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Crystal structure of bis(*N*-methyl-*N*-phenylamino)trisulfane

George Barany,[a]* Matthew J. Henley,[a] Lauren A. Polski,[a] Alayne L. Schroll[b] and Victor G. Young Jr[a]

[a]Department of Chemistry, University of Minnesota, Minneapolis, MN 55455, USA, and [b]Department of Chemistry, Saint Michael's College, Colchester, VT 05439, USA. *Correspondence e-mail: barany@umn.edu

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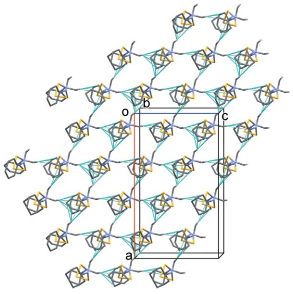
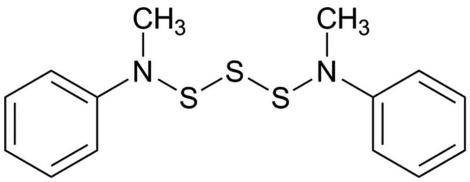
The title compound, $C_{14}H_{16}N_2S_3$, crystallized with two independent molecules [(**1*a***) and (**1*b***)] in the asymmetric unit. Both molecules display a pseudo-*trans* conformation. The two consecutive S—S bond lengths of the trisulfane unit of molecule (**1*a***) are 2.06 (3) and 2.08 (3) Å, and 2.08 (3) and 2.07 (2) Å for molecule (**1*b***). Torsion angles about each of the two S—S bonds are 86.6 (2) and 87.0 (2)° for (**1*a***), and −84.6 (2) and −85.9 (2)° for (**1*b***). The core atoms, *viz*. the N—S—S—S—N moiety, of the two molecules superimpose well if one is inverted on the other, but the phenyl groups do not. Thus, the two units are essentially conformational enantiomers. In molecule (**1*a***), the two phenyl rings are inclined to one another by 86.7 (3)°, and in molecule (**1*b***), by 81.1 (3)°. In the crystal, molecules are linked *via* C—H···π interactions, forming sheets lying parallel to (010).



1. Chemical context

The reactions of substrates with one or two sulfanyl chloride, acid chloride, and/or (alkoxydichloromethyl)sulfanyl moieties have been of interest to our laboratory for some time (Barany *et al.*, 1983; Barany & Mott, 1984; Schroll & Barany, 1986; Schroll *et al.*, 1990; Schroll *et al.*, 2012). In some of these experiments, bis[methyl(phenyl)amino]trisulfane was a component of more complicated mixtures of polysulfanes with varying numbers of S atoms. One such mixture was separated by preparative HPLC at 298 K, eluting with methanol–water (17:3). The fraction containing the title compound (dissolved in the eluting solvent) was cooled to 277 K, after which the trisulfane was obtained directly in crystalline form.



2. Structural commentary

The title compound, (**1**), was obtained in crystalline form after preparative HPLC, as described by Schroll & Barany (1986). The proposed molecular structure of (**1**) was confirmed by single-crystal X-ray analysis at 173 K. The molecules do not take advantage of the twofold axis provided as an available symmetry option by the *Fdd*2 space group. Instead, there are two molecules, (**1*a***) and (**1*b***), in the asymmetric unit (Fig. 1), and both of them display a pseudo-*trans* conformation (see

