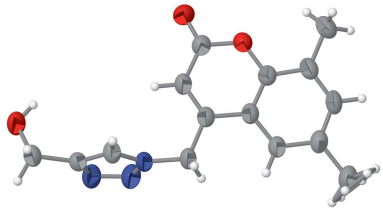
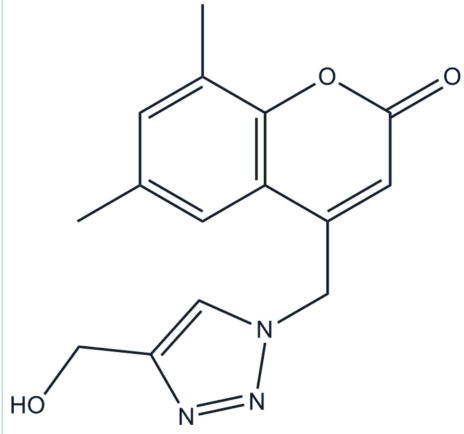
In the paper by El-Khatatneh *et al.* [*IUCrData* (2016), **1**, x161644], the scheme and chemical name in the title are corrected.

In the paper by El-Khatatneh *et al.* (2016), the chemical scheme should be as shown here.

3D view



Chemical scheme



The chemical name in the title is then corrected as '4-[(4-Hydroxymethyl-1*H*-1,2,3-triazol-1-yl)methyl]-6,8-dimethyl-2*H*-chromen-2-one'.

References

El-Khatatneh, N., Chandra, Shamala, D., Shivashankar, K. & Mahendra, M. (2016). *IUCrData*, **1**, x161644.

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Keywords: crystal structure; chromene; triazole; hydrogen bonds.

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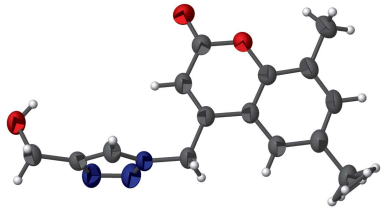
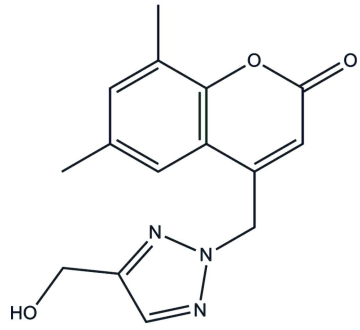
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{15}N_3O_3$, the dihedral angle between the triazole ring and coumarin ring system [r.m.s. deviation = 0.040 Å] is 77.40 (6)°. The O atom of the hydroxymethyl group deviates from the triazole ring plane by 1.345 (1) Å. In the crystal, inversion dimers linked by pairs of O—H···O hydrogen bonds generate $R_2^2(22)$ loops; C—H···O and C—H···N interactions link the dimers into a three-dimensional network.

3D view



Chemical scheme



Structure description

Coumarin derivatives represent an important class of natural and synthetic heterocycles that are often linked to a broad array of biological activities (Gaspar *et al.*, 2015). As part of our ongoing studies of coumarin–triazole derivatives (El-Khatatneh *et al.*, 2016), the title compound (Fig. 1) was synthesized and its crystal structure is now reported.

The dihedral angle between the triazole ring and coumarin ring system [r.m.s. deviation = 0.040 Å] is 77.40 (6)°. Key inter-ring torsion angles include 97.34 (15)° for N19—N15—C14—C13) and −173.30 (13)° for C6—C13—C14—N15. The O atom of the hydroxymethyl group is displaced from the triazole ring plane by 1.345 (1) Å.

In the crystal, inversion dimers linked by pairs of O—H···O hydrogen bonds (Table 1) generate $R_2^2(22)$ loops. The dimers are linked by weak C—H···O and C—H···N hydrogen bonds, generating a three-dimensional network (Fig. 2).

