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N′-[(1*E*)-(3,5-Dichloro-2-hydroxyphenyl)(phenyl)methylene]-4-methoxybenzohydrazide

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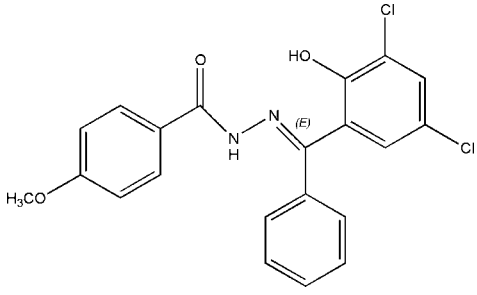
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.005 Å; R factor = 0.054; wR factor = 0.207; data-to-parameter ratio = 13.6.

The title compound, $C_{21}H_{15}Cl_2N_2O_3$, displays a *trans* configuration with respect to the C=N double bond. The molecular conformation is stabilized by an intramolecular O—H···N hydrogen bond.

Related literature

For related compounds, see: Salem (1998); Chang & Ji (2007).



Experimental

Crystal data

$C_{21}H_{15}Cl_2N_2O_3$
M_r = 414.25
Triclinic, $P\overline{1}$
a = 8.9814 (9) Å
b = 10.8867 (11) Å
c = 11.5291 (13) Å
α = 89.623 (2)°
β = 72.700 (1)°
γ = 66.947 (2)°
V = 982.57 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 273 (2) K
0.18 × 0.15 × 0.10 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.949, T_{max} = 0.965
5254 measured reflections
3459 independent reflections
2262 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.207
S = 1.00
3459 reflections
255 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2	0.82	1.82	2.528 (3)	145

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: GD2026).

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supporting information

Acta Cryst. (2008). E**64**, o198 [https://doi.org/10.1107/S1600536807064069]

N′-[(1*E*)-(3,5-Dichloro-2-hydroxyphenyl)(phenyl)methylene]-4-methoxybenzohydrazide

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S1. Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions and their biological activity (Salem, 1998; Chang *et al.*, 2007).As an extension of work on the structural characterization of aroylhydrazone derivatives,the title compound, (I),was synthesized and its crystal structure is reported here.

The title molecule displays a *trans* conformation with respect to the C7=N2 double bond (Fig. 1). The three benzene rings, C1–C6 (A), C9–C14 (B) and C16–C21 (C) make dihedral angles of 10.69 (15)(A/B) °, 79.64 (13) (B/C) °, 73.13 (12)(A/C) °. The molecular conformation is stabilized by intramolecular O—H···N hydrogen bond.(Table 1.) .

S2. Experimental

4-methoxybenzohydrazide (0.01 mol,1.66 g) was dissolved in anhydrous ethanol (50 ml), and (3,5-dichloro-2-hydroxyphenyl)(phenyl)methanone (0.01 mol, 2.67 g) was added. The reaction mixture was refluxed for 6 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 78%). The compound (1.0 mmol,0.41 g) was dissolved in dimethylformamide (30 ml) and kept at room temperature for 20 d to obtain colourless single crystals suitable for X-ray diffraction.

S3. Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms,*C*—*H*(methyl) = 0.96 Å,*C*—H(aromatic) = 0.93 Å, O—H = 0.82 Å, and N—H =0.86 Å and with U_{iso}(H) =1.5*U*~eq~(*C*~methyl~,*O*) and 1.2U_{eq}($C_{aromatic}$,*N*).

