Cooling Tower Wastewater Pilot Plant Trial Report

BOC Limited Kooragang Island

27 October 2016



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Document Control			Approved for Issue			
Project No.	Rev	Author	Reviewer	Name	Signature	Date
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1 Introduction

1.1 Overview

BOC Limited Kooragang Island, herein referred to as BOC Kooragang, owns and operates a gas facility for the production and supply of gas products located at 9 Egret Street Kooragang, New South Wales. The facility operates 24 hours per day, 7 days per week. BOC Kooragang holds NSW Environmental Protection Authority (EPA) Environmental Protection Licence (EPL) 20165. The Scheduled Activities in the EPL include chemical production and chemical storage.

BOC Kooragang currently possess two (2) cooling towers onsite. Currently the cooling tower blowdown (waste) water continues to two (2) 10,000 litre capacity storage tanks onsite, totalling a capacity of 20,000 litres storage onsite. The wastewater is collected by an approved waste contractor approximately once per week.

BOC Kooragang are researching the possibility of utilising the cooling tower wastewater for irrigation purposes in specific grassed areas of the site. Water quality analysis performed from 2014 to 2016 showed that the wastewater contains fluoride concentrations which are above the applicable guideline limits.

1.2 Objective

MJM Environmental (MJM) was engaged by BOC Kooragang to undertake a treatment options assessment to research the possibility of utilising the cooling tower wastewater onsite.

In order to verify key process design parameters for the treatment options assessment, a bench-scale jar testing investigation was completed to trial current defluoridation techniques including coagulation and pH precipitation. The results of the investigation showed these methods were ineffective in removing the high levels of fluoride.

Further research into current defluoridation techniques was undertaken.

A widely accepted defluoridation method is adsorption, which involves passing the water through a filter column packed with an adsorbent media.

In October 2016 MJM fabricated a pilot-scale plant to trial the use of the widely-used adsorbent Activated Alumina (AA) at the Kooragang site. The following report summarises the results from the pilot plant trial.

2 Pilot Plant Methodology

2.1 Treated Water Quality Targets

The treated water quality targets for the trial were adopted from the *Australian and New Zealand Environment and Conservation Council* (ANZECC) 2000 guidelines. The analysis results were compared to the ANZECC guidelines presented in *Section 4: Primary Industries - 4.2 Water Quality for irrigation and general water use*. The guidelines are presented in Table 2-1 below.

The aim of the investigation was to achieve the treatment targets for fluoride.

Table 2-1: Treated water quality targets

Parameter	Unit	Recommended Irrigation Threshold ¹	
Fluoride	mg/L	1.0 ²	2.0 ³

¹ Australian and New Zealand Environment and Conservation Council (ANZECC) 2000 guidelines - Section 4: Primary Industries - 4.2 Water Quality for irrigation and general water use.

² Short-term trigger value (STV) – The STV is the maximum concentration (mg/L) of contaminant in the irrigation water which can be tolerated for a shorter period of time (20 years).

³ Long-term trigger value (LTV) – The LTV is the maximum concentration (mg/L) of contaminant in the irrigation water which can be tolerated assuming 100 years of irrigation.

2.2 Activated Alumina Media

AA is a granular porous material consisting mainly of aluminium oxide. AA has a very high surface area. The application of AA in the defluoridation of water has been accepted worldwide. The specifications of the media used in the pilot trial are presented in Table 2-2.

Parameter	Unit	Specification
Media Supplier	-	Pacific Water Technology Pty Ltd
Chemical Composition:	%	
Al ₂ O ₃		> 93
SiO ₂		0.06
Fe ₂ O ₃		0.03
Na ₂ O		0.5
BET-surface	m²/g	300 ± 20
Pore-volume	ml/g	0.4 ± 0.02
LOI at 400°C	%	<6
Specific Wearability	%	0.05
Bulk Density	kg/L	0.7 ± 0.02
Crush Strength	N	120
Fluoride Uptake	mg/kg	2,500

Table 2-2: Activated alumina specifications

2.3 Pilot Plant Design

The pilot plant consisted of an up-flow filter which was designed for a raw water fluoride concentration of 6 to 7 mg/L. The filter column was constructed from 50mm PVC pressure pipe with a filter height of approximately 1,200 mm. A 50 mm PVC barrel union and perforated flange were installed at the base of the filter column which supported the AA media filter bed. The outlet of the filter column consisted of a 25mm clear vinyl tubing.

