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The Crystal and Molecular Structure of the Iodine Complex of 1-Oxa-4-selenacyclohexane, $C_4H_8OSe\cdot I_2$

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The molecular complex $C_4H_8OSe\cdot I_2$ forms deep purple monoclinic crystals with $a = 6.232$ A, $b = 16.089$ A, $c = 9.162$ A, and $\beta = 101.12°$. The space group symmetry is $P2_1/c$ with four molecules in the unit cell. The trial structure was refined by use of three-dimensional, full-matrix, least-squares procedures involving some 1420 unique observed reflections. The molecule approximates the symmetry **m** with the six-membered ring in the chair configuration and the iodine molecule bonded to selenium in the axial position. The Se–I(2) bond distance of 2.755 A is the shortest of its kind observed so far and the I(1)–I(2) distance of 2.956 A is unusually long. The Se–I(2)–I(1) bond angle is 174.8°. Selenium forms a weaker bond with a second iodine molecule with Se–I(1) = 3.708 A and the angle Se–I(1)–I(2) = 77.4°. The oxygen atom is not involved in bonding outside its own ring.

Introduction

The present communication reports another in a series of investigations of the structures of halogen addition compounds of organoselenides and organotellurides carried out in these laboratories.[1] With one exception,[2] the previous studies have involved donor molecules where only one kind of atom was available for combination with the halogen. The present complex was of interest because it offered the possibility of iodine bonding not only with selenium (on which primary bonding was anticipated) but also, more weakly, with oxygen. Although no involvement of oxygen in the complex bonding was found, the selenium–iodide bond system was observed to be of a new type and hence of interest in its own right.

Experimental Section

1-Oxa-4-selenacyclohexane (1,4-oxaselenane) was synthesized by the method of McCullough and Lefohn.[3] The iodine addition compound was prepared by mixing approximately stoichiometric quantities of the components dissolved in carbon tetrachloride. The resulting solid was recrystallized from ethylene chloride, and specimens for the X-ray study were grown by evaporation of solutions in that solvent. The crystals were mostly six-sided prisms elongated on the *a* axis of the monoclinic unit. Because of the high absorption, molybdenum radiation was used for the initial diffraction photographs. However, owing to an unfortunate combination of circumstances (fluorescence of Se in Mo K radiation, a high temperature factor, and overlap of diffraction spots on integrated molybdenum Weissenberg photographs), the intensity data were collected with Cu Kα radiation and corrected for absorption. Since the crystals are somewhat volatile, the selected specimens were sealed in thin-walled capillaries.

Weissenberg and precession photographs showed monoclinic diffraction symmetry. The unit cell dimensions were determined by means of $0kl$ Weissenberg photographs prepared with Cu K radiation and precession photographs of the $hk0$ and $h0l$ nets prepared with Mo K radiation. The $hk0$ reflections of a small quartz crystal were superimposed on these films for calibration purposes.[4] On the basis of $a = 4.9131$ A for quartz, the lattice parameters for the iodine complex were found to be: $a = 6.232 \pm 0.010$ A, $b = 16.089 \pm 0.015$ A, $c = 9.162 \pm 0.015$ A, and $\beta = 101.12 \pm 0.10°$. The density observed by flotation was 2.96 g cm^{-3} while that calculated for four molecules in the unit cell was 2.983 g cm^{-3}. The photographs showed systematic extinctions for $h0l$ with l odd and for $0k0$ with k odd, thus indicating the space group $P2_1/c$.

The intensity data were taken from multiple-film, integrated, Weissenberg photographs about the *a* axis prepared by use of Cu Kα radiation. Six levels, $0kl$ through $5kl$, were photographed in this manner. The intensities were estimated visually by use of a set of timed, integrated exposures prepared with the same crystal, and the initial interlayer scaling was based on the exposure times at constant X-ray intensity. In addition to the usual geometric factors, corrections were made for absorption in the specimen. The crystal used for the intensity data was a six-sided prism with principal faces of the form $\{010\}$ (0.050 mm between opposite pairs) and of the form $\{011\}$ (0.070 mm between opposite pairs). Absorption corrections were made on the assumption of a cylindrical specimen with the same cross-sectional area, 4.0×10^{-3} mm^2. For Cu Kα radiation the value of μR was calculated to be 2.0, and transmission factors were estimated by use of tables given by Buerger.[5]

Determination and Refinement of the Structure

Two-dimensional Patterson summations on $0kl$ and $hk0$ were prepared, but consideration of these did not lead to a satisfactory trial structure. Although a three-dimensional Patterson synthesis probably would have served the purpose, it was of interest to try a new program for phase determination written by Long.[6] This program for the IBM 7094 is based on a reiterative application of Sayre's equation.[7] The present structure was one of several cases which Long used in developing and testing the program. For $C_4H_8OSeI_2$, 189 reflections with values of $|E| \geq 1.50$ were used. The solution which gave the highest consistency index ($C = 1.000$) and lowest number of cycles (4) proved to be the true solution, and all predicted signs were correct. A Fourier synthesis based on these $|E|$ values and signs gave the heavy-atom trial parameters shown below. The structure determination and refinement

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[1] H. Hope and J. D. McCullough, *Acta Cryst.*, **17**, 712 (1964), and the references given therein.

[2] H. Hope and J. D. McCullough, *ibid.*, **15**, 806 (1962).

[3] J. D. McCullough and A. Lefohn, *Inorg. Chem.*, **5**, 150 (1966).

[4] A. Pabst, *Am. Mineralogist*, **42**, 664 (1957).

[5] M. J. Buerger, "Crystal-Structure Analysis," John Wiley and Sons, Inc., New York, N. Y., 1960, pp 210, 213.

[6] R. E. Long, Ph.D. Thesis, University of California at Los Angeles, 1965.

[7] D. Sayre, *Acta Cryst.*, **5**, 60 (1952).

	x	y	z
I(1)	0.08	0.36	0.42
I(2)	0.43	0.26	0.34
Se	0.77	0.18	0.26

were then carried out in the nine following steps.

(1) The positions of the three heavy atoms were refined by least-squares procedures with isotropic temperature factors taken initially as 4.0 for I(1), 3.5 for I(2), and 3.0 for Se. These values were based on results obtained in previous studies of similar iodine complexes.

(2) The interlayer scaling was checked and adjusted by use of the ratios $\Sigma|F_o|/\Sigma|F_c|$ for the various layers. The $|F_c|$ values were based on the heavy atoms only at this stage.

(3) The positions of the heavy atoms were again refined by least-squares procedures with anisotropic temperature parameters derived from the refined isotropic values of 4.4 for I(1), 3.5 for I(2), and 3.7 for Se.

(4) A three-dimensional difference synthesis was prepared in which the heavy atoms were subtracted at their refined positions. The positions of the four carbon atoms and the oxygen atom were clearly revealed.

