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HYDRO ALUMINIUM KURRI KURRI PTY LTD

CWS WASTE MANAGEMENT OPTION 4 REMEDIATION DESIGN AND PROPOSED VALIDATION OF TREATMENT

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Date	**15/03/2018**
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Approved by	**Fiona Robinson**
Description	**Ramboll were engaged by Hydro Aluminium Kurri Kurri (Hydro) to develop a plan of validation for the treatment of Capped Waste Stockpile materials in accordance with the Remedial Option 4, treatment with lime.**

Ref AS130515

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1. INTRODUCTION

This plan presents the process proposed to treat the aluminium smelter wastes within the Capped Waste Stockpile (CWS) so that it can be certified as ‘approved aluminium smelter wastes’ and disposed of in accordance with EHC Licence Number 05 and the *Chemical Control Order in Relation to Aluminium Smelter Wastes Containing Fluoride and/or Cyanide* (CCO).

The former Hydro Aluminium Kurri Kurri Smelter (the Smelter) is located on Hart Road, Loxford near Kurri Kurri in New South Wales, Australia. The area of land owned and managed by Hydro Aluminium Kurri Kurri Pty Ltd (Hydro) incorporates the former Smelter area (the Smelter Site) and the surrounding buffer zone (comprising approximately 2000 hectares of land in total) (the Hydro Land). Smelting activities ceased at the Smelter Site in September 2012, and in May 2014 Hydro formally announced the closure of the Smelter.

Demolition and remediation of the Smelter are key tasks to facilitating the future redevelopment of the Smelter. Remediation of the site includes remediation of a legacy landfill currently capped and situated within the Smelter footprint known as the Capped Waste Stockpile (CWS). Other remediation activities comprise the excavation and removal of contaminated soils, disposal of demolition waste materials and recycling of recyclable materials (Wastes).

A remedial options study involving a comprehensive semi-quantitative net environmental benefit analysis identified that placement of the wastes within a new purpose built Containment Cell in a geologically suitable area of the Hydro Land was the most beneficial remediation strategy. In response to the study, the NSW Environment Protection Authority (EPA) advised that the onsite containment of waste (contaminated wastes and non-recyclable demolition waste) is appropriate, however any waste in the CWS that contains levels of leachable fluoride and/or cyanide above the thresholds set out in the CCO would need to be treated to reduce these levels prior to disposal to the Containment Cell.

As such, Hydro is evaluating options for the treatment of the waste to meet the CCO for leachable fluoride concentrations. Leachable cyanide concentrations from the CWS have been documented to be below the CCO requirement. The option under consideration is treatment by application of calcium from gypsum, to chemically fix any leached fluoride to form the stable precipitate calcium fluoride (fluorite or fluorspar).

This Validation of Treatment plan has been developed to explore the treatment requirements and the manner in which these treatments can be applied in the remediation works.

This plan details: the laboratory treatability trials to evaluate if the waste can be successfully treated to below the CCO; the method for treatment of the waste during the remediation works; and the data collection proposed to validate the treatment of the waste so that it can be certified as ‘approved aluminium smelter waste’ and disposed of in accordance with the requirements of EHC Licence Number 05 and the CCO.

This Plan is provided to the EPA for review and consideration. Hydro requests the EPA to confirm in writing that the sampling, analysis and testing procedures set out in this Plan are appropriate for the purposes of:

- certifying that the aluminium smelter waste is ‘approved aluminium smelter waste’ for the purposes of the EHC Licence; and
- testing whether the aluminium smelter waste contains ‘leachable fluoride’ or ‘leachable cyanide’ for the purposes of the CCO.

1.1 Objective

The objective of the proposed treatment is to treat the aluminium smelter waste within the CWS so that it can be certified as 'approved aluminium smelter waste' for the purposes of EHC Licence Number 05 and the CCO.

1.2 Certification of Aluminium Smelter Waste

Condition 5.1 of the EHC Licence provides that aluminium smelter waste may only be disposed of if certified as being 'approved aluminium smelter waste'.

'Approved aluminium smelter waste' is defined in the EHC Licence to mean:

> *'… aluminium smelter wastes containing fluoride and/or cyanide, that contains neither leachable fluoride nor leachable cyanide.'*

'Leachable fluoride' is defined in the CCO to mean:
"Leachable fluoride" in relation to aluminium smelter wastes means those wastes that when subjected to a test as specified by the Commission, being either the United States Environmental Protection Agency "Toxicity Characteristic Leaching Procedure (TCLP, method 13xx)" or other similar specified test, produce a leachate containing more than 150mg. L-1 of fluoride.

'Leachable cyanide' is defined in the CCO to mean:

"Leachable cyanide" in relation to aluminium smelter wastes means those wastes that when subjected to a test as specified by the Commission, being either the United States Environmental Protection Agency "Toxicity Characteristic Leaching Procedure (TCLP, Method 13xx)" or other similar specified test, produce a leachate containing more than 10mg. L-1 of cyanide.

1.3 Background

The Capped Waste Stockpile (CWS) is known to contain, amongst other materials, spent pot lining. Leachable fluoride in spent pot lining occurs from cryolite (Na_3AlF_6) and sodium fluoride (NaF) that are used as a flux in the smelting process. Cyanides form when nitrogen reacts with sodium and carbon during the smelting process. Previous TCLP analysis of CWS waste found leachable fluorides on a 95% upper confidence mean of 337mg/L, and cyanides below 10mg/L (Ramboll Environ, 2016).

The Capped Waste Stockpile is an on-site stockpile comprising mixed smelter wastes that were capped in 1995. The stockpile originated during early site operations between 1969 and 1992, when smelter wastes were stored within onsite storage facilities situated along the eastern smelter boundary. Mixed wastes including Spent Pot Lining was stored in this area. In the mid 1980's changes to legislation regarding the storage of aluminium smelter wastes resulted in the improvement of storage and waste management on the site. These improvements resulted in the consolidation of wastes into one stockpile and the capping of that stockpile, now referred to as the Capped Waste Stockpile. The capping of the Capped Waste Stockpile in 1995 was designed to reduce ongoing leachate generation.

Ramboll undertook an intrusive (core drilling) investigation in October/November 2015 at the Capped Waste Stockpile. The objective of the investigation was to provide an assessment on the composition of the waste material, the underlying soil, and the groundwater conditions beneath the Capped Waste Stockpile. A total of six boreholes were drilled and subsequently developed with groundwater monitoring wells. Waste, soil, and groundwater were collected and analysed in a laboratory for a wide range of contaminants of concern.

The Capped Waste Stockpile comprises smelter derived waste of various physical and chemical states. The intended treatment is to remove the waste and mix with a dry calcium source. The waste and calcium source will then be disposed in a purposely designed, located and constructed containment cell.

1.4 Scope of Work

The scope of work comprised the following elements:

- Section 2 – incorporates characteristics of the waste as determined from testing of the waste materials
- Section 3 – outlines the test methods to be followed to evaluate the proposed treatment
- Section 4 – outlines the remediation design including the methods proposed for treatment and containment
- Section 5 – outlines the validation methodology that will be implemented during the remediation works to validate compliance with the treatment methodology
- Section 6 - presents the results of preliminary testing completed on the waste to date

2. CHARACTERISATION OF THE CAPPED WASTE STOCKPILE

2.1 Historical information

The CWS comprises approximately 365,000 tonnes of mixed historical wastes arising from the smelter operations and impacted soils lying below the stockpile. The volume of the capped waste stockpile has been approximated by survey, drilling and conversion on assume bulk density. The contents of the CWS have been approximated from historical site documents and includes spent pot lining, steel, waste anodes, asbestos containing materials and other smelter related wastes. The estimated volumes of the key waste categories within the CWS is presented in **Figure 2-1**. Whilst site knowledge of the CWS content is documented, and investigations support this information, there remains uncertainty in the actual contents of the stockpile that will not be realised until the stockpile is excavated. This uncertainty results in project, environmental and health and safety risks for all management options. These risks are being managed through various management plans to be implemented during the works and prepared in consultation with the EPA and SafeWork Australia.

