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5,5′-(1,3-Phenylenedimethylene)bis-(2-amino-4-*tert*-butylthiazol-3-ium) dibromide monohydrate

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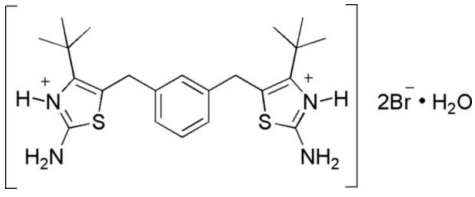
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.006 Å; R factor = 0.030; wR factor = 0.112; data-to-parameter ratio = 17.0.

Geometric parameters of the title salt, $C_{22}H_{32}N_4S_2^{2+}\cdot 2Br^-\cdot H_2O$, are in the usual ranges. The crystal packing is stabilized by N–H···O, N–H···Br and O–H···Br hydrogen bonds.

Related literature

For related literature, see: He *et al.* (2006); Saïd El et *et al.* (2002); Xu *et al.* (2007).



Experimental

Crystal data

$C_{22}H_{32}N_4S_2^{2+}\cdot 2Br^-\cdot H_2O$
M_r = 594.47
Triclinic, $P\overline{1}$
a = 8.9104 (5) Å
b = 9.9136 (5) Å
c = 15.8574 (8) Å
α = 87.742 (1)°
β = 78.019 (1)°
γ = 71.805 (1)°
V = 1301.24 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.30 mm^{-1}
T = 173 (2) K
0.48 × 0.38 × 0.16 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.248, T_{max} = 0.590
9833 measured reflections
4962 independent reflections
3929 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.112
S = 1.10
4962 reflections
292 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Br1	0.88	2.45	3.266 (3)	155
N2—H2A···Br1	0.88	2.78	3.523 (3)	143
N2—H2B···O1W	0.88	1.91	2.770 (4)	164
N3—H3A···Br2	0.88	2.46	3.268 (3)	153
N4—H4B···Br1^{i}	0.88	2.63	3.456 (3)	157
O1W—H1B···Br2^{ii}	0.860 (10)	2.563 (13)	3.413 (3)	170 (4)
O1W—H1C···Br1^{i}	0.86 (3)	2.47 (4)	3.315 (3)	167 (4)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2340).

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supplementary materials

Acta Cryst. (2007). E**63**, o2533 [**doi:10.1107/S1600536807018296**]

5,5'-(1,3-Phenylenedimethylene)bis(2-amino-4-*tert*-butylthiazol-3-ium) dibromide monohydrate

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Comment

The 2-aminothiazole group is a common functionality in medicinal chemistry. It is known to be a ligand of estrogen receptors, as well as a novel class of adenosine receptor antagonists. Some are also used as fungicide, inhibiting in vivo the growth of Xanthomonas, as an ingredient of herbicides or as schistosomicidal drugs (Saïd El *et al.*, 2002). Two 2-aminothiazoles crystal structures were reported (He *et al.*, 2006; Xu *et al.*, 2007). We report here the synthesis and structure of the title 2-aminothiazoles derivative 5,5'-(1,3-phenylenebis(methylene))bis(2-amino-4-tert-butylthiazol-3-ium) dibromide monohydrate (I).

The molecular structure of (I) is illustrated in Fig.1. The dihedral angles between the central aromatic ring and the two thiazole rings are 76.7 (2) and 80.4 (2)°. The crystal packing is stabilized by N–H···O, N–H···Br and O–H···Br hydrogen bonds.

Experimental

5,5'-(1,3-Phenylene)bis(2,2-dimethylpentan-3-one) (0.033 mol) was dissolved in 200 ml e thanol and the mixture was stirred and heated to reflux. Cupric bromide (0.132 mol) was added to the reaction mixture in batches and the course of the reaction was followed by TLC analysis. After the reaction had finished, the mixture was filtered and concentrated in vacuo. The resulting residue was taken up in dichloromethane, washed with 10% hydrochloric acid (30 mL three times), then washed with water until the solution was neutral, dried over anhydrous sodium sulfate and concentrated in vacuo to give 5,5'-(1,3-phenylene)bis(4-bromo-2,2- dimethylpentan-3-one), yield 90.8%. Then a solution of thiourea (0.055 mol) and cupric bromide (0.028 mol) in ethanol (65 ml) was refluxed for 9 h. The solvent was evaporated and the precipitate formed was filtered off, dried, giving colourless crystals of (I), yield 63.2%. M.p. 463.6-464.5 K.