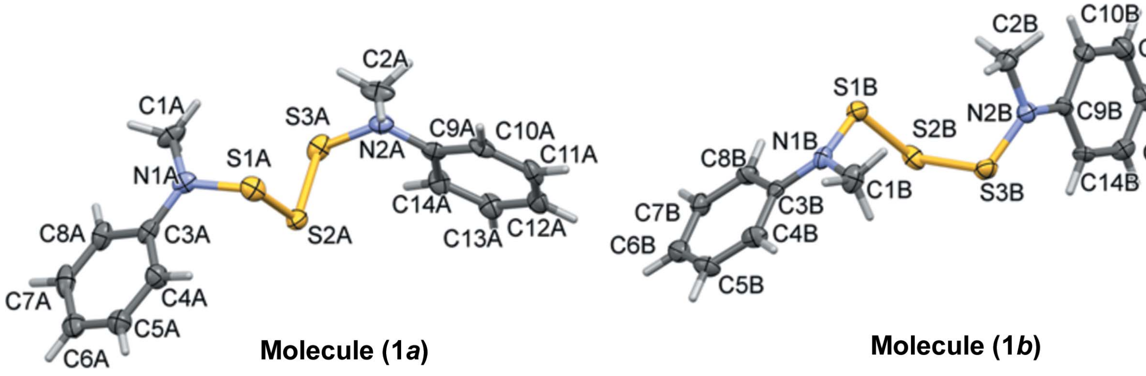


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

later). All bond distances and angles in both molecules are within expected ranges. Selected geometric parameters for compound (**1**) are given in Table 1. The two consecutive S—S bond lengths (comprising the trisulfane) of molecule (**1*a***) are 2.064 (3) and 2.078 (3) Å, and for molecule (**1*b***) are 2.076 (3) and 2.067 (2) Å. These values are similar to the value of 2.07 Å reported for the S—S bond length in elemental sulfur (S_8). Torsion angles about each of the two S—S bonds (comprising the trisulfane) are, respectively, 86.6 (2) and 87.0 (2)° for (**1*a***), and −84.6 (2) and −85.9 (2)° for (**1*b***). The core atoms, *viz*. the N—S—S—S—N moiety, of the two units superimpose well if one is inverted on the other, but the phenyl groups do not. Thus, the two units are essentially conformational enantiomers. Moreover, with respect to the four measured torsion angles, which range in absolute value from 84.6 (2) to 87.0 (2)°, these are slightly smaller than the theoretical optimum of 90.0° (Pauling, 1949; Torrico-Vallejos *et al.*, 2010). Finally, given the presence of three consecutive linearly connected sulfur atoms, representing two dihedral angles close to 90°, it is noteworthy that both of the molecules in the asymmetric unit display a pseudo-*trans* conformation (torsion angles +,+ or -,- across the two S—S bonds). The theoretically possible pseudo-*cis* (torsion angles +,- or -,+) conformation (Meyer, 1976) was not observed for these structures.

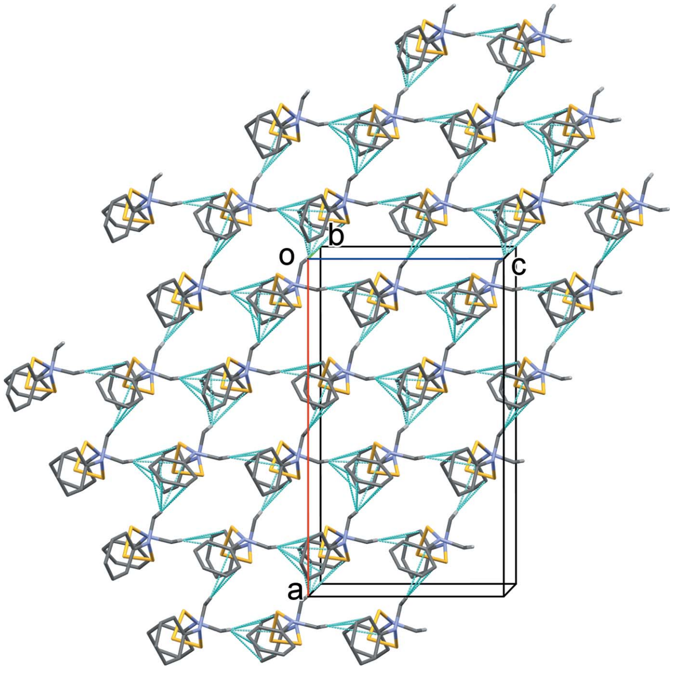


Figure 2
A view along the *b* axis of the crystal packing of the title compound. The dashed lines indicate the C—H$\cdots\pi$ interactions (see Table 2 for details). Only the H atoms involved in these interactions have been included for clarity.

3. Supramolecular features

In the crystal of (**1**), molecules are linked *via* C—H$\cdots\pi$ interactions, forming sheets lying parallel to (010) (see Table 2 and Fig. 2).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.36, February 2015; Groom & Allen, 2014) revealed the presence of two compounds (see Fig. 3) that also have an N—S—S—S—N moiety, *viz*. bis(oxamido)trisulfane, (**2**) (CSD refcode GEHPUE; Brunn *et al.*, 1988), and bis[*tert*-butyl(di-

Table 1
Selected geometric parameters (Å, °) of the title compound (**1**), and the comparison compounds (**2**) and (**3**).

	(**1*a***)	(**1*b***)	(**2**)	(**3**)
S1—N1	1.664 (5)	1.653 (5)	1.693 (2)	1.668 (2)
S1—S2	2.064 (3)	2.076 (3)	2.040 (1)	2.102 (1)
S2—S3	2.078 (3)	2.067 (2)	2.045 (1)	2.082 (1)
S3—N2	1.663 (6)	1.649 (5)	1.687 (2)	1.680 (2)
N1—S1—S2	106.9 (2)	107.3 (2)	105.0 (1)	110.0 (1)
S1—S2—S3	106.05 (11)	105.41 (11)	105.2 (2)	104.7 (1)
N2—S3—S2	107.6 (2)	107.2 (2)	103.8 (1)	110.3 (1)
N1—S1—S2—S3	86.6 (2)	−84.6 (2)	93.2 (7)	109.7 (2)
S1—S2—S3—N2	87.0 (2)	−85.9 (2)	−89.5 (2)	95.9 (1)

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

*Cg*1, *Cg*2, *Cg*3, and *Cg*4 are the centroids of rings C3*A*–C8*A*, C9*A*–C14*A*, C3*B*–C8*B*, and C9*B*–C14*B*, respectively.

