

Studies of stannylated-1,2-dithiolato compounds: Synthesis and crystal structure of co-crystalline 4,5-bis[(triphenylstannyl)methylthiolato]-1,2-dithiole-3-thione (Ph_3SnCH_2)₂(dmt) and 5-[(triphenylstannyl)methylthiolato]-4- [(triphenylstannyl)thiolato]-1,2-dithiole-3-thione (Ph_3SnCH_2)(Ph_3Sn)(dmt) [72:28]

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Abstract

The synthesis of 4,5-bis[(triphenylstannyl)methylthiolato]-1,2-dithiole-3-thione in admixture with 4-[(triphenylstannyl)thiolato]-5-[(triphenylstannyl)methylthiolato]-1,2-dithiole-3-thione is reported from the reaction between $\text{Ph}_3\text{SnCH}_2\text{I}$ and caesium 4,5-dithiolato-1,2-dithiole-3-thione. X-ray structure determinations at 120 and 298 K reveal that the compounds co-crystallise to form solid solution species, displaying 4-coordinate, distorted tetrahedral tin centres in both components. Crystallisation results in the spontaneous resolution of enantiomeric forms of both components.

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1. Introduction

We have reported the synthesis and characterisation of various tin complexes of 4,5-dithiolato-1,3-dithiole-2-thione, $[\text{C}_3\text{S}_5]^{2-}$, dmit²⁻, (**1**), and 4,5-dithiolato-1,3-dithiole-2-one, $[\text{C}_3\text{S}_4\text{O}]^{2-}$, dmio²⁻, also known as dmio²⁻, (**2**) [1–6], including [(Ph_3SnCH_2)₂(dmit)] (**3**) [6], [(Ph_3SnCH_2)₂(dmio)] (**4**) [6], [(Ph_3Sn)₂(dmit)] (**5**) [1] and [(Ph_3Sn)₂(dmio)] (**6**) [1],

see Fig. 1. A large number of studies of dmit compounds have been reported, a smaller number for dmio compounds [7] and fewer still for the compounds of the unsymmetrical isomer of dmit²⁻, 4,5-dithiolato-1,2-dithiole-3-thione, dmt²⁻ (**7**), the latter being available on rearrangement of suitable dmit species (e.g., [8,9]).

We have carried out the reaction of $\text{Ph}_3\text{SnCH}_2\text{I}$ with caesium 4,5-dithiolato-1,2-dithiole-3-thione, Cs_2dmt . As we now report, the compound, obtained after work-up and recrystallisation, was a co-crystalline mixture of 4,5-bis[(triphenylstannyl)methylthiolato]-1,2-dithiole-3-thione (Ph_3SnCH_2)₂(dmt) (**8**) and 4-(triphenylstannylmethyl)thiolato-5-(triphenylstannyl)thiolato-1,2-dithiole-3-thione, (Ph_3SnCH_2)(Ph_3Sn)(dmt) (**9**). This was particularly

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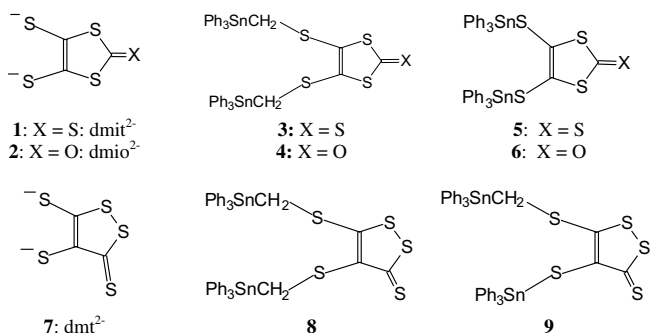


Fig. 1. Species mentioned in this article.

intriguing since the same batch of $\text{Ph}_3\text{SnCH}_2\text{I}$ had been used in the preparations of **3** and **4** and no formation of a $\text{Ph}_3\text{Sn-S}$ compound had been indicated in those syntheses.