(5) The parameters for all atoms (except hydrogen) were refined by the method of least squares. Anisotropic temperature parameters were used on I and Se and isotropic parameters on C and O.

(6) The interlayer scaling was again checked, and small adjustments were made by the procedure indicated above. The $|F_c|$ value included all atoms except hydrogen.

(7) A final set of least-squares cycles was computed, including a set of esd values for all parameters. The results are listed in Tables I and II. Refinement was carried out until the shifts in parameters were much less than their respective esd values. The maximum ratio of final shift to esd was 0.18, and the average was 0.08.

TABLE I

FINAL ATOMIC POSITIONAL PARAMETERS IN $C_4H_8OSeI_2$ WITH THEIR ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

Atom	x	y	z
I(1)	0.0757	0.3707	0.4233
	(0.0002)	(0.0001)	(0.0001)
I(2)	0.4226	0.2681	0.3347
	(0.0002)	(0.0001)	(0.0001)
Se	0.7704	0.1850	0.2569
	(0.0004)	(0.0001)	(0.0002)
C(1)	0.6610	0.0790	0.1779
	(0.0033)	(0.0012)	0.0020)
C(2)	0.5555	0.0297	0.2882
	(0.0044)	(0.0015)	(0.0027)
C(3)	0.7593	0.0797	0.5126
	(0.0032)	(0.0013)	(0.0021)
C(4)	0.9121	0.1385	0.4523
	(0.0038)	(0.0013)	(0.0023)
O	0.7114	0.0100	0.4188
	(0.0024)	(0.0009)	(0.0015)

TABLE II

FINAL THERMAL PARAMETERS WITH THEIR ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

(A) Anisotropic Parameters[a] for Heavy Atoms in A^2

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
I(1)	6.16	3.46	6.00	0.28	1.58	0.02
	(0.09)	(0.07)	(0.06)	(0.04)	(0.05)	(0.01)
I(2)	5.92	2.72	4.57	−0.11	0.80	0.00
	(0.09)	(0.05)	(0.07)	(0.04)	(0.04)	(0.01)
Se	6.68	2.76	5.12	0.13	1.62	0.03
	(0.13)	(0.07)	(0.10)	(0.06)	(0.08)	(0.01)

(B) Isotropic Parameters for Light Atoms in A^2

	C(1)	C(2)	C(3)	C(4)	O
B	5.01	6.09	5.26	5.55	5.60
	(0.32)	(0.46)	(0.33)	(0.38)	(0.26)

[a] The relationships between the tabulated B values and the b values in the anisotropic temperature factor expression are given by: $B_{11} = 4a^2(\sin^2 \beta)b_{11}$, $B_{22} = 4b^2b_{22}$, $B_{12} = 2ab(\sin \beta)b_{12}$, etc.

(8) A final difference Fourier was computed in which all atoms except hydrogen were subtracted. This showed fluctuations within the range ±1.5 e A^{-3} with esd of the electron density ~0.62 e A^{-3}. Although 12 of the maxima were of the right order of magnitude to be H atoms, their coordinates did not correspond to expected positions.

(9) A final set of structure factors was computed and listed in Table III. The final values of the usual index, R, were as follows: (a) all 1435 observed reflections except 16 designated by E in Table III and believed to show effects of extinction, $R = 0.100$; (b) all observed reflections (except 16 designated by E) plus 295 reciprocal lattice points within the geometric range for which the intensity was below the observational level, $R = 0.110$; (c) all 1730 reciprocal lattice points listed in Table III, $R = 0.119$.

Computing Procedures

The Patterson, Fourier, and difference syntheses involved in this study were computed by use of an unpublished routine written for the IBM 7094 by P. K. Gantzel and H. Hope in these laboratories. The structure factor part of the routine provides for either isotropic or anisotropic temperature factors, and the phasing of the Fourier synthesis may be based on any number of specified atoms. The contribution and phase of each atom is printed out for each reflection. The least-squares refinement was carried out by use of ACA Computer Program No. 317 (UCLALS-1) written by P. K. Gantzel, R. A. Sparks, and K. N. Trueblood for the IBM 7094. This program minimizes the weighted sum of the squares of the quantity ($KF_o - GF_c$) by a full-matrix routine, where K and G are scale factors. The program provides for several weighting options and for either isotropic or anisotropic temperature factors on the individual atoms. The anisotropic temperature factors are of the form

$$\exp[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)]$$

The weighting scheme used for the observed reflections was that of Hughes[8] with $4F_o(\min) = 2.8$. The unobserved reflections were not included in the refinement. The standard deviations of the positional and temperature parameters were estimated from the inverse matrix of the normal equations.

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(8) E. W. Hughes, *J. Am. Chem. Soc.*, **63**, 1737 (1941).

TABLE III
OBSERVED AND CALCULATED STRUCTURE FACTORS[a]

