

SYNTHESIS, STRUCTURES, AND PROPERTIES OF PLATINUM METAL TELLURIDES¹

W. O. J. GROENEVELD MEIJER,² *Queen's University,
Kingston, Ontario, Canada.*

ABSTRACT

Tellurides of all platinum metals have been synthesized and characterized by *x*-ray and optical methods. Ditellurides of ruthenium, osmium, rhodium, and iridium have pyrite type structures, ditellurides of platinum and palladium are of the cadmium iodide type, iridium and palladium monotellurides have nickel arsenide type structures.

INTRODUCTION AND ACKNOWLEDGEMENTS

Only one platinum metal mineral, sperrylite (PtAs₂) is known to occur with certainty in the Sudbury ores. Since tellurium is one of the products recovered from the ores and no tellurium-bearing platinum metal minerals have been reported, the synthesis of the tellurides of the platinum metals was undertaken in order to obtain data which might eventually be of mineralogical, and therefore of geochemical, interest. More specifically, their synthesis was performed so that optical and crystallographical observations derived from them would be of assistance in their possible ultimate detection in polished sections of the ores.

The work is limited to a description of the products obtained by dry fusion of the metals in various atomic proportions in a vacuum. Some observations are included on phase relationships, especially in the system palladium-tellurium.

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SYNTHESIS

In principle, two methods are available for the preparation of compounds of the platinum metals with metalloids such as tellurium:

- (1) By a "wet" procedure; for instance through the reaction of hydrogen telluride (H₂Te) on a platinum metal salt in solution, and
- (2) By a "dry" procedure; by direct fusion of the weighed constituents.

The latter method is the more favorable for our purposes. When homogeneous (one phase) products are obtained, it is not necessary to

¹ Extracted from a portion of an unpublished Ph.D. thesis by the author: *The Geochemistry of the Platinum Metals with Respect to Their Occurrence in Nickeliferous Sulphide Deposits—Queen's University, 1954.* This work was carried out with the aid of a scholarship from the Research Council of Ontario (1953).

² Now with McPhar Geophysics Limited, 36 Cranfield Road, Toronto, Canada.

analyze the product since we may assume that it will have a composition equal to that of the charge which is weighed out in definite stoichiometric proportions. Otherwise hindersome, secondary reactions such as hydrolysis, oxidation, and disproportionation can be avoided or controlled by heating the charges in evacuated and sealed tubes.

As the platinum metals melt at high to very high temperatures and tellurium melts at a relatively low temperature (452° C. at atmospheric pressure), the reactions take place in the heterogeneous systems solid-vapor or solid-liquid. It is thus of importance that the solid phase be present in a finely divided state. For that reason, the platinum metals were used in either their "sponge" or "black" form. The chemicals used for this work are listed in Table 1.

Charges of 1.2 to 0.5 gm. were prepared by weighing out the platinum metal and the powdered tellurium in proportions, corresponding to the

TABLE 1. CHEMICALS USED FOR FUSIONS

| |
|--|
| Te, metallic, coarsely crystalline, Fisher, C. P. |
| Pd, sponge, Falconbridge .062% other PM's; .009% Fe, .026% SiO ₂ ; traces of Ni and Ag. |
| Pt, sponge, Falconbridge .042% other PM's; .042% SiO ₂ ; traces of Fe and Ni. |
| Ir, sponge, Falconbridge .14% other PM's; .12% Ni, .03% Fe, .13% Si. |
| Rh, sponge, Falconbridge .23% other PM's; .07% Ni, .07% Fe, trace of Ag. |
| Ru, sponge, Falconbridge .05% Rh, .13% Ni, .05% Fe; traces of Ag, Pt, Pd. |
| Os, black, Johnson, Matthey & Mallory, spectrographically pure. |

expected formulae of the compounds to be prepared. The powders were mixed and inserted into Vycor or Vitreosil glass tubes 7–8 mm. bore, 25 cm long sealed at one end. The tubes were then attached to a vacuum pump by airtight rubber tubing and collapsed in the center by heating with an oxygen-hydrogen flame until only a small passageway remained. After evacuation and with the pump running, the narrow constriction was sealed with the flame. During the sealing operation, small amounts of tellurium sublimate from the charge, and condense immediately above the sealed join. For this reason a slight excess of the metalloid was generally added over the calculated amount.