A submersible pump was used to pump the wastewater through the filter column. The inlet consisted of a 25mm clear vinyl tubing and 20mm PVC ball valve. A recirculation loop consisting of a 20mm PVC ball valve and 16mm clear vinyl tubing was installed to adjust the flowrate.

The design criteria for the pilot plant are presented below in Table 2-3.

Parameter	Unit	Value
Filter Bed Height	mm	1,050
Filtration Rate	mL/min	300

Table 2-3: Pilot plant design criteria

2.4 Pilot Trial Program

The pilot trial program used for the investigation is shown below in Table 2-4.

Various sources state that more effective fluoride removal occurs at lower pH, with the ideal pH for treatment being pH 5.5. Therefore the trial was conducted with and without pH correction to determine whether it is required to achieve the fluoride target limit.

Pilot Test No.	Objective			
Raw Water Quality Analysis				
Raw	To determine and analyse the raw water quality.			
Activated Alumina Adsorption with pH Correction				
PT1	Determine the effectiveness of pH correction and activated alumina adsorption for fluoride removal			
Activated Alumina Adsorption without pH Correction				
PT2	Determine the effectiveness of activated alumina adsorption for fluoride removal			

Table 2-4: Pilot trial summary

2.5 Waste Classification

At the completion of the pilot trial samples of the AA media were submitted to and analysed by a NATA accredited laboratory to determine the waste class based on the NSW EPA's *Waste Classification Guidelines* (2014).

The guidelines state that classification of waste can be performed using chemical assessment. The chemical assessment process is based around the waste's potential to release chemical contaminants into the environment through contact with liquids, which leads to the production of leachates.

The two measurable properties of chemical contaminants used to classify waste are:

- <u>Classifying a waste using the Specific Contaminant Concentration (SCC)</u>: This is an initial screening of a waste for contaminants in mg/kg. The SCC limit for each contaminant alone is used. In this report the SCC is obtained through inductively coupled plasma mass spectrometry (ICP) analysis only. Where the contaminant exceeds the threshold value for the contaminant for General Solid Waste, further assessment using the toxicity characteristics leaching procedure (TCLP) may be used.
- 2. <u>Classifying a waste using SCC and Toxicity Characteristics Leaching Procedure (TCLP)</u>: This uses the SCC in conjunction with the leachable concentration of a contaminant in mg/L through TCLP analysis.

For the purposes of the investigation at BOC Kooragang both SCC and TCLP were utilised for waste classification purposes.

3 Results

3.1 Raw Water Quality

Analysis of the raw water quality data used for each of the pilot tests is presented below in Table 3-1.

Parameter	Unit	Unit Results – Raw PT1	
рН	pH Unit	8.1	8.2
Temperature	°C	20.1	18.0
Turbidity	NTU	0.99	0.54
Colour (True)	Hazen	32	8
Fluoride (Dissolved)	mg/L	7.0	6.8

Table 3-1: Raw water quality

3.2 Activated Alumina Adsorption with pH Correction (PT1)

The objective of the trial was to determine whether pH correction and AA adsorption would provide significant fluoride removal.

Clean 20 litre drums were filled with the cooling tower wastewater and hydrochloric acid was gradually added to achieve reduced a pH of approximately 5 to 5.5. The drums were then poured into a large bin which contained the pilot plant submersible pump. The pump was then started and the flowrate calibrated using a measuring cylinder and stop watch.

The trial was undertaken on 11 and 12 October 2016. Samples were taken every 0.5 hours. All samples were filtered through 0.45 μ m filter paper prior to analysis. Fluoride analysis was undertaken onsite using a portable water quality testing instrument (colorimeter). The results are tabulated below in Table 3-2.