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
$C1A—H1AA\cdots Cg2^{i}$	0.98	2.91	3.810 (7)	153
$C2A—H2AA\cdots Cg3^{ii}$	0.98	2.76	3.658 (8)	153
$C1B—H1BA\cdots Cg4^{iii}$	0.98	2.73	3.575 (7)	145
$C2B—H2BA\cdots Cg1^{ii}$	0.98	2.98	3.870 (7)	151

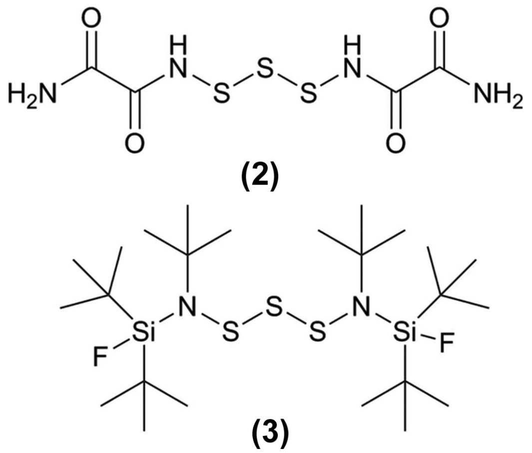
Symmetry codes: (i) $x - \frac{1}{4}, -y + \frac{3}{4}, z + \frac{1}{4}$; (ii) $-x + \frac{1}{2}, -y + 1, z + \frac{1}{2}$; (iii) $x + \frac{1}{4}, -y + \frac{5}{4}, z + \frac{1}{4}$.

tert-butylfluorosilyl)amino]trisulfane, (**3**) (SOTLAO; Klingebiel *et al.*, 1991). Unlike the title compound, (**1**), compounds (**2**) and (**3**) each have a unique conformation in the unit cell ($Z' = 1$). Selected geometric parameters of (**1**) and the comparison compounds, (**2**) and (**3**), are given in Table 1. While the average S—S bond length of the title compound is *ca* 2.07 Å, the corresponding value is longer (2.09 Å) in (**3**) and shorter (2.04 Å) in (**2**). The absolute value of the average torsion angle of the title compound (**1**) is *ca* 86.0°, while the corresponding value is larger (93.2 and −89.5°) and closer to the theoretical optimum in (**2**), and significantly larger (109.7 and 95.9°) in (**3**).

Note regarding nomenclature: In the discussion above, a consistent nomenclature scheme has been used that differs from the names used in the original publications, *viz.* bis(oxamido)trisulfan, (**2**) (Brunn *et al.*, 1988) and 1,3-bis[*tert*-butyl(di-*tert*-butylfluorsilyl)amino]trisulfan, (**3**) (Klingebiel *et al.*, 1991).

5. Synthesis and crystallization

The title compound, (**1**), was synthesized and obtained in crystalline form after preparative HPLC, as described by Schroll & Barany (1986): compound (**37**) in that publication.



(2)

(3)

Figure 3
Compounds that also have an N—S—S—S—N moiety, *viz.* bis(oxamido)trisulfane, (**2**) (CSD refcode, GEHPUE; Brunn *et al.*, 1988), and bis[*tert*-butyl(di-*tert*-butylfluorosilyl)amino]trisulfane, (**3**) (SOTLAO; Klingebiel *et al.*, 1991).

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{16}N_2S_3$
M_r	308.47
Crystal system, space group	Orthorhombic, *Fdd*2
Temperature (K)	173
a, *b*, *c* (Å)	19.284 (3), 56.440 (8), 11.1695 (15)
V (Å^3)	12157 (3)
Z	32
Radiation type	Mo *Kα*
μ (mm^{-1})	0.48
Crystal size (mm)	0.25 × 0.22 × 0.04
Data collection	
Diffractometer	Bruker *SMART* CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2001)
$T_{\min}$, $T_{\max}$	0.890, 0.981
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15884, 4978, 3097
$R_{\rm int}$	0.075
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.597
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.129, 1.06
No. of reflections	4978
No. of parameters	347
No. of restraints	1
H-atom treatment	H-atom parameters constrained
	$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 36.8709P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.43, −0.31
Absolute structure	2194 Friedel pairs (Flack, 1983)
Absolute structure parameter	0.08 (12)

Computer programs: *SMART* and *SAINT* (Bruker, 2001), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95–0.98 Å and $U_{\rm iso}(H) = 1.5U_{\rm eq}(C)$ for methyl H atoms and $1.2U_{\rm eq}(C)$ for other H atoms.

