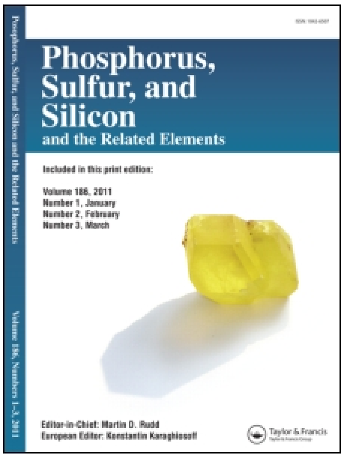


Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

SELFSTACKING SYSTEMS, PART 6.[1] HOST LATTICE FUNCTION OF 2,3,8,9-TETRAMETHOXYDIBENZO[*c,e*][1,2]-DICHALCOGENINS IN THEIR ELECTRICALLY CONDUCTING IODINE COMPLEXES

Joachim Behrens[a]; Winfried Hinrichs[b]; Thorsten Link[a]; Christian Schiffling[a]; Günter Klar[a]
[a] Institut für Anorganische und Angewandte Chemie der Universität Hamburg, Hamburg, Germany [b] Institut für Kristallographie der Freien Universität Berlin, Berlin, Germany

To cite this Article Behrens, Joachim , Hinrichs, Winfried , Link, Thorsten , Schiffling, Christian and Klar, Günter(1995) 'SELFSTACKING SYSTEMS, PART 6.[1] HOST LATTICE FUNCTION OF 2,3,8,9-TETRAMETHOXYDIBENZO[*c,e*][1,2]-DICHALCOGENINS IN THEIR ELECTRICALLY CONDUCTING IODINE COMPLEXES', Phosphorus, Sulfur, and Silicon and the Related Elements, 101: 1, 235 — 244

To link to this Article: DOI: 10.1080/10426509508042522

URL: http://dx.doi.org/10.1080/10426509508042522

Phosphorus, Sulfur, and Silicon, 1995, Vol. 101, pp. 235–244
Reprints available directly from the publisher
Photocopying permitted by license only

SELFSTACKING SYSTEMS, PART 6.[1] HOST LATTICE FUNCTION OF 2,3,8,9-TETRAMETHOXYDIBENZO[*c*,*e*][1,2]-DICHALCOGENINS IN THEIR ELECTRICALLY CONDUCTING IODINE COMPLEXES

JOACHIM BEHRENS,† WINFRIED HINRICHS,‡ THORSTEN LINK,†
CHRISTIAN SCHIFFLING† and GÜNTER KLAR*·†

†Institut für Anorganische und Angewandte Chemie der Universität Hamburg, Martin-Luther-King-Platz 6, D-20146 Hamburg, Germany; ‡Institut für Kristallographie der Freien Universität Berlin, Takustraße 6, D-14195 Berlin, Germany

(Received October 25, 1994; in final form December 8, 1994)

The structures of 2,3,8,9-tetramethoxydibenzo[*c*,*e*][1,2]dithiin (*o*-S_2, **1**) and -diselenin (*o*-Se_2, **3**) have been determined by single-crystal X-ray diffraction methods. Both compounds have a columnar structure in which the orientation of the molecules alternates with respect to their dichalcogeno bridges. The same kind of stacks had been found before in the electrically conducting iodine complex [*o*-S_2]$I_{1.16}$ (**2**) with channels parallel to the stacks in which chains of disordered polyiodide ions are intercalated, *i.e.* the host lattice of **2** is performed in **1**. *o*-Se_2-(**3**) also forms an iodine complex of composition [*o*-Se_2]I_x (x = 1.5–2.5) (**4**) which is, however, thermally unstable loosing iodine already at room temperature. For reasons of comparison the X-ray structure of 3,3′,4,4′-tetramethoxybiphenyl (**5**) has also been determined.

Key words: 2,3,8,9-Tetramethoxydibenzo[*c*,*e*][1,2]dithiin and -diselenin X-ray structures, 3,3′4,4′-tetramethoxybiphenyl X-ray structure, iodine complexes, host lattices, polyiodide chains.

