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N,*N*′-Bis(1,3-dimethylimidazolidin-2-ylidene)-2,2′-dithiodianiline

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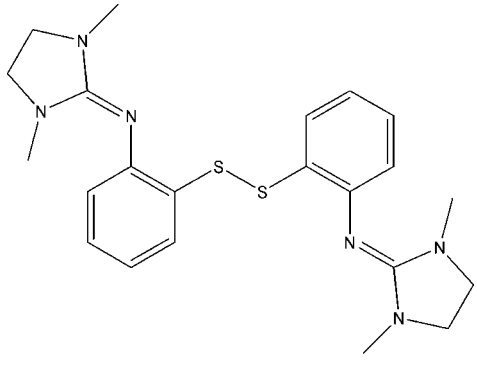
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.088; data-to-parameter ratio = 15.2.

The molecular structure of the title compound, $C_{22}H_{28}N_6S_2$, is the first crystallographically determined guanidine ligand containing sulfur. It shows two guanidyl groups bridged by a diphenyldisulfanyl linker with localized C=N bonds. The C—S—S—C group has a folded nonplanar conformation like that found in H_2O_2 or H_2S_2, with a torsion angle of 84.65 (10)°. The S—S bond length is 2.0435 (7) Å. Molecules are linked by C—H···N hydrogen bonds. The crystal studied was an inversion twin with approximately 2:1 components.

Related literature

For related literature, see: Allen *et al.* (1987); Gomes de Mesquita (1967); Harmjanz (1997); Herres *et al.* (2005); Kaitner & Pavlovic (1997); Lee & Bryant (1970); Neuba *et al.* (2007); Pohl *et al.* (2000); Schneider (2000); Waden (1999); Wittmann *et al.* (2001).



Experimental

Crystal data

$C_{22}H_{28}N_6S_2$
M_r = 440.62
Monoclinic, *Pc*
a = 8.2794 (12) Å
b = 10.1065 (14) Å
c = 13.7018 (19) Å
β = 104.333 (3)°
V = 1110.8 (3) Å^3
Z = 2
Mo *K*α radiation
μ = 0.26 mm^{-1}
T = 120 (2) K
0.42 × 0.39 × 0.36 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.898, T_{max} = 0.912
8801 measured reflections
4209 independent reflections
4074 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.088
S = 1.06
4209 reflections
276 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 1554 Friedel pairs
Flack parameter: 0.34 (6)

Table 1
Selected geometric parameters (Å, °).

S1—C11	1.786 (2)	N3—C1	1.362 (3)
S1—S2	2.0435 (7)	N4—C18	1.297 (3)
S2—C12	1.781 (2)	N4—C17	1.401 (3)
N1—C1	1.299 (3)	N5—C18	1.375 (3)
N1—C6	1.401 (3)	N6—C18	1.373 (3)
N2—C1	1.383 (3)		
C11—S1—S2	104.29 (6)	C1—N1—C6	123.32 (19)
C12—S2—S1	105.15 (7)	C18—N4—C17	121.27 (18)
C11—S1—S2—C12	84.65 (10)	S1—S2—C12—C13	9.0 (2)
S2—S1—C11—C10	0.55 (19)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···N2^{i}	0.95	2.54	3.463 (3)	165

Symmetry code: (i) $x, -y+1, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2431).

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supplementary materials

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N,*N*'-Bis(1,3-dimethylimidazolidin-2-ylidene)-2,2'-dithiodianiline

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Comment

The synthesis and characterization of molecules containing nitrogen and sulfur as donor functions is important for biomimetic coordination chemistry. The use of this molecules as ligands in the synthesis of copper-complexes as mimics for active centres like the CuA in cytochrome-c-oxidase and N_2O-reductase is currently of considerable interest in bioinorganic chemistry. In search of multifunctional ligands we have extended our studies to guanidyl-type systems with N-donor functions. The first derivative, the ligand bis(tetramethyl-guanidino)propylene and its complexes with Cu, Fe, Ni and Mn have recently been investigated (Harmjanz, 1997; Waden, 1999; Pohl *et al.*, 2000; Schneider, 2000; Wittmann *et al.*, 2001; Herres *et al.*, 2005, Neuba *et al.*, 2007). We have now developed the title compound with disulfide group as novel ligand with redoxactivity for use in biomimetic copper-sulfur chemistry.

