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1,5-Bis[1-(2-hydroxyphenyl)ethylidene]- carbonohydrazide dimethylformamide monosolvate

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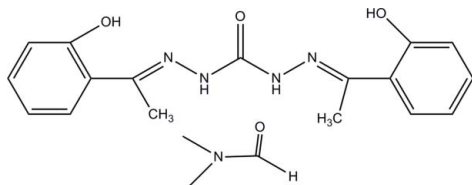
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;
R factor = 0.051; wR factor = 0.165; data-to-parameter ratio = 13.7.

In the title compound, $\text{C}_{17}\text{H}_{18}\text{N}_4\text{O}_3 \cdot \text{C}_3\text{H}_7\text{NO}$, the main disubstituted urea and solvate molecules are linked by pairs of $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds. In the main molecules, the benzene rings form a dihedral angle of 15.59 (13°) and two intramolecular $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds influence the molecular conformation. In the crystal structure, weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ interactions link the hydrogen-bonded pairs into chains along the b axis. The chains associate *via* $\text{C}-\text{H} \cdots \pi$ interactions.

Related literature

For a related structure, see: Zukerman-Schpector *et al.* (2009).
For the bioactivity of carbonohydrazide derivatives, see:
Loncle *et al.* (2004); Li *et al.* (2004).



Experimental

Crystal data

 $\text{C}_{17}\text{H}_{18}\text{N}_4\text{O}_3 \cdot \text{C}_3\text{H}_7\text{NO}$
 $M_r = 399.45$
Monoclinic, $P2_1/c$
 $a = 16.6372$ (15) Å
 $b = 7.5880$ (9) Å
 $c = 16.2967$ (14) Å

 $\beta = 94.472$ (1) $^\circ$
 $V = 2051.1$ (4) Å³
 $Z = 4$
Mo $K\alpha$ radiation

 $\mu = 0.09$ mm⁻¹
 $T = 298$ K
 $0.47 \times 0.46 \times 0.23$ mm

Data collection

 Bruker SMART APEX CCD area-
detector diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.958$, $T_{\max} = 0.979$

 10277 measured reflections
3596 independent reflections
1712 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.059$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.165$
 $S = 1.04$
3596 reflections

 263 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³
Table 1

 Hydrogen-bond geometry (Å, $^\circ$).

Cg is the centroid of the C12–C17 ring.

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N1}-\text{H1} \cdots \text{O4}$	0.86	2.02	2.805 (3)	151
$\text{N3}-\text{H3} \cdots \text{O4}$	0.86	2.09	2.858 (4)	148
$\text{O2}-\text{H2A} \cdots \text{N2}$	0.82	1.83	2.548 (3)	145
$\text{O3}-\text{H3A} \cdots \text{N4}$	0.82	1.83	2.546 (3)	145
$\text{C6}-\text{H6} \cdots \text{O1}^i$	0.93	2.57	3.241 (4)	129
$\text{C10}-\text{H10A} \cdots \text{Cg}^{ii}$	0.96	2.66	3.536 (4)	153

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; 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: CV2762).

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Zukerman-Schpector, J., Affan, M. A., Foo, S. W. & Tiekink, E. R. T. (2009). *Acta Cryst.* **E65**, o2951.

supporting information

Acta Cryst. (2010). E66, o2645 [doi:10.1107/S1600536810037517]

1,5-Bis[1-(2-hydroxyphenyl)ethylidene]carbonohydrazide dimethylformamide monosolvate

Lingyun Du and Wenkang Zhang

S1. Comment

Carbonohydrazide derivatives exhibit various bioactivities such as antibacteriale antifungal, anticonvulsant and anticancer activities (Loncle *et al.*, 2004; Li *et al.*, 2004). Herewith we present the crystal structure of the title compound (I), which is a new carbonohydrazide derivative.

Crystals of (I) comprise equal quantities of a disubstituted urea molecule (M) and a solvent *N,N*-dimethylformamide molecule (Fig. 1). The bond lengths and angles of the title compound are normal and correspond to those observed in *N''*,*N'''*-bis (1-(2-hydroxyphenyl)ethylidene)carbonohydrazide dimethyl sulfoxide solvate (Zukerman-Schpector *et al.*, 2009). The molecular conformation of M is influenced by two intramolecular O—H \cdots N hydrogen bonds (Table 1). Two benzene rings - C4-C9 and C12-C17, respectively - form a dihedral angle of 15.59 (13) $^\circ$.