2. Experimental

2.1. Synthesis

All the synthetic procedures were carried out under argon atmospheres using standard Schlenk techniques with solvents pre-dried prior use. Compounds, $\text{Ph}_3\text{SnCH}_2\text{I}$ [10] and $(\text{PhCO})_2\text{dmt}$ [8], were obtained by published procedures: Cs_2dmt was obtained from $(\text{PhCO})_2\text{dmt}$, by a similar procedure used to obtain Cs_2dmit from $(\text{PhCO})_2\text{dmit}$ [8]. The labile salt, Cs_2dmt , was stored under argon to avoid oxidation. Infrared spectra were obtained in CsI discs using a Nicolet Magna 760 FT-IR instrument. NMR spectra were run on Varian 300 MHz or Bruker 400 MHz Instruments. Melting points were measured on a Melt-TempII. Elemental analyses were obtained using a Perkin–Elmer 2400 apparatus.

2.2. Reaction between $\text{Ph}_3\text{SnCH}_2\text{I}$ and Cs_2dmt

(Iodomethyl)triphenylstannane (0.758 g, 1.55 mmol), was added under argon to a solution of Cs_2dmt (0.356 g, 0.77 mmol), prepared from $(\text{PhCO})_2\text{dmt}$ and CsOH . The red reaction mixture was stirred for 12 h, in which time the solution colour changed from red to yellow. The reaction mixture was filtered and the precipitate collected and recrystallized from chloroform/methanol. M.p. 158–159 °C.

¹H NMR (300 MHz, CDCl_3): δ : 2.82 (s, 2H, $J^{19,117}\text{Sn-}^1\text{H} = 47.0, 45.0$ Hz), 2.89 (s, 1.5H, $J^{19,117}\text{Sn-}^1\text{H} = 43.2, 41.2$ Hz), 7.50–7.70 (m, 30H, Ph).

¹³C (75 MHz, CDCl_3): δ : 10.5 (CH_2), 10.7 (CH_2), 128.9 ($^{19,117}\text{Sn-}^{13}\text{C} = 54$ Hz, *m*-Ph), 129.8 (*p*-Ph), 135.5 ($J^{19,117}\text{Sn-}^{13}\text{C} = 557, 530$ Hz, *i*-Ph), 136.7 ($^{19,117}\text{Sn-}^{13}\text{C} = 39$ Hz, *o*-Ph), 137.3 (C=C), 185.2 (C=C), 208.9 (C=S).

¹¹⁹Sn NMR (113 MHz, CDCl_3): δ : -115.6, -115.9, -120.2.

2.3. X-ray crystallography

2.3.1. Data collection

Intensity data were obtained with Mo $\text{K}\alpha$ radiation on two different sample crystals, one at 298 K and the other at 120 K. At 298 K, the data were obtained on a Bruker P4/Bruker-Nonius Kappa-CCD diffractometer under the control of XSCANS [11] and, for data reduction, SAINT [12] software. In this case no correction for absorption was carried out. At 120 K the data were obtained by means of the Enraf Nonius KappaCCD area detector diffractometer of the EPSRC National crystallographic service at the University of Southampton, UK. Now data collection was carried out under the control of the program COLLECT [13] and data reduction and unit cell refinement were achieved with the COLLECT and DENZO [14] programs. Correction for absorption, by comparison of the intensities of equivalent reflections, was applied using the program SADABS [15]. The program ORTEP-3 for Windows [16] was used in the preparation of the figures and SHELXL-97 [17] and PLATON [18] in the calculation of molecular geometry.