The crystals suitable for X-ray structure determination were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

All H atoms with exception of the water H atoms were refined using a riding model with N—H distances of 0.88 A° and C— H distances ranging from 0.95to 0.99Å and with $U_{iso}(H) = 1.2U_{eq}(C, N)$ or $1.5Ueq(C_{methyl})$. The coordinates of the water H atoms were refined with an O—H distance restraint of 0.86 (1) Å, a H—H distance restraint of 1.38 (1) Å, and $U_{iso}(H) = 1.2U_{eq}(O)$.

Figures

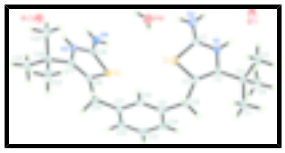


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids.

5,5'-(1,3-Phenylenedimethylene)bis(2-amino-4-tert-butylthiazol-3-ium) dibromide monohydrate

Crystal data

$C_{22}H_{32}N_4S_2^{2+}\cdot 2(Br^-)\cdot H_2O$	$Z = 2$
$M_r = 594.47$	$F_{000} = 608$
Triclinic, $P\overline{1}$	$D_x = 1.517$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.9104$ (5) Å	Cell parameters from 4865 reflections
$b = 9.9136$ (5) Å	$\theta = 2.5$–27.0º
$c = 15.8574$ (8) Å	$\mu = 3.30$ mm^{-1}
$\alpha = 87.742$ (1)º	$T = 173$ (2) K
$\beta = 78.019$ (1)º	Block, colourless
$\gamma = 71.805$ (1)º	0.48 × 0.38 × 0.16 mm
$V = 1301.24$ (12) Å^3	

Data collection

Bruker SMART 1000 CCD diffractometer	4962 independent reflections
Radiation source: fine-focus sealed tube	3929 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.024$
$T = 173$(2) K	$\theta_{max} = 26.0$º
ω scans	$\theta_{min} = 1.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10\rightarrow10$
$T_{min} = 0.248$, $T_{max} = 0.590$	$k = -12\rightarrow12$
9833 measured reflections	$l = -19\rightarrow19$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 1.5162P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.112$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.10$	$\Delta\rho_{max} = 0.75$ e Å^{-3}

4962 reflections
292 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

$\Delta\rho_{min} = -0.43$ e Å^{-3}
Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
Br1	0.25498 (5)	1.24569 (4)	1.18698 (2)	0.02619 (12)
Br2	0.55055 (6)	0.84294 (4)	0.39514 (3)	0.03703 (14)
S1	0.22272 (12)	0.85025 (10)	0.96410 (6)	0.0236 (2)
S2	0.44511 (11)	0.64652 (10)	0.72660 (6)	0.0206 (2)
C1	0.2366 (4)	0.9915 (4)	1.0193 (2)	0.0203 (8)
C2	0.1230 (4)	0.8691 (4)	1.1295 (2)	0.0186 (7)
C3	0.1364 (4)	0.7853 (4)	1.0620 (2)	0.0202 (8)
C4	0.0552 (4)	0.8669 (4)	1.2255 (2)	0.0221 (8)
C5	−0.0800 (5)	1.0070 (4)	1.2520 (3)	0.0324 (10)
H5A	−0.0359	1.0864	1.2405	0.049*
H5B	−0.1266	1.0067	1.3136	0.049*
H5C	−0.1640	1.0174	1.2187	0.049*
C6	0.1913 (5)	0.8533 (5)	1.2740 (3)	0.0364 (10)
H6A	0.2746	0.7612	1.2594	0.055*
H6B	0.1475	0.8603	1.3362	0.055*
H6C	0.2388	0.9297	1.2573	0.055*
C7	−0.0139 (6)	0.7448 (5)	1.2501 (3)	0.0336 (10)
H7A	−0.0946	0.7479	1.2158	0.050*
H7B	−0.0647	0.7538	1.3115	0.050*
H7C	0.0732	0.6543	1.2386	0.050*
C8	0.0896 (5)	0.6526 (4)	1.0542 (3)	0.0281 (9)
H8A	0.0995	0.5981	1.1075	0.034*
H8B	−0.0250	0.6808	1.0491	0.034*
C9	0.1919 (4)	0.5578 (4)	0.9776 (2)	0.0215 (8)
C10	0.1505 (4)	0.5771 (4)	0.8974 (2)	0.0200 (8)
H10	0.0555	0.6504	0.8908	0.024*
C11	0.2456 (4)	0.4908 (4)	0.8263 (2)	0.0182 (7)

