

Factual review of TR-20-14 - Fraser King

<i>Reviewer:</i> Fraser King, Integrity Consulting
<i>Type of review:</i> Factual review
<i>Review document:</i> The report “ <i>Corrosion of copper in bentonite after 20 years exposure in the field tests LOT S2 and LOT A3.</i> ”, 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.
Yes, just about meets the criterion. It is pretty obvious what the aims and scope of the report are from the title, although these are not specifically defined in the text. Perhaps they could be defined in the Abstract or Summary that should be added to the report.

- 2 Methodologies and models are verified and validated or proven.
Yes, meets the criterion. The experimental methods are defined in detail in Section 2, including a discussion of calibration and quality checks on the methods.

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- 3 Used methodologies and models are applicable and have been used within its possibilities and limits.
Yes, meets the criterion. The analytical methods that have been used are appropriate for the intended purposes.
- 4 It should be possible to evaluate the quality of cited references of importance for the conclusions reached.
Yes, meets the criterion. All but one of the cited references are published (one is in preparation).
- 5 Factual information shall be supported by relevant references.
Yes, meets the criterion. I did not find any statement that required an additional reference to support it.
- 6 The conclusions reached shall be supported by the analyses, other findings in the report and/or by supporting documents.
Yes, just about meets the criterion. As discussed below, I have some comments about the evidence for the existence of Cu₂S which is heavily relied upon to support the suggestion that the experiment represents a transitional phase from aerobic to anaerobic corrosion. But other than that, the analyses and conclusions are fully supported.
- 7 The conclusions should be clearly reported and in such way that the fulfilment of the stated objectives can be judged.
Yes, just about meets the criterion. As noted above, the aims and scope of the report are not specifically defined in the Introduction, although they are clear and addressed in the Conclusions.

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>

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1	General	Proposed mechanism based on aerobic/anaerobic transitional phase	<p>It is suggested that the observations can be explained on the basis that the 20-year end-point corresponds to a transition from aerobic to anaerobic conditions. Hence, Cu₂O formed under aerobic conditions is being transformed to Cu₂S by incoming sulfide.</p> <p>Unfortunately there is no clear indication of when initial O₂ was consumed. Indeed there is no clear statement in the report that it has been consumed, and I think such a statement (if correct) should be added.</p> <p>I agree that the <u>oxic</u> phase may be over, but I think conditions may still be <u>aerobic</u> due to the presence of Cu(II) (here I use the term “aerobic” to indicate relatively oxidising conditions). There is not a lot of discussion about the presence of Cu(II), but I think the [Cu] profiles in the bentonite provide very clear evidence. Copper concentration profiles in bentonite are of two general types (King et al. AECL-11831, 1997, Figure 2). Short, steep profiles with high interfacial [Cu] (as measured here) are indicative of Cu(II), while long, shallow profiles with low [Cu] are indicative of Cu(I). In some of our early work (AECL-10397, Corros. Sci. <u>33</u>, 1992, 1979-1995), we found evidence for a maximum interfacial [Cu] that was equal to the CEC of the buffer material on the assumption of Cu(II). I note from your figure 31/32 that an interfacial [Cu] of 1 wt.% is equivalent to about 50 % of a CEC of 80 meq/100g. In aerobic systems, the distribution of oxidised Cu is dominated by adsorbed Cu(II). Instead of using XRF to measure the Cu content of buffer, it is interesting to elute the Cu by suspending the clay sample first in DIW (to remove precipitated and pore-water Cu) and then in acid (to desorb the adsorbed Cu(II)). Generally, the latter fraction is much greater than the water-washed fraction.</p> <p>So, I agree that the 20-year mark might correspond to a transitional phase, but I would argue the system is still aerobic due to the presence of Cu(II).</p>	<p>Changed description of O₂ driven corrosion in 1.2, Cu(II) is now discussed.</p> <p>Added discussion of Cu(II), its probable presence in the clay as indicated by the steep profile, in section 4.2 Corrosion products. Also added reference to the suggested papers.</p> <p>Regarding the identification of Cu₂S with XRD it must be noted that both TEM and Cu/S ratios in EDS indicate Cu₂S or a stoichiometrically related phase.</p>
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			I am less convinced by the argument that you are at the aerobic/anaerobic transition (i.e., the transition to sulfide-dominated conditions) as I am not overwhelmed by the evidence for Cu ₂ S, as discussed below.	
2	General	Question of O ₂ transport control	<p>With regard to the discussion about the rate-determining process in association with Figures 43/44, my estimate for the activation energy is 30 kJ/mol for the pipe (based on Fig 43) and only 2.3 kJ/mol for the coupons (Figure 44). For diffusion under saturated conditions, I would expect an activation energy of 15 kJ/mol (which is related to the temperature dependence of the viscosity of water). Thus, the value for the pipe looks a bit too high and that for the coupons a bit too low, although the quality of the coupon data is not that good.</p> <p>I wouldn't rule out the possibility of O₂ transport control for the pipe, but there might well be some advective component (at least initially), and I am not sure what the temperature dependence would be for a combination of diffusive and advective transport.</p>	I think its difficult to compute activation energies from these data since we are not sure how long time it took for O ₂ to be consumed. Furthermore, the time of O ₂ corrosion might have been different in the two test parcels due to different saturation times. But I realised that the temperature intervals are not fully comparable and therefore changed the discussion and omitted the plot of the coupons.