2.3.2. Structure solution and refinement

Initially the structures at the different temperatures were treated independently. In both cases the initial partial solution, obtained by the heavy atom method as implemented in the program SHELXS-97 [17], was expanded and refined by means of the program SHELXL-97 [17]. It became apparent that the molecules in the two samples were enantiomers. Thus, in order to ensure complete consistency in the atom labelling scheme used in the two refinements, the refinement against the 298 K data was repeated with suitably transformed (x, y, z replaced by $x, 1 - y, z$) coordinates from the 120 K refinement as the starting model. In the later stages of both refinements, hydrogen atoms were placed in calculated positions with aryl and methylene hydrogen atoms at C–H distances of 0.93 and 0.97 Å, respectively, at 298 K and 0.95 and 0.99 Å, respectively, at 120 K and in all cases refined with a riding model with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. Finally, in both cases, complete refinement was achieved with the introduction into the structural models of a partially occupied site for a sulphur atom [S(6)] associated with partial omission of both the methylene group [C(6)] and sulphur atom S(4) (see Fig. 2). Thus S(6) indicates relocation of the sulphur atom S(4) when the methylene group [C(6)] is absent. In other words, the structural model now represents a solid solution between 4,5-bis[(triphenylstannyl)methylthiolato]-1,2-dithiole-3-thione, the major component (**8**), and 4-[(triphenylstannyl)thiolato]-5-[(triphenylstannyl)methylthiolato]-1,2-dithiole-3-thione, the minor component (**9**). The ratio of major to minor components was refined to values of 0.712(15):0.288(15) at 298 K and 0.718(9):0.282(9) at 120 K and are, within error, the same for both sample crystals, i.e., 0.72:0.28. It was the presence of a residual electron density peak of $\sim 3 \text{ e}/\text{\AA}^3$ in the refinement, without S(6), against the 120 K data (and $\sim 1 \text{ e}/\text{\AA}^3$ for the 298 K

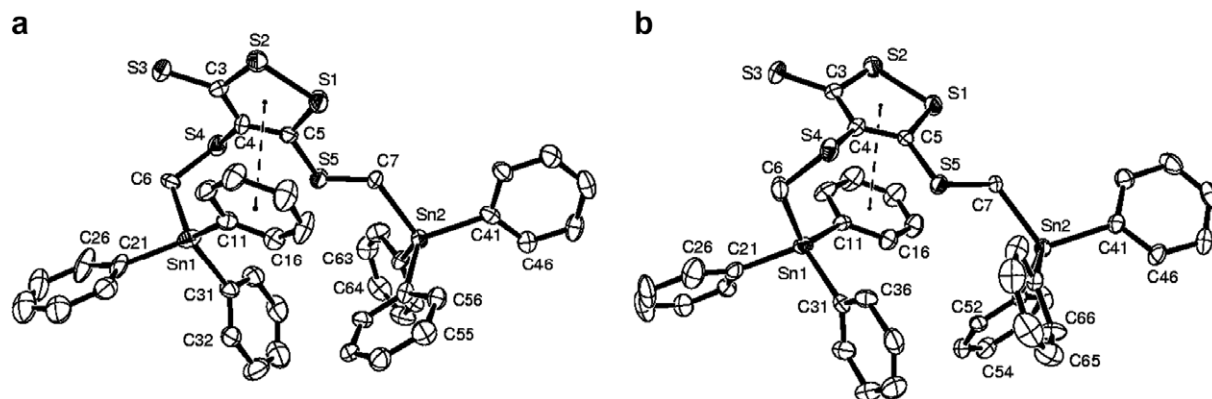


Fig. 2. Molecule of **8** (a) at 120 K and (b) at 298 K. Dashed lines join the centroids of rings R(1), defined as S(1)/S(2)/C(3)–C(5), and R(2), defined by C(11)–C(16). The mirror image relationship between the enantiomers is seen by comparison of (b), in which S(1) and S(2) of ring R(1) and ring R(2) lie below the plane of the paper, with (a) where these features lie above it. All hydrogen atoms have been omitted for clarity and only selected atoms of the benzene rings are labelled. Ellipsoids are shown at the 20% probability level in (b) and at the 50% probability level in (a).

data), which prompted the introduction of $S(6)$ into the structural models. In the refinement against the 120 K data, the Flack asymmetry parameter [19] was refined to take account of the partial racemic twinning of the sample crystal. Crystal data and structure refinement details are given in Table 1 and selected geometric parameters in Table 2. The residual electron density [$1.57 \text{ e}/\text{\AA}^3$] in the 120 K determination was sited close to tin.