Table 3-2.1111(Sulls						
Sample Interval Hr	Fluoride (Dissolved) – Onsite Analysis	Recommended Irrigation Thresholds ¹				
Raw	7.0	1.0 ²				
0.5	0.40	2.0 ³				
1	0.34					
1.5	0.39					
2	0.50					
2.5	0.42					
3	0.53					
3.5	0.51					
4	0.54					

Table 3-2: PT1 results

¹ Australian and New Zealand Environment and Conservation Council (ANZECC) 2000 guidelines - Section 4: Primary Industries - 4.2 Water Quality for irrigation and general water use.

² Short-term trigger value (STV) – The STV is the maximum concentration (mg/L) of contaminant in the irrigation water which can be tolerated for a shorter period of time (20 years).

³ Long-term trigger value (LTV) – The LTV is the maximum concentration (mg/L) of contaminant in the irrigation water which can be tolerated assuming 100 years of irrigation.

As can be seen in Table 3-2 all results from the colorimeter were reported to be below the recommended irrigation threshold limits. The maximum fluoride removal occurred after one hour with 95% removal achieved.

3.3 Activated Alumina Adsorption without pH Correction (PT2)

The objective of the trial was to determine whether AA adsorption without pH correction would provide significant fluoride removal.

Clean 20 litre drums were filled with the cooling tower wastewater and poured into a large bin which contained the pilot plant submersible pump. The pump was then started and the flowrate calibrated using a measuring cylinder and stop watch.

The trial was undertaken on 18 October 2016. Samples were taken every 0.5 hours. All samples were filtered through 0.45 μ m filter paper prior to analysis. Fluoride analysis was undertaken onsite a portable water quality testing instrument (colorimeter). Additional fluoride samples were submitted and analysed at a NATA accredited laboratory for confirmation of the results. The results are tabulated below in Table 3-3.

Table 3-3: PT1 results

Sample Interval Hr	Fluoride (Dissolved) – Onsite Analysis mg/L	Fluoride (Dissolved) – NATA Laboratory mg/L	Recommended Irrigation Thresholds ¹
Raw	6.8	6.5	1.0 ²
0.5	0.49	0.2	2.0 ³
1	0.57	<0.1	
1.5	0.60	0.2	
2	0.58	0.1	
2.5	0.59	0.3	
3	0.56	0.2	

¹ Australian and New Zealand Environment and Conservation Council (ANZECC) 2000 guidelines - Section 4: Primary Industries - 4.2 Water Quality for irrigation and general water use.

² Short-term trigger value (STV) – The STV is the maximum concentration (mg/L) of contaminant in the irrigation water which can be tolerated for a shorter period of time (20 years).

³ Long-term trigger value (LTV) – The LTV is the maximum concentration (mg/L) of contaminant in the irrigation water which can be tolerated assuming 100 years of irrigation.

As can be seen in Table 3-3 all treated wastewater results were below the recommended irrigation threshold limits. The result of the sample taken after one hour and analysed by the NATA laboratory was below the Limit of Reporting. The NATA laboratory report is presented in Appendix A.

Based on the laboratory results the maximum fluoride removal occurred after one hour with 99% removal achieved.

3.4 Waste Classification

At the completion of the trial samples of the media were analysed for fluoride to determine the waste classification and disposal options according to the NSW EPA's *Waste Classification Guidelines* (2014).

Three samples of the AA media were collected and submitted to Australian Laboratory Services (ALS), a NATA accredited laboratory with accreditation number 825 located at Mayfield West, Newcastle, 2304. Raw laboratory results are presented in Appendix B. The results are presented in Table 3-4 below.

ltem	Unit	Sample 1	Sample 2	Sample 3	General Solid Waste Limit	
SCC ¹	mg/kg	40	<40	<40	10,000	
TCLP ²	mg/L	0.2	0.2	0.1	150	
1 SCC – Specific Contaminant Concentration						

Table 3-4: Activated alumina waste classification results

¹ SCC – Specific Contaminant Concentration

² TCLP – Toxicity Characteristics Leaching Procedure

As can be seen above fluoride concentrations for all samples were below the prescribed limits for general solid waste.

