

Andrei S. Batsanov\* and  
Igor F. PerepichkaDepartment of Chemistry, University of  
Durham, South Road, Durham DH1 3LE,  
EnglandCorrespondence e-mail:  
a.s.batsanov@durham.ac.uk

## Key indicators

Single-crystal X-ray study  
 $T = 150$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å  
 $R$  factor = 0.041  
 $wR$  factor = 0.106  
Data-to-parameter ratio = 9.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.9-Dicyanomethylene-4,5-dinitrofluorene-  
2,7-disulfonamideThe title compound,  $\text{C}_{16}\text{H}_8\text{N}_6\text{O}_8\text{S}_2$ , has a twisted fluorene moiety due to steric repulsion between the 4- and 5-nitro groups.

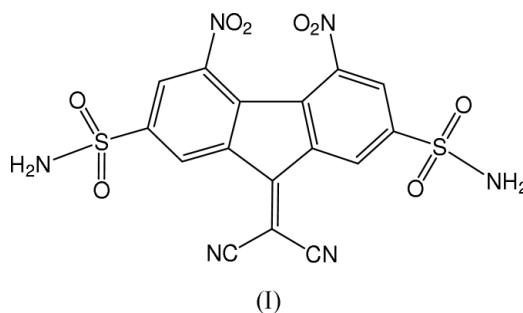
Received 15 September 2004

Accepted 22 September 2004

Online 30 September 2004

## Comment

2,4,5,7-Tetranitro-9-dicyanomethylene fluorene (DTeF) (Silverman *et al.*, 1974) is a strong electron acceptor, used as a component of charge-transfer complexes (CTC) (Perepichka *et al.*, 1998; Batsanov, Bryce *et al.*, 2001; Batsanov, Perepichka *et al.*, 2001). We also prepared and structurally characterized similar complexes with 2,7-bis(*n*-butylsulfonyl)- and 2,7-bis(phenylsulfonyl)-9-dicyanomethylene-4,5-dinitrofluorene (Perepichka *et al.*, 2000). The title compound, (I), was prepared in the course of the same study.



The fluorene system of (I), like that of DTeF, adopts a twisted conformation (Fig. 1), due to steric repulsion between the nitro groups in positions 4 and 5 [intramolecular contacts  $\text{O}42 \cdots \text{N}5 = 2.646$  (5) Å and  $\text{O}51 \cdots \text{N}4 = 2.674$  (5) Å]. The strain is relieved by (i) both nitro groups tilting out of the aromatic plane in opposite directions and (ii) the fluorene moiety itself twisting substantially. Thus, the 13 C atoms of the fluorene moiety show an average deviation of 0.13 Å from their mean plane. Both benzene rings adopt 'sofa' conformations, atoms C11 and C12 deviating by 0.12 and 0.06 Å from the planes of the essentially planar moieties C1–C4/C10 and C5–C8/C13, respectively. The latter planes form a dihedral angle of 15.2 (3)°. The twist around the  $\text{C}9=\text{C}14$  bond, *i.e.* the dihedral angle between the C9/C10/C13/C14 and C9/C14/C15/C16/N15/N16 planes, is 11.5 (3)°. The bond lengths and angles in (I) (Table 1) are consistent with those observed in other distorted fluorene systems.

The  $\text{NH}_2$  groups adopt *trans* orientations with respect to the mean fluorene plane. All amine H atoms participate in intermolecular hydrogen bonds (Table 2), although one of these ( $\text{H}71 \cdots \text{O}41$ ) is very weak.