The most interesting feature are the torsion angles τ C–S1–S2–C' and C–C'–S1–S2 which indicate pπ-dπ interactions between the fully occupied pz orbital on the carbon C atom (part of the aromatic π system) with an empty d orbital on the S atom as reported for 2,2'-diaminodiphenyl disulfide (Lee & Bryant, 1970). The S–S bond length in disulfide compounds is correlated with the C–S–S–C torsion angles, being around 2.031 Å (τ = 75–105°) or 2.070 Å (t = 0–20°) (Allen *et al.*, 1987). For (I) τ is 84.65 (10)° and the S1–S2 bond length 2.0435 (7) Å matching these ranges. As may be seen from the geometric bonding parameters (Table 1), the guanidyl double bonds C1═ N1 and C18═N14 are clearly localized.

The packing pattern shows intermolecular C–H···N hydrogen interactions (Table 2) with molecules stacked in [010] direction.

Experimental

A solution of dimethylethylenechloroformamidinium chloride (5.07 g, 30 mmol) in dry MeCN was added dropwise to an ice-cooled solution of 2,2'-dithiodianiline (3.73 g, 15 mmol) and triethylamine (4.18 ml, 3.03 g, 30 mmol) in dry MeCN. After 3 h under reflux, a solution of NaOH (1.2 g, 30 mmol) in water was added. The solvents and NEt_3 were then evaporated under vacuum. In order to deprotonate the bis-hydrochloride, 50 wt% KOH (aqueous, 15 ml) was added and the free base was extracted into the THF phase (3 *x* 80 ml). The organic phase was dried with Na_2SO_4. After filtration, the solvent was evaporated under reduced pressure. Colourless crystals suitable for X-ray diffraction were obtained by diffusion of Et_2O into a cold saturated MeCN solution.

^{1}H NMR (500 MHz, $CDCl_3$): δ 2.65 (s, 12H, Me), 3.3 (s, 8H, CH_2), 6.79 (m, 4H, CH_{arom}), 6.95 (m, 2H, CH_{arom}), 7.45 (m, 2H, CH_{arom}); ^{13}C NMR (125 MHz, $CDCl_3$): δ 43.8 (Me), 48.4 (CH_2), 121.1 (CH_{arom}), 122.0 (CH_{arom}), 125.2 (CH_{arom}), 125.8 (CH_{arom}), 128.9 (C_{quart}), 147.2 (C_{quart}), 155.5 (C_{gua}); IR (KBr, \v, cm^{-1}): 3035 (w), 2946 (*m*), 2858 (*m*), 1636 (*versus*, C═ N), 1613 (*versus*, C═N), 1568 (*versus*), 1441 (*s*), 1283 (*s*), 1034 (*s*), 966 (*m*), 740 (*m*). EI—MS: m/*z* (%)

440 (40) [M^+], 344 (70), 248 (85), 220 (90), 165 (80), 124 (90), 80 (75), 44 (80), 28 (30); Elemental analysis (M = 440.63 g mol^{-1}): calcd. for $C_{22}H_{28}N_6S_2$: C: 59.97; H: 6.41; N: 19.07; found C: 60.05, H: 6.62, N: 19.00.

Refinement

Hydrogen atoms located from difference Fourier maps were refined at idealized positions riding on the carbon atoms with isotropic displacement parameters U_{iso}(H) = 1.2U(Ceq) or 1.5U(CH_3). All CH_3 hydrogen atoms were allowed to rotate but not to tip. The crystal was refined as an inversion twin with the ratio of the two domains of 0.34 (6)/0.66 (6).

Figures

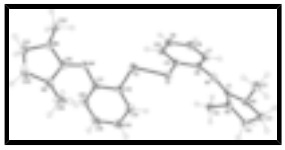

Fig. 1. Molecular structure of I. Displacement ellipsoids are drawn at the 50% probability level.

Fig. 2. Crystal packing of I viewed along [010] with hydrogen bond indicated as dashed lines. H-atoms not involved are omitted.

