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SUBJECT EXTERNAL PUBLICATIONS

GRANITE CK

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B.C. Open File 92H/10W
092HNE 199

Shell Canada Calgary Nov.16, 1977

Re: Shell Placer Tests Tulameen River 1967-68

History:

About 20,000 oz of Pt recovered from Tulameen area prior to 1967.

An analysis of Pt grains resulted in values

77.07% Pt
0.19% Pd
2.57% Rh
1.14% Ir
3.39% Cu
8.59% Fe
10.51% iridosmine (Ir + Os alloy)
1.69% Cr

In 1964 Tulameen Mines trenched to depth of 25 feet at placer lease 1364 in the Tulameen River about 20 miles upstream from Princeton B.C.

Tulameen Platinum Mines Ltd. (N.P.L.) found that a mechanical and chemical process was required to separate Pt and Au and used a process developed by Rao and Koch which is as follows:

Gravel is crushed and mechanically processed to produce black sand at a ratio of 50:1. The black sand is treated with concentrated HCl which leaves metallic Pt and PtO_2. The residue is calcined at 450° C converting metal sulphides to oxides. The residue is treated with concentrated H_2SO_4 and washed with H_2O and dried. The dry residue is placed in a reaction vessel with a catalyst and gaseous chlorine is added under 100 to 200 lbs pressure at less than 100° C. Residue cooled and treated with HCl to convert PtCl to soluble chloroplatinic species. Mixture is filtered and the filtrate containing Pt and other soluble species of noble metals is treated with sodium borohydride which produces a metallic black powder. The black powder is filtered off and dried with a recovery of 2 grams of black powder from 10 lbs of black sand. Pt is present in amounts from >0 to 98% weight. (500 lbs gravel $\lessapprox$ 2 gms Pt). Recoverable Pt from the sample used in the process is:

$\approx$ 280 g/t black sand

or $\approx$ 5.6 g/t gravel

Insufficient work was done to determine whether or not this sample is representative of the entire deposit.

Table 1
Tulameen Pt Mines Ltd.
Analytical Data
New Samples Oct.14/66
Analytical Data - R & D

Sample	Tulameen	Prelim. Spec. Analys.	Precise wet Analysis
(1) Gravel 50 t/day processed by 2 men.			
(2) Black Sand Processed from gravel 1 t/day		>10% wt. Fe 1-10 Al,Mg,Si,Ti 0.1-1 Cr,Cu,Mn,Sn,V <0.1 Ca,Pb Ag <0.1% wt. Iridium Detected ≤500 ppm →	4 oz Ir/ton
(3) Processed Black Sand 0.15 ton/day It is used as feedstock to Tulameen's Chlorination Method for rec of Pt		>10% wt. Si 1-10 Al,Cr,Fe,Ti 0.1-1 Mn,Na <0.1 V,Ca,Cu,Mg,Sn Platinum 0.1±0.01% wt. Silver 0.04±0.02 Gold Detected <50 ppm	
(4) Black powder Rec product 400 g/50 tons gravel or 400 g/1 ton Black sand	Platinum	>10% wt. Pt,Cu 1-10 As,Fe,Si,Ti 0.1-1 Sb,Ca,Mg,Ni,Sn <0.1 Al,B,Cr,Mn Platinum 50±10% wt. Silver 0.1 Au detected (≤ 50 ppm) Rhodium detected (≤100 ppm) Iridium detected (≤500 ppm)	Pt 69±0.5% wt.

Shell Involvement Tulameen Project

I Bulk Sample

In 1967 Shell obtained a 10 ton sample from Tulameen Platinum Mines Ltd. Placer Lease 1365. Shell split the sample returning one half to Tulameen Platinum Mines Ltd. and carried out a study of the retained 5 tons of samples. Shell's conclusions resulting the study are as follows:

(1) Rao and Koch process used by Tulameen Platinum Mines Ltd. fails:

   (a) Precious metal content is far below the value required for a breakeven operation based on estimated chemical/operating costs.

