

7th January 2016

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Dear Gareth

Results for the Treatment Trial for Molybdenum at Surrey Street, Glossop

Synopsis

The site was formally an Iron Foundry (Ferro Alloys), used to produce metals and metalloids from 1860s to 1990s. It is likely that the site processes which have been conducted on site have generated the ashy made ground which seems to contain the contaminant of concern – Molybdenum.

The Molybdenum contamination is currently in a form which is mobile and it is migrating off site into the water course below the site.

The core objective is to undertake a lab based trial to replicate site conditions and best case conditions to prove that a chemical reaction can be successfully undertaken to physically change the available Molybdenum so that it falls out of suspension.

Following the bulk sampling, the recovered material was handed directly to the laboratory so that all sub-sampling, commissioning, bench testing and reporting was undertaken independently.

The results obtained from the laboratory show the complex nature and variability of the contaminated ashy material when test conditions tried to replicate site conditions, however it also showed that the proposed chemical reaction has accrued and can be successfully reproduced on site as a treatment process under our mobile plant permit.

One of the bulk samples selected by the laboratory was ground, sieved to ensure the sample was homogenized. This was then subject to the same testing and yielded very positive results showing that the chemical reaction was in line with expectations whereby the leachable Molybdenum had signification decreased but the solid totals for the Molybdenum remain unchanged.

Sampling, Testing, Results and Discussion

After obtaining 4 bulk samples from the site on 30th October 2015, as the form of contamination Molybdenum does not exhibit any simple detectable olfactory signature an assumption was made based on the ground conditions encountered that the contamination would be present within the ashy made ground.

All samples recovered were obtained using a JCB to break through the concrete slab and then dig through the made ground to allow visual inspection to take place and enable further consideration to the ground conditions for the proposed remediation treatment methodology.

The ashy made ground did not exhibit any obvious signs that it could be contaminated, however given the nature of the contamination, historic use of the site and the likely hood that the ash will contain metalloids this material became the main focus of the trials.

Prior to commencing with the bench testing the 4 recovered bulk samples were subject to total and leachable testing to determine if Molybdenum was present in sufficient quantities to warrant further use in the bench testing to prove the chemical reaction to reduce the available Molybdenum.

Please find below the table of results denoting the bench testing conducted to best emulate the site conditions and poor mixing of the material in case sections of the ashy material have become cohesive hence the reduced surface area and limited chemical interaction.

Sample	Treatment	Untreated 2:1 leachate (mg/l)	Treated 2:1 Leachate (mg/l)	Total in Treated sample (mg/kg)
AR 1	Control	47	5	4869
	3:1 Ferric Sulphate	85	3	5737
	6:1 Ferric Sulphate	47	12	6790
	Sodium Hydroxide only	46	47	7027
AR 2	Control	10	3	1002
	3:1 Ferric Sulphate	15	< 1	1784
	6:1 Ferric Sulphate	14	25	1139
	Sodium Hydroxide only	< 1	14	1126
AR 3	Control	41	< 1	3913
	3:1 Ferric Sulphate	47	< 1	3850
	6:1 Ferric Sulphate	37	50	3318
	Sodium Hydroxide only	40	28	2663
AR 4	Control	59	29	3966
	3:1 Ferric Sulphate	61	7	6292
	6:1 Ferric Sulphate	59	< 1	2850
	Sodium Hydroxide only	61	79	4737

The results for our preferred treatment methodology is to use a 3:1 Ferric Sulphate to Molybdenum and then return the pH back to neutral using Sodium Hydroxide to cause the Molybdenum to fall out of suspension and flocculate. The higher ratio of 6:1 was used to check to see if a greater recovery of Molybdenum could be enacted to further improve the treatment methodology. However the results do not warrant the significant cost increases for such limited gains.

The final stage of testing used just Sodium Hydroxide due to information from an alleged former site employee during the site investigation, where it was claimed that the ash was mixed with acid. However this has not yielded results in line with expectations due to the limited amount of molybdenum reacted and later present in the treated sampling round of testing.

The control material tested which did not undergo any form of treatment seems to start with high levels of Molybdenum and then diminish significantly for the second test. This cannot be easily explained as just inconsistency with levels of molybdenum within the sample matrix. Due to the complex nature of the material this may partly account for the differing levels but it is not possible to draw a defining conclusion as to why the control has a higher level within the untreated and low levels of treated Molybdenum.

In order to add a further element of confidence an extra line of bench testing was conducted to further prove that the proposed treatment methodology will reduce the amount of available Molybdenum.

The table below is the bench testing to show the best case, as the sample was ground and mixed prior to testing, thus yielding very close similar results as the total Molybdenum remains the same in each of the 4 sub samples.

