λ = X-ray wave length, cm.,
$r_0 = e^2/mc^2 = 2\cdot81784 \times 10^{-13}$ cm.,
A = atomic weight,
N = Avogadro's number, $6\cdot02472 \times 10^{23}$ mol.$^{-1}$,
μ_m = mass obsorption coefficient, cm.^{2}g.$^{-1}$.

Table 8. *Comparison between observed and calculated values for* $\Delta f''$

Calculated values according to the formula of Townsend, Jeffrey & Pangis (1959)

		Mo $K\alpha$	Cu $K\alpha$	Fe $K\alpha$	Cr $K\alpha$
Th	$\Delta f_o''$	$14\cdot4 \pm 1\cdot2$	$18\cdot9 \pm 1\cdot6$	$25\cdot5 \pm 3\cdot4$	$22\cdot3 \pm 3\cdot6$
	$\Delta f_c''$	10·6	18·4	24·6	28·6
U	$\Delta f_o''$	$12\cdot9 \pm 2\cdot5$	$16\cdot0 \pm 2\cdot5$	$20\cdot0 \pm 5\cdot4$	$30\cdot5 \pm 6\cdot3$
	$\Delta f_c''$	12·6	18·6	25·0	27·1
Pu	$\Delta f_o''$	$10\cdot0 \pm 2\cdot7$	$20\cdot3 \pm 1\cdot9$	$32\cdot6 \pm 5\cdot0$	$34\cdot7 \pm 5\cdot9$
	$\Delta f_c''$	5·4	22·5	33·9	38·7

As an example we will consider the calculation of $\Delta f''$ for Pu for Cu $K\alpha$ radiation. $\lambda = 1\cdot5418 \times 10^{-8}$ cm., $A = 242$, and $\mu_m = 488$ cm.^{2}g.$^{-1}$ and the calculated $\Delta f'' = 22\cdot5$, which is in good agreement with the observed value of $\Delta f'' = 21\cdot0 \pm 1\cdot9$. Table 8 lists these values along with the calculated and observed $\Delta f''$ for the other elements and radiations. Considering the difficulty in obtaining observed values of $\Delta f''$ and the assumptions involved in the theoretical formula, (equation (13) being strictly valid only for $\sin\theta/\lambda = 0$), the agreement is quite good and in this case may be described as semi-quantitative.

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The Refinement of the Structure of the Complex of Iodine with 1,4-Diselenane, $C_4H_8Se_2 . 2\,I_2$

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The structure of the molecular complex $C_4H_8Se_2 . 2\,I_2$ has been refined by three-dimensional, full-matrix least-squares procedures. There are two molecules of the complex in the unit cell which has the dimensions:

$$a = 6\cdot876 \pm 0\cdot007,\ b = 6\cdot325 \pm 0\cdot007,\ c = 17\cdot68 \pm 0\cdot01\ \text{Å};\ \beta = 118°30' \pm 20'\,.$$

The space group is $P2_1/c$. The molecules are thus required only to be centrosymmetric but the molecular symmetry is actually $2/m$ within the standard deviations. The observed bond distances and angles are: I_1–$I_2 = 2\cdot870 \pm 0\cdot003$, I_2–Se $= 2\cdot829 \pm 0\cdot004$, Se–$C_1 = 1\cdot947 \pm 0\cdot024$, Se–$C_2 = 1\cdot980 \pm 0\cdot024$, C_1–$C_2 = 1\cdot568 \pm 0\cdot030$ Å; I_1–I_2–Se $= 180\cdot0 \pm 0\cdot3°$, I_2–Se–$C_1 = 101\cdot5 \pm 1\cdot0°$, I_2–Se–$C_2 = 100\cdot5 \pm 1\cdot0°$, C_1–Se–$C_2 = 100\cdot5 \pm 1\cdot8°$, Se–$C_1$–$C_2' = 117\cdot2 \pm 2\cdot0°$, Se–$C_2$–$C_1' = 113\cdot0 \pm 2\cdot0$.

The structure of the diselenane part of the molecule is but slightly changed by complexing with iodine, however, the I–I bond in the complex is 0·21 Å longer than that in solid I_2. In contrast to the equatorial bonding of iodine to sulfur in the dithiane complex, the iodine is bonded to selenium in axial positions in the diselenane complex.

Introduction

A preliminary study of the structures of the iodine complexes of 1,4-dithiane and 1,4-diselenane (McCullough, Chao & Zuccaro, 1959) and the three-dimen-

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sional refinement of the structure of the dithiane complex (Chao & McCullough, 1960) have been reported. The present communication is concerned with the three-dimensional refinement of the structure of the diselenane complex.

Experimental

The substance $C_4H_8Se_2.2\,I_2$ was first reported and described by McCullough & Tideswell (1954). Crystals suitable for X-ray diffraction studies can be prepared by recrystallization from ethylene chloride by slowly cooling solutions saturated at a higher temperature. The predominant habit of the purple colored crystals is that of needles extended along the *b* axis of the monoclinic unit. Because of the volatility of the complex, the selected crystals were sealed in thin-walled X-ray capillaries.

Rotation and Weissenberg photographs about the *b* axis were prepared with Mo $K\alpha$ radiation. The intensity data were obtained by use of the multiple-film Weissenberg procedure, interleaving 0·0025 cm. brass foils between the films. Precession photographs of the *hk*0, 0*kl* and several other nets which include 0*k*0 as a common row were also prepared with Mo $K\alpha$ radiation. On the precession camera, intensity data were obtained by making graduated sets of timed exposures of each net. Intensities were estimated visually by use of calibrated comparison strips and were corrected by application of appropriate factors to give $|F_0|^2$ values. The crystals used in the intensity measurements were six-sided prisms elongated on *b* with a nearly uniform thickness of 0·08–0·10 mm. The corresponding value of $\mu r = 0{\cdot}5$ is small enough so that omission of absorption corrections was considered justified.

Measurement of the photographs indicated a monoclinic unit cell with the dimensions:

$$a = 6{\cdot}876 \pm 0{\cdot}007,\ b = 6{\cdot}325 \pm 0{\cdot}007,\ c = 17{\cdot}68 \pm 0.01\ \text{Å};$$
$$\beta = 118°30' \pm 20'\ .$$

The above values are based on Mo $K\alpha = 0{\cdot}7107$ Å. The pycnometric density of a small sample of the crystals was 3·41 g.cm.$^{-3}$ while that calculated on the basis of 2 $C_4H_8Se_2.2\,I_2$ in the unit cell is 3·549 g.cm.$^{-3}$. The only systematic extinctions are those for *h*0*l* with *l* odd and 0*k*0 with *k* odd. The space group $P2_1/c$ is thus indicated and the molecules are required to be centrosymmetric.

Determination of the structure

Patterson summations on (100), (010) and (001) indicated trial parameters for I and Se which served to fix the starting phases for two-dimensional Fourier refinement. The heavy atom parameters which resulted from this refinement are given in Set 1 in Table 1. Since the carbon atom positions were not unambiguously determined in the two-dimensional work, a three-dimensional difference synthesis was carried out. In this synthesis, some 930 $|F_o|$ values derived from the observed reflections and the corresponding F_c values based on the iodine and selenium parameters in Set 1 were used. Well-defined carbon maxima appeared at the positions indicated in Set 1 in Table 1.

Table 1. *Atomic positonal parameters in* $C_4H_8Se_2.2\,I_2$

		Set 1	Set 2	
		Fourier parameters	Final L.S. parameters	Standard deviation
I_1	*x*	0·770	0·7786	0·0003
	y	0·920	0·9189	0·0003
	z	0·173	0·1738	0·0001
I_2	*x*	0·472	0·4758	0·0003
	y	0·259	0·2627	0·0003
	z	0·139	0·1401	0·0001
Se	*x*	0·192	0·1848	0·0006
	y	0·607	0·6066	0·0006
	z	0·110	0·1103	0·0002
C_1	*x*	0·14	0·1454	0·0030
	y	0·72	0·7117	0·0032
	z	0·00	0·0004	0·0011
C_2	*x*	0·90	0·9003	0·0030
	y	0·44	0·4555	0·0032
	z	0·07	0·0717	0·0011

Refinement of the structure

The preceding calculations were carried out on SWAC. However, since it was necessary to move this computer to a new building, a process which required almost a year, three-dimensional refinement was carried out on the IBM 709 computer at the Western Data Processing Center in the UCLA School of Business Administration. The first three-dimensional refinement routine available on the IBM 709 was one patterned after the full-matrix least-squares routine written for SWAC. In all other respects, the refinement procedure was closely similar to that used for the iodine complex of dithiane.