Synthesis and crystallization

A mixture of propargyl alcohol (1.9 mmol), sodium azide (0.14 g, 2.0 mmol), copper(I) iodide (10 mol%) and triethylamine (0.19 g, 1.9 mmol) in 20 ml of acetone was taken in a

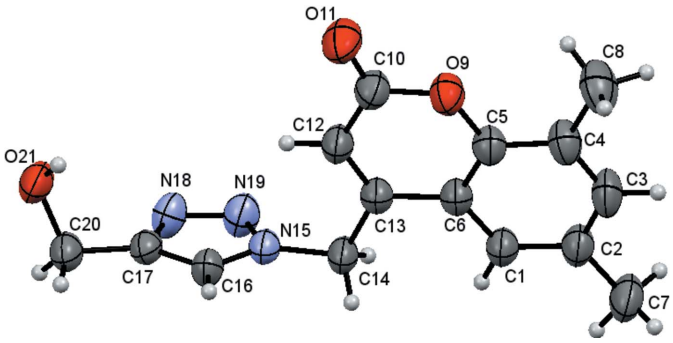


Figure 1
The molecular structure of the title compound with 50% probability displacement ellipsoids.

round-bottom flask and stirred for 1 h. To this mixture, 4-bromomethylcoumarin (1.9 mmol) was added and the stirring continued for 8 h (the reaction was monitored by TLC). After the completion of the reaction, the copper catalyst was filtered through celite and the product was extracted with diethyl ether (3.10 ml). The solvent was removed under vacuum. The crude product was dried and recrystallized from ethyl acetate solution to give colourless blocks.

Yield 92%; colourless solid; m.p. 210–212 °C; IR (KBr, cm^{-1}): 1742 cm^{-1} (lactone C=O), 3311 cm^{-1} (OH); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.70 (*s*, 1H, OH), 2.37 (*s*, 3H, C_6-CH_3) 2.42 (*s*, 3H, C_8-CH_3) 4.83 (*s*, 2H, $-CH_2O-$), 5.43 (*s*, 1H, C_3-H), 5.70 (*s*, 2H, $-CH_2N-$), 7.21-7.24 (*m*, 1H, C_7-H), 7.60 (*s*, 1H, C_5-H), 7.75 (*s*, 1H, Tr—H) p.p.m. ^{13}C NMR (100 MHz, DMSO-d_6): δ 15.0, 20.3, 49.0, 55.0, 113.0, 116.5, 122.0, 123.8, 125.3, 133.1, 134.5, 148.6, 149.5, 150.6, 159.5 p.p.m. Analysis

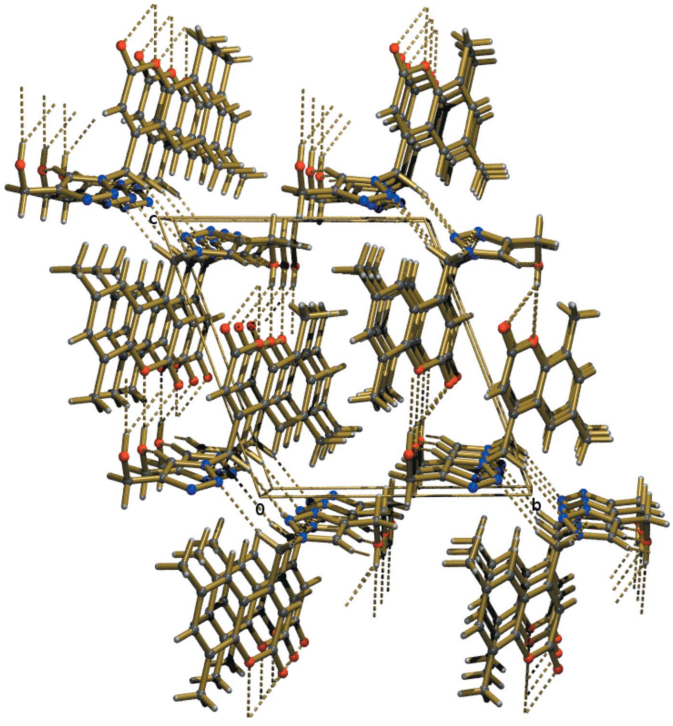


Figure 2
The packing viewed along [100] with hydrogen bonds indicated by dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
O21—H21···O11^{i}	0.82	2.10	2.9155 (19)	176
C14—H14A···N19^{ii}	0.97	2.55	3.486 (2)	162
C14—H14B···N18^{iii}	0.97	2.41	3.344 (2)	162
C16—H16···O21^{iii}	0.93	2.47	3.284 (2)	146

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{15}N_3O_3$
M_r	285.30
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	6.0265 (16), 11.062 (3), 11.848 (3)
α, β, γ (°)	108.812 (7), 103.950 (8), 100.848 (8)
V (Å^3)	694.5 (3)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.80
Crystal size (mm)	0.30 × 0.20 × 0.10
Data collection	
Diffractometer	Bruker X8 Proteum
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8218, 2217, 2142
R_{int}	0.030
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.587
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.114, 1.04
No. of reflections	2217
No. of parameters	194
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.14, −0.14

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS* (Sheldrick, 2008), *SHELXL2016/4* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008).