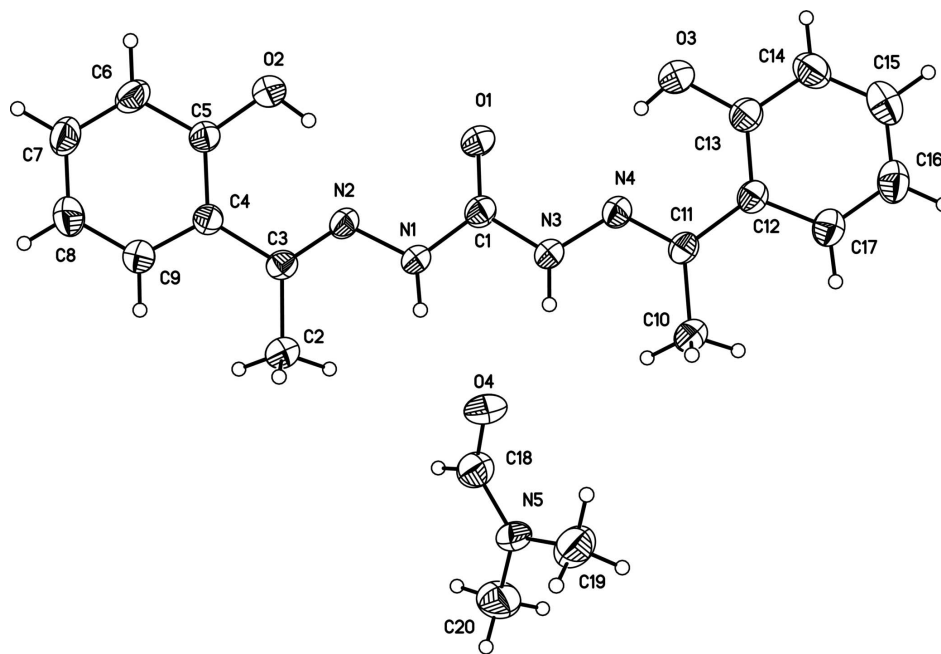
In the crystal structure, one M molecule and solvate molecule are paired *via* N—H \cdots O hydrogen bonds (Table 1). Weak intermolecular C—H \cdots O interactions (Table 1) link hydrogen-bonded pairs into chains along the *b* axis. The chains associate *via* C—H \cdots π interactions (Table 1).

S2. Experimental

2-Hydroxyacetophenone (10.0 mmol) and carbohydrazide (5.0 mmol) were mixed in 50 ml flash After stirring 3 h at 373 K, the resulting mixture was cooled to room temperature, and recrystallized from DMF, and afforded the title compound as a crystalline solid. Elemental analysis: calculated for C₂₀H₂₅N₅O₄: C 60.14, H 6.31, N 17.53%; found: C 60.23, H 6.45, N 17.64%.

S3. Refinement

All H atoms were placed in geometrically idealized positions (N—H 0.86 and C—H = 0.93–0.96 Å, O—H = 0.82 Å) and treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}$ of the parent atom.

**Figure 1**

The content of asymmetric unit of the title compound showing the atomic numbering scheme and 30% probability displacement ellipsoids.

1,5-Bis[1-(2-hydroxyphenyl)ethylidene]carbonohydrazide dimethylformamide monosolvate

Crystal data

$C_{17}H_{18}N_4O_3 \cdot C_3H_7NO$

$M_r = 399.45$

Monoclinic, $P2_1/c$

$a = 16.6372$ (15) Å

$b = 7.5880$ (9) Å

$c = 16.2967$ (14) Å

$\beta = 94.472$ (1)°

$V = 2051.1$ (4) Å³

$Z = 4$

$F(000) = 848$

$D_x = 1.294$ Mg m⁻³

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

Cell parameters from 1764 reflections

$\theta = 2.5$ – 21.7 °

$\mu = 0.09$ mm⁻¹

$T = 298$ K

Block, colourless

$0.47 \times 0.46 \times 0.23$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.958$, $T_{\max} = 0.979$

10277 measured reflections

3596 independent reflections

1712 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.059$

$\theta_{\text{max}} = 25.0$ °, $\theta_{\text{min}} = 2.5$ °

$h = -13 \rightarrow 19$

$k = -9 \rightarrow 9$

$l = -19 \rightarrow 19$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.051$ $wR(F^2) = 0.165$ $S = 1.04$

