An Investigation into

THE HYDROMETALLURGICAL UPGRADING OF GRAPHITE FLOTATION CONCENTRATE

from the

ROCKSTONE DEPOSIT

prepared for

5042078 ONTARIO INC.

Project 14748-03 – Final Report
May 18, 2022

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Introduction

SGS Natural Resources in Lakefield, Ontario was requested to conduct a brief investigation into the potential for hydrometallurgical upgrading of a flotation concentrate generated from the Rockstone graphite deposit by Mr. Ken Kukkee of 5042078 Ontario Inc. The flotation test program (SGS Project 14748-02) successfully upgraded the graphite sample to ~80% carbon content, short of the intended 95% purity. The scope of the current test program included chemical head characterization and two pressurized alkaline leaches with atmospheric acid leaching.

Testing was commenced once flotation concentrate became available in October 2021 and concluded in January 2022. Test results were forwarded to Mr. Kukkee as they became available.

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Report preparation by: M. Archer
Reviewed by: C. Mina, J. Brown

Testwork Summary

1. Flotation Concentrate Characterization

Several small flotation concentrate samples were blended and assayed for total ash content with the resultant ash assayed for 19 impurity elements (values corrected to original sample mass) by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES). The mass fraction of ash remaining after combustion was 20%, indicating a Cg purity of 80%. Analysis of the ash product is summarized in Table 1. Major impurities included 4.78% Si, 2.26% Fe, 1.81% Al, 0.51% Mg, 0.50% K, 0.41% Na, and 0.11% Ca.

Table 1: Results of the Head Ash Analysis by ICP-OES

Elemental constituent, g/t								
Al	***Ba***	***Ca***	***Cr***	***Cu***	***Fe***	***K***	***Mg***	***Mn***
18100	164	1060	470	152	22600	5030	5100	139
Mo	***Na***	***Ni***	***P***	***Si***	***Ti***	***V***	***Zn***	***Zr***
50	4080	<204	38	47800	491	36	3740	49.4

2. Test Procedures

Each test consisted of two individual leach steps – the first a pressure alkaline leach and the second an atmospheric acid leach. The intermediate graphite product was washed thoroughly before advancing to the next step.

2.1. Pressure Alkaline Leach (PAL) Testing

In the first pressure alkaline leach, graphite flotation concentrate was mixed with 200 g/L NaOH solution to produce a slurry of 20% (w/w) solids. This mixture was sealed in a Monel autoclave vessel and heated to 250°C with 345 kPa argon overpressure. The slurry was mixed at the target temperature, for one hour and then cooled, depressurized, and filtered. The residue was washed five times with hot deionized water (DI) with each wash equivalent to 1 m^3/t flotation concentrate – although the first wash in each test was marginally larger as required to rinse the Monel autoclave.

It was observed in the first test that a 20% (w/w) slurry did not produce any primary filtrate after the pressure leach. The wet reaction mass was thoroughly washed with the combined liquor assayed to complete a mass balance. The second test was conducted under similar conditions, but with a target slurry density of 10% (w/w) solids instead, and with a large first wash (450 mL). The second test was also completed without argon overpressure.

After each pressure alkaline leach, filtrate and wash samples were titrated for total alkalinity reported as NaOH and analyzed for a 30-element scan by ICP-OES. Analysis of the intermediate solids was not conducted in an effort to maximize sample for the second stage acid leaching.

2.2. Atmospheric Acid Leach (AL) Testing

In the first atmospheric acid leach, partially purified graphite (PAL residue) was mixed with DI water to produce a slurry of 20% (w/w) solids. This mixture was heated in a glass reaction kettle while adding a target dosage of acid (to achieve a solution free acidity of 20 g/L H_2SO_4). The slurry was mixed at the target temperature, following acid addition, for two hours followed by filtration. The residue was thoroughly washed (3 × 300 mL DI water) with an overall wash ratio of 18 m^3/t flotation concentrate. This wash ratio is not optimized but was selected to ensure adequate washing.

It was observed in the first test that a 20% (w/w) slurry was difficult to mix throughout the reaction time, though this test did produce primary filtrate. The second test was conducted under similar conditions, but at a target slurry density of 10% (w/w) solids.

After each atmospheric acid leach, filtrate and wash samples were titrated for free sulphuric acid and analyzed for a 30-element scan by ICP-OES. The final upgraded graphite samples were assayed for total ash content with the resultant ash assayed for 19 impurity elements (values corrected to original sample mass) by ICP-OES.

