significant improvement in the R values, to suppress the anomalies concerning the out-of-plane vibrations of the deuterium atoms in the central ring and to find consistent potentials from neutron and X-ray diffraction data. The barrier height, found to be about 0·6 kcal mol^{-1}, is in good agreement with that given by potential-energy calculations. This barrier energy is of the same order as the thermal energy kT of any mode at room temperature, or even at 200 K. But it is only for the internal g mode that this energy is sufficient to bring the rings near the barrier top. For the central ring the doubly peaked distribution function is an argument for an order–disorder type of transition. However, the smallness of the double-well barrier height suggests that the p-terphenyl structural phase transition is near the boundary between the order–disorder and displacive regimes.

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The Crystal and Molecular Structure of *trans*-1,3,5-Trimethyl-1,3,5-triphenylcyclotrisilthiane: a Stable Twist-Boat Conformation

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The title compound, $C_{21}H_{24}Si_3S_3$, crystallizes in the triclinic system, space group $P\bar{1}$, with unit-cell dimensions $a = 10·941$ (8), $b = 9·423$ (8), $c = 12·252$ (8) Å, $\alpha = 80·02$ (12), $\beta = 75·80$ (12) and $\gamma = 99·40$ (12)°. The structure was solved by the Patterson method and refined by block-diagonal least squares to a final R of 0·059 for 2086 observed reflexions. The structure consists of alternating Si and S atoms in a six-membered ring. A methyl group and a phenyl group are attached to each Si atom. The novelty of this structure is that the Si–S heterocyclic ring is in the twist-boat conformation.

Introduction

In chair–chair equilibrations of various six-membered rings, the boat and twist-boat conformations are postulated as discrete intermediates. The thermodynamic calculations of Beckett, Pitzer & Spitzer (1947) and Howlett (1957) indicate that the boat forms of six-membered ring carbocycles are of higher energy (3–12 kcal mol^{-1}) than the corresponding chair conformations. This is confirmed by the direct experimental evidence of Johnson, Margrave, Bauer, Frisch, Dreger & Hubbard (1960) and of Jensen & Bushweller (1969). Similarly, reports for other six-membered ring systems such as C–O (Moerman, 1937), C–N (Gutowsky & Temussi, 1967), C–S (Moerman & Wiebenga, 1937; Campaigne, Chamberlain & Edwards, 1962), Si–N (Moedritzer & van Wazer, 1966; Yokoi & Yamasaki,

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1953) and S—S (Donohue, Caron & Goldish, 1961), conclude that the chair conformation is the most predominant conformer.

The conformation of the Si—S six-membered heterocycle has been in dispute since it was first isolated by Etienne (1952). The electron diffraction measurements made by Yokoi, Nomura & Yamasaki (1955) on $[(CH_3)_2SiS]_3$ in the vapour state at 200°C could not distinguish between the chair and boat conformations, but definitely ruled out the planar ring which is the conformation of the analogous Si—O heterocycle (Roth & Harker, 1948). In their dipole-moment study, Kurita & Kondo (1954) concluded that the Si—S ring in $[(CH_3)_2SiS]_3$ was mostly in the chair conformation while Kriegsmann & Clauss (1959) showed by IR spectroscopy that the most probable structure for the Si—S heterocycle, also in $[(CH_3)_2SiS]_3$, was the boat conformation. The variable-temperature PMR of trans-1,3,5-trimethyl-1,3,5-triphenylcyclotrisilthiane in a number of solvents could be explained by a rapid exchange between the two chair conformations (Pazdernik, 1970). In order to establish the conformation of the Si—S heterocycle a crystal structure analysis was necessary. Because $[(CH_3)_2SiS]_3$ is liquid at room temperature (m.p. 17–18°C), we chose to determine the structure of $[CH_3C_6H_5SiS]_3$ since PMR indicated that it has all possible conformers in solution.

Experimental

The crystals of $[CH_3C_6H_5SiS]_3$ were synthesized by reaction between methyl(phenyl)dichlorosilane with H_2S. The crystals obtained, always those of the trans isomer, were isolated as colourless prisms from a saturated hexane solution. The cis compound could not be isolated. Precession films indicated that the crystals of trans-$[CH_3C_6H_5SiS]_3$ were triclinic. The unit-cell dimensions obtained by least squares from diffractometer measurements of 12 reflexions are listed in Table 1. The $P\bar{1}$ space group was chosen on the basis of the distribution of the normalized structure factors which very strongly suggested a centrosymmetric space group.

