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(*E*)-4-Amino-*N*′-(5-chloro-2-hydroxybenzylidene)benzohydrazide

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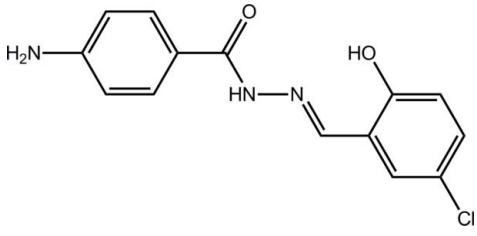
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.084; data-to-parameter ratio = 16.2.

The title compound, $C_{14}H_{12}ClN_3O_2$, displays an *E* conformation with respect to the C=N double bond. The dihedral angle between the benzene rings is 41.3 (5)°. The molecular structure is stabilized by an intramolecular O—H···N hydrogen bond. In the crystal, N—H···O and weak N—H···Cl hydrogen bonds link the molecules into a three-dimensional architecture. In addition, there are weak C—H···π stacking interactions.

Related literature

For the biological properties of Schiff base and hydrazone compounds, see: Kucukguzel *et al.* (2006); Khattab (2005); Karthikeyan *et al.* (2006). For closely related structures and background references, see: Bernhardt *et al.* (2003, 2005); Armstrong *et al.* (2003); Cao (2009); Yang (2009); Zhou & Yang (2010); Zhang *et al.*, (2009).



Experimental

Crystal data

$C_{14}H_{12}ClN_3O_2$
M_r = 289.72
Orthorhombic, $Pna2_1$
a = 9.3375 (16) Å
b = 9.7194 (16) Å
c = 14.214 (3) Å
V = 1290.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 296 K
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.947, T_{max} = 0.970
8516 measured reflections
2944 independent reflections
2866 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.084
S = 1.10
2944 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 1407 Friedel pairs
Flack parameter: 0.09 (9)

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C9–C14 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.86	2.12	2.921 (2)	156
N1—H1A···Cl1^{ii}	0.86	2.94	3.680 (2)	145
N1—H1B···O1^{iii}	0.86	2.19	2.978 (3)	151
O2—H2···N3	0.82	1.88	2.588 (3)	145
C5—H5···$Cg2^{iv}$	0.93	2.92	3.473 (3)	120
C14—H14···$Cg1^{v}$	0.93	2.83	3.641 (3)	147

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z$; (ii) $x, y, z-1$; (iii) $-x+2, -y+1, z-\frac{1}{2}$; (iv) $-x+2, -y+2, z-\frac{1}{2}$; (v) $x+\frac{3}{2}, -y+\frac{1}{2}, z$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5088).

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supporting information

Acta Cryst. (2012). E**68**, o1546–o1547 [doi:10.1107/S160053681201803X]

(*E*)-4-Amino-*N*′-(5-chloro-2-hydroxybenzylidene)benzohydrazide

Zhong-Feng Shi and Jia-Ming Li

S1. Comment

Schiff bases are an important class of compounds in the medicinal and pharmaceutical fields and have been found to play a role in the development of coordination chemistry as they readily form stable complexes with most transition metals. These complexes show interesting properties, *e.g.* their ability to reversibly bind oxygen, catalytic activity in hydrogenation of olefins and transfer of an amino group, photochromic properties, and complexing ability towards toxic metals (Karthikeyan *et al.*, 2006; Khattab, 2005; Kucukguzel *et al.*, 2006). Recently, hydrazone Schiff base compounds (Cao, 2009; Yang, 2009; Zhou & Yang, 2010; Zhang *et al.*, 2009) derived from the reaction of aldehydes with hydrazines have been demonstrated to possess excellent biological activities, such as anti-bacterial, anti-convulsant, and anti-tubercular (Bernhardt & Caldwell *et al.*, 2003; Bernhardt & Chin *et al.*, 2005; Armstrong *et al.*, 2003). In order to explore new anti-bacterial materials, a new hydrazone derivatives was prepared and characterized. It can be seen that in addition to the presence of the typical functional group —CO—NH—N═ CH—, the compound has chloro, hydroxy and amino substituents, which may enhance its biological properties.