3596 reflections

263 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.8303P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL*, $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0049 (10)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.15121 (15)	0.6195 (3)	0.48890 (15)	0.0541 (7)
H1	0.1563	0.6254	0.5417	0.065*
N2	0.08378 (15)	0.6805 (3)	0.44431 (15)	0.0491 (7)
N3	0.27455 (15)	0.4912 (4)	0.49563 (15)	0.0555 (8)
H3	0.2756	0.5015	0.5483	0.067*
N4	0.33674 (15)	0.4171 (3)	0.45841 (15)	0.0510 (7)
N5	0.23936 (17)	0.5436 (4)	0.78637 (16)	0.0602 (8)
O1	0.20609 (13)	0.5357 (3)	0.37093 (14)	0.0717 (8)
O2	0.02424 (14)	0.7754 (4)	0.30242 (13)	0.0772 (8)
H2A	0.0592	0.7362	0.3357	0.116*
O3	0.38946 (14)	0.3282 (4)	0.32185 (14)	0.0826 (8)
H3A	0.3577	0.3729	0.3518	0.124*
O4	0.22303 (16)	0.6047 (4)	0.65032 (15)	0.0825 (9)
C1	0.20997 (19)	0.5488 (4)	0.4450 (2)	0.0511 (9)
C2	0.0191 (2)	0.7253 (5)	0.57328 (18)	0.0606 (10)
H2B	0.0483	0.6243	0.5950	0.091*
H2C	-0.0361	0.7172	0.5863	0.091*
H2D	0.0428	0.8305	0.5973	0.091*
C3	0.02260 (19)	0.7313 (4)	0.48137 (18)	0.0445 (8)
C4	-0.04633 (18)	0.7964 (4)	0.42688 (19)	0.0455 (8)
C5	-0.04176 (19)	0.8178 (4)	0.3417 (2)	0.0516 (9)
C6	-0.1065 (2)	0.8871 (5)	0.2935 (2)	0.0620 (10)
H6	-0.1023	0.9033	0.2374	0.074*

C7	-0.1762 (2)	0.9319 (5)	0.3270 (2)	0.0706 (11)
H7	-0.2193	0.9779	0.2940	0.085*
C8	-0.1824 (2)	0.9086 (5)	0.4098 (3)	0.0792 (12)
H8	-0.2299	0.9381	0.4330	0.095*
C9	-0.1183 (2)	0.8417 (5)	0.4584 (2)	0.0634 (10)
H9	-0.1235	0.8264	0.5143	0.076*
C10	0.40492 (19)	0.3582 (5)	0.59426 (19)	0.0617 (10)
H10A	0.4233	0.4721	0.6132	0.093*
H10B	0.4426	0.2699	0.6148	0.093*
H10C	0.3531	0.3346	0.6139	0.093*
C11	0.39830 (18)	0.3552 (4)	0.50206 (19)	0.0470 (8)
C12	0.46219 (18)	0.2798 (4)	0.4551 (2)	0.0485 (8)
C13	0.4547 (2)	0.2668 (5)	0.3684 (2)	0.0588 (9)
C14	0.5149 (2)	0.1875 (5)	0.3275 (2)	0.0756 (12)
H14	0.5082	0.1747	0.2706	0.091*
C15	0.5839 (2)	0.1277 (5)	0.3690 (3)	0.0799 (12)
H15	0.6244	0.0772	0.3404	0.096*
C16	0.5933 (2)	0.1423 (5)	0.4528 (3)	0.0738 (11)
H16	0.6404	0.1021	0.4813	0.089*
C17	0.5336 (2)	0.2159 (4)	0.4947 (2)	0.0610 (10)
H17	0.5410	0.2237	0.5517	0.073*
C18	0.1982 (2)	0.5477 (5)	0.7144 (2)	0.0700 (11)
H18	0.1458	0.5041	0.7116	0.084*
C19	0.3198 (2)	0.6151 (6)	0.7959 (2)	0.0948 (14)
H19A	0.3380	0.6399	0.7427	0.142*
H19B	0.3553	0.5311	0.8239	0.142*
H19C	0.3195	0.7218	0.8275	0.142*
C20	0.2055 (2)	0.4702 (6)	0.8576 (2)	0.0931 (14)
H20A	0.1503	0.4381	0.8436	0.140*
H20B	0.2080	0.5561	0.9010	0.140*
H20C	0.2356	0.3676	0.8757	0.140*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0494 (16)	0.072 (2)	0.0391 (15)	0.0127 (15)	-0.0055 (13)	-0.0015 (14)
N2	0.0449 (16)	0.0560 (18)	0.0448 (16)	0.0048 (14)	-0.0071 (14)	-0.0012 (13)
N3	0.0466 (16)	0.077 (2)	0.0421 (15)	0.0101 (15)	-0.0048 (13)	-0.0037 (15)
N4	0.0432 (15)	0.0593 (18)	0.0493 (17)	0.0022 (14)	-0.0032 (13)	-0.0033 (14)
N5	0.0661 (19)	0.073 (2)	0.0406 (17)	0.0032 (17)	0.0014 (15)	0.0090 (15)
O1	0.0632 (15)	0.106 (2)	0.0436 (15)	0.0167 (14)	-0.0101 (12)	-0.0117 (14)
O2	0.0669 (16)	0.117 (2)	0.0473 (14)	0.0275 (15)	0.0014 (13)	0.0008 (14)
O3	0.0693 (17)	0.125 (2)	0.0517 (15)	0.0146 (16)	-0.0044 (13)	-0.0063 (15)
O4	0.099 (2)	0.107 (2)	0.0418 (15)	0.0096 (17)	0.0026 (14)	0.0109 (15)
C1	0.0455 (19)	0.058 (2)	0.048 (2)	-0.0022 (17)	-0.0055 (17)	-0.0060 (18)
C2	0.064 (2)	0.068 (2)	0.049 (2)	0.0049 (19)	-0.0020 (17)	0.0008 (18)
C3	0.049 (2)	0.042 (2)	0.0421 (19)	-0.0037 (16)	-0.0026 (16)	-0.0045 (15)
C4	0.0480 (19)	0.0426 (19)	0.045 (2)	0.0011 (16)	-0.0012 (16)	-0.0055 (16)

