Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Tris(methylammonium thiocyanurate) monohydrate

**Yimin Hou[a] and Yunxia Yang[b]***

[a]Henan University of Traditional Chinese Medicine, Zhengzhou 450008, People's Republic of China, and [b]Key Laboratory of Polymer Materials of Gansu Province, Ministry of Education, College of Chemistry and Chemical Engineering, Northwest Normal University, Lanzhou 730070, Gansu, People's Republic of China
Correspondence e-mail: yangyx80@nwnu.edu.cn

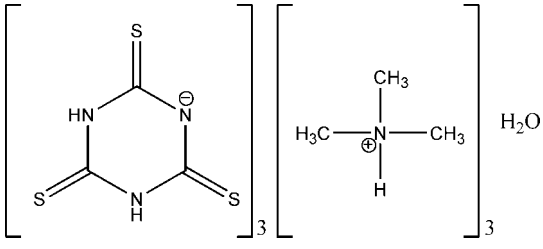
Received 18 November 2010; accepted 1 December 2010

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(N–C) = 0.004 Å; $R$ factor = 0.036; $wR$ factor = 0.103; data-to-parameter ratio = 16.1.

In the title compound, $3[(CH_3)_3HN^+]\cdot 3C_3H_2N_3S_3^-\cdot H_2O$, two independent trithiocyanurate anions construct a planar hydrogen-bonded ribbon with two N—H···S hydrogen bonds linking each pair of adjacent anions in the chain. The third independent anion and the water molecule form a chain by way of N—H···S and O—H···S contacts, which propagates parallel to the ribbon. The chains and ribbons are cross-linked by O—H···S hydrogen bonds, generating sheets. The three independent trimethylammonium cations are contained between the sheets by way of various N—H···S and N—H···N contacts.

## Related literature

For hydrogen-bond formation in the compounds of trithiocyanuric acid, see: Dean *et al.* (2004).



## Experimental

### Crystal data

$3C_3H_{10}N^+\cdot 3C_3H_2N_3S_3^-\cdot H_2O$
$M_r$ = 727.23
Triclinic, $P\overline{1}$
$a$ = 11.3466 (1) Å
$b$ = 12.6474 (1) Å
$c$ = 12.8135 (1) Å
$\alpha$ = 76.950 (1)°
$\beta$ = 84.762 (1)°
$\gamma$ = 82.274 (1)°
$V$ = 1771.51 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.60 mm$^{-1}$
$T$ = 296 K
0.55 × 0.40 × 0.09 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan *SADABS* (Bruker, 2007)
$T_{min}$ = 0.735, $T_{max}$ = 0.948
14356 measured reflections
6243 independent reflections
5406 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.016

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.103
$S$ = 1.05
6243 reflections
388 parameters
12 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$WA$···S3 | 0.86 | 2.57 | 3.284 (2) | 140 |
| O1$W$—H1$WB$···S9$^{i}$ | 0.87 | 2.52 | 3.355 (2) | 161 |
| N2—H2···S4$^{ii}$ | 0.85 (1) | 2.48 (1) | 3.3196 (17) | 168 (2) |
| N3—H3···S6$^{iii}$ | 0.85 (1) | 2.48 (1) | 3.3203 (17) | 168 (2) |
| N4—H4···S2$^{ii}$ | 0.86 (1) | 2.45 (1) | 3.2962 (17) | 169 (2) |
| N5—H5···S1$^{iii}$ | 0.86 (1) | 2.49 (1) | 3.3316 (17) | 167 (2) |
| N7—H7···O1$W$ | 0.86 (1) | 2.03 (1) | 2.878 (3) | 172 (3) |
| N9—H9···S8$^{iv}$ | 0.86 (1) | 2.63 (1) | 3.470 (2) | 167 (3) |
| N10—H10···N6 | 0.87 (1) | 1.95 (1) | 2.804 (3) | 168 (3) |
| N11—H11···N1 | 0.86 (1) | 1.95 (1) | 2.795 (2) | 164 (3) |
| N12—H12···S7$^{v}$ | 0.85 (1) | 2.69 (2) | 3.439 (2) | 148 (3) |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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: *SHELXL97* and *publCIF* (Westrip, 2010).

We thank Northwest Normal University for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5752).

## References

Bruker (2007). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconson, USA.
Dean, P. A. W., Jennings, M., Houle, T. M., Craig, D. C., Dance, I. G., Hook, J. M. & Scudder, M. L. (2004). *CrystEngComm*, **6**, 543–548.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

# supporting information

*Acta Cryst.* (2011). E**67**, o44 [https://doi.org/10.1107/S1600536810050312]

## Tris(methylammonium thiocyanurate) monohydrate

**Yimin Hou and Yunxia Yang**

### S1. Comment

Trithiocyanuric acid, which can be regarded as the polymer of three thiourea molecules, have a strong tendency to form various hydrogen bonds (Dean *et al.* 2004). Here we report the crystal structure of the title ammonium water-trithiocyanurate, $3[(CH_3)_3HN^+].3C_3H_2N_3S_3^-.H_2O$, (I). In this structure, two independent trithiocyanurate anions containing S1 and S4 atoms firstly form the hydrogen-bonded ribbon parallel to (010) plane by four N—H···S hydrogen bonds, and then between two planar ribbons, there exist a totally different waving ribbon composed of the third trithiocyanurate anion and the only water molecule, in which the anion links the water molecule with varied N—H···S and O—H···S contacts (Fig. 2). With the existence of one independent O—H···S interaction between the aforementioned planar hydrogen-bonded ribbon and the waving ribbon, two planar ribbons and one waving ribbon can yield the larger hydrogen-bonded unit as shown in Fig. 3. The ammonium cations, with the existence of N—H···S and N—H···N interactions, are stably accommodated to the intervals of these separated units to form the stable crystal structure.