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Crystal structure of bis(*N*-methyl-*N*-phenylamino)trisulfane

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Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

Bis(*N*-methyl-*N*-phenylamino)trisulfane

Crystal data

$C_{14}H_{16}N_2S_3$
$M_r = 308.47$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 19.284$ (3) Å
$b = 56.440$ (8) Å
$c = 11.1695$ (15) Å
$V = 12157$ (3) Å^3
$Z = 32$
$F(000) = 5184$
$D_x = 1.348$ Mg m^{-3}
Melting point: 353 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1945 reflections
$\theta = 2.4$–$24.9°$
$\mu = 0.48$ mm^{-1}
$T = 173$ K
Plate, colorless
$0.25 \times 0.22 \times 0.04$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{\min} = 0.890$, $T_{\max} = 0.981$
15884 measured reflections
4978 independent reflections
3097 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.075$
$\theta_{\max} = 25.1°$, $\theta_{\min} = 1.4°$
$h = 0\rightarrow22$
$k = 0\rightarrow67$
$l = -13\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.129$
$S = 1.06$
4978 reflections
347 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
$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 36.8709P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.43$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}
Absolute structure: 2194 Friedel pairs (Flack, 1983)
Absolute structure parameter: 0.08 (12)

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*	$U_{\rm iso}$*/$U_{\rm eq}$
S1A	0.16203 (9)	0.35562 (3)	0.42989 (17)	0.0509 (5)
S2A	0.13888 (9)	0.37674 (3)	0.28446 (18)	0.0512 (5)
S3A	0.05789 (9)	0.39854 (3)	0.33839 (17)	0.0529 (5)
N1A	0.1096 (2)	0.33225 (10)	0.4230 (5)	0.0430 (14)
N2A	0.0925 (3)	0.42227 (10)	0.4033 (5)	0.0460 (14)
C1A	0.0371 (3)	0.33682 (13)	0.4515 (6)	0.0555 (19)
H1AA	0.0155	0.3222	0.4809	0.083*
H1AB	0.0129	0.3422	0.3794	0.083*
H1AC	0.0343	0.3491	0.5133	0.083*
C2A	0.1179 (4)	0.41859 (13)	0.5256 (6)	0.063 (2)
H2AA	0.1143	0.4334	0.5706	0.095*
H2AB	0.1665	0.4135	0.5230	0.095*
H2AC	0.0899	0.4064	0.5649	0.095*
C3A	0.1249 (3)	0.31310 (11)	0.3459 (6)	0.0428 (18)
C4A	0.1941 (3)	0.30690 (11)	0.3200 (6)	0.0501 (19)
H4AA	0.2307	0.3164	0.3502	0.060*
C5A	0.2096 (4)	0.28734 (13)	0.2520 (7)	0.059 (2)
H5AA	0.2567	0.2832	0.2383	0.071*
C6A	0.1577 (4)	0.27368 (13)	0.2037 (7)	0.056 (2)
H6AA	0.1684	0.2605	0.1544	0.067*
C7A	0.0894 (5)	0.27962 (12)	0.2284 (7)	0.063 (2)
H7AA	0.0531	0.2699	0.1980	0.076*
C8A	0.0733 (3)	0.29903 (12)	0.2953 (7)	0.0514 (19)
H8AA	0.0260	0.3030	0.3076	0.062*
C9A	0.1215 (3)	0.44089 (11)	0.3334 (7)	0.0408 (17)
C10A	0.1734 (3)	0.45591 (12)	0.3798 (6)	0.0458 (19)
H10A	0.1906	0.4533	0.4584	0.055*
C11A	0.1992 (3)	0.47418 (11)	0.3132 (7)	0.052 (2)
H11A	0.2340	0.4841	0.3462	0.062*
C12A	0.1756 (4)	0.47837 (12)	0.2001 (8)	0.055 (2)
H12A	0.1935	0.4912	0.1542	0.066*
C13A	0.1253 (4)	0.46368 (12)	0.1536 (7)	0.055 (2)
H13A	0.1086	0.4665	0.0749	0.066*
C14A	0.0987 (4)	0.44514 (12)	0.2171 (7)	0.0514 (19)
H14A	0.0646	0.4352	0.1821	0.062*