As shown in Fig. 1, the molecule displays an *E* configuration with respect to the C═N double bond. The dihedral angle between the two benzene rings is 41.3 (5)°. The bond lengths and angles are as expected for a compound of this type and agree with the other ligands belonging to the hydrazone series, mentioned above. The C8═N3 and C7═O1 bond lengths of 1.287 (3) and 1.238 (3) Å, respectively, conform to the values for double bonds. Whereas the C1—N1, C7—N2, C10—O2 and N2—N3 [1.367 (3), 1.370 (3), 1.355 (3) and 1.368 (3) Å, respectively] bond lengths correspond to a single bond. In the crystal packing, it is noted that amino H (H1A, H1B) and amide H2A are involved in forming intermolecular N—H···O and N—H···Cl hydrogen bonds (Fig. 2 and Table 1), linking the molecules into a three-dimensional supramolecular structure. In addition, neighbouring molecules are also interact through weak C—H···π stacking interactions, Table 1.

S2. Experimental

To a methanol solution (20 ml) of 5-chloro-2-hydroxybenzaldehyde (1 mmol, 0.157 g) and 4-aminobenzohydrazide (1 mmol, 0.151 g), a few drops of acetic acid were added. The mixture was refluxed for 2 h and then cooled to room temperature to give a yellow solution. Crystals of the title compound were formed by gradual evaporation of the solvent over a period of six days at room temperature.

S3. Refinement

H-atoms were placed in calculated positions (C—H = 0.93, O—H = 0.82 and N—H = 0.86-0.89 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C or N) and 1.5 U_{eq}(O).

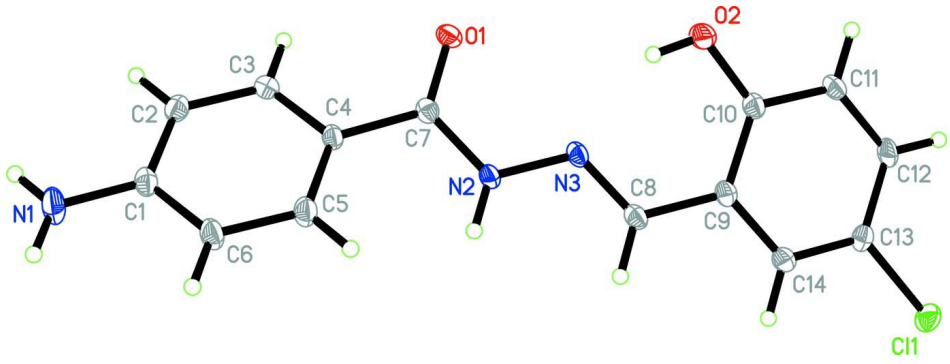

Figure 1

The molecular structure of the title compound, with displacement ellipsoids at the 30% probability level.