N,*N*'-Bis(1,3-dimethylimidazolidin-2-ylidene)-2,2'-dithiodianiline

Crystal data

$C_{22}H_{28}N_6S_2$	F_{000} = 468
M_r = 440.62	D_x = 1.317 Mg m^{-3}
Monoclinic, *Pc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P -2yc	Cell parameters from 913 reflections
a = 8.2794 (12) Å	θ = 2.5–28.2º
b = 10.1065 (14) Å	μ = 0.26 mm^{-1}
c = 13.7018 (19) Å	*T* = 120 (2) K
β = 104.333 (3)º	Prism, colourless
V = 1110.8 (3) Å^3	0.42 × 0.39 × 0.36 mm
Z = 2	

Data collection

Bruker SMART APEX diffractometer	4209 independent reflections
Radiation source: sealed tube	4074 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.033
T = 120(2) K	θ_{max} = 27.9º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan	*h* = −10→9

(SADABS; Bruker, 2002)
T_{min} = 0.898, T_{max} = 0.912
8801 measured reflections
k = −13→13
l = −15→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.088
S = 1.06
4209 reflections
276 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.0758P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: none
Absolute structure: Flack H D (1983), Acta Cryst. A39, 876-881
Flack parameter: 0.34 (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 $F^2 > 2$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	0.55549 (6)	0.30126 (4)	0.67932 (4)	0.02459 (12)
S2	0.65522 (6)	0.12261 (4)	0.73258 (4)	0.02580 (12)
N1	0.4194 (2)	0.50987 (16)	0.54669 (13)	0.0256 (4)
N2	0.3726 (2)	0.72951 (16)	0.57965 (14)	0.0254 (4)
N3	0.5600 (2)	0.69499 (14)	0.49018 (14)	0.0263 (4)
N4	0.8884 (2)	−0.09539 (16)	0.77503 (14)	0.0272 (4)
N5	1.0402 (2)	−0.1713 (2)	0.94000 (15)	0.0329 (4)
N6	0.9051 (2)	−0.31590 (17)	0.82600 (16)	0.0338 (5)
C1	0.4521 (2)	0.63399 (19)	0.53619 (15)	0.0232 (4)
C2	0.2088 (3)	0.7044 (2)	0.5944 (2)	0.0382 (6)
H2A	0.2052	0.6150	0.6215	0.057*
H2B	0.1258	0.7120	0.5298	0.057*
H2C	0.1842	0.7692	0.6420	0.057*