## Experimental

4,5-Dinitro-9-fluorenone-2,7-disulfonyl dichloride [(II); 200 mg, 0.43 mol] (Mysyk *et al.*, 1997) was dissolved in dry dioxane (10 ml) and ammonia (35% solution in water, 0.2 ml) was added dropwise with intense stirring. The mixture was stirred at room temperature for 1 h and poured into water. The solid was filtered off, washed with warm water, dried and recrystallized from acetone, yielding pale yellow crystals of 4,5-dinitro-9-fluorenone-2,7-disulfonamide, (III) (130 mg, yield 71%, m.p. >573 K).  $^1\text{H}$  NMR (400 MHz, acetone- $d_6$ ):  $\delta$  8.63 (2H, *d*,  $J = 1.5$  Hz, H-3,6), 8.50 (2H, *d*,  $J = 1.5$  Hz, H-1,8), 7.19 (4H, *s*,  $\text{SO}_2\text{NH}_2$ );  $^{13}\text{C}$  NMR (100 MHz, acetone- $d_6$ ):  $\delta$  186.56 (C=O), 149.28, 147.15, 139.40, 136.61, 128.86, 126.07. Analysis found: C 36.42, H 1.95, N 13.18, S 14.89%;  $\text{C}_{13}\text{H}_8\text{N}_4\text{O}_9\text{S}_2$  requires: C 36.45, H 1.88, N 13.08, S 14.97%. Compound (III) (300 mg, 0.70 mmol) and malononitrile (100 mg, 167 mmol) in dimethylformamide (1.5 ml) were stirred at room temperature for 4 h and diluted with methanol (5 ml), resulting in precipitation. After keeping this solution at 273 K for 6 h, the solid obtained was filtered off and washed with water, yielding crude product (I) as a yellow-green solid. This was dissolved in a minimal amount of hot acetone and diluted with a fourfold volume of hot methanol. On cooling, a bright yellow solid was collected, washed with methanol and dried, giving 210 mg of (I) (yield 63%), m.p. >573 K.  $^1\text{H}$  NMR (300 MHz, acetone- $d_6$  + *ca* 0.2 drop  $\text{CF}_3\text{CO}_2\text{D}$ ):  $\delta$  9.36 (2H, *d*,  $J = 1.4$  Hz, H-1,8), 8.71 (2H, *d*,  $J = 1.4$  Hz, H-3,6), 7.32 (4H, *s*,  $\text{SO}_2\text{NH}_2$ ). Analysis found: C 40.22, H 1.65, N 17.78, S 13.37;  $\text{C}_{16}\text{H}_8\text{N}_6\text{O}_8\text{S}_2$  requires: C 40.34, H 1.69, N 17.64, S 13.46%. Single crystals of (I) of X-ray quality were obtained by slow evaporation (over several days) at room temperature of a solution of (I) (10 mg) in PhCl (10 ml) and acetonitrile (3 ml).

### Crystal data

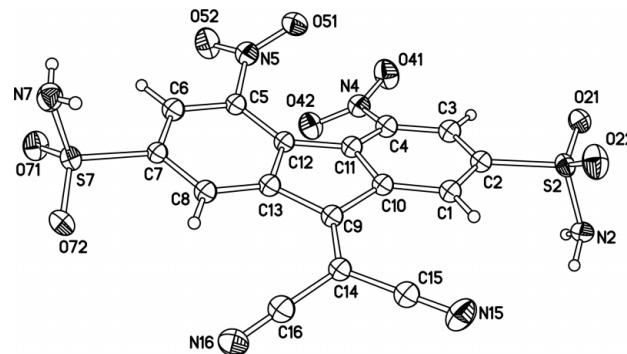
$\text{C}_{16}\text{H}_8\text{N}_6\text{O}_8\text{S}_2$	Cu $K\alpha$ radiation
$M_r = 476.40$	Cell parameters from 25 reflections
Orthorhombic, $Pna2_1$	$\theta = 15.5\text{--}25.0^\circ$
$a = 9.574$ (2) Å	$\mu = 3.30$ mm $^{-1}$
$b = 10.778$ (6) Å	$T = 150$ (2) K
$c = 17.461$ (5) Å	Needle, yellow
$V = 1801.8$ (12) Å $^3$	$0.45 \times 0.06 \times 0.05$ mm
$Z = 4$	
$D_x = 1.756$ Mg m $^{-3}$	

### Data collection

Rigaku AFC-6S four-circle diffractometer	1730 reflections with $I > 2\sigma(I)$
$2\theta/\omega$ scans	$R_{\text{int}} = 0.033$
Absorption correction: $\psi$ scan (TEXSAN; Molecular Structure Corporation, 1989)	$\theta_{\text{max}} = 75.1^\circ$
$T_{\text{min}} = 0.745$ , $T_{\text{max}} = 0.848$	$h = -1 \rightarrow 12$
2381 measured reflections	$k = -1 \rightarrow 13$
1942 independent reflections	$l = -1 \rightarrow 21$
	3 standard reflections every 147 reflections
	intensity decay: 0.9%

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.7842P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.106$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.37$ e Å $^{-3}$
1942 reflections	$\Delta\rho_{\text{min}} = -0.43$ e Å $^{-3}$
199 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	Flack parameter = 0.01 (3)



**Figure 1**  
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

**Table 1**

Selected geometric parameters (Å, °).

