

# THE EFFECT OF THE MINERALOGY OF THE PLATINUM GROUP METALS ON THEIR LEACHABILITY DURING THE PLATSOL® LEACH PROCESS

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## ABSTRACT

The mineralogy of the platinum group metals is fairly complex, and it is therefore not surprising that the different minerals have variable responses to metallurgical processes, in particular, flotation and leaching.

The PLATSOL® leach process was developed to extract simultaneously base metals, gold and platinum group metals from various materials. Extensive research has indicated that most PGM's were amenable to the PLATSOL process, with the notable exception of cooperite PtS.

Further work showed that a thermal pretreatment at 500-700°C transformed the structure of the cooperite and similar refractory minerals (Pt,Pd)S into Pt metal and Pt-Pd alloys that responded well to the PLATSOL® process.

Examples are presented of the mineralogy of PGM concentrates as produced, in the residue from the PLATSOL® leach and after thermal pretreatment.

## INTRODUCTION

Most of today's platinum production originates from South Africa: in 2002, it produced 4.45 M oz. of platinum, or 74.5% of the world supply (1). The process commonly used in South Africa involves the production of a flotation concentrate, that is then processed to metal as per the simplified flowsheet presented in Figure 1 (2).

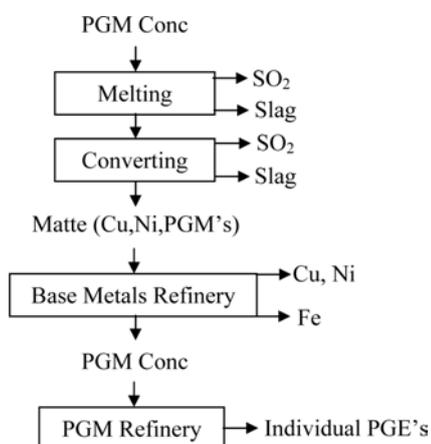


Figure 1. Conventional PGM concentrate treatment

Although efficient, the process imposes strict conditions on the quality of the concentrates treated: they typically assay  $\geq 120\text{-}150\text{ g/t } \Sigma\text{ PGE}$  (Platinum Group Elements), with a limit on certain gangue elements, notably chromite  $\text{Cr}_2\text{O}_3$ . (3)

There are numerous deposits around the world where such quality PGM (Platinum Group Metals) concentrates cannot be produced, or only at the expense of a significant loss of recovery. With the present state of the technology, these deposits cannot be economically developed, and await the development of a new process concept.

The NorthMet deposit in Minnesota, USA, is a typical example of a low grade PGM ore for which the full values of its PGM content could not be realised using conventional techniques. The PLATSOL®

process was developed in 2000 by International PGM Technologies at SGS Lakefield, to maximise the revenues from the ore by leaching, in a single step, the base (Cu, Ni, Co) and precious (Pt, Pd, Au) metals in a bulk concentrate.

NorthMet bulk flotation concentrate assayed  $\sim 15\%$  Cu and  $3\%$  Ni, but only  $14\text{ g/t}$  (Au + Pt + Pd). The PLATSOL® process consists in high temperature ( $>200^\circ\text{C}$ ) pressure oxidation in a sulphuric acid medium spiked with a few grams per litre of chloride. Details of the application of the PLATSOL® process to the NorthMet concentrate are in the open literature (4 - 6). PM's (precious metals) extraction during the PLATSOL® process were excellent and confirmed during a 10 day pilot plant.

The applicability of the PLATSOL<sup>®</sup> process to various other PGM concentrates was attempted, and it was discovered that some concentrates did not respond well to PLATSOL<sup>®</sup>.

This paper describes the mineralogical investigations undertaken to find the reasons for the refractory nature of some concentrates. The findings of such studies eventually led to the development of a pretreatment process to unlock the PGM's from the refractory concentrates.

## EXPERIMENTAL

### EXPERIMENTAL SET-UP

The pressure leaching experiments were conducted in a 2L Parr titanium autoclave fitted with dual radial axial impellers, cooling coil and external resistance heating. Some of the roasting tests were carried out in a muffle furnace, where no attempt was made to control the gaseous atmosphere above the sample. When control of the gas phase was required, the experiments were conducted in a glass rotating tube furnace with external resistance heating.

### MINERALOGICAL

Detailed PGM mineralogy was conducted at SGS Lakefield. Platinum Group Minerals (PGM's) were located by systematically scanning 30 mm diameter particulate mounts made of as-received products. Location of PGM grains was performed in automated mode with a Leo 440 Scanning Electron Microscope (SEM) using QemSCAN technology. Use of Back-Scattered Electron (BSE) detector, allows atomic number contrast imaging to detect high-atomic number species. Once high-BSE grains were located, PGM's were characterized by Energy Dispersive X-Ray analysis using an Oxford ISIS system. Routine analysis included semi-quantitative identification of common PGM using internal standards and photographing all occurrences and characterizing grains and host minerals where locked. An account of liberation, grain size and association was kept in order to record all potential mineralogical influences on exposure and recovery.

