ACTA CHEMICA SCANDINAVICA 26 (1972) 873–888

Structures of Linear Multisulphur Systems

II. The Crystal and Molecular Structure of 7-(5-*t*-Butyl-1,2-dithiole-3-ylidene)-4,5,6,7-tetrahydro-1,2-benzodithiole-3-thione

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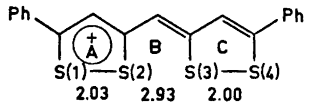
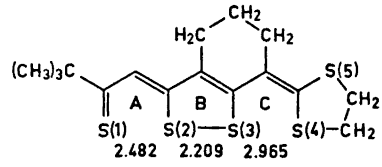
The structure of $C_{14}H_{16}S_5$ has been determined by X-ray crystallographic methods. 2719 unique reflections were measured twice on a computer-controlled four-circle diffractometer using the $\theta-2\theta$ scan technique and Mo$K\alpha$ radiation. The structure was solved by the Patterson method and refined by full-matrix least-squares technique to an R of 0.077. Standard deviations in S – S, S – C, C – C, and C – H bond lengths are 0.003 Å, 0.007 – 0.009 Å, 0.010 – 0.013 Å, and 0.06 – 0.11 Å, respectively. Four sulphur atoms are lying on a row with distances S(1) – S(2) = 2.063 Å, S(2) – S(3) = 2.863 Å, S(3) – S(4) = 2.062 Å.

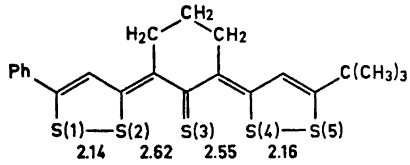
In 6a-thiathiophthenes the sulphur-sulphur bonds are in the region between a single bond and van der Waals distance. The partial covalent bonding between the sulphur atoms may be described in terms of a delocalized σ-system comprising the three sulphur atoms. Additionally there is a delocalized system of π-electrons which may extend across the sulphur atoms.[1]



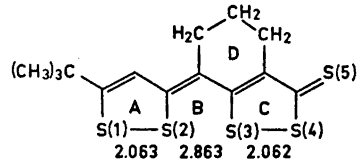
(I)



(II)



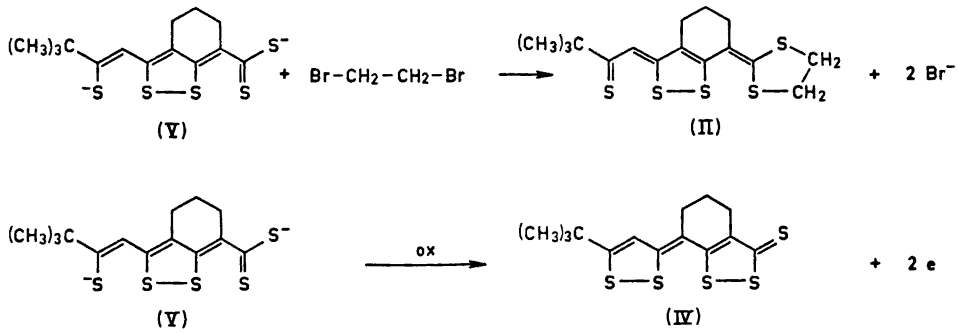
(III)



(IV)

The question of whether such "abnormal" partial bonding between sulphur atoms also exists in analogous compounds with more than three sulphur atoms in a row, has been studied in this laboratory for some time. So far, reports have been given on the structure determinations of three such compounds (I, II, and III).[2-4]

Chemically, molecule IV in the present investigation closely resembles molecule II. Both of the compounds are obtained from the dianion V by reaction with ethylene dibromide and by oxydation, respectively.[5]



In compound II there is a bridging ethylene group between S(4) and S(5)' Thus S(4) is engaged in an S – C σ-bond in a direction *trans* to S(3). The ethylene group is not present in compound IV and this would be expected to strongly affect the sulphur-sulphur bonding scheme.

EXPERIMENTAL

The compound has been synthesized by Stavaux and Lozac'h [5] who kindly supplied a sample. The crystals grew as deep red, thin plates from an ethylacetate solution by slow evaporation. Preliminary cell dimensions and space group were determined by precession film methods. Final cell parameters were derived from the least-squares treatment of the 2θ, χ, and ϕ settings of 12 reflections measured on a computer-controlled four-circle diffractometer using Mo$K\alpha$ radiation ($\lambda = 0.71069$ Å). The density as measured by flotation is 1.49 g cm^{-3}.

Crystal data

$C_{14}H_{16}S_5$	M.w. 344.598
Crystal system	monoclinic
Systematic extinctions	hkl, when $h+k=2n+1$
	$h0l$, when $l=2n+1$
Space group	$C2/c$ or Cc
Cell dimensions	
a = 22.264(4) Å,	b = 9.122(2) Å
c = 15.216(3) Å,	β = 91.683(8)°
V = 3089 Å^3	
D_m = 1.49 g cm^{-3},	D_x = 1.482 g cm^{-3} for $Z=8$
μ = 7.02 cm^{-1} (for Mo$K\alpha$ radiation).	

The crystals had a high mosaic spread, and in a number of cases the reflections were observed to be split. None of the crystals examined were ideal for accurate X-ray crystallographic work. The crystal selected for data collection had dimensions 0.013 mm × 0.425 mm × 0.500 mm, approximately in the directions of the a, b, and c axes, respectively, and was mounted along the c^* axis. 2719 unique reflections with $2\theta < 50°$ were measured by the $\theta - 2\theta$ scan technique on a computer-controlled 4-circle diffractometer using niobium-filtered Mo$K\alpha$ radiation ($\lambda = 0.71069$ Å). Scan ranges were calculated according to the relationship $\Delta 2\theta = A + B \tan\theta$.[6] Values for A and B were estimated by measuring the sufficient scan ranges for a number of reflections distributed at different locations in the reciprocal space. Backgrounds were measured for 10 sec at each of the scan range extremes, and the net count calculated as $N = N_{Pk} - (N_{B1} + N_{B2}) \times T/20$. N_{Pk} is the number of counts in a scan, N_{B1} and N_{B2} are the background counts and T is the scan time in sec.

During the data collection three reference reflections were remeasured for every 200 reflections. The fluctuation in net intensity of the individual standard reflection approached 15 %. However, no evidence of crystal deterioration was found. As no fault in the electronics of the instrument could be detected, this large variation in intensities was believed to be caused by small movements of the crystal. Due to the high mosaic spread, the dimensions of the receiving aperture may become critical,[7] and any movement of the crystal may cause a different amount of the peak to be "cut off". The data were recollected, leaving the receiving aperture wide open. However, the stability of the reference reflections was not significantly better than during the first data collection. It was therefore decided to combine the two data sets. Scale factors based on the mean value of the reference reflections bracketing each group of 200 reflections were evaluated for each data set. The net count for each reflection was then calculated as $N = \sum_{i=1}^{2} k_i[N_{Pki} - (N_{B1i} + N_{B2i}) \times T/20]$, where k_i is the appropriate scale factors for the group of reflections. The standard deviation in measurement due to counting statistics is $\sigma_c = [\sum_{i=1}^{2} (N_{Pki} + (N_{B1i} + N_{B2i}) \times T/20)^2]^{\frac{1}{2}}$. Of the 2719 unique reflections, 871 were found to be less than $3\sigma_c$. These were coded as unobserved, and set equal to $3\sigma_c$. The total error in the net intensity of each reflection was taken as

$$\sigma_I = [\sigma_c^2 + (0.03\ N)^2]^{\frac{1}{2}} \sum_{i=1}^{2} (k_i)/2$$

where 0.03 N is an estimate of the random error due to instrument instability, and movement of the crystal. The "instability factor" 0.03 was arrived at by inspection of a plot of $(k_1 + k_2)/2$.

The data were corrected for Lorentz and polarization effects. The standard deviations in the structure factors were calculated as $\sigma_F = [F^2 + \sigma_I(Lp)^{-1}]^{\frac{1}{2}} - F$. Near the end of the refinement an absorption correction was applied using the Gaussian integration method, described by Busing and Levy.[8] This correction was found necessary even though the linear absorption coefficient, μ, is fairly low. Due to the uneven shape of the crystal the attenuation caused by absorption was found to range from 1 % to 25 %.

STRUCTURE DETERMINATION AND REFINEMENT

The statistical distribution of E-values [9] clearly indicates that the space group is centrosymmetric, thus $C2/c$ rather than Cc was chosen. The successful solution and refinement confirms this choice of space group.

The four sulphur atoms lying on a row were located from a three-dimensional Patterson map. A structure factor calculation based on the four sulphur atoms gave an R of 0.59. Phases determined by these atoms were used to calculate a three-dimensional electron density map. From this map the fifth sulphur atom and eight carbon atoms were located. One more such cycle revealed three additional carbon atoms. The terminal carbon atoms on the

t-butyl group could not be located until the sulphur positions had been adjusted by one cycle of full-matrix least-squares refinement. In the initial refinement cycles all atoms were assigned isotropic temperature factors. Later, anisotropic thermal parameters were introduced, first on sulphur, then on carbon atoms in two consecutive cycles. The refinement converged at an R of 0.09.

At this stage all of the 16 hydrogen atoms were located from a three-dimensional ΔF synthesis. These atoms were included in the further refinement with isotropic thermal parameters, and all of them refined to reasonable positions.

At the conclusion of the refinement all shifts in parameters were less than a tenth of the corresponding standard deviation, and the conventional agreement factor ($R = \sum||F_o| - |F_c||/\sum|F_o|$) is 0.077 omitting unobserved reflections. The function minimized in the refinement was $w(|F_o| - |(1/k)F_c|)^2$ where $w = 1/\sigma_F^2$. The unobserved reflections were given zero weight in the refinement except if $F_c > 3\sigma_F$, in this case the reflections are included in the refinement with weight calculated as shown above.

Table 1a. Positional parameters at the conclusion of the least-squares refinement. Standard deviations are listed in parentheses and refer to the last decimal places.

