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1-(4,6-Dimethylpyrimidin-2-yl)-3-(3,5-dinitrobenzoyl)thiourea monohydrate

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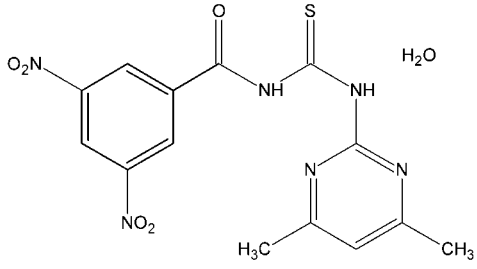
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.105; data-to-parameter ratio = 11.2.

The organic molecule in the title molecule, $C_{14}H_{12}N_6O_5S\cdot H_2O$, is roughly planar with a maximum deviation of 0.156 (2) Å. An intramolecular N—H···N hydrogen bond occurs. In the crystal, intermolecular N—H···O and O—H···O hydrogen-bonding interactions connect the molecules into a two-dimensional network that lies parallel to (101).

Related literature

For background to this study and our previous work on the structural chemistry of *N*,*N*′-disubstituted thiourea, see: Saeed *et al.* (2011).



Experimental

Crystal data

$C_{14}H_{12}N_6O_5S\cdot H_2O$
M_r = 394.37
Monoclinic, $P2_1/n$
a = 6.7892 (6) Å
b = 10.1823 (9) Å
c = 24.267 (2) Å
β = 92.901 (1)°
V = 1675.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 297 K
0.40 × 0.16 × 0.14 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.909, T_{max} = 0.967
9067 measured reflections
2942 independent reflections
2499 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.06
2942 reflections
263 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4*N*···O6	0.88 (2)	1.97 (2)	2.849 (2)	171.9 (19)
N3—H3*N*···N6	0.83 (2)	2.00 (2)	2.705 (2)	141.8 (19)
O6—H6*A*···O3^{i}	0.82 (4)	2.32 (4)	3.119 (3)	168 (4)
O6—H6*B*···O2^{ii}	0.73 (4)	2.44 (4)	3.143 (3)	164 (4)
O6—H6*B*···O1^{ii}	0.73 (4)	2.62 (4)	3.248 (3)	146 (4)

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2402).

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supporting information

Acta Cryst. (2011). E**67**, o1094 [doi:10.1107/S1600536811012323]

1-(4,6-Dimethylpyrimidin-2-yl)-3-(3,5-dinitrobenzoyl)thiourea monohydrate

Sohail Saeed, Naghmana Rashid, Wing-Tak Wong and Rizwan Hussain

S1. Comment

The background to this study has been described in our previous work on the structural chemistry of N,N′-disubstituted thiourea (Saeed *et al.*, 2011). In continuation of our studies, the crystal structure of the title compound, is presented in this paper.

In the title molecule (Fig. 1), the nitro groups N1/O1/O2 and N2/O3/O4 are oriented are 5.05 (11) and 9.40 (15)°, respectively, with rest to the mean-plane of the phenyl ring C1—C6. Moreover, the 4,6-dimethyl-pyrimidinyl ring plane, C9—C14/N5/N6, makes a dihedral angle of 1.31 (5)° with the the plane formed by the atoms C7/O5/N3/C8/S1/N4.

The structure is stabilized by intra-molecular N—H···O and N—H···N hydrogen bonding interactions. The inter-molecular hydrogen bonds O—H···O link the molecules into a two dimensinal network (Tab. 1 and Fig. 2).

S2. Experimental

A solution of 3,5-dinitrobenzoyl chloride (0.01 mol) in anhydrous acetone (75 ml) and 3% tetrabutylammonium bromide as a phase-transfer catalyst in anhydrous acetone was added dropwise to a suspension of dry potassium thiocyanate (0.01 mol) in acetone (50 ml) and the reaction mixture was refluxed for 50 min. After cooling to room temperature, a solution of 2-amino-4,6-dimethylpyrimidine (0.01 mol) in anhydrous acetone (25 ml) was added dropwise and the resulting mixture refluxed for 3 h. Hydrochloric acid (0.1 N, 300 ml) was added, and the solution was filtered. The solid product was washed with water and purified by re-crystallization from ethanol.