For more detailed analysis of minerals, Electron Microprobe Analysis (EMPA) was performed using a Jeol 733 Superprobe. Information regarding complex zoning and quantitative mineral chemistry was obtained using a ZAF correction procedure and mineral standards from SRM and SPI for the PGE suite of elements.

### CONCENTRATES TESTED

Numerous concentrates from around the world have been tested. Table 1 presents the chemical analyses of some of the concentrates that responded well to the PLATSOL<sup>®</sup>, i.e. with (Pt + Pd) extractions during the leach equal or greater than 90%, while Table 2 presents the chemical analyses of some of the refractory concentrates.

ELEMENTS	CONCENTRATE TYPES										
	A	B	C	D	E	F	G	H	I	J	K
Cu%	14.7	5.8	-	12.0	4.2	2.4	5.5	3.9	0.9	0.2	0.7
Ni%	3.05	4.1	-	3.01	7.7	0.9	10.3	14.9	0.1	0.1	0.3
Fe%	32.9	17.1	-	33.9	35.9	19.2	-	27.9	32.7	16.6	28.7
S%	26.7	16.3	-	24.5	34.1	-	25.3	28.8	0.8	0.5	<1
Au g/t	1.41	12.4	0.5	0.9	0.5	3.0	2.1	1.2	0.8	9.9	1.5
Pt g/t	2.22	12.3	1.9	0.8	1.4	5.3	11	2.4	3.4	72.0	5.3
Pd g/t	9.9	14.3	23.6	2.8	2	10.1	7.5	9.6	20.6	62.3	23.3
MgO%	2.0.	-	-	-	-	-	-	-	-	-	-

Table I. Chemical Analyses of Cu/Ni/PGM Sulphide Concentrates Amenable to PLATSOL<sup>®</sup>

ELEMENTS	CONCENTRATE TYPES									
	L	M	N	O	P	Q	R	S	T*	
Cu%	0.27	4.21	-	-	-	-	-	-	-	
Ni%	0.16	2.38	-	-	-	-	-	-	-	
Fe%	7.29	14.9	-	-	-	-	-	-	-	
S%	0.23	9.91	0.09	0.37	-	9.9	6.7	9.7	-	
Au g/t	-	9.6	-	-	-	-	-	-	-	
Pt g/t	17.5	54.8	7.4	27.6	7.7	46.3	490	54.8	322	
Pd g/t	10.9	42.6	4.9	17.0	4.9	14.5	292	42.6	134	
MgO%	20.9	16.2	-	-	-	-	-	-	-	
Cr <sub>2</sub> O <sub>3</sub> %	10.1	1.29	-	-	-	-	-	-	-	
*Used automobile catalyst										

Table II. Chemical Analyses of Cu/Ni/PGM Sulphide Concentrates Refractory to PLATSOL<sup>®</sup>

Concentrate A was the NorthMet concentrate for which the PLATSOL<sup>®</sup> was initially developed. Comparing the two sets of concentrates (Tables I and II) from a general point of view, it appears that the concentrates amenable to the PLATSOL<sup>®</sup> process are those containing high values of sulphide and iron; even concentrates I, J, K initially contained high sulphide values; but they had been pretreated by bacteria to dissolve the base metal sulphides, prior to the PLATSOL<sup>®</sup> test.

## RESULTS

### METALLURGICAL

Tables III and IV present the PLATSOL<sup>®</sup> leach extractions of the concentrates described in Table 1 (amenable to PLATSOL<sup>®</sup>) and Table II (refractory).

ELEMENTS	% RECOVERY										
	CONCENTRATE TYPES										
	A	B	C	D	E	F	G	H	I	J	K
Cu	99.5	99.9	-	99.3	99	99.3	99.7	99.6	99.3	85.0	99.2
Ni	99.5	99	-	93.8	99.6	98.7	98.9	98.7	96.7	-	95.5
Au	90	99	97.9	97.1	84.3	96.0	97.9	82.9	91.5	96.4	53.6
Pt	95	95	98.5	97.4	98.2	99.0	97.9	93.9	93.9	99.1	95.3
Pd	96	94	99.7	98.1	93.9	98.4	96.2	95.6	94.9	96.3	96.3