### S2. Experimental

Trithiocyanuric acid (0.044 g, 0.25 mmol) was dissolved in a water-ethanol (1:2 *v*/*v*) mixture and a 33% solution of trimethyl amine was added to neutralize the acid. Colorless block crystals separated after several weeks.

### S3. Refinement

Nitrogen-bound H-atoms were placed in calculated positions (N—H 0.86 Å) and were included in the refinement in the riding model approximation, with $U$(H) set to 1.5$U$(N). The water H-atom was similarly treated.

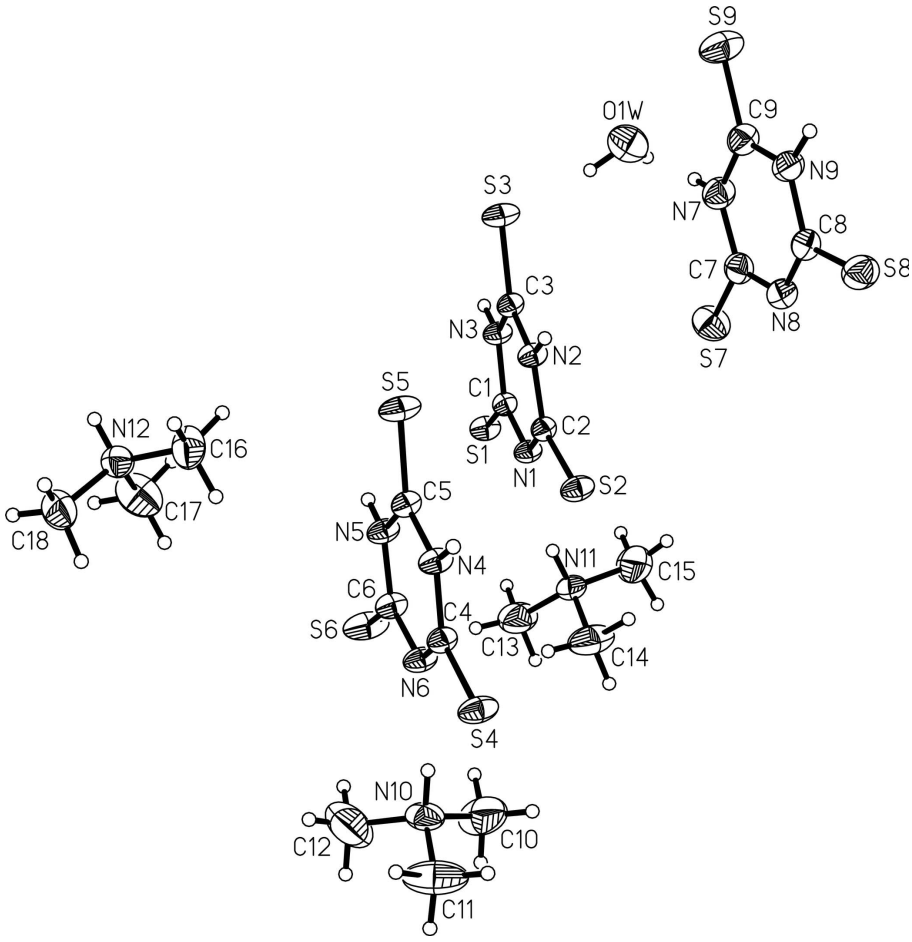



**Figure 1**

The title compound at the 30% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

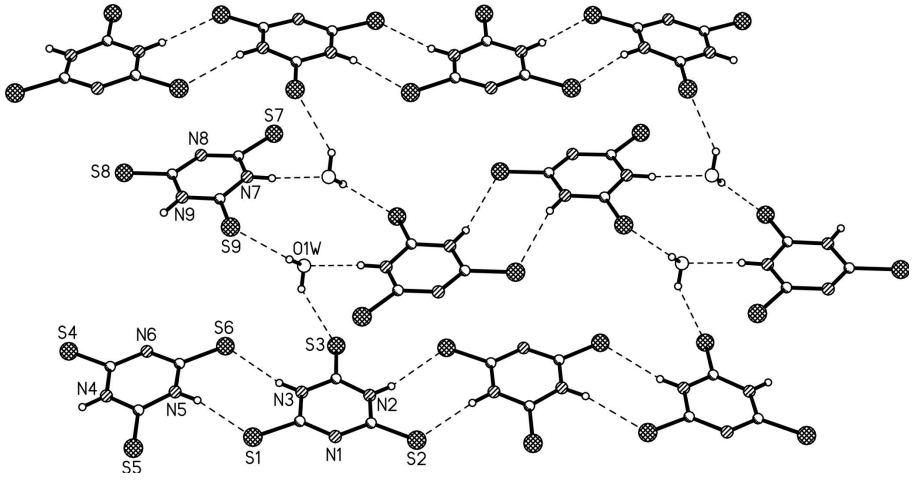



**Figure 2**

Hydrogen-bonded linking pattern of the separated unit in the crystal structure of the title compound.

