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PHASE RELATIONS IN THE SYSTEM Pt-Pd-Sb-Te

AT 1000°C, 800°C AND 600°C

BY

© WON SA KIM, M.Sc.

A thesis submitted to the Faculty of
Graduate Studies and Research in partial fulfilment
of the requirements for the degree of Doctor of Philosophy.

Department of Geology
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March 1984
The undersigned hereby recommend to the
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ABSTRACT

Phase relations in the ternary portions of the system Pt-Pd-Sb-Te have been investigated at 1000°C, 800°C and/or 600°C using mixes sealed in evacuated silica glass capsules.

In the Pt-Sb-Te system at 1000°C, Pt, PtSb, PtSb2, PtTe2, Pt3Te4 and two liquids are stable, forming four 3-phase and ten 2-phase assemblages. At 800°C, 2 additional phases, PtTe and Pt2Te3, become stable, resulting in seven 3-phase and fourteen 2-phase fields. At 600°C, Pt34Sb7, Pt3Sb, Pt3Sb2, Sb2Te3 and Sb appear, one liquid field disappears and the other, along the Sb-Te join, splits into two liquid fields, resulting in twelve 3-phase and twenty-five 2-phase assemblages. All the binary phases show some Sb-Te substitution with the most significant, 28.5 at.% found in both PtSb2 (1000°C and 800°C) and PtTe2 (1000°C).

In the Pd-Pt-Sb system, the 1000°C isothermal section consists of (Pd,Pt) solid solution, Pd20Sb7, Pd8Sb3, Pd31Sb12, Pd5Sb2, PtSb, PtSb2 and one liquid, forming one 3-phase and nine 2-phase assemblages. 2 more phases, PdSb and Pd5Sb3, become stable at 800°C and the liquid field splits into two fields, producing one 3-phase and sixteen 2-phase assemblages. The Pt-Pd substitution in most Pt-Sb and Pd-Sb phases increases significantly as temperature decreases from 1000°C to 800°C.

At 1000°C, the stable phases of the Pd-Sb-Te system are Pd, Pd20Sb7, Pd8Sb3, Pd31Sb12, Pd5Sb2 and one liquid with one 3-phase and nine 2-phase assemblages confirmed. The
substitution of Te for Sb reaches 42 at.% in Pd$_{20}$Sb$_7$, 38 at.% in Pd$_8$Sb$_3$, 38.3 at.% in Pd$_{31}$Sb$_{12}$ and 16.5 at.% in Pd$_5$Sb$_2$.

A spot-check of the previously published phase relations confirmed the Pt-Sb, Pt-Te and Pd-Sb binary phase diagrams. In the Pd-Te system, a new phase, Pd$_7$Te$_3$, was found and the phase relations in the region 0 - 50 at.% Te were re-determined. Phase relations in the Sb-Te system were only partly confirmed.

The X-ray powder pattern of Pd$_8$Te$_3$ was indexed on an orthorhombic cell with $a=12.843(3)$, $b=15.126(3)$, $c=11.304(2)$ Å and that of Pd$_7$Te$_3$ on a monoclinic cell with $a=7.444(1)$, $b=13.918(2)$, $c=8.873(2)$ Å, $\beta=92.46(2)^\circ$. The X-ray powder patterns of Pd$_5$Sb$_3$ and Pt$_{34}$Sb$_7$, not indexable on the cells previously proposed, were indexed respectively on an orthorhombic cell with $a=3.362(1)$, $b=17.484(7)$, $c=6.934(2)$ Å and a tetragonal cell with $a=3.948(3)$, $c=16.85(1)$ Å.

Optical properties, micro-indentation hardness and X-ray powder data are included for most of the synthetic phases.
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CHAPTER I

INTRODUCTION

The platinum group elements commonly occur in mafic or ultramafic igneous rocks in the form of native elements, alloys, or compounds with S, Sb, Te, As, Se, Bi, Sn, Fe, Pb, Hg, Ni, Cu, etc. Most of the platinum group minerals exhibit complex compositions mainly due to multi-element substitutions. In "Platinum-Group Elements: Mineralogy, Geology, Recovery" (edited by Cabri, 1981), the most comprehensive book on the platinum group elements for mineralogists and geologists, 78 established platinum group minerals and 29 unnamed ones were listed; the latter considered to be probable new species. In the same book physical properties, cell parameters and microprobe analyses were compiled for these named and unnamed minerals.

In this study, phase relations of the system involving the elements Pt, Pd, Sb and Te were selected because minerals of Pt and Pd with Sb and Te comprise over 20 percent of the recognized platinum group minerals, including geversite [PtSb$_2$], stumplite [PtSb], genkinite [(Pt,Pd)$_4$Sb$_3$], moncheite [PtTe$_2$], mertieite-I [(Pd,Cu)$_{5+x}$(Sb,As)$_{2-x}$], mertieite II [Pd$_8$Sb$_3$], stibiopalladinite [Pd$_{5+2x}$Sb$_{2-x}$], sudburyite [PdSb], telargpalite [(Pd,Ag)$_{4+8}$Te], kotulskite [PdTe], merenskyite [PdTe$_2$], keithconnite [Pd$_{3-x}$Te$_x$], telluropalladinite [Pd$_9$Te$_4$], borovskite [Pd$_3$SbTe$_4$], testibiopalladinite [PdSbTe], and native platinum and palladium. In addition,
experimental data on the phase relations of the ternary systems of Pt-Pd-Sb-Te are very sparse, and most studies have been restricted to the binary portions of this system. Compilation of most of the binary data can be found in Hansen and Anderko (1958), Elliott (1965), Shunk (1969), and Moffatt (1979). Experimental data for the ternary portions of this system found in the literature consist of two isothermal sections, namely, Pt-Sb-Te at 650°C (El-Boragy and Schubert, 1971b) and Pd-Sb-Te at 400°C (El-Boragy and Schubert, 1971a).

The main objective of this thesis is to explore the phase chemistry and to determine stable phase assemblages in the ternary portion of the condensed system Pt-Pd-Sb-Te by constructing the phase diagrams using mineral synthesis data. Six ternary isothermal sections were investigated, including three ternary portions of the system Pt-Sb-Te at 1000°C, 800°C and 600°C; two ternary portions of the system Pt-Pd-Sb at 1000°C and 800°C; and one ternary portion of the system Pd-Sb-Te at 1000°C. In addition, (1) six binary portions which bound the ternary portions mentioned above were examined, because they are the foundation of the ternary systems to be studied and considerable disagreement exists on these binary phase diagrams in the literature, and (2) optical, physical, and X-ray powder data of the synthetic phases determined in this study were compiled.
CHAPTER II

METHODS OF INVESTIGATION

1. Synthesis

A. Starting materials

High-purity elements, platinum (wire), palladium (wire and powder), antimony (ingot) and tellurium (ingot) used in this study were obtained from Johnson Matthey Chemical Ltd. Spectrographic analyses of the materials, provided by the same company, are given in Table 1. All materials were stored in evacuated desiccators to ensure a contamination-free environment.

B. Preparation of runs

Silica capsules were used as containers for all experiments. The high melting point and low thermal expansion coefficient of silica glass allows it to be heated up to 1000°C in the furnace and subsequently quenched in ice water without breaking. The capsules were prepared from transparent silica tubes with 5 mm inside diameter and 1 mm wall thickness. Silica glass tubes were cut into approximately 12 cm lengths, and were later sealed at one end using a flame of acetylene gas and oxygen. The 12 cm length of tubes were cleaned inside and out with acetone, and then dried in a desiccator for at least one day.

Platinum and palladium wires of desired weight were cleaned with acetone and made into coils or cut into small pieces. Large fragments of antimony and tellurium were
<table>
<thead>
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<th>élément</th>
<th>type</th>
<th>purity</th>
</tr>
</thead>
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<tr>
<td>platinum (Pt)</td>
<td>wire (0.25 mm diameter)</td>
<td>99.9995% purity with Fe, Ca, Cu, Mg, Si, Ag, all&lt;0.0005%</td>
</tr>
<tr>
<td></td>
<td>powder</td>
<td>99.9996% purity with Si, Ca, Cu, Fe, Mg, Ag, all&lt;0.0004%</td>
</tr>
<tr>
<td>palladium (Pd)</td>
<td>wire (0.25 mm diameter)</td>
<td>99.999% purity with Fe, Ca, Ag, Si, Cu, Mg, all&lt;0.001%</td>
</tr>
<tr>
<td></td>
<td>powder</td>
<td>99.9999% purity with Bi, Si, all&lt;0.0001%</td>
</tr>
<tr>
<td>antimony (Sb)</td>
<td>broken ingot</td>
<td>100% purity with no other elements detected</td>
</tr>
<tr>
<td>tellurium (Te)</td>
<td>broken ingot</td>
<td></td>
</tr>
</tbody>
</table>

Source: Johnson Matthey Chemical Ltd.
crushed into small pieces. A balance, Mettler 20, was used for weighing the starting materials. The method of loading by difference was adopted: i.e., the capsule was weighed first, and one element was added, and then the tube was reweighed. This process was repeated until the required amounts of each element were added to the capsule. In cases when palladium powder was used, an amount of reagent which had been approximately weighed was swept through a funnel to the bottom of the tube using a soft brush. Since a very small amount of powder always adheres to the glass walls, these were freed as much as possible by tapping or immersing the closed end of the tube in the ultrasonic vibrator. This process was carried out very carefully to ensure the accurateness of the composition of the mixes. The net weight of the mixes ranged from 250 to 350 mg in most cases.

After loading the reagents into the tube, a tightly fitting silica glass rod about 2 cm long was placed on top of the mixture in order to keep the mix in place and to reduce the vapor volume on heating. The tube was then necked down, using a torch, to a capillary size located just above the rod. The sample end of the tube was wrapped with wet asbestos cloth to prevent heating of the reagents. The capsule was then evacuated with a high vacuum pump for 5 to 10 minutes, and sealed off with a torch.

C. Furnace

Two types of furnaces were employed for the
experiments. The first type of the furnace used was the Lindberg Hevi-Duty Model 55031 Tube Furnace which is a horizontal, cylindrical, electrical muffle furnace. Temperatures were controlled by a Sirect Mark 2 Proportional Temperature Controller. Temperatures were measured by 7553-5 Type K-3 Universal Potentiometer and Type E Guarded DC Galvanometer with reference junction at 0°C, using chromel-alumel thermocouples for temperatures at or below 800°C and platinum thermocouples for temperatures above 800°C. The thermocouples were calibrated to the melting point of cadmium chloride (568°C) and sodium chloride (801.5°C). The thermocouple was suspended approximately 0.5 cm below the top of the heating chamber of a furnace, and the charges were placed on the floor of the heating chamber (see Appendix 1). It was assumed that the charges were heated to the temperature recorded by the thermocouple. Temperatures were checked daily and found to be constant within ± 2°C. Besides six furnaces that were used at Carleton University, four furnaces of a similar type at the Canada Centre for Mineral and Technology (CANMET) were also used.

The second type used was the Astro Model 1000A Furnace, a graphite resistance furnace capable of operating up to 3000°C in an evacuated condition. This was used for the runs requiring temperatures higher than 1000°C. An optical pyrometer was used to measure the temperatures with an accuracy of ± 10°C.
D. Heating and quenching

All runs, except a few for temperatures higher than 1000°C, were loaded in the muffle furnaces using tongs and heated at selected temperatures for a period from a few days to a few weeks until all the elements appeared to have reacted (based on visual examination of the reaction product under a binocular microscope). To ensure homogeneity, most samples were opened, and the contents were finely ground. The charges were then pelletized with a 4.8 mm internal diameter press at about 1476 kg/cm², in order to speed up the next reaction. Each pellet was then re-loaded into a silica glass capsule and sealed in the same way as described earlier. Samples were returned to the furnace for a further heating period ranging from a few days to six months. For the cases where one of the reaction products was liquid, shorter heating periods were used. In a few cases where reaction proceeded slowly, grinding and pelletizing of a run product was repeated two or three times.

2. Analytical Methods

A. Reflecting microscopy

A representative sample of each run product was taken for examination in a polished section. In order to minimize the time needed for preparation of polished sections, eight to twelve samples were embedded in one mount. This also reduced the amount of time for loading
and unloading the polished sections during the electron microprobe work. Optical properties of each phase and textural relationships among the phases present were examined using a conventional reflecting microscope.

In some cases, the colour contrast between phases was enhanced after the polished surface was carbon-coated. This was helpful in distinguishing one phase from the other(s), especially between those which have almost identical optical properties. Colours in oil were also observed.

B. X-ray diffraction

1) Gandolfi cameras with a diameter of 114.6 mm were used for identification of phases and for comparison of X-ray diffraction powder patterns of similar phases. Small fragments of the reaction products were picked off with a needle under the binocular microscope. They were mounted on a glass fiber, which was then mounted in the camera. All Gandolfi X-ray patterns were obtained at 35 KV and 30 mA using filtered CuKα radiation with approximately 6 hour exposure.

2) When accurate X-ray data were required, a Philips automated X-ray diffractometer consisting of a vertical goniometer (PW 1050), a microprocessor (PW 1710) and a printing recorder (PW 8210) was used. Smear amounts of finely ground samples of approximately 100 to 150 mg were used for this purpose. A survey diffractometer tracing was
first obtained with a scanning speed of the goniometer of 0.1 or 0.05\(^\circ\) (2\(\theta\)) per second, using filtered CuK\(\alpha\) radiation. All the runs were operated at 40 KV and 50 mA. The PM 8210 printing recorder labels automatically the d-values based on the \(\lambda = 1.54059\)\(\AA\) to all peaks on the diffractometer tracing. More reliable d-values were obtained, after the completion of each scan, with a routine built into the microprocessor for searching for the maximum intensity.

Cell parameters for each phase were then refined by a least-squares method using d-values of reflections in the high 2\(\theta\) angle region (2\(\theta \) > 35\(^\circ\)) where K\(\alpha\), and K\(\alpha\), peaks are resolved.

C. Electron microprobe analysis

Chemical compositions of individual phases in each run product were determined using a Cambridge MK5 Electron Microprobe Analyser. Synthetic compounds and spectrographically pure elements (Pt, Pd, Sb and Te) were used as standards for electron microprobe analyses. All analyses were carried out at an accelerating voltage of 25 KV, with a 1 - 2 \(\mu\)m beam, and a specimen current of 50 nanoamperes measured on a synthetic PtSb\(_2\) standard. X-ray lines and standards used for samples in different systems are shown in Table 2. X-ray intensities were converted to element concentrations by the EMPADR VII computer program of Rucklidge and Gasparrini (1969). Errors of microprobe analyses are approximately 1\% of major elements present.
Table 2. Standards and X-ray lines for electron microprobe analyses.

<table>
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<tr>
<th>X-ray lines</th>
<th>Pt-Sb</th>
<th>Pt-Te</th>
<th>Pd-Sb</th>
<th>Pd-Te</th>
<th>Pt-Pd</th>
<th>Sb-Te</th>
<th>Pt-Sb-Te</th>
<th>Pt-Pd-Sb</th>
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<td>(\text{PtTe}_2)</td>
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<td>(\text{Pt}_3\text{Sb}_2)</td>
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<td>(\text{PtTe})</td>
<td>(\text{PdSb})</td>
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<td></td>
<td>(\text{PdSb})</td>
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<td>(\text{PdSb})</td>
</tr>
<tr>
<td>PdLα</td>
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<td>(\text{PtTe}_2)</td>
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<td>(\text{Pt}_3\text{Sb}_2)</td>
<td></td>
<td></td>
<td>(\text{Pt}_3\text{Sb}_2)</td>
<td>(\text{PdSb})</td>
<td>(\text{PdSb})</td>
</tr>
<tr>
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<td>(\text{PdTe})</td>
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<td></td>
<td></td>
<td>(\text{PdSb})</td>
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<td>(\text{PdSb})</td>
<td>(\text{PdSb})</td>
<td>(\text{PdSb})</td>
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<td>(\text{Pt}_3\text{Sb}_2)</td>
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<td>(\text{PdTe})</td>
<td>(\text{PdTe})</td>
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</tbody>
</table>
The composition of the phases were calculated on the basis of number of atoms per formula from the average of several analyses.

D. Differential thermal analysis

Differential thermal analyses were carried out for a few samples in the binary system Sb–Te using a Fisher Series 100A DTA/TGA system. All analyses were made in a nitrogen atmosphere to avoid oxidation of samples on heating, and with the specimen thermocouples shielded within very fine silica glass tubing (3 mm ID/4 mm OD) to prevent reaction between the thermocouples and samples. Sample weight was approximately 150 mg. Powdered Al₂O₃ was used as a reference material for all runs. The heating rate was 10°C per minute. The thermogram was recorded on a dual-channel strip chart recorder. The DTA temperatures quoted in this thesis were for heating.

E. Micro-indentation hardness

Micro-indentation hardness was measured for the synthetic phases using a Leitz DURimet small-hardness tester. The tester was checked for accuracy and was found to perform well within specifications. Vickers hardness number (VHN) was calculated based on the formula,

\[ VHN = \frac{1854.4 \times P}{d^2} \]

where \( P \) = the load in grams and \( d \) = average length of the
diagonals of indentation in μm. Duration of each test was 25 seconds (15 seconds for lowering the testing diamond on to the specimen and 10 seconds for indentation). Distances of the diagonals of the hardness indentation were measured by means of the 40-power measuring objective with numerical aperture 0.70.

As recommended by the Commission on Ore Microscopy of the International Mineralogical Association (Criddle, 1980; Henry, 1980), Vickers hardness numbers were determined using a standard load of 100 gf (gram force), whenever the grain size of a phase permitted. Smaller loads were, however, used for fine-grained phases.
CHAPTER III
THE BINARY SYSTEMS

The phase relationships of the three bounding binary systems of a given ternary system are the foundation upon which the knowledge of the ternary system is built. Thus it is important to review and re-examine the binary systems before work on the ternary system begins. The system Pt-Pd-Sb-Te under investigation includes four ternary systems, Pt-Sb-Te, Pd-Sb-Te, Pt-Pd-Sb, and Pt-Pd-Te, which, in turn, involve six binary systems, Pt-Sb, Pt-Te, Pd-Sb, Pd-Te, Pd-Pt, and Sb-Te. Experimental data on the phase relations of the six binary portions of the Pt-Pd-Sb-Te system are numerous, and most of them are compiled in detail by Hansen and Anderko (1958), Elliott (1965), Shunk (1969), Savitsky et al. (1978), Moffatt (1979), and Berlincourt et al. (1981). However, there are regions within each binary system where phase relations remain ambiguous. Some binary compounds have been previously synthesized at specific temperatures without any attempt to determine their stability or compositional range. Some of the phases previously reported have not been confirmed by others. Furthermore, compositions for some phases in previously published phase diagrams have been revised by later investigators, due to use of more sophisticated analytical methods.

In this study, phase relationships of each binary system were re-examined by spot checks across the whole
compositional range at temperatures between 400° and 1000°C to confirm the phases reported. Special attention was given to the controversial region(s) where more concentrated study was made. Mineralogical study on each synthetic phase and comparison with naturally occurring minerals will be discussed separately in Chapter V.

1. The Pt-Sb System

A. Previous study: Phase relations of this system were first investigated by Friedrich and Leroux (1909) and later by Nemilow and Woronow (1936). Fig. 1 given by Hansen and Anderko (1958) represents a compromise between that of Friedrich and Leroux (1909) and Nemilow and Woronow (1936). In the phase diagram, thermal data of Friedrich and Leroux (1909) and Nemilow and Woronow (1936) are shown as circles and crosses, respectively. As shown in Fig. 1, there is a substantial agreement from 50 to 100 at.% Sb, as indicated by the existence of the PtSb and PtSb₂ phases and a liquidus line between PtSb₂ and Sb. However, there is a considerable difference in the 10 - 50 at.% Sb region of the phase diagrams. Friedrich and Leroux (1909) placed the Pt₄Sb - PtSb eutectic at 33.6 at.% Sb, 690±10°C, and reported that the Pt₅Sb₂ phase was formed peritectoidally at 637±8°C. In contrast, Nemilow and Woronow (1956) placed the Pt₄Sb - PtSb eutectic at a lower temperature (633±8°C), but at the same composition. They also reported that the Pt₄Sb and PtSb phases undergo transformations at
Fig. 1. Phase diagram of the Pt-Sb system by Hansen and Anderko (1958). A compromise between those of Friedrich and Leroux (1909, Ref. 1) and Nemilow and Woronow (1956, Ref. 2).

Fig. 2. Phase diagram of the Pt-Sb system by Bhan et al. (1969).
671° and 660°C, respectively, and did not find the Pt₅Sb₂ phase. Bhan et al. (1969) re-investigated this system in the region 0 - 50 at.% Sb and their results are shown in Fig. 2. These new data include: a eutectic point between Pt₄Sb and Pt₃Sb₂ at 31 at.% Sb and 633°C; the Pt₃Sb phase melts incongruently to form Pt₄Sb and liquid (30 at.% Sb) at 682°C; Pt₃Sb₂ melts incongruently at 732°C forming PtSb and liquid (33 at.% Sb). The formula for the phase previously known as Pt₄Sb was revised by Bhan and Schubert (1969) to Pt₄+Sb as the Pt content of the phase was about 81 to 82 at.% Pt.

B. Present study: The present investigation was aimed primarily at the confirmation of the existence of Pt₃Sb and Pt₃Sb₂ reported by Bhan and Schubert (1969), and Pt₅Sb₂ reported by Friedrich and Leroux (1909).

All the runs prepared for this system and phases identified from their reaction products are shown in Table 3. At 600°C, run 101 yielded two phases, Pt₄+Sb and Pt containing 6.1 at.% Sb in solid solution. Run 107 quenched from 650°C consisted of Pt₄+Sb and Pt₃Sb. The composition of the Pt₄Sb phase was determined by microprobe analyses to range from Pt₈₃Sb₁₇ (run 101) to Pt₈₂.₇Sb₁₇.₃ (run 107). The Pt : Sb ratio is clearly not 4 : 1 but closer to 4.85 : 1, and therefore, the formula Pt₃₄Sb₇ is assigned to this phase. This is supported by run 107 (with a starting composition of Pt₈₀Sb₂₀ and quenched from 650°C), which gave a two-phase product consisting of Pt₃₄Sb₇ and Pt₃Sb. As
<table>
<thead>
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<th>Run no.</th>
<th>Composition (at.%)</th>
<th>Temp. (°C)</th>
<th>Heating period (days)</th>
<th>Phases present</th>
<th>Composition (at.%)</th>
</tr>
</thead>
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<td>Pt</td>
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<td>15.45</td>
<td>1000</td>
<td>4 (\times) 4</td>
<td></td>
</tr>
<tr>
<td>466</td>
<td>5.00</td>
<td>95.00</td>
<td>1000</td>
<td>3 (\times) 3</td>
<td></td>
</tr>
</tbody>
</table>

Abbreviations: L = liquid, 1h = initial heating, 2h = second heating after grinding and pelleting, 3h = third heating after grinding and pelleting.
Bhan and Schubert (1969) and Bhan et al. (1969) did not publish the X-ray powder pattern of their Pt$_4$Sb phase, the structural identity of Pt$_{34}$Sb$_7$ with Pt$_4$Sb could not be confirmed.

The Pt$_3$Sb and Pt$_3$Sb phases were confirmed in runs 103 and 104, but the Pt$_5$Sb$_2$ phase was not found in this study. Attempts to synthesize the single phase Pt$_{34}$Sb$_7$ (run 114) and Pt$_3$Sb (run 108) were not successful, even after repeated heating and grinding of the mixes. This is apparently due to the slow reaction rate at the lower temperature range, as evidenced by the presence of unreacted Pt in the run products.

At 800°C, Pt, PtSb and PtSb$_2$ are the only stable solid phases. Two different liquids with relatively limited compositional fields also exist at this temperature. Phase relations at 1000°C are basically the same as those at 800°C. The Sb content of Pt coexisting with liquid varies from 10 at.% Sb at 800°C to 7.5 at.% Sb at 1000°C. These values are comparable with those of 10 at.% Sb at 800°C and 8 at.% Sb at 1000°C given by Friedrich and Leroux (1909) and Nemilow and Woronow (1956), but are much higher than the values of 4 at.% Sb at 800°C and 3 at.% Sb at 1000°C derived from the phase diagram of Bhan et al. (1969). The composition of liquid phases coexisting with Pt at 800°C and with PtSb at 1000°C could not be determined due to the separation of solids from the former liquids during quenching.

In the region between PtSb$_2$ and Sb, run 352 (with a
starting composition of Pt$_{14.98}$Sb$_{85.02}$, quenched from 800°C, yielded PtSb$_2$ in equilibrium with a liquid of Pt$_{0.1}$Sb$_{99.9}$ (Fig. 3) and run 460 (with a starting composition of Pt$_{20.29}$Sb$_{79.9}$), quenched from 1000°C, yielded an assemblage of PtSb$_2$ and liquid of Pt$_{0.4}$Sb$_{99.6}$ (Fig. 4). While the reaction product of the third run (466, with a bulk composition of Pt$_3$Sb$_{95}$ and quenched from 1000°C) consisted of PtSb$_2$ and a liquid whose composition could not be determined accurately due to the separation of minute dendrites of PtSb$_2$ composition from an original liquid during quenching (Fig. 5). Nevertheless, it indicates that the liquidus curve over much of the range between PtSb$_2$ and Sb must be somewhere above 1000°C. The possible liquidus line between PtSb$_2$ and Sb must be much steeper than that shown in the previously published phase diagrams (Figs. 1 and 2).

The experimental results obtained from this investigation showed that the phase diagram of Bhan et al. (1969) is essentially correct, and only minor revisions are necessary. A revised phase diagram is given in Fig. 6, in which starting compositions of the runs examined are represented with dots. Electron microprobe analyses of the Pt-Sb phases are shown in Table 4.

The important features of this phase diagram are summarized as follows:

(i) Seven solid phases are stable at 600°C, namely, Pt, Pt$_{34}$Sb$_7$, Pt$_3$Sb, Pt$_3$Sb$_2$, PtSb, PtSb$_2$, and Sb. Among these
Fig. 3. Euhedral and subhedral grains of PtSb₂ (dark grey) coexist with former liquid (dark yellow) quenched from 800°C. Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 4. Subhedral grains of PtSb₂ (dark grey) are enclosed by former liquid (yellow) quenched from 1000°C. Carbon-coated. Plane polarized light.
Fig. 5. A large subhedral crystal of PtSb₂ (dark yellow) surrounded by former liquid (yellow) in the quenched product of run 466 from 1000°C. The matrix contains numerous dendritic crystals of PtSb₂ composition. Carbon-coated. Plane polarized light.
Fig. 6. Revised phase diagram of the Pt-Sb system. Starting compositions and temperatures of the runs are represented with dots. Temperatures of phase transformations shown in this diagram have been taken from the previously published data (Fig. 1 and Fig. 2).
Table 4. Electron microprobe analyses of the Pt-Sb phases.

<table>
<thead>
<tr>
<th>Phases</th>
<th>Composition (wt.%</th>
<th>Run no.</th>
<th>Temp.(°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Pt</td>
<td>Sb</td>
<td>total</td>
</tr>
<tr>
<td>Pt$<em>{34}$Sb$</em>{7}$</td>
<td>89.1</td>
<td>11.5</td>
<td>100.6</td>
</tr>
<tr>
<td></td>
<td>89.7</td>
<td>11.6</td>
<td>101.3</td>
</tr>
<tr>
<td>Pt$_{3}$Sb</td>
<td>83.1</td>
<td>17.3</td>
<td>100.4</td>
</tr>
<tr>
<td></td>
<td>83.3</td>
<td>17.1</td>
<td>100.4</td>
</tr>
<tr>
<td>Pt$<em>{3}$Sb$</em>{2}$</td>
<td>71.4</td>
<td>29.5</td>
<td>100.9</td>
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<td></td>
<td>70.7</td>
<td>29.5</td>
<td>100.2</td>
</tr>
<tr>
<td>PtSb</td>
<td>62.1</td>
<td>38.7</td>
<td>100.8</td>
</tr>
<tr>
<td></td>
<td>62.2</td>
<td>38.9</td>
<td>101.1</td>
</tr>
<tr>
<td></td>
<td>61.1</td>
<td>39.4</td>
<td>100.5</td>
</tr>
<tr>
<td>Pt$_{2}$Sb</td>
<td>45.1</td>
<td>56.0</td>
<td>101.1</td>
</tr>
<tr>
<td></td>
<td>44.8</td>
<td>55.6</td>
<td>100.4</td>
</tr>
</tbody>
</table>
only Pt, PtSb and PtSb\textsubscript{2} are stable at 800° and up to at least 1000°C. No compositional variation has been observed in these phases.

(ii) The position of the liquidus between PtSb\textsubscript{2} and Sb, up to 1000°C, is revised.

2. The Pt-Te System

A. Previous study: Roessler (1897) first prepared PtTe\textsubscript{2} by heating platinum with an excess of tellurium in an open furnace. He also reported the PtTe and Pt\textsubscript{2}Te phases obtained by melting the PtTe\textsubscript{2} phase on charcoal in an oxidizing flame with a blowpipe. Thomassen (1929) synthesized the PtTe\textsubscript{2} phase and determined its cell parameters. Grønvold et al. (1960) made a single phased PtTe and provided its X-ray powder diffraction data. The Pt\textsubscript{2}Te of Roessler (1897) was not found by Grønvold et al. (1960).

Phase relations of the Pt-Te system were first investigated by Gimpl et al. (1963a) and later by Bhan et al. (1969). Their phase diagrams are reproduced here in Fig. 7 and Fig. 8. Two main differences exist between them: (i) in Gimpl et al., there are only two intermediate phases, PtTe and PtTe\textsubscript{2} between the Pt and Te, but in Bhan et al., two more phases, Pt\textsubscript{3}Te\textsubscript{4} and Pt\textsubscript{2}Te\textsubscript{3} are present, and (ii) in Gimpl et al., there is no liquidus curve between the PtTe\textsubscript{2} and Te phases, whereas in Bhan et al., a liquidus
Fig. 7. Phase diagram of the Pt-Te system by Gimpl et al. (1963a).

Fig. 8. Phase diagram of the Pt-Te system by Bhan et al. (1969).
curve joining the melting points of the PtTe₂ phase and Te is present. According to the phase diagram of Bhan et al. (Fig. 8), the Pt₃Te₄ phase appears to have a compositional range of about 2 at.% and melts incongruently at 985°C to form PtTe₂ and liquid. The Pt₂Te₃ phase decomposes at 875°C to form Pt₃Te₄ and PtTe₂.

E. Present study: The present investigation was mainly concerned with the confirmation of the existence or absence of all the binary phases and of the previously reported liquidus curve between 66.7 and 100.0 at.% Te. Starting compositions for the runs and their reaction products are given in Table 5. At 650°C, run 81 yielded a reaction product of PtTe and Pt containing less than 0.03 at.% Te in solid solution. The Pt₃Te₄ and Pt₂Te₃ phases were confirmed in runs whose starting compositions lie in the region of 50 - 66.6 at.% Te (runs 83, 84 and 85). But the Pt₂Te phase reported by Roessler (1897) was not found.