From comprehensive site records and site knowledge, the Capped Waste Stockpile is known to contain:

- Spent pot lining;
- Carbon Plant shot blast refuse, including grit and dust;
- Carbon Plant dust collector product;
- Collar mix (coke, pitch) spillage;
- Carbon Plant floor sweepings;
- Packing coke oversize;
- Contaminated bath;
- Rotary breaker oversize;
- Pot lining mix (hot ramming paste);
- Rodding mix (coke, graphite, pitch and anthracene oil);
- Stud joining mix;
- Pitch spills/ pencil pitch;
- Aluminium swarf;
- Scrap aluminium billets;
- Anode cover material;
- Butt from spent anodes;
- Ahead of schedule anodes;
- Dross;
- Pot bottom aluminium;
- Consumable gaskets and insulation material (synthetic mineral fibre and asbestos);
- General rubbish, including plastic, wood and steel.

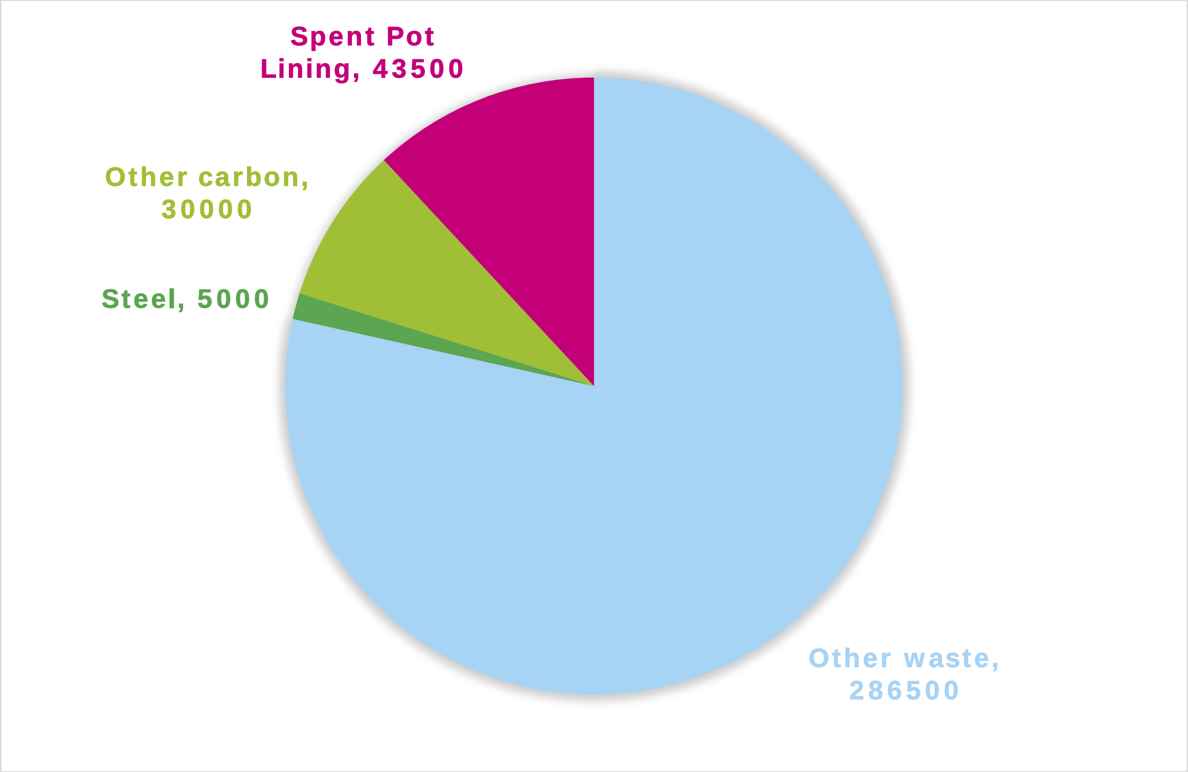

Figure 2-1 Waste Categories by Mass (T)

2.2 Physical appearance

A substantial amount of information is available to describe the physical appearance of the CWS materials. The wastes are described as being black in colour, generally dry and of small to very large sizing and with strength ranging from soft to very hard. The following photos of the CWS provide an indication of waste appearance.

2.3 Chemical Characteristics

Drilling of six boreholes and recovery of a 100mm diameter continuous core was completed in November 2015. Core material has been retained at the Smelter for testing. Initial testing of this core material has been completed to determine the chemical characteristics of the waste. **Table 2-1** outlines the waste characteristics in comparison to the *NSW EPA (2014) Waste Classification Guidelines. Part 1: Classifying Waste, 2014* and the CCO.

Table 2-1 Waste classification of untreated material using chemical assessment – all categories

Analyte	Unit	No. of samples	Min conc.[1]	Max conc.	No. above GL[2]	95% UCL[3]	Waste Classification (max. conc.)	Waste Classification (95% UCL)	Adequately Characterised Yes/No [9]
TRH C6 - C10	mg/kg	48	<25	n/a	0	-	**GSW[4]**	**GSW**	Yes
Toluene	mg/kg	48	<0.5	n/a	0	-	**GSW**	**GSW**	Yes
Benzo(a)pyrene	mg/kg	57	<0.05	832	46	193.4	**HAZ[5]**	**HAZ**	Yes
Benzo(a)pyrene in TCLP	mg/L	54	<0.001	0.002	0	-	**GSW**	**GSW**	Yes
Total +ve PAHs	mg/kg	57	0.5	6320	41	1546	**HAZ**	**HAZ**	Yes
Total +ve PAHs in TCLP	mg/L	54	NIL +ve	0.522	0	-	**GSW**	**GSW**	Yes
Arsenic	mg/kg	36	<4	850	7	189.4	**RSW[6]**	**GSW**	Yes
Arsenic in TCLP	mg/L	8	<0.05	0.08	0	-	**GSW**	**GSW**	Yes
Cadmium	mg/kg	36	<0.4	1	0	-	**GSW**	**GSW**	Yes
Lead	mg/kg	36	18	640	18	351.9	**GSW**	**GSW**	Yes
Lead in TCLP	mg/L	8	<0.03	0.56	0	-	**GSW**	**GSW**	Yes
Mercury	mg/kg	48	<0.1	0.2	0	-	**GSW**	**GSW**	Yes
Molybdenum	mg/kg	36	<1	12	0	-	**GSW**	**GSW**	Yes
Nickel	mg/kg	36	34	170	33	73.49	**GSW**	**GSW**	Yes
Nickel in TCLP	mg/L	8	0.02	0.1	0	-	**GSW**	**GSW**	Yes
Silver	mg/kg	36	<1	2	0	-	**GSW**	**GSW**	Yes
Free Cyanide in soil	mg/kg	36	<0.5	8.8	0	-	**GSW**	**GSW**	Yes
Total Cyanide	mg/kg	63	4	734	2	155.1	**GSW**	**GSW**	Yes
Total Cyanide in Neutral Leach (ASLP)	mg/L	54	0.198	10	0	-	**GSW**	**GSW**	Yes
Total Fluoride	mg/kg	55	184	51,700	47	30,585	**HAZ**	**RSW**	Yes
Fluoride in TCLP	mg/L	54	42	909	30	337	**>CCO[7]**	**>CCO[7]**	Yes
Asbestos ID	g/kg	55	Not Present	Present	23 [8]	-	**Asbestos Waste**	**Asbestos Waste**	n/a

[1] Minimum/Maximum Concentration

[2] Number of samples above Guidelines (NSW EPA Waste Classification / NSW EPA Chemical Control Order) – includes CT1, CT2, SCC1, and SCC2 for total concentrations

[3] 95% Upper Confidence Limit, calculated using ProUCL Version 5.1

[4] General Solid Waste

[5] Hazardous Waste

[6] Restricted Solid Waste

[7] NSW EPA Chemical Control Order in Relation to Aluminium Smelter Wastes Containing Fluoride and/or Cyanide

[8] All detections of asbestos, including samples less than g/kg detection limits

[9] Determined using Procedure B of the NSW EPA Sampling Design Guideline 1995, Number of samples required for determining the average concentration.

n/a – not applicable

'-' indicates 95% UCL not calculated, as maximum concentration did not exceed the guidelines

CWS material was also analysed for Organisation for Economic Co-operation and Development (OECD) Dangerous Goods Class 4.3 substances which in contact with water emit flammable gases. Sixteen samples of the CWS waste material were randomly collected from the core trays (MW201 through MW206) by Ramboll personnel and forwarded to SGS Australia Pty Ltd laboratory for analyses under CoC protocol.

Laboratory testing was carried out under the methodology described in UN Manual of tests and Criteria, Part III, Section 33.4 – Division 4.3. The substances were brought into contact with water under a variety of conditions, in which spontaneous ignition can be observed and a measure of the amount of gas evolution was taken. The evolved gas was then assessed for flammability.

