because generally we are only trying to find the coordinates of one atom, and they can be determined one at a time. We can concentrate on the spectra which can be measured accurately, and neglect such as are uncertain because a weak intensity is hard to measure against the background. Yet such intensities may correspond to F values which affect a Fourier considerably. Termination of a Fourier series may influence the accuracy of the peak position, but in comparing spectra it is of no importance. A selected set can be measured out to a high order without undue labour, and so a high resolution can be attained, as in the example of an $(0k0)$ series for ox haemoglobin given above.

If the crystals were very closely isomorphous, if the added heavy atom were on one definite site, if there were no error in scaling the measurements of the protein against those of the protein with added heavy atom, if all spectra could be included up to an order where they become negligibly small, and if the random errors were small compared with the difference produced by the addition of the heavy atom, even for the spectra which are faint against the background, any method of calculation would lead to accurate positions for the heavy atom. Actually, however, we are dealing with necessarily imperfect and incomplete data because of all these sources of error. Evidence must be weighed and judgement exercised in arriving at the best estimates of the coordinates. It is of first importance to fix these coordinates as accurately as possible, because they will eventually be needed to determine the phases of the protein structure factors. It is the purpose of this note to draw attention to the possible advantages of a method of representing the observations, which enables one to weigh the evidence of all the most significant and trustworthy data plotted in each case on the one chart.

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The Crystal Structure of Iridium Diselenide

By Luisa Brahde Barricelli
Kjemisk Institutt A, Universitetet i Oslo, Blindern, Norway

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The structure of $IrSe_2$ has been determined, using single-crystal data. The unit cell is orthorhombic with $a = 20{\cdot}94$, $b = 5{\cdot}93$, $c = 3{\cdot}74$ Å. There are eight iridium and sixteen selenium atoms in the unit cell. The space group is *Pnam*. The iridium atom has octahedral selenium environment, the coordination distances are three Ir–Se = 2·44 Å and three Ir–Se = 2·52 Å. Half of the selenium atoms are surrounded by three iridium atoms at three corners of a distorted tetrahedron, and by a selenium atom at a distance Se–Se = 2·57 Å at the fourth corner. The other half of the selenium atoms are coordinated in the same way, but the distance Se–Se = 3·27 Å in this case, which implies that very little bond character is left. An explanation of the divergence in the Se–Se distances and of the difference in the Ir–Se octahedral bond lengths is attempted.

Introduction

The iridium selenides have been studied in this institute by Søvold (1954). He confirmed *inter alia* the existence of the compound $IrSe_2$, which was first prepared by Wöhler, Ewald & Krall (1933).

Søvold succeeded in preparing a few crystals of $IrSe_2$ and used a prismatic one with cross-section $0{\cdot}07 \times 0{\cdot}07$ mm.^{2} and length 0·1 mm. along the shortest axis to take Weissenberg photographs about the three axes, using Cu $K\alpha$ radiation ($\lambda = 1{\cdot}542$ Å). With these photographs he determined the unit-cell dimensions, and from the systematic absences of reflexions the two possible space groups *Pnam* and *Pna*, with *Pnam* as the more probable.

By combining the X-ray data and the pycnometrically determined densities Søvold concluded that the diselenide possibly is a non stoichiometric compound, with a selenium content corresponding to the formula $IrSe_{2-x}$, where $x \approx 0{\cdot}1$. The calculations here will be based on the composition $IrSe_2$.

Moreover, Søvold measured the magnetic susceptibility of iridium diselenide and found a weak and nearly temperature-independent paramagnetism.