S2—O22	1.424 (4)	C6—C7	1.384 (6)
S2—O21	1.433 (4)	C7—C8	1.394 (6)
S2—N2	1.612 (4)	C8—C13	1.387 (7)
S2—C2	1.785 (5)	C9—C14	1.358 (6)
S7—O72	1.428 (4)	C9—C13	1.469 (6)
S7—O71	1.432 (3)	C9—C10	1.475 (6)
S7—N7	1.606 (4)	C10—C11	1.410 (6)
S7—C7	1.784 (5)	C11—C12	1.473 (6)
C1—C2	1.383 (7)	C12—C13	1.429 (6)
C1—C10	1.396 (6)	C14—C15	1.435 (6)
C2—C3	1.381 (7)	C14—C16	1.446 (7)
C3—C4	1.393 (6)	C15—N15	1.144 (7)
C4—C11	1.387 (7)	C16—N16	1.146 (7)
C4—N4	1.476 (6)	N4—O41	1.218 (5)
C5—C12	1.387 (6)	N4—O42	1.236 (5)
C5—C6	1.391 (6)	N5—O52	1.219 (5)
C5—N5	1.465 (6)	N5—O51	1.239 (5)
C2—C1—C10	117.7 (4)	C14—C9—C10	127.1 (4)
C3—C2—C1	122.6 (4)	C13—C9—C10	106.8 (4)
C3—C2—S2	117.4 (4)	C1—C10—C11	121.5 (4)
C1—C2—S2	120.0 (4)	C1—C10—C9	130.4 (4)
C2—C3—C4	118.2 (5)	C11—C10—C9	108.0 (4)
C11—C4—C3	121.7 (4)	C4—C11—C10	117.4 (4)
C11—C4—N4	122.2 (4)	C4—C11—C12	134.2 (4)
C3—C4—N4	115.8 (4)	C10—C11—C12	108.3 (4)
C12—C5—C6	122.6 (4)	C5—C12—C13	116.8 (4)
C12—C5—N5	122.7 (4)	C5—C12—C11	135.2 (4)
C6—C5—N5	114.5 (4)	C13—C12—C11	107.8 (4)
C7—C6—C5	118.5 (4)	C8—C13—C12	121.7 (4)
C6—C7—C8	121.9 (4)	C8—C13—C9	130.2 (4)
C6—C7—S7	117.8 (3)	C12—C13—C9	107.9 (4)
C8—C7—S7	120.2 (4)	C9—C14—C15	123.4 (4)
C13—C8—C7	118.3 (4)	C9—C14—C16	122.2 (4)
C14—C9—C13	125.9 (4)	C15—C14—C16	114.4 (4)
C1—C2—S2—N2	79.0 (4)	C8—C7—S7—N7	96.3 (4)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
N2—H21 $\cdots$ O72 <sup>i</sup>	0.89	2.03	2.878 (6)	159
N2—H22 $\cdots$ O52 <sup>ii</sup>	0.89	2.10	2.925 (5)	153
N7—H71 $\cdots$ O41 <sup>iii</sup>	0.89	2.51	3.116 (6)	126
N7—H72 $\cdots$ N2 <sup>iv</sup>	0.89	2.24	3.103 (6)	162

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

Owing to an insufficient number of observed reflections, only the S, O and N atoms were refined with anisotropic displacement parameters, the C atoms being refined in isotropic approximation. Amine H atoms were refined in isotropic approximation, then constrained with the same bond direction but idealized N–H bond lengths (0.89 Å). Other H atoms were treated as riding in idealized positions, with C–H bond lengths of 0.95 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

The authors thank Professor M. R. Bryce for fruitful advice. IFP thanks the Royal Society of Chemistry for an International Author grant.

## References

- Batsanov, A. S., Bryce, M. R., Chesney, A., Howard, J. A. K., John, D. E., Moore, A. J., Wood, C. L., Gershtenman, H., Becker, J. Y., Khodorkovsky, V. Y., Ellern, A., Bernstein, J., Perepichka, I. F., Rotello, V., Gray, M. & Cuello, A. O. (2001). *J. Mater. Chem.* **10**, 2181–2191.
- Batsanov, A. S., Perepichka, I. F., Bryce, M. R. & Howard, J. A. K. (2001). *Acta Cryst.* **C57**, 1299–1302.
- Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1989). *TEXSAN*. Version 5.1. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Mysyk, D. D., Perepichka, I. F. & Sokolov, N. I. (1997). *J. Chem. Soc. Perkin Trans. 2*, pp. 537–545.
- Perepichka, I. F., Kuz'mina, L. G., Perepichka, D. F., Bryce, M. R., Goldenberg, L. M., Popov, A. F. & Howard, J. A. K. (1998). *J. Org. Chem.* **63**, 6484–6493.
- Perepichka, I. F., Popov, A. F., Orekhova, T. V., Bryce, M. R., Andrievskii, A. M., Batsanov, A. S., Howard, J. A. K. & Sokolov, N. I. (2000). *J. Org. Chem.* **65**, 3053–3063.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Silverman, J., Yannoni, N. F. & Krukoni, A. P. (1974). *Acta Cryst.* **B30**, 1474–1480.