Table 2. *Anisotropic temperature parameters in* $C_4H_8Se_2.2\,I_2$

(Standard deviations in parentheses)

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
I_1	4·72	4·33	4·95	2·50	3·26	−0·59
	(0·08)	(0·08)	(0·08)	(0·16)	(0·12)	(0·35)
I_2	3·11	4·10	3·47	0·71	2·32	0·43
	(0·08)	(0·08)	(0·08)	(0·16)	(0·12)	(0·35)
Se	3·08	3·58	3·20	0·54	2·18	1·54
	(0·18)	(0·18)	(0·18)	(0·30)	(0·20)	(0·35)
C_1	4·6	3·3	5·0	−0·4	3·6	−0·3
	(0·8)	(1·0)	(1·0)	(1·2)	(1·2)	(1·3)
C_2	4·9	1·5	6·5	1·40	6·9	1·0
	(0·8)	(1·0)	(1·0)	(1·4)	(1·4)	(1·6)

Table 3. *Comparison of observed and calculated structure factors for* $C_4H_8Se_2 . 2\,I_2$

The F_o values enclosed in parentheses are the minimum observable values for unobserved reflections.

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	h k l	F_o	F_c
00 00 02	112.8	93.6-	06 00 04	69.4	71.5-	03 01 14-	25.5	24.2-	00 02 03	83.5	82.1-	03 02 12	30.1	34.3-	00 03 05	32.2	25.4-
00 00 04	259.1	315.1-	06 00 06	(18.4)	11.2-	03 01 13-	19.3	20.3	00 02 04	74.4	72.3	03 02 13	(17.8)	2.7-	00 03 06	116.0	111.3-
00 00 06	158.4	170.1	06 00 08	57.5	54.6	03 01 12-	133.2	146.9	00 02 05	75.3	70.4-	03 02 14	28.5	25.4	00 03 07	29.4	33.1
00 00 08	63.8	61.9	06 00 10	(20.5)	5.5-	03 01 11-	42.3	44.4-	00 02 06	31.3	28.0-	03 02 15	20.8	20.8-	00 03 08	75.7	75.7
00 00 10	34.2	32.5-	07 00 18-	(18.0)	0.5	03 01 10-	24.7	23.4-	00 02 07	136.0	158.4	03 02 16	19.3	11.8	00 03 09	21.6	18.5
00 00 12	11.9	4.9	07 00 16-	(17.3)	1.7	03 01 09-	52.7	52.9	00 02 08	102.2	108.7-	04 02 20-	26.8	26.1-	00 03 10	10.0	10.0
00 00 14	22.6	14.0-	07 00 14-	(16.8)	3.9-	03 01 08-	144.4	165.8-	00 02 09	11.3	12.8-	04 02 19-	(17.0)	6.4	00 03 11	(13.1)	3.4-
00 00 16	(16.3)	13.2	07 00 12-	(16.5)	15.1-	03 01 07-	58.3	64.4	00 02 10	63.5	66.1	04 02 18-	35.4	32.3	00 03 12	(13.9)	3.7
00 00 18	(17.7)	3.3	07 00 10-	43.4	40.6	03 01 06-	85.7	91.2	00 02 11	71.3	79.2-	04 02 17-	35.0	34.1	00 03 13	(14.4)	1.4-
00 00 20	(19.0)	0.4-	07 00 08-	29.1	15.1	03 01 05-	137.5	156.4-	00 02 12	41.0	43.4	04 02 16-	13.4	16.5	00 03 14	(15.5)	6.7-
00 00 22	(20.5)	7.7	07 00 06-	78.3	81.1-	03 01 04-	88.9	93.0	00 02 13	15.6	14.5	04 02 15-	21.2	21.3-	00 03 15	(15.9)	4.7-
01 00 18-	27.0	28.5	07 00 04-	31.5	25.0	03 01 03-	39.3	33.9	00 02 14	65.7	73.6-	04 02 14-	45.7	46.4-	00 03 16	(17.5)	9.7-
01 00 16-	12.6	13.1	07 00 02-	81.8	80.8	03 01 02-	67.3	67.0-	00 02 15	(15.8)	11.0	04 02 13-	59.1	62.6-	01 03 16-	41.8	47.2
01 00 14-	106.1	115.6-	07 00 00	42.8	42.2-	03 01 01-	119.2	122.4	00 02 16	(16.4)	2.2	04 02 12-	(15.2)	9.7	01 03 15-	15.2	18.8-
01 00 12-	43.5	39.8	07 00 02	47.0	37.2-	03 01 00	10.7	16.6-	00 02 17	(17.1)	13.8	04 02 11-	53.6	58.5	01 03 14-	(13.5)	4.5
01 00 10-	221.5	252.5	07 00 04	31.0	27.8	03 01 01	53.3	54.8-	00 02 18	34.7	37.2	04 02 10-	40.9	42.3	01 03 13-	(13.5)	5.1-
01 00 08-	175.0	198.4-	08 00 16-	42.2	31.0-	03 01 02	24.8	27.0	01 02 19-	15.0	11.6-	04 02 09-	28.4	26.4	01 03 12-	116.0	135.7-
01 00 06-	187.9	221.9-	08 00 14-	38.6	32.2	03 01 03	(8.6)	3.6	01 02 18-	34.3	31.9-	04 02 08-	(12.0)	1.2-	01 03 11-	33.2	39.2
01 00 04-	246.2	302.2	08 00 12-	44.3	37.5	03 01 04	16.7	13.6	01 02 17-	34.7	39.6-	04 02 07-	34.8	34.9-	01 03 10-	59.2	63.5
01 00 02-	28.1	26.5	08 00 10-	57.3	53.5-	03 01 05	26.4	23.3-	01 02 16-	(11.7)	6.6	04 02 06-	36.4	31.3-	01 03 09-	(11.0)	4.8
01 00 00	80.5	79.3-	08 00 08-	30.4	19.1-	03 01 06	27.5	25.4-	01 02 15-	37.3	37.8	04 02 05-	37.5	34.6	01 03 08-	154.2	167.1
01 00 02	77.4	62.1	08 00 06-	51.3	43.9	03 01 07	30.5	25.5-	01 02 14-	36.3	32.6	04 02 04-	9.1	10.5	01 03 07-	43.7	43.1-
01 00 04	52.5	51.8-	08 00 02-	18.5	15.8-	03 01 08	(14.7)	6.1	01 02 13-	30.4	28.2	04 02 03-	61.3	60.2-	01 03 06-	199.2	186.9-
01 00 06	47.4	48.1	00 01 01	30.4	29.7-	03 01 09	55.3	61.4	01 02 12-	24.4	23.7-	04 02 02-	64.5	64.6	01 03 05-	(7.9)	3.9
01 00 08	22.3	17.5	00 01 02	34.8	40.4-	03 01 10	33.4	37.4	01 02 11-	39.9	43.9-	04 02 01-	(12.3)	2.3-	01 03 04-	83.0	69.1-
01 00 10	22.6	21.8-	00 01 03	86.9	102.7-	03 01 11	(12.8)	5.6-	01 02 10-	29.9	28.5-	04 02 00	53.7	47.8-	01 03 03-	48.4	47.9
01 00 12	35.1	30.5	00 01 04	7.5	7.6	03 01 12	29.0	29.2-	01 02 09-	30.4	32.1	04 02 01	101.7	102.5	01 03 02-	150.1	148.0
01 00 14	42.5	39.5-	00 01 05	160.5	169.0	03 01 13	30.1	31.2-	01 02 08-	12.2	16.1	04 02 02	51.1	51.5-	01 03 01-	14.5	13.1
01 00 16	(17.3)	5.3-	00 01 06	86.3	90.8	03 01 14	26.6	21.1-	01 02 07-	68.7	70.3-	04 02 03	47.7	45.2-	01 03 00	36.2	35.6-
01 00 18	50.9	47.8	00 01 07	9.8	7.7	03 01 15	16.8	11.3	01 02 06-	54.7	51.0	04 02 04	84.5	87.4	01 03 01	29.9	25.8-
01 00 20	17.5	12.8-	00 01 08	80.0	80.6-	03 01 16	33.5	31.0	01 02 05-	24.3	27.3-	04 02 05	51.8	50.6-	01 03 02	(7.8)	2.7-
01 00 22	29.8	22.9-	00 01 09	114.4	124.9-	04 01 20-	30.8	25.3-	01 02 04-	84.2	82.0-	04 02 06	(11.8)	8.1	01 03 03	22.0	13.2-
02 00 20-	44.3	39.5	00 01 10	68.8	72.7-	04 01 19-	15.0	17.8	01 02 03-	190.3	197.0	04 02 07	29.9	27.8	01 03 04	19.4	14.4-
02 00 18-	78.2	77.6-	00 01 11	44.2	48.2	04 01 18-	43.3	43.4-	01 02 02-	112.5	111.2-	04 02 08	59.0	62.8-	01 03 05	(9.7)	5.5
02 00 16-	35.5	35.4-	00 01 12	102.0	111.0	04 01 17-	(15.7)	4.1	01 02 01-	120.3	113.0-	04 02 09	(16.8)	11.6	01 03 06	47.9	42.8-
02 00 14-	118.0	125.8	00 01 13	20.7	22.0	04 01 16-	52.3	52.7	01 02 00	190.1	181.2	04 02 10	29.3	24.7	01 03 07	(11.3)	8.7