$C_{15}H_{15}N_3O_3$. Calculated for: C, 63.15; H, 5.30; N, 14.73%. Found: C, 63.08; H, 5.26; N, 14.68%.

Refinement

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

Acknowledgements

MM thanks UGC, New Delhi, Government of India, for awarding a project under the title F. No. 41–920/2012(SR) dated: 25–07–2012. SD is grateful to the Council of Scientific and Industrial Research, New Delhi, India, for financial assistance [Grant No. 02 (0172)/13/EMR-II].

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full crystallographic data

IUCrData (2016). **1**, x161644 [https://doi.org/10.1107/S2414314616016448]

4-[(4-Hydroxymethyl-2*H*-1,2,3-triazol-2-yl)methyl]-6,8-dimethyl-2*H*-chromen-2-one

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4-[(4-Hydroxymethyl-2*H*-1,2,3-triazol-2-yl)methyl]-6,8-dimethyl-2*H*-chromen-2-one

Crystal data

$C_{15}H_{15}N_3O_3$
M_r = 285.30
Triclinic, $P\overline{1}$
a = 6.0265 (16) Å
b = 11.062 (3) Å
c = 11.848 (3) Å
α = 108.812 (7)°
β = 103.950 (8)°
γ = 100.848 (8)°
V = 694.5 (3) Å^3
Z = 2
$F(000)$ = 300
D_x = 1.364 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 2217 reflections
θ = 7.2–64.7°
μ = 0.80 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
8218 measured reflections
2217 independent reflections
2142 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 64.7°, θ_{min} = 7.2°
h = −6→7
k = −12→12
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.114
S = 1.04
2217 reflections
194 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2 + 0.126P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O9	0.37402 (19)	0.76949 (10)	0.45209 (9)	0.0574 (3)	
O21	−0.04447 (19)	1.36437 (12)	0.82987 (11)	0.0658 (3)	
H21	−0.055031	1.305321	0.763928	0.099*	
O11	0.0867 (2)	0.85556 (13)	0.39863 (11)	0.0757 (4)	
C6	0.6271 (2)	0.84311 (13)	0.66749 (13)	0.0452 (3)	
N19	0.2818 (2)	1.08969 (13)	0.92565 (13)	0.0596 (4)	
N15	0.44456 (19)	1.12365 (11)	0.87345 (10)	0.0450 (3)	
C13	0.5024 (2)	0.94266 (13)	0.70280 (13)	0.0458 (3)	
C5	0.5580 (3)	0.75918 (14)	0.54097 (13)	0.0485 (3)	
N18	0.1788 (2)	1.18550 (14)	0.94803 (13)	0.0598 (4)	
C1	0.8117 (3)	0.82578 (14)	0.75347 (14)	0.0500 (4)	
H1	0.860125	0.880475	0.838505	0.060*	
C12	0.3208 (3)	0.94819 (14)	0.61462 (14)	0.0523 (4)	
H12	0.240245	1.011061	0.638265	0.063*	
C4	0.6663 (3)	0.66137 (15)	0.49641 (14)	0.0560 (4)	
C2	0.9229 (3)	0.72895 (15)	0.71413 (15)	0.0542 (4)	
C17	0.2756 (2)	1.28047 (13)	0.91088 (12)	0.0454 (3)	
C20	0.1846 (3)	1.39858 (15)	0.91808 (14)	0.0556 (4)	