S1B	0.34517 (9)	0.60013 (3)	0.43382 (17)	0.0503 (5)
S2B	0.35517 (9)	0.62207 (3)	0.28572 (18)	0.0504 (5)
S3B	0.44058 (9)	0.64310 (3)	0.32075 (16)	0.0473 (5)
N1B	0.3972 (2)	0.57722 (9)	0.4127 (5)	0.0406 (13)
N2B	0.4133 (3)	0.66599 (9)	0.3995 (5)	0.0410 (14)
C1B	0.4706 (3)	0.58075 (12)	0.4394 (7)	0.056 (2)
H1BA	0.4893	0.5665	0.4775	0.084*
H1BB	0.4758	0.5943	0.4936	0.084*
H1BC	0.4958	0.5839	0.3649	0.084*
C2B	0.3957 (4)	0.66091 (11)	0.5245 (6)	0.0502 (18)
H2BA	0.4010	0.6754	0.5724	0.075*
H2BB	0.4269	0.6486	0.5554	0.075*
H2BC	0.3477	0.6554	0.5293	0.075*
C3B	0.3785 (3)	0.55805 (10)	0.3387 (6)	0.0360 (15)
C4B	0.4264 (3)	0.54146 (11)	0.2998 (6)	0.0470 (18)
H4BA	0.4739	0.5435	0.3201	0.056*
C5B	0.4072 (4)	0.52205 (12)	0.2322 (6)	0.0528 (19)
H5BA	0.4413	0.5108	0.2098	0.063*
C6B	0.3392 (4)	0.51874 (12)	0.1965 (7)	0.052 (2)
H6BA	0.3259	0.5056	0.1486	0.062*
C7B	0.2916 (3)	0.53537 (11)	0.2336 (6)	0.0467 (18)
H7BA	0.2444	0.5335	0.2110	0.056*
C8B	0.3098 (3)	0.55452 (10)	0.3020 (6)	0.0418 (16)
H8BA	0.2753	0.5656	0.3249	0.050*
C9B	0.3837 (3)	0.68621 (10)	0.3444 (7)	0.0369 (16)
C10B	0.3412 (3)	0.70186 (10)	0.4077 (6)	0.0446 (18)
H10B	0.3288	0.6984	0.4881	0.054*
C11B	0.3175 (4)	0.72197 (13)	0.3558 (8)	0.060 (2)
H11B	0.2898	0.7326	0.4015	0.072*
C12B	0.3328 (4)	0.72757 (12)	0.2363 (8)	0.054 (2)
H12B	0.3158	0.7417	0.2003	0.065*
C13B	0.3730 (3)	0.71203 (11)	0.1735 (7)	0.0481 (17)
H13B	0.3840	0.7153	0.0922	0.058*
C14B	0.3981 (3)	0.69158 (11)	0.2259 (7)	0.0436 (17)
H14B	0.4257	0.6810	0.1800	0.052*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0414 (11)	0.0603 (11)	0.0510 (14)	0.0036 (8)	−0.0136 (9)	−0.0069 (10)
S2A	0.0612 (11)	0.0511 (10)	0.0414 (12)	−0.0137 (8)	0.0091 (10)	−0.0026 (10)
S3A	0.0373 (10)	0.0635 (11)	0.0577 (14)	−0.0058 (8)	−0.0102 (9)	0.0142 (10)
N1A	0.032 (3)	0.054 (3)	0.043 (4)	0.006 (2)	−0.002 (3)	0.008 (3)
N2A	0.049 (3)	0.054 (4)	0.035 (4)	0.005 (3)	0.000 (3)	0.001 (3)
C1A	0.029 (4)	0.085 (5)	0.053 (5)	0.000 (3)	0.002 (3)	0.019 (4)
C2A	0.070 (5)	0.090 (5)	0.029 (5)	0.029 (4)	−0.007 (4)	−0.003 (4)
C3A	0.033 (4)	0.050 (4)	0.046 (5)	−0.006 (3)	−0.005 (3)	0.017 (4)
C4A	0.038 (4)	0.055 (4)	0.057 (6)	−0.003 (3)	−0.008 (4)	−0.005 (4)