The intensity data were measured on an automatic Picker diffractometer with Ni-filtered Cu $K\alpha$ radiation, up to $2\theta = 110°$. The θ–2θ scan technique was used with a scan rate of $1°$ (2θ) min^{-1} and a minimum scan width of $1.5°$ (2θ). Background counts of 20 s were

taken at each end of the scan range. It should be mentioned that while the data were collected, a mechanical malfunction of the instrument interrupted the measurements for two weeks. The intensities of three reference reflexions, measured after each group of 30 reflexions, showed an overall decrease of 13 to 18% of their initial values indicating a significant crystal decomposition. The data reduction was performed with the programs* of Ahmed, Hall, Pippy & Huber (1966). Of the 2345 measured reflexions, 2086 with $I/\sigma(I) \geq 2.50$ were retained for the structure determination and refinement. The crystal dimensions measured perpendicular to $|001|$, $|101|$ and $[1\bar{2}1]$ were $0.22 \times 0.38 \times 0.45$ mm. Because of the relatively large crystal size and absorption coefficient the intensities were corrected for absorption. The transmission factor ranged from 0.32 to 0.55. The X-ray scattering factors were those of Cromer & Waber (1965) for Si, S and C and those of Stewart, Davidson & Simpson (1965) for H. The anomalous dispersion corrections for S and Si were taken from Cromer (1965).

Structure determination and refinement

The Si—S six-membered ring was located on a Patterson map. All the remaining C atoms were found by a combination of Fourier syntheses and structure-factor calculations. At this stage, $R = \Sigma \Delta F/\Sigma |F_o|$ had the value of 0.26. Four cycles of block-diagonal least-squares refinement, the quantity minimized being $\Sigma w(|F_o| - |F_c|)^2$, lowered this value to 0.145. Anisotropic refinement followed by a Fourier difference map revealed most of the phenyl H atoms. These were included in the structure-factor calculation ($R = 0.072$). However, no more H atoms could be found on a subsequent Fourier difference map. The theoretical positions of the missing H atoms were computed assuming a C—H bond distance of 1.0 Å and sp^2 or sp^3 geometry. The methyl H atoms were placed so that the methyl groups were in staggered positions with respect to the Si—S and Si—C bonds. When all the H atoms were included in the structure-factor calculation, with isotropic temperature factors as listed in Table 2, R was lowered to 0.059.† The relatively high value of R is attributed to the crystal decomposition during the intensity measurements. A weight of unity was taken for

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* The computer programs used were locally modified versions of the following: NRC-2: data reduction; NRC-3: absorption correction (Ahmed et al., 1966); FORDAP: Fourier and Patterson maps (A. Zalkin); NUCLS: least-squares refinement (R. J. Doedens & J. A. Ibers), with block-diagonal approximation option introduced by J. Sygusch; ORTEP: stereo drawings (C. K. Johnson).

† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32328 (10 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Crystal data

$C_{21}H_{24}Si_3S_3$, $M_r = 456.87$, m.p. $114°C$, $F(000) = 480$
Triclinic, $a = 10.941$ (8), $b = 9.423$ (8), $c = 12.252$ (8) Å
$\alpha = 80.02$ (12), $\beta = 75.80$ (12), $\gamma = 99.40$ (12)°, $V = 1177.6$ Å^3
$\rho_{obs} = 1.272$, $\rho_{cal} = 1.289$ g cm^{-3}, $Z = 2$
Space group $P\bar{1}$, μ(Cu $K\alpha$) = 42 cm^{-1}, λ(Cu $K\alpha$) = 1.54178 Å