S3. Refinement

All of the C-bound H atoms were observable from difference Fourier map but were placed at geometrically idealized positions with C—H = 0.93 and 0.96Å for phenyl and methyl H-atoms, respectively. The C-bound H-atoms were refined using riding model with U_{iso}(H) = 1.2U_{eq}(C). Both the N– and O-bound H-atoms were located from difference Fourier map and refined isotropically.

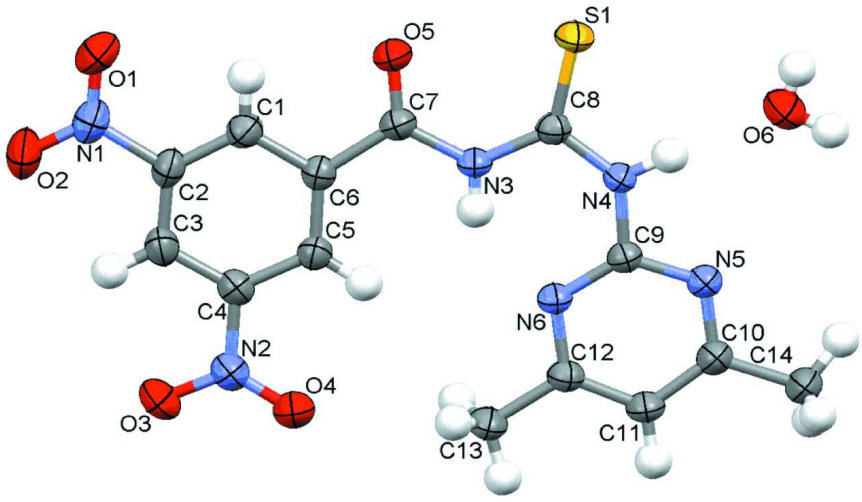

Figure 1

An *ORTEP* plot of the title molecule drawn with 50% probability thermal ellipsoids showing the atom numbering scheme.

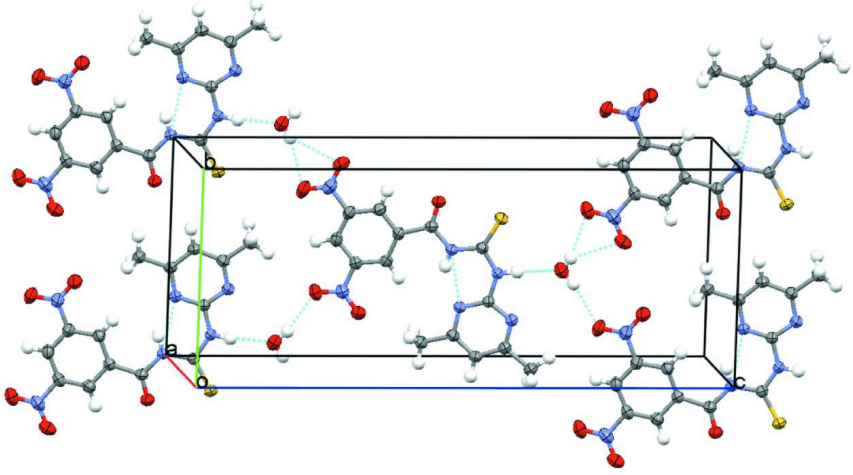

Figure 2

Packing diagram of the title compound viewed down the *a* axis.

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Crystal data

$C_{14}H_{12}N_6O_5S\cdot H_2O$
$M_r = 394.37$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.7892\ (6)$ Å
$b = 10.1823\ (9)$ Å
$c = 24.267\ (2)$ Å
$\beta = 92.901\ (1)°$
$V = 1675.4\ (3)$ Å^3
$Z = 4$
$F(000) = 816$
$D_x = 1.563$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11914 reflections
$\theta = 2.0–25.0°$
$\mu = 0.24$ mm^{-1}
$T = 297$ K
Prism, yellow
0.40 × 0.16 × 0.14 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.909, T_{max} = 0.967
9067 measured reflections
2942 independent reflections
2499 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°, θ_{min} = 2.6°
h = −8→8
k = −11→12
l = −23→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.06
2942 reflections
263 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.9069P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0044 (6)