02 00 12-	25.8	25.2-	00 01 14	21.7	16.5	04 01 15-	51.9	54.3-	01 02 01	160.5	152.3-	04 02 11	(18.0)	8.1	01 03 08	31.7	32.5
02 00 10-	65.0	66.8-	00 01 15	22.7	15.5-	04 01 14-	26.0	25.9	01 02 02	32.1	30.1	04 02 12	28.4	28.1	01 03 09	17.8	15.0
02 00 08-	34.9	39.9	00 01 16	72.4	72.6-	04 01 13-	32.1	35.1	01 02 03	110.4	104.2	05 02 18-	(16.8)	11.1-	01 03 10	16.5	14.0-
02 00 06-	64.5	61.1-	00 01 17	(17.0)	7.7	04 01 12-	55.0	58.7-	01 02 04	210.8	203.0-	05 02 17-	(16.2)	14.4-	01 03 11	(14.1)	3.6
02 00 04-	47.7	48.6	00 01 18	(17.9)	8.7	04 01 11-	49.6	55.7	01 02 05	18.3	16.7	05 02 16-	(15.9)	12.4-	01 03 12	(14.9)	8.1
02 00 02-	6.1	11.6	00 01 19	16.3	13.8-	04 01 10-	(12.5)	0.3	01 02 06	75.4	73.2	05 02 15-	(15.4)	6.0	01 03 13	19.1	14.8-
02 00 00	71.8	60.1-	00 01 20	29.3	29.1	04 01 09-	56.8	58.2-	01 02 07	17.5	16.1	05 02 14-	18.6	18.4	01 03 14	25.0	17.0
02 00 02	65.9	61.7	01 01 20-	36.4	28.9	04 01 08-	27.2	26.2	01 02 08	101.3	98.9	05 02 13-	26.6	24.4-	01 03 15	(17.0)	4.2-
02 00 04	96.1	96.3-	01 01 19-	35.0	28.6-	04 01 07-	(9.1)	1.3	01 02 09	12.7	10.8-	05 02 12-	23.8	20.9	01 03 16	36.5	37.2-
02 00 06	17.9	16.5	01 01 18-	21.3	16.8	04 01 06-	9.1	10.3-	01 02 10	95.6	93.2-	05 02 11-	12.2	18.2	02 03 20-	32.5	32.0
02 00 08	130.1	144.4	01 01 17-	21.1	14.6	04 01 05-	22.1	21.5-	01 02 11	66.7	65.2-	05 02 10-	51.8	52.6-	02 03 19-	(17.3)	7.7-
02 00 10	100.1	103.5-	01 01 16-	37.5	38.9-	04 01 04-	31.0	28.4-	01 02 12	26.2	25.9-	05 02 09-	64.9	63.8	02 03 18-	29.1	32.7
02 00 12	74.9	73.2-	01 01 15-	43.8	44.7	04 01 03-	9.1	7.1-	01 02 13	35.1	34.0	05 02 08-	13.5	10.1-	02 03 17-	(16.1)	15.1-
02 00 14	82.6	82.6	01 01 14-	(14.3)	8.1	04 01 02-	21.8	19.4	01 02 14	48.3	45.7	05 02 07-	67.4	68.1-	02 03 16-	70.5	75.9-
02 00 16	(18.4)	13.5	01 01 13-	47.5	47.5-	04 01 01-	101.5	107.7	01 02 15	32.6	29.4	05 02 06-	89.2	100.0	02 03 15-	11.9	10.7
02 00 18	31.8	33.2-	01 01 12-	28.8	2.82	04 01 00	42.0	39.1	01 02 16	(17.5)	1.8	05 02 05-	39.9	36.7-	02 03 14-	(10.0)	4.1-
03 00 18-	(16.2)	13.7	01 01 11-	16.6	9.1-	04 01 01	48.1	46.9-	02 02 18-	(16.6)	16.9	05 02 04-	40.6	36.2-	02 03 13-	18.8	17.5
03 00 16-	(15.0)	9.6-	01 01 10-	(11.5)	13.2-	04 01 02	71.3	75.4-	02 02 17-	(15.9)	15.5-	05 02 03-	65.6	65.2	02 03 12-	67.5	76.6
03 00 14-	15.7	13.4	01 01 09-	(11.0)	6.8-	04 01 03	64.6	66.6-	02 02 16-	(15.2)	8.6	05 02 02-	81.0	87.6-	02 03 11-	(15.2)	6.2-
03 00 12-	(12.6)	3.0	01 01 08-	7.2	10.2-	04 01 04	17.7	20.1-	02 02 15-	(14.8)	10.5	05 02 01-	(14.2)	7.5	02 03 10-	41.5	43.6-
03 00 10-	35.1	28.9-	01 01 07-	26.8	28.6-	04 01 05	52.3	55.6	02 02 14-	41.0	43.5-	05 02 00	72.0	74.9	02 03 09-	24.9	27.3-
03 00 08-	26.1	20.0	01 01 06-	43.6	41.8	04 01 06	82.8	91.7	02 02 13-	59.3	64.1	05 02 01	(14.8)	1.3	02 03 08-	7.4	8.6-
03 00 06-	90.5	89.6-	01 01 05-	158.7	179.3	04 01 07	(15.7)	3.5	02 02 12-	(12.4)	7.1-	05 02 02	34.9	31.2	02 03 07-	6.8	6.4-
03 00 04-	62.6	59.0	01 01 04-	72.6	71.9	04 01 08	17.2	20.6-	02 02 11-	78.2	83.3-	05 02 03	20.7	19.3-	02 03 06-	12.1	9.8-
03 00 02-	144.3	146.8	01 01 03-	70.5	71.0-	04 01 09	(16.8)	9.0-	02 02 10-	107.2	119.5	05 02 04	58.2	61.6-	02 03 05-	34.6	28.4
03 00 00	244.9	264.7-	01 01 02-	130.2	128.2-	04 01 10	60.6	61.1-	02 02 09-	62.7	65.5-	05 02 05	23.5	24.6-	02 03 04-	56.9	46.0-
03 00 02	64.6	65.0-	01 01 01-	208.5	212.1-	04 01 11	(18.0)	5.8	02 02 08-	45.1	46.0-	05 02 06	(16.8)	2.8	02 03 03-	11.9	8.1
03 00 04	223.8	249.6	01 01 00	31.5	31.0-	04 01 12	25.1	26.4	02 02 07-	108.0	120.4	05 02 07	35.2	35.8	02 03 02-	66.0	58.0
03 00 06	52.6	50.8-	01 01 01	148.8	162.0	04 01 13	(19.3)	14.7-	02 02 06-	148.0	167.6-	05 02 08	30.1	30.5	02 03 01-	(8.3)	2.8-
03 00 08	107.6	116.4-	01 01 02	239.3	274.2	04 01 14	24.8	23.4	02 02 05-	(8.5)	3.8-	05 02 09	(18.5)	9.9	02 03 00	20.9	21.7-
03 00 10	40.3	38.7	01 01 03	19.3	13.7	05 01 11-	48.8	48.7	02 02 04-	148.1	161.7	06 02 18-	17.5	11.4	02 03 01	6.4	5.3-
03 00 12	16.9	24.2	01 01 04	57.1	55.3-	05 01 10-	(12.8)	2.8	02 02 03-	18.8	15.0-	06 02 17-	32.1	29.8-	02 03 02	11.9	10.3
03 00 14	(18.3)	6.7-	01 01 05	67.3	66.8-	05 01 09-	59.2	63.0-	02 02 02-	92.6	86.1	06 02 16-	32.4	33.5	02 03 03	29.0	32.5-
03 00 16	(19.7)	8.4-	01 01 06	180.4	202.5-	05 01 08-	61.8	65.8-	02 02 01-	41.7	35.9-	06 02 15-	(16.1)	4.8-	02 03 04	18.9	22.3
03 00 18	(21.0)	2.6-	01 01 07	45.0	48.7	05 01 07-	46.4	43.2-	02 02 00	175.2	194.4-	06 02 14-	41.7	42.5-	02 03 05	(11.5)	2.7
03 00 20	(22.0)	3.6	01 01 08	108.5	117.0	05 01 06-	20.1	17.2	02 02 01	107.9	108.4-	06 02 13-	42.5	41.0	02 03 06	99.0	108.3-
04 00 16-	15.0	16.0-	01 01 09	55.1	54.7-	05 01 05-	74.2	78.9	02 02 02	34.3	33.2	06 02 12-	36.4	36.4-	02 03 07	37.0	35.0
04 00 14-	47.6	47.1	01 01 10	81.9	89.2	05 01 04-	92.1	97.3	02 02 03	120.0	117.1	06 02 11-	(15.2)	4.3-	02 03 08	39.0	42.1
04 00 12-	28.2	25.7	01 01 11	(13.9)	5.9	05 01 03-	13.5	12.5-	02 02 04	120.1	115.8	06 02 10-	65.8	67.0	02 03 09	(13.9)	7.3-
04 00 10-	160.8	174.9-	01 01 12	65.6	68.2-	05 01 02-	79.2	81.3-	02 02 05	54.5	50.9	06 02 09-	12.9	12.4-	02 03 10	69.3	80.0
04 00 08-	49.0	46.2	01 01 13	53.3	55.6	05 01 01-	28.3	16.2-	02 02 06	41.1	41.9-	06 02 08-	(14.5)	0.4-	02 03 11	12.3	24.3-
04 00 06-	213.5	237.0	01 01 14	14.1	19.1-	05 01 00	81.9	83.7-	02 02 07	98.7	103.7-	06 02 07-	21.2	18.8-	02 03 12	66.3	73.5-