H20A	0.178228	1.440812	1.002359	0.067*	
H20B	0.295413	1.462794	0.903203	0.067*	
C14	0.5903 (3)	1.03629 (14)	0.83948 (13)	0.0517 (4)	
H14A	0.597759	0.983779	0.891184	0.062*	
H14B	0.751444	1.090828	0.859203	0.062*	
C10	0.2480 (3)	0.85893 (15)	0.48426 (14)	0.0552 (4)	
C3	0.8477 (3)	0.64959 (16)	0.58574 (16)	0.0614 (4)	
H3	0.923601	0.585243	0.558673	0.074*	
C16	0.4471 (2)	1.24121 (13)	0.86378 (13)	0.0477 (3)	
H16	0.545138	1.286513	0.831682	0.057*	
C7	1.1162 (3)	0.70920 (19)	0.80743 (18)	0.0718 (5)	
H7A	1.168804	0.636256	0.764247	0.108*	0.31 (2)
H7B	1.055076	0.689718	0.869294	0.108*	0.31 (2)
H7C	1.248294	0.789024	0.848366	0.108*	0.31 (2)
H7D	1.145979	0.773742	0.890357	0.108*	0.69 (2)
H7E	1.259707	0.720281	0.785311	0.108*	0.69 (2)
H7F	1.066488	0.620975	0.806239	0.108*	0.69 (2)
C8	0.5915 (4)	0.57393 (19)	0.35871 (16)	0.0760 (5)	
H8A	0.690144	0.613072	0.319554	0.114*	
H8B	0.427523	0.565734	0.318815	0.114*	
H8C	0.609121	0.487213	0.349891	0.114*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O9	0.0673 (7)	0.0594 (6)	0.0445 (5)	0.0320 (5)	0.0145 (5)	0.0135 (4)
O21	0.0540 (6)	0.0732 (7)	0.0627 (7)	0.0362 (5)	0.0113 (5)	0.0124 (5)
O11	0.0765 (8)	0.0868 (8)	0.0507 (6)	0.0440 (7)	0.0024 (6)	0.0120 (6)
C6	0.0468 (7)	0.0422 (7)	0.0487 (7)	0.0192 (6)	0.0170 (6)	0.0156 (6)
N19	0.0560 (7)	0.0609 (7)	0.0744 (9)	0.0269 (6)	0.0276 (6)	0.0316 (6)
N15	0.0420 (6)	0.0456 (6)	0.0436 (6)	0.0198 (5)	0.0113 (5)	0.0105 (5)
C13	0.0457 (7)	0.0434 (7)	0.0477 (7)	0.0186 (6)	0.0143 (6)	0.0143 (6)
C5	0.0549 (8)	0.0481 (7)	0.0481 (8)	0.0228 (6)	0.0189 (6)	0.0197 (6)
N18	0.0546 (7)	0.0692 (8)	0.0696 (8)	0.0322 (6)	0.0297 (6)	0.0294 (7)
C1	0.0504 (8)	0.0486 (7)	0.0508 (8)	0.0237 (6)	0.0155 (6)	0.0144 (6)
C12	0.0518 (8)	0.0524 (8)	0.0514 (8)	0.0272 (6)	0.0136 (6)	0.0136 (6)
C4	0.0723 (10)	0.0526 (8)	0.0518 (8)	0.0309 (7)	0.0283 (7)	0.0180 (7)
C2	0.0564 (8)	0.0527 (8)	0.0607 (9)	0.0290 (7)	0.0213 (7)	0.0218 (7)
C17	0.0420 (7)	0.0472 (7)	0.0401 (7)	0.0183 (6)	0.0094 (5)	0.0082 (5)
C20	0.0553 (8)	0.0534 (8)	0.0509 (8)	0.0274 (6)	0.0126 (6)	0.0080 (6)
C14	0.0488 (8)	0.0502 (8)	0.0502 (8)	0.0271 (6)	0.0099 (6)	0.0093 (6)
C10	0.0560 (8)	0.0583 (8)	0.0493 (8)	0.0268 (7)	0.0117 (7)	0.0163 (7)
C3	0.0766 (11)	0.0591 (9)	0.0647 (9)	0.0434 (8)	0.0329 (8)	0.0237 (7)
C16	0.0467 (7)	0.0452 (7)	0.0535 (8)	0.0203 (6)	0.0196 (6)	0.0156 (6)
C7	0.0739 (11)	0.0756 (11)	0.0714 (11)	0.0479 (9)	0.0187 (9)	0.0242 (9)
C8	0.1070 (15)	0.0748 (11)	0.0542 (10)	0.0497 (11)	0.0331 (10)	0.0167 (8)

Geometric parameters (Å, º)