C3	0.4088 (3)	0.86078 (19)	0.54545 (18)	0.0309 (5)
H3A	0.4261	0.9263	0.6008	0.037*
H3B	0.3179	0.8918	0.4886	0.037*
C4	0.5678 (3)	0.8371 (2)	0.51246 (18)	0.0310 (5)
H4A	0.5699	0.8899	0.4519	0.037*
H4B	0.6673	0.8591	0.5669	0.037*
C5	0.7065 (3)	0.6331 (2)	0.46938 (19)	0.0320 (5)
H5A	0.6830	0.6092	0.3979	0.048*
H5B	0.7348	0.5533	0.5106	0.048*
H5C	0.8003	0.6952	0.4855	0.048*
C6	0.4730 (2)	0.40803 (19)	0.49286 (16)	0.0243 (4)
C7	0.4507 (3)	0.4075 (2)	0.38863 (18)	0.0312 (5)
H7A	0.4041	0.4829	0.3506	0.037*
C8	0.4953 (3)	0.2983 (2)	0.33941 (18)	0.0343 (5)
H8A	0.4807	0.3001	0.2684	0.041*
C9	0.5613 (3)	0.1865 (2)	0.39408 (19)	0.0330 (5)
H9A	0.5916	0.1117	0.3606	0.040*
C10	0.5827 (3)	0.18485 (18)	0.49752 (18)	0.0282 (4)
H10A	0.6288	0.1088	0.5350	0.034*
C11	0.5374 (2)	0.29307 (17)	0.54681 (16)	0.0224 (4)
C12	0.8746 (3)	0.14206 (19)	0.75131 (15)	0.0246 (4)
C13	0.9518 (3)	0.2624 (2)	0.74463 (18)	0.0313 (5)
H13A	0.8864	0.3404	0.7290	0.038*
C14	1.1245 (3)	0.2698 (2)	0.76067 (19)	0.0378 (5)
H14A	1.1769	0.3522	0.7556	0.045*
C15	1.2188 (3)	0.1562 (3)	0.78401 (19)	0.0370 (5)
H15A	1.3365	0.1608	0.7949	0.044*
C16	1.1434 (3)	0.0360 (2)	0.79161 (17)	0.0317 (5)
H16A	1.2101	−0.0412	0.8077	0.038*
C17	0.9701 (3)	0.02614 (19)	0.77599 (15)	0.0249 (4)
C18	0.9436 (3)	−0.1845 (2)	0.84325 (17)	0.0268 (4)
C19	1.0216 (4)	−0.0560 (3)	1.0000 (2)	0.0419 (6)
H19A	0.9221	−0.0665	1.0259	0.063*
H19B	1.0099	0.0236	0.9580	0.063*
H19C	1.1201	−0.0473	1.0566	0.063*
C20	1.0484 (3)	−0.2999 (2)	0.9920 (2)	0.0411 (6)
H20A	0.9661	−0.3045	1.0335	0.049*
H20B	1.1613	−0.3165	1.0354	0.049*
C21	1.0063 (3)	−0.3963 (2)	0.9055 (2)	0.0383 (6)
H21A	1.1080	−0.4284	0.8873	0.046*
H21B	0.9428	−0.4729	0.9214	0.046*
C22	0.8467 (4)	−0.3676 (2)	0.7259 (2)	0.0429 (6)
H22A	0.9403	−0.4068	0.7041	0.064*
H22B	0.7980	−0.2959	0.6798	0.064*
H22C	0.7620	−0.4355	0.7253	0.064*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0266 (3)	0.0221 (2)	0.0259 (2)	0.00218 (18)	0.00804 (19)	0.00260 (18)
S2	0.0213 (2)	0.0223 (2)	0.0330 (3)	−0.00125 (17)	0.00521 (19)	0.00647 (19)
N1	0.0261 (9)	0.0233 (7)	0.0274 (9)	0.0044 (6)	0.0064 (7)	0.0029 (6)
N2	0.0258 (9)	0.0230 (7)	0.0263 (9)	0.0023 (6)	0.0047 (7)	−0.0028 (6)
N3	0.0282 (10)	0.0219 (7)	0.0296 (10)	0.0016 (6)	0.0088 (7)	0.0014 (6)
N4	0.0230 (9)	0.0245 (7)	0.0306 (9)	0.0001 (6)	−0.0001 (7)	0.0027 (7)
N5	0.0265 (10)	0.0428 (10)	0.0273 (10)	−0.0012 (8)	0.0025 (8)	0.0063 (8)
N6	0.0294 (10)	0.0259 (8)	0.0419 (12)	0.0045 (7)	0.0008 (9)	0.0063 (7)
C1	0.0196 (10)	0.0277 (9)	0.0197 (10)	0.0042 (7)	0.0000 (8)	0.0017 (7)
C2	0.0278 (12)	0.0357 (11)	0.0533 (17)	0.0040 (9)	0.0143 (11)	−0.0082 (10)
C3	0.0346 (12)	0.0245 (9)	0.0316 (12)	0.0037 (8)	0.0045 (9)	0.0000 (8)
C4	0.0332 (12)	0.0229 (9)	0.0346 (12)	−0.0009 (8)	0.0043 (9)	−0.0012 (8)
C5	0.0273 (11)	0.0335 (11)	0.0375 (12)	0.0011 (8)	0.0121 (9)	0.0009 (8)
C6	0.0199 (10)	0.0250 (8)	0.0275 (11)	0.0011 (7)	0.0049 (8)	0.0009 (7)
C7	0.0262 (11)	0.0344 (10)	0.0296 (11)	0.0053 (8)	0.0006 (9)	0.0037 (9)
C8	0.0327 (13)	0.0448 (12)	0.0244 (11)	−0.0012 (9)	0.0054 (9)	−0.0046 (9)
C9	0.0332 (13)	0.0322 (10)	0.0352 (13)	−0.0012 (8)	0.0117 (10)	−0.0105 (9)
C10	0.0259 (11)	0.0225 (8)	0.0365 (12)	0.0008 (7)	0.0085 (9)	−0.0012 (8)
C11	0.0172 (9)	0.0244 (8)	0.0255 (10)	−0.0021 (7)	0.0048 (7)	−0.0001 (7)
C12	0.0214 (10)	0.0276 (9)	0.0235 (10)	−0.0028 (7)	0.0029 (8)	0.0006 (7)
C13	0.0316 (12)	0.0257 (9)	0.0352 (12)	−0.0040 (8)	0.0055 (9)	0.0040 (8)
C14	0.0332 (13)	0.0407 (11)	0.0368 (13)	−0.0137 (9)	0.0038 (10)	0.0074 (9)
C15	0.0224 (11)	0.0543 (13)	0.0326 (12)	−0.0061 (10)	0.0036 (9)	0.0097 (10)
C16	0.0234 (11)	0.0409 (11)	0.0292 (11)	0.0032 (8)	0.0039 (8)	0.0061 (9)
C17	0.0250 (10)	0.0285 (9)	0.0198 (9)	−0.0021 (7)	0.0032 (7)	0.0014 (7)
C18	0.0182 (10)	0.0292 (9)	0.0311 (11)	0.0012 (7)	0.0025 (8)	0.0017 (7)
C19	0.0423 (14)	0.0529 (13)	0.0310 (13)	−0.0202 (11)	0.0100 (10)	−0.0038 (10)
C20	0.0300 (13)	0.0553 (14)	0.0368 (14)	0.0031 (10)	0.0060 (10)	0.0198 (11)
C21	0.0261 (12)	0.0350 (11)	0.0524 (16)	0.0036 (8)	0.0071 (10)	0.0190 (10)
C22	0.0491 (16)	0.0256 (10)	0.0465 (15)	−0.0001 (9)	−0.0020 (12)	−0.0008 (9)