As the reactions in question are exothermic in character, all mixed charges were first brought to reaction by inserting the sealed tubes in a steel shell and heating this slowly in a furnace from room temperature to approximately 850° C. They were left at this temperature for 2 hours and then allowed to cool over a period of 12 hours in the furnace.

With palladium and platinum compounds, the reaction product is compact and apparently homogeneous. In such cases the tubes were

broken open and the nuggets were sawed in halves. One half was mounted in bakelite for polished section study, the other reserved for x -ray examination. With compounds of the other platinum metals the products were generally loosely sintered and very fine-grained. Good x -ray photographs were obtained from these powders. In order to increase the grain size, the sintered fusion products were reground and sealed anew in evacuated silica glass tubing with the addition of a small amount of tellurium. Since the reaction had clearly taken place, the tubes were now heated for 24 or more hours over the hottest gas flame obtainable with a Mecker burner. In some instances, the grain size did increase so that with repetition of the fusion and grinding processes, polished sections could be prepared. Since the products were still porous in those cases, they were impregnated with a solution of bakelite in acetone under reduced pressure.

All x -ray photographs were made by the Debye-Scherrer powder method using a camera with an internal diameter of 57.3 mm.¹ Full correction for film shrinkage was applied. The fusion products belonged either to cubic (pyrite type) or hexagonal (niccolite and cadmium iodide types) systems. After indexing the films by standard methods outlined in Henry, *et al.* (1951), unit cell dimensions were calculated from the more precise back reflections and the interplanar spacings calculated from these dimensions.

INDIVIDUAL PRODUCTS

Ruthenium Ditelluride (RuTe₂)

Ruthenium ditelluride has been synthesized by Thomassen (1929) by dry fusion in vacuum. The structure was determined as being that of pyrite with $a_0 = 6.360 \pm .002$ Å. Wöhler, *et al.* (1933) also synthesized and analyzed the compound by heating ruthenium trichloride with an excess of tellurium in a porcelain boat in a carbon dioxide stream at atmospheric pressure.

In this series of syntheses the compound was prepared by fusing the metals in their correct atomic proportion. The black sintered product showed no crystal forms under the binocular microscope. By refusion in vacuum, a coarser grained product was obtained. Thomassen (1929) reports that the compound melts at a temperature, estimated at between 400 and 600° C. At no time was a liquid phase observed here although much higher temperatures were applied.

Under the microscope, in polished section, the following properties were determined on the porous subhedral aggregate: It is isotropic, fine-grained and has a high (F) Talmage hardness. The color is white, the

¹ Using $\text{CuK}\alpha_1 = 1.54050$ Å. (Ni filter).

reflection is strong. Etch reactions performed after Short (1940) are negative with KOH, KCN, HCl, and FeCl₃. With HgCl₂ an iridescent, in part grey, stain is produced which is easily rubbed off. HNO₃ produces slight effervescence between grain boundaries; a brownish stain with some iridescent colors is produced. The effects are probably due to tellurium, which is found sparsely in the section.

An excellent powder diagram (Table 2) was obtained from which all

TABLE 2. RUTHENIUM DITELLURIDE—RuTe₂: X-RAY POWDER DATA
Cubic, Pyrite Structure; $a_0 = 6.377 \pm .001 \text{ \AA}$