Based on the NSW EPA's *Waste Classification Guidelines* (2014) the AA media used in the trial can also be classified as a general solid waste (non-putrescible) for the following reasons:

- Does not readily decay under standard conditions
- Does not emit offensive odours
- Does not attract vermin or other vectors (such as flies, birds and rodents).

Please note that upon commissioning of the treatment plant and subsequent production of the filter waste, the media must be sampled and analysed before disposal to landfill.

4 Discussion

In October 2016 MJM carried out a pilot-scale plant trial at the BOC Kooragang site. The objective of the trial was to determine the effectiveness of activated alumina adsorption for removing fluoride from the cooling tower blowdown wastewater. Two trials were conducted which were:

- AA adsorption with pH correction; and
- AA adsorption without pH correction.

As a result of the trials it was found that AA adsorption was effective in removing the fluoride to below the recommended irrigation threshold limits.

It was found that pH correction was not required with a maximum fluoride removal of 99% achieved with AA adsorption without pH correction.

At the completion of the trial, samples of the media were collected and analysed to determine the waste classification and disposal options according to EPA's *Waste Classification Guidelines* (2014). Based on the guidelines the AA media used in the trial can be classified as a general solid waste (non-putrescible). If BOC proceed with the AA adsorption treatment option it is recommended that further waste classification be undertaken on the filter media due to the likelihood of elevated fluoride levels.

It is recommended that BOC proceed with the treatment options assessment and short-list AA adsorption as the preferred treatment option.

Appendix A – NATA Laboratory Wastewater Results



CERTIFICATE OF ANALYSIS

Work Order	ES1623422	Page	: 1 of 4
Client	: MJM ENVIRONMENTAL PTY LTD	Laboratory	: Environmental Division Sydney
Contact	: MR ADAM BUCIOR	Contact	
Address	: OFFICE 1, 335 WHARF ROAD	Address	: 277-289 Woodpark Road Smithfield NSW Australia 2164
	NEWCASTLE NSW, AUSTRALIA 2300		
Telephone	: +61 02 49264222	Telephone	: +61-2-8784 8555
Project	: 034 1612	Date Samples Received	: 18-Oct-2016 15:57
Order number	:	Date Analysis Commenced	: 19-Oct-2016
C-O-C number	:	Issue Date	: 20-Oct-2016 16:50
Sampler	: AB		Hac-MRA NAIA
Site	:		
Quote number	:		Accreditation No. 825
No. of samples received	: 8		Accredited for compliance with
No. of samples analysed	: 8		ISO/IEC 17025 - Testing

This report supersedes any previous report(s) with this reference. Results apply to the sample(s) as submitted. This document shall not be reproduced, except in full.

This Certificate of Analysis contains the following information:

- General Comments
- Analytical Results

Additional information pertinent to this report will be found in the following separate attachments: Quality Control Report, QA/QC Compliance Assessment to assist with Quality Review and Sample Receipt Notification.

Signatories

This document has been electronically signed by the authorized signatories below. Electronic signing is carried out in compliance with procedures specified in 21 CFR Part 11.

Signatories	Position	Accreditation Category
Ankit Joshi	Inorganic Chemist	Sydney Inorganics, Smithfield, NSW
Wisam Marassa	Inorganics Coordinator	Sydney Inorganics, Smithfield, NSW



General Comments

The analytical procedures used by the Environmental Division have been developed from established internationally recognized procedures such as those published by the USEPA, APHA, AS and NEPM. In house developed procedures are employed in the absence of documented standards or by client request.

Where moisture determination has been performed, results are reported on a dry weight basis.

Where a reported less than (<) result is higher than the LOR, this may be due to primary sample extract/digestate dilution and/or insufficient sample for analysis.

Where the LOR of a reported result differs from standard LOR, this may be due to high moisture content, insufficient sample (reduced weight employed) or matrix interference.