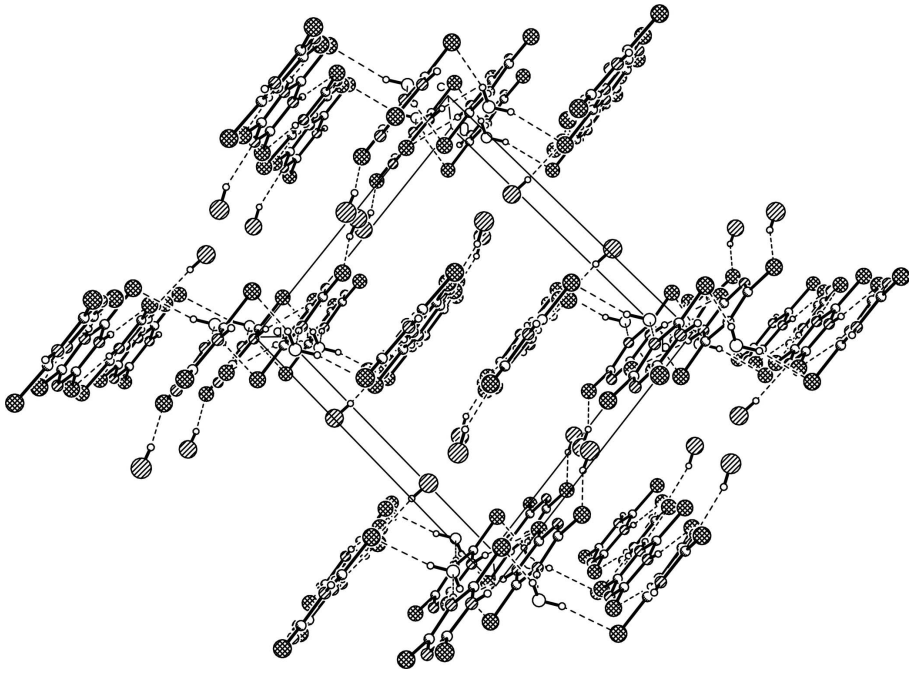


**Figure 3**

Packing diagram of the title compound; all hydrogen atoms bonded to carbon and carbon atoms of the trimethylammonium cations are omitted for clarity and the cations are represented with the hatched spheres.

**Tris(methylammonium thiocyanurate) monohydrate**

*Crystal data*

$3C_3H_{10}N^+·3C_3H_2N_3S_3^-·H_2O$
$M_r = 727.23$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 11.3466$ (1) Å
$b = 12.6474$ (1) Å
$c = 12.8135$ (1) Å
$\alpha = 76.950$ (1)°
$\beta = 84.762$ (1)°
$\gamma = 82.274$ (1)°
$V = 1771.51$ (3) Å$^3$
$Z = 2$
$F(000) = 764$
$D_x = 1.363$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8448 reflections
$\theta = 2.4$–$27.6$°
$\mu = 0.60$ mm$^{-1}$
$T = 296$ K
Block, colorless
0.55 × 0.40 × 0.09 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan *SADABS* (Bruker, 2007)
$T_{min} = 0.735$, $T_{max} = 0.948$
14356 measured reflections
6243 independent reflections
5406 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
$h = -13 \rightarrow 13$
$k = -15 \rightarrow 14$
$l = -15 \rightarrow 15$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.103$
$S = 1.05$
6243 reflections
388 parameters
12 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
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.8093P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.43$ e Å$^{-3}$
$\Delta\rho_{min} = -0.28$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.56389 (5) | 0.64769 (5) | −0.12395 (4) | 0.04362 (14) |
| N1 | 0.55437 (14) | 0.63614 (14) | 0.08580 (12) | 0.0364 (4) |
| C1 | 0.49478 (16) | 0.64115 (15) | −0.00121 (15) | 0.0339 (4) |
| O1W | 0.0658 (2) | 0.88067 (19) | −0.01243 (17) | 0.0861 (6) |
| H1WA | 0.1088 | 0.8189 | −0.0116 | 0.129* |
| H1WB | 0.0952 | 0.9280 | −0.0651 | 0.129* |
| S2 | 0.56375 (5) | 0.62637 (5) | 0.29317 (4) | 0.04494 (15) |
| N2 | 0.37391 (14) | 0.62546 (14) | 0.18974 (13) | 0.0370 (4) |
| H2 | 0.335 (2) | 0.624 (2) | 0.2500 (12) | 0.055* |
| C2 | 0.49461 (17) | 0.62957 (15) | 0.18250 (15) | 0.0343 (4) |
| S3 | 0.16216 (5) | 0.63313 (6) | 0.11585 (5) | 0.05253 (16) |
| N3 | 0.37320 (14) | 0.64138 (14) | 0.01016 (13) | 0.0377 (4) |
| H3 | 0.337 (2) | 0.646 (2) | −0.0466 (13) | 0.057* |
| C3 | 0.30893 (17) | 0.63316 (16) | 0.10501 (15) | 0.0364 (4) |
| S4 | 0.80586 (5) | 0.34932 (6) | 0.59567 (4) | 0.05156 (16) |
| N4 | 0.61463 (14) | 0.35390 (15) | 0.49243 (13) | 0.0407 (4) |
| H4 | 0.577 (2) | 0.361 (2) | 0.5519 (13) | 0.061* |
| C4 | 0.73630 (17) | 0.34937 (17) | 0.48581 (16) | 0.0381 (4) |
| S5 | 0.40260 (5) | 0.35735 (6) | 0.42068 (5) | 0.05557 (17) |
| N5 | 0.61589 (15) | 0.34088 (15) | 0.31875 (13) | 0.0404 (4) |
| H5 | 0.581 (2) | 0.343 (2) | 0.2620 (14) | 0.061* |
| C5 | 0.54998 (17) | 0.35067 (17) | 0.41032 (16) | 0.0380 (4) |
| S6 | 0.80673 (5) | 0.33438 (7) | 0.18635 (5) | 0.0681 (2) |
| N6 | 0.79689 (14) | 0.34433 (16) | 0.39149 (13) | 0.0428 (4) |