Table 2. *Fractional coordinates, thermal parameters and their e.s.d.'s*

The thermal parameters are the coefficients of the expression $T = \exp[-2\pi^2(U_{11}a^{*2}h^2 + \cdots + 2U_{12}a^*b^*hk + \cdots]$ for S. Si and C atoms and $T = \exp[-8\pi^2 U(\sin^2 \theta/\lambda^2)]$ for H atoms. The coordinates are in fractions of unit-cell edges $\times 10^5$ for S and Si and $\times 10^4$ for C and H atoms. All thermal parameters are given $\times 10^4$. The standard deviations refer to the least significant digit.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	30595 (20)	11736 (21)	41344 (15)	852 (14)	563 (12)	401 (10)	287 (10)	−100 (9)	−203 (9)
S(2)	41064 (17)	33477 (20)	12724 (15)	519 (11)	506 (11)	505 (11)	135 (9)	−46 (9)	−112 (9)
S(3)	8692 (17)	29567 (21)	29724 (15)	538 (11)	660 (12)	460 (10)	99 (9)	−69 (9)	−255 (9)
Si(1)	36467 (19)	11816 (20)	23277 (15)	584 (12)	415 (11)	404 (11)	141 (9)	−53 (9)	−198 (9)
Si(2)	23494 (18)	41421 (21)	14392 (15)	542 (12)	452 (11)	428 (11)	147 (9)	−115 (9)	−208 (9)
Si(3)	18415 (19)	27288 (21)	43023 (15)	626 (13)	453 (11)	368 (10)	117 (10)	−38 (9)	−220 (9)
C(1)	2429 (7)	−85 (8)	1956 (6)	644 (50)	518 (47)	558 (47)	41 (39)	−17 (39)	−276 (38)
C(2)	2832 (7)	6119 (8)	1401 (7)	701 (53)	478 (46)	673 (51)	172 (40)	−204 (42)	−224 (39)
C(3)	532 (8)	1921 (9)	5662 (6)	791 (57)	611 (52)	437 (44)	82 (43)	27 (39)	−220 (39)
C(11)	5220 (7)	615 (8)	2121 (6)	667 (50)	417 (41)	438 (40)	193 (36)	−62 (35)	−195 (33)
C(12)	6149 (8)	1177 (9)	2613 (7)	669 (53)	669 (54)	749 (55)	314 (43)	−128 (43)	−398 (45)
C(13)	7315 (9)	743 (12)	2483 (8)	762 (63)	1120 (80)	976 (71)	359 (57)	−236 (53)	−502 (62)
C(14)	7591 (8)	−254 (10)	1867 (8)	701 (59)	795 (63)	959 (67)	397 (50)	−28 (50)	−168 (52)
C(15)	6689 (9)	−874 (9)	1341 (7)	958 (67)	677 (57)	682 (55)	463 (50)	−7 (48)	−264 (46)
C(16)	5540 (8)	−447 (9)	1466 (6)	762 (56)	573 (50)	575 (48)	216 (43)	−73 (41)	−267 (40)
C(21)	1671 (7)	3847 (8)	210 (6)	600 (46)	486 (43)	427 (41)	224 (36)	−158 (35)	−252 (35)
C(22)	605 (8)	2797 (10)	321 (6)	718 (55)	846 (61)	517 (47)	80 (46)	−240 (41)	−283 (44)
C(23)	121 (9)	2657 (11)	−619 (7)	798 (62)	1017 (73)	751 (60)	160 (53)	−329 (49)	−368 (54)
C(24)	718 (9)	3586 (12)	−1656 (7)	993 (69)	1351 (86)	636 (55)	681 (64)	−431 (50)	−634 (57)
C(25)	1776 (10)	4661 (11)	−1788 (7)	1204 (82)	958 (74)	562 (54)	83 (62)	−327 (54)	−117 (50)
C(26)	2231 (9)	4795 (10)	−852 (7)	955 (67)	904 (67)	453 (48)	92 (53)	−222 (46)	−161 (45)
C(31)	2756 (7)	4530 (9)	4358 (6)	641 (51)	690 (53)	425 (43)	90 (42)	−145 (37)	−193 (38)
C(32)	4097 (8)	5002 (8)	3910 (7)	731 (54)	495 (47)	685 (52)	63 (41)	−155 (43)	−249 (41)
C(33)	4731 (9)	6389 (10)	3917 (8)	769 (61)	815 (64)	768 (60)	−72 (51)	−276 (49)	−71 (50)
C(34)	4003 (10)	7346 (9)	4444 (7)	1361 (84)	477 (51)	677 (56)	−12 (52)	−424 (56)	−78 (43)
C(35)	2707 (9)	6913 (9)	4886 (7)	1218 (76)	513 (51)	688 (56)	282 (50)	−230 (52)	−340 (44)
C(36)	2082 (8)	5532 (8)	4865 (7)	857 (59)	487 (47)	667 (52)	209 (43)	−170 (44)	−328 (41)