C12	0.3822 (5)	0.3825 (4)	0.8373 (3)	0.0245 (8)
H12	0.4462	0.3210	0.7899	0.029*
C13	0.4262 (5)	0.3631 (4)	0.9168 (3)	0.0267 (9)
H13	0.5213	0.2898	0.9233	0.032*
C14	0.3320 (5)	0.4501 (4)	0.9872 (3)	0.0244 (8)
H14	0.3626	0.4365	1.0416	0.029*
C15	0.2030 (5)	0.5180 (4)	0.7381 (2)	0.0230 (8)
H15A	0.0853	0.5649	0.7452	0.028*
H15B	0.2305	0.4261	0.7069	0.028*
C16	0.2921 (4)	0.6108 (4)	0.6849 (2)	0.0193 (7)
C17	0.2797 (4)	0.6721 (4)	0.6087 (2)	0.0194 (7)
C18	0.4898 (4)	0.7414 (4)	0.6372 (2)	0.0211 (8)
C19	0.1713 (5)	0.6716 (4)	0.5460 (2)	0.0226 (8)
C20	0.0880 (6)	0.8264 (5)	0.5248 (3)	0.0393 (11)
H20A	0.0201	0.8782	0.5776	0.059*
H20B	0.0209	0.8277	0.4828	0.059*
H20C	0.1700	0.8717	0.5004	0.059*
C21	0.2753 (5)	0.5883 (5)	0.4639 (3)	0.0318 (9)
H21A	0.3551	0.6347	0.4371	0.048*
H21B	0.2064	0.5859	0.4236	0.048*
H21C	0.3311	0.4911	0.4785	0.048*
C22	0.0409 (5)	0.6038 (5)	0.5842 (3)	0.0368 (11)
H22A	0.0917	0.5035	0.5950	0.055*
H22B	−0.0304	0.6113	0.5437	0.055*
H22C	−0.0222	0.6532	0.6387	0.055*
N1	0.1818 (4)	0.9832 (3)	1.10288 (19)	0.0204 (6)
H1A	0.1822	1.0465	1.1402	0.024*
N2	0.2944 (4)	1.0916 (3)	0.9839 (2)	0.0284 (8)
H2A	0.2988	1.1597	1.0163	0.034*
H2B	0.3290	1.0906	0.9277	0.034*
N3	0.3936 (4)	0.7450 (3)	0.58320 (19)	0.0193 (6)
H3A	0.4003	0.7903	0.5344	0.023*
N4	0.6064 (4)	0.8003 (3)	0.6226 (2)	0.0269 (7)
H4A	0.6239	0.8455	0.5745	0.032*
H4B	0.6666	0.7943	0.6610	0.032*
O1W	0.4233 (4)	1.0337 (3)	0.80968 (19)	0.0356 (7)
H1B	0.432 (5)	1.054 (5)	0.7559 (10)	0.043*
H1C	0.513 (3)	0.971 (4)	0.814 (2)	0.043*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0306 (2)	0.0268 (2)	0.0245 (2)	−0.01054 (17)	−0.01016 (16)	−0.00086 (15)
Br2	0.0494 (3)	0.0318 (2)	0.0241 (2)	−0.0117 (2)	0.00306 (19)	0.00451 (17)
S1	0.0337 (5)	0.0247 (5)	0.0150 (4)	−0.0140 (4)	−0.0037 (4)	0.0005 (4)
S2	0.0254 (5)	0.0225 (4)	0.0181 (4)	−0.0113 (4)	−0.0083 (4)	0.0029 (3)
C1	0.0217 (19)	0.0184 (17)	0.0213 (19)	−0.0062 (15)	−0.0059 (15)	0.0018 (14)
C2	0.0203 (18)	0.0198 (17)	0.0158 (17)	−0.0053 (15)	−0.0052 (14)	−0.0011 (14)

