

The Crystal Structures of $C_{33}H_{23}S_4I_3$ and $C_9H_7S_2I_3$; Triiodide Salts of Positively Charged Unsaturated Cyclic Disulfides

Asbjørn Hordvik, Knut Jynge and Lars K. Hansen

Department of Chemistry, Institute of Mathematical and Physical Sciences, University of Tromsø, Box 953, N-9001 Tromsø, Norway

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The crystal structures of $C_{33}H_{23}S_4I_3$ (**1**) and $C_9H_7S_2I_3$ (**2**) have been determined by X-ray crystallographic methods. There are two different I_3^- ions in crystals of **1**, both in two-fold positions. The I–I distances are 2.920(1) Å in one and 2.935(1) Å in the other. The corresponding I–I angles are 180.0° and 175.0(2)°, respectively. There are two unsaturated five-membered disulfide rings in the cation of **1**, each carrying one-half positive charge. The average S–S and C–S bond lengths in the rings are 2.048(5) Å and 1.726(12) Å. There are three S···I close contacts in the crystal structure, 3.760(4) Å, 3.796(3) Å, and 3.730(4) Å, respectively.

The I–I bond lengths in compound **2** are 2.876(6) Å and 2.973(5) Å, respectively, with I–I angle = 178.3(2)°. There are I···S close contacts in the crystal structure, in “linear” S–S···I···S–S arrangements [I···S = 3.75(2) Å, S–S···I = 157(1)°, S···I···S = 132(1)°] and in triangular $\begin{array}{c} \cdot \cdot S^- \\ | \\ I \cdot \cdot S^- \\ \cdot \cdot S^- \end{array}$ arrangements [I···S = 3.72(2) Å, S···I···S = 33(1)°].

Dedicated to Professor Olav Foss on his 70th birthday

Compound **1** of this structure study had been obtained as a by-product in a synthesis,¹ and we were asked to help find out what kind of a compound it was.

Compound **2** had been briefly looked at previously earlier by one of us (A.H.).² It was of interest because it had been formulated as a I_2^- salt, and possibly might contain I_4^{2-} ions in two-fold positions. However, several years elapsed before crystals suitable for intensity measurements were obtained.

Our study revealed interesting crystal structures. Both **1** and **2** proved to be triiodide salts of positively charged unsaturated cyclic disulfides. We hope that the present results may throw further light on the structure of such disulfides and the way in which they interact with triiodide ions.

Structure analysis

Compound 1. Crystals of compound **1** from chlorobenzene are thin rectangular needles; they are shiny red-brown in reflected light, and green in

transmitted light. The chlorobenzene solution is dark green.

Crystal data for compound 1. $C_{33}H_{23}S_4I_3$; M.W. = 928.50, space group $I2/c$ (No. 15), $a = 28.254(5)$, $b = 9.627(2)$, $c = 25.283(6)$ Å, $\beta = 105.50(2)^\circ$, $V = 6626.8$ Å³, $Z = 8$, $D_c = 1.861$ g cm⁻³, D_m (flotation) = 1.85 g cm⁻³, $\mu(\text{CuK}\alpha) = 251.1$ cm⁻¹.

The non-standard space group $I2/c$ was chosen because it gives a β angle of 105.5° as compared to that of 123.1° which the standard space group $C2/c$ (No. 15) would require.

A crystal of dimensions 0.083×0.60×0.012 mm was used for the X-ray analysis, which was carried out on a CAD4 diffractometer using graphite-monochromated $\text{CuK}\alpha$ radiation ($\lambda = 1.5418$ Å).

The intensities of 6838 independent reflections within $\theta = 75^\circ$ were measured at $t = 20^\circ\text{C}$ by the ω - 2θ technique. Reflections for which $I > 2\sigma(I)$, where $\sigma(I)$ is based on counting statistics, were regarded as observed. By this criterion 4062 re-

flections were considered observed. Lp and absorption corrections were applied.³

The composition (relative) of compound **1** was assumed to be $C_{25}H_{17}O_2I_2S_3$,¹ corresponding to a formula weight of 699 D, as compared to that of 1843 D calculated from the unit cell volume, the measured density, and $Z = 4$. This indicated a

molecular composition of $C_{66}H_{45}O_5I_5S_8$. MULTAN⁴ found all the atoms of the real molecule (or salt) twice, and a centre of symmetry was clearly indicated. Further calculations were therefore based on $I/2c$.