H= 2, L= -9		
4	35	31
5	16	-16
6	48	-45
7	15	16
8	18	19
9	8	-7
10	3U	6
11	3U	4
12	2U	1
13	10	-12
H= 2, L= -10		
0	8	2
1	8	7
2	14	12
3	8	3
4	39	-34
5	8	-7
6	33	30
7	3U	-3
8	9	-10
9	2U	1
10	8	9
11	1U	3
H= 2, L= 11		
1	6	7
2	12	11
3	2U	-3
4	14	-14
5	12	-12
6	10	13
7	11	15
H= 3, L= 0		
1	189E	-240
2	27	35
3	44	50
4	18	-21
5	6	9
6	7	-3
7	72	63
8	29	22
9	66	-55
10	4U	-6
11	11	-13
12	3U	-5
13	21	17
14	20	16
15	28	27
16	2U	-4
17	25	-27
18	2U	-0
19	13	-19
H= 3, L= 1		
1	28	29
2	74	-81
3	24	-28
4	11	14
5	35	-42
6	48	54
7	3U	-6
8	22	26
9	82	79
10	101	-93
11	118	-106
12	66	58
13	56	50
14	3U	-3
15	18	-17
16	10	10
17	13	14
18	22	-25
H= 3, L= 2		
0	193	191
1	48	48
2	94	-95
3	79	-83
4	14	15
5	23	21
6	20	-21
7	32	34
8	74	82
9	29	-29
10	24	-24
11	3U	-11
12	24	-23
13	26	25
14	3U	4
15	10	-10
16	23	21
17	2U	-4
18	1U	-0
H= 3, L= 3		
1	55	54
2	3U	5
3	108	-104
4	11	-10
5	11	-10
6	32	32
7	33	33
8	50	-51
9	14	15
10	53	52
11	25	-22
12	64	-57
13	24	-24
14	46	42
15	22	20
16	24	-24
17	15	17
H= 3, L= 4		
0	28	-24
1	38	37
2	65	64
3	8	-9
4	88	-83
5	25	27
6	34	33
7	59	-54
8	26	27
9	58	55
10	3U	1
11	3U	-4
12	32	-30
13	15	-17
14	12	10
15	13	13
16	10	13
17	1U	-3
H= 3, L= 5		
1	8	11
2	46	45
3	19	20
4	8	-8
5	61	-59
6	35	-33
7	53	49
8	15	15
9	9	-13
10	5U	-3
11	3U	5
12	11	10
13	24	-21
14	29	-27
15	27	28
H= 3, L= 6		
0	3U	4
1	3U	6
2	3U	-4
3	56	49
4	12	-11
5	71	-63
6	4U	8
7	23	24
8	15	-13
9	3U	-5
10	10	10
11	19	19
12	2U	-3
13	10	-10
14	1U	-5
H= 3, L= 7		
1	3U	6
2	12	10
3	12	-12
4	16	15
5	24	25
6	34	-34
7	21	-22
8	16	16
9	8	10
10	2U	6
11	2U	-2
12	1U	3
H= 3, L= 8		
0	3U	0
1	6	6
2	20	-18
3	2U	-1
4	31	30
5	2U	3
6	20	-21
7	13	-15
8	7	8
9	10	14
H= 3, L= 9		
1	2U	-7
2	10	11
3	10	11
4	10	-11
5	1U	3
6	7	10
H= 3, L= -1		
1	19	-19
2	89E	-125
3	2U	-2
4	66	65
5	17	16
6	56	55
7	15	15
8	3U	-0
9	115	-95
10	94	82
11	128	107
12	33	30
13	65	-61
14	28	26
15	31	31
16	2U	6
17	19	-21
18	26	-31
19	10	12
H= 3, L= -2		
0	161	-150
1	70	69
2	72	74
3	119	126
4	2U	-2
5	54	63
6	61	64
7	38	37
8	113	-106
9	61	-55
10	42	37
11	27	-25
12	27	24
13	47	44
14	23	-20
15	15	-14
16	8	-8
17	9	-9
18	1U	-6
H= 3, L= -3		
1	92	-90
2	13	11
3	139	136
4	43	-41
5	20	22
6	86	91
7	45	-46
8	57	-60
9	3U	-3
10	41	35
11	9	10
12	38	-35
13	46	42
14	53	51
15	37	-37
16	34	-37
17	7	-14
18	18	23
H= 3, L= -4		
0	29	-28
1	3U	-2
2	83	-80
3	15	15
4	134	131
5	29	29
6	79	-78
7	65	-68
8	16	-17
9	63	62
10	15	-16
11	3U	-4
12	46	44
13	23	-22
14	2U	-7
15	16	17
16	32	-32
17	11	-15
18	14	19
H= 3, L= -5		
1	14	-5
2	34	32
3	16	-9
4	20	18
5	108	103
6	65	-68
7	84	-84
8	25	26
9	3U	5
10	3U	4
11	25	23
12	9	10
13	24	24
14	35	-35
15	37	-39
16	19	22
17	17	23
H= 3, L= -6		
0	18	-15
1	15	13
2	3U	3
3	88	77
4	11	10
5	102	-99
6	3U	-1
7	45	44
8	20	-24
9	10	-12
10	7	3
11	30	33
12	10	11
13	12	-13
14	5	-4
15	22	-25
16	7	-6
H= 3, L= -7		
1	20	-17
2	49	48
3	43	41
4	8	8
5	48	-46
6	48	-49
7	32	35
8	17	20
9	11	-14
10	8	8
11	2U	7
12	2U	4
13	10	-10
14	25	-26
15	14	18
H= 3, L= -8		
0	20	-15
1	18	17
2	32	29
3	3U	4
4	47	-44
5	30	6
6	24	25
7	40	-43
8	2U	0
9	33	39
10	2U	6
11	2U	1
12	12	-14
13	14	-17
14	4	4
H= 3, L= -9		
1	21	18
2	19	19
3	33	-32
4	13	-14
5	2U	5
6	6	5
7	6	10
8	14	-17
9	2U	1
10	25	25
11	1U	-2
12	15	-19
H= 3, L= -10		
0	41	37
1	22	20
2	18	-16
3	13	-15
4	2U	1
5	2U	2
6	10	-10
7	2U	6
8	19	22
9	1U	-3
H= 3, L= -11		
1	1U	3
2	1U	-5
3	3	-4
4	3	5
5	6	-8
6	1U	-2
H= 4, L= 0		
1	11	4
2	1U	9
3	95	-94
4	2U	4
5	97	105
6	39	41
7	30	-27
8	64	-55
9	30	-1
10	28	24
11	43	-34
12	17	16
13	27	25
14	18	-16
15	22	21
16	7	8
17	24	-27
H= 4, L= 1		
1	13	-14
2	84	-85
3	26	25
4	35	36
5	51	-55
6	39	42
7	57	54
8	25	-25
9	35	-34
10	23	-21
11	22	20
12	14	-14
13	26	-25
14	39	36
15	30	30
16	8	-9
17	19	-24
H= 4, L= 2		
0	35	-31
1	62	-63
2	57	57
3	20	10
4	63	-64
5	6	-10
6	6	6
7	65	66
8	14	18
9	54	-49
10	2U	-4
11	2U	2
12	24	-20
13	24	22
14	18	18
15	1U	-2
16	1U	1
17	1U	0
H= 4, L= 3		
1	48	44
2	19	-19
3	55	-49
4	3U	-2
5	3U	6
6	3U	0
7	20	19
8	30	31
9	21	22
10	58	-56
11	23	-24
12	44	40
13	7	-5
14	19	-21
15	9	9
16	10	13
H= 4, L= 4		
0	98	91
1	22	-22
2	69	-60
3	3U	8
4	3U	-0
5	3U	-6
6	16	-18
7	2U	-4
8	32	31
9	2U	8
10	14	-13
11	2U	-1
12	10	-10
13	5	-6
14	5	-2
15	5	6
H= 4, L= 5		
1	14	14
2	14	13
3	15	-15
4	2U	1