04 00 04-	160.7	170.8-	01 01 15	29.4	29.0-	05 01 01	(14.5)	15.9	02 02 08	40.8	39.3-	06 02 06-	62.6	67.1-	02 03 13	(16.0)	5.0
04 00 02-	140.6	144.5-	01 01 16	25.0	26.1	05 01 02	87.6	92.0	02 02 09	(13.9)	9.9	06 02 05-	11.5	10.9-	02 03 14	26.8	25.2-
04 00 00	132.7	131.8	01 01 17	15.9	18.3-	05 01 03	34.2	34.2-	02 02 10	15.8	17.3	06 02 04-	28.5	29.1	03 03 18-	(16.5)	4.5-
04 00 02	(13.1)	14.1	02 01 16-	17.3	20.4	05 01 04	26.8	25.9	02 02 11	23.3	19.9	06 02 03-	55.5	56.1	03 03 17-	(15.9)	4.0
04 00 04	38.4	33.8-	02 01 15-	40.0	41.6	05 01 05	25.0	23.0	02 02 12	21.7	20.7	06 02 02-	34.2	33.8	03 03 16-	(15.2)	2.7
04 00 06	(15.0)	8.4-	02 01 14-	(10.1)	1.2	05 01 06	57.7	61.3-	02 02 13	(16.8)	5.3-	06 02 01-	(13.8)	2.5-	03 03 15-	(14.6)	14.9
04 00 08	(16.2)	9.3-	02 01 13-	52.8	56.6-	05 01 07	23.3	25.2	02 02 14	(17.4)	11.3-	06 02 00	30.4	27.6-	03 03 14-	(14.1)	11.0-
04 00 10	(17.3)	14.3	02 01 12-	55.3	58.3-	05 01 08	(15.7)	3.4-	02 02 15	(17.8)	13.6	06 02 01	42.4	44.0-	03 03 13-	(13.5)	7.6-
04 00 12	(18.5)	4.6-	02 01 11-	63.7	67.1-	05 01 09	32.5	29.2-	02 02 16	(18.2)	11.7-	06 02 02	17.1	17.8-	03 03 12-	24.8	25.9
04 00 14	(19.7)	0.7-	02 01 10-	36.2	36.3	05 01 10	(18.9)	22.6	03 02 21-	20.7	22.1-	07 02 16-	30.7	26.5-	03 03 11-	(12.4)	14.4-
04 00 16	(21.0)	5.6	02 01 09-	113.8	127.1	05 01 11	(19.4)	5.2-	03 02 20-	29.8	25.0	07 02 15-	(15.0)	6.5	03 03 10-	(12.0)	8.5-
04 00 18	(22.2)	11.8-	02 01 08-	143.7	161.4	05 01 12	(20.1)	1.6-	03 02 19-	(17.0)	7.1-	07 02 14-	28.4	23.9	03 03 09-	(11.5)	2.2-
05 00 20-	42.7	37.7-	02 01 07-	18.9	11.7-	05 01 13	(20.6)	1.6-	03 02 18-	38.9	34.1-	07 02 13-	28.2	27.6	03 03 08-	(11.0)	5.0
05 00 18-	44.3	43.4	02 01 06-	124.4	138.7-	06 01 15-	36.7	36.4	03 02 17-	42.1	41.3	07 02 12-	14.4	11.0	03 03 07-	23.0	21.1-
05 00 16-	62.1	62.6	02 01 05-	55.9	60.8-	06 01 14-	31.2	29.4	03 02 16-	37.4	34.6-	07 02 11-	16.6	17.4-	03 03 06-	(10.2)	7.9-
05 00 14-	107.2	109.2-	02 01 04-	152.4	169.6-	06 01 13-	15.5	12.3-	03 02 15-	(16.4)	6.9-	07 02 10-	34.3	29.3-	03 03 05-	19.4	16.7
05 00 12-	28.5	39.7-	02 01 03-	77.7	78.0	06 01 12-	61.3	67.8-	03 02 14-	79.8	88.5	07 02 09-	35.1	33.5-	03 03 04-	100.2	105.9-
05 00 10-	122.7	121.9	02 01 02-	257.4	281.3	06 01 11-	20.3	11.1-	03 02 13-	18.3	21.1-	07 02 08-	(16.4)	4.8-	03 03 03-	33.5	38.3
05 00 08-	21.3	13.7-	02 01 01-	97.1	100.3-	06 01 10-	28.3	25.0-	03 02 12-	15.3	6.8	07 02 07-	29.8	26.5	03 03 02-	111.7	113.1
05 00 06-	45.6	43.1-	02 01 00	90.3	84.7	06 01 09-	(15.0)	6.9	03 02 11-	18.9	21.4-	07 02 06-	18.7	19.5	03 03 01-	45.3	42.3-
05 00 04-	(13.2)	5.9	02 01 01	49.5	43.8	06 01 08-	88.1	98.8	03 02 10-	94.3	113.1-	07 02 05-	(16.7)	8.8	03 03 00	93.0	96.0
05 00 02-	21.9	11.4-	02 01 02	192.2	198.2-	06 01 07-	32.7	28.8-	03 02 09-	32.0	32.4-	07 02 04-	(16.8)	2.6	03 03 01	30.6	35.0-
05 00 00	25.2	26.1	02 01 03	112.6	114.7	06 01 06-	18.4	12.7-	03 02 08-	71.8	78.2	07 02 03-	(17.0)	7.4-	03 03 02	171.2	188.9-
05 00 02	(14.9)	10.2-	02 01 04	24.7	26.7	06 01 05-	33.8	35.4	03 02 07-	107.2	120.1	07 02 02-	17.8	22.3	03 03 03	28.6	32.6
05 00 04	(15.8)	4.0-	02 01 05	123.9	128.8-	06 01 04-	83.3	85.1-	03 02 06-	78.0	84.9	07 02 01-	(17.5)	5.6	03 03 04	(12.2)	3.9
05 00 06	21.3	13.9	02 01 06	83.4	87.0	06 01 03-	26.8	18.3	03 02 05-	(10.1)	6.5	07 02 00	(17.9)	2.6	03 03 05	(14.5)	13.0
05 00 08	35.1	33.3-	02 01 07	26.4	22.7-	06 01 02-	31.3	28.0	03 02 04-	85.8	89.2-	07 02 01	24.0	19.3-	03 03 06	109.1	127.0
05 00 10	18.9	11.7	02 01 08	(13.2)	13.3-	06 01 01-	53.0	56.8-	03 02 03-	144.5	153.5-	08 02 10-	12.1	10.8	03 03 07	22.0	20.8-
05 00 12	32.3	31.3	02 01 09	65.3	65.9	06 01 00	34.8	37.2	03 02 02-	23.4	19.7-	08 02 09-	29.1	13.5-	03 03 08	38.5	44.3-
05 00 14	(21.2)	22.5-	02 01 10	19.6	20.4-	06 01 01	18.6	9.6	03 02 01-	60.3	61.7	08 02 08-	15.8	12.3	03 03 09	(15.5)	10.8-
06 00 20-	48.4	39.0	02 01 11	(15.1)	6.9-	06 01 02	25.5	20.6-	03 02 00	52.0	52.5	08 02 07-	12.9	9.9	03 03 10	41.2	43.2-
06 00 18-	23.9	14.6-	02 01 12	(15.7)	8.0	06 01 03	27.7	27.4	03 02 01	43.2	40.6	08 02 06-	24.4	26.0-	03 03 11	(16.7)	5.4
06 00 16-	24.7	23.2-	02 01 13	(16.4)	1.8	06 01 04	17.9	14.7-	03 02 02	27.2	23.6	08 02 05-	20.6	20.0	03 03 12	(17.3)	16.2
06 00 14-	(15.7)	10.8	02 01 14	(17.0)	12.2	06 01 05	(18.0)	6.6-	03 02 03	32.3	32.4-	08 02 04-	(13.0)	3.2-	03 03 13	(18.0)	4.5
06 00 12-	(15.2)	0.6	02 01 15	(17.8)	9.8-	06 01 06	(18.5)	8.6	03 02 04	33.1	28.6-	08 02 03-	21.3	21.1-	03 03 14	(17.9)	9.9
06 00 10-	14.6	15.0	02 01 16	(18.5)	10.4-	06 01 07	(19.0)	1.6-	03 02 05	37.2	35.6	08 02 02-	23.7	30.7	04 03 14-	16.4	19.5-
06 00 08-	(14.7)	5.1-	03 01 20-	25.3	21.8	06 01 08	(19.4)	4.3	03 02 06	(13.4)	12.0-	08 02 01-	(19.3)	11.6-	04 03 13-	(14.3)	8.1
06 00 06-	(14.7)	1.9	03 01 19-	34.5	33.5	06 01 09	(20.1)	9.2-	03 02 07	45.5	44.5-	08 02 00	14.0	11.5-	04 03 12-	69.6	81.6
06 00 04-	(14.9)	11.4	03 01 18-	27.6	25.0	06 01 10	(20.5)	9.4-	03 02 08	48.8	49.4	00 03 01	53.7	48.6	04 03 11-	37.6	38.0-
06 00 02-	41.9	41.2-	03 01 17-	(15.7)	9.8-	00 02 00	52.5	52.1-	03 02 09	25.7	20.3-	00 03 02	265.4	282.4	04 03 10-	(12.4)	13.6
06 00 00	43.5	39.6	03 01 16-	58.7	67.9-	00 02 01	82.4	81.0	03 02 10	(15.9)	8.5-	00 03 03	43.3	39.6-	04 03 09-	(12.2)	7.1-
06 00 02	48.4	46.7	03 01 15-	26.5	21.4-	00 02 02	20.5	19.2-	03 02 11	47.0	50.8	00 03 04	140.0	129.3-	04 03 08-	141.8	156.4-