Determination of the structure

In the calculation of the Fourier projections it seemed safer to use the space group $Pna2_1$, because only one of the special positions of the space group *Pnam* proved to be possible by consideration of the available space. It turns out, however, that this structure can be described by the special positions of the space group *Pnam*. The orientation and origin of the space group $Pna2_1$ is preferred, for it has the advantage of making it easy to compare the structure of $IrSe_2$ with the structure of marcasite, to which it has some similarity. Therefore, the orientation *Pnam* is chosen with

$$a = 20\cdot94,\ b = 5\cdot93,\ c = 3\cdot74\ \text{Å},\ \alpha = \beta = \gamma = 90°;$$
$$V = 464\cdot4\ \text{Å}^3;\ d = 9\cdot68\ \text{g.cm.}^{-3};\ Z = 8\ (Z_{\text{calc.}} = 7\cdot72);$$
$$a:b:c = 3\cdot531:1:0\cdot631\ .$$

The lattice constants are determined from a powder photograph of $IrSe_2$ taken with Cr $K\alpha$ radiation ($\lambda = 2\cdot291$ Å).

The systematic absences are: $0kl$ reflections when $k+l$ is odd, $h0l$ reflections when h is odd; $hk0$ reflections have no conditions. The special positions are:

$$x, y, 0;\ \bar{x}, \bar{y}, \tfrac{1}{2};\ \tfrac{1}{2}-x, \tfrac{1}{2}+y, \tfrac{1}{2};\ \tfrac{1}{2}+x, \tfrac{1}{2}-y, 0\ .$$

Since there are eight iridium and sixteen selenium atoms in the unit cell, the parameters of two iridium and four selenium atoms are to be determined.

Intensity measurements were carried out visually on Weissenberg photographs, using the multiple-film technique. A total of 146 $hk0$ reflexions were recorded, out of the 157 theoretically accessible, and 50 $h0k$ reflexions out of the 57 theoretically accessible. Corrections for the Lorentz and polarization factors were made. For the calculation of the structure amplitudes the Thomas–Fermi model of the atomic scattering function was used, where the values of f were taken from the *International Tables*. Corrections for the temperature factor $\exp\,[-B \sin^2\theta/\lambda^2]$ were attempted, where the constant B was determined by comparison between the calculated and observed structure factors. The constant was determined to be $B = -0\cdot5$ Å^2. This means that the ratio between the calculated and observed amplitudes for different values of $\sin\theta/\lambda$ is not very far from constant, and that the temperature and absorption factor nearly neutralizes each other. An attempt to correct the observed amplitudes for absorption, and the theoretical atomic scattering function for the temperature factor (calculated as before), gave a much too high positive value of the constant B and not as good a correlation between the observed and calculated F values. Therefore, no further corrections were applied.

The [001] *projection*

A Patterson F^2 projection along the c axis was difficult to interpret. However, with the help of Buerger's (1951) minimum-function method the approximate coordinates of the iridium atoms were determined. A consideration of the available space, combined with the Patterson projection and numerous refinements, led to a preliminary location of the selenium atoms. By a difference synthesis $F_o - F_{c,\text{Ir}}$ a greater accuracy in the determination of the selenium atomic coordinates was achieved. At last, the electron-density map, shown in Fig. 1(*a*), was obtained, where all atoms are well resolved and the iridium atoms are easily distinguished by the greater height of the peaks.