5	9	-7
6	2U	-1
7	11	11
8	13	-15
9	28	30
10	30	32
11	32	-34
12	23	-26
13	11	12
14	8	12
H= 4, L= 6		
0	2U	-7
1	51	47
2	13	15
3	19	-17
4	15	-17
5	5	6
6	2U	-2
7	16	-16
8	8	9
9	13	14
10	1U	-2
11	1U	-0
12	5	-8
H= 4, L= 7		
1	2U	2
2	21	22
3	2U	0
4	6	-8
5	4	-6
6	4	-7
7	9	12
8	1U	0
9	11	-14
10	5	7
H= 4, L= 8		
0	8	-9
1	8	-11
2	7	9
3	16	16
4	5	-7
5	11	-14
6	7	8
7	1U	-1
H= 4, L= -1		
1	16E	-38
2	9	-14
3	2U	2
4	51	-53
5	72	71
6	104	99
7	73	-62
8	48	-43
9	2U	-1
10	17	-16
11	9	4
12	2U	-0
13	22	20
14	35	35
15	35	-36
16	29	-32
17	9	12
H= 4, L= -2		
0	37	-30
1	7	-6
2	33	-33
3	11	-10
4	131	135
5	2U	5
6	88	-82
7	34	30
8	15	15
9	22	-20
10	23	-20
11	6	4
12	46	42
13	11	11
14	8	8
15	7	-6
16	36	-38
17	1U	-1
H= 4, L= -3		
1	63	-62
2	35	-35
3	50	50
4	62	59
5	29	34
6	65	-67
7	60	-63
8	32	35
9	2U	-4
10	10	-8
11	10	8
12	14	14
13	36	31
14	25	-25
15	34	-34
16	25	25
17	1U	-0
H= 4, L= -4		
0	43	-33
1	34	-29
2	27	24
3	85	78
4	16	11
5	67	-65
6	41	40
7	6	-4
8	80	-81
9	12	11
10	36	35
11	16	16
12	26	24
13	26	-24
14	19	-19
15	1U	-4
16	1U	2
17	10	13
H= 4, L= -5		
1	22	-21
2	84	78
3	29	27
4	36	-31
5	2U	-4
6	17	-16
7	16	17
8	6	9
9	42	-44
10	16	17
11	54	48
12	8	9
13	31	-29
14	20	-22
15	14	14
16	1U	3
H= 4, L= -6		
0	49	-45
1	94	82
2	39	32
3	14	-10
4	24	-24
5	6	-4
6	6	-7
7	42	-41
8	15	16
9	37	36
10	7	-8
11	2U	2
12	7	-7
13	15	-15
14	11	11
15	6	-7
H= 4, L= -7		
1	12	12
2	19	17
3	15	-12
4	6	3
5	3U	3
6	23	-22
7	2U	4
8	17	-16
9	23	24
10	51	50
11	25	-25
12	35	-35
13	8	7
14	7	8
H= 4, L= -8		
0	92	74
1	2U	4
2	48	-42
3	10	10
4	2U	-2
5	11	-9
6	5	-7
7	5	-6
8	14	15
9	8	9
10	8	-7
11	3	6
12	6	-7
13	5	-7
H= 4, L= -9		
1	17	15
2	7	6
3	20	-17
4	6	-6
5	4	-3
6	2U	-4
7	6	7
8	14	15
9	12	13
10	18	-19
11	10	-14
H= 4, L= -10		
0	6	-6
1	17	-17
2	15	15
3	11	10
4	21	-20
5	6	-6
6	1U	-1
7	10	11
8	10	12
H= 4, L= -11		
1	1U	2
2	9	-12
3	1U	1
H= 5, L= 0		
1	35	-32
2	49	-46
3	5	5
4	64	57
5	8	-8
6	24	-21
7	58	48
8	29	-22
9	49	-39
10	2U	0
11	8	3
12	22	20
13	15	17
14	9	-10
15	3	-6
H= 5, L= 1		
1	2	7
2	56	-47
3	18	-16
4	11	8
5	57	48
6	33	31
7	33	-27
8	17	-15
9	15	15
10	6	-6
11	9	-9
12	8	-8
13	17	17
14	21	24
15	16	-20
H= 5, L= 2		
0	4U	-6
1	2U	2
2	2U	-3
3	61	-47
4	2U	1
5	71	60
6	16	-15
7	21	-21
8	16	14
9	2U	-2
10	9	-11
11	20	-21
12	4	-1
13	10	11
14	4	5
H= 5, L= 3		
1	2U	5
2	10	-8
3	28	5
4	2U	-16
5	33	-30
6	48	38
7	32	30
8	14	-14
9	4	-6
10	2U	-5
11	4	-1
12	3	-3
13	7	-9
14	11	17
H= 5, L= 4		
0	6	5
1	10	-7
2	23	21
3	2U	-1
4	46	-40
5	2U	-2
6	29	24
7	16	16
8	8	-8
9	13	-15
10	7	9
11	1U	-3
12	9	11
H= 5, L= 5		
1	13	13
2	10	-11
3	18	-18
4	13	12
5	7	-7
6	17	-18
7	11	13
8	12	16
9	1U	-0
10	6	-8
11	2	-3
H= 5, L= 6		
0	16	17
1	11	-13
2	7	-8
3	15	15
4	1U	2
5	10	-11
6	8	-10
7	1U	1
8	14	19
9	1U	1
H= 5, L= 7		
1	1U	1
2	8	11
3	5	-5
4	3	-6
5	1U	2
H= 5, L= -1		
1	33	-38
2	14	-20
3	57	55
4	7	2
5	7	3
6	9	-7
7	22	-19
8	30	26
9	13	-12
10	48	-42
11	16	15
12	40	38
13	12	16
14	19	-23
15	7	-11
16	8	10
H= 5, L= -2		
0	105	-103
1	26	-26
2	62	57
3	32	34
4	5	7
5	9	-9
6	13	10
7	19	-17
8	55	-47
9	15	13
10	18	16
11	2U	2
12	16	17
13	10	-10
14	1U	-2
15	6	7
16	9	-15
H= 5, L= -3		
1	21	-18
2	28	23
3	18	17
4	2U	-1
5	21	19
6	16	-15
7	7	-9
8	15	-16
9	49	-43
10	46	40
11	55	52
12	27	-26
13	19	-22
14	3	5
15	1U	3
H= 5, L= -4		
0	2U	0
1	118	95
2	15	-12
3	18	-13
4	23	22
5	5	-4
6	5	4
7	22	-24
8	15	-13
9	27	25
10	8	6
11	6	5
12	10	11
13	6	-8
14	5	-8
15	7	-10
H= 5, L= -5		
1	9	-9
2	52	41
3	5	2
4	24	-22
5	5	-4
6	17	-19
7	12	-15
8	5	-2
9	27	29
10	24	26
11	32	-35
12	6	-8
13	2U	23
14	9	-11
H= 5, L= -6		
0	45	40
1	19	-15
2	19	-16
3	44	40
4	10	8
5	28	-28
6	17	-16
7	13	16
8	28	32
9	17	20
10	10	-11
11	5	8
12	6	-7
13	14	-19
H= 5, L= -7		
1	21	21
2	2U	1
3	30	-30
4	13	13
5	2U	-3
6	23	-26
7	8	9
8	15	20
9	4	-1
10	2	-3
11	1U	1
12	6	11
H= 5, L= -8		
0	6	6
1	7	5
2	14	14
3	4	6
4	33	-36
5	9	-11
6	15	18
7	10	13
8	1U	3
9	8	-13
10	4	3
H= 5, L= -9		
1	4	4
2	4	-2
3	4	5
4	5	-5
5	17	-21
6	8	11
7	13	18
8	5	-6
H= 5, L= -10		
0	4	5
1	3	-1
2	3	-2
3	9	-12
4	3	-5
H= 0, L= 0		
2	127E	-227
4	16	-16
6	22	-18
8	138	133
10	35	-30
12	45	-40
14	5U	-4
16	78	61
18	16	-12
20	50	-49
H= 0, L= 1		
1	78	76
2	2U	8
3	119E	-164
4	5	10
5	38	-55
6	41	-57
7	27	31
8	94	-93
9	98	96
10	200	179
11	139	-122
12	162	-141