3. Results and discussion

3.1. General

The product obtained after workup of the reaction between Cs_2dmt and $\text{Ph}_3\text{SnCH}_2\text{I}$ was shown by X-ray crystallography to be a co-crystallised mixture of 4,5-bis[(triphenylstannylmethyl)-thiolato]-1,2-dithiole-3-thione

Table 1
Crystal data and structure refinement

Compound	8:9 [0.712(15):0.288(15)]	8:9 [0.718(9):0.282(9)]
Empirical formula	$\text{C}_{40.712}\text{H}_{33.424}\text{S}_5\text{Sn}_2$	$\text{C}_{40.718}\text{H}_{33.436}\text{S}_5\text{Sn}_2$
Formula weight	920.33	920.41
Temperature (K)	298(2)	120(2)
Wavelength (Å)	0.71073	0.71073
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Orthorhombic, $P2_12_12_1$
Unit cell dimensions		
a (Å)	14.952(3)	14.7676(2)
b (Å)	15.375(3)	15.2218(2)
c (Å)	17.200(3)	17.0395(2)
Volume (Å ³)	3954.0(14)	3830.30(9)
Z , calculated density (Mg/m ³)	4, 1.546	4, 1.596
Absorption coefficient (mm ⁻¹)	1.555	1.605
$F(000)$	1831	1831
Crystal size (mm)	0.20 × 0.20 × 0.10	0.14 × 0.05 × 0.04
θ Range for data collection (°)	5.11–27.53	3.01–27.54
Index ranges	–18 ≤ h ≤ 19 –19 ≤ k ≤ 19 –20 ≤ l ≤ 22	–19 ≤ h ≤ 19 –19 ≤ k ≤ 19 –21 ≤ l ≤ 22
Reflections collected/unique [R_{int}]	33,600/8984 [0.1077]	78,638/8797 [0.0676]
Reflections observed [$I > 2\sigma(I)$]	4796	8107
Absorption correction	None	Semi-empirical from equivalents
Maximum and minimum transmission	Not applicable	0.9386 and 0.6984
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	8984/0/438	8797/0/439
Goodness-of-fit on F^2	1.098	1.269
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.063$, $wR_2 = 0.1010$	$R_1 = 0.054$, $wR_2 = 0.1185$
R indices (all data)	$R_1 = 0.1485$, $wR_2 = 0.1263$	$R_1 = 0.0608$, $wR_2 = 0.1204$
Absolute structure parameter	0.01(4)	0.10(4)
Largest difference in peak and hole ($\text{e}/\text{\AA}^3$)	0.63 and –0.723	0.82 and –1.57

Table 2
Selected geometric parameters, (Å, °) for the co-crystallised mixture of **8** and **9** at 298 K and 120 K