3. Combined Test Results

The pressure alkaline leach tests consumed 75.6 kg/t NaOH for PAL1 and 469 kg/t for PAL2. This difference can be attributed to the changes in slurry density between the two tests but should be further investigated with additional testing to confirm the caustic requirement. Test details along with select solution assays are summarized in Table 2.

The atmospheric acid leach tests consumed 143.1 kg/t H_2SO_4 for PAL1-AL1 and 160.2 kg/t for PAL2-AL1. Test details along with select solution assays are summarized in Table 3.

Table 2: Pressurized Alkaline Leach (PAL) Test Results Summary

Test ID	NaOH Consumed, kg/t	NaOH Add'n, kg/t	Liquor Assays				Alkalinity, g/L NaOH
			Al, mg/L	Fe, mg/L	K, mg/L	Zn, mg/L	
PAL1 Filt+Wash	75.6	672.3	271	45.6	205	73.1	37.8
PAL2 Filt	468.8	1512.6	410	31.7	481	271	150.2
PAL2 Comb. Wash	N/A	N/A	53.0	6.1	64	33.4	N/A

Table 3: Atmospheric Acid Leach (AL) Test Results Summary

Test ID	H_2SO_4 Consumed, kg/t	H_2SO_4 Add'n, kg/t	Filtrate + Wash Assays			
			Al, mg/L	Fe, mg/L	Mg, mg/L	Na, mg/L
PAL1-AL1 Filt	143.1	509.8	2770	5550	1070	3480
PAL1-AL1 Wash	N/A	N/A	230	464	92.8	290
PAL2-AL1 Filt	160.2	337.2	1290	1870	345	1710
PAL2-AL1 Wash	N/A	N/A	228	335	64.6	291

Through this two-stage process, the graphite flotation concentrate was upgraded from approximately 80% purity (measured by difference via combustion, assuming all combusted material was graphite content) to an average of 95% purity (93.2% for the residue of PAL1-AL1, 96.1% for PAL2-AL1 residue) with an overall mass loss of approximately 20%. The main remaining impurities after PAL2-AL1 included 0.9% Si, 0.6% Fe, 0.2% Al, 0.2% Mg, and 0.1% Na. Final acid leach residue assays are compared to the initial flotation concentrate in Table 4. Full test details are available in Appendix A.

Table 4: Flotation Concentrate and Acid Leach Residue Assay Summary

Sample ID	Flot Con	PAL1-AL1 Res	PAL2-AL1 Res
Cg, %	80.0	93.2	96.1
Al, g/t	18100	5020	2420
Ba, g/t	164	298	219
Ca, g/t	1060	215	245
Cr, g/t	470	101	112
Cu, g/t	152	122	111
Fe, g/t	22600	4190	6010
K, g/t	5030	1880	185
Mg, g/t	5100	1880	2350
Mn, g/t	139	38.8	48.3
Mo, g/t	50	44	49
Na, g/t	4080	736	772
Ni, g/t	<204	252	188
P, g/t	38	95	<20
Si, g/t	47800	18700	8970
Ti, g/t	491	299	222
V, g/t	36	15	<8
Zn, g/t	3740	235	49
Zr, g/t	49.4	290	20.8

Conclusions and Recommendations

Graphite flotation concentrate produced under SGS Project 14748-02 at 80% graphite purity was subjected to a pair of hydrometallurgical upgrading tests consisting of two stages (pressure alkaline leach and atmospheric acid leach). This pair of tests demonstrated that the flotation concentrate could be further upgraded through hydrometallurgical processing steps to a purity of up to 96.1%. The main impurities remaining in the leach residue include silicon, aluminum, iron, and magnesium. These process steps, performed on a suitable flotation concentrate, bring the graphite purity in line with the originally proposed goal for flotation alone – above 95%.

There is potential to optimize these process steps by adjusting variables in both the pressurized alkaline leach (caustic concentration, test temperature, reaction time) and the atmospheric acid leach (acid addition, acid type, test temperature, reaction time), but it is not expected that these changes will improve graphite purity; rather, these optimization steps would seek to minimize overall costs. Any further removal of impurities to increase graphite purity will likely require additional hydrometallurgical process steps such as acidic fluoride leaching. It is recommended that additional flotation concentrate be made available for these purposes.