| <i>I</i> | <i>d</i> (meas) | <i>hkl</i> | <i>d</i> (calc) | <i>I</i> | <i>d</i> (meas) | <i>hkl</i> | <i>d</i> (calc) |
|---------------|-----------------|------------|-----------------|----------------|-----------------|------------|-----------------|
| $\frac{1}{2}$ | 3.70 | 111 | 3.68 | 6 | 1.126 | 440 | 1.127 |
| 4 | 3.16 | 200 | 3.19 | 2 | 1.060 | 600 | 1.062 |
| 10 | 2.82 | 210 | 2.85 | 2 | 1.046 | 610 | 1.049 |
| 9 | 2.57 | 211 | 2.60 | 4 | 1.033 | 611 | 1.034 |
| 3 | 2.24 | 220 | 2.25 | 1 | 1.008 | 620 | 1.008 |
| $\frac{1}{2}$ | 2.11 | 300 | 2.12 | 4 | .972 | 533 | .973 |
| 10 | 1.905 | 311 | 1.92 | $1\frac{1}{2}$ | .961 | 622 | .962 |
| 2 | 1.823 | 222 | 1.84 | 4 | .951 | 630 | .951 |
| 4 | 1.752 | 320 | 1.77 | 3 | .940 | 631 | .940 |
| 7 | 1.694 | 321 | 1.70 | 1 | .921 | 444 | .921 |
| 2 | 1.582 | 400 | 1.593 | 1 | .884 | 640 | .884 |
| $\frac{1}{2}$ | 1.466 | 331 | 1.463 | 4 | .877 | 720 | .877 |
| 2 | 1.418 | 420 | 1.426 | 5 | .8680 | 721 | .868 |
| 4 | 1.386 | 421 | 1.390 | 2 | .8530 | 642 | .853 |
| 2 | 1.355 | 332 | 1.359 | 10 | .8319 | 731 | .830 |
| 2 | 1.296 | 422 | 1.301 | 7 | .8181 | 650 | .817 |
| 6 | 1.223 | 333 | 1.227 | 8 | .8117 | 732 | .810 |
| 5 | 1.181 | 520 | 1.183 | 6 | .7989 | 800 | .798 |
| 5 | 1.161 | 521 | 1.163 | 2 | .7752 | 820 | .774 |

lines could be indexed. The unit cell edge with $a_0 = 6.377 \pm .001 \text{ \AA}$ is slightly larger than that obtained by Thomassen (1929).

Osmium Ditelluride (OsTe₂)

Osmium ditelluride has been synthesized by Thomassen (1929) by dry fusion of the elements in vacuum. The substance was found to have a cubic pyrite-type structure with $a_0 = 6.369 \pm .003 \text{ \AA}$.

In this work the compound was synthesized in the usual manner. It was not possible to obtain a polished section from the loosely textured and very fine-grained fusion product. Thomassen (1929) reports that the compound melts at a temperature estimated between 500 and 600° C. No such melting was observed in this work. At high temperatures, the

compound dissociates and tellurium condenses at cooler portions of the evacuated tube.

The powder photograph could be readily indexed on the basis of a cubic unit cell with $a_0 = 6.398_5 \pm .001$ Å. The photograph shows one extra line ($d = 2.07$, Table 3) which corresponds to the strongest osmium line

TABLE 3. OSMIUM DITELLURITE OsTe_2 : X-RAY POWDER DATA
Cubic, Pyrite Structure; $a_0 = 6.398_5 \pm .001$ Å

| <i>I</i> | <i>d</i> (meas) | <i>hkl</i> | <i>d</i> (calc) | <i>I</i> | <i>d</i> (meas) | <i>hkl</i> | <i>d</i> (calc) |
|----------|-----------------|------------|-----------------|---------------|-----------------|------------|-----------------|
| 1 | 3.67 | 111 | 3.69 | 1 | 1.038 | 611 | 1.038 |
| 2 | 3.18 | 200 | 3.20 | 3 | 1.010 | 620 | 1.011 |
| 4 | 2.84 | 210 | 2.86 | 4 | .973 | 533 | .975 |
| 3 | 2.60 | 211 | 2.61 | 3 | .962 | 622 | .964 |
| 3 | 2.25 | 220 | 2.26 | 3 | .951 | 630 | .953 |
| 2 | 2.07 | | | 2 | .9414 | 631 | .943 |
| 10 | 1.909 | 311 | 1.93 | 2 | .8949 | 711 | .895 |
| 1 | 1.836 | 222 | 1.845 | 2 | .8859 | 640 | .886 |
| 1 | 1.770 | 320 | 1.773 | 3 | .8781 | 720 | .878 |
| 3 | 1.702 | 321 | 1.710 | 3 | .8707 | 721 | .870 |
| 1 | 1.426 | 420 | 1.430 | 3 | .8541 | 642 | .855 |
| 1 | 1.392 | 421 | 1.396 | $\frac{1}{2}$ | .8465 | 722 | .847 |
| 2 | 1.360 | 332 | 1.362 | 10 | .8331 | 731 | .8328 |
| 2 | 1.304 | 422 | 1.306 | 4 | .8192 | 650 | .8194 |
| 5 | 1.229 | 333 | 1.230 | 6 | .8122 | 732 | .8125 |
| 2 | 1.184 | 520 | 1.187 | 6 | .7995 | 800 | .7997 |
| 1 | 1.167 | 521 | 1.168 | 1? | .7894 | 811 | .787 |
| 4 | 1.127 | 440 | 1.130 | ? | .7824 | 733 | .781 |
| 1 | 1.080 | 531 | 1.081 | 6 | .7759 | 820 | .776 |
| 2 | 1.067 | 442 | 1.067 | | | | |