Compound **1** proved to be a triiodide. Oxygen atoms were not located. It should be noted in this

Table 1. Fractional atomic coordinates, and equivalent isotropic thermal parameters $U_{eq} = (U_{11} \cdot U_{22} \cdot U_{33})^{1/3}$ ($\times 10^2$) for iodine, sulphur and carbon in compound **1**.

Atom	x	y	z	$U_{eq}/\text{\AA}^2$
I(1)	0.25000	0.25000	0.75000	4.01(9)
I(2)	0.24228(3)	0.11438(9)	0.64474(4)	5.52(9)
I(11)	0.00000	0.40842(13)	0.25000	5.48(10)
I(12)	-0.06959(3)	0.42174(10)	0.14069(4)	6.29(9)
S(1)	0.0649(1)	0.9793(4)	0.2437(1)	5.7(3)
S(2)	0.0727(1)	1.0476(4)	0.3220(1)	6.2(3)
S(01)	0.1671(1)	0.2441(3)	0.1838(1)	4.3(2)
S(02)	0.2017(1)	0.1147(3)	0.2467(1)	4.3(2)
C(3)	0.1060(4)	0.9080(12)	0.3539(5)	4(1)
C(4)	0.1156(4)	0.8109(13)	0.3193(5)	4(1)
C(5)	0.0964(4)	0.8245(12)	0.2624(5)	4(1)
C(6)	0.0978(4)	0.7302(12)	0.2189(5)	4(1)
C(7)	0.1213(4)	0.6008(12)	0.2324(4)	4(1)
C(8)	0.1271(3)	0.4908(11)	0.1990(4)	3(1)
C(03)	0.1912(4)	0.2176(11)	0.2987(4)	4(1)
C(04)	0.1666(4)	0.3380(11)	0.2815(5)	4(1)
C(05)	0.1516(4)	0.3705(11)	0.2250(4)	4(1)
C(11)	0.1210(4)	0.9042(13)	0.4142(5)	5(1)
C(12)	0.1396(5)	0.7865(14)	0.4430(6)	5(1)
C(13)	0.1521(5)	0.7860(16)	0.4994(6)	6(1)
C(14)	0.1480(5)	0.9006(19)	0.5280(6)	8(2)
C(15)	0.1297(6)	1.0207(16)	0.5005(6)	7(2)
C(16)	0.1164(5)	1.0214(14)	0.4450(6)	6(1)
C(21)	0.0692(4)	0.7771(12)	0.1625(4)	4(1)
C(22)	0.0844(4)	0.8906(14)	0.1370(5)	5(1)
C(23)	0.0532(6)	0.9434(14)	0.0891(6)	7(1)
C(24)	0.0095(5)	0.8794(18)	0.0662(6)	7(1)
C(25)	-0.0051(4)	0.7644(16)	0.0894(6)	6(1)
C(26)	0.0241(4)	0.7155(12)	0.1376(5)	4(1)
C(31)	0.1101(4)	0.4911(12)	0.1376(4)	4(1)
C(32)	0.1326(4)	0.5747(13)	0.1062(5)	6(1)
C(33)	0.1147(5)	0.5730(15)	0.0497(5)	7(1)
C(34)	0.0755(6)	0.4920(17)	0.0238(6)	7(1)
C(35)	0.0540(5)	0.4118(15)	0.0541(6)	6(1)
C(36)	0.0709(4)	0.4093(13)	0.1100(5)	5(1)
C(41)	0.2084(4)	0.1710(11)	0.3558(5)	3(1)
C(42)	0.2285(4)	0.0377(14)	0.3683(5)	5(1)
C(43)	0.2431(5)	-0.0079(15)	0.4221(6)	6(1)
C(44)	0.2402(5)	0.0792(15)	0.4642(5)	6(1)
C(45)	0.2220(5)	0.2082(15)	0.4523(5)	6(1)
C(46)	0.2064(4)	0.2573(13)	0.399505	5(1)

Table 2. Fractional atomic coordinates, and equivalent isotropic thermal parameters $U_{eq} = (U_{11} \cdot U_{22} \cdot U_{33})^{1/3}$ ($\times 10^2$) for iodine and sulfur, and isotropic thermal parameters U_{iso} ($\times 10^2$) for carbon in compound 2.