	x	y	z	U		x	y	z	U
H(12)	6016	1774	3013	1021	H(34)	4628	8325	4330	1109
H(13)	8071	1022	2827	1089	H(35)	2272	7499	5192	1103
H(14)	8378	−654	1847	1071	H(36)	1155	5291	5114	1021
H(15)	6824	−1797	928	918	H[Me(1)]	2688	−150	1095	1154
H(16)	4982	−919	1144	610	H[Me(2)]	1603	265	2066	1198
H(22)	284	1984	1006	699	H[Me(3)]	2238	−1051	2473	1198
H(23)	−649	1871	−514	646	H[Me(4)]	2147	6645	1347	1265
H(24)	388	3444	−2243	711	H[Me(5)]	3546	6621	665	1234
H(25)	2134	5377	−2395	1168	H[Me(6)]	3201	6193	2071	1047
H(26)	2942	5811	−916	619	H[Me(7)]	87	964	5607	1344
H(32)	4567	4389	3587	680	H[Me(8)]	−29	2654	5763	994
H(33)	5631	6472	3565	698	H[Me(9)]	868	1825	6246	1327

each reflexion. At the end of the refinement the average shift-to-σ ratio was less than 0.3. A final electron density map showed no significant residual density, the extreme fluctuations being −0.25 and +0.31 e Å⁻³.

Results and discussion

The positional and thermal parameters and their standard deviations are presented in Table 2. A view of the molecule and its thermal ellipsoids is shown in Fig. 1.

The structure consists of alternating Si and S atoms in a six-membered ring, with a methyl group and a

phenyl group attached to each Si atom. There are two methyl groups and one phenyl group on the same side of the Si—S heterocycle which shows this to be in the *trans* configuration. In this arrangement the Si(1)—C(1) and Si(3)—C(31) bonds and the Si(3)—C(3) and Si(2)—C(21) bonds are nearly parallel to each other. The remaining methyl and phenyl groups are in 'axial' positions. The novelty of this structure is that the Si—S heterocycle is neither in the chair nor in the boat but in the twist-boat conformation, one of the few to be observed in the solid state. This result leads us to presume that for Si—S heterocycles the energy of the twist-boat conformation is not that different from the energies of either the chair or the boat conformations.
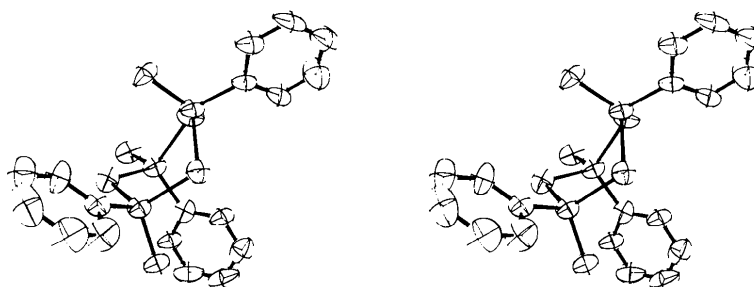
Fig. 1. Stereoscopic view of the molecule.

Torsion angles

The torsion angles of this compound are compared, in Table 3, with those calculated for ideal boat and chair conformations. These ideal geometries assume that all ring bonds are equivalent and that all angles are tetrahedral. The chair values listed are for atom S(3) to be in the down position. Inspection of a molecular model revealed strong interactions between a methyl group and a S lone pair for both boat 1 and boat 2 conformations. It is therefore reasonable for the structure to adopt the observed intermediate conformation. The twist-boat conformation of trans-$[CH_3C_6H_5SiS]_3$ is roughly $\frac{1}{3}$ from one and $\frac{2}{3}$ from the other boat conformation since the torsion angles of interest vary from 0 to 60° as the molecule passes from one boat form through the twist-boat to the other boat conformation.

