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Author  
2020-05-25 Paul Wersin  
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## Factual review of TR-20-14 - Paul Wersin

<b>Review statement/Response statement</b>
<i>Reviewer:</i> Paul Wersin, Uni Bern
<i>Type of review:</i> Factual review
<i>Review document:</i> The report “Corrosion of copper in bentonite after 20 years exposure in the field tests LOT S2 and LOT A3.”, SKBdoc id 1900516, SKBdoc version 0.2. Appendices A-H with the following SKBdoc id’s, 1900518, 1900519, 1900520, 1900521, 1900522, 1900523, 1900524, 1900525, version 0.1 for all appendices.
<i>Review plan:</i> TR-20-14 Review instructions, SKBdoc id 1895707, SKBdoc version 0.5

## Summary

**Assessment of fulfilment of review criteria:**

Address each criterion with "Yes, meets the criterion" or "No, see comment XX"

- |   |  |
|---|--|
| 1 | The objective, scope and premises for the report and analyses should be clearly and traceably presented. <b>No, see comment 0</b>  |
| 2 | Methodologies and models are verified and validated or proven. <b>Yes, meets the criterion</b>   |
| 3 | Used methodologies and models are applicable and have been used within its possibilities and limits. <b>In general yes, but regarding SEM-EDS, see comments 16, 18, 21, 52</b> |
| 4 | It should be possible to evaluate the quality of cited references of importance for the conclusions reached. <b>Yes, meets the criterion</b>                                   |
| 5 | Factual information shall be supported by relevant references. <b>Yes, meets the criterion</b>   |
| 6 | The conclusions reached shall be supported by the analyses, other findings in the report and/or by supporting documents. <b>Yes, meets the criterion</b>                       |
| 7 | The conclusions should be clearly reported and in such way that the fulfilment of the stated objectives can be judged. <b>No, see comment 0</b>                                |

## Review comments/questions

## Response statement

No.	Section.	Concerning <i>[Filled in by reviewer]</i>	Comment / Question <i>[Filled in by reviewer]</i>	Corrections / Measure <i>[Filled in by author]</i>
<i>[ID ]</i>	<i>[Page, headline, section ]</i>	<i>[Content, e.g. text, figure, table, etc. that the comment concerns. For example, text that is to be commented may be quoted.]</i>	<i>[Specification of comment and/or question, including motivation. If needed, provide advices, instructions and suggestion for improvements]</i>	<i>[Brief description of how the review comment will be handled ]</i>
0	General	Objectives of report	These are not explicitly stated.	Added to section 1.1.

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1	General	Overall comment	A comprehensive study that has provided valuable new data and confirmed the conceptual view of Cu canister corrosion in KBS-3 type settings. Careful microscopic analysis has enabled to unambiguously identify Cu <sub>2</sub> S as corrosion product which had not been possible in previous in-situ studies.	Ok.
2	1.2	Fate of O <sub>2</sub>	It is important to note that considerations on O <sub>2</sub> should include the possibility of leakage of air through plug, cable conduits and/or EDZ. For example, this was clearly the case in the FEBEX experiment.	We cannot be certain that no leakage has occurred in the ways mentioned, but there is no sign of significant in-leakage from the corrosion analysis. Examination of clay from earlier test parcels have indicated reducing conditions, I think.
3	1.2	Description FEBEX experiment	O <sub>2</sub> could in fact be measured until the end of experiment (see Fernandez & Giroud, 2017, Nagra NAB 16-003.	Ok, good to know.
4	1.5	General comment	Good to mention these limitations so clearly!	Ok.
5	1.5	Tightness of system	Can it be assured that the system was tight against air ingress?	Tried to clarify tightness of the copper pipe in 1.5: "The lower part of the copper pipe was welded to a bottom plate, also made of copper. The upper part of the copper pipe (ca 0.7 m) was in direct contact with the atmosphere in the Äspö tunnel (Figure 1). Since the upper end of the pipe was open, the interior of the pipe was filled with air during the whole exposure. In order to assure that the weld at the bottom plate was not leaking air into the clay system, the tightness of the weld was tested using helium gas (Sandén and Nilsson, 2020). The corrosion of these surfaces (upper part and inner surface) has thus not occurred under repository-like conditions."