At 800°C PtTe, Pt₃Te₄ and PtTe₂ are the stable binary phases. Run 88, with a starting composition of Pt₂Te₃, yielded Pt₃Te₄ and Pt₂Te₃ with very small amounts of other phases of different compositions, indicating that the product of run 88 was not equilibrated. Runs 94, 97, 98 and 99, with starting compositions between 57.0 and 66.0 at.% Te, invariably yielded Pt₃Te₄ and PtTe₂ but not Pt₂Te₃, indicating that the Pt₂Te₃ phase decomposes at a temperature below 800°C and much lower than the temperature 875°C given by Bhan et al. (1969). Run 94 is identical in starting
Table 5. Experimental results of the system Pt-Te.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Composition (at. %)</th>
<th>Temp. (°C)</th>
<th>Heating period (days)</th>
<th>Phase present</th>
<th>Composition (at. %)</th>
</tr>
</thead>
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<td></td>
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<td></td>
<td>1h 2h 3h 4h</td>
<td></td>
<td>Pt &amp; Te</td>
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<td>650</td>
<td>7 9 7 10</td>
<td>Pt</td>
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</tr>
<tr>
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<td>50.00 50.00</td>
<td>650</td>
<td>7 9 7 10</td>
<td>Pt &amp; Te</td>
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</tr>
<tr>
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<td>47.95 52.05</td>
<td>650</td>
<td>7 9 7 10</td>
<td>Pt &amp; Te &amp; Te4</td>
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</tr>
<tr>
<td>84</td>
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<td>650</td>
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<td>Pt &amp; Te &amp; Te4</td>
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</tr>
<tr>
<td>85</td>
<td>35.99 64.01</td>
<td>650</td>
<td>7 9 7 10</td>
<td>Pt &amp; Te &amp; Te4</td>
<td>39.9 60.1</td>
</tr>
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<td>800</td>
<td>18 17 15</td>
<td>Pt, Te, Te2</td>
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<td>42.87 57.13</td>
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<td>Pt &amp; Te &amp; Te4</td>
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<td>800</td>
<td>8 5</td>
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<td>89</td>
<td>33.33 66.67</td>
<td>1000</td>
<td>5 7</td>
<td>Pt &amp; Te</td>
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<td>66.49 33.51</td>
<td>1000</td>
<td>6</td>
<td>Pt</td>
<td>99.6 0.4</td>
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<tr>
<td>91</td>
<td>39.17 60.83</td>
<td>650</td>
<td>21 6</td>
<td>non-equilibrium</td>
<td></td>
</tr>
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<td>92</td>
<td>36.99 63.01</td>
<td>700</td>
<td>11 10 10</td>
<td>Pt &amp; Te &amp; Te4</td>
<td>43.1 56.9</td>
</tr>
<tr>
<td>93</td>
<td>44.49 55.01</td>
<td>1000</td>
<td>8</td>
<td>Pt &amp; Te &amp; Te4</td>
<td>33.6 66.4</td>
</tr>
<tr>
<td>94</td>
<td>37.99 62.01</td>
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<td>8 2 6</td>
<td>Pt &amp; Te &amp; Te4</td>
<td>43.0 57.0</td>
</tr>
<tr>
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<td>4 8 4</td>
<td>Pt</td>
<td>99.7 0.3</td>
</tr>
<tr>
<td>96</td>
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<td>4 8</td>
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<td>4 8</td>
<td>Pt &amp; Te &amp; Te4</td>
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<tr>
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</tr>
<tr>
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<td>4 8</td>
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<tr>
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<td>33.30 66.70</td>
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<td>2 20</td>
<td>Pt &amp; Te &amp; Te4</td>
<td>33.3 66.7</td>
</tr>
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<td>455</td>
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<td>1000</td>
<td>2 5</td>
<td>Pt &amp; Te</td>
<td>33.3 66.7</td>
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<td>1000</td>
<td>3 6 1</td>
<td>Pt &amp; Te &amp; Te4</td>
<td>42.7 57.3</td>
</tr>
<tr>
<td>465</td>
<td>9.99 90.01</td>
<td>1000</td>
<td>3 4</td>
<td>Pt &amp; Te</td>
<td>33.2 66.8</td>
</tr>
</tbody>
</table>

Abbreviations: L = liquid, 1h = initial heating, 2h = second heating, 3h = third heating, 4h = fourth heating.
composition with the mix used by Bhan et al. (1969), the reaction product of which was reported to contain 70% of Pt$_2$Te$_3$ and 30% of PtTe$_2$. Run 92, quenched from 700°C after heating for 31 days, gave a two-phase product consisting of Pt$_3$Te$_4$ and PtTe$_2$, indicating that the decomposition temperature of Pt$_2$Te$_3$ would be somewhere between 650°C and 700°C. Attempts to synthesize single-phase Pt$_2$Te$_3$ at 600°C (run 91) and 800°C (run 88) were unsuccessful.

The thermal stability of Pt$_3$Te$_4$ was another point of interest as run 93 (with a starting composition of Pt$_{45}$Te$_{55}$ and quenched from 1000°C) showed an equilibrium assemblage of Pt$_3$Te$_4$ and solidified liquid (Fig. 9). This result was supported by run 462 which, after repeated heating to 1000°C, yielded Pt$_3$Te$_4$ and PtTe$_2$ (Fig. 10). The melting point of the Pt$_3$Te$_4$ phase is, therefore, above 1000°C, higher than the melting point of 985°C reported by Bhan et al. (1969).

Solid solubility of Te in Pt was determined to be 0.3 - 0.4 at.% at 800 - 1000°C. The composition of the liquid coexisting with Pt at 1000°C is near 37 at.% Te (runs 455 and 90, Table 5), considerably different from that of approximately 31 at.% Te estimated by Gimpi et al. (1963a, Fig. 7) and Bhan et al. (1969, Fig. 8). Thus the position of the liquidus line between 0 at.% Te and 32 - 35 at.% Te in their phase diagrams (Figs. 7 and 8) must be displaced towards the Te-rich side by perhaps as much as 5 at.%.
Fig. 9. Rounded grains of Pt$_3$Te$_6$ (yellow) coexisting with former liquid (dark yellow and brown) in the run 93 quenched from 1000°C. The liquid separates into metastable phases (dark yellow and light brown). Black areas are pits and scratch marks. Carbon-coated. Plane polarized light.

Fig. 10. Fine grains of Pt$_3$Te$_6$ (yellow) and PtTe$_2$ (dark yellow) in the run 462 quenched from 1000°C. Black areas are pits. Carbon-coated. Plane polarized light.
Fig. 11. Rounded grains of platinum (light yellow) surrounded by former liquid (dark yellow) quenched from 1000°C (run 455). Black areas are pits and scratch marks. Carbon-coated. Plane polarized light.

Fig. 12. Large, rounded grains of PtTe₂ (pale brown) with pitted or unpitted polished surface existing in former liquid (pale green) quenched from 1000°C (run 458). Acicular crystals of PtTe₂ (orange yellow) present in matrix. Black areas are pits. Crossed nicols.
location of the eutectic point between Pt and PtTe also requires revision. Extrapolation of the revised liquidus curve, from the melting point (1768.4°C) of Pt through the composition of the liquid (about 37 at.% Te) coexisting with Pt at 1000°C to the solidus line between Pt and PtTe, gives a projected eutectic point approximately 40 at.% Te, 870°C (Fig. 14).

The region between PtTe₂ and Te was also of interest because of the uncertainty over the position of the liquidus line. Reaction products of runs with starting compositions between PtTe₂ and Te and quenched from 800°C and 1000°C consisted of PtTe₂ and a solidified liquid phase of nearly pure Te composition. Run 86 produced an assemblage of PtTe₂ and a quenched liquid phase of pure Te composition at 800°C, and run 458 also yielded PtTe₂ and liquid of 99.8 at.% Te at 1000°C (Fig. 12). Run 465, with a starting composition of Pt₁₀Te₉₀, which falls outside of the postulated liquidus line of Bhan et al. (1969), gave a reaction product consisting of needle-like PtTe₂ crystals and liquid of 99.8 at.% Te (Fig. 13). These data suggest that most part of the liquidus curve exists above 1000°C. Below 1000°C, the liquidus curve is close to 100 at.% Te line. The revised phase diagram of this system is shown in Fig. 14.

The salient feature of the phase relations of the Pt-Te system are:

(i) There are four Pt-Te binary phases, namely, PtTe₂, Pt₃Te₄, Pt₂Te₃, and PtTe₂. The compositions of these
Fig. 13. Crystals of PtTe$_2$ (creamy yellow) in former liquid of 99.8 at.% Te quenched from 1000°C (run 465). Note that some PtTe$_2$ crystals are pitted and others are unpitted on the polished surface. Black areas are pits. Plane polarized light.
Fig. 14. Revised phase diagram of the Pt-Te system. Starting compositions and temperatures of the runs are represented with dots. Temperatures of transformation in parentheses are estimated in this study, and others are taken from the data of Gimp et al. (1963b) and Bhan et al. (1969).
Table 6. Electron microprobe analyses of the Pt-Te phases.

<table>
<thead>
<tr>
<th>Phases</th>
<th>Composition (wt.%</th>
<th>Run no.</th>
<th>Temp. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Pt</td>
<td>Te</td>
<td>total</td>
</tr>
<tr>
<td>---------</td>
<td>-------</td>
<td>-------</td>
<td>-----------</td>
</tr>
<tr>
<td>PtTe</td>
<td>60.6</td>
<td>39.7</td>
<td>100.3</td>
</tr>
<tr>
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<tr>
<td></td>
<td>43.0</td>
<td>56.5</td>
<td>99.5</td>
</tr>
</tbody>
</table>
appear to be constant (Table 6).

(ii) The Te content in platinum and the Pt in tellurium in solid solution are negligible, less than 0.5 at.% up to 1000°C.

(iii) The composition of the liquid coexisting with Pt at 1000°C is 36.6 at.% Te. The eutectic point between Pt and PtTe is near 40 at.% Te at 870°C.

(iv) Stability of the Pt$_2$Te$_3$ phase above 700°C is questionable.

(v) The melting point of the Pt$_3$Te$_4$ phase is apparently above 1000°C.

(vi) The position of the liquidus between PtTe$_2$ and Te is revised as shown in Fig. 14.

3. The Pd-Sb System

A. Previous study: The phase relationships of this system were first investigated by Sander (1912) and later by Grigorjew (1932). There is substantial agreement between their results. The phase diagram presented by Hansen and Anderko (1958), on the basis of the two sets of data, is shown in Fig. 15. Four binary phases were reported, namely, Pd$_3$Sb, Pd$_5$Sb$_3$, PdSb, and PdSb$_2$. Of these, PdSb$_2$ had been identified earlier by Roessler (1895). Bäls and Schubert (1969) reported an additional phase, Pd$_2$Sb, which is stable at below 580°C and decomposes into Pd$_5$Sb$_2$ and Pd$_5$Sb$_3$ at about 580°C. El-Boragy et al. (1970) confirmed
Fig. 15. Phase diagram of the Pd-Sb system by Hansen and Anderko (1958). A compromise between those of Sander (1912, Ref. 1) and Grigorjew (1932, Ref. 2).

Fig. 16. Phase relations for the portion 23 - 30 at.% Sb of the Pd-Sb system (El-Boragy and Schubert, 1971a).
Fig. 17. Phase relations for the portion 25-29 at.%Sb of the Pd-Sb system (Wopersnow and Schubert, 1977).
the Pd$_5$Sb$_2$ phase and reported an additional, structurally related, non-stoichiometric Pd$_5$Sb$_2$- phase. El-Boragy and Schubert (1971a) re-investigated phase equilibria in the region between 24 and 30 at.% Sb (Fig. 16) and reported a new phase, Pd$_8$Sb$_3$. The same portion of the phase diagram was later revised by Wopersnow and Schubert (1977) as shown in Fig 17.

Desborough et al. (1973) reported that they were unable to synthesize the Pd$_3$Sb phase which was shown by El-Boragy and Schubert (1971a) to be stable over the temperature range 500 - 800°C. Cabri et al. (1975) first recognized that the Pd : Sb ratio in the Pd$_3$Sb phase is closer to 2.9 : 1 rather than 3 : 1. This was confirmed by Wopersnow and Schubert (1977), who revised the formula of this phase to Pd$_{20}$Sb$_7$, and at the same time revised the formula for the Pd$_5$Sb$_2$- phase of El-Boragy et al. (1970) to Pd$_{31}$Sb$_{12}$. According to Fig. 17, compositions of the Pd$_{20}$Sb$_7$, Pd$_8$Sb$_3$, Pd$_{31}$Sb$_{12}$, and Pd$_5$Sb$_2$ phases are 25.9 - 26.1, 27.2 - 27.5, 27.7 - 28.3, and 28.5 - 29.0 at.% Sb, respectively.

B. Present study: According to the phase diagram of El-Boragy and Schubert (1971a, Fig. 16) and Wopersnow and Schubert (1977, Fig. 17), phase relations in the region 20 - 30 at.% Sb appear to be complicated due to the presence of phases with similar compositions. The present investigation was thus primarily to verify the phases previously reported, particularly in the region 0 - 50
at.% Sb.

All the runs prepared and experimental results obtained from their reaction products are given in Table 7. Runs, with starting compositions in the range 0 - 25 at.% Sb and quenched from 510 - 1000°C (runs 31, 39, 43, 50, 52 and 266) invariably yielded two phases, Pd and Pd₂₀Sb₇. Solid solubility of Sb in Pd is 21.0 at.% Sb at 800°C and 16.5 at.% Sb at 1000°C. These data differ considerably from those previously reported: approximately 14 at.% Sb (Sander, 1912), 16 at.% Sb (Grigorjew, 1932), and 17 at.% Sb (Pratt et al., 1968) with no temperature dependence up to 1000°C. The composition of the Pd₂₀Sb₇ phase coexisting with Pd is constant within the analytical error, at 510 - 1000°C, ranging from 74.0 - 74.4 at.% Pd as shown in Table 7. It is clear that the Pd : Sb ratio is 2.88 - 2.85 : 1, rather than 3 : 1, supporting the correctness of the designation Pd₂₀Sb₇ for this phase. The Pd₈Sb₃, Pd₃₁Sb₁₂, and Pd₅Sb₂ phases were confirmed. The Pd₂Sb phase reported by Bälz and Schubert (1969) was also confirmed (Fig. 18). It is stable below 570°C and decomposes into Pd₅Sb₂ and Pd₅Sb₃ at between 570°C and 600°C, in agreement with the temperature of 580°C determined by Bälz and Schubert (1969). The Pd₅Sb₃ phase is confirmed to be stable at 600 - 800°C, and has a homogeneous range of less than 1 at.% at 700°C (Table 7).

Run 60-5, quenched from 700°C, gave an assemblage of PdSb and a liquid of 66.2 at.% Sb. Run 60-6, quenched from


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Abbreviation: L = liquid, 1h = initial heating, 2h = second heating, 3h = third heating.
Fig. 18. Grains of Pd$_2$Sb (yellow) intergrown with those of PdSb (brown) quenched from 570°C (run 56). The PdSb grains contain inclusions of Pd$_2$Sb. Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 19. Large grains of PdSb$_2$ (dark yellow) in former liquid (light yellow) quenched from 650°C (run 60-6). The matrix contains many dendritic crystals of PdSb$_2$ composition. Black areas are pits. Carbon-coated. Plane polarized light.
Fig. 20. Revised phase diagram of the Pd-Sb system. A compromise between those of Sander (1912), Grigorjev (1932), El-Boragy and Schubert (1971a), and Wopereanow and Schubert (1977) and the present study. Starting compositions and temperatures of the runs are represented with dots.
Table 8. Electron microprobe analyses of the Pd-Sb phases.

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650°C, consisted of large crystals of PdSb₂ and very small amounts of liquid containing minute dendrites of PdSb₂ (Fig. 19). Solid solubility of Pd in Sb at 510 - 520°C was negligible.

Revised phase relations are shown in Fig. 20. All the single phases present in this system were successfully synthesized and confirmed by X-ray diffraction. Electron microprobe analyses of the Pd-Sb phases are shown in Table 8.

The important features of this binary system are summarized as follows:

(i) All the binary phases previously reported are confirmed: Pd_{20}Sb₇, Pd₈Sb₃, Pd₃₅Sb₁₂, Pd₅Sb₂, Pd₂Sb, Pd₅Sb₃, PdSb, and PdSb₂.

(ii) The Pd₅Sb₃ phase is stable between 580°C and 845°C. Its compositional range, however, is narrower than that indicated by Hansen and Anderko (1958).

(iii) Sb content in Pd is as high as 16.5 at.% at 1000°C and 21.0 at.% at 800°C, whereas Pd in Sb is negligible at 510 - 520°C.

4. The Pd-Te System

A. Previous study: The phase PdTe was first prepared by Tibbals, Jr. in 1909 (Hansen and Anderko, 1958). Later, Thomassen (1929a) synthesized PdTe and PdTe₂ and determined their crystal symmetry. Grønvold and Røst (1956) synthesized Pd₄Te, Pd₃Te, Pd₅Te₂ and Pd₂Te by reacting pure
Pd or Te with PdTe at below 650°C. They reported the PdTe phase to have a very narrow range of homogeneity and did not find any extended mutual solubility between PdTe and PdTe₂, although a continuous transition from monotelluride to ditelluride of 3d transition elements were known.

Medvedeva et al. (1961) first investigated the phase relations in the region 20 - 100 at.% Te and confirmed the existence of all the six previously reported binary phases. According to their phase diagram (Fig. 21), the Pd₄Te₇, Pd₃Te, Pd₅Te₂ and Pd₂Te phases melt incongruently at about 1000°C, 820°C, 760°C and 590°C, respectively. The PdTe and PdTe₂ phases melt congruently at 720°C and 740°C, respectively. A liquidus curve exists between PdTe and PdTe₂ and below it there is a concave solidus curve. The field bounded by the liquidus and solidus curves is shown as a three-phase area (PdTe + PdTe₂ + liquid) which violates the Phase Rule as indicated by Elliott (1965). A continuous solid solution forms between PdTe (NiAs structure) and PdTe₂ [Cd(OH)₂ structure] at temperatures of 640 - 690°C, and breaks down into PdTe-rich and PdTe₂-rich solid solutions below 640°C. Guggenheim et al. (1961) supported the existence of the continuous solid solution between PdTe and PdTe₂ by their measurements of superconducting transition temperatures, but did not specify temperatures nor experimental procedures.

Kjekshus and Pearson (1965) re-investigated this problematic region using high temperature X-ray powder diffraction, density and magnetic susceptibility
Fig. 21. Phase diagram of the Pd–Te system (Medvedeva et al., 1961).
measurements. They found a continuous solid solution between PdTe and PdTe₂ above 660 to 670°C and restricted limits of homogeneity range for PdTe and PdTe₂ at lower temperatures (probably below 500°C), corroborating results of Medvedeva et al. (1961). Hoffman and MacLean (1976) reported that, between 575°C±10°C and 710°C±10°C, PdTe and PdTe₂ formed a complete solid solution which was unquenchable; below 570°C±10°C, PdTe and PdTe₂ occur as distinct phases. They also stated that the liquidus relations between PdTe and PdTe₂ were not clear but assumed that they might be similar to the feldspar liquidus relations between albite (NaAlSi₃O₈) and orthoclase (KAlSi₃O₈).

The problem in this phase diagram was further increased as new phases such as Pd₃Te₂ (El-Boragy and Schubert, 1971a), Pd₂₀Te₇ (Wopersnow and Schubert, 1977), Pd₉Te₄ (Matkovic and Schubert, 1978) were reported since Medvedeva et al. (1961) published their Pd–Te phase diagram. Cabri et al. (1979) pointed out that the Pd–Te phase diagram of Medvedeva et al. (1961) was unreliable and required major modification, as their unpublished detailed work in the partial system Pd–PdTe revealed many complications in the phase relations. Cabri (unpublished) found two new phases, Pd₉Te₂ and Pd₈Te₃.

B. Present study: In view of the above controversies the whole range of this system was re-investigated in this
study. The starting compositions of the runs and the experimental results are shown in Table 9. The same experimental results are also presented diagrammatically in Fig. 21. Unpublished DTA data provided by Dr. Cabri were incorporated in this study.

The Pd phases in reaction products, quenched from 620°C, 800°C and 1000°C, contain respectively 0.1 at.% Te (run 61), 14.8 at.% Te (runs 79-1 and 79-3) and 10.8 at.% Te (run 79-2) in solid solution. The solid phase coexisting with Pd at 620°C (run 61) and with Pd_{20}Te_{7} at 620 - 630°C (runs 62, 63, 64 and 65) has a composition ranging from Pd_{81.3}Te_{18.7} to Pd_{80.6}Te_{19.4} or simply Pd_{17}Te_{4}. This phase falls compositionally between the Pd_{4}Te phase of Gronvold and Rost (1956) and the Pd_{9}Te_{2} phase of Gabri. However, the X-ray powder pattern (Table 57) of Pd_{17}Te_{4} is not exactly the same as those of the Pd_{4}Te phase (Gronvold and Rost, 1956), nor the Pd_{9}Te_{2} phase (Gabri, unpublished). DTA data show that the melting point of the Pd_{17}Te_{4} is 760°C, significantly different from that of Pd_{4}Te (about 1000°C). At 760°C, Pd_{17}Te_{4} melts incongruently, forming Pd solid solution and liquid. The equilibrium assemblages of Pd solid solution + liquid at 800°C observed in runs 79-1 and 79-3 and at 1000°C observed in run 79-2 provide further support that the melting point of the Pd_{17}Te_{4} phase is lower than 800°C.

The Pd_{20}Te_{7} phase of Wopersnow and Schubert (1977), of composition 73.6 - 74.0 at.% Pd, is confirmed. The Pd_{3}Te phase of Gronvold and Rost (1956) is identical to Pd_{20}Te_{7} in
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Abbreviations: L = liquid, 1h = first heating, 2h = second heating, 3h = third heating.
Fig. 22. Revised Pd–Te phase diagram. DTA data (open circles) and temperatures in parentheses are from Cabri (unpublished). Bulk compositions and temperatures of runs are represented with dots. Possible liquidus curves between PdTe and PdTe$_2$ are represented by dotted lines. Dashed lines indicate uncertainty.
X-ray powder diffraction data. DTA data of Pd$_{20}$Te$_7$ shows two endothermic peaks, at 745°C and 885°C; the former is interpreted as a polymorphic transformation and the latter as melting of this phase. Four run products (78, 802, 822 and 837) of a single-phased Pd$_{20}$Te$_7$ exhibit, under the microscope, polysynthetic or lamellar twinning (Figs. 23 and 24).

The assemblages Pd solid solution + quenched liquid with 77.6 at.% Pd observed in run 79-2 (Fig. 25) and Pd$_{20}$Te$_7$ + quenched liquid with 75.9 at.% Pd, observed in run 823 (Fig. 26), both quenched from 800°C, indicate a liquid field in the region 77.6 - 75.9 at.% Pd at 800°C. The eutectic point between Pd$_{17}$Te$_6$ and Pd$_{20}$Te$_7$ is placed at 76.5 at.% Pd, 720°C, the temperature being determined by DTA.

Pd$_8$Te$_3$, the new phase found by Cabri (unpublished), is confirmed and known to have a composition of Pd$_{72.7}$Te$_{27.3}$. Although compositionally this phase falls in the solid solution range 74.7 - 72.0 at.% Pd proposed for Pd$_{20}$Te$_7$ by Wopersnow and Schubert (1977), the X-ray powder pattern of the Pd$_8$Te$_3$ phase (Table 58 in Chapter V) is apparently different from that of the Pd$_{20}$Te$_7$ phase and is indexed on the basis of orthorhombic symmetry. DTA data for this composition showed endothermic reactions, at 270°C, 680°C, before melting sets in at 900°C. The two former reactions are interpreted as polymorphic transitions. The X-ray powder pattern of Pd$_8$Te$_3$ quenched from 800°C (run 841), however, is identical to that of Pd$_8$Te$_3$ quenched from 410°C.
Fig. 23. The Pd$_{20}$Te$_{7}$ phase quenched from 600°C (run 802). Polysynthetic twinning developing along the NE direction (lower left, higher relief) overlaps the cross-hatched pattern. Black areas are pits, former gas bubbles, and scratch marks. Plane polarized light.

Fig. 24. Lamellar twinning in Pd$_{20}$Te$_{7}$ quenched from 800°C (run 822). Black areas are pits and fractures. Crossed nicols.
Fig. 25. Rounded grains of \( \text{Pd}\) solid solution (pink) enclosed by former liquid (pinkish grey) quenched from \(800^\circ\)C (run 79-2). Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 26. \(\text{Pd}_{20}\text{Te}_7\) grains (green) coexisting with former liquid (pale green) quenched from \(800^\circ\)C (run 823). Black areas are pits. Crossed nicols.
(run 67), 600° (run 823) and 640° (run 843), indicating that the structure of the high form (above 680°C) is not always quenchable. Lamellar twinning, most likely a product of the phase transformations, is very common in the product of run 841 (Fig. 27).

A new phase was encountered between \( \text{Pd}_8\text{Te}_3 \) and \( \text{Pd}_9\text{Te}_4 \). It has a composition from 70.0 - 71.0 at.% Pd and therefore is referred to as \( \text{Pd}_7\text{Te}_3 \). The \( \text{Pd}_7\text{Te}_3 \) phase seems to melt at about 830°C and showed two phase transitions, at 470°C and 595°C, as evidenced by DTA data. The high form (above 595°C) is not quenchable and the lamellar twinning observed in the \( \text{Pd}_7\text{Te}_3 \) phase quenched from above 600°C (runs 808, 844 and 848) is most likely due to the phase transformations. X-ray powder data of the \( \text{Pd}_7\text{Te}_3 \) phase, quenched from 600°C, were indexed based on monoclinic symmetry, and its cell parameters were determined (Table 59 in Chapter V). The \( \text{Pd}_7\text{Te}_3 \) phase coexists with liquid at 800°C (run 844) and 700°C (run 846, Fig. 28). Runs with starting compositions between \( \text{Pd}_8\text{Te}_3 \) and \( \text{Pd}_7\text{Te}_3 \), quenched from 410 - 800°C (runs 840, 843, 839, 838, 803, 814, 67 and 68), yielded lamellar intergrowths of the two phases (Fig. 29).

The \( \text{Pd}_9\text{Te}_4 \) phase reported by Matkovic and Schubert (1978) is confirmed, but the phase with \( \text{Pd}_2\text{Te} \) composition was not found. From the X-ray diffraction data there is little doubt that the \( \text{Pd}_2\text{Te} \) phase of Gronvold and Rost (1956) is the same phase as \( \text{Pd}_9\text{Te}_4 \). According to the DTA data, \( \text{Pd}_9\text{Te}_4 \) has a polymorphic transformation at 460°C and melts at 605°C to form \( \text{Pd}_7\text{Te}_3 \) and liquid (Fig. 30).
Fig. 27. $\text{Pd}_9\text{Te}_3$ showing a polysynthetic (lower half) and lamellar twinning (upper half) in run 841 quenched from $800^\circ\text{C}$. Black areas are pits and former gas bubbles. Plane polarized light.

Fig. 28. Round to irregular grains of $\text{Ni-PtTe}_2$ (dark brown), a new phase, existing in extract liquid (yellow) quenched from $700^\circ\text{C}$ (run 846). Lamellar twinning observed under higher magnification. Carbon-coated. Plane polarized light.
Fig. 29. Photomicrograph showing intergrowths of Pd₈Te₃ (pale green) and Pd₇Te₃ (yellow) lamellae (run 814). Black areas are former gas bubbles. Plane polarized light.

Fig. 30. Large rounded grains of Pd₉Te₄ (pinkish grey) coexisting with former liquid (yellowish pink) in run 805. Lamellar twinning in the Pd₉Te₄ phase is not visible in this photomicrograph. Black areas are pits. Carbon-coated. Plane polarized light.
Lamellar twinning was also observed for the Pd₉Te₄ phase quenched from 510°C (run 815), apparently a result of the phase transformation.

The Pd₃Te₂ phase, first synthesized at 430°C by El-Boragy and Schubert (1971a), was confirmed by X-ray diffraction and microprobe analysis. The Pd₃Te₂ phase decomposes into Pd₉Te₄ and PdTe at 500°C according to DTA data. The eutectic point between Pd₉Te₄ and PdTe is placed at 39.0 at.% Te, 500°C as run products (824 and 826), quenched from 500°C, show myrmekitic intergrowths of the Pd₉Te₄ and PdTe phases (Fig. 31). The Pd₃Te₂ phase coexists with Pd₉Te₄ and with PdTe in the region 40 – 50 at.% Te, up to 500°C.

The PdTe phase has a stoichiometric composition below 450°C (runs 807 and 834), but becomes progressively depleted in Pd as temperature increases. It has 50.4 at.% Te at 500°C (runs 824 and 826), 51.0 at.% Te at 600°C (runs 77 and 806) and 51.8 at.% Te at 700°C (runs 810 and 813, Fig. 32).

The PdTe₂ phase does not take Te in solid solution at 600 – 710°C, but the phase relations in the region between PdTe and PdTe₂ remain unclear. The phase diagram of this region presented by Medvedeva et al. (1961) could neither be confirmed nor denied on the basis of the present experimental data. Runs 827, 828 and 829 quenched from 710°C all yielded PdTe and PdTe₂ intergrowths, similar in texture to perthite. Although these runs all fell in the region between the liquidus and solidus of Medvedeva et al.
Fig. 31. A eutectic texture showing myrmekitic intergrowths of Pd₄Te₄ (yellow) and PdTe (brown) quenched from 500°C (run 826). Carbon-coated. Plane polarized light.

Fig. 32. Large grain of PdTe (yellowish-pink) coexisting with former liquid in run 813 quenched from 700°C. The matrix contains numerous dendritic grains. Black areas are pits and scratch marks. Plane polarized light.
(1961), the presence of a liquid phase could not be verified due to small grain size. Run 821, 57.5 at.% Te in bulk composition and quenched from 60°C, yielded small patches of PdTe$_2$ solid solution of 65.0 at.% Te in a matrix of fine lamellar intergrowths of PdTe$_2$ and PdTe$_2$ (Figs. 33 and 34) which were probably also solid solutions but could not be confirmed by electron microprobe analysis. Run 818, with an identical bulk composition as run 821 but quenched from 670°C, resulted in coarser lamellae of PdTe$_2$ solid solution of 62.5 at.% Te in a matrix similar to that of run 821 (Fig. 35). Thus, the complete solid solution between PdTe and PdTe$_2$ at 640°C and 690°C (Medvedeva et al., 1961; Guggenheim et al., 1961; Kjekshus and Pearson, 1965) and at 575°C ± 10°C - 710°C ± 10°C (Hoffman and MacLean, 1976) was not verified.

The textures observed from the quenched phases rather suggest two episodes of exsolution taking place on quenching: patches of PdTe$_2$ first exsolved from the PdTe-PdTe$_2$ solid solution and subsequently, at lower temperatures, fine lamellae of PdTe$_2$ exsolved from the remaining matrix. A previously published exsolution curve (Medvedeva et al., 1961) has not explained the existence of the second phase of exsolution.

The shape of the liquidus curve between PdTe and PdTe$_2$ is perhaps similar to those between AgF and AgF-ZnF$_2$ in the AgF - ZnF$_2$ system (DeVries and Rustum, 1953) on the basis of a difference in the crystal structures between the PdTe and PdTe$_2$ phases. Regarding the critical temperature of the
Fig. 33. Exsolution of small patches of PdTe$_2$ solid solution (grayish pink) in the matrix of fine intergrowths of PdTe and PdTe$_2$ lamellae (grayish brown) in run 821. Black areas are pits, fractures, and former gas bubbles. Plane polarized light.

Fig. 34. Same as Figure 33, but greater magnification showing detail of exsolution lamellae of PdTe solid solution (greenish brown) and PdTe$_2$ solid solution (brownish yellow). Black areas are former gas bubbles. Plane polarized light.
Fig. 35. Coarser lamellae of $\text{PdTe}_2$ (light green) in the matrix of fine intergrowth of $\text{PdTe}$ (dark green) and $\text{PdTe}_2$ (light green) lamellae quenched from $670^\circ\text{C}$ (run 818). Crossed nicols.

