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Results from Electrochemical leach Experiments under the DOE Technical Assistant Program

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Changing the World's Energy Future

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Idaho National Laboratory

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Introduction

The activities described in this (report) are related to the analysis and testing of graphite and mine tailing samples provided by the Recipient for the extraction and recovery of embedded metals. This report focused on recovery of platinum group metals (PGMs), which appear in relatively high abundance, particularly technically important elements ruthenium and rhodium. Work performed involved assessment of materials followed by extraction tests using two methods. A novel electrochemically-driven route was compared to a chemical route for extraction of the metals. The electrochemically driven route showed similar results while producing the lixiviant in the electrochemical cell, hence reducing chemical requirements.

Sampling and characterization

Two mining products, graphite ore, and mine tailings were received from the Recipient and analyzed for metal composition focusing on the content of precious and PGMs. A total of 605.5 grams of graphite and 915.6 grams of mine tailings were received, homogenized and sieved into three different fractions: < 75 µm, 75~355 µm and >355 µm. Between 1-5 g of materials were taken from each fraction of the two different products for analysis purpose. Digestion was performed in freshly prepared aqua regia 3:1 $HCl:HNO_3$. Analysis of base, precious, and PGMs was performed through atomic absorption spectrometry (AAS) and total reflection X-ray fluorescence (TXRF), respectively. The elemental analysis composition, and weight distribution, for each one of the fractions is shown in Table 1.

Note: Technical details for the leaching methods have been removed from this version to protect intellectual property

Table 1. Size distribution and elemental composition of Recipient's graphite and tailings

	Graphite Ore (<75 μm)	Graphite Ore (75-355 μm)	Graphite Ore (>355 μm)	Tailings (<75 μm)	Tailings (75-355 μm)	Tailings (>355 μm)
Fraction weight	86.98%	11.52%	1.50%	7.17%	40.56%	52.28%
Cr	0.01%	0.01%	0.01%	0.00%	0.00%	0.00%
Ag	0.11%	0.11%	0.10%	0.00%	0.00%	0.00%
Cu	0.38%	0.37%	0.37%	0.02%	0.00%	0.00%
Al	0.21%	0.32%	0.30%	0.30%	0.19%	0.16%
Mn	0.03%	0.04%	0.03%	0.11%	0.05%	0.04%
Fe	25.83%	22.88%	24.03%	7.68%	3.60%	2.37%
Co	0.01%	0.00%	0.01%	0.14%	0.00%	0.00%
Sn	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%
Ni	0.01%	0.01%	0.01%	0.08%	0.00%	0.00%
Cd	0.07%	0.06%	0.07%	0.01%	0.00%	0.00%
Pb	1.48%	1.40%	1.52%	0.16%	0.03%	0.00%
Zn	4.95%	4.74%	4.86%	0.37%	0.06%	0.01%
Au	**0.01%**	**0.01%**	**0.01%**	**0.00%**	**0.00%**	**0.00%**
Pd	**0.05%**	**0.00%**	**0.05%**	**0.02%**	**0.00%**	**0.00%**
Ir	**0.00%**	**0.00%**	**0.00%**	**0.00%**	**0.00%**	**0.00%**
Ru	**0.24%**	**0.24%**	**0.50%**	**0.32%**	**0.09%**	**0.12%**
Rh	**0.18%**	**0.15%**	**0.32%**	**0.20%**	**0.04%**	**0.02%**
Undigested	32.52%	36.57%	3.57%	79.14%	89.66%	91.12%

During the digestion of the graphite sample a significant amount of bubbling was observed, which is an indicative of organic matter content. Mass balance analysis on the digestion of graphite and tailings show that on average only 92% of the total mass of the tailings samples was accounted within the digested metals and dissolved samples, while only 56.2% of the total mass balance was accounted for the graphite sample. As this is based on metal composition, this confirms the presence of organic and non-metal material that was digested as evidenced by the bubbles formed (CO_2).

Based on the metal analysis, the feedstock and fraction with the highest PGM and precious metals (Ag, Au) content is the graphite sample > 355 µm. However, only 9.1 g of sample were collected in this fraction, which it is not enough to perform the planned testing. The fractions with the next highest content of PGMs and precious metals are tailings < 75 µm, and graphite < 75 µm, respectively.

X-ray diffraction analysis was performed for the graphite and tailing samples with particle size < 75 µm. X-ray analysis shown in Figure 1 show silica SiO_2 is a major common component for both the graphite and tailing samples. The graphite sample appears to have a significant amount of pyrite ($FeSO_2$), which