INTRODUCTION

2,3,8,9-Tetramethoxydibenzo[*c*,*e*][1,2]dithiin (a biphenyl derivative with a dithio bridge and two *ortho*-standing methoxy substituents at each phenyl ring, therefore abbreviated as *o*-S_2, **1**) reacts with elementary iodine to give a dark blue, electrically conducting complex of composition [*o*-S_2]$I_{1.16}$ (**2**).[2,3] This compound is characterized by stacks of partially oxidized *o*-S_2 units in an alternating orientation with respect to their dithio bridges and by disordered polyiodide ions in channels parallel to the stacks. **2** crystallizes from hot concentrated solution of the components, but is also formed when crystals of **1** are exposed to an atmosphere of iodine.

This solid state reaction suggests that the channels for the polyiodide ions are already preformed in the crystal structure of **1**. In order to prove this hypothesis, a structure determination of **1** was carried out. Furthermore, in order to enlarge the experimental basis, the investigations were extended to the corresponding diselenin *o*-Se_2 (**3**) and its iodine complex **4**.

---

*Author to whom all correspondence should be addressed.

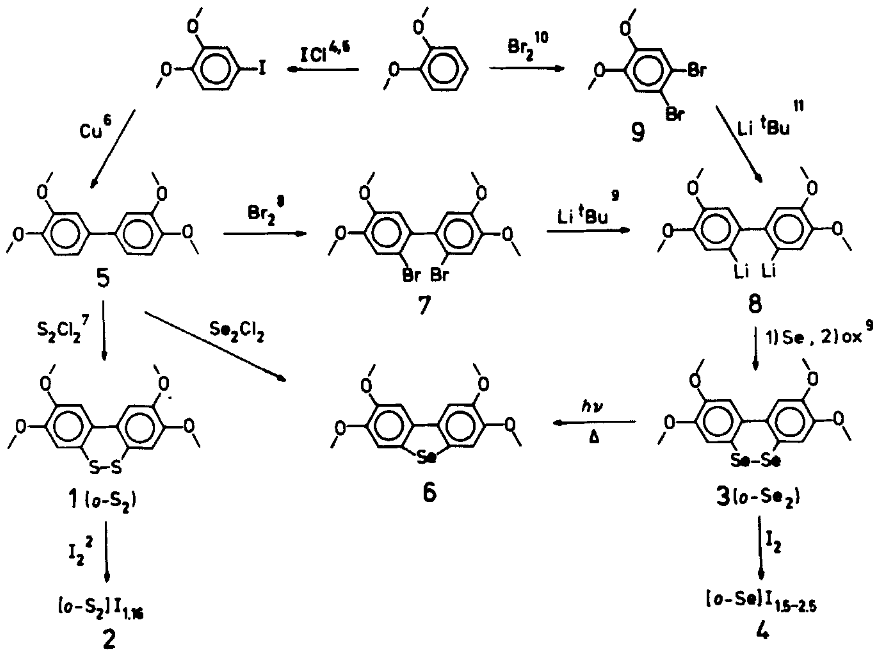
SYNTHESES OF COMPOUNDS

Although the syntheses of the dichalcogenins **1** and **3** have already been known—a synopsis is given in Scheme I—they could be improved by variation of the reaction conditions.

In contrast to *disulfur dichloride* which directly reacts with 3,3′,4,4′-tetramethoxybiphenyl (**5**) to the dithiin **1**,[7] *diselenium dichloride* only reacts on warming to give—by simultaneous separation of selenium—instead of the diselenin **3** the also known[9] 2,3,7,8-tetramethoxydibenzoselenophene (**6**):

Under an atmosphere of dry nitrogen diselenium dichloride (1 ml, 12.1 mmol) was added to the solution of 3,3′4,4′-tetramethoxybiphenyl (**5**) (3.3 g, 12.1 mmol) in glacial acid (100 ml). The mixture was heated to 120°C for 3 hours, then poured on ice and extracted with chloroform. The residue of the chloroform solution was crystallized from ethanol giving 1.6 g (38%) 2,3,7,8-tetramethoxyselenophene (**6**), mp. 180°C (lit.[9] 181°C).