C5	0.051 (2)	0.058 (2)	0.044 (2)	0.0062 (17)	-0.0033 (17)	-0.0065 (17)
C6	0.067 (2)	0.068 (3)	0.048 (2)	0.007 (2)	-0.0143 (19)	-0.0001 (18)
C7	0.058 (2)	0.081 (3)	0.070 (3)	0.019 (2)	-0.015 (2)	-0.005 (2)
C8	0.053 (2)	0.103 (3)	0.080 (3)	0.022 (2)	0.004 (2)	-0.006 (3)
C9	0.057 (2)	0.078 (3)	0.055 (2)	0.011 (2)	0.0026 (19)	-0.001 (2)
C10	0.061 (2)	0.070 (2)	0.053 (2)	0.0082 (19)	-0.0060 (17)	0.0022 (19)
C11	0.0440 (19)	0.047 (2)	0.049 (2)	-0.0037 (16)	-0.0047 (16)	-0.0020 (16)
C12	0.045 (2)	0.046 (2)	0.054 (2)	-0.0063 (16)	-0.0020 (17)	0.0011 (17)
C13	0.053 (2)	0.065 (2)	0.057 (2)	-0.0051 (19)	-0.0014 (19)	-0.0055 (19)
C14	0.070 (3)	0.093 (3)	0.066 (3)	-0.004 (2)	0.017 (2)	-0.012 (2)
C15	0.068 (3)	0.074 (3)	0.101 (4)	0.006 (2)	0.028 (3)	-0.004 (3)
C16	0.055 (2)	0.072 (3)	0.094 (3)	0.012 (2)	0.004 (2)	0.011 (2)
C17	0.055 (2)	0.058 (2)	0.070 (2)	0.0031 (19)	-0.001 (2)	0.0056 (19)
C18	0.070 (3)	0.075 (3)	0.064 (3)	0.000 (2)	-0.001 (2)	-0.001 (2)
C19	0.072 (3)	0.132 (4)	0.078 (3)	-0.003 (3)	-0.009 (2)	0.010 (3)
C20	0.116 (3)	0.100 (3)	0.066 (3)	0.013 (3)	0.024 (3)	0.025 (2)

Geometric parameters (Å, °)

N1—C1	1.365 (4)	C7—C8	1.372 (5)
N1—N2	1.369 (3)	C7—H7	0.9300
N1—H1	0.8600	C8—C9	1.375 (5)
N2—C3	1.283 (4)	C8—H8	0.9302
N3—N4	1.361 (3)	C9—H9	0.9300
N3—C1	1.374 (4)	C10—C11	1.498 (4)
N3—H3	0.8600	C10—H10A	0.9600
N4—C11	1.289 (4)	C10—H10B	0.9600
N5—C18	1.311 (4)	C10—H10C	0.9600
N5—C19	1.440 (4)	C11—C12	1.473 (4)
N5—C20	1.441 (4)	C12—C17	1.394 (4)
O1—C1	1.208 (3)	C12—C13	1.411 (4)
O2—C5	1.352 (3)	C13—C14	1.384 (5)
O2—H2A	0.8200	C14—C15	1.364 (5)
O3—C13	1.358 (4)	C14—H14	0.9300
O3—H3A	0.8200	C15—C16	1.367 (5)
O4—C18	1.232 (4)	C15—H15	0.9299
C2—C3	1.504 (4)	C16—C17	1.368 (5)
C2—H2B	0.9600	C16—H16	0.9300
C2—H2C	0.9600	C17—H17	0.9300
C2—H2D	0.9600	C18—H18	0.9300
C3—C4	1.480 (4)	C19—H19A	0.9600
C4—C9	1.382 (4)	C19—H19B	0.9600
C4—C5	1.405 (4)	C19—H19C	0.9600
C5—C6	1.387 (4)	C20—H20A	0.9600
C6—C7	1.363 (5)	C20—H20B	0.9600
C6—H6	0.9300	C20—H20C	0.9600
C1—N1—N2	116.4 (3)	C11—C10—H10A	109.5