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3	General	Evidence for Cu ₂ S	<p>One of the main conclusions from the report is that it marks the transition from oxic to anaerobic conditions, the latter evidenced by the presence of Cu₂S. However, I found the evidence for Cu₂S to be a bit thin. I am not an XRD expert, but I was not convinced by the data in Figure 9. The other definitive information is from the TEM, which is only discussed in Appendix C. Given the importance for the proposed mechanism of the identification of Cu₂S, I suggest some of the TEM discussion from App. C should be included in the main text. Again, I am not a TEM expert so how strong this evidence is for the presence of Cu₂S is unclear to me.</p> <p>As discussed below, I think we should be careful about using the EDS data as evidence for Cu₂S in any of its forms.</p>	<p>Agree that TEM data should not be considered 100% conclusive, just like XRD and EDS, it is semi-quantitative at best. The analysed volume can contain C and S that are in the vicinity rather than only particles of Cu₂S. Then there is uncertainty of which phase is there but probably a stoichiometry near Cu₂S. So some caution should be taken in the description of the results which I believe has been applied in the text.</p>

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4	General	Use of EDS data	I was surprised that, having stated in Section 2.2 that the EDS data should only be treated as qualitative, you then go on to repeatedly use it in a quantitative sense, even to the point of almost distinguishing digenite from djurleite from chalcocite (Section 3.5.1.4). Because of the quantitative manner in which you use the EDS data, I suggest re-phrasing the discussion in Section 2.2.	We use it only in a semi-quantitative way. For example, in 3.5.1.4 we say at the end “This indicates that the visible corrosion product, is a copper sulfide, with a Cu/S ratio of around 2, including the possibility of digenite (1.8), djurleite (1.9) and chalcocite (2.0).” We don’t believe that the EDS is accurate enough to allow quantification of the ratio to a degree can be used to distinguish between compounds with such similar ratios as 1.8 and 2.0. Rephrased in 2.2: “Due to the morphology and filtering effects of corrosion products the analysis results should be interpreted as semi-quantitative at best, and as such an accuracy of the measurement values cannot be strictly defined.”

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5	General	Microbial activity	<p>There is no discussion of the possibility of microbial activity and, in particular, of sulfate reduction. The only sulfide source that is mentioned is the ground water, where the concentration is low. I note that there are some type of microbial sensor in S2, layers 22 and 30.</p> <p>I suggest adding some discussion about the possibility of in situ microbial activity and sulfide production.</p>	<p>Now mention SRB in 1.2. Added comment on the “bacteria” treatment of coupons in S2 in 2.1 and discussed their (lack of) effect in 4.2.</p>
6	General	More background information about the LOT test	<p>I appreciate that the reader can find the information elsewhere, but I would have found some additional information about the experiment to have been very useful. For example: (a) what was the boundary condition between the outside of the buffer segments and the host rock? Was there an initial gap? Was a borehole liner used?, (b) what was the initial degree of saturation of the buffer rings?, (c) what about the time-dependent saturation behaviour?, (d) what about the pore-water chemistry, especially the [Cl]?</p>	<p>Added discussion in 1.2 concerning sources of air (gaps, pores, Ti-tubes) with reference to Sandén and Nilsson (2020). The effect of these O₂ sources on the corrosion process will be discussed in section 4.3. Added discussion on saturation periods in 4.3 with reference to Sandén and Nilsson (2020).</p> <p>Changed 1.3 “Temperature” to “Physicochemical development” and added discussion on densities, pressure and humidity from Sandén and Nilsson (2020). Added Table 2, which contains chemical data for the supply water.</p>