The diselenin **3**, however, can be obtained according to Engman[9] when 2,2′-dibromo-4,4′,5,5′tetramethoxybiphenyl (**7**) is lithiated by lithium *tert.*-butyl, the dilithio compound **8** thereby formed is reacted with selenium and the selenium insertion product is finally oxidized by air. By lowering the temperature during the lithiation and subsequent addition of selenium the yield could be raised from 4 to 16%. The synthesis can also be carried out in form of a one-pot-reaction by producing the dilithio compound **8** from the easily accessible 1,2-dibromo-4,5-dimethoxybenzene (**9**) and lithium *tert.*-butyl in 2:3 molar ratio:



SCHEME I

Under an atmosphere of dry nitrogen a 1.6 M solution of lithium *tert.*-butyl in hexane (1.6 M, 9.4 ml, 15 mmol) was added at −40°C to the solution of 1,2-dibromo-4,5-dimethoxybenzene (**9**) (3.0 g, 10 mmol) in tetrahydrofurane (THF) (50 ml). The mixture was allowed to reach room temperature within 1 hour, was again cooled to −30°C and dry, powdered selenium (0.8 g, 10 mmol) was added. The mixture was then stirred without any further cooling for 4 hours, non-reacted selenium was filtered off and the solution refluxed for 5 hours at the air. The residue of the THF solution was chromatographed on neutral aluminium oxide, 2,3,8,9-tetramethoxydibenzo[*c*,*e*][1,2]diselenin (**3**) being eluated by chloroform. Yield 235 mg (11%), mp. 206°C (lit.[9] 208°C) after crystallization from chloroform/petroleum ether.

The diselenin **3** decomposes to selenium and the selenophene **6** on exposition to light or standing at room temperature for a longer time.

Black lustrous iodine complexes **4** precipitate when solutions of **3** in acetonitril (ca. 0.15 mmol per 30 ml) and iodine (molar ratio 1:2) are boiled for a short time and then cooled to room temperature. These complexes slowly liberate iodine on standing thus leading to variable compositions $[o\text{-}Se_2]I_x$ (x = 1.5–2.5). Complexes **4** are also formed, when the crystals of **3** are exposed to an atmosphere of iodine at 80°C, however, the crystals obtained had not the quality required for crystal structure determination.

CRYSTAL STRUCTURE DETERMINATIONS

X-ray analyses were performed for the dichalcogenins **1** and **3** and, for reasons of comparison, for the parent biphenyl **5** as well. The results can be found in Tables I and II, the scheme of atom numbering in Figure 1. All crystallographic data have been deposited with the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen and are available on quoting the depository number CSD #58757, the names of authors, and journal citation.

TABLE I
Data collection and structure refinement parameter

	1	**3**	**5**
Crystallographic Section			
compound	$C_{16}H_{16}O_4S_2$	$C_{16}H_{16}O_4Se_2$	$C_{16}H_{18}O_4$
M [g mol^{-1}]	336.43	430.22	274.32
crystal system	monoclinic P	monoclinic P	monoclinic P
space group	$P2_1/a$	$P2_1/n$	$P2_1/c$
a [pm]	2332.9(5)	1303.9(2)	1254.9(4)
b [pm]	715.1(1)	725.7(1)	718.5(3)
c [pm]	1313.5(3)	1733.6(3)	1663.7(6)
β [°]	133.84(1)	100.80(2)	107.71(9)
V [10^6 pm^3]	1580.5	1611.4	1428.8(9)
Z	4	4	4
d (calc) [g cm^{-3}]	1.41	1.77	1.28

TABLE I (*Continued*)

	1	**3**	**5**
Data Collection			
diffractometer	CAD4 (Enraf Nonius)	Syntex $P2_1$	Syntex$P2_1$
radiation	Cu-K_α	Mo-K_α	Mo-K_α
data collection mode	$\omega/2\Theta$-scan	$\omega/2\Theta$-scan	$\omega/2\Theta$-scan
scan range [°]	$4.5<2\Theta<60$	$4.5<2\Theta<50$	$4.5<2\Theta<50$
independent reflections	2334	2851	2537
reflections with $Fo \geq 3\sigma(Fo)$	2137	2273	1360
refined parameters	205	215	186
μ [cm^{-1}]	31.3	44.6	0.9
absorption correction	DIFABS[12]	none	none
Structural analysis and Refinement			
solution by	direct methods	Patterson methods	direct methods
method of refinement	full matrix LSQ, hydrogen atoms with fixed distances of 96 pm and isotropic U	full matrix LSQ, hydrogen atoms with fixed distances of 96 pm and isotropic U	full matrix LSQ, hydrogen atoms with fixed distances of 96 pm and isotropic U
R; Rw with $w = 1/\sigma^2(F)$	0.051; 0.056	0.042; 0.034	0.076; 0.073
programs used	SHELX-76[13], SHELXS-86[14]	SHELX-76[13], SHELXS-86[14]	SHELX-76[13], SHELXS-86[14]

Molecular Structures

The molecular structures of compounds **1**, **3** and **5** are given in Figure 2, the mean values of the bond lengths and angles in Table III. In Table IV typical structural features are compared with those of reference compounds.