	X/a	*Y/b*	*Z/c*
S(1)	0.32157(10)	0.48745(30)	0.03034(14)
S(2)	0.40156(9)	0.39966(29)	0.07770(13)
S(3)	0.51545(9)	0.29271(26)	0.14213(13)
S(4)	0.58239(10)	0.15989(30)	0.19449(14)
S(5)	0.66057(11)	−0.03532(30)	0.10156(18)
C(1)	0.3365(3)	0.4555(8)	−0.0806(5)
C(2)	0.3898(4)	0.3957(10)	−0.0957(5)
C(3)	0.4310(3)	0.3558(9)	−0.0253(5)
C(4)	0.4852(3)	0.2835(9)	−0.0362(5)
C(5)	0.5246(3)	0.2352(9)	0.0329(5)
C(6)	0.5711(3)	0.1336(9)	0.0219(5)
C(7)	0.6043(4)	0.0853(10)	0.0961(6)
C(8)	0.5029(4)	0.2467(13)	−0.1289(6)
C(9)	0.5673(4)	0.1924(13)	−0.1366(6)
C(10)	0.5836(4)	0.0785(11)	−0.0692(6)
C(11)	0.2846(3)	0.4884(9)	−0.1451(5)
C(12)	0.3090(5)	0.4893(16)	−0.2387(6)
C(13)	0.2372(4)	0.3700(13)	−0.1370(8)
C(14)	0.2573(5)	0.6366(12)	−0.1255(8)
H(2)	0.402(2)	0.357(6)	−0.149(4)
H(81)	0.471(3)	0.185(8)	−0.166(5)
H(82)	0.494(4)	0.332(10)	−0.163(6)
H(91)	0.572(5)	0.149(12)	−0.197(7)
H(92)	0.595(4)	0.294(11)	−0.133(6)
H(101)	0.623(5)	0.025(12)	−0.069(7)
H(102)	0.555(6)	−0.020(16)	−0.080(9)
H(121)	0.323(4)	0.392(10)	−0.248(5)
H(122)	0.273(5)	0.501(12)	−0.291(7)
H(123)	0.338(5)	0.557(11)	−0.241(6)
H(131)	0.218(4)	0.351(10)	−0.076(6)
H(132)	0.252(3)	0.279(9)	−0.141(5)
H(133)	0.209(4)	0.378(9)	−0.183(5)
H(141)	0.240(4)	0.638(11)	−0.071(7)
H(142)	0.224(5)	0.660(11)	−0.166(7)
H(143)	0.286(4)	0.721(11)	−0.121(6)

Table 1b. Thermal parameters with the corresponding standard deviations in parentheses. The anisotropic thermal parameters are defined by the expression $T_i = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$ and the isotropic parameters by $T_i = \exp(-8\pi^2 U \sin^2\theta/\lambda^2)$. For non-hydrogen atoms the values are multiplied by 10^4, for hydrogen atoms by 10^3.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
S(1)	339(11)	688(18)	277(10)	163(12)	−41(11)	26(8)
S(2)	340(11)	669(17)	219(10)	140(11)	−52(11)	6(8)
S(3)	341(11)	498(15)	243(10)	83(11)	−18(10)	−20(8)
S(4)	390(12)	597(16)	346(11)	78(12)	37(12)	−81(9)
S(5)	407(13)	508(16)	671(16)	172(12)	17(13)	−119(11)
C(1)	306(41)	227(43)	286(40)	−6(34)	−96(34)	46(32)
C(2)	340(43)	394(51)	205(37)	56(38)	−5(37)	20(32)
C(3)	308(42)	319(46)	257(38)	−50(36)	0(36)	41(31)
C(4)	152(35)	341(48)	317(39)	−10(33)	−11(37)	45(29)
C(5)	227(37)	336(47)	258(38)	−84(34)	12(35)	18(31)
C(6)	153(36)	324(47)	427(46)	−1(33)	23(38)	79(32)
C(7)	320(43)	326(49)	446(48)	−45(38)	57(41)	−68(36)
C(8)	387(50)	596(67)	300(43)	148(47)	−58(48)	37(38)
C(9)	408(49)	624(70)	291(45)	83(49)	−39(47)	80(37)
C(10)	369(48)	383(55)	400(48)	127(44)	−59(43)	48(38)
C(11)	314(41)	313(48)	324(42)	−14(37)	−30(37)	−20(33)
C(12)	428(57)	715(86)	298(48)	33(60)	−16(52)	−16(41)
C(13)	317(51)	458(71)	635(70)	−102(48)	102(54)	−110(48)
C(14)	457(59)	363(59)	538(62)	115(48)	89(49)	−48(50)

	U (Å^2)		U (Å^2)		U (Å^2)
H(2)	0(13)	H(101)	74(33)	H(131)	46(25)
H(81)	26(19)	H(102)	115(48)	H(132)	17(20)
H(82)	39(25)	H(121)	30(23)	H(133)	28(21)
H(91)	73(32)	H(122)	69(31)	H(141)	58(30)
H(92)	57(28)	H(123)	50(30)	H(142)	61(29)
				H(143)	46(26)

The following atomic scattering factors were used; for sulphur those of Dawson,[10] for carbon those of Berghuis *et al.*[11] and for hydrogen those of Stewart *et al.*[12]

RESULTS AND DISCUSSION

The final positional and thermal parameters are listed in Table 1a and b. Table 2 is a list of the observed and final calculated structure factors multiplied by a factor of 10. Interatomic distances and angles are summarized in Tables 3 and 4.

The sequence S(1), S(2), S(3) is approximately linear, while the fourth sulphur atom deviates significantly from this line, the angle S(1), S(2), S(3)/S(3), S(4) being 163°. Table 5 lists the atomic deviations from least-squares planes through various parts of the molecule. The terminal dithiole rings A and C are planar, while the central ring B is non-planar. There is a dihedral angle of 17.0° between the planes of rings A and C. This angle is attained mainly by a twist around C(3)···C(5) and a bending around S(3)−C(5). A similar distortion, although to a smaller degree, is also found in molecule II (Fig. 1).