O9—C10	1.3712 (18)	C4—C8	1.505 (2)
O9—C5	1.3833 (18)	C2—C3	1.395 (2)
O21—C20	1.4118 (18)	C2—C7	1.503 (2)
O21—H21	0.8200	C17—C16	1.3635 (19)
O11—C10	1.2089 (19)	C17—C20	1.4952 (19)
C6—C5	1.392 (2)	C20—H20A	0.9700
C6—C1	1.404 (2)	C20—H20B	0.9700
C6—C13	1.4554 (18)	C14—H14A	0.9700
N19—N18	1.3135 (18)	C14—H14B	0.9700
N19—N15	1.3375 (17)	C3—H3	0.9300
N15—C16	1.3395 (18)	C16—H16	0.9300
N15—C14	1.4513 (16)	C7—H7A	0.9600
C13—C12	1.345 (2)	C7—H7B	0.9600
C13—C14	1.5080 (19)	C7—H7C	0.9600
C5—C4	1.392 (2)	C7—H7D	0.9600
N18—C17	1.352 (2)	C7—H7E	0.9600
C1—C2	1.3829 (19)	C7—H7F	0.9600
C1—H1	0.9300	C8—H8A	0.9600
C12—C10	1.442 (2)	C8—H8B	0.9600
C12—H12	0.9300	C8—H8C	0.9600
C4—C3	1.384 (2)		

C10—O9—C5	122.09 (11)	N15—C14—H14B	108.6
C20—O21—H21	109.5	C13—C14—H14B	108.6
C5—C6—C1	117.93 (12)	H14A—C14—H14B	107.6
C5—C6—C13	118.08 (12)	O11—C10—O9	115.82 (13)
C1—C6—C13	123.98 (12)	O11—C10—C12	126.45 (14)
N18—N19—N15	106.63 (11)	O9—C10—C12	117.73 (13)
N19—N15—C16	110.78 (11)	C4—C3—C2	123.61 (13)
N19—N15—C14	119.15 (12)	C4—C3—H3	118.2
C16—N15—C14	130.04 (12)	C2—C3—H3	118.2
C12—C13—C6	119.78 (12)	N15—C16—C17	105.51 (12)
C12—C13—C14	123.64 (12)	N15—C16—H16	127.2
C6—C13—C14	116.58 (11)	C17—C16—H16	127.2
O9—C5—C6	120.68 (12)	C2—C7—H7A	109.5
O9—C5—C4	116.33 (13)	C2—C7—H7B	109.5
C6—C5—C4	122.99 (13)	H7A—C7—H7B	109.5
N19—N18—C17	109.68 (12)	C2—C7—H7C	109.5
C2—C1—C6	121.27 (14)	H7A—C7—H7C	109.5
C2—C1—H1	119.4	H7B—C7—H7C	109.5
C6—C1—H1	119.4	C2—C7—H7D	109.5
C13—C12—C10	121.56 (13)	H7A—C7—H7D	141.1
C13—C12—H12	119.2	H7B—C7—H7D	56.3
C10—C12—H12	119.2	H7C—C7—H7D	56.3
C3—C4—C5	116.29 (14)	C2—C7—H7E	109.5
C3—C4—C8	121.84 (14)	H7A—C7—H7E	56.3
C5—C4—C8	121.87 (14)	H7B—C7—H7E	141.1
C1—C2—C3	117.89 (14)	H7C—C7—H7E	56.3
C1—C2—C7	120.78 (14)	H7D—C7—H7E	109.5
C3—C2—C7	121.33 (13)	C2—C7—H7F	109.5
N18—C17—C16	107.39 (12)	H7A—C7—H7F	56.3
N18—C17—C20	121.83 (13)	H7B—C7—H7F	56.3
C16—C17—C20	130.68 (14)	H7C—C7—H7F	141.1
O21—C20—C17	112.70 (12)	H7D—C7—H7F	109.5
O21—C20—H20A	109.1	H7E—C7—H7F	109.5
C17—C20—H20A	109.1	C4—C8—H8A	109.5
O21—C20—H20B	109.1	C4—C8—H8B	109.5
C17—C20—H20B	109.1	H8A—C8—H8B	109.5
H20A—C20—H20B	107.8	C4—C8—H8C	109.5
N15—C14—C13	114.71 (11)	H8A—C8—H8C	109.5
N15—C14—H14A	108.6	H8B—C8—H8C	109.5
C13—C14—H14A	108.6		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O21—H21···O11^{i}	0.82	2.10	2.9155 (19)	176
C14—H14*A*···N19^{ii}	0.97	2.55	3.486 (2)	162

C14—H14*B*···N18^{iii}	0.97	2.41	3.344 (2)	162
C16—H16···O21^{iii}	0.93	2.47	3.284 (2)	146

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