C3	0.0233 (19)	0.0264 (19)	0.0134 (17)	−0.0107 (16)	−0.0053 (14)	0.0050 (14)
C4	0.024 (2)	0.028 (2)	0.0159 (18)	−0.0114 (16)	−0.0016 (15)	0.0003 (15)
C5	0.033 (2)	0.035 (2)	0.024 (2)	−0.0055 (19)	−0.0003 (17)	−0.0074 (17)
C6	0.033 (2)	0.059 (3)	0.020 (2)	−0.017 (2)	−0.0093 (18)	0.009 (2)
C7	0.049 (3)	0.037 (2)	0.018 (2)	−0.024 (2)	0.0030 (18)	−0.0007 (17)
C8	0.036 (2)	0.027 (2)	0.023 (2)	−0.0152 (18)	−0.0002 (17)	−0.0008 (16)
C9	0.025 (2)	0.0196 (18)	0.0246 (19)	−0.0163 (16)	−0.0022 (16)	0.0018 (15)
C10	0.0200 (18)	0.0158 (17)	0.0247 (19)	−0.0071 (15)	−0.0040 (15)	0.0030 (14)
C11	0.0250 (19)	0.0178 (17)	0.0157 (17)	−0.0124 (15)	−0.0047 (15)	0.0022 (13)
C12	0.025 (2)	0.0180 (18)	0.029 (2)	−0.0059 (16)	−0.0005 (16)	−0.0035 (15)
C13	0.027 (2)	0.0209 (19)	0.036 (2)	−0.0083 (16)	−0.0126 (18)	0.0084 (16)
C14	0.033 (2)	0.0241 (19)	0.025 (2)	−0.0180 (17)	−0.0120 (17)	0.0107 (16)
C15	0.028 (2)	0.0255 (19)	0.0192 (19)	−0.0127 (16)	−0.0061 (16)	0.0005 (15)
C16	0.0208 (18)	0.0202 (18)	0.0171 (17)	−0.0050 (15)	−0.0060 (14)	−0.0023 (14)
C17	0.0186 (18)	0.0173 (17)	0.0206 (18)	−0.0036 (14)	−0.0027 (15)	−0.0033 (14)
C18	0.0218 (19)	0.0186 (17)	0.0194 (18)	−0.0027 (15)	−0.0026 (15)	0.0011 (14)
C19	0.026 (2)	0.0261 (19)	0.0170 (18)	−0.0064 (16)	−0.0087 (15)	−0.0010 (15)
C20	0.044 (3)	0.029 (2)	0.038 (3)	0.006 (2)	−0.022 (2)	−0.0026 (19)
C21	0.033 (2)	0.035 (2)	0.026 (2)	−0.0091 (19)	−0.0047 (18)	−0.0092 (18)
C22	0.033 (2)	0.062 (3)	0.023 (2)	−0.021 (2)	−0.0126 (18)	0.001 (2)
N1	0.0249 (16)	0.0202 (15)	0.0178 (15)	−0.0091 (13)	−0.0041 (13)	−0.0023 (12)
N2	0.039 (2)	0.0273 (17)	0.0233 (17)	−0.0194 (16)	−0.0014 (15)	0.0008 (14)
N3	0.0250 (16)	0.0194 (15)	0.0151 (15)	−0.0075 (13)	−0.0072 (13)	0.0025 (12)
N4	0.0320 (19)	0.0346 (19)	0.0218 (17)	−0.0197 (16)	−0.0089 (14)	0.0063 (14)
O1W	0.0374 (18)	0.0344 (17)	0.0258 (15)	−0.0018 (14)	−0.0016 (13)	0.0029 (13)