| | | | | |
|---|---|---|---|---|
| C6 | 0.73742 (18) | 0.34035 (19) | 0.30705 (16) | 0.0424 (5) |
| N10 | 1.04639 (17) | 0.32035 (19) | 0.3879 (2) | 0.0643 (6) |
| H10 | 0.9696 (10) | 0.331 (3) | 0.398 (3) | 0.096* |
| C10 | 1.0768 (4) | 0.4212 (3) | 0.3201 (4) | 0.1236 (17) |
| H10A | 1.0568 | 0.4792 | 0.3580 | 0.185* |
| H10B | 1.0332 | 0.4372 | 0.2565 | 0.185* |
| H10C | 1.1607 | 0.4145 | 0.3004 | 0.185* |
| N11 | 0.79117 (16) | 0.68045 (18) | 0.06442 (19) | 0.0581 (5) |
| H11 | 0.7233 (16) | 0.655 (2) | 0.078 (3) | 0.087* |
| C11 | 1.1012 (3) | 0.2929 (4) | 0.4905 (3) | 0.1159 (16) |
| H11A | 1.0825 | 0.3528 | 0.5260 | 0.174* |
| H11B | 1.1860 | 0.2787 | 0.4786 | 0.174* |
| H11C | 1.0711 | 0.2289 | 0.5345 | 0.174* |
| N12 | 0.50621 (19) | 0.00292 (17) | 0.28412 (18) | 0.0569 (5) |
| H12 | 0.4444 (18) | −0.015 (3) | 0.262 (2) | 0.085* |
| C12 | 1.0776 (4) | 0.2293 (4) | 0.3325 (4) | 0.1397 (19) |
| H12A | 1.0415 | 0.2473 | 0.2648 | 0.210* |
| H12B | 1.0489 | 0.1645 | 0.3759 | 0.210* |
| H12C | 1.1626 | 0.2169 | 0.3205 | 0.210* |
| C13 | 0.8621 (3) | 0.6173 (3) | −0.0050 (3) | 0.0944 (11) |
| H13A | 0.8258 | 0.6298 | −0.0722 | 0.142* |
| H13B | 0.8667 | 0.5410 | 0.0286 | 0.142* |
| H13C | 0.9409 | 0.6392 | −0.0175 | 0.142* |
| C14 | 0.8474 (3) | 0.6615 (3) | 0.1690 (3) | 0.0917 (11) |
| H14A | 0.7998 | 0.7031 | 0.2150 | 0.137* |
| H14B | 0.9258 | 0.6841 | 0.1566 | 0.137* |
| H14C | 0.8527 | 0.5852 | 0.2026 | 0.137* |
| C15 | 0.7683 (4) | 0.7964 (3) | 0.0190 (4) | 0.1141 (15) |
| H15A | 0.7320 | 0.8074 | −0.0480 | 0.171* |
| H15B | 0.8420 | 0.8280 | 0.0072 | 0.171* |
| H15C | 0.7156 | 0.8308 | 0.0678 | 0.171* |
| C16 | 0.4718 (3) | 0.0885 (2) | 0.3457 (3) | 0.0755 (8) |
| H16A | 0.4210 | 0.0612 | 0.4073 | 0.113* |
| H16B | 0.4300 | 0.1511 | 0.3012 | 0.113* |
| H16C | 0.5420 | 0.1090 | 0.3687 | 0.113* |
| C17 | 0.5840 (3) | 0.0438 (3) | 0.1878 (3) | 0.0974 (11) |
| H17A | 0.6051 | −0.0125 | 0.1479 | 0.146* |
| H17B | 0.6551 | 0.0639 | 0.2098 | 0.146* |
| H17C | 0.5423 | 0.1066 | 0.1434 | 0.146* |
| C18 | 0.5605 (3) | −0.0983 (2) | 0.3525 (3) | 0.0858 (10) |
| H18A | 0.5062 | −0.1218 | 0.4127 | 0.129* |
| H18B | 0.6331 | −0.0850 | 0.3776 | 0.129* |
| H18C | 0.5775 | −0.1541 | 0.3116 | 0.129* |
| N7 | 0.12163 (19) | 0.92130 (18) | 0.18856 (17) | 0.0574 (5) |
| H7 | 0.107 (3) | 0.916 (3) | 0.1258 (13) | 0.086* |
| S7 | 0.34408 (7) | 0.92115 (7) | 0.10863 (6) | 0.0737 (2) |
| C7 | 0.2383 (2) | 0.92934 (19) | 0.2065 (2) | 0.0538 (6) |
| S8 | 0.19623 (6) | 0.96108 (6) | 0.50398 (6) | 0.06475 (19) |

