Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Hydroxy-3-methoxybenzaldehyde 2,4-dinitrophenylhydrazone pyridine monosolvate

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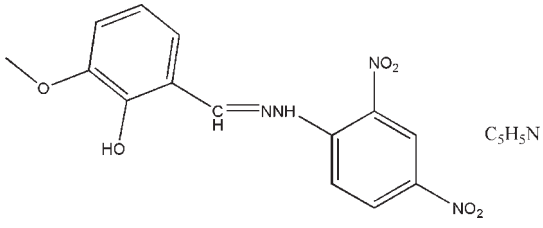
Received 25 July 2010; accepted 28 July 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.143; data-to-parameter ratio = 16.2.

The Schiff base molecule of the title compound, $C_{14}H_{12}N_4O_6\cdot C_5H_5N$, was obtained from the condensation reaction of 2-hydroxy-3-methoxybenzaldehyde and 2,4-dinitrophenylhydrazine. The C=N bond of the Schiff base has a *trans* arrangement and the dihedral angle between the two benzene rings is 3.49 (10)°. An intramolecular N—H···O hydrogen bond generates an S(6) ring. In the crystal, O—H···O hydrogen bonds link the Schiff base molecules.

Related literature

For background to Schiff bases, see: Kahwa *et al.* (1986); Santos *et al.* (2001). For a related structure, see: Ohba (1996).



Experimental

Crystal data

$C_{14}H_{12}N_4O_6\cdot C_5H_5N$ M_r = 411.38
Triclinic, $P\overline{1}$
a = 6.9020 (18) Å
b = 7.6240 (12) Å
c = 19.073 (3) Å
α = 95.112 (13)°
β = 91.199 (17)°
γ = 107.024 (19)°
V = 954.7 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.21 × 0.19 × 0.17 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.973, T_{max} = 0.978
7517 measured reflections
4401 independent reflections
1852 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.143
S = 0.82
4401 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1B···O4^{i}	0.82	2.53	3.319 (2)	162
N2—H2A···O3	0.86	2.03	2.635 (2)	126

Symmetry code: (i) $-x+1, -y-1, -z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5577).

References

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Ohba, S. (1996). *Acta Cryst.* C**52**, 2118–2119.
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supporting information

Acta Cryst. (2010). E**66**, o2204 [https://doi.org/10.1107/S1600536810029971]

2-Hydroxy-3-methoxybenzaldehyde 2,4-dinitrophenylhydrazone pyridine monosolvate

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S1. Comment

The chemistry of Schiff base has attracted a great deal of interest in recent years. These compounds play an important role in the development of various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of our in the study of the coordination chemistry of Schiff bases, we synthesized the title compound, (I), and determined its crystal structure.

The molecular structure of (I) is shown in Fig.1. The benzene ring and the 2,4-dinitro benzene ring are nearly coplanar, making a dihedral angle of 3.49 (10)°. The dinitro group is coplanar with C9-benzene ring with a dihedral angle of 0.58 (8). Bond lengths and bond angles agree with those of other dinitrophenylhydrazone derivatives (Ohba, 1996).

Intramolecular N—H···O and intermolecular O—H···O hydrogen bonds are certainly responsible for the planar conformation of the molecule.

S2. Experimental

2,4-Dinitrophenylhydrazine (1 mmol, 0.198 g) was dissolved in anhydrous ethanol (15 ml), H_2SO_4(98%, 0.5 ml) was then added and the mixture was stirred for several minitutes at 351 K. Then, 2-hydroxy-3-methoxybenzaldehyde (1 mmol, 0.152 g) in ethanol (8 mm l) was added dropwise and the mixture was stirred at refluxing temperature for 3 h. The product was isolated and recrystallized from pyridine, red blocks of (I) were obtained after two weeks.

S3. Refinement

All H atoms were positioned geometrically and refined as riding with C—H = 0.93 (aromatic), 0.96 Å(methyl) and N—H = 0.86 Å, with U_{iso}(H) = 1.2U_{eq}(CH, CH2 or NH) and U_{iso}(H) = 1.5U_{eq}(C).