Atom	x	y	z	$U_{eq}/U_{iso}/\text{\AA}^2$
I(1)	0.0000	0.0298(1)	0.0000	5.3(3)
I(2)	0.0002(7)	0.1116(1)	0.3430(8)	3.6(2)
I(3)	-0.0003(7)	0.1932(2)	0.6658(8)	5.2(3)
S(1)	-0.1165(16)	0.5558(7)	-0.0538(22)	5(1)
S(2)	0.1143(16)	0.5564(6)	-0.0533(21)	5(1)
C(3)	0.136(7)	0.5951(24)	0.175(9)	5(2)
C(4)	-0.003(8)	0.6114(26)	0.261(10)	6(2)
C(5)	-0.138(8)	0.5948(29)	0.176(11)	6(2)
C(41)	-0.004(8)	0.6466(20)	0.463(9)	6(2)
C(42)	0.133(7)	0.6624(22)	0.554(8)	5(2)
C(43)	0.132(7)	0.6940(23)	0.737(9)	6(2)
C(44)	-0.005(11)	0.7098(36)	0.828(15)	7(2)
C(45)	-0.141(12)	0.6940(24)	0.737(9)	6(2)
C(46)	-0.140(12)	0.6624(25)	0.554(10)	5(2)

connection that the “missing” iodine and the “assumed” oxygens roughly balance with respect to formula weight.

The structure was refined by means of CRYLSQ of X-RAY-76⁵ to an R value of 0.05. The hydrogen positions were calculated, and hydrogen parameters were included in the structure factor calculations but were not refined. All calculations on compound 1 were carried out on the CYBER 171 computer at the University of Tromsø.

Compound 2. Crystals of compound 2 are thin orange needles and flakes. The space group for these crystals has been reported to be either $Imma$ or $Im2a$.² We chose to let the additional symmetry elements reveal themselves by carrying out the structure analysis in the monoclinic setting Ia .

Crystal data for compound 2. $C_9H_7S_2I_3$; M.W. = 559.99, space group Ia (No. 9), $a = 8.882(1)$, $b = 24.565(3)$, $c = 6.387(1)$ Å, $\beta = 90.02(2)^\circ$, $V = 1393.6$ Å³, $Z = 4$, $D_c = 2.669$ g cm⁻³, D_m (floatation) = 2.63 g cm⁻³, $\mu(\text{MoK}\alpha) = 70.5$ cm⁻¹.

A crystal of dimension 0.2×0.8×0.05 mm was used for the X-ray analysis on the CAD4 diffractometer, using graphite-monochromated MoK α radiation ($\lambda = 0.71069$ Å).

The intensities of 1225 independent reflections within $\theta = 25^\circ$ were measured at room temper-

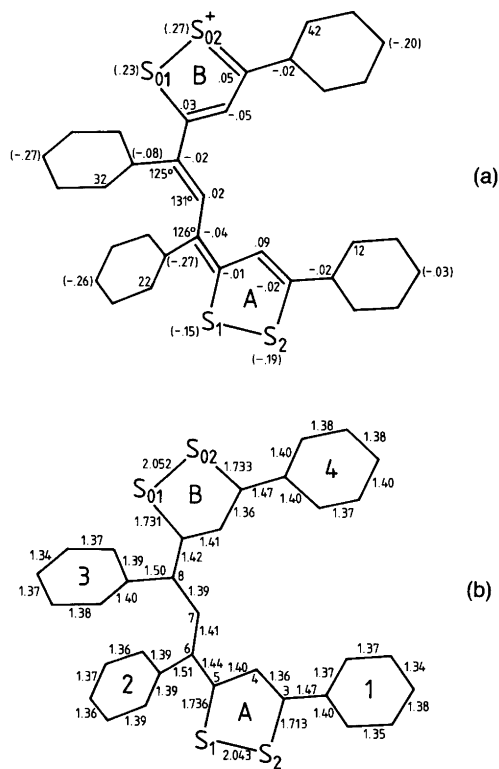


Fig. 1. The structure of the cation of compound 1, showing (a) deviations from a least-squares plane, and (b) bond lengths and designation of rings. The numbering of atoms is also indicated.

