# INCORPORATION OF BLACK MASS RECYCLING INTO A HYDROMETALLURGICAL REFINERY

By

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# ABSTRACT

To maximise cash flow, typically, the highest grade material is processed early in the mine life, inevitably resulting in increasingly under-utilised refinery capacity over time. For an integrated nickel laterite mine and refinery, such as the proposed Sunrise Energy Metals project located in central NSW, the introduction of black mass from recycled Ni/Co-rich lithium-ion batteries represents an opportunity to better utilise an existing asset. Moreover, nickel and cobalt products with a proportion of recycled metal content are attractive to markets such as the European Union, where the reuse of the metals is mandated.

A conceptual process for incorporating Ni and Co from black mass has been developed. Bench-scale testwork on representative samples of Ni- and Co-rich black mass have established the optimum conditions for the recovery of Ni and Co and removal of foreign impurities (Li, F, P, organics) in the minimum number of steps to produce a material suitable for introduction into the refinery.

Keywords: black mass, recycling, lithium-ion batteries, nickel, cobalt

# INTRODUCTION

During the life of the Sunrise operation, as for most mines, the refinery capacity is expected to be progressively underutilised, as the highest grade ore is processed in the first years of the operation to maximise cash flow. As such, in later years, there is scope for the refinery to process additional Ni and Co units from a source that does not require the autoclave for leaching. This equipment will be fully utilised as progressively lower-grade ore is processed. Black mass from end-of-life Li-ion batteries (LiBs) fits this requirement since it is a high-grade material that can be leached under atmospheric conditions.

Black mass is an intermediate product from the recycling of LiBs. LiBs from electric vehicles (EVs), which are expected to make up the bulk of the battery recycle stream in the future (along with stationary energy storage), are usually organised into modules (arrays of cells) and then further into battery packs (arrays of modules). Battery packs and modules are typically dismantled, and then the cells are mechanically processed by crushing and various physical separation techniques to remove solvents and larger particles (metallic and polymeric), leaving the black mass, which is predominantly a mixture of cathode and anode powders. For Nickel Manganese Cobalt (NMC)-type batteries, the black mass is the most valuable portion of the cell, as it contains Ni and Co in the cathode active material,  $\text{LiNi}_x\text{Mn}_y\text{Co}_z\text{O}_2$ . LFP batteries, which use LiFePO<sub>4</sub> as the cathode powder, are not of interest for the Sunrise refinery as they do not contain Ni or Co.

Depending on the preceding mechanical processing steps, the black mass will contain varying proportions of the following materials:

- Cathode powder, consisting of cathode active material (LiNi<sub>x</sub>Mn<sub>y</sub>Co<sub>z</sub>O<sub>2</sub>), containing the value metals, carbon black (conductive additive), binder (PVDF)
- Anode powder (coated, spheronised graphite)
- Residual metallic copper (anode foil)
- Residual metallic aluminium (cathode foil)
- Residual electrolyte, consisting of salt (usually LiPF<sub>6</sub>) and solvent (usually a mixture of organic carbonates)
- Residual steel (cell casing)

Black mass is now a traded commodity, with payability based on the Ni, Co and Li content. Market prices are quoted by metal market analysts such as Platts.<sup>1</sup> There is currently significant interest and innovation in black mass processing, but the industry has not coalesced on a "standard" flowsheet.

Figure 1 shows a generic flowsheet for producing black mass from LiBs followed by hydrometallurgical processing to recover Ni, Co and Li. The steps involved are leaching (acidic and reducing), solid/liquid separation to remove the residue (anode powder), impurity removal (precipitation), base metal recovery (either precipitation, solvent extraction, or ion exchange) and separation, and finally lithium recovery (e.g. by precipitation as lithium carbonate). To maximise the value, the production of separate Ni and Co products is desirable, requiring further separation processes. All these steps require capital investment. Conversely, incorporating the impure base metal product into an existing refinery is a more efficient way to use existing capital equipment.