Fig. 36. Grains of $\text{PdTe}_2$ (dark yellow) coexisting with former liquid (pale green) quenched from $710^\circ\text{C}$ (run 832). The matrix contains numerous blades of $\text{PdTe}_2$ composition. Black areas are pits. Carbon-coated. Plane polarized light.
exsolution curve, preference is given to 575°±10°C (Hoffman and MacLean, 1976) rather than 640°C (Medvedeva et al., 1961) on the basis of the texture observed in the run 821 quenched from 600°C.

Six runs with bulk compositions between PdTe₂ and Te were prepared and heated at 600 - 710°C. Electron microprobe analyses and textural relations showed that quenched products of runs 809, 832 and 835 consisted of PdTe₂ in equilibrium with a liquid in which a second generation of PdTe₂ was produced during quenching (Fig. 36), causing a shift of the composition of the final liquid to 99.2 - 99.4 at.% Te. Runs 836, 842 and 847, falling outside of and heated to temperatures above the liquidus of Medvedeva et al. (1961), yielded products of a liquid with dendritic solids of compositions from 65.7 to 75.0 at.% Te, apparently metastable phases crystallized during quenching (Fig. 37). The data, although sparse, indicate that the phase diagram in the region between PdTe₂ and Te proposed by Medvedeva et al. (1961) is essentially correct.

Based on the present study, the phase relations in the Pd - PdTe portion of Medvedeva et al. (1961) have been revised as shown in Fig. 22. Further work is required in the region between PdTe and PdTe₂ to clarify the liquidus relations, solid solution and exsolution problems, because PdTe (kotulskite) and PdTe₂ (merenskyite) are among the more common platinum-group minerals. Microprobe analyses of the Pd-Te phases are shown in Table 10.

The important features of this phase diagram are
Fig. 37. Dendritic or flake-like crystals (yellow) of variable composition in former liquid (pale green), quenched from 700°C (run 847). Plane polarized light.
summarized as follows:

(i) There are eight Pd-Te phases: Pd$_{17}$Te$_4$, Pd$_{20}$Te$_7$, Pd$_8$Te$_3$, Pd$_7$Te$_3$, Pd$_9$Te$_4$, Pd$_3$Te$_2$, PdTe and PdTe$_2$. Of these, Pd$_7$Te$_3$ is the new phase established in this study.

(ii) The Pd$_{17}$Te$_4$ phase melts incongruently at 760°C.

(iii) The Pd$_{20}$Te$_7$ phase has a polymorphic transformation at 745°C before it melts incongruently at 885°C.

(iv) Cabri's (unpublished) new phase Pd$_8$Te$_3$ is confirmed as stoichiometric (72.7 at.% Pd). It has two polymorphic transformations, at 270°C and 680°C before it melts at 900°C.

(v) The composition of the new phase Pd$_7$Te$_3$ ranges from 70.0 to 71.0 at.% Pd. It has two polymorphic transformations, at 470°C and 595°C, and melts probably incongruently at about 830°C.

(vi) The Pd$_9$Te$_4$ phase, corresponding to the Pd$_2$Te phase of Gronvold and Rost (1956), has a polymorphic transformation at 460°C and melts incongruently at 605°C.

(vii) The Pd$_3$Te$_2$ phase is confirmed. It decomposes at 500°C to form high Pd$_9$Te$_4$ and PdTe.

(viii) The PdTe phase becomes Pd-poor from 450°C to its melting point, 720°C (depletion of Pd up to 2 at.%).

(ix) A eutectic point between the Pd$_{17}$Te$_4$ and Pd$_{20}$Te$_7$ phases is placed at 23.5 at.% Te and 720°C. Another eutectic point between Pd$_9$Te$_4$ and PdTe is located near 39.0 at.% Te and 500°C.

(x) Te content in Pd at 600°C is negligible (0.1 at.%),
Table 10. Electron microprobe analyses of the Pd-Te phases.

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<td>44.4</td>
<td>55.6</td>
<td>100.0</td>
</tr>
<tr>
<td></td>
<td>43.7</td>
<td>56.6</td>
<td>100.3</td>
</tr>
<tr>
<td>PdTe$_2$</td>
<td>29.5</td>
<td>70.6</td>
<td>100.1</td>
</tr>
<tr>
<td></td>
<td>29.3</td>
<td>70.9</td>
<td>100.2</td>
</tr>
<tr>
<td></td>
<td>29.6</td>
<td>70.7</td>
<td>100.3</td>
</tr>
</tbody>
</table>
but it abruptly increases to 14.8 at.% at 800°C and then decreases to 10.8 at.% at 1000°C.

5. The Pd-Pt System

A. Previous study: It has been generally believed that Pt and Pd form a continuous solid solution at temperatures below the solidus curve, based on the measurements of electrical conductivity, temperature coefficient of electrical resistance and thermoelectric force (Geibel, 1911), thermal conductivity (Schultze, 1911) and magnetic susceptibility (Vogt, 1932). However, as miscibility gaps were observed in some binary systems of the platinum group elements, Pd-Ir (Raub and Plate, 1957), Pd-Rh (Raub, 1959) and Pt-Ir (Raub and Plate, 1956), Raub (1959) suggested that a similar miscibility gap may exist between Pt and Pd. He estimated the critical temperature of the gap to be about 780°C, deduced from possible relationship found between the difference in the melting points of the pure elements of the Pd-Rh, Pd-Ir, and Pt-Ir systems and the critical temperatures of the miscibility gaps determined experimentally. He stated that thermodynamic equilibrium in these binary systems was reached by solid state diffusion and therefore must be very slow. He cited the example that in the Pt-Ir system, true equilibrium was not reached even after annealing at 1200°C for more than one year.

Thus he suggested that alloys of the platinum group elements should be made from the melt. A hypothetical phase diagram
Fig. 38. Phase diagram of the Pt-Pd system in Moffatt (1979).
of the Pt-Pd system was given in Moffatt (1979), and is shown in Fig. 38.

**B. Present study**: The preparation of homogeneous alloys of Pt and Pd by diffusion was attempted on eight mixes sealed in silica capsules and annealed at 1000°C up to 6 months. None of the reaction products was found to have attained equilibrium, and some contained unreacted reagents. Consequently, a high temperature graphite furnace was used to prepare the alloys from the melt. Three mixes (runs 180, 181 and 182) were fused in alumina crucibles at 1800°C under vacuum for a few minutes, and then cooled to room temperature in three hours. Melting of the mixes was confirmed under the binocular microscope. The inner walls of the crucibles were found to be coated with a thin greyish film of presumably Pd, Pt or both. All run products consisted of a solid phase and a former liquid phase in different proportions, depending on the bulk composition. The solid phase formed round, creamy white grains which are enclosed by a former liquid of grey colour (Figs. 39 and 40).

Electron microprobe analyses of the solid phases (Table 11) shows that they are compositionally homogeneous and the total Pt and Pd is less than 100 wt.%. The liquid phase is variable in composition and the total Pt and Pd is also smaller than 100 wt.%. Small pieces of products of runs 180, 181 and 182 were
Fig. 39. Grains of solid phase (creamy yellow) in former liquid (greenish grey) quenched from 800°C (run 183). Plane polarized light.

Fig. 40. Rounded grains of solid phase (creamy yellow) and interstitial former liquid (greenish grey) in run 188. Carbon-coated. Plane polarized light.
<table>
<thead>
<tr>
<th>Run no.</th>
<th>Composition(at.%): Pd 79.08 20.92</th>
<th>Thermal history: a, b</th>
<th>Phases: S</th>
<th>Composition(wt.%): Pd 40.6 S 56.1 total 96.7</th>
</tr>
</thead>
<tbody>
<tr>
<td>183</td>
<td>&quot; &quot; &quot; a, b</td>
<td>S</td>
<td></td>
<td>40.5 55.5 96.0</td>
</tr>
<tr>
<td>184</td>
<td>&quot; &quot; &quot; a, c</td>
<td>S</td>
<td></td>
<td>40.0 55.8 95.8</td>
</tr>
<tr>
<td>181</td>
<td>50.10 49.90  a</td>
<td>S</td>
<td></td>
<td>23.9 74.6 98.5</td>
</tr>
<tr>
<td>185</td>
<td>&quot; &quot; &quot; a, b</td>
<td>S</td>
<td></td>
<td>24.0 74.7 98.7</td>
</tr>
<tr>
<td>182</td>
<td>19.82 80.18  a</td>
<td>S</td>
<td></td>
<td>6.5 92.7 99.3</td>
</tr>
<tr>
<td>187</td>
<td>&quot; &quot; &quot; a, b</td>
<td>S</td>
<td></td>
<td>6.2 92.2 98.4</td>
</tr>
<tr>
<td>188</td>
<td>&quot; &quot; &quot; a, c</td>
<td>S</td>
<td></td>
<td>6.7 92.0 98.7</td>
</tr>
</tbody>
</table>

* a: annealed at 1800°C.
b: reheated to 800°C and quenched.
c: reheated to 1000°C and quenched.

** S: solid.
L: liquid.
reheated in evacuated, sealed silica capsules at 800°C and 1000°C for one to two weeks and then quenched in ice water. The process significantly coarsened the grain size, but did not affect the composition of the solid phase (Table 11).

Electron microprobe energy dispersive spectroscopy indicated minor amounts of Fe and Al in the solid phase and only Si in the liquid phase from run 183. Subsequent re-analyses of the samples for these minor elements gave 3.6 wt.% Fe and 0.6 wt.% Al in the solid phase and less than 1 wt.% Si in the liquid phase which is not sufficient to account for the 4.6 wt.% deficiency in that phase.

According to the experimental data for the Pt–C and Pd–C systems (Hansen and Andersen, 1958), up to 1.5 wt.% of carbon may be dissolved in the melt of Pt but not in solid Pt, and at least 2.45 wt.% of carbon in the melt of Pd. It is therefore reasonable to assume that carbon, presumably from the graphite furnace, makes up the remainder of the analyses. The Fe, Al and Si contaminants were probably derived from the furnace and crucibles.

The experimental results obtained in this study are inconclusive in confirming the presence or absence of a miscibility gap between Pt and Pd, mainly due to the contamination of the Pt–Pd alloys which seems to be unavoidable when working at such a high temperature (near the melting point of Pt and Pd) by using silica capsules, alumina crucibles and a graphite furnace. Other crucibles, such as tungsten or molybdenum, are for the same reason also considered to be unsuitable.
6. The Sb-Te System

A. Previous study: Fay and Ashley (1902) first investigated the phase relations of this system. They found a eutectic point between Sb$_2$Te$_3$ and Te at 89 at.% Te, 424°C. Hansen and Anderko (1958) constructed a phase diagram (Fig. 41) based on all the data reported before 1951. Abrikosov et al. (1959) re-investigated the partial system between Sb and Sb$_2$Te$_3$ and found two intermediate phases, β and γ, whose homogeneity range covers 17.3 - 36.9 at. % Te and 40.9 - 53.8 at.% Te, respectively (Fig. 42). They were unable to synthesize single-phased Sb$_2$Te$_3$ with a stoichiometric composition. The solid solubility of Te in Sb was estimated to be about 1 at.% Te at 500°C.

Brown and Lewis (1962) investigated the region between Sb and Sb$_2$Te$_3$, making alloys at near the solidus temperature and agitating the surface containing the capsules to ensure thorough reaction between reagents. They compiled a phase diagram (Fig. 43) in which liquidus curves were taken from Hansen and Anderko (1958) and solidus lines and phase boundaries were determined from X-ray diffraction data. They reported that X-ray diffraction lines of alloys of compositions between 11 and 60 at.% Te could be indexed based on the Sb$_2$Te$_3$ cell, except for a few X-ray diffraction lines of alloys with less than 40 at.% Te, and found progressive changes in cell dimensions with composition.
Fig. 41. Phase diagram of the Sb-Te system by Hansen and Anderko (1958) compromising experimental data published prior to 1958.

Fig. 42. Phase diagram of the Sb-Te system by Abrikosov et al. (1959).
Fig. 43. Phase diagram of the Sb-Te system (Brown and Lewis, 1962).

Fig. 44. Phase diagram of the Sb-Te system showing a compositional variation of the Sb\textsubscript{2}Te\textsubscript{3} phase (Poretskaya et al., 1963).
Thus, they concluded that a single phase (referred to as $\delta$) existed with a homogeneity range from 11 to 60 at.% Te. They suggested that the $\beta$ and $\gamma$ phases of Abrikosov et al. (1959) could be products of non-equilibrium.

Poretskaya et al. (1963) examined the compositional variation of the $\text{Sb}_2\text{Te}_3$ phase with temperature. As shown in Fig. 44, they found the $\text{Sb}_2\text{Te}_3$ phase to be stoichiometric when quenched from temperatures close to its melting point, 621 - 622$^\circ$C, but Te-poor when quenched from lower temperatures, 59.4 at.% Te at 500$^\circ$C and 59.2 at.% Te at 400$^\circ$C.

B. Present study: As discrepancies in the phase relations in the Sb-Te system were mainly in the region between Sb and $\text{Sb}_2\text{Te}_3$, the present study is therefore concentrated in this region. Bulk compositions of the experimental runs and their reaction products are shown in Table 12. Runs (2, 3, 8, 11 and 12), with bulk compositions in the region 3 - 10 at.% Te and quenched from 420 - 600$^\circ$C, invariably yielded products consisting of Sb and another phase, tentatively referred to as X because of its questionable identity (Fig. 45). The solid solubility of Te in Sb was not significant (less than 1 at.% up to 600$^\circ$C), confirming the results of Abrikosov et al. (1959). The composition limit of the X-phase on the Sb side was 12 - 13 at.% Te, and that on the Te side was not known due to failure in obtaining equilibrium assemblages in the region.
Table 12. Experimental data of the system Sb-Te.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Composition (at.%)</th>
<th>Temp. (°C)</th>
<th>Heating period (days)</th>
<th>Phases present</th>
<th>Composition (at.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sb</td>
<td>Te</td>
<td>1h</td>
<td>2h</td>
<td>3h</td>
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<tr>
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<td>79.92</td>
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<tr>
<td>2</td>
<td>90.00</td>
<td>10.00</td>
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<td>62</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>94.82</td>
<td>5.18</td>
<td>21</td>
<td>42</td>
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<tr>
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<td>40.25</td>
<td>11</td>
<td>28</td>
<td>30</td>
</tr>
<tr>
<td>5</td>
<td>55.08</td>
<td>44.92</td>
<td>12</td>
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<td>30</td>
</tr>
<tr>
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<td>43.14</td>
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</tr>
<tr>
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<td>80.16</td>
<td>16</td>
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<td>14</td>
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</tr>
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<td>83.00</td>
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<td>21</td>
<td>5.99</td>
<td>94.91</td>
<td>15</td>
<td>7</td>
<td></td>
</tr>
</tbody>
</table>

Abbreviations: L = liquid, X = phase unknown, 1h = first heating, 2h = second heating, 3h = third heating, Sb₂Te₃* = expected to exist but not confirmed.
Fig. 45. Antimony grains (creamy yellow) coexisting with X-phase (pale brown) of 11-12 at.% Sb in composition quenched from 500°C (run 12). Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 46. Crystals of Sb₂Te₃ (yellowish brown) exsolved from former liquid when quenched from 800°C (run 16). Black areas are pits. Carbon-coated. Crossed nicols.
20 - 60 at.% Te. These run products (1, 4, 5, 6, 8 and 10) appeared to be composed of a single phase under a reflecting microscope, and their X-ray powder films showed systematic gradual shifts of X-ray lines with bulk composition of the runs, suggestive of a solid solution series. However, electron microprobe analyses revealed wide variations in composition in every run, leaving no doubt of their non-equilibrium nature.

The liquidus curve in the region between Sb and Sb$_2$Te$_3$, determined by DTA (Table 13), was essentially in agreement with that of Hansen and Anderko (1958) and Abrikosov et al. (1959), with only minor differences in temperatures of thermal reactions. It is also consistent with the results of runs 18 and 19. Contrary to the results of Abrikosov et al. (1959) and Poretskaya et al. (1963), single-phased Sb$_2$Te$_3$ was successfully synthesized at 500°C from a stoichiometric mix (run 14). The composition range of the Sb$_2$Te$_3$ phase was 60.3 at.% Te at 430°C (run 7) and 58.7 - 60.0 at.% Te at 600°C (runs 19 and 20).

In the region between Sb$_2$Te$_3$ and Te, the experimental results of run 16 (Fig. 46) and run 17 could all be explained in terms of previously published phase diagrams, e.g., Brown and Lewis (1962). The Te-rich liquid presumably could not be readily quenched, resulting in non-equilibrium assemblages in runs 7, 16 and 17. In these runs, solids presumably crystallized during quenching, causing the liquid to shift composition until reaching the eutectic point at 89 at.% Te. Thus, this portion of the
Table 13. Differential thermal analysis data.

<table>
<thead>
<tr>
<th>Run no.</th>
<th>Composition (at.%)</th>
<th>Peaks</th>
</tr>
</thead>
<tbody>
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<td></td>
<td>Sb</td>
<td>Te</td>
</tr>
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<td>94.82</td>
<td>5.18</td>
</tr>
<tr>
<td></td>
<td></td>
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</tr>
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<td>2</td>
<td>85.09</td>
<td>14.91</td>
</tr>
<tr>
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</tr>
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<td>79.92</td>
<td>20.08</td>
</tr>
<tr>
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<td>74.97</td>
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<td>5</td>
<td>55.08</td>
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<td>9</td>
<td>50.10</td>
<td>49.90</td>
</tr>
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</tr>
<tr>
<td>6</td>
<td>43.14</td>
<td>56.86</td>
</tr>
<tr>
<td></td>
<td></td>
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</tbody>
</table>

Abbreviations: endo. = endothermal, exo. = exothermal.
Fig. 47. Revised phase diagram of the Sb-Te system. Temperatures of transformation in parentheses were determined in this study.
phase diagram of Brown and Lewis (1962) is adopted without modifications in the revised phase diagram (Fig. 47).

The important experimental data from the present investigation are: 

(i) Sb coexists with an X-phase of 12 - 13 at.% Te in composition below 549°C. The phase relations between the X-phase and the Sb₂Te₃ phase below 510°C are uncertain.

(ii) The Sb₂Te₃ phase has a narrow solid solution range of about 1.3 at.% at 600°C. A single-phased Sb₂Te₃ was successfully synthesized at 500°C from a stoichiometric mix.
CHAPTER IV

PHASE RELATIONS OF THE TERNARY SYSTEMS

Ternary phase diagrams constructed in this study are presented in order of decreasing temperatures, if the systems were investigated at more than one temperatures.

1. Phase Relations of the Pt-Sb-Te System

A. Previous study: An isothermal section at 650°C was constructed by El-Boragy and Schubert (1971b, Fig. 48) and is apparently the only one in the literature.

B. Present study: Phase relationships of three isothermal sections were constructed. Known minerals in the Pt-Sb-Te system are platinum [Pt], geversite [PtSb₂], stumplflite [PtSb], moncheite [PtTe₂], and tellurantimony [Sb₂Te₃].

1) Isothermal section at 1000°C

Starting compositions of the runs prepared and their reaction products are shown in Table 14. The same experimental results are also presented diagrammatically in Fig. 49 and Fig. 50. As shown in Figs. 49 and 50, five solid phases are stable at this temperature: Pt, PtSb, PtSb₂, PtTe₂, and Pt₃Te₄. In addition, two different liquid fields exist: (i) a large liquid field extending from the Pt-Sb join to the Pt-Te binary join and (ii) a liquid field restricted to near the Sb-Te binary join. To distinguish these two liquids in this text, the former with
Fig. 48. Isothermal section of the system Pt-Sb-Te at 650°C (El-Boragy and Schubert, 1971b).
<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Composition (at%)</th>
<th>Heating Period (days)</th>
<th>Phases present</th>
<th>Composition (at%)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Pt  Sb  Te</td>
<td>1h  2h</td>
<td></td>
<td>Pt  Sb  Te</td>
<td></td>
</tr>
<tr>
<td>401</td>
<td>9.96 59.92 30.12</td>
<td>7  11</td>
<td>Gv</td>
<td>33.3  64.2  2.5</td>
<td>Pt_{1.00} Sb_{1.93} Te_{0.07}</td>
</tr>
<tr>
<td>402</td>
<td>10.00 40.01 49.99</td>
<td>7  11</td>
<td>Gv</td>
<td>33.2  58.6  8.2</td>
<td>Pt_{1.00} Sb_{1.76} Te_{0.24}</td>
</tr>
<tr>
<td>403</td>
<td>20.00 69.95 10.05</td>
<td>7  11</td>
<td>Gv</td>
<td>33.6  65.4  1.0</td>
<td>Pt_{1.01} Sb_{1.96} Te_{0.03}</td>
</tr>
<tr>
<td>404</td>
<td>20.00 10.00 70.00</td>
<td>7  11</td>
<td>Me</td>
<td>32.2  7.5  60.3</td>
<td>Pt_{0.97} Te_{1.81} Sb_{0.22}</td>
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<tr>
<td>405</td>
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<td>7  11</td>
<td>Gv</td>
<td>33.7  54.8  11.5</td>
<td>Pt_{1.01} Sb_{1.64} Te_{0.35}</td>
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<tr>
<td>406</td>
<td>30.00 40.03 29.96</td>
<td>7  11</td>
<td>Gv</td>
<td>33.3  48.4  18.3</td>
<td>Pt_{1.00} Sb_{1.45} Te_{0.55}</td>
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<tr>
<td>407</td>
<td>30.00 20.01 49.99</td>
<td>18 12</td>
<td>Gv</td>
<td>33.2  15.7  51.1</td>
<td>Pt_{1.00} Te_{1.53} Sb_{0.47}</td>
</tr>
<tr>
<td>408</td>
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<td>19 12</td>
<td>Me</td>
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<td>Pt_{0.99} Te_{1.71} Sb_{0.30}</td>
</tr>
<tr>
<td>409</td>
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<td>18 12</td>
<td>Gv</td>
<td>33.6  50.2  16.6</td>
<td>Pt_{1.00} Sb_{1.50} Te_{0.50}</td>
</tr>
<tr>
<td>410</td>
<td>42.86 5.01 52.13</td>
<td>18 12</td>
<td>Gv</td>
<td>33.7  34.6  15.4</td>
<td>Pt_{1.00} Te_{1.35} Sb_{0.66}</td>
</tr>
<tr>
<td>411</td>
<td>48.99 42.99 8.02</td>
<td>16 12</td>
<td>St</td>
<td>33.4  32.5  14.1</td>
<td>Pt_{1.00} Sb_{1.58} Te_{0.42}</td>
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<tr>
<td>412</td>
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<td>16 12</td>
<td>Me</td>
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</tr>
<tr>
<td>413</td>
<td>49.98 7.52 42.50</td>
<td>7</td>
<td>L</td>
<td>33.8  30.0  51.2</td>
<td>Pt_{1.00} Sb_{1.50} Te_{0.50}</td>
</tr>
<tr>
<td>414</td>
<td>59.92 28.97 10.01</td>
<td>16 12</td>
<td>L</td>
<td>33.4  30.0  51.2</td>
<td>Pt_{1.00} Sb_{1.50} Te_{0.50}</td>
</tr>
<tr>
<td>415</td>
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<td>16 12</td>
<td>Gv</td>
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</tr>
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<td>7  9</td>
<td>Gv</td>
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<td>Gv</td>
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Abbreviations: 1h = first heating, 2h = second heating, L = liquid, Gv = geversite, Mc = mancheite, St = stumplite.
Fig. 49. Phase relations in the Pt-rich portion of the Pt-Sb-Te system at 1000°C. Solid circles and numerals represent bulk compositions and run numbers.
Fig. 50. Phase relations in the Pt-poor portion of the Pt-Sb-Te system at 1000°C. Solid circles and numerals represent bulk compositions and run numbers.
a large areal extent is referred to as liquid(1) and the latter, confined to near the Sb-Te join, is referred to as liquid(2). The exact boundaries of the two liquid fields were often impossible to determine because of the separation of the liquid phase into metastable phases during quenching. In such cases, the boundary points of the liquid(1) field were approximated by using the compositions of the metastable phases averaged over a large number of grains. These points are indicated in the phase diagrams by open circles. For liquid(2), electron microprobe analyses showed that Pt content in the metastable phases, including solid and matrix, is less than 1 at.% in most cases, suggesting that the overall Pt content of the original liquid(2) probably does not exceed 1 at.%.

Thus, approximate compositions of the liquid(2) in equilibrium with any solid phase may be estimated indirectly by drawing a tie line from the composition of the solid phase to the Sb-Te binary join through the bulk composition of that particular run, because they are in univariant condition.

There are four 3-phase fields: PtSb + liquid(1) + PtSb₂, PtTe₂ + liquid(1) + Pt₃Te₄, PtSb₂ + liquid(1) + PtTe₂, and PtSb₂ + liquid(2) + PtTe₂. The observed 3-phase assemblages are shown in Figures 51 to 54. Ten 2-phase fields exist: Pt + liquid(1), PtSb + liquid(1), PtSb₂ + liquid(1), Pt₃Te₄ + liquid(1), PtTe₂ + liquid(1), PtSb₂ + liquid(2), PtTe₂ + liquid(2), PtSb + PtSb₂, PtTe₂ + Pt₃Te₄,
Fig. 51. An assemblage of PtSb (dark yellow) + PtSb₂ (yellow) + former liquid(1) quenched from 1000°C (run 411). The matrix separates into metastable phases during quenching. Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 52. An assemblage of PtTe₂ (dark yellow), Pt₃Te₄ (creamy yellow), and former liquid(1) quenched from 1000°C (run 422). Bladed crystals (light brown) exsolved from former liquid. Black areas are former gas-bubbles and pits. Carbon-coated. Plane polarized light.
Fig. 53. Photomicrograph showing an assemblage of PtSb₂ (bright yellow) and former liquid(1) (dark yellow) quenched from 1000°C (run 467). Black areas are pits. Plane polarized light.

Fig. 54. Subhedral and rounded crystals of PtSb₂ (a) coexisting with anhedral and acicular grains of PtTe₂ (b) and former liquid(2) quenched from 1000°C (run 406). The matrix contains small bladed crystals (c) of Sb₂Te₃ composition exsolved from former liquid. Black areas are pits. Carbon-coated. Plane polarized light.
and \( \text{PtSb}_2 + \text{PtTe}_2 \). The remainder of the phase diagram consists of solid or liquid single-phase fields.

All the solid phases present are solid solutions. Platinum in equilibrium with liquid(1) contains up to about 7 at.% Sb in solid solution, but negligible amounts of Te (less than 0.5 at.%), corresponding well to the earlier results for the Pt-Sb and Pt-Te systems. The composition of the platinum solid solution varies with the composition of the coexisting liquid(1): solid solubility of Sb in Pt decreases as Te content of the liquid increases. PtSb becomes Pt-poor as substitution of Te for Sb and Pt increases, and that coexisting with liquid(1) and PtSb\(_2\) (run 411) is Pt\(_{47}\)Sb\(_{47}\)Te\(_6\), showing the largest departure of 3 at.% Pt from its stoichiometry. PtSb\(_2\) and PtTe\(_2\) exhibit the greatest solid solution ranges among the solid phases but do not form complete solid solution between them: approximately 28.5 at.% Te substitutes for Sb in PtSb\(_2\) and the same amount of Sb substitutes for Te in PtTe\(_2\). The PtSb\(_2\) and PtTe\(_2\) solid solutions in equilibrium with liquid(1) are richer in Sb by 3 and 6 at.%, respectively, than those in equilibrium with liquid(2). The Pt\(_3\)Te\(_4\) phase shows solid solution of only 2 at.% by substitution of Sb for Te.

2) Isothermal section at 800°C

Starting compositions of the runs and their reaction products are listed in Table 15. The same experimental results are also presented diagrammatically in Fig. 55 and Fig. 56. In addition to the five stable phases at 1000°C,
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Abbreviations: 1h = first heating, 2h = second heating, L = liquid, Gv = grinstein, Mc = munnite, St = stannite.
Fig. 55. Phase relations in the Pt-rich portion of the Pt-Sb-Te system at 800°C. Solid circles and numerals represent bulk compositions and run numbers. Thick lines indicate narrow 2-phase fields.
Fig. 56. Phase relations in the Pt-poor portion of the Pt-Sb-Te system at 800°C. Solid circles and numerals represent bulk compositions and run numbers. Thick lines indicate narrow 2-phase fields.
Pt$_2$Te$_3$ and PtTe appear as stable phases at 800°C. The large area of liquid(1) at 1000°C is reduced to a small liquid between approximately 29 and 35 at.% Sb on the Pt-Sb binary join and extending 3 at.% towards the Te side. Liquid(2) remains as a thin strip along the Sb-Te join.

There are seven 3-phase fields: Pt + liquid(1) + PtTe, PtSb + liquid(1) + PtTe$_2$, PtSb + PtSb$_2$ + PtTe$_2$, PtSb$_2$ + liquid(2) + PtTe$_2$, PtTe$_2$ + liquid(1) + Pt$_2$Te$_3$, Pt$_2$Te$_3$ + liquid(1) + Pt$_3$Te$_4$, and Pt$_3$Te$_4$ + liquid(1) + PtTe. The observed 3-phase assemblages are shown in Figures 57 to 63. Fourteen 2-phase fields exist: Pt + PtTe, Pt + liquid(1), PtSb + liquid(1), PtSb$_2$ + PtSb, PtSb + PtTe$_2$, PtSb$_2$ + liquid(2), PtTe$_2$ + liquid(2), PtTe$_2$ + Pt$_2$Te$_3$, Pt$_3$Te$_4$ + Pt$_2$Te$_3$, PtTe + Pt$_3$Te$_4$, PtSb$_2$ + PtTe$_2$, PtTe$_2$ + liquid(1), Pt$_3$Te$_4$ + liquid(1), and Pt$_2$Te$_3$ + liquid(1). Of these, the Pt$_2$Te$_3$ + liquid(1) field is too narrow to permit drawing of boundaries, because the Pt$_2$Te$_3$ phases in equilibrium with Pt$_3$Te$_4$ and liquid(1) (runs 354, 351 and 347) and with PtTe$_2$ and liquid(1) (runs 329 and 349) are nearly identical in composition. The field is thus indicated by a thicker tie line in Fig. 55 and Fig. 56. The remainder of this isothermal section consists of single-phase solid or liquid fields.

Solid solubility of Sb in Pt is about 10 at.% and that of Te is negligible (less than 0.3 at.%). PtSb$_2$ in equilibrium with PtTe$_2$ and liquid(2) (run 308) contains 28.5 at.% Te substituting for Sb, which is the limit of the PtSb$_2$
Fig. 57. Photomicrograph showing an assemblage of platinum (dark yellow) + PtTe (brown) + former liquid(1) (greenish yellow) quenched from 800°C (run 318). Black areas are former gas-bubbles and pits. Carbon-coated. Plane polarized light.