Table 3. Bond lengths (Å) and bond angles (°) in compound 1.

Bond lengths		Bond angles	
I(1)–I(2)	2.920(1)	I(2)–I(1)–I(2')	180.0
I(11)–I(12)	2.935(1)	I(12)–I(11)–I(12')	175.0(3)
S(1)–S(2)	2.043(5)	S(1)–S(2)–C(3)	96.1(8)
S(1)–C(5)	1.736(12)	S(2)–C(3)–C(11)	118.5(11)
S(2)–C(3)	1.713(11)	S(2)–C(3)–C(4)	114.7(10)
C(3)–C(4)	1.36(2)	C(4)–C(3)–C(11)	126.8(11)
C(3)–C(11)	1.47(2)	C(3)–C(4)–C(5)	120.5(11)
C(4)–C(5)	1.40(2)	C(4)–C(5)–S(1)	113.0(11)
C(5)–C(6)	1.44(2)	C(4)–C(5)–C(6)	130.2(11)
		S(1)–C(5)–C(6)	116.8(11)
		C(5)–S(1)–S(2)	95.5(9)
C(11)–C(12)	1.37(2)	C(3)–C(11)–C(12)	122.3(12)
C(12)–C(13)	1.37(2)	C(3)–C(11)–C(16)	120.9(12)
C(13)–C(14)	1.34(3)	C(16)–C(11)–C(12)	116.8(13)
C(14)–C(15)	1.38(2)	C(11)–C(12)–C(13)	120.6(14)
C(15)–C(16)	1.35(2)	C(12)–C(13)–C(14)	121.5(14)
C(16)–C(11)	1.40(2)	C(13)–C(14)–C(15)	119.5(14)
		C(14)–C(15)–C(16)	119.4(16)
		C(15)–C(16)–C(11)	122.2(13)
		C(5)–C(6)–C(21)	114.7(10)
C(5)–C(6)	1.44(2)	C(5)–C(6)–C(7)	118.8(12)
C(6)–C(7)	1.41(2)	C(7)–C(6)–C(21)	126.3(11)
C(6)–C(21)	1.51(2)	C(6)–C(21)–C(22)	121.3(10)
C(21)–C(22)	1.39(2)	C(6)–C(21)–C(26)	119.6(14)
C(22)–C(23)	1.39(2)	C(26)–C(21)–C(22)	118.8(12)
C(23)–C(24)	1.36(2)	C(21)–C(22)–C(23)	119.3(12)
C(24)–C(25)	1.37(2)	C(22)–C(23)–C(24)	119.8(16)
C(25)–C(26)	1.36(2)	C(23)–C(24)–C(25)	121.8(14)
C(26)–C(21)	1.39(2)	C(24)–C(25)–C(26)	118.9(12)
		C(25)–C(26)–C(21)	121.4(15)
		C(6)–C(7)–C(8)	130.6(11)
C(7)–C(8)	1.39(2)	C(7)–C(8)–C(05)	117.6(11)
C(8)–C(05)	1.42(2)	C(7)–C(8)–C(31)	124.8(10)
C(8)–C(31)	1.50(2)	C(05)–C(8)–C(31)	117.6(12)
C(31)–C(32)	1.40(2)	C(8)–C(31)–C(32)	121.4(11)
C(32)–C(33)	1.38(2)	C(8)–C(31)–C(36)	120.9(14)
C(33)–C(34)	1.37(2)	C(36)–C(31)–C(32)	117.7(11)
C(34)–C(35)	1.34(2)	C(31)–C(32)–C(33)	119.0(12)
C(35)–C(36)	1.37(2)	C(32)–C(33)–C(34)	121.6(17)
C(36)–C(31)	1.39(2)	C(33)–C(34)–C(35)	119.2(14)
		C(34)–C(35)–C(36)	121.0(14)
		C(35)–C(36)–C(31)	121.5(15)
S(01)–S(02)	2.052(4)	C(8)–C(05)–C(04)	128.8(12)
S(01)–C(05)	1.731(12)	C(8)–C(05)–S(01)	117.8(9)
S(02)–C(03)	1.733(12)	C(04)–C(05)–S(01)	113.4(9)
C(03)–C(04)	1.36(2)	C(05)–S(01)–S(02)	96.0(4)
C(04)–C(05)	1.41(2)	S(01)–S(02)–C(03)	95.6(5)
C(03)–C(41)	1.47(2)	S(02)–C(03)–C(04)	114.9(10)
		S(02)–C(03)–C(41)	119.6(8)
		C(04)–C(03)–C(41)	125.5(12)
		C(03)–C(04)–C(05)	120.0(12)

cont'd

Table 3. (contd)