# Figure 1: Generic flowsheet for production of black mass from LiBs and hydrometallurgical recovery of Ni, Co and Li

Figure 2 shows the proposed flowsheet for the recovery of Ni and Co from black mass to deliver these metals into a Ni/Co refinery. Based on an initial literature review, sulfuric acid and a reductant (hydrogen peroxide or sodium metabisulfite) were selected as the leaching reagents.



Figure 2: Conceptual flowsheet for hydrometallurgical processing of black mass and incorporation into an existing base metal refinery

Pre-treatment with NaOH has been reported to be effective at AI removal.<sup>2-4</sup> It would also be expected to be effective at removing most of the soluble Li, P, F and organics. Leaching must break down the structure of the Li metal oxide to solubilise the Li, Ni, Mn and Co. The formal oxidation states of Ni, Mn and Co in NMC cathode active materials are +2, +4 and +3,<sup>5</sup> although Ni can also be present as +3 and Co as +4.<sup>6</sup> Due to the high oxidation states of Co and Mn, a reductant is required. Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) can act as a reductant relative to Ni, Mn and Co in high oxidation states (i.e. +3 or higher). The amount of reductant required depends on the amount of each metal and its oxidation state, so it is difficult to predict. The reactions below are for a generic LiMO<sub>2</sub> (where M = Ni, Co, Mn), in which the average oxidation state of the transition metal is +3.

H<sub>2</sub>O<sub>2</sub> as reductant: H<sub>2</sub>O<sub>2</sub> + 2LiMO<sub>2</sub> + 3H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  2MSO<sub>4</sub> + Li<sub>2</sub>SO<sub>4</sub> + 4H<sub>2</sub>O + O<sub>2</sub>

SMBS as reductant: Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> + 4LiMO<sub>2</sub> +  $6H_2SO_4 \rightarrow 4MSO_4 + 2Li_2SO_4 + 2NaHSO_4 + 5H_2O_4$ 

Figure 3 shows the concept of incorporating black mass recycling into the Sunrise refinery flowsheet. The objective is to add additional units of Ni and Co at a point where further processing is minimised, new impurities are not introduced and outlets exist for contained impurities. The Sunrise flowsheet processes laterite ore via High Pressure Acid Leach (HPAL), followed by Partial Neutralisation of the leached slurry using limestone before selectively recovering Ni and Co by continuous Resin-in-Pulp ion exchange technology (cRIP). The eluate from cRIP contains some free acid and is neutralised in the Eluate Neutralisation (EN) stage with lime to remove residual Fe, Al and Cu.

Given the Sunrise flowsheet, by converting the pre-treated black mass into a clarified leach liquor, precipitating a hydroxide product, and adding this product to the EN stage, these objectives can be achieved. Specifically:

- Pre-treatment with NaOH removes residual electrolyte (lithium hexafluorophosphate, organics) and some of the Al
- Leaching solubilises the Ni and Co (along with Li, Mn, Cu, Fe, Al)
- Leach residue consists of C and F (in graphite and PVDF), removed by filtration
- Neutralisation to pH 10 precipitates everything except Li, Na, F
- Bulk hydroxide precipitate is added to Eluate Neutralisation (EN) stage (minimal Li, F, P)
- Bulk hydroxide precipitate dissolves in EN and partially offsets the neutralisation requirement by consuming free acid
- Fe, AI and Cu are removed in EN (recycled to PN)
- Mn, Ca and Zn are removed in Impurity Solvent Extraction
- Co is recovered as CoSO<sub>4</sub>.7H<sub>2</sub>O via Cobalt SX and Crystallisation
- Ni is recovered as NiSO4.6H2O via Nickel SX and Crystallisation



Figure 3: Conceptual black mass recycling process (right) and incorporation into Sunrise refinery flowsheet (left)

# SAMPLES

NMC-type LiB black mass samples were obtained from four sources (Table 1). The samples from Switzerland and the USA were used for testing, as the Australian sample had a low Ni content and only a limited amount of sample was received from Japan. Table 2 summarises the elemental composition of the samples. Note that "moisture" likely includes some volatile organic solvent and water (measured as mass loss after drying at 105°C).