Appendix A – Test Details

Project: 14748-03 **Date:** 5-Oct-21
Client: 5042078 Ontario Inc. **Technologist(s):** R.Heilmann

Test: PAL1
Objective: To investigate graphite impurity removal through two-step caustic/acid process

Sample: Flot Con

H&S: NaOH, Graphite. Review MSDS

Conditions:

	target	actual	
Leach Feed:	50	50	g as received
% Moisture:	0%	0	
Dry equiv:	50	50	g
Calculated pulp weight:	250	250	
Lixiviant to add:	200	200	g 200 g/L NaOH
Lixiviant type:	NaOH	NaOH	
target Lixiviant Concentration:	**200**	200.0	g/L NaOH
% solids (after reagent):	20.0	20.0	% solids
Total Time (h):	1		
Temperature (°C):	250		(temperature controlled with heating mantle)

Vessel: Monel Autoclave
Vessel Volume: 500 mL
Agitator RPM: **600** RPM
Target Nitrogen Over pressure: **50** psi

50	mL this is the volume of each hot- DI water wash
5	<-> this is the number of washes

Assays:

#	Elements		Streams
2	liquor samples -	ICP, NaOH	final liquor, combined wash
1	solid sample -	Ash + ICP	feed solids

Instructions:

1. Review MSDS/HAZOP: High press/temp, NaOH, Graphite. Clave-hood, vent, PPE.
2. Prepare 500 mL lixiviant (200 g 50% NaOH diluted to 500 mL). Add the feed to the lixiviant under gentle but firm mixing - do not allow vortex.
3. Seal the vessel and carry out the test - record data and observations.
4. At the conclusion of the test, shut off heat, cool down the vessel to 90°C. Ensure that that the vessel is fully depressurized before opening. Remove the head assembly and filter the sample as hot as possible.
5. Collect filtrate, measure its volume when cold, record SG and perform NaOH titration.
6. Wash on filter as per the parameters shown, label and store all washes separately. DO NOT combine the Primary Filtrate and wash solutions - sample separately.
7. Weigh the wet cake.
8. Determine % moist by drying a small subsample to constant weight @ 105ºC. Main portion wet cake-cut: direct to acid leaching - see separate sequence.
9. Combine equal volumes of each wash solution to create a single composite sample to determine SG, NaOH (by titration).
10. Store the filtrate and wash solutions in separate sealed & marked containers until needed for other testwork or disposed.
11. Record all data, verify balance, summarize results.
12. Further instructions may be provided regarding wash solution handling.

Autoclave Data

D time min	Elapsed Time min	RPM	Temp °C	Pressure (psig) Total meas	Steam	Over	Ar	Off-Gas Flow mL/min	Ar %	Remarks
					calculated					
07:00	0:00	400	22	0				-	-	heat on
07:53	0.0	600	247	177				-	-	increased temperature SP to 252°C
08:00	0	600	250	215		**0**		-	-	injecting Ar at 50psi over the 215 psi steam pressure, temperature dropped to 240°C, pressure is increasing
08:10	10	600	251	294		**79**		-	-	temp. cycling down to 244°C, pressure increasing
08:20	20	600	251	325		**110**		-	-	temp. cycling down to 244°C, pressure settling
08:30	30	600	250	322		**107**		-	-	temp. settled, reduced pressure, pressure increasing
08:40	40	600	251	321		**106**		-	-	temperature settled, pressure settled
08:50	50.0	600	250	327		**112**		-	-	
09:00	60.0	600	250	323		**108**		-	-	end test, cooling
	210		**250**	**322**	**0**	**107**	**0**	**0**	**0**	

NaOH Data *Fill out SG data. Enter aliquot data in weight or volume basis. Enter vol of titrant. Enter type of acid (HCl, H2SO4 or HNO3)*

Sample #	SG g/mL	Sample Aliquot (wght or vol based) g	mL calc	mL pipet	Aliquot mL	Titrant N	mL	Which Base	Stoich mol/mol	MW g/mole	g/L NaOH	g NaOH
Comb. PLS + Wash	1.046			5	5	0.5	9.45	NaOH	1	40.000	37.8	29.8

sum: 29.8 g NaOH (100%)

Sampling INFO

Sample #	Weight pulp, g	PLS, g	Volume PLS, mL	**emf at room T**	**pH at room T**	SG g/mL	Calc PLS Vol, mL	Wet res, g	Dry res, g	%H2O	Colours PLS	Residue	Filtration fst /slw	Pulp % solids
Comb. PLS + Wash	252	825.5	789			1.046	198.2	107.5	44.8	58.3	yell	black	fst	17.8

Final Filtration:

Diameter of filtration paper:	110		Filter Cake		Washing time:	2	min
type of paper (Whatman ##):	GF		Tare	11.7	Clarity of wash:	clear	
Filtration time:	2	min	Tare+W	119.2	Volume of wash:	~580	mL
Clarity of filtrate:	clear		Tare+D	56.5	Colour of wash:	ww	
Colour of filtrate:	yellow				Colour of solids:	black	
Cake thickness:	3.0	mm	Moisture Subsample				
			Tare	4.2			
% Moisture	58.3		Tare+W	18.6			
% Weightloss:	10.4		Tare+D	10.2			

