

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1,2-Bis(2-hydroxy-5-methylbenzylidene)-hydrazine

B. Saravanan,^a A. Jayamani,^b N. Sengottuvelan,^b
G. Chakkaravarthi^{c*} and V. Manivannan^{a*}^aCentre for Research and Development, PRIST University, Vallam, Thanjavur 613 403, India, ^bDepartment of Chemistry, DDE, Alagappa University, Karaikudi 630 003, India, and ^cDepartment of Physics, CPCL Polytechnic College, Chennai 600 068, India

Correspondence e-mail: chakkaravarthi_2005@yahoo.com, crystallography2010@gmail.com

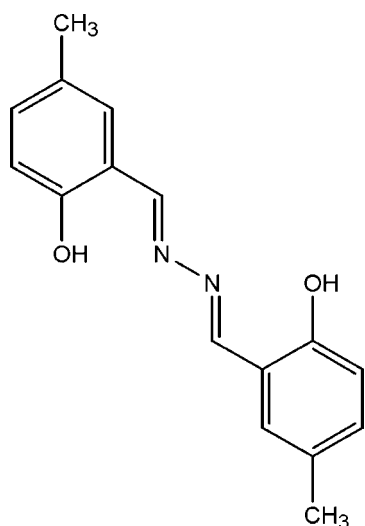
Received 27 July 2013; accepted 1 August 2013

Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.050; wR factor = 0.154; data-to-parameter ratio = 16.0.

The molecular structure of the title compound, $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_2$, is stabilized by intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds with $S(6)$ graph-set motifs, so that the molecule is almost planar, with a $\text{C}=\text{N}-\text{N}=\text{C}$ torsion angle of $-179.7(2)^\circ$ and a dihedral angle of $1.82(12)^\circ$ between the aromatic rings. In the crystal, weak $\text{C}-\text{H}\cdots\pi$ interactions lead to the formation of a three-dimensional network.

Related literature

For the biological activity of Schiff base ligands, see: Kelley *et al.* (1995); Pandeya *et al.* (1999); Singh & Dash (1988); Tarafder *et al.* (2002). For standard bond lengths, see: Allen *et al.* (1987). For related structures, see: Chantrapromma *et al.* (2010); Fun *et al.* (2010). For graph-set motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_2$
 $M_r = 268.31$
 Orthorhombic, $P2_12_12_1$
 $a = 6.0108(5)$ Å
 $b = 7.3394(5)$ Å
 $c = 31.674(2)$ Å
 $V = 1397.32(17)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 295$ K
 $0.22 \times 0.18 \times 0.16$ mm

Data collection

Bruker Kappa APEXII diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.982$, $T_{\max} = 0.987$
 5699 measured reflections
 2952 independent reflections
 1780 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.154$
 $S = 1.02$
 2952 reflections
 185 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 and Cg2 are the centroids of the C1–C6 and C10–C15 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2A}\cdots\text{N2}$	0.82	1.91	2.635 (3)	146
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.93	2.646 (3)	145
$\text{C5}-\text{H5}\cdots\text{Cg1}^i$	0.93	2.84	3.519 (3)	130
$\text{C14}-\text{H14}\cdots\text{Cg2}^{ii}$	0.93	2.85	3.519 (3)	130

Symmetry codes: (i) $-x - 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x + \frac{1}{2}, -y + \frac{5}{2}, -z + 1$.

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: SHELXL97.

The authors acknowledge the Department of Chemistry, IIT Madras, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5296).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Bruker (2004). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Chantrapromma, S., Jansrisewangwong, P. & Fun, H.-K. (2010). *Acta Cryst.* **E66**, o2994–o2995.
 Fun, H.-K., Jansrisewangwong, P. & Chantrapromma, S. (2010). *Acta Cryst.* **E66**, o2401–o2402.
 Kelley, J. L., Linn, J. A., Bankston, D. D., Burchall, C. J., Soroko, F. E. & Cooper, B. R. (1995). *J. Med. Chem.* **38**, 3676–3679.
 Pandeya, S. N., Sriram, D., Nath, G. & De Clercq, E. (1999). *Pharm. Acta Helv.* **74**, 11–17.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

Singh, W. M. & Dash, B. C. (1988). *Pesticides*, **22**, 33–37.
Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.

