

X-RAY CRYSTAL ANALYSIS OF THIRTEEN  
COMMON METALS.<sup>1</sup>

BY ALBERT W. HULL.

## SYNOPSIS.

*Crystal Structure of Thirteen Metals Determined by X-ray Analysis.*—The author's method of powder analysis was used. In the case of anisometric substances, the crystal system and axial ratio were found by means of plots by the method described in the preceding paper.<sup>2</sup> The lattices of *chromium*, *molybdenum* and *tantalum* were found to be body centered cubes with sides 2.895, 3.143 and 3.272 Å. respectively; *cobalt alpha*, *nickel*, *rhodium*, *palladium*, *iridium* and *platinum* have face centered cubic lattices with the sides of the cubes 3.554, 3.540, 3.820, 3.950, 3.805 and 3.930 Å. respectively; *cobalt beta*, *zinc*, *cadmium* and *ruthenium* have hexagonal lattices of the "close-packed" type with axial ratios 1.63, 1.86, 1.89 and 1.59 respectively and with the triangular sides 2.514, 2.670, 2.960 and 2.686 Å. respectively; and *indium* has a face-centered tetragonal lattice with axial ratio 1.06 and the side of elementary prism 4.58 Å. The structures found for cadmium, zinc and indium are close packed arrangements of solid prolate spheroids while that for ruthenium is a close packed arrangement of oblate spheroids.

## INTRODUCTION.

THE extension of the Bragg method of crystal analysis to powdered materials has been described elsewhere<sup>3</sup> and need be only briefly summarized here. The photographs were taken with the same apparatus and in the same manner as those previously described, and the interpretations were made in the same manner, except that the process of guessing the correct lattice was greatly simplified by the use of logarithmic plots of crystal spacings against axial ratio. These plots are fully described in the preceding paper.<sup>4</sup>

For convenience of reference, the arrangement of apparatus is reproduced in Fig. 1. Intense characteristic X-rays of wave-length 0.712 Å. were produced by a water-cooled molybdenum Coolidge tube, running at 30,000 volts constant potential and 30 milliamperes. A filter, consisting of a thin sheet of cellulose binding material containing .05 gram

<sup>1</sup> The results of these analysis have been briefly announced elsewhere: Co, Ni, Cr, Mo, and Pt in Proc. A. I. E. E., 38, 1189, 1919; the rest in Science, 52, 227, Sept. 3, 1920.

<sup>2</sup> Graphical Determination of Hexagonal and Tetragonal Crystal Structures from X-Ray Data, by Albert W. Hull and Wheeler P. Davey. This number, pp. 549-570.

<sup>3</sup> PHYS. REV., 10, 661, 1917.

<sup>4</sup> Loc. cit.

of  $ZrO_2$  per  $cm.^2$ , absorbed almost completely all wave-lengths except the K-alpha doublet, giving a monochromatic source. The smooth face of the X-ray target made an angle of  $10^\circ$  with the line joining it to the two defining slits. These were each 2 mm. wide and 1 cm. high, and 9 and 13 cm. respectively from the focal spot. This gave an intense beam of approximately  $1^\circ$  angular width, with a halo, due to rays pro-

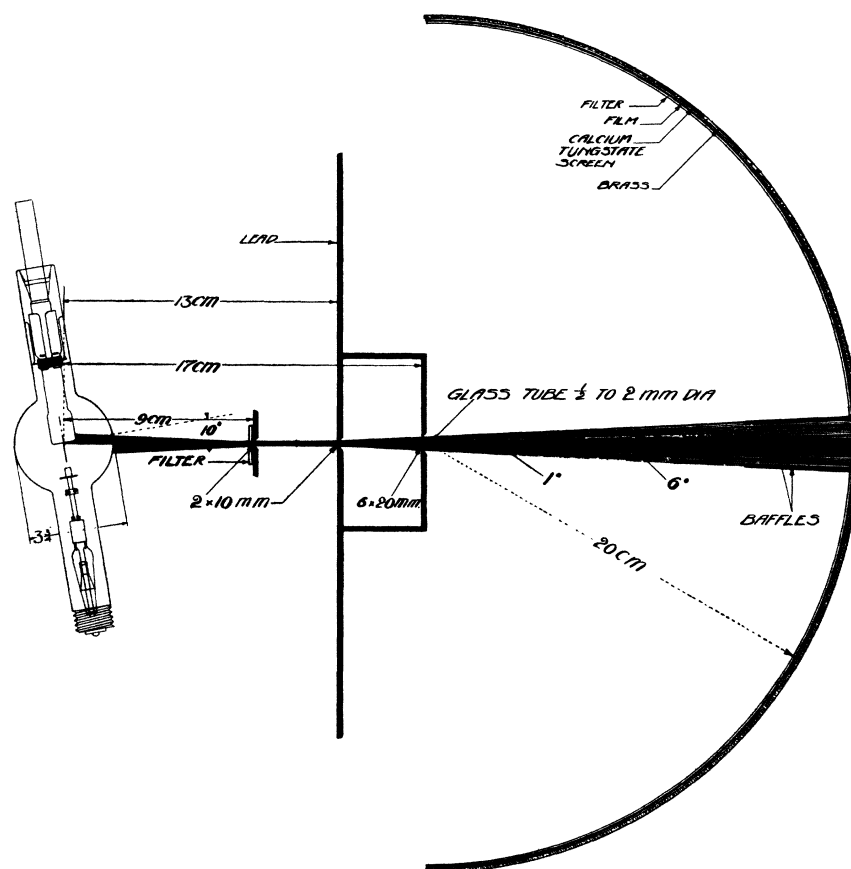


Fig. 1.

ceeding from the face and stem of the target, and secondary rays from other parts of the tube and lead box, of about  $6^\circ$ . A third slit, 6 mm. wide and 2 cm. high, was placed 4 cm. beyond the second defining slit; *i.e.*, 17 cm. from the focal spot. This slit was wide enough so that no primary rays which were able to get through the first two slits could strike it, and its function was to prevent secondary rays from the second slit from reaching the photographic film, except for an angular width of about  $8^\circ$  in the center.