Other Notes / Observations

NaOH Added:	33.6	g NaOH, 100%	NaOH Addition:	672.3 kg/t
NaOH Left:	29.8	g NaOH, 100%		
NaOH consumption:	75.6	kg/t Feed		

Metallurgical Balance

Element	Units	Feed	Primary Filt	Wash	PAL1 Res	Extraction to liquor	Accountability out/in	Calc Head	Primary Filt
Quant (mL/g)		50.0	789	0	45				
		Assay (mg/L, %, g/t)				%	%	%, g/t	Distribution, %
Cg	%	80.0			84	--	94.4	75.5	--
Si	mg/L, g/t	47800			23600	0.0	44	21142	--
Al	mg/L, g/t	18100	271		14200	23.6	94	16998	25
Ba	mg/L, g/t	164	0.36		250	3.5	140	230	2
Ca	mg/L, g/t	1060	2.7		2070	4.0	179	1897	2
Co	mg/L, g/t	<20	0.7		<20	55.2	145	<29	38
Cr	mg/L, g/t	470	<0.1		506	0.3	97	455	0
Cu	mg/L, g/t	152	0.2		573	2.1	340	516	1
Fe	mg/L, g/t	22600	45.6		23700	3.2	97	21951	3
K	mg/L, g/t	5030	205		1810	64.3	97	4857	67
Mg	mg/L, g/t	5100	0.17		5570	0.1	98	4992	0
Mn	mg/L, g/t	139	0.72		217	8.2	148	206	6
Mo	mg/L, g/t	50	0.8		44	25.3	104	52	24
Na	mg/L, g/t	4080	24100		12900	97.4	100	--	97
Ni	mg/L, g/t	204	1.0		436	7.7	199	406	4
P	mg/L, g/t	38	12		267	498.4	1128	429	44
Ti	mg/L, g/t	491	1.42		507	4.6	97	477	5
V	mg/L, g/t	36	1.6		15	70.2	107	39	65
Zn	mg/L, g/t	3740	73.1		2400	30.9	88	3304	35
Zr	mg/L, g/t	49.4			291	--	528	261	--

Project: 14748-03 **Date:** 8-Oct-21
Client: 5042078 Ontario Inc. **Technologist:** A. Rashleigh

Test: PAL1-AL1
Objective: To investigate graphite impurity removal through two-step caustic/acid process

Sample: PAL1 Caustic Residue

H&S: H_2SO_4. Review MSDS

Conditions:

	target	actual	
Leach Feed wet:	93.1	88.3	g as received
Target % solids (before reagent):	20.0	20.0	% solids
Leach Feed dry:	38.8	36.8	g
Calculated pulp weight:	194.0	184.0	g
Water to add:	100.9	95.7	g DI water
Total Time (h):	2		
Temperature (°C):	30		(temperature controlled with heating mantle)
Target Acidity:	20		g/L, H_2SO_4

Assays:

#	Elements		Streams
2	liquor samples -	ICP	final PLS and wash
1	solid sample -	Ash	washed residue

Instructions:

1. Review Acid Leach SOP, perform FLRA if required
2. Prepare target weight of feed and DI water into a reactor. Commence heatup.
3. Charge acid **slowly** into leach reactor. Temperature will increase during acid addition.
4. Monitor and record pH and T during test. Record any observations, weights of sample taken, reagents/water added to/from test in log sheet.
5. At end of test, the pulp + reactor was weighed and filtered.
6. The contents of the reactor were washed out onto the filter. An acidic filtrate sample was obtained and submitted for analysis.
7. The residue was displacement washed using a known amount of DI water. 3 × 300 mL, combined.
8. Volume recorded. Primary filtrate and wash separately submitted.
9. The washed filtercake was dried and weighed and submitted for analysis.

Test Data:

Time		*Reactor*				*Reagents / Feed*				*Comments*
(24 h)	(min) elapsed	Temp °C	pH	ORP		Graphite g	H_2O g	H_2SO_4 96% g		
8:35		20.9	8.99	78		88.3	97			
8:45		26.1	1.63	414				20.6		Acid added
8:53		29.4	-0.06	414						FA check - 158.6 g/L
9:45		30.6	1.29	430						
10:45		32.2	0.14	431						End test - filter
Totals/Avg:		29.6	0.8			88.3	97	20.60	0.0	

Free Acid Data *Fill out SG data. Enter aliquot data in weight or volume basis. Enter vol of titrant. Enter type of acid (HCl, H2SO4 or HNO3)*