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.
The structure was solved by direct methods (*SHELXS97*) and expanded using Fourier techniques. All non-H atoms were refined anisotropically.
All of the C-bound H atoms are observable from difference Fourier map but are all placed at geometrical positions with C—H = 0.93 and 0.96Å for phenyl and methyl H-atoms. All C-bound H-atoms are refined using riding model with U_{iso}(H) = 1.2U_{eq}(Carrier). Both the N– and O-bound H-atoms were located from difference Fourier map and refined isotropically.
Highest peak is 0.20 at (0.0792, 0.5848, 0.1447) [0.83Å from Hl4C] Deepest hole is -0.23 at (0.0397, 0.8302, 0.0937) [0.74Å from Sl]

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.64692 (10)	0.68130 (5)	0.59394 (2)	0.05020 (19)
O1	0.9131 (3)	0.89155 (18)	0.30790 (8)	0.0777 (6)
O2	1.0041 (4)	0.7610 (2)	0.24468 (8)	0.0851 (7)
O3	0.9757 (3)	0.29505 (18)	0.27197 (7)	0.0759 (6)
O4	0.8719 (4)	0.21594 (17)	0.34703 (7)	0.0802 (6)
O5	0.7636 (3)	0.73999 (15)	0.48300 (6)	0.0608 (5)
O6	0.5934 (4)	0.4521 (2)	0.70525 (8)	0.0692 (6)
H6A	0.561 (5)	0.395 (4)	0.7269 (16)	0.108 (14)*

H6B	0.569 (6)	0.512 (4)	0.7202 (16)	0.108 (15)*
N1	0.9439 (3)	0.7823 (2)	0.29029 (8)	0.0563 (5)
N2	0.9151 (3)	0.30735 (18)	0.31844 (8)	0.0519 (5)
N3	0.7393 (2)	0.52460 (16)	0.50834 (6)	0.0338 (4)
H3N	0.753 (3)	0.447 (2)	0.4988 (8)	0.035 (6)*
N4	0.6648 (2)	0.42786 (15)	0.59103 (7)	0.0354 (4)
H4N	0.632 (3)	0.440 (2)	0.6254 (10)	0.037 (5)*
N5	0.6445 (3)	0.21615 (16)	0.61919 (7)	0.0405 (4)
N6	0.7339 (2)	0.26275 (15)	0.52709 (6)	0.0346 (4)
C1	0.8532 (3)	0.6919 (2)	0.37878 (8)	0.0395 (5)
H1	0.8383	0.7775	0.3913	0.047*
C2	0.9061 (3)	0.6685 (2)	0.32582 (8)	0.0416 (5)
C3	0.9278 (3)	0.5444 (2)	0.30505 (8)	0.0437 (5)
H3	0.9645	0.5305	0.2691	0.052*
C4	0.8928 (3)	0.4414 (2)	0.33990 (8)	0.0399 (5)
C5	0.8407 (3)	0.45841 (19)	0.39404 (8)	0.0363 (4)
H5	0.8188	0.3865	0.4166	0.044*
C6	0.8221 (3)	0.58592 (19)	0.41368 (8)	0.0341 (4)
C7	0.7720 (3)	0.62534 (18)	0.47127 (8)	0.0362 (4)
C8	0.6868 (3)	0.54090 (18)	0.56219 (8)	0.0330 (4)
C9	0.6830 (3)	0.29543 (18)	0.57718 (8)	0.0331 (4)
C10	0.6548 (3)	0.0876 (2)	0.60875 (8)	0.0417 (5)
C11	0.7017 (3)	0.04196 (19)	0.55717 (8)	0.0429 (5)
H11	0.7055	−0.0477	0.5499	0.051*
C12	0.7424 (3)	0.13190 (19)	0.51685 (8)	0.0371 (4)
C13	0.7985 (4)	0.0930 (2)	0.46069 (8)	0.0496 (6)
H13A	0.9262	0.1281	0.4539	0.060*
H13B	0.7032	0.1269	0.4338	0.060*
H13C	0.8021	−0.0010	0.4581	0.060*
C14	0.6125 (4)	−0.0021 (2)	0.65554 (9)	0.0600 (7)
H14A	0.6208	0.0462	0.6895	0.072*
H14B	0.7073	−0.0722	0.6573	0.072*
H14C	0.4824	−0.0381	0.6498	0.072*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0823 (4)	0.0302 (3)	0.0389 (3)	0.0073 (2)	0.0114 (3)	−0.0046 (2)
O1	0.1246 (17)	0.0442 (10)	0.0658 (12)	−0.0034 (10)	0.0201 (11)	0.0142 (9)
O2	0.1375 (18)	0.0706 (13)	0.0507 (11)	−0.0010 (12)	0.0386 (11)	0.0165 (9)
O3	0.1253 (16)	0.0596 (11)	0.0457 (10)	0.0000 (10)	0.0324 (10)	−0.0116 (8)
O4	0.1496 (19)	0.0412 (9)	0.0524 (11)	−0.0108 (11)	0.0316 (11)	−0.0044 (8)
O5	0.1047 (13)	0.0309 (8)	0.0490 (9)	−0.0032 (8)	0.0266 (9)	−0.0014 (7)
O6	0.1083 (16)	0.0545 (11)	0.0466 (10)	0.0065 (11)	0.0221 (10)	−0.0072 (9)
N1	0.0725 (14)	0.0516 (12)	0.0452 (11)	−0.0031 (10)	0.0085 (10)	0.0142 (9)
N2	0.0741 (13)	0.0450 (11)	0.0374 (10)	−0.0029 (9)	0.0101 (9)	−0.0037 (8)
N3	0.0430 (9)	0.0269 (8)	0.0320 (8)	0.0018 (7)	0.0057 (7)	−0.0014 (7)
N4	0.0495 (10)	0.0294 (8)	0.0278 (8)	0.0022 (7)	0.0070 (7)	−0.0019 (6)