The samples from Australia and Japan were additionally characterised by XRD (Table 3). This characterisation revealed that the Ni content of the Australian sample was likely from NiMH batteries rather than NMC-type LiBs. A Ni-containing Li phase was not detected, and LCO (Li<sub>0.98</sub>CoO<sub>2</sub>) was the dominant phase (Co-only cathode powder typically used in batteries for electronics and power tools).

Source	Description	Sample
Australia	Mechanical processing of used batteries to produce black mass for sale	10 kg Black powder with mm-sized metal and plastic particles
Japan	Collect and resell black mass and scrap (used battery and production)	300 g Black powder
Switzerland	Battery recycler	10 kg Fine black powder with cm-sized strips of lightweight shredded material, no metallic pieces visible
USA	Battery recycler	10 kg Agglomerated black powder, mm-sized metal particles, strong solvent odour

# Table 1: Sources and descriptions of black mass samples



Switzerland Figure 4: Black mass samples, as received

Element	Australia	Japan	Switzerland	USA
AI	2.77	0.0369	3.87	0.38
Ba	0.00826	N/A	N/A	N/A
С	35.6	32.8	23.6	27.9
Са	0.0255	0.0037	0.0983	0.0203
Со	25.2	6.46	7.17	18.7
Cr	0.0103	0.0003	0.006	0.00532
Cu	3.72	0.116	1.52	0.437
F	2.42	0.84	1.69	1.93
Fe	0.0929	0.0035	6.10	0.0941
K	0.0317	< 0.007	0.019	0.0028
Li	3.55	4.18	2.67	3.17
Mg	0.0602	0.00252	0.0316	0.0466
Mn	2.78	6.44	5.85	4.65
Na	0.058	0.0459	0.03	0.061
Ni	2.10	20.7	10.8	6.46
Р	0.451	< 0.0003	0.471	0.275
S	0.110	0.078	0.0695	0.0402
Si	0.105	0.0054	0.048	0.0071
Sn	0.0178	N/A	N/A	N/A
Ti	0.0366	N/A	N/A	N/A
Zn	0.00902	< 0.0001	0.069	0.0101
LOI @ 850°C	39.0	35.5	38.9	33.2
Moisture (105°C)	5.25	0.139	3.5	15.4

# Table 2: Analysis of black mass samples (as received) (elemental analysis as % w/w, drybasis, LOI and moisture as % w/w, as received basis)

# Table 3: Phase analysis by XRD (Switzerland and USA samples were not analysed)

Phase name/Formula	Australia	Japan
Graphite; C	Major	Major
Lithium Cobalt Oxide; Li <sub>0.98</sub> CoO <sub>2</sub>	Major	ND
Lithium Cobalt Nickel Oxide; Li(Co <sub>0.2</sub> Ni <sub>0.8</sub> )O <sub>2</sub>	ND	Major
Lithium Dimanganate; LiMn <sub>2</sub> O <sub>4</sub>	Minor	ND
Amorphous Content	Undetermined	Undetermined

# **BENCH SCALE TESTWORK**

All solid analyses are reported on a dry mass basis. Mass balances for Ni, Co and Li were 87-105%, with an average of 98%. Accurate fluorine analysis for solids and liquors required alkaline digestion to convert all fluorine to inorganic fluoride before quantification with an ion-selective electrode. Carbon was measured by CHN analysis (combustion). Other elements were measured by ICP-AES (solids were digested by acid before analysis).