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6	2.1	Sampling procedure	It would be useful to dwell a bit more on the sampling procedure: for example how long was exposure to air on-site and during preparation? Were there measures (if any) to reduce/minimise exposure to air?	I have added a sentence about this, estimating total exposure to air < 1 hour.
7	2.1	Bentonite samples	It should be clarified if bentonite samples were dried (and how) for the different analyses.	This was further clarified.
8	Table 2	Reference for unexposed pipe	Was there no ref. sample for the Cu pipes?	A reference pipe has been analysed regarding its content of O and H. Added sample in the table of samples and analysis techniques in section 2.1 and in the result section.
9	2.2	Method description	More details on SEM/EDS method would be useful. E.g. type of detectors, carbon coating, description of EDS quantification method (standardless?)	A description of the EDS quantification was added.
10	2.4	Gravimetric analysis	Not clear (at least not for a non-expert) how corrosion depth was calculated from mass-loss data which do not seem to have reached constant weight (see Appendix D).	Method explained a bit further.
11	2.7	XRF analysis of bentonite samples	Description not clear regarding sample drying and sample preparation in general. What does "if needed, dried at 60°C" mean? It is important to know what the water content of the sample is.	If the water content is too high the ball mill does not work. The bentonite needs to be a bit dry in order to mill it. More info about this was added to the sample handling section in the introduction. The final water content was measured using standard method (105C /24h) and is shown in Table 10. More info about this was added to the sample handling section in the introduction.

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12	2.7	XRD of bentonite samples	Same comment regarding drying of samples	If the water content is too high the ball mill does not work. The bentonite needs to be a bit dry in order to mill it. More info about this was added to the sample handling section in the introduction. The final water content in the samples for XRD is not important as long as the basal reflections does not interfere with the reflections from the other phases.
13	3.1	Sample prep.	Was sample preparation carried out in air or under O <sub>2</sub> -free conditions? If in air, then what is impact of oxidation?	In this study all preparation was done in air. We are not looking on the oxidation state of any element, and the XRF method is blind to water and oxygen, so I cannot see any impact from this. More info about this was added to the sample handling section in the introduction.
14	3.2.1	"..XRD directly onto the surfaces."	Not clear	Clarified that it was in the as received state.
15	3.2.1	Ca and S analyses	From combined Ca and S mappings it should be possible to clearly identify locations of CaSO <sub>4</sub>	Yes, however, I only focused on the Cu and wanted to show the correlation between Cu and S. In the dataset shown (EDS map) most of the S correlates to Cu. Gypsum seems to enrich into larger crystals and is perhaps not so finely distributed as the Cu-sulfide. In this report the focus is very much on Cu, in the next report we will focus on the bentonite, and the fate of the minerals etc.
16	3.2.1	EDS: Analysis of O	The analysis of light elements such as O is prone to large errors and is usually not reported. Moreover, if sample is embedded in resin, the analyses of C and O are not meaningful.	The sample was not embedded. More info about this was added to the SEM sample handling section. In the EDS map figure O correlates with Si and Al, and anti-correlates with Cu and S. No interpretation of this was done, however it is perfectly compatible with the interpretation that we see Cu-sulfide (Cu, S) in montmorillonite (Si,Al,O). For EDS spot analysis I only evaluate the ratio of Cu and S, the other elements are included for anyone interested to use with caution. If they are included or not only affects the normalisation to 100% and does not affect the Cu/S ratio.
17	Fig. 11 and others	Figure legend etc.	Font very small and difficult to read	Most of the figures were remade or improved.