The powdered material to be examined was mounted in a thin-walled glass tube, of diameter  $\frac{1}{2}$  to 2 mm., according to density, directly in front of the third slit, and at the center of a semicircular brass frame of 20 cm. radius. Upon this frame was mounted a strip of standard Eastman portrait film 4 cm. wide and 60 cm. long, backed by a calcium tungstate intensifying screen. A second filter<sup>1</sup> sometimes of .0025 cm. iron, but generally and preferably a thin strip of cellulose containing .025 g. of  $ZrO_2$  per cm.<sup>2</sup>, was placed directly in front of the film to absorb fluorescent secondary rays from the powder. These rays, though they produce no lines, are often 100 times as intense as the scattered rays which produce the line patterns, and their fog completely obscures the lines. The exposure was usually over night, about 15 hours.

A typical film is shown in Fig. 2.

The films were measured on a standard Keuffel and Esser log log slide rule, with movable scale removed. The film was fastened with paper clips to the rule, with its center line at the zero of the scale of equal parts. The glass slider was then moved over the film, its index line placed successively over the centers of the lines on the film, viewed as a transparency, and the distances in metric inches read off on the scale of equal parts at the bottom. The lines on both sides of the center were read independently, so that any scratch or false line, as well as mistakes in measurement, could be detected by comparison, and the accuracy of reading could be estimated. The readings on the two sides always agreed within about  $\frac{1}{4}$  of 1 per cent.

These distances of the lines from the center of the film were reduced to crystal degrees by dividing by 2 (radius of film  $\times 2\pi/360$ ), or 0.2777, and then to planar spacings in Ångstroms by taking the quotient  $\lambda/(2 \sin \theta)$  according to Bragg's law.

In the following tables only the planar spacings, with the estimated intensities of the lines from which they are calculated, will be given. The scale of intensities is chosen so that intensity 1 represents the weakest line that can be identified with certainty. Lines with intensity  $\frac{1}{2}$  or even  $\frac{1}{4}$  are considered reliable if found in identical positions on both sides of the film.

For comparison with the observed spacings, the calculated spacings are given; *i.e.*, those of a theoretical lattice of the type assumed, as calculated by the equations published in an earlier paper.<sup>2</sup>

The calculation of the theoretical intensities of the lines requires a knowledge of the effect of temperature, absorption, size of crystals,

<sup>1</sup> The first filter was between the X-ray tube and first slit.

<sup>2</sup> PHYS. REV., 10, 671, 677, 1917.

shape of atoms, and variation of ideal reflecting power with angle, in addition to the number of cooperating planes. The effect of all these factors except the last, is to produce a nearly uniform decrease of intensity with increasing angle. The large fluctuations in intensity of consecutive lines are due to the variation in the number of different sets of planes which cooperate to produce the respective lines. Hence a comparison of the number of cooperating planes with the observed intensities is a valuable check on the correctness of the identification of the lines. These numbers are given in the second column.

As a final check the densities calculated from the X-ray data, by dividing the mass of the atoms in an elementary cell by the volume of the cell, are compared with the best values obtained by standard methods, as given in Van Nostrand's Chemical Annual for 1918.

TABLE I.

*Chromium.*

A sample of very fine chromium powder, from the Metal and Thermit Company, gave the following spacings:

Intensity of Line.	Number of Coöperating Planes.	Planar Spacings.		Indices of Form.
		Observed.	Calculated.	
100	6	2.048	2.047	100
20	3	1.448	1.448	100 (2)
50	12	1.183	1.183	211
10	6	1.025	1.024	110 (2)
30	12	.915	.915	310
5	4	.837	.836	111 (2)
30	24	.774	.774	321
$\frac{1}{2}$	3	.723	.724	100 (4)
20	18	.682	.683	{ 411 110 (3)
5	12	.648	.647	210
2	12	.617	.617	332
1	12	.591	.591	211 (2)
15	36	.567	.567	{ 431 510

Type of Lattice.....	Body-centered Cube.
Side of elementary cube.....	2.895 Å.
Distance between nearest atoms.....	2.508 Å.
Density { from X-ray measurements.....	7.07
{ from standard methods.....	6.92

The calculated spacings are those of a *body centered cubic lattice*, the side of the cube being 2.895 Å., and the distance between nearest atoms 2.508 Å.

Since there are two atoms per elementary cube, the ideal density is

$$\frac{\text{mass}}{\text{volume}} = \frac{2M}{d_0^3} = \frac{2 \times 52.0 \times 1.650}{(2.895)^3} = 7.07;$$

$M$  = mass of 1 atom in grams;  $d_0$  = side of elementary cube in cm.