   (b) Riffle table concentration of Pt and Au in black sand is ineffective, resulting in a loss of over 90% of the values into tailings.

(2) A new recovery process is required.

(3) Precious metals in 20 mesh fraction offers incentive for further exploratory programs with the following objectives:

   (a) Determination of lateral-vertical distribution of 20 mesh fraction in Tulameen Valley.

   (b) Pt/Au values in 20 mesh fraction

   (c) Search for possible recovery methods.

The 10 ton sample is not necessarily representative of valley but is representative of material in which Tulameen worked. There appears to be some discrepancy between the values obtained in Tulameen Platinum's first sample and that taken by Shell.

Table II

Gravity Separation On Riffle Table

Distribution of Pt/Au in Riffle Table Cuts

Shell Sample - May

	Particle Size Cut	Mat. Bal. Around Riffle Table Separation wt. % of Particle Size Cut			Pt/Au Assays of Riffle Table Cuts /Ton of Particle Size Cut			Pt/Gold Loss to Tailings
		Feed	Blk Sand Conc	Tailings	Feed*	Blk Sand Conc	Tailings**	Cut %
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
(1)	2½+ in	100	1	99	2.45	0.02	2.43	99
(2)	3/4-2½ in	100	4	96	1.65	0.05	1.60	97
(3)	6 mesh-3/4"	100	2	98	1.65	0.03	1.62	98
(4)	20-6 mesh	100	2	98	1.71	0.07	1.64	96
(5)	65-20 mesh	100	4	96	7.75	0.42	7.33	94
(6)	65 mesh	100	3	97	9.86	1.66	8.20	83

*Assuming Pt @ $160/Troy oz.
Au @ $35/Troy oz.

**obtained by difference (5)-(6)

II Placer drilling was carried out in three lines of drill holes on the Tulameen valley floor, about 10 to 17 miles, upstream from Princeton. Twenty four holes, in three lines, totalling 2500 feet were sampled at 4 foot intervals and each sample was screened to 5 particle sizes. Bedrock was reached in line 2 but only in 3 holes at the edge of the valley in the other two lines.

Assays were disappointingly low, with minor exceptions from the drill holes near the bulk sample pit the levels were less than 0.3 ppm Pt (20 mesh size fraction). During analysis, when it became evident that the values were low, it was decided not to assay every sample. However a minimum of two samples per hole were assayed.

Conclusions:

(1) There is some question regarding the Shell drilling method producing <u>representative assays</u> of the entire volume of gravels.

(2) The form and manner in which the Pt occurs is not clearly established. Tulameen Platinum Mines Limited and Shell found that <u>a process of chemical recovery of Pt</u> as well as mechanical concentration was necessary. There is some thought about Pt being associated with magnetite within rock fragments.

(3) The cost of treating the gravel containing Pt, as conducted by Shell, was prohibitive.

K.E. Northcote

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TULAMEENITE, A NEW PLATINUM-IRON-COPPER MINERAL FROM PLACERS IN THE TULAMEEN RIVER AREA, BRITISH COLUMBIA †

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Abstract

Tulameenite (Pt_2FeCu) is a new mineral found in placer deposits along the valleys of the Tulameen and Similkameen Rivers in south-central British Columbia, Canada. Tulameenite is tetragonal with $a = b =$ 3.891(2) and $c =$ 3.577(2)7Å, D calc. = 15.6 g/cc. The six strongest lines of the x-ray diffraction powder pattern are 2.179(10) (111), 1.946(7) (020), 1.317(5) (022), 1.163(8) (131), 1.094(8) (113), and 1.016(6) (023,132).

Tulameenite is white in reflected light in air and in oil and is very weakly anisotropic. Reflectance values are: 470 nm, 65.3 and 61.0; 546 nm, 66.5 and 60.0; 589 nm, 65.5 and 61.5; and 650 nm, 64.9 and 61.1%. Micro-indentation hardness values for a 50 g load range from 420-456 kg/sq. mm, with an average of 442. The mineral is ferromagnetic.