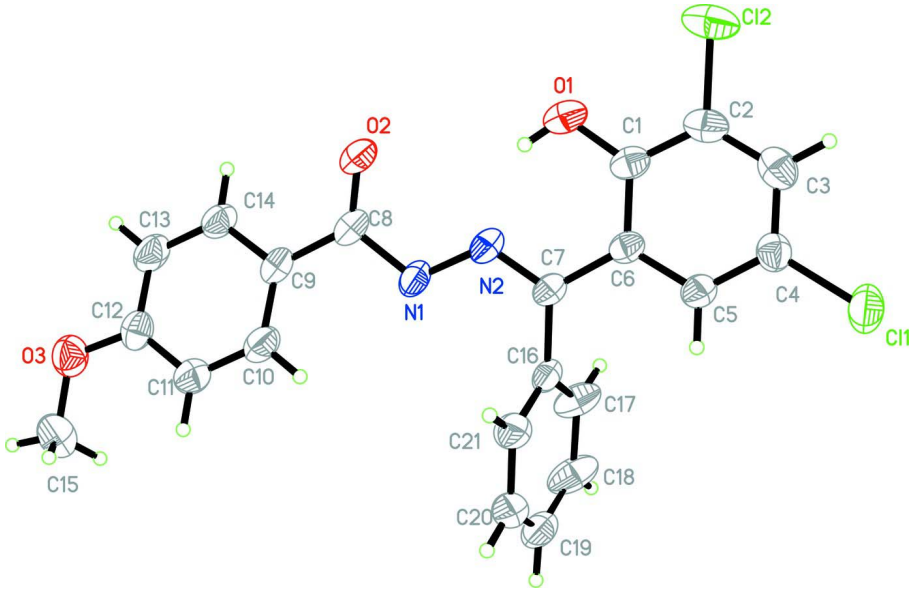

Figure 1

The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

N′-[(1E)-(3,5-Dichloro-2-hydroxyphenyl)(phenyl)methylene]-4- methoxybenzohydrazide

Crystal data

$C_{21}H_{15}Cl_2N_2O_3$
$M_r = 414.25$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
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$b = 10.8867\ (11)$ Å
$c = 11.5291\ (13)$ Å
$\alpha = 89.623\ (2)°$
$\beta = 72.700\ (1)°$
$\gamma = 66.947\ (2)°$
$V = 982.57\ (18)$ Å^3
$Z = 2$
$F(000) = 426$
$D_x = 1.400$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1372 reflections
$\theta = 2.6–23.0°$
$\mu = 0.36$ mm^{-1}
$T = 273$ K
Block, yellow
0.18 × 0.15 × 0.10 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.949$, $T_{max} = 0.965$
5254 measured reflections
3459 independent reflections
2262 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.9°$
$h = -10\rightarrow10$
$k = -12\rightarrow8$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.207$
$S = 1.00$
3459 reflections
255 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
$w = 1/[\sigma^2(F_o^2) + (0.135P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.51883 (14)	1.15784 (10)	0.47405 (9)	0.0944 (4)
Cl2	0.19492 (12)	1.40951 (10)	0.93386 (11)	0.1083 (5)
O1	0.4694 (3)	1.2048 (2)	0.99251 (19)	0.0763 (7)
H1	0.5549	1.1501	1.0051	0.115*
O2	0.7413 (3)	1.0462 (2)	1.16739 (19)	0.0795 (7)
O3	1.3151 (3)	0.5644 (3)	1.3043 (2)	0.0845 (7)
N1	0.8610 (3)	0.9057 (3)	0.9878 (2)	0.0634 (7)
N2	0.7483 (3)	0.9984 (2)	0.9403 (2)	0.0602 (7)
C1	0.4846 (4)	1.1863 (3)	0.8740 (3)	0.0598 (8)
C2	0.3621 (4)	1.2797 (3)	0.8308 (3)	0.0705 (9)
C3	0.3711 (4)	1.2716 (3)	0.7099 (4)	0.0758 (10)
H3	0.2875	1.3359	0.6838	0.091*
C4	0.5050 (4)	1.1674 (3)	0.6276 (3)	0.0651 (8)
C5	0.6262 (4)	1.0708 (3)	0.6671 (3)	0.0592 (8)
H5	0.7149	0.9998	0.6113	0.071*
C6	0.6180 (3)	1.0778 (3)	0.7901 (2)	0.0533 (7)
C7	0.7493 (4)	0.9727 (3)	0.8309 (2)	0.0530 (7)
C8	0.8492 (4)	0.9403 (3)	1.1074 (3)	0.0621 (8)
C9	0.9766 (4)	0.8393 (3)	1.1529 (3)	0.0590 (8)
C10	1.1299 (4)	0.7433 (4)	1.0769 (3)	0.0724 (9)
H10	1.1552	0.7415	0.9925	0.087*
C11	1.2451 (4)	0.6506 (4)	1.1240 (3)	0.0740 (9)
H11	1.3464	0.5863	1.0713	0.089*
C12	1.2110 (4)	0.6527 (3)	1.2492 (3)	0.0659 (8)
C13	1.0590 (5)	0.7504 (4)	1.3255 (3)	0.0768 (10)
H13	1.0346	0.7532	1.4099	0.092*
C14	0.9449 (4)	0.8425 (3)	1.2786 (3)	0.0682 (9)
H14	0.8449	0.9080	1.3314	0.082*
C15	1.4627 (5)	0.4539 (4)	1.2293 (4)	0.0973 (12)