The density given by Van Nostrand is 6.92.

TABLE II.

Molybdenum.

Very fine powder, by reduction of oxide in H<sub>2</sub>. Excellent photograph.

Intensity of Line.	Number of Cooperating Planes	Spacing of Planes.		Indices of Forms.
		Observed.	Calculated.	
100	6	2.215	2.221	110
50	3	1.569	1.571	100 (2)
100	12	1.283	1.284	211
35	6	1.109	1.111	110 (2)
60	12	.993	.993	310
10	4	.907	.907	111 (2)
70	24	.839	.839	321
5	3	.784	.785	100 (4)
30	18	.739	.740	{ 110 (3) 411
20	12	.702	.702	210 (2)
20	12	.669	.670	332
20	12	.641	.641	211 (2)
35	36	.616	.616	{ 431 510
25	24	.574	.574	521
5	6	.554	.555	110 (4)
25	24	.538	.538	{ 530 433
20	15	.523	.525	{ 100 (3) 221

Type of Lattice.....	Body-centered Cube.
Side of elementary cube.....	3.143 Å.
Distance between nearest atoms.....	2.720 Å.
Density {	
from X-ray data.....	10.16
by standard methods.....	10.28

These spacings are identical, within experimental error, with those of tungsten. In order to detect the difference, if any, a special photograph was taken, giving tungsten and molybdenum on the same film, one above the other. This was accomplished by filling the lower half of a small glass tube with Mo and the upper half with W, and placing it symmetrically in the X-ray beam so that both halves received equal radiation. A brass septum prevented overlapping of the scattered radiation from the two samples.

This photograph showed the first 6 lines of W exactly above those of Mo. The seventh W line was noticeably displaced from that of Mo, and the fifteenth lines were displaced by the difference between the components of the clearly resolved  $\alpha$  doublet (cf. Fig. 1), about 2 mm. The Mo lines were the farther from the center, on both sides, thus eliminating any error due to imperfect alignment of the samples.

This gives tungsten a spacing  $\frac{1}{4}$  per cent. *greater* than that of Mo, viz., 3.150.

TABLE III.

*Tantalum.*

Filaments, taken from lamp, crushed. Very faint photograph.

Intensity of Line.	Number of Coöperating Planes.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
10	6	2.315	2.315	110
3	3	1.636	1.636	100 (2)
15	12	1.335	1.335	211
4	6	1.157	1.158	110 (2)
3	12	1.033	1.033	310
2	4	.946	.944	111 (2)
8	24	.872	.874	321
$\frac{1}{2}$	3	.818	.819	100 (4)
3	18	.773	.773	{ 110 (3) 411
$\frac{1}{2}$	12	.732	.732	210 (2)
$\frac{1}{2}$	12	.697	.697	332
$\frac{1}{2}$	12	.667	.667	211 (2)
1	36	.643	.642	{ 431 510

Type of Lattice.....	Body-centered Cube.
Side of elementary cube.....	3.272 Å.
Distance between nearest atoms.....	2.833 Å.
Density { from X-ray data.....	17.09
{ by standard methods.....	14.49 <sup>1</sup>

## COBALT.

The powder photographs of cobalt show two crystalline forms, one face-centered cubic and the other hexagonal close packed, both stable at room temperature.

The relation of these two forms to each other, and especially to heat treatment and mechanical working, has not yet been studied.

Tests were made upon samples prepared in three different ways.

1. Two carefully analyzed samples of very pure cobalt were kindly

<sup>1</sup> This is the value given by Van Nostrand. The most recent determination cited by Landolt & Börnstein (v. Bolton, Zs. Electroch., 11, 45, '05) gives 16.64, in better agreement with the X-ray data.

furnished for this investigation by Dr. H. T. Kalmus. Filings from these samples gave identical patterns of lines, with the spacings given in Table IV. They show a *perfect hexagonal close-packed lattice* with no

TABLE IV.

Cobalt (1).<sup>1</sup>

Filings from cast cobalt (Kalmus), 99.7 per cent. pure.

Number of Cooperating Planes, <i>N</i> .	Phase Factor, <i>P</i> .	<i>NF</i> .	Intensity of Line (Estimated).	Spacing of Planes.		Indices of Form.
				Observed.	Calculated.	
3	$\frac{1}{4}$	.75	30	2.182	2.176	10 $\bar{1}$ 0
1	1	1.	25	2.055	2.051	0001 (2)
6	$\frac{3}{4}$	4.5	50	1.920	1.920	10 $\bar{1}$ 1
6	$\frac{1}{4}$	1.5	2	1.490	1.493	10 $\bar{1}$ 2
3	1	3.	20	1.250	1.257	11 $\bar{2}$ 0
6	$\frac{3}{4}$	4.5	5	1.145	1.158	10 $\bar{1}$ 3
6	$\frac{1}{4}$	1.5	...	...	1.089	10 $\bar{1}$ 0 (2)
6	1	6.	15	1.064	1.072	11 $\bar{2}$ 2
6	$\frac{3}{4}$	4.5	5	1.045	1.051	20 $\bar{2}$ 1
1	1	1.	$\frac{1}{2}$	1.013	1.026	0001 (4)
6	$\frac{1}{4}$	1.5	$\frac{1}{2}$	.953	.961	10 $\bar{1}$ 1 (2)
6	$\frac{1}{4}$	1.5	...	...	.928	10 $\bar{1}$ 4
6	$\frac{3}{4}$	4.5	2	.839	.853	20 $\bar{2}$ 3
6	$\frac{1}{4}$	1.5	...	...	.823	21 $\bar{3}$ 0
12	$\frac{3}{4}$	9.	2	.797	.807	21 $\bar{3}$ 1
6	1	6.	2	.788	.794	11 $\bar{2}$ 4
6	$\frac{3}{4}$	4.5	1	.752	.768	10 $\bar{1}$ 5
12	$\frac{1}{4}$	3. } (wide)			.765	21 $\bar{3}$ 2
6	$\frac{1}{4}$	1.5	$\frac{1}{4}$	.738	.747	10 $\bar{1}$ 2 (2)
3	1	3.	$\frac{1}{4}$	.717	.727	10 $\bar{1}$ 0 (3)