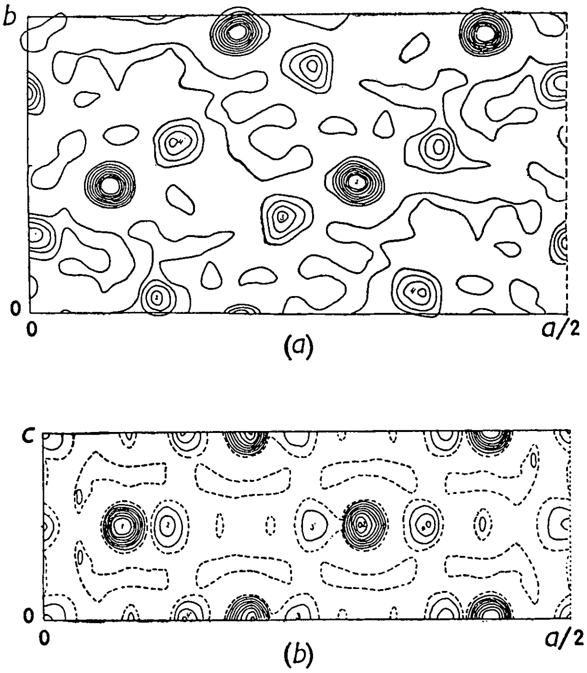

Fig. 1. (*a*) Iridium diselenide [001] Fourier projection. Contours are drawn at arbitrary equidistant levels, starting from 0. (*b*) Iridium diselenide [010] Fourier projection. Contours are drawn at arbitrary equidistant levels, starting from 0 (broken line).

The final x and y coordinates for the iridium atoms were determined from the map. A correction of up to two units in the third figure was made on some of the selenium atomic coordinates determined from this electron-density map. This departure from the coordinates determined from the map seems plausible, because the same coordinates determined from a map where the iridium atoms are subtracted give corrections in the same directions. Moreover, the parameters chosen give a better correlation factor.

The coordinates of the iridium and selenium atoms are given in Table 1.

Table 1. *Atomic coordinates*

	x/a	y/b	z/c
Ir_1	0·076	0·427	0·500
Ir_2	0·304	0·442	0·500
Se_1	0·007	0·270	0·000
Se_2	0·121	0·045	0·500
Se_3	0·237	0·324	0·000
Se_4	0·363	0·074	0·500

A detailed consideration of the errors has not been attempted, but the coordinates of the iridium and