Geometric parameters (Å, °)

S1—C1	1.730 (4)	C12—H12	0.9500
S1—C3	1.775 (4)	C13—C14	1.389 (6)
S2—C18	1.717 (4)	C13—H13	0.9500
S2—C16	1.765 (4)	C14—H14	0.9500
C1—N2	1.312 (5)	C15—C16	1.518 (5)
C1—N1	1.322 (5)	C15—H15A	0.9900
C2—C3	1.344 (5)	C15—H15B	0.9900
C2—N1	1.406 (5)	C16—C17	1.341 (5)
C2—C4	1.519 (5)	C17—N3	1.407 (5)
C3—C8	1.515 (5)	C17—C19	1.524 (5)
C4—C7	1.524 (5)	C18—N4	1.320 (5)
C4—C5	1.532 (5)	C18—N3	1.324 (5)
C4—C6	1.535 (6)	C19—C22	1.527 (6)
C5—H5A	0.9800	C19—C21	1.530 (5)
C5—H5B	0.9800	C19—C20	1.540 (5)
C5—H5C	0.9800	C20—H20A	0.9800
C6—H6A	0.9800	C20—H20B	0.9800
C6—H6B	0.9800	C20—H20C	0.9800
C6—H6C	0.9800	C21—H21A	0.9800
C7—H7A	0.9800	C21—H21B	0.9800
C7—H7B	0.9800	C21—H21C	0.9800

C7—H7C	0.9800	C22—H22A	0.9800
C8—C9	1.511 (5)	C22—H22B	0.9800
C8—H8A	0.9900	C22—H22C	0.9800
C8—H8B	0.9900	N1—H1A	0.8800
C9—C10	1.386 (5)	N2—H2A	0.8800
C9—C14	1.398 (5)	N2—H2B	0.8800
C10—C11	1.395 (5)	N3—H3A	0.8800
C10—H10	0.9500	N4—H4A	0.8800
C11—C12	1.387 (5)	N4—H4B	0.8800
C11—C15	1.517 (5)	O1W—H1B	0.860 (10)
C12—C13	1.386 (6)	O1W—H1C	0.86 (3)
C1—S1—C3	90.67 (17)	C13—C14—C9	119.7 (4)
C18—S2—C16	90.78 (18)	C13—C14—H14	120.2
N2—C1—N1	124.8 (3)	C9—C14—H14	120.2
N2—C1—S1	125.2 (3)	C11—C15—C16	112.0 (3)
N1—C1—S1	110.0 (3)	C11—C15—H15A	109.2
C3—C2—N1	111.1 (3)	C16—C15—H15A	109.2
C3—C2—C4	133.6 (3)	C11—C15—H15B	109.2
N1—C2—C4	115.3 (3)	C16—C15—H15B	109.2
C2—C3—C8	132.8 (3)	H15A—C15—H15B	107.9
C2—C3—S1	111.1 (3)	C17—C16—C15	132.2 (3)
C8—C3—S1	116.1 (3)	C17—C16—S2	110.9 (3)
C2—C4—C7	113.0 (3)	C15—C16—S2	116.9 (3)
C2—C4—C5	108.3 (3)	C16—C17—N3	111.6 (3)
C7—C4—C5	108.4 (3)	C16—C17—C19	131.8 (3)
C2—C4—C6	108.4 (3)	N3—C17—C19	116.6 (3)
C7—C4—C6	108.8 (3)	N4—C18—N3	124.1 (3)
C5—C4—C6	109.8 (3)	N4—C18—S2	125.1 (3)
C4—C5—H5A	109.5	N3—C18—S2	110.8 (3)
C4—C5—H5B	109.5	C17—C19—C22	112.0 (3)
H5A—C5—H5B	109.5	C17—C19—C21	108.8 (3)
C4—C5—H5C	109.5	C22—C19—C21	109.0 (3)
H5A—C5—H5C	109.5	C17—C19—C20	108.5 (3)
H5B—C5—H5C	109.5	C22—C19—C20	108.4 (4)
C4—C6—H6A	109.5	C21—C19—C20	110.2 (3)
C4—C6—H6B	109.5	C19—C20—H20A	109.5
H6A—C6—H6B	109.5	C19—C20—H20B	109.5
C4—C6—H6C	109.5	H20A—C20—H20B	109.5
H6A—C6—H6C	109.5	C19—C20—H20C	109.5
H6B—C6—H6C	109.5	H20A—C20—H20C	109.5
C4—C7—H7A	109.5	H20B—C20—H20C	109.5
C4—C7—H7B	109.5	C19—C21—H21A	109.5
H7A—C7—H7B	109.5	C19—C21—H21B	109.5
C4—C7—H7C	109.5	H21A—C21—H21B	109.5
H7A—C7—H7C	109.5	C19—C21—H21C	109.5
H7B—C7—H7C	109.5	H21A—C21—H21C	109.5
C9—C8—C3	113.2 (3)	H21B—C21—H21C	109.5
C9—C8—H8A	108.9	C19—C22—H22A	109.5
C3—C8—H8A	108.9	C19—C22—H22B	109.5