When sampling time information is not provided by the client, sampling dates are shown without a time component. In these instances, the time component has been assumed by the laboratory for processing purposes.

Where a result is required to meet compliance limits the associated uncertainty must be considered. Refer to the ALS Contact for details.

Key : CAS Number = CAS registry number from database maintained by Chemical Abstracts Services. The Chemical Abstracts Service is a division of the American Chemical Society.

LOR = Limit of reporting

^ = This result is computed from individual analyte detections at or above the level of reporting

ø = ALS is not NATA accredited for these tests.

~ = Indicates an estimated value.

Page : 3 of 4 Work Order : ES1623422 Client : MJM ENVIRONMENTAL PTY LTD Project : 034 1612



Sub-Matrix: WATER (Matrix: WATER)		Client sample ID			RAW	30 MIN	1 HR	1.5 HR
	Cl	ient samplii	ng date / time	[18-Oct-2016]	[18-Oct-2016]	[18-Oct-2016]	[18-Oct-2016]	[18-Oct-2016]
Compound	CAS Number	LOR	Unit	ES1623422-001	ES1623422-002	ES1623422-003	ES1623422-004	ES1623422-005
				Result	Result	Result	Result	Result
EA010P: Conductivity by PC Titrator								
Electrical Conductivity @ 25°C		1	µS/cm	1520	1490			
ED045G: Chloride by Discrete Analys	ser							
Chloride	16887-00-6	1	mg/L	300	297			
ED093F: Dissolved Major Cations								
Sodium	7440-23-5	1	mg/L	229	179			
ED093F: SAR and Hardness Calculat	ions							
Sodium Adsorption Ratio		0.01	-	7.00	3.97			
EK040P: Fluoride by PC Titrator								
Fluoride	16984-48-8	0.1	mg/L	0.2	6.5	0.2	<0.1	0.2
EK059G: Nitrite plus Nitrate as N (NC	Dx) by Discrete Ana	lyser						
Nitrite + Nitrate as N		0.01	mg/L	2.08	1.69			
EK061G: Total Kjeldahl Nitrogen By I	Discrete Analyser							
Total Kjeldahl Nitrogen as N		0.1	mg/L	1.0	1.6			
EK062G: Total Nitrogen as N (TKN +	NOx) by Discrete Ar	alyser						
^ Total Nitrogen as N		0.1	mg/L	3.1	3.3			
EK067G: Total Phosphorus as P by E)iscrete Analvser							
Total Phosphorus as P		0.01	mg/L	0.02	1.89			



Sub-Matrix: WATER (Matrix: WATER)	Client sample ID			2 HR	2.5 HR	3 HR	
	Cl	ient sampli	ng date / time	[18-Oct-2016]	[18-Oct-2016]	[18-Oct-2016]	
Compound	CAS Number	LOR	Unit	ES1623422-006	ES1623422-007	ES1623422-008	
				Result	Result	Result	
EA010P: Conductivity by PC Titrator							
Electrical Conductivity @ 25°C		1	µS/cm				
ED045G: Chloride by Discrete Analys	ser						
Chloride	16887-00-6	1	mg/L				
ED093F: Dissolved Major Cations							
Sodium	7440-23-5	1	mg/L				
ED093F: SAR and Hardness Calculat	ions						
Sodium Adsorption Ratio		0.01	-				
EK040P: Fluoride by PC Titrator							
Fluoride	16984-48-8	0.1	mg/L	0.1	0.3	0.2	
EK059G: Nitrite plus Nitrate as N (NC	Dx) by Discrete Ana	lyser					
Nitrite + Nitrate as N		0.01	mg/L				
EK061G: Total Kjeldahl Nitrogen By I	Discrete Analyser						
Total Kjeldahl Nitrogen as N		0.1	mg/L				
EK062G: Total Nitrogen as N (TKN +	NOx) by Discrete Ar	nalyser					
^ Total Nitrogen as N		0.1	mg/L				
EK067G: Total Phosphorus as P by D)iscrete Analys <u>er</u>						
Total Phosphorus as P		0.01	mg/L				