Tarafder, M. T. H., Kasbollah, A., Saravanan, N., Crouse, K. A., Ali, A. M. & Khor, T. O. (2002). *J. Biochem. Mol. Biol. Biophys.* **6**, 85–91.

supporting information

Acta Cryst. (2013). E69, o1394–o1395 [doi:10.1107/S160053681302148X]

1,2-Bis(2-hydroxy-5-methylbenzylidene)hydrazine

B. Saravanan, A. Jayamani, N. Sengottuvelan, G. Chakkaravarthi and V. Manivannan

S1. Comment

Schiff base ligands exhibit anti-cancer, anti-fungal, anti-tumour and anti-HIV activities (Pandeya *et al.*, 1999; Singh & Dash, 1988; Kelley *et al.*, 1995; Tarafder *et al.*, 2002). In the molecular structure of the title compound (Fig. 1), the bond distances are within the normal range (Allen *et al.*, 1987) and are comparable with the related structures (Chantrapromma *et al.*, 2010; Fun *et al.*, 2010). In the molecule, two aromatic rings are almost co-planar, with a dihedral angle of $1.82 (12)^\circ$. The hydroxy groups form intramolecular O—H \cdots N hydrogen bonds (O1—H1 \cdots N1 and O2—H2A \cdots N2; Table 1) with S(6) graph-set motifs (Bernstein *et al.*, 1995). The crystal structure also exhibits weak intermolecular C—H \cdots π (Table 1) interactions which forms a three dimensional network.

S2. Experimental

The title compound was synthesized by mixing a solution (1:2 molar ratio) of hydrazine hydrate (0.20 ml, 4 mmol) and 2-hydroxy-5-methylbenzaldehyde (1.08 g, 8 mmol) in ethanol (30 ml). The resulting solution was refluxed for 4 h, yielding (65%) the pale yellow crystalline solid. The resultant solid was filtered off and washed with methanol. Pale Yellow single crystals of the title compound suitable for X-ray structure determination were recrystallized from dimethylformamide by slow evaporation at room temperature over several days.

S3. Refinement

H atoms were positioned geometrically with C—H = 0.93–0.96 Å and O—H = 0.82 Å and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{O, methyl C})$.

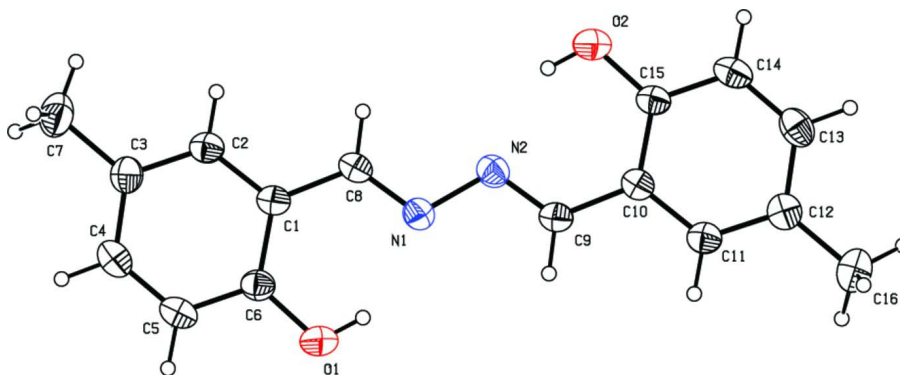


Figure 1

The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids for non-H atoms.