Some compositional, optical, and physical properties are also given for associated cubic iron-bearing platinum.

Introduction

Platinum-bearing placers have been known to occur in the Tulameen River area of south-central British Columbia for nearly a century. They were actively worked for platinum and gold in the early 1900's and since that time have received intermittent attention from various mining companies. Because of the continued interest in the placers and their possible economic importance, and because no mineralogical reports have been published on them, the Mines Branch considered that a detailed mineralogical investigation would be helpful in providing guidelines for prospecting and for appraisal and beneficiation of the placers. Accordingly, samples were obtained from several sources for examination. To date, ten minerals of the platinum group have been recognized in these samples, including two new minerals. One of the new minerals — an alloy of platinum, iron and copper, has been named tulameenite *, for the locality. The second new mineral is currently under study.

The geology of the Tulameen River area has been described by Camsell (1913). More recently, Findlay (1969) reported on the petrology of the Tulameen igneous rock complex. This complex is generally considered to be a "zoned" intrusion after Nobel & Taylor (1960) or a "concentric" intrusion after Jackson & Thayer (1972).

Samples and Procedures

Detrital platinum and platinum-rich grains from placer concentrates, and synthetic platinum alloys, were examined in this study. The detrital samples and their localities are listed in Table 1. Grains from the Similkameen River were obtained from the National Mineral Collection, Ottawa. Grains from the Tulameen River (Nos. 1 and 2) were recovered from a concentrate provided by B.H. Levelton and Associates of Vancouver for the Mines Branch Standards Program. Grains (G.D.) from the junction of the Tulameen River and Bear Creek were separated from a sample previously submitted to the Mines Branch for beneficiation tests.

The samples were studied by ore microscopy, x-ray diffraction analysis, and electron probe microanalysis. The samples were mounted in cold-setting plastic, polished on lead laps, and lightly buffed on a cloth lap using minus 0.2-μ alumina. The reflectance values were obtained with a Leitz MPE microscope photometer equipped with a Dumont 6467 photo-multiplier tube and a continuous-band interference filter. A silicon standard calibrated by the National Physical Laboratory, Great Britain, was used as a reference. A 16.5:1 objective with a numerical aperture of 0.40 was used. The micro-indentation hardness was measured with a Leitz-Durimet tester.

The x-ray data were obtained by the film method using Gandolfi and Debye-Scherrer cameras and Fe-filtered Co radiation. Film shrinkage corrections were applied, the intensities visually estimated, and unit cell parameters were refined by a least-squares computational method.

† Mineral Research Program — Non-sulphide research contribution No. 33.

* The mineral tulameenite and the name have been approved by the Commission on New Minerals and Minerals Names, I.M.A.

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Compositions were determined using a Materials Analysis Company model 400 electron-probe microanalyser using, as standards, iridium and nickel metals and synthetic PtFe, Pt_3Fe, $PtSb_2$, and PtCu. Corrections were applied using Edition VII of the program by Rucklidge (1967). The synthesis of Pt–Fe, Pt–Cu and Pt–Fe–Cu compositions were necessary to provide standards for the electron probe and for x-ray powder diffraction. High-purity reagents were used for synthesis, the suppliers and reagents being: (a) Johnson, Matthey and Co.: Fe and Pt (reduced J.M. 844 sponge and J.M.C. 1010), and (b) American Smelting and Refining Co. 99.999% Cu rod (reduced filings) and 99.999% Sb. After several failures at obtaining homogeneous products the following technique was found to be successful, but the Pt–Fe–Cu compositions took the longest to homogenize. The samples were weighed in silica-glass tubes, evacuated, sealed and placed in a horizontal furnace at 1200°C. The tubes were removed from the furnace after four weeks, opened, and the samples were crushed in a mortar or cut into small fragments with a side cutter. They were then replaced in tubes and, after evacuation and sealing, were annealed for a further period of three to four weeks at 1200°C. The furnace was then cooled at a rate of about 200°C/day down to 400°C. The tubes were removed, air-quenched, some of the sample removed for polished section and some drilled with a small high-speed drill. The drill cuttings were placed in silica-glass capillaries which were themselves placed in a silica tube and annealed at 300°C for one to twelve days prior to removal for x-ray diffraction analysis.