13	8	11
14	60	53
15	3	8
16	41	-32
17	38	35
18	24	23
19	43	-38
20	8	-9
H= 0, L= 2		
0	69	-81
1	213E	276
2	128E	154
3	52	-59
4	138	-164
5	13	9
6	3	3
7	95	-121
8	88	109
9	128	134
10	24	-26
11	18	16
12	60	-54
13	45	-40
14	58	49
15	3	-10
16	3	1
17	20	18
18	3U	-6
19	22	27
20	3	4
H= 0, L= 3		
1	23	-21
2	176E	210
3	38	36
4	78	-93
5	42	-53
6	45	-59
7	87	100
8	20	22
9	68	-79
10	27	30
11	77	73
12	21	20
13	101	-84
14	73	-64
15	64	56
16	3	6
17	38	-34
18	47	43
19	25	24
H= 0, L= 4		
0	26	-25
1	32	-31
2	15	18
3	169	183
4	31	-31
5	142	-159
6	73	79
7	4U	-0
8	109	-117
9	30	32
10	44	50
11	38	47
12	18	17
13	56	-48
14	39	-33
15	10	-11
16	17	17
17	44	35
18	14	-13
19	1	-6
H= 0, L= 5		
1	43	-45
2	3	-5
3	49	52
4	87	93
5	52	54
6	123	-130
7	58	-63
8	48	48
9	14	13
10	5U	6
11	5U	3
12	13	9
13	48	43
14	54	-48
15	45	-40
16	49	42
17	10	9
18	23	-24
H= 0, L= 6		
0	31	-32
1	20	22
2	50	-48
3	8	11
4	125	125
5	20	-21
6	109	-107
7	5	6
8	18	16
9	5U	6
10	29	-29
11	15	13
12	44	37
13	3	-5
14	13	9
15	3U	-8
16	48	-41
17	3	8
H= 0, L= 7		
1	18	-20
2	20	20
3	18	21
4	52	-51
5	58	59
6	56	58
7	47	-49
8	32	-31
9	4U	4
10	7	7
11	10	9
12	4	-6
13	24	21
14	23	21
15	31	-28
16	23	-26
H= 0, L= 8		
0	27	-28
1	18	16
2	4U	2
3	57	-53
4	4U	5
5	50	47
6	23	25
7	23	-23
8	50	-49
9	7	9
10	17	18
11	19	-17
12	14	14
13	10	9
14	15	-14
H= 0, L= 9		
1	16	-15
2	33	-31
3	22	21
4	23	21
5	15	-16
6	13	13
7	21	21
8	8	-8
9	24	-23
10	3U	-3
11	21	19
12	8	-6
H= 0, L= 10		
0	21	-22
1	28	-25
2	22	22
3	2	7
4	12	-11
5	3U	-6
6	3U	2
7	24	22
8	4U	2
9	22	-21
10	2U	-0
H= 0, L= 11		
1	4	6
2	4	-5
3	10	-11
4	2U	1
5	2U	3
6	1U	2
H= 1, L= 0		
2	70E	111
3	24	32
4	241E	-308
5	27	-32
6	136	133
7	170	161
8	30	30
9	151	-131
10	21	16
11	4U	5
12	95	-82
13	66	54
14	15	17
15	23	-23
16	61	57
17	21	18
18	34	32
19	13	-15
20	2U	1
H= 1, L= 1		
1	179	193
2	49	-64
3	160E	-190
4	72	92
5	2	4
6	65	-90
7	73	87
8	89	96
9	22	20
10	96	-86
11	11	-10
12	85	72
13	72	-64
14	7U	-62
15	62	53
16	55	50
17	27	26
18	46	-40
19	47	-44
20	22	25
H= 1, L= 2		
0	256	270
1	93	-109
2	111	-126
3	145	167
4	28	-35
5	70	-84
6	84	-99
7	44	-56
8	113	149
9	46	58
10	69	-67
11	17	16
12	53	-45
13	61	-55
14	30	23
15	28	24
16	12	13
17	8	7
18	3U	3
19	2U	-6
20	27	-33
H= 1, L= 3		
1	37	41
2	96	105
3	9	-10
4	66	-71
5	26	-34
6	32	-39
7	4U	-1
8	10	-15
9	107	129
10	79	87
11	131	133
12	33	-33
13	85	73
14	18	-17
15	25	-25
16	14	-10
17	48	28
18	43	34
19	15	-17
H= 1, L= 4		
0	21	24
1	235	257
2	35	-35
3	51	-54
4	10	12
5	12	-14
6	4U	-0
7	56	-59
8	4U	-5
9	62	65
10	5U	4
11	13	11
12	4U	-7
13	19	-19
14	10	-9
15	32	-29
16	10	10
17	28	26
18	2U	-2
19	14	17
H= 1, L= 5		
1	4U	-6
2	63	67
3	10	6
4	13	-11
5	24	25
6	37	-39
7	24	24
8	14	-18
9	68	-69
10	84	84
11	81	82
12	44	-44
13	64	-56
14	4U	3
15	21	22
16	11	-10
17	14	-13
18	20	24
H= 1, L= 6		
0	128	-127
1	31	-31
2	78	79
3	73	71
4	11	-13
5	40	-37
6	25	26
7	35	-37
8	52	-53
9	24	26
10	21	22
11	4U	7
12	15	15
13	34	-29
14	3U	-3
15	10	7
16	12	-11
17	5	5
H= 1, L= 7		
1	44	-42
2	5U	5
3	50	50
4	13	12
5	11	-7
6	28	-29
7	23	-20
8	37	35
9	7	-8
10	32	-31
11	4U	6
12	45	39
13	17	16
14	36	-33
15	11	-12
H= 1, L= 8		
0	26	20
1	13	-11
2	36	-31
3	12	14
4	67	58
5	20	-21
6	24	-22
7	35	32
8	11	-14
9	27	-25
10	2	2
11	2	6
12	23	20
13	3U	8
14	4	-6
H= 1, L= 9		
1	4U	-3
2	18	-18
3	12	-13
4	4U	0
5	33	31
6	10	12
7	28	-27
8	10	-9
9	8	5
10	2U	5
11	2U	-1
H= 1, L= 10		
0	5	-8
1	3U	4
2	3U	2
3	27	-23
4	8	8
5	31	28
6	4	-5
7	12	-14
8	2U	2
9	1U	6
H= 1, L= -1		
1	55	-30
2	149	-152
3	64	82
4	47	-62
5	145E	-203
6	132	153
7	162	164
8	86	-78
9	19	-17
10	41	-36
11	30	-23
12	29	-24
13	60	-47
14	83	73
15	78	69
16	58	-47
17	53	-46
18	3U	-4
19	35	32
20	2U	-4
H= 1, L= -2		
0	69	66
1	43	-51
2	19	23
3	182E	-219
4	3U	-2
5	237E	285
6	21	21
7	72	-92
8	35	34
9	32	30
10	5U	-6
11	83	-74
12	11	-11
13	52	42
14	4U	5
15	75	61
16	4U	4
17	81	-69
18	3U	-3
19	15	14
20	2U	-2
H= 1, L= -3		
1	3U	-5
2	127	-131
3	93	-101
4	30	-29
5	118	140
6	122	148
7	177	-146
8	42	50
9	17	19
10	20	-23
11	11	5
12	16	-14
13	39	32
14	82	69
15	71	-57
16	45	-39
17	50	45
18	7	-9
19	34	-34
H= 1, L= -4		
0	37	39
1	50	-51
2	88	88
3	9	-13
4	174	174
5	12	-17
6	75	-80
7	111	123
8	4U	-2
9	112	-122
10	11	-13
11	4U	-4
12	63	57
13	51	47
14	4U	-6
15	23	-19