2-[[2-(2-Hydroxy-5-methylbenzylidene)hydrazin-1-ylidene]methyl]-4-methylphenol

Crystal data

$C_{16}H_{16}N_2O_2$	$F(000) = 568$
$M_r = 268.31$	$D_x = 1.275 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: P 2ac 2ab	Cell parameters from 2658 reflections
$a = 6.0108 (5) \text{ \AA}$	$\theta = 2.4\text{--}27.2^\circ$
$b = 7.3394 (5) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 31.674 (2) \text{ \AA}$	$T = 295 \text{ K}$
$V = 1397.32 (17) \text{ \AA}^3$	Block, yellow
$Z = 4$	$0.22 \times 0.18 \times 0.16 \text{ mm}$

Data collection

Bruker Kappa APEXII diffractometer	5699 measured reflections
Radiation source: fine-focus sealed tube	2952 independent reflections
Graphite monochromator	1780 reflections with $I > 2\sigma(I)$
ω and ϕ scans	$R_{\text{int}} = 0.023$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 27.2^\circ$, $\theta_{\text{min}} = 2.6^\circ$
$T_{\text{min}} = 0.982$, $T_{\text{max}} = 0.987$	$h = -6 \rightarrow 7$
	$k = -9 \rightarrow 9$
	$l = -40 \rightarrow 39$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.154$	$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
2952 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
185 parameters	$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.1393 (4)	0.8872 (3)	0.28106 (7)	0.0424 (6)
C2	-0.0535 (4)	0.9211 (3)	0.24097 (7)	0.0467 (6)
H2	0.0874	0.9726	0.2387	0.056*
C3	-0.1689 (5)	0.8812 (4)	0.20427 (8)	0.0528 (7)
C4	-0.3790 (5)	0.8051 (4)	0.20879 (8)	0.0558 (7)

H4	-0.4604	0.7765	0.1847	0.067*
C5	-0.4700 (5)	0.7708 (3)	0.24753 (9)	0.0540 (7)
H5	-0.6106	0.7185	0.2493	0.065*
C6	-0.3559 (4)	0.8129 (4)	0.28389 (8)	0.0474 (7)
C7	-0.0682 (6)	0.9219 (4)	0.16150 (7)	0.0766 (10)
H7A	-0.0947	0.8212	0.1428	0.115*
H7B	0.0891	0.9405	0.1645	0.115*
H7C	-0.1352	1.0299	0.1500	0.115*
C8	-0.0071 (4)	0.9275 (3)	0.31799 (7)	0.0454 (6)
H8	0.1363	0.9726	0.3145	0.055*
C9	-0.0019 (4)	0.9213 (3)	0.42464 (7)	0.0451 (6)
H9	-0.1451	0.8758	0.4282	0.054*
C10	0.1302 (5)	0.9612 (3)	0.46142 (7)	0.0429 (6)
C11	0.0395 (5)	0.9322 (3)	0.50158 (7)	0.0478 (7)
H11	-0.1016	0.8810	0.5035	0.057*
C12	0.1501 (5)	0.9762 (3)	0.53841 (8)	0.0527 (7)
C13	0.3625 (6)	1.0501 (4)	0.53396 (8)	0.0578 (8)
H13	0.4417	1.0814	0.5581	0.069*
C14	0.4593 (5)	1.0786 (4)	0.49519 (8)	0.0556 (7)
H14	0.6019	1.1271	0.4935	0.067*
C15	0.3451 (5)	1.0352 (3)	0.45871 (8)	0.0460 (6)
C16	0.0438 (6)	0.9530 (4)	0.58087 (8)	0.0718 (9)
H16A	-0.0435	1.0588	0.5874	0.108*
H16B	0.1573	0.9378	0.6019	0.108*
H16C	-0.0504	0.8474	0.5805	0.108*
N1	-0.0818 (3)	0.9027 (3)	0.35543 (6)	0.0498 (6)
N2	0.0716 (4)	0.9465 (3)	0.38727 (6)	0.0498 (6)
O1	-0.4551 (3)	0.7801 (3)	0.32150 (5)	0.0680 (6)
H1	-0.3751	0.8171	0.3406	0.102*
O2	0.4451 (3)	1.0657 (3)	0.42094 (6)	0.0650 (6)
H2A	0.3623	1.0328	0.4018	0.098*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0381 (15)	0.0357 (12)	0.0534 (14)	0.0023 (12)	-0.0003 (12)	0.0034 (10)
C2	0.0442 (15)	0.0392 (13)	0.0565 (16)	0.0000 (13)	-0.0007 (12)	0.0069 (11)
C3	0.0594 (19)	0.0491 (15)	0.0499 (15)	0.0053 (16)	-0.0034 (13)	0.0016 (12)
C4	0.0553 (19)	0.0488 (15)	0.0633 (17)	0.0011 (16)	-0.0152 (15)	-0.0051 (13)
C5	0.0412 (15)	0.0494 (14)	0.0715 (17)	-0.0039 (14)	-0.0070 (15)	-0.0016 (14)
C6	0.0417 (16)	0.0458 (14)	0.0548 (15)	0.0020 (14)	0.0002 (13)	0.0028 (11)
C7	0.102 (3)	0.076 (2)	0.0512 (16)	-0.006 (2)	-0.0028 (17)	0.0065 (14)
C8	0.0386 (16)	0.0415 (13)	0.0562 (15)	-0.0024 (13)	-0.0057 (12)	0.0003 (12)
C9	0.0395 (15)	0.0413 (13)	0.0546 (14)	-0.0010 (13)	0.0000 (12)	0.0035 (11)
C10	0.0412 (17)	0.0358 (12)	0.0518 (14)	0.0041 (12)	-0.0031 (12)	0.0002 (10)
C11	0.0465 (17)	0.0399 (13)	0.0570 (15)	0.0003 (13)	-0.0003 (13)	0.0042 (12)
C12	0.058 (2)	0.0425 (14)	0.0572 (16)	0.0059 (15)	-0.0035 (14)	0.0008 (12)
C13	0.061 (2)	0.0471 (15)	0.0656 (18)	0.0036 (16)	-0.0186 (15)	-0.0005 (13)