Table 2. Observed and calculated structure factors multiplied by a factor of 10. Reflections with observed intensities less than the threshold value of $3\sigma_c$ are marked with < signs.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
-26	0	2	229	35	-20	0	6	1012	-1016	-18	4	11	249	-236	-16	6	9	<211	158	-14	6	5	241	228
-26	2	1	508	-553	-20	0	8	214	181	-18	6	1	263	205	-16	6	10	<201	31	-14	6	6	<187	-131
-26	3	1	<206	-74	-20	0	10	423	403	-18	6	2	236	176	-16	8	1	333	248	-14	6	7	221	-174
-26	3	2	318	-299	-20	0	12	411	446	-18	6	3	<207	192	-16	8	2	<215	128	-14	6	8	476	-474
-26	3	3	453	-462	-20	2	1	220	210	-18	6	4	<205	-143	-16	8	3	370	-335	-14	6	9	203	-134
-26	1	1	<206	-74	-20	2	2	232	178	-18	6	5	278	-266	-16	8	4	273	267	-14	6	10	4197	-87
-26	1	2	229	-276	-20	2	3	452	461	-18	6	6	236	-208	-16	8	5	<198	23	-14	6	11	<205	21
-26	1	3	<206	-70	-20	2	4	<182	93	-18	6	7	421	-430	-15	7	1	263	227	-14	8	1	458	-490
-26	1	4	<207	25	-20	2	5	212	182	-18	6	8	<208	-127	-15	7	2	345	-339	-14	8	2	389	-345
-26	1	5	212	46	-20	2	6	<192	-100	-18	6	9	236	81	-15	7	3	448	486	-14	8	3	378	-436
-26	1	6	<210	-54	-20	2	7	214	287	-17	7	1	<197	-54	-15	7	4	<198	111	-14	8	4	<196	92
-24	0	2	540	-584	-20	2	8	348	-393	-17	7	2	281	-278	-15	7	5	199	-170	-14	8	5	245	235
-24	0	4	654	-656	-20	2	9	<204	-138	-17	7	3	500	462	-15	7	6	<191	-62	-14	8	6	286	260
-24	0	6	403	-440	-20	2	10	<205	-106	-17	7	4	317	220	-15	7	7	<208	-226	-14	8	7	<204	38
-24	0	8	<211	-40	-20	2	11	225	34	-17	7	5	258	293	-15	7	8	<200	-41	-13	9	1	<213	108
-24	2	1	<200	-157	-20	4	1	<193	-93	-17	7	6	<207	97	-15	7	9	<206	-167	-13	9	2	226	-117
-24	2	2	<213	-210	-20	4	2	<193	-86	-17	7	7	<210	-94	-15	5	1	<207	243	-13	9	3	294	-228
-24	2	3	<196	-15	-20	4	3	268	-243	-17	5	1	<184	35	-15	5	2	545	547	-13	9	4	254	-248
-24	2	4	414	-443	-20	4	4	<192	-11	-17	5	2	385	377	-15	5	3	<180	-33	-13	9	5	<212	-127
-24	2	5	234	-164	-20	4	5	<192	-84	-17	5	3	<185	-21	-15	5	4	195	31	-13	7	1	<184	3
-24	2	6	<193	-64	-20	4	6	<208	172	-17	5	4	<184	101	-15	5	5	<179	-83	-13	7	2	218	-233
-24	2	7	334	-253	-20	4	7	<197	-106	-17	5	5	465	452	-15	5	6	560	-520	-13	7	3	<198	-200
-24	4	1	523	567	-20	4	8	272	239	-17	5	6	210	-109	-15	5	7	493	-494	-13	7	4	<186	-100
-24	4	2	299	310	-20	4	9	<201	-30	-17	5	7	<192	127	-15	5	8	301	326	-13	7	5	<189	-61
-24	4	3	227	-108	-20	4	10	<211	162	-17	5	8	<198	157	-15	5	9	<194	7	-13	7	6	227	-202
-24	4	4	<225	-280	-20	6	1	524	-539	-17	5	9	<206	-149	-15	5	10	473	458	-13	7	7	<206	213
-24	5	1	<216	-166	-20	6	2	298	-322	-17	5	10	378	370	-15	5	11	281	300	-13	7	8	336	-279
-24	5	2	523	575	-20	6	3	<197	25	-17	5	11	<207	97	-15	5	12	231	208	-13	7	9	<197	-19
-23	5	3	365	397	-20	6	4	<208	102	-17	3	1	274	-256	-15	3	1	218	172	-13	7	10	254	-220
-23	3	1	310	272	-20	6	5	<209	92	-17	3	2	289	240	-15	3	2	<164	-52	-13	5	1	461	-411
-23	3	2	<195	24	-20	6	6	<209	-95	-17	3	3	<175	-48	-15	3	3	532	-569	-13	5	2	331	286
-23	3	3	<194	-51	-19	7	1	<216	-127	-17	3	4	<178	-11	-15	3	4	494	-510	-13	5	3	391	-390
-23	3	4	<204	77	-19	7	2	509	-533	-17	3	5	252	268	-15	3	5	456	-447	-13	5	4	<177	-135
-23	3	5	<195	-66	-19	7	3	<218	-217	-17	3	6	265	283	-15	3	6	281	301	-13	5	5	<175	-22
-23	3	6	<202	-167	-19	7	4	352	-328	-17	3	7	722	714	-15	3	7	340	393	-13	5	6	254	-252
-23	3	7	<201	-17	-19	7	5	<212	-81	-17	3	8	230	-211	-15	3	8	272	271	-13	5	7	272	-299
-23	1	1	<195	130	-19	5	1	<190	41	-17	3	9	362	381	-15	3	9	355	346	-13	5	8	318	-288
-23	1	2	220	205	-19	5	2	<197	-184	-17	3	10	390	-386	-15	3	10	259	261	-13	5	9	236	198
-23	1	3	<196	33	-19	5	3	203	-243	-17	3	11	276	-251	-15	3	11	210	203	-13	5	10	449	-454
-23	1	4	347	-345	-19	5	4	413	-356	-17	3	12	282	252	-15	3	12	597	582	-13	5	11	242	-166
-23	1	5	230	253	-19	5	5	292	-248	-17	3	13	263	-229	-15	3	13	<248	218	-13	5	12	301	-288
-23	1	6	<200	-56	-19	5	6	<199	-34	-17	1	1	998	-997	-15	3	14	<209	57	-13	5	13	<221	-185
-23	1	7	250	-210	-19	5	7	<197	81	-17	1	2	853	827	-15	1	1	724	-736	-13	3	1	655	603
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-22	0	8	<217	42	-19	3	4	<193	-262	-17	1	8	477	-464	-15	1	7	202	-171	-13	3	7	<177	-27
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-22	2	7	<202	-53	-19	1	1	565	-611	-16	0	4	404	424	-15	1	15	337	-333	-13	3	15	<202	-3
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-22	2	9	<211	-55	-19	1	3	323	334	-16	0	8	1398	1670	-14	0	4	570	-595	-13	1	2	821	-815
-22	4	1	426	554	-19	1	4	212	-190	-16	0	10	203	110	-14	0	6	584	581	-13	1	3	<160	42
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-21	3	7	219	-175	-18	2	7	399	422	-16	4	5	203	-222	-14	4	2	278	275	-12	2	1	515	467
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-21	1	7	601	-658	-18	4	4	640	-672	-16	6	2	609	627	-14	4	12	250	182	-12	2	11	<176	75
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-21	1	9	<219	-214	-18	4	6	<194	-208	-16	6	4	<190	129	-14	4	14	690	651	-12	2	13	<193	91
-21	1	10	495	465	-18	4	7	<192	145	-16	6	5	<188	64	-14	6	1	<179	-54	-12	2	14	339	-341
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Table 2. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
-12	4	2	553	528	-10	2	3	890	903	-9	1	3	346	340	-7	7	13	278	-158	-6	8	3	<184	94
-12	4	3	<176	-190	-10	2	4	655	689	-9	1	4	1088	-1083	-7	5	1	637	644	-6	8	4	368	374
-12	4	4	<176	-33	-10	2	5	176	-115	-9	1	5	334	331	-7	5	2	<164	-133	-6	8	5	321	254
-12	4	5	222	-150	-10	2	6	671	680	-9	1	6	<151	-40	-7	5	3	347	-362	-6	8	6	<186	63
-12	4	6	<174	96	-10	2	7	<161	-17	-9	1	7	762	-747	-7	5	4	1028	-1023	-6	8	7	300	314
-12	4	7	261	283	-10	2	8	<167	30	-9	1	8	346	338	-7	5	5	672	-664	-6	8	8	204	-157
-12	4	8	317	288	-10	2	9	<172	104	-9	1	9	<168	110	-7	5	6	300	-267	-6	8	9	241	-151
-12	4	9	285	-309	-10	2	10	<176	-132	-9	1	10	359	314	-7	5	7	299	285	-6	8	10	213	-42
-12	4	10	216	-184	-10	2	11	374	-357	-9	1	11	<174	81	-7	5	8	<177	58	-6	8	11	241	239
-12	4	11	<191	61	-10	2	12	640	644	-9	1	12	574	561	-7	5	9	<171	-4	-6	10	1	272	191
-12	4	12	317	-325	-10	2	13	<180	-52	-9	1	13	572	-576	-7	5	10	<175	-36	-6	10	2	211	19
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-12	4	14	<207	-99	-10	2	15	<195	80	-9	1	15	419	368	-7	5	12	480	-446	-6	10	4	247	186
-12	6	1	311	289	-10	2	16	<200	-28	-9	1	16	403	456	-7	5	13	<192	-30	-6	10	5	217	60
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-11	9	1	226	-216	-10	6	6	<182	132	-8	2	12	533	539	-7	1	3	220	215	-5	7	12	<201	75
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-11	3	13	329	333	-9	5	5	627	-612	-8	8	8	257	-225	-6	4	4	692	-709	-5	1	10	193	178
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-11	1	1	421	427	-9	5	8	876	880	-8	8	11	230	147	-6	4	7	<162	5	-5	1	13	204	-155
-11	1	2	1217	1209	-9	5	9	271	-287	-8	10	1	<203	96	-6	4	8	186	131	-5	1	14	250	-207
-11	1	3	<147	50	-9	5	10	<182	-4	-8	10	2	234	55	-6	4	9	378	342	-5	1	15	<190	-35
-11	1	4	<152	100	-9	5	11	<183	-31	-8	10	3	235	99	-6	4	10	269	259	-5	1	16	<193	-115
-11	1	5	243	238	-9	5	12	<198	-152	-8	10	4	<208	-161	-6	4	11	220	166	-5	1	17	<205	-20
-11	1	6	334	-350	-9	5	13	301	338	-7	9	1	<196	15	-6	4	12	399	-392	-4	0	2	1437	-1388
-11	1	7	388	-354	-9	5	14	<200	55	-7	9	2	214	143	-6	4	13	324	375	-4	0	4	1441	-1409
-11	1	8	186	143	-9	5	15	<207	15	-7	9	3	221	-278	-6	4	14	226	-185	-4	0	6	4292	3998
-11	1	9	437	-446	-9	3	1	481	478	-7	9	4	<200	-67	-6	4	15	<212	-210	-4	0	8	3290	3111
-11	1	10	<164	45	-9	3	2	1491	-1428	-7	9	5	<198	42	-6	4	16	344	-358	-4	0	10	449	-424
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-10	0	14	556	514	-9	3	15	<208	95	-7	7	9	352	372	-6	6	13	<205	-185	-4	2	10	766	779
-10	0	16	261	134	-9	3	16	<216	-38	-7	7	10	218	204	-6	6	14	<202	41	-4	2	11	885	918
-10	2	1	1180	-1152	-9	1	1	455	433	-7	7	11	<199	85	-6	8	1	557	587	-4	2	12	374	411
-10	2	2	471	-459	-9	1	2	423	467	-7	7	12	<210	97	-6	8	2	<182	96	-4	2	13	213	-173

