Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(4-Nitrobenzoyl)thiosemicarbazide monohydrate: a three-dimensional hydrogen-bonded framework structure

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.091
Data-to-parameter ratio = 14.1

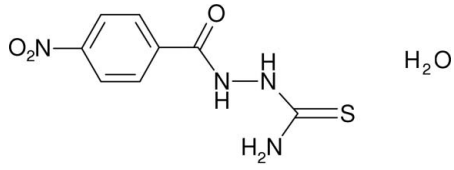
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 May 2006
Accepted 23 May 2006

In the title compound, $C_8H_8N_4O_3S\cdot H_2O$, strong hydrogen bonding results in the formation of a number of chains and dimers, which combine to give a three-dimensional hydrogen-bonded framework.

Comment

Acylthiosemicarbazides are versatile compounds, having a large spectrum of biological properties (Bhat *et al.*, 1967; Guersoy & Karali, 1995; Plumitallo *et al.*, 2004). They are, in addition, useful precursors of various biologically active heterocyclic compounds, including triazoles (Kane *et al.*, 1994; Palaska *et al.*, 2002), thiadiazoles (Oruc *et al.*, 2004; Palaska *et al.*, 2002) and oxadiazoles (Palaska *et al.*, 2002; Yale & Losee, 1966). Certain acylthiosemicarbazide–transition metal complexes have also been shown to possess useful biological activities (Shen *et al.*, 1997; Singh & Singh, 2001). As part of our interest in acylthiosemicarbazide compounds, we now report the crystal structure of 1-(4-nitrobenzoyl)thiosemicarbazide monohydrate, (I).



(I)

Within the asymmetric unit of (I), the O atom of the solvent water molecule acts as an H-atom acceptor for the amide group of the organic molecule (Fig. 1). The *p*-nitro group is rotated from the essentially planar aryl group by an angle of 13.07 (12)°, whereas the CN(O) group is twisted by 10.77 (12)°.

The hydrogen bonding (Table 2) at the basic level produces a mixture of chains and dimers. The combination of the hydrogen bond described above, together with O1*W*—H1*WA*···O7^{ii} [symmetry code: (ii) $x+1, y, z$] leads to a $C_2^2(9)$ chain (Bernstein *et al.*, 1995) along [010]. Another chain, $C(12)$, forms along [100] *via* the N9—H9*A*···O42^{v} hydrogen bond [symmetry code: (v) $x, y, z-1$]. These combine to form an $R_5^6(34)$ ring (Fig. 2); the disparity between the number of donors and acceptors results from the amide acting as a double donor. The rings link to create a sheet normal to [010] (Fig. 2).

All other hydrogen bonds involve S as an acceptor and result in dimers. In the first, the hydrogen bond within the asymmetric unit combines with O1*W*—H1*WA*···S1^{1}

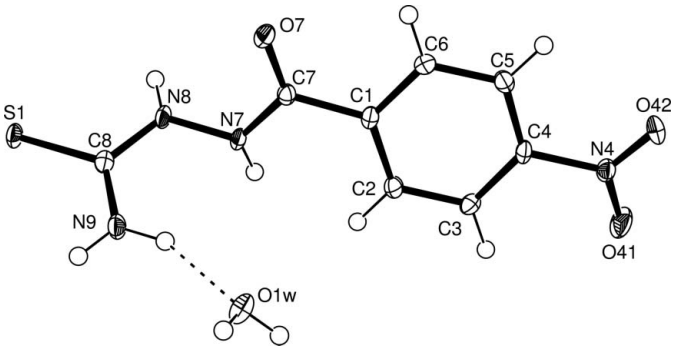


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as circles of arbitrary radius. The dashed line indicates a hydrogen bond.