H15A	1.4296	0.4067	1.1785	0.146*
H15B	1.5162	0.3938	1.2804	0.146*
H15C	1.5416	0.4867	1.1787	0.146*
C16	0.8695 (4)	0.8437 (3)	0.7508 (2)	0.0533 (7)
C17	0.8065 (5)	0.7546 (4)	0.7229 (3)	0.0823 (11)
H17	0.6896	0.7767	0.7513	0.099*
C18	0.9169 (7)	0.6327 (4)	0.6528 (3)	0.0980 (14)
H18	0.8745	0.5722	0.6352	0.118*
C19	1.0902 (6)	0.6004 (4)	0.6088 (3)	0.0872 (12)
H19	1.1650	0.5178	0.5623	0.105*
C20	1.1502 (5)	0.6900 (4)	0.6338 (3)	0.0888 (12)
H20	1.2664	0.6698	0.6020	0.107*
C21	1.0411 (4)	0.8108 (4)	0.7059 (3)	0.0732 (9)
H21	1.0846	0.8704	0.7240	0.088*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1206 (9)	0.0939 (7)	0.0808 (7)	−0.0388 (6)	−0.0560 (6)	0.0253 (5)
Cl2	0.0701 (7)	0.0784 (7)	0.1367 (10)	−0.0121 (5)	−0.0020 (6)	−0.0271 (6)
O1	0.0759 (15)	0.0805 (16)	0.0590 (14)	−0.0308 (12)	−0.0038 (10)	−0.0138 (11)
O2	0.0978 (18)	0.0806 (16)	0.0510 (13)	−0.0251 (14)	−0.0263 (12)	−0.0083 (12)
O3	0.0870 (17)	0.0971 (18)	0.0738 (15)	−0.0362 (15)	−0.0339 (13)	0.0216 (14)
N1	0.0846 (18)	0.0668 (16)	0.0454 (13)	−0.0317 (14)	−0.0290 (12)	0.0054 (11)
N2	0.0777 (17)	0.0639 (16)	0.0463 (13)	−0.0351 (14)	−0.0215 (12)	0.0012 (11)
C1	0.0614 (18)	0.0613 (18)	0.0589 (18)	−0.0339 (16)	−0.0098 (14)	−0.0040 (15)
C2	0.0571 (19)	0.0590 (19)	0.087 (2)	−0.0243 (16)	−0.0104 (17)	−0.0079 (17)
C3	0.071 (2)	0.066 (2)	0.101 (3)	−0.0302 (18)	−0.039 (2)	0.015 (2)
C4	0.070 (2)	0.0637 (19)	0.069 (2)	−0.0283 (17)	−0.0309 (16)	0.0112 (16)
C5	0.0643 (18)	0.0567 (18)	0.0554 (17)	−0.0244 (15)	−0.0179 (14)	−0.0001 (14)
C6	0.0551 (16)	0.0565 (17)	0.0498 (16)	−0.0276 (14)	−0.0122 (13)	−0.0019 (13)
C7	0.0636 (18)	0.0545 (17)	0.0455 (15)	−0.0302 (14)	−0.0154 (13)	0.0020 (12)
C8	0.080 (2)	0.073 (2)	0.0429 (16)	−0.0417 (19)	−0.0183 (15)	0.0023 (15)
C9	0.074 (2)	0.0704 (19)	0.0458 (16)	−0.0408 (17)	−0.0215 (14)	0.0058 (14)
C10	0.081 (2)	0.088 (2)	0.0477 (17)	−0.037 (2)	−0.0169 (16)	0.0012 (17)
C11	0.071 (2)	0.086 (2)	0.0569 (19)	−0.0280 (19)	−0.0154 (16)	0.0015 (17)
C12	0.074 (2)	0.080 (2)	0.0621 (19)	−0.0468 (18)	−0.0279 (16)	0.0175 (17)
C13	0.095 (3)	0.091 (3)	0.0427 (16)	−0.037 (2)	−0.0204 (17)	0.0117 (17)
C14	0.081 (2)	0.075 (2)	0.0438 (16)	−0.0306 (18)	−0.0156 (15)	0.0047 (15)
C15	0.078 (3)	0.105 (3)	0.107 (3)	−0.032 (2)	−0.035 (2)	0.025 (3)
C16	0.0698 (19)	0.0551 (17)	0.0394 (14)	−0.0264 (15)	−0.0221 (13)	0.0045 (12)
C17	0.103 (3)	0.083 (2)	0.062 (2)	−0.057 (2)	−0.0013 (18)	−0.0132 (18)
C18	0.154 (4)	0.075 (3)	0.064 (2)	−0.067 (3)	−0.005 (2)	−0.0078 (19)
C19	0.127 (4)	0.060 (2)	0.053 (2)	−0.013 (2)	−0.033 (2)	0.0035 (16)
C20	0.074 (2)	0.094 (3)	0.077 (2)	−0.006 (2)	−0.0326 (19)	−0.018 (2)
C21	0.068 (2)	0.079 (2)	0.072 (2)	−0.0252 (18)	−0.0255 (16)	−0.0107 (17)