Fig. 58. Photomicrograph showing an assemblage of PtSb (a), PtTe₂ (b), and former liquid(1) (c) quenched from 800°C (run 348). PtSb crystals are mostly elongated. Black areas are former gas-bubbles and pits. Carbon-coated. Plane polarized light.
Fig. 59. Fine intergrowths of PtSb₂ (greenish yellow), PtSb (pale orange), and PtTe₂ (pale brown) quenched from 800°C (run 343). Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 60. Small grains of PtSb₂ (a) coexisting with PtTe₂ (b) and former liquid (c) quenched from 800°C (run 308). Black areas are pits. Carbon-coated. Plane polarized light.
Fig. 61. Photomicrograph showing an assemblage of $\text{Pt}_2\text{Te}_3$ (yellow) + $\text{PtTe}_2$ (pale brown) + former liquid(l) (greenish yellow) quenched from 800°C (run 349). PtTe$_2$ crystals are elongated. Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 62. Photomicrograph showing an assemblage $\text{Pt}_2\text{Te}_3$ (a), $\text{Pt}_3\text{Te}_4$ (b), and former liquid(l) (c) quenched from 800°C (run 354). Black areas are former gas-bubbles and pits. Carbon-coated. Plane polarized light.
Fig. 63. Pt$_3$Te$_4$ (a) coexisting with PtTe (b) and former liquid (c) quenched from 800°C (run 334). Black areas are pits. Carbon-coated. Plane polarized light.
solid solution at 800°C. PtSb₂ coexisting with PtSb and PtTe₂ shows a substitution of 24.8 - 25.5 at.\% Te for Sb. The composition of PtTe₂ in equilibrium with PtSb and PtSb₂ was not determined by microprobe analysis because PtTe₂ grains were too finely intergrown with other phases, although its presence was confirmed by X-ray diffraction. The composition limit for Sb solid solution in PtTe₂ is close to Pt₃₃.₃Sb₁₂.₅Te₂₅.₂, as determined from the PtTe₂ in equilibrium with PtSb₂ in run 327. The other binary phases also show lesser amounts of Sb solid solution. The substitution of Te for Sb in PtSb is about 10 at.\% and the substitution of Sb for Te in Pt₂Te₃ and Pt₃Te₄, and PtTe are 6.7, 5.2, and 2.0 at.\%, respectively. Although PtTe₂ was not found at 800°C in the Pt-Te binary system, Pt₂(Te,Sb)₃ was observed and its X-ray powder pattern was identical to that of Pt₂Te₃. It suggests that the presence of small concentrations of Sb in Pt₂Te₃ may have stabilized the structure at 800°C.

3) Isothermal section at 600°C

Starting compositions of the runs and their reaction products are listed in Table 16. The experimental results are presented diagrammatically in Fig. 64 and Fig. 65. Compositions of some solid phases, although their presence was confirmed by optical and/or X-ray diffraction methods, could not be obtained by microprobe analysis because they are too fine grained or finely intergrown with other phases.
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Abbreviations: 1h = first heating, 2h = second heating, Gv = geversite, Mc = moncheite, St = stumpflite, Ti = tellurantimon. L = liquid.
Fig. 64. Phase relations in the Pt-rich portion of the Pt-Sb-Te system at 600°C. Solid circles and numbers represent bulk compositions and run numbers. Thick lines indicate narrow 2-phase fields.
Fig. 65. Phase relations in the Pt-poor portion of the Pt-Sb-Te system at 600°C. Solid circles and numerals represent bulk compositions and run numbers. Thick lines indicate narrow 2-phase fields. Dashed boundary lines indicate uncertainty.
At 600°C, five additional compounds appear as stable phases: $\text{Pt}_3\text{Sb}_7$, $\text{Pt}_3\text{Sb}$, $\text{Pt}_3\text{Sb}_2$, $\text{Sb}_2\text{Te}_3$ (tellurantimony) and Sb (antimony). Liquid(1) disappears completely, whereas liquid(2) splits into two liquids along the Sb-Te binary join at 7 - 50 at.% Te and 66.6 - 100 at.% Te due to the appearance of $\text{Sb}_2\text{Te}_3$. The two liquids are here referred to as liquid(2a) at 7 - 50 at.% Te and liquid(2b) at 66.6 - 100 at.% Te. The left boundary of the liquid(2a) was taken from the Sb-Te binary phase diagram (Fig. 47) as the polished surface of run product 642 did not contain adequate samples of the quenched liquid phase for analysis.

Twelve 3-phase fields exist in the phase diagram:

$\text{Pt} + \text{Pt}_3\text{Sb}_7 + \text{PtTe}_1, \text{Pt}_3\text{Sb}_7 + \text{Pt}_3\text{Sb} + \text{PtTe}, \text{Pt}_3\text{Sb} + \text{PtTe} + \text{Pt}_3\text{Te}_4, \text{Pt}_3\text{Sb} + \text{Pt}_3\text{Sb}_2 + \text{Pt}_3\text{Te}_4, \text{Pt}_3\text{Sb}_2 + \text{Pt}_3\text{Te}_4 + \text{Pt}_2\text{Te}_3, \text{Pt}_2\text{Te}_3 + \text{Pt}_3\text{Sb}_2 + \text{PtTe}_2, \text{Pt}_3\text{Sb}_2 + \text{PtSb} + \text{PtTe}_2, \text{PtSb} + \text{PtSb}_2 + \text{PtTe}_2, \text{PtSb}_2 + \text{Sb} + \text{liquid(2a)}, \text{PtSb}_2 + \text{liquid(2a)} + \text{Sb}_2\text{Te}_3, \text{PtSb}_2 + \text{Sb}_2\text{Te}_3 + \text{liquid(2b)}, \text{and PtSb}_2 + \text{PtTe}_2 + \text{liquid(2b)}. Some of these 3-phase assemblages are shown in Figures 66 to 73. The remainder of this diagram consists of 2-phase and single-phase fields. The two-phase fields, $\text{Pt}_3\text{Sb} + \text{PtTe}, \text{Pt}_3\text{Sb} + \text{Pt}_3\text{Te}_4, \text{Pt}_3\text{Sb}_2 + \text{Pt}_3\text{Te}_4, \text{Pt}_3\text{Sb}_2 + \text{Pt}_2\text{Te}_3$ and $\text{PtSb} + \text{PtTe}$ are too narrow to be shown diagrammatically, and therefore they are represented by thick tie lines in Fig. 64 and Fig. 65.

The composition of the liquids coexisting with $\text{PtSb}_2$ or $\text{PtTe}_2$ was impossible to determine by electron microprobe analysis due to separation of the former liquid into metastable phases during quenching (runs 610, 605, 607, 614
Fig. 66. Photomicrograph showing an assemblage of platinum (a), Pt$_2$Sb$_7$ (b), and PtTe (c) quenched from 600°C (run 643). Black areas are pits. Plane polarized light.

Fig. 67. Photomicrograph showing an assemblage of Pt$_3$Sb (a) + PtTe (b) + Pt$_3$Te$_4$ (c) quenched from 600°C (run 648). Black areas are pits. Crossed nicols.
Fig. 68. Massive Pt$_3$Sb (a) and fine intergrowths (b) of Pt$_3$Sb$_2$ and Pt$_3$Te$_4$ quenched from 600°C (run 624). Grain boundaries between Pt$_3$Sb$_2$ and Pt$_3$Te$_4$ are not clearly visible. Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 69. Photomicrograph showing an assemblage of Pt$_3$Sb$_2$ (cream) + PtTe$_2$ (brown) + Pt$_2$Te$_3$ (orange) quenched from 600°C (run 635). Black areas are pits. Carbon-coated. Plane polarized light.
Fig. 70. Photomicrograph showing an assemblage of Pt$_3$Sb$_2$ (yellowish pink) + PtSb (dark yellow) + PtTe$_2$ (orange brown) quenched from 600°C (run 637). Black areas are pits. Plane polarized light.

Fig. 71. Euhedral and subhedral crystals of PtSb$_2$ (yellowish white) coexisting with acicular crystals of Sb$_2$Te$_3$ (creamy yellow) and former liquid(I) (pale brown) quenched from 600°C (run 646). Black areas are former gas-bubbles and pits. Plane polarized light.
Fig. 72. Subhedral crystals of PtSb₂ (gray) in massive Sb₂Te₃ (yellowish pink) and former liquid(1) (yellow) quenched from 600°C (run 647). Black areas are pits. Plane polarized light.

Fig. 73. Small grains of PtSb₂ (yellowish pink) and PtTe₂ (brown) in former liquid(2) which separated into metastable Sb₂Te₃ (acicular, dark brown) and liquid (orange brown) quenched from 600°C (run 606). Black areas are pits. Carbon-coated. Plane polarized light.
and 608). Their Pt contents, however, were determined indirectly, by averaging the analyses of a number of grains of the metastable phases to be less than 1 at.%. The quenched products of the liquid(2b) in equilibrium with PtTe₂ and PtSb₂ contain metastable Sb₂Te₃ and an amorphous phase (apparently solidified liquid) of 90 at.% Te (Fig. 73), and is comparable with the results of run 16 of the Sb-Te system. The composition of this liquid would be somewhere between 68 at.% and 76 at.% Te, the estimated composition of liquids coexisting with PtSb₂ in run 605 and with PtTe₂ in run 607. Due to uncertainties in the composition of this liquid, boundaries of the PtSb₂ + PtTe₂ + liquid(2) field are shown in Fig. 65 by dashed lines.

Platinum may contain up to 9 at.% Sb, but little Te (runs 643 and 627). PtSb₂ in equilibrium with PtTe₂ and liquid(2b), with 27.0 - 28.5 at.% Te in substitution for Sb (runs 606, 609 and 612), defines the limit of the PtSb₂ solid solution with PtTe₂. The PtSb₂ in equilibrium with PtSb and PtTe₂ is close to its ideal composition, containing less than 1 at.% Te for Sb. Pt₃Sb₂ contains up to 5 at.% Te, substituting for Sb. PtTe₂ shows solid solution, with Sb substituting for Te up to 16.5 at.%. The substitutions of Sb for Te in the Pt₂Te₃ and Pt₃Te₄ phases are restricted to 8.3 and 3.5 at.%, respectively.

A comparison of the phase relations between the isothermal section at 650°C (Fig. 48) given by El-Boragy and Schubert (1971b) and that at 600°C (Fig. 64 and Fig. 65)
constructed in this study shows substantial agreement in the region 33.3 - 100 at.% Pt, except that the phase Pt\textsubscript{4}Sb of El-Boragy and Schubert has now been redefined as Pt\textsubscript{3.4}Sb\textsubscript{7}. However, there is considerable disagreement in phase relations for the 0 - 33.3 at.% Pt portion. Besides Sb and Sb\textsubscript{2}Te\textsubscript{3} present in this study, El-Boragy and Schubert (1971b) showed, on the Sb-Te join of their phase diagram (Fig. 50), the presence of four additional solid phases with the compositions of 19 - 21, 25 - 37, 40 - 52, and 100 at.% Te. However, phase diagrams of the Sb-Te system (Hansen and Anderko, 1958; Abrikosov et al., 1959; and Brown and Lewis, 1962) all illustrate that liquid is the only stable phase at 650°C, supported by the well established melting points of the stable solid phases in the system: Sb (630.5°C; Hansen and Anderko, 1958), Sb\textsubscript{2}Te\textsubscript{3} (621 - 622°C; Hansen and Anderko, 1958) and Te (450°C; Kracek, 1941).

It is possible that all the solid phases on the Sb-Te join of the phase diagram given by El-Boragy and Schubert (1971b) are metastable, and are formed during quenching. Similar metastable phases formed during quenching have been frequently encountered in this study. A more reasonable phase diagram for this region at 650°C is perhaps similar to that for 800°C of this study (Fig. 56) where a liquid is the only phase within the entire range of the Sb-Te binary join.

2. Phase Relations of the Pd-Pt-Sb System

There are no published data on the Pd-Pt-Sb system.
Phase relations of the Pd-Pt-Sb system were investigated at 1000° and 800°C. As this ternary system involves the Pt-Pd binary, the phase relations of which have not been confirmed in this study, the interpretation of this portion of the experimental data is based on the work of Raub (1959), who reported that platinum and palladium form a continuous solid solution in the temperature range, 1000 - 800°C.

Established minerals in the Pd-Pt-Sb system are platinum [Pt], mertieite II [Pd₈Sb₃], sudburyite [PdSb], palladium [Pd], stumplite [PtSb], genkinite [(Pt,Pd)₄Sb₃], stibiopalladinite [Pd₅₋ₓSb₂₋ₓ] and mertieite I [formula uncertain, close to (Pd,Cu)₅₋ₓ(Sb,As)₂₋ₓ].

1) Isothermal section at 1000°C

Starting compositions of the runs and their reaction products are shown in Table 17. The results are interpreted and presented in the form of a phase diagram as shown in Fig. 74 and Fig. 75. Binary phases stable at this temperature, as predicted from the results of the Pd-Sb and Pt-Sb binary systems in the previous chapter, are Pd₂₀Sb₇, Pd₈Sb₃, Pd₃₁Sb₁₂ and Pd₅Sb₂ on the Pd-Sb join, PtSb and PtSb₂ on the Pt-Sb join, and (Pd,Pt) solid solutions along the Pd-Pt join.

The isothermal section at 1000°C is characterized by the presence of an inverted V-shaped liquid field composed of a thin strip along the Pd-Sb join, linked to a band centered roughly at 35-40 at.% Sb, parallel to the Pt-Pd
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Table 12: Experimental results of the system Fe-FeS-P at 1000°C
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Abbreviations: 1h = first heating, 2h = second heating, L = liquid, Av = aversite, St = stumplite, Met II = mertieite II, A = alloy of Pt, Pd, and Sb. * Average of 7 - 16 analyses.
Fig. 74. Phase relations in the Pd-rich portion of the Pd-Pt-Sb system at 1000°C. Solid circles and numbers represent bulk compositions and identification of individual runs. Dashed field boundaries indicate approximation.
Fig. 75. Phase relations in the Pd-poor portion of the Pd-Pt-Sb system at 1000°C. Solid circles and numbers indicate bulk compositions and identification of the runs examined. Dashed field boundaries represent approximation.
join and a large field of Pd-Pt solid solution.

The boundary of the Pt-Pd solid solution field was

determined from the compositions of the (Pd, Pt) alloys in
equilibrium with $\text{Pd}_{20}\text{Sb}_7$ or liquid. Confirmation of the
boundary using charges (510, 511, 519, 520, 521, 522, 523,
and 527) with bulk compositions within the (Pd, Pt) solid
solution field was unsuccessful, because the run products
contained non-equilibrium assemblages consisting of Pd-Sb or
Pt-Sb binary compounds with variable compositions and
unreacted Pt and Pd. Portions of the boundaries of the
liquid field could not be determined with precision, because
metastable solid phases were formed during quenching (Fig.
76 to Fig. 77). In such cases, the composition of the
liquid in-equilibrium with a solid phase was approximated by
averaging a large number of analyses of the metastable
phases. The boundaries so derived are represented by
dashed lines in the phase diagrams (Fig. 74 and Fig. 75).

Solid solubility of Sb in the (Pd, Pt) alloy is found to
range from 16 - 17 at.% (at the Pd-Sb binary) to 7 - 8 at.%
(at the Pt-Sb binary join). The Pd-Sb binary compounds may
contain Pt in substitution for Pd up to at least 24 at.% in
$\text{Pd}_{20}\text{Sb}_7$ (run 537, Fig. 78), 22 at.% in $\text{Pd}_8\text{Sb}_3$ (run 588) and
$\text{Pd}_{31}\text{Sb}_{12}$ (run 518, Fig. 79), and 19.6 at.% in $\text{Pd}_5\text{Sb}_2$ (run
587). The upper limits of the substitutions, however, are
not determined. The Pd content of 18 at.% (run 545, Fig.
80) in the PtSb, coexisting with PtSb$_2$ and liquid, defines
the upper limit of the Pd substitution for Pt in this
compound at 1000°C. The substitution of Pd for Pt in PtSb$_2$
Fig. 76. Photomicrograph showing dendritic crystals (brown) exsolved from an original liquid quenched from 1000°C (run 554). Black areas are pits. Plane polarized light.

Fig. 77. Photomicrograph showing rounded grains of (Pt,Pd) alloy (pale green) surrounded by former liquid which separated into metastable phases (darker green) when quenched from 1000°C (run 595). Crossed nicols.
Fig. 78. Irregular grains of (Pd,Pt) alloy (a) coexisting with Pd$_{20}$Sb$_7$ (b) quenched from 1000°C (run 537). Black areas are former gas-bubbles. Carbon-coated. Plane polarized light.

Fig. 79. Large grains of Pd$_5$Sb$_2$ (pinkish grey) and former liquid (yellow) quenched from 1000°C (run 518). The matrix contains dendritic crystals of Pd$_5$Sb$_2$ composition. Black areas are pits. Carbon-coated. Plane polarized light.
Fig. 80. Photomicrograph showing an assemblage of PtSb (dark yellow) + PtSb₂ (greenish yellow) + former liquid (dark brown) quenched from 1000°C (run 545). Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 81. A large crystal of PtSb₂ (yellow) surrounded by former liquid (brown) which separated into metastable phases coloured yellow, brown, and dark brown when quenched from 1000°C (run 506). Black areas are pits. Carbon-coated. Plane polarized light.
is rather restricted, about 7.2 at.% (run 506, Fig. 81).

There are at least nine 2-phase assemblages in the system at 1000°C, namely: Pd$_{20}$Sb$_7$ + (Pd, Pt) alloy, (Pd, Pt) alloy + liquid, Pd$_5$Sb$_2$ + liquid, PtSb$_2$ + liquid, PtSb + liquid (Fig. 82), PtSb$_2$ + PtSb (Fig. 83), Pd$_{20}$Sb$_7$ + Pd$_8$Sb$_3$, Pd$_8$Sb$_3$ + Pd$_{31}$Sb$_{12}$, and Pd$_{31}$Sb$_{12}$ + Pd$_5$Sb$_2$. The phase relations are not clear in the shaded area of the phase diagram because (1) the liquids coexisting with solid phases separate into metastable phases during quenching and (2) distinction of structurally and compositionally similar Pd$_{31}$Sb$_{12}$ and Pd$_5$Sb$_2$ is practically impossible by X-ray diffraction and electron microprobe analysis.

The boundaries of fields [Pd$_{20}$Sb$_7$ + (Pd, Pt) alloy, (Pd, Pt) alloy + liquid, Pd$_5$Sb$_2$ + liquid, Pd$_{20}$Sb$_7$ + Pd$_8$Sb$_3$, Pd$_8$Sb$_3$ + Pd$_{31}$Sb$_{12}$, and Pd$_{31}$Sb$_{12}$ + Pd$_5$Sb$_2$] are not fully determined due to the presence of an area near the middle of the phase diagram where the phase relations are not clear. The lines in the Pd$_{31}$Sb$_{12}$ + Pd$_5$Sb$_2$ field could not be established because the compositional difference between the two is too small. There is only one 3-phase field, PtSb$_2$ + PtSb + liquid, that is firmly established. The liquid in equilibrium with PtSb$_2$ and PtSb has the composition of Pd$_{30.3}$Pt$_{30.6}$Sb$_{39.1}$. 3-phase fields must also be present in the vicinity between the (Pd, Pt) solid solution and the liquid fields. However, because of the presence of the problematic area in that region, it is not possible to determine their boundaries.
Fig. 82. Large grains of PtSb (orange) in former liquid (brown) quenched from 1000°C (run 543). Small yellow speck in the middle right is a dust mark. Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 83. PtSb₂ (creamy white) surrounded by massive PtSb (orange) quenched from 1000°C (run 549). Black areas are former gas-bubbles. Carbon-coated. Plane polarized light.
2) Isothermal section at 800°C

Starting compositions of the runs and their reaction products are shown in Table 18. The interpretation of the experimental results is shown diagrammatically in Fig. 84 and Fig. 85. In addition to the binary phases observed at 1000°C, Pd₅Sb₃ and PdSb (sudburyite) appear as stable solid phases at 800°C.

The main features of the phase diagram at 800°C are similar to those at 1000°C, except that the boundary of the \( \text{Pd} \), \( \text{Pt} \) solid solution field, as determined from the compositions of the \( \text{Pd} \), \( \text{Pt} \) alloys coexisting with \( \text{Pd}_{20}\text{Sb}_7 \), \( \text{Pd}_8\text{Sb}_3 \), or liquid, shifted towards the Sb-rich side to about 21 at.% and 10 at.% Sb along the Pd-Sb and Pt-Sb joins respectively as determined for the Pd-Sb and Pt-Sb binary systems.

The inverted V-shaped liquid field at 1000°C is broken into two liquid fields due to the appearance of PdSb, a band of liquid field from the 40-48 at.% Sb on the Pd-Sb join to the 30-40 at.% Sb on the Pt-Sb join [liquid(1)] and a thin strip of liquid field from 52 to 100 at.% Sb along the Pd-Sb join [liquid(2)]. Both liquids appear to be unquenchable, except in runs 211, 215, 226, and 227, forming metastable phases during quenching (Fig. 86 and Fig. 87), and thus making it difficult to delineate their boundaries. The composition of the liquid(1) in equilibrium with solid phase was approximated by averaging a large number of probe analyses of the metastable phases. For liquid(2), electron
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Abbreviations: 1h = first heating, 2h = second heating, 3h = third heating, L = liquid, Gv = geversite, St = stempflite, Sd = sudburyite, A = alloy of Pt, Pd, and Sb. * Average of 7 - 10 analyses.
Fig. 84. Phase relations in the upper half of the Pd-Pt-Sb system at 800°C. Solid circles and numbers represent bulk compositions and identification of runs examined. Approximate boundaries of liquid field are shown by dotted lines.
Fig. 85. Phase relations in the lower half of the Pd-Pt-Sb system at 800°C. Solid circles and numbers represent bulk compositions and identification of the runs examined. Dashed field boundaries indicate uncertainty. Approximate liquid-field boundaries are represented by dotted lines.
Fig. 86. Photomicrograph showing rounded grains of PtSb (orange) and former liquid which separated into metastable phase (orange brown) when quenched from 800°C (run 241). Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 87. Euhedral and subhedral crystals of PtSb₂ (yellowish pink) in former liquid which separated into metastable phases (orange) when quenched from 800°C (run 245). Many PtSb₂ grains contain inclusions of former liquid. Black areas are pits. Carbon-coated. Plane polarized light.
microprobe analyses showed that Pt content in the metastable phases, including solid and matrix, is about 0.1 - 0.3 at.%, suggesting that the overall Pt content of the original liquid(2) probably does not exceed 0.5 at.%. Thus, approximate compositions of the liquid(2) in equilibrium with Ptsb₂ are estimated by drawing a tie line from the composition of the solid phase to the Pd-Sb join through the bulk composition if that particular run. These approximate boundary points are indicated in the phase diagram (Fig. 84 and Fig. 85) by open circles, and boundaries of the liquid fields are, therefore, denoted by dashed lines.

The Pd₂₀Sb₇ phase contains up to at least 18.4 at.% Pt in substitution for Pd (run 265, Fig. 88). The upper limit of the substitution is not determined, but is expected to be less than 19.7 at.% Pt as it is restricted within the Pd₈Sb₃ - (Pd,Pt) alloy tie line of run 221. This is considerably less than the known range at 1000°C. Pd₈Sb₃ occurs in equilibrium with (Pd,Pt) alloys (runs 219 and 221, Fig. 89) or liquid (run 217, Fig. 90), and shows the most significant solid solution range among the binary phases at this temperature, with a substitution of up to at least 44.7 at.% Pt for Pd (much greater than the known range of 22 at.% at 1000°C). The upper limit is unknown. The Pd₈Sb₃ phase becomes enriched in Sb content as the amount of substitution of Pt for Pd increases: 27.6 at.% Sb (run 221), 28.6 at.% Sb (run 219), and 29.5 at.% Sb (run 217). The Pt-Pd substitution range of the Pd₃₁Sb₁₂ phase is not certain,
Fig. 88. Photomicrograph showing massive $\text{Pd}_{20}\text{Sb}_{7}$ (grayish brown) with small inclusions of (Pt,Pd) alloy (yellowish pink) quenched from $800^\circ\text{C}$ (run 265). Black areas are former gas-bubbles and pits. Carbon-coated. Plane polarized light.

Fig. 89. $\text{Pd}_8\text{Sb}_3$ (orange-yellow) containing small inclusions of (Pd,Pt) alloy (creamy yellow) quenched from $800^\circ\text{C}$ (run 219). Black areas are pits. Plane polarized light.
Fig. 90. Large Pd$_2$Sb$_3$ grains (a) surrounded by former liquid which separated into metastable phases (b) when quenched from 800°C (run 217). Black areas are former gas-bubbles and pits. Plane polarized light.

Fig. 91. Photomicrograph showing rounded crystals of Pd$_2$Sb$_2$ (grayish brown) in former liquid (grayish pink) quenched from 800°C (run 259). Black areas are pits. Carbon-coated. Plane polarized light.
because none of the reaction products, except for a single-phased one (run 252), contains the phase. The Pd₅Sb₂ phase shows a significant increase in Pt-Pd substitution range as compared with that at 1000°C; the phase coexisting with liquid (run 259) has 38.5 at.% Pt (Fig. 91). Substitution of up to 8 at.% Pt and 18 at.% Pt for Pd is confirmed for the Pd₅Sb₃ phase (run 222) and PdSb (run 240, Fig. 92), respectively. The Pd content (18 at.% of the PdSb₂ coexisting with PdSb and liquid (runs 215 and 227, Fig. 93) defines the limit of Pd substitution for Pt in that phase at this temperature. PdSb may contain up to 46 at.% Pd in substitution for Pt (run 243, Fig. 94), a significant increase over the known range at 1000°C.

Phase relations for the region between the tie lines of Pd₅Sb₃ - (Pd, Pt) alloy (run 219) and liquid(1) - (Pt, Pd) alloy (run 206) were left undetermined, because runs with bulk compositions (207, 213, 260, 262, and 263) falling in this field did not produce equilibrium assemblages, as indicated by the presence of compounds with variable compositions and small amounts of unreacted Pt-metal.

There are at least sixteen 2-phase fields in the phase diagram, namely: (Pd, Pt) alloy + Pd₁₀Sb₇, (Pd, Pt) alloy + Pd₅Sb₃, (Pd, Pt) alloy + liquid(1), Pd₅Sb₂ + Pd₅Sb₃, Pd₅Sb₂ + liquid(1), Pd₅Sb₃ + liquid(1), PdSb + liquid(1), PtSb₂ + liquid(2), PtSb₂ + liquid(1), PtSb₂ + PdSb, PtSb + liquid(1), Pd₁₀Sb₇ + Pd₅Sb₃, Pd₆Sb₃ + Pd₃₁Sb₁₂, Pd₃₁Sb₁₂ + Pd₅Sb₂, PdSb + liquid(2), and PtSb₂ + PtSb. With the exception of the PtSb₂ - liquid(2) field, the boundaries of
Fig. 92. Rounded grains of PdSb (grayish pink) enclosed by former liquid that separated into metastable phases (brown and orange) when quenched from 800°C (run 240). Black areas are pits. Plane polarized light.

Fig. 93. Photomicrograph showing an assemblage of PtSb₂ (a), PdSb (b), and former liquid (c) quenched from 800°C (run 227). Black areas are former gas-bubbles and pits. Plane polarized light.
Fig. 94. Photomicrograph showing an assemblage of PtSb (orange) + PtSb₂ (grayish pink) quenched from 800°C (run 243). Black areas are former gas-bubbles and pits. Plane polarized light.
these fields are not fully determined due to the uncertainties in the 3-phase fields.

There is only one 3-phase field \([\text{PtSb}_2 + \text{PdSb} + \text{liquid}(2)]\) the boundaries of which are known with certainty. In the Sb-rich half of the phase diagram, two hypothetical 3-phase fields may be postulated as shown by dotted boundary lines. These are: \(\text{PtSb}_2 + \text{PdSb} + \text{liquid}(1)\) and \(\text{PtSb}_2 + \text{PdSb} + \text{liquid}(1)\). Three additional 3-phase fields are postulated in the Sb-poor half of the phase diagram and represented by dotted lines. They are: \(\text{Pd}_2\text{Sb}_7 + \text{Pd}_8\text{Sb}_3\), \(\text{Pd}_5\text{Sb}_2 + \text{Pd}_5\text{Sb}_3 + \text{liquid}(1)\), and \(\text{Pd}_8\text{Sb}_3 + \text{liquid}(1) + (\text{Pt},\text{Pd})\text{ alloy}\). Further experimental data are required to confirm the boundary lines.

The phase with genkinite composition \([(\text{Pt},\text{Pd})_4\text{Sb}_3]\), synthesized by Cabri et al. (1977) at about 675°C, has not been observed at 1000°C and 800°C.

3. Phase Relations of the Pd-Sb-Te System

A. Previous study: An isothermal section of the Pd-Sb-Te system at 400°C was constructed by El-Boragy and Schubert (1971a), and it is apparently the only ternary diagram in the literature (Fig. 95).

B. Present study: Phase relations of the Pd-Sb-Te system at 1000°C were investigated in this study. Starting compositions of the runs and results of their reaction
Fig. 95. Isothermal section of the Pd-Sb-Te system at 400°C (El-Boragy and Schubert, 1971a).
products are shown in Table 19. The interpretation of the experimental results are presented diagrammatically in Fig. 96. The isothermal section at 1000°C is characterized by the presence of a large liquid field that extends from Te20 on the Pd-Te join and Sb35 on the Pd-Sb join all the way to the Sb-Te join. Quenching of the liquid often caused formation of metastable phases. For example, reaction products of run 902, Pd10Sb60Te30 in bulk composition, consisted of small dendrites of Pd34Sb57Te9 in a nonhomogeneous matrix (Fig. 97). In such a case, the presence of the liquid phase in the quenched run product was recognized from the texture of the phases in the polished sections, and the composition of the liquid, needed for determination of the boundary, was approximated by averaging the 7–15 microprobe analyses of the metastable phases and the matrix. The liquid field boundaries so derived are shown by dashed lines in the phase diagram.

Stable solid phases at 1000°C are Pd20Sb7, Pd8Sb3, Pd31Sb12, and Pd5Sb2, and palladium. Palladium contains 16.5 at.% Sb and 10.8 at.% Te in solid solution along the Pd-Sb and Pd-Te binary join, respectively. The Pd content of the Pd20Sb7 phase ranges from 74.6 at.% (run 941; 954, 965, and 940) – 75.4 at.% (run 919) on the Pd-rich side to 74.0 at.% Pd (run 973) on the Pd-poor side. The range covers the phase with Pd3Sb2 composition. The Pd20Sb7 phase may have up to about 42 at.% Te in substitution for Sb9 (run 968). Single-phased products of Pd20Sb7 solid solution
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Abbreviations: 1h = first heating, 2h = second heating, Average of 7 - 16 analyses.
Fig. 96. Phase relations in the Pd-rich portion of the Pd-Sb-Te system at 1000°C. The lower portion of this diagram is an area of liquid that extends to the Sb-Te join. Solid circles represent bulk compositions and numeral run numbers. Dotted field boundary indicates uncertainty.
were synthesized in runs 946 and 947. \( \text{Pd}_8\text{Sb}_3 \) and \( \text{Pd}_{31}\text{Sb}_{12} \)
may contain up to 36 - 38 and 38.3 at.% Te respectively in substitution for Sb (runs 952, 970, and 957; Fig. 98), whereas \( \text{Pd}_5\text{Sb}_2 \) contains only up to 16.5 at.% Te (run 939, Fig. 99).

There are nine 2-phase fields: \( \text{Pd} + \text{Pd}_{20}\text{Sb}_7 \), \( \text{Pd}_{20}\text{Sb}_7 + \text{Pd}_8\text{Sb}_3 \), \( \text{Pd}_8\text{Sb}_3 + \text{Pd} + \text{liquid} \), \( \text{Pd}_{20}\text{Sb}_7 + \text{Pd}_8\text{Sb}_3 \), \( \text{Pd}_8\text{Sb}_3 + \text{Pd}_{31}\text{Sb}_{12} \), \( \text{Pd}_{31}\text{Sb}_{12} + \text{Pd}_5\text{Sb}_2 \), and \( \text{Pd}_5\text{Sb}_2 + \text{liquid} \).