	298 K	120 K		298 K	120 K
<i>Both 8 and 9</i>					
Sn(1)–C(11)	2.132(10)	2.146(8)	C(21)–Sn(1)–C(11)	110.2(4)	110.7(3)
Sn(1)–C(21)	2.117(13)	2.132(9)	C(31)–Sn(1)–C(11)	109.5(4)	109.0(3)
Sn(1)–C(31)	2.107(10)	2.149(7)	C(31)–Sn(1)–C(21)	108.5(4)	109.5(3)
Sn(2)–C(7)	2.174(9)	2.174(7)	C(41)–Sn(2)–C(7)	106.0(4)	105.3(3)
Sn(2)–C(41)	2.137(9)	2.126(7)	C(51)–Sn(2)–C(7)	104.2(4)	102.9(3)
Sn(2)–C(51)	2.122(10)	2.128(7)	C(61)–Sn(2)–C(7)	111.8(4)	111.6(3)
Sn(2)–C(61)	2.134(10)	2.141(7)	C(51)–Sn(2)–C(41)	109.6(4)	109.3(3)
S(1)–C(5)	1.710(10)	1.716(8)	C(61)–Sn(2)–C(41)	113.5(4)	115.4(3)
S(1)–S(2)	2.055(4)	2.061(3)	C(61)–Sn(2)–C(51)	111.3(4)	111.4(3)
S(2)–C(3)	1.719(11)	1.713(8)	C(5)–S(1)–S(2)	94.5(4)	94.0(3)
C(3)–C(4)	1.435(13)	1.426(11)	C(3)–S(2)–S(1)	97.5(4)	97.2(3)
C(4)–C(5)	1.385(13)	1.366(10)	C(4)–C(3)–S(2)	113.1(8)	113.4(6)
C(3)–S(3)	1.639(11)	1.666(8)	C(5)–C(4)–C(3)	117.6(9)	118.0(7)
C(5)–S(5)	1.732(10)	1.725(7)	C(4)–C(5)–S(1)	117.2(7)	117.4(6)
S(5)–C(7)	1.769(10)	1.783(8)	S(2)–C(3)–S(3)	118.2(6)	117.5(5)
			C(4)–C(3)–S(3)	128.7(9)	129.1(6)
			C(4)–C(5)–S(5)	123.6(8)	123.8(6)
			S(1)–C(5)–S(5)	119.2(6)	118.7(5)
			C(5)–S(5)–C(7)	103.0(5)	102.4(4)
			S(5)–C(7)–Sn(2)	113.9(5)	114.3(4)
<i>8 only</i>					
Sn(1)–C(6)	2.079(16)	2.146(11)	<i>9 only</i>		
C(4)–S(4)	1.726(11)	1.748(8)	Sn(1)–S(6)	2.476(15)	2.534(8)
S(4)–C(6)	1.835(19)	1.804(11)	C(4)–S(6)	1.896(16)	1.884(10)
C(11)–Sn(1)–C(6)	115.8(6)	117.1(3)	C(11)–Sn(1)–S(6)	101.4(4)	101.3(2)
C(21)–Sn(1)–C(6)	98.3(6)	99.2(4)	C(21)–Sn(1)–S(6)	123.1(4)	123.6(3)
C(31)–Sn(1)–C(6)	113.8(6)	110.9(4)	C(31)–Sn(1)–S(6)	103.4(4)	101.9(3)
C(3)–C(4)–S(4)	123.8(8)	122.3(6)	C(3)–C(4)–S(6)	118.7(8)	117.9(6)
C(5)–C(4)–S(4)	117.7(8)	118.5(6)	C(5)–C(4)–S(6)	120.3(8)	121.3(6)
C(4)–S(4)–C(6)	100.3(8)	101.5(5)	C(4)–S(6)–Sn(1)	108.9(6)	106.4(4)
S(4)–C(6)–Sn(1)	116.6(8)	116.1(6)			

(Ph₃SnCH₂)₂(dmt) (**8**) and 4-(triphenylstannylmethyl)-thiolato-5-(triphenylstannyl)thiolato-1,2-dithiole-3-thione, (Ph₃SnCH₂)(Ph₃Sn)(dmt) (**9**), in a mole ratio of 0.715:0.285. The ¹¹⁹Sn NMR spectrum of the co-crystallised mixture in CDCl₃ solution exhibited signals at –115.6, –115.9, and –120.2 ppm. The former two values relate to tin in the Ph₃SnCH₂S groups and the –120.2 ppm to tin in the Ph₃SnS fragment.