General Mineralogy

The main constituent of the placers in the Tulameen area is quartz with variable quantities of rock fragments and boulders. Magnetite and chromite are common heavy minerals; their relative proportion varies with location. Minor heavy minerals are garnets, (ferroan spessartite and manganoan almandine), hematite, zircon, sulphides (principally pyrite), ilmenite, gold, and hydrated iron oxides. Platinum-group minerals, as discrete mono-minerallic grains and as complex multi mineral nuggets and flakes, occur as minor constituents of these placer deposits. Tulameenite occurs associated with cubic iron-bearing platinum as rounded to irregular areas up to about 400 μm in diameter, as free grains, or as grains with complex inclusions (Fig. 1). When occurring as an outer zone on a nugget or flake of cubic iron-bearing platinum, tulameenite is distinguished by its relatively inferior polished surface (Fig. 2).

TABLE 1. ELECTRON PROBE ANALYSES OF TULAMEENITE AND CUBIC IRON-BEARING PLATINUM

Sample, locality & no. of analyses	weight per cent -- average and range						Total	Formula
	Pt	Ir	Fe	Cu	Ni	Sb		
tulameenite								
Grain A (G.D.) Tulameen R. (10)	76.65 75.5-77.6	not detected	10.55 8.6-12.3	6.95 5.7-8.3	3.77 3.0-4.7	2.07 1.6-2.9	99.99	$Pt_{2.04}(Fe_{0.98}Cu_{0.56}Ni_{0.34}Sb_{0.08})_{\Sigma=1.96}$
Grain B (G.D.) Tulameen R. (9)	74.87 70.7-76.9	not detected	8.39 6.4-9.8	9.38 6.9-11.9	3.52 2.1-4.6	3.52 2.7-5.0	99.68	$Pt_{2.00}(Fe_{0.78}Cu_{0.76}Ni_{0.32}Sb_{0.14})_{\Sigma=2.00}$
Grain 5 Similkameen R. (10)	73.98 73.56-74.78	1.99 1.98-1.99	10.38 9.80-11.09	13.13 12.18-13.99	not detected	not detected	99.48	$(Pt_{1.94}Ir_{0.06})(Cu_{1.06}Fe_{0.94})_{\Sigma=2.00}$
Grain 9 Similkameen R. (10)	73.68 72.12-75.09	1.95 1.94-1.96	11.22 10.13-12.05	11.25 10.15-13.09	not detected	not detected	98.10	$(Pt_{1.98}Ir_{0.06})(Fe_{1.04}Cu_{0.92})_{\Sigma=1.96}$
Grain 1 (#2258) Area A-Tul. R. (8)	76.66 76.19-77.19	0.22 0.22-0.22	11.94 11.43-12.14	8.68 8.45-8.98	0.50 0.49-0.51	1.40 1.31-1.50	99.40	$(Pt_{2.06}Ir_{0.006})(Fe_{1.12}Cu_{0.72}Sb_{0.06}Ni_{0.04})_{\Sigma=1.94}$
Grain 1 (#2258) Area B-Tul. R. (8)	77.24 77.00-77.55	0.20 0.18-0.21	12.21 12.12-12.28	8.43 8.29-8.56	0.51 0.49-0.53	1.23 1.14-1.30	99.82	$(Pt_{2.06}Ir_{0.006})(Fe_{1.14}Cu_{0.70}Sb_{0.06}Ni_{0.04})_{\Sigma=1.94}$
Grain 1 (#2258) Area C-Tul. R. (9)	77.31 76.91-78.12	not detected	11.23 10.40-12.45	9.17 8.70-9.43	0.60 0.58-0.64	1.04 0.96-1.20	99.35	$Pt_{2.08}(Fe_{1.06}Cu_{0.76}Ni_{0.06}Sb_{0.04})_{\Sigma=1.92}$
Grain 1 (#2258) Area D Tulameen River (10)	77.83 76.93-78.33	0.25 0.24-0.25	12.87 12.59-13.07	8.01 7.87-8.15	0.56 0.52-0.58	0.65 0.59-0.70	100.17	$(Pt_{2.06}Ir_{0.006})(Fe_{1.20}Cu_{0.66}Ni_{0.06}Sb_{0.02})_{\Sigma=1.94}$
cubic iron-bearing platinum								
Grain 5 Similkameen R. (10)	84.85 83.8-85.76	2.22 2.22-2.24	10.83 10.58-11.11	1.46 1.43-1.50	not detected	not detected	99.3	$Pt_{2.62}Fe_{1.17}Cu_{0.14}Ir_{0.07}$
Grain 9 Similkameen R. (7)	83.20 82.8-83.4	2.00 1.99-2.00	11.99 11.7-12.2	0.88 0.84-0.94	not detected	not detected	98.07	$Pt_{2.56}Fe_{1.29}Cu_{0.08}Ir_{0.06}$
Grain 2 (#2258) Tulameen R.	90.22	not detected	9.69	0.22	not detected	not detected	100.13	$Pt_{2.9}Fe_{1.1}Cu_{0.02}$
Grain C (G.D.) Tulameen R.	87.40	not detected	8.97	2.59	0.47	not detected	99.43	$Pt_{2.27}Fe_{0.98}Cu_{0.25}Ni_{0.05}$