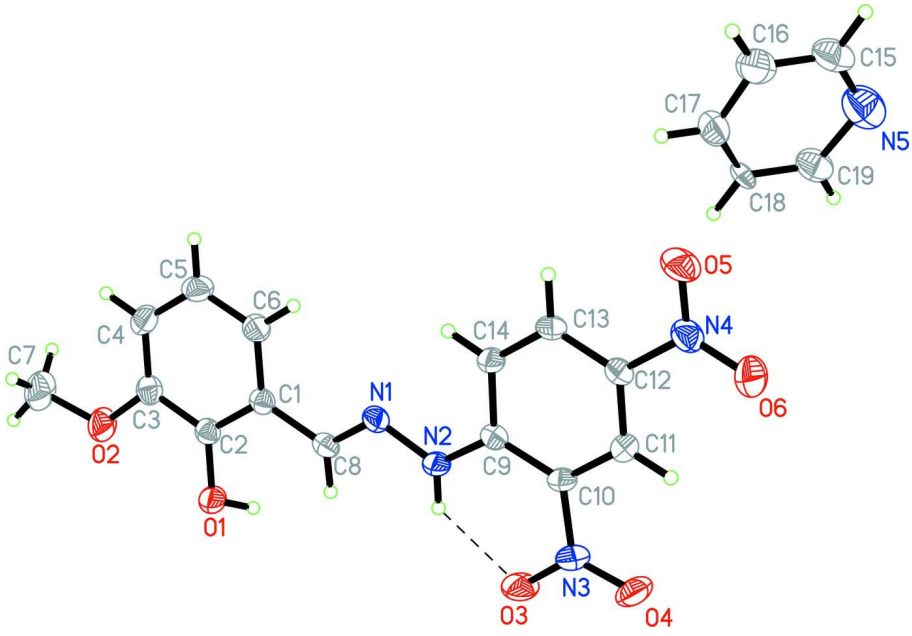

Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 30% probability level. The intramolecular hydrogen bond is indicatd with dashed lines.

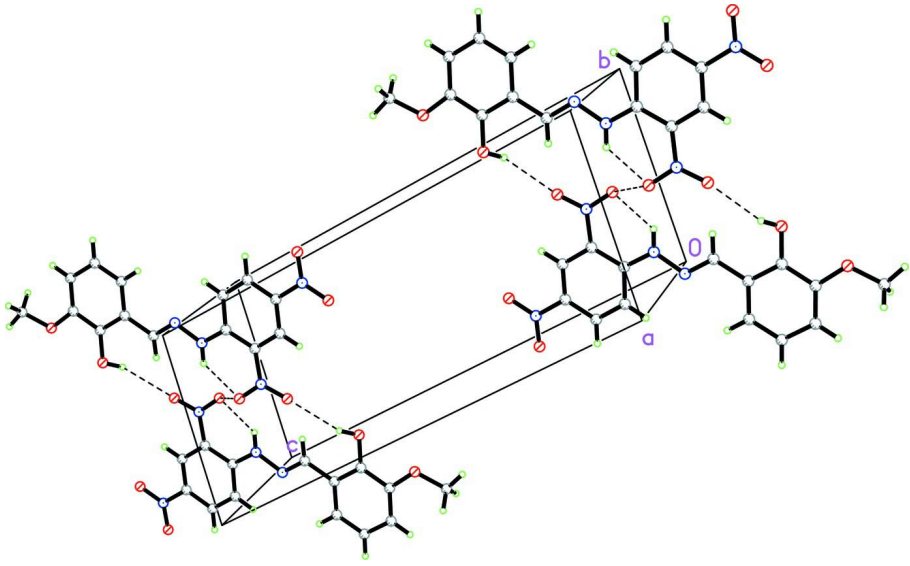

Figure 2

The partial packing of (I).

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γ = 107.024 (19)°
V = 954.7 (3) Å^3
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F(000) = 418
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Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 1741 reflections
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Graphite monochromator
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7517 measured reflections
4401 independent reflections
1852 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 29.3°, θ_{min} = 3.2°
h = −8→9
k = −10→8
l = −23→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.143
S = 0.82
4401 reflections
271 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
$w = 1/[\sigma^2(F_o^2) + (0.078P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.7466 (3)	0.0112 (2)	0.01896 (10)	0.0424 (5)
N3	0.6500 (3)	−0.4948 (2)	−0.11703 (12)	0.0485 (5)
O1	0.7209 (2)	0.00916 (18)	0.23110 (8)	0.0549 (5)
H1B	0.6315	−0.0690	0.2069	0.082*
C1	0.7623 (3)	0.1767 (3)	0.13134 (12)	0.0390 (5)
N2	0.7090 (3)	−0.1535 (2)	−0.02176 (10)	0.0429 (5)
H2A	0.6742	−0.2561	−0.0030	0.051*
C2	0.7567 (3)	0.1720 (3)	0.20412 (13)	0.0412 (6)
O3	0.6370 (3)	−0.5111 (2)	−0.05341 (11)	0.0677 (5)
C12	0.7671 (3)	−0.1328 (3)	−0.23637 (12)	0.0386 (5)