One 3-phase field of \( \text{Pd} + \text{Pd}_{20}\text{Sb}_7 + \text{liquid} \) (run 963, Fig. 100) is confirmed between the two univariant fields of \( \text{Pd} + \text{Pd}_{20}\text{Sb}_7 \) and \( \text{Pd} + \text{liquid} \). There are three additional invariant fields whose exact boundaries were not determined experimentally. These are \( \text{Pd}_{20}\text{Sb}_7 + \text{Pd}_8\text{Sb}_3 + \text{liquid} \), \( \text{Pd}_8\text{Sb}_3 + \text{Pd}_{31}\text{Sb}_{12} + \text{liquid} \), and \( \text{Pd}_{31}\text{Sb}_{12} + \text{Pd}_5\text{Sb}_2 + \text{liquid} \). The difficulties encountered in the determination of boundaries of the invariant fields are as follows: (i) compositions of the solid phases are very similar, (ii) the liquid coexisting with solid phases tends to separate into metastable phases during quenching, making it difficult to determine the composition of the liquid accurately, and (iii) the \( \text{Pd}_{31}\text{Sb}_{12} \) phase and the structurally related \( \text{Pd}_5\text{Sb}_2 \) phase have almost identical optical properties and X-ray diffraction patterns. The difference in their chemical compositions is well within the error range of electron microprobe analysis. Thus the two phases in the same run product cannot be distinguished with confidence.

Nevertheless, the approximate boundaries of the 3-phase
Fig. 97. Photomicrograph showing dendrites (grey) exsolved from original liquid when quenched from 1000°C (run 902). Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 98. Rounded grains of Pd₆Sb₃ (greyish brown) and interstitial former liquid quenched from 1000°C (run 952). Black areas are former gas-bubbles and pits. Carbon-coated. Plane polarized light.
Fig. 99. Oriented grains of $\text{Pd}_5\text{Sb}_2$ (brown) in former liquid which separated into metastable phases (reddish brown) when quenched from $1000^\circ\text{C}$ (run 939). Black areas are pits. Carbon-coated. Plane polarized light.

Fig. 10B. Photomicrograph showing an assemblage of Pd (creamy yellow) + $\text{Pd}_{32}\text{Sb}_7$ (dark yellow) + former liquid (dark yellow) quenched from $1000^\circ\text{C}$ (run 963). Black areas are pits and former gas-bubbles. Carbon-coated. Plane polarized light.
field may be postulated on the basis of the known tie lines of the neighbouring 2-phase fields. These are shown by dotted lines in Fig. 101.

El-Boragy and Schubert (1971a) reported that complete solid solution exists at 400°C between Pd3Sb and Pd3Te (presently Pd20Sb7 and Pd20Te7) and between PdSb and PdTe. To verify these solid solutions at higher temperatures, charges with bulk compositions of Pd74.22Sb14.99Te10.79, Pd74.19Sb10.02Te15.79, and Pd74.20Sb5.01Te20.19, were heated at 700°C for 27 days and charges with bulk compositions of Pd49.99Sb40.01Te10.00, Pd49.99Sb25.00Te25.01, and Pd48.70Sb11.80Te39.50, were heated at 670°C for 28 days. In all cases, homogeneous single-phased products with the Pd20(Sb,Te)7, and Pd(Sb,Te) structures were obtained, suggesting that complete solid solution between Pd20Sb7 and Pd20Te7 persists at least up to 700°C and the complete solid solution between PdSb and PdTe up to 670°C. X-ray data for these solid solutions will be discussed in the next chapter.

The synthetic phase with testibiopalladite composition [PdSbTe] and a cobaltite structure, first synthesized by Hulliger (1963) at unknown temperature, does not occur at 1000°C. The synthetic equivalent of borovskite (Pd3SbTe4; Fleischer, 1974) was not found by El-Boragy and Schubert (1971a) at 400°C nor in this study at 1000°C.

The phase diagram (Fig. 95) of El-Boragy and Schubert (1971a) shows a 3-phase field of antimony + PdSb2 + Sb2Te3, with Sb2Te3 as the only intermediate compound between Sb and
Fig. 101. Phase diagram of the Pd–Sb–Te system at 1000°C showing approximate boundaries of the possible three-phase assemblages (dashed lines) not determined experimentally. Dotted field boundary indicates uncertainty.
Te at 400°C. The study on the Sb-Te binary in this thesis shows the existence of a phase X, stable below 549°C with a composition of Sb$_{88}$Te$_{12}$. However, the composition range of the phase X on the Te-side is not known. Thus, the 3-phase field, if exists, must be defined by PdSb$_2$, phase X, and Sb$_{88}$Te$_{12}$. 
CHAPTER V

PROPERTIES OF THE SYNTHETIC PHASES

Because previous studies of the synthetic Pt-Pd-Sb-Te compounds were largely restricted to crystallographic work, data of their optical properties and micro-indentation hardness are very sparse or non-existent. The synthetic phases obtained in this study were examined to obtain data which provide a basis for better understanding each phase and contribute to characterizations of the natural compounds. The work in this study is limited to a determination of qualitative optical characteristics, micro-indentation hardness data, and compositional variations of pure elements and the phases synthesized in this study. In addition, the cell parameters of the purest binary phases and elements have been re-determined or newly determined, and are compared with previously published X-ray data by earlier authors. In all cases where natural analogues of synthetic compounds occur and their optical, physical, or X-ray data are known, properties of the synthetic and natural phases have been compared.

1. Platinum

Native platinum usually contains a few per cent of Fe and lesser amounts of Ir, Os, Rh, Pd, Cu, Au, Ni, etc. and contains greater than 80 at.% Pt (Cabri and Feather, 1975). Optical, physical and X-ray data for native platinum are shown in Table 20.
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<td>isotropic(^{(5)})</td>
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<td>(\text{VHN}_{50,100} = 580) (553-588)(^{(8)})</td>
<td>(\bigcirc)</td>
</tr>
<tr>
<td><strong>Melting point ((^{\circ})C)</strong></td>
<td>1768.4(^{(2)})</td>
<td>(\bigcirc)</td>
<td>(\bigcirc)</td>
</tr>
<tr>
<td></td>
<td>1769.9(^{(3)})</td>
<td>(\bigcirc)</td>
<td>(\bigcirc)</td>
</tr>
<tr>
<td><strong>Symmetry</strong></td>
<td>isometric, (\text{Fm}3\text{m})(^{(4)})</td>
<td>(\bigcirc)</td>
<td>(\bigcirc)</td>
</tr>
<tr>
<td><strong>Cell parameters (Å)</strong></td>
<td>(a = 3.9226(2))</td>
<td>(\bigcirc)</td>
<td>(\bigcirc)</td>
</tr>
<tr>
<td></td>
<td>(a = 3.9231)(^{(4)})</td>
<td>(\bigcirc)</td>
<td>(\bigcirc)</td>
</tr>
<tr>
<td></td>
<td>(a = 3.923)(^{(3)})</td>
<td>(\bigcirc)</td>
<td>(\bigcirc)</td>
</tr>
<tr>
<td></td>
<td>(a = 3.877)(^{(3)})</td>
<td>(\bigcirc)</td>
<td>(\bigcirc)</td>
</tr>
<tr>
<td></td>
<td>(a = 3.851)(^{(1)})(^{(8)})</td>
<td>(\bigcirc)</td>
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Table 21. X-ray powder diffraction data of pure Pt and native platinum.

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<td></td>
</tr>
<tr>
<td>h k l</td>
<td>(d_c(\text{Å}))</td>
<td>(d_o(\text{Å}))</td>
<td>I/I(_o)</td>
</tr>
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<td>-----</td>
<td>---------</td>
<td>---------</td>
<td>--------</td>
</tr>
<tr>
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<td>2.236</td>
<td>100</td>
</tr>
<tr>
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<td>1.960</td>
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<tr>
<td>h k l</td>
<td>(d_c(\text{Å}))</td>
<td>(d_o(\text{Å}))</td>
<td>I/I(_o)</td>
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<table>
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<tr>
<td>h k l</td>
<td>(d_c(\text{Å}))</td>
<td>(d_o(\text{Å}))</td>
<td>I/I(_o)</td>
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</table>

1. This study. 99.9995\% purity, Johnson Matthey Chemical Ltd.
CuK\(_\alpha_1\) radiation (\(\lambda = 1.54059\)Å), automated diffractometer.
2. PDF 4-0802. 3 additional high angle reflections not reported.
Properties of pure-element Pt are shown in Table 20 and are compared with those of native Pt. The micro-indentation hardness value for pure Pt metal is close to that of native platinum of unknown composition given by Craig and Vaughan (1981), but significantly smaller than those of native platinum containing about 10 – 11 at.% Fe (Tarkian and Stumpfl, 1975; Câbri et al., 1973). The microhardness value VHN\(_{50}\) = 580 (533-588) of the native platinum grains from placer deposits in Tulameen River, British Columbia is significantly greater than VHN\(_{50}\) = 345 - 466 of 'primary platinum' with a similar composition. Ramdohr (1960) suggested that the microhardness of placer platinum may be increased by 'cold working' during river transport. Pt contains up to 11.0 at.% Sb in solid solution at 1000°C, 10 at.% Sb at 800°C, and 7.5 at.% Sb at 600°C, but Te content in solid solution is negligible (<0.5 at.%). The refined cell parameter for pure-element Pt used in this study is a = 3.9226(2)Å; this compares closely with that of Raub (1959), but is considerably larger than those for native Pt containing 27 % Fe and 16 % Fe (Table 20). X-ray powder diffraction patterns of the pure metal and native platinum are compared in Table 21.

2. Palladium

Not much is known about native palladium. Previously reported optical properties of palladium are shown in Table 22.
<table>
<thead>
<tr>
<th>Property</th>
<th>Pd (This study)$^{(1)}$</th>
<th>Pd</th>
<th>Native Pd$^{(4)}$</th>
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<tr>
<td>Colour</td>
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<td></td>
<td>white with yellowish tinge</td>
</tr>
<tr>
<td>Bireflectance/pleochroism</td>
<td>none</td>
<td></td>
<td>none</td>
</tr>
<tr>
<td>Anisotropism</td>
<td>isotropic</td>
<td></td>
<td>isotropic</td>
</tr>
<tr>
<td>Polishing characteristic</td>
<td>takes a good polish</td>
<td></td>
<td>takes a good polish</td>
</tr>
<tr>
<td>Micro-indentation</td>
<td>VHN$_{100}$ = 116.3 (109-123) for 5 measurements</td>
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<tr>
<td>hardness, mean (range)</td>
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</tr>
<tr>
<td>Melting point</td>
<td>1554$^\circ$C$^{(2)}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Symmetry</td>
<td>isometric, Fm3m$^{(3)}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cell parameters</td>
<td>a = 3.8881(5)</td>
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<td>a = 3.8898$^{(3)}$</td>
</tr>
<tr>
<td>(Å)</td>
<td></td>
<td></td>
<td>a = 3.890(2)</td>
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</tbody>
</table>

$^{(1)}$ 99.999% purity, Johnson Matthey Chemical Ltd., $^{(2)}$ Raub (1959), $^{(3)}$ PDF 5-681, $^{(4)}$ Picot and Johann (1982).
Table 23. X-ray powder diffraction data of pure Pd metal.

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<th>k</th>
<th>l</th>
<th>d_c(Å)</th>
<th>d_o(Å)</th>
<th>I/I_o</th>
<th>d_o(Å)</th>
<th>I/I_o</th>
</tr>
</thead>
<tbody>
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<td>1</td>
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<td>1</td>
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<td>1.1730</td>
<td>24</td>
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<td>1.1224</td>
<td>1.1225</td>
<td>13</td>
<td>1.1232</td>
<td>8</td>
</tr>
</tbody>
</table>

1. This study. 99.9996% purity, Johnson Matthey Chemical Ltd., CuKα radiation (λ = 1.54059Å), automated diffractometer.

2. PDF 5-681. 3 additional high angle reflections not reported.
Properties of the pure-element Pd determined in this study and by others are shown in Table 22. Solid solubility of Sb in the pure element Pd is 16.5 at.% at 1000°C, 21.0 at.% at 800°C, and 0.2 at.% at 510°C. Amounts of Te dissolved in palladium are 10.8 at.% at 1000°C, 14.8 at.% at 800°C, and 0.1 at.% at 600°C. X-ray powder diffraction data for the pure Pd metal are shown in Table 23.

3. Antimony

Previously published properties of the native antimony, and X-ray and melting point data of the pure element antimony are shown in Table 24.

Properties of pure-element antimony determined in this study are shown in Table 24. Optical properties of pure antimony are identical to those of native antimony, as shown in Table 24. The micro-indentation hardness value \( VHN_{100} = 69.9 \) (62.0 - 78.5) for pure Sb is slightly lower than the range 84 - 98 for native Sb given by Craig and Vaughan (1981). Solid solubility of Te in Sb is about 1 at.% up to 600°C and that of Pt or Pd is negligible. The X-ray powder pattern of pure-element Sb is indexed on a hexagonal cell with \( a = 4.3056(8) \) and \( c = 11.250(3) \). X-ray powder data are given in Table 25.

4. Tellurium

Previously published properties of native tellurium and X-ray and melting point data of pure tellurium are shown in
<table>
<thead>
<tr>
<th></th>
<th>pure Sb (This study)(^{(1)})</th>
<th>pure Sb</th>
<th>native Sb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colour</td>
<td>white with yellowish tinge in air and in oil</td>
<td>white with a very light creamy shade(^{(5)})</td>
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</tr>
<tr>
<td>Bireflectance/pleochroism</td>
<td>nil to very weak</td>
<td>weak(^{(6)}) extremely weak(^{(5)})</td>
<td></td>
</tr>
<tr>
<td>Anisotropism</td>
<td>weak to moderate (dark olive grey to brownish grey) in air and slightly enhanced in oil</td>
<td>strong; yellowish grey to brownish grey(^{(6)}); orange brown to yellowish brown(^{(5)})</td>
<td></td>
</tr>
<tr>
<td>Polishing characteristic</td>
<td>takes a good polish</td>
<td>takes a good polish(^{(6)})</td>
<td></td>
</tr>
<tr>
<td>Micro-indentation</td>
<td>(VHN_{100} = 69.9\ (62.0-78.5)) for 5 measurements</td>
<td>(VHN_{100} = 84-98)(^{(6)})</td>
<td></td>
</tr>
<tr>
<td>hardness mean (range)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Melting point</td>
<td>(624^\circ\ C(^{(2)}), 630^\circ\ C(^{(3)})</td>
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</tr>
<tr>
<td>Symmetry</td>
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<td>Rhombohedral, (R\bar{3}m)(^{(7)})</td>
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<tr>
<td>Cell parameters</td>
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<td>(a = 4.307, c = 11.273\AA)(^{(4)})</td>
<td>(a = 4.310, c = 11.28\AA)(^{(7)})</td>
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<tr>
<td>Others</td>
<td>cleavages well developed and twinning common</td>
<td>lamellar twinning, concentric and botryoidal form common</td>
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Table 25. X-ray powder diffraction data of antimony.

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<th>l</th>
<th>d_c(Å)</th>
<th>d_o(Å)</th>
<th>I/I_o</th>
<th>d_o(Å)</th>
<th>I/I_o</th>
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1. 99.9999% purity, Johnson Matthey Chemical Ltd. CuKα, radiation (λ = 1.54059Å), automated diffractometer.
2. PIF 5-562. 12 additional high angle reflections not reported.
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<tr>
<th>Property</th>
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<th>Native Tellurium</th>
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<tr>
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<td>White with yellowish tinge in air and oil</td>
<td>White, gray (4)</td>
<td>Distinct (4)</td>
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<tr>
<td>Bireflectance/pliochroism</td>
<td>Nil</td>
<td>Strong: light gray to brownish gray (4)</td>
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</tr>
<tr>
<td>Anisotropism</td>
<td>Strong: yellowish green to extinction in air; olive gray to extinction in oil</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Polishing characteristics</td>
<td>Takes a good polish</td>
<td>Scratches difficult to avoid</td>
<td></td>
</tr>
<tr>
<td>Micro-indentation hardness, mean (range)</td>
<td>$VHN_{100} = 37.4 (35.9 - 38.5)$ for 5 indentations</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Melting point</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Symmetry</td>
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<td>Hexagonal, $P3_1 21 (6)$</td>
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<tr>
<td>Cell parameters</td>
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<td>$a = 4.4572, c = 5.9290 \AA (3)$</td>
<td>$a = 4.454, c = 5.92 \AA (6)$</td>
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<tr>
<td>Others</td>
<td>Cleavages good</td>
<td></td>
<td>Cleavages well-developed (4)</td>
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Table 27. X-ray powder diffraction data of tellurium.

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<th>d_o(Å)</th>
<th>I/I_o</th>
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1. 100% purity, Johnson Matthey Chemical Ltd. CuKα radiation (λ = 1.54059Å), automated diffractometer.
2. PDF 4-554. 10 additional high angle reflections not reported.
Table 26, where they are compared with the properties of pure-element Te determined in this study. Note that the birefringence is distinct for native Te, whereas it is not noticeably distinct for the pure element. Micro-indentation hardness measurements give $V_{HN100} = 37.4 (35.9 - 38.5)$ for 5 indentations. Tellurium does not contain any noticeable amount of Sb, Pt, or Pd in solid solution.

5. Pt$_{34}$Sb$_7$

Bhan and Schubert (1969) reported Pt$_{4+}$Sb to be cubic, with $a = 3.99\AA$. Pt$_{4+}$Sb melts incongruently at 755°C to form Pt and liquid of composition 30 at.% Sb, according to the Pt-Sb phase diagram given by Bhan et al. (1969). No other data for this phase have been reported. A natural analogue of this phase is not known.

In this study a phase having a composition of Pt$_{83}$Sb$_{17}$ was produced at 600° and 650°C, and the chemical formula Pt$_{34}$Sb$_7$ was assigned to it. Microscopic examinations of the Pt$_{34}$Sb$_7$ phase coexisting with Pt or Pt$_3$Sb show that under reflected light it is pale brownish grey or yellowish grey in air and in oil, showing no birefringence. Micro-indentation hardness measurements gave $V_{HN100} = 225 (206 - 243)$ for 4 indentations. The Pt$_{34}$Sb$_7$ phase does not have any significant composition range nor solid solution. X-ray powder diffractometer data for this phase were not obtainable, because an attempt to synthesize a single-phased Pt$_{34}$Sb$_7$ was unsuccessful. The X-ray powder pattern shown
Table 28. X-ray powder diffraction data of synthetic \( \text{Pt}_{34}\text{Sb}_7 \) *.

<table>
<thead>
<tr>
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<th>k</th>
<th>l</th>
<th>( d_c (\text{Å}) )</th>
<th>( d_o (\text{Å}) )</th>
<th>( I/I_o )</th>
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<td>6</td>
<td>1.191</td>
<td>1.192</td>
<td>40</td>
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</tbody>
</table>

* X-ray powder pattern was obtained from a mixture of \( \text{Pt}_{34}\text{Sb}_7 + \text{Pt}_3\text{Sb} + \text{PtTe} \) (run 631) quenched from 600°C. CuKα radiation (\( \lambda = 1.5418\text{Å} \)), 114.6 mm Gandolfi camera.
in Table 28 was obtained from Pt$_{34}$Sb$_7$ coexisting with PtTe and Pt$_3$Sb (run 631), using a 114.6 mm diameter Gandolfi camera. The present X-ray powder pattern cannot be indexed according to the cubic cell with $a = 3.99$Å given by Bhan and Schubert (1969) for the Pt$_4$Sb phase. However, the X-ray powder data of Pt$_{34}$Sb$_7$ are indexable on a tetragonal cell with $a = 3.948(3)$ and $c = 16.85(1)$Å. Identity of Pt$_{34}$Sb$_7$ with Pt$_{4}$Sb (Bhan and Schubert, 1969; Bhan et al., 1969) and Pt$_4$Sb (Nemilow and Woronow, 1936) is not confirmed, because the X-ray powder patterns of the previously reported phases were not published.

6. Pt$_3$Sb

Pt$_3$Sb was reported to be tetragonal (ZrAl$_3$ structure), $a = 3.94$, $c = 16.96$Å (Bhan et al., 1969). However, its X-ray powder diffraction data have not been published. According to Bhan et al. (1969), it melts incongruently at 682°C, forming Pt$_4$Sb and liquid of composition about 30 at.% Sb. No other data for this phase have been found in the literature.

An attempt in this study to synthesize single-phase Pt$_3$Sb was unsuccessful. Pt$_3$Sb coexisting with Pt$_{34}$Sb$_7$ or with Pt$_3$Sb$_2$ is pale greyish yellow in air with very weak bireflectance. It is weakly anisotropic, ranging from brownish grey to greyish brown in air. Micro-indentation hardness tests gave VHN$_{25} = 216$ (183 - 240) for 3 measurements. Microprobe analyses of Pt$_3$Sb coexisting with Pt$_{34}$Sb$_7$ or Pt$_3$Sb$_2$ show that it is stoichiometric in
composition, with less than 1 at.% of Te in substitution for Sb at 600°C in the ternary system Pt-Sb-Te. The X-ray powder pattern shown in Table 29 was obtained from Pt₃Sb coexisting with Pt₃Sb₂, using a 114.6 mm diameter Gandolfi camera. The X-ray reflection lines are indexed on the basis of a tetragonal cell, with a = 3.9455(7), c = 16.959(5) Å. It is noted that there are 2 unindexable, weak reflections (2.482 and 2.450 Å) that apparently do not belong to Pt₃Sb₂. Bhan and Schubert (1969) also noted a few weak X-ray reflections (d values not specified) in their Guinier film that did not fit well to the ZrAl₃ structure. Bhan et al. (1969) reported that Pt₃Sb has a very weak X-ray line indexable on a tetragonal cell by doubling the a-axis. They suggested that this small discrepancy from the ZrAl₃ structure might have been resulted from slight distortion of the structure. However, it is not certain whether the problematic weak X-ray lines mentioned by Bhan and Schubert (1969) and Bhan et al. (1969) are identical to those found in the present study, because the earlier investigators have not specified the X-ray lines. Further investigation is needed to resolve this issue.

7. Pt₃Sb₂

Pt₃Sb₂ was reported to be orthorhombic, space group Ibam, with a = 6.44(6), b = 10.93(9), c = 5.31(9) Å for the phase quenched from 510°C (Bhan et al., 1969). Cabri et al. (1977) synthesized the same phase at about 675°C and
Table 29. X-ray powder diffraction data of synthetic Pt$_3$Sb.

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* This study. Run 103 (a mixture of 80% Pt$_3$Sb and 20% Pt$_3$Sb$_2$), quenched from 600°C. CuKα radiation ($\lambda = 1.5418$ Å), 114.6 mm Gandolfi camera. X-ray reflections indexed on the basis of a tetragonal cell (ZrAl$_3$ structure).
gave the cell parameters: \( a = 6.445(4), b = 10.947(4), c = 5.320(3) \text{Å} \). \( \text{Pt}_3\text{Sb}_2 \) melts incongruently at 732°C, forming \( \text{PtSb} \) and liquid of composition 33 - 34 at.% Sb (Bhan et al., 1969). The X-ray powder pattern of this phase given by Bhan et al. (1969) is shown in Table 30.

\( \text{Pt}_3\text{Sb}_2 \), synthesized at 600°C in this study, is pale brownish pink or creamy yellow in reflected light in air with a weak bireflectance from cream to pale greenish yellow. Anisotropism is moderate to strong in air from greyish brown to light bluish green, and strong to distinct in oil from pale yellow to greenish blue. The microindentation hardness value is \( \text{VHN}_{100} = 275 (240 - 306) \), based on 5 indentations. Microprobe analyses show that \( \text{Pt}_3\text{Sb}_2 \) is close to stoichiometric in composition. The substitution of Te for Sb in \( \text{Pt}_3\text{Sb}_2 \) is about 5 at.% at 600°C. The X-ray powder diffraction data of \( \text{Pt}_3\text{Sb}_2 \) quenched from 600°C (Table 30) do not agree well with those of Bhan et al. (1969), showing discrepancies in designations of the relative intensities and missing reflections which seem to be due to the different methods used in recording the data. The cell parameters refined in this study are \( a = 6.414(7), b = 10.941(6), c = 5.28(1) \text{Å} \).

No natural analogue of the \( \text{Pt}_3\text{Sb}_2 \) phase is known. Tarkian and Stumpfl (1975) reported an unnamed mineral \( (\text{Pt}_{1.76-1.78} \text{Pd}_{1.24-1.22})_3 \text{Sb}_{2.05-2.09} \) or \( \text{Pt}_{1.69} \text{Pd}_{1.18} \text{Sb}_{1.96} \text{Bi}_{0.06} \text{Sn}_{0.09} \text{Fe}_{0.01} \) from Driekop mine, South Africa, which was speculated by Cabri et al. (1977) as
Table 30. X-ray powder data of synthetic Pt₃Sb₂.

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</table>

  w: weak, m: medium, st: strong.

(2) Run 109, quenched from 600°C. CuKα radiation (λ = 1.54059 Å), automated diffractometer.
a possible Sn-bearing, Rh-free variety of genkinite on the basis of similarity in composition and reflectance at the 546 nm and 650 nm. They also pointed out considerable differences between the two, especially in reflectance values at 470 and 589 nm and in micro-indentation hardness values. However, the qualitative optical properties and VHN value of unnamed mineral of Tarkian and Stumpfl are close to those of the synthetic Pt3Sb2. Thus, the possibility that the unnamed mineral may be the natural analogue of the Pt3Sb2 phase cannot be totally dismissed.

8. PtSb

Synthetic PtSb was reported to be hexagonal (NiAs-type), space group P63/mmc, with a = 4.130(4), c = 5.472(5) Å (Thomassen, 1929a). PtSb melts at 1050°C (Friedrich and Leroux, 1909), 1040°C (Nemilow and Woronow, 1936), and 1043°C (Bhan et al., 1969). Neither optical nor micro-indentation hardness data for the phase have been reported.

The mineral corresponding to the synthetic PtSb was first found by Stumpfl (1961) from the Driekop mine, South Africa, and was named stumplite by Johan and Picot (1972). Mineralogical data for the stumplite are shown in Table 31. The crystal structure of stumplite was determined by Johan and Picot (1972) as hexagonal (NiAs-type) with a = 4.175, c = 5.504 Å.

PtSb was synthesized in this study and its optical, micro-indentation hardness, and X-ray data are given in Table 31. Qualitative optical properties of the synthetic
Table 31. Properties of synthetic PtSb and stumplite.

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<thead>
<tr>
<th></th>
<th>synthetic PtSb(1)</th>
<th>synthetic PtSb</th>
<th>stumplite(6)</th>
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</thead>
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<td>Colour</td>
<td>cream in air</td>
<td></td>
<td>cream in air</td>
</tr>
<tr>
<td>Bireflectance/pleochroism</td>
<td>weak</td>
<td></td>
<td>weak</td>
</tr>
<tr>
<td>Anisotropism</td>
<td>strong</td>
<td></td>
<td>strong</td>
</tr>
<tr>
<td>Polishing characteristic</td>
<td>takes a good polish</td>
<td></td>
<td>takes a good polish</td>
</tr>
<tr>
<td>Micro-indentation VHN$_{50}$ = 417 (296-473) for hardness, 5 measurements,</td>
<td></td>
<td>VHN$_{50}$ = 385</td>
<td></td>
</tr>
<tr>
<td>Mean (range) VHN$_{100}$ = 424 (376-464) for 4 measurements</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Melting point</td>
<td>1050°C(2), 1040°C(3), 1043°C(4)</td>
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<tr>
<td>Cell parameters</td>
<td>$a = 4.1318(6), c = 5.483(1)$Å for PtSb quenched from 1000°C</td>
<td>$a = 4.130(4)$, $c = 5.472(5)$Å(5) for PtSb slowly cooled to room temperature from 600°C(5)</td>
<td>$a = 4.175, c = 5.504$Å</td>
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</table>

<table>
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<th>d_0 (Å)</th>
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<td>1.0535</td>
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1. This study. Run 122, quenched from 1000° C. CuKα radiation (λ=1.54059Å), automated diffractometer.
2. This study. Run 110, quenched from 800° C. CuKα radiation (λ=1.54059Å), automated diffractometer.
3. Thomsen (1929b). FeKα radiation (λ=1.934Å), 57.65 mm diameter camera.
4. PIF 25-1482.
PtSb and stumpflite are virtually identical. The microhardness value (VHN$_{50}$ = 385) of stumpflite is well in the range of that measured from the synthetic compound (Table 31). Microprobe data for the phase indicate that it is stoichiometric at least up to 800°C and has a slight deficiency of Pt (about 1 at.% at 1000°C). The cell parameters of the pure PtSb phase vary slightly depending on temperatures from which the phase was quenched: \( a = 4.1318(6) \text{Å} \), \( c = 5.483(1) \text{Å} \) (800°C), and \( a = 4.1420(5) \text{Å} \), \( c = 5.486(1) \text{Å} \) (1000°C). Te substitution for Sb in PtSb is restricted to 2 at.% at 600°C and may reach about 10 at.% at 800°C and 6 at.% at 1000°C, whereas Pd substitution for Pt is about 23 at.% at 800°C and decreases to 9 at.% at 1000°C. The refined cell parameters are \( a = 4.1435(3) \text{Å} \), \( c = 5.4888(8) \text{Å} \) for Pt$_{1.00}$Sb$_{0.93}$Te$_{0.07}$ quenched from 800°C and \( a = 4.1329(3) \text{Å} \), \( c = 5.4793(4) \text{Å} \) for Pt$_{0.98}$Sb$_{0.96}$Te$_{0.06}$ quenched from 1000°C. X-ray powder diffraction data for the pure PtSb phase quenched from 1000°C and 800°C and for stumpflite are shown in Table 32.

9. PtSb$_2$

Synthetic PtSb$_2$, first found by Roessler (1895), was reported to be cubic (pyrite-type), space group Pa3, with \( a = 6.428(3) \text{Å} \) (Thomassen, 1929a). A more reliable value of \( a = 6.4400(4) \text{Å} \) was later given by Kjekshus (1960). Melting points of PtSb$_2$ were reported to be 1226°C (Friedrich and Leroux, 1909), 1230°C (Thomassen, 1929a), and 1210°C...
Table 33. Properties of synthetic PtSb$_2$ and geversite.

<table>
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<tr>
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<th>synthetic PtSb$_2$</th>
<th>geversite</th>
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<td></td>
<td>nil$^5$</td>
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<tr>
<td>Anisotropism</td>
<td>isotropic</td>
<td></td>
<td>isotropic$^5$</td>
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<tr>
<td>Polishing characteristic</td>
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<td></td>
<td>takes a good polish$^5$</td>
</tr>
<tr>
<td>Micro-indentation hardness, mean (range)</td>
<td>VHN$<em>{50}$ = 663.5 (566-766) for 4 indentations, VHN$</em>{100}$ = 701 (681-724) for 4 indentations</td>
<td></td>
<td>VHN$<em>{50}$ = 726-766$^5$, VHN$</em>{50}$ = 435$^7$</td>
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<tr>
<td>Melting point</td>
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<td></td>
<td>1230°C$^1$, 1210°C$^2$, 1226°C$^3$</td>
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<td>a = 6.428(3)Å$^1$</td>
<td>a = 6.440Å$^5$</td>
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Table 34. X-ray powder diffraction data of synthetic PtSb₂.

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<th>PtSb₁.₄₉Te₀.₅₁</th>
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<th>d_o (Å)</th>
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<td>0.9888 0.9889 13</td>
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(1) This study. Run 111, quenched from 1000°C. CuKα, radiation (λ = 1.54059Å), automated diffractometer.

(2) PDF 14-141.

(3) This study. Run 452, quenched from 1000°C. CuKα, radiation (λ = 1.54059Å), automated diffractometer.
(Nemilow and Woronow, 1936). No optical properties for the phase have been reported.

Geversite, the natural analogue of synthetic PtSb$_2$, was found from the Driekop mine, South Africa by Stumpfi (1961). Optical data of geversite are given in Table 33. Johan and Picot (1972) gave the micro-indentation hardness for geversite as VHN$_{50} = 435$, but later Tarkian and Stumpfi (1975) gave a considerably higher value of VHN$_{50} = 726 - 766$. No X-ray powder pattern of geversite has been published.