16	30	-30
17	7	6
18	24	23
19	11	-13
H= 1, L= -5		
1	86	-93
2	43	-46
3	125	125
4	46	48
5	10	-5
6	42	-44
7	48	-55
8	56	62
9	13	-13
10	67	-71
11	4U	8
12	55	52
13	41	39
14	39	-33
15	41	-34
16	37	33
17	23	-19
18	29	-29
H= 1, L= -6		
0	140	-139
1	76	-75
2	74	69
3	75	70
4	4U	11
5	11	-16
6	51	49
7	19	-21
8	86	-84
9	8	17
10	32	34
11	7	2
12	22	22
13	22	-21
14	17	-13
15	18	16
16	9	-10
17	2U	-3
H= 1, L= -7		
1	22	-23
2	49	46
3	19	13
4	31	-27
5	27	25
6	8	-9
7	4U	-0
8	4U	-7
9	61	62
10	41	43
11	80	75
12	17	-19
13	39	-35
14	3U	-2
15	15	12
16	2U	-5
H= 1, L= -8		
0	24	-26
1	108	96
2	4U	3
3	37	-34
4	11	10
5	4U	-0
6	4U	-5
7	26	-24
8	10	-7
9	18	20
10	9	-3
11	3U	2
12	7	7
13	3U	-5
14	2U	1
15	17	-14
H= 1, L= -9		
1	4U	5
2	28	26
3	10	8
4	7	-8
5	4U	-2
6	15	-16
7	9	-11
8	3U	-4
9	28	27
10	36	32
11	33	-30
12	19	-18
13	22	26
H= 1, L= -10		
0	42	33
1	8	-13
2	31	-27
3	30	26
4	15	9
5	14	-11
6	14	-13
7	11	-10
8	13	13
9	12	11
10	8	-7
H= 1, L= -11		
1	11	11
2	2U	4
3	17	-16
4	2U	4
5	2U	2
6	12	-13
7	1U	5
H= 2, L= 0		
1	51	59
2	39	-47
3	172E	-205
4	13	-10
5	134	146
6	91	-92
7	4U	-4
8	180	155
9	39	-31
10	94	-76
11	56	-48
12	56	-44
13	54	47
14	40	36
15	11	11
16	13	-13
17	38	-33
18	7	9
19	9	7
H= 2, L= 1		
1	87	88
2	3U	11
3	77	-88
4	103	-121
5	55	-68
6	134	165
7	89	108
8	72	-73
9	4U	1
10	5U	-5
11	5U	-7
12	15	-13
13	60	-53
14	71	58
15	70	60
16	61	-51
17	3U	-3
18	34	34
19	11	-13
H= 2, L= 2		
0	47	45
1	9	4
2	54	56
3	15	14
4	198	-220
5	14	16
6	126	151
7	17	26
8	18	-21
9	18	21
10	36	37
11	17	-14
12	68	-62
13	5U	-8
14	4U	-11
15	10	12
16	70	61
17	3U	-5
18	31	-32
19	2U	-1
H= 2, L= 3		
1	56	60
2	20	-17
3	23	-24
4	91	93
5	99	-107
6	96	-108
7	83	91
8	50	56
9	5U	-4
10	5U	4
11	20	-18
12	5U	11
13	50	-35
14	42	-38
15	53	48
16	46	43
17	19	-16
18	21	-25
19	1U	1
H= 2, L= 4		
0	44	42
1	13	-13
2	4U	7
3	111	110
4	4U	7
5	128	-127
6	44	-47
7	29	30
8	82	85
9	5U	-8
10	27	-27
11	29	37
12	18	-21
13	35	-30
14	29	23
15	21	-19
16	3U	-6
17	31	31
18	7	8
H= 2, L= 5		
1	18	16
2	81	77
3	58	-56
4	41	-40
5	34	34
6	36	-34
7	51	-51
8	21	22
9	41	41
10	14	16
11	38	-36
12	18	15
13	31	27
14	36	-35
15	26	-24
16	2U	5
17	22	26
H= 2, L= 6		
0	44	43
1	65	65
2	50	-47
3	12	-9
4	55	53
5	12	12
6	5U	-3
7	54	-55
8	4U	-10
9	52	54
10	15	6
11	4U	1
12	20	14
13	18	-18
14	17	-17
15	2U	3
16	2U	3
H= 2, L= 7		
1	24	-23
2	14	15
3	35	31
4	4U	-3
5	11	-7
6	4U	-7
7	4U	-2
8	27	-27
9	15	-16
10	49	48
11	13	14
12	40	-37
13	2U	-0
14	13	14
H= 2, L= 8		
0	77	-71
1	18	18
2	44	40
3	4U	-1
4	4U	-5
5	4U	-0
6	4U	10
7	3U	0
8	25	-21
9	8	-4
10	8	9
11	2U	2
12	2U	5
13	1U	2
H= 2, L= 9		
1	8	-6
2	3U	-1
3	8	9
4	3U	0
5	3U	4
6	3U	-3
7	3U	-3
8	12	12
9	15	-16
10	15	-18
H= 2, L= 10		
0	2U	4
1	23	-24
2	2U	-9
3	10	12
4	10	11
5	7	-5
6	2U	1
H= 2, L= -1		
1	57	63
2	215	-230
3	21	14
4	71E	101
5	44	53
6	71	76
7	72	-69
8	23	18
9	116	99
10	40	-37
11	94	-85
12	5U	-12
13	89	76
14	69	61
15	47	-46
16	9	-8
17	37	35
18	47	-46
19	25	-26
20	19	27
H= 2, L= -2		
0	90	87
1	204E	-235
2	140	-148
3	38	44
4	57	63
5	4U	5
6	4U	-1
7	99	117
8	68	70
9	109	-103
10	11	14
11	11	-8
12	36	32
13	47	39
14	34	-31
15	17	16
16	9	9
17	18	-18
18	3U	1
19	19	-20
20	1U	-7
H= 2, L= -3		
1	54	-56
2	45	-50
3	92	99
4	13	-20
5	21	26
6	39	47
7	27	-30
8	37	43
9	70	-73
10	164	-157
11	102	93
12	113	100
13	11	-15
14	39	-34
15	4U	-2
16	17	18
17	24	-24
18	26	-22
19	22	26
H= 2, L= -4		
0	277	-291
1	4U	-9
2	153	159
3	16	-17
4	14	11
5	29	27
6	31	29
7	20	18
8	57	-64
9	11	-14
10	16	16
11	5U	-7
12	22	21
13	20	17
14	4U	6
15	4U	3
16	46	-39
17	3U	-4
18	2U	6
19	1U	0
H= 2, L= -5		
1	75	-78
2	4U	-1
3	77	78
4	18	17
5	19	20
6	5U	-4
7	35	-38
8	45	-49
9	52	-56
10	87	90
11	68	66
12	83	-77
13	4U	-6
14	47	41
15	8	-10
16	24	-21
17	20	-20
18	8	9
H= 2, L= -6		
0	36	36
1	93	92
2	72	-66
3	41	-40
4	84	82
5	14	16
6	5U	6
7	53	-55
8	49	-47
9	48	50
10	11	15
11	4U	3
12	35	34
13	16	-16
14	26	-23
15	3U	-3
16	2U	-1
17	2U	3
H= 2, L= -7		
1	5U	-5
2	78	76
3	5U	5
4	42	-40
5	32	31
6	22	-24
7	52	-54
8	11	12
9	23	25
10	4U	9
11	25	-25
12	9	11
13	40	37
14	31	-27
15	37	-33
16	2U	4
H= 2, L= -8		
0	5U	3
1	19	-16
2	5U	0
3	65	60
4	19	20
5	53	-53
6	29	-29
7	5U	6
8	36	36
9	4U	8
10	17	-17
11	18	17
12	3U	-2
13	18	-16
14	11	13
15	1U	-7
H= 2, L= -9		
1	4U	7
2	10	6
3	12	-10