## supporting information

*Acta Cryst.* (2004). E60, o1892–o1894 [https://doi.org/10.1107/S160053680402361X]

## 9-Dicyanomethylene-4,5-dinitrofluorene-2,7-disulfonamide

Andrei S. Batsanov and Igor F. Perepichka

## 9-Dicyanomethylene-4,5-dinitrofluorene-2,7-disulfonamide

*Crystal data*

$C_{16}H_8N_6O_8S_2$

$M_r = 476.40$

Orthorhombic,  $Pna2_1$

$a = 9.574$  (2) Å

$b = 10.778$  (6) Å

$c = 17.461$  (5) Å

$V = 1801.8$  (12) Å<sup>3</sup>

$Z = 4$

$F(000) = 968$

$D_x = 1.756$  Mg m<sup>-3</sup>

Melting point > 573 K

Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å

Cell parameters from 25 reflections

$\theta = 15.5$ – $25.0^\circ$

$\mu = 3.30$  mm<sup>-1</sup>

$T = 150$  K

Needle, yellow

$0.45 \times 0.06 \times 0.05$  mm

*Data collection*

Rigaku AFC-6S four-circle  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$2\theta/\omega$  scans

Absorption correction:  $\psi$  scan

(TEXSAN; Molecular Structure Corporation,  
1989)

$T_{\min} = 0.745$ ,  $T_{\max} = 0.848$

2381 measured reflections

1942 independent reflections

1730 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 75.1^\circ$ ,  $\theta_{\min} = 4.8^\circ$

$h = -1 \rightarrow 12$

$k = -1 \rightarrow 13$

$l = -1 \rightarrow 21$

3 standard reflections every 147 reflections

intensity decay: 0.9%

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.106$

$S = 1.03$

1942 reflections

199 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.7842P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.37$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.43$  e Å<sup>-3</sup>

Absolute structure: Flack (1983), 28 Friedel  
pairs [CHECK]

Absolute structure parameter: 0.01 (3)

*Special details*

**Experimental.** Needle-like crystal aligned approximately along the  $\varphi$  axis. 118 Friedel pairs has been measured. exptl\_absorpt\_correction\_type psi-scan (TEXSAN; Molecular Structure Corporation, 1989), on 108  $\psi$ -scans of 3 reflections

**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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S2	0.32267 (12)	0.29279 (10)	0.30553 (7)	0.0227 (2)
S7	0.85953 (12)	0.95636 (10)	-0.00931 (7)	0.0214 (2)
C1	0.4962 (5)	0.4139 (4)	0.2047 (3)	0.0215 (9)*
H1	0.5019	0.3380	0.1773	0.026*
C2	0.4190 (5)	0.4233 (4)	0.2714 (3)	0.0212 (9)*
C3	0.4144 (5)	0.5305 (4)	0.3147 (3)	0.0248 (9)*
H3	0.3639	0.5331	0.3615	0.030*
C4	0.4858 (5)	0.6344 (4)	0.2879 (3)	0.0242 (10)*
C5	0.6182 (5)	0.8599 (4)	0.1692 (3)	0.0207 (9)*
C6	0.6941 (5)	0.9288 (4)	0.1162 (3)	0.0236 (10)*
H6	0.6960	1.0168	0.1187	0.028*
C7	0.7667 (5)	0.8662 (4)	0.0596 (3)	0.0185 (9)*
C8	0.7648 (5)	0.7372 (4)	0.0541 (3)	0.0213 (9)*
H8	0.8123	0.6958	0.0138	0.026*
C9	0.6638 (5)	0.5371 (4)	0.1149 (3)	0.0201 (9)*
C10	0.5655 (5)	0.5201 (4)	0.1790 (3)	0.0200 (9)*
C11	0.5517 (5)	0.6348 (4)	0.2171 (3)	0.0211 (9)*
C12	0.6193 (5)	0.7312 (4)	0.1702 (3)	0.0195 (9)*
C13	0.6921 (5)	0.6707 (4)	0.1093 (3)	0.0209 (9)*
C14	0.7247 (5)	0.4468 (4)	0.0721 (3)	0.0207 (9)*
C15	0.6810 (5)	0.3196 (4)	0.0736 (3)	0.0253 (10)*
C16	0.8411 (5)	0.4717 (5)	0.0216 (3)	0.0278 (10)*
N2	0.4291 (4)	0.1993 (4)	0.3479 (2)	0.0237 (9)
H21	0.4669	0.2361	0.3885	0.028*
H22	0.4937	0.1670	0.3171	0.028*
N4	0.4993 (4)	0.7382 (4)	0.3425 (2)	0.0272 (9)*
N5	0.5253 (4)	0.9336 (4)	0.2179 (2)	0.0276 (9)*
N7	0.7527 (4)	1.0028 (4)	-0.0741 (2)	0.0290 (9)
H71	0.7014	1.0593	-0.0498	0.035*
H72	0.7186	0.9356	-0.0971	0.035*
N15	0.6447 (5)	0.2185 (4)	0.0715 (3)	0.0365 (11)
N16	0.9357 (5)	0.4914 (4)	-0.0167 (3)	0.0386 (11)
O21	0.2300 (4)	0.3387 (3)	0.3634 (2)	0.0324 (8)