The laboratory concluded that for the samples provided, no gas, including flammable gas, was observed to evolve throughout the duration of the test for each sample.

The number of samples required to calculate an average was determined using the data set obtained for each analyte. This calculation is outlined in Procedure B of the NSW EPA *Sampling Design Guidelines* (1995) and uses the average and standard deviation in combination with a confidence limit and the target concentration, to determine the number of samples required to demonstrate that an average concentration is below a target guideline. This method is applicable for determining the number of samples required to ascertain the average concentration of a stockpile. The analysis completed shows that, based on the data set variability, sufficient samples have been collected to determine the average concentration. These average concentrations are presented as 95% Upper confidence limits of the mean and are compared to the guideline concentrations as required by both the Waste Classification Guidelines, and the Chemical Control Order.

On the basis of the chemical assessment, following the classification hierarchy, material within the CWS is considered to be Hazardous Special Wastes on the basis of elevated concentrations of leachable fluoride, benzo(a)pyrene, total PAHs and the presence of asbestos fibres.

3. PRELIMINARY TREATMENT RESULTS

In 2017, Hydro commissioned Ramboll to undertake preliminary testing (Stage 1) of chemical fixation treatment methods for the Capped Waste Stockpile samples. The objective of the Stage 1 testing was to provide a preliminary indication of the efficacy of calcium in achieving the CCO compliance with respect to leachable fluoride and leachable cyanide.

Treatment study 1 involved the addition of dry hydrated lime ($Ca(OH)_2$) at 10% w/w using the moisture inherent in the CWS Waste. A bulk sample was made by homogenising subsamples from each of the recovered cores. A portion of the bulk sample was then taken and mixed with the dry lime. Six subsamples from each bulk sample were then taken and analysed for:

- NSW Landfill Suite for waste characterisation (including Total Recoverable Hydrocarbons, Benzene, Toluene, Ethylbenzene, Xylenes, Naphthalene (BTEXN), Polycyclic Aromatic Hydrocarbons (PAHs), Organochlorine Pesticides (OCP), Organophosphate Pesticides (OPP), Polychlorinated Biphenyls (PCB), heavy metals, Phenols, Cyanide, Fluoride)
- Leachable Cyanide (CN) by Australian Standard Leaching Procedure (ASLP)
- Leachable Fluoride (F) by Toxicity Characteristic Leaching Procedure (TCLP)

Each sample was left for 24 hours in an enclosed space and the head space sampled for gas generation. Gases were analysed using a landfill gas analyser for methane, and using Kitigawa and Dräger detector tubes for ammonia.

Testing of the initial resultant mixtures showed no change in the waste classification of the material for contaminants in comparison with the untreated material. There were, however, reductions in total concentrations observed, likely to be attributed to the dilution effect of adding the hydrated lime as reductions in total concentrations of arsenic, lead, and nickel were somewhat consistent with the dilution factor of the reagents. Treated and untreated concentrations of leachable cyanide and leachable fluoride are shown in comparison to the CCO in **Figure 3-1** and **Figure 3-2**.

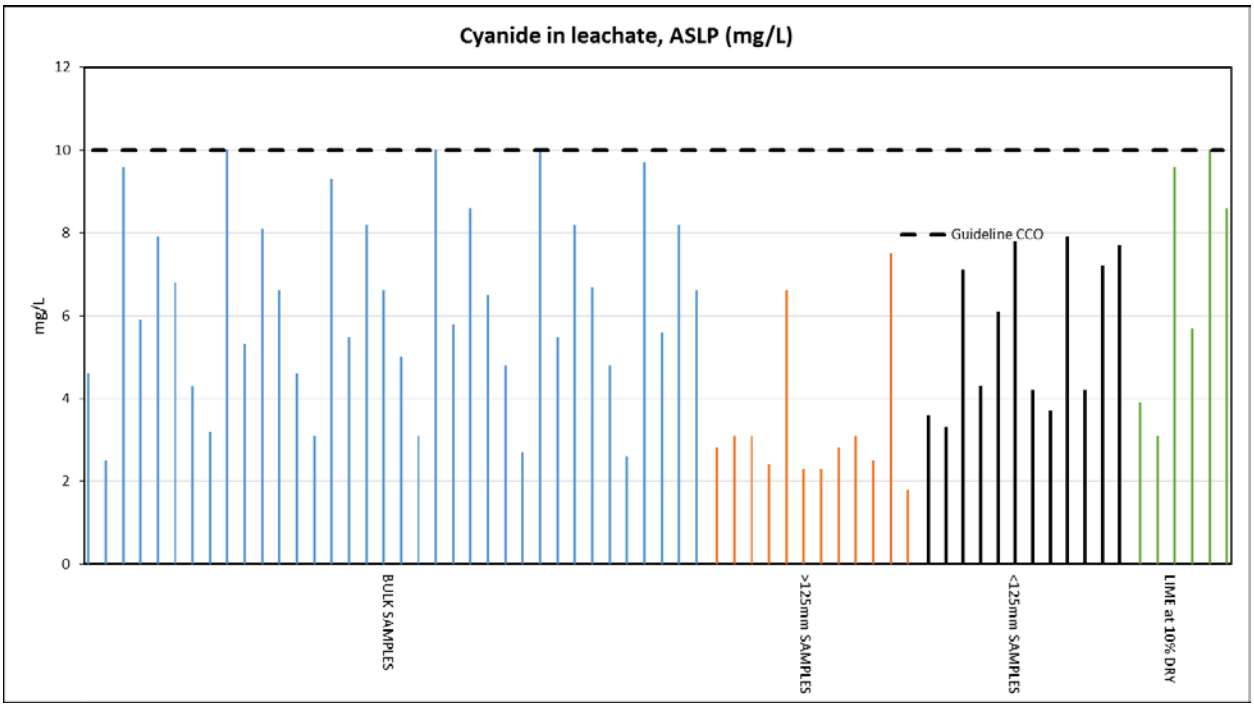

Figure 3-1 Leachable cyanide concentrations in untreated and treated CWS waste samples

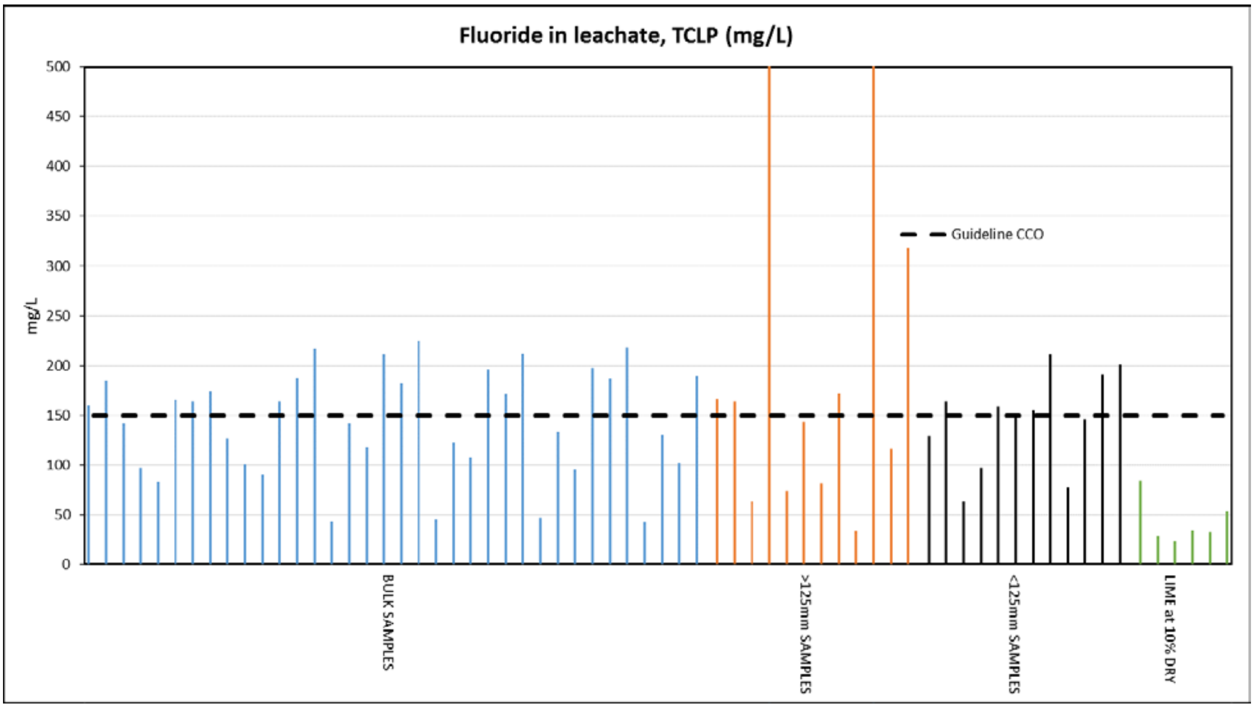

Figure 3-2 Leachable fluoride concentrations in untreated and treated CWS waste samples

The preliminary testing identified that calcium sourced from hydrated lime, at an application of 10% was effective when added dry at reducing the leachable fluoride to a level compliant with the CCO. Concentrations of leachable cyanide remained consistent with the untreated data set.