C9—C8—H8B	108.9	H22A—C22—H22B	109.5
C3—C8—H8B	108.9	C19—C22—H22C	109.5
H8A—C8—H8B	107.8	H22A—C22—H22C	109.5
C10—C9—C14	119.3 (3)	H22B—C22—H22C	109.5
C10—C9—C8	121.1 (3)	C1—N1—C2	117.1 (3)
C14—C9—C8	119.7 (4)	C1—N1—H1A	121.4
C9—C10—C11	121.3 (3)	C2—N1—H1A	121.4
C9—C10—H10	119.4	C1—N2—H2A	120.0
C11—C10—H10	119.4	C1—N2—H2B	120.0
C12—C11—C10	118.8 (3)	H2A—N2—H2B	120.0
C12—C11—C15	120.6 (3)	C18—N3—C17	115.9 (3)
C10—C11—C15	120.6 (3)	C18—N3—H3A	122.0
C13—C12—C11	120.5 (4)	C17—N3—H3A	122.0
C13—C12—H12	119.7	C18—N4—H4A	120.0
C11—C12—H12	119.7	C18—N4—H4B	120.0
C12—C13—C14	120.4 (4)	H4A—N4—H4B	120.0
C12—C13—H13	119.8	H1B—O1W—H1C	106 (4)
C14—C13—H13	119.8		
C3—S1—C1—N2	−179.9 (4)	C8—C9—C14—C13	−179.9 (3)
C3—S1—C1—N1	0.8 (3)	C12—C11—C15—C16	−84.1 (4)
N1—C2—C3—C8	−178.6 (4)	C10—C11—C15—C16	94.0 (4)
C4—C2—C3—C8	−0.3 (8)	C11—C15—C16—C17	−172.4 (4)
N1—C2—C3—S1	−0.2 (4)	C11—C15—C16—S2	9.0 (4)
C4—C2—C3—S1	178.0 (3)	C18—S2—C16—C17	−1.1 (3)
C1—S1—C3—C2	−0.3 (3)	C18—S2—C16—C15	177.8 (3)
C1—S1—C3—C8	178.4 (3)	C15—C16—C17—N3	−177.7 (4)
C3—C2—C4—C7	−0.9 (6)	S2—C16—C17—N3	0.9 (4)
N1—C2—C4—C7	177.3 (3)	C15—C16—C17—C19	−0.1 (7)
C3—C2—C4—C5	−121.0 (5)	S2—C16—C17—C19	178.6 (3)
N1—C2—C4—C5	57.2 (4)	C16—S2—C18—N4	−177.6 (3)
C3—C2—C4—C6	119.9 (5)	C16—S2—C18—N3	1.0 (3)
N1—C2—C4—C6	−61.9 (4)	C16—C17—C19—C22	8.7 (6)
C2—C3—C8—C9	−153.1 (4)	N3—C17—C19—C22	−173.8 (3)
S1—C3—C8—C9	28.6 (5)	C16—C17—C19—C21	−111.9 (4)
C3—C8—C9—C10	−88.1 (4)	N3—C17—C19—C21	65.6 (4)
C3—C8—C9—C14	91.0 (4)	C16—C17—C19—C20	128.3 (4)
C14—C9—C10—C11	0.2 (5)	N3—C17—C19—C20	−54.2 (4)
C8—C9—C10—C11	179.3 (3)	N2—C1—N1—C2	179.5 (4)
C9—C10—C11—C12	1.2 (5)	S1—C1—N1—C2	−1.1 (4)
C9—C10—C11—C15	−176.9 (3)	C3—C2—N1—C1	0.9 (5)
C10—C11—C12—C13	−2.0 (5)	C4—C2—N1—C1	−177.7 (3)
C15—C11—C12—C13	176.2 (3)	N4—C18—N3—C17	178.0 (3)
C11—C12—C13—C14	1.4 (6)	S2—C18—N3—C17	−0.6 (4)
C12—C13—C14—C9	0.0 (6)	C16—C17—N3—C18	−0.2 (4)
C10—C9—C14—C13	−0.8 (5)	C19—C17—N3—C18	−178.2 (3)