PtSb$_2$ was synthesized at 600 - 1000°C in the present study. Qualitative optical data for the phase observed in this study compare well with those of geversite (Table 33). Micro-indentation hardness for synthetic PtSb$_2$ is VHN$_{50} = 664$ (566 - 766) and VHN$_{100} = 701$ (681 - 724), which compare closely with that of geversite given by Tarkian and Stumpfi (1975). A single-phased stoichiometric PtSb$_2$ quenched from 1000°C has a = 6.4373(2)Å (Table 34). Te substitution for Sb in PtSb$_2$ may reach 27 - 28.5 at.% in the temperature range 600 - 1000°C and Pd substitution for Pt is 18 at.% at 800°C and 7.2 at.% at 1000°C. The cell dimension increases significantly with the amount of Te in substitution for Sb, as evidenced by a = 6.4844(9)Å for Pt$_{1.00}$Sb$_{1.49}$Te$_{0.51}$ quenched from 1000°C.

10. PtTe

Gronvold et al. (1960) first provided X-ray powder diffraction data for synthetic PtTe and indexed the X-ray
Table 35. X-ray powder data of synthetic PtTe.

<table>
<thead>
<tr>
<th>hk l</th>
<th>d_c(Å)</th>
<th>d_o(Å)</th>
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<th>I/I_o</th>
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<td>1.900</td>
<td>w+</td>
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<td>9</td>
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<td>10</td>
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<tr>
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<td>1.0984</td>
<td>1.0984</td>
<td>15</td>
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</tbody>
</table>

(1) Run 82, quenched from 650 °C. CuKα radiation (λ = 1.54059Å), automated diffractometer.

reflections on an orthorhombic cell with \( a = 6.6144 \), \( b = 5.6360 \), and \( c = 11.865\) Å. They also suggested that a possible phase transformation from hexagonal (NiAs structure) to orthorhombic structure might exist because crystals with platy and hexagonal habit, formed at near the melting point, disintegrated to a powder after slow cooling to room temperature for some time. Bhan et al. (1969) determined the crystal structure of PtTe, using single crystals synthesized at 830°C. The compound is monoclinic, space group \( \text{C}2/\text{m} \), with \( a = 6.865\) Å, \( b = 3.962\), \( c = 7.044\) Å, \( \beta = 108.98^\circ \). The PtTe phase was reported to be formed by peritectic reaction of PtTe\(_2\) and liquid at 920°C (Gimpl et al., 1963a) and at 935°C (Bhan et al., 1969). Gimpl et al. (1963a) reported the PtTe phase to be strongly anisotropic, and gave a micro-indentation hardness value of 55 for a 100g load.

PtTe was synthesized at 650°C in this study. Under reflected light it is yellowish grey in air and pale brownish grey in oil. The bireflectance varies from nil to very weak. It is moderately anisotropic, from brownish grey to light bluish grey in air and enhanced in oil. The micro-indentation hardness value is \( \text{VHN}_{15} = 63.1 \) (50.4 - 85.9) for 4 indentations. The PtTe phase is stoichiometric and does not show any compositional range in the Pt-Te binary system. In the Pt-Te-Sb ternary system it may contain about 2 at.% of Sb in substitution for Te in the temperature range 600 - 800°C. The X-ray powder diffraction pattern of the PtTe phase (Table 35) is
comparable to that given by Bhan et al. (1969). The X-ray
powder data were indexed on the basis of a monoclinic cell
and refined by a least-squares method to give cell
parameters $a = 6.857(3)$, $b = 3.952(4)$, $c = 6.988(3)\text{Å}$, $\beta = 108.99(8)^\circ$.

11. Pt$_3$Te$_4$

Bhan et al. (1969) reported Pt$_3$Te$_4$ to be monoclinic,
space group $C2/m$, with $a = 6.906$, $b = 3.991$, $c = 12.019\text{Å}$,
$\beta = 101.03^\circ$. According to the Pt–Te phase diagram (Bhan et
al., 1969), Pt$_3$Te$_4$ has a compositional range of
approximately 2 at.% and melts at 985°C to form PtTe$_2$ and
liquid of composition of about 49 at.% Te. Neither optical
data nor microhardness value of the phase has been reported.

Under reflected light, the Pt$_3$Te$_4$ synthesized in this
study is pale yellow in air, pale yellow with a pink tinge
in oil, and is not birefringent. Anisotropism is moderate
from reddish grey to yellowish grey both in air and oil.
Since the grain size of the synthetic Pt$_3$Te$_4$ phase is small,
its micro-indentation hardness was determined using a 15g
load. The VHN$_{15}$ values for 4 measurements range from 68.2
to 80.4, with an average of 73.2.

Electron microprobe analyses of Pt$_3$Te$_4$ indicate that it
has a compositional range of 56.8 – 57.5 at.% Te. Sb
substitutions for Te in Pt$_3$Te$_4$ are 1.7 at.% at 1000°C, 5.2
at.% at 800°C, and 3.5 at.% at 600°C.

Because the Pt$_3$Te$_4$ phase is stable at 1000°C, the
melting point of the phase must be higher than 985°C given
Table 55. X-ray powder data of synthetic $\text{Pt}_2\text{Te}_4$.

<table>
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<tr>
<th></th>
<th>$\text{Pt}_2\text{Te}_4$</th>
<th>$\text{Pt}_2\text{Te}_4$ (This study)</th>
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<tr>
<td></td>
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<td>(2)</td>
</tr>
<tr>
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<td>$a = 6.90_{2}$, $b = 3.99_{1}$</td>
<td>$a = 6.942_{4}$, $b = 3.980_{2}$</td>
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<tr>
<td></td>
<td>$c = 12.01_{2}$, $\beta = 101.03^\circ$</td>
<td>$c = 12.00_{1}$, $\beta = 100.4_{1}$</td>
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</table>

<table>
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<tr>
<th>h k l</th>
<th>$d_o (\AA)$</th>
<th>$I/I_o$</th>
<th>$d_o (\AA)$</th>
<th>$d_o (\AA)$</th>
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<tr>
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<td></td>
<td></td>
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<tr>
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</table>

D-values converted from ϑ-values. w: weak. m: medium.
s t: strong.

(2) Run 87, quenched from 800°C. CuKα radiation ($λ = 1.540591\AA$), automated diffractometer.
by Bhan et al. (1969). The X-ray powder pattern of $\text{Pt}_3\text{Te}_4$
quenched from 800°C (Table 36) are comparable to that of Bhan et al. (1969), except for differences in the relative intensities which seem to be due to the use of different recording methods. The refined cell dimensions are $a = 6.942(4)$, $b = 3.980(2)$, $c = 12.00(1)$Å, $\beta = 100.4(1)^\circ$; were obtained using the present X-ray powder data and indices assigned by Bhan et al.

12. $\text{Pt}_2\text{Te}_3$

Bhan et al. (1969) determined the crystal structure of $\text{Pt}_2\text{Te}_3$ as monoclinic, space group C2/m, with $a = 6.933$, $b = 4.002$, $c = 17.119$Å, $\beta = 97.75^\circ$. According to the phase diagram given by Bhan et al. (1969), $\text{Pt}_2\text{Te}_3$ decomposes at 875°C to form $\text{Pt}_3\text{Te}_4$ and $\text{PtTe}_2$. No physical properties of this phase have been reported, and the natural equivalent of this phase is not known.

Attempts in this study to synthesize a single-phase $\text{Pt}_2\text{Te}_3$ at 600°C and 800°C were unsuccessful. Under reflected light the $\text{Pt}_2\text{Te}_3$ phase coexisting with $\text{PtTe}_2$ or with $\text{Pt}_3\text{Te}_4$ is white with a yellow tinge in air. No bireflectance is observed in air. It is weakly anisotropic, from light greenish grey to reddish grey. Due to the fine grain size, the micro-indentation hardness was measured with a 10g load, resulting in a VHN value of 62.7 for a single indentation. The $\text{Pt}_2\text{Te}_3$ phase exists at 650°C, but is not stable between 700 - 1000°C, suggesting
Table 37. X-ray powder data of synthetic Pt₄Te₃.

<table>
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<th>k</th>
<th>l</th>
<th>dₒ (Å)</th>
<th>I/Iₒ</th>
<th>dₓ (Å)</th>
<th>dᵧ (Å)</th>
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(2) Run 94, quenched from 800°C. CuKα radiation (λ = 1.54059 Å), automated diffractometer.
that the decomposition temperature is somewhere between 650°C and 700°C. However, Pt₂Te₃ containing about 1 at.% Sb is confirmed at 800°C in the Pt-Sb-Te system. Pt₂Te₃ has a compositional range of 60.1 - 67.6 at.% Te at 650°C in the binary system. The substitution of Sb for Te is about 6.7 at.% at 800°C and 4.3 at.% at 600°C. The X-ray powder pattern of Pt₂Te₃ quenched from 800°C is shown in Table 37, and is comparable to that given by Bhan et al. (1969). Using the present X-ray powder data and the indices assigned by Bhan et al. (1969), the cell dimensions refined by a least-squares method are a = 6.910(1), b = 3.985(1), c = 17.115(3)Å, β = 97.82(4)°.

13. PtTe₂

Thomassen (1929a) reported PtTe₂ as hexagonal (CdI₂ structure), space group P6₃mc, with a = 4.010(4), c = 5.201(5)Å. This was later confirmed by Groeneveld Meijer (1955). Cell parameters of the phase were re-determined by Groenveld et al. (1960) as a = 4.0259 and c = 5.2209Å. Groeneveld Meijer (1955) reported that PtTe₂ is white and non-birefringent in reflected light and moderately anisotropic.

Moncheite, (Pt,Pd)(Te,Bi)₂ from the Monchegorsk deposits, U.S.S.R. and first described by Genkin et al. (1963) is the natural equivalent of the synthetic PtTe₂ phase. Moncheite has since been reported from many localities such as Sudbury, Ontario (Cabri and Laflamme, 1976) and Stillwater complex, Montana (Cabri et al., 1979)
Table 38. Properties of synthetic PtTe$_2$ and moncheite.

<table>
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<tr>
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<th>PtTe$_2$ (This study)</th>
<th>PtTe$_2$</th>
<th>moncheite</th>
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<tr>
<td><strong>Colour</strong></td>
<td>white with yellowish tinge</td>
<td>white$^{(1)}$</td>
<td>bright greyish or white$^{(6)}$</td>
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<tr>
<td><strong>Bireflectance/pleochroism</strong></td>
<td>nil to very weak in air. and oil</td>
<td>nil$^{(1)}$</td>
<td>weak in air, distinct in oil$^{(6)}$</td>
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<tr>
<td><strong>Anisotropism</strong></td>
<td>strong pinkish grey to extinction, brownish grey to dark grey</td>
<td>moderate$^{(1)}$</td>
<td>strong: light yellowish brown to dark brown in air$^{(6)}$</td>
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<tr>
<td><strong>Polishing characteristic</strong></td>
<td>vary from good to fair probably depending on orientation of crystals</td>
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<td></td>
</tr>
<tr>
<td><strong>Micro-indentation hardness, mean (range)</strong></td>
<td>VHN$<em>{10} = 84.2$ (76.2-90.7) and VHN$</em>{100} = 50.7$ (44.7-54.9) on clean surface, VHN$<em>{10} = 83.2$ (70.7-92.0) and VHN$</em>{100} = 66.8$ on pitted surface</td>
<td>VHN$_{10} = 92$ (73-111)$^{(7)}$</td>
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</tr>
<tr>
<td><strong>Melting point</strong></td>
<td>1100°C$^{(2)}$, 1150°C$^{(3)}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Symmetry</strong></td>
<td>Hexagonal, P6$_3$mc$^{(4)}$</td>
<td>Hexagonal$^{(4)}$</td>
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<tr>
<td><strong>Cell parameters</strong></td>
<td>a = 4.025(1), c = 5.222(1)Å for PtTe$_2$ quenched from 1000°C. a = 4.024(4), c = 5.2207(4)Å for PtTe$_2$ quenched from 800°C.</td>
<td>a = 4.010(4), c = 5.201(5)Å$^{(4)}$, a = 4.0259, c = 5.2209Å$^{(6)}$</td>
<td>a = 4.049(4), c = 5.288(5)Å$^{(8)}$</td>
</tr>
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Table 39. X-ray powder data of synthetic PtTe$_2$ and moncheite.

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<th>PtTe$_2$</th>
<th>PtTe$_2$</th>
<th>moncheite$^4$</th>
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<td>$a = 4.0259$</td>
<td>$a = 4.010(4)$</td>
<td>$a = 4.049$</td>
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<td>$c = 5.2207(4)\text{Å}$</td>
<td>$c = 5.2209\text{Å}$</td>
<td>$c = 5.201(5)\text{Å}$</td>
<td>$c = 5.288\text{Å}$</td>
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<td>$d_o(\text{Å})$</td>
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1. This study. Run 100, quenched from 800°C. CuK$\alpha$ radiation ($\lambda = 1.54059\text{Å}$), automated diffractometer.
2. Grønvold et al. (1960). CuK$\alpha$ radiation ($\lambda = 1.54059\text{Å}$), 114.6 mm diameter camera. 18 additional high angle reflections not reported.
3. Thomassen (1929a). FeK$\alpha$ radiation ($\lambda = 1.934\text{Å}$), 57.65 mm diameter camera.
4. PDF 15-392. Pt$_{0.63-0.79}$Pd$_{0.36-0.21}$Te$_{1.34-1.82}$Bi$_{0.66-0.18}$. 

and is one of a few well-characterized platinum group minerals.

The qualitative optical properties of the phase, synthesized in this study, are in good agreement to those of moncheite, as shown in Table 38. The polished surface of the synthetic phase is either unpitted or pitted, probably depending on the orientation of the grain. Micro-indentation hardness tests gave poor diamond imprints, accompanied by sets of parallel fractures, similar to those described for moncheite by Cabri and Laflamme (1976). Micro-indentation hardness values for a 10g load of the synthetic PtTe₂ compare closely with those for moncheite given by Cabri and Laflamme (1976). PtTe₂ shows substitution of Sb for Te from 15 - 16.5 at.% at 600°C, 19.5 at.% at 800°C, to 28.5 at.% at 1000°C. The cell parameters of the phase quenched from 1000°C and 800°C, refined by a least-squares method using X-ray powder diffraction data, are respectively a = 4.025(1), c = 5.222(1)Å and a = 4.0242(4), c = 5.2207(4)Å, in good agreement with those given by Grønvold et al. (1960). Sb substitution for Te causes the cell parameters to become smaller, as evidenced by a = 4.018(1), c = 5.175(1)Å for PtTe₁.53Sb₀.47 (run 453, 1000°C) and a = 4.022(1), c = 5.1790(8)Å for Pt₀.98Te₁.65Sb₀.37 (run 335, 800°C). X-ray powder diffraction data for PtTe₂ quenched from 800°C are compared with those of other authors and those for moncheite in Table 39.
14. Pd$_{20}$Sb$_7$ - Pd$_{20}$Te$_7$

The phases Pd$_{20}$Sb$_7$ and Pd$_{20}$Te$_7$ are closely related, structurally and chemically, and are therefore treated under the same heading. Wopersnow and Schubert (1977) determined the structure of Pd$_{20}$Sb$_7$ to be rhombohedral, space group R3, with $a = 11.734(1)$, $c = 11.021(2)$ Å. The melting point of the Pd$_3$Sb phase was reported to be 1220°C (Sander, 1912) and 1185°C (Grigorjew, 1932). No further data for this phase have been published. A natural analogue of synthetic Pd$_{20}$Sb$_7$ is not yet known. Although the formula Pd$_3$Sb was originally given for stibiopalladinite by Adam (1927), it was later revised to Pd$_{5+x}$Sb$_{2-x}$ ($x \approx 0.05$) by Gabri and Chen (1976).

The Pd$_{20}$Sb$_7$ phase synthesized in the present study has a compositional range of 74.0 - 74.5 at.% Pd between 510° and 1000°C. Under reflected light, Pd$_{20}$Sb$_7$ is yellowish white in air and creamy yellow in oil, showing no bireflectance. Anisotropism is very weak in air, brownish grey to bluish grey, and it is slightly enhanced in oil. Micro-indentation hardness measurements gave VHN$_{100}$ = 516 (488 - 548) for 5 indentations.

The extent of Pt substitution for Pd in Pd$_{20}$Sb$_7$ is at least 18.4 at.% at 800°C and increased to 27 at.% at 1000°C. Te substitutes for Sb up to 42 at.% at 1000°C.

X-ray powder diffraction patterns of the Pd$_{20}$Sb$_7$ phase, quenched from 1000°, 800°, and 600°C, were obtained and indexed on hexagonal axes according to Wopersnow and
Table 40. X-ray powder diffraction data of Pd$_{20}$Sb$_7$.

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(1) Wert automatic diffractometer.

(2) Run at 0°C, CuKα radiation, Guinier camera.

v: weak, m: medium, s: strong.
### Table 41. Cell dimensions of Pd$_{20}$Sb$_7$ - Pd$_{20}$Te$_7$ compounds

<table>
<thead>
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<th>Run no.</th>
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* CuK$_α$, radiation (λ = 1.54059Å), automated diffractometer.

The X-ray powder diffraction data for the intermediate phase from which the cell parameters were derived are given in Appendix II.

** Quenched from different annealing temperatures.
Schubert (1977). The refined cell dimensions are shown in Table 40. The X-ray powder data of Pd$_{20}$Sb$_7$ synthesized at 800°C in this study agree with those of Wopernsnow and Schubert (1977) except for some discrepancies in the designations of the relative intensities which may be due to the different methods used in recording the data (Table 41).

Wopernsnow and Schubert (1977) reported Pd$_{20}$Te$_7$ as rhombohedral, space group R3, with $a = 11.797(1)$, $c = 11.172(2)$Å. No further data for this phase have been reported.

Pd$_{20}$Te$_7$ was synthesized between 600°C and 800°C. DTA data showed that Pd$_{20}$Te$_7$ undergoes a polymorphic transformation at 745°C before it melts at 885°C. The high-temperature structure is apparently not quenchable, as the phases quenched from temperatures above and below the transition point gave identical X-ray diffraction patterns. The X-ray powder pattern of Pd$_{20}$Te$_7$ is indexed according to Wopernsnow and Schubert (1977) on hexagonal axes. The refined cell parameters are $a = 11.797(3)$, $c = 11.150(4)$Å, in good agreement with those reported by Wopernsnow and Schubert (1977). X-ray powder patterns are given in Table 42.

Under reflected light Pd$_{20}$Te$_7$ is cream in air and in oil, and is non-bireflectant. Anisotropism varies from weak (dark grey to extinction) to strong (orange brown to dark blue) in air and in oil. Micro-indentation hardness measurements gave VHN$_{100}$ = 515 (488 - 548) for 5 indentations.

Electron microprobe analysis and X-ray diffraction of Pd$_{20}$(Sb,Te)$_7$ solid solutions in run products #1004 - #1006
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<th>d_c(Å)</th>
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\( v \): weak, \( m \): medium, \( s \): strong.

(2) Run 78, quenched from 800°C. CuKα radiation (\( \lambda = 1.54059\) Å), automated diffractometer.
were done to check the possibility of a relationship between the cell dimensions and the composition of the phase. As shown in Table 41, it is apparent that the cell parameters, both a and c, increase as the composition of the phase becomes closer to the Pd$_{20}$Te$_7$ end-members.

Keithconnite, Pd$_{3-x}$Te (0.14 < x < 0.43), was discovered from Stillwater complex, Montana by Cabri et al. (1979). Keithconnite was reported to be rhombohedral, space group R3, with a = 11.45(1), c = 11.40(1)Å. Cabri et al. (1979) also reported that they were unable to synthesize keithconnite.

Keithconnite is compositionally close to Pd$_{20}$Te$_7$ and Pd$_8$Te$_3$. The X-ray powder pattern of keithconnite given by Cabri et al. (1979) is similar to that of Pd$_{20}$Te$_7$, but different from that of Pd$_8$Te$_3$. Although some similarities exist between keithconnite and Pd$_{20}$Te$_7$, as indicated by Cabri et al. (1979), such as optical properties, a few reflections (e.g. 2.74, 1.87, 1.65, 1.51Å) of keithconnite cannot be correlated with Pd$_{20}$Te$_7$ as indexed in Table 43. Confirmation of equivalence of keithconnite with any Pd–Te synthetic phase requires better X-ray and micoprobe data for keithconnite.

15. Pd$_8$Sb$_3$

Pd$_8$Sb$_3$ was reported to be rhombohedral, with a = 7.57(9), c = 42.80(3)Å (El-Boragy and Schubert, 1971a). Cabri et al. (1975) confirmed the phase and determined the
cell dimensions of their synthetic \( \text{Pd}_8\text{Sb}_3 \) to be \( a = 7.60(1), c = 42.904(1) \text{Å} \), which are in good agreement with those reported by El-Boragy and Schubert (1971a). Wopersnow and Schubert (1976) determined the space group of the \( \text{Pd}_8\text{Sb}_3 \) to be \( \text{R3c} \), and gave the cell dimensions as \( a = 7.6152(7), c = 43.032(7) \text{Å} \). Micro-indentation hardness was reported by Cabri et al. (1975) to be \( \text{VHN}_{50} = 516 \) (501 - 545).

The group II mertieite (mertieite II) from Goodnews Bay, Alaska described by Desborough et al. (1973), is now generally accepted as the natural analogue of the synthetic \( \text{Pd}_8\text{Sb}_3 \) phase (Cabri, 1981a). Mertieite II was originally thought to have the approximate formula \( (\text{Pd}, \text{Cu})_{5+x} \text{(Sb, As)}_2 \text{Sb}_3 \) \( x \), where \( x = 0.1 - 0.2 \). Cabri et al. (1975) restudied two grains of the type material and found mertieite II to be stoichiometric \( \text{Pd}_8 \text{(Sb, As)}_3 \). They also showed by X-ray single crystal and powder diffraction work that the mineral is rhombohedral, with \( a = 7.546(2), c = 43.15(1) \text{Å} \). The cell parameters and the X-ray powder diffraction data of mertieite II are in good agreement with those reported for the synthetic \( \text{Pd}_8\text{Sb}_3 \) (Table 43).

\( \text{Pd}_8\text{Sb}_3 \) was synthesized in this study between 600° and 1900°C. The optical properties of synthetic \( \text{Pd}_8\text{Sb}_3 \) are essentially identical to those of mertieite II (Table 44). However, the micro-indentation hardness value of the synthetic \( \text{Pd}_8\text{Sb}_3 \) is significantly lower than that of the synthetic \( \text{Pd}_8\text{Sb}_3 \) of Cabri et al. (1975) and mertieite II (Cabri, 1981a). The amount of Pt in substitution for Pd
Table 43. Cell parameters of synthetic Pd₈Sb₃ and mertieite II.

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<th>Compound</th>
<th>a(Å)</th>
<th>c(Å)</th>
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(1) Run 44, quenched from 600°C. CuKα₁ radiation (λ = 1.54059Å), automated diffractometer.

(2) Run 60-3, quenched from 1000°C. CuKα₁ radiation (λ = 1.54059Å), automated diffractometer.

(3) Run 579, quenched from 1000°C. CuKα₁ radiation (λ = 1.54059Å), automated diffractometer.

(4) Run 948, quenched from 1000°C. CuKα₁ radiation (λ = 1.54059Å), automated diffractometer.
Table 44. Properties of synthetic Pd₈Sb₃ and mertieite II.

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<th>Pd₈Sb₃</th>
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<td>moderate(1), distinct: dark brownish grey to extinction(4)</td>
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<td>takes a good polish</td>
</tr>
<tr>
<td>Micro-indentation hardness, mean (range)</td>
<td>VHN₅₀ = 324 (241 - 349) for four indentations.</td>
<td>VHN₅₀ = 516 (501 - 545) for ten indentations(1)</td>
<td>VHN₅₀ = 544 (511 - 588)(4)</td>
</tr>
<tr>
<td>Symmetry</td>
<td></td>
<td></td>
<td>Rhombohedral, R3c(2)</td>
</tr>
<tr>
<td>Cell parameters</td>
<td>a=7.604(1), c=42.943(4)Å</td>
<td>a=7.6152(7), c=43.032(7)Å(2)</td>
<td>Rhombohedral(1)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>a=7.579, c=42.803Å(3)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>a=7.60(1), c=42.904(1)Å(1)</td>
<td></td>
</tr>
</tbody>
</table>

Table 45. X-ray powder diffraction data of synthetic \( \text{Fe}_2\text{SiO}_4 \) and merrillite II.

<table>
<thead>
<tr>
<th>( \text{Fe}_2\text{SiO}_4 )</th>
<th>( \text{Fe}_2\text{SiO}_4 )</th>
<th>merrillite II</th>
<th>( \text{Fe}_2\text{SiO}_4 )</th>
<th>( \text{Fe}_2\text{SiO}_4 )</th>
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<tbody>
<tr>
<td>( \text{h k l} )</td>
<td>( d_h )</td>
<td>( \sigma_h )</td>
<td>( d_k )</td>
<td>( \sigma_k )</td>
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<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
</tr>
<tr>
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<td>4.027</td>
<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
</tr>
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<td>1.486</td>
<td>6.143</td>
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<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
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<td>1.486</td>
<td>6.143</td>
</tr>
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<td>1.486</td>
<td>6.143</td>
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<td>1.486</td>
<td>6.143</td>
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<td>1.486</td>
<td>6.143</td>
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<td>1.486</td>
<td>6.143</td>
</tr>
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<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
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<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
</tr>
<tr>
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<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
</tr>
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<td>1.486</td>
<td>6.143</td>
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<td>4.116</td>
<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
</tr>
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<td>1.486</td>
<td>6.143</td>
</tr>
<tr>
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<td>4.116</td>
<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
</tr>
<tr>
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<td>4.116</td>
<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
</tr>
<tr>
<td>1 1 1</td>
<td>4.116</td>
<td>1.137</td>
<td>1.486</td>
<td>6.143</td>
</tr>
</tbody>
</table>

(1) Wagner and Schubert (1976). \( \text{Fe}_2\text{SiO}_4 \), synthesized from 725°C, \( \theta \) = 7.45°(4), \( \sqrt{a} = 3.205(3) \). Cu diffracted.

Goniometer camera. \( \text{h}-\text{values converted to \( \theta \)-values. w; weak; m; medium; s; strong}.\)

(2) From \( 400°C \) (\( \text{Fe}_2\text{SiO}_4 \), \( \theta \) = 7.46°(4), \( \sqrt{a} = 3.207(3) \). Cu diffracted, \( \omega \) = 1.56°(4), automated diffractometer.

(3) Cahn et al. (1975). \( \text{Fe}_2\text{SiO}_4 \), \( \theta \) = 7.46°(4), \( \sqrt{a} = 3.210(3) \). Cu diffracted, 12.5 cm Gandolfi camera.
in Pd₈Sb₃ may reach 44.7 at.% at 800°C and 22 at.% at 1000°C. Te may substitute for Sb up to 36 - 38 at.% at 1000°C.

The X-ray powder diffraction pattern of Pd₈Sb₃ quenched from 600°C is compared with those given by other authors in Table 45. The cell parameters of the Pd₈Sb₃ phases synthesized in this study were refined by a least-squares method using powder diffraction data indexed on hexagonal axes. The refined cell parameters of these phases are well within the range of those reported by other authors for synthetic Pd₈Sb₃ and mertieite II (Table 43). The data in Table 43 show that the temperature at which the phase is synthesized has little effect on the cell parameters, whereas substitutions of Pt for Pd and Te for Sb affects the c-parameter only. Mertieite II has a smaller a-parameter and a larger c-parameter as compared with the synthetic phases, perhaps due to compositional difference.

16. Pd₃₁Sb₁₂

According to El-Boragy et al. (1970), Pd₃₁Sb₁₂ is rhombohedral with a = 7.613, c = 42.240Å, structurally related to Pd₅Sb₂ which is also rhombohedral with a = 7.606 and c = 13.863Å. The c parameter of Pd₃₁Sb₁₂ is approximately three times of that of Pd₅Sb₂. The X-ray powder diffraction patterns of Pd₃₁Sb₁₂ and Pd₅Sb₂ are also quite similar. Optical and physical data of Pd₃₁Sb₁₂ are nonexistent in the literature.
Pd$_{31}$Sb$_{12}$ was synthesized in this study at 800°C and its X-ray powder diffraction pattern is essentially identical to that of Pd$_5$Sb$_2$ given by El-Boragy et al., except for minor discrepancies in designating relative intensities and d-values of some lines (Table 46). The refined cell parameters are a = 7.6006(1) and c = 41.945(8)Å, in good agreement with those given by El-Boragy et al. (1970). The compositional range of this phase given by Wopersnow and Schubert (1977) was not confirmed in this study. The atomic substitution of Pt for Pd and Te for Sb in Pd$_{31}$Sb$_{12}$ may reach 22 at.% and 38.3 at.% respectively at 1000°C.

Under reflected light Pd$_{31}$Sb$_{12}$ is yellowish white in air and pale yellow in oil, showing no bireflectance. It is moderately anisotropic from brownish grey to greenish grey in air and in oil. Micro-indentation hardness measurements give VHN$_{50}$ = 467 (423 - 494) and VHN$_{100}$ = 463 (437 - 498) for four indentations each.

The mineral stibiopalladinite is closely related to the synthetic Pd$_{31}$Sb$_{12}$ phase. Stibiopalladinite was originally thought to be Pd$_5$Sb but later analysis (DesBorough et al., 1973; Clark et al., 1974) on material from Potgietersrust, South Africa suggested the formula to be more accurately Pd$_5$Sb$_2$. Subsequent detailed study by Cabri and Chen (1976) of the mineral from the locality of its original description, with electron microprobe analysis using synthetic standards, showed the composition of the mineral to be either Pd$_{5+x}$Sb$_{2-x}$ with x = 0.05 or Pd$_{8-x}$Sb$_{3+x}$ with x = 0.065. They chose the formula Pd$_{5+x}$Sb$_{2-x}$ because of its
Table 46. X-ray powder diffraction data of synthetic \( \text{Ba}_2\text{Br}_2 \text{Cl}_2 \) and stibiopalladatite.

<table>
<thead>
<tr>
<th>( \text{Ba}_2\text{Br}_2\text{Cl}_2 )</th>
<th>( \text{Ba}_2\text{Br}_2\text{Cl}_2 ) (This study)</th>
<th>( \text{Stibiopalladatite} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( a=7.41 )</td>
<td>( a=7.406 \pm 0.006 )</td>
<td>( a=7.398 \pm 0.002 )</td>
</tr>
<tr>
<td>( c=2.19 \pm 0.01 )</td>
<td>( c=2.18 \pm 0.01 )</td>
<td>( c=2.18 \pm 0.01 )</td>
</tr>
<tr>
<td>( \lambda_{\text{CuK} \alpha} = 1.5405 )</td>
<td>( \lambda_{\text{CuK} \alpha} = 1.5405 )</td>
<td>( \lambda_{\text{CuK} \alpha} = 1.5405 )</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>( d_i )</th>
<th>( \lambda_i )</th>
<th>( I_i )</th>
<th>( h_i )</th>
<th>( k_i )</th>
<th>( l_i )</th>
<th>( d_i )</th>
<th>( \lambda_i )</th>
<th>( I_i )</th>
<th>( h_i )</th>
<th>( k_i )</th>
<th>( l_i )</th>
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</thead>
<tbody>
<tr>
<td>0 0 2</td>
<td>3.967</td>
<td>w</td>
<td>1.000</td>
<td>1.000</td>
<td>1.000</td>
<td>0 0 2</td>
<td>3.967</td>
<td>w</td>
<td>1.000</td>
<td>1.000</td>
<td>1.000</td>
</tr>
<tr>
<td>1 1 0</td>
<td>3.542</td>
<td>v</td>
<td>1.000</td>
<td>1.000</td>
<td>1.000</td>
<td>1 1 0</td>
<td>3.542</td>
<td>v</td>
<td>1.000</td>
<td>1.000</td>
<td>1.000</td>
</tr>
<tr>
<td>0 0 1</td>
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<td>3.067</td>
<td>v</td>
<td>1.000</td>
<td>1.000</td>
<td>1.000</td>
</tr>
</tbody>
</table>

(1) Zlavenec et al. (1977). \( \text{Ba}_2\text{Br}_2\text{Cl}_2 \) (originally \( \text{Ba}_2\text{Br}_2\text{Cl}_2 \)), quenched from 500°C. CuK\( \alpha \) radiation. Guinier camera.

w = weak, m = medium, s = strong. D-values converted from F-values.