and whose presence is not unexpected, considering the observed dissociation of the compound. The intensities of the other lines are quite similar to the corresponding lines of ruthenium ditelluride.

Rhodium (Di?) Telluride (RhTe_{2+x})

Some controversy exists in the literature as to the composition of rhodium-tellurium compounds. Wöhler, *et al.* (1933) synthesized and analyzed a ditelluride. Strangely enough, the authors remark, no compound analogous in composition to Rh_2Se_5 is formed. Biltz (1937) re-analyzed the compound and found a formula $\text{RhTe}_{2.45}$. He reports that the substance has a pseudo-pyrite structure. This structure was reported for $\text{RhSe}_{2.48}$, where pyrite lines were split into several distinct lines,

However, Biltz (1937) only reports that "slight line splitting is likely" for $\text{RhTe}_{2.45}$, and no further x -ray data are given for the compound.

A telluride of rhodium was prepared from a charge of the elements, mixed in the atomic proportion of $\text{RhTe}_{2.5}$. After several heat treatments, a fine-grained product was obtained, which was rendered coherent by an excess of tellurium. The compound is isotropic, its color is white in reflected light and slightly darker than the tellurium with which it was intergrown. The Talmage hardness is high (F). Etch reactions are negative with KOH , KCN , FeCl_3 , and HCl . With HNO_3 faint intergranular

TABLE 4. RHODIUM (DI?) TELLURIDE— RhTe_{2+x} X-RAY POWDER DATACubic, Pyrite Structure; $a_0 = 6.428 \pm .001 \text{ \AA}$

| I | d (meas) | hkl | d (calc) | I | d (meas) | hkl | d (calc) |
|-----|------------|-------|------------|---------------|------------|-------|------------|
| 2 | 3.17 | 200 | 3.22 | 2 | 1.052 | 610 | 1.058 |
| 10 | 2.83 | 210 | 2.87 | 5 | 1.039 | 611 | 1.043 |
| 9 | 2.58 | 211 | 2.53 | 1 | 1.006 | 620 | 1.017 |
| 2 | 2.24 | 220 | 2.27 | 3 | .978 | 533 | .980 |
| 10 | 2.91 | 311 | 1.940 | 1 | .966 | 622 | .969 |
| 1 | 1.83 | 222 | 1.857 | 4 | .958 | 630 | .958 |
| 3 | 1.76 | 320 | 1.782 | 4 | .9461 | 631 | .948 |
| 8 | 1.70 | 321 | 1.719 | 1 | .9264 | 444 | .928 |
| 2 | 1.59 | 400 | 1.608 | $\frac{1}{2}$ | .8898 | 640 | .891 |
| 2 | 1.43 | 420 | 1.438 | 6 | .8828 | 720 | .884 |
| 4 | 1.394 | 421 | 1.403 | 8 | .8744 | 721 | .875 |
| 2 | 1.360 | 332 | 1.370 | 3 | .8592 | 642 | .860 |
| 1 | 1.304 | 422 | 1.312 | 10 | .8368 | 731 | .837 |
| 6 | 1.231 | 333 | 1.238 | 8 | .8229 | 650 | .823 |
| 5 | 1.187 | 520 | 1.193 | 9 | .8161 | 732 | .816 |
| 4 | 1.166 | 521 | 1.173 | 6 | .8035 | 800 | .804 |
| 6 | 1.128 | 440 | 1.137 | 6 | .7797 | 820 | .780 |
| 1 | 1.068 | 600 | 1.071 | 5 | .7755 | 821 | .774 |

effervescence was observed which is likely due to the presence of tellurium.