Geometric parameters (Å, °)

S1—C11	1.786 (2)	C6—C11	1.409 (3)
S1—S2	2.0435 (7)	C7—C8	1.390 (3)
S2—C12	1.781 (2)	C7—H7A	0.9500
N1—C1	1.299 (3)	C8—C9	1.391 (3)
N1—C6	1.401 (3)	C8—H8A	0.9500
N2—C1	1.383 (3)	C9—C10	1.384 (3)
N2—C2	1.442 (3)	C9—H9A	0.9500
N2—C3	1.463 (3)	C10—C11	1.385 (3)
N3—C1	1.362 (3)	C10—H10A	0.9500
N3—C5	1.454 (3)	C12—C13	1.388 (3)
N3—C4	1.466 (2)	C12—C17	1.407 (3)
N4—C18	1.297 (3)	C13—C14	1.394 (3)

N4—C17	1.401 (3)	C13—H13A	0.9500
N5—C18	1.375 (3)	C14—C15	1.380 (4)
N5—C19	1.457 (3)	C14—H14A	0.9500
N5—C20	1.475 (3)	C15—C16	1.382 (3)
N6—C18	1.373 (3)	C15—H15A	0.9500
N6—C22	1.435 (3)	C16—C17	1.401 (3)
N6—C21	1.448 (3)	C16—H16A	0.9500
C2—H2A	0.9800	C19—H19A	0.9800
C2—H2B	0.9800	C19—H19B	0.9800
C2—H2C	0.9800	C19—H19C	0.9800
C3—C4	1.513 (3)	C20—C21	1.507 (4)
C3—H3A	0.9900	C20—H20A	0.9900
C3—H3B	0.9900	C20—H20B	0.9900
C4—H4A	0.9900	C21—H21A	0.9900
C4—H4B	0.9900	C21—H21B	0.9900
C5—H5A	0.9800	C22—H22A	0.9800
C5—H5B	0.9800	C22—H22B	0.9800
C5—H5C	0.9800	C22—H22C	0.9800
C6—C7	1.394 (3)		
C11—S1—S2	104.29 (6)	C10—C9—H9A	120.2
C12—S2—S1	105.15 (7)	C8—C9—H9A	120.2
C1—N1—C6	123.32 (19)	C9—C10—C11	120.54 (19)
C1—N2—C2	120.12 (17)	C9—C10—H10A	119.7
C1—N2—C3	109.76 (19)	C11—C10—H10A	119.7
C2—N2—C3	118.61 (18)	C10—C11—C6	120.6 (2)
C1—N3—C5	124.81 (16)	C10—C11—S1	124.58 (16)
C1—N3—C4	110.31 (18)	C6—C11—S1	114.86 (15)
C5—N3—C4	117.79 (18)	C13—C12—C17	120.3 (2)
C18—N4—C17	121.27 (18)	C13—C12—S2	123.86 (17)
C18—N5—C19	120.3 (2)	C17—C12—S2	115.80 (15)
C18—N5—C20	109.09 (19)	C12—C13—C14	120.5 (2)
C19—N5—C20	115.5 (2)	C12—C13—H13A	119.8
C18—N6—C22	121.73 (19)	C14—C13—H13A	119.8
C18—N6—C21	110.3 (2)	C15—C14—C13	119.5 (2)
C22—N6—C21	120.65 (19)	C15—C14—H14A	120.3
N1—C1—N3	131.85 (19)	C13—C14—H14A	120.3
N1—C1—N2	119.5 (2)	C14—C15—C16	120.6 (2)
N3—C1—N2	108.66 (17)	C14—C15—H15A	119.7
N2—C2—H2A	109.5	C16—C15—H15A	119.7
N2—C2—H2B	109.5	C15—C16—C17	121.0 (2)
H2A—C2—H2B	109.5	C15—C16—H16A	119.5
N2—C2—H2C	109.5	C17—C16—H16A	119.5
H2A—C2—H2C	109.5	N4—C17—C16	122.52 (19)
H2B—C2—H2C	109.5	N4—C17—C12	119.12 (19)
N2—C3—C4	102.41 (16)	C16—C17—C12	118.11 (18)
N2—C3—H3A	111.3	N4—C18—N6	121.5 (2)
C4—C3—H3A	111.3	N4—C18—N5	130.2 (2)
N2—C3—H3B	111.3	N6—C18—N5	108.33 (18)
C4—C3—H3B	111.3	N5—C19—H19A	109.5