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18	Fig. 11	EDS analyses	Again: O is very imprecise. Recommend to normalise without O. This would enable better comparison of contents of S. Regarding concentrations below 1%: Could a peak be identified for example for Ca? Otherwise might be artefact. This comment refers to all EDS data.	I agree with you to be careful when including elements in low quantities. However, we know that all these elements are very common in bentonite. Actually we even do expect them all to be in this sample and even in this region of interest. We see them in XRF all the time, and the levels detected here are more or less what we expect. If the software finds a bit of Ca in a bentonite, I think it is ok to include it, but I am very well aware of the high relative uncertainties of such a number. The exception here is carbon, most of the signal comes from the adhesive film used to stick the sample to the sample holder. A comment about this was added for improved clarity. Since only the Cu/S ratio was used, and there is little scattering between the different sites, I see no problem with this procedure. Normalisation by excluding elements have no or insignificant influence on the Cu/S ratio.
19	3.2.1, last section	Typo	Chalcocite	Corrected.
20	Table 7	Reference sample	Any EDS for ref. sample?	No EDS done for reference pipes but for ref coupons, added in 3.3.1: "For comparison, the level of S in EDS spectra for the reference coupons was in most cases below detection and in all cases less than 1 at-% (Figures C27-C32)."
21	Fig. 17	Na	Was this element measured? Was it below detection? Comment also refers to most other EDS data.	Where elements are not included they were not detected.
22	Fig. 19	GDOES data	How do these data compare to corresponding EDS data?	Qualitatively similar trends. Discussed in 4.1.

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23	3.5	XRF data	Again: What about LOI and H <sub>2</sub> O in the XRF samples?	There is no LOI in the XRF, as stated the samples were made by compaction. The water content is shown in Table 10 for the samples close to the copper inserts. The water content in the sample in contact with the heater was not determined; however, all samples had comparable water content (more or less in equilibrium with the ambient relative humidity) and hence had no or insignificant impact on the measurement (see Table 11; the Cu quantification works really well). For the data evaluation a dry density of 1.5 g/cm <sup>3</sup> was used, which seemed like a nice number close to an average of observed data reported in Sandén and Nilsson, 2020.
24	3.5	Last sentence of intro	Not clear what is meant. In principle, the Cu stemming from Cu corrosion can be in the corrosion layer on the coupon surface <u>or</u> in the adjacent bentonite.	Clarified and added discussion of thickness of the oxide layers adherent to the surfaces.
25	3.5.1	“Based on earlier experience...”	A possibility would be to prepare a sample normal to the metal/clay contact and do SEM/EDS analysis.	The SEM/EDS is not at all as good for quantification as XRF at these low levels of copper. With XRF you measure a homogenised sample with a large surface. With SEM/EDS you will have big variations depending on where in the material you look as the Cu-sulfide in very inhomogenously distributed.
26	3.5.1	Sample prep.	How was sample of 0-10 mm prepared?	A line was marked at 10 mm distance, this line was cut with a small saw.
27	3.5.1	Fig. 24	“All bentonite at 5 mm distance from copper plate” How accurately could this distance be achieved?	This was done fairly accurately. The really big error (assumption), is the estimation of how complete a sample is from a perfect sample. This error is very difficult to estimate, but is the biggest uncertainty, hence the “+- 20% “was added to cope with this.

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28	3.5.1.1	0.03-0.05 wt%	What is accuracy and error?	<p>Table 9 shows that the repeatability of the XRF measurements was very high (low SD).</p> <p>Table 11 show that the bias for the Cu measurements was very low.</p> <p>As stated in the previous comment, the uncertainty in the final result is not a function of the XRF measurement, it is a function of how to estimate how complete the sample of bentonite was in relation to a perfect sample. As not all bentonite surrounding the insert was available, this was roughly estimated, and this is by far the biggest error.</p> <p>The relative error in bias is around 1% in Table 11, while the numbers in the final statement are given with only one significant figure, and as a range with a pretty large error margin, so I would say the final statement is pretty solid:</p> <p>“Based on the assumptions above regarding the sample representativeness, the amount of copper leaving the coupons into the bentonite in the different samples is probably in the range of 0.01-0.02 g <math>\pm</math>20%. It may be noted that this range, including the estimated uncertainty in the method, is in rather good agreement with the mass-losses determined by standardised gravimetric methods”</p>
29	3.5.1.3	Calculation Cu in bentonite	It is not clear if wt% values refer to dry or wet material.	The XRF does not measure hydrogen or water. The included elements are normalised to 100% as oxides. To calculate the amount of copper in the clay, the dry mass should be used. This was clarified further.
30	3.5.1.3	Last sentence not clear	I don't understand this statement. Here you are measuring the amount of Cu transferred to the clay. Why should this be the same as the Cu measured in the surface layer?	The mass loss using standard gravimetric methods is estimating the total corrosion of the copper. This can be compared with the copper in the bentonite if the amount of corrosion products still present on the heater are negligible (here they seem to be ca 350 nm, ie. Ca 200 nm copper corrosion).