An excellent x -ray powder photograph was obtained with very clearly resolved $K\alpha_1$ and $K\alpha_2$ doublet in the back reflection region. No line splitting of any kind was observed and all lines could be indexed on the basis of a cubic unit cell with $a_0 = 6.428 \pm .001 \text{ \AA}$ (Table 4). The intensity distribution is very similar to that of ruthenium telluride and a pyrite structure is clearly indicated for this compound. There is thus no evidence to advance in favor of a so-called pseudo-pyrite structure although the lack of tellurium lines on the powder photograph, which was

taken before the compound was prepared for polished section by refusing it with excess tellurium, does tend to corroborate the RhTe_{2+x} formula.

Iridium (Di?) Telluride (IrTe_{2+x})

Wöhler, *et al.* (1933) report the existence of two iridium tellurides: IrTe_2 and IrTe_3 . Biltz (1937) analyzed a compound, saturated with tellurium and found $\text{IrTe}_{2.89}$. He includes the substance in the pseudopyrite structural classification, although it is stated that a slight splitting of pyrite lines is only "likely." No further x -ray observations are mentioned.

In this work an iridium telluride was prepared from the charge $\text{IrTe}_{2.5}$. As with osmium, this compound loses tellurium at higher temperatures. A satisfactory polished section could not be prepared of the friable, sintered product.

The powder photograph of the product was easily indexed on the basis of a cubic unit cell with $a_0 = 6.411 \pm .002 \text{ \AA}$. No effects of line splitting could be observed in the very clear film. The intensities of the lines (Table 5) are very similar to those measured in the other compounds of the platinum metals already described with pyrite structures.

Iridium Monotelluride (IrTe)

In the preparation of iridium ditelluride it was noticed that the product lost tellurium at higher temperatures. The x -ray powder photograph did not show iridium lines and as the diagram seemed amenable to indexing on a hexagonal lattice of the niccolite type, a fusion product was prepared of the composition IrTe .

Iridium monotelluride formed a friable fine-grained sintered product. It was impossible to prepare a polished section of the compound; attempts to fuse it in a hollow carbon electrode in the arc resulted in its dissociation to metallic iridium.

The powder photograph was indexed (Table 6) on the basis of an orthohexagonal unit cell with $a_0 = 3.930 \text{ \AA}$, $c_0 = 5.386 \text{ \AA}$, and $c = 1.370$. After a first trial indexing, more accurate interplanar spacings were obtained with the Philips x -ray diffractometer on a few planes with $00l$, $hk0$, and hkl type indices.

Palladium Ditelluride PdTe_2

Thomassen (1929) prepared palladium ditelluride and found for it a cadmium iodide type structure with $a_0 = 4.028 \pm .003 \text{ \AA}$, $c_0 = 5.118 \pm .004 \text{ \AA}$, and $c = 1.271$. Wöhler, *et al.* (1933) prepared the ditelluride as the only compound in the system Pd-Te.

TABLE 5. IRIIDIUM (DI?) TELLURIDE—IrTe_{2+x} X-RAY POWDER DATA
Cubic, Pyrite Structure; $a_0 = 6.411 \pm .002 \text{ \AA}$