H3A—C3—H3B	109.2	N5—C19—H19B	109.5
N3—C4—C3	102.73 (17)	H19A—C19—H19B	109.5
N3—C4—H4A	111.2	N5—C19—H19C	109.5
C3—C4—H4A	111.2	H19A—C19—H19C	109.5
N3—C4—H4B	111.2	H19B—C19—H19C	109.5
C3—C4—H4B	111.2	N5—C20—C21	102.4 (2)
H4A—C4—H4B	109.1	N5—C20—H20A	111.3
N3—C5—H5A	109.5	C21—C20—H20A	111.3
N3—C5—H5B	109.5	N5—C20—H20B	111.3
H5A—C5—H5B	109.5	C21—C20—H20B	111.3
N3—C5—H5C	109.5	H20A—C20—H20B	109.2
H5A—C5—H5C	109.5	N6—C21—C20	101.97 (19)
H5B—C5—H5C	109.5	N6—C21—H21A	111.4
C7—C6—N1	124.29 (18)	C20—C21—H21A	111.4
C7—C6—C11	118.10 (18)	N6—C21—H21B	111.4
N1—C6—C11	117.25 (19)	C20—C21—H21B	111.4
C8—C7—C6	121.1 (2)	H21A—C21—H21B	109.2
C8—C7—H7A	119.4	N6—C22—H22A	109.5
C6—C7—H7A	119.4	N6—C22—H22B	109.5
C7—C8—C9	120.0 (2)	H22A—C22—H22B	109.5
C7—C8—H8A	120.0	N6—C22—H22C	109.5
C9—C8—H8A	120.0	H22A—C22—H22C	109.5
C10—C9—C8	119.7 (2)	H22B—C22—H22C	109.5
C11—S1—S2—C12	84.65 (10)	S1—S2—C12—C13	9.0 (2)
C6—N1—C1—N3	−12.6 (3)	S1—S2—C12—C17	−172.24 (15)
C6—N1—C1—N2	169.64 (18)	C17—C12—C13—C14	1.0 (4)
C5—N3—C1—N1	−21.9 (4)	S2—C12—C13—C14	179.77 (19)
C4—N3—C1—N1	−171.5 (2)	C12—C13—C14—C15	−0.4 (4)
C5—N3—C1—N2	156.0 (2)	C13—C14—C15—C16	0.0 (4)
C4—N3—C1—N2	6.4 (2)	C14—C15—C16—C17	−0.1 (4)
C2—N2—C1—N1	−28.6 (3)	C18—N4—C17—C16	45.6 (3)
C3—N2—C1—N1	−171.40 (18)	C18—N4—C17—C12	−140.4 (2)
C2—N2—C1—N3	153.2 (2)	C15—C16—C17—N4	174.7 (2)
C3—N2—C1—N3	10.4 (2)	C15—C16—C17—C12	0.6 (3)
C1—N2—C3—C4	−21.8 (2)	C13—C12—C17—N4	−175.4 (2)
C2—N2—C3—C4	−165.25 (19)	S2—C12—C17—N4	5.8 (3)
C1—N3—C4—C3	−19.5 (2)	C13—C12—C17—C16	−1.1 (3)
C5—N3—C4—C3	−171.50 (19)	S2—C12—C17—C16	−179.95 (17)
N2—C3—C4—N3	23.9 (2)	C17—N4—C18—N6	−156.8 (2)
C1—N1—C6—C7	−50.4 (3)	C17—N4—C18—N5	24.8 (4)
C1—N1—C6—C11	136.7 (2)	C22—N6—C18—N4	19.0 (4)
N1—C6—C7—C8	−174.8 (2)	C21—N6—C18—N4	169.2 (2)
C11—C6—C7—C8	−2.0 (3)	C22—N6—C18—N5	−162.3 (2)
C6—C7—C8—C9	0.9 (4)	C21—N6—C18—N5	−12.1 (3)
C7—C8—C9—C10	−0.2 (4)	C19—N5—C18—N4	34.9 (4)
C8—C9—C10—C11	0.6 (3)	C20—N5—C18—N4	171.7 (2)
C9—C10—C11—C6	−1.7 (3)	C19—N5—C18—N6	−143.7 (2)
C9—C10—C11—S1	178.80 (17)	C20—N5—C18—N6	−6.8 (3)
C7—C6—C11—C10	2.3 (3)	C18—N5—C20—C21	21.7 (3)

