

among his observed  $d$ -spacings. In addition he records appearance of a line at 5.61 Å which he cannot account for, suggesting that it may be a line due to ammonioborite, data for which were not then available to him. Comparison of his data with the calculated  $d$ -spacings for larderellite (Table 3) shows that the material in his sample gives additionally not only several sassolite lines and the unidentified 5.61 Å line, but also a series of lines clearly due neither to sassolite nor to ammonioborite. These observed lines are listed in Table 4 and compared with the observed  $d$ -spacings associated with lines of strong intensity of  $\text{KB}_5\text{O}_6(\text{OH})_4 \cdot 2\text{H}_2\text{O}$  ( $\text{KB}_5\text{O}_8 \cdot 4\text{H}_2\text{O}$ , Clark and Christ, 1959). The matching of these two sets of observed  $d$ -spacings is preliminary evidence for the appearance of  $\text{KB}_5\text{O}_6(\text{OH})_4 \cdot 2\text{H}_2\text{O}$  in nature; confirming chemical tests would be of interest.

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THE CRYSTAL STRUCTURE OF POTARITE ( $\text{PdHg}$ ) WITH  
SOME COMMENTS ON ALLOPALLADIUM

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Potarite is a palladium amalgam which occurs in the Potaro river region of British Guiana (Palache, Berman, and Frondel 1944). It was first described by Harrison and Bourne (1924-5) and more fully characterized by Spencer (1928). Spencer further surveyed the known synthetic palladium amalgams and suggested that potarite might be identical with allopalladium. Cissarz (1930) reexamined simultaneously samples of potarite and allopalladium and found them to be different sub-

stances. He described potarite as an isomorphous ground mass with small, irregular, anisotropic inclusions.

M. A. Peacock (1945) examined a sample of potarite by the *x*-ray powder method and reported that it gave a simple cubic pattern with  $a_0 = 5.21 \text{ \AA}$  with a unit of cell  $\text{Pd}_4\text{Hg}_4$ . This gave a calculated density of 14.48 g./ml. compared with Spencer's value of 14.88 g./ml. Peacock further reported that attempts to directly synthesize the material had not been successful. He suggested that the structure might be that attributed to FeSi with a space group  $P2_13$ .

A powder photograph of a synthetic alloy PdHg obtained by Bittner and Nowotny (1952) indicated a face-centered cell  $a_0 = 4.284 \text{ \AA}$ ,  $c_0 = 3.692 \text{ \AA}$ , containing  $\text{Pd}_2\text{Hg}_2$ . In our work we have indexed our patterns for the smaller body centered tetragonal cell  $a_0 = 3.020_5 \text{ \AA}$ ,  $c_0 = 3.706_7 \text{ \AA}$ , cell content PdHg. The crystal structure is that of AuCu type  $L1_0$ . The space group is  $P 4/mmm$ . Trial-structure calculations with Hg at 0, 0, 0 and Pd at  $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$  are in agreement with the observed line intensities.

#### EXPERIMENTAL

Samples of PdHg which are identical with the mineral potarite may be prepared either by displacement of palladium from  $\text{Pd}(\text{NO}_3)_2$  solution by mercury or by direct synthesis from powdered palladium and mercury in sealed evacuated tubes. The samples used by Bittner and Nowotny were prepared by direct synthesis from the elements. The compound PdHg can be easily prepared by heating the elements in a sealed Pyrex tube for twelve hours at  $400^\circ \text{ C}$ . Compositions high in mercury should be annealed for a considerable time at  $100^\circ \text{ C}$ . to insure uniformity of product. A mixture of palladium powder and mercury in equimolar amounts, which is kept at room temperature, does not show formation of potarite at an appreciable rate. In fact, it has long been known that the rate of penetration of palladium by mercury at low temperatures is slight (Horsford, 1852). A meta-stable system containing an alloy higher in mercury than PdHg and unreacted palladium are found by *x*-ray examination of this system. However, if the system is heated for 4 hours at  $200^\circ \text{ C}$ ., the *x*-ray powder pattern is that of PdHg.

A table showing the observed inter-planar spacings for a natural sample of potarite and for synthetic PdHg prepared from solution and from direct combination of the elements is given. The data given in Table I have been corrected for absorption and beam divergence using the method outlined by Azaroff and Buerger (1958). This method has been programed for use with the Datatron 205 electronic digital computer, and so may be rapidly and easily applied. Patterns were ob-

TABLE I. MEASURED AND CALCULATED SPACINGS OF PdHg.