Sample	Treatment	Untreated 2:1 leachate (mg/l)	Treated 2:1 Leachate (mg/l)	Total in Treated sample (mg/kg)
AR3	Control	30	17	3800
	3:1 Ferric Sulphate	29	1	3700
	6:1 Ferric Sulphate	28	3	3500
	Sodium Hydroxide only	28	49	3600

The results were obtained by grinding the sample into a course powder and sieving the material in order to use just 2mm size and below particles to gain a fully harmonized sample, which was obtained based on the untreated and total treated level results for Molybdenum.

The control result has been rerun as it does not contain the same level of certainty as the other set of results obtained from this data set. However it is much improved but still shows a reduction in Molybdenum despite not undergoing any treatment which cannot be directly explained.

Based on the results obtained by emulating best case and site derived conditions we are confident that the ashy material containing the Molybdenum can be treated in line with the laboratory testing to achieve the same results.

Summary of upscaling treatment

In order to up scale the physical treatment element we proposed to use a conveyor belt based system to allow a thin layer of material to be spread across the belt while adding the necessary chemicals in 2 phases (first pass adding Ferric Sulphate). The same process will be repeated but using the a different chemical (second pass adding Sodium Hydroxide) to increase the pH from an acidic material to a neutral pH level thus completing the reaction to cause the molybdenum to flocculate and fall out of suspension and reduce the mobility within the groundwater and prevent off site migration.

The order of works will be to break out the hard surfaced material covering the site, then to stockpile the ashy material prior to undergoing mass treatment as denoted above. The excavation void will be allowed to fill with groundwater which will be subsequently pumped out, treated and discharged from site under consent from the Statutory Water Undertaker.

Testing will be conducted to monitor the levels and availability of Molybdenum before, during and after completing the treatment phasing. Validation testing will then be conducted on the treated stockpile to determine if the material can be replaced within the site or subjected to another round of treatment to further reduce the levels of leachable Molybdenum from the ash/made ground.

Based on the laboratory bench testing and results we are confident that this alternative treatment methodology will work and we would be happy to present our findings with you to the Environment Agency should you wish to carry this forward.

If you require any further information we would be happy to discuss our proposals with you at a convenient time and venue.

Yours sincerely



Jon Berry
Environmental and Remediation Manager



L15/2216/ARD/002

Project Reference – Surrey Street, Glossop

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Report revision	Prepared by	Date	Checked by	Date	Issued by	Date
0	P. Swanston	January 2016	J. Gane	January 2016	J. Gane	January 2016

1.0 INTRODUCTION

1.1 Nicholls Colton Analytical (NCA) were instructed by Mr J Berry, on behalf of A R Demolition (ARD) to undertake a lab trial to assess viability of a proposed remediation strategy. The basis of this was to reduce the leachability of Molybdenum in samples of soil obtained from site at Surrey Street, Glossop.

NCA were instructed to:

- Undertake initial leachate testing for each sample (2:1ratio in accordance with BS EN12457)
- Undertake various trials of the proposed remediation strategy, including two different concentrations of Ferric Sulphate followed by treatment with Sodium Hydroxide, treatment with Sodium Hydroxide only and no treatment at all as a control
- Undertake leachate testing for each sample (2:1ratio in accordance with BS EN12457) post treatment

1.2 Laboratory testing was undertaken by Nicholls Colton Analytical's testing laboratory, 7-11 Harding Street, Leicester LE1 4DH.

All results and interpretations / opinions are outside the scope of any accreditation.

1.3 Four samples were initially received for analysis on 30th October 2015:

NCA Sample Number	Client Reference	Date Sampled	Time Sampled
15-30611	AR 1	30/10/15	AM
15-30612	AR 2	30/10/15	AM
15-30613	AR 3	30/10/15	AM
15-30614	AR 4	30/10/15	AM

Laboratory trial work was carried out in the period 18th November 2015 to 5th January 2016.

2. Initial Leachable Molybdenum Stabilisation Test

For each sample (AR1, AR2, AR3 & AR4) a representative portion was taken and split into four sub-samples.