O2	0.7845 (3)	0.3159 (2)	0.31832 (9)	0.0670 (5)
C8	0.7250 (3)	0.0051 (3)	0.08483 (13)	0.0399 (5)
H8A	0.6856	−0.1080	0.1035	0.048*
C10	0.7001 (3)	−0.3115 (2)	−0.13946 (12)	0.0367 (5)
C3	0.7921 (3)	0.3370 (3)	0.24820 (13)	0.0476 (6)
C6	0.8044 (3)	0.3474 (3)	0.10356 (13)	0.0477 (6)
H6A	0.8102	0.3526	0.0551	0.057*
C14	0.7760 (3)	0.0185 (3)	−0.12116 (12)	0.0400 (5)
H14A	0.7952	0.1268	−0.0918	0.048*
C11	0.7186 (3)	−0.3009 (3)	−0.21097 (12)	0.0399 (6)
H11A	0.6983	−0.4075	−0.2415	0.048*
O4	0.6221 (3)	−0.6271 (2)	−0.16096 (10)	0.0679 (6)
N4	0.7912 (3)	−0.1221 (3)	−0.31121 (11)	0.0534 (5)
C5	0.8368 (3)	0.5061 (3)	0.14741 (15)	0.0549 (7)
H5A	0.8634	0.6184	0.1284	0.066*
C13	0.7946 (3)	0.0271 (3)	−0.19136 (12)	0.0407 (6)
H13A	0.8261	0.1407	−0.2096	0.049*
O5	0.8451 (3)	0.0308 (2)	−0.33278 (10)	0.0713 (6)
C9	0.7280 (3)	−0.1511 (3)	−0.09170 (12)	0.0354 (5)
C4	0.8307 (3)	0.5021 (3)	0.21998 (15)	0.0533 (7)
H4A	0.8528	0.6111	0.2493	0.064*
O6	0.7599 (3)	−0.2658 (3)	−0.35043 (10)	0.0813 (6)
C18	0.3624 (4)	0.0818 (3)	−0.36369 (12)	0.0432 (6)
H18A	0.3344	0.0297	−0.3214	0.052*
C7	0.8415 (5)	0.4749 (4)	0.36657 (16)	0.0861 (10)
H7A	0.8292	0.4398	0.4138	0.129*
H7B	0.7546	0.5500	0.3588	0.129*
H7C	0.9796	0.5435	0.3601	0.129*
N5	0.2789 (4)	0.0847 (4)	−0.48479 (16)	0.0962 (9)
C19	0.2479 (4)	0.0124 (4)	−0.42066 (17)	0.0663 (8)
H19A	0.1386	−0.0923	−0.4179	0.080*
C17	0.5183 (5)	0.2274 (4)	−0.36801 (16)	0.0732 (9)
H17A	0.6012	0.2766	−0.3277	0.088*
C15	0.4455 (5)	0.2404 (4)	−0.48748 (17)	0.0736 (8)
H15A	0.4736	0.2948	−0.5293	0.088*
C16	0.5660 (5)	0.3114 (4)	−0.42861 (18)	0.0760 (9)
H16A	0.6783	0.4146	−0.4293	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0478 (11)	0.0423 (10)	0.0358 (13)	0.0098 (8)	0.0002 (9)	0.0099 (9)
N3	0.0542 (13)	0.0366 (11)	0.0584 (16)	0.0161 (9)	0.0098 (11)	0.0130 (11)
O1	0.0749 (11)	0.0411 (9)	0.0432 (11)	0.0083 (7)	−0.0017 (9)	0.0069 (7)
C1	0.0306 (11)	0.0417 (12)	0.0435 (16)	0.0081 (9)	−0.0015 (10)	0.0074 (10)
N2	0.0563 (12)	0.0342 (9)	0.0379 (12)	0.0107 (8)	0.0036 (10)	0.0109 (8)
C2	0.0362 (12)	0.0440 (12)	0.0425 (16)	0.0099 (9)	−0.0003 (11)	0.0060 (11)
O3	0.1054 (15)	0.0491 (10)	0.0544 (14)	0.0253 (9)	0.0186 (11)	0.0247 (9)