A preliminary study was subsequently undertaken to assess efficacy of four other calcium source options. These were gypsum ($CaSO_4.2H_2O$), agricultural lime ($CaCO_3$), hydrated lime ($Ca(OH)_2$) and calcium chloride (CaCl). Testing in this instance followed the Australian Standard Leachate Procedure (ASLP) and results are presented in **Table 3-1**.

Table 3-1 ASLP analysis of capped waste stockpile with 10% w/w dry calcium addition

ASLP	Units	PQL	CWS waste with 10% agricultural lime	CWS waste with 10% hydrated lime	CWS waste with 10% calcium chloride	CWS waste with 10% gypsum
Free Cyanide	mg/L	0.004	<0.004	<0.004	0.005	0.007
Fluoride	mg/L	0.1	210	100	28	32

From the results in **Table 3-1** and in **Figure 3-1** and **Figure 3-2** it is evident that gypsum and calcium chloride provide the best performance with respect to reducing fluoride. Leachable concentrations of fluoride are practically the same (28 mg/ L for calcium chloride and 32 mg/ L for gypsum) and both well below the CCO criteria. Neither the calcium chloride nor the gypsum have any chemical affect on cyanide and leachable cyanide concentrations are already below the CCO requirements in the untreated CWS material.

On the basis of the above performance, Treatability Trials are proposed to be completed as described in **Section 6**. Gypsum is selected as the preferred calcium source, when compared to calcium chloride, due to the following:

- It was found to be effective at reducing leachable fluoride concentrations with minimal pH change.

- Gypsum is documented within NSW as having been previously used for this purpose at the Tomago Aluminium Wallaroo Landfill. Successful results have been documented in this application specifically:
    - A review of available monitoring results at the Wallaroo landfill which have not shown adverse issues relating to the use of gypsum. Monitoring results from surrounding groundwater and surface water locations, dating from 2012, are available on the Tomago Aluminium website at: http://www.tomago.com.au/health-safety/monitoring-results/previous-monitoring-results
    - It has been adopted and continues to be regulated by the EPA as a method for the treatment of aluminium smelter waste.
- The concept of “gypsum fixing” is commonly used within the aluminium smelting industry for the treatment of fluoride containing waste.
- There are commercial and practical reasons for the selection of gypsum as opposed to calcium chloride:
    - Gypsum is more readily available than calcium chloride. Given the significant quantity that is required in a relatively short amount of time, this makes gypsum advantageous. In fact, it may be possible to use gypsum derived from recycled building material and other secondary sources.
    - Calcium chloride is significantly more expensive than gypsum. Initial enquiries that Hydro has made to suppliers of both gypsum and calcium chloride indicated that calcium chloride is nine to ten times more expensive. For example, if a 10% application rate was used, this could result in a $80 million to $90 million price difference ($10 million for gypsum compared to $90 to $100 million for calcium chloride).
      Given that both products result in very similar concentrations of cyanide and fluoride (and at concentrations well below the CCO limits) such a significant additional cost provides little to no benefit.

The chemical reaction achieved through the addition of gypsum to the waste is as follows:

$$2NaF + CaSO_4.2H_2O = CaF_2 + Na_2SO_4 + 2H_2O$$

The addition of gypsum has no effect on cyanide, except where a pH change may result in CN dissolution or formation. To test the performance of gypsum within the range concentrations present in the waste material an extensive testing method is proposed.

4. REMEDIATION DESIGN

On the basis of the testing undertaken of the CWS materials and the preliminary treatability tests, the proposed remediation design to achieve compliance with the CCO and the appropriate long term management of the CWS materials comprises:

1) Excavation of the material from the CWS
2) Treatment through addition of gypsum at a level pre-determined to achieve CCO compliance followed by
3) Placement within a purposely design containment cell
4) Long term management of the cell

With the exception of the addition of gypsum, this proposal has been previously presented to the EPA and the Department of Planning in the Demolition and Remediation EIS and as Option 4 in the CWS Management Options Study. Specifically, the Containment Cell design has progressed to detailed design, which has previously been provided to the EPA as Appendix 5 of Volume 1 of the CWS Management Options Study. In principle the design incorporates the following:

- Triple base lining system consisting of low permeability clay overlain by two geocomposite liners each comprising a geosynthetic clay liner and high density polyethylene liner.
- Primary and secondary leachate and groundwater collection system
- Liner durability testing using site won leachate which demonstrated liner performance was unaffected by the leachate
- Dry entombment work methodology to minimise moisture entrainment
- Double capping system comprising a linear low density polyethylene liner and 1.5 m soil and vegetation layer
- Precautionary gas venting system
- Leachate collection including ability for long term periodic pump out

Figure 4-1 and **Figure 4-2** shown liner and capping detailed cross-sections.

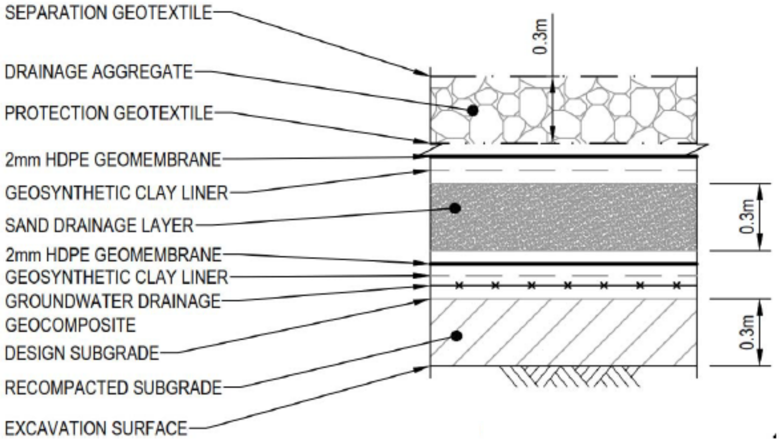

Figure 4-1 Cross section showing liner elements

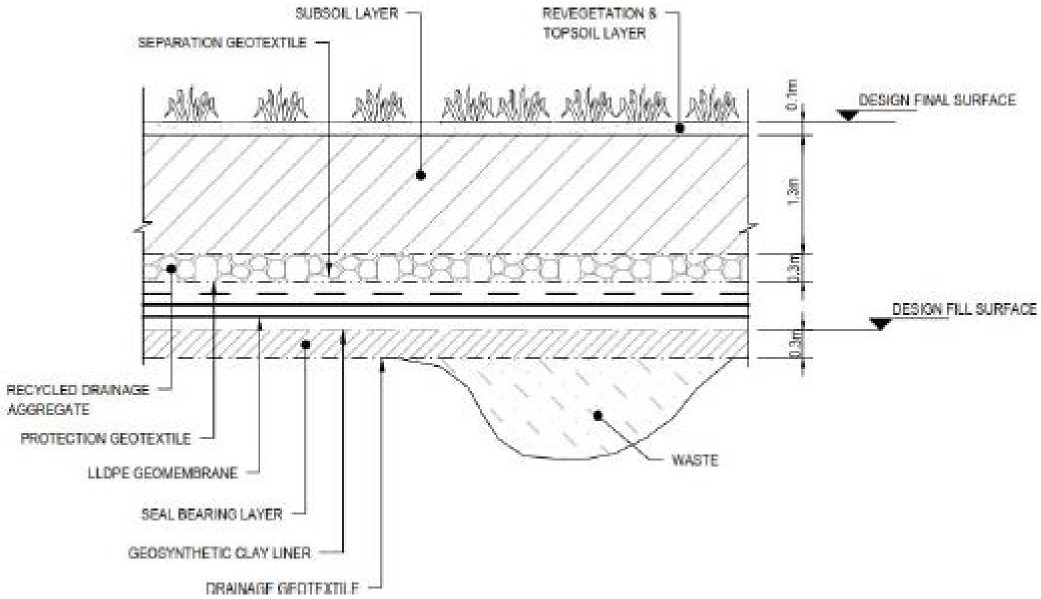

Figure 4-2 Cross section showing capping elements

The process of adding gypsum to the waste materials is:

- Waste will be loaded to trucks and driven over a weighbridge to ascertain total weight
- Gypsum will be added to the loaded waste at the pre-determined w/w percentage using a front end loader with weighing system attached within a specified tolerance
- The truck will be driven to the containment cell and the waste end dumped at the filling face
- The waste will be pushed out by bull dozer and compacted in accordance with the cell filling requirements

Mixing of the waste with gypsum will occur through this process. When considering the waste mass as a whole, the proposed containment will incorporate approximately 17,000 individual 20T truck loads of waste each with the addition of gypsum. Through this method of placement the gypsum addition is considered to be mixed on a macro scale. This level of mixing is sufficient when considering that the waste itself is variable in concentration and highly heterogeneous and that any pathway of leachate through the cell will inevitably pass through gypsum when designed in this manner. Also noting that the containment cell proposal is dry entombment and the generation of leachate is designed to be negligible with an estimated generation rate of 400L/year.[1]

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[1] GHD Hydro Aluminium Kurri Kurri Pty Ltd Containment Cell Design Report October 2017, Appendix C

5. VALIDATION OF TREATMENT

The validation proposed to demonstrate compliance with the CCO, and to enable certification of the waste as ‘approved aluminium smelter waste’ under the EHC Licence, involves:

- Laboratory treatability trials described in **Section 6** to demonstrate efficacy of the gypsum application with respect to meeting the CCO
- Mass based records of the addition of gypsum to the pre-determined w/w percentage
- Documentation supporting the correct construction of the Containment Cell and the placement of the waste within the cell

As part of the remediation and validation requirements, a validation report will be prepared for review and sign off by an independent EPA Accredited Site Auditor.

6. TREATABILTY TRIALS

The description of the treatability trials follows the Data Quality Objective and Data Quality Indicator (DQO/DQI) process outlined in NEPM (2013).

6.1 Step 1: State the Problem

- Aluminium Smelter By-products with concentrations of leachable fluoride above 150 mg/L is proposed to be disposed in an on-site purpose built containment cell. The CCO for aluminium smelting by-products prohibits the disposal of these wastes with concentrations of leachable fluoride and leachable cyanide above 150 mg/L and 10 mg/L respectively when determined by a specific test. The waste therefore requires treatment to achieve concentrations below these levels prior to disposal.
- The material contains asbestos that requires management during treatment. These requirements form part of the EIS and are not discussed here.
- Other wastes on the site may also not meet the CCO and will require treatment. These will be managed on a case by case basis.

6.2 Step 2: Identify the Decisions/Goal of the Study

This treatment plan outlines the methodology to be adopted to validate that the waste can be treated and that the application rate is sufficient to allow chemical fixation to occur should the waste leach.

Treatment will be considered successful when:

- Treatability trials are shown to meet data quality requirements and are considered reliable
- Treatability trials show that following the application of a determined mass percentage of gypsum to the waste the leachable concentrations of fluoride and cyanide are below the CCO concentration limits, as determined by the 95% UCL of the mean for the data set derived from the repeatability testing

6.3 Step 3: Identify Inputs to the Decision

The following inputs to the decision making process are required:

- Laboratory methodology for completing the treatability trial
- Results of treatability trial for leachable fluoride and leachable cyanide

6.4 Step 4: Define the Study Boundary

The study boundary is the material within the CWS.

6.5 Step 5: Development of Decision Rules or Analytical Approach

The decision rules can be defined as:

- If the results of the analytical data quality control assessment are acceptable, then the data will be deemed suitable for the purpose of the project. In this regard, data will be assessed against completeness, comparability, representativeness, precision and accuracy

- If the 95% UCL of the data set for leachable fluoride and leachable cyanide are below 150 mg/L and 10 mg/L respectively then the effective application rate for gypsum to be applied to the waste has been successfully determined

To meet these decision rules, the types of data quality required and the quality of analytical data undertaken by the commercial laboratories are summarised in **Section 6.6.1**. In order to assess the success of achieving the DQOs for the project, Data Quality Indicators (DQI) were considered. DQIs are outlined in **Section 6.6.2**.

6.6 Step 6: Specific Limits of Decision Error

6.6.1 Laboratory QA/QC

The laboratory quality assurances and quality controls are provided below:

- All laboratories will be NATA accredited.
- Laboratory quality assurance testing completed in accordance with NEPM requirements.
- Appropriate LORs adopted for validation criteria.
- Comparative and approved analytical methods between laboratories.

6.6.2 Assessment of DQIs

Field and laboratory data must be assessed against the following DQIs:

- Accuracy;
- Precision;
- Completeness;
- Representativeness; and
- Comparability.

Accuracy

Accuracy is defined as the nearness of a result to the true value, where all random errors have been statistically removed. Internal accuracy is measured using percent recovery '%R' and external accuracy is measured using the Relative Percent Difference '%RPD'.

Internal accuracy will be tested utilising:

Surrogates	Surrogates are QC monitoring spikes, which are added to all field and QA/QC samples at the beginning of the sample extraction process in the laboratory, where applicable. Surrogates are closely related to the organic target analytes being measured, are to be spiked at similar concentrations, and are not normally found in the natural environment
Laboratory control samples	An externally prepared and supplied reference material containing representative analytes under investigation. These will be undertaken at a frequency of one per analytical batch
Matrix spikes	Field samples which are injected with a known concentration of contaminant and then tested to determine the potential for adsorption onto the matrix. These will be undertaken at a frequency of 5%

Recovery data shall be categorised into one of the following control limits:

- 70%-130%R confirming acceptable data, note that there are some larger %R for intractable substances;
- 69%-20%R indicates discussion required. May be considered acceptable data, or may be regarded with uncertainty;
- 10-19 %R indicating that the data should be treated as an estimate result; and
- <10 %R indicating that the data should be rejected.

External accuracy will be determined by the submission of inter-laboratory duplicates at a frequency of 5%. The external duplicate samples are to be obtained by mixing and then splitting the primary sample to create two identical sub samples. Field triplicate samples are to be labelled with a unique identification that does not reveal the association between the primary and triplicate samples e.g., QA1.

It must be noted that significant variation in duplicate results is often observed (particularly for solid matrix samples) due to sample heterogeneity or concentrations reported near the LOR.

Data for primary and duplicate is collated and reported as a relative percent difference (RPD) of the mean concentration of both samples. If results show greater than 30% difference, a review should be conducted of the cause (e.g. instrument calibration, extraction efficiency, appropriateness of the method used, etc.).

Any data which does not conform to these acceptance criteria will be examined for determination of suitability for the purpose of site characterisation.

Precision
The degree to which data generated from replicate or repetitive measurements differ from one another due to random errors. Precision is measured using the standard deviation 'SD' or Relative Percent Difference '%RPD'.

Internal precision will be determined by the undertaking of laboratory duplicates, where two sub samples from a submitted sample are analysed. These will be undertaken at a frequency of 10%. A RPD analysis is calculated and results compared to the laboratory acceptance criteria and any outliers identified by the laboratory will be assessed.

External precision will be determined by the submission of intra-laboratory duplicates at a frequency of 10%. The external duplicate samples are to be obtained by mixing and then splitting the primary sample to create two identical sub samples. Field duplicate samples are to be labelled with a unique identification that does not reveal the association between the primary and duplicate samples e.g., QA1.

It must be noted that significant variation in duplicate results is often observed (particularly for solid matrix samples) due to sample heterogeneity or concentrations reported near the LOR.

Data for primary and duplicate is collated and reported as a relative percent difference (RPD) of the mean concentration of both samples. If results show greater than 30% difference, a review should be conducted of the cause (e.g. instrument calibration, extraction efficiency, appropriateness of the method used, etc.).

Any data which does not conform to these acceptance criteria will be examined for determination of suitability for the purpose of site characterisation.

Blank samples will be submitted with the analytical samples and analysed for the contaminants of concern. One field blank per matrix type each batch samples/each day.

The laboratory will additionally undertake a method blank with each analytical batch of samples. Laboratory method blank analyses are to be below the LORs. Results will be examined and any positive results will be reviewed. Positive blank results may not be subtracted from sample results.