Table 2. *Observed and calculated values of the* ($h\ k\ 0$) *structure factors*

$h\ k\ l$	F_c	F_o
2 0 0	−17·9	16·4
4 0 0	− 9·3	11·2
6 0 0	−24·4	19·4
8 0 0	−15·0	17·2
10 0 0	56·9	52·2
12 0 0	10·9	22·4
14 0 0	65·6	67·2
16 0 0	87·1	87·3
18 0 0	−75·8	79·9
20 0 0	−11·1	14·9
22 0 0	−15·6	15·7
24 0 0	16·7	34·3
26 0 0	77·4	88·1
1 1 0	−36·7	25·4
2 1 0	12·0	10·4
3 1 0	−61·6	40·6
4 1 0	54·6	39·6
5 1 0	87·3	52·2
6 1 0	9·2	10·4
7 1 0	9·7	10·4
8 1 0	−21·8	23·9
9 1 0	34·2	34·3
10 1 0	3·1	11·2
11 1 0	17·8	23·9
12 1 0	−24·7	25·4
13 1 0	−127·8	105·3
14 1 0	54·5	53·0
15 1 0	12·0	14·9
16 1 0	− 5·7	0
17 1 0	3·6	19·4
18 1 0	48·3	57·5
19 1 0	44·0	39·6
20 1 0	12·5	17·9
21 1 0	27·3	29·1
22 1 0	− 7·6	11·9
23 1 0	−36·4	32·8
24 1 0	−10·3	11·9
25 1 0	19·6	20·2
26 1 0	18·5	29·1
0 2 0	85·7	56·7
1 2 0	−69·8	39·6
2 2 0	−26·4	20·9
3 2 0	13·7	14·2
4 2 0	−79·3	58·2
5 2 0	−76·4	57·5
6 2 0	−22·1	23·1
7 2 0	−20·4	23·9
8 2 0	−69·5	61·9
9 2 0	88·3	70·2
10 2 0	28·1	27·6
11 2 0	24·2	26·1
12 2 0	−43·8	44·0
13 2 0	−17·2	29·1
14 2 0	20·6	28·4
15 2 0	−19·7	23·9
16 2 0	31·5	42·5
17 2 0	−50·2	55·2
18 2 0	−65·2	72·4
19 2 0	14·2	14·9
20 2 0	−19·5	23·1
21 2 0	− 9·9	11·9
22 2 0	−31·0	27·6
23 2 0	27·9	29·9
24 2 0	6·6	5·2
25 2 0	40·8	41·0
1 3 0	17·9	20·2
2 3 0	6·4	0
3 3 0	−28·0	26·9
4 3 0	109·4	79·1
5 3 0	40·6	41·0
6 3 0	−33·4	39·6
7 3 0	− 6·5	0
8 3 0	−32·7	42·5
9 3 0	37·8	44·0
10 3 0	−49·5	51·5
11 3 0	− 2·5	0
12 3 0	−48·0	51·5
13 3 0	−20·5	27·6
14 3 0	43·5	38·1
15 3 0	4·5	0
16 3 0	−14·5	19·4
17 3 0	24·5	33·6
18 3 0	31·4	38·1
19 3 0	− 8·1	14·2
20 3 0	24·8	38·8
21 3 0	26·6	32·1
22 3 0	−90·4	88·1
23 3 0	−27·7	36·6
24 3 0	−29·0	30·6
0 4 0	7·0	15·7
1 4 0	−25·9	24·6
2 4 0	11·9	17·2
3 4 0	−16·1	20·2
4 4 0	14·9	20·9
5 4 0	−36·3	42·5
6 4 0	32·8	35·1
7 4 0	−57·5	56·7
8 4 0	19·5	27·6
9 4 0	133·2	121·0
10 4 0	27·6	32·8
11 4 0	6·7	0
12 4 0	− 6·9	9·0
13 4 0	6·4	7·5
14 4 0	− 3·4	0
15 4 0	−24·9	26·1
16 4 0	15·0	18·7
17 4 0	−48·1	54·5
18 4 0	19·3	20·9
19 4 0	34·9	42·5
20 4 0	21·1	17·9
1 5 0	22·1	23·1
2 5 0	4·0	7·5
3 5 0	− 5·1	9·0
4 5 0	88·1	88·8
5 5 0	−55·3	61·2
6 5 0	−37·5	38·8
7 5 0	−11·4	14·9
8 5 0	− 1·7	10·4
9 5 0	−26·4	38·8
10 5 0	−17·4	17·9
11 5 0	− 3·5	0
12 5 0	−20·1	27·6
13 5 0	19·3	26·9
14 5 0	40·3	46·3
15 5 0	21·6	24·6
16 5 0	2·3	0
17 5 0	−21·3	29·1
18 5 0	36·8	44·8
19 5 0	−25·7	22·4
20 5 0	48·2	43·3
0 6 0	−77·0	73·1
1 6 0	−30·7	37·3
2 6 0	−27·7	34·3
3 6 0	20·7	31·3
4 6 0	26·5	35·1
5 6 0	−28·4	41·0
6 6 0	− 3·9	11·9
7 6 0	−33·8	39·6
8 6 0	38·1	38·8
9 6 0	52·1	53·0
10 6 0	−35·0	36·6
11 6 0	− 2·8	0
12 6 0	− 8·7	11·9
13 6 0	− 8·5	20·2
14 6 0	−56·6	56·7
15 6 0	−14·9	16·4
16 6 0	−32·3	28·4
1 7 0	40·3	48·5
2 7 0	0·4	0
3 7 0	35·7	47·8
4 7 0	13·0	20·2
5 7 0	−50·9	55·2
6 7 0	−29·8	33·6
7 7 0	6·1	9·0
8 7 0	− 2·7	9·0
9 7 0	−12·3	11·9
10 7 0	27·3	26·1

selenium atoms are probably reliable to ±0·01 Å and ±0·03 Å, respectively (those of Se_4 are less reliable).

The correlation between calculated and observed amplitudes is given by $R = \Sigma||F_c|-|KF_o||\div\Sigma|KF_o|$, where the F_c corresponding to observed values too weak to be detected are taken in the summation with their half value. The constant K, by which the observed amplitudes are multiplied, is determined by the method of least squares by the formula $K = \Sigma F_oF_c/\Sigma F_o^2$. The result obtained was $R = 0{\cdot}18$.