| <i>I</i> | <i>d</i> (meas) | <i>hkl</i> | <i>d</i> (calc) | <i>I</i> | <i>d</i> (meas) | <i>hkl</i> | <i>d</i> (calc) |
|---------------|-----------------|------------|-----------------|----------|-----------------|------------|-----------------|
| 2 | 3.14 | 200 | 3.21 | 1 | 1.063 | 600 | 1.070 |
| 5 | 2.81 | 210 | 2.87 | 1 | 1.048 | 610 | 1.055 |
| 5 | 2.57 | 211 | 2.62 | 3 | 1.036 | 611 | 1.041 |
| 2 | 2.23 | 220 | 2.27 | 2 | 1.009 | 620 | 1.014 |
| 7 | 1.91 | 311 | 1.934 | 4 | .975 | 533 | .979 |
| $\frac{1}{2}$ | 1.82 | 222 | 1.851 | 1 | .964 | 622 | .967 |
| 1 | 1.755 | 320 | 1.780 | 4 | .954 | 630 | .956 |
| 3 | 1.692 | 321 | 1.715 | 3 | .9432 | 631 | .946 |
| 1 | 1.422 | 420 | 1.433 | 1 | .8872 | 640 | .889 |
| 2 | 1.383 | 421 | 1.400 | 3 | .8802 | 720 | .881 |
| 1 | 1.350 | 332 | 1.368 | 4 | .8715 | 721 | .873 |
| 2 | 1.299 | 422 | 1.310 | 3 | .8559 | 642 | .857 |
| 4 | 1.223 | 333 | 1.235 | 10 | .8340 | 731 | .835 |
| 3 | 1.181 | 520 | 1.191 | 5 | .8205 | 650 | .821 |
| 2 | 1.161 | 521 | 1.171 | 6 | .8141 | 732 | .815 |
| 4 | 1.126 | 440 | 1.134 | 5 | .8013 | 800 | .802 |
| | | | | 8 | .7776 | 820 | .778 |

TABLE 6. IRIIDIUM MONOTELLURIDE—IrTe: X-RAY POWDER DATA
Hexagonal, niccolite structure; $a_0 = 3.930 \text{ \AA}$, $c_0 = 5.386 \text{ \AA}$, $c = 1.370$

| <i>I</i> | <i>d</i> (meas) | <i>hk·l</i> | <i>d</i> (calc) | <i>I</i> | <i>d</i> (meas) | <i>hk·l</i> | <i>d</i> (calc) |
|---------------|-----------------|-------------|-----------------|---------------|-----------------|-------------|-----------------|
| 3 | 5.29 | 00.1 | 5.40 | 2 | 1.131 | 30.0 | 1.134 |
| 1 | 3.34 | 10.0 | 3.40 | 4 | 1.107 | 30.1 | 1.111 |
| 10 | 2.83 | 10.1 | 2.88 | 3 | 1.044 | 21.3 | 1.045 |
| $\frac{1}{2}$ | 2.64 | 00.2 | 2.69 | 2 | 1.026 | 10.5 | 1.027 |
| 6 | 2.09 | 10.2 | 2.11 | 3 | .9815 | 22.0 | .9828 |
| 6 | 1.94 | 11.0 | 1.96 | $\frac{1}{2}$ | .9601 | 30.3 | .9594 |
| 2 | 1.824 | 11.1 | 1.847 | $\frac{1}{2}$ | .9479 | 31.0 | .9438 |
| $\frac{1}{2}$ | 1.779 | 00.3 | 1.796 | 3 | .9296 | 31.1 | .9302 |
| 3 | 1.606 | 20.1 | 1.625 | 2 | .9107 | 20.5 | .9102 |
| 3 | 1.576 | 11.2 | 1.588 | 4 | .8917 | 31.2 | .8912 |
| 4 | 1.428 | 20.2 | 1.437 | 6 | .8684 | 10.6 | .8684 |
| 1 | 1.339 | 00.4 | 1.346 | 2 | .8412 | 40.1 | .8406 |
| 1 | 1.317 | 11.3 | 1.326 | 4 | .8361 | 31.3 | .8353 |
| 3 | 1.244 | 21.1 | 1.251 | 5 | .8273 | 21.5 | .8262 |
| 2 | 1.229 | 20.3 | 1.235 | 3 | .8117 | 40.2 | .8113 |
| 4 | 1.156 | 21.2 | 1.160 | 8 | .7945 | 20.6 | .7938 |
| | | | | 1 | .7826 | 30.5 | .7820 |

In this work, palladium ditelluride was formed by fusing the elements in the correct atomic proportions. Metallic nuggets were obtained which were homogeneous in all respects. The compound is coarsely crystalline and shows the excellent platy cleavage, peculiar to compounds with the sheet-like cadmium iodide type structure.