Type of Lattice.....	Hexagonal Close-packed.
Axial ratio.....	1.633
Side of elementary triangular prism.....	2.514 Å.
Distance between nearest atoms.....	2.514 Å.
Density { from X-ray data.....	8.66
{ by standard methods.....	8.718

trace of a cubic form. The same filings, after annealing in H<sub>2</sub> at 600° for 6 hours, showed a mixture of cubic and hexagonal close packing in about equal proportions. It was not found possible by annealing at this temperature to cause the crystallization to go over entirely from hexagonal to cubic form.

<sup>1</sup> This film was taken in a cassette of only 10 cm. radius, with the Co filings spread on a thin film nearly 3 mm. wide, and there is a systematic error in the readings, amounting to about 1 per cent. for the largest spacings. The accuracy is sufficient for the purpose, which is to show the perfect hexagonal character of the lattice. The correctness of the value chosen for the fundamental spacing was checked by other films.



2. A sample of fine powder, prepared by reduction of cobalt oxide in  $H_2$  at about  $600^\circ$ , gave the spacings shown in Table V. These correspond to a *perfect cubic close-packed lattice*. A very faint trace of the hexagonal form is present, as shown by the first and third lines, 2.162 Å. and 1.922 Å. respectively.

TABLE V.

Cobalt (2).

Fine cobalt powder, from oxide reduced in  $H_2$  at  $600^\circ$ . Lines broken and irregular, due to large crystals, making the estimation of intensities difficult, but not interfering with the measurement of the spacings.

Intensity of Line.	Number of Coöperating Planes.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
1	..	2.162	....	...
12	4	2.035	2.051	111
$\frac{1}{4}$	..	1.922	....	...
1	3	1.775	1.777	100 (2)
10	6	1.257	1.257	110 (2)
12	12	1.071	1.072	311
1	4	1.024	1.025	111 (2)
$\frac{1}{4}$	3	.888	.888	100 (4)
$\frac{1}{2}$	12	.814	.815	311
5	12	.795	.794	210 (2)
1	12	.726	.725	211 (2)
2	16	.686	.684	{ 511 111 (3)
1	6	.630	.628	110 (4)
1	24	.603	.601	531
1	15	.595	.594	{ 100 (6) 221 (2)

Type of Lattice.....	Face-centered Cubic.
Side of elementary cube.....	3.554
Distance between nearest atoms.....	2.514
Density { from X-ray data.....	8.66
{ by standard methods.....	8.718

3. A third sample, prepared by dissolving a portion of sample 1, pure Kalmus cobalt, in  $H_2SO_4$  and electrolyzing, showed a *mixture of about equal amounts of the two forms*. The spacings are tabulated in Table VI., together with the theoretical spacings of both the cubic and hexagonal lattices. It is seen that practically *all the spacings of both lattices are present*.

In Table IV. two extra columns have been added, to take account of the fact that in the hexagonal close-packed lattice the number of planes which coöperate to produce a line is not a correct index of the intensity to be expected, even for consecutive lines, since in some sets of planes the planes are not equally spaced, and hence their reflections are not all in

phase. The number,  $N$ , of cooperating sets of planes must be multiplied by the "phase factor,"  $F$ , of each set of planes, which is the ratio of the square of the sum of the amplitudes of reflection from all the planes in the set *added as vectors*, to the square of their algebraic sum. The product  $NF$  will be seen to represent very well the fluctuations in intensity of consecutive lines, thus confirming their identification.

TABLE VI.

*Cobalt* (3).

Fine powder, obtained by rapid electrolysis of sulphate. Lines rather diffuse.

Intensity of Line.	Spacing of Planes.		
	Observed.	Theoretical Face-centered Cubic.	Theoretical Hexagonal Close-packed.
5	2.158	....	2.176
20	2.037	2.051	2.051
20	1.915	....	1.920
3	1.773	1.777	....
$\frac{1}{2}$	1.484	....	1.493
10	1.250	1.257	1.257
2	1.148	....	1.158
...	....	....	1.089
12	1.066	1.072	1.072
3	1.045	....	1.051
2	1.017	1.025	1.025
1	.952	...	.961
...	...	...	.928
...	...	.888	...
$\frac{1}{4}$	.846	...	.853
...	...	...	.823
...	...	.815	...
$\frac{1}{2}$	.802	...	.807
1	.787	.794	.794
...	...	...	.768
...	...	...	.765
...	...	...	.747
$\frac{1}{4}$	.723	.725	.727
$\frac{1}{4}$	.701	...	.706
$\frac{1}{4}$	.682	.684	.684

## NICKEL.

In a previous paper<sup>1</sup> some preliminary measurements on nickel were reported, which seemed to indicate two crystal forms, one face-centered cubic, the other body-centered cubic. The photographs were very poor, and the results were given as questionable.