Appendix B – NATA Laboratory Waste Classification Results



CERTIFICATE OF ANALYSIS

Work Order	ES1623476	Page	: 1 of 4
Client	: MJM ENVIRONMENTAL PTY LTD	Laboratory	: Environmental Division Sydney
Contact	: MS BRIGID KELLY	Contact	:
Address	: OFFICE 1, 335 WHARF ROAD NEWCASTLE NSW, AUSTRALIA 2300	Address	: 277-289 Woodpark Road Smithfield NSW Australia 2164
Telephone	: +61 49264222	Telephone	: +61-2-8784 8555
Project	: 034 1612	Date Samples Received	: 19-Oct-2016 09:59
Order number	: 49264222	Date Analysis Commenced	: 20-Oct-2016
C-O-C number	:	Issue Date	: 25-Oct-2016 16:55
Sampler	: ADAM BUCIOR		IC-MRA NATA
Site			
Quote number	:		Accreditation No. 825
No. of samples received	: 3		Accredited for compliance with
No. of samples analysed	: 3		ISO/IEC 17025 - Testing

This report supersedes any previous report(s) with this reference. Results apply to the sample(s) as submitted. This document shall not be reproduced, except in full.

This Certificate of Analysis contains the following information:

- General Comments
- Analytical Results

Additional information pertinent to this report will be found in the following separate attachments: Quality Control Report, QA/QC Compliance Assessment to assist with Quality Review and Sample Receipt Notification.

Signatories

This document has been electronically signed by the authorized signatories below. Electronic signing is carried out in compliance with procedures specified in 21 CFR Part 11.

Signatories	Position	Accreditation Category
Ankit Joshi Merrin Avery	Inorganic Chemist Supervisor - Inorganic	Sydney Inorganics, Smithfield, NSW Newcastle - Inorganics, Mayfield West, NSW
Wisam Marassa	Inorganics Coordinator	Sydney Inorganics, Smithfield, NSW



General Comments

The analytical procedures used by the Environmental Division have been developed from established internationally recognized procedures such as those published by the USEPA, APHA, AS and NEPM. In house developed procedures are employed in the absence of documented standards or by client request.

Where moisture determination has been performed, results are reported on a dry weight basis.

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^ = This result is computed from individual analyte detections at or above the level of reporting

ø = ALS is not NATA accredited for these tests.

~ = Indicates an estimated value.

Page	: 3 of 4
Work Order	: ES1623476
Client	: MJM ENVIRONMENTAL PTY LTD
Project	: 034 1612



Sub-Matrix: SOIL (Matrix: SOIL)	Client sample ID			MEDIA -1	MEDIA-2	MEDIA-3	
	Cl	ient samplii	ng date / time	[19-Oct-2016]	[19-Oct-2016]	[19-Oct-2016]	
Compound	CAS Number	LOR	Unit	ES1623476-001	ES1623476-002	ES1623476-003	
				Result	Result	Result	
EK040T: Fluoride Total							
Fluoride	16984-48-8	40	mg/kg	40	<40	<40	
EN33: TCLP Leach							
Initial pH		0.1	pH Unit	6.2	6.1	6.1	
After HCI pH		0.1	pH Unit	1.9	1.9	1.9	
Extraction Fluid Number		1	-	1	1	1	
Final pH		0.1	pH Unit	5.1	5.1	5.1	



Sub-Matrix: TCLP LEACHATE (Matrix: WATER)		Clie	ent sample ID	MEDIA -1	MEDIA-2	MEDIA-3	
	Client sampling date / time			[19-Oct-2016]	[19-Oct-2016]	[19-Oct-2016]	
Compound	CAS Number LOR Unit		ES1623476-001	ES1623476-002	ES1623476-003	 	
				Result	Result	Result	
EK040P: Fluoride by PC Titrator							
Fluoride	16984-48-8	0.1	mg/L	0.2	0.2	0.1	