Sample #		SG g/mL	Sample Aliquot (wght or vol based) g	mL calc	mL pipet	Aliquot mL	Titrant N	mL	Which Acid	Stoich mol/mol	MW g/mole	g/L acid	g acid	
Final	Filtrate				1	1	0.2	10.32	H_2SO_4	2	98.080	101	7.2	
Final	Wash				5	5	0.2	4.58	H_2SO_4	2	98.080	9	7.0	
												sum:	14.2 g	H2SO4 (100%)

Sampling INFO

Sample #		Weight pulp, g	Weight PLS, g	Volume PLS, mL	**emf at room T**	**pH at room T**	SG g/mL	Calc PLS Vol, mL	Wet res, g	Dry res, g	%H2O	Colours PLS	Colours Residue	Filtration fst /slw	Pulp % solids
Final	Filtrate	194	79.3	72		0.43	1.109	144.2	79.5	33.6	57.7	lt. green	black		17.4
Final	Wash		784.3	777		1.17	1.009	777.5				vp green	black		

Final Filtration:

Diameter of filtration paper:	90		Washing time:	90	min
type of paper (Whatman ##):	3		Clarity of wash:	clear	
Filtration time:	30	min	Volume of wash:		mL
Clarity of filtrate:	clear		Colour of wash:	very pale green	
Colour of filtrate:	light green		Colour of solids:	black	
Cake thickness:					

% Moisture	57.7
% Weightloss:	13.4

Other Notes / Observations

Mixing of the pulp was very difficult. The pulp tended to stay clumped to the side of the reactor with just a vortex in the middle where the impellor was. Even extremely high RPM did not remedy the build-up (tried 1800 RPM)

Acid Consumption

Acid Added:	19.8	g H_2SO_4, 100%	Acid Addition:	509.8 kg/t Feed
Acid Left:	14.2	g H_2SO_4, 100%		
Acid Consumption:	143.1	kg/t Feed		

Metallurgical Balance

Element	Units	AL1 Feed	Pri. Filt	Wash	Final res	Extraction to liquor	Stage Ext'n from flot con	Accountability out/in	Calc Head (AL1 Feed)	Pri. Filt	Final res
Quant (mL/g)		37	72	777	34						
		Assay (mg/L, %, g/t)				%	%	%	%, g/t	Distribution, %	
Cg	%	84.3			93.18	--	--	100.9	85.1	--	--
Si	mg/L, g/t	23600			18700	0.0	0.0	72	17078	--	--
Al	mg/L, g/t	14200	2770	230	5020	72.2	55	104	14831	36	31
Ba	mg/L, g/t	250	0.18	0.10	298	1.0	1	110	275	0	99
Ca	mg/L, g/t	2070	522	46.0	215	96.0	92	105	2183	46	9
Co	mg/L, g/t	<20	3.9	0.4	<20	80.2	36	172	34	22	53
Cr	mg/L, g/t	506	116	9.9	101	85.9	86	104	527	43	18
Cu	mg/L, g/t	573	151	13.8	122	102.1	100	122	697	42	16
Fe	mg/L, g/t	23700	5550	464	4190	86.9	84	103	24423	44	16
K	mg/L, g/t	1810	47	4	1880	9.7	3	105	1893	5	91
Mg	mg/L, g/t	5570	1070	92.8	1880	72.6	73	103	5758	36	30
Mn	mg/L, g/t	217	59.4	7.33	38.8	124.6	114	141	306	38	12
Mo	mg/L, g/t	44	0.7	<0.6	44	31.9	24	123	54	3	74
Na	mg/L, g/t	12900	3480	290	736	100.0	3	105	13567	50	5
Ni	mg/L, g/t	436	67	6	252	59.0	54	112	487	27	47
P	mg/L, g/t	267	60	<8	95	107.0	--	140	372	31	23
Ti	mg/L, g/t	507	68.4	5.79	299	50.4	48	104	528	25	52
V	mg/L, g/t	15	0.6	<0.2	15	36.0	11	127	19	6	72
Zn	mg/L, g/t	2400	700	65.5	235	114.4	79	123	2960	46	7
Zr	mg/L, g/t	291			290	0.0	--	91	265	0	100

Project: 14748-03
Client: 5042078 Ontario Inc.