| | | | | |
|---|---|---|---|---|
| N8 | 0.26245 (17) | 0.94371 (16) | 0.30300 (17) | 0.0542 (5) |
| C8 | 0.1747 (2) | 0.95025 (18) | 0.3790 (2) | 0.0514 (5) |
| S9 | −0.11137 (7) | 0.92925 (8) | 0.23683 (7) | 0.0800 (2) |
| N9 | 0.05886 (18) | 0.94998 (17) | 0.35405 (17) | 0.0529 (5) |
| H9 | 0.0012 (19) | 0.964 (2) | 0.3986 (19) | 0.079* |
| C9 | 0.0290 (2) | 0.9339 (2) | 0.2601 (2) | 0.0539 (6) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0367 (3) | 0.0641 (3) | 0.0296 (3) | −0.0079 (2) | 0.0031 (2) | −0.0103 (2) |
| N1 | 0.0296 (8) | 0.0493 (10) | 0.0317 (8) | −0.0075 (7) | −0.0002 (6) | −0.0106 (7) |
| C1 | 0.0329 (9) | 0.0372 (10) | 0.0318 (10) | −0.0057 (8) | −0.0009 (8) | −0.0073 (8) |
| O1W | 0.0859 (15) | 0.0935 (15) | 0.0732 (13) | 0.0028 (12) | −0.0087 (11) | −0.0124 (11) |
| S2 | 0.0367 (3) | 0.0688 (4) | 0.0331 (3) | −0.0108 (2) | −0.0038 (2) | −0.0153 (2) |
| N2 | 0.0309 (8) | 0.0528 (10) | 0.0290 (8) | −0.0096 (7) | 0.0027 (6) | −0.0114 (7) |
| C2 | 0.0327 (9) | 0.0379 (10) | 0.0334 (10) | −0.0064 (8) | −0.0011 (8) | −0.0091 (8) |
| S3 | 0.0302 (3) | 0.0844 (4) | 0.0445 (3) | −0.0137 (3) | −0.0001 (2) | −0.0138 (3) |
| N3 | 0.0314 (8) | 0.0541 (10) | 0.0287 (8) | −0.0080 (7) | −0.0024 (7) | −0.0095 (7) |
| C3 | 0.0330 (10) | 0.0435 (11) | 0.0339 (10) | −0.0084 (8) | −0.0005 (8) | −0.0092 (8) |
| S4 | 0.0354 (3) | 0.0880 (4) | 0.0377 (3) | −0.0090 (3) | −0.0046 (2) | −0.0251 (3) |
| N4 | 0.0304 (8) | 0.0639 (11) | 0.0310 (9) | −0.0094 (8) | 0.0018 (7) | −0.0164 (8) |
| C4 | 0.0321 (10) | 0.0489 (11) | 0.0354 (10) | −0.0077 (8) | −0.0004 (8) | −0.0127 (9) |
| S5 | 0.0297 (3) | 0.0942 (5) | 0.0464 (3) | −0.0143 (3) | 0.0000 (2) | −0.0197 (3) |
| N5 | 0.0323 (8) | 0.0617 (11) | 0.0308 (9) | −0.0107 (8) | −0.0010 (7) | −0.0150 (8) |
| C5 | 0.0337 (10) | 0.0475 (11) | 0.0344 (10) | −0.0096 (8) | −0.0011 (8) | −0.0102 (8) |
| S6 | 0.0400 (3) | 0.1345 (7) | 0.0371 (3) | −0.0152 (3) | 0.0055 (2) | −0.0340 (4) |
| N6 | 0.0305 (8) | 0.0669 (12) | 0.0346 (9) | −0.0093 (8) | 0.0005 (7) | −0.0176 (8) |
| C6 | 0.0351 (10) | 0.0594 (13) | 0.0351 (11) | −0.0085 (9) | 0.0001 (8) | −0.0143 (9) |
| N10 | 0.0338 (10) | 0.0679 (14) | 0.0813 (15) | −0.0089 (10) | 0.0012 (10) | 0.0042 (11) |
| C10 | 0.101 (3) | 0.090 (3) | 0.164 (4) | −0.046 (2) | −0.049 (3) | 0.041 (3) |
| N11 | 0.0320 (9) | 0.0626 (13) | 0.0851 (15) | −0.0111 (9) | 0.0005 (10) | −0.0259 (11) |
| C11 | 0.0499 (17) | 0.194 (4) | 0.083 (2) | −0.014 (2) | 0.0008 (16) | 0.011 (3) |
| N12 | 0.0542 (12) | 0.0575 (12) | 0.0608 (13) | −0.0030 (10) | −0.0084 (10) | −0.0165 (10) |
| C12 | 0.128 (4) | 0.116 (3) | 0.172 (5) | 0.047 (3) | −0.033 (3) | −0.048 (3) |
| C13 | 0.0535 (17) | 0.131 (3) | 0.116 (3) | −0.0040 (18) | −0.0003 (17) | −0.068 (2) |
| C14 | 0.0481 (16) | 0.145 (3) | 0.091 (2) | −0.0186 (18) | −0.0029 (15) | −0.040 (2) |
| C15 | 0.092 (3) | 0.065 (2) | 0.185 (4) | −0.0214 (18) | −0.031 (3) | −0.011 (2) |
| C16 | 0.086 (2) | 0.0609 (16) | 0.082 (2) | −0.0163 (15) | 0.0168 (16) | −0.0243 (15) |