C1—N1—H1	121.8	C11—C10—H10B	109.5
N2—N1—H1	121.8	H10A—C10—H10B	109.5
C3—N2—N1	120.0 (3)	C11—C10—H10C	109.5
N4—N3—C1	116.7 (3)	H10A—C10—H10C	109.5
N4—N3—H3	121.6	H10B—C10—H10C	109.5
C1—N3—H3	121.6	N4—C11—C12	115.4 (3)
C11—N4—N3	120.2 (3)	N4—C11—C10	122.7 (3)
C18—N5—C19	120.2 (3)	C12—C11—C10	121.8 (3)
C18—N5—C20	121.3 (3)	C17—C12—C13	116.4 (3)
C19—N5—C20	118.5 (3)	C17—C12—C11	121.2 (3)
C5—O2—H2A	109.5	C13—C12—C11	122.4 (3)
C13—O3—H3A	109.5	O3—C13—C14	117.2 (3)
O1—C1—N1	124.8 (3)	O3—C13—C12	122.7 (3)
O1—C1—N3	123.5 (3)	C14—C13—C12	120.1 (3)
N1—C1—N3	111.6 (3)	C15—C14—C13	121.2 (4)
C3—C2—H2B	109.5	C15—C14—H14	119.4
C3—C2—H2C	109.5	C13—C14—H14	119.4
H2B—C2—H2C	109.5	C14—C15—C16	119.7 (4)
C3—C2—H2D	109.5	C14—C15—CG1	59.7 (2)
H2B—C2—H2D	109.5	C14—C15—H15	120.2
H2C—C2—H2D	109.5	C16—C15—H15	120.2
N2—C3—C4	115.1 (3)	C15—C16—C17	120.0 (4)
N2—C3—C2	123.6 (3)	C15—C16—H16	120.0
C4—C3—C2	121.3 (3)	C17—C16—H16	120.0
C9—C4—C5	116.9 (3)	C16—C17—C12	122.5 (4)
C9—C4—C3	120.9 (3)	C16—C17—H17	118.8
C5—C4—C3	122.2 (3)	C12—C17—H17	118.8
O2—C5—C6	116.4 (3)	O4—C18—N5	125.4 (4)
O2—C5—C4	123.3 (3)	O4—C18—H18	117.3
C6—C5—C4	120.3 (3)	N5—C18—H18	117.3
C7—C6—C5	121.0 (3)	N5—C19—H19A	109.5
C7—C6—H6	119.5	N5—C19—H19B	109.5
C5—C6—H6	119.5	H19A—C19—H19B	109.5
C6—C7—C8	119.6 (3)	N5—C19—H19C	109.5
C6—C7—H7	120.2	H19A—C19—H19C	109.5
C8—C7—H7	120.2	H19B—C19—H19C	109.5
C7—C8—C9	120.0 (4)	N5—C20—H20A	109.5
C7—C8—H8	120.0	N5—C20—H20B	109.5
C9—C8—H8	120.0	H20A—C20—H20B	109.5
C8—C9—C4	122.2 (3)	N5—C20—H20C	109.5
C8—C9—H9	118.9	H20A—C20—H20C	109.5
C4—C9—H9	118.9	H20B—C20—H20C	109.5

Hydrogen-bond geometry (Å, °)

Cg is the centroid of the C12–C17 ring.

<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>
N1—H1···O4	0.86	2.02	2.805 (3)	151

N3—H3···O4	0.86	2.09	2.858 (4)	148
O2—H2A···N2	0.82	1.83	2.548 (3)	145
O3—H3A···N4	0.82	1.83	2.546 (3)	145
C6—H6···O1 ⁱ	0.93	2.57	3.241 (4)	129
C10—H10A···Cg ⁱⁱ	0.96	2.66	3.536 (4)	153

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