Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Hydroxy-5-[(*E*)-(1*H*-indol-3-ylmethylidene)azaniumyl]benzoate

M. Nawaz Tahir[a]* and Hazoor Ahmad Shad[b]

[a]Department of Physics, University of Sargodha, Sargodha, Pakistan, and [b]Depart-Department of Chemistry, Govt. M. D. College, Toba Tek Singh, Punjab, Pakistan
Correspondence e-mail: dmntahir_uos@yahoo.com

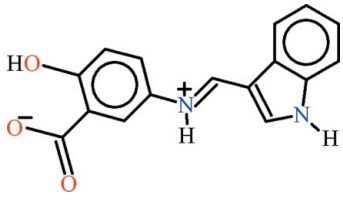
Received 18 November 2010; accepted 22 November 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.057; wR factor = 0.152; data-to-parameter ratio = 12.1.

The zwitterionic title compound, $C_{16}H_{12}N_2O_3$, was obtained as a result of the condensation of 5-aminosalicylic acid and 1*H*-indole-3-carbaldehyde. The whole molecule is roughly planar: the 4-hydroxyanilinic group and the 1*H*-indole-3-carbaldehyde moieties are only slightly twisted, making a dihedral angle of 7.77 (11)°, whereas, the carboxylate group makes a dihedral angle of 3.34 (45)° with the parent 4-hydroxyanilinic group. S(6) ring motifs are formed due to intramolecular O—H···O hydrogen bonding. In the crystal, intermolecular N—H···O and C—H···O hydrogen bonds build up pseudo-rings with $R_1^2(4)$, $R_2^1(7)$ and $R_2^2(14)$ ring motifs. These pseudo-dimers are further linked by N—H···O hydrogen bonds into a chain extending along [101]. C—H···π interactions also occur, along with offset π–π interactions between the anilinic phenyl and the heterocyclic five-membered rings with a centroid–centroid distance of 3.5716 (19) Å.

Related literature

For background to our ongoing work on the synthesis and ligand properties of Schiff bases derived from 2-hydroxy-5-aminobenzoic acid, see: Tahir *et al.* (2010*a*,*b*). For graph-set notation, see: Bernstein *et al.* (1995); Etter (1990).



Experimental

Crystal data

$C_{16}H_{12}N_2O_3$
M_r = 280.28
Monoclinic, $P2_1/n$
a = 7.3463 (6) Å
b = 15.5496 (12) Å
c = 11.5310 (8) Å
β = 104.619 (4)°
V = 1274.57 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.24 × 0.14 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.980, T_{max} = 0.988
9888 measured reflections
2313 independent reflections
1169 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.152
S = 1.00
2313 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

*Cg*3 is the centroid of the C10–C15 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	1.90	2.735 (3)	162
N2—H2···O3^{ii}	0.86	1.95	2.805 (3)	171
O3—H3···O2	0.82	1.70	2.448 (3)	150
C16—H16···O1^{i}	0.93	2.48	3.279 (4)	144
C16—H16···O2^{i}	0.93	2.55	3.439 (4)	160
C5—H5···*Cg*3^{iii}	0.93	2.92	3.643 (4)	136

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors acknowledge the provision of funds for the purchase of the diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2627).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tahir, M. N., Shad, H. A., Khan, M. N. & Tariq, M. I. (2010*a*). *Acta Cryst.* E**66**, o2923.
Tahir, M. N., Tariq, M. I., Ahmad, S. & Sarfraz, M. (2010*b*). *Acta Cryst.* E**66**, o2553–o2554.

supporting information

Acta Cryst. (2010). E**66**, o3314 [https://doi.org/10.1107/S1600536810048579]

2-Hydroxy-5-[(*E*)-(1*H*-indol-3-ylmethylidene)azaniumyl]benzoate

M. Nawaz Tahir and Hazoor Ahmad Shad

S1. Comment

As a part of our on going project related to synthesize various Schiff bases of 2-hydroxy-5-aminobenzoic acid and then their metal complexation (Tahir *et al.*, 2010*a,b*), we report here the title compound (I).