| C17 | 0.097 (3) | 0.110 (3) | 0.072 (2) | 0.010 (2) | 0.0189 (18) | −0.0126 (19) |
| C18 | 0.092 (2) | 0.0576 (17) | 0.106 (3) | 0.0014 (15) | −0.032 (2) | −0.0100 (16) |
| N7 | 0.0571 (12) | 0.0642 (13) | 0.0535 (12) | −0.0103 (10) | −0.0052 (10) | −0.0158 (10) |
| S7 | 0.0663 (4) | 0.0802 (5) | 0.0686 (4) | −0.0057 (4) | 0.0147 (3) | −0.0129 (4) |
| C7 | 0.0520 (13) | 0.0428 (12) | 0.0649 (15) | −0.0075 (10) | 0.0010 (11) | −0.0089 (11) |
| S8 | 0.0578 (4) | 0.0798 (5) | 0.0621 (4) | −0.0077 (3) | −0.0098 (3) | −0.0245 (3) |
| N8 | 0.0469 (11) | 0.0538 (11) | 0.0646 (13) | −0.0095 (9) | −0.0011 (9) | −0.0174 (10) |
| C8 | 0.0485 (13) | 0.0445 (12) | 0.0630 (15) | −0.0089 (10) | −0.0031 (11) | −0.0136 (11) |
| S9 | 0.0556 (4) | 0.1196 (7) | 0.0727 (5) | −0.0239 (4) | −0.0104 (3) | −0.0271 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N9 | 0.0463 (11) | 0.0605 (12) | 0.0567 (12) | −0.0117 (9) | −0.0006 (9) | −0.0200 (10) |
| C9 | 0.0527 (14) | 0.0547 (14) | 0.0558 (14) | −0.0122 (11) | −0.0021 (11) | −0.0122 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C1 | 1.6819 (19) | C11—H11C | 0.9600 |
| N1—C1 | 1.341 (2) | N12—C16 | 1.470 (3) |
| N1—C2 | 1.348 (2) | N12—C18 | 1.471 (4) |
| C1—N3 | 1.374 (2) | N12—C17 | 1.486 (4) |
| O1W—H1WA | 0.8619 | N12—H12 | 0.851 (10) |
| O1W—H1WB | 0.8691 | C12—H12A | 0.9600 |
| S2—C2 | 1.6723 (19) | C12—H12B | 0.9600 |
| N2—C3 | 1.346 (3) | C12—H12C | 0.9600 |
| N2—C2 | 1.371 (2) | C13—H13A | 0.9600 |
| N2—H2 | 0.850 (10) | C13—H13B | 0.9600 |
| S3—C3 | 1.6585 (19) | C13—H13C | 0.9600 |
| N3—C3 | 1.349 (2) | C14—H14A | 0.9600 |
| N3—H3 | 0.854 (10) | C14—H14B | 0.9600 |
| S4—C4 | 1.675 (2) | C14—H14C | 0.9600 |
| N4—C5 | 1.347 (3) | C15—H15A | 0.9600 |
| N4—C4 | 1.370 (2) | C15—H15B | 0.9600 |
| N4—H4 | 0.857 (10) | C15—H15C | 0.9600 |
| C4—N6 | 1.345 (3) | C16—H16A | 0.9600 |
| S5—C5 | 1.658 (2) | C16—H16B | 0.9600 |
| N5—C5 | 1.354 (3) | C16—H16C | 0.9600 |
| N5—C6 | 1.373 (3) | C17—H17A | 0.9600 |
| N5—H5 | 0.855 (10) | C17—H17B | 0.9600 |
| S6—C6 | 1.682 (2) | C17—H17C | 0.9600 |
| N6—C6 | 1.340 (3) | C18—H18A | 0.9600 |
| N10—C10 | 1.436 (4) | C18—H18B | 0.9600 |
| N10—C11 | 1.456 (4) | C18—H18C | 0.9600 |
| N10—C12 | 1.473 (5) | N7—C9 | 1.347 (3) |
| N10—H10 | 0.865 (10) | N7—C7 | 1.385 (3) |
| C10—H10A | 0.9600 | N7—H7 | 0.856 (10) |
| C10—H10B | 0.9600 | S7—C7 | 1.666 (3) |
| C10—H10C | 0.9600 | C7—N8 | 1.346 (3) |
| N11—C13 | 1.447 (4) | S8—C8 | 1.680 (3) |
| N11—C15 | 1.448 (4) | N8—C8 | 1.336 (3) |
| N11—C14 | 1.493 (4) | C8—N9 | 1.381 (3) |
| N11—H11 | 0.863 (10) | S9—C9 | 1.658 (3) |
| C11—H11A | 0.9600 | N9—C9 | 1.346 (3) |
| C11—H11B | 0.9600 | N9—H9 | 0.857 (10) |
| | | | |
| C1—N1—C2 | 119.69 (16) | C18—N12—H12 | 105 (2) |
| N1—C1—N3 | 118.93 (16) | C17—N12—H12 | 107 (2) |
| N1—C1—S1 | 122.20 (14) | N10—C12—H12A | 109.5 |
| N3—C1—S1 | 118.87 (14) | N10—C12—H12B | 109.5 |
| H1WA—O1W—H1WB | 106.8 | H12A—C12—H12B | 109.5 |