Table 2. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
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-4	2	15	<185	59	-3	1	3	1305	1371	-1	7	7	310	301	0	6	4	448	405	1	1	13	460	-515
-4	2	16	<203	92	-3	1	4	1158	-1189	-1	7	8	<180	-53	0	6	5	552	-557	1	1	14	332	376
-4	2	17	295	-231	-3	1	5	1404	-1502	-1	7	9	221	258	0	6	6	796	830	1	1	15	427	468
-4	4	1	813	-843	-3	1	6	<150	17	-1	7	10	<191	44	0	6	7	<171	-14	1	1	16	<213	-216
-4	4	2	320	-309	-3	1	7	448	-489	-1	7	11	<197	98	0	6	8	190	170	1	1	17	365	400
-4	4	3	450	-455	-3	1	8	633	692	-1	7	12	<205	-145	0	6	9	464	508	2	0	0	1493	1471
-4	4	4	534	525	-3	1	9	253	296	-1	7	13	290	-245	0	6	10	<187	23	2	0	2	1015	-900
-4	4	5	496	-537	-3	1	10	834	856	-1	9	1	165	128	0	6	11	303	344	2	0	4	957	902
-4	4	6	266	313	-3	1	11	<191	66	-1	9	2	<151	-1	0	6	12	378	-416	2	0	6	1337	1287
-4	4	7	<155	11	-3	1	12	<175	60	-1	9	3	327	-330	0	6	13	<203	-4	2	0	8	391	380
-4	4	8	221	242	-3	1	13	285	248	-1	9	4	<154	-105	0	6	14	313	-270	2	0	10	476	-479
-4	4	9	<174	47	-3	1	14	<180	143	-1	9	5	<156	-64	0	6	15	<213	47	2	0	12	1018	-958
-4	4	10	<172	-46	-3	1	15	<185	-50	-1	9	6	354	338	0	8	0	357	335	2	0	14	616	619
-4	4	11	311	302	-3	1	16	<191	-87	-1	9	7	<161	-65	0	8	1	388	-338	2	0	16	368	349
-4	4	12	<191	-50	-3	1	17	<205	95	-1	9	8	235	233	0	8	2	313	272	2	2	0	3964	-3835
-4	4	13	662	706	-2	0	2	2036	1937	-1	9	9	658	749	0	8	3	<198	175	2	2	1	2319	-2293
-4	4	14	307	333	-2	0	4	1450	-1373	-1	9	10	838	-931	0	8	4	324	252	2	2	2	880	-833
-4	4	15	205	-126	-2	0	6	423	368	-1	9	11	<187	-35	0	8	5	248	233	2	2	3	182	193
-4	4	16	<215	-1	-2	0	8	1055	-1077	-1	9	12	663	-760	0	8	6	<188	156	2	2	4	440	491
-4	6	1	356	-330	-2	0	10	<164	66	-1	9	13	<200	-163	0	8	7	248	-235	2	2	5	655	-656
-4	6	2	392	400	-2	0	12	534	524	-1	9	14	<202	81	0	8	8	<209	188	2	2	6	650	673
-4	6	3	<170	-105	-2	0	14	388	395	-1	9	15	<203	113	0	8	9	<193	133	2	2	7	477	569
-4	6	4	272	-238	-2	0	16	210	-118	-1	9	16	<209	8	0	8	10	<206	-43	2	2	8	271	280
-4	6	5	623	-659	-2	0	18	641	-661	-1	3	1	968	1007	0	8	11	318	361	2	2	9	329	340
-4	6	6	293	266	-2	2	1	2448	2544	-1	3	2	1350	-1399	0	8	12	<214	-92	2	2	10	714	783
-4	6	7	<178	-58	-2	2	2	<155	-10	-1	3	3	<151	-95	0	10	0	<209	65	2	2	11	1179	-1304
-4	6	8	184	97	-2	2	3	298	294	-1	3	4	199	169	0	10	1	<208	111	2	2	12	587	658
-4	6	9	242	209	-2	2	4	205	261	-1	3	5	223	-282	0	10	2	<200	26	2	2	13	456	-522
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-4	6	12	562	602	-2	2	7	1405	-1563	-1	3	8	816	-969	0	10	5	<213	-143	2	2	16	495	538
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-4	6	14	<211	156	-2	2	9	<155	-46	-1	3	10	314	-308	0	10	7	<212	-132	2	4	0	388	-389
-4	8	1	250	-188	-2	2	10	378	440	-1	3	11	226	137	1	9	0	522	504	2	4	1	471	-542
-4	8	2	<181	63	-2	2	11	<178	-192	-1	3	12	568	-621	1	9	1	417	-452	2	4	2	519	-499
-4	8	3	400	-435	-2	2	12	364	411	-1	3	13	202	252	1	9	2	<199	-153	2	4	3	715	-756
-4	8	4	229	213	-2	2	13	255	-247	-1	3	14	350	-408	1	9	3	<201	-170	2	4	4	1059	-1096
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-4	8	6	<200	212	-2	2	15	440	483	-1	3	16	251	305	1	9	5	329	349	2	4	6	876	961
-4	8	7	421	460	-2	2	16	<202	56	-1	3	17	<209	-25	1	9	6	<205	166	2	4	7	663	661
-4	8	8	242	237	-2	2	17	228	-184	-1	1	1	1146	1189	1	9	7	288	242	2	4	8	953	1090
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-4	8	10	<208	160	-2	4	2	<150	56	-1	1	3	307	349	1	9	9	<209	-71	2	4	10	<176	141
-4	8	11	<206	58	-2	4	3	248	236	-1	1	4	637	-747	1	9	10	<212	-17	2	4	11	207	134
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-4	10	2	<207	-56	-2	4	5	<153	167	-1	1	6	961	-1061	1	7	1	311	-290	2	4	13	293	-349
-4	10	3	202	32	-2	4	6	351	-418	-1	1	7	289	270	1	7	2	193	187	2	4	14	<195	105
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-3	9	3	<198	73	-2	4	12	738	-898	-1	1	13	<185	88	1	7	8	239	175	2	6	3	252	-223
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-3	9	5	215	-10	-2	4	14	301	327	-1	1	15	<185	123	1	7	10	226	77	2	6	5	894	-967
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-3	9	8	<203	-89	-2	6	1	<167	-118	-1	1	18	<226	-191	1	7	13	<214	-86	2	6	8	<187	126
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-3	7	5	195	-202	-2	6	7	<176	57	0	0	12	329	-284	1	5	5	<163	180	2	6	14	244	197
-3	7	6	221	-203	-2	6	8	476	-495	0	0	14	<196	166	1	5	6	817	894	2	8	0	346	-350
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-3	7	13	<211	-137	-2	6	15	228	214	0	2	4	512	568	1	5	13	<200	234	2	8	7	227	-218
-3	5	1	298	287	-2	8	1	589	-574	0	2	5	257	-201	1	5	14	<196	-61	2	8	8	265	-268
-3	5	2	329	332	-2	8	2	<185	29	0	2	6	1694	-1829	1	5	15	<210	175	2	8	9	<197	98
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-3	5	13	<194	-80	-2	10	1	<200	32	0	2	17	222	160	1	3	9	385	-460	3	9	0	<201	-107
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-3	3	1	622	644	-2	10	4	434	-448	0	4	2	297	292	1	3	12	212	104	3	9	3	<206	182
-3	3	2	378	378	-2	10	5	<212	-108	0	4	3	214	235	1	3	13	<192	-115	3	9	4	<196	96
-3	3	3	<151	114	-2	10	6	<230	-305	0	4	4	<154	154	1	3	14	<192	-176	3	9	5	307	322
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-3	3	6	<149	14	-1	9	3	219	-256	0	4	7	607	630	1	3	17	356	370	3	9	8	<202	49
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-3	3	10	514	539	-1	9	7	335	303	0	4	11	442	427	1	1	3	674	-713	3	7	2	304	295
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-3	3	15	<201	-99	-1	7	2	400	375	0	4	16	296	-338	1	1	8	<163	118	3	7	7	726	-769
-3	3	16	349	368	-1	7	3	311	-311	0	6	0	492	480	1	1	9	227	-217	3	7	8	<187	-135
-3	3	17	219	-279	-1	7	4	233	147	0	6	1	632	648	1	1	10	439	-489	3	7	9	232	-286
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Table 2. Continued.