NCA Sample Number	Client Reference	NCA Sample	Client Reference	NCA Sample	Client Reference	NCA Sample	Client Reference
15-31492	AR 1/1	15-31496	AR 2/1	15-31500	AR 3/1	15-31504	AR 4/1
15-31493	AR 1/2	15-31497	AR 2/2	15-31501	AR 3/2	15-31505	AR 4/2
15-31494	AR 1/3	15-31498	AR 2/3	15-31502	AR 3/3	15-31506	AR 4/3
15-31495	AR 1/4	15-31499	AR 2/4	15-31503	AR 3/4	15-31507	AR 4/4

2.1. Leachate Analysis – Pre-Treatment

A leachate for each of the sixteen sub-samples was prepared (2:1 ratio in accordance with BS EN12457) and then analysed for Molybdenum (in accordance with In-House Method Statement MS-CL-ICP Metals Water). Results summarised in Table 1 below:

Table 1: Initial Leachate Analysis

NCA Sample Number	Client Reference	Molybdenum Pre-Treatment (mg/l)
15-31492	AR 1/1	47
15-31493	AR 1/2	85
15-31494	AR 1/3	47
15-31495	AR 1/4	46
15-31496	AR 2/1	10
15-31497	AR 2/2	15
15-31498	AR 2/3	14
15-31499	AR 2/4	<1
15-31500	AR 3/1	41
15-31501	AR 3/2	47
15-31502	AR 3/3	37
15-31503	AR 3/4	40
15-31504	AR 4/1	59
15-31505	AR 4/2	61
15-31506	AR 4/3	60
15-31507	AR 4/4	61

2.2. Treatment

For each of the original samples split into four, the first test portion was left untreated as a control. The second test portion was treated with Ferric Sulphate solution at a ratio 3 parts Ferric Sulphate to 1 part Molybdenum, the Ferric Sulphate was dissolved in water and the solution stirred into the sample then left for 24hours. The test portion then had 1M Sodium Hydroxide solution added until the sample pH was within the range pH 7-8 and left for a further 24hours. The third test portion was treated in the same way but with Ferric Sulphate solution at a ratio of 6 parts Ferric Sulphate to 1 part Molybdenum. The fourth test portion had no Ferric Sulphate added but a similar amount of water to those treated and then 1M Sodium Hydroxide added until the sample pH was within the range pH 7-8 and left for a further 24hours. See Table 2 for summary of treatment for each sample. All samples were then air-dried ready for further analysis.

Table 2: Summary of Chemical Treatment

NCA Sample Number	Client Reference	Test Portion	Amount of Ferric Sulphate Added	Amount of Water Added	Amount of 1M Sodium Hydroxide added
15-31492	AR 1/1	250g	None	None	None
15-31493	AR 1/2	250g	9.78g	30ml	43ml
15-31494	AR 1/3	250g	19.57g	30ml	150ml
15-31495	AR 1/4	250g	None	30ml	20ml
15-31496	AR 2/1	250g	None	None	None
15-31497	AR 2/2	250g	1.47g	10ml	0ml*
15-31498	AR 2/3	250g	2.93g	20ml	0ml*
15-31499	AR 2/4	250g	None	15ml	20ml
15-31500	AR 3/1	250g	None	None	None
15-31501	AR 3/2	250g	6.61g	30ml	20ml
15-31502	AR 3/3	250g	13.21g	30ml	70ml
15-31503	AR 3/4	250g	None	30ml	20ml
15-31504	AR 4/1	250g	None	None	None
15-31505	AR 4/2	250g	6.20g	30ml	30ml
15-31506	AR 4/3	250g	12.40g	30ml	60ml
15-31507	AR 4/4	250g	None	30ml	30ml

*Sample pH was within the range 7-8pH units without requiring addition of Sodium Hydroxide

2.3. Leachate Analysis – Post-Treatment

Following treatment a leachate for each of the sixteen samples was prepared (2:1 ratio in accordance with BS EN12457) and then analysed for Molybdenum (in accordance with In-House Method Statement MS-CL-ICP Metals Water).

Results summarised in Table 3 below:

Table 3: Treated Leachate Analysis

NCA Sample Number	Client Reference	Molybdenum Post-Treatment (mg/l)
15-31492	AR 1/1	5.3
15-31493	AR 1/2	2.9
15-31494	AR 1/3	12
15-31495	AR 1/4	47
15-31496	AR 2/1	4.2
15-31497	AR 2/2	3.5
15-31498	AR 2/3	<1
15-31499	AR 2/4	25
15-31500	AR 3/1	14
15-31501	AR 3/2	<1
15-31502	AR 3/3	<1
15-31503	AR 3/4	51
15-31504	AR 4/1	29
15-31505	AR 4/2	7.7
15-31506	AR 4/3	<1
15-31507	AR 4/4	79

2.4. Soil Analysis – Post-Treatment

A sample from each of the treated test portions was prepared and analysed for total Molybdenum (in accordance with In-House Method Statement MS-CL-ICP Metals).