In Table 2 the structure factors calculated from the x and y coordinates are listed, together with the observed structure amplitudes.

The [010] *projection*

A Patterson projection along the b axis indicated that the parameters along the c axis for all atoms were either 0 or $\frac{1}{2}$ in accordance to the special positions of the space group *Pnam*. The x and z coordinates having been fixed (see Table 1), the phases of the observed structure factors were determined and the electron-density map shown in Fig. 1(b) was obtained.

The structure factors calculated from the x and z coordinates are compared with the observed amplitudes in Table 3. The correlation factor obtained, following the same procedure as before, is $R = 0{\cdot}24$. The less good correlation between the calculated and observed amplitudes is probably to some extent due to absorption effects, which are larger for this projection. A few of the highest intensities observed are also determined with less accuracy.

From Fig. 1(b) one can see that in this projection there are quite strong peaks caused by the termination-of-series error. A rough calculation of the radius for these effects shows that the false peak below atom Ir_1 in Fig. 1(b) is built up of six such termination-of-series effects from the surrounding atoms. Otherwise, all atoms are well resolved and the iridium atoms are easily distinguished. The peak corresponding to the Se_3 atom is weaker than the other Se atom peaks. Assuming that the compound is non stoichiometric, as Søvold did, this might possibly indicate the site of a selenium vacancy in the lattice. Therefore, a calculation of the structure factors was tried, omitting one selenium atom from these positions. A better correla-

Table 3. *Observed and calculated values of the* (*h* 0 *l*) *structure factors*

h k l	F_c	F_o	*h k l*	F_c	F_o	*h k l*	F_c	F_o	*h k l*	F_c	F_o
2 0 0	−17·9	3·5	6 0 1	60·6	43·7	6 0 2	−20·4	20·7	10 0 3	48·2	48·2
4 0 0	9·3	10·3	8 0 1	25·0	29·3	8 0 2	−17·6	19·6	12 0 3	35·1	40·6
6 0 0	−24·4	20·5	10 0 1	63·1	45·5	10 0 2	48·2	43·7	14 0 3	−27·2	28·3
8 0 0	−15·0	20·0	12 0 1	42·3	43·7	12 0 2	9·9	17·5	16 0 3	5·6	10·4
10 0 0	56·9	55·7	14 0 1	−33·3	33·7	14 0 2	56·9	54·9	18 0 3	−21·8	29·8
12 0 0	10·9	26·6	16 0 1	6·8	14·1	16 0 2	77·4	81·6	20 0 3	37·5	62·6
14 0 0	65·6	75·8	18 0 1	−23·7	34·2	18 0 2	−65·4	83·1			
16 0 0	87·1	81·7	20 0 1	−43·2	59·0	20 0 2	−10·2	16·8	0 0 4	143·2	126·8
18 0 0	−75·8	93·1	22 0 1	114·6	133·2	22 0 2	−14·9	21·2	2 0 4	− 9·6	14·5
20 0 0	−11·1	21·3	24 0 1	18·8	30·0	24 0 2	15·6	9·1	4 0 4	− 5·2	10·7
22 0 0	−15·6	23·6	26 0 1	26·7	30·5				6 0 4	−15·0	16·5
24 0 0	16·7	40·6				2 0 3	− 4·6	0	8 0 4	−14·7	19·7
26 0 0	77·4	91·8	0 0 2	206·8	131·8	4 0 3	−100·3	85·5	10 0 4	36·9	35·3
			2 0 2	−13·6	18·4	6 0 3	43·2	47·9	12 0 4	7·6	15·6
2 0 1	− 5·9	0	4 0 2	− 7·2	11·7	8 0 3	18·3	29·8	14 0 4	45·6	41·8
4 0 1	−141·5	80·8									

tion with the observed amplitudes was obtained in this projection, but in the *xy* projection the correlation was not so good. Since no peculiarities suggesting an ordered distribution of the selenium vacancies could be detected on the Weissenberg photographs, this question was not studied further.