H	K	L	F(O)	F(C)
3	7	11	4194	3
3	7	12	222	51
3	7	13	4205	42
3	5	0	232	227
3	5	1	603	570
3	5	2	659	-666
3	5	3	525	560
3	5	4	1255	-1298
3	5	5	530	-571
3	5	6	4167	41
3	5	7	205	-161
3	5	8	4169	52
3	5	9	232	-257
3	5	10	4182	40
3	5	11	289	-321
3	5	12	228	282
3	5	13	266	312
3	5	14	4204	249
3	5	15	4205	-96
3	3	0	1214	-1225
3	3	1	405	-404
3	3	2	854	-865
3	3	3	348	401
3	3	4	332	-379
3	3	5	771	766
3	3	6	1362	-1503
3	3	7	349	-351
3	3	8	4165	142
3	3	9	359	-403
3	3	10	1094	1122
3	3	11	4175	103
3	3	12	221	-175
3	3	13	4180	-122
3	3	14	4201	200
3	3	15	313	-329
3	3	16	4224	79
3	3	17	4224	71
3	1	0	1119	-1061
3	1	1	859	801
3	1	2	1018	963
3	1	3	1179	-1143
3	1	4	1935	-1863
3	1	5	547	532
3	1	6	301	-275
3	1	7	237	239
3	1	8	226	-257
3	1	9	295	259
3	1	10	4172	112
3	1	11	376	-377
3	1	12	4185	-4
3	1	13	535	-536
3	1	14	4196	54
3	1	15	515	554
3	1	16	401	464
3	1	17	4214	-63
4	0	0	237	-258
4	0	2	517	-488
4	0	4	193	205
4	0	6	203	179
4	0	8	355	-345
4	0	10	2645	-2662
4	0	12	1842	-1824
4	0	14	526	500
4	0	16	4205	-27
4	2	0	3002	-2847
4	2	1	1570	-1527
4	2	2	4156	139
4	2	3	4151	-100
4	2	4	1010	984
4	2	5	924	-656
4	2	6	4152	4
4	2	7	631	712
4	2	8	1107	-1166
4	2	9	175	112
4	2	10	183	-165
4	2	11	4178	-122
4	2	12	4179	-131
4	2	13	4183	90
4	2	14	4185	-81
4	2	15	290	-315
4	2	16	326	360
4	2	17	4211	114
4	4	0	1105	1076
4	4	1	848	-887
4	4	2	723	673
4	4	3	255	-275
4	4	4	767	-823
4	4	5	1468	1552
4	4	6	406	-402
4	4	7	188	210
4	4	8	665	-712
4	4	9	4172	-89
4	4	10	4176	-195
4	4	11	4173	-35
4	4	12	321	305
4	4	13	4205	-123
4	4	14	215	134
4	4	15	4203	-135
4	4	16	4210	-135
4	4	17	4167	124
4	6	1	547	535
4	6	2	187	-165
4	6	3	4170	31
4	6	4	486	-519
4	6	5	4178	-42
4	6	6	1085	-1223
4	6	7	4175	76
4	6	8	237	-213
4	6	9	4181	-83
4	6	10	194	101
4	6	11	444	-493
4	6	12	226	146
4	6	13	4204	-190
4	6	14	308	298
4	8	0	199	153
4	8	1	4181	-40
4	8	2	4187	-135
4	8	3	252	239
4	8	4	464	-455
4	8	5	232	229
4	8	6	292	-298
4	8	7	4205	181
4	8	8	4204	-154
4	8	9	420	-386
4	8	10	4213	-106
4	8	11	4212	-76
4	10	0	4209	61
4	10	1	4208	-63
4	10	2	221	-137
4	10	3	4210	-141
4	10	4	4214	-178
4	10	5	4204	-29
4	10	6	304	300
5	9	0	4193	-40
5	9	1	4194	17
5	9	2	4192	-68
5	9	3	4198	83
5	9	4	4204	147
5	9	5	265	195
5	9	6	4206	172
5	9	7	4205	-41
5	9	8	225	176
5	9	9	225	87
5	7	0	4177	-11
5	7	1	288	293
5	7	2	4174	-90
5	7	3	4179	18
5	7	4	4183	185
5	7	5	4186	-167
5	7	6	4183	25
5	7	7	4189	-133
5	7	8	310	-325
5	7	9	303	338
5	7	10	4195	53
5	7	11	4203	-52
5	7	12	4206	-102
5	7	13	4217	-110
5	5	0	1656	1580
5	5	1	679	648
5	5	2	359	-350
5	5	3	4167	-115
5	5	4	501	-492
5	5	5	414	-406
5	5	6	318	283
5	5	7	567	608
5	5	8	277	-316
5	5	9	4182	-52
5	5	10	610	-681
5	5	11	4186	176
5	5	12	4186	-38
5	5	13	4193	70
5	5	14	206	194
5	5	15	283	-285
5	3	0	4149	53
5	3	1	209	-209
5	3	2	877	858
5	3	3	422	-428
5	3	4	369	378
5	3	5	262	-289
5	3	6	1179	-1182
5	3	7	237	-203
5	3	8	362	335
5	3	9	473	479
5	3	10	366	-322
5	3	11	804	799
5	3	12	444	-394
5	3	13	4187	-54
5	3	14	219	-242
5	3	15	542	-543
5	3	16	227	-198
5	1	0	1641	1577
5	1	1	884	859
5	1	2	1954	-1903
5	1	3	632	-582
5	1	4	182	130
5	1	5	982	-915
5	1	6	1656	1492
5	1	7	4147	-71
5	1	8	187	-131
5	1	9	1369	1292
5	1	10	1123	-1036
5	1	11	296	-277
5	1	12	836	-753
5	1	13	389	-347
5	1	14	424	-398
5	1	15	474	430
5	1	16	4202	-13
5	1	17	377	-339
6	0	0	310	-326
6	0	2	356	317
6	0	4	235	318
6	0	6	172	-91
6	0	8	805	739
6	0	10	908	-838
6	0	12	221	174
6	0	14	562	-547
6	0	16	924	-895
6	2	0	840	790
6	2	1	1305	1288
6	2	2	735	698
6	2	3	1137	-1075
6	2	4	1306	1173
6	2	5	1762	-1573
6	2	6	4153	-64
6	2	7	215	209
6	2	8	347	320
6	2	9	274	259
6	2	10	1178	1054
6	2	11	1118	1059
6	2	12	271	241
6	2	13	571	503
6	2	14	598	-585
6	2	15	4195	16
6	2	16	4210	-130
6	2	17	230	-159
6	4	0	3491	3303
6	4	1	567	-452
6	4	2	750	711
6	4	3	568	555
6	4	4	4157	22
6	4	5	4155	76
6	4	6	289	267
6	4	7	847	-831
6	4	8	4170	-54
6	4	9	332	290
6	4	10	4177	52
6	4	11	650	665
6	4	12	4183	13
6	4	13	373	395
6	4	14	4194	-113
6	4	15	373	-306
6	4	16	264	-98
6	6	0	4254	100
6	6	1	4186	17
6	6	2	557	508
6	6	3	327	315
6	6	4	4181	147
6	6	5	4185	-150
6	6	6	340	-345
6	6	7	290	-234
6	6	8	4192	131
6	6	9	4185	8
6	6	10	4201	-128
6	6	11	4202	-173
6	6	12	373	370
6	6	13	4206	-49
6	6	14	4202	-26
6	8	0	194	-120
6	8	1	232	-72
6	8	2	4180	-77
6	8	3	403	364
6	8	4	4198	183
6	8	5	571	591
6	8	6	4189	-55
6	8	7	299	280
6	8	8	4197	-36
6	8	9	359	-286
6	8	10	4209	24
6	8	11	264	201
6	10	0	4202	53
6	10	1	4202	-52
6	10	2	4221	-226
6	10	3	4209	97
6	10	4	264	237
6	10	5	4214	-111
7	9	0	4190	14
7	9	1	522	-522
7	9	2	4199	179
7	9	3	4192	-88
7	9	4	4199	-95
7	9	5	383	333
7	9	6	225	176
7	9	7	4203	93
7	9	8	354	286
7	7	0	268	-218
7	7	1	764	-736
7	7	2	440	401
7	7	3	4181	-74
7	7	4	501	504
7	7	5	4181	45
7	7	6	303	-254
7	7	7	361	363
7	7	8	4193	-69
7	7	9	339	280
7	7	10	647	642
7	7	11	417	-440
7	7	12	243	-33
7	5	0	316	291
7	5	1	210	-167
7	5	2	4160	70
7	5	3	805	-746
7	5	4	683	642
7	5	5	310	326
7	5	6	715	673
7	5	7	650	620
7	5	8	231	-181
7	5	9	195	-200
7	5	10	358	-325
7	5	11	4192	-113
7	5	12	240	230
7	5	13	315	-320
7	5	14	490	483
7	5	15	4218	153
7	3	0	1974	-1892
7	3	1	1116	-1043
7	3	2	1517	1461
7	3	3	482	-421
7	3	4	1055	989
7	3	5	246	211
7	3	6	4153	24
7	3	7	306	-273
7	3	8	283	271
7	3	9	4170	-74
7	3	10	4176	156
7	3	11	366	359
7	3	12	667	633
7	3	13	285	244
7	3	14	4194	-96
7	3	15	4200	-107
7	3	16	4207	139
7	1	0	4149	58
7	1	1	4151	-2
7	1	2	4149	82
7	1	3	162	-110
7	1	4	872	801
7	1	5	1029	-987
7	1	6	458	427
7	1	7	274	242
7	1	8	277	275
7	1	9	713	694
7	1	10	917	843
7	1	11	787	-720
7	1	12	425	392
7	1	13	310	287
7	1	14	209	129
7	1	15	4467	427
7	1	16	4205	-134
7	1	17	326	-307
8	0	0	485	-487
8	0	2	1844	-1832
8	0	4	3717	-3558
8	0	6	516	552
8	0	8	917	872
8	0	10	4175	106
8	0	12	817	806
8	0	14	280	216
8	0	16	289	-186
8	2	0	1863	-1763
8	2	1	1970	1924
8	2	2	753	-738
8	2	3	866	-843
8	2	4	290	-292
8	2	5	386	-365
8	2	6	4155	-114
8	2	7	414	-374
8	2	8	944	852
8	2	9	4167	15
8	2	10	1397	1284
8	2	11	730	669
8	2	12	4183	-67
8	2	13	4187	116
8	2	14	249	198
8	2	15	384	324
8	2	16	288	309
8	4	0	296	-260
8	4	1	4161	116
8	4	2	552	-533
8	4	3	293	292
8	4	4	452	400
8	4	5	1094	-806
8	4	6	621	608
8	4	7	606	-615
8	4	8	4167	0
8	4	9	4181	136
8	4	10	237	-254
8	4	11	4179	91
8	4	12	622	-599
8	4	13	212	133
8	4	14	329	302
8	4	15	471	-387
8	6	0	970	-933
8	6	1	4169	19
8	6	2	211	202
8	6	3	745	-800
8	6	4	4176	-55
8	6	5	791	-816
8	6	6	515	521
8	6	7	224	148
8	6	8	215	114
8	6	9	244	200
8	6	10	224	-202
8	6	11	580	-564
8	6	12	246	167
8	6	13	4211	-189
8	8	0	227	-77
8	8	1	4198	-21
8	8	2	4192	137
8	8	3	4191	-112
8	8	4	210	-216
8	8	5	511	518
8	8	6	263	-247
8	8	7	204	111
8	8	8	4211	118
8	8	9	4204	-57
8	8	10	239	130
8	10	0	4209	139
8	10	1	4203	-1
8	10	2	4206	4
8	10	3	4213	-43
8	10	4	4218	137
9	9	0	243	205
9	9	1	4203	5
9	9	2	4203	-106
9	9	3	260	269
9	9	4	432	-399
9	9	5	4217	129
9	9	6	4209	108
9	9	7	255	-151
9	7	0	325	-282
9	7	1	4181	37
9	7	2	288	-268
9	7	3	388	396
9	7	4	4184	-93
9	7	5	4194	-118
9	7	6	448	-451
9	7	7	4189	0
9	7	8	4196	-1
9	7	9	4200	-89
9	7	10	262	242
9	7	11	4202	-73
9	7	12	4212	-182
9	5	0	170	129
9	5	1	356	335
9	5	2	522	-552
9	5	3	4166	87
9	5	4	431	-449
9	5	5	702	718
9	5	6	4179	139
9	5	7	361	-359
9	5	8	4176	15
9	5	9	502	-536
9	5	10	381	-459
9	5	11	4198	-65
9	5	12	281	-243
9	5	13	4197	-38
9	5	14	4226	279
9	3	0	678	-658
9	3	1	245	217
9	3	2	598	588
9	3	3	1130	1127
9	3	4	1142	-1130
9	3	5	425	432
9	3	6	407	-440
9	3	7	735	-773
9	3	8	236	-260
9	3	9	633	-654
9	3	10	4195	212
9	3	11	4180	58
9	3	12	4189	1
9	3	13	220	255
9	3	14	370	-382
9	3	15	4222	1
9	3	16	283	353
9	1	0	663	665
9	1	1	850	865
9	1	2	956	-933
9	1	3	929	955
9	1	4	1417	-1418
9	1	5	970	-959
9	1	6	995	-972
9	1	7	225	238
9	1	8	195	-191
9	1	9	4164	-49
9	1	10	329	331
9	1	11	769	-730
9	1	12	4184	76
9	1	13	322	303
9	1	14	440	398
9	1	15	290	238
9	1	16	258	-266
10	0	0	1533	1566
10	0	2	528	-538
10	0	4	838	-803
10	0	6	4162	146
10	0	8	1831	-1786
10	0	10	872	-872
10	0	12	822	798
10	0	14	230	176
10	0	16	4209	-132
10	2	0	514	-492
10	2	1	707	701
10	2	2	532	552
10	2	3	4152	-116
10	2	4	636	617
10	2	5	1011	1032
10	2	6	969	-998
10	2	7	367	343
10	2	8	424	-416
10	2	9	4168	-59
10	2	10	236	-140
10	2	11	718	-709
10	2	12	4195	-150
10	2	13	311	-286
10	2	14	4198	32
10	2	15	813	779
10	2	16	4207	-39
10	4	0	4161	-116
10	4	1	404	-369
10	4	2	705	-688
10	4	3	871	885