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[a] The data are separated into groups having common values of h and l. The three columns in each group list values of k, F_o, and F_c, in that order. Unobserved reflections are indicated by U, and the values of F_o given correspond to the minimum observable intensities. Reflections believed to have been affected by extinction are indicated by E.

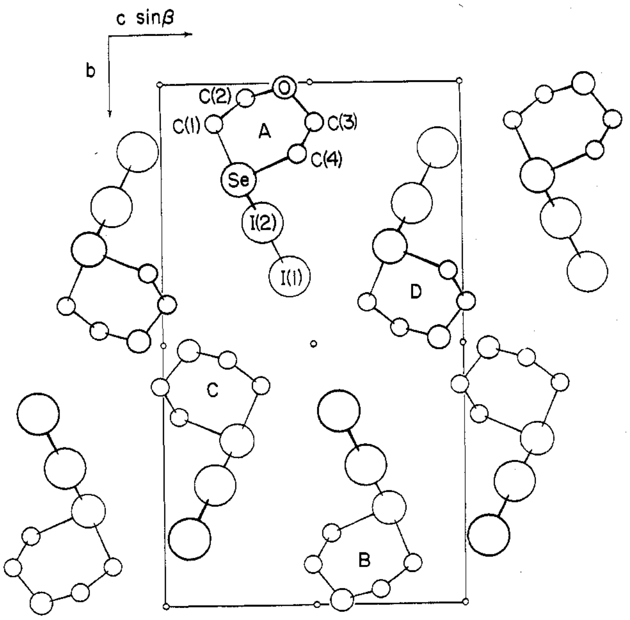

Figure 1.—Projection of the structure of $C_4H_8OSeI_2$ down the *a* axis.

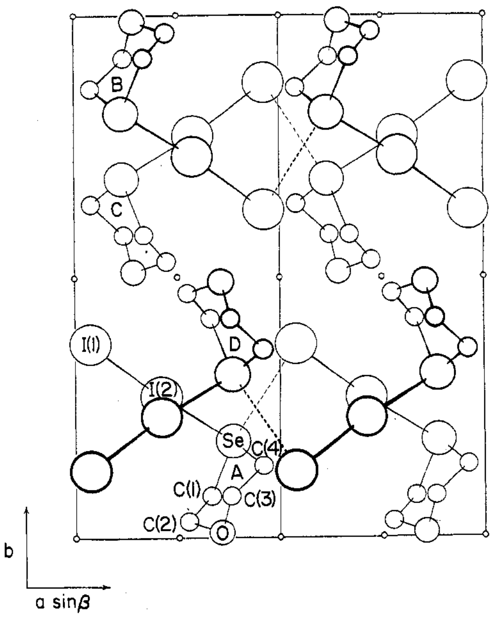

Figure 2.—Projection of the structure of $C_4H_8OSeI_2$ down the *c* axis.

The atomic scattering factors used in the calculations were the neutral atom values given in the "International Tables for X-Ray Crystallography,"[9] corrected for dispersion for Cu Kα radiation.

Discussion of the Structure

Projections of the crystal structure of $C_4H_8OSeI_2$ down the crystallographic *a* and *c* axes are shown in Figures 1 and 2, respectively, and the structurally interesting bond distances, bond angles and intermolecular packing distances are given in Tables IV, V, and VI.

TABLE IV

BOND DISTANCES IN $C_4H_8OSeI_2$ AND THEIR ESTIMATED STANDARD DEVIATIONS

Bond	Distance, A	Esd, A	Bond	Distance, A	Esd, A
I(1)–I(2)	2.956	0.003	O–C(2)	1.42	0.04
Se–I(2)	2.755	0.004	O–C(3)	1.41	0.04
Se···I(1)[a]	3.708	0.004	C(1)–C(2)	1.53	0.04
Se–C(1)	1.93	0.03	C(3)–C(4)	1.52	0.04
Se–C(4)	1.98	0.03			

[a] Intermolecular, between molecules related by the lattice translation, **a**.