Bond distances and angles

The interatomic bond distances and angles are schematically summarized in Fig. 2. The standard

deviations of these quantities derived from the calculated e.s.d.'s of the fractional coordinates, uncorrected for the block-diagonal approximation, amount to 0·003 Å and 0·2° for bond distances and angles,
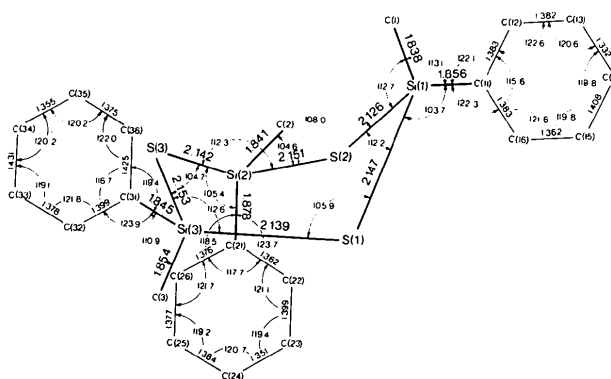

Fig. 2. Schematic view of the molecule showing the bond distances and angles.

Table 3. Torsion angles (°)

Endocyclic torsion angles compared with those of standard conformations

Angle	Chair	Boat 1	This work	Twist	Boat 2
			Conformations		
Si(3)—S(1)—Si(1)—S(2)	60	60	41·3 (2)	33·2	0
Si(2)—S(2)—Si(1)—S(1)	−60	−60	−68·2 (2)	−70·6	−60
Si(1)—S(2)—Si(2)—S(3)	60	0	20·1 (2)	33·2	60
Si(3)—S(3)—Si(2)—S(2)	−60	60	43·1 (2)	33·2	0
Si(2)—S(3)—Si(3)—S(1)	60	−60	−72·3 (2)	−70·6	−60
Si(1)—S(1)—Si(3)—S(3)	−60	0	27·8 (2)	33·2	60

Exocyclic torsion angles

Si(1)—S(1)—Si(3)—C(3)	141·9 (3)		Si(2)—S(2)—Si(1)—C(1)	57·2 (3)
Si(1)—S(1)—Si(3)—C(31)	−97·6 (3)		Si(2)—S(2)—Si(1)—C(11)	−179·8 (3)
Si(3)—S(1)—Si(1)—C(1)	−85·3 (3)		Si(2)—S(3)—Si(3)—C(3)	172·9 (3)
Si(3)—S(1)—Si(1)—C(11)	153·3 (3)		Si(2)—S(3)—Si(3)—C(31)	52·9 (3)
Si(1)—S(2)—Si(2)—C(2)	143·3 (3)		Si(3)—S(3)—Si(2)—C(2)	−75·1 (3)
Si(1)—S(2)—Si(2)—C(21)	−97·0 (3)		Si(3)—S(3)—Si(2)—C(21)	162·8 (3)

Table 4. *Comparison of bond distances* (Å) *and angles* (°) *in* Si—S *cycles*

	This work	Bart & Daly (1968)	Yokoi *et al.* (1955)*		Büssem *et al.* (1935)
Si—S	2·143 (3)	2·128 (2)	2·15 (3)	2·18 (3)	2·14
Si—C	1·852 (8)	1·854 (5)			
S—Si—S	112·4 (2)	111·8 (1)	115	105†	100†
Si—S—Si	106·2 (2)	104·5 (1)	110	75†	80†
S—Si—C	104·8, 111·4 (3)	107·0 (2)			

* Electron diffraction in the vapour state at 200°C.
† Four-membered rings.

Table 5. S—Si—C *bond angles in trimethyltriphenyl-cyclotrisilthiane* (°)

All these angles haves e.s.d.'s of 0·3°.

S(1)—Si(1)—C(1)	110·4	S(1)—Si(1)—C(11)	103·7
S(2)—Si(1)—C(1)	112·7	S(2)—Si(1)—C(11)	104·3
S(2)—Si(2)—C(2)	104·6	S(2)—Si(2)—C(21)	110·0
S(3)—Si(2)—C(2)	113·3	S(3)—Si(2)—C(21)	105·3
S(3)—Si(3)—C(3)	104·9	S(3)—Si(3)—C(31)	111·3
S(1)—Si(3)—C(3)	106·0	S(1)—Si(3)—C(31)	110·8

respectively, between S and Si atoms; they are of the order of 0·013 Å and 0·9° when C atoms are involved.