Optical, Physical, and Chemical Properties of Tulameenite and Associated Iron-bearing Platinum

Tulameenite

Tulameenite is white in reflected light in oil and air, though a slightly darker white than cubic iron-bearing platinum. No bireflectance could be observed and the mineral is very weakly anisotropic. The reflectance for the four wavelengths 470, 546, 589, and 650 nm is 61.0, 60.0, 61.5, and 61.1% for the minimum position and 65.3, 66.5, 65.5 and 64.9% for the maximum position, all respectively. The reflectances were measured on two different areas for grains #5 and #9 (Table 1).

The micro-indentation hardness values were obtained from five indentations on grain #9 and four indentations on grain #5 with a 50-g load. The range of micro-indentation hardness is 420 to 456 kg/mm², with an average value of 442. Tulameenite is distinctly ferromagnetic and is attracted to a steel needle.

The analyses of eight tulameenite grains by the electron microprobe are given in Table 1 and some are plotted on a ternary Pt–Fe–Cu diagram (Fig. 3). The analyses correspond to the formula $(Pt,Ir)_2(Fe,Cu,Ni,Sb)_2$ or Pt_2FeCu. These analyses are discussed in more detail in a subsequent section.

The *x*-ray diffraction powder data for tulameenite and synthetic Pt_2FeCu are given in Table 2. The density of synthetic Pt_2FeCu was measured with a Berman balance using hexachloro-1, 3-butadiene. The highest value obtained was 14.9 g/cc which is reasonably close to a theoretical value of 15.6 for the compound with Z = 2. Voids in the synthetic sample account for the lower measured density.