In each of the three compounds the methoxy groups are nearly coplanar to the phenyl rings they belong to, all in *exo*-positions. An analogous situation has been found for 2,3,7,8-tetramethoxythianthrene, too, where the consequences with respect to CO bond lengths and CCO bond angles have already been discussed in detail.[15]

In the biphenyl moiety, which is the main component of compounds **1**, **3** and **5**, the two aryl rings are twisted against each other, the angles between the planes increase in the order **1** < **3** < **5**. Taking the non-bridged biphenyl **5** as a standard, the dithio bridge in **1** seems to be a tighter clamp than the diseleno bridge in **3**. The clamping effect of the dichalcogeno bridges can also be seen from the bond angles at the atoms C(11) and C(21) by which the two aryl rings of the biphenyl unit are connected (see Table III).

Compounds **1** and **3** can also be compared with acyclic diaryl dichalcogenides the conformations of which, due to electronic effects, depend on the nature of the substituents at the aryl rings.[16] This is well documented for diaryl disulfides by a wealth of structure determinations, but not yet for diaryl diselenides for which not enough X-ray analyses have been done until now to allow the formation of mean values. Whereas the bond lengths in the cyclic and acyclic dichalcogenides do not differ significantly, partly tremendous changes of the bond and dihedral angles are caused by cyclization, thus minimizing the steric stress.

TABLE II

Refined atomic parameters with e.s.d. s in parentheses

Atom	1 (E = S)			3 (E = Se)			5		
	x/a	y/b	z/c	x/a	y/b	z/c	x/a	y/b	z/c
E(1)	0.40458(6)	0.1775(1)	0.3019(1)	0.0840(1)	-0.1893(1)	0.0892(1)	-	-	-
E(2)	0.42710(5)	-0.0244(1)	0.2220(1)	0.1936(1)	0.0462(1)	0.0634(1)	-	-	-
O(14)	0.1715(1)	0.0935(5)	0.2637(3)	-0.0937(3)	-0.0845(7)	0.3329(2)	0.4754(3)	0.6014(7)	0.0825(3)
O(15)	0.0718(1)	0.0161(5)	-0.0002(3)	0.0761(3)	-0.0034(6)	0.4275(2)	0.3304(4)	0.3496(7)	0.0794(3)
O(24)	0.3259(1)	0.0832(5)	-0.2674(3)	0.5915(3)	-0.0762(7)	0.1731(2)	0.0104(4)	0.7170(7)	0.4569(3)
O(25)	0.1832(1)	0.1528(5)	-0.3855(3)	0.5665(3)	-0.1487(6)	0.3135(2)	-0.0749(3)	0.6299(7)	0.3010(3)
C(11)	0.2518(2)	0.0777(5)	0.0613(4)	0.1845(4)	-0.0734(7)	0.2458(3)	0.2939(5)	0.6374(9)	0.2531(4)
C(12)	0.3049(2)	0.1172(6)	0.2055(4)	0.0919(4)	-0.1193(7)	0.1973(3)	0.3768(5)	0.767(1)	0.2565(4)
C(13)	0.2804(2)	0.1205(6)	0.2769(4)	-0.0036(4)	-0.1198(8)	0.2234(3)	0.4387(5)	0.760(1)	0.2004(4)
C(14)	0.2026(2)	0.0890(6)	0.2059(4)	-0.0058(4)	-0.0839(8)	0.3012(2)	0.4208(5)	0.620(1)	0.1416(4)
C(15)	0.1478(2)	0.0472(6)	0.0611(4)	0.0859(4)	-0.0379(8)	0.3512(3)	0.3408(5)	0.4839(9)	0.1385(4)
C(16)	0.1731(2)	0.0387(5)	-0.0074(4)	0.1795(4)	-0.0291(7)	0.3242(3)	0.2774(5)	0.4947(9)	0.1936(4)
C(17)	0.2264(3)	0.1152(8)	0.4127(5)	-0.1899(4)	-0.1078(13)	0.2810(4)	0.5528(6)	0.743(1)	0.0782(4)
C(18)	0.0136(2)	-0.0257(7)	-0.1459(4)	0.1664(4)	0.0349(9)	0.4838(3)	0.2703(7)	0.187(1)	0.0849(5)
C(21)	0.2754(2)	0.0780(6)	-0.0183(4)	0.2895(4)	-0.0716(7)	0.2218(3)	0.2221(5)	0.6514(8)	0.3096(4)
C(22)	0.3515(2)	0.0394(6)	0.0412(4)	0.3051(4)	-0.0244(7)	0.1466(3)	0.2672(5)	0.7044(9)	0.3929(4)
C(23)	0.3706(2)	0.0413(6)	-0.0383(4)	0.4056(4)	-0.0289(8)	0.1286(3)	0.1992(5)	0.7276(9)	0.4443(4)
C(24)	0.3143(2)	0.0786(6)	-0.1791(4)	0.4907(4)	-0.0706(8)	0.1853(3)	0.0853(6)	0.6970(9)	0.4128(4)
C(25)	0.2360(2)	0.1182(6)	-0.2441(4)	0.4771(4)	-0.1121(8)	0.2613(3)	0.0390(5)	0.6475(8)	0.3284(4)
C(26)	0.2179(2)	0.1199(6)	-0.1633(4)	0.3768(4)	-0.1150(7)	0.2777(3)	0.1068(5)	0.6224(8)	0.2776(4)
C(27)	0.4052(2)	0.0575(8)	-0.2083(4)	0.6086(4)	-0.0583(12)	0.0952(3)	0.0525(7)	0.757(1)	0.5447(4)
C(28)	0.1028(2)	0.1869(7)	-0.4573(4)	0.5589(4)	-0.1842(9)	0.3922(3)	-0.1293(5)	0.6165(9)	0.2121(4)