Results summarised in Table 4 below:

Table 4: Total Molybdenum Analysis Post-Treatment

NCA Sample Number	Client Reference	Molybdenum Post-Treatment (mg/kg)
15-31492	AR 1/1	4900
15-31493	AR 1/2	5700
15-31494	AR 1/3	6800
15-31495	AR 1/4	7000
15-31496	AR 2/1	1000
15-31497	AR 2/2	1800
15-31498	AR 2/3	1100
15-31499	AR 2/4	1100
15-31500	AR 3/1	3900
15-31501	AR 3/2	3900
15-31502	AR 3/3	3300
15-31503	AR 3/4	2700
15-31504	AR 4/1	4000
15-31505	AR 4/2	6300
15-31506	AR 4/3	2900
15-31507	AR 4/4	4700

3. Further Stabilisation Test

The initial stabilisation testing showed some variation in results within the samples. This was attributed to the non-homogeneous nature of contamination within the samples. As a result further stabilisation testing was carried out using sample AR3 which was homogenised by drying and grinding prior to the trials.

3.1. Leachate Analysis – Pre-Treatment

A leachate for each of the four samples was prepared (2:1 ratio in accordance with BS EN12457) and then analysed for Molybdenum (in accordance with In-House Method Statement MS-CL-ICP Metals Water). Results summarised in Table 5 below:

Table 5: Further Trial - Initial Leachate Analysis

NCA Sample Number	Client Reference	Molybdenum Pre-Treatment (mg/l)
15-31500	AR 3/1	30
15-31501	AR 3/2	29
15-31502	AR 3/3	28
15-31503	AR 3/4	28

3.2. Soil Analysis – Pre-Treatment

A sample from each of the test portions was prepared and analysed for total Molybdenum (in accordance with In-House Method Statement MS-CL-ICP Metals) prior to any treatment. Results summarised in Table 6 below:

Table 6: Further Trial - Total Molybdenum Analysis Pre-Treatment

NCA Sample Number	Client Reference	Molybdenum Pre-Treatment (mg/kg)
15-31500	AR 3/1	3700
15-31501	AR 3/2	3600
15-31502	AR 3/3	3600
15-31503	AR 3/4	3700

3.3. Ferric Sulphate Treatment

As before test the first test portion was left untreated as a control.

The second test portion was treated with Ferric Sulphate solution at a ratio 3 parts Ferric Sulphate to 1 part Molybdenum, the Ferric Sulphate was dissolved in water and the solution stirred into the sample then left for 24hours. The test portion then had 1M Sodium Hydroxide solution added until the sample pH was within the range pH 7-8 and left for a further 24hours.

The third test portion was treated in the same way but with Ferric Sulphate solution at a ratio of 6 parts Ferric Sulphate to 1 part Molybdenum.

The fourth test portion had no Ferric Sulphate added but a similar amount of water to those treated and then 1M Sodium Hydroxide added until the sample pH was within the range pH 7-8 and left for a further 24hours.

See Table 7 for summary of treatment for each sample.

All samples were then air-dried ready for further analysis.

Table 7: Further Trial - Summary of Chemical Treatment

NCA Sample Number	Client Reference	Test Portion	Amount of Ferric Sulphate Added	Amount of Water Added	Amount of 1M Sodium Hydroxide added
15-31500	AR 3/1	250g	None	None	None
15-31501	AR 3/2	250g	6.61g	30ml	0ml*
15-31502	AR 3/3	250g	13.21g	30ml	70ml
15-31503	AR 3/4	250g	None	30ml	10ml

*Sample pH was within the range 7-8pH units without requiring addition of Sodium Hydroxide

3.4. Leachate Analysis – Post-Treatment

Following treatment a leachate for each of the four samples was prepared (2:1 ratio in accordance with BS EN12457) and then analysed for Molybdenum (in accordance with In-House Method Statement MS-CL-ICP Metals Water). Results summarised in Table 8 below:

Table 8: Further Trial - Treated Leachate Analysis

NCA Sample Number	Client Reference	Molybdenum Post-Treatment (mg/l)
15-31500	AR 3/1	17
15-31501	AR 3/2	1.0
15-31502	AR 3/3	3.0
15-31503	AR 3/4	49

3.5. Soil Analysis – Post-Treatment

A sample from each of the treated test portions were prepared and analysed for total Molybdenum (in accordance with In-House Method Statement MS-CL-ICP Metals). Results summarised in Table 9 below:

Table 9: Further Trial - Total Molybdenum Analysis Post-Treatment

NCA Sample Number	Client Reference	Molybdenum Post-Treatment (mg/kg)
15-31500	AR 3/1	3800
15-31501	AR 3/2	3700
15-31502	AR 3/3	3500
15-31503	AR 3/4	3600



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