C14	0.0401 (17)	0.0508 (15)	0.0759 (19)	-0.0037 (15)	-0.0086 (14)	0.0047 (14)
C15	0.0379 (16)	0.0436 (14)	0.0564 (15)	-0.0008 (13)	-0.0007 (13)	0.0036 (12)
C16	0.091 (3)	0.0697 (18)	0.0550 (16)	0.005 (2)	0.0022 (17)	0.0006 (14)
N1	0.0458 (13)	0.0551 (13)	0.0485 (11)	-0.0018 (12)	-0.0044 (10)	0.0023 (10)
N2	0.0476 (13)	0.0490 (12)	0.0527 (11)	-0.0004 (11)	-0.0053 (11)	0.0021 (9)
O1	0.0460 (12)	0.0922 (16)	0.0658 (12)	-0.0134 (12)	0.0067 (10)	0.0055 (12)
O2	0.0490 (12)	0.0778 (14)	0.0683 (11)	-0.0112 (12)	0.0037 (10)	0.0053 (11)

Geometric parameters (Å, °)

C1—C2	1.393 (3)	C9—H9	0.9300
C1—C6	1.415 (3)	C10—C11	1.400 (3)
C1—C8	1.445 (3)	C10—C15	1.404 (3)
C2—C3	1.385 (3)	C11—C12	1.381 (3)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.388 (4)	C12—C13	1.395 (4)
C3—C7	1.514 (3)	C12—C16	1.499 (3)
C4—C5	1.367 (4)	C13—C14	1.375 (4)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.376 (3)	C14—C15	1.381 (3)
C5—H5	0.9300	C14—H14	0.9300
C6—O1	1.354 (3)	C15—O2	1.357 (3)
C7—H7A	0.9600	C16—H16A	0.9600
C7—H7B	0.9600	C16—H16B	0.9600
C7—H7C	0.9600	C16—H16C	0.9600
C8—N1	1.281 (3)	N1—N2	1.404 (3)
C8—H8	0.9300	O1—H1	0.8200
C9—N2	1.277 (3)	O2—H2A	0.8200
C9—C10	1.440 (3)		
C2—C1—C6	117.9 (2)	C10—C9—H9	119.0
C2—C1—C8	119.9 (2)	C11—C10—C15	118.2 (2)
C6—C1—C8	122.3 (2)	C11—C10—C9	119.3 (3)
C3—C2—C1	122.8 (2)	C15—C10—C9	122.5 (2)
C3—C2—H2	118.6	C12—C11—C10	123.0 (3)
C1—C2—H2	118.6	C12—C11—H11	118.5
C2—C3—C4	117.0 (2)	C10—C11—H11	118.5
C2—C3—C7	120.6 (3)	C11—C12—C13	116.5 (3)
C4—C3—C7	122.4 (3)	C11—C12—C16	121.8 (3)
C5—C4—C3	122.0 (3)	C13—C12—C16	121.7 (3)
C5—C4—H4	119.0	C14—C13—C12	122.5 (3)
C3—C4—H4	119.0	C14—C13—H13	118.8
C4—C5—C6	120.7 (3)	C12—C13—H13	118.8
C4—C5—H5	119.6	C13—C14—C15	120.1 (3)
C6—C5—H5	119.6	C13—C14—H14	119.9
O1—C6—C5	118.5 (2)	C15—C14—H14	119.9
O1—C6—C1	122.0 (2)	O2—C15—C14	118.6 (3)
C5—C6—C1	119.5 (2)	O2—C15—C10	121.7 (2)