Table 2. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
10	4	4	288	282	12	0	4	378	422	13	1	2	627	644	15	3	13	<212	-82	17	1	6	539	537
10	4	5	442	458	12	0	6	701	662	13	1	3	1039	-1069	15	1	0	279	-286	17	1	7	219	-130
10	4	6	<165	-19	12	0	8	498	-513	13	1	4	640	676	15	1	1	223	164	17	1	8	313	313
10	4	7	<172	104	12	0	10	<223	104	13	1	5	<164	-62	15	1	2	336	-314	17	1	9	<200	193
10	4	8	389	-425	12	0	12	266	241	13	1	6	693	685	15	1	3	1129	-1152	17	1	10	216	198
10	4	9	574	-571	12	0	14	275	187	13	1	7	717	720	15	1	4	631	-603	17	1	11	221	-203
10	4	10	1151	-1160	12	2	0	184	202	13	1	8	<173	-22	15	1	5	236	265	17	1	12	<217	-168
10	4	11	<196	-56	12	2	1	667	-649	13	1	9	<181	-176	15	1	6	201	-173	17	1	13	<223	208
10	4	12	673	-678	12	2	2	1886	1900	13	1	10	322	-339	15	1	7	274	275	18	0	0	<278	76
10	4	13	<200	-29	12	2	3	690	699	13	1	11	281	-201	15	1	8	336	-336	18	0	2	329	319
10	4	14	<203	21	12	2	4	629	596	13	1	12	443	398	15	1	9	<187	-183	18	0	4	577	-573
10	4	15	243	-256	12	2	5	863	874	13	1	13	274	-267	15	1	10	229	-152	18	0	6	589	-630
10	6	0	424	-410	12	2	6	644	-647	13	1	14	484	458	15	1	11	<196	-111	18	0	8	321	274
10	6	1	210	-141	12	2	7	<167	46	13	1	15	<208	107	15	1	12	<199	87	18	0	10	337	347
10	6	2	<182	-161	12	2	8	596	601	14	0	0	<322	-311	15	1	13	<203	39	18	0	12	666	645
10	6	3	442	-411	12	2	9	<184	27	14	0	2	1808	-1835	15	1	14	420	-395	18	2	0	620	651
10	6	4	333	320	12	2	10	694	682	14	0	4	325	314	16	0	0	536	-534	18	2	1	329	325
10	6	5	439	-427	12	2	11	734	-765	14	0	6	1085	1048	16	0	2	642	-684	18	2	2	842	844
10	6	6	615	650	12	2	12	<200	171	14	0	8	<179	-112	16	0	4	1009	1021	18	2	3	.722	-734
10	6	7	326	304	12	2	13	<199	-144	14	0	10	423	-409	16	0	6	<188	-1	18	2	4	<182	69
10	6	8	401	-469	12	2	14	<222	<248	14	0	12	638	-656	16	0	8	351	-357	18	2	5	544	-568
10	6	9	<194	94	12	2	15	293	283	14	0	14	523	470	16	0	10	972	-978	18	2	6	<186	49
10	6	10	342	-303	12	4	0	<183	9	14	2	0	980	-982	16	0	12	429	-426	18	2	7	<188	169
10	6	11	277	-221	12	4	1	335	-310	14	2	1	303	-286	16	0	14	<230	-99	18	2	8	373	414
10	6	12	<206	53	12	4	2	253	248	14	2	2	<173	-132	16	2	0	403	-388	18	2	9	377	-354
10	6	13	<214	111	12	4	3	547	607	14	2	3	268	-254	16	2	1	<168	-86	18	2	10	646	720
10	8	0	<192	90	12	4	4	285	-293	14	2	4	497	-516	16	2	2	536	564	18	2	11	242	194
10	8	1	239	-185	12	4	5	688	675	14	2	5	692	-709	16	2	3	434	-411	18	2	12	<213	73
10	8	2	<197	172	12	4	6	416	-439	14	2	6	345	328	16	2	4	736	782	18	4	0	414	393
10	8	3	<198	97	12	4	7	<179	-52	14	2	7	375	363	16	2	5	949	-1011	18	4	1	282	-257
10	8	4	<201	-120	12	4	8	284	302	14	2	8	514	535	16	2	6	<179	112	18	4	2	236	217
10	8	5	572	559	12	4	9	588	-566	14	2	9	432	432	16	2	7	196	200	18	4	3	245	250
10	8	6	382	-366	12	4	10	<193	81	14	2	10	<197	176	16	2	8	333	-297	18	4	4	<208	213
10	8	7	<238	-37	12	4	11	327	280	14	2	11	564	-591	16	2	9	<187	136	18	4	5	<193	-88
10	8	8	<202	-15	12	4	12	229	178	14	2	12	<206	-189	16	2	10	289	-288	18	4	6	516	508
10	8	9	<210	90	12	4	13	222	-86	14	2	13	<208	-13	16	2	11	224	219	18	4	7	592	-597
10	10	0	374	369	12	4	14	302	-229	14	2	14	<214	27	16	2	12	268	-276	18	4	8	<195	-37
10	10	1	238	-16	12	6	0	317	272	14	4	0	<172	-98	16	2	13	295	281	18	4	9	<212	135
11	9	0	353	346	12	6	1	322	329	14	4	1	<170	-44	16	4	0	231	145	18	4	10	<210	159
11	9	1	<232	0	12	6	2	<183	-30	14	4	2	893	-909	16	4	1	249	-190	18	4	11	<217	110
11	9	2	<232	-19	12	6	3	469	-486	14	4	3	<175	40	16	4	2	<181	6	18	6	0	<202	-79
11	9	3	<232	30	12	6	4	425	400	14	4	4	1570	-1582	16	4	3	<183	-56	18	6	1	<207	-175
11	9	4	<138	35	12	6	5	619	-616	14	4	5	463	483	16	4	4	199	-54	18	6	2	492	491
11	9	5	<203	-28	12	6	6	419	462	14	4	6	<137	-172	16	4	5	269	269	18	6	3	<204	-73
11	9	6	<209	78	12	6	7	220	191	14	4	7	242	-334	16	4	6	205	-164	18	6	4	<220	232
11	7	0	210	-211	12	6	8	222	191	14	4	8	<186	128	16	4	7	<196	-158	18	6	5	243	230
11	7	1	503	521	12	6	9	<209	91	14	4	9	<187	-58	16	4	8	640	-694	18	6	6	<212	121
11	7	2	302	225	12	6	10	543	643	14	4	10	<198	-72	16	4	9	325	398	18	6	7	<209	50
11	7	3	223	-189	12	6	11	<210	41	14	4	11	419	451	16	4	10	364	-415	18	6	8	235	234
11	7	4	276	229	12	6	12	<209	96	14	4	12	300	374	16	4	11	327	303	19	7	0	<216	81
11	7	5	674	-658	12	8	0	<205	27	14	4	13	381	<396	16	4	12	<222	204	19	7	1	427	-406
11	7	6	394	-392	12	8	1	<200	-58	14	6	0	<192	132	16	6	0	275	284	19	7	2	<221	163
11	7	7	216	103	12	8	2	355	317	14	6	1	502	510	16	6	1	<191	107	19	7	3	326	-327
11	7	8	270	230	12	8	3	260	296	14	6	2	508	-534	16	6	2	<196	172	19	7	4	<212	-7
11	7	9	338	360	12	8	4	<199	101	14	6	3	216	-195	16	6	3	<204	-187	19	5	0	406	-430
11	7	10	232	87	12	8	5	<201	-33	14	6	4	<189	-36	16	6	4	309	291	19	5	1	222	85
11	7	11	416	479	12	8	6	<209	155	14	6	5	270	-242	16	6	5	403	391	19	5	2	364	-363
11	5	0	<172	130	12	8	7	500	-537	14	6	6	<192	-59	16	6	6	517	<561	19	5	3	523	-543
11	5	1	465	461	12	8	8	281	362	14	6	7	<198	132	16	6	7	655	731	19	5	4	396	452
11	5	2	525	-506	13	5	0	<209	106	14	6	8	<201	57	16	6	8	<217	-143	19	5	5	<202	-131
11	5	3	246	-165	13	9	1	219	-105	14	6	9	<205	60	16	6	9	<218	105	19	5	6	365	384
11	5	4	243	224	13	9	2	<214	-68	14	6	10	354	434	16	6	10	<214	-82	19	5	7	247	205
11	5	5	361	-374	13	9	3	<204	-52	14	6	11	<213	-7	16	8	0	324	263	19	5	8	331	-345
11	5	6	531	519	13	9	4	<224	181	14	8	0	280	-203	16	8	1	<225	-205	19	5	9	<215	-117
11	5	7	296	-320	13	7	0	375	-356	14	8	1	<198	47	16	8	2	<209	12	19	3	0	721	-737
11	5	8	219	185	13	7	1	<191	-102	14	8	2	226	139	16	8	3	383	391	19	3	1	<185	-94
11	5	9	568	635	13	7	2	396	375	14	8	3	<210	195	16	8	4	<231	-237	19	3	2	376	395
11	5	10	424	-439	13	7	3	512	-521	14	8	4	<204	-7	16	8	5	<226	201	19	3	3	341	379
11	5	11	250	184	13	7	4	<194	-107	14	8	5	245	-236	17	7	0	209	163	19	3	4	<192	-120
11	5	12	<202	-31	13	7	5	<197	53	14	8	6	<212	49	17	7	1	<204	-33	19	3	5	252	244
11	5	13	<206	-113	13	7	6	<199	-33	14	8	7	292	-293	17	7	2	313	374	19	3	6	251	203
11	3	0	434	419	13	7	7	<204	123	15	7	0	<193	-76	17	7	3	<202	-93	19	3	7	324	-322
11	3	1	<158	-169	13	7	8	416	410	15	7	1	<204	190	17	7	4	<202	46	19	3	8	349	387
11	3	2	316	-273	13	7	9	<214	-185	15	7	2	<199	159	17	7	5	<201	-1	19	3	9	<202	-108
11	3	3	581	552	13	7	10	<213	154	15	7	3	382	401	17	7	6	<210	57	19	3	10	<210	-130
11	3	4	328	311	13	5	0	<178	35	15	7	4	407	<378	17	7	7	<211	-2	19	3	11	<206	-12
11	3	5	231	238	13	5	1	769	-790	15	7	5	386	443	17	5	0	309	275	19	1	0	<176	-55
11	3	6	588	534	13	5	2	<173	21	15	7	6	224	-201	17	5	1	287	294	19	1	1	287	-234
11	3	7	<171	-124	13	5	3	426	-483	15	7	7	251	-217	17	5	2	<187	84	19	1	2	477	-471
11	3	8	233	-152	13	5	4	<135	-209	15	7	8	<206	33	17	5	3	<205	-174	19	1	3	239	-313
11	3	9	<173	-40	13	5	5	<161	-12	15	5	0	361	336	17	5	4	548	543	19	1	4	<182	-23
11	3	10	167	87	13	5	6	224	237	15	5	1	<181	60	17	5	5	365	<400	19	1	5	<184	0
11	3	11	266	242	13	5	7	283	245	15	5	2	425	-411	17	5	6	375	335	19	1	6	<196	195
11	3	12	400	-407	13	5	8	<193	123	15	5	3	673	664	17	5	7	278	238	19	1	7	246	175
11	3	13	<197	-36	13	5	9	<199	153	15	5	4	275	-272	17	5	8	431	-410	19	1	8	254	-271
11	3	14	640	-619	13	5	10	<209	220	15	5	5	211	-115	17	5	9	<210	-125	19	1	9	256	-202
11	3	15	245	-155	13	5	11	525	-545	15	5	6	<201	-174	17	5	10	372	-326	19	1	10	345	304
11	1	0	868	866	13	5	12	474	496	15	5	7	<201	61	17	3	0	<176	71	19	1	11	538	-588
11	1	1	525	500	13	5	13	<226	95	15	5	8	651	-687	17	3	1	463	484	19	1	12	386	355
11	1	2	340	368	13	3	0	<169	42	15	5	9	<212	-198	17	3	2	649	686	20	0	0	307	-392
11	1	3	195	-200	13	3	1	748	-740	15	5	10	<219	-185	17	3	3	240	298	20	0	2	1007	-1052
11	1	4	696	679	13	3	2	<172	158	15	5	11	347	-356	17	3	4	229	223	20	0	4	1326	-1371
11	1	5	344	-316	13	3	3	272	-292	15	5	12	342	389	17	3	5	433	-444	20	0	6	386	-415
11	1	6	194	-154	13	3	4	256	220	15	3	0	254	270	17	3	6	216	-236	20	0	8	245	-224
11	1	7	914	942	13	3	5	254	181	15	3	1	253	-244	17	3	7	509	-546	20	0	10	<208	-2
11	1	8	<173	51	13	3	6	189	-74	15	3	2	331	-331	17	3	8	778	803	20	2	0	206	94
11	1	9	473	-478	13	3	7	263	265	15	3	3	321	372	17	3	9	390	340	20	2	1	277	279
11	1	10	738	-726	13	3	8	209	131	15	3	4	263	<266	17	3	10	239	-199	20	2	2	514	-528
11	1	11	477	-496	13	3	9	394	334	15	3	5	266	278	17	3	11	562	614	20	2	3	<188	-25
11	1	12	303	-244	13	3	10	822	868	15	3	6	1007	-1018	17	3	12	431	-474	20	2	4	447	-435
11	1	13	134	142	13	3	11	<196	98	15	3	7	265	-249	17	1	0	515	493	20	2	5	422	436
11	1	14	<168	-59	13	3	12	<193	-90	15	3	8	<183	3	17	1	1	319	278	20	2	6	225	-170
11	1	15	<211	99	13	3	13	378	-405	15	3	9	<205	82	17	1	2	324	-327	20	2	7	<192	-71
11	1	16	<210	-112	13	3	14	392	358	15	3	10	269	216	17	1	3	659	-660	20	2	8	334	334
12	0	0	2568	2858	13	1	0	1125	-1189	15	3	11	429	<452	17	1	4	<181	-73	20	2	9	368	-366
12	0	2	413	349	13	1	1	391	357	15	3	12	396	-318	17	1	5	325	-294	20	2	10	<225	245