These experiments have been repeated with great care. Many photo-

<sup>1</sup> PHYS. REV., 10, 692, 1917.

graphs have been taken of nickel powder prepared in different ways, including electrolysis, reduction of oxide in hydrogen, filings from cast nickel, and the same annealed in hydrogen at different temperatures. *All gave identical patterns of lines, showing face centered cubic structure.* The samples used in the preliminary tests have been lost so that these tests cannot be checked; but sufficient data has been taken to show that the centered cubic form, if it exists, does not appear under ordinary conditions of preparation.

The following measurements, obtained with fine-nickel powder prepared by rapid electrolysis of nickel ammonium sulphate, are typical:

TABLE VII.

*Nickel.*

Fine powder, by electrolysis of nickel ammonium sulphate.

Intensity of Line.	Number of Coöperating Planes.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
50	4	2.038	2.042	111
25	3	1.766	1.770	100 (2)
20	6	1.252	1.253	110 (2)
30	12	1.067	1.065	311
5	4	1.022	1.022	111 (2)
1	3	.884	.885	100 (4)
10	12	.812	.812	331
8	12	.791	.791	210 (2)
5	12	.723	.722	211 (2)
5	16	.681	.681	{ 111 (3) 511
$\frac{1}{2}$	6	.625	.625	110 (4)
4	24	.598	.598	531
2	15	.590	.591	{ 100 (6) 221

Type of Lattice	Face-centered Cubic.
Side of elementary cube	3.540 Å.
Distance between nearest atoms	2.505 Å.
Density { from X-ray data	8.72
by standard methods	8.6-8.93

## ZINC.

The planar spacings obtained from measurements of the powder diffraction pattern do not fit an hexagonal lattice with the axial ratio 1.356 calculated from crystallographic measurements.<sup>1</sup> The determination of the true axial ratio, 1.860, by means of graphs representing planar spacings as a function of axial ratio, is described in the preceding

<sup>1</sup> See Groth, *Chemische Krystallographie*, Vol. I.

TABLE VIII.

*Rhodium.*

Fine powder, from electrolysis of pure rhodium chloride.

Intensity of Line.	Number of Coöperating Planes.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
50	4	2.200	2.203	111
10	3	1.908	1.910	100 (2)
10	6	1.350	1.353	110 (2)
15	12	1.150	1.150	311
2	4	1.100	1.103	111 (2)
...	3	...	.955	100 (4)
2	12	.878	.875	331
1.5	12	.853	.853	210 (2)
1.0	12	.781	.779	211 (2)
$\frac{1}{2}$	16	.736	.731	{ 111 (3) 511
...	6	...	.675	110 (4)
$\frac{1}{2}$	24	.647	.646	531

Type of Lattice.....Face-centered Cubic.  
 Side of elementary cube..... 3.820 Å.  
 Distance between nearest atoms..... 2.700  
 Density { from X-ray data..... 12.18  
 by standard method..... 12.1

TABLE IX.

*Palladium.*

Powder very fine, lines diffuse and faint.

Intensity of Line.	Number of Coöperating Planes.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
10	4	2.274	2.280	111
4	3	1.966	1.975	100 (2)
10	6	1.398	1.399	110 (2)
15	12	1.192	1.189	311
...	4	...	1.140	111 (2)
...	3	...	.987	100 (4)
3	12	.909	.906	331
1	12	.886	.882	210 (2)
1	12	.807	.806	211 (2)
$\frac{2}{3}$	16	.759	.760	{ 111 (3) 511
...	6	...	.697	110 (2)
$\frac{1}{2}$	24	.668	.668	531
$\frac{1}{4}$	15	.656	.660	{ 100 (6) 221 (2)

Type of Lattice.....Face-centered Cubic.  
 Side of elementary cube..... 3.950  
 Distance between nearest atoms..... 2.795 Å.  
 Density { from X-ray data..... 11.40  
 by standard methods..... 11.4 to 11.9

TABLE X.  
*Iridium.*

Powder very fine, lines diffuse and faint.

Intensity of Line.	Number of Coöperating Planes.	Spacing of Planes.		Indices of Forms.
		Observed.	Calculated.	
4	4	2.185	2.193	111
2	3	1.898	1.902	100 (2)
4	6	1.346	1.347	110 (2)
4	12	1.150	1.145	311
1	4	1.092	1.096	111 (2)
..	3	...	.952	100 (4)
2	12	.872	.872	331
$\frac{1}{2}$	12	.847	.850	210 (2)
$\frac{1}{2}$	12	.776	.776	211 (2)
$\frac{1}{2}$	16	.732	.732	{ 111 (3) 511
..	6	...	.672	100 (6)
2	24	.642	.643	531

Type of Lattice.....Face-centered Cubic  
 Side of elementary cube..... 3.805 Å.  
 Distance between nearest atoms..... 2.690 Å.  
 Density { from X-ray data.....23.15  
 by standard methods.....22.42

TABLE XI.  
*Platinum.*

Intensity of Line.	Number of Coöperating Planes.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
10	4	2.265	2.266	111
4	3	1.958	1.964	100 (2)
10	6	1.387	1.390	110 (2)
15	12	1.183	1.183	311
2	4	1.137	1.134	111 (2)
1	3	.983	.983	100 (4)
5	12	.902	.901	331
5	12	.878	.878	210 (2)
4	12	.800	.802	211 (2)
2	16	.758	.756	{ 111 (3) 511
..	6	...	.694	110 (4)
2	24	.663	.664	531
1	15	.658	.657	{ 100 (6) 221 (2)

Type of Lattice.....Face-centered Cubic.  
 Side of elementary cube..... 3.930 Å.  
 Distance between nearest atoms..... 2.780 Å.  
 Density { from X-ray data.....21.23  
 by standard methods.....19.96

paper in this number, and only the summary of results need be given here.