Table III. Results of the Standard PLATSOL® Test - Non-Refractory Concentrates

ELEMENTS	% RECOVERY									
	CONCENTRATE TYPES									
	L	M	N	O	P	Q	R	S	T*	
Pt	36.6	37.0	64.0	34.4	40.9	33.9	51.1	34	72.1	
Pd	70.2	73.8	90.7	65.6	85.7	78.1	80.1	78	86	

\*Used automobile catalyst

Table IV. PLATSOL® Results on Refractory PGM Concentrates

### MINERALOGICAL

Due to the good leachability of the PGM's from the non-refractory concentrates, limited mineralogical studies were conducted on these test products. Figure 2 presents the PGM distribution in the NorthMet concentrate feed to the PLATSOL® process; the description of the PGM's (Pt + Pd) minerals in the PLATSOL® leach residue from the said concentrates is presented in Table V.

PLATSOL® leach extractions were excellent, and only 5% of the PGM minerals resulted in the leach residue and all were locked within reaction products (iron oxy-sulphates). These findings indicate that the reasons for the non-leachability of some of the PGM's in the NorthMet concentrate were physical (entrapment) rather than chemical (refractoriness).

Based on these very promising results, the applicability of the PLATSOL® process was extended to other Cu/Ni/PGE sulphide concentrates, and also to other precious metal containing feed stocks, such as matte, automobile catalysts, copper-gold and refractory (non preg-ropper) gold concentrates, as well as silver concentrates (7 - 11).

Continued testing of the PLATSOL® on other Cu/Ni/PGE concentrates produced results where PGM extractions, and, more particularly platinum extractions, were not good (Table IV).

It is well known that the mineralogy of platinum-group minerals is much more complex than that of gold, with new minerals continuously being identified (12). After a few unsuccessful metallurgical tests to improve PLATSOL® leach recoveries, it was hypothesized that there were certain PGM minerals that were refractory to the PLATSOL® conditions. A systematic mineralogical study was conducted to identify the PGM minerals in the PLATSOL® residues, and hopefully develop a strategy to overcome their refractory behaviour.

A detailed mineralogical examination was conducted on various feeds/residues to the PLATSOL® process; the results will be presented here for only a few examples due to limitations to the length of the paper. Three examples are presented in Figures 3, 4 and 5 for refractory concentrates.

Figures 6 to 8 present photomicrographs of several concentrates (mostly refractory) and their PLATSOL® leach residues.

These results, together with others not presented here, indicated clearly that some PGM minerals were not responding to the PLATSOL® leach process. The main refractory mineral was shown to be cooperite (PtS) and other associated cooperite minerals (Pt, Pd) S, (Pt, Ru) S. Further examination of representative leach residues showed that the refractory Pt minerals were overwhelmingly liberated, indicating that the non-leachability of those minerals under PLATSOL® conditions was due to chemical rather than physical (encapsulation, rimming) reasons. An example of a well-liberated cooperite grain in a PLATSOL® residue is shown in Figure 9. Clearly preserved crystal faces and forms, together with grain sizes corresponds to feed sizes, provides evidence that these crystals have survived leaching intact.

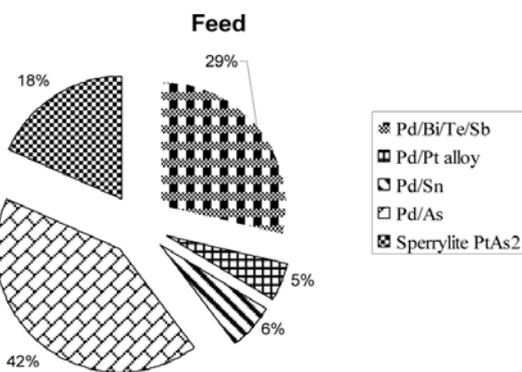


Figure 2. PGM Minerals Distribution in NorthMet Feed to the PLATSOL® Leach

PGM MINERALS	OCCURRENCE
PdSn	With or within SnO <sub>2</sub> , Fe-OX rim
Pt AsS	Fe-SO <sub>4</sub> rim
Pd/Sb/S	Fe-Ox, Fe-SO <sub>4</sub>
Pd/Sb	Fe-Ox

Table V. NorthMet PLATSOL® Residue: Description of PGM Minerals

PGM Distribution in Feed

PGM Distribution in PLATSOL® Residue

acid addition were tested. Some of the results are presented in Table VI.