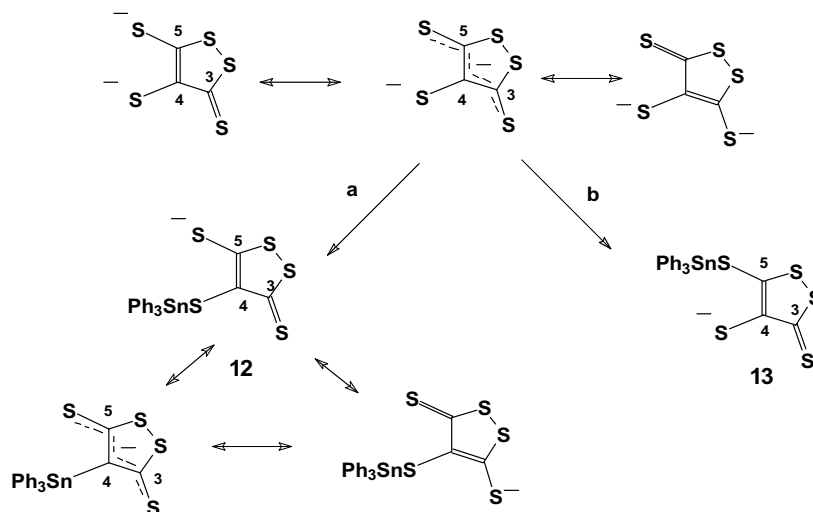
The preparation of **8/9** was carried out using the same batch of Ph₃SnCH₂I as used in the preparations of (Ph₃SnCH₂)₂dmit, **3**, and (Ph₃SnCH₂)₂dmio, **4**. While **8/9** were prepared using Cs₂dmt, **3** and **4** were obtained from the zincate compounds, [NEt₄]₂[Zn(dmit)₂] or [NEt₄]₂[Zn(dmio)₂]. The zincates are in general much milder reagents than the alkali–metal salts. Furthermore, Cs₂dmt is appreciable more reactive than either Cs₂dmit or Cs₂dmio.

The bulk sample of Ph₃SnCH₂I was found, by NMR spectroscopy, not to contain any Ph₃SnCl. This is significant since Ph₃SnCH₂I had been prepared from reaction of Ph₃SnCl with ICH₂ZnI, and a triphenyltin halide is an obvious precursor of the Ph₃Sn–S linked compound, **9**. A possible pathway to **9** is via a metal exchange reaction

between Ph₃SnCH₂I and Cs₂dmt, for example to give [CsCH₂I] (**10**) and Cs(Ph₃Sn)(dmt) (**11**) species. Compound **11** on reaction with Ph₃SnCH₂I would yield **9**. Compound **10** is considered to be a highly reactive and short lived carbenoid reagent, much more so than the lithium analogue. Metal exchanges between iodomethylstannanes and lithium compounds, to give iodomethyl–lithium carbenoid reagents, have been well utilised in synthesis [20]. Neither compound **10** nor products of CH₂ transfer from it were detected.

The ratio of Ph₃SnCH₂–S to Ph₃Sn–S links in the co-crystallised mixture **8/9** is 1.715:0.285 [ca. 6:1] indicates that the major reaction is the direct alkylation of dmt^{2–} by Ph₃SnCH₂I.

A further issue is the regiochemistry of **9**: the Ph₃Sn link to the dmt moiety occurs only at S-4. A restricted Hartree–Fock (RHF) calculation, with 6-31G(d) basis set on the resonance stabilized dmt^{2–} dianion indicates that S(4) bears a larger negative charge than S(3) and S(5) (–0.609 vs. –0.476). Thus S(5) will be the more reactive towards electrophilic reagents, and thus pathway a in Scheme 1 will be followed. An additional rationale for pathway a is that the species formed, **12**, is a resonance stabilized anion, which



Scheme 1.

is not be the case for **13**, a result of attack at S-3 or S-5 pathway b in Scheme 1.