Date: 6-Jan-22 10
Technologist(s): A. Rashleigh

Test: PAL2
Objective: To investigate graphite impurity removal through two-step caustic/acid process

Sample: Flot Con

H&S: NaOH, Graphite. Review MSDS

Conditions:

	target	actual	
Leach Feed:	50	50	g as received
% Moisture:	0%	0	
Dry equiv:	50	50	g
Calculated pulp weight:	500	500	
Lixiviant to add:	450	450	g 200 g/L NaOH
Lixiviant type:	NaOH	NaOH	
target Lixiviant Concentration:	**200**	200.0	g/L NaOH
% solids (after reagent):	10.0	10.0	% solids
Total Time (h):	1		
Temperature (°C):	250		(temperature controlled with heating mantle)

Vessel: Monel Autoclave
Vessel Volume: 500 mL
Agitator RPM: **600** RPM
Target Nitrogen Over pressure: **50** psi

50	mL this is the volume of each hot- DI water wash
5	<-> this is the number of washes

Assays:

#	Elements	Streams
2	liquor samples - ICP, NaOH	final liquor, combined wash
0	solid sample -	

Instructions:

1. Review MSDS/HAZOP: High press/temp, NaOH, Graphite. Clave-hood, vent, PPE.
2. Prepare 500 mL lixiviant (200 g 50% NaOH diluted to 500 mL). Add the feed to the lixiviant under gentle but firm mixing - do not allow vortex.
3. Seal the vessel and carry out the test - record data and observations.
4. At the conclusion of the test, shut off heat, cool down the vessel to 90°C. Ensure that that the vessel is fully depressurized before opening. Remove the head assembly and filter the sample as hot as possible.
5. Collect filtrate, measure its volume when cold, record SG and perform NaOH titration.
6. Wash on filter as per the parameters shown, label and store all washes separately. DO NOT combine the Primary Filtrate and wash solutions - sample separately.
7. Weigh the wet cake.
8. Determine % moist by drying a small subsample to constant weight @ 105ºC. Main portion wet cake-cut: direct to acid leaching - see separate sequence.
9. Combine equal volumes of each wash solution to create a single composite sample to determine SG, NaOH (by titration).
10. Store the filtrate and wash solutions in separate sealed & marked containers until needed for other testwork or disposed.
11. Record all data, verify balance, summarize results.
12. Further instructions may be provided regarding wash solution handling.

Autoclave Data

D time min	Elapsed Time min	RPM	Temp °C	Pressure (psig) Total meas	Steam calculated	Over calculated	Ar calculated	Off-Gas Flow mL/min	Off-Gas Ar %	Remarks
								-	-	heat on
6:20		700	28	0				-	-	Begin heating
7:37		729	250	345				-	-	At temperature
7:52	15	730	249	335				-	-	
8:07	30	733	250	321				-	-	
8:22	45	735	248	313				-	-	
8:37	60	735	252	331				-	-	End test - cool and filter
								-	-	
								-	-	
	150		**250**	**324**	**0**	**0**	**0**	**0**	**0**	

NaOH Data *Fill out SG data. Enter aliquot data in weight or volume basis. Enter vol of titrant. Enter type of acid (HCl, H2SO4 or HNO3)*

Sample #	SG g/mL	Sample Aliquot (wght or vol based) g	mL calc	mL pipet	Aliquot mL	Titrant N	Titrant mL	Which Base	Stoich mol/mol	MW g/mole	g/L NaOH	g NaOH
Final Filtrate	1.157			1	1	0.5	7.51	NaOH	1	40.000	150.2	27.4
WASH 1	1.065			1	1	0.5	2.76	NaOH	1	40.000	55.2	23.8
WASH 2	1.011			5	5	0.5	2.73	NaOH	1	40.000	10.9	0.7
WASH 3	1.004			5	5	0.5	1.25	NaOH	1	40.000	5.0	0.3
											sum:	27.4 g NaOH (100%)

Sampling INFO

Sample #	Weight pulp, g	Weight PLS, g	Volume PLS, mL	**emf at room T**	**pH at room T**	SG g/mL	Calc PLS Vol, mL	Wet res, g	Dry res, g	%H2O	Colours PLS	Colours Residue	Filtration fst /slw	Pulp % solids
Final Filtrate	429	211.2	182.6	**-814**	**12.17**	1.1566					yllw-orang	black		
WASH 1		458.7	430.8	**-573**	**12.62**	1.0647					lt yllw	black		
WASH 2		64	63.3	**-250**	**12.82**	1.0109					vp yllw	black		
WASH 3		58.7	58.5	**-248**	**12.71**	1.0038					ww	black		
WASH 4		59.4	59.1	**-249**	**12.66**	1.0046					ww	black		
WASH 5		91.3	91.2	**-236**	**12.63**	1.0011					ww	black		
Comp WASH		50.7	50			1.018		98.4	40.2	59.2	ww	black		#DIV/0!