Bond lengths		Bond angles	
C(41)–C(42)	1.40(2)	C(03)–C(41)–C(42)	120.6(12)
C(42)–C(43)	1.38(2)	C(03)–C(41)–C(46)	121.7(10)
C(43)–C(44)	1.38(2)	C(46)–C(41)–C(42)	117.8(12)
C(44)–C(45)	1.35(2)	C(41)–C(42)–C(43)	120.5(13)
C(45)–C(46)	1.37(2)	C(42)–C(43)–C(44)	120.3(13)
C(46)–C(41)	1.40(2)	C(43)–C(44)–C(45)	119.1(13)
		C(44)–C(45)–C(46)	122.7(13)
		C(45)–C(46)–C(41)	119.6(12)

ature by the ω -2 θ technique. 991 of these with intensity values greater than $2\sigma(I)$ were regarded as observed. Lp and absorption corrections were applied.³

The structure was solved by the use of Patterson methods and refined using CRYLSQ of X-RAY-76⁵ to an R value of 0.05. The phenyl group was assigned an idealised structure during last stages of refinement, and its atomic parameters were then not refined. The structure analysis showed that compound **2**, like **1**, is a triiodide, and that the space group should be $Im2a$ (No. 46); the b and c axes given above then have to be interchanged (cf. Fig. 3). Structure refinement in the orthorhombic space group was considered unnecessary. The results give an adequate description of the crystal structure and of the ionic interactions, which for us was the major point of interest.

All calculations on **2** were carried out on the

UNIVAC 1110 computer at the University of Bergen.

Final atomic coordinates for I, S, and C atoms in compounds **1** and **2** are given in Tables 1 and 2, respectively. Hydrogen positions, anisotropic temperature parameters and structure factor lists are available from one of the authors (A.H.) on request. Scattering factors given by Cromer and Mann⁶ were used for I, S, and C. For hydrogen, the scattering factor curve given by Stewart *et al.*⁷ was used.

Discussion

Compound **1** was found to be a triiodide salt of the cation whose structure is shown in Fig. 1. In the crystal structure there are two slightly different triiodide ions, both in two-fold positions. One of them has its central iodine atom on a two-fold axis, and the other has its central iodine in a

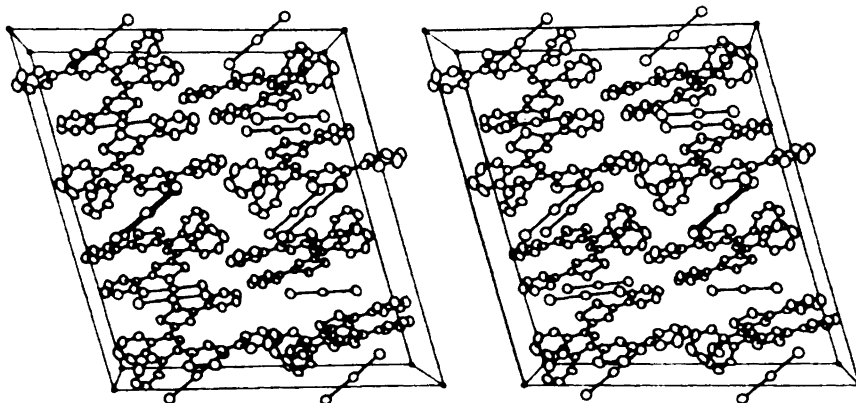


Fig. 2. A stereoscopic view of the crystal structure of compound **1** along the b axis.