The Si—S distances, the S—Si—S and Si—S—Si bond angles are compared in Table 4 with the corresponding values in tetra(methylsilicon) hexasulphide (Bart & Daly, 1968), tetramethylcyclodisilthiane and hexamethylcyclotrisilthiane (Yokoi, Nomura & Yamasaki, 1955). The values in $(SiS_2)_n$ (Büssem, Fischer & Gruner, 1935) have also been included. The Si—C distances average 1·852 (8) Å and range from 1·838 to 1·878 Å. Vidal (1975) listed distinct values for Si—C_{sp^2} (1·873 Å) and Si—C_{sp^3} (1·885 Å). In our case these distances are indistinguishable. The average C_{sp^2}—C_{sp^3} bond distance is 1·382 (12) Å, in good agreement with previous averages (Brisse & Sygusch, 1974).

Each Si atom has approximately tetrahedral geometry. When the S—Si—C angles are considered, it is found that the angles are grouped around two mean values of 104·8 (3) and 111·4 (3)°. There is an equal number of angles in each group and the values range from 103·7 to 106·0° in one group and from 110·0 to

113·3° in the other. These angles, which could not all be shown on Fig. 2, are listed in Table 5. The reason for these angular differences is not clearly resolved, although intramolecular interactions can explain some of them.

Mean planes

The least-squares planes have been calculated for each of the phenyl groups and are listed in Table 6. The C atoms deviate by less than two e.s.d.'s from their respective planes. However, the Si atoms are significantly outside the phenyl mean planes, which may indicate some intermolecular or packing interactions.

Table 6. *Least-squares planes for the phenyl groups*

Phenyl group	$n = 1$	2	3
χ^2	0·04	5·68	6·13
Atom		Deviation from plane (Å)	
C(n1)	−0·001 (7)	0·013 (7)	0·007 (7)
C(n2)	0·000 (8)	−0·004 (9)	−0·011 (8)
C(n3)	0·001 (10)	−0·005 (10)	0·011 (9)
C(n4)	0·000 (9)	0·006 (11)	−0·008 (9)
C(n5)	0·001 (8)	0·003 (11)	0·005 (9)
C(n6)	0·001 (8)	−0·013 (10)	−0·005 (8)
Si(n)*	−0·035 (2)	−0·038 (2)	0·073 (2)

Equations for the least-squares planes

Plane 1 $0·0943X + 0·5615Y + 0·8221Z + 0·7926 = 0$
Plane 2 $0·6699X − 0·7020Y − 0·2417Z + 1·7506 = 0$
Plane 3 $−0·4261X + 0·1819Y − 0·8862Z − 5·0495 = 0$

* Atom not included in the least-squares plane calculations.
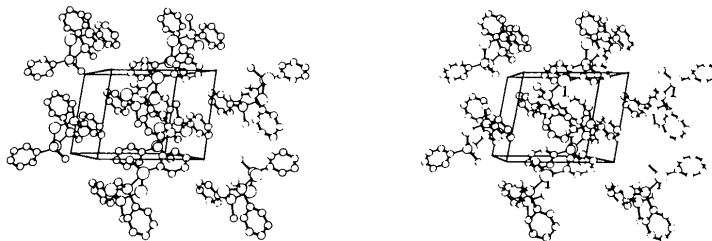

Fig. 3. Molecular packing.

Packing of the molecules

A view of the packing of the molecules in one unit cell is shown in Fig. 3. The molecules are packed in such a way that the phenyl group on Si(3) is facing its centrosymmetric counterpart at a distance of 3·50 Å. The axial phenyl and the methyl groups on Si(1) are opposite to the corresponding methyl and phenyl groups of the centrosymmetric molecule. There is only one short contact between heavy atoms. The distance between the S(2) atoms at positions (x,y,z) and $(1 - x, 1 - y, -z)$ is only 3·860 (3) Å. The shortest intermolecular distances are all reasonable. For S—C distances the minimum value is 3·89 Å while it is 4·20 Å for Si—C distances. The shortest intermolecular C—C distances are of the order of 3·64 Å. That there are no close contacts appears to be because of the flexibility of the heterocycle which allows for the observed deviations from an ideal structure.

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