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31	Table 9	Chemical analysis	What about H <sub>2</sub> O?	You cannot measure hydrogen with XRF. Elements with lower atomic number than Na are not measured, this was further clarified in the introduction of the method. The dry mass of the clay is used for the calculation, and this works fine as shown in Table 11.
32	Table 10	Water content	Determined by mass loss @ 60 °C. In that case the determined water content may be too low. The standard is 105 °C.	No, the water content was determined using standard procedure (105 C, 24 h). This was clarified. The 60 C is only used if needed to dry the bentonite in order to be able to mill it.
33	Fig. 26	Readability	Fonts very small.	Figure was improved.
34	Fig. 26	Elemental maps	Combining Cu and S maps would better illustrate Cu <sub>x</sub> S-rich areas	Actually I think you see this better by not combining them. The Cu and S clearly have almost the same pattern which anti-correlates to Al, Si and O patterns.
35	Fig. 27	Figure quality	Rather poor	Figure was improved.
36	Table 13	EDS data	C and O data are very uncertain. Where does C come from? Resin? Would be better to normalise without these elements.	C comes mainly from the adhesive film used to stick the sample to the sample holder. Normalising without C would have no or negligible impact on the Cu/S ratio.
37	Figs. 28 & 29		What do peaks represent?	As stated in the figure text, the diffraction pattern of the bentonite sample with the corrosion product is compared to a calculated diffraction pattern of chalcocite.
38	3.5.2	Title	Do you mean pipes?	With heater I mean the warm copper cylinder in the bentonite. It is not really a heater, but it is heating the bentonite, and we commonly call it the heater.

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39	3.5.2.1	Drying	Same comment as above: Was all water removed?	No. Only enough in order to be able to mill but still compact the bentonite. If the the clay is too wet you cannot mill it. If it is too dry you cannot compact it properly. The XRF does not measure hydrogen, and the impact from water content on the measurement is very small and negligible. All about drying, impact from water and measurement of water content was further clarified.
40	3.5.2.2	S and Ca enrichment at heater	What about anhydrite?	Both gypsum and anhydrite can be present depending on the conditions during the experiment, as well as how fresh the sample is as water can hydrate anhydrite by internal movement within the bentonite samples. Gypsum was changed to gypsum/anhydrite, and a small section discussing gypsum/anhydrite was added.
41	3.5.2.2	Last sentence	Another explanation would be that hotter parts had higher O <sub>2</sub> concentrations because they remained unsaturated for longer time.	Yes that is true. This was added to the text.
42	Fig. 30		Ca could be plotted too	Yes I totally agree. Initially it was. However, as the focus in this report is on copper, and to make it more clear, Ca was finally not plotted. In the next report more focus will be on bentonite and its minerals, and in that report we will focus much more on precipitates such as gypsum/anhydrite.
43	3.5.2.3	Calculation of Cu amount	Why is saturated density used if material was “dry”? In general, it would be more precise to use the measured densities, if they are available.	This was a mistake that has been corrected. The calculation was updated using 1.5 g/cm <sup>3</sup> which was close to an averaged measured dry density from Sandén and Nilsson, 2020. The true heater diameter was also used in the updated calculation.
44	3.5.2.3	General	Perhaps all calculations do not have to be put in such detail in the text. An example could be given in a Table instead.	Yes, I agree. The amount of calculations shown have been reduced. However, I do want this to be as transparent as possible to avoid errors and to avoid someone thinks we are hiding something.