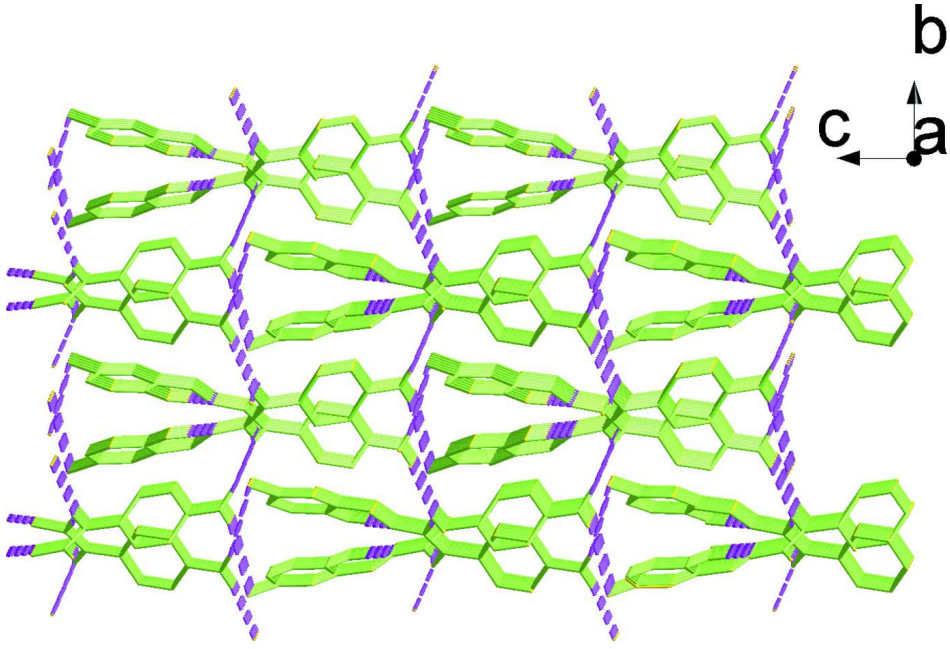

Figure 2

Crystal packing in the title compound where molecules are linked *via* N—H···O and N—H···Cl hydrogen bonds (dashed lines). Except for those involved in hydrogen-bonding interactions, H atoms have been omitted for clarity.

(*E*)-4-Amino-*N*′-(5-chloro-2-hydroxybenzylidene)benzohydrazide

Crystal data

$C_{14}H_{12}ClN_3O_2$
$M_r = 289.72$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 9.3375\ (16)$ Å
$b = 9.7194\ (16)$ Å
$c = 14.214\ (3)$ Å
$V = 1290.0\ (4)$ Å^3
$Z = 4$

$F(000) = 600$
$D_x = 1.487$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6341 reflections
$\theta = 1.4$–$27.5°$
$\mu = 0.30$ mm^{-1}
$T = 296$ K
Block, yellow
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.947, T_{max} = 0.970
8516 measured reflections
2944 independent reflections
2866 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −11→12
k = −12→9
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.084
S = 1.10
2944 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.2086P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 1407 Friedel pairs
Absolute structure parameter: 0.09 (9)

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. 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 $F^2 > 2$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}
Cl1	0.75084 (6)	0.97018 (6)	0.79216 (7)	0.03468 (17)
O1	1.14409 (16)	0.65129 (17)	0.27805 (12)	0.0267 (4)
O2	1.19222 (18)	0.8254 (2)	0.51735 (12)	0.0343 (4)
H2	1.1593	0.8184	0.4641	0.051*
N1	0.8561 (3)	0.6238 (2)	−0.12949 (16)	0.0377 (5)
H1A	0.7980	0.6813	−0.1551	0.045*
H1B	0.8867	0.5542	−0.1610	0.045*
N2	0.94718 (19)	0.78349 (19)	0.30263 (14)	0.0224 (4)
H2A	0.8682	0.8164	0.2816	0.027*
N3	0.9907 (2)	0.8088 (2)	0.39290 (13)	0.0224 (4)
C1	0.8999 (2)	0.6432 (2)	−0.03874 (17)	0.0262 (5)
C2	0.9933 (3)	0.5508 (2)	0.00555 (18)	0.0270 (5)
H2B	1.0272	0.4748	−0.0273	0.032*
C3	1.0352 (2)	0.5718 (2)	0.09760 (17)	0.0255 (5)
H3	1.0982	0.5103	0.1257	0.031*