In polished section, palladium ditelluride is homogeneous. The compound is white, lighter in color and more creamy than native tellurium. It is very easily scratched with a steel needle (Talmage hardness *B*). The cleavage plane (0001) is not well developed but shows up by application of a sharp object. The compound is quite strongly anisotropic: bluish and yellowish colors are observed. The etch reactions are negative for KOH, HgCl₂, KCN, and HCl. FeCl₂ forms a brown stain in two to five minutes. With HNO₃ faint effervescence was observed with rapid discoloration of the reagent droplet to orange brown; fumes stain slightly. In a fusion product of composition PdTe₃, the ditelluride is seen with native tellurium.

Interplanar spacings obtained from powder diffraction photographs check well with those calculated from data presented by Thomassen (1929).

Palladium Monotelluride (PdTe)

Thomassen (1929) also prepared palladium monotelluride by dry fusion of the elements in evacuated tubes. For the compound a nickel arsenide structure was found with $a_0 = 4.127 \pm .004 \text{ \AA}$, $c_0 = 5.663 \pm .005 \text{ \AA}$, and $c = 1.372$.

The fusion product of palladium monotelluride forms a brassy compact product. In polished section it is very strongly anisotropic with colors ranging from deep blue to yellow. Its color is creamy white, the Talmage hardness is *C*; no cleavage was observed, either in hand specimen or polished section. The etch reactions are similar to those obtained with the ditelluride: negative with KOH, HgCl₂, KCN, and HCl; FeCl₃ leaves after five minutes an orange-brown stain. A reaction occurs with HNO₃ but no effervescence was observed, the reaction droplet turns deep red-brown but no precipitate forms within 30 seconds as with PdTe₂.

Interstitally between the larger monotelluride grains, a small amount of a myrmekitic intergrowth of monotelluride and an isotropic (?) compound appears. This fine-grained palladium monotelluride extinguished parallel with the larger bounding crystals, which show sutured contacts towards the intergrowth. This texture is not likely eutectic in origin, rather does it appear to be a product of a peritectic reaction, e.g.:



A polished section was also prepared from a fusion product of composition Pd_2Te_3 . Macroscopically it is very similar to the ditelluride but under the microscope, the product is seen to consist of two phases, one the ditelluride, the other an exsolution product, deposited along (0001) directions of the former. The latter is very fine-grained and differs from the monotelluride and the ditelluride by the absence of strong anisotropism. This exsolution product was too fine-grained to be isolated for powder diffraction work.

Thus, aside from PdTe and PdTe_2 , two other phases are indicated: one isotropic phase, occurring as a myrmekitic intergrowth with palladium monotelluride and another fine-grained phase appearing as an exsolution product in PdTe_2 .

Interplanar spacings calculated for palladium monotelluride are very similar to those calculated from Thomassen's data (1929).

Platinum Ditelluride (PtTe_2)

Roessler (1897) prepared platinum ditelluride by heating platinum with an excess of tellurium in an open furnace. Wöhler, *et al.* (1933) also prepared and analyzed the compound. Thomassen (1929) synthesized it from the elements and determined the structure. It has a cadmium iodide type structure with $a_0 = 4.010 \pm .004$ Å, $c_0 = 5.201 \pm .005$ Å, and $c = 1.297$. No other tellurides are reported by Wöhler or Thomassen although the latter attempted to prepare the monotelluride. Roessler (1897) reports the existence of PtTe and Pt_2Te which he obtained by melting the ditelluride in an oxidizing flame of a blowpipe on charcoal.

In this work the ditelluride was prepared in the usual manner. It formed a sintered product after fusion; refusion at higher temperatures yielded a product from which a satisfactory polished section could be prepared. No liquid phase was observed.

In polished section the aggregate of anhedral crystals takes on a good polish. The Talmage hardness is *B*. In reflected light PtTe_2 is white, with the same intensity of reflection as tellurium or slightly darker. It is moderately anisotropic. Etch reactions with HgCl_2 , KCN , HCl , KOH and FeCl_3 are negative. HNO_3 produces locally slow intergranular effervescence. HNO_3 fumes tarnish the compound locally although some of these effects are possibly due to small amounts of tellurium.

The *x*-ray powder photograph of PtTe_2 is slightly diffuse. It is difficult to obtain a good mount of the compound due to its softness and flaky nature. Measured interplanar spacings agree well with those calculated from Thomassen's data (1929).