C12	0.0393 (12)	0.0444 (12)	0.0343 (14)	0.0142 (9)	0.0037 (10)	0.0097 (10)
O2	0.0895 (13)	0.0634 (10)	0.0400 (12)	0.0122 (9)	0.0021 (10)	−0.0021 (9)
C8	0.0380 (13)	0.0438 (12)	0.0390 (15)	0.0116 (9)	−0.0002 (11)	0.0123 (10)
C10	0.0346 (12)	0.0317 (11)	0.0448 (15)	0.0093 (9)	0.0043 (10)	0.0104 (10)
C3	0.0421 (13)	0.0523 (14)	0.0452 (17)	0.0094 (10)	0.0008 (12)	0.0036 (12)
C6	0.0434 (13)	0.0469 (13)	0.0480 (16)	0.0039 (10)	−0.0011 (12)	0.0123 (11)
C14	0.0472 (13)	0.0343 (11)	0.0377 (15)	0.0100 (9)	0.0037 (11)	0.0070 (10)
C11	0.0398 (13)	0.0358 (11)	0.0430 (16)	0.0102 (9)	0.0029 (11)	0.0016 (10)
O4	0.0970 (14)	0.0336 (9)	0.0729 (14)	0.0187 (9)	0.0124 (11)	0.0037 (9)
N4	0.0635 (14)	0.0614 (13)	0.0397 (14)	0.0235 (11)	0.0074 (10)	0.0110 (11)
C5	0.0510 (15)	0.0418 (13)	0.069 (2)	0.0058 (11)	0.0002 (13)	0.0178 (12)
C13	0.0457 (13)	0.0338 (11)	0.0427 (15)	0.0083 (9)	0.0071 (11)	0.0150 (10)
O5	0.1020 (15)	0.0721 (12)	0.0476 (13)	0.0311 (10)	0.0184 (10)	0.0256 (9)
C9	0.0302 (11)	0.0384 (11)	0.0376 (15)	0.0082 (9)	0.0039 (10)	0.0101 (10)
C4	0.0508 (15)	0.0434 (13)	0.060 (2)	0.0067 (11)	−0.0025 (13)	−0.0023 (12)
O6	0.1247 (17)	0.0754 (12)	0.0429 (12)	0.0308 (11)	0.0063 (11)	−0.0032 (10)
C18	0.0617 (15)	0.0504 (13)	0.0200 (13)	0.0172 (12)	0.0036 (11)	0.0137 (10)
C7	0.115 (3)	0.084 (2)	0.053 (2)	0.0256 (18)	0.0025 (18)	−0.0192 (16)
N5	0.102 (2)	0.120 (2)	0.080 (2)	0.0501 (18)	0.0147 (17)	0.0221 (18)
C19	0.0695 (19)	0.0749 (17)	0.058 (2)	0.0208 (14)	0.0169 (16)	0.0237 (15)
C17	0.099 (2)	0.0744 (19)	0.054 (2)	0.0404 (17)	−0.0206 (18)	0.0007 (16)
C15	0.084 (2)	0.0792 (19)	0.067 (2)	0.0295 (17)	0.0219 (18)	0.0330 (17)
C16	0.081 (2)	0.0683 (17)	0.079 (3)	0.0186 (15)	−0.0033 (19)	0.0186 (17)

Geometric parameters (Å, º)