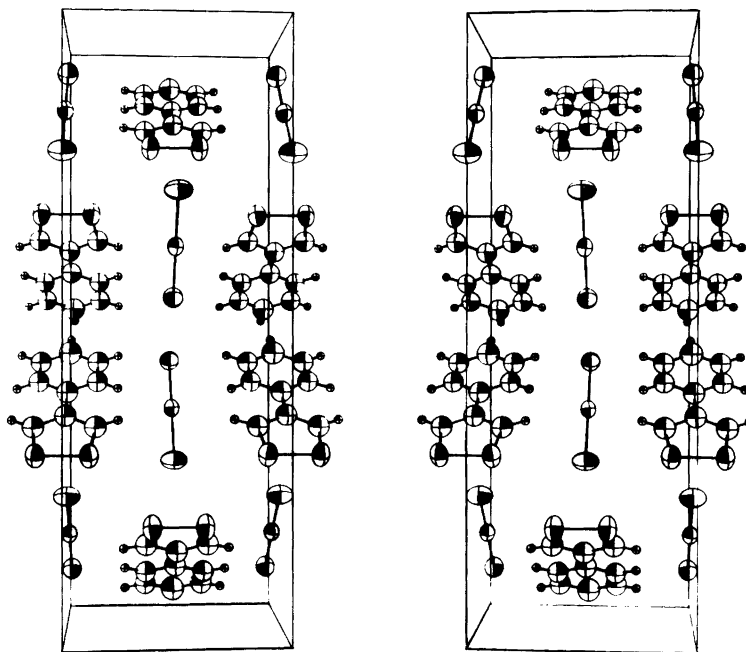


Fig. 3. A stereoscopic view of the crystal structure of compound **2** along the *c* axis.

centre of symmetry (cf. Fig. 2). Thus, half of either of these triiodide ions belongs to the asymmetric unit.

The I–I bond lengths in the “centrosymmetric” triiodide ion are 2.920(1) Å, and the I–I–I bond angle is 180°. In the other I_3^- ion the I–I bond lengths are 2.935(1) Å and the I–I–I angle 175.0(3)°. These values agree with reported values for triiodide ions.^{8–11}

Fig. 1a illustrates the geometry of the cation. Deviations of atoms from a least-squares plane are given. This plane has been calculated with unit weights, and comprises the atoms for which the deviations are given without parentheses. The planes of rings 2 and 3 both form an angle of 70.4° with this plane, and the corresponding angles for the planes of the other rings are 6.0 for ring 1, 4.0 for ring 4, 6.0 for ring A, and 7.3° for ring B. Each of the six rings is almost exactly planar.

The phenyl rings 2 and 3 are rather close and may repel each other to some degree, as indicated by the three bond angle values given in the figure.

Bond lengths and angles in compound **1** are

given in Table 3, and the bond lengths in the cation are also shown in Fig. 1b. The symmetry with respect to corresponding bond lengths in the two halves of the ion should be noted.

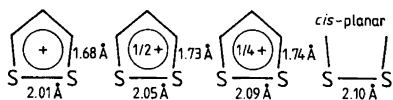
The symmetry with respect to bond lengths makes it tempting to look at the cation as being equivalent to a cyanine dye (see Ref. 12 and references cited therein). This implies that the positive charge is equally distributed between the two halves of the ion, and that the two unsaturated five-membered cyclic disulfides, as a rough estimate, carry one-half positive charge each. This in turn may probably explain why the S–S bonds in the present compound [average length 2.048(5) Å] are found to be somewhat longer than the S–S bonds in 1,2-dithiolylium ions; e.g. S–S = 2.004(5) Å in 4-phenyl-1,2-dithiolylium thiocyanate¹³ and S–S = 2.021(4) Å in 4-phenyl-1,2-dithiolylium chloride monohydrate.¹⁴ Furthermore, the average C–S bond length in the present compound, 1.726(12) Å, is greater than that [1.676(9) Å] for the two mentioned 1,2-dithiolylium ions.

The 1,2-dithiolylium ion has a sextet of π -electrons and is aromatic.¹⁵ When electrons are add-

ed to such a system, the population of antibonding π -orbitals may increase, thus weakening the π -bonding. This is in accordance with longer S-S and C-S bonds in the present compound than in the 1,2-dithiolium ions.