O22	0.2694 (4)	0.2294 (4)	0.2402 (2)	0.0374 (9)
O41	0.4083 (4)	0.7526 (4)	0.3904 (2)	0.0384 (9)
O42	0.6069 (4)	0.8012 (3)	0.3374 (2)	0.0328 (8)
O51	0.4081 (4)	0.8909 (3)	0.2326 (2)	0.0309 (8)
O52	0.5671 (4)	1.0355 (3)	0.2378 (2)	0.0405 (10)
O71	0.9091 (4)	1.0654 (3)	0.0289 (2)	0.0296 (8)
O72	0.9576 (4)	0.8737 (3)	-0.0438 (2)	0.0313 (8)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S2	0.0195 (5)	0.0265 (5)	0.0222 (5)	-0.0038 (4)	-0.0003 (5)	0.0048 (5)
S7	0.0202 (5)	0.0226 (5)	0.0213 (5)	-0.0031 (4)	0.0013 (5)	-0.0003 (5)
N2	0.020 (2)	0.0255 (18)	0.025 (2)	0.0030 (15)	-0.0008 (17)	0.0011 (16)
N7	0.029 (2)	0.032 (2)	0.026 (2)	-0.0028 (17)	-0.0022 (19)	0.0052 (18)
N15	0.050 (3)	0.024 (2)	0.036 (3)	0.002 (2)	0.002 (2)	-0.0027 (18)
N16	0.037 (3)	0.033 (2)	0.046 (3)	0.0059 (19)	0.016 (2)	-0.002 (2)
O21	0.0257 (18)	0.0314 (18)	0.0402 (19)	0.0000 (15)	0.0115 (18)	0.0068 (16)
O22	0.041 (2)	0.042 (2)	0.0292 (19)	-0.0135 (18)	-0.0079 (18)	0.0041 (17)
O41	0.042 (2)	0.041 (2)	0.0321 (19)	-0.0055 (18)	0.0155 (18)	-0.0097 (18)
O42	0.033 (2)	0.0359 (18)	0.0297 (18)	-0.0071 (15)	-0.0014 (17)	-0.0050 (17)
O51	0.0259 (18)	0.0340 (18)	0.0327 (19)	0.0028 (15)	0.0033 (16)	-0.0009 (16)
O52	0.045 (2)	0.0264 (18)	0.050 (2)	-0.0054 (17)	0.020 (2)	-0.0156 (18)
O71	0.034 (2)	0.0239 (16)	0.0305 (18)	-0.0077 (15)	-0.0018 (17)	-0.0069 (15)
O72	0.0233 (17)	0.0338 (19)	0.0368 (19)	0.0021 (15)	0.0099 (18)	0.0034 (16)