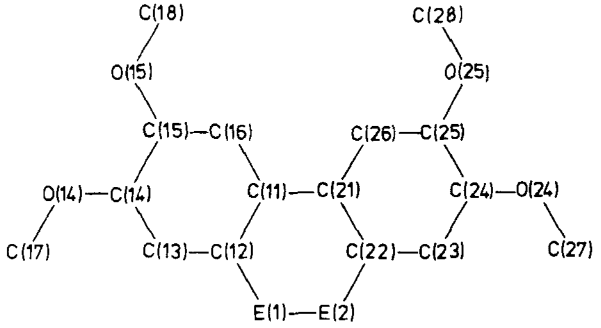


FIGURE 1 Scheme of atom numbering, **1**: E = S, **3**: E = Se, **5**: E(1)—E(2) = H,H.

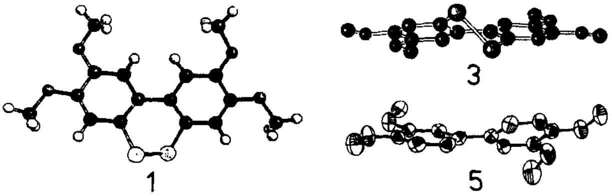


FIGURE 2 Molecular structures of compounds **1**, **3** and **5**.

TABLE III
Bond lengths [pm] and angles [°] (mean values)

bond	1 (E=S)	3 (E=Se)	5	angle	1 (E=S)	3 (E=Se)	5
E-E	205.6(2)	232.4(1)	--	E-E-C	97.8(1)	94.0(2)	--
E-C	177.2(5)	192.2(5)	--	E-C(2)-C(1)	120.5(4)	122.9(4)	--
O-C(Ar)	136.4(6)	136.6(6)	136.3(8)	E-C(2)-C(3)	117.7(4)	155.9(4)	--
O-C (Me)	141.8(7)	141.2(6)	142.3(9)	C(4)-O(4)-C(7)	116.5(5)	117.5(4)	117.9(6)
C(1)-C(1')	148.1(8)	150.4(6)	149.0(9)	C(5)-O(5)-C(8)	118.0(4)	118.9(4)	118.4(6)
C(1)-C(2)	139.1(7)	138.8(6)	138.3(9)	O-C-C(H)	125.5(5)	121.1(4)	124.9(6)
C(2)-C(3)	139.4(8)	140.2(7)	138.8(9)	O-C-C(O)	115.1(5)	115.4(4)	115.4(6)
C(3)-C(4)	136.4(7)	137.1(7)	137.5(10)	C(1')-C(1)-C(2)	123.5(5)	124.5(4)	121.0(6)
C(4)-C(5)	140.7(7)	139.6(6)	139.3(9)	C(1')-C(1)-C(6)	119.8(5)	118.4(4)	120.5(6)
C(5)-C(6)	138.3(8)	138.8(6)	138.4(9)	C(2)-C(1)-C(6)	116.9(5)	117.2(4)	118.6(6)
C(6)-C(1)	140.5(7)	139.8(6)	139.7(9)	C(3)-C(2)-C(1)	121.7(5)	121.3(5)	120.9(6)
				C(4)-C(3)-C(2)	120.7(5)	120.7(5)	120.3(6)
				C(5)-C(4)-C(3)	119.4(5)	119.0(5)	119.7(6)
				C(6)-C(5)-C(4)	119.6(5)	120.1(5)	119.8(6)
				C(1)-C(6)-C(5)	121.9(5)	121.8(5)	121.0(6)