Description of the structure

The structure determined is shown in Fig. 2. In this structure each iridium atom is surrounded octahedrally by six selenium atoms. The interatomic distances are listed in Table 4. From the table it is seen that three Ir–Se distances are shorter than the other three, 2·44 Å against 2·52 Å. The three closer selenium atoms are all situated on one side of the iridium atom. The deviation from the regular octahedral angles is generally within ±7°, but in one case reaches −11°. Each selenium atom is surrounded tetrahedrally by three iridium atoms at the same distances. In the fourth tetrahedral direction one half of the selenium atoms have a selenium atom at a distance of 2·57 Å. The other half have the nearest selenium atom at the much larger Se–Se distance of 3·27 Å. The latter distance indicates that very little bond character is left. The largest deviation from the regular tetrahedral angle is +15°.

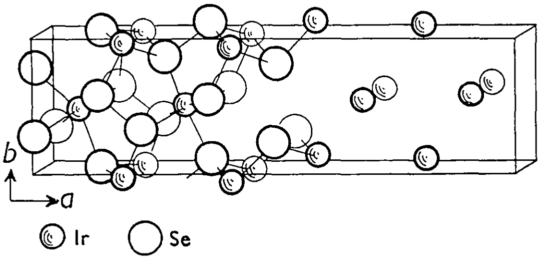

Fig. 2. The unit cell of iridium diselenide. In the right half of the cell the selenium atoms are not drawn.

Table 4. *Interatomic distances*

By the Ir–2 Se distances is meant that the two selenium atoms lie $c = 3{\cdot}74$ Å over each other.

The sign $^-$ means that the *y* coordinate of the atom is negative. Index ^{1} means that the atom is moved in accordance with the space group *Pnam*.

Ir–Se bond distances

$\mathrm{Ir_1–2\,Se_1} = 2{\cdot}54$ Å
$\mathrm{Ir_1–2\,Se_4^1} = 2{\cdot}43$ Å
$\mathrm{Ir_1–Se_1^1} = 2{\cdot}50$ Å
$\mathrm{Ir_1–Se_2} = 2{\cdot}45$ Å

$\mathrm{Ir_2–2\,Se_2^1} = 2{\cdot}52$ Å
$\mathrm{Ir_2–2\,Se_3} = 2{\cdot}44$ Å
$\mathrm{Ir_2–Se_4} = 2{\cdot}51$ Å
$\mathrm{Ir_2–Se_3^1} = 2{\cdot}42$ Å

$m \pm s = 2{\cdot}520 \pm 0{\cdot}017$ Å
$m \pm s = 2{\cdot}435 \pm 0{\cdot}015$ Å

Se–Se distance in tetrahedral bond direction

$\mathrm{Se_3–Se_4} = 2{\cdot}57$ Å
$\mathrm{Se_2–Se_1^{1-}} = 3{\cdot}27$ Å

The octahedral and tetrahedral environment of the iridium and selenium atoms, respectively, leads one to compare the structure of iridium diselenide with that of compounds of the marcasite or pyrite type.

The following divergences are to be pointed out:

The axial ratio of iridium diselenide ($a{:}b{:}c = 3{\cdot}531{:}1{:}0{\cdot}631$) shows that a/b is nearly four times greater than for marcasite-type structures (e.g. in $\mathrm{FeSe_2}$ $a{:}b{:}c = 0{\cdot}838{:}1{:}0{\cdot}626$ (Tengnér, 1938)). In iridium diselenide only half of the selenium atoms are bonded to one other selenium atom and there are three longer and three shorter Ir–Se octahedral bond distances. In the marcasite-type structure all metalloid atoms are bonded in pairs and there are usually two shorter and four longer M–X octahedral bond distances.