Table 2. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
20	4	0	345	-315	21	5	6	285	-258	22	0	2	<206	4	23	5	0	<206	9	24	2	0	248	225
20	4	1	235	229	21	3	0	<203	73	22	0	4	290	278	23	5	1	<206	79	24	2	1	235	-305
20	4	2	682	-758	21	3	1	<197	71	22	0	6	246	249	23	5	2	<220	-168	24	2	2	563	606
20	4	3	566	587	21	3	2	269	336	22	0	8	680	-707	23	3	0	540	565	24	2	3	<201	119
20	4	4	<211	144	21	3	3	824	879	22	2	0	253	251	23	3	1	<204	-107	24	2	4	<212	54
20	4	5	347	-348	21	3	4	500	-533	22	2	1	232	-99	23	3	2	<206	189	24	2	5	316	342
20	4	6	272	241	21	3	5	367	386	22	2	2	534	520	23	3	3	<213	164	24	2	6	<212	48
20	4	7	315	-327	21	3	6	253	-295	22	2	3	<209	-146	23	3	4	<214	-211	24	4	0	<211	108
20	4	8	<226	-17	21	3	7	<206	-125	22	2	4	369	367	23	3	5	<211	-58	24	4	1	<209	-33
20	4	9	<206	78	21	3	8	<201	23	22	2	5	730	762	23	3	6	398	431	24	4	2	<213	40
20	6	0	531	-559	21	3	9	<213	-81	22	2	6	<204	-39	23	1	0	225	146	24	4	3	<215	91
20	6	1	258	208	21	1	0	327	299	22	2	7	391	396	23	1	1	238	-181	25	3	0	<209	59
20	6	2	<205	-14	21	1	1	<183	-84	22	2	8	218	126	23	1	2	305	319	25	3	1	<208	-25
20	6	3	295	-282	21	1	2	<190	77	22	2	9	<210	-1	23	1	3	401	-446	25	3	2	279	-229
20	6	4	251	72	21	1	3	<192	57	22	4	0	<202	31	23	1	4	387	353	25	1	0	717	-743
20	6	5	310	-233	21	1	4	<201	186	22	4	1	<198	-12	23	1	5	<206	-57	25	1	1	214	-41
21	5	0	<210	-163	21	1	5	<193	-107	22	4	2	<202	-53	23	1	6	286	277	25	1	2	<228	-237
21	5	1	<206	54	21	1	6	760	-759	22	4	3	354	360	23	1	7	431	443	25	1	3	576	-625
21	5	2	500	-499	21	1	7	359	398	22	4	4	252	264	23	1	8	320	216	25	1	4	<219	110
21	5	3	<201	0	21	1	8	500	-542	22	4	5	<206	-63	24	0	0	262	-198	25	1	5	<213	-63
21	5	4	<202	-54	21	1	9	<223	-211	22	4	6	<214	-123	24	0	2	<210	-56	26	0	0	274	-267
21	5	5	455	429	21	1	10	<213	-81	22	4	7	<215	109	24	0	4	522	554	26	0	2	640	-653
					22	0	0	274	-308	22	6	0	<208	-3	24	0	6	462	406	26	2	0	220	30

Table 3. Bond lengths with the corresponding standard deviations in parentheses. Bond lengths corrected for rigid body motion are listed in brackets.

Bond		Bond	
S(1) – S(2)	2.063(3) [2.065] Å	C(11) – C(12)	1.538(12)[1.549] Å
S(1) – C(1)	1.754(7) [1.765]	C(11) – C(13)	1.518(13)[1.528]
S(2) – S(3)	2.863(3) [2.865]	C(11) – C(14)	1.515(13)[1.521]
S(2) – C(3)	1.762(8) [1.774]	C(2) – H(2)	0.93(6)
S(3) – S(4)	2.062(3) [2.064]	C(8) – H(81)	1.06(7)
S(3) – C(5)	1.761(7) [1.773]	C(8) – H(82)	0.95(9)
S(4) – C(7)	1.727(9) [1.739]	C(9) – H(91)	1.00(11)
S(5) – C(7)	1.667(9) [1.670]	C(9) – H(92)	1.11(10)
C(1) – C(2)	1.332(11)[1.336]	C(10) – H(101)	1.01(11)
C(1) – C(11)	1.523(10)[1.525]	C(10) – H(102)	1.11(15)
C(2) – C(3)	1.436(11)[1.439]	C(12) – H(121)	0.96(9)
C(3) – C(4)	1.390(10)[1.394]	C(12) – H(122)	1.13(10)
C(4) – C(5)	1.419(10)[1.422]	C(12) – H(123)	0.89(10)
C(4) – C(8)	1.512(12)[1.523]	C(13) – H(131)	1.05(9)
C(5) – C(6)	1.403(11)[1.406]	C(13) – H(132)	0.90(8)
C(6) – C(7)	1.403(11)[1.407]	C(13) – H(133)	0.93(7)
C(6) – C(10)	1.508(12)[1.518]	C(14) – H(141)	0.92(11)
C(8) – C(9)	1.525(13)[1.528]	C(14) – H(142)	0.97(10)
C(9) – C(10)	1.498(14)[1.503]	C(14) – H(143)	0.99(10)

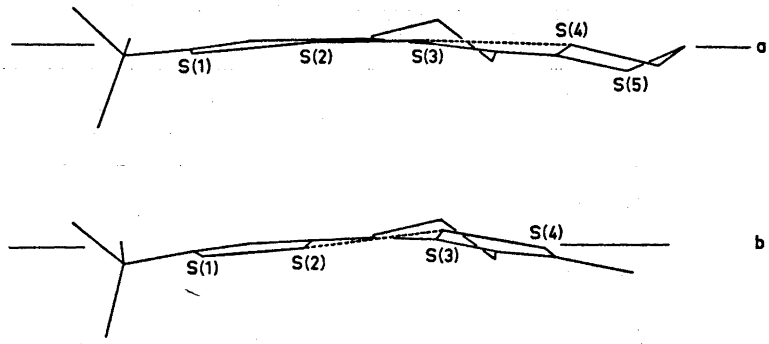


Fig. 1. Atomic deviations from the least-squares planes through rings A, B, and C for molecules II and IV, respectively.

Table 4. Intramolecular bond angles(°) with the corresponding standard deviations in parentheses.

Angle		Angle	
C(1) – S(1) – S(2)	94.9(3)	C(8) – C(9) – H(91)	109(6)
S(1) – S(2) – S(3)	177.0(1)	C(8) – C(9) – H(92)	104(5)
S(1) – S(2) – C(3)	96.7(3)	C(10) – C(9) – H(91)	108(6)
S(3) – S(2) – C(3)	83.0(3)	C(10) – C(9) – H(92)	115(5)
S(2) – S(3) – S(4)	162.8(1)	H(91) – C(9) – H(92)	108(8)
S(2) – S(3) – C(5)	84.5(3)	C(6) – C(10) – C(9)	110.5(7)
S(4) – S(3) – C(5)	95.0(3)	C(6) – C(10) – H(101)	110(6)
S(3) – S(4) – C(7)	96.7(3)	C(6) – C(10) – H(102)	107(7)
S(1) – C(1) – C(2)	115.4(5)	C(9) – C(10) – H(101)	122(6)
S(1) – C(1) – C(11)	115.3(5)	C(9) – C(10) – H(102)	109(7)
C(2) – C(1) – C(11)	129.1(6)	H(101) – C(10) – H(102)	96(10)
C(1) – C(2) – C(3)	121.8(6)	C(1) – C(11) – C(12)	108.5(6)
C(1) – C(2) – H(2)	126(3)	C(1) – C(11) – C(13)	108.9(7)
C(3) – C(2) – H(2)	110(3)	C(1) – C(11) – C(14)	110.5(7)
S(2) – C(3) – C(2)	111.1(5)	C(12) – C(11) – C(13)	110.1(8)
S(2) – C(3) – C(4)	124.1(5)	C(12) – C(11) – C(14)	109.3(8)
C(2) – C(3) – C(4)	124.7(6)	C(13) – C(11) – C(14)	109.5(7)
C(3) – C(4) – C(5)	125.4(6)	C(11) – C(12) – H(121)	105(5)
C(3) – C(4) – C(8)	117.8(6)	C(11) – C(12) – H(122)	113(5)
C(5) – C(4) – C(8)	116.7(6)	C(11) – C(12) – H(123)	108(6)
S(3) – C(5) – C(4)	121.3(5)	H(121) – C(12) – H(122)	103(7)
S(3) – C(5) – C(6)	114.6(5)	H(121) – C(12) – H(123)	113(8)
C(4) – C(5) – C(6)	124.0(6)	H(122) – C(12) – H(123)	114(8)
C(5) – C(6) – C(7)	119.0(6)	C(11) – C(13) – H(131)	120(5)
C(5) – C(6) – C(10)	119.1(6)	C(11) – C(13) – H(132)	116(5)
C(7) – C(6) – C(10)	121.9(7)	C(11) – C(13) – H(133)	110(5)
S(4) – C(7) – C(6)	114.7(6)	H(131) – C(13) – H(132)	94(7)
S(4) – C(7) – S(5)	116.6(4)	H(131) – C(13) – H(133)	113(7)
C(6) – C(7) – S(5)	128.6(6)	H(132) – C(13) – H(133)	105(7)
C(4) – C(8) – C(9)	114.5(6)	C(11) – C(14) – H(141)	112(6)
C(4) – C(8) – H(81)	115(4)	C(11) – C(14) – H(142)	112(6)
C(4) – C(8) – H(82)	106(5)	C(11) – C(14) – H(143)	117(5)
C(9) – C(8) – H(81)	114(4)	H(141) – C(14) – H(142)	104(9)
C(9) – C(8) – H(82)	114(5)	H(141) – C(14) – H(143)	101(8)
H(81) – C(8) – H(82)	91(7)	H(142) – C(14) – H(143)	110(8)
C(8) – C(9) – C(10)	112.5(7)		

Table 5. Least-squares planes through each of the five-membered rings, and atomic deviations from these planes.