<i>hkl</i>	<i>d</i> (soln)	<i>d</i> (direct combination)	<i>d</i> (potarite-annealed)	<i>d</i> (calculated)
001				3.707
100	3.032	3.029	3.033	3.020
101	2.346	2.343	2.342	2.342
110	2.143	2.141	2.142	2.136
002				1.853
111	1.847	1.850	1.846	1.850
102				1.580
200	1.516	1.509	1.515	1.510
112				1.400
201	1.397	1.399	1.398	1.399
210		1.354		1.351
211	1.273	1.272	1.272	1.269
003				1.236
202	1.170	1.170	1.171	1.171
103	1.139	1.141	1.141	1.144
212				1.092
113				1.069
220	1.071	1.072		1.068
221	1.027			1.026
300				1.007
301	.973 <sub>7</sub>	.973 <sub>5</sub>	.973 <sub>2</sub>	.971 <sub>6</sub>
203				.956 <sub>3</sub>
310	.957 <sub>9</sub>	.956 <sub>5</sub>	.956 <sub>5</sub>	.955 <sub>2</sub>
004				.926 <sub>7</sub>
222	.926 <sub>6</sub>	.926 <sub>0</sub>	.925 <sub>9</sub>	.925 <sub>3</sub>
311				.925 <sub>0</sub>
213	.910 <sub>2</sub>	.911 <sub>5</sub>	.910 <sub>7</sub>	.911 <sub>7</sub>
104				.885 <sub>9</sub>
302				.884 <sub>7</sub>
114				.850 <sub>1</sub>
312	.850 <sub>0</sub>	.850 <sub>2</sub>	.850 <sub>0</sub>	.849 <sub>0</sub>
320				.837 <sub>7</sub>
321	.819 <sub>2</sub>	.818 <sub>7</sub>	.818 <sub>3</sub>	.817 <sub>1</sub>
223				.807 <sub>9</sub>
204	.789 <sub>4</sub>	.789 <sub>0</sub>		.789 <sub>8</sub>
303		.780 <sub>8</sub>	.780 <sub>3</sub>	.780 <sub>5</sub>

The blank spaces in the table represent "d" distances for faint lines which have been observed in pure PdHg patterns exposed under ideal conditions. The last column of interplanar distances were calculated from our  $a_0 = 3.020_5 \text{ \AA}$  and  $c_0 = 3.706_7 \text{ \AA}$ . By the method of least squares,  $a_0$  and  $c_0$  were recalculated as  $3.026 \text{ \AA}$  and  $3.072 \text{ \AA}$  respectively.

tained using  $\text{CuK}\alpha$  radiation  $\lambda(\text{K}\alpha_1) = 1.5405 \text{ \AA}$ ,  $\lambda(\text{K}\alpha_2) = 1.5443 \text{ \AA}$ ,  $\lambda(\text{K}\alpha \text{ weighted average}) = 1.5418 \text{ \AA}$ .

It is interesting to note that the potarite sample obtained from the

TABLE II. TETRAGONAL AND CUBIC INDEXING OF PdHg POWDER PHOTOGRAPH

Tetragonal Index	$d$ (observed)	$d$ (calculated with Peacock $a=5.21$ )	Cubic Index
100	3.03	3.01	111
101	2.34	2.33	210
110	2.14	2.13	211
111 } 002 }	1.85	1.84	220
200	1.51	1.50	222
201	1.40	1.39	321

U. S. National Museum gave broad diffraction lines and thus was quite imperfectly crystalline. This sample was then annealed while still sealed in a Lindemann capillary. Upon heating at 100° C. for 6 hours it recrystallized and sharp diffraction lines were obtained. It is thus apparent that the mineral has not been subjected to elevated temperatures. The sample which Professor Peacock had examined showed normal line breadth and so we may assume that different degrees of crystalline perfection exist in naturally occurring potarite.

It is interesting to compare the indexing assigned by Professor Peacock with that for the tetragonal cell. This is shown in Table II. We have reexamined Professor Peacock's films and find that lines which cannot be explained by his cell may be observed though they are extremely weak. This of course, illustrates the intrinsic danger always present in the interpretation of a powder pattern containing only a few lines.

It would seem that the poorly crystalline mineral appears to be isotropic on microscopic examination and that small islands of crystalline material comprise the "inclusions." The poorly crystalline nature is in accord with an experimental specific gravity of 14.88, rather lower than the theoretical calculated  $x$ -ray value 15.09.

Synthetic samples containing less than 15 atomic per cent mercury show a solid solution of mercury in the palladium lattice. This is in accord with the observation of Berzelius (1813) that it is very difficult to remove the last traces of mercury from palladium amalgam. In the mercury rich region, two diffraction patterns have been obtained. These are substances of very low symmetry judged from the complexity of the  $x$ -ray patterns.

#### ALLOPALLADIUM

Attempts to obtain a sample of allopalladium have been unsuccessful. However, no hexagonal phase of the system PdHg has been found in the range Pd to PdHg<sub>7</sub>. Thus we must conclude that the hexagonal mineral

described by Zincken (1829), Cissarz (1930), and Schneiderhöhn and Ramdohr (1931) is almost certainly not a simple palladium amalgam.

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## A NOTE ON SO-CALLED "PRESSURE INDEPENDENT" MINERALS

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In a recent note on the stability and synthesis of uvarovite,  $3\text{CaO} \cdot \text{Cr}_2\text{O}_3 \cdot 3\text{SiO}_2$ , Glasser (1959) objected to a statement by Hall (1958) which tended to give the impression that uvarovite, like diamond,