The zinc lattice is found to be a combination of two simple right triangular prism lattices, each of axial ratio 1.860, and side of elementary prism 2.670 Å., with the atoms of one lattice at the centers of the prisms of the other lattice. The cartesian coördinates of the positions of the atoms are therefore

$$m, n, pc,$$

$$m + \frac{1}{3}, n + \frac{2}{3}, (p + \frac{1}{2})c,$$

TABLE XII.

Zinc.

Fine powder, from oxide reduced in H<sub>2</sub> at 600° C

Intensity of Line.	Number of Coöperating Planes × Phase Factor.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
3	1.0	2.472	2.466	0001 (2)
1	0.75	2.293	2.295	10 $\bar{1}$ 0
10	4.5	2.077	2.080	10 $\bar{1}$ 1
2	1.5	1.684	1.678	10 $\bar{1}$ 2
10	4.5	1.339	1.337	10 $\bar{1}$ 3
10	3.0	1.332	1.326	11 $\bar{2}$ 0
$\frac{1}{2}$	1.0	1.235	1.232	0001 (4)
7	6.0	1.172	1.166	11 $\bar{2}$ 2
$\frac{1}{2}$	0.75	1.152	1.148	10 $\bar{1}$ 0 (2)
4	4.5	1.121	1.119	20 $\bar{2}$ 1
$\frac{1}{2}$	1.5	1.088	1.087	10 $\bar{1}$ 4
1	1.5	1.044	1.041	10 $\bar{1}$ 1 (2)
2	4.5	.947	.948	20 $\bar{2}$ 3
(doublet, not resolved)	{ 2 2	{ 4.5 6.0	{ .913 .910	{ 10 $\bar{1}$ 5 11 $\bar{2}$ 4
...	1.5	...	.874	21 $\bar{3}$ 0
3	9.0	.859	.861	21 $\bar{3}$ 1
...	1.5	...	.845	10 $\bar{1}$ 2 (2)
1	{ 1.0 3.0	.827	{ .828 .825	0001 (6) 21 $\bar{3}$ 2
...	1.5	...	.778	10 $\bar{1}$ 6
2	{ 9.0 3.0	.773	{ .773 .771	21 $\bar{3}$ 3 10 $\bar{1}$ 0 (3)
1	4.5	.756	.754	20 $\bar{2}$ 5
2	6	.737	.736	30 $\bar{3}$ 2
$\frac{1}{2}$	3	.717	.715	21 $\bar{3}$ 4
$\frac{1}{2}$	6	.703	.703	11 $\bar{2}$ 6

Type of Lattice.....	Hexagonal Close-packed
Axial ratio.....	1.860
Side of elementary triangular prism.....	2.670 Å.
Distance between nearest atoms {	{ in basal plane..... 2.670 Å.
	{ in pyramid planes..... 2.920 Å.
Density {	{ from X-ray data..... 7.04
	{ by standard methods..... 7.142

where  $m$ ,  $n$ , and  $p$  have all possible integral values,  $c$  is the axial ratio 1.860, and the unit is the side of the elementary triangular prism, 2.670 Å.

The lattice is shown in Fig. 3. *It is one of the two closest packed arrangements of prolate spheroids whose axes are in the ratio 1.140 : 1.*

Each atom has six nearest neighbors in regular hexagonal arrangement about it (in the basal plane), at distances of 2.670 Å. (between centers), and six others almost as near, viz., 2.92 Å., three in the plane below, and three in exactly similar positions in the plane above.

The density is

$$\begin{aligned}\rho &= \frac{\text{mass of 1 atom}}{\text{volume of triangular prism}} \\ &= \frac{65.37 \times 1.650}{\frac{1}{4}\sqrt{3} \times (2.670)^3 \times 1.860} = 7.04.\end{aligned}$$

#### CADMIUM.

The interpretation of the data obtained from powder photographs of cadmium, with the help of graphs of planar spacings against axial ratio, has been described in the preceding paper by A. W. Hull and W. P. Davey. The lattice is found to be hexagonal, of the close-packed type, with axial ratio 1.89, instead of the value 1.335 found in the literature.

A new and slightly better photograph gave the data in Table XIII.