Finally, a comparison of **1** and of its iodine complex **2** is of interest. The partial oxidation of the dithiin moiety in **2** should facilitate mesomeric interactions with the methoxy substituents leading to a certain double bond character especially of the C(Ar)—O, C(Ar)—S and C(11)—C(21) bonds. Though the trends found for **1** and **2** and, in part, for **5** and biphenyl go into the right direction, the experimental

TABLE IV

Typical structural data of **1**, **2**, **3** and of some reference compounds for comparison

	1 (E=5)	**2**[2] (E=S)	ArSSAr[16] *)	**3** (E=Se)	$[(ClC_6H_4Se^{\cdot})_2]^{17}$	**5**	$[(C_6H_5)_2]^{18-20}$
bond lengths (pm)							
E-E	205.6(2)	205.5(2)	207.2±0.8	232.4(1)	233.3(5)	--	--
C-E	177.2(5)	176.0(7)	177.1±0.3	192.2(5)	193(3)	--	--
C(1)-C(1')	148.1(8)	147.7(7)	--	--	--	149.0(9)	150.5(10)
bond angle (°)							
C-E-E	97.8(1)	98.3(2)	103.0±0.4	94.0(2)	101.5	--	--
angles between planes (°)							
C(1)SS'/SS'C(1')	58.7	55.6	82.1±5.4	55.6	74.5	--	--
C(1)SS/Ar	42.4	41.3	83.3±1.3	44.9	67.7	--	--
Ar/Ar'	26.8	25.1	28.0±5.5	30.5	41.5	42.2	0

*) mean values for diaryl disulfides with electron-donating substituents

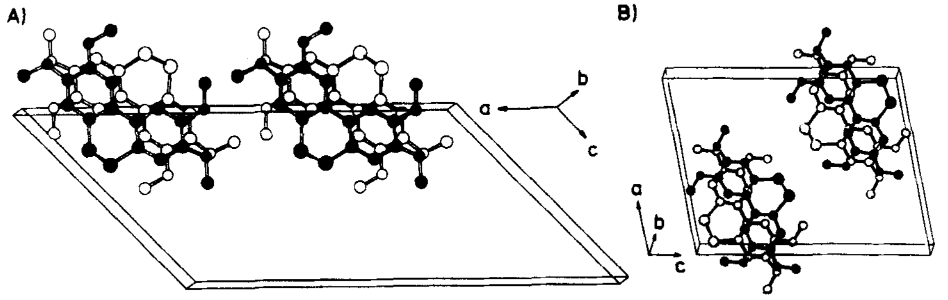


FIGURE 3 Unit cells of compounds **1** (A) and **3** (B).

errors of the structure determinations are too large to allow reliable statements. On the other hand, with an increasing oxidation state an increasing levelling of the dithiin unit should be found. Indeed, with regard to **1** the dithiin of **2** is more flattened according to the bond and dihedral angles.

Crystal Structures

Figure 3 shows the crystal structures of the homologous dichalcogenins **1** and **3** to be built according to the same structural principles giving columnar structures with stacks parallel to the b-axis of each unit cell.

In these stacks an AB, AB, . . . sequence is formed since successive molecules of a stack are rotated by 180° around their longitudinal axis through C(11)—C(21). In this way steric repulsion between the dichalcogeno bridges is avoided and an eclipsed arrangement of the biphenyl units made possible. The two substacks of parallel aryl rings thus formed differ by their setting angles relative to the a,c-plane (Figure 4).