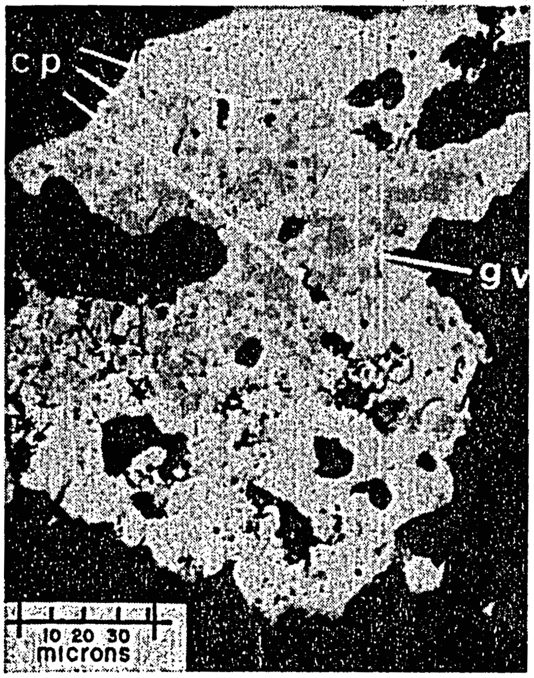

FIG. 1. Photomicrograph of a complex tulameenite grain (Grain B, G.D.) showing inclusion of geversite (gv) and chalcopyrite (cp) with, on the lower part, darker grey inclusions of a new Ir(Rh)-Sb-S mineral. Black areas are pits.

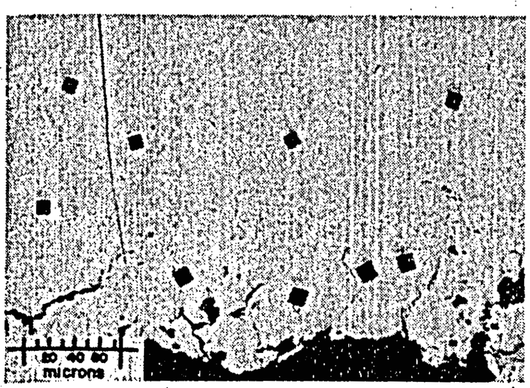

FIG. 2. Photomicrograph of part of grain #9, Similkameen River, showing cubic iron-bearing platinum (white, smooth surface) with associated zone of tulameenite (fractured surface, lower part of photograph) and larger micro-indentation. Note the larger indentations in the relatively softer tulameenite.

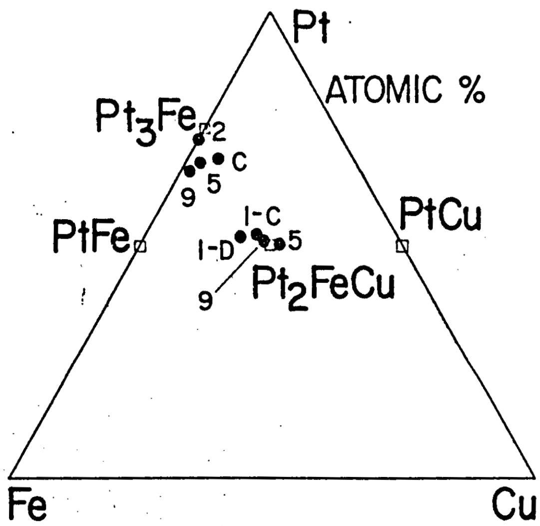

FIG. 3. The Pt-Fe-Cu ternary. Synthetic compounds are denoted by squares; natural samples by closed circles. Sample numbers refer to those in the table. For both tulameenite and cubic iron-bearing platinum, the minor Ir is calculated as Pt, but the other minor elements are calculated as Cu in the former and as Fe in the latter.

Cubic iron-bearing platinum

The analyses for four grains of cubic iron-bearing platinum associated with tulameenite are also given in Table 1 and these are shown in Fig. 3. The formulae are calculated on the basis of 4 formula weights per unit cell because the x-ray diffraction powder data for grain #5 shows the same pattern as for pure platinum, but with $a = 3.851(1)$Å. This is in contrast to synthetic Pt–Fe alloys of similar compositions which give a different cubic diffraction pattern and this problem is still under study.

The micro-indentation hardness was measured for the cubic iron-bearing platinum and found to be VHN(50) = 553-588 (av. 580 for five indentations) in grain #5 and VHN(50) = 580-633 (av. 600 for five indentations) in grain #9. These values are higher than those reported by Yushko-Zakharova *et al.* (1970) for similar compositions.