# **Pre-treatment**

Previous testing established that 1.0 M NaOH at 60°C for 2 hours was more effective than water or 0.1 M NaOH at removing soluble F, P, and organics (as well as Li and some AI). Pre-treatment was conducted in a 5 L jacketed glass reactor equipped with an overhead stirrer and condenser. Black

mass was added to the solution of NaOH and stirred for the required time, then filtered by vacuum filtration. The filter cake was washed with deionised water and used for leaching tests without drying. As shown in Table 4, substantial F and P were removed, and a portion of the Al (Co was used as a tie element for the removal calculation). Only a small amount of Li was removed, which is consistent with most of the Li in the black mass being present in the cathode powder as LiMO<sub>2</sub> and only a minority in the residual electrolyte as LiPF<sub>6</sub>.



Figure 5: Filtrate from NaOH pre-treatment

Element	Feed	Residue	Filtrate	Removal
	% w/w	% w/w	mg/L	%
AI	3.87	4.01	1440	14%
С	23.6	23.2	N/A	N/A
Ca	0.0983	0.104	< 0.009	0%
Со	7.17	8.15	< 2	0%
Cr	0.006	0.00856	< 0.09	0%
Cu	1.52	1.57	18.5	0%
F	1.69	1.1	3000	78%
Fe	6.1	7.07	< 3	0%
K	0.019	0.004	48	102%
Li	2.67	2.9	395	7%
Mg	0.0316	0.0378	< 0.04	0%
Mn	5.85	6.52	< 0.5	0%
Na	0.03	0.418	21800	67%
Ni	10.8	12.6	< 4	0%
Р	0.471	0.286	664	61%
S	0.0695	0.056	138	84%
Si	0.048	0.0658	80	75%
Zn	0.069	0.0818	3.4	2%

Table 4: Composition of black mass feed, treated residue, filtrate, and removal efficiency

# Leaching

The leaching test program was designed to systematically vary reductant and acid dosage, reductant type and pulp density while also obtaining kinetic data. Leach tests were conducted at 60°C in a 1 L jacketed glass reactor equipped with an overhead stirrer and condenser. Black mass was added to the solution of H<sub>2</sub>SO<sub>4</sub> pre-heated to 60°C, followed by slow addition of the reductant (either H<sub>2</sub>O<sub>2</sub> or SMBS) to control the heat and gas generation. Slurry samples were periodically withdrawn to obtain reaction kinetics. At the end of each test, the slurry was filtered by vacuum filtration. The filter cake was repulped with deionised water and then filtered again.

#### General observations

- The addition of black mass to the H<sub>2</sub>SO<sub>4</sub> solution resulted in heat generation
- The addition of H<sub>2</sub>O<sub>2</sub> to the mixture of H<sub>2</sub>SO<sub>4</sub> and black mass resulted in heat generation and substantial gas generation, which caused the solid material to froth up (presumably the hydrophobic graphite particles)
- The addition of SMBS also resulted in heat and gas generation, both of which were more significant as the dose of SMBS was increased
- Filtration of the solid residue was rapid, and very little material passed through Whatman #4 filter paper (pore size approximately 25 µm), except for tests with high terminal pH which were difficult to filter
- Leaching was rapid, reaching the maximum extent within 30-60 minutes



Figure 6: Black mass leaching – note frothing

#### Results

Table 5 summarises the conditions and outcome of each test. Low acid dosages, below a threshold, resulted in high terminal pH (> 2.5) and a difficult-to-filter residue, presumably due to Al/Fe hydroxide gel formation. High reductant dosages above a threshold resulted in a final ORP < 500 mV. Test #15 was done in a 5 L reactor at a larger scale, and it did not have as good accountability as the smaller scale tests. The leaching extent is likely underestimated, given that the residue had comparable Ni, Co and Li levels to test #13 (Table 6). Both C and F are higher in the residue compared to the feed.