TABLE V

BOND ANGLES IN $C_4H_8OSeI_2$ AND THEIR ESTIMATED STANDARD DEVIATIONS

Atoms	Angle, deg	Esd, deg
I(1)–I(2)–Se	174.8	0.3
I(2)–Se–C(1)	107.2	1.0
I(2)–Se–C(4)	101.5	1.0
I(2)–Se–I(1)[a]	82.5	0.4
C(1)–Se–I(1)[a]	169.8	1.0
C(4)–Se–I(1)[a]	80.0	1.0
Se–I(1)–I(2)[a]	77.4	0.3
C(1)–Se–C(4)	94	2
Se–C(1)–C(2)	112	2
Se–C(4)–C(3)	110	2
C(1)–C(2)–O	109	3
C(4)–C(3)–O	110	3
C(2)–O–C(3)	111	3

[a] Intermolecular, between two molecules related by the lattice translation, **a**.

TABLE VI

SHORTEST INTERMOLECULAR PACKING DISTANCES

First atom (position A)[a]	Second atom, position,[a] and lattice translation			Distance, A
Se	I(1)	D	$10\bar{1}$	4.01
I(2)	Se	D	000	4.12
I(1)	C(1)	D	$\bar{1}00$	3.88
I(1)	C(4)	A	$\bar{1}00$	3.89
I(1)	C(1)	C	000	3.91
I(1)	C(3)	D	$00\bar{1}$	3.98
I(1)	C(1)	D	000	4.03
I(2)	C(1)	D	000	4.05
I(2)	C(4)	A	$\bar{1}00$	4.09
C(3)	O	B	$0\bar{1}0$	3.44
O	C(2)	B	$0\bar{1}0$	3.47
C(3)	C(2)	B	$0\bar{1}0$	3.40

[a] Positions: A = (x, y, z), B = $(\bar{x}, \bar{y}, \bar{z})$, C = $(\bar{x}, {}^1/_2 + y, {}^1/_2 - z)$, D = $(x, {}^1/_2 - y, {}^1/_2 + z)$.

Although molecules of the complex are not required crystallographically to have any symmetry above the identity operation, the symmetry **m** is approximated. Deviations from this symmetry are indicated by differences in corresponding pairs of bond distances and angles in Tables IV and V. Figure 3 shows two views of the molecule idealized to the symmetry **m**.

The details of the molecular structure of $C_4H_8OSeI_2$ are similar to those found in the iodine complexes of 1,4-diselenane[10] and tetrahydroselenophene.[1] The 1,4-oxaselenane ring is in the chair form, and the iodine molecule is bonded to selenium in the axial position. On the other hand, the intermolecular arrangement in $C_4H_8OSeI_2$ is different from that found in either of the related iodine complexes previously studied. In the 1,4-diselenane complex, there are weak secondary bonds

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(9) "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, Table 3.3.1 A.

(10) G. Y. Chao and J. D. McCullough, *Acta Cryst.*, **14**, 940 (1961).

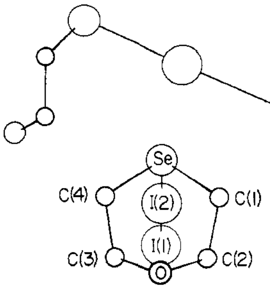
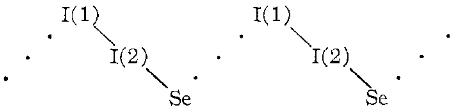


Figure 3.—Two views of the molecular structure of $C_4H_8OSeI_2$ idealized to the symmetry **m**. The upper view is a projection of the molecule parallel to the plane of the carbon atoms and perpendicular to the Se–I–I line. The lower view is a projection of the molecule perpendicular to the plane of the carbon atoms.

between an Se atom of one molecule and I(1) of another at a distance of 3.89 A. As a result, the molecules are linked together in helical chains about alternate 2_1 screw axes. In the tetrahydroselenophene complex, there are much stronger secondary Se–I(1) bonds at 3.64 A in a nearly linear arrangement

···I(1)—I(2)—Se···I(1)—I(2)—Se···

parallel to the *c* axis of the orthorhombic cell. The secondary Se–I(1) bonds in the 1,4-oxaselenane complex are of intermediate strength (3.71 A) and result in the formation of nonlinear chains, thus



with bond angles of 77.4° at I(1) and 82.5° at Se. The general direction of these chains is along the *a* axis of the monoclinic unit as shown in Figure 2.

The observed Se–I bond distance of 2.755 A is the shortest of its kind observed so far. Consistent with this short Se–I distance, the observed I–I distance of 2.956 A is unusually long. In fact, an examination of reported I–I distances in the I_2 *molecule* parts of complexes reveals no other I–I distance this long. In making this comparison, one should not include the longer I···I distances observed between I_2 molecules and I^- ions in polyiodides such as I_3^-, I_5^-, etc. A summary of observed distances in Se–I–I systems is given in Table VII, and a more general summary of heavy-atom bonding systems in related halogen addition compounds of some organoselenides and organotellurides has been given by Hope and McCullough.[1]

TABLE VII

SUMMARY OF OBSERVED Se–I AND I–I DISTANCES IN IODINE COMPLEXES OF SOME SELENIDES

Complex	Se–I distance, A	I–I distance, A
$C_4H_8Se_2 \cdot 2I_2$	2.829	2.870
$C_4H_8Se \cdot I_2$	2.762	2.914
$C_4H_8OSe \cdot I_2$	2.755	2.956

The observed Se–C distances are 1.93 and 1.98 A. Although these differ by almost twice the standard deviation of 0.03 A for the individual values, the difference is not believed to be significant. The average of the two values, 1.955 A, is in good agreement with the value 1.960 A (esd ~ 0.025 A) found in the tetrahydroselenophene complex and with the average of the two values (1.947 and 1.980 A, esd ~ 0.024 A, average 1.964 A) found in the 1,4-diselenane complex. The two C–O distances of 1.42 and 1.41 A differ by one-third their esd value of 0.03 A, and the two C–C distances of 1.53 and 1.52 A (esd ~ 0.04 A) are also in good agreement. The average bond lengths (1.415 A for C–O and 1.525 A for C–C) are within their respective esd's of expected values.

Ideally, the four carbon atoms in the molecule should be coplanar, and this was found to be the case within the experimental errors of the determination. The displacements (in A) of the six ring atoms from a least-squares plane fit to the four carbon atoms are: C(1), 0.004; C(2), −0.005; C(3), 0.005; C(4), −0.004; O, −0.761; Se, 0.942.

Because of the cylindrical approximation made in estimating the absorption corrections, the individual thermal parameters are probably in error by much more than their listed esd values. Nevertheless, the reported values are quite similar in magnitude to those found in the iodine complexes of 1,4-diselenane and tetrahydroselenophene. As in those cases, the thermal parameters for the heavy atoms are all unusually high, and the values for the terminal iodine atom, I(1), are highest of all.

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