The title compound (I) is Zwitterion similar to 2-hydroxy-5-{[(*E*)-4-methoxybenzylidene]azaniumyl}benzoate (Tahir *et al.*, 2010*b*). In (I), the group A (C2—C7/N1/O3) of 5-aminosalicylic acid moiety and the 1*H*-indole-3-carbaldehyde moiety B (C8—C15/N2) are almost planar with r. m. s. deviations of 0.0258 and 0.0175 Å, respectively (Fig. 1). The dihedral angle between A/B is 7.77 (11)°. The carboxylate group C (O1/C1/O2) is oriented at a dihedral angle of 3.34 (45)° with the parent group A. The observed values of C═N and C═O are 1.298 (5) and 1.254 (4) Å, respectively compared to 1.291 (2) and 1.242 (2) Å observed in related compound (Tahir *et al.*, 2010*b*).

In the title compound S(6) ring motif (Etter, 1990; Bernstein *et al.*, 1995), is formed due to intramolecular H-bonding of O—H···O type (Table 1, Fig. 1). The N—H···O type of H-bondings build up a pseudo dimer with $R_2^2(14)$ ring motifs (Table 1, Fig. 2). The C—H···O and N—H···O types of H-bondings (Table 2) complete $R_1^2(4)$ and $R_2^1(7)$ ring motifs (Table 1, Fig. 2). The pseudo dimers are interlinked due to H-bonding of N—H···O type (Table 1, Fig. 2) resulting in dimensional polymeric chains extending along the crystallographic [1 0 1] axis. A C—H···π (Table 1) and offset π–π interaction between the benzene ring (C2—C7) of anilinic and heterocyclic five membered (N2/C15/C10/C9/C16) groups at a distance of 3.5716 (19) Å, with plane to plane distance of 3.25 and 3.41 Å and mean offset angle of 20.9°, play important role in stabilizing the molecules.

S2. Experimental

Equimolar quantities of 1*H*-indole-3-carbaldehyde and and 5-amino-2-hydroxybenzoic acid were refluxed in methanol along with few drops of acetic acid as catalyst for 30 min resulting in orange yellow solution. The solution was kept at room temperature which affoarded orange needles after five days.

S3. Refinement

The H-atoms were positioned geometrically (O–H = 0.82, N–H = 0.86, C–H = 0.93 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = 1.2U_{eq}(C, N, O).

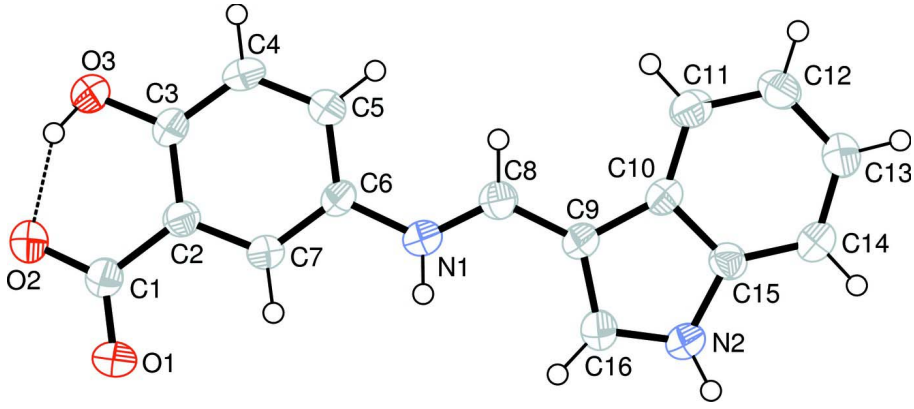


Figure 1

View of the title compound with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii. The dotted line represent the intramolecular H-bonding.