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45	3.5.2.3	Calculation corrosion depth	Cu in corrosion layer is not included in this calculation.	Yes that is true. Actually it says “estimation of “ not “calculation of”. This difference was however further clarified.
46	3.5.2.4	Phases close to heater	Was accumulation of other salts, such as NaCl observed?	No, so far only Ca-sulfate has been identified as accumulated salt. Looking on the Cl content in XRF data indicate no accumulation that I can see. Possibly carbonates can have been accumulated however, as XRF does not measure carbon, this is not as easy to detect. We will later send samples for carbon and sulfur analysis using EGA and we will then have more information. Additionally XRD data can be used for this, but I have not yet looked on that in detail.
47	Figs. 34 & 35	Legend	Not legible, gypsum peaks could be indicated.	The entire figure was updated, including indication of gypsum.
48	4	General	It would be nice to have a summary table with all the corrosion depth estimates obtained from mass loss, EDS etc.	We refer to the tables in the result section instead.
49	4.1	Organisation	This section describes macroscopic observations. Why isn't it in the Results chapter?	Agree, the section has been moved into the results chapter.
50	4.1	Corrosion features of pipes	These seem very corroded, especially the bottom of S3 pipe. Any chance that crack has developed such that O <sub>2</sub> could have escaped to the outer parts?	Im not sure which parts of the pipes the reviewer means here. We haven't made a pressure/gas tightness test after retrieval but my judgement is that its highly unlikely that the pipe had cracked. Its quite difficult to judge from the photos how much corrosion there was. Our judgement from the visual examination of the outer part of the pipes and bottom plates is that it was very little corrosion, which is in agreement with the estimates of corrosion by copper in the clay or by mass-loss. Regarding the bottom of the interior of the pipes, these were difficult to get good photos of, but also here its our judgement that the corrosion was modest.
51	4.2	1 <sup>st</sup> sentence	Include Cu in list of most common elements	Done.

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52	4.2	levels of O	Again: it's difficult to obtain reliable data for O from SEM analysis	Agree, but we only use the EDS O-levels qualitatively or at most semi-quantitatively, discussing relative amounts of e.g. O and S. We don't aim at precise predictions or explanations of corrosion products, absolute quantities of oxides, stoichiometries etc.
53	4.2	"..SiO <sub>2</sub> phase Al <sub>2</sub> O <sub>3</sub> phase .."	These are rather <u>components</u> of bentonite	Agree, changed.
54	4.2	Ti-tubes (Fig. 2)	Are these the "1W tubes 7" in Fig. 2?	That's correct. Added explanation and references to Figure 2 at two places in the report.
55	4.3	Rates not representative of long-term corrosion	The obtained corrosion rates are also not representative of O <sub>2</sub> induced corrosion, but rather represent "mixed" rates.	Agree, added the following: "However, the integrated corrosion rates were calculated by dividing the mass-loss with the whole exposure period of 20 years, and since most of the corrosion of the coupons occurred during the initially oxygenated conditions, these rates are thus not representative for the long-term corrosion, which rate is expected to be controlled by the diffusion of sulfide through the bentonite clay.
56	4.3	Slow saturation mentioned for block 8	In my view, slow saturation is a key factor, the heat helps to slow down the water saturation.	Agree.
57	Figs. 43 & 44	T-dependence of corrosion depth	See comment above. Corrosion rate is also dependent on O <sub>2</sub> availability, which is higher for longer times in hot areas.	Agree, but it is already discussed in 4.2.
58	Table 22	Volume gap bentonite-Cu pipe	1.3 dm <sup>3</sup> seems very small. Please check. My estimate in TR-13-17 is about twice this value (the other volumes are similar).	It seems that a Cu-bentonite distance of 2 mm was used in TR-13-17, while the actual distance according to TR-20-11 was 1 mm. The outer diameter of the copper pipes were 108 mm, while the inner diameter of the hole in the bentonite rings were 110 mm. See 2.3.4 in TR-20-11.
59	4.4	Ref. Kober & Wersin	Should be Wersin & Kober	Changed.
60	4.4	Briggs et al. 2020; Martino et al. 2020, SKB 2019	Not in reference list	Added references.
61	4.4	"..copper components seems to be affected .."	Typo	Corrected.