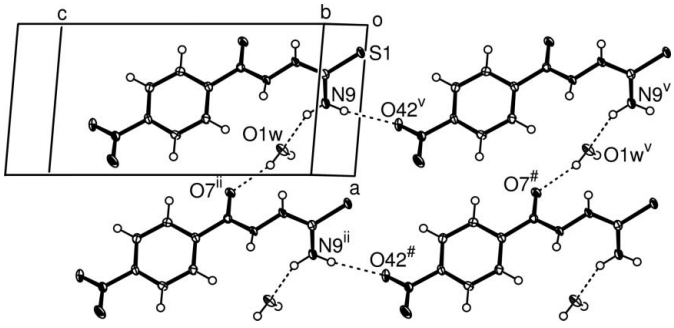


Figure 2
Part of the crystal structure of (I), showing the formation of a hydrogen-bonded $R_5^6(34)$ ring which links with others to give sheets. Atoms marked with (ii), (v) or a hash (#) are at the symmetry positions $(1 + x, y, z)$, $(x, y, -1 + z)$ and $(1 + x, y, -1 + z)$, respectively. Dashed lines indicate hydrogen bonds.

[symmetry code: (i) $1 - x, 1 - y, -z$] to form an $R_4^4(12)$ ring. The other two are simpler motifs; N7—H7···S1^{iii} [symmetry code: (iii) $1 - x, 2 - y, -z$] giving an $R_2^2(10)$ ring and N8—H8···S1^{iv} [symmetry code: (iv) $-x, 2 - y, -z$] forming an $R_2^2(8)$ motif. The former two dimers combine with the above-described hydrogen bond to give a chain along [010] (Fig. 3). The sheet shown in Fig. 2 and the chain shown in Fig. 3 thus combine to give a three-dimensional hydrogen-bonded framework.

Experimental

A solution of potassium thiocyanate (0.73 g, 12.5 mmol) and concentrated HCl (1.25 ml) was added to a stirred solution of 4-nitrobenzoylhydrazide (1.5 g, 8.3 mmol) (Hosamani & Pattanashettar, 2004) in methanol (21 ml). The mixture was evaporated to dryness on a steam bath, further methanol (21 ml) was added and the mixture heated for 1 h on a steam bath. The resulting solid was successively washed with water and a small volume of ethanol, and recrystallized from acetone, yielding 2.1 g (70%) of yellow 1-(4-nitrobenzoyl)thiosemicarbazide (m.p. 489 K). ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.71 (*s*, 1H, CONHNH), 9.44 (*s*, 1H, CONH), 8.33 (*d*, 2H, J = 8.5 Hz, Ar—H), 8.13 (*d*, 2H, J = 8.5 Hz, Ar—H), 7.95 (*s*, 1H, $CSNH_2$), 7.79 (*s*, 1H, $CSNH_2$).

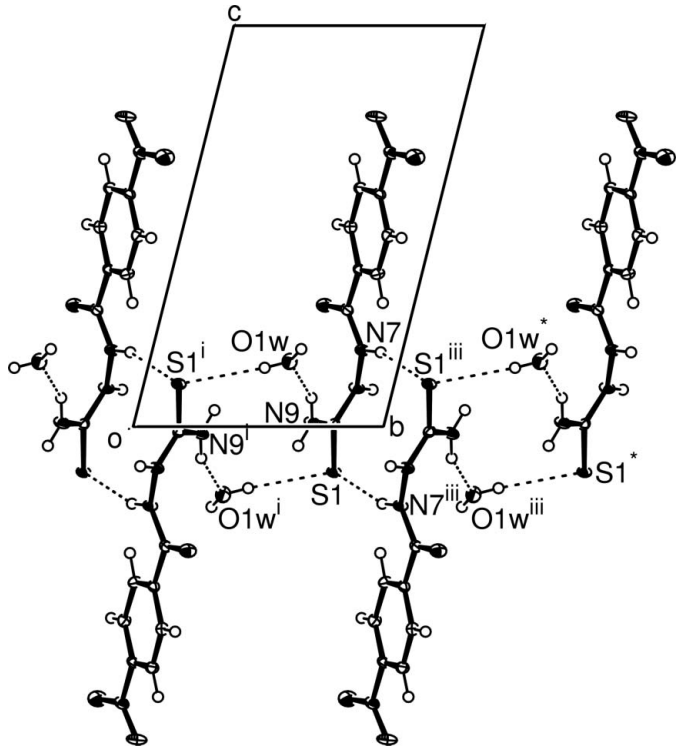


Figure 3
Part of the crystal structure of (I), showing the formation of hydrogen-bonded dimers linked to form a chain. Atoms marked with (i), (iii) or an asterisk (*) are at the symmetry positions $(1 - x, 1 - y, -z)$, $(1 - x, 2 - y, -z)$ and $(x, 1 + y, z)$ respectively. Dashed lines indicate hydrogen bonds.