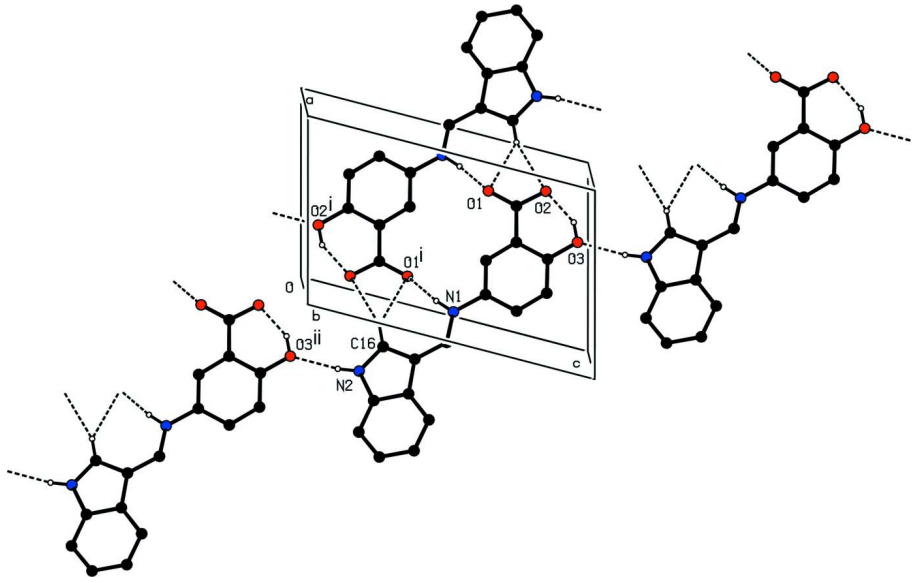


Figure 2

Partial packing view showing the formation of dimers through different ring motifs and infinite one dimensional polymeric chains. [Symmetry codes: (i) -*x*+2, -*y*, -*z*; (ii) *x*-1, *y*, *z*-1]

2-Hydroxy-5-[(*E*)-(1*H*-indol-3-ylmethylidene)azaniumyl]benzoate

Crystal data

$C_{16}H_{12}N_2O_3$
$M_r = 280.28$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.3463\ (6)$ Å
$b = 15.5496\ (12)$ Å
$c = 11.5310\ (8)$ Å
$\beta = 104.619\ (4)°$
$V = 1274.57\ (17)$ Å^3
$Z = 4$

$F(000) = 584$
$D_x = 1.461$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1169 reflections
$\theta = 2.3–25.3°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Needle, orange
0.24 × 0.14 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.20 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.980, T_{max} = 0.988
9888 measured reflections
2313 independent reflections
1169 reflections with $I > 2\sigma(I)$
R_{int} = 0.080
θ_{max} = 25.3°, θ_{min} = 2.3°
h = −8→8
k = −18→18
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.152
S = 1.00
2313 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
O1	1.2830 (3)	0.00635 (16)	0.14061 (19)	0.0527 (10)
O2	1.3616 (3)	0.01396 (16)	0.34045 (19)	0.0529 (10)
O3	1.1429 (3)	0.07152 (17)	0.44973 (17)	0.0486 (9)
N1	0.6066 (4)	0.10087 (18)	0.0169 (2)	0.0394 (10)
N2	0.1653 (4)	0.11644 (18)	−0.3115 (2)	0.0408 (10)
C1	1.2446 (5)	0.0237 (2)	0.2380 (3)	0.0375 (12)
C2	1.0566 (4)	0.0584 (2)	0.2369 (3)	0.0334 (11)
C3	1.0142 (5)	0.0819 (2)	0.3454 (3)	0.0364 (11)
C4	0.8368 (5)	0.1138 (2)	0.3417 (3)	0.0459 (13)
C5	0.7012 (5)	0.1219 (2)	0.2364 (3)	0.0424 (14)
C6	0.7423 (4)	0.0983 (2)	0.1288 (3)	0.0338 (11)
C7	0.9190 (4)	0.0666 (2)	0.1312 (3)	0.0349 (11)
C8	0.4406 (5)	0.1355 (2)	−0.0070 (3)	0.0387 (11)
C9	0.3080 (5)	0.1370 (2)	−0.1184 (3)	0.0344 (11)
C10	0.1205 (4)	0.1715 (2)	−0.1405 (3)	0.0324 (11)
C11	0.0164 (5)	0.2123 (2)	−0.0705 (3)	0.0410 (11)
C12	−0.1644 (5)	0.2377 (2)	−0.1229 (3)	0.0451 (12)