Leach	Sample	Redu	ctant	H₂SO₄	Pulp density	PLS ORP	PLS pH	Ni	Со	Li
#	source	type	mol/kg	mol/kg	g/L	mV	-	%	%	%
1	СН	$H_2O_2$	8.17	11.38	157	612	< 1	92	72	90
2	СН	$H_2O_2$	4.12	11.47	159	609	< 1	88	62	89
3	СН	SMBS	0.23	11.31	159	675	< 1	72	54	85
4	СН	$H_2O_2$	4.22	5.60	162	533	2.7	52	40	66
5	US	$H_2O_2$	10.24	12.57	155	572	< 1	90	67	84
6	US	$H_2O_2$	4.92	12.44	160	561	< 1	78	55	81
7	US	SMBS	0.74	12.35	153	584	< 1	70	61	82
8	СН	SMBS	0.65	15.86	122	718	< 1	82	66	87
9	СН	SMBS	2.14	16.52	113	369	< 1	92	92	92
10	СН	SMBS	0.67	12.25	119	686	< 1	84	70	86
11	СН	SMBS	0.69	6.71	282	533	3.4	69	54	69
12	СН	SMBS	0.69	10.08	282	893	< 1	80	63	80
13	СН	SMBS	1.51	14.82	298	293	1.0	92	92	91
14	СН	SMBS	1.14	11.89	257	398	1.1	85	85	86
15	СН	SMBS	1.18	9.24	254	367	1.1	79	79	79

# Table 5: Test conditions and leaching extents for selected elements (based on liquor out vs solid in)

CH = Switzerland, US = United States; ORP vs. Ag|AgCl

#### Table 6: Composition of feed, PLS and solid residue from leaching test #13

Element	Feed	PLS	Solid residue
	mg/kg	mg/L	mg/kg
Ag	44	0.4	170
AI	41100	13100	4110
Ba	281	< 0.07	1090
С	236000	N/A	839000
Ca	1270	257	762
Со	81300	24800	987
Cr	95	10.8	229
Cu	16700	5050	465
F	8510	126	27000
Fe	70500	20800	16600
K	70	8	80
Li	28300	8600	290
Mg	391	103	81.4
Mn	65100	20100	362
Na	1010	23700	200
Ni	126000	38600	3010
Р	2450	733	450
Pb	818	14.1	2970
S	363	137000	2230
Si	570	157	524
Zn	892	292	64.8

Figure 7 plots Co leaching extent against acid addition, grouped by ORP and reductant. The data show that the addition of sufficient reductant to result in a final ORP <500 mV in the same acid dose

range leads to higher Co leaching. There was also a dependence on the acid dosage within each group of tests, i.e. leaching efficiency deteriorated below a threshold level of acid addition.



Figure 7: Effect of acid addition and reductant on Co leaching (filled symbols = Switzerland, open symbols = USA)

Figure 8 plots Co leaching extent against ORP, grouped by acid addition and reductant. The data show that low ORP is required for high Co leaching, very low acid results in poor leaching, very high acid at low ORP does not improve leaching, and none of the  $H_2O_2$  dosages resulted in low enough ORP to achieve high Co leaching.



Figure 8: Effect of ORP and acid addition on Co leaching, grouped by reductant type

# **Metal recovery**

#### Solution purification

Despite the very fine particle size of the graphite (typically around 15  $\mu$ m), filtration of the leach residue was surprisingly facile using regular filter paper and vacuum filtration. The free acid remaining in the clarified PLS was then neutralised using calcium carbonate (20% w/w slurry) at 50°C, producing a clean and readily filterable gypsum residue (Figure 9) with minimal loss of Li, Ni and Co (Table 7).

Element	Feed	Precipitate	Filtrate	Precipitation
	mg/L	mg/kg	mg/L	%
AI	8720	327	8759	<0.1%
Ca	161	241200	580	N/A
Со	17200	395.8	17150	<0.1%
Cr	6.6	1.69	6.69	<1%
Cu	3720	120.7	3655	<0.1%
F	86.4	500	97	13%
Fe	12000	427.8	11990	<0.1%
K	4	42	9	23%
Li	5960	200	5960	<0.1%
Mg	72.9	560	135	17%
Mn	13900	461	13800	<0.1%
Na	12400	2414	12570	<1%
Ni	26600	593.9	26340	<0.1%
Р	486	70	457	<1%
S	83800	212400	79500	5.6%
Si	98.1	170	108	3.9%
Zn	202	7.69	174	<0.1%