Crystal data

$C_8H_8N_4O_3S\cdot H_2O$
M_r = 258.26
Triclinic, $P\overline{1}$
a = 6.0621 (2) Å
b = 7.3991 (3) Å
c = 12.2661 (5) Å
α = 75.9684 (16)°
β = 85.112 (2)°
γ = 88.903 (2)°
V = 531.83 (4) Å^3
Z = 2
D_x = 1.613 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 120 (2) K
Slab, pale yellow
0.45 × 0.45 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.688, T_{max} = 0.928
8670 measured reflections
2425 independent reflections
2178 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.091
S = 1.12
2425 reflections
172 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 0.2095P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 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
O1W—H1WA···S1^{i}	0.79 (2)	2.61 (2)	3.3472 (13)	156.5 (18)
O1W—H1WA···O7^{ii}	0.81 (2)	2.01 (2)	2.7944 (15)	162.7 (19)
N7—H7···S1^{iii}	0.831 (19)	2.608 (19)	3.4096 (13)	162.4 (16)
N8—H8···S1^{iv}	0.854 (19)	2.49 (2)	3.3382 (13)	172.0 (16)
N9—H9A···O42^{v}	0.84 (2)	2.26 (2)	3.0834 (17)	164.8 (18)
N9—H9B···O1W	0.89 (2)	1.94 (2)	2.7754 (16)	153.8 (17)

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

All H atoms were located in difference maps; those in the aryl ring were then treated as riding atoms, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C). All other H atoms were refined freely.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1997) and *PLATON* (Spek, 2003).

We are indebted to the EPSRC for the use of both the Chemical Database Service at Daresbury, England, primarily for access to the Cambridge Structural Database (Fletcher *et al.*, 1996), and the X-ray service at the University of Southampton, England, for data collection.

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supporting information

Acta Cryst. (2006). E**62**, o2563–o2565 [https://doi.org/10.1107/S1600536806019301]

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Crystal data

$C_8H_8N_4O_3S·H_2O$
$M_r = 258.26$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.0621$ (2) Å
$b = 7.3991$ (3) Å
$c = 12.2661$ (5) Å
$\alpha = 75.9684$ (16)°
$\beta = 85.112$ (2)°
$\gamma = 88.903$ (2)°
$V = 531.83$ (4) Å^3
$Z = 2$
$F(000) = 268$
$D_x = 1.613$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2242 reflections
$\theta = 2.9$–27.5°
$\mu = 0.32$ mm^{-1}
$T = 120$ K
Slab, pale yellow
0.45 × 0.45 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker–Nonius KappaCCD
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.688$, $T_{max} = 0.928$
8670 measured reflections
2425 independent reflections
2178 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.4°$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.091$
$S = 1.12$
2425 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 0.2095P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Special details

Experimental. IR ν_{max} (KBr, cm^{-1}): 3515, 3429, 3157, 1683, 1630, 1605, 1522, 1348, 1264, 714. ^{13}C NMR (125 MHz, DMSO-d_6): δ 181.92, 164.30, 149.20, 138.24, 129.30, 123.22.