C3—C7—H7A	109.5	C14—C15—C10	119.7 (2)
C3—C7—H7B	109.5	C12—C16—H16A	109.5
H7A—C7—H7B	109.5	C12—C16—H16B	109.5
C3—C7—H7C	109.5	H16A—C16—H16B	109.5
H7A—C7—H7C	109.5	C12—C16—H16C	109.5
H7B—C7—H7C	109.5	H16A—C16—H16C	109.5
N1—C8—C1	121.8 (2)	H16B—C16—H16C	109.5
N1—C8—H8	119.1	C8—N1—N2	113.7 (2)
C1—C8—H8	119.1	C9—N2—N1	113.9 (2)
N2—C9—C10	122.0 (2)	C6—O1—H1	109.5
N2—C9—H9	119.0	C15—O2—H2A	109.5
C6—C1—C2—C3	-1.5 (4)	C15—C10—C11—C12	1.3 (4)
C8—C1—C2—C3	178.2 (2)	C9—C10—C11—C12	-176.3 (2)
C1—C2—C3—C4	0.3 (4)	C10—C11—C12—C13	-0.9 (4)
C1—C2—C3—C7	179.6 (2)	C10—C11—C12—C16	176.6 (2)
C2—C3—C4—C5	0.2 (4)	C11—C12—C13—C14	0.0 (4)
C7—C3—C4—C5	-179.1 (3)	C16—C12—C13—C14	-177.6 (2)
C3—C4—C5—C6	0.7 (4)	C12—C13—C14—C15	0.6 (4)
C4—C5—C6—O1	178.5 (2)	C13—C14—C15—O2	179.7 (2)
C4—C5—C6—C1	-1.9 (4)	C13—C14—C15—C10	-0.3 (4)
C2—C1—C6—O1	-178.2 (2)	C11—C10—C15—O2	179.4 (2)
C8—C1—C6—O1	2.1 (4)	C9—C10—C15—O2	-3.1 (4)
C2—C1—C6—C5	2.3 (4)	C11—C10—C15—C14	-0.6 (3)
C8—C1—C6—C5	-177.4 (2)	C9—C10—C15—C14	176.9 (2)
C2—C1—C8—N1	178.2 (2)	C1—C8—N1—N2	179.0 (2)
C6—C1—C8—N1	-2.0 (4)	C10—C9—N2—N1	-179.07 (19)
N2—C9—C10—C11	179.4 (2)	C8—N1—N2—C9	-179.7 (2)
N2—C9—C10—C15	2.0 (4)		

Hydrogen-bond geometry (\AA , $^\circ$)

*Cg*1 and *Cg*2 are the centroids of the C1—C6 and C10—C15 rings, respectively.

<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>
O2—H2A...N2	0.82	1.91	2.635 (3)	146
O1—H1...N1	0.82	1.93	2.646 (3)	145
C5—H5... <i>Cg</i> 1 ⁱ	0.93	2.84	3.519 (3)	130
C14—H14... <i>Cg</i> 2 ⁱⁱ	0.93	2.85	3.519 (3)	130

Symmetry codes: (i) $-x-1, y-1/2, -z+1/2$; (ii) $x+1/2, -y+5/2, -z+1$.