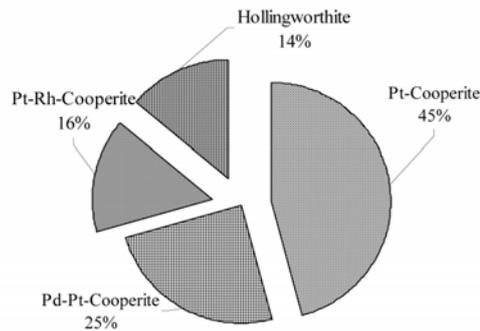
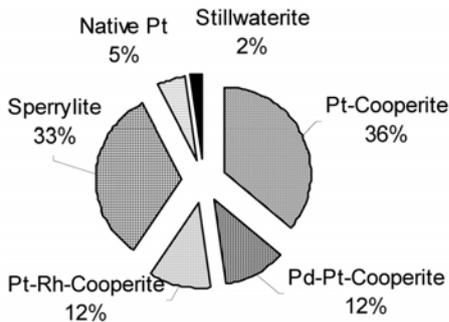


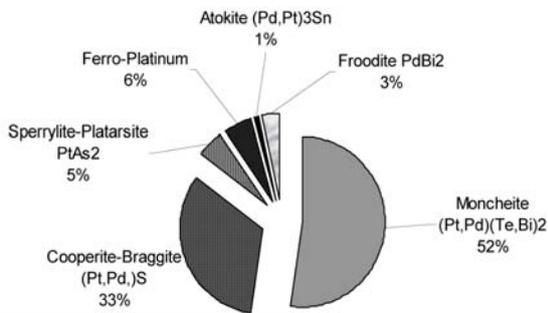
Figure 3. PGM Distribution in the Feed and Residue from a PLATSOL® Leach Example 1 of a refractory concentrate

None of these attempts was successful to improve the leachability of some of the PGM minerals with the exception of a high temperature pre-treatment procedure. Results after the pretreatment were spectacular (Table VI), and a more detailed investigation was carried out. Some results have been presented earlier (15,16).

PGM Distribution in Feed

PGM Distribution in PLATSOL® Residue

Pre-treatment tests were conducted in a muffle furnace or a tube furnace, depending on the level of gaseous atmosphere control required. The main parameters examined were temperature, retention time and gaseous atmosphere. The results are presented graphically in Figures 10 and 11 to show the effects of temperature and retention time, respectively. The effect of gaseous atmosphere in the furnace on PLATSOL® extractions is presented in Table VII.



100% Cooperite-braggite (Pt,Pd)S

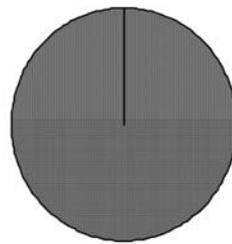


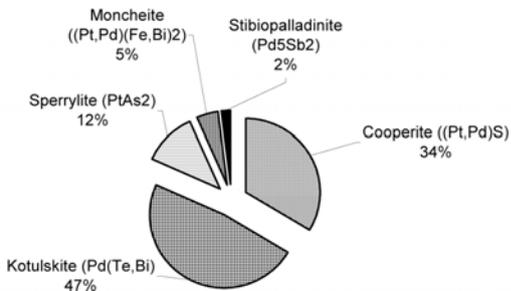
Figure 4 – PGM Minerals Present in the Feed and Residue from a PLATSOL® Leach Example 2 of a refractory concentrate

The results presented in Figure 11 indicated the optimum roasting temperature for both platinum and palladium minerals is around 650°C, at one hour retention time.

PGM Distribution in Feed

PGM Distribution in PLATSOL® Residue

As indicated in Figure 10, the effect of the pre-treatment is fairly rapid, and the maximum improvement is achieved between 30 and 60 minutes at 650°C. The trends in Figure 10 show that there are no negative effects from overextending the roasting pre-treatment to 240 minutes.



100% Cooperite-braggite (Pt,Pd)S

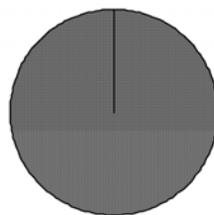


Figure 5 – PGM Minerals Present in the Feed and Residue from a PLATSOL® Leach Example 3 of a refractory concentrate

Cooperite has a tetragonal structure, and a theoretical composition of 85.9% Pt, 14.1% S, although the platinum can be substituted by various quantities of Ni, Pd, Rh, Ru. Cooperite and its dimorphous braggite are commonly found in the Merensky Reef of the Bushveld Complex (13). It is well known that some PGM concentrates are fairly difficult to dissolve, and it was reported as early as the 1920's that cooperite was non-reactive to aqua regia (14).

### IMPROVEMENTS TO THE PLATSOL® PROCESS

Two concentrates that were refractory to the PLATSOL® process were selected for a more detailed study. The chemical analyses of the two concentrates have been presented in Table II. Concentrate L contained only 0.23% S, while concentrate M was a more typical sulphide concentrate.