One might possibly extend this line of reasoning by looking at an equivalent disulphide ion carrying less than one-half positive charge. The 2:3 complex of tetrathiotetracene and iodine, $\text{TTT}_2^+ \cdot \text{I}_3^-$ may be such a compound.¹⁶ TTT_2^+ is described as a cation-radical which carries one positive charge and contains four unsaturated five-membered disulfide rings. This may leave each ring with roughly one quarter of a positive charge, and one would therefore expect that the S-S and C-S bonds should be longer than those in the present compound. In agreement with this, the average S-S and C-S bond lengths in TTT_2^+ are 2.087(4) Å and 1.735(8) Å, respectively.

One may conclude that the above indicates a clear trend, which, when the proposed bond length, 2.10 Å, for the S-S bond in a *cis* planar disulfide group is also taken into account,^{17,18} may be summarized as shown in Scheme 1. A stereo-



Scheme 1.

scopic view of the crystal structure of compound **1** is given in Fig. 2. It should be noted that the two different triiodide ions approach the cation rather similarly from opposite sides. The centrosymmetric I_3^- ion forms an angle of 26.6° with the *a-c* plane.

There are three distances between sulfur and iodine which are shorter than the corresponding sum of van der Waals radii, i.e. 3.85–4.00 Å;¹⁹ these distances are: $\text{S}(02) \cdots \text{I}(1) = 3.760(4)$, $\text{S}(02) \cdots \text{I}(2) = 3.796(3)$ and $\text{S}(2) \cdots \text{I}(12) = 3.730(4)$ Å. Note that S(02) and S(2) are symmetrically positioned in the cation (cf. Fig. 1). S(02) approaches the centrosymmetric triiodide ion in a triangular arrangement, and although S(2) approaches the other triiodide ion in a similar way, only one of the $\text{S}(2) \cdots \text{I}$ contacts there is shorter than the corresponding van der Waals distance.

The crystal structure of compound **2** is shown in Fig. 3. Standard deviations in atomic parameters for the C and S atoms are rather poor (cf. Table 2), but there is no indication that the di-

mensions of the cation are different from those quoted above for the thiocyanate and the chloride monohydrate.^{13,14}

The two I-I bond lengths in the triiodide ion of **2** are different, viz. $\text{I}(1)-\text{I}(2) = 2.973(5)$ and $\text{I}(2)-\text{I}(3) = 2.876(6)$ Å, and the $\text{I}(1)-\text{I}(2)-\text{I}(3)$ angle is 178.3(2)°. I(1) participates in two close contacts in the crystal structure (cf. Fig. 3). It forms a

triangular $\text{I} \cdots \begin{array}{c} \text{S}^- \\ | \\ \text{S}^- \end{array}$ arrangement with the disulfide

group of one cation, and approaches the plane of the disulfide ring of another at a distance of 3.72(2) Å. The $\text{I} \cdots \text{S}$ distances in the triangular arrangement are 3.70(2) Å and the $\text{S} \cdots \text{I} \cdots \text{S}$ angle is 32(1)°. There is a similar arrangement in the crystal structure of 4-phenyl-1,2-dithiolium iodide, where the $\text{I} \cdots \text{S}$ distances are 3.55(1) Å and the $\text{S} \cdots \text{I} \cdots \text{S}$ angle is 33(1)°.²⁰

The central iodine atom of the present structure participates in close contacts with sulfur in a "linear" $\text{S} \cdots \text{I} \cdots \text{S} \cdots \text{S} \cdots \text{S}$ arrangement. The $\text{S} \cdots \text{I}$ distances there are 3.75(2) Å, the $\text{S}-\text{S} \cdots \text{I}$ angle 157(1)°, and the $\text{S} \cdots \text{I} \cdots \text{S}$ angle 132(1)°. Equivalent dimensions in a similar arrangement in the mentioned iodide salt are 3.50(1) Å, 158(1)° and 136(1)°, respectively.

It is interesting to note that the $\text{I} \cdots \text{S}$ contacts are shorter in the iodide where iodine carries more negative charge. This supports the assumption that partial bonding between iodine and sulfur may be established through partial transfer of charge from iodine to sulfur in these compounds.²⁰

Since I(1) participates in two close contacts and I(3) in only a rather weak contact with I(3) of a neighbouring ion [$\text{I} \cdots \text{I} = 4.24(2)$ Å], one might expect I(1)–I(2) to be a longer bond than I(2)–I(3).²¹

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