(2) Run 47 (\( \text{Ba}_2\text{Br}_2\text{Cl}_2 \)), quenched from 800°C. CuK\( \alpha \) radiation (\( \lambda = 1.5405 \)), automated diffractometer.

(3) Coates and Chen (1974). CuK\( \alpha \) radiation (\( \lambda = 1.5405 \)), 114.4 mm Guinier camera.
### Table 47. Properties of synthetic Pd$_{31}$Sb$_{12}$ and stibiopalladinite.

<table>
<thead>
<tr>
<th></th>
<th>Pd$<em>{31}$Sb$</em>{12}$ (This study)</th>
<th>Pd$<em>{31}$Sb$</em>{12}$</th>
<th>Stibiopalladinite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colour</td>
<td>Yellowish white in air and</td>
<td></td>
<td>white$^{(2)}$, yellowish white in air and</td>
</tr>
<tr>
<td></td>
<td>pale yellow in oil</td>
<td></td>
<td>rather yellow in oil$^{(3)}$</td>
</tr>
<tr>
<td>Bireflectance/</td>
<td>not observed</td>
<td></td>
<td>not observed$^{(2)}$, weak in air and</td>
</tr>
<tr>
<td>Pleochroism</td>
<td></td>
<td></td>
<td>enhanced in oil$^{(3)}$</td>
</tr>
<tr>
<td>Anisotropism</td>
<td>moderate: brownish grey to</td>
<td></td>
<td>weak to distinct: reddish brown to bright</td>
</tr>
<tr>
<td></td>
<td>greenish grey in air and</td>
<td></td>
<td>grey in air; various shades of brown in</td>
</tr>
<tr>
<td></td>
<td>in oil</td>
<td></td>
<td>oil$^{(2)}$</td>
</tr>
<tr>
<td>Micro-indentation</td>
<td>$\text{VHN}_50 = 467$ (423 - 494) for four indentations</td>
<td>$\text{VHN}_50 = 605$ (585 - 625) for 2 indentations$^{(2)}$</td>
<td></td>
</tr>
<tr>
<td>hardness, mean (range)</td>
<td>$\text{VHN}_{100} = 463$ (437 - 498) for four indentations</td>
<td>$\text{VHN}_{50} = 327$$^{(4)}$</td>
<td>$\text{VHN}_{100} = 610$ (603 - 617) for 4 indentations$^{(2)}$</td>
</tr>
<tr>
<td>Symmetry</td>
<td>Rhombohedral$^{(1)}$</td>
<td></td>
<td>Hexagonal, $P6_3/mmc$, $P6_3/mc$ or $P6_2c$$^{(5)}$</td>
</tr>
<tr>
<td>Cell parameters</td>
<td>$a = 7.6006(9)$, $c = 41.945(8)$</td>
<td>$a = 7.613$</td>
<td>Hexagonal or orthorhombic (P22$_12$2)$$^{(3)}$</td>
</tr>
<tr>
<td></td>
<td>$c = 42.240$</td>
<td></td>
<td>$a = 7.598(2)$, $c = 28.112(9)$$^{(5)}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>$a = 12.80$, $b = 15.04$, $c = 11.36$$^{(3)}$</td>
</tr>
</tbody>
</table>

close relationship to synthetic Pd$_5$Sb$_2$– (presently Pd$_{31}$Sb$_{12}$) of El-Boragy et al. (1970).

According to Desborough et al. (1973), the symmetry of stibiopalladinite is either hexagonal or orthorhombic, but they excluded the hexagonal symmetry on the basis of their optical data. They gave the parameters of the orthorhombic cell with P2$_2$12 space group as $a = 12.80$, $b = 15.04$, $c = 11.36\AA$. However, the stibiopalladinite studied by Cabri and Chen (1976) is hexagonal, P6$_3$/mmc, P6$_3$mc, or P62c, with $a = 7.607(2)$ and $7.598(2)$, $c = 28.220(6)$ and $28.112(9)\AA$. The discrepancy was attributed to possible twinning in the crystals used by Desborough et al. (1973) or polytypism in the mineral.

The close relationship between Cabri and Chen's stibiopalladinite and synthetic Pd$_{31}$Sb$_{12}$ is substantiated by the fact that a direct comparison of the Gandolfo X-ray diffraction patterns of the two substances shows an excellent accordance with minor difference, in the back reflection region and in relative intensities of some lines. Most of the lines in the X-ray powder pattern of Pd$_{31}$Sb$_{12}$ may be indexed on the basis of the stibiopalladinite cell of Cabri and Chen (1976) with $a = 7.604(4)$ and $c = 28.09(1)\AA$.

Properties of synthetic Pd$_{31}$Sb$_{12}$ and stibiopalladinite are given in Table 47.

17. Pd$_5$Sb$_2$

The Pd$_5$Sb$_2$ was first observed by Bälz and Schubert
Table 44. X-ray powder diffraction data of synthetic Pd$_2$Rh$_2$.

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<th>b</th>
<th>k</th>
<th>l</th>
<th>d$_c$(A)</th>
<th>d$_e$(A)</th>
<th>I/I$_0$</th>
<th>d$_c$(A)</th>
<th>d$_e$(A)</th>
<th>I/I$_0$</th>
<th>d$_c$(A)</th>
<th>d$_e$(A)</th>
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<td>2.333</td>
<td>1.039</td>
<td>2.333</td>
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(1) Run 45, measured from 600°C. CuK$_\alpha$ radiation ($\lambda = 1.54059\AA$). automated diffractometer.
(2) EagleBerg et al. (1970). Guinier camera with CuK$_\alpha$ radiation. $\theta$-values converted from $\theta$-values.
$\omega$, weak; $\alpha$, medium; $\omega$, strong.
(3) Cobet and Chen (1976). 134.6mm Gandolfi camera, CuK$_\alpha$ radiation ($\lambda = 1.54154\AA$).
as a decomposition product of Pd$_2$Sb, and was later confirmed by El-Boragy et al. (1970), Cabri and Chen (1976), and Wopersnow and Schubert (1977). According to El-Boragy et al. (1970) and Cabri and Chen (1976), the Pd$_5$Sb$_2$ phase is hexagonal, space group P6$_3$cm, with $a = 7.60\text{ Å}$ and $c = 13.86\text{ Å}$. According to the Pd-Sb phase diagram of Wopersnow and Schubert (1977), Pd$_5$Sb$_2$ has a compositional range of 28.5 - 29.0 at.% Sb. The physical properties of Pd$_5$Sb$_2$ were not reported. A natural analogue of the synthetic Pd$_5$Sb$_2$ is not yet known.

The Pd$_5$Sb$_2$ phase was synthesized in this study between 1000° and 510°C. The phase has a narrow composition range of about 1.5 at.% in terms of Pd (run 51 and run 53, Table 8). It is white with a creamy tint under reflected light in air, with no visible bireflectance. It is weakly anisotropic from brownish grey to greenish grey in air. Micro-indentation hardness measurements give VHN$_{100}$ = 522 (483 - 560) for four indentations. The extent of Pt substituting for Pd reaches 27.5 at.% at 800°C, but decreases significantly to 15.6 at.% at 1000°C. The amount of Te in substitution for Sb is restricted to 16.5 at.% at 1000°C. The X-ray powder diffraction data of Pd$_5$Sb$_2$ compare well with those given by El-Boragy et al. (1970) and Cabri and Chen (1976), as do the cell parameters calculated from the powder data (Table 48).

18. Pd$_5$Sb$_2$
Pd$_5$Sb$_3$ was reported to be hexagonal (NiAs-type structure), with $a = 4.45$ and $c = 5.83\AA$ on the basis of X-ray powder diffraction data (Schubert et al., 1953). There is no X-ray powder diffraction pattern nor physical properties reported for this phase in the literature. There is no natural equivalent of this phase.

Pd$_5$Sb$_3$ synthesized between 600$^\circ$C and 800$^\circ$C has a narrow composition range of about 2 at.% in terms of Pd. Pt substitution for Pd and Sb reaches 8 at.% at 800$^\circ$C. Below 570$^\circ$C, bulk compositions of the Pd$_5$Sb$_3$ composition produce PdSb and Pd$_2$Sb. Thus, the lower thermal stability limit of Pd$_5$Sb$_3$ must be somewhere between 570$^\circ$ and 600$^\circ$C. In this thesis the temperature 580$^\circ$C, the upper stability limit of Pd$_2$Sb at which it decomposes into Pd$_5$Sb$_3$ and Pd$_5$Sb$_2$ (BäIz and Schubert, 1969), is tentatively accepted as the lower stability limit of Pd$_5$Sb$_3$.

Under reflected light Pd$_5$Sb$_3$ is pale yellowish pink in air and in oil, showing no bireflectance. It is weakly anisotropic, from brown to brownish pink in air and from brownish pink to greenish pink in oil. The micro-indentation hardness value is $VHN_{100} = 394$ (339 - 433) for five indentations.

The X-ray powder diffraction data of Pd$_5$Sb$_3$ do not fit on the hexagonal cell given by Schubert et al. (1953). However, comparison of the X-ray powder pattern of Pd$_5$Sb$_3$ with that of Pd$_2$Sb shows an excellent accordance, except for a minor difference in the bulk reflection region.
Table 49. X-ray powder diffraction data of synthetic Pd$_{5}$Sb$_{3}$.

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* Run 60-4, quenched from 700°C. Pd$_{62.4}$Sb$_{37.6}$. CuK$_\alpha$ radiation ($\lambda$=0.154059Å), automated diffractometer.
Therefore, the X-ray powder data of \( \text{Pd}_5\text{Sb}_3 \) were re-indexed according to the \( \text{Pd}_2\text{Sb} \) cell (orthorhombic, space group \( \text{Cmc}_2_1 \)). The cell parameters refined by a least-squares method, using the present X-ray data, are \( a = 3.362(1) \), \( b = 17.484(7) \), \( c = 6.934(2) \AA \) (Table 49).

19. \( \text{Pd}_2\text{Sb} \)

Bälz and Schubert (1969) reported \( \text{Pd}_2\text{Sb} \) to be orthorhombic, space group \( \text{Cmc}_2_1 \), with \( a = 3.354 \), \( b = 17.444 \), \( c = 6.909 \AA \). The \( \text{Pd}_2\text{Sb} \) phase was reported to decomposed at 580°C into \( \text{Pd}_5\text{Sb}_3 \) and \( \text{Pd}_5\text{Sb}_2 \). No further information is given about this phase, and no natural analogue of the synthetic \( \text{Pd}_2\text{Sb} \) is known.

The \( \text{Pd}_2\text{Sb} \) phase was synthesized in this study at 500°C and 570°C. Under reflected light \( \text{Pd}_2\text{Sb} \) is white with a creamy tint in both air and oil and is weakly bireflectant. Anisotropism is strong, from brownish gray to greenish blue in air and from yellowish pink to dark greenish blue in oil. The micro-indentation hardness test gives \( \text{VHN}_{100} = 293 \) (242 - 322) for four indentations. The X-ray powder diffraction data of \( \text{Pd}_2\text{Sb} \) are comparable with those given by Bälz and Schubert (1969), except that the d-values obtained in the present study are consistently larger (Table 50). The cell parameters calculated by a least-squares method are \( a = 3.366(1), b = 17.523(3), c = 6.929(2) \AA \).
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<td>v</td>
</tr>
</tbody>
</table>

(1) Run 40, quenched from 500°C. CuKα, radiation (λ = 1.54059Å), automated diffractometer.

v: weak, m: medium, s: strong.
20. PdSb - PdTe

The PdSb and PdTe phases are isostructural and form continuous solid solutions at 400°C (El-Boragy and Schubert, 1971a). They are, therefore, described under the same heading.

PdSb was first reported by Sander (1912) to melt congruently at 805°C, and later at 800°C by Grigorjew (1932). The structure was determined by Thomassen (1928) and confirmed by Pratt et al. (1968) to be of NiAs-type hexagonal, space group P63/mmc with \( a = 4.076 \), \( c = 5.591 \AA \). Cabri and Laflamme (1974a) re-determined the cell parameters as \( a = 4.079(3), c = 5.587(3) \AA \).

The PdSb phase was synthesized in the temperature range of 500 - 700°C. The X-ray powder diffraction patterns and the cell parameters of this synthetic phase compare well with those reported for PdSb by other authors (Table 51). The substitution of Pd by Pt reaches 18 at.% at 800°C.

Te substitution for Sb in PdSb phase is known to have no restrictions at 400°C, giving rise to a complete Pd(Sb,Te) solid solution (El-Boragy and Schubert, 1971a). However, the phase relations and the thermal stability of the Pd(Sb,Te) solid solutions between 400°C and 1000°C are not known. To elucidate the thermal stability of the Pd(Sb,Te) solid solution, three charges (runs 1004, 1005, and 1006; Table 52) with intermediate compositions were prepared at 670°C for 2 weeks, and homogeneous solid solutions were obtained in each case. Thus the complete Pd(Sb,Te) solid
Table 51. X-ray powder diffraction data of synthetic PdSb and sudburyite.

<table>
<thead>
<tr>
<th>h</th>
<th>k</th>
<th>l</th>
<th>d_c (Å)</th>
<th>d_o (Å)</th>
<th>I/I_o</th>
<th>d_o (Å)</th>
<th>I/I_o</th>
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</table>

(1) Run 42, quenched from 600°C. CuKα, radiation (λ = 1.54059 Å), automated diffractometer.

(2) Cabri and Laflamme (1974a). 114.6mm Debye-Scherrer camera, Cu radiation.

(3) Cabri and Laflamme (1974a). 57.3mm Gandolfi camera, Co radiation.
Table 52. Cell parameters of the synthetic PdSb - PdTe solid solutions.

<table>
<thead>
<tr>
<th>Run no.</th>
<th>Analyses (at.%)</th>
<th>Temperature (°C)</th>
<th>Cell parameters*</th>
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<tr>
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<td>Pd</td>
<td>Sb</td>
<td>Te</td>
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<td>50.0</td>
<td>-</td>
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<td>25.7</td>
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<tr>
<td>820</td>
<td>48.6</td>
<td>-</td>
<td>51.4</td>
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</table>

* X-ray powder diffraction data for the intermediate members from which the cell parameters were calculated are given in Appendix II.
solution is stable at least at 670°C and perhaps up to the melting point of PdTe (720°C).

The a and c cell parameters of the intermediate members increase almost linearly with increasing Te contents (Table 52). Pure PdSb is stoichiometric up to its melting point, but Te substitution for Sb causes the phase to become slightly deficient in Pd relative to total (Sb,Te).

Under reflected light synthetic PdSb is creamy yellow in air and in oil. Bireflectance is weak from creamy yellow to pale greenish yellow in air and moderate from greyish purple to dark greyish purple in oil. It is strongly anisotropic from dark greyish green to greyish pink in air and in oil. Micro-indentation hardness measurements give $VHN_{25} = 291$ (270 - 306) and $VHN_{100} = 294$ (279 - 302) for 4 indentations each.

Sudburyite, ideally PdSb, first described from Sudbury, Ontario (Cabri and Laflamme, 1974a), is the natural analogue of the synthetic PdSb phase. The mineral contains Ni in substitution for Pd, ranging from 2 to 2.9 at.%. Its X-ray powder diffraction pattern and physical properties compare well with those of the synthetic phase, except that the mineral is less anisotropic (Table 53), perhaps due to a composition difference.

PdTe was first prepared by Thomassen (1929a) and confirmed as a stable phase by Groeneveld Meijer (1955), Grønvold and Røst (1956) and other authors. It is isostructural with PdSb, being hexagonal, $P6_3/mmc$ with $a=$(...
<table>
<thead>
<tr>
<th>Property</th>
<th>PdSb (This study)</th>
<th>PdSb</th>
<th>Sudburyite (4)</th>
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<tr>
<td>Colour</td>
<td>creamy yellow in air and in oil</td>
<td></td>
<td>white with a yellowish tint in air and yellow in oil</td>
</tr>
<tr>
<td>Bireflectance/pleochroism</td>
<td>weak in air: creamy yellow to pale greenish yellow, moderate in oil: greyish purple to dark greyish purple.</td>
<td></td>
<td>not observed</td>
</tr>
<tr>
<td>Anisotropism</td>
<td>very strong in air and in oil: greyish green to greyish pink</td>
<td></td>
<td>weak to moderate in air, moderate in oil.</td>
</tr>
<tr>
<td>Melting point</td>
<td>$805^\circ\text{C}(1)$, $800^\circ\text{C}(2)$</td>
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<td></td>
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<tr>
<td>Micro-indentation hardness, mean (range)</td>
<td>$VHN_{25}^{} = 291$ (270-306) for 4 indentations, $VHN_{100}^{} = 294$ (279-302) for 4 indentations</td>
<td>$VHN_{25}^{} = 281$, $311$</td>
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</tr>
<tr>
<td>Symmetry</td>
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<td>Hexagonal, $P6_3/mmc$</td>
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</tr>
<tr>
<td>Cell parameters ($\bar{\lambda}$)</td>
<td>$a = 4.0753(4)$, $c = 5.5910(5)$</td>
<td>$a = 4.076(1)$, $c = 5.591(1)$ (3)</td>
<td>$a = 4.06(2)$, $c = 5.59(2)$</td>
</tr>
<tr>
<td></td>
<td>$a = 4.079(3)$, $c = 5.587(3)$ (4)</td>
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<td></td>
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</table>

4.1521 and c = 5.6719Å (Grønvold and Røst, 1956). The melting point of pure PdTe is 720°C (Medevedeva et al., 1961). The qualitative optical properties of PdTe were given by Groeneveld Meijer (1955) (Table 54).

PdTe was synthesized between 400°C and 700°C. Electron microprobe analyses show that PdTe synthesized below 450°C has a 1:1 Pd:Te ratio, whereas when synthesized above 500°C it becomes slightly enriched in Te with increasing temperature: 50.4 at.% at 500°C, 51.0 at.% at 600°C, and 51.8 at.% at 700°C, corresponding to Pd_{0.98}Te, Pd_{0.96}Te, and Pd_{0.93}Te respectively. This is consistent with the observations of the Pd(Sb,Te) solid solutions synthesized at 670°C, where the Te-rich members are slightly Pd-poor. The cell dimensions of PdTe are a = 4.130(3), c = 5.661(5)Å, in good agreement with those given by Thomassen (1929a) and Grønvold and Røst (1956). The X-ray powder diffraction data of PdTe are identical to those in the literature (Table 55) except that the weak line 1.081Å was not detected in this study.

Under reflected light PdTe is pale yellow in air and in oil. Bireflectance of the phase is absent to very weak in air and slightly enhanced in oil. It is strongly anisotropic, from dark yellow to greyish violet in air and in oil. Micro-indentation hardness value is VHN_{15} = 186 (171 - 193) and VHN_{100} = 197 (195 - 198) for four indentations of each load.

Kotulskite is the natural analogue of the synthetic
Table 5A. Properties of synthetic PdTe and kotulskite.

<table>
<thead>
<tr>
<th></th>
<th>PdTe (This study)</th>
<th>PdTe</th>
<th>Kotulskite</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Colour</strong></td>
<td>pale yellow in air and in oil</td>
<td>creamy white (1)</td>
<td>cream in air (5), cream or pale yellow in air and more yellow in oil (6)</td>
</tr>
<tr>
<td><strong>Bireflectance/pleochroism</strong></td>
<td>nil to very weak in air, weak in oil (pale brown to pale yellow)</td>
<td>none (1)</td>
<td>distinct in air and in oil: light cream to darker greyish cream (6), none (7)</td>
</tr>
<tr>
<td><strong>Anisotropism</strong></td>
<td>strong in air and in oil: dark yellow to greyish violet</td>
<td>strong in air (deep blue to yellow) (1)</td>
<td>strong in air: brownish- to greyish blue (5), grey to dark bluish grey (6)</td>
</tr>
<tr>
<td><strong>Melting point</strong></td>
<td></td>
<td>720°C (2)</td>
<td></td>
</tr>
<tr>
<td><strong>Micro-indentation hardness, mean (range)</strong></td>
<td>VHN&lt;sub&gt;15&lt;/sub&gt; = 186 (171-193) for four indentations, VHN&lt;sub&gt;100&lt;/sub&gt; = 197 (195-198) for four indentations</td>
<td>VHN&lt;sub&gt;15&lt;/sub&gt; = 292 (281-315) for four indentations (7), VHN&lt;sub&gt;15&lt;/sub&gt; = 236 (6)</td>
<td></td>
</tr>
<tr>
<td><strong>Symmetry</strong></td>
<td></td>
<td>Hexagonal, P6&lt;sub&gt;3&lt;/sub&gt; /mmc</td>
<td>Hexagonal, P6&lt;sub&gt;3&lt;/sub&gt; /mmc</td>
</tr>
<tr>
<td><strong>Cell parameters</strong></td>
<td>a = 4.135(1), c = 5.657(2)</td>
<td>a = 4.127(4), c = 5.663(5) (3)</td>
<td>a = 4.19(1), c = 5.67(1) (5)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>a = 4.1521, c = 5.6719 (4)</td>
<td></td>
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</tbody>
</table>

Table 55. X-ray powder diffraction data of synthetic PdTe and kotulskite.

<table>
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<tr>
<th>h k l</th>
<th>d_c(Å)</th>
<th>d_o(Å)</th>
<th>I/I_o</th>
<th>d_o(Å)</th>
<th>I/I_o</th>
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<th>I/I_o</th>
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(1) Run 820, quenched from 600°C. CuKα radiation (λ = 1.54059Å), automated diffractometer.

(2) PDF 29-971. 114.6mm camera with CuKα radiation (λ = 1.5418Å). Additional 15 high angle reflections are not reported.

(3) Génkin et al. (1963). 57.3mm camera. Pd_{1.0}Te_{1.2}Bi_{0.4}.
PdTe phase. The mineral was first described from Monchegorsk deposits, U.S.S.R. by Genkin et al. (1963), who assigned to the mineral the formula Pd(Te,Bi)\(_{1-2}\), later revised by Kingston (1966) to Pd(Te,Bi). Cabri (1981a) gives the ideal formula as PdTe and general formula as Pd(Te,Bi). The mineral has been found from many other deposits such as Sudbury, Ontario, and Stillwater complex, Montana.

Qualitative optical properties (Table 54) and X-ray powder diffraction pattern of kotulskite (Table 55) compare well with those of the synthetic equivalent, except that the micro-indentation hardness values of kotulskite are higher than those of synthetic PdTe. Genkin et al. (1963) reported two additional X-ray lines, 1.68 and 2.85\(\AA\), for kotulskite which are not present in the X-ray powder pattern of synthetic PdTe (Table 55). The presence of the two lines may be attributed to impurities.

21. PdSb\(_2\)

PdSb\(_2\) is cubic, space group Pa3 (Thomassen, 1928; Furuseth et al., 1965), with \(a = 6.439\,\AA\) (Thomassen, 1928), 6.4584(5)\(\AA\) (Furuseth et al., 1965), and 6.460\(\AA\) (Pratt et al., 1968). The melting point of PdSb\(_2\) was measured to be 677 - 680\(^\circ\)C by Sander (1912) and by Grigorjew (1932). Neither optical nor micro-indentation hardness data of this phase are reported. A natural equivalent of this phase has not been found.
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(1) Run 41, quenched from 600°C. CuKα radiation (λ = 1.54059 Å), automated diffractometer.

(2) Thomassen (1928). 57.65mm camera, FeKα radiation (λ = 1.934 Å). D-values converted from θ-values.
PdSb₂ was synthesized between 510°C and 650°C. Within the temperature range the compound has a stoichiometric composition. The solid solubility of Pt and Te in PdSb₂ is not known. The x-ray powder diffraction pattern of this phase compares well with the only pattern documented (Table 56), except for variations in intensities. The difference in relative intensities is perhaps due to the different method of recording and the different radiation employed. The refined cell parameter a = 6.488(3)Å is in excellent agreement with that given by Furuseth et al. (1965).

PdSb₂ is creamy white, under reflected light, with no bireflectance both in air and oil. Micro-indentation hardness measurements give VHN₁₀₀ = 484 (464 - 503) for 5 indentations.

22. Pd₁₇Te₄

Gabri (personal communication, 1983) did not find Pd₄Te (Grønvold and Røst, 1956) with a Pd:Te ratio of 4:1; instead, he found a phase having a composition of Pd₈₂Te₁₈ to which he assigned the formula Pd₉Te₂. Neither optical nor micro-indentation hardness data of Pd₄Te and Pd₉Te₂ are known.

In this study, a phase having a composition ranging from Pd₆ₐ₈₆Te₁₉.₄ to Pd₈₁₃Te₁₈.₇ has been found to exist between 610°C and 700°C and the formula Pd₁₇Te₄ is assigned to it. The X-ray powder pattern of this phase, coexisting
Table 57. X-ray powder diffraction data of synthetic Pd₄Te₄, Pd₉Te₂, and Pd₁₇Te₄.

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Plus 33 lines

(1) Gronvold and Rost (1956). Annealed at 500°C. CuKα radiation, 114.6 mm camera. D-values converted from θ-values. α = 12.674Å.

v: weak, m: medium, st: strong.

(2) Cabri (unpublished). Run 519 (Pd₉Te₂ with minor Pd₈Te₄ phase), quenched from 700°C. Cu radiation (λ = 1.789Å), Guinier camera.

(3) This study. Run 301 (Pd₁₇Te₄ with minor amount of Pd₂₀Te₇), quenched from 700°C. CuKα radiation (λ = 1.5418Å), 114.6 mm Gandolfini camera.

(4) This study. Run 61 (Pd + Pd₁₇Te₄), quenched from 620°C. CuKα radiation (λ = 1.5418Å), 114.6 mm Gandolfini camera.
with Pd (run 61) at 620°C or Pd₁₀Te₇ (runs 62, 63, and 64) at 620°C, is similar, to that of Pd₂₀Te₇ but different from that coexisting with Pd₂₀Te₇ at 700°C (run 801). The X-ray powder patterns (Table 57) of Pd₁₇Te₄, the Pd₄Te of Grønvold and Røse (1956) and the Pd₉Te₂ synthesized at 710°C by Cabri (unpublished) are not exactly the same. As DTA data does not indicate any reactions for this composition before it melts incongruently at 760°C, the differences in the X-ray powder patterns of these phases cannot be explained in terms of phase transformations.

Under reflected light Pd₁₇Te₄ synthesized at 700°C is light pinkish grey in air and in oil. The microindentation hardness value is VHN₁₀ = 259 (234 - 289) for four indentations. Pd₁₇Te₄ melts incongruently at 760°C, forming Pd and liquid.

Telargpalite, with a general formula (Pd,Ag)₄₆Te, was reported from Oktyabr deposit, U.S.S.R. by Kovalenker et al. (1975) who also reported it to be cubic with a = 12.60(2)Å. X-ray powder data of telargpalite are not similar to those of Pd₁₇Te₄, Pd₉Te₂, and Pd₄Te, although Berlincourt et al. (1981) considered telargpalite to be a natural equivalent of the synthetic Pd₄Te phase. More work is required to fully characterize the Pd₁₇Te₄ phase.

23. Pd₈Te₃

Pd₈Te₃ was first synthesized by Cabri in 1979 (Cabri, 1981a). However, neither X-ray data nor physical
properties of the synthetic phase are reported.

Pd$_8$Te$_3$ was synthesized between 410$^\circ$ and 800$^\circ$C. Pd$_8$Te$_3$ is found to have a composition of Pd$_{72.7}$Te$_{27.3}$ with no detectable composition range. The X-ray powder diffraction pattern of Pd$_8$Te$_3$ (Table 58) quenched from 800$^\circ$C is indexable on the basis of an orthorhombic cell with $a = 12.843(3)$, $b = 15.126(3)$, $c = 11.304(2)\text{Å}$ (Table 58).

Under reflected light Pd$_8$Te$_3$ is creamy yellow or pale grey in air and in oil with weak to moderate birefringence. It is strongly anisotropic from dark greyish blue to pale orange in air and slightly stronger in oil. DTA data show 2 endothermic reactions at 270$^\circ$ and 680$^\circ$C before it melts at 900$^\circ$C, which are interpreted as polymorphic transformations of the phase. The lamellar twinning, which is common in quenched grains, is probably due to the phase transformations. Micro-indentation hardness value was not measured because of the lamellar twinning present in the quenched phase.

Gandolfi X-ray diffraction patterns of synthetic Pd$_8$Te$_3$ of Cabri (personal communication, 1984) show two different structures depending upon the temperatures from which the phase was quenched (i.e., 500$^\circ$ and 250$^\circ$C), suggesting the existence of two polymorphs. X-ray powder pattern of the phase quenched from 250$^\circ$C is identical to that obtained in this study quenched from 800$^\circ$C. It is interpreted that the high temperature structure of this phase is not always quenchable or quench from 800$^\circ$C was not fast enough.

Cabri (1981) found an unnamed Pd$_8$Te$_3$ mineral in heavy
Table 58. X-ray powder diffraction data of synthetic Pd$_8$Te$_3$.

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plus many more lines

(1) Run 841 (Pd$_{0.73}$Te$_{0.27}$), quenched from 800°C. CuK$_\alpha$, radiation ($\lambda = 1.5405$Å), automated diffractometer. Orthorhombic, a = 12.848(3), b = 15.126(3), c = 11.304(2)Å.

(2) #504, quenched from 250°C. 114.6 mm Gandolfi camera, CuK$_\alpha$, radiation ($\lambda = 1.5405$Å).

(3) #504, quenched from 500°C. 114.6 mm Gandolfi camera, CuK$_\alpha$, radiation ($\lambda = 1.5405$Å).
mineral concentrates from Stillwater Complex, Montana, U.S.A.. According to him the mineral has the same optical properties as those of synthetic Pd₈Te₃ and is therefore considered to be the natural analogue. However, an unindexed X-ray powder pattern of the unnamed Pd₈Te₃ (Cabri, 1981) does not agree with those of the two synthetic phases of Pd₈Te₃. Confirmation of equivalence of the unnamed Pd₈Te₃ to the synthetic Pd₈Te₃ must await better microprobe analyses and X-ray diffraction work on the natural phase.

24. Pd₇Te₃

Pd₇Te₃, with a composition ranging from 70 to 71 at.% Pd, is a previously unreported phase and was synthesized between 410⁰ and 800⁰C. According to the DTA data, Pd₇Te₃ undergoes phase transformations at 470⁰ and 595⁰C before it melts at near 830⁰C.

X-ray powder diffraction patterns of the Pd₇Te₃ are almost identical to that of compositionally similar Pd₉Te₄ except for a few diffuse X-ray lines in the pattern of Pd₉Te₄. Thus, X-ray powder data for Pd₇Te₃ (run 808), quenched from 600⁰C, are indexed as Pd₉Te₄. Monoclinic cell parameters refined by a least-squares method, using the present X-ray powder data, are \( \alpha = 7.444(1) \), \( \beta = 13.918(2) \), \( c = 8.873(2) \text{Å} \), \( \beta = 92.46(2)⁰ \) (Table 59). The calculated density of the phase is 10.19 g/cm³, using the present cell dimensions with \( Z = 5 \).