Geometric parameters (Å, º)

Cl1—C4	1.738 (3)	C10—C11	1.376 (5)
Cl2—C2	1.722 (3)	C10—H10	0.9300
O1—C1	1.340 (4)	C11—C12	1.382 (5)
O1—H1	0.8200	C11—H11	0.9300
O2—C8	1.217 (4)	C12—C13	1.389 (5)
O3—C12	1.358 (4)	C13—C14	1.365 (5)
O3—C15	1.427 (4)	C13—H13	0.9300
N1—N2	1.366 (4)	C14—H14	0.9300
N1—C8	1.394 (4)	C15—H15A	0.9600
N2—C7	1.291 (3)	C15—H15B	0.9600
C1—C2	1.391 (5)	C15—H15C	0.9600
C1—C6	1.408 (4)	C16—C21	1.367 (4)
C2—C3	1.374 (5)	C16—C17	1.381 (4)
C3—C4	1.378 (4)	C17—C18	1.380 (5)
C3—H3	0.9300	C17—H17	0.9300
C4—C5	1.375 (4)	C18—C19	1.381 (5)
C5—C6	1.399 (4)	C18—H18	0.9300
C5—H5	0.9300	C19—C20	1.354 (5)
C6—C7	1.479 (4)	C19—H19	0.9300
C7—C16	1.492 (4)	C20—C21	1.379 (5)
C8—C9	1.469 (5)	C20—H20	0.9300
C9—C10	1.387 (4)	C21—H21	0.9300
C9—C14	1.390 (4)		
C1—O1—H1	109.5	C10—C11—H11	119.9
C12—O3—C15	118.3 (3)	C12—C11—H11	119.9
N2—N1—C8	116.5 (3)	O3—C12—C11	124.7 (3)
C7—N2—N1	120.2 (2)	O3—C12—C13	116.8 (3)
O1—C1—C2	118.3 (3)	C11—C12—C13	118.5 (3)
O1—C1—C6	123.8 (3)	C14—C13—C12	121.1 (3)
C2—C1—C6	117.8 (3)	C14—C13—H13	119.4
C3—C2—C1	122.3 (3)	C12—C13—H13	119.4
C3—C2—Cl2	119.6 (3)	C13—C14—C9	120.7 (3)
C1—C2—Cl2	118.1 (3)	C13—C14—H14	119.6
C2—C3—C4	119.5 (3)	C9—C14—H14	119.6
C2—C3—H3	120.3	O3—C15—H15A	109.5
C4—C3—H3	120.3	O3—C15—H15B	109.5
C5—C4—C3	120.1 (3)	H15A—C15—H15B	109.5
C5—C4—Cl1	120.2 (3)	O3—C15—H15C	109.5
C3—C4—Cl1	119.8 (3)	H15A—C15—H15C	109.5
C4—C5—C6	120.9 (3)	H15B—C15—H15C	109.5
C4—C5—H5	119.5	C21—C16—C17	119.2 (3)
C6—C5—H5	119.5	C21—C16—C7	121.4 (3)
C5—C6—C1	119.3 (3)	C17—C16—C7	119.4 (3)
C5—C6—C7	120.1 (3)	C18—C17—C16	120.0 (4)
C1—C6—C7	120.6 (3)	C18—C17—H17	120.0