Table 7. Composition of reed, precipitate and mitrate nominee acto neutransation test
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Figure 9: Gypsum residue from neutralisation of free acid in black mass PLS

The next step was conceptualised to be a partial neutralisation to precipitate Fe and Al, followed by precipitation of a mixed hydroxide containing Ni and Co. However, upon testing, when the pH was raised to around 4 using 400 g/L NaOH at 40°C, the residue was not filterable, most likely due to the formation of aluminium hydroxide gel, given that the concentration of Al in the PLS was high (8.7 g/L). Bearing in mind that the destination for the mixed Ni-Co hydroxide is the Eluate Neutralisation stage of the Sunrise flowsheet, a bulk hydroxide precipitate containing Al and Fe is viable since these

elements are removed during the Eluate Neutralisation step. Furthermore, the bulk hydroxide can offset part of the lime demand in the Eluate Neutralisation unit operation.

#### Bulk hydroxide precipitation

Accordingly, another batch of filtrate from free acid neutralisation (160 mL) was neutralised with 400 g/L NaOH at 40°C to around pH 10. A sticky, brown-coloured solid with a reasonable filtration rate on filter paper using vacuum filtration was formed (Figure 10). After washing three times with deionised water, the precipitate residue (152 g) contained 76% moisture and the composition as shown in Table 8. The high residual level of Na and S indicates that washing efficiency was low, and a repulp wash would be required. Nevertheless, the desired outcome of low P and F (foreign elements in the Sunrise refinery) was achieved, with these elements present at 2.1 and 60 mg/L, respectively. Further testing should be done with other reagents, such as sodium carbonate, lime, and magnesium oxide.



Figure 10: Bulk hydroxide precipitate from neutralisation of black mass PLS with NaOH

Element	Feed liquor	Precipitate	Filtrate
	mg/L	mg/kg	mg/L
AI	8759	38890	2.4
Ва	0.04	1.57	0.01
Ca	580	1904	115
Co	17150	76960	5.5
Cr	6.69	33.9	< 0.06
Cu	3655	16490	0.99
Fe	11990	53950	2.84
К	9	< 20	13
Li	5960	4842	3410
Mg	135	681	0.29
Mn	13800	61400	6.9
Na	12570	52060	54400
Ni	26340	117500	9.6
Р	457	2050	2.1
S	79500	70410	44300
Si	108	534	1.2
Zn	174	887	0.06
F	97	683	60

Table 8: Composition of feed, precipitate and filtrate from bulk hydroxide neutralisation test

#### Lithium recovery

The bulk precipitation filtrate contained a high sodium concentration (54 g/L), a moderate concentration of Li (3.4 g/L) and minimal levels of other metals. Due to the low solubility of lithium carbonate, particularly as temperature increases, the addition of sodium carbonate to this solution should precipitate lithium carbonate.<sup>7</sup> This is usually done at 50°C using a solution of Na<sub>2</sub>CO<sub>3</sub> to maximise the solubility of Na<sub>2</sub>CO<sub>3</sub>.

Lithium precipitation: Li<sub>2</sub>SO<sub>4</sub> + Na<sub>2</sub>CO<sub>3</sub> → Li<sub>2</sub>CO<sub>3 (s)</sub> + Na<sub>2</sub>SO<sub>4</sub>

In this study, solid sodium carbonate was added to the filtrate from the bulk hydroxide precipitation to recover Li as crude  $Li_2CO_3$  (see Table 9). Despite adding a large molar excess of  $Na_2CO_3$ , Li recovery was only 39%. The resulting precipitate also contained a high level of Na and S, and the filtrate contained 2.3 g/L Li. Residual base metals remaining in the feed liquor after bulk precipitation were concentrated in the lithium precipitate.

For efficient Li recovery, the liquor would need to be concentrated before adding Na<sub>2</sub>CO<sub>3</sub>, for example, by evaporation or membrane filtration.