Under reflected light Pd₇Te₃ is greyish pink in air, and
Table 59. X-ray powder diffraction data of synthetic Pd$_7$Te$_3$.

Monoclinic, $a = 7.444(1)$, $b = 13.918(2)$, $c = 8.873(2)$ Å, $β = 92.46(2)°$

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Run 808, quenched from 600°C. CuKα$_1$ radiation ($λ = 1.54059$ Å), automated diffractometer.
in oil, and is weakly bireflectant. It is weakly anisotropic from greyish brown with a red tinge to extinction in air and slightly stronger in oil. Micro-indentation hardness tests were not performed because of the presence of fine, lamellar twinning in the grains, probably due to phase transformations.

25. Pd$_9$Te$_4$

Pd$_9$Te$_4$ is monoclinic, space group P2$_1$/c, with a = 7.458(1), b = 13.938(1), c = 8.839(1) Å, β = 91.97(1)° (Matkovic and Schubert, 1978). The structure of Pd$_9$Te$_4$ is related to that of Pd$_5$Sb$_2$ with replacement, partial occupancy, and inner deformation (Matkovic and Schubert, 1978). According to the DTA data, Pd$_9$Te$_4$ undergoes a phase transformation at 460°C and melts incongruently at 605°C to form Pd$_7$Te$_3$ and liquid. A phase with Pd$_5$Te$_2$ stoichiometry was not encountered in this study. The Pd$_2$Te reported by Grønvold and Røst (1956) is apparently identical to Pd$_9$Te$_4$ according to X-ray powder diffraction data (Table 60).

Pd$_9$Te$_4$ was synthesized between 400°C and 600°C; above 605°C, and up to 830°C, Pd$_7$Te$_3$ occurs in equilibrium with a liquid. Microprobe analyses indicate that Pd$_9$Te$_4$ has a narrow compositional range of 69.4 - 69.2 at.% Pd. The X-ray powder diffraction patterns of Pd$_9$Te$_4$, quenched from above and below the phase transformation temperature (460°C), are identical and compare well with those given by Matkovic and Schubert (1978) (Table 60); the structure of
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(2) Run 815. quenched from 310°C. CuKα radiation (λ = 1.54059Å), automated diffractometer.
(3) Grunewald and Rout (1956). 114.8mm camera. w: weak. m: medium. s: strong.
Table 61. Properties of synthetic Pd₉Te₄ phase and telluropalladinite.

<table>
<thead>
<tr>
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<th>Pd₉Te₄ (This study)</th>
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<th>telluropalladinite&lt;sup&gt;(3)&lt;/sup&gt;</th>
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<td>cream with yellowish tint</td>
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<tr>
<td>Bireflectance/</td>
<td>nil to very weak in air:</td>
<td></td>
<td>not observed in air but</td>
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<td>pleochroism</td>
<td>pinkish brown to creamy</td>
<td></td>
<td>observed in oil</td>
</tr>
<tr>
<td>yellow</td>
<td></td>
<td></td>
<td>moderate (grey to extinction) to</td>
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<tr>
<td>Anisotropism</td>
<td>moderate in air: grey to</td>
<td></td>
<td>strong (light brownish grey to</td>
</tr>
<tr>
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<td>extinction</td>
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<td></td>
<td>oil</td>
</tr>
<tr>
<td>Melting point</td>
<td></td>
<td>605°C&lt;sup&gt;(1)&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>Micro-indentation</td>
<td>VHN&lt;sub&gt;15&lt;/sub&gt; = 176</td>
<td></td>
<td>VHN&lt;sub&gt;15&lt;/sub&gt; = 388 (376 - 399) for 4</td>
</tr>
<tr>
<td>hardness, mean (range)</td>
<td>146 - 210</td>
<td></td>
<td>indentations</td>
</tr>
<tr>
<td></td>
<td>for 4 indentations</td>
<td></td>
<td>VHN&lt;sub&gt;15&lt;/sub&gt; = 302 (296 - 308) for 5</td>
</tr>
<tr>
<td>Symmetry</td>
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<td></td>
<td>indentations</td>
</tr>
<tr>
<td>Cell parameters</td>
<td>a = 7.449(1)</td>
<td>a = 7.458(1)</td>
<td>a = 7.45(1)</td>
</tr>
<tr>
<td></td>
<td>b = 13.916(3)</td>
<td>b = 13.938(1)</td>
<td>b = 13.95(2)</td>
</tr>
<tr>
<td></td>
<td>c = 8.841(4)Å</td>
<td>c = 8.839(1)Å</td>
<td>c = 8.82(2)Å</td>
</tr>
<tr>
<td></td>
<td>θ = 92.01(5)°</td>
<td>θ = 91.97(1)°</td>
<td>θ = 91.9°</td>
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</tbody>
</table>

<sup>(1)</sup> Cabri (Personal communication, 1983).  <sup>(2)</sup> Matkovic and Schubert (1978).  <sup>(3)</sup> Cabri et al. (1979).
the high temperature form is therefore not quenchable. The lamellar twinning, which is common in the phase quenched from above 510°C, is probably due to the phase transformation.

The refined cell dimensions, \( a = 7.449(1) \), \( b = 13.916(3) \), \( c = 8.841(4) \) Å, \( \beta = 92.01(5)^\circ \), are in agreement with those reported by Matkovic and Schubert (1978). The optical properties and micro-indentation hardness of \( \text{Pd}_9\text{Te}_4 \) are given in Table 61.

The mineral telluropalladinite, with a general formula of \( \text{Pd}_9(\text{Te},\text{As},\text{Bi})_4 \) or ideally \( \text{Pd}_9\text{Te}_4 \), was first described by Cabri et al. (1979) from the Stillwater complex, Montana, U.S.A. and is considered to be the natural analogue of the synthetic \( \text{Pd}_9\text{Te}_4 \) phase. The properties of telluropalladinite compare well with those of the synthetic phase (Tables 60 and 61), except that the mineral is considerably harder, perhaps due to compositional differences.

26. \( \text{Pd}_3\text{Te}_2 \)

\( \text{Pd}_3\text{Te}_2 \), first synthesized by El-Boragy and Schubert (1971a), was reported as orthorhombic, space group \( \text{Amam} \), with \( a = 7.900(2) \), \( b = 12.687(3) \), \( c = 3.858(1) \) Å (Matkovic and Schubert, 1977). According to the later authors, \( \text{Pd}_3\text{Te}_2 \) is stable at least up to 480°C. No further information of this phase has been reported, and the natural analogue of \( \text{Pd}_3\text{Te}_2 \) is not known.
<table>
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<th>d_o (Å)</th>
<th>I/I_o</th>
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<td>1.068</td>
<td>6</td>
<td>1.068</td>
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</table>

(1) 834, quenched from 450°C. CuKα, radiation (λ = 1.54059 Å), 2θ, measured diffractometer.

Pd$_3$Te$_2$ was synthesized at 400° and 450°C; DTA data show that it decomposes at 500°C into Pd$_9$Te$_4$ and PdTe. Electron microprobe analyses indicate that Pd$_3$Te$_2$ is stoichiometric, with no measurable compositional range. The extent of substitution of Pt for Pd and Sb for Te has not been determined, because this phase is not stable in the temperature range of the ternary section investigated (1000°C).

The X-ray powder pattern of Pd$_3$Te$_2$ quenched from 450°C (Table 62) was indexed on the orthorhombic cell of Matkovic and Schubert (1977). The refined cell parameters are a = 7.875(2), b = 12.675(4), c = 3.853(1)Å, in agreement with those reported by Matkovic and Schubert (1977).

Under reflected light the synthetic Pd$_3$Te$_2$ phase is pale pinkish brown with a grey tint in air. It is very weakly bireflectant and weakly anisotropic from greyish brown to grey in air. Micro-indentation hardness tests give VHN$_{15}$ = 275 (257 - 290) for four indentations.

27. PdTe$_2$

Thomassen (1929a) reported PdTe$_2$ as hexagonal, space group P3$_1$cm, with a = 4.028(3) and c = 5.118(4)Å. Kjekshus and Grønvold (1959) studied PdTe$_2$ with a high temperature X-ray camera and showed a progressive increase in cell dimensions from a = 4.0365, c = 5.1262Å at 20°C to a = 4.0632, c = 5.2303Å at 574°C. Optical properties of the PdTe$_2$ phase were described by Groeneveld Meijer (1953) (Table 63).
### Table 63. Properties of synthetic PdTe₂ and merenskyite.

<table>
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<tr>
<th></th>
<th>PdTe₂ (This study)</th>
<th>PdTe₂</th>
<th>merenskyite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colour</td>
<td>white with creamy tint in air, white in oil</td>
<td>creamy white in air</td>
<td>white in air, white with creamy tint in oil</td>
</tr>
<tr>
<td>Bireflectance/pleochroism</td>
<td>absent in air and very weak in oil</td>
<td>not observed in air</td>
<td>weak in air and enhanced in oil, absent</td>
</tr>
<tr>
<td>Anisotropism</td>
<td>very weak (brownish grey to pale greenish grey) to moderate (dark grey to pale greenish yellow) in air and in oil</td>
<td>strong (bluish and yellowish colours) in air</td>
<td>strong (dark brown to light greenish grey) in air, weak in air and moderate in oil for Pt-rich one</td>
</tr>
<tr>
<td>Melting point</td>
<td></td>
<td>740°C</td>
<td></td>
</tr>
<tr>
<td>Micro-indentation hardness, mean (range)</td>
<td>VHN₁₀₀ = 115 (110 - 121) for 5 indentations</td>
<td></td>
<td>VHN₇₆ = 158</td>
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<td>Hexagonal, P₃₃₃m</td>
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<tr>
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<td>a = 3.978, c = 5.125Å</td>
</tr>
<tr>
<td></td>
<td></td>
<td>c = 5.1378(7)Å</td>
<td>a = 4.036, c = 5.132Å</td>
</tr>
</tbody>
</table>

PdTe$_2$ was reported to melt at 740°C (Medvedeva et al., 1961) and 730°C (Guggenheim et al., 1961).

PdTe$_2$ was synthesized in this study at temperatures between 600° and 710°C. Electron microprobe analyses of PdTe$_2$ indicate no measurable deviations from stoichiometry. The X-ray powder diffraction data and the cell parameters calculated by a least-squares method are in good agreement with those reported for PdTe$_2$ by other authors (Tables 63 and 64).

The complete solid solution between PdTe$_2$ and PdTe at temperatures between 640° and 690°C reported by previous authors (Medvedeva et al., 1961; Guggenheim et al., 1961; Kjekshus and Pearson, 1965; Hoffman and MacLean, 1976) could not be confirmed. Data from this study suggest that this high-temperature solid solution, if it truly exists, is not quenchable. The charges of compositions intermediate between PdTe and PdTe$_2$ split into PdTe$_2$-rich and PdTe-rich members on quenching from 600° and 670°C. Partial solid solutions of up to 4.1 mole% PdTe in PdTe$_2$, however, are quenchable.

The optical properties of PdTe$_2$ synthesized in this study are compared with those for PdTe$_2$ given by Groeneveld Meijer (1955) in Table 63. The differences are probably due to orientation of grains studied.

The mineral merenskyite, (Pd,Pt)(Te,Bi)$_2$ or ideally PdTe$_2$ (Kingston, 1966), is the natural analogue of the synthetic PdTe$_2$ phase. The X-ray powder diffraction data and physical properties of the mineral are comparable with
Table 64. X-ray powder diffraction data of synthetic PdTe₂ and merenskyite.

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<th>d₀(Å)</th>
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(1) Run 817, quenched from 700°C. CuKα radiation (λ = 1.54059Å), automated diffractometer.
(2) PDF 29-970. CuKα radiation (λ = 1.5418Å).
(3) Kingston (1966). \((\text{Pd,Pt})(\text{Te,Bi})_2\). 60mm camera, Cu radiation.
those for the synthetic phase (Tables 63 and 64).

Since Kingston (1966) first reported the fine
intergrowth of kotulskite (PdTe) and merenskyite (PdTe₂)
from the Merensky Reef, South Africa, similar occurrences
have been documented by other authors, including Cabri
also reported a mineral with Pd:Te ratio of 3:5 which was
considered to be unusual because no synthetic phase is known
to exist between PdTe and PdTe₂ according to the Pd-Te phase
diagram. Later, Cabri (1974) found, from the Stillwater
Complex, Montana, intimately intergrown lamellae of
kotulskite and merenskyite (confirmed by scanning electron
microscopy and energy dispersive X-ray analysis). Cabri
(1974) suggested that the unusual 3:5 stoichiometry for
Pd:Te shown by Kingston (1966) may merely represent the bulk
composition of what is actually a two-phase mixture (i.e.,
kotulskite and merenskyite) and probably is due to an
extremely fine intergrowth of the two minerals, which is
beyond the resolution of the average ore microscope and the
electron microprobe analyser.

28. Sb₂Te₃

Sb₂Te₃ is rhombohedral, space group R̅3m (Fay and
Ashley, 1902), with a = 4.25, c = 6.07Å (Dönges, 1951).
Semiletov (1956) also obtained hexagonal symmetry for Sb₂Te₃
by electron diffraction, and re-determined the cell
dimensions as a = 4.24(2)Å, c = 29.90(10)Å (=5 x 6.07Å).
The larger c-dimension was later confirmed by many authors (Black and Conwell, 1957; Bekebrede and Guentert, 1962; Brown and Lewis, 1962; Kuznetov and Palkina, 1963). The melting point of $\text{Sb}_2\text{Te}_3$ determined by various authors range from 616° to 629°C (Table 65).

$\text{Sb}_2\text{Te}_3$ was synthesized between 400° and 600°C in this study. It was also found as quenching products of Te-rich liquid. The X-ray powder diffraction pattern of $\text{Sb}_2\text{Te}_3$ synthesized in this study and the refined cell parameters are in good agreement with those for $\text{Sb}_2\text{Te}_3$ by other authors (Tables 65 and 66). $\text{Sb}_2\text{Te}_3$ has a compositional range of 59 - 60 at.% Te at 600°C and its composition limit on the Te-side is 60.3 at.% Te at 430°C, contrary to the results of Poretskaya et al. (1963) who reported deficiencies of Te in the phase as temperature decreases. $\text{Sb}_2\text{Te}_3$ does not contain any detectable amount of Pt in solid solution at 600°C.

Synthetic crystals of $\text{Sb}_2\text{Te}_3$ are hexagonal; because they are generally tabular, most crystals in polished sections appear lath-shaped. The compound is very brittle and fractures easily during micro-hardness testing under heavy loads (e.g., 100 gf). The micro-indentation hardness of the compound was, therefore, determined using a 15g load. The properties of the synthetic $\text{Sb}_2\text{Te}_3$ are given in Table 65.

The mineral tellurantimony, $\text{Sb}_{1.91}\text{Te}_3$ or ideally $\text{Sb}_2\text{Te}_3$, was first reported by Thorpe and Harris (1973) from
Table 65. Properties of synthetic Sb$_2$Te$_3$ and tellurantimony.

<table>
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<tr>
<th></th>
<th>Sb$_2$Te$_3$ (This study)</th>
<th>Sb$_2$Te$_3$</th>
<th>Tellurantimony$^{(10)}$</th>
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<tr>
<td>Colour</td>
<td>creamy yellow in air and in oil</td>
<td>pinkish cream in air</td>
<td></td>
</tr>
<tr>
<td>Bireflectance/</td>
<td>nil to very weak in air and in oil: creamy yellow to light greyish yellow</td>
<td>weak in air: pink to cream</td>
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<tr>
<td>pleochroism</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Anisotropism</td>
<td>moderate in air and in oil: dark brownish grey to light grey</td>
<td>moderate: pink to dark grey</td>
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<tr>
<td>Micro-indentation</td>
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<td>VHN$_{25}$ = 49.8 (39.6 - 61.3)</td>
<td></td>
</tr>
<tr>
<td>hardness, mean</td>
<td>(range)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Melting point</td>
<td>$629^{(1)}, 616^{(2)}, 621.6^{(3)}, 618.5^{(4)}$</td>
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<td>($^\circ$C)</td>
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<td>Rhombohedral, R$_3$m</td>
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<tr>
<td>Cell parameters</td>
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<td>$a=4.25^{(2)}, c=29.96^{(10)}^{(5)}$, $a=4.25, c=30.3^{(6)}$, $a=4.26, c=30.4^{(7)}$, $a=4.264, c=30.42^{(8)}$, $a=4.264, c=30.428^{(9)}$</td>
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Table 66. X-ray powder diffraction data of synthetic $\text{Sb}_2\text{Te}_3$ and tellurantimony.

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<td>$a = 4.262(1)$ c=30.435(2)Å</td>
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plus 4 lines

(1) PDF 15-874. Cu radiation ($\lambda=1.5405Å$), diffractometer.

(2) Run 14, quenched from 500°C. CuKα radiation ($\lambda=1.54059Å$), automated diffractometer.

(3) Thorpe and Harris (1973). 57.3mm Gandolfi camera, Ni-filtered Co radiation.
Mattagami Lake mine, Quebec and is the natural analogue of the synthetic Sb₂Te₃. The X-ray powder diffraction pattern, cell parameters and physical properties of tellurantimony compare well with those of the synthetic compound (Tables 65 and 66).

29. X-phase

A phase with a composition limit on the Sb-side ranges from 88.4 to 87.0 at.% Sb, having unknown boundary on the Te-side, was encountered in this study. This phase, tentatively referred to as 'X-phase' due to its questionable identity, was synthesized at 420°, 510°, and 520°D (runs 3, 2, 11, 12). The X-phase may be identical to the Sb-rich phase of Brown and Lewis (1962), that has a composition extending from 11 to 60 at.% Te, or it may be an independent phase for which the uncertain compositional boundary may be due to failure in obtaining equilibrium in the region 20 - 60 at.% Te.

The X-ray powder data of the X-phase coexisting with almost pure antimony (run 2), indexed on hexagonal axes, are given in Table 67, together with the cell parameters refined by a least-squares method using the powder data. The c-parameter of the phase is approximately 3 times that of antimony.

Under reflected light the X-phase is pale brownish yellow in air, and shows weak bireflectance. It is weakly anisotropic from brownish yellow to yellowish brown.
### Table 67. X-ray powder diffraction data of synthetic 'phase-X'

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\alpha &= 4.328(2) \\
c &= 33.65(2) \text{Å}
\end{align*}
\]

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<th>(d_o (\text{Å}))</th>
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Run 2, quenched from 510°C, CuKα radiation (\(\lambda = 1.54059\) Å), automated diffractometer.
CHAPTER VI
APPLICATION OF THE EXPERIMENTAL DATA
TO NATURAL ORES

Most primary platinum-group minerals, including Pt-Pd-Sb-Te compounds, are commonly associated with Cu-Fe-Ni sulphides, which in turn almost exclusively occur in close with association with mafic or ultramafic igneous rocks. Craig and Kullerud (1969) reported that 1149°C and 1105°C respectively are the liquidus and solidus temperatures of typical Ni- and Cu-bearing sulphide masses. Effects of almost ubiquitous additional phases such as magnetite and H₂O on the crystallization of such ores were determined to lower the melting temperature of the sulphide ores by 85°C (Craig, 1966) and by less than 20°C (Craig and Kullerud, 1969). The present experiments were conducted in rigid silica tubes in which vapour is an inherent phase.

However, the effects of pressure of a vapour phase on the phase relations of sulphide systems have been reported to be about 15° - 20°C/Kb by Kullerud (1959) and Kullerud and Yoder (1959), 10°C/Kb by Kullerud et al. (1969), and almost negligible by Barton and Toulmin (1966). The effects of confining pressure on phase relations in the ternary systems investigated for this thesis have not been studied, but are assumed to be small by analogy with other known systems.

In view of the above discussions, the experimental results obtained from this study can be applied in the following manner, assuming no complications due to the presence of
additional elements: (1) the thermal stability limit known for an individual phase and assemblages can give a measure of formation temperatures; (2) a natural mineral assemblage containing more than one phase as found in a synthetic assemblage may be used to evaluate whether it is formed at equilibrium or not. If it is an equilibrium mineral assemblage, it is possible to explain whether the mineral assemblage arose by liquidus or subliquidus reactions. As suggested by Skinner et al. (1976), the temperature 1000°C is adopted to serve to divide higher temperature liquidus processes from lower temperature subsolidus reactions.

1. The Pt-Sb-Te System

Only five solid phases, Pt (platinum), PtSb (stumpflite), PtSb₂ (geversite), PtTe₂ (moncheite), and Pt₃Te₄, coexist with liquid at 1000°C. This suggests that these phases may crystallize from the melt and may be expected to occur as primary igneous minerals. These phases, except for Pt₃Te₄ whose natural equivalent is not known, may occur individually or form assemblages such as geversite + stumpflite and geversite + moncheite. The other phases such as Pt₃₄Sb₇, Pt₃Sb, Pt₃Sb₂, PtTe, Pt₂Te₃, Sb₂Te₃ (tellurantimonite), and Sb (native antimony) may form in nature by reactions at subliquidus temperatures.

Platinum cannot coexist in equilibrium with geversite, stumpflite, or moncheite over the temperature ranges of 1000° - 600°C. Stumpfl (1961) reported a mineral assemblage consisting of Pt-Fe alloy, geversite, and/or
stumpflite, reported by Stumpfl (1961) from the Driekop mine, Transvaal, South Africa, may be interpreted to have been formed at disequilibrium or at liquidus temperatures in the presence of Fe. Common mineral intergrowths of stumpflite and geversite, also reported by Stumpfl (1961) from the same locality, are not certain whether they have been formed from liquidus or subliquidus temperature reactions, because such an assemblage persists over the temperature ranges of 1000° to 600°C.

2. The Pd-Pt-Sb System

Of nine solid phases existing in the temperature range of 1000 - 800°C in the Pd-Pt-Sb system, seven phases are stable at 1000°C: (Pt,Pd) alloy, Pd$_{20}$Sb$_7$, Pd$_8$Sb$_3$ (mertieite II), Pd$_{31}$Sb$_{12}$, Pd$_5$Sb$_2$, PtSb (stumpflite), and PtSb$_2$ (geversite). We can therefore expect these to have been formed by reactions at liquidus temperatures. The (Pt,Pd) alloy coexisting with liquid at 1000° and 800°C is high in Pt content. Thus, (Pt,Pd) alloy crystallized from the melt at liquidus temperatures may be expected to be enriched in Pt relative to Pd. Any known mineral assemblages formed at liquidus temperatures must consist of stumpflite and geversite, because the other possible assemblages include at least one phase whose natural analogue is not yet known. The other two phases in this system, PdSb (sudburyite) and Pd$_5$Sb$_3$, may be formed in nature by reactions at subliquidus temperatures.
3. The Pd-Sb-Te System

Five solid phases are stable at 1000°C: Pd (palladium), Pd$_{20}$Sb$_7$, Pd$_8$Sb$_3$ (mertieite II), Pd$_3$Sb$_{12}$, and Pd$_5$Sb$_2$. These phases exist in equilibrium with liquid, and therefore they are the phases expected to occur as primary igneous minerals and must not occur with Pd-Te minerals, because no Pd-Te compounds are stable at 1000°C.

4. Minerals formed by Subsolidus Reactions

Any mineral assemblages including one or more of the minerals such as sudburyite (805°C), kotulskite (720°C), and merenskyite (740°C) may be considered to form by subsolidus reactions based on the melting points of their synthetic equivalents.
CHAPTER VII
SUMMARY OF DATA

The Pt-Sb phase diagram reported by Bhan et al. (1969) is essentially correct, except that the liquidus between PtSb₂ and Sb has been revised. The X-ray powder diffraction pattern of the Pt₃₄Sb₇ phase has been indexed on a tetragonal cell with \( a = 3.948(3), c = 16.85(1) \text{Å} \).

The Pt-Te phase diagram given by Bhan et al. (1969) is mostly correct. The melting point of Pt₃Te₄ and the decomposition temperature of Pt₂Te₃ must be re-investigated.

The Pd-Sb phase diagram summarized by Hansen and Anderko (1958), later partly revised by Wopersnow and Schubert (1977), has been confirmed. X-ray powder data of Pd₅Sb₃ are indexable on an orthorhombic cell with \( a = 3.362(1), b = 17.484(7), c = 6.934(2) \text{Å} \).

In the Pd-Te system, a new phase, Pd₉Te₃, was found and the phase relations in the field 0 - 50 at.\% Te were redetermined. The X-ray powder diffraction patterns of Pd₈Te₃ and Pd₅Te₃ have been indexed on an orthorhomic cell with \( a = 12.843(3), b = 15.126(3), c = 11.304(2) \text{Å} \) and a monoclinic cell with \( a = 7.444(1), b = 13.918(2), c = 8.873(2) \text{Å}, \beta = 92.46(2)° \), respectively.

Phase relations of the Sb-Te system have been partly confirmed.
In the Pt-Sb-Te system at 1000°C, Pt, PtSb, PtSb$_2$, 
PtTe$_2$, Pt$_3$Te$_4$ and two liquids occur, forming four 3-phase 
and ten 2-phase assemblages. Two additional phases, PtTe 
and Pt$_2$Te$_3$, appear at 800°C, resulting in seven 3-phase and 
fourteen 2-phase assemblages. At 600°C, Pt$_3$Sb$_7$, Pt$_3$Sb, 
Pt$_3$Sb$_2$, Sb$_2$Te$_3$ and Sb become stable, and one liquid 
disappears and the other, along the Sb-Te join, splits into 
2 liquid fields, resulting in twelve 3-phase and twenty-five 
2-phase assemblages. Of these stable assemblages of solids 
are: PtSb + PtSb$_2$, PtTe$_2$ + Pt$_3$Te$_4$ and PtSb$_2$ + PtTe$_2$ at 
1000°C; PtSb + PtSb$_2$ + PtTe$_2$, Pt + PtTe, PtSb$_2$ + PtSb, PtSb 
+ PtTe$_2$, PtTe$_2$ + Pt$_2$Te$_3$, Pt$_2$Te$_3$ + Pt$_3$Te$_4$, PtTe + Pt$_3$Te$_4$ and 
PtSb$_2$ + PtTe$_2$ at 800°C; Pt + Pt$_3$Sb$_7$, PtTe, Pt$_3$Sb$_7$ + 
Pt$_3$Sb + PtTe, Pt$_3$Sb + Pt$_3$Te$_4$, Pt$_3$Sb + Pt$_3$Sb$_2$ + 
Pt$_3$Te$_4$, Pt$_3$Sb$_2$ + Pt$_3$Te$_4$ + Pt$_2$Te$_3$, Pt$_2$Te$_3$ + Pt$_3$Sb$_2$ + PtTe, 
Pt$_3$Sb$_2$ + PtSb + PtTe$_2$, PtSb + PtSb$_2$ + PtTe$_2$, Pt + PtTe, 
Pt$_3$Sb$_7$ + PtTe, Pt$_3$Sb + PtTe, Pt$_3$Sb + Pt$_3$Te$_4$, Pt$_3$Sb$_2$ + 
Pt$_3$Te$_4$, Pt$_3$Sb$_2$ + Pt$_2$Te$_3$, Pt$_3$Sb$_2$ + PtTe$_2$, PtSb + PtTe$_2$, PtSb 
+ PtSb$_2$, Pt$_3$Sb$_2$ + Pt$_3$Te$_4$, Pt$_3$Sb$_2$ + Sb, PtSb$_2$ + Sb$_2$Te$_3$, Pt + 
Pt$_3$Sb$_7$, Pt$_3$Sb$_7$ + Pt$_3$Sb, Pt$_3$Sb + Pt$_3$Sb$_2$, Pt$_3$Sb$_2$ + PtSb, 
PtSb + PtSb$_2$, Pt$_2$Te$_3$ + PtTe$_2$, Pt$_3$Te$_4$ + Pt$_2$Te$_3$ and PtTe + 
Pt$_3$Te$_4$ at 600°C.

In the Pt-Pd-Sb system, the 1000°C isothermal section 
consists of (Pd,Pt) solid solution, Pd$_2$O$_7$Sb$_7$, Pd$_3$Sb$_3$, 
Pd$_3$Sb$_4$, Pd$_5$Sb$_2$, PtSb, PtSb$_2$ and a large liquid, forming at 
least one 3-phase and nine 2-phase fields. At 800°C, two 
more phases, PtSb and Pd$_3$Sb$_3$, become stable, resulting in at
least one 3-phase and sixteen 2-phase fields confirmed.

The assemblages of solids are: Pd$_{20}$Sb$_7$ + (Pd, Pt) alloy, PtSb$_2$ + PtSb, Pd$_{20}$Sb$_7$ + Pd$_8$Sb$_3$, Pd$_8$Sb$_3$ + Pd$_{31}$Sb$_{12}$, Pd$_{31}$Sb$_{12}$ + Pd$_5$Sb$_2$ at 1000°C; (Pd, Pt) alloy + Pd$_{20}$Sb$_7$, (Pd, Pt) alloy + Pd$_8$Sb$_3$, Pd$_5$Sb$_2$ + Pd$_5$Sb$_3$, PtSb$_2$ + PdSb, Pd$_{20}$Sb$_7$ + Pd$_8$Sb$_3$, Pd$_8$Sb$_3$ + Pd$_{31}$Sb$_{12}$, Pd$_{31}$Sb$_{12}$ + Pd$_5$Sb$_2$ and PtSb$_2$ + PtSb at 800°C.

In the Pd-Sb-Te system, the isothermal section of 1000°C consists of Pd$_{20}$Sb$_7$, Pd$_8$Sb$_3$, Pd$_{31}$Sb$_{12}$, Pd$_5$Sb$_2$, and one large liquid with nine 2-phase and one 3-phase fields confirmed.

The stable assemblages of solid phases are: Pd + Pd$_{20}$Sb$_7$, Pd$_{20}$Sb$_7$ + Pd$_8$Sb$_3$, Pd$_8$Sb$_3$ + Pd$_{31}$Sb$_{12}$ and Pd$_{31}$Sb$_{12}$ + Pd$_5$Sb$_2$. 
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Geol., 71, 1352-1363.


Thomassen, L. (1928): Über Darstellung und Kristallstrukturen des Mono- und Diantimonides von


APPENDICES
Appendix I. Cut-away sketch of a furnace showing the relative position of the thermocouple (a) and the silica capsule (b).
Appendix II. X-ray powder diffraction data of the \( \text{Pd}_{20}(\text{Sb},\text{Te})_7 \) solid solutions.

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(2) Run 974, quenched from 1000°C. CuK\(_\alpha\) radiation (\( \lambda = 1.54059\AA \)), automated diffractometer.

(3) Run 944, quenched from 1000°C. CuK\(_\alpha\) radiation (\( \lambda = 1.54059\AA \)), automated diffractometer.
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(4) Run 1001, quenched from 700°C. CuK$_\alpha$ radiation (\(\lambda = 1.54056\)), automated diffractometer.

(5) Run 1002, quenched from 700°C. CuK$_\alpha$ radiation (\(\lambda = 1.54056\)), automated diffractometer.

(6) Run 1003, quenched from 700°C. CuK$_\alpha$ radiation (\(\lambda = 1.54056\)), automated diffractometer.
Appendix III. X-ray powder diffraction data of Pd(Sb,Te) solid solutions.

<table>
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<th>PdSb&lt;sub&gt;0.8&lt;/sub&gt;Te&lt;sub&gt;0.2&lt;/sub&gt;</th>
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(1) Run 1004. (2) Run 1005. (3) Run 1006. All quenched from 670°C.
CuKα<sub>1</sub> radiation (λ = 1.54059Å), automated diffractometer.