C4	0.9840 (2)	0.6846 (2)	0.14908 (16)	0.0220 (4)
C5	0.8906 (3)	0.7754 (3)	0.10492 (17)	0.0286 (5)
H5	0.8548	0.8503	0.1382	0.034*
C6	0.8504 (3)	0.7562 (3)	0.01244 (17)	0.0308 (5)
H6	0.7895	0.8193	−0.0161	0.037*
C7	1.0335 (2)	0.7040 (2)	0.24690 (16)	0.0220 (4)
C8	0.9037 (2)	0.8682 (2)	0.45000 (17)	0.0239 (5)
H8	0.8149	0.8985	0.4294	0.029*
C9	0.9471 (2)	0.8871 (2)	0.54757 (16)	0.0230 (4)
C10	1.0863 (2)	0.8585 (2)	0.57817 (17)	0.0247 (5)
C11	1.1199 (2)	0.8632 (2)	0.67355 (18)	0.0274 (5)
H11	1.2124	0.8434	0.6930	0.033*
C12	1.0177 (3)	0.8968 (2)	0.73943 (16)	0.0268 (5)
H12	1.0402	0.8979	0.8031	0.032*
C13	0.8807 (3)	0.9291 (2)	0.70915 (17)	0.0256 (5)
C14	0.8452 (3)	0.9260 (2)	0.61485 (16)	0.0253 (5)
H14	0.7534	0.9497	0.5959	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0388 (3)	0.0439 (3)	0.0213 (3)	0.0096 (2)	0.0028 (2)	−0.0028 (3)
O1	0.0207 (7)	0.0360 (8)	0.0235 (9)	0.0020 (6)	−0.0044 (6)	0.0024 (7)
O2	0.0268 (9)	0.0562 (11)	0.0198 (8)	0.0067 (8)	−0.0026 (7)	0.0004 (8)
N1	0.0550 (14)	0.0359 (11)	0.0221 (11)	0.0034 (10)	−0.0080 (10)	−0.0054 (9)
N2	0.0218 (8)	0.0301 (9)	0.0155 (8)	0.0004 (7)	−0.0036 (7)	0.0002 (7)
N3	0.0232 (9)	0.0287 (9)	0.0152 (8)	−0.0022 (7)	−0.0037 (7)	0.0020 (7)
C1	0.0289 (11)	0.0308 (12)	0.0188 (10)	−0.0055 (9)	−0.0003 (8)	0.0001 (9)
C2	0.0284 (11)	0.0270 (10)	0.0254 (11)	0.0000 (9)	0.0027 (9)	−0.0042 (10)
C3	0.0228 (10)	0.0289 (11)	0.0247 (12)	0.0021 (9)	−0.0020 (9)	−0.0010 (9)
C4	0.0227 (10)	0.0267 (10)	0.0166 (10)	−0.0023 (8)	−0.0013 (8)	−0.0002 (8)
C5	0.0374 (12)	0.0291 (11)	0.0195 (11)	0.0054 (9)	−0.0026 (9)	−0.0010 (9)
C6	0.0403 (14)	0.0317 (11)	0.0206 (11)	0.0065 (10)	−0.0077 (10)	0.0004 (9)
C7	0.0236 (10)	0.0239 (10)	0.0185 (11)	−0.0033 (8)	−0.0003 (9)	0.0029 (8)
C8	0.0235 (10)	0.0288 (11)	0.0195 (11)	−0.0007 (8)	−0.0047 (8)	0.0018 (9)
C9	0.0271 (11)	0.0239 (10)	0.0180 (10)	−0.0009 (8)	−0.0033 (8)	0.0010 (9)
C10	0.0263 (11)	0.0274 (10)	0.0205 (11)	0.0000 (8)	−0.0032 (9)	−0.0010 (9)
C11	0.0258 (11)	0.0325 (11)	0.0239 (12)	0.0019 (9)	−0.0093 (9)	−0.0012 (9)
C12	0.0360 (12)	0.0299 (11)	0.0146 (10)	0.0002 (9)	−0.0062 (9)	−0.0001 (9)
C13	0.0322 (12)	0.0248 (10)	0.0197 (11)	0.0011 (8)	0.0015 (9)	−0.0022 (9)
C14	0.0252 (11)	0.0293 (10)	0.0215 (11)	0.0019 (9)	−0.0033 (8)	0.0009 (9)

Geometric parameters (Å, º)

Cl1—C13	1.741 (2)	C4—C5	1.392 (3)
O1—C7	1.235 (2)	C4—C7	1.479 (3)
O2—C10	1.355 (3)	C5—C6	1.381 (3)
O2—H2	0.8200	C5—H5	0.9300

N1—C1	1.367 (3)	C6—H6	0.9300
N1—H1A	0.8600	C8—C9	1.458 (3)
N1—H1B	0.8600	C8—H8	0.9300
N2—N3	1.369 (3)	C9—C10	1.399 (3)
N2—C7	1.370 (3)	C9—C14	1.403 (3)
N2—H2A	0.8600	C10—C11	1.393 (3)
N3—C8	1.287 (3)	C11—C12	1.378 (3)
C1—C6	1.396 (3)	C11—H11	0.9300
C1—C2	1.402 (3)	C12—C13	1.390 (3)
C2—C3	1.382 (3)	C12—H12	0.9300
C2—H2B	0.9300	C13—C14	1.382 (3)
C3—C4	1.402 (3)	C14—H14	0.9300
C3—H3	0.9300		