Positive results may be acceptable if sample analyte concentrations are significantly greater than the amount reported in the blank (ten times for laboratory reagents such as methylene chloride, chloroform, and acetone etc., and five times for all other analytes). Alternatively, the laboratory LOR may be raised to accommodate blank anomalies provided that regulatory guidelines are not compromised by any adjustment made to the LOR.

Completeness
The completeness of the data set will be judged as:

- The percentage of data retrieved from the field compared to the proposed scope of works. The acceptance criterion is 95%.
- The percentage of data regarded as acceptable based on the above data quality objectives. 95% of the retrieved data must be reliable.
- The reliability of data based on cumulative sub-standard performance of data quality objectives.

Where two or more data quality objectives indicate less reliability than what the acceptance criteria dictates, the data will be considered with uncertainty.

Representativeness

Sufficient samples must have been collected from the soil present at the site. This will be calculated for soil samples by *Procedure B, NSWEPA Sampling Design Guidelines*, 1995.

Samples must be collected and preserved in accordance with the sampling methodology proposed in Step 7 to ensure that the sample is representative of the assessed stratum.

Comparability

The data must show little to no inconsistencies with results and field observations and include likely associates e.g. TPH C6-C9 and BTEX. This is achieved through maintaining a level of consistency in techniques used to collect samples and ensuring analysing laboratories use consistent analysis techniques and reporting methods as discussed above.

A summary of the DQIs and the corresponding measures to be applied for the validation are presented in **Table 6-1.**

Table 6-1 Summary of DQIs

Field	Laboratory	Acceptability Limits
Completeness		
All critical locations sampled All samples collected (from grid and a depth) Standard Operating Procedures (SOPs) appropriate and complied with Experienced sampler Documentation correct	All critical samples analysed according to SAQP. All analytes analysed according to SAQP. Appropriate methods Appropriate laboratory detection limits Sample documentation complete Sample holding times complied with	As per NEPM 2013
Comparability		
Same SOPs used on each occasion Experienced and same field personnel Climatic conditions appropriate for the type of analyte with respect to the geographical location. Climatic conditions noted during sampling. Same types of samples collected	Same analytical methods used for all samples Same laboratory detection limits Same laboratories (NATA accredited) Same units	As per NEPM 2013
Representativeness		
Appropriate media sampled according to SAQP All media identified in SAQP sampled	All samples analysed according to SAQP	As per NEPM 2013
Precision		
Sampling methodology appropriate and sampling completed in accordance with methodology. Collection of Duplicates (intra-laboratory) and triplicates (inter-laboratory)	Analysis of: Field duplicates analysed 1 in 10 samples Field triplicates analysed 1 in 20 samples Laboratory duplicates analysed	RPD<= 30% RPD<= 30% RPD<= Lab spec

Field	Laboratory	Acceptability Limits
Accuracy		
Sampling methodology appropriate and sampling completed in accordance with methodology. Collection of Field blanks	Analysis of:	
	Field blanks	Non-detect
	Method blanks	Non-detect
	Matrix spikes	70-130%
	Surrogate spikes	70-130%
	Laboratory control spikes	70-130%

6.7 Step 7: Optimise the Design for Obtaining Data

The following outlines the laboratory proposed to meet the project objectives.

6.7.1 Preparation of the waste sample

Preparation of the waste sample follows the guidance provided in the SPCC *Procedures for the Sampling and Analysis of Aluminium Smelter Wastes for the Determination of Leachable Fluoride and/or Leachable Cyanide, 19 December 1986*, and included in **Appendix A**. Variations are made to allow for the treatment application.

Approximately 200 kg of waste remains on-site from core recovery drilling works completed in 2015. The waste was collected by vertically coring through the full depth of the waste emplacement using a 100mm diameter continuous coring machine (sonic method). The cores are considered representative of the materials within the CWS as the cores are collected from the full vertical profile at six spatially discrete locations.

The bulk sample will be subsampled at the laboratory to comprise ten, 10 kg samples each formed from the collection of 25 equal and representative samples from the bulk sample. The 10kg samples will be separated by coning or riffling to create ten, 1 kg samples. The 1 kg samples will be subject to the treatment outlined below.

6.7.2 Stage 1. Determination of preferred gypsum.

Two types of gypsum are available to supply and are comprised of a recycled and a mined gypsum product, with levels of purity as described below:

- Super Ag Gypsum – purity 92% - fine recycled
- Mined Gypsum – purity 94%

Bench scale testing will initially be completed through the addition of 10% gypsum to 100% weight of waste, that is 100 grams of gypsum to 1 kg of waste.

Each sample will be tumbled to achieving mixing. A sample will be collected following Appendix A whereby treated waste is subsampled by coning and quartering, or riffling, to create a 200 g sample that is subject to leachate analysis. On two occasions a second sample will also be collected for analysis.

In accordance with Appendix A, all material for the leaching test will be capable of passing through a mesh of not greater than 9.5mm, and crushing or grinding will be used as required, using standard laboratory practices, when preparing the 200 g sub sample.

The test for producing a leachate will be *the US Environment Protection Agency method 13XX (known as TCLP)* as described in Appendix A, *and modified with the additional conditions that*

1. *Distilled or deionised water shall be used as the extraction fluid, and*
2. *The filter material used shall be cellulose.*

The method was previously approved by the EPA for demonstrating approved aluminium smelter waste was suitable for disposal in the Tomago Aluminium Wallaroo Landfill.

Analysis of the elutriate will be completed for fluoride and free cyanide as described in **Section 6.7.5**.

Six tests for each gypsum type will be undertaken making a total of 12 tests. In two mixes, a replicate will also be collected and analysed, making a total of 14 analysis sets.

6.7.3 Stage 2. Determination of the rate of application

Based on the findings of Stage 1, further testing using the selected gypsum will be completed to determine the application range. The application ranges will be estimated from Stage 1 testing, and will incorporate two increments either side of this estimate.

Six tests will be completed at each application rate of 24 tests. In two mixes, a replicate will also be collected and analysed, making a total of 26 analysis sets.

6.7.4 Stage 3. Verification of application

Once the application rate is determined with a high degree of confidence, replicate testing will be carried out. Replicate testing will comprise the addition of gypsum at the application rate to thirty samples. Three replicate samples will also be completed making a total of 33 analysis.

A summary of the testing proposed is outlined in **Table 6-2.**

Table 6-2 Summary of proposed testing to verify treatment method

Stage	Purpose	Detail	Total
1	Testing gypsum source	Addition of 10% w/w Super Ag Gypsum	6
		Mined Gypsum	6
		Replicate samples	2
2	Testing application rates	Rate -2	6
		Rate -1	6
		Rate + 1	6
		Rate + 2	6
		Replicate	2
3	Test of repeatability	Addition of selected gypsum at selected application rate	30
		Replicates	3
		Total	66
		Replicates	7

6.7.5 Laboratory Method

Appendix A describes recommended test methods for leachable cyanide and leachable fluoride. Variation from the prescribed test method for leachable cyanide and leachable fluoride analysis is proposed as follows:

- Leachable cyanide will be determined by on-line UV catalytic digestion/diffusion. The advantages with this method are that the specific UV digestion restricts interference from Thiocyanate, which is prevalent with the distillation method; and is also less likely to form Cyanide from Cyanide pre-cursors. Hence, this method is highly preferable.
- For leachable fluoride, an Ion Selective Electrode (ISE) based on APHA 4500-F- C 23rd Edition is proposed. The ISE method uses a complexing agent to minimise the effect of soluble Aluminium in the sample, however, where Aluminium is very high the method is modified by adding extra complexing agent (called CDTA) to the sample prior to analysis and/or diluting the sample. Samples are spiked to check for recovery of Fluoride given the Aluminium effect to make sure there are no false negatives. The SPCC recommended method of distillation is prone to loss and is not preferred.

6.7.6 Reporting

At the completion of testing a report will be prepared for review by the EPA. The report will include:

- Details of all testing completed
- Presentation of all laboratory data
- Evaluation of quality assurance and quality control
- Calculation of 95% upper confidence limits and comparison to Criteria
- Summary of results and conclusions on the preferred treatment including a review of the addition of gypsum on the proposed containment cell design

7. PROGRESS REPORT

Hydro has commenced laboratory trials following the laboratory methodology outlined above. Trials have involved:

- Stage 1 Analysis of preferred gypsum supply; and
- Stage 2 Determination of gypsum application rate.

Preliminary results of this analysis are included here to provide a progress update.