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}
C1	0.4146 (2)	0.75286 (19)	0.39533 (11)	0.0124 (3)
C2	0.6269 (2)	0.82803 (19)	0.38368 (11)	0.0145 (3)
H2	0.6979	0.8770	0.3106	0.019*
C3	0.7348 (2)	0.83152 (19)	0.47840 (12)	0.0141 (3)
H3	0.8784	0.8843	0.4714	0.018*
C4	0.6275 (2)	0.75598 (19)	0.58338 (11)	0.0129 (3)
C5	0.4188 (2)	0.67728 (19)	0.59826 (12)	0.0143 (3)
H5	0.3507	0.6251	0.6716	0.019*
C6	0.3118 (2)	0.67690 (19)	0.50267 (12)	0.0134 (3)
H6	0.1677	0.6247	0.5103	0.017*
N4	0.74170 (19)	0.75903 (17)	0.68434 (10)	0.0156 (3)
O41	0.90680 (19)	0.85720 (17)	0.67255 (9)	0.0266 (3)
O42	0.66489 (18)	0.66376 (17)	0.77561 (9)	0.0242 (3)
C7	0.2872 (2)	0.74619 (19)	0.29655 (11)	0.0128 (3)
O7	0.11625 (16)	0.65565 (15)	0.30868 (9)	0.0186 (2)
N7	0.3713 (2)	0.84764 (17)	0.19467 (10)	0.0142 (3)
H7	0.476 (3)	0.921 (3)	0.1890 (15)	0.018*
N8	0.2502 (2)	0.86376 (18)	0.10162 (10)	0.0156 (3)
H8	0.133 (3)	0.930 (3)	0.0979 (15)	0.020*
C8	0.3325 (2)	0.80758 (19)	0.01032 (11)	0.0131 (3)
N9	0.5224 (2)	0.71739 (18)	0.01309 (11)	0.0177 (3)
H9A	0.574 (3)	0.684 (3)	−0.0449 (17)	0.023*
H9B	0.598 (3)	0.689 (3)	0.0745 (17)	0.023*
S1	0.18825 (6)	0.85785 (5)	−0.10755 (3)	0.01538 (12)
O1W	0.83048 (19)	0.56199 (16)	0.16378 (10)	0.0208 (2)
H1WA	0.932 (3)	0.595 (3)	0.1938 (17)	0.027*
H1WA	0.867 (3)	0.468 (3)	0.1476 (17)	0.027*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0146 (6)	0.0122 (6)	0.0116 (6)	0.0024 (5)	−0.0043 (5)	−0.0042 (5)
C2	0.0157 (7)	0.0159 (7)	0.0109 (6)	−0.0004 (5)	−0.0020 (5)	−0.0008 (5)
C3	0.0125 (6)	0.0145 (7)	0.0157 (7)	−0.0013 (5)	−0.0033 (5)	−0.0032 (5)

C4	0.0162 (7)	0.0135 (6)	0.0107 (6)	0.0035 (5)	−0.0054 (5)	−0.0053 (5)
C5	0.0160 (7)	0.0153 (7)	0.0115 (6)	0.0015 (5)	−0.0006 (5)	−0.0033 (5)
C6	0.0120 (6)	0.0137 (6)	0.0146 (7)	0.0004 (5)	−0.0014 (5)	−0.0035 (5)
N4	0.0160 (6)	0.0195 (6)	0.0134 (6)	0.0036 (5)	−0.0042 (4)	−0.0071 (5)
O41	0.0258 (6)	0.0332 (7)	0.0223 (6)	−0.0090 (5)	−0.0111 (4)	−0.0060 (5)
O42	0.0260 (6)	0.0371 (7)	0.0093 (5)	−0.0008 (5)	−0.0023 (4)	−0.0049 (5)
C7	0.0137 (6)	0.0138 (6)	0.0119 (6)	0.0029 (5)	−0.0029 (5)	−0.0049 (5)
O7	0.0153 (5)	0.0252 (6)	0.0158 (5)	−0.0047 (4)	−0.0040 (4)	−0.0046 (4)
N7	0.0136 (6)	0.0192 (6)	0.0107 (6)	−0.0024 (5)	−0.0044 (4)	−0.0040 (5)
N8	0.0151 (6)	0.0218 (6)	0.0111 (6)	0.0038 (5)	−0.0067 (4)	−0.0050 (5)
C8	0.0157 (7)	0.0116 (6)	0.0112 (6)	−0.0032 (5)	−0.0021 (5)	−0.0009 (5)
N9	0.0192 (6)	0.0238 (7)	0.0121 (6)	0.0066 (5)	−0.0059 (5)	−0.0071 (5)
S1	0.0180 (2)	0.0187 (2)	0.01036 (19)	0.00160 (13)	−0.00624 (13)	−0.00371 (13)
O1W	0.0208 (6)	0.0193 (6)	0.0236 (6)	−0.0006 (4)	−0.0108 (4)	−0.0047 (5)

Geometric parameters (Å, º)