N1—C6—C11—C10	175.74 (19)	C19—N5—C20—C21	160.8 (2)
C7—C6—C11—S1	−178.12 (16)	C18—N6—C21—C20	25.1 (3)
N1—C6—C11—S1	−4.7 (2)	C22—N6—C21—C20	175.6 (2)
S2—S1—C11—C10	0.55 (19)	N5—C20—C21—N6	−27.1 (2)
S2—S1—C11—C6	−178.97 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8A···N2^{i}	0.95	2.54	3.463 (3)	165

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

Fig. 1

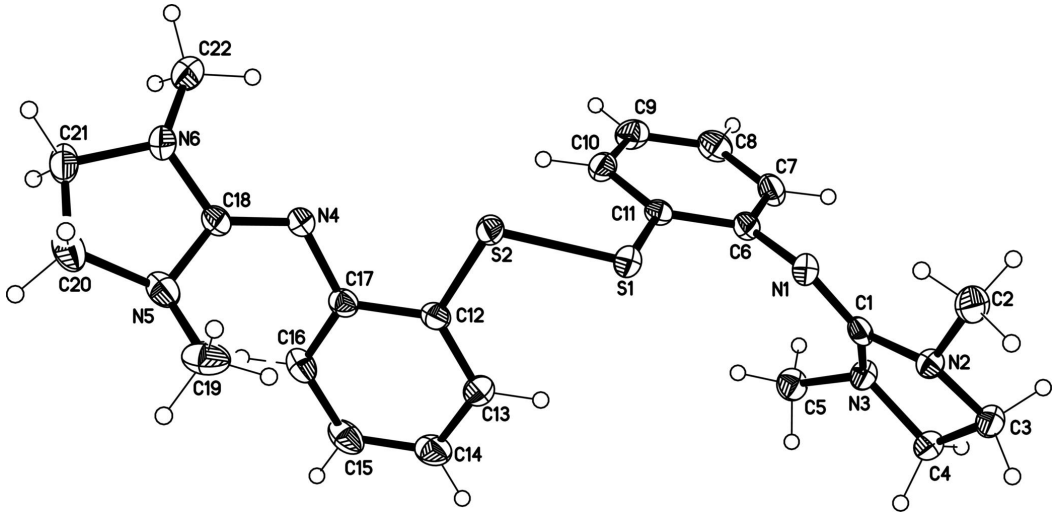
C22
C21
N6
C18
N4
C20
N5
C19
C16
C17
C12
S2
S1
C9
C10
C8
C7
C11
C6
N1
C1
C2
N2
C5
N3
C3
C4
C13
C14
C15

Fig. 2