C13	−0.2477 (5)	0.2216 (2)	−0.2434 (3)	0.0476 (14)
C14	−0.1494 (5)	0.1805 (2)	−0.3140 (3)	0.0411 (11)
C15	0.0346 (5)	0.1569 (2)	−0.2617 (3)	0.0350 (11)
C16	0.3256 (5)	0.1038 (2)	−0.2278 (3)	0.0411 (12)
H1	0.63809	0.07659	−0.04221	0.0472*
H2	0.14581	0.10162	−0.38548	0.0489*
H3	1.24072	0.05315	0.43705	0.0584*
H4	0.80901	0.13014	0.41293	0.0551*
H5	0.58271	0.14295	0.23633	0.0512*
H7	0.94585	0.05043	0.05962	0.0420*
H8	0.40583	0.16216	0.05637	0.0463*
H11	0.06906	0.22218	0.01060	0.0494*
H12	−0.23290	0.26623	−0.07704	0.0542*
H13	−0.37122	0.23872	−0.27642	0.0567*
H14	−0.20470	0.16904	−0.39435	0.0495*
H16	0.43238	0.07717	−0.24027	0.0491*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0458 (16)	0.076 (2)	0.0390 (14)	0.0109 (13)	0.0160 (12)	−0.0088 (13)
O2	0.0373 (15)	0.079 (2)	0.0397 (15)	0.0091 (13)	0.0048 (12)	−0.0035 (14)
O3	0.0402 (16)	0.0743 (19)	0.0303 (13)	0.0077 (14)	0.0070 (11)	−0.0027 (12)
N1	0.0406 (19)	0.0466 (19)	0.0307 (15)	0.0039 (16)	0.0087 (13)	−0.0042 (14)
N2	0.0439 (19)	0.0481 (19)	0.0292 (15)	0.0048 (15)	0.0071 (14)	−0.0045 (14)
C1	0.036 (2)	0.040 (2)	0.035 (2)	0.0021 (17)	0.0063 (17)	−0.0006 (17)
C2	0.034 (2)	0.036 (2)	0.0306 (18)	−0.0016 (16)	0.0087 (15)	−0.0013 (16)
C3	0.037 (2)	0.041 (2)	0.0285 (18)	0.0015 (18)	0.0033 (16)	0.0003 (16)
C4	0.050 (2)	0.060 (3)	0.0279 (18)	0.008 (2)	0.0104 (17)	−0.0090 (18)
C5	0.037 (2)	0.054 (3)	0.036 (2)	0.0112 (18)	0.0086 (16)	−0.0048 (18)
C6	0.035 (2)	0.038 (2)	0.0260 (17)	0.0012 (17)	0.0031 (15)	−0.0027 (16)
C7	0.034 (2)	0.042 (2)	0.0295 (18)	0.0013 (16)	0.0093 (15)	−0.0038 (16)
C8	0.041 (2)	0.040 (2)	0.0348 (19)	0.0065 (18)	0.0090 (16)	0.0011 (17)
C9	0.036 (2)	0.037 (2)	0.0288 (17)	0.0008 (16)	0.0056 (15)	−0.0030 (16)
C10	0.037 (2)	0.031 (2)	0.0279 (18)	−0.0014 (16)	0.0058 (15)	0.0007 (15)
C11	0.048 (2)	0.042 (2)	0.0332 (19)	0.0006 (19)	0.0109 (17)	−0.0011 (17)
C12	0.044 (2)	0.048 (2)	0.047 (2)	0.0103 (19)	0.0181 (18)	0.0001 (19)
C13	0.040 (2)	0.056 (3)	0.045 (2)	0.0095 (19)	0.0075 (18)	0.004 (2)
C14	0.043 (2)	0.047 (2)	0.0308 (19)	0.0011 (19)	0.0047 (17)	0.0003 (17)
C15	0.037 (2)	0.034 (2)	0.0352 (19)	0.0039 (17)	0.0115 (16)	0.0028 (16)
C16	0.037 (2)	0.046 (2)	0.038 (2)	0.0066 (18)	0.0050 (17)	−0.0019 (18)