N5	0.0561 (10)	0.0331 (9)	0.0327 (9)	−0.0012 (7)	0.0063 (7)	0.0004 (7)
N6	0.0429 (9)	0.0296 (8)	0.0315 (8)	0.0024 (7)	0.0038 (7)	−0.0008 (7)
C1	0.0421 (11)	0.0363 (11)	0.0403 (11)	−0.0004 (8)	0.0036 (9)	0.0033 (8)
C2	0.0458 (11)	0.0443 (12)	0.0349 (11)	−0.0030 (9)	0.0042 (9)	0.0102 (9)
C3	0.0495 (12)	0.0506 (12)	0.0316 (10)	−0.0043 (10)	0.0071 (9)	0.0013 (9)
C4	0.0459 (11)	0.0409 (11)	0.0330 (10)	−0.0012 (9)	0.0036 (8)	−0.0025 (8)
C5	0.0390 (10)	0.0375 (10)	0.0325 (10)	−0.0035 (8)	0.0033 (8)	0.0020 (8)
C6	0.0337 (10)	0.0364 (10)	0.0323 (10)	−0.0009 (8)	0.0025 (7)	0.0020 (8)
C7	0.0409 (11)	0.0305 (10)	0.0374 (10)	−0.0013 (8)	0.0041 (8)	−0.0001 (8)
C8	0.0351 (10)	0.0317 (9)	0.0321 (10)	0.0014 (8)	0.0013 (8)	−0.0011 (8)
C9	0.0369 (10)	0.0304 (9)	0.0320 (10)	0.0008 (8)	0.0023 (8)	0.0002 (8)
C10	0.0557 (12)	0.0342 (10)	0.0353 (10)	−0.0034 (9)	0.0044 (9)	0.0017 (8)
C11	0.0602 (13)	0.0287 (10)	0.0403 (11)	0.0009 (9)	0.0073 (9)	−0.0020 (8)
C12	0.0438 (11)	0.0326 (10)	0.0350 (10)	0.0028 (8)	0.0031 (8)	−0.0024 (8)
C13	0.0744 (15)	0.0351 (11)	0.0403 (12)	0.0044 (10)	0.0132 (11)	−0.0043 (9)
C14	0.105 (2)	0.0363 (12)	0.0400 (12)	−0.0073 (12)	0.0160 (12)	0.0025 (10)

Geometric parameters (Å, º)