N2—C7—C6	115.7 (2)	C16—C17—H17	120.0
N2—C7—C16	123.0 (3)	C17—C18—C19	120.2 (4)
C6—C7—C16	121.2 (2)	C17—C18—H18	119.9
O2—C8—N1	121.7 (3)	C19—C18—H18	119.9
O2—C8—C9	123.9 (3)	C20—C19—C18	119.4 (3)
N1—C8—C9	114.4 (3)	C20—C19—H19	120.3
C10—C9—C14	118.0 (3)	C18—C19—H19	120.3
C10—C9—C8	123.5 (3)	C19—C20—C21	120.8 (4)
C14—C9—C8	118.5 (3)	C19—C20—H20	119.6
C11—C10—C9	121.3 (3)	C21—C20—H20	119.6
C11—C10—H10	119.3	C16—C21—C20	120.4 (3)
C9—C10—H10	119.3	C16—C21—H21	119.8
C10—C11—C12	120.3 (3)	C20—C21—H21	119.8

C8—N1—N2—C7	−178.8 (2)	O2—C8—C9—C14	19.9 (5)
O1—C1—C2—C3	177.5 (3)	N1—C8—C9—C14	−160.2 (3)
C6—C1—C2—C3	−2.3 (5)	C14—C9—C10—C11	2.2 (5)
O1—C1—C2—Cl2	−2.2 (4)	C8—C9—C10—C11	−179.9 (3)
C6—C1—C2—Cl2	177.9 (2)	C9—C10—C11—C12	−0.9 (5)
C1—C2—C3—C4	0.3 (5)	C15—O3—C12—C11	−6.0 (5)
Cl2—C2—C3—C4	−179.9 (2)	C15—O3—C12—C13	173.2 (3)
C2—C3—C4—C5	1.5 (5)	C10—C11—C12—O3	178.8 (3)
C2—C3—C4—Cl1	−179.1 (2)	C10—C11—C12—C13	−0.3 (5)
C3—C4—C5—C6	−1.3 (5)	O3—C12—C13—C14	−179.0 (3)
Cl1—C4—C5—C6	179.3 (2)	C11—C12—C13—C14	0.2 (5)
C4—C5—C6—C1	−0.7 (4)	C12—C13—C14—C9	1.1 (6)
C4—C5—C6—C7	179.8 (3)	C10—C9—C14—C13	−2.3 (5)
O1—C1—C6—C5	−177.4 (3)	C8—C9—C14—C13	179.7 (3)
C2—C1—C6—C5	2.5 (4)	N2—C7—C16—C21	−67.8 (4)
O1—C1—C6—C7	2.1 (4)	C6—C7—C16—C21	114.8 (3)
C2—C1—C6—C7	−178.1 (3)	N2—C7—C16—C17	111.3 (3)
N1—N2—C7—C6	177.9 (2)	C6—C7—C16—C17	−66.2 (4)
N1—N2—C7—C16	0.3 (4)	C21—C16—C17—C18	1.5 (5)
C5—C6—C7—N2	169.1 (2)	C7—C16—C17—C18	−177.6 (3)
C1—C6—C7—N2	−10.4 (4)	C16—C17—C18—C19	−1.1 (6)
C5—C6—C7—C16	−13.3 (4)	C17—C18—C19—C20	−0.8 (6)
C1—C6—C7—C16	167.2 (2)	C18—C19—C20—C21	2.1 (6)
N2—N1—C8—O2	1.7 (4)	C17—C16—C21—C20	−0.2 (5)
N2—N1—C8—C9	−178.2 (2)	C7—C16—C21—C20	178.9 (3)
O2—C8—C9—C10	−157.9 (3)	C19—C20—C21—C16	−1.7 (5)
N1—C8—C9—C10	22.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2	0.82	1.82	2.528 (3)	145