Geometric parameters (Å, º)

O1—C1	1.254 (4)	C9—C10	1.440 (5)
O2—C1	1.283 (4)	C9—C16	1.399 (5)
O3—C3	1.339 (4)	C10—C15	1.399 (5)
O3—H3	0.8200	C10—C11	1.397 (5)

N1—C6	1.418 (4)	C11—C12	1.371 (5)
N1—C8	1.298 (5)	C12—C13	1.392 (5)
N2—C16	1.335 (4)	C13—C14	1.375 (5)
N2—C15	1.388 (5)	C14—C15	1.384 (5)
N1—H1	0.8600	C4—H4	0.9300
N2—H2	0.8600	C5—H5	0.9300
C1—C2	1.480 (5)	C7—H7	0.9300
C2—C7	1.379 (5)	C8—H8	0.9300
C2—C3	1.412 (5)	C11—H11	0.9300
C3—C4	1.385 (5)	C12—H12	0.9300
C4—C5	1.368 (5)	C13—H13	0.9300
C5—C6	1.398 (5)	C14—H14	0.9300
C6—C7	1.382 (4)	C16—H16	0.9300
C8—C9	1.403 (5)		

C3—O3—H3	109.00	C9—C10—C11	134.9 (3)
C6—N1—C8	127.8 (3)	C10—C11—C12	119.1 (3)
C15—N2—C16	110.1 (3)	C11—C12—C13	121.4 (3)
C8—N1—H1	116.00	C12—C13—C14	120.7 (3)
C6—N1—H1	116.00	C13—C14—C15	117.8 (3)
C16—N2—H2	125.00	C10—C15—C14	122.5 (3)
C15—N2—H2	125.00	N2—C15—C10	107.6 (3)
O2—C1—C2	117.3 (3)	N2—C15—C14	129.9 (3)
O1—C1—O2	123.3 (3)	N2—C16—C9	109.4 (3)
O1—C1—C2	119.4 (3)	C3—C4—H4	119.00
C3—C2—C7	118.9 (3)	C5—C4—H4	119.00
C1—C2—C3	120.1 (3)	C4—C5—H5	120.00
C1—C2—C7	121.0 (3)	C6—C5—H5	120.00
O3—C3—C4	121.1 (3)	C2—C7—H7	119.00
O3—C3—C2	120.1 (3)	C6—C7—H7	119.00
C2—C3—C4	118.8 (3)	N1—C8—H8	117.00
C3—C4—C5	121.9 (3)	C9—C8—H8	117.00
C4—C5—C6	119.4 (3)	C10—C11—H11	120.00
C5—C6—C7	119.2 (3)	C12—C11—H11	120.00
N1—C6—C5	122.7 (3)	C11—C12—H12	119.00
N1—C6—C7	118.0 (3)	C13—C12—H12	119.00
C2—C7—C6	121.7 (3)	C12—C13—H13	120.00
N1—C8—C9	126.8 (3)	C14—C13—H13	120.00
C8—C9—C10	125.5 (3)	C13—C14—H14	121.00
C8—C9—C16	128.2 (3)	C15—C14—H14	121.00
C10—C9—C16	106.3 (3)	N2—C16—H16	125.00
C11—C10—C15	118.4 (3)	C9—C16—H16	125.00
C9—C10—C15	106.7 (3)		

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the C10–C15 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	1.90	2.735 (3)	162
N2—H2···O3^{ii}	0.86	1.95	2.805 (3)	171
O3—H3···O2	0.82	1.70	2.448 (3)	150
C16—H16···O1^{i}	0.93	2.48	3.279 (4)	144
C16—H16···O2^{i}	0.93	2.55	3.439 (4)	160
C5—H5···*Cg*3^{iii}	0.93	2.92	3.643 (4)	136

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