Initial attempts to improve the extractions of PGM's relied on modifying the standard PLATSOL® leach conditions. In particular, increasing the temperature, retention time, adding a strong oxidiser as a catalyst, increasing chloride addition and decreasing

The results in Table VII show that the gaseous atmosphere in the furnace affects the efficiency of the pre-treatment, but the effect is not extreme, compare extractions with a nitrogen atmosphere (Test 6) with those of an air atmosphere (Test 1). The results indicate that the muffle furnace atmosphere had sufficient air and was oxidising enough even when purged with nitrogen to produce the desired effect (compare tests 1 and 5).

Under all these conditions, base metal (Cu, Ni) extractions were not affected and remained very high.

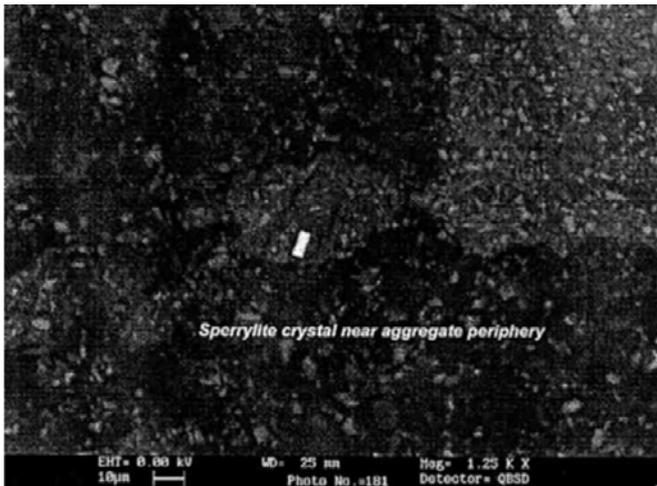


Figure 6A. BSE Photomicrograph illustrating a rectangular-shaped sperrylite crystal (PtAs<sub>2</sub>) accompanied by silicate gangue slimes. NorthMet feed.

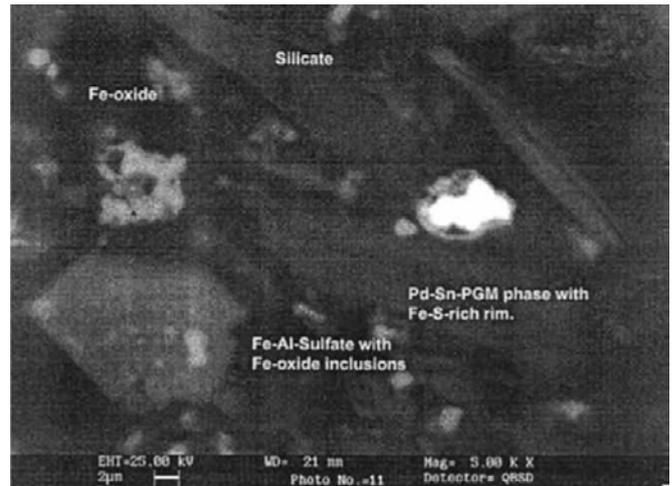


Figure 6B. Pd-Sn-PGM phase with semi-spherical overgrowth of Fe-sulfate. Also present are well-formed, Fe-Al-Sulfate with Fe-oxide inclusions, discrete Fe-oxide and leached/partially leached silicates. NorthMet PLATSOL® leach residue.

Having determined that a pre-treatment would benefit most refractory Pt concentrates, the process involving pre-treatment followed by a standard PLATSOL® leach process (“Optimised” PLATSOL®) was applied to several concentrates that had previously been shown to be refractory to the PLATSOL® process. The results of the optimised PLATSOL® leach are presented in Table XI for the same concentrates presented in Table V.

Significantly higher PGM extractions, more particularly Pt extraction, resulted from the introduction of a pre-treatment step prior to the conventional PLATSOL® leach. It is noticeable that the optimised PLATSOL® process also improves platinum and palladium leach extractions from used automobile catalyst (Sample T).

### IMPROVEMENTS TO THE PLATSOL® PROCESS - MINERALOGICAL STANDPOINT

The examples given above have clearly indicated the beneficial effect of a high temperature pre-treatment of some PGE minerals prior to the PLATSOL® leach process. A detailed mineralogical examination was conducted to understand the effect of the pre-treatment on the concentrate. A complete set of results is presented in Figure 12 below, showing the PGM assemblages of the concentrate, its PLATSOL® leach residue, and the same concentrate after 2-hour pre-treatment at 650°C in a muffle furnace.