C1—C2	1.3943 (19)	N4—O42	1.2281 (16)
C1—C6	1.3973 (19)	C7—O7	1.2234 (17)
C1—C7	1.5022 (18)	C7—N7	1.3551 (18)
C2—C3	1.3867 (19)	N7—N8	1.3890 (16)
C2—H2	0.9500	N7—H7	0.831 (19)
C3—C4	1.3840 (19)	N8—C8	1.3411 (18)
C3—H3	0.9500	N8—H8	0.854 (19)
C4—C5	1.382 (2)	C8—N9	1.3180 (18)
C4—N4	1.4742 (17)	C8—S1	1.7135 (14)
C5—C6	1.3882 (19)	N9—H9A	0.84 (2)
C5—H5	0.9500	N9—H9B	0.89 (2)
C6—H6	0.9500	O1W—H1WA	0.81 (2)
N4—O41	1.2257 (16)	O1W—H1WA	0.79 (2)
C2—C1—C6	120.01 (12)	O41—N4—C4	118.21 (11)
C2—C1—C7	123.07 (12)	O42—N4—C4	118.00 (12)
C6—C1—C7	116.92 (12)	O7—C7—N7	122.55 (12)
C3—C2—C1	120.21 (12)	O7—C7—C1	121.35 (12)
C3—C2—H2	119.9	N7—C7—C1	116.10 (12)
C1—C2—H2	119.9	C7—N7—N8	119.14 (12)
C4—C3—C2	118.22 (12)	C7—N7—H7	121.3 (12)
C4—C3—H3	120.9	N8—N7—H7	117.8 (13)
C2—C3—H3	120.9	C8—N8—N7	121.51 (12)
C5—C4—C3	123.23 (13)	C8—N8—H8	119.3 (12)
C5—C4—N4	118.35 (12)	N7—N8—H8	118.1 (12)
C3—C4—N4	118.42 (12)	N9—C8—N8	119.39 (13)
C4—C5—C6	117.89 (13)	N9—C8—S1	121.85 (11)
C4—C5—H5	121.1	N8—C8—S1	118.75 (10)
C6—C5—H5	121.1	C8—N9—H9A	118.7 (13)
C5—C6—C1	120.44 (13)	C8—N9—H9B	122.6 (12)
C5—C6—H6	119.8	H9A—N9—H9B	118.7 (17)

C1—C6—H6	119.8	H1WA—O1W—H1WA	107 (2)
O41—N4—O42	123.78 (12)		
C6—C1—C2—C3	−1.5 (2)	C5—C4—N4—O42	12.54 (18)
C7—C1—C2—C3	179.39 (12)	C3—C4—N4—O42	−167.28 (13)
C1—C2—C3—C4	1.1 (2)	C2—C1—C7—O7	169.10 (13)
C2—C3—C4—C5	0.1 (2)	C6—C1—C7—O7	−10.07 (19)
C2—C3—C4—N4	179.88 (12)	C2—C1—C7—N7	−11.73 (19)
C3—C4—C5—C6	−0.9 (2)	C6—C1—C7—N7	169.10 (12)
N4—C4—C5—C6	179.33 (11)	O7—C7—N7—N8	5.9 (2)
C4—C5—C6—C1	0.5 (2)	C1—C7—N7—N8	−173.27 (11)
C2—C1—C6—C5	0.6 (2)	C7—N7—N8—C8	−121.29 (15)
C7—C1—C6—C5	179.85 (12)	N7—N8—C8—N9	7.2 (2)
C5—C4—N4—O41	−167.02 (13)	N7—N8—C8—S1	−172.06 (10)
C3—C4—N4—O41	13.16 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···S1^{i}	0.79 (2)	2.61 (2)	3.3472 (13)	156.5 (18)
O1*W*—H1*WA*···O7^{ii}	0.81 (2)	2.01 (2)	2.7944 (15)	162.7 (19)
N7—H7···S1^{iii}	0.831 (19)	2.608 (19)	3.4096 (13)	162.4 (16)
N8—H8···S1^{iv}	0.854 (19)	2.49 (2)	3.3382 (13)	172.0 (16)
N9—H9*A*···O42^{v}	0.84 (2)	2.26 (2)	3.0834 (17)	164.8 (18)
N9—H9*B*···O1*W*	0.89 (2)	1.94 (2)	2.7754 (16)	153.8 (17)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*+1, *y*, *z*; (iii) −*x*+1, −*y*+2, −*z*; (iv) −*x*, −*y*+2, −*z*; (v) *x*, *y*, *z*−1.