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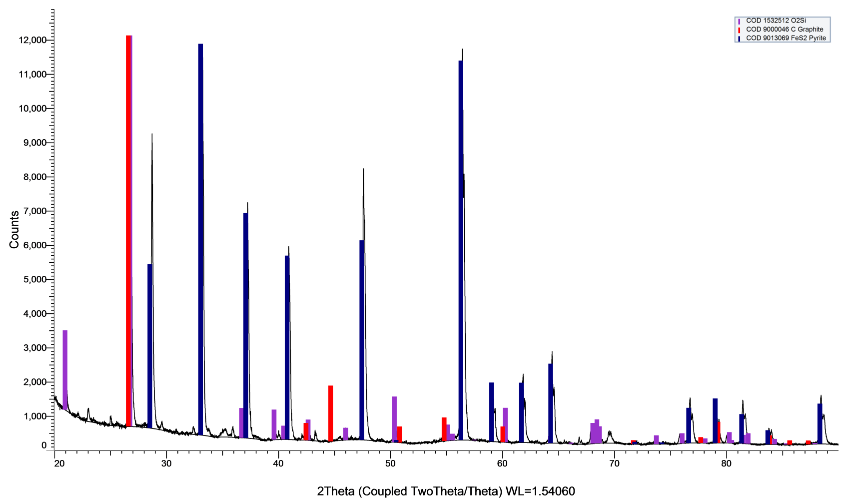
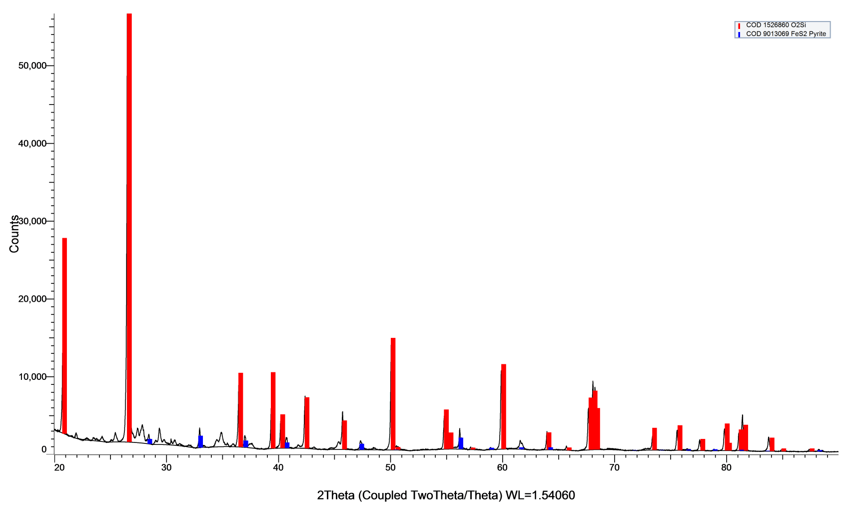
agrees with the elemental composition analysis where iron is over 20% of the total weight. However, XRD analysis did not show an adequate identification for graphite as a major component within the sample and the presence of graphite in the provided sample could not be verified.



a)



b)

Figure 1. XRD analysis of (a) graphite, and (b) tailing samples received from Recipient.

1. PGM leaching analysis

Based on the sample availability (high PGM concentration and sample amount) the tailings fraction < 75 µm was selected for PGM extraction experiments. A baseline hydrometallurgical extraction experiment was performed according to a PGM leaching procedure found in the literature. In brief the tailings were added to an acidic chloride solution containing the lixiviant. The solid suspension was heated to 65 °C

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and the extraction performed for 6 hours. Results of the hydrometallurgical extraction efficiency are presented on Table 2.

Table 2. PGM extraction efficiency through chemical leaching.

Extraction efficiency chemical leaching				
Au	Pd	Ir	Ru	Rh
59%	90%	100%	79%	74%

2. Electrochemical leaching

Electrochemical leaching experiments were performed at room temperature using an acidic chloride environment at room temperature where a lixiviant was produced in the electrochemical cell. Two electrochemical tests were performed at two different current densities with the leaching results presented in Table 2. The results show that at the higher current density the leaching efficiency of PGMs was very promising, with the benefits that the process can operate at lower temperature, with lower acid concentrations, while generating the lixiviant in-situ.

Table 2. Extraction efficiency of the electrochemical leaching of mine tailings at 11.1 and 16.67 mA/cm^2.

Current density	Extraction efficiency electrochemical leaching				
	Au	Pd	Ir	Ru	Rh
11.11 mA/cm2	8%	57%	100%	25%	32%
16.67 mA/cm2	6%	92%	100%	60%	76%

XRD analysis of the processed tailings were also performed and shown in Figure 2. However, no significant changes were observed on the tailings structure.

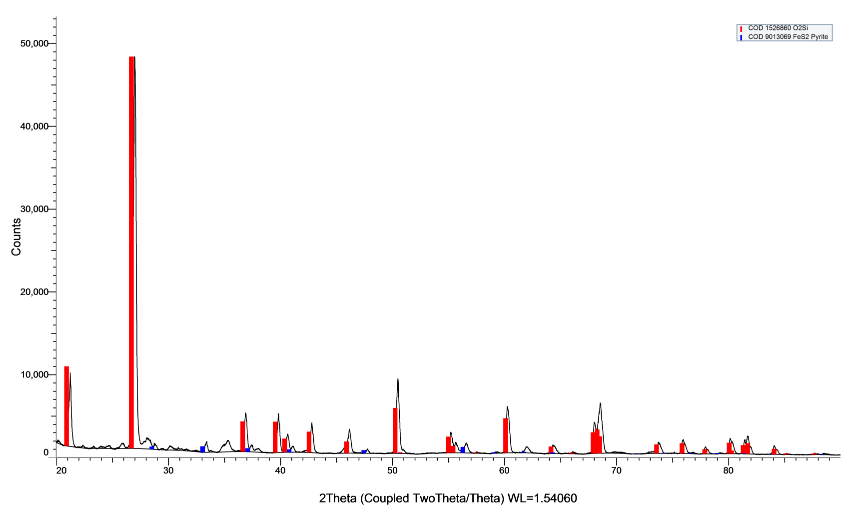

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Figure 2. XRD analysis of the processed mine tailings after PGMs extraction.

Proposed steps for process optimization and scale-up

The results show that leaching through the electrochemically generated lixiviant can produce extraction efficiencies comparable with traditional chemical leaching but with lower chemical consumption. The system employs simple construction which enables scalability. However, the limited results presented in Table 2 show that the leaching efficiency is affected by the operating current density where an optimization of this parameter is recommended as the first step toward scale-up. Additional parametric optimizations should be performed prior to scale-up to assess temperature, pulp density, and acid concentration.

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