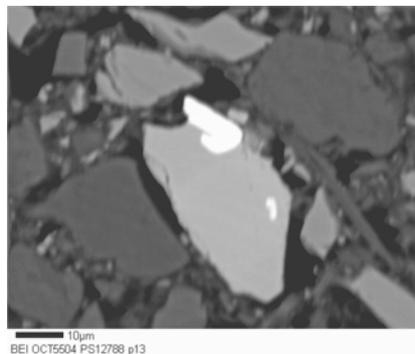


Figure 7A. Pd-Cooperite attached to Pyrrhotite (4.8x4.0µm). Feed to PLATSOL®

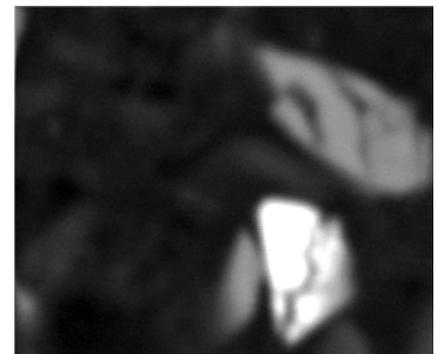


Figure 7B. Liberated Rh-Pt Cooperite (3.0x2.5µm). PLATSOL® Leach Residue



Figure 8A. Platarsite (4.3x3.1µm) – Sperrylite (8.1x4.6µm) Binary. Feed to PLATSOL®

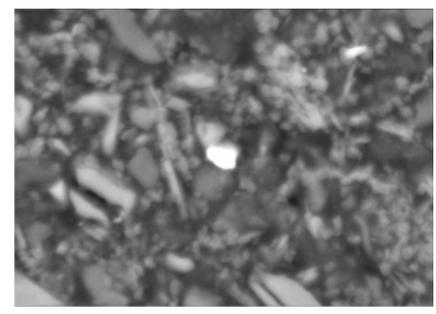


Figure 8B. Liberated Cooperite (2.9x1.5µm). PLATSOL® Leach Residue

Examination of the PLATSOL® leach residue showed that about 90% of the PGM minerals that had not dissolved belong to the cooperite family (Pt, Pd, Rh, Ru)S, the remainder being represented by a Pt-Pd-Ru sulpho-arsenide mineral. Direct PLATSOL® leaching (without pretreatment) of that concentrate recovered only 53% Pt and 76.6% Pd.

After pre-treatment, all the “cooperite” and the Pt-Pd sulpho-arsenides have

been transformed to various minerals such as native metals and various alloys. These newly formed PGM species respond much better to the PLATSOL® leach conditions, since recoveries of platinum and palladium increased to 95% and 96%, respectively, after pre-treatment. Photomicrographs of a pretreated refractory concentrate are presented in Figure 13.

TEST CONDITIONS	CONC L			CONC M	
	% EXTRACTION			% EXTRACTION	
	Au	Pt	Pd	Pt	Pd
Standard PLATSOL®	93.2	36.6	70.2	37.0	73.8
Higher Temperature, more NaCl	51.3	41.9	62.4	-	-
Additional Oxidant (V <sub>2</sub> O <sub>5</sub> )	97.4	39.3	70.9	-	-
Longer retention	-	-	-	33.9	78.1
MgO pre-leach	-	-	-	37.3	80.3
Roasting pre-treatment	91.7	93.7	95.3	98.3	94.3

Table VI. Attempts to Improve PLATSOL® Leach Extractions

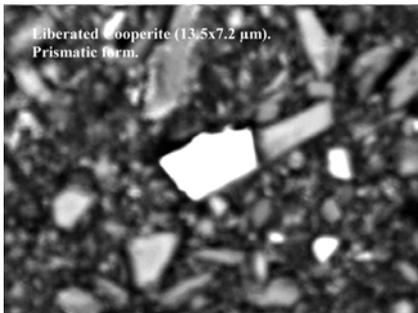


Figure 9. Liberated Cooperite Grain in a PLATSOL® Residue

### DISCUSSION

The mineralogy of PGM's is fairly complex, but, due to the invention of powerful investigative tools such as the electron microprobe (EMP), the scanning electron microscope (SEM), and energy dispersive spectrometers (EDS), significant advances have been made in recent years and new discoveries are made every year.

However, according to Cabri(12), less than about 25 PGM's may be considered to be common, and can be classified into 6 families, as shown in Figure 14.