The lattice is exactly like that of zinc, except for axial ratio, viz., *a close-packed arrangement of prolate spheroids whose axes are in the ratio 1.158 : 1.* The cartesian coördinates of the positions of the atoms are:

$$\begin{aligned}m, n, pc, \\ m + \frac{1}{3}, n + \frac{2}{3}, (p + \frac{1}{2})c,\end{aligned}$$

where  $m$ ,  $n$ , and  $p$  represent all possible integral numbers,  $c$  is the axial ratio, 1.890, and the unit is the side of the elementary triangular prism, 2.960. The lattice is therefore a combination of two simple lattices of right triangular prisms, each of side 2.960 Å. and height  $1.89 \times 2.960 = 5.60$  Å., with the atoms of one lattice at the centers of the prisms of the other, and vice versa. Each atom is surrounded by six others, in the basal plane, in a regular hexagon about it, at distances of 2.960 Å., and by six others, almost as near, viz., 3.28 Å., three in the plane below and three in exactly similar arrangement in the plane above.

$$\text{The density} = \frac{\text{mass of one atom}}{\text{volume of triangular prism}} = 8.74.$$





## RUTHENIUM.

There is no crystallographic data regarding ruthenium. The procedure was therefore as follows: A powder photograph was taken in the usual manner, the lines measured and the experimental crystal spacings calculated. These spacings were then laid off on a strip of paper, as described in the preceding article, and this strip moved over the different plots until a position was found on one of them where the experimental lines exactly matched the theoretical lines of the plot.

TABLE XIV.

*Ruthenium.*

Intensity of Line.	No. of Cooperating Planes $\times$ Phase Factor.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
3	0.75	2.315	2.325	10 $\bar{1}$ 0
$\frac{1}{4}$	1.0	2.160	2.134	0001 (2)
10	4.5	2.040	2.040	10 $\bar{1}$ 1
2	1.5	1.572	1.575	10 $\bar{1}$ 2
5	3.0	1.343	1.343	11 $\bar{2}$ 0
5	4.5	1.212	1.214	10 $\bar{1}$ 3
...	0.75	...	1.163	10 $\bar{1}$ 0 (2)
5	6.0	1.139	1.136	11 $\bar{2}$ 2
4	4.5	1.123	1.122	20 $\bar{2}$ 1
...	1.0	...	1.067	0001 (4)
$\frac{1}{2}$	1.5	1.024	1.020	10 $\bar{1}$ 1 (2)
1	1.5	.968	.971	10 $\bar{1}$ 4
2	4.5	.902	.901	20 $\bar{2}$ 3
...	1.5	...	.879	21 $\bar{3}$ 0
3	9.0	.864	.861	21 $\bar{3}$ 1
2	6.0	.838	.836	11 $\bar{2}$ 4
$\frac{1}{4}$	3.0	.816	.813	21 $\bar{3}$ 2
$\frac{1}{2}$	4.5	.803	.802	10 $\bar{1}$ 5
...	1.5	...	.788	10 $\bar{1}$ 2 (2)
$\frac{1}{4}$	3.0	.779	.776	10 $\bar{1}$ 0 (3)
1	9.0	.750	.748	21 $\bar{3}$ 3
1	6.0	.730	.728	30 $\bar{3}$ 2

Type of Lattice .....	Hexagonal Close-packed.
Axial ratio .....	1.59
Side of elementary triangular prism .....	2.686 Å.
Distance between nearest atoms	{ in basal plane .....
	{ in pyramid planes .....
	2.686 Å.
	2.640 Å.
Density { from X-ray data .....	12.56
	{ by standard methods .....
	12.26

The experimental values were found to agree exactly with an hexagonal close packed type of lattice, of axial ratio 1.59.—The indices of the crystal forms corresponding to each of the experimental spacings can be read off on the plot (cf. Fig. 3, p. 556, this number) and any one of them, e.g., the 11 $\bar{2}$ 0

spacing, can be used to calculate the side of the elementary triangular prism. The density is then calculated as the ratio of the mass in the unit triangular prism, viz., one atom, to its volume. The side of the unit prism is thus found to be 2.686 Å., and the ideal density of ruthenium 12.56.

*This lattice is a close packed arrangement of oblate spheroids whose axes are in the ratio 0.973 : 1.* It is composed, like that of Zn and Cd, of two intermeshed lattices of right triangular prisms, each of side 2.686 Å. and height  $1.59 \times 2.686 = 4.272$  Å., the atoms of the first lattice being in the centers of the prisms of the second and vice versa.

Each atom has as nearest neighbor the six atoms of the unit prism of which it is the center, at distances of 2.640 Å., and six others almost as near, in a regular hexagon about it (in the basal plane) at distances of 2.686 Å.

INDIUM.

The interpretation of the X-ray diffraction pattern of indium powder, with the help of graphs of axial ratio against crystal spacings, has been described in the foregoing article, and need only be summarized here.

Indium was found to consist of a *face-centered tetragonal lattice of atoms, with axial ratio 1.06.*

TABLE XV.