Discussion

The interesting feature of iridium diselenide that one half of the selenium atoms are bonded in pairs, and the other half not, leads to the conclusion that the iridium atom is in valence state 3. Assuming that the iridium and selenium atoms form covalent d^2sp^3 and sp^3 bonds respectively, this means that three of the twelve electrons needed for six octahedral bonds are delivered by the iridium atom, four are delivered by three selenium atoms which have a free electron pair in the fourth bond direction, and five by three of the selenium atoms bonded in pairs.

This gives for each Ir–Se bond:

$$\tfrac{3}{6}+\tfrac{1}{2}(\tfrac{4}{3}+\tfrac{5}{3}) = 2 \text{ electrons} .$$

According to Pauling & Huggins (1934), the octahedral radius for Ir(III) is 1·32 Å and the tetrahedral radius for Se is 1·14 Å, giving Ir–Se and Se–Se distances of 2·46 Å and 2·32 Å, respectively. In the structure of iridium diselenide three Ir–Se distances are 0·06 Å longer and three 0·02 Å shorter than the calculated one, whereas the Se–Se bond distance is 0·25 Å longer (Table 4). The close agreement between the Ir–Se distances and those for octahedral and tetrahedral bonds makes it reasonable to assume that mainly $5d^26s6p^3$ and $4s4p^3$ orbitals are used in the bond formation. The great divergence from the tetrahedral angle and the large Se–Se bond distance indicate, however, that these bonds are hardly purely tetrahedral. The Se–Se and the three longer Ir–Se bond distances observed agree better with the metallic radii given by Pauling (1947). With coordination number six for iridium and four for selenium, the radius of both atoms are 1·26 Å and all metallic bond distances 2·52 Å. This is in perfect agreement with the three observed Ir–Se distances of 2·52 Å and is within the possible error of determination for the observed Se–Se bond distance of 2·57 Å.

This good agreement between the observed and the metallic atomic distances, together with the fact that the compound has a small and almost temperature-independent paramagnetic susceptibility and metallic lustre, can be taken as an indication that the Se–Se and three of the Ir–Se bonds have metallic character.

The fact that the three other Ir–Se distances are shorter can be explained by supposing that the iridium atom, with its three electrons in the $5d^26s6p^3$ orbitals participating in bond formation, forms three normal and three coordinated covalent bonds and that only the three so-called coordinated bonds are metallic. This would mean that in the structure of iridium diselenide a small difference in energy between normal covalent and coordinated bonds is retained, owing to the metallic character of the components. The metallic character of the bonds can be due to electrons of the selenium atoms being exited to the conduction band, or to the possibly non stoichiometric composition of $IrSe_2$, mentioned in the introduction, which might give a sufficient deficiency of electrons to make some of the $5d^26s6p^3$ and $4s4p^3$ orbitals metallic (Pauling, 1949).

The structure of iridium diselenide would then have layers with metallic bonds alternating with layers with normal covalent bonds perpendicular to the *a* axis. In this connexion it would be of great interest to find out whether iridium diselenide has an enhanced electrical conductivity in the *bc* plane in order to test this hypothesis. There are, however, practical difficulties in carrying out such measurements because of the extremely small size of the crystals obtained.

A parallelism between the number of electrons contributed by the metal atom to the bonds and the octahedral M–X bond distances seems to exist also in structures of the marcasite type. With two shorter and four longer octahedral bond distances, one would expect the metal atom to contribute with two electrons to the bonds and form two normal and four coordinated covalent bonds. This agrees completely with the valence state assumed for the metal in the sulphides, selenides and tellurides with marcasite-like structures.

In corresponding compounds where the metalloids are phosphorus, arsenic and antimony, a deficiency of electrons is present in the d^2sp^3 and sp^3 orbitals supposedly used in bond formation. But this is no obstacle to the assumption that some of the bonds in these structures have more metallic character.

The author intends to continue the study of this problem.

The author is indebted to Prof. Haakon Haraldsen for suggesting the determination of this structure and for his interest in this study.

Thanks are also due to the American Association of University Women for having given her possibilities to specialize in the field of X-ray diffraction.

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