Equation of planes (X, Y and Z monoclinic coordinates in Å)	Atoms defining the plane and their deviation from the plane (Å)				
(1) $0.42212X + 0.90529Y - 0.05994Z = 7.01010$	S(1) 0.0098	S(2) −0.0066	C(1) −0.0128	C(2) 0.0081	C(3) 0.0016
(2) $0.44249X + 0.89043Y - 0.11988Z = 7.12032$	S(2) 0.0607	S(3) 0.0749	C(3) 0.0606	C(4) 0.0280	C(5) −0.1028
(3) $0.65915X + 0.74275Y - 0.13699Z = 9.23740$	S(3) 0.0140	S(4) −0.0127	C(5) 0.0134	C(6) 0.0033	C(7) 0.0089

Angle between plane (1) and plane (2) 3.7°
Angle between plane (1) and plane (3) 17.0°
Angle between plane (2) and plane (3) 15.1°

The six-membered ring D may be described as a slightly twisted boat with one stem (C(4), C(5), C(6)) flattened.

The most important bond lengths and angles are shown in Fig. 2. The interatomic distances in the sulphur sequence are markedly different from

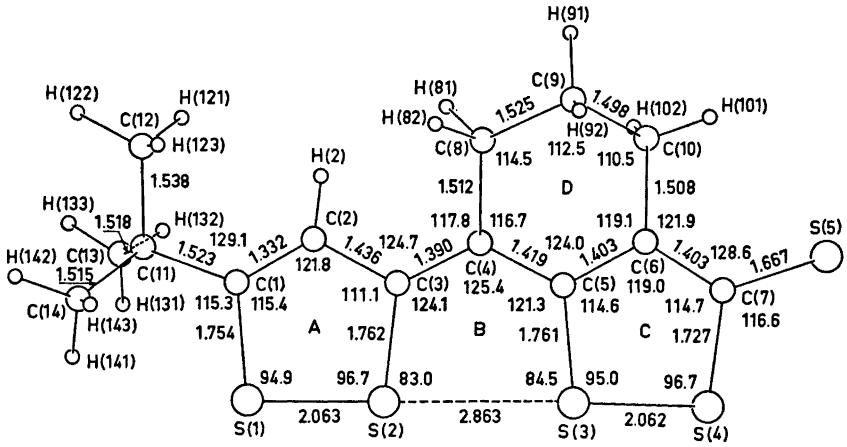
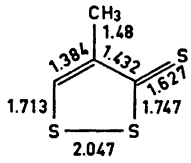
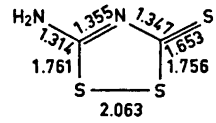


Fig. 2. Bond distances and angles.

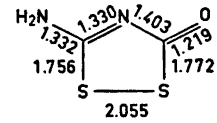
those found in the related molecule II. Chemically, molecule II differs from molecule IV only by having an ethylene group attached to S(4) and S(5). In molecule II, rings A and B apparently constitute a thiathiophthene system, and this system does not seem to be perturbed to any noticeable degree by the close contact to S(4). In the present molecule, IV, the two terminal S–S bonds are both shorter than a sulphur-sulphur single bond of 2.10 Å.[13] The central sulphur-sulphur distance is approximately 0.3 Å longer than the longest S–S bond observed in a thiathiophthene[14] but is still well below the van der Waals distance. A similar sulphur sequence is found in the symmetrical four-sulphur compound I where the S–S distances are 2.03(3), 2.93(3), and 2.00(3) Å.[2] The terminal disulphide rings in the present molecule may be compared with similar isolated unsaturated five-membered disulphides (VI, VII, and VIII).[15–18] The S–S bonds in the range 2.047–2.063 Å are significantly shorter than a single bond and this shortening is assumed to be caused by π-bonding.[19] The cyclic carbon-sulphur bonds in VII and VIII



(VI)



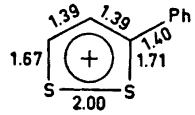
(VII)



(VIII)

are similar to the corresponding bond distances in the present molecule, indicating a comparable amount of π-conjugation in the rings. The S(1) – S(2) and S(3) – S(4) distances are not significantly different from those in the isolated disulphide rings. Thus the close contact of 2.863(3) Å between S(2) and S(3) does not seem to produce a lengthening of the terminal sulphur-sulphur bonds.

The geometry of the five-membered disulphide rings in molecule I may by analogy be compared with that of the 3-phenyl-dithiolium ion (IX).[20]



(IX)

Judging from the C – S distances in the two molecules, there is a comparable amount of π-conjugation in rings A, C and in IX. Thus, the bond lengths of the terminal disulphide groups in I are not significantly different from the S – S bond length expected for a dithiolium ion with this amount of π-bonding.

In each of the four-sulphur compounds I, II, and IV, one of the S – S distances are in the region 2.86 – 2.96 Å. It is not evident in any of these cases that the short contact has brought about a lengthening of an adjacent sulphur-sulphur bond.

There are a few short intramolecular S···H and H···H distances in molecule IV. S(1)···H(141) and S(5)···H(101) are 2.72 and 2.75 Å, respectively, as compared to corresponding distances of 2.71 and 2.51 Å in molecule II. H(2) in the present molecule has short contacts to H(81), H(82), and H(121) of 2.21, 2.07, and 2.30 Å, respectively.

In Fig. 3 the packing of molecules in the unit cell is shown. The molecules are stacked on top of one another across centers of symmetry. Distances

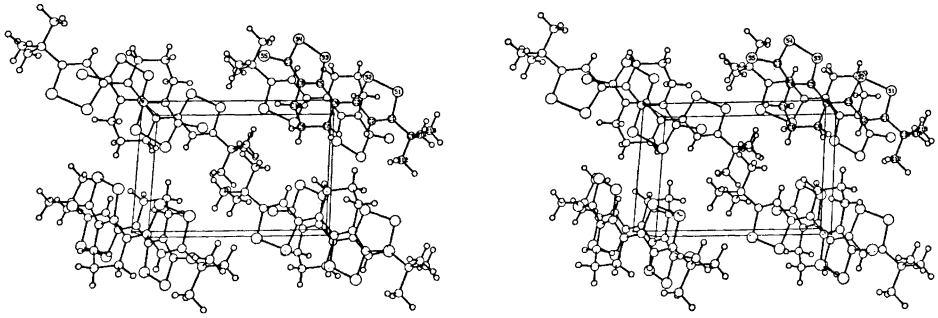


Fig. 3. Stereoscopic drawing showing the packing of molecules. The *a*-axis runs horisontally, the *c*-axis from top to bottom and the positive *b*-axis out of the paper. The box indicated defines ($\frac{1}{2} \times \frac{1}{2} \times \frac{1}{2}$) of the unit cell. The drawing was done by computer using the ORTEP plotting program.[21]

between the least-squares planes of the molecules are 3.64 and 4.22 Å, alternatingly.

There is a short intermolecular S(3)···S(3′) contact of 3.374(3) Å across a twofold axis. This indicates that approximately 3.40 Å is a reasonable estimate for the sulphur-sulphur van der Waals distance.[3]

THERMAL MOTION

Thermal ellipsoids have been plotted at 50 % probability level using the ORTEP program [21] (Fig. 4). The thermal motion of the 11 atoms in rings A, B,

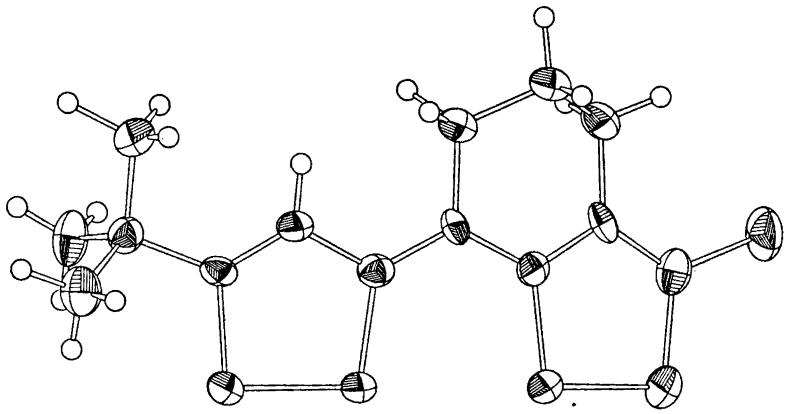


Fig. 4. Thermal ellipsoids at 50 % probability level.

and C were analyzed in terms of rigid body motion according to the method described by Schomaker and Trueblood.[22] The r.m.s. difference between observed and calculated U_{ij} values was 0.0035 Å. The maximum angle of libration is 6.5°, and the corresponding principal axis of libration is almost parallel to the S(1)···S(4) direction. Bond distances corrected for rigid body motion using Cruickshank's [23] method are listed in Table 3 together with the uncorrected ones. The lengthenings range from 0.002 to 0.012 Å.

All calculations were carried out on an IBM 360/50H computer. The programs, except when otherwise noted, originate from the Chemistry Department, Weizmann Institute of Science, Rehovoth, Israel, and have been modified for IBM 360/50H by Dr. D. Rabinovich and Cand. real. K. Åse.

Acknowledgement. The author wants to thank Drs. N. Lozac'h and M. Stavaux, University of Caen, for supplying a sample of the compound, and Dr. L. H. Jensen, Department of Biological Structure, University of Washington, for the use of laboratory facilities during the initial stage of this work.

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Received June 4, 1971.