C10—O2—H2	109.5	C1—C6—H6	119.6
C1—N1—H1A	120.0	O1—C7—N2	121.3 (2)
C1—N1—H1B	120.0	O1—C7—C4	123.1 (2)
H1A—N1—H1B	120.0	N2—C7—C4	115.54 (19)
N3—N2—C7	117.90 (19)	N3—C8—C9	118.7 (2)
N3—N2—H2A	121.1	N3—C8—H8	120.6
C7—N2—H2A	121.1	C9—C8—H8	120.6
C8—N3—N2	118.93 (19)	C10—C9—C14	118.3 (2)
N1—C1—C6	120.1 (2)	C10—C9—C8	122.0 (2)
N1—C1—C2	121.5 (2)	C14—C9—C8	119.6 (2)
C6—C1—C2	118.4 (2)	O2—C10—C11	117.7 (2)
C3—C2—C1	120.6 (2)	O2—C10—C9	122.0 (2)
C3—C2—H2B	119.7	C11—C10—C9	120.4 (2)
C1—C2—H2B	119.7	C12—C11—C10	120.9 (2)
C2—C3—C4	120.8 (2)	C12—C11—H11	119.6
C2—C3—H3	119.6	C10—C11—H11	119.6
C4—C3—H3	119.6	C11—C12—C13	119.0 (2)
C5—C4—C3	118.3 (2)	C11—C12—H12	120.5
C5—C4—C7	122.7 (2)	C13—C12—H12	120.5
C3—C4—C7	118.9 (2)	C14—C13—C12	121.2 (2)
C6—C5—C4	121.0 (2)	C14—C13—Cl1	119.69 (19)
C6—C5—H5	119.5	C12—C13—Cl1	119.13 (18)
C4—C5—H5	119.5	C13—C14—C9	120.3 (2)
C5—C6—C1	120.8 (2)	C13—C14—H14	119.9
C5—C6—H6	119.6	C9—C14—H14	119.9

C7—N2—N3—C8	−171.4 (2)	N2—N3—C8—C9	176.14 (19)
N1—C1—C2—C3	−179.5 (2)	N3—C8—C9—C10	8.1 (3)
C6—C1—C2—C3	−0.2 (4)	N3—C8—C9—C14	−167.4 (2)
C1—C2—C3—C4	0.9 (4)	C14—C9—C10—O2	−177.5 (2)
C2—C3—C4—C5	−0.5 (3)	C8—C9—C10—O2	6.9 (3)
C2—C3—C4—C7	−179.1 (2)	C14—C9—C10—C11	2.7 (3)
C3—C4—C5—C6	−0.7 (4)	C8—C9—C10—C11	−172.9 (2)
C7—C4—C5—C6	177.9 (2)	O2—C10—C11—C12	179.7 (2)

C4—C5—C6—C1	1.4 (4)	C9—C10—C11—C12	−0.4 (4)
N1—C1—C6—C5	178.3 (2)	C10—C11—C12—C13	−1.4 (4)
C2—C1—C6—C5	−1.0 (4)	C11—C12—C13—C14	1.0 (4)
N3—N2—C7—O1	3.3 (3)	C11—C12—C13—Cl1	179.46 (18)
N3—N2—C7—C4	−177.71 (18)	C12—C13—C14—C9	1.3 (4)
C5—C4—C7—O1	−158.2 (2)	Cl1—C13—C14—C9	−177.17 (17)
C3—C4—C7—O1	20.4 (3)	C10—C9—C14—C13	−3.1 (3)
C5—C4—C7—N2	22.8 (3)	C8—C9—C14—C13	172.6 (2)
C3—C4—C7—N2	−158.6 (2)		

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C1–C6 and C9–C14 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.86	2.12	2.921 (2)	156
N1—H1*A*···Cl1^{ii}	0.86	2.94	3.680 (2)	145
N1—H1*B*···O1^{iii}	0.86	2.19	2.978 (3)	151
O2—H2···N3	0.82	1.88	2.588 (3)	145
C5—H5···*Cg*2^{iv}	0.93	2.92	3.473 (3)	120
C14—H14···*Cg*1^{v}	0.93	2.83	3.641 (3)	147

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*; (ii) *x*, *y*, *z*−1; (iii) −*x*+2, −*y*+1, *z*−1/2; (iv) −*x*+2, −*y*+2, *z*−1/2; (v) *x*+3/2, −*y*+1/2, *z*.