Table 3 (*cont.*)

h k l	F_o	F_c
04 03 07-	39.2	42.2
04 03 06-	68.1	71.2
04 03 05-	(11.7)	10.0-
04 03 04-	122.0	137.0
04 03 03-	32.2	32.2-
04 03 02-	111.0	119.3-
04 03 01-	(12.7)	0.8
04 03 00	36.9	38.5-
04 03 01	(13.0)	14.1
04 03 02	56.8	64.4
04 03 03	(14.4)	6.9
04 03 04	(14.4)	3.2
04 03 05	(14.9)	11.1-
04 03 06	(15.3)	0.6-
04 03 07	(15.9)	8.4-
04 03 08	(16.6)	11.5-
04 03 09	(17.0)	1.6
04 03 10	(17.5)	2.2-
05 03 18-	40.0	43.0-
05 03 17-	(16.3)	16.3
05 03 16-	46.6	52.1
05 03 15-	11.0	14.2-
05 03 14-	40.8	45.6
05 03 13-	(14.5)	14.0-
05 03 12-	72.2	85.2-
05 03 11-	(13.9)	12.2
05 03 10-	(13.5)	1.4
05 03 09-	(13.3)	10.6
05 03 08-	58.4	65.9
05 03 07-	(13.2)	0.2-
05 03 06-	19.5	16.8-
05 03 05-	24.8	19.0-
05 03 04-	(13.3)	3.8-
05 03 03-	(13.5)	6.2-
05 03 02-	14.4	11.0-
05 03 01-	14.7	16.9
05 03 00	(14.6)	2.2-
05 03 01	(14.8)	0.2
05 03 02	17.3	20.8
05 03 03	(15.4)	0.7
05 03 04	(16.0)	11.0-
05 03 05	(16.6)	1.5
05 03 06	(17.0)	14.9
06 03 16-	(16.7)	14.1-
06 03 15-	(16.3)	8.6-
06 03 14-	(15.8)	5.0-
06 03 13-	(15.6)	4.8
06 03 12-	(15.5)	0.7-
06 03 11-	(15.4)	16.9
06 03 10-	(15.2)	1.7
06 03 09-	(15.0)	5.9-
06 03 08-	(15.0)	16.1
06 03 07-	(15.0)	4.8-
06 03 06-	(15.0)	8.5-
06 03 05-	(15.0)	2.8
06 03 04-	(15.2)	15.8
06 03 03-	(15.4)	12.7-
06 03 02-	(15.7)	7.5-
06 03 01-	(15.9)	12.0
06 03 00	38.4	40.8-
06 03 01	21.8	17.9
06 03 02	41.6	45.1
06 03 03	14.0	15.8-
06 03 04	27.4	25.3
06 03 05	(16.8)	8.0-
06 03 06	44.4	48.6-
07 03 10-	(16.6)	16.0-
07 03 09-	(16.5)	7.4
07 03 08-	44.6	50.4
07 03 07-	(16.5)	21.8-
07 03 06-	(16.8)	3.0
07 03 05-	(16.8)	0.9
07 03 04-	56.5	66.3-
07 03 03-	(17.3)	19.6
07 03 02-	19.7	25.3
07 03 01-	(17.6)	3.2-
07 03 00	37.4	42.4
07 03 01	(18.4)	8.2-
07 03 02	28.8	28.7-
07 03 03	(19.2)	0.7
08 03 14-	35.2	32.0-
08 03 13-	(18.2)	11.1
08 03 12-	31.6	30.7
08 03 11-	(18.0)	8.7-
08 03 10-	29.0	22.6
08 03 09-	(18.0)	5.6-
08 03 08-	35.7	37.9-
08 03 07-	(18.3)	5.2
08 03 06-	(18.4)	5.1-
08 03 05-	(18.5)	4.6
08 03 04-	(18.6)	21.1
08 03 03-	(19.0)	0.8
00 04 00	(3.4)	1.2
00 04 01	37.3	34.9
00 04 02	(5.5)	3.0
00 04 03	35.7	37.3-
00 04 04	21.7	17.3-
00 04 05	13.9	10.4-
00 04 06	43.6	34.5
00 04 07	87.4	77.9
00 04 08	44.5	42.3
00 04 09	41.6	43.1-
00 04 10	66.3	68.9-
00 04 11	31.1	31.5-
00 04 12	23.8	18.8-
00 04 13	32.5	35.7
00 04 14	58.9	65.1
00 04 15	(16.1)	7.7-
00 04 16	(16.9)	0.0
00 04 17	(17.5)	4.7-
00 04 18	33.8	32.4-
01 04 19-	(18.1)	6.6
01 04 18-	29.0	22.6
01 04 17-	28.5	25.0-
01 04 16-	(16.1)	10.5
01 04 15-	(15.5)	7.9
01 04 14-	(14.7)	18.0-
01 04 13-	(14.1)	18.6
01 04 12-	(13.4)	5.3
01 04 11-	(12.7)	7.2-
01 04 10-	(12.0)	0.5-
01 04 09-	24.0	21.4
01 04 08-	(10.4)	7.0-
01 04 07-	47.0	38.0-
01 04 06-	(9.0)	4.4
01 04 05-	(8.1)	7.8
01 04 04-	39.5	35.5
01 04 03-	79.8	87.6
01 04 02-	(15.5)	14.1
01 04 01-	90.1	88.1-
01 04 00	78.8	103.8-
01 04 01	27.8	22.4-
01 04 02	19.6	19.6
01 04 03	94.6	92.8
01 04 04	137.8	143.0
01 04 05	46.2	42.9-
01 04 06	63.8	58.9-
01 04 07	(11.5)	2.9
01 04 08	89.7	91.5-
01 04 09	31.7	36.2
01 04 10	52.6	55.4
01 04 11	35.2	37.0-
01 04 12	37.8	36.1
01 04 13	(15.7)	5.3
01 04 14	24.8	22.5-
01 04 15	18.3	21.1
01 04 16	(17.8)	11.0-
02 04 19-	(17.7)	1.5-
02 04 18-	(16.9)	3.6-
02 04 17-	(16.2)	13.1-
02 04 16-	(15.7)	8.4
02 04 15-	25.4	13.7
02 04 14-	(14.3)	10.3
02 04 13-	29.3	30.2
02 04 12-	17.1	16.1-
02 04 11-	49.4	51.2-
02 04 10-	48.4	47.6-
02 04 09-	13.3	10.0
02 04 08-	53.8	49.5
02 04 07-	73.9	67.5
02 04 06-	108.6	95.9
02 04 05-	69.1	59.7-
02 04 04-	145.8	122.5-
02 04 03-	36.6	26.9
02 04 02-	104.2	87.4-
02 04 01-	48.6	41.7
02 04 00	150.1	144.1
02 04 01	88.6	87.0-
02 04 02	28.3	26.2
02 04 03	44.4	41.3
02 04 04	82.6	84.7-
02 04 05	50.0	52.6
02 04 06	(12.2)	0.5
02 04 07	47.9	45.7-
02 04 08	21.9	24.5
02 04 09	(14.3)	2.9
02 04 10	(14.9)	1.9
02 04 11	(15.7)	14.9
02 04 12	(16.2)	6.1-
02 04 13	(17.0)	0.2
02 04 14	(17.7)	0.6
02 04 15	(18.3)	2.6
03 04 14-	62.8	67.5-
03 04 13-	14.7	19.6
03 04 12-	(13.2)	11.1-