3.2. Crystal structure

Structure determinations were carried out at two different temperatures, 120 and 298 K, using a different crystal for each determination. The atom arrangements and labelling schemes for **8** at 120 K and at 298 K are shown in Fig. 2, those for **9** at 120 K are shown in Fig. 3. The enantiomer of **9**, detected in the 298 K determination has not been drawn. The replacement of substituents between **8** [CH₂S] and **9** [S] occurs exclusively at the 4 position. All atoms, apart from C(6), S(4) and S(6), are in sites with full occupancy and are therefore fully ordered and common to both molecules. The distorted tetrahedral geometries at the tin centres in both components are clearly indicated by the data given in Table 2a. There are no interactions between tin centres in either component, nor are there any Sn–S intra- or intermolecular molecular interactions. However,

there are π – π stacking interactions between the essentially planar dmt and a phenyl ring in each of **8** and **9**.

The dmt ring overlaps with the phenyl ring, defined by C(11)–C(16), in both components, **8** and **9**, with a dihedral angle between the least-squares planes and the distance between the ring centroids of 12.5(6)° and 3.838 Å at 298 K [11.5(4)° and 3.798 Å, at 120 K], respectively. As there are two ways in which this ring overlap can be brought about, enantiomeric forms of each component in the solid state are formed. Both enantiomers must be present to the same extent in the bulk material, but clearly individual crystals are formed solely containing one enantiomer, i.e., spontaneous resolution has resulted on forming the crystalline state. The formations of enantiomers indicate that the formations of **8** and **9** must be derived from non-planar, non-symmetric intermediates. We suggest that intermediates, **12** and **13**, shown in Scheme 1, are indeed non-planar and that different enantiomers result from whether S(3) or S(5), in either intermediate, is attacked. Further details of the parameters of the intramolecular contacts are available in supplementary Table S1.

Evidence for delocalisation within the dmt ring is obtained from the C(3)–C(4) and C(4)–C(5) bond lengths of 1.435(13) and 1.385(13) Å at 298 K [1.426(11) and 1.366(10) Å at 120 K], respectively, as these values are between those expected for C–C and C=C bonds. Indications of resonance also are found in the C(3)–S(3) and C(4)–S(4) bonds of 1.639(11) and 1.726(11) Å, at 298 K [1.666(8) and 1.748(8) Å, at 120 K], respectively.

The structures of relatively few dmt complexes have been published: these include Me₂dmt, **13**, [21], [Bu₄N]₂[V(dmt)₃] (**14**) [22], [Bu₄N]₂[Ni(dmt)₂] (**15**) [8], [Bu₄N]₂[Cu(dmt)₄] (**16**) [23] and K₂dmt [24]. The geometric parameters within the dmt ring in **13**–**16** are similar to those found in **8** and **9**; the dmt²⁻ dianion in the ionic compound, K₂dmt, has exact C_{2v} symmetry, with intra-ring C–C bond length 1.420(7) Å.

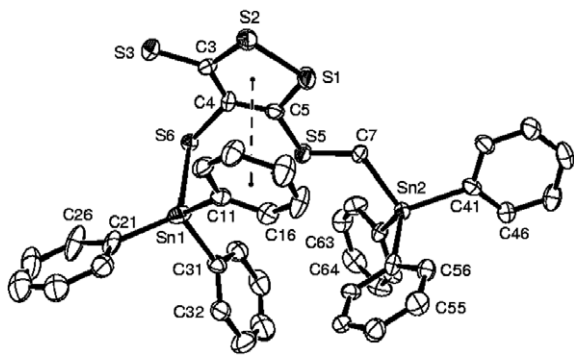


Fig. 3. Molecule of **9** at 120 K. Dashed lines join the centroids of rings R(1) and R(2). All hydrogen atoms have been omitted for clarity and only selected atoms of the benzene rings are labelled. Ellipsoids are shown at the 50% probability level.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.jorganchem.2007.12.010](https://doi.org/10.1016/j.jorganchem.2007.12.010).

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