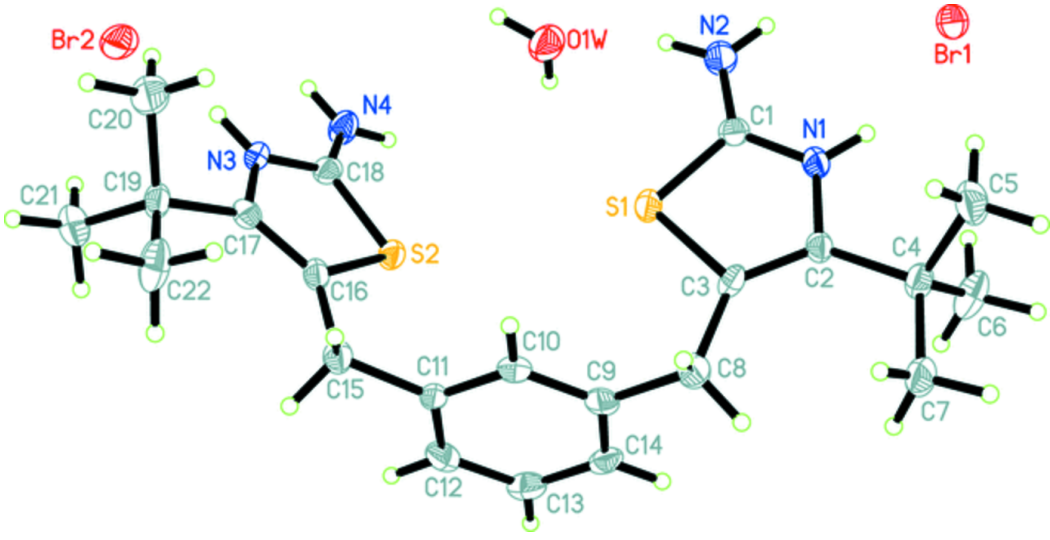
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N1—H1A···Br1	0.88	2.45	3.266 (3)	155
N2—H2A···Br1	0.88	2.78	3.523 (3)	143
N2—H2B···O1W	0.88	1.91	2.770 (4)	164
N3—H3A···Br2	0.88	2.46	3.268 (3)	153
N4—H4B···Br1^{i}	0.88	2.63	3.456 (3)	157
O1W—H1B···Br2^{ii}	0.860 (10)	2.563 (13)	3.413 (3)	170 (4)
O1W—H1C···Br1^{i}	0.86 (3)	2.47 (4)	3.315 (3)	167 (4)

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

Fig. 1


Br2
C20
N4
N3
C18
C21
C19
C17
S2
C22
C16
C15
C11
C10
C9
C8
O1W
N2
C1
N1
S1
C3
C2
C4
C5
C6
C7
Br1
C14
C12
C13