Final Filtration:

Diameter of filtration paper:	110		Filter Cake		Washing time:	10	min
type of paper (Whatman ##):	GF		Tare	29.5	Clarity of wash:	clear	
Filtration time:	5	min	Tare+W	127.9	Volume of wash:		mL
Clarity of filtrate:	clear		Tare+D	69.7	Colour of wash:	colorless	
Colour of filtrate:	yellow-orange				Colour of solids:	black	
Cake thickness:		mm	Moisture Subsample				
			Tare	19.4			
% Moisture	59.2		Tare+W	20.9			
% Weightloss:	19.6		Tare+D	20.0			

Other Notes / Observations

No N2/Ar overpressure was applied during test
Comp Wash solution was 10 mL each of WASH 1-5

NaOH Added:	75.6	g NaOH, 100%	NaOH Addition:	1512.6 kg/t
NaOH Left:	27.4	g NaOH, 100%		
NaOH consumption:	964.1	kg/t Feed		

Metallurgical Balance

Element	Units	Feed	Primary Filt	Wash	Extraction to liquor	Accountability out/in	Calc Head	Primary Filt
Quant (mL/g)		50.0	183	703				
		Assay (mg/L, %, g/t)			%	%	%, g/t	Distribution, %
Cg	%	80.0			--	100.0	0.0	--
Si	mg/L, g/t	47800			--	100	0	--
Al	mg/L, g/t	18100	410	53.0	8.3	100	2242	67
Ba	mg/L, g/t	164	1.56	0.030	3.5	100	6	93
Ca	mg/L, g/t	1060	1.2	3.1	0.4	100	48	9
Co	mg/L, g/t	<20	<0.3	<0.3	5.5	100	5	21
Cr	mg/L, g/t	470	0.1	<0.1	0.1	100	2	21
Cu	mg/L, g/t	152	7.0	1.6	16.8	100	48	53
Fe	mg/L, g/t	22600	31.7	6.1	0.5	100	202	57
K	mg/L, g/t	5030	481	64	34.9	100	2656	66
Mg	mg/L, g/t	5100	0.09	0.19	0.0	100	3	11
Mn	mg/L, g/t	139	0.41	0.08	1.1	100	3	57
Mo	mg/L, g/t	50	1.1	<0.6	8.0	100	12	32
Na	mg/L, g/t	4080	79800	9820	33.4	100	--	68
Ni	mg/L, g/t	204	<0.6	<0.6	1.1	100	11	21
P	mg/L, g/t	38	12	<5	115.3	300	114	38
Ti	mg/L, g/t	491	2.01	0.32	1.5	100	12	62
V	mg/L, g/t	36	3.5	0.4	35.5	100	18	69
Zn	mg/L, g/t	3740	271	33.4	26.5	100	1459	68
Zr	mg/L, g/t	49.4			--	100	0	--

Project: 14748-03 **Date:** 7-Jan-22
Client: 5042078 Ontario Inc. **Technologist:** Y.A

Test: PAL2-AL1
Objective: To investigate graphite impurity removal through two-step caustic/acid process

Sample: PAL2 Caustic Residue

H&S: H_2SO_4. Review MSDS

Conditions:

	target	actual	
Leach Feed wet:	96.9	96.6	g as received
Target % solids (before reagent):	10.0	9.8	% solids
Leach Feed dry:	39.6	39.5	g
Calculated pulp weight:	395.7	402.4	g
Water to add:	298.8	305.8	g DI water
Total Time (h):	2		
Temperature (°C):	30		(temperature controlled with heating mantle)
Target Acidity:	20		g/L, H_2SO_4

Assays:

#	Elements		Streams
2	liquor samples -	ICP	final PLS and wash
1	solid sample -	Ash	washed residue

Instructions:

1. Review Acid Leach SOP, perform FLRA if required
2. Prepare target weight of feed and DI water into a reactor. Commence heatup.
3. Charge acid **slowly** into leach reactor. Temperature will increase during acid addition.
4. Monitor and record pH and T during test. Record any observations, weights of sample taken, reagents/water added to/from test in log sheet.
5. At end of test, the pulp + reactor was weighed and filtered.
6. The contents of the reactor were washed out onto the filter. An acidic filtrate sample was obtained and submitted for analysis.
7. The residue was displacement washed using a known amount of DI water. 3 × 300 mL, combined.
8. Volume recorded. Primary filtrate and wash separately submitted.
9. The washed filtercake was dried and weighed and submitted for analysis.