7.1 Stage 1 Preliminary Results, Gypsum source selection

Gypsum samples were sourced from a NSW provider and comprised:

- Rehab Gypsum – purity 75+% - coarse recycled
- Super Ag Gypsum (SAG) – purity 92% - fine recycled
- Mined Gypsum – purity 94%

Of the above, Rehab gypsum was observed to have a high paper content and was rejected from further investigation.

Stage 1 involved the application of 10% by weight of two gypsum sources to the waste, with waste samples prepared as outlined in **Section 6.7.1**. **Figure 7-1** and **Figure 7-2** provide a graphical representation of this data.

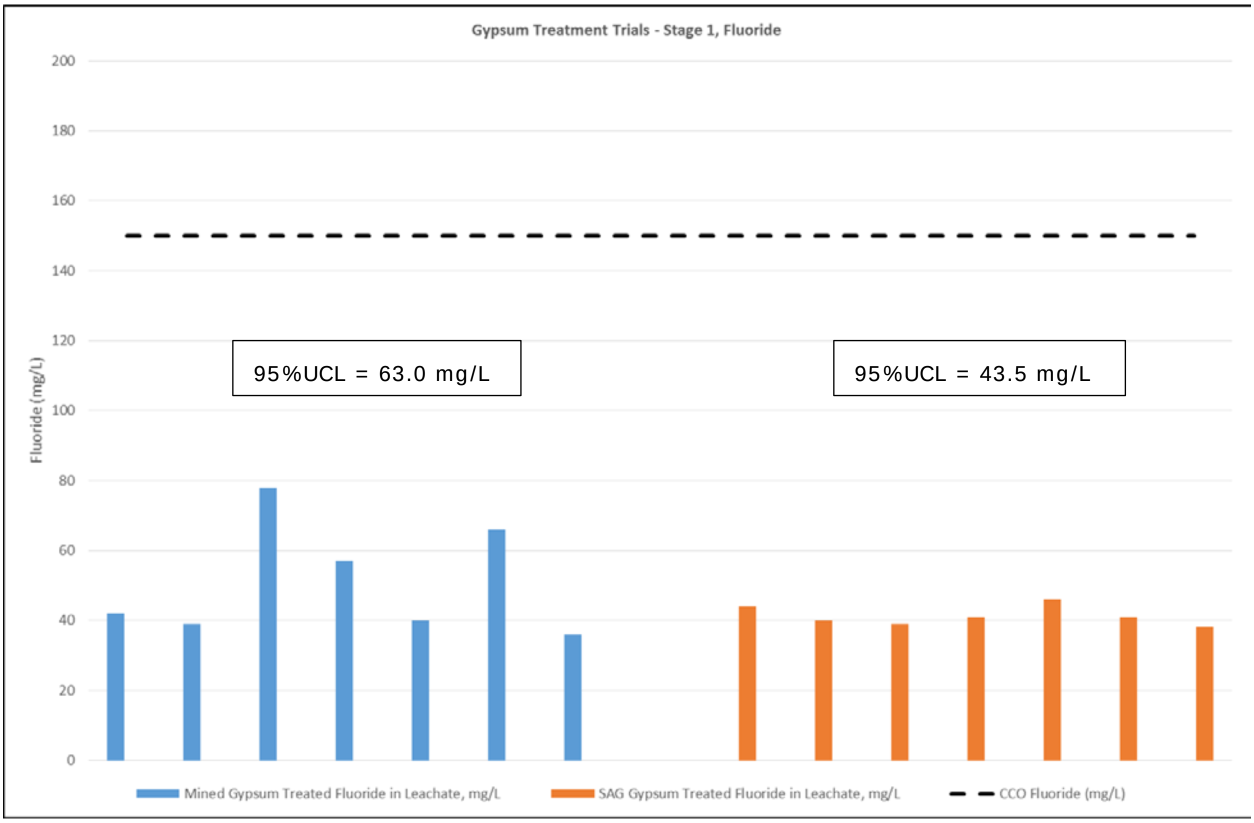

Figure 7-1 Comparison of Leachable F from waste treated with alternate Gypsum sources

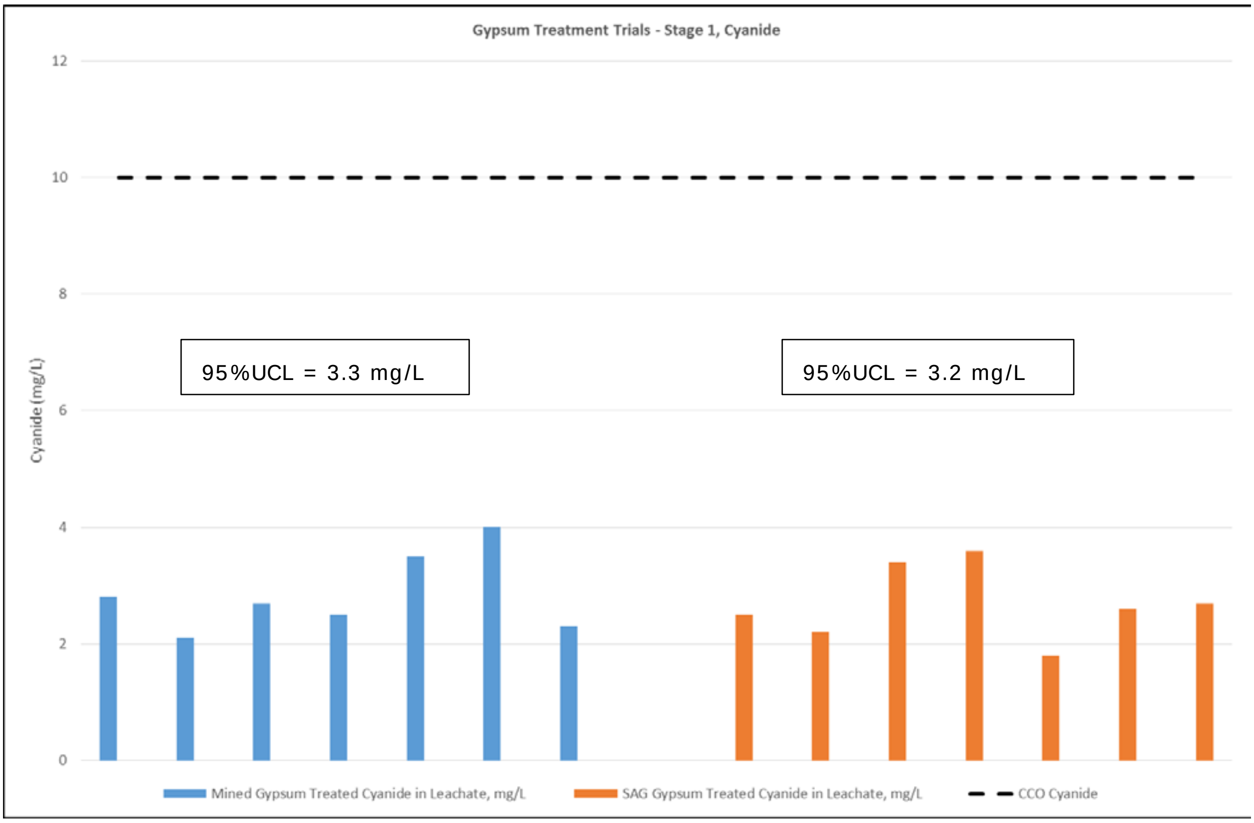

Figure 7-2 Comparison of Leachable CN from waste treated with alternate Gypsum sources

The results show that both gypsum applications meet the CCO criteria for both leachable F and leachable CN. SAG was selected moving forward on the basis of:

- Preference to use a recycled product
- Cost
- Availability of supply

7.2 Stage 2 Preliminary Results, Gypsum Application Rate

Following the methodology outlined in **Section 6.7.3**, trials were completed using various application rates. Rates of 5%, 7% and 15% were selected. The 95% UCLs determined from these trials are presented in **Table 7-1.**

Table 7-1 Comparison of Application Rates

Application w/w	pH in leachate, pH units	Leachable Results mg/L	
SAG Gypsum added	pH 95%UCL	CN 95% UCL	F 95% UCL
5%	8.2	3.4	116.1
7%	8.4	3.3	114.9
10%	7.4	3.2	43.4
15%	7.5	2.9	39.7

w/w refers to the added weight. For example, where the initial weight is 1kg, 10% of this weight is added so the final weight is 1.1kg.

The results show that in all cases, the leachable concentrations are below the CCO. To illustrate the variability in the data sets, box and whisker plots were developed for pH and leachable fluoride.