03 04 11-	(12.7)	6.6
03 04 10-	101.2	108.8
03 04 09-	56.1	58.5-
03 04 08-	31.6	26.1-
03 04 07-	66.2	61.1
03 04 06-	99.6	97.4-
03 04 05-	38.4	38.8
03 04 04-	46.5	39.6
03 04 03-	73.5	74.2-
03 04 02-	33.0	31.3
03 04 01-	24.7	20.0
03 04 00	22.6	19.0-
03 04 01	18.1	17.0
03 04 02	(11.7)	5.1-
03 04 03	16.1	10.4-
03 04 04	(12.7)	1.5-
03 04 05	23.7	15.3
03 04 06	(13.8)	2.3-
03 04 07	32.0	25.1-
03 04 08	(15.1)	11.4-
03 04 09	(15.5)	5.9
03 04 10	(16.4)	13.1
03 04 11	(17.0)	24.9
03 04 12	(17.6)	13.9
04 04 19-	22.7	12.5-
04 04 18-	25.7	25.1-
04 04 17-	27.4	25.1
04 04 16-	27.8	34.9-
04 04 15-	29.2	4.2
04 04 14-	38.4	37.6
04 04 13-	39.3	41.4-
04 04 12-	17.9	13.3
04 04 11-	17.4	15.0
04 04 10-	26.7	25.3-
04 04 09-	(12.7)	9.2
04 04 08-	(12.4)	3.7
04 04 07-	(12.2)	4.9-
04 04 06-	(12.0)	3.9-
04 04 05-	15.6	16.1
04 04 04-	(12.0)	7.2-
04 04 03-	40.6	40.4-
04 04 02-	(12.4)	2.1-
04 04 01-	16.4	15.6
04 04 00	22.7	18.5
04 04 01	37.4	38.5
04 04 02	(13.6)	8.1
04 04 03	44.4	49.3-
04 04 04	46.9	51.0-
04 04 05	(15.1)	1.6-
04 04 06	(15.7)	2.8
04 04 07	24.3	24.5
04 04 08	46.3	51.4
04 04 09	(17.3)	16.3-
04 04 10	(17.9)	17.1-
04 04 11	(18.5)	3.5
04 04 12	24.8	30.1-
05 04 16-	(16.2)	0.9-
05 04 15-	(15.7)	1.8-
05 04 14-	(15.3)	3.6-
05 04 13-	22.5	22.9-
05 04 12-	(14.5)	7.7
05 04 11-	17.0	17.0
05 04 10-	(14.1)	9.6
05 04 09-	19.5	19.2
05 04 08-	(13.6)	8.3-
05 04 07-	43.7	49.4-
05 04 06-	41.0	39.4-
05 04 05-	(13.6)	13.6
05 04 04-	29.3	28.1
05 04 03-	22.3	26.6
05 04 02-	58.7	60.0
05 04 01-	31.6	34.3-
05 04 00	55.0	57.7-
05 04 01	13.9	16.8
05 04 02	41.1	42.7-
05 04 03	(15.9)	5.7
05 04 04	51.4	54.0
05 04 05	29.6	29.4-
05 04 06	(17.3)	14.0
05 04 07	(17.9)	12.6
05 04 08	25.9	27.5-
06 04 14-	21.2	24.8
06 04 13-	(15.8)	7.6
06 04 12-	22.2	19.3
06 04 11-	23.3	21.2-
06 04 10-	49.8	54.8-
06 04 09-	18.1	18.7
06 04 08-	(15.1)	10.8-
06 04 07-	(15.1)	5.2-
06 04 06-	65.7	71.6
06 04 05-	32.0	32.2-
06 04 04-	(15.5)	8.8-
06 04 03-	22.4	30.6
06 04 02-	44.0	47.0-
06 04 01-	(16.1)	9.7
06 04 00	17.5	15.1
06 04 01	23.8	24.3-
07 04 16-	31.6	31.4
07 04 15-	(17.5)	9.8-
07 04 14-	(17.3)	20.8-
07 04 13-	20.5	23.0
07 04 12-	30.0	27.7-
07 04 11-	(16.7)	0.3
07 04 10-	28.3	26.1
07 04 09-	28.3	22.1-
07 04 08-	(16.7)	11.4
07 04 07-	(16.7)	7.2
07 04 06-	(16.7)	11.9-
07 04 05-	(17.0)	3.9
07 04 04-	(17.1)	0.4
07 04 03-	(17.3)	2.7
07 04 02-	(17.7)	1.8
07 04 01-	(17.8)	0.6
00 05 01	15.2	13.2-
00 05 02	48.8	43.8-
00 05 03	42.1	48.2
00 05 04	14.5	20.8-
00 05 05	78.3	65.4-
00 05 06	64.8	52.0
00 05 07	27.1	23.9-
00 05 08	(11.5)	4.0-
00 05 09	59.7	58.8
00 05 10	38.7	38.0-
00 05 11	(13.5)	6.1-
00 05 12	28.2	28.0
00 05 13	19.5	24.8-
00 05 14	(15.6)	8.5
00 05 15	(16.7)	5.8-
00 05 16	24.3	22.9-
00 05 17	19.6	5.8
01 05 17-	16.8	14.5-
01 05 16-	22.9	13.6-
01 05 15-	23.2	20.9-
01 05 14-	22.2	19.0
01 05 13-	19.9	26.4
01 05 12-	16.4	15.2
01 05 11-	(12.9)	5.5-
01 05 10-	19.9	19.2-
01 05 09-	14.8	5.9-
01 05 08-	39.1	32.0-
01 05 07-	33.8	29.4
01 05 06-	22.5	16.2
01 05 05-	65.2	69.4-
01 05 04-	51.2	64.3
01 05 03-	(6.5)	2.3-
01 05 02-	40.6	39.7-
01 05 01-	90.5	92.1
01 05 00	33.4	47.1-
01 05 01	44.1	45.5-
01 05 02	78.0	78.3
01 05 03	48.5	48.7-
01 05 04	9.3	7.8-
01 05 05	17.6	14.5
01 05 06	69.7	61.2-
01 05 07	(11.6)	13.4
01 05 08	40.0	41.9
01 05 09	26.7	23.3
01 05 10	21.8	18.3
01 05 11	23.9	23.8-
01 05 12	30.2	32.9-
01 05 13	32.6	22.6-
01 05 14	(16.8)	3.0
01 05 15	(17.3)	21.1
01 05 16	(18.3)	17.2
01 05 17	(18.8)	4.7
02 05 17-	(18.3)	3.9
02 05 16-	22.2	20.3
02 05 15-	22.5	24.2-
02 05 14-	14.4	17.0
02 05 13-	16.8	13.7
02 05 12-	41.1	42.3-
02 05 11-	44.6	49.5
02 05 10-	(12.0)	9.5-
02 05 09-	53.3	48.1-
02 05 08-	80.3	68.8
02 05 07-	36.2	31.9-
02 05 06-	48.2	37.6-
02 05 05-	50.2	39.9
02 05 04-	63.7	51.7-
02 05 03-	15.6	15.1
02 05 02-	91.5	79.7
02 05 01-	25.1	19.4
02 05 00	(8.9)	1.1
02 05 01	45.4	44.5-
02 05 02	58.5	55.6-
02 05 03	32.9	30.6-
02 05 04	32.7	33.4
02 05 05	58.7	65.7