| | | | |
|---|---|---|---|
| C3—N2—C2 | 124.13 (16) | N10—C12—H12C | 109.5 |
| C3—N2—H2 | 116.5 (17) | H12A—C12—H12C | 109.5 |
| C2—N2—H2 | 119.1 (17) | H12B—C12—H12C | 109.5 |
| N1—C2—N2 | 118.82 (17) | N11—C13—H13A | 109.5 |
| N1—C2—S2 | 121.75 (14) | N11—C13—H13B | 109.5 |
| N2—C2—S2 | 119.43 (14) | H13A—C13—H13B | 109.5 |
| C3—N3—C1 | 124.00 (16) | N11—C13—H13C | 109.5 |
| C3—N3—H3 | 118.8 (18) | H13A—C13—H13C | 109.5 |
| C1—N3—H3 | 117.2 (18) | H13B—C13—H13C | 109.5 |
| N2—C3—N3 | 114.34 (17) | N11—C14—H14A | 109.5 |
| N2—C3—S3 | 122.95 (14) | N11—C14—H14B | 109.5 |
| N3—C3—S3 | 122.71 (15) | H14A—C14—H14B | 109.5 |
| C5—N4—C4 | 124.14 (17) | N11—C14—H14C | 109.5 |
| C5—N4—H4 | 117.5 (18) | H14A—C14—H14C | 109.5 |
| C4—N4—H4 | 118.4 (18) | H14B—C14—H14C | 109.5 |
| N6—C4—N4 | 119.15 (17) | N11—C15—H15A | 109.5 |
| N6—C4—S4 | 121.71 (15) | N11—C15—H15B | 109.5 |
| N4—C4—S4 | 119.14 (14) | H15A—C15—H15B | 109.5 |
| C5—N5—C6 | 123.76 (17) | N11—C15—H15C | 109.5 |
| C5—N5—H5 | 119.5 (18) | H15A—C15—H15C | 109.5 |
| C6—N5—H5 | 116.3 (18) | H15B—C15—H15C | 109.5 |
| N4—C5—N5 | 114.15 (17) | N12—C16—H16A | 109.5 |
| N4—C5—S5 | 122.96 (15) | N12—C16—H16B | 109.5 |
| N5—C5—S5 | 122.90 (15) | H16A—C16—H16B | 109.5 |
| C6—N6—C4 | 119.38 (17) | N12—C16—H16C | 109.5 |
| N6—C6—N5 | 119.31 (17) | H16A—C16—H16C | 109.5 |
| N6—C6—S6 | 122.21 (15) | H16B—C16—H16C | 109.5 |
| N5—C6—S6 | 118.48 (15) | N12—C17—H17A | 109.5 |
| C10—N10—C11 | 113.8 (3) | N12—C17—H17B | 109.5 |
| C10—N10—C12 | 110.8 (4) | H17A—C17—H17B | 109.5 |
| C11—N10—C12 | 109.4 (3) | N12—C17—H17C | 109.5 |
| C10—N10—H10 | 104 (2) | H17A—C17—H17C | 109.5 |
| C11—N10—H10 | 110 (2) | H17B—C17—H17C | 109.5 |
| C12—N10—H10 | 108 (2) | N12—C18—H18A | 109.5 |
| N10—C10—H10A | 109.5 | N12—C18—H18B | 109.5 |
| N10—C10—H10B | 109.5 | H18A—C18—H18B | 109.5 |
| H10A—C10—H10B | 109.5 | N12—C18—H18C | 109.5 |
| N10—C10—H10C | 109.5 | H18A—C18—H18C | 109.5 |
| H10A—C10—H10C | 109.5 | H18B—C18—H18C | 109.5 |
| H10B—C10—H10C | 109.5 | C9—N7—C7 | 123.6 (2) |
| C13—N11—C15 | 115.1 (3) | C9—N7—H7 | 118 (2) |
| C13—N11—C14 | 109.7 (2) | C7—N7—H7 | 118 (2) |
| C15—N11—C14 | 110.3 (3) | N8—C7—N7 | 118.7 (2) |
| C13—N11—H11 | 107 (2) | N8—C7—S7 | 122.06 (19) |
| C15—N11—H11 | 108 (2) | N7—C7—S7 | 119.2 (2) |
| C14—N11—H11 | 107 (2) | C8—N8—C7 | 120.0 (2) |
| N10—C11—H11A | 109.5 | N8—C8—N9 | 118.8 (2) |
| N10—C11—H11B | 109.5 | N8—C8—S8 | 123.78 (19) |