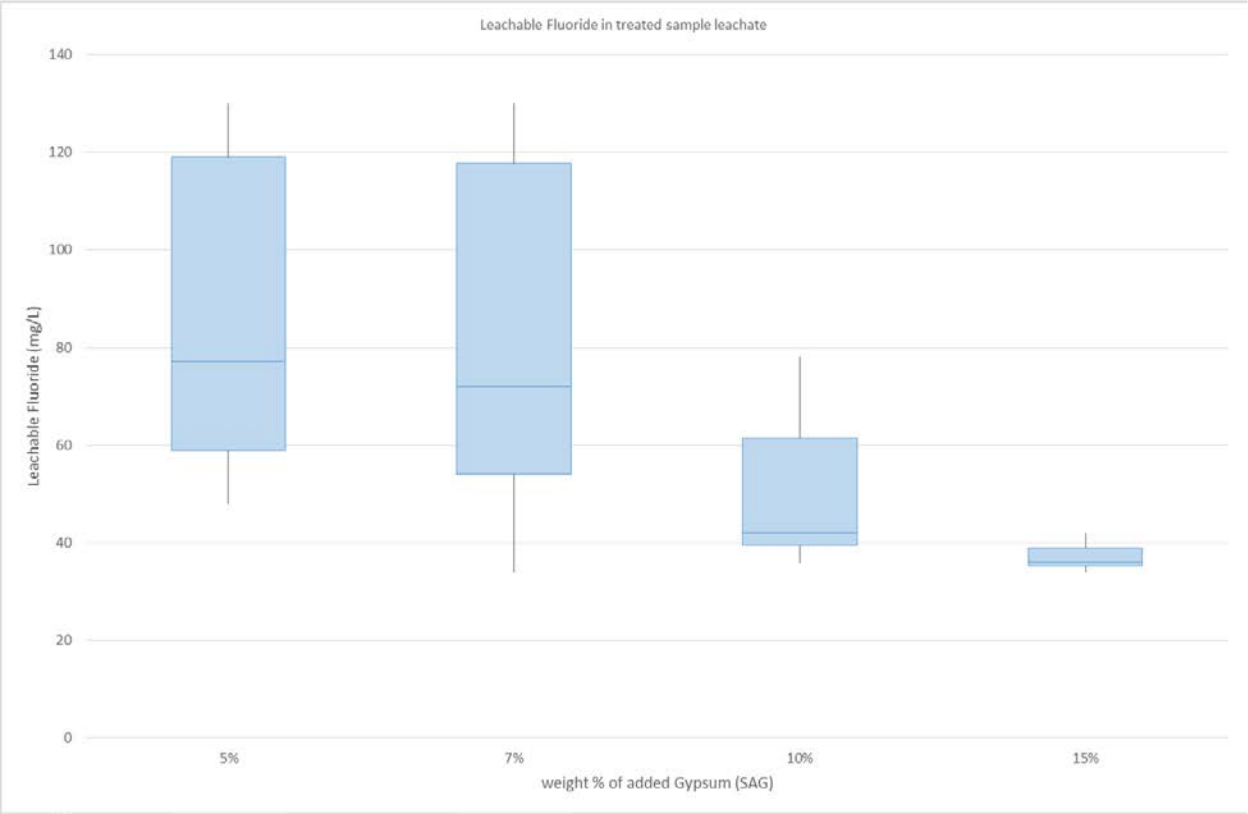

Figure 7-3 Comparison of Leachable CN from waste treated with alternate Gypsum sources

Figure 7-3 shows that the data set variability improves with increased gypsum application. This observation may also be an artefact of the small sample size and the variability in the waste materials. Stage 3 testing will allow for further evaluation of the waste variability and the response to the gypsum application.

Table 7-1 also shows that pH decreases with increasing gypsum addition from 7% to 10% but that these pH values are in the neutral range. In response to the EPA requests regarding reaction times and the potential for the application of gypsum to result in a lowering of the pH over time, longer duration leachate testing has been completed to assess pH only. This method is in addition to that described in **Section 6.7** and involved tumbling the sample for 48 hours, rather than the standard 18 hours ± 3. The results tabulated in **Table 7-2** show that pH does not decrease further with the additional test duration.

Table 7-2 Comparison of Application Rates

Application w/w SAG 7%	pH in leachate, pH units
Test duration	pH 95%UCL
18 hours	8.4
48 hours	8.5

8. CONCLUSIONS

The methodology outlined provides a practical and effective procedure for achieving compliance with the CCO, and certification of the treated CWS material as ‘approved aluminium smelter waste’ under the EHC Licence. The proposed method for application of the gypsum is consistent with minimising the health, safety and environmental risks as described in the EIS. The proposed approach continues to minimise the entrainment of moisture within the Containment Cell wastes in order to reduce long term management requirements.

Based on the results of preliminary testing Hydro concludes that a 10% gypsum application rate is the preferred option. Hydro will proceed with replication tests of the 10% gypsum application rate.

Hydro requests that the EPA provide a written response confirming that the sampling, analysis and testing procedures set out in this Plan are appropriate for the purposes of:

- certifying that the aluminium smelter waste is ‘approved aluminium smelter waste’ for the purposes of the EHC Licence; and
- testing whether the aluminium smelter waste contains ‘leachable fluoride’ or ‘leachable cyanide’ for the purposes of the CCO.

Following determination of the application rate, Hydro will prepare the detailed methodology for the application of gypsum to the waste during the remediation works in accordance with the methodology outlined in **Section 4**. This methodology will be reviewed by the NSW EPA and the NSW EPA accredited Site Auditor, Mr Ross McFarland.

9. REFERENCES

NSW EPA 2014, Waste Classification Guidelines – Part 1: Classifying Waste, New South Wales Environmental Protection Agency, November 2014.

Organisation for Economic Cooperation and Development (OECD), 2015, Recommendations on the Transport of Dangerous Goods – Manual of Tests and Criteria Sixth Revised Edition, United Nations Publications, Sales No. E.09 VIII.3.

Ramboll Environ, 2016. Capped Waste Stockpile Assessment, Document Reference AS130456, April 2016

Ramboll Environ, 2017. Capped Waste Stockpile Waste Management Options Evaluation Study, Document Reference AS130525, October 2017

United States Environmental Protection Agency (USEPA), 1992, Method 1311: Toxicity Characteristic Leaching Procedure, part of Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Document SW-846, July 1992

EnviroLab in-house method INORG-004

Procedures for the Sampling and Analysis of Aluminium Smelter Wastes for the Determination of Leachable Fluoride and/or Leachable Cyanide, 19 December 1986

APPENDIX A

Procedures for the Sampling and Analysis of Aluminium Smelter Wastes for the Determination of Leachable Fluoride and/or Leachable Cyanide 19 December 1986

Sampling

Samples for analysis are to be formed by taking equal and representative samples from bulk lots of aluminium smelter wastes to make a composite sample. At least 25 representative composite samples shall be made for the evaluation of leachable fluoride and/or leachable cyanide content of any accumulation of wastes from a nominated production source or sources.

Preparation

1. Samples, to be tested by the leaching test, shall be prepared from each composite sample by coning and quartering, or riffling. A final riffle pair of sub samples shall be produced, one sample for company use and the other for the Commission, each being at least 200g in size.
2. All material for the leaching test shall be capable of passing through a mesh of not greater than 9.5mm, and crushing or grinding shall be used as required, using standard laboratory practices, when preparing the sub samples in 1 above.

Leaching

The representative sub samples shall be leached using the US Environment Protection Agency method 13XX (known as TCLP) with the additional conditions that

1. Distilled or deionised water shall be used as the extraction fluid, and
2. The filter material used shall be cellulose.

Precautions shall be taken to ensure that gaseous fluoride and cyanide compounds are not lost during the leaching test.

Analysis

1. The cyanide content of the leachate shall be determined using the method –
   - 1.1. known as "The Woods River Modification (Shell Oil Company) of the Roberts and Jackson method for cyanide" described in Analyst, 1971, 96, 209-212. or
   - 1.2. for cyanide available for chlorination, described in "Standard Methods for the Examination of Water and Wastewater", 15th edition, 1980, Section 412.

   Precautions shall be taken to ensure that oxidising agents and sulphides do not interfere with the analysis.
2. The fluoride content of the leachate shall be determined by a sample distillation followed by potentiometric or colorimetric analysis as described in "Standard Methods for the Examination of Water and Wastewater", 15th edition, 1930, Section 413.

   Precautions shall be taken to ensure that interfering substances, such as aluminium and iron, do not interfere with the analysis.
3. The pH of the leachate shall be determined.