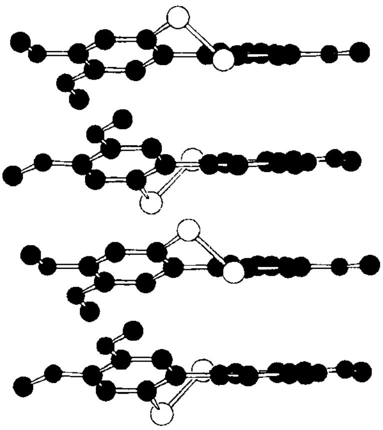


FIGURE 4 Side view of a stack in the crystal of **1**.

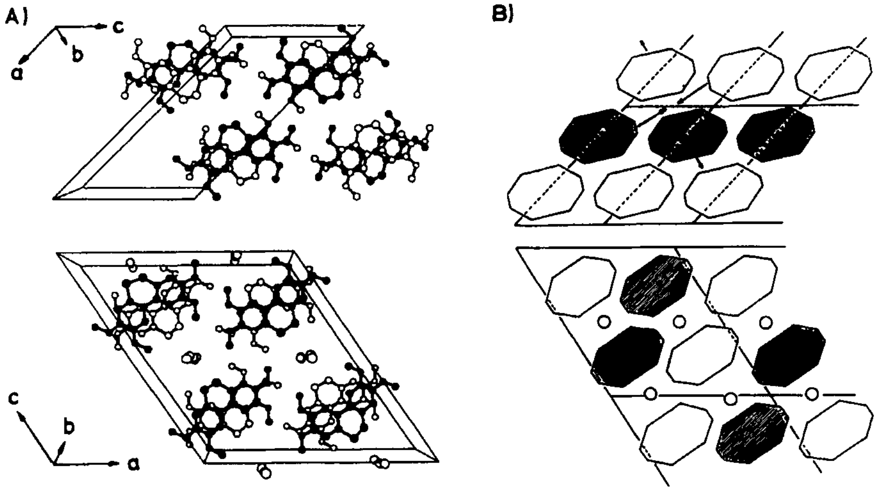


FIGURE 5 Comparison of the crystal structures of **1** and of its iodine complex **2**: A) view down the b-axis, B) diagrammatic representation of the structural changes during the transition **1** – – –> **2** (□, ▨ stacks of *R*- and *S*-enantiomers, resp., ○ channels for the iodine atoms).

Due to their non-planarity the dichalcogenins **1** and **3** possess axial chirality. Here, each of the two enantiomers is used as a building unit of a separate stack, the two kind of stacks showing a 1:1 ratio as is required for a racemate.

The same kind of stacking is also realized in **2**, the iodine complex of **1**.[2] In **2** the iodine atoms of the polyiodide ions, though disordered, are located in channels between the stacks. It can be seen from Figure 5 that the arrangement of the stacks in **2** is already preformed in **1**, *i.e.* a host function can be attributed to the lattice of **1** as has been suggested.

An estimate of the spatial requirements of the iodine in **2** is obtained assuming a cylindrical channel for the iodine atoms with the van der Waals radius of iodine

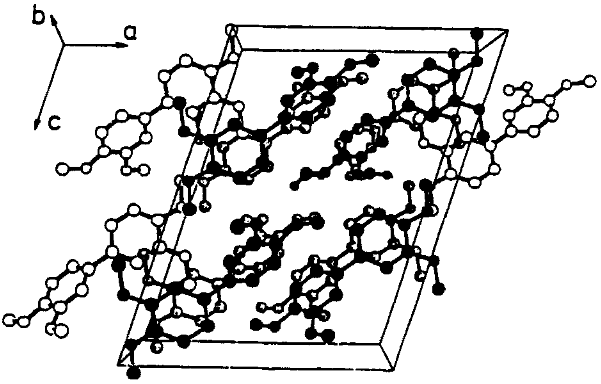


FIGURE 6 Contents of the unit cell of **5** (●) and stacking mode of the molecules.

(215 pm[21]) as radius and as distance between the iodine atoms in the channel the mean value of bonding and non-bonding distances (321 pm) in the iodine chains in the crystal of $(CH_3)_4NI_5$.[22,23] With these assumptions the volume needed for 1.16 iodine atoms is calculated to be $54 \cdot 10^6$ pm^3, whereas in comparison with **1** the volume per dithiin unit is increased by $35 \cdot 10^6$ pm^3 in **2**. This seems to indicate that about 35% of the volume required for the intercalation of the iodine atoms is already provided in the crystal of **1**. However, a closer look at Figure 5 shows the situation to be more complicated. The gap between neighbouring stacks with width of 365 pm in **1** is expended to 705 pm in **2** (increase of volume) but, at the same time, the rows of stacks are interlinked (decrease of volume). A further slight decrease of volume during the transition **1** – – –> **2** is achieved by a closer packing of the dithiin molecules by shortening the distances between the aryl rings of successive molecules from 388 to 350 pm. In addition, the intercalation of iodine is accompanied by small shifts in b-direction of the individual stacks relative to each other.