02 05 06	26.2	23.6
02 05 07	(13.2)	0.7-
02 05 08	22.7	23.0-
02 05 09	31.2	30.9-
02 05 10	18.4	14.4-
02 05 11	(15.8)	11.8
02 05 12	(16.7)	11.2
03 05 18-	20.4	21.4
03 05 17-	(16.7)	7.5-
03 05 16-	26.7	24.5-
03 05 15-	24.6	25.4
03 05 14-	14.2	12.1-
03 05 13-	(14.2)	7.6
03 05 12-	41.6	45.2
03 05 11-	(12.9)	1.9-
03 05 10-	22.6	19.2-
03 05 09-	35.4	31.5-
03 05 08-	35.4	32.8-
03 05 07-	(11.0)	5.1-
03 05 06-	47.1	41.2
03 05 05-	80.9	74.9
03 05 04-	(10.2)	0.6-
03 05 03-	22.3	19.7-
03 05 02-	33.9	32.8-
03 05 01-	47.3	46.6-
03 05 00	(11.3)	3.7
03 05 01	35.9	38.6
03 05 02	29.6	27.2
03 05 03	(12.4)	6.2-
03 05 04	19.3	16.0
03 05 05	(13.3)	2.2
03 05 06	38.7	31.2-
03 05 07	23.3	25.5
03 05 08	(15.4)	10.7-
03 05 09	23.5	26.1-
03 05 10	25.7	27.9
03 05 11	(20.7)	7.0-
04 05 16-	15.7	16.1
04 05 15-	26.1	28.3
04 05 14-	14.6	8.8-
04 05 13-	21.2	21.3-
04 05 12-	15.3	12.7-
04 05 11-	21.5	25.0-
04 05 10-	11.1	14.9
04 05 09-	34.4	35.6
04 05 08-	18.4	15.8
04 05 07-	(14.3)	6.9-
04 05 06-	(14.3)	6.5-
04 05 05-	(14.3)	3.3
04 05 04-	38.7	42.3-
04 05 03-	26.9	27.9
04 05 02-	(14.5)	9.5
04 05 01-	41.5	47.3-
04 05 00	47.2	49.9
04 05 01	(13.3)	3.1
04 05 02	34.7	31.8-
04 05 03	45.5	49.9
04 05 04	21.7	22.5-
04 05 05	16.8	15.8-
04 05 06	31.0	35.2
04 05 07	22.7	19.8-
05 05 12-	25.1	23.9
05 05 11-	31.8	31.3-
05 05 10-	19.7	24.8
05 05 09-	15.4	15.1
05 05 08-	42.0	46.6-
05 05 07-	40.7	42.4
05 05 06-	24.3	6.8-
05 05 05-	34.7	36.3-
05 05 04-	44.7	48.4
05 05 03-	20.7	20.4-
05 05 02-	26.2	28.0-
05 05 01-	20.2	20.9
05 05 00	26.3	24.2-
05 05 01	14.9	10.6
05 05 02	31.5	31.5
06 05 15-	22.5	23.9-
06 05 14-	18.1	23.9
06 05 13-	(16.1)	7.3-
06 05 12-	29.3	26.5-
06 05 11-	23.1	20.6
06 05 10-	(15.5)	7.7-
06 05 09-	(15.5)	3.2
06 05 08-	31.1	31.1
06 05 07-	10.6	4.1-
06 05 06-	10.6	12.8-
06 05 05-	22.6	21.6-
06 05 04-	21.7	15.1-
06 05 03-	(15.8)	1.5-
06 05 02-	15.7	14.1
06 05 01-	32.2	29.4
06 05 00	(16.8)	2.3
00 06 00	103.2	105.6-
00 06 01	34.8	36.6
00 06 02	27.8	28.5
00 06 03	28.8	35.0
00 06 04	59.4	66.0
00 06 05	25.0	23.4-
00 06 06	56.6	44.9-
00 06 07	20.5	17.8-
00 06 08	17.5	12.9-
00 06 09	(12.4)	9.6
00 06 10	13.3	13.7
00 06 11	(13.9)	3.8
00 06 12	(14.7)	2.2
01 06 14-	33.1	34.4
01 06 13-	20.2	25.7-
01 06 12-	(13.9)	3.9-
01 06 11-	(13.3)	3.7-
01 06 10-	65.9	62.2-
01 06 09-	34.6	37.1
01 06 08-	52.5	44.4
01 06 07-	(10.1)	3.3
01 06 06-	44.7	44.9
01 06 05-	30.3	37.3-
01 06 04-	44.5	64.3-
01 06 03-	(7.2)	13.6-
01 06 02-	(6.0)	5.8
01 06 01-	(6.0)	16.1
01 06 00	17.1	23.4
01 06 01	(7.5)	6.4
01 06 02	(8.0)	14.0-
01 06 03	10.5	8.8-
01 06 04	(9.5)	10.9
01 06 05	(10.3)	4.6-
01 06 06	(11.3)	12.7-
01 06 07	(12.0)	3.8-
01 06 08	(12.8)	0.6-
01 06 09	(13.5)	1.4-
01 06 10	(14.1)	10.0
01 06 11	(14.9)	8.6
01 06 12	(15.6)	11.4-
02 06 14-	24.6	30.9-
02 06 13-	(14.4)	2.6
02 06 12-	(13.6)	10.5
02 06 11-	(12.1)	14.9
02 06 10-	(12.2)	12.2
02 06 09-	(11.5)	3.9
02 06 08-	14.1	15.8-
02 06 07-	20.9	17.5-
02 06 06-	18.3	16.8
02 06 05-	(9.2)	6.6-
02 06 04-	(8.8)	10.1-
02 06 03-	(8.4)	5.8
02 06 02-	(8.4)	3.7-
02 06 01-	(8.8)	6.6-
02 06 00	17.4	12.8
02 06 01	(9.5)	4.8
02 06 02	22.1	20.0-
02 06 03	(10.8)	3.3-
02 06 04	26.2	24.3
02 06 05	26.2	25.4-
02 06 06	(12.8)	1.8
02 06 07	(13.5)	6.5
02 06 08	38.5	39.2-
02 06 09	23.3	23.8
02 06 10	26.8	24.9
03 06 10-	(12.5)	6.2
03 06 09-	(12.0)	2.3
03 06 08-	(11.8)	6.5-
03 06 07-	(11.2)	0.9-
03 06 06-	28.6	24.8
03 06 05-	25.1	21.0-
03 06 04-	20.0	12.8-
03 06 03-	20.5	22.7
03 06 02-	49.2	46.6-
03 06 01-	26.5	24.5
03 06 00	57.4	58.8
03 06 01	36.3	37.0-
03 06 02	24.9	19.8
03 06 03	22.1	20.4-
03 06 04	56.6	63.1-
03 06 05	23.0	23.0
03 06 06	15.1	13.9
03 06 07	19.2	11.8
03 06 08	31.8	30.1
03 06 09	(16.3)	8.8-
03 06 10	21.6	14.2-
04 06 10-	49.6	48.6
04 06 09-	32.9	35.5-
04 06 08-	(12.0)	4.3-
04 06 07-	(12.5)	5.1
04 06 06-	57.2	63.0-
04 06 05-	34.4	36.9
04 06 04-	41.5	38.6
04 06 03-	(12.5)	1.0-
04 06 02-	33.4	32.2
04 06 01-	15.2	17.0-
04 06 00	35.0	36.5-