| | | | |
|---|---|---|---|
| H11A—C11—H11B | 109.5 | N9—C8—S8 | 117.46 (18) |
| N10—C11—H11C | 109.5 | C9—N9—C8 | 123.9 (2) |
| H11A—C11—H11C | 109.5 | C9—N9—H9 | 116 (2) |
| H11B—C11—H11C | 109.5 | C8—N9—H9 | 119 (2) |
| C16—N12—C18 | 111.1 (2) | N9—C9—N7 | 114.7 (2) |
| C16—N12—C17 | 110.3 (2) | N9—C9—S9 | 121.64 (19) |
| C18—N12—C17 | 112.8 (3) | N7—C9—S9 | 123.69 (19) |
| C16—N12—H12 | 110 (2) | | |

| | | | |
|---|---|---|---|
| C2—N1—C1—N3 | 1.1 (3) | N4—C4—N6—C6 | −2.1 (3) |
| C2—N1—C1—S1 | −179.13 (15) | S4—C4—N6—C6 | 177.44 (17) |
| C1—N1—C2—N2 | 1.6 (3) | C4—N6—C6—N5 | −0.4 (3) |
| C1—N1—C2—S2 | −178.39 (15) | C4—N6—C6—S6 | 179.78 (17) |
| C3—N2—C2—N1 | −3.5 (3) | C5—N5—C6—N6 | 3.4 (3) |
| C3—N2—C2—S2 | 176.45 (16) | C5—N5—C6—S6 | −176.76 (17) |
| N1—C1—N3—C3 | −2.3 (3) | C9—N7—C7—N8 | 4.3 (4) |
| S1—C1—N3—C3 | 177.94 (16) | C9—N7—C7—S7 | −175.60 (19) |
| C2—N2—C3—N3 | 2.4 (3) | N7—C7—N8—C8 | −0.2 (3) |
| C2—N2—C3—S3 | −177.34 (16) | S7—C7—N8—C8 | 179.74 (18) |
| C1—N3—C3—N2 | 0.6 (3) | C7—N8—C8—N9 | −4.6 (3) |
| C1—N3—C3—S3 | −179.71 (15) | C7—N8—C8—S8 | 176.47 (18) |
| C5—N4—C4—N6 | 1.9 (3) | N8—C8—N9—C9 | 6.1 (4) |
| C5—N4—C4—S4 | −177.66 (17) | S8—C8—N9—C9 | −174.96 (19) |
| C4—N4—C5—N5 | 0.9 (3) | C8—N9—C9—N7 | −2.2 (3) |
| C4—N4—C5—S5 | −179.48 (17) | C8—N9—C9—S9 | 177.35 (18) |
| C6—N5—C5—N4 | −3.5 (3) | C7—N7—C9—N9 | −3.1 (4) |
| C6—N5—C5—S5 | 176.84 (17) | C7—N7—C9—S9 | 177.44 (19) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*WA*···S3 | 0.86 | 2.57 | 3.284 (2) | 140 |
| O1*W*—H1*WB*···S9$^{i}$ | 0.87 | 2.52 | 3.355 (2) | 161 |
| N2—H2···S4$^{ii}$ | 0.85 (1) | 2.48 (1) | 3.3196 (17) | 168 (2) |
| N3—H3···S6$^{iii}$ | 0.85 (1) | 2.48 (1) | 3.3203 (17) | 168 (2) |
| N4—H4···S2$^{ii}$ | 0.86 (1) | 2.45 (1) | 3.2962 (17) | 169 (2) |
| N5—H5···S1$^{iii}$ | 0.86 (1) | 2.49 (1) | 3.3316 (17) | 167 (2) |
| N7—H7···O1*W* | 0.86 (1) | 2.03 (1) | 2.878 (3) | 172 (3) |
| N9—H9···S8$^{iv}$ | 0.86 (1) | 2.63 (1) | 3.470 (2) | 167 (3) |
| N10—H10···N6 | 0.87 (1) | 1.95 (1) | 2.804 (3) | 168 (3) |
| N11—H11···N1 | 0.86 (1) | 1.95 (1) | 2.795 (2) | 164 (3) |
| N12—H12···S7$^{v}$ | 0.85 (1) | 2.69 (2) | 3.439 (2) | 148 (3) |

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