C5A	0.058 (5)	0.057 (5)	0.062 (6)	−0.004 (4)	0.001 (4)	0.000 (4)
C6A	0.072 (6)	0.055 (5)	0.041 (5)	−0.008 (4)	−0.002 (4)	0.006 (3)
C7A	0.084 (6)	0.049 (5)	0.056 (6)	−0.024 (4)	−0.029 (4)	0.018 (4)
C8A	0.042 (4)	0.053 (4)	0.060 (6)	−0.010 (3)	−0.015 (4)	0.014 (4)
C9A	0.030 (4)	0.049 (4)	0.043 (5)	0.014 (3)	−0.001 (3)	−0.005 (3)
C10A	0.042 (4)	0.059 (4)	0.037 (5)	0.011 (3)	−0.013 (3)	−0.018 (3)
C11A	0.047 (4)	0.035 (4)	0.074 (7)	0.001 (3)	−0.012 (4)	−0.018 (4)
C12A	0.061 (5)	0.035 (4)	0.069 (6)	0.005 (3)	0.007 (4)	−0.002 (4)
C13A	0.067 (5)	0.050 (4)	0.050 (6)	0.002 (4)	−0.012 (4)	0.000 (4)
C14A	0.052 (4)	0.050 (4)	0.052 (6)	−0.005 (3)	−0.019 (4)	−0.004 (4)
S1B	0.0489 (11)	0.0517 (10)	0.0502 (14)	−0.0096 (8)	0.0123 (10)	−0.0097 (9)
S2B	0.0595 (11)	0.0440 (10)	0.0476 (12)	0.0073 (8)	−0.0123 (10)	−0.0093 (9)
S3B	0.0400 (10)	0.0478 (10)	0.0540 (14)	0.0062 (7)	0.0123 (9)	0.0058 (8)
N1B	0.039 (3)	0.041 (3)	0.041 (4)	−0.011 (2)	−0.004 (3)	0.005 (3)
N2B	0.039 (3)	0.045 (3)	0.039 (4)	−0.004 (2)	−0.002 (3)	−0.002 (3)
C1B	0.045 (4)	0.061 (4)	0.062 (6)	−0.011 (3)	−0.016 (4)	0.009 (4)
C2B	0.057 (4)	0.061 (4)	0.033 (5)	−0.014 (3)	−0.003 (4)	0.002 (4)
C3B	0.039 (4)	0.040 (4)	0.028 (4)	−0.007 (3)	0.004 (3)	0.007 (3)
C4B	0.037 (4)	0.057 (4)	0.047 (5)	0.013 (3)	0.000 (4)	0.009 (4)
C5B	0.050 (5)	0.056 (4)	0.052 (5)	0.016 (3)	0.002 (4)	−0.008 (4)
C6B	0.060 (5)	0.047 (4)	0.049 (6)	0.001 (3)	0.006 (4)	−0.001 (3)
C7B	0.041 (4)	0.050 (4)	0.049 (5)	−0.012 (3)	−0.001 (3)	−0.005 (3)
C8B	0.034 (4)	0.045 (4)	0.047 (5)	0.004 (3)	0.001 (3)	−0.002 (3)
C9B	0.029 (3)	0.037 (4)	0.045 (5)	−0.007 (3)	0.002 (3)	−0.014 (3)
C10B	0.051 (4)	0.045 (4)	0.037 (5)	−0.004 (3)	0.001 (3)	−0.005 (3)
C11B	0.061 (5)	0.048 (5)	0.071 (7)	0.003 (4)	0.000 (5)	−0.015 (4)
C12B	0.051 (4)	0.041 (4)	0.071 (6)	0.004 (3)	−0.013 (4)	−0.007 (4)
C13B	0.042 (4)	0.057 (4)	0.045 (5)	−0.002 (3)	0.005 (4)	0.005 (4)
C14B	0.044 (4)	0.042 (4)	0.044 (5)	0.004 (3)	0.017 (3)	−0.006 (3)

Geometric parameters (Å, º)

S1A—N1A	1.664 (5)	S1B—N1B	1.653 (5)
S1A—S2A	2.064 (3)	S1B—S2B	2.076 (3)
S2A—S3A	2.078 (3)	S2B—S3B	2.067 (2)
S3A—N2A	1.663 (6)	S3B—N2B	1.649 (5)
N1A—C3A	1.413 (8)	N1B—C3B	1.408 (8)
N1A—C1A	1.457 (7)	N1B—C1B	1.460 (7)
N2A—C9A	1.424 (8)	N2B—C9B	1.416 (8)
N2A—C2A	1.465 (8)	N2B—C2B	1.465 (8)
C1A—H1AA	0.9800	C1B—H1BA	0.9800
C1A—H1AB	0.9800	C1B—H1BB	0.9800
C1A—H1AC	0.9800	C1B—H1BC	0.9800
C2A—H2AA	0.9800	C2B—H2BA	0.9800
C2A—H2AB	0.9800	C2B—H2BB	0.9800
C2A—H2AC	0.9800	C2B—H2BC	0.9800
C3A—C8A	1.393 (8)	C3B—C4B	1.386 (8)
C3A—C4A	1.410 (9)	C3B—C8B	1.401 (8)

C4A—C5A	1.373 (9)	C4B—C5B	1.381 (9)
C4A—H4AA	0.9500	C4B—H4BA	0.9500
C5A—C6A	1.373 (9)	C5B—C6B	1.383 (9)
C5A—H5AA	0.9500	C5B—H5BA	0.9500
C6A—C7A	1.388 (10)	C6B—C7B	1.377 (9)
C6A—H6AA	0.9500	C6B—H6BA	0.9500
C7A—C8A	1.362 (10)	C7B—C8B	1.369 (8)
C7A—H7AA	0.9500	C7B—H7BA	0.9500
C8A—H8AA	0.9500	C8B—H8BA	0.9500
C9A—C14A	1.392 (9)	C9B—C14B	1.386 (9)
C9A—C10A	1.410 (9)	C9B—C10B	1.396 (8)
C10A—C11A	1.365 (9)	C10B—C11B	1.354 (9)
C10A—H10A	0.9500	C10B—H10B	0.9500
C11A—C12A	1.363 (10)	C11B—C12B	1.403 (10)
C11A—H11A	0.9500	C11B—H11B	0.9500
C12A—C13A	1.378 (9)	C12B—C13B	1.365 (9)
C12A—H12A	0.9500	C12B—H12B	0.9500
C13A—C14A	1.365 (9)	C13B—C14B	1.382 (9)
C13A—H13A	0.9500	C13B—H13B	0.9500
C14A—H14A	0.9500	C14B—H14B	0.9500