*Indium.*

Intensity of Line.	Number of Coöperating Planes.	Spacing of Planes.		Indices of Form.
		Observed.	Calculated.	
20	4	2.70	2.693	111
$\frac{1}{2}$	1	2.42	2.428	001 (2)
5	2	2.29	2.292	100 (2)
2	4	1.675	1.665	101 (2)
$\frac{1}{2}$	2	1.617	1.620	110 (2)
1	4	1.450	1.449	113
2	8	1.392	1.390	311
2	4	1.348	1.347	111 (2)
...	1	....	1.215	001 (4)
$\frac{1}{4}$	2	1.150	1.146	100 (4)
1	8	1.080	1.082	133

Type of Lattice.....	Face-Centered Tetragonal.
Axial ratio.....	1.06
Side of elementary tetragonal prism.....	4.58 Å.
Height of elementary tetragonal prism.....	4.86 Å.
Distance between nearest atoms	{ in basal plane..... 3.24 Å.
	{ in pyramid planes..... 3.33 Å.
Density { from X-ray data.....	7.42
	{ by standard methods..... 7.12

The coördinates of the atoms are

$$\begin{aligned} m, n, pc, \\ m + \frac{1}{2}, n + \frac{1}{2}, pc, \\ m + \frac{1}{2}, n, (p + \frac{1}{2})c, \\ m, n + \frac{1}{2}, (p + \frac{1}{2})c, \end{aligned}$$

where  $m$ ,  $n$ , and  $p$  have all possible integral values,  $c$  is the axial ratio, 1.06, and the unit of length is the side of the elementary face-centered square which is the base of the unit prism. The lattice obviously consists of four intermeshed lattices of right tetragonal prisms, each of side 4.58 Å. and height 4.86 Å. It is shown in Fig. 4.

Each atom has four nearest neighbors, viz., the four corner atoms of the 001 face at whose center it is, at distances of 3.24 Å., and eight others almost as near, four in the centers of the lateral faces of the cube above and four similarly below, at distances of 3.33 Å.

*This lattice is a close packed arrangement for prolate spheroids with axes in the ratio 1.06 : 1. It is an alternative arrangement to the hexagonal type exemplified by Zn and Cd, corresponding to the cubic and hexagonal arrangements, respectively, of close-packed spheres.*

RESEARCH LABORATORY,  
GENERAL ELECTRIC CO.,  
Schenectady, N. Y.

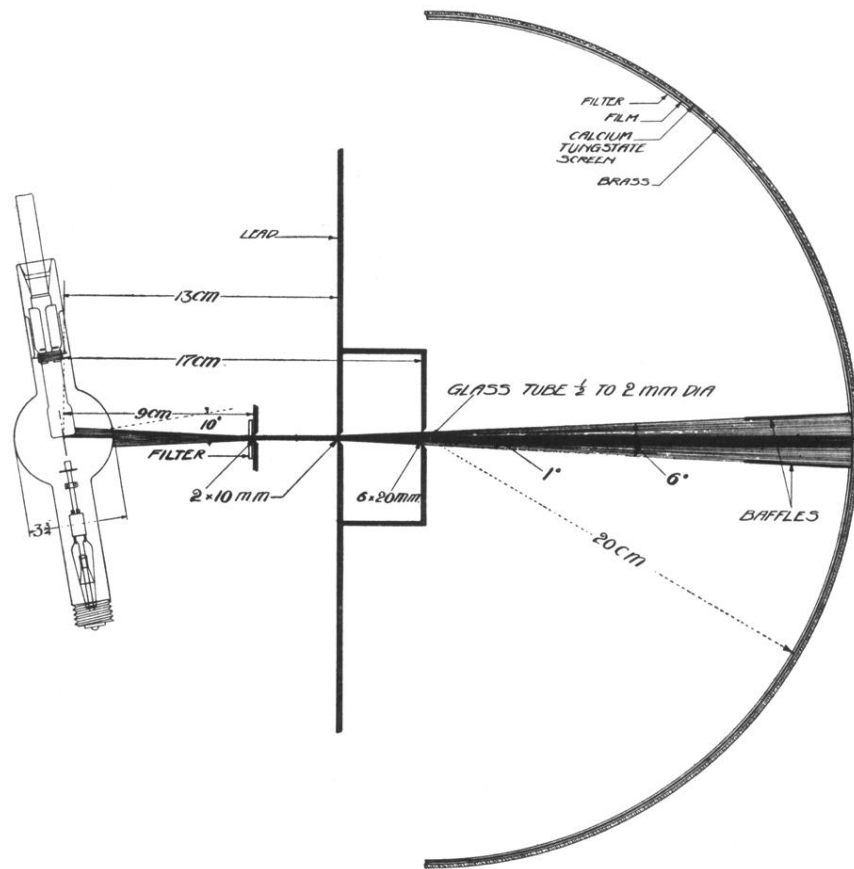


Fig. 1.



Fig. 2.

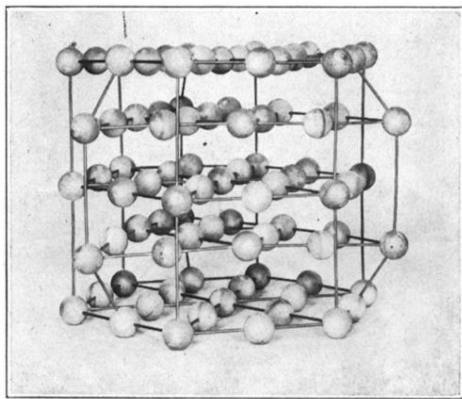


Fig. 3.

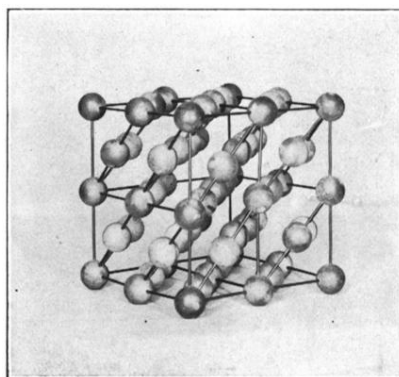


Fig. 4.