Figure 11: Crude lithium carbonate precipitate from liquor remaining after bulk hydroxide precipitation

Table 9:	Composition of feed liquor, precipitate and filtrate from lithium	carbonate
	precipitation test	

Element	Feed	Precipitate	Filtrate
	mg/L	mg/kg	mg/L
AI	2.4	121.2	1.8
Ва	0.01	8.29	0.04
Ca	115	10110	12
Со	5.5	475.6	0.65
Cr	< 0.06	2.13	< 0.06
Cu	0.99	72.89	0.72
F	60	1490	63
Fe	2.84	274.9	1.15
K	13	35	18
Li	3410	137900	2280
Mg	0.29	49.4	0.15
Mn	6.9	657	0.6
Na	54400	56810	128100
Ni	9.6	806.2	1.2
Р	2.1	21.2	3.2
S	44300	26550	52300

Si	1.2	61.8	5.1
Zn	0.06	20.1	0.04

#### **Overall recovery**

Table 10 lists the composition of intermediates through the black mass treatment process and the overall recovery to the refinery. Note that the metal contents are depressed in the hydroxide precipitate due to the high concentration of Na, S and Li due to insufficient washing. Importantly, F is very efficiently rejected by the black mass flowsheet.

Table 10:	Summary of	of composition o	f intermediate	products and	overall recovery	to refinery
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	Unit	Ni	Co	Li	Mn	С	F	Р	AI	Fe
Black mass	% w/w	10.8	7.17	2.67	5.85	23.6	1.69	0.47	3.87	6.1
Pre-treated residue	% w/w	12.6	8.15	2.9	6.52	23.2	1.1	0.29	4.01	7.07
Leach residue	% w/w	0.3	0.1	0.03	0.04	83.9	2.7	0.05	0.41	1.66
PLS	g/L	38.6	24.8	8.6	20.1	N/A	0.13	0.73	13.1	20.8
Gypsum precipitate	% w/w	0.06	0.04	0.02	0.05	N/A	0.05	<0.01	0.03	0.04
Hydroxide precipitate	% w/w	11.8	7.7	0.48	6.14	N/A	0.07	0.21	3.89	5.4
Recovery to refinery	%	85%	86%	14%	86%	0%	1%	35%	76%	83%

#### IMPLICATIONS

This bench scale proof of concept has confirmed that it is feasible to process black mass and produce a Ni- and Co-containing material suitable for introduction into a conventional Ni-Co refinery. Given that the black mass feed material is very high grade and leaching was effective at 300 g/L solids content, the size of the equipment can be relatively small, thus the capital cost of the process is expected to be low (especially since the refinery is sunk capital) and the operating cost is the more important factor. The major contributors to the operating cost are the cost of the black mass (based on the Ni, Co and Li content), the reagent cost for the black mass portion of the flowsheet and the additional reagent costs in the refinery to process the additional metal. These are offset to some extent by lower lime requirement in Eluate Neutralisation and higher refinery utilisation.

The optimised dosages of SMBS and  $H_2SO_4$  found for the Swiss black mass sample will necessarily change based on the composition of the black mass, but knowing that the terminal ORP needs to be below 500 mV (vs. Ag|AgCI) and the terminal pH should be around 1 provide the guidelines to quickly optimise the dosages for any given sample.

# CONCLUSIONS

There is already a market for black mass and this will only grow with time from the increasing supply of end-of-life EV batteries. This proof of concept has shown that a simple flowsheet with minimal steps can convert black mass into a form suitable to feed to a Ni-Co refinery. The point in the refinery flowsheet at which this should be added is as far downstream as possible and where outlets exist for impurities contained in the converted black mass. This alleviates building a standalone refinery for black mass and provides an existing refinery with a supply of recycled metal content to meet market requirements. Battery recycling is expected to be enforced by legislation and regulation for a minimum recycled metal content in all batteries and is already imposed in the European Union.

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