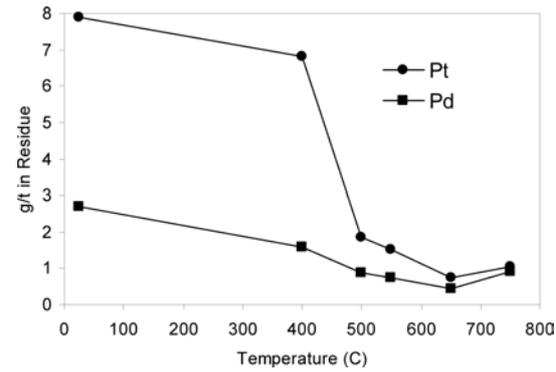


Figure 11. Effect of Pretreatment Temperature on Pt and Pd in Residue from PLATSOL® Leach (Concentrate M; Tube Furnace; 1 h, 100 mL/min air)

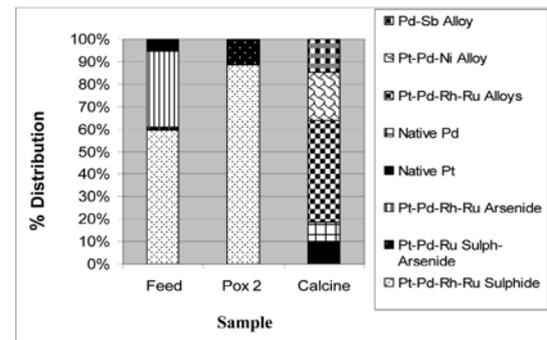


Figure 12. PGM Assemblages in a Concentrate, its PLATSOL® Leach Residue, and the Concentrate after Pre-Treatment at 650°C.

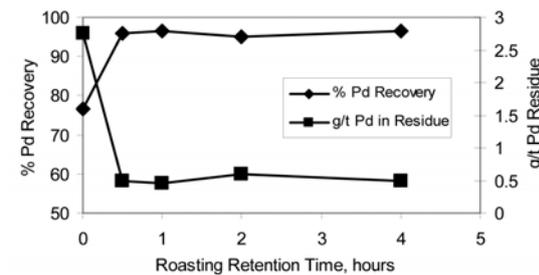
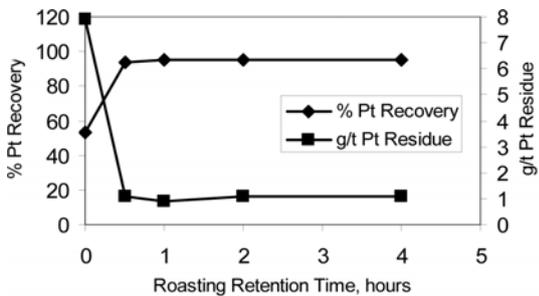


Figure 10. Effect of Pre-Roasting Residence Time on PLATSOL® Leach Extractions for Pt, Pd; Concentrate M; Tube Furnace, 650° C, 100 mL/min Air

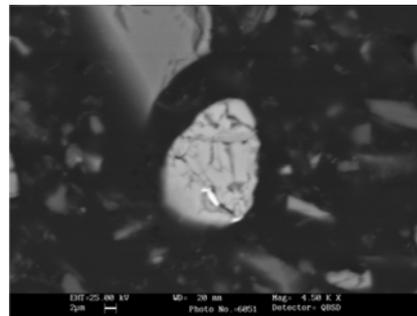


Figure 13A. Calcined product feed to PLATSOL® leach: Base metal sulphides converted to Fe-Ni metallic phase.

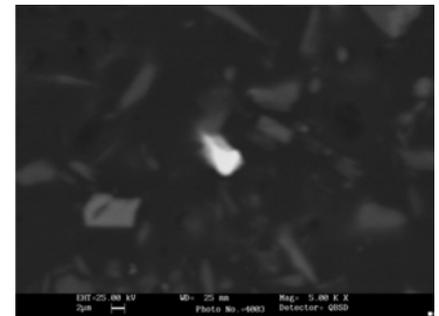


Figure 13B. Calcined product feed to PLATSOL® leach: Pt alloy associated with Fe-Ni phase.

TEST #	TEMP (°C)	TIME (HRS)	FURNACE TYPE	GAS FLOW (mL/min)	% EXTRACTION (PLATSOL® LEACH)	
					Pt	Pd
1	650	4	A	Air (100)	95	96
2	550	4	A	Air (300)	93	95
3	650	2	A	Air (500)	95	95
5	650	4	B	-	94	95
6	650	4	A	Nitrogen (500)	91	93
A = Tube furnace B = Muffle furnace						