*Geometric parameters (Å, °)*

S2—O22	1.424 (4)	C7—C8	1.394 (6)
S2—O21	1.433 (4)	C8—C13	1.387 (7)
S2—N2	1.612 (4)	C8—H8	0.9500
S2—C2	1.785 (5)	C9—C14	1.358 (6)
S7—O72	1.428 (4)	C9—C13	1.469 (6)
S7—O71	1.432 (3)	C9—C10	1.475 (6)
S7—N7	1.606 (4)	C10—C11	1.410 (6)
S7—C7	1.784 (5)	C11—C12	1.473 (6)
C1—C2	1.383 (7)	C12—C13	1.429 (6)
C1—C10	1.396 (6)	C14—C15	1.435 (6)
C1—H1	0.9500	C14—C16	1.446 (7)
C2—C3	1.381 (7)	C15—N15	1.144 (7)
C3—C4	1.393 (6)	C16—N16	1.146 (7)
C3—H3	0.9499	N2—H21	0.8901
C4—C11	1.387 (7)	N2—H22	0.8900
C4—N4	1.476 (6)	N4—O41	1.218 (5)
C5—C12	1.387 (6)	N4—O42	1.236 (5)
C5—C6	1.391 (6)	N5—O52	1.219 (5)
C5—N5	1.465 (6)	N5—O51	1.239 (5)
C6—C7	1.384 (6)	N7—H71	0.8901

C6—H6	0.9500	N7—H72	0.8900
O22—S2—O21	120.6 (3)	C13—C8—H8	120.8
O22—S2—N2	107.1 (2)	C7—C8—H8	120.9
O21—S2—N2	106.5 (2)	C14—C9—C13	125.9 (4)
O22—S2—C2	107.2 (2)	C14—C9—C10	127.1 (4)
O21—S2—C2	106.5 (2)	C13—C9—C10	106.8 (4)
N2—S2—C2	108.6 (2)	C1—C10—C11	121.5 (4)
O72—S7—O71	119.4 (2)	C1—C10—C9	130.4 (4)
O72—S7—N7	108.4 (2)	C11—C10—C9	108.0 (4)
O71—S7—N7	106.5 (2)	C4—C11—C10	117.4 (4)
O72—S7—C7	105.8 (2)	C4—C11—C12	134.2 (4)
O71—S7—C7	107.3 (2)	C10—C11—C12	108.3 (4)
N7—S7—C7	109.1 (2)	C5—C12—C13	116.8 (4)
C2—C1—C10	117.7 (4)	C5—C12—C11	135.2 (4)
C2—C1—H1	121.2	C13—C12—C11	107.8 (4)
C10—C1—H1	121.2	C8—C13—C12	121.7 (4)
C3—C2—C1	122.6 (4)	C8—C13—C9	130.2 (4)
C3—C2—S2	117.4 (4)	C12—C13—C9	107.9 (4)
C1—C2—S2	120.0 (4)	C9—C14—C15	123.4 (4)
C2—C3—C4	118.2 (5)	C9—C14—C16	122.2 (4)
C2—C3—H3	120.8	C15—C14—C16	114.4 (4)
C4—C3—H3	121.0	N15—C15—C14	177.0 (6)
C11—C4—C3	121.7 (4)	N16—C16—C14	178.2 (6)
C11—C4—N4	122.2 (4)	S2—N2—H21	110.1
C3—C4—N4	115.8 (4)	S2—N2—H22	114.0
C12—C5—C6	122.6 (4)	H21—N2—H22	111.9
C12—C5—N5	122.7 (4)	O41—N4—O42	125.1 (4)
C6—C5—N5	114.5 (4)	O41—N4—C4	118.5 (4)
C7—C6—C5	118.5 (4)	O42—N4—C4	116.3 (4)
C7—C6—H6	120.6	O52—N5—O51	125.0 (4)
C5—C6—H6	120.8	O52—N5—C5	117.0 (4)
C6—C7—C8	121.9 (4)	O51—N5—C5	117.9 (4)
C6—C7—S7	117.8 (3)	S7—N7—H71	103.2
C8—C7—S7	120.2 (4)	S7—N7—H72	107.3
C13—C8—C7	118.3 (4)	H71—N7—H72	124.7
C1—C2—S2—N2	79.0 (4)	C8—C7—S7—N7	96.3 (4)

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H21 $\cdots$ O72 <sup>i</sup>	0.89	2.03	2.878 (6)	159
N2—H22 $\cdots$ O52 <sup>ii</sup>	0.89	2.10	2.925 (5)	153
N7—H71 $\cdots$ O41 <sup>iii</sup>	0.89	2.51	3.116 (6)	126
N7—H72 $\cdots$ N2 <sup>iv</sup>	0.89	2.24	3.103 (6)	162

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