N1A—S1A—S2A	106.9 (2)	N1B—S1B—S2B	107.3 (2)
S1A—S2A—S3A	106.05 (11)	S3B—S2B—S1B	105.41 (11)
N2A—S3A—S2A	107.6 (2)	N2B—S3B—S2B	107.2 (2)
C3A—N1A—C1A	118.0 (5)	C3B—N1B—C1B	118.2 (5)
C3A—N1A—S1A	120.5 (4)	C3B—N1B—S1B	122.0 (4)
C1A—N1A—S1A	115.6 (4)	C1B—N1B—S1B	116.9 (4)
C9A—N2A—C2A	119.0 (6)	C9B—N2B—C2B	118.6 (5)
C9A—N2A—S3A	120.9 (5)	C9B—N2B—S3B	121.9 (5)
C2A—N2A—S3A	115.3 (5)	C2B—N2B—S3B	115.4 (4)
N1A—C1A—H1AA	109.5	N1B—C1B—H1BA	109.5
N1A—C1A—H1AB	109.5	N1B—C1B—H1BB	109.5
H1AA—C1A—H1AB	109.5	H1BA—C1B—H1BB	109.5
N1A—C1A—H1AC	109.5	N1B—C1B—H1BC	109.5
H1AA—C1A—H1AC	109.5	H1BA—C1B—H1BC	109.5
H1AB—C1A—H1AC	109.5	H1BB—C1B—H1BC	109.5
N2A—C2A—H2AA	109.5	N2B—C2B—H2BA	109.5
N2A—C2A—H2AB	109.5	N2B—C2B—H2BB	109.5
H2AA—C2A—H2AB	109.5	H2BA—C2B—H2BB	109.5
N2A—C2A—H2AC	109.5	N2B—C2B—H2BC	109.5
H2AA—C2A—H2AC	109.5	H2BA—C2B—H2BC	109.5
H2AB—C2A—H2AC	109.5	H2BB—C2B—H2BC	109.5
C8A—C3A—C4A	116.9 (7)	C4B—C3B—C8B	116.3 (6)
C8A—C3A—N1A	122.3 (6)	C4B—C3B—N1B	122.2 (6)
C4A—C3A—N1A	120.8 (6)	C8B—C3B—N1B	121.5 (6)
C5A—C4A—C3A	121.2 (6)	C5B—C4B—C3B	121.8 (6)
C5A—C4A—H4AA	119.4	C5B—C4B—H4BA	119.1
C3A—C4A—H4AA	119.4	C3B—C4B—H4BA	119.1

C4A—C5A—C6A	120.7 (7)	C4B—C5B—C6B	121.3 (6)
C4A—C5A—H5AA	119.6	C4B—C5B—H5BA	119.3
C6A—C5A—H5AA	119.6	C6B—C5B—H5BA	119.3
C5A—C6A—C7A	118.6 (8)	C7B—C6B—C5B	116.9 (7)
C5A—C6A—H6AA	120.7	C7B—C6B—H6BA	121.5
C7A—C6A—H6AA	120.7	C5B—C6B—H6BA	121.5
C8A—C7A—C6A	121.3 (7)	C8B—C7B—C6B	122.3 (6)
C8A—C7A—H7AA	119.3	C8B—C7B—H7BA	118.8
C6A—C7A—H7AA	119.3	C6B—C7B—H7BA	118.8
C7A—C8A—C3A	121.2 (7)	C7B—C8B—C3B	121.2 (6)
C7A—C8A—H8AA	119.4	C7B—C8B—H8BA	119.4
C3A—C8A—H8AA	119.4	C3B—C8B—H8BA	119.4
C14A—C9A—C10A	117.7 (6)	C14B—C9B—C10B	117.5 (6)
C14A—C9A—N2A	121.0 (6)	C14B—C9B—N2B	120.7 (6)
C10A—C9A—N2A	121.4 (6)	C10B—C9B—N2B	121.8 (7)
C11A—C10A—C9A	120.8 (7)	C11B—C10B—C9B	120.8 (7)
C11A—C10A—H10A	119.6	C11B—C10B—H10B	119.6
C9A—C10A—H10A	119.6	C9B—C10B—H10B	119.6
C12A—C11A—C10A	121.0 (7)	C10B—C11B—C12B	121.6 (7)
C12A—C11A—H11A	119.5	C10B—C11B—H11B	119.2
C10A—C11A—H11A	119.5	C12B—C11B—H11B	119.2
C11A—C12A—C13A	118.6 (7)	C13B—C12B—C11B	117.6 (7)
C11A—C12A—H12A	120.7	C13B—C12B—H12B	121.2
C13A—C12A—H12A	120.7	C11B—C12B—H12B	121.2
C14A—C13A—C12A	122.1 (7)	C12B—C13B—C14B	121.2 (7)
C14A—C13A—H13A	119.0	C12B—C13B—H13B	119.4
C12A—C13A—H13A	119.0	C14B—C13B—H13B	119.4
C13A—C14A—C9A	119.8 (7)	C13B—C14B—C9B	121.2 (6)
C13A—C14A—H14A	120.1	C13B—C14B—H14B	119.4
C9A—C14A—H14A	120.1	C9B—C14B—H14B	119.4