Table VII. Concentrate B; Effect of Gaseous Atmosphere on PLATSOL® Extractions

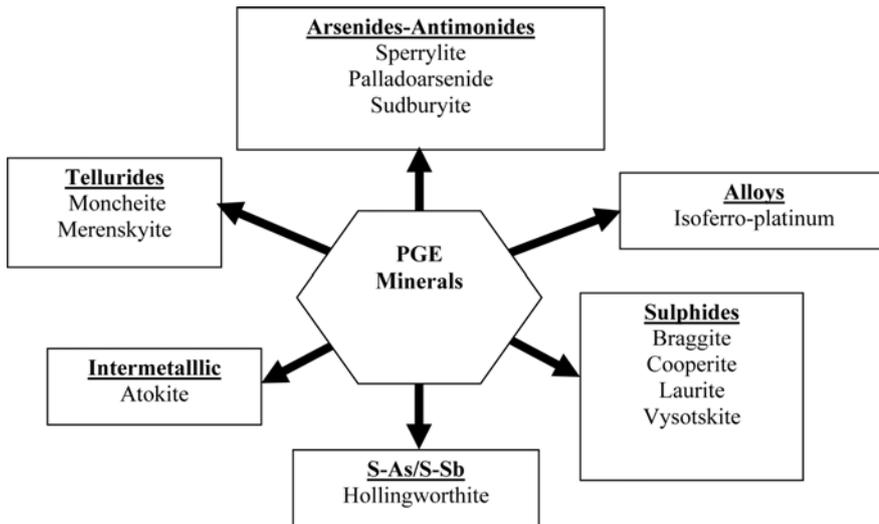


Figure 14. Most Common PGM Mineral Families (Cabri(12))

Given the complexity of PGM mineralogy, it has proven very difficult to develop a leach process successful for all types of minerals. The PLATSOL® process developed for Cu/Ni/PGM sulphide concentrates from the NorthMet deposit in Minnesota has been applied successfully to a variety of other PGM feedstocks of similar mineralogy.

However, continued testing of the process indicated that PGM minerals of the cooperite PtS extended family (Pt, Pd, Rh, Ru)S did not respond favourably to the conditions of the PLATSOL® leach process.

Thermal pre-treatment steps have often been used to improve the subsequent leaching process, and that has also been the case for the treatment of PGM's concentrates (17, 18, 19). Clearly, the efficiency of a pre-treatment step can only be measured in regards to the subsequent leach process it is designed to improve. The TML process (19) showed that the optimum temperature for the pre-treatment of the Hartley oxide ore was in the 300-450°C for leaching platinum using Geobrom®. The USBM process for the Stillwater concentrate (17) indicated that temperatures around 800°C were required to achieve ≥90% Pt dissolution using their subsequent aqueous chlorination leach process. Because of the efficiency of the PLATSOL® leach process, temperatures between 500 and 700°C were sufficient to give acceptable Pt dissolutions, without affecting Cu and Ni extractions.

A roasting pre-treatment at temperatures ranging from 500-700°C in an oxidising environment converts the cooperite into metallic elements or alloys, and these newly formed compounds respond well to the conditions of the PLATSOL® leach process. Moreover, the pre-treatment had no noticeable effect on the dissolutions of Cu and Ni during the PLATSOL® leach.

SAMPLE	% RECOVERY						
	N	O	P	Q	R	S	T*
Pt	99.0	93.7	98.0	98.3	91.8	98.3	93.7
Pd	99.6	95.3	95.9	94.3	90.5	94.3	95.2

\*Used automobile catalyst

Table VIII. Optimized PLATSOL® Results for Various Refractory Concentrates

## CONCLUSIONS

The PLATSOL® leach process is a high temperature chloride-assisted leach process that has been developed and piloted for the NorthMet bulk Cu/Ni/PGM concentrate. Its application has been successfully extended to numerous other concentrates that are similar from a mineralogical point of view.

The detailed mineralogical investigations described in this paper have allowed to identify PGM minerals that are not leachable under typical PLATSOL® conditions. The most common refractory mineral identified in PLATSOL® leach residues is cooperite PtS. Other refractory minerals identified to date are braggite (Pd,Pt,Ni)S, Vysotskite PdS, hollingworthite ((Rh,Pt,Pd)AsS). Therefore, it appears that the PGE-S bond is fairly stable under PLATSOL® conditions.

Various attempts to modify the PLATSOL® leach conditions failed completely, such as higher temperature, longer retention time, lower acidity and catalyst addition. Refractory PGM minerals can be floated quite effectively from the PLATSOL® leach residue, but the concentrate so produced cannot be simply recycled to the autoclave.

Metallurgical tests have indicated that a thermal pretreatment at temperatures between 450°- 650°C was transforming most refractory minerals (i.e. sulphides) into metallic PGE or alloys, which in turn respond well to the PLATSOL® process.

Based on these results, it has become obvious that a thorough mineralogical examination of the PGM occurrences in an ore or concentrate should greatly help determining if the given product would be amenable directly to the PLATSOL® process, or if it will require a thermal pretreatment (optimized PLATSOL®). The application of more sophisticated mineralogical methods in identifying mineralogical controls on leaching, such as coatings and trace-element compositions, is being investigated.

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