The final positional parameters are given as Set 2 in Table 1 and the anisotropic temperature parameters are listed in Table 2. The observed structure factors are compared in Table 3 with those calculated on the basis of the final positional and temperature parameters. The atomic scattering factors used for iodine and selenium were those of Thomas & Umeda (1957), corrected for dispersion for Mo $K\alpha$ radiation (Dauben & Templeton, 1955). For carbon, the diamond values of McWeeney (1954) were used. The final value of the usual residual, *R*, was 0·095% for all observed reflections.

Discussion of the structure

A projection of the structure of $C_4H_8Se_2.2\,I_2$ on (010) is shown in Fig. 1(*a*) while the molecular structure of the complex is shown in Fig. 2(*a*). The corresponding projection and molecular structure of $C_4H_8S_2.2\,I_2$ are given in Figs. 1(*b*) and 2(*b*) respectively. The observed

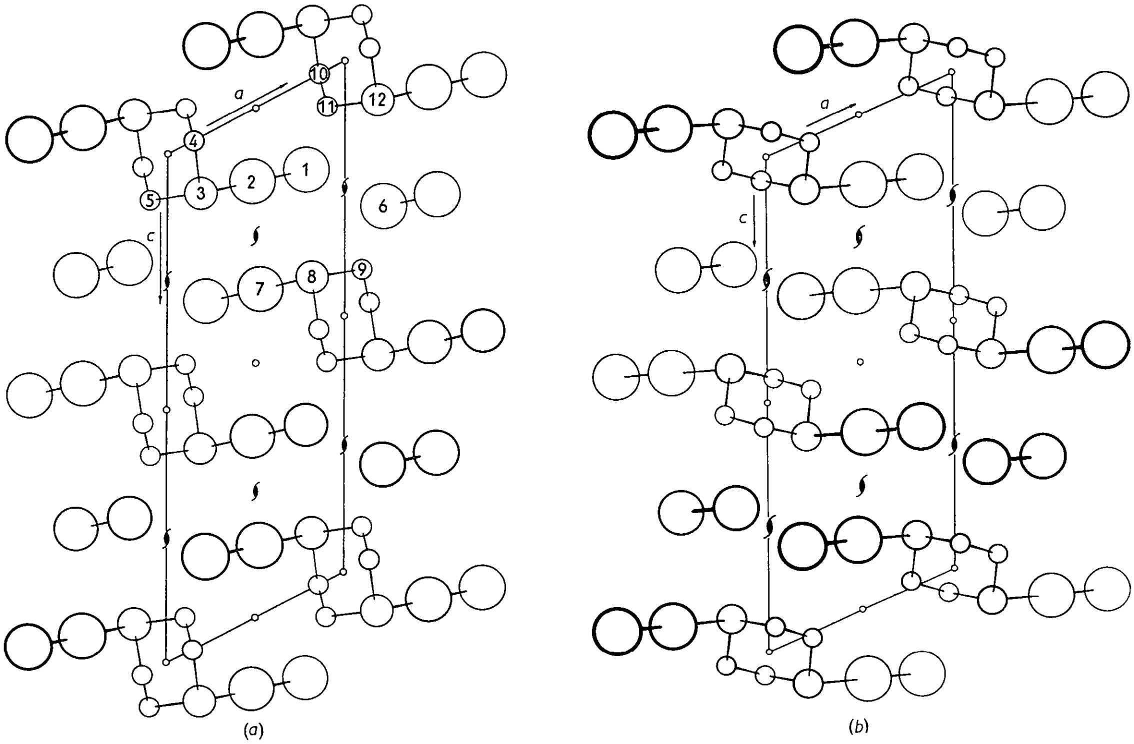

Fig. 1. (a) Projection of the structure of $C_4H_8Se_2.2\,I_2$ down the b axis.
(b) Projection of the structure of $C_4H_8S_2.2\,I_2$ down the b axis.

bond distances and bond angles for the diselenane complex are given in Table 4 where they are compared with the results of Marsh & McCullough (1951) for 1,4-diselenane. The structurally important non-bonded separations are given in Table 5.

Although the $C_4H_8Se_2.2\,I_2$ molecule is required only to be centrosymmetric, it has, within the standard deviations of the present study, the symmetry $2/m$. Deviations from the higher symmetry are much smaller than those found in $C_4H_8S_2.2\,I_2$. On the other hand, it appears that the 1,4-diselenane molecule is affected more by complexing with iodine than is the 1,4 dithiane molecule. The structure of the latter is affected less than the standard deviations involved but the Se · · · Se distance across the diselenane ring is lengthened from $3{\cdot}66 \pm 0{\cdot}02$ to $3{\cdot}746 \pm 0{\cdot}005$ Å by complexing with iodine. At the same time, the angles $Se–C_1–C_2'$ and $Se–C_2–C_1'$ are increased from the average value of $108 \pm 3°$ to an average of $115 \pm 2°$.

The most striking differences between the structures of the iodine complexes of 1,4-dithiane and 1,4-diselenane are the angles of attachment of iodine to the ring systems and the bonded distances involving iodine. These differences are shown in Fig. 2 and in Table 6. In the dithiane complex, the iodine molecules are attached to the ring in the equatorial positions whereas the attachment in the diselenane complex is in the axial positions. The reason for this interesting difference in bonding is not at present clear as there does not appear to be any appreciable intramolecular crowding in either form of the iodine complex with either dithiane or diselenane.

A comparison of the S–I and Se–I distances indicates a stronger bond in the latter case. This is also suggested by the significantly longer I–I bond in the diselenane complex and by the lower dissociation constant of $C_4H_8Se_2.I_2$ ($K = 3{\cdot}55 \times 10^{-3}$ mol./l.) when compared to that of $C_4H_8S_2.I_2$ ($K = 1{\cdot}30 \times 10^{-2}$ mol./l.) in carbon tetrachloride solution at 25 °C (McCullough & Zimmermann, 1961). It is interesting to note that

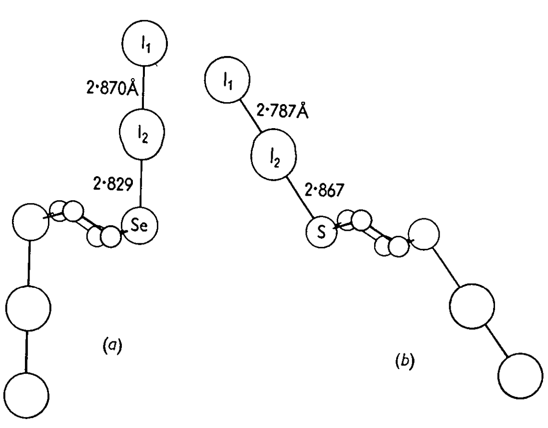

Fig. 2. (a) The molecular structure of $C_4H_8Se_2.2\,I_2$.
(b) The molecular structure of $C_4H_8S_2.2\,I_2$.

Table 4. *Bond distances and angles in* $C_4H_8Se_2.2\,I_2$ *compared with those in* $C_4H_8Se_2$ (*Marsh & McCullough*, 1951)

(*a*) Distances in Å. (Standard deviations in parentheses)

	$C_4H_8Se_2.2\,I_2$	$C_4H_8Se_2$
I_1–I_2	2·870 (0·003)	—
I_2–Se	2·829 (0·004)	—
Se–C_1	1·947 (0·024)	1·99 (0·04)
Se–C_2	1·980 (0·024)	2·04 (0·06)
C_1–C_2	1·568 (0·030)	1·54 (0·04)
Se · · · Se	3·746 (0·005)	3·66 (0·02)

(*b*) Angles in degrees. (Standard deviations in parentheses)

	$C_4H_8Se_2.2\,I_2$	$C_4H_8Se_2$
I_1–I_2–Se	180·0 (0·3)	—
I_2–Se–C_1	101·5 (1·0)	—
I_2–Se–C_2	100·5 (1·0)	—
I_2–Se · · · Se′	89·4 (0·5)	—
C_1–Se–C_2	100·5 (1·8)	97·6 (3·5)
Se–C_1–C_2′	117·2 (2·0)	108·5 (3·0)
Se–C_2–C_1′	113·0 (2·0)	107·8 (4·0)

Table 5. *Shortest non-bonded interatomic separations in* $C_4H_8Se_2.2\,I_2$

(Standard deviations: I–I, 0·003 Å; I–Se, 0·004 Å; I–C, 0·002 Å)
(See Fig. 1 for identification numbers of atoms)

(*a*) Atoms within a given molecule

I–C	2–4	3·74 Å
	2–5	3·75

(*b*) Atoms in different molecules

I–I	1– 2′	4·550 Å
	1– 6	4·329
I–Se	1– 8	3·889
	1–12	4·022
	2– 8	4·008
I–C	1– 9	3·92
	1–10	4·06
	1–11	4·11
	2–10	4·37

Table 6. *Comparison of bond distances in the iodine complexes of diselenane and dithiane*

Complex	Bond	Obs. dist.	Radius sum	Diff.
$C_4H_8Se_2.2\,I_2$	Se–I	2·829 Å	2·50 Å	0·33 Å
	I–I	2·870	2·66	0·21
$C_4H_8S_2.2\,I_2$	S–I	2·867	2·37	0·50
	I–I	2·787	2·66	0·13

the first stage of the dissociation (corresponding to the loss of the first of the two iodine molecules) is complete in both complexes in carbon tetrachloride solution at 25 °C.

The behavior of the temperature parameters in $C_4H_8Se_2.2\,I_2$ is similar to that in the dithiane complex. In the present study $B_{ii}(I_1) > B_{ii}(I_2) > B_{ii}(Se)$. As pointed out in the discussion of the dithiane complex, this apparently anomolous behavior of higher temperature parameters for the heavier atoms is understandable in view of the nature of the structure.

The structure of the iodine complexes of dithiane and diselenane represent an interesting case of pseudo isomorphism. The close similarity of the unit-cell dimensions and the fact that both structures are based on $P2_1/c$ strongly suggest that the two substances are isomorphous. However, the great difference in molecular structures prevents true isomorphism. The difference between the two structures is greatest in the *y* parameters, as shown by comparison of the *F* values for 0*k*0 in the two substances which are as follows:

	$C_4H_8S_2.2\,I_2$		$C_4H_8Se_2.2\,I_2$	
	F_o	F_c	F_o	F_c
020	123	−121·8	53	−52·1
040	112	−104·0	—	1·2
060	74	70·4	103	105·6

A study of the structure of the iodine complex of 1,4-selenothiane is planned.

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