S1—C8	1.6528 (19)	C1—C6	1.395 (3)
O1—N1	1.213 (3)	C1—H1	0.9300
O2—N1	1.218 (3)	C2—C3	1.372 (3)
O3—N2	1.226 (2)	C3—C4	1.375 (3)
O4—N2	1.206 (2)	C3—H3	0.9300
O5—C7	1.204 (2)	C4—C5	1.389 (3)
O6—H6A	0.82 (4)	C5—C6	1.391 (3)
O6—H6B	0.73 (4)	C5—H5	0.9300
N1—C2	1.475 (3)	C6—C7	1.509 (3)
N2—C4	1.472 (3)	C10—C11	1.387 (3)
N3—C8	1.382 (2)	C10—C14	1.496 (3)
N3—C7	1.390 (2)	C11—C12	1.379 (3)
N3—H3N	0.83 (2)	C11—H11	0.9300
N4—C8	1.359 (2)	C12—C13	1.487 (3)
N4—C9	1.397 (2)	C13—H13A	0.9600
N4—H4N	0.88 (2)	C13—H13B	0.9600
N5—C9	1.337 (2)	C13—H13C	0.9600
N5—C10	1.336 (3)	C14—H14A	0.9600
N6—C9	1.323 (2)	C14—H14B	0.9600
N6—C12	1.357 (2)	C14—H14C	0.9600
C1—C2	1.373 (3)		

H6A—O6—H6B	101 (4)	C1—C6—C7	113.88 (17)
O1—N1—O2	123.7 (2)	O5—C7—N3	123.49 (18)
O1—N1—C2	118.43 (19)	O5—C7—C6	119.51 (17)
O2—N1—C2	117.9 (2)	N3—C7—C6	117.00 (16)
O4—N2—O3	123.56 (19)	N4—C8—N3	115.18 (16)
O4—N2—C4	118.69 (17)	N4—C8—S1	117.87 (14)
O3—N2—C4	117.75 (18)	N3—C8—S1	126.94 (14)

C8—N3—C7	125.51 (17)	N6—C9—N5	128.26 (17)
C8—N3—H3N	114.5 (14)	N6—C9—N4	119.63 (16)
C7—N3—H3N	119.9 (14)	N5—C9—N4	112.11 (16)
C8—N4—C9	132.86 (16)	N5—C10—C11	121.06 (18)
C8—N4—H4N	114.2 (14)	N5—C10—C14	116.15 (18)
C9—N4—H4N	112.9 (14)	C11—C10—C14	122.79 (19)
C9—N5—C10	115.67 (17)	C12—C11—C10	118.78 (18)
C9—N6—C12	115.51 (16)	C12—C11—H11	120.6
C2—C1—C6	119.31 (19)	C10—C11—H11	120.6
C2—C1—H1	120.3	N6—C12—C11	120.68 (17)
C6—C1—H1	120.3	N6—C12—C13	116.40 (17)
C1—C2—C3	122.81 (18)	C11—C12—C13	122.92 (18)
C1—C2—N1	118.21 (19)	C12—C13—H13A	109.5
C3—C2—N1	118.97 (18)	C12—C13—H13B	109.5
C2—C3—C4	116.83 (18)	H13A—C13—H13B	109.5
C2—C3—H3	121.6	C12—C13—H13C	109.5
C4—C3—H3	121.6	H13A—C13—H13C	109.5
C3—C4—C5	123.19 (19)	H13B—C13—H13C	109.5
C3—C4—N2	117.74 (17)	C10—C14—H14A	109.5
C5—C4—N2	119.06 (17)	C10—C14—H14B	109.5
C4—C5—C6	118.16 (17)	H14A—C14—H14B	109.5
C4—C5—H5	120.9	C10—C14—H14C	109.5
C6—C5—H5	120.9	H14A—C14—H14C	109.5
C5—C6—C1	119.68 (17)	H14B—C14—H14C	109.5
C5—C6—C7	126.44 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*N*···O6	0.88 (2)	1.97 (2)	2.849 (2)	171.9 (19)
N3—H3*N*···N6	0.83 (2)	2.00 (2)	2.705 (2)	141.8 (19)
O6—H6*A*···O3^{i}	0.82 (4)	2.32 (4)	3.119 (3)	168 (4)
O6—H6*B*···O2^{ii}	0.73 (4)	2.44 (4)	3.143 (3)	164 (4)
O6—H6*B*···O1^{ii}	0.73 (4)	2.62 (4)	3.248 (3)	146 (4)

Symmetry codes: (i) $x-1/2, -y+1/2, z+1/2$; (ii) $x-1/2, -y+3/2, z+1/2$.