Test Data:

Time		*Reactor*				*Reagents / Feed*				*Comments*
(24 h)	(min) elapsed	Temp °C	pH	ORP		Graphite g	H_2O g	H_2SO_4 96% g		
8:35		22.3	10.94	-368		96.6	299			
8:39		26.5	10.96	-365			7	0.4		
8:45		29.8	7.55	-94						
8:52		34.3	2.64	357				4.20		
9:08		34.7	1.83	403				1.60		FA 2.6 g/L
9:25		36.0	1.26	439				4.90		FA 14.02 g/L
9:35		35.5	1.06	470				1.80		FA 18.9 g/L
9:42	0	35.3	0.99	476				1.00		FA 21.5 g/L
Totals/Avg:		32.4	2.9			96.6	306	13.90	0.0	

Free Acid Data *Fill out SG data. Enter aliquot data in weight or volume basis. Enter vol of titrant. Enter type of acid (HCl, H2SO4 or HNO3)*

Sample #		SG g/mL	Sample Aliquot (wght or vol based) mL	mL calc	mL pipet	Aliquot mL	Titrant N	mL	Which Acid	Stoich mol/mol	MW g/mole	g/L acid	g acid	
Final	Filtrate	1.034	1.00	0.97	1	0.967	0.2	2.13	H_2SO_4	2	98.080	22	5.1	
Final	Wash	1.005	5.0	4.97	5	4.9741	0.2	2.10	H_2SO_4	2	98.080	4	1.9	
												sum:	7.0 g	H2SO4 (100%)

Sampling INFO

Sample #		Weight pulp, g	Weight PLS, g	Volume PLS, mL	**emf at room T**	**pH at room T**	SG g/mL	Calc PLS Vol, mL	Wet res, g	Dry res, g	%H2O	Colours PLS	Colours Residue	Filtration fst /slw	Pulp % solids
Final	Filtrate	377	245.2	237	483	1.33	1.034	334.0	86.3	31.3	63.7				8.3
Final	Wash		457.2	455	509	1.61	1.005	454.8							

Final Filtration:

Diameter of filtration paper:	90		Washing time:		min
type of paper (Whatman ##):	3		Clarity of wash:		
Filtration time:		min	Volume of wash:		mL
Clarity of filtrate:			Colour of wash:		
Colour of filtrate:			Colour of solids:		
Cake thickness:					

% Moisture	63.7
% Weightloss:	20.9

Other Notes / Observations

Acid Consumption

Acid Added:	13.3	g H_2SO_4, 100%	Acid Addition:	337.2 kg/t Feed
Acid Left:	7.0	g H_2SO_4, 100%		
Acid Consumption:	160.2	kg/t Feed		

Metallurgical Balance

Element	Units	AL1 Feed	Pri. Filt	Wash	Final res
Quant (mL/g)		39	237	455	31
		Assay (mg/L, %, g/t)			
Cg	%				96.11
Si	mg/L, g/t				8970
Al	mg/L, g/t		1290	228	2420
Ba	mg/L, g/t		0.098	0.072	219
Ca	mg/L, g/t		192	35.0	245
Co	mg/L, g/t		<0.8	<0.8	<20
Cr	mg/L, g/t		44.1	7.9	112
Cu	mg/L, g/t		36	6.7	111
Fe	mg/L, g/t		1870	335	6010
K	mg/L, g/t		15	<3	185
Mg	mg/L, g/t		345	64.6	2350
Mn	mg/L, g/t		17.1	3.41	48.3
Mo	mg/L, g/t		<0.6	<0.6	49
Na	mg/L, g/t		1710	291	772
Ni	mg/L, g/t		26.8	5.2	188
P	mg/L, g/t		<5	<5	<20
Ti	mg/L, g/t		30.9	5.86	222
V	mg/L, g/t		<0.2	<0.2	<8
Zn	mg/L, g/t		65.9	12.7	49
Zr	mg/L, g/t				20.8

Extraction to liquor	Stage Ext'n from flot con	Accountability out/in	Calc Head (AL1 Feed)	Pri. Filt	Final res
%	%	%	%, g/t	Distribution, %	
--	--	76.6	76.2	--	--
--	--	12	7116	--	--
52.6	48	62	12301	63	16
0.7	1	89	175	0	99
123.7	123	139	1752	66	11
76.8	73	164	30	16	53
61.1	61	76	445	60	20
226.6	188	295	381	57	23
54.2	54	71	19868	57	24
4.2	3	9	271	33	54
44.4	44	74	4682	44	40
83.7	83	106	180	57	21
22.5	21	106	49	7	79
2.5	2	3	14244	72	4
91.9	91	154	370	44	40
--	--	--	104	29	15
42.5	42	72	429	43	41
16.0	10	45	10	12	64
19.1	14	20	581	68	7
--	--	--	--	--	--