Discussion

Tulameenite has a range of composition with minor Ir replacing Pt and with Ni replacing Cu and Fe to the extent that some grains can be considered a nickeloan variety (Table 1). Minor Sb was detected in some grains. There also appears to be some substitution of Fe for Cu, up to about 1.29 Fe to 0.71 Cu (recalculating grain #1 area D without the Ni and Sb). Conversely, however, there appears to be little replacement of Cu for Fe in the samples examined. Experiments along the PtFe–Pt_2FeCu join indicate that the maximum substitution of Fe for Cu in tulameenite is up to between $Pt_2Fe_{1.68}Cu_{0.32}$ and $Pt_2Fe_{1.28}Cu_{0.72}$.

Pt_2FeCu gives an x-ray diffraction powder pattern that differs from the patterns for both PtFe (Table 2) and PtCu. Early in the study the authors believed that natural Pt_2FeCu would correspond to the "unamed mineral Q" of Cabri (1972) which has also been referred to as "tetragonal ferroplatinum" by Mikheev (1961) and Genkin (1968). However, the x-ray powder data indicate that these minerals have tetragonal cells with different crystal structures.

A preliminary investigation of the magnetic properties of synthetic PtFe and Pt_2FeCu by our colleague, Mr. J.L. Horwood, indicates that they are both ferromagnetic but that PtFe is more so, by several orders of magnitude.

Conclusion

This study has defined the new mineral tulameenite and has described its occurrence and relationship with the relatively non-magnetic cubic iron-bearing platinum alloys. This relationship is, in itself, a useful guide for the sampling and/or mining of such placer deposits. This study also indicates that more detailed work needs to be done on natural PtFe, on iron-bearing platinum, and on the phase relations in the Pt–Fe–Cu system. Some of this work is underway in our laboratories.

TABLE 2. X-RAY DIFFRACTION POWDER DATA

Tulameenite (gr. #5) a=3.891(2) c=3.577(2)Å				Pt_2FeCu a=3.885(1) c=3.588(1)Å			PtFe a=3.847(1) c=3.715(1)Å			
I	d_{meas}	d_{calc}	hkl	I	d_{meas}	d_{calc}	I	d_{meas}	d_{calc}	hkl
3	3.569	3.576	001	3	3.568	3.588	3	3.690	3.715	001
4	2.753	2.751	110	4	2.746	2.747	3	2.714	2.720	110
10	2.179	2.181	111	10	2.180	2.181	10	2.195	2.194	111
7	1.946	1.945	020	6	1.942	1.942	4	1.918	1.923	020
2	1.789	1.788	002	4	1.794	1.794 ?	3	1.857	1.857	002
4	1.709	1.709	021	4	1.707	1.708	3	1.707	1.708	021
3	1.500	1.499	112	4	1.502	1.502	3	1.534	1.534	112
4	1.375	1.375	220	4	1.374	1.373	3	1.361	1.360	220
5	1.317	1.317	022	7	1.319	1.318	5	1.338	1.336	022
2	1.285	1.284	221	3	1.283	1.282	4	1.280	1.282	030
1	1.230	1.230	130	4	1.229	1.228	2	1.243	1.238	003
			003	1	1.196	1.196	3	1.218	1.216	130
8	1.163	1.163	131	9	1.163	1.162	9	1.156	1.156	311
							6	1.127	1.127	113
8	1.093	1.094	113	8	1.096	1.096	6	1.098	1.097	222
							1/2	1.068	1.067	230
							5	1.042	1.041	023
6	1.016	1.016	023	3	1.018	1.018	6	1.018	1.017	132
			132	6	1.014	1.013				
3b	0.9730	0.9728	040	5	0.9713	0.9712	5	0.9626	0.9618	040
2b	0.9433	0.9438	140	6	0.9420	0.9422	5	0.9324	0.9311	041
2b	0.9175	0.9172	230	5	0.9157	0.9157	6	0.9180	0.9157	223
			223	6	0.9022	0.9020	5b	0.9088	0.9068	330

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