In **1** the shortest S ··· S contacts between sulfur atoms of adjacent stacks (460 and 466 pm) are larger than the van der Waals distance (370 pm[21]); in **2**, however, the corresponding values (735 and 777 pm) are shorter than the van der Waals distance of an arrangement S ··· I ··· S (800 pm) indicating interactions between the sulfur atoms of the dithiins in the stacks and the iodine atoms intercalated between them.

The non-bridged biphenyl **5** also forms a columnar structure in the solid state (Figure 6), again with stacks extended in b-direction. In these loose packed stacks one of the aryl rings is arranged parallel to the a,c-plane with distances between successive rings of 690 pm, the second one is inclined to this plane leading to inter-ring distances of 404 pm.

ACKNOWLEDGEMENTS

We thank the Fonds der Chemischen Industrie for financial support. The grant to J. B. by the Alfried Krupp von Bohlen und Halbach-Stiftung is gratefully acknowledged.

REFERENCES

1. Part 5: E. Sánchez Martínez, R. Díaz Calleja and G. Klar, *Synthetic Metals*, **38,** 93 (1990).
2. W. Hinrichs, J. Kopf, K.-W. Stender and G. Klar, *Z. Naturforsch, Teil B*, **40,** 39 (1985).
3. K. Stender, G. Klar, M. Peo, W. Bauhofer and S. Roth, *Mol. Cryst. Liq. Cryst.*, **120,** 277 (1985).
4. D. E. Janssen, J. VanAllen and C. V. Wilson, *J. Org. Chem.*, **20,** 1326 (1955).
5. F. W. Bennett and A. G. Sharpe, *J. Chem. Soc.*, 1383 (1950).
6. C. Seer and E. Karl, *Monatsh. Chem.*, **34,** 631 (1913).
7. K.-W. Stender, N. Wölki and G. Klar, *Phosphorus, Sulfur and Silicon*, **42,** 111 (1989).
8. W. Baker, J. W. Barton, J. F. W. McOmie, R. J. Penneck and M. L. Watts, *J. Chem. Soc.*, 3986 (1961).
9. L. Engman, *J. Heterocycl. Chem.*, **21,** 413 (1984).
10. H. W. Underwood, Jr., O. L. Baril and G. C. Toone, *J. Am Chem. Soc.*, **52,** 4087 (1930).
11. H. Gilman and B. J. Gaj, *J. Org. Chem.*, **22,** 447 (1957).
12. N. Walker and D. Stuart, *Acta Crystallogr., Ser. A*, **39,** 158 (1983).
13. G. M. Sheldrick, SHELX-76, *A Program for Crystal Structure Determination*, University of Cambridge, England, 1976.
14. G. M. Sheldrick SHELXS-86, *Programs for Crystal Structure Solution*, Universität Göttingen, 1986.
15. W. Hinrichs, H.-J. Riedel and G. Klar, *J. Chem. Res.*, (S) 334; (M) 3501 (1982).
16. M. Dötze and G. Klar, *J. Chem. Res.*, (S) 226 (1993).
17. F. H. Kruse, R. E. Marsh and J. D. McCullough, *Acta Crystallogr.*, **10,** 201 (1957).
18. J. Trotter, *Acta Crystallogr.*, **14,** 1135 (1961).
19. A. Hargreaves and S. Hasan Rizvi, *Acta Crystallogr.*, **15,** 365 (1962).
20. G.-P. Charbonneau and Y. Delugeard, *Acta Cryst., Sect. B*, **32,** 1420 (1976).
21. "Handbook of Chemistry and Physics," CRC Press Cleveland, Oh, 58th edn., 1977–1978, p. D-178.
22. R. J. Hach and R. E. Rundle, *J. Am. Chem. Soc.*, **73,** 4321 (1951).
23. J. Broekema, E. E. Havinga and E. H. Wiebenga, *Acta Crystallogr.*, **10,** 596 (1957).