N1—C8	1.272 (3)	C14—C13	1.353 (3)
N1—N2	1.369 (2)	C14—C9	1.408 (3)
N3—O4	1.219 (2)	C14—H14A	0.9300
N3—O3	1.233 (2)	C11—H11A	0.9300
N3—C10	1.444 (3)	N4—O5	1.227 (2)
O1—C2	1.344 (2)	N4—O6	1.231 (2)
O1—H1B	0.8200	C5—C4	1.388 (4)
C1—C2	1.393 (3)	C5—H5A	0.9300
C1—C6	1.402 (3)	C13—H13A	0.9300
C1—C8	1.467 (3)	C4—H4A	0.9300
N2—C9	1.344 (3)	C18—C19	1.305 (3)
N2—H2A	0.8600	C18—C17	1.310 (3)
C2—C3	1.405 (3)	C18—H18A	0.9300
C12—C11	1.362 (3)	C7—H7A	0.9600
C12—C13	1.389 (3)	C7—H7B	0.9600
C12—N4	1.447 (3)	C7—H7C	0.9600
O2—C3	1.362 (3)	N5—C19	1.381 (4)
O2—C7	1.407 (3)	N5—C15	1.396 (4)
C8—H8A	0.9300	C19—H19A	0.9300
C10—C11	1.380 (3)	C17—C16	1.371 (4)
C10—C9	1.421 (3)	C17—H17A	0.9300
C3—C4	1.370 (3)	C15—C16	1.354 (4)

C6—C5	1.366 (3)	C15—H15A	0.9300
C6—H6A	0.9300	C16—H16A	0.9300
C8—N1—N2	117.18 (18)	O5—N4—C12	118.34 (19)
O4—N3—O3	122.35 (19)	O6—N4—C12	119.0 (2)
O4—N3—C10	119.5 (2)	C6—C5—C4	121.0 (2)
O3—N3—C10	118.15 (19)	C6—C5—H5A	119.5
C2—O1—H1B	109.5	C4—C5—H5A	119.5
C2—C1—C6	119.0 (2)	C14—C13—C12	120.38 (19)
C2—C1—C8	120.1 (2)	C14—C13—H13A	119.8
C6—C1—C8	120.8 (2)	C12—C13—H13A	119.8
C9—N2—N1	118.40 (17)	N2—C9—C14	119.45 (18)
C9—N2—H2A	120.8	N2—C9—C10	124.01 (19)
N1—N2—H2A	120.8	C14—C9—C10	116.5 (2)
O1—C2—C1	119.27 (19)	C3—C4—C5	119.7 (2)
O1—C2—C3	121.0 (2)	C3—C4—H4A	120.2
C1—C2—C3	119.7 (2)	C5—C4—H4A	120.2
C11—C12—C13	120.8 (2)	C19—C18—C17	117.8 (3)
C11—C12—N4	119.12 (19)	C19—C18—H18A	121.1
C13—C12—N4	120.05 (19)	C17—C18—H18A	121.1
C3—O2—C7	118.4 (2)	O2—C7—H7A	109.5
N1—C8—C1	119.9 (2)	O2—C7—H7B	109.5
N1—C8—H8A	120.1	H7A—C7—H7B	109.5
C1—C8—H8A	120.1	O2—C7—H7C	109.5
C11—C10—C9	121.50 (19)	H7A—C7—H7C	109.5
C11—C10—N3	115.69 (19)	H7B—C7—H7C	109.5
C9—C10—N3	122.8 (2)	C19—N5—C15	116.6 (3)
O2—C3—C4	125.1 (2)	C18—C19—N5	124.1 (3)
O2—C3—C2	114.6 (2)	C18—C19—H19A	118.0
C4—C3—C2	120.4 (2)	N5—C19—H19A	118.0
C5—C6—C1	120.3 (2)	C18—C17—C16	123.7 (3)
C5—C6—H6A	119.9	C18—C17—H17A	118.1
C1—C6—H6A	119.9	C16—C17—H17A	118.1
C13—C14—C9	121.41 (19)	C16—C15—N5	119.4 (3)
C13—C14—H14A	119.3	C16—C15—H15A	120.3
C9—C14—H14A	119.3	N5—C15—H15A	120.3
C12—C11—C10	119.34 (19)	C15—C16—C17	118.3 (3)
C12—C11—H11A	120.3	C15—C16—H16A	120.8
C10—C11—H11A	120.3	C17—C16—H16A	120.8
O5—N4—O6	122.7 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O4^{i}	0.82	2.53	3.319 (2)	162
N2—H2*A*···O3	0.86	2.03	2.635 (2)	126

Symmetry code: (i) −*x*+1, −*y*−1, −*z*.