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62	4.4	“ .. has been extensively studied over the years.”	Provide reference(s)	Added Chen J et al. 2011, 2014, Gordon et al. 2018, Martino et al. 2014, Sharma 1980.
63	5	“ ... within a depth where elements..”	Typo	Corrected.
64	5	Cu content in bentonite as measure for corrosion depth	Again: Cu in corrosion layer should be added to get the depth.	See reply to comment 30. Discussion rewritten.
65	5	“.. more extensive <u>oxic</u> corrosion of the pipes..”	Suggest to add “oxic”	Done.
66	App. A	General	It would be nice to have a sketch with location of samples.	Figure 2 shows position/location of coupons. Pipe samples were taken from blocks 21-23.
67	App. A	Orientation of pipes etc.	It may be useful to know orientation in space of Cu tubes and even for coupons. This may help in data interpretation & modelling. Is there some reference line parallel to tube axis? In this context what does 0°, 90° ... refer to in Fig. A-12 and A-14?	This type of information is presented in TR-20-11.
68	App. B	General	Interesting collection of images. There does not seem to be much description or analysis of most of these images in the main text. There, a rather small selection of these images is presented.	We have reduced the number of photos by omitting duplicates in Appendices B and C (micrographs with or without EDS data). We have also omitted micrographs showing the same features at different magnifications. Note that we use the SEM observations to collect pit data.
69	Table B-1	Measurement uncertainty	How precise is this estimate?	Manual measurement, note added in text.
70	App. C, C.4	General	How does this appendix relate to Appendix B?	I have added a comment in C.4 about how App B contains a comprehensive set of SEM images but App C is just representative areas for the whole sample with EDS data.

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71	App. C, C.4	General	Again: sketch with location of samples would be useful.	Coupon positions shown in Fig 2.
72	C.4	1 <sup>st</sup> sentence	Any idea to which depth samples were probed?	Described in section 2.1 (less than 1um)
73	C.4	Origin of S, Cl, Mg and Fe	Couldn't, the origin be, at least partly the clay?	Agree, and added.
74	C.4	“Smaller amounts of copper sulphide were indicated, with a tendency for more sulphur further from the copper surface, and more oxygen closer to the copper surface”.	Any more specific information from all these samples that could be mentioned here?	Longer discussion in the report itself, several references to Appendix C in section 3 and 4.
75	App. D	Pickling curves and data	For someone not familiar with method: would be good to explain how mass loss calculated from cuves which are still rising.	Description of method expanded.
76	App. D	Pickling curves	x-axis unit is missing, I guess it is minutes	Clarified in text.
77	App. G	General	A lot of data- does not seem to be fully ”digested” yet – note e.g. general remarks on EDS analyses.	Note that we use the micrographs to measure pits.
78	App. G	Sample preparation	Were all samples prepared in air atmosphere?	Yes.
79	Fig. G-7		Which of the 3 areas? Where exactly was measurement carried out? Backscatter image?	I added ref to fig 2 and 3 which show the areas analysed. These are SE images.
80	Fig. G-8		See comment above. A bit peculiar that light area has much less Cu than dark area.	It is the bentonite that coated a lot of the surface actually.

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81	Fig. G-9		It seems that inner points 8 & 9 has higher S content than outer points → is somewhat at odds with description in text and other analyses	Agree, the trend of higher S outwards can not be declared for the pipes as it could for the coupons. Added: "Many of the cross-sections were very low in S (<3 at-%) and only occasionally high levels (>10 at-%) were detected. Due to the low levels of S, the presence of bentonite, and significant levels of Ca (precipitation of CaSO <sub>4</sub> ) it was difficult to analyse the distribution of Cu-S phases in the corrosion products."
82	Fig. G-11		Same comment as #81	See reply to 81.
83	Fig. G-20		How explain high Si? Could sample have somehow been contaminated by the clay?	Yes it is likely the clay – the samples were not cleaned prior to analyses and during exposure and installation/removal they were in contact with the clay.
84	Fig. G-37		High Cl content. What about Na, below detection?	Na was not detected.