TABLE 7. SUMMARY OF PROPERTIES DETERMINED ON FUSION PRODUCTS

| Substance | Structure Type | Lattice Dimensions | Nature of Product | Anisotropism X/Nicols | Pleochroism 1 Nicol | Color | Bright-ness | Hard-ness | Cleavage Parting | Etch Reactions | | | |
|---------------------|--------------------|--|--------------------|--------------------------|------------------------|--------------|-------------|-----------|-------------------|------------------|-------------------|-------------------|-------------------------|
| | | | | | | | | | | HCl, KOH, KCN | HgCl ₂ | FeCl ₃ | HNO ₃ |
| RuTe ₂ | FeS ₂ * | 6.377 | sintered aggregate | isotropic | none | white | strong | F | none | neg. | irides. stain? | neg. | sl. eff. inter-granular |
| OsTe ₂ | FeS ₂ * | 6.398 | loose powder | — | — | — | — | — | — | — | — | — | — |
| RhTe _{2+z} | FeS ₂ * | 6.428 | sintered aggregate | isotropic | none | white | strong | F | none | neg. | fg'd ppte? | neg. | sl. eff. inter-granular |
| IrTe _{2+z} | FeS ₂ * | 6.411 | loose powder | — | — | — | — | — | — | — | — | — | — |
| IrTe | NiAs | a ₀ = 3.930 c ₀ = 5.386 | loose powder | — | — | — | — | — | — | — | — | — | — |
| PdTe ₂ | CdI ₂ | a ₀ = 4.028 c ₀ = 5.118 | c. gr'd nugget | moderately strong | none | creamy white | med. str. | B. | pinacoidal (0001) | neg. | neg. | brown stain | sl. eff. irid. st. |
| PdTe | NiAs | a ₀ = 4.127 c ₀ = 5.663 | med. gr'd nugget | strong | none | creamy white | med. str. | C | none | neg. | neg. | brown stain | no. eff. pos. stain |
| PtTe ₂ | CdI ₂ | a ₀ = 4.010 c ₀ = 5.201 | sintered aggregate | moderate | none | white | med. str. | B | pinacoidal (0001) | neg. | neg. | neg. | sl. eff. fumes stain |

* Pyrite type structure.

CONCLUSIONS

The dry fusion procedure of synthesis has been found successful for preparing the following compounds:

RuTe₂, OsTe₂, RhTe_{2+x}, IrTe_{2+x}, IrTe, PdTe₂, PdTe, and PtTe₂.

Only palladium compounds form melts; other products do not melt in vacuum over the hottest gas flame available—in contrast to published accounts—indeed, they tend to dissociate at very high temperatures.

The platinum metal tellurides RuTe₂, OsTe₂, RhTe_{2+x}, and IrTe_{2+x} all belong structurally to the pyrite group. Their interplanar spacings as well as the densities of their lines on *x*-ray powder photographs are consequently very similar; the unit cell edges are within the range $6.377 \leq a_0 \leq 6.428$ Å. The ditellurides of platinum and palladium are both hexagonal and belong to the cadmium iodide type which is characterized by a strong cleavage parallel (0001). IrTe and PdTe have niccolite structures. Properties determined on all products are shown in Table 7.

REFERENCES

- BILTZ, W. (1937): Notiz zu Selen- und Tellurverbindungen von Iridium and Rhodium. *Z. anorg. algem. Chem.*, **253**, 282–285.
- HENRY, N. F. M., LIPSON, H., & WOOSTER, W. A. (1951): *The interpretation of x-ray diffraction photographs*. Macmillan & Co., London.
- ROESSLER, C. (1897): Über Tellurverbindungen des Platins. *Z. anorg. Chem.*, **15**, 405–411.
- SHORT, M. N. (1940): Microscopic determination of the ore minerals. 2nd ed., *U. S. Geol. Surv., Bull.* **914**.
- THOMASSEN, L. (1929): Über Kristallstrukturtypen einiger binärer Verbindungen der Platinmetalle. *Z. phys. Chem.*, **48**, 277–287.
- WÖHLER, L., EWALD, K., & KRALL, H. G. (1933): Die Sulfide, Selenide, und Telluride der sechs Platinmetalle. *Ber. Deu. Chem. Ges.*, **66**, part II, Section B, pp. 1638–1652.