N1A—S1A—S2A—S3A	86.6 (2)	N1B—S1B—S2B—S3B	−84.6 (2)
S1A—S2A—S3A—N2A	87.0 (2)	S1B—S2B—S3B—N2B	−85.9 (2)
S2A—S1A—N1A—C3A	80.2 (5)	S2B—S1B—N1B—C3B	−79.9 (5)
S2A—S1A—N1A—C1A	−72.2 (5)	S2B—S1B—N1B—C1B	80.3 (5)
S2A—S3A—N2A—C9A	77.9 (5)	S2B—S3B—N2B—C9B	−83.1 (5)
S2A—S3A—N2A—C2A	−77.0 (5)	S2B—S3B—N2B—C2B	73.7 (5)
C1A—N1A—C3A—C8A	1.3 (9)	C1B—N1B—C3B—C4B	5.8 (9)
S1A—N1A—C3A—C8A	−150.5 (5)	S1B—N1B—C3B—C4B	165.7 (5)
C1A—N1A—C3A—C4A	−176.4 (6)	C1B—N1B—C3B—C8B	−175.2 (6)
S1A—N1A—C3A—C4A	31.7 (8)	S1B—N1B—C3B—C8B	−15.3 (8)
C8A—C3A—C4A—C5A	−2.5 (10)	C8B—C3B—C4B—C5B	−2.3 (10)
N1A—C3A—C4A—C5A	175.4 (6)	N1B—C3B—C4B—C5B	176.7 (6)
C3A—C4A—C5A—C6A	2.3 (12)	C3B—C4B—C5B—C6B	2.4 (11)
C4A—C5A—C6A—C7A	−2.2 (11)	C4B—C5B—C6B—C7B	−1.4 (11)
C5A—C6A—C7A—C8A	2.5 (11)	C5B—C6B—C7B—C8B	0.5 (11)
C6A—C7A—C8A—C3A	−2.8 (11)	C6B—C7B—C8B—C3B	−0.6 (11)
C4A—C3A—C8A—C7A	2.8 (10)	C4B—C3B—C8B—C7B	1.4 (10)

N1A—C3A—C8A—C7A	−175.1 (6)	N1B—C3B—C8B—C7B	−177.6 (6)
C2A—N2A—C9A—C14A	−179.5 (6)	C2B—N2B—C9B—C14B	−179.3 (5)
S3A—N2A—C9A—C14A	26.5 (8)	S3B—N2B—C9B—C14B	−23.2 (8)
C2A—N2A—C9A—C10A	−0.7 (8)	C2B—N2B—C9B—C10B	3.1 (9)
S3A—N2A—C9A—C10A	−154.7 (5)	S3B—N2B—C9B—C10B	159.1 (5)
C14A—C9A—C10A—C11A	1.1 (9)	C14B—C9B—C10B—C11B	−2.5 (10)
N2A—C9A—C10A—C11A	−177.8 (6)	N2B—C9B—C10B—C11B	175.2 (6)
C9A—C10A—C11A—C12A	−0.1 (10)	C9B—C10B—C11B—C12B	1.8 (11)
C10A—C11A—C12A—C13A	−0.3 (10)	C10B—C11B—C12B—C13B	−0.3 (10)
C11A—C12A—C13A—C14A	−0.2 (11)	C11B—C12B—C13B—C14B	−0.4 (10)
C12A—C13A—C14A—C9A	1.1 (11)	C12B—C13B—C14B—C9B	−0.4 (10)
C10A—C9A—C14A—C13A	−1.5 (10)	C10B—C9B—C14B—C13B	1.8 (9)
N2A—C9A—C14A—C13A	177.4 (6)	N2B—C9B—C14B—C13B	−175.9 (6)

Hydrogen-bond geometry (Å, º)

*Cg*1, *Cg*2, *Cg*3, and *Cg*4 are the centroids of rings C3A–C8A, C9A–C14A, C3B–C8B, and C9B–C14B, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1*A*—H1*AA*···*Cg*2^{i}	0.98	2.91	3.810 (7)	153
C2*A*—H2*AA*···*Cg*3^{ii}	0.98	2.76	3.658 (8)	153
C1*B*—H1*BA*···*Cg*4^{iii}	0.98	2.73	3.575 (7)	145
C2*B*—H2*BA*···*Cg*1^{ii}	0.98	2.98	3.870 (7)	151

Symmetry codes: (i) *x*−1/4, −*y*+3/4, *z*+1/4; (ii) −*x*+1/2, −*y*+1, *z*+1/2; (iii) *x*+1/4, −*y*+5/4, *z*+1/4.