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7	Title page	Summary	A summary or abstract should be added.	Done.
8	Page 3, Section 1.2	“Exactly how long it took to reach oxygen free conditions in the LOT test parcels is not known.”	It is not definitively stated in the report that O ₂ -free conditions had been established in the experiment, and this would probably be a good location to clearly state that all of the initial O ₂ had been consumed some time during the 20-year period (if indeed that is the case).	Added discussion of sources of air/O ₂ and that the effect of this on the corrosion process will be discussed in section 4.2. Then follows a discussion of the time for O ₂ depletion found in various experiments.
9	Page 3	Cathodic reduction of H ₂ O	It’s not a major point, but I noted that you showed electron transfer to H ₂ O, rather than to HS ⁻ or H ₂ S.	Changed to H ₂ S since it might be more conventional, although I’m not convinced about the actual mechanism.
10	Figure 3		Do you think these temperature profiles are representative for the majority of the exposure period? How long did it take to reach steady state? As noted above, I think it would be useful to add something here about the saturation time. Also, it would be useful to either show the location of the corrosion coupons on the figure or to make note of their location in the figure caption.	Added info on the early temperature development in 1.3.
11	Page 6	Limitations of LOT, fourth bullet point	You should explain here the relevance of there being air inside the pipe, as it was not immediately clear to me.	Added: “The corrosion of these surfaces (upper part and inner surface) has thus not occurred under repository-like conditions.”

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12	Page 6	Limitations of LOT, last bullet point	Again, it might be useful to emphasize here that, although the time to consume the initial O ₂ is unknown, it is believed that the experiment became O ₂ -free during the course of the 20-year exposure period.	Added: "However, as discussed above, it is implied by other experiments that the time for O ₂ depletion in a setup like LOT is probably less than a year."
13	Table 2	Unexposed coupons	It's a bit confusing to have the same ID # for the reference coupons as for two of the A3 coupons!	Fully agree but that's the way they were identified at installation, by carving in a letter in the surface. There is no risk of confusion during analysis though, since the reference coupons had a different appearance (not corroded) and also different dimensions (see Appendix A).
14	Section 2.2	Preparation of coupons	You might say a little more about how the bentonite was removed from the coupon surfaces, as I know UWO found they had to go to great lengths.	As explained in 3.1.1 we used hand tools of plastic or wood to remove the bulk bentonite. Some bentonite was visibly sticking to the surface and was washed off using deionized water. Obviously, there are still microscopic deposits left according to XRD and EDS.

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15	Section 2.3	Samples selected from cross-sectioning	You might explain the rationale for selecting these two particular coupons for cross-sectioning.	Added: "Using 1 out of 4 coupons per test parcel for this purpose was a trade off between the value of information gained from examination of cross-sections, and the statistical significance of the gravimetric analysis. The particular coupons were chosen as to examine one coupon from each test parcel, and one coupon from each block position."
16	P 23	First paragraph	Here is an example of the use of EDS to positively identify chalcocite. If the EDS is qualitative, then I think you should be more cautious.	See reply to comment 4. Added: "From the EDS analysis, the sulfur rich particles and layers appear to have Cu:S ratios close to that of Cu ₂ S (chalcocite)." I changed "corresponding to" to "close to".
17	Table 5 and 6	Point of reference for pit depth measurements	You should note in the text or table caption what point of reference you used to estimate the pit depths (presumably the corroded surface rather than the original surface).	The corroded surface. Added to the text about the pit depth measurement method.

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18	Table 6	Effect of temperature	Is there an effect of temperature on the depth and number of pits? The average of the 5 deepest pits is greater for S2 than A3, and there are more of them on average. More pitting at the lower temperature?	The coupons in block 30 (OP and KL) had similar low temperatures ca 25degC, while the coupons in block 22 (IJ and MN) had higher temp 50-60 deg. Looking at the data in the table you refer to, it seems to be the other way around, although few data. No change made.
19	Figure 14	Effect of temperature	Apart from the reference coupons, could you plot the pit depths against temperature (on the x-axis) instead?	See reply to above comment. Since the data set is small, I suggest to make the suggested analysis for a larger data set combining data from several field tests.
20	P 26	Discussion below Table 7	You appear to be building a story here that relates the presence of S to the existence of pits. Is that what you mean to do?	The origin of pits is unclear, but if its related to corrosion it is most probably the O2 corrosion. Clarified in the discussion.
21	Figure 22	Visible unevenly distributed corrosion products	The build-up of corrosion products in the corners is not clearly visible from the photo.	I improved the figure by adding arrows, and added an additional closeup picture in black/white with enhanced contrast to further increase visibility.
22	Section 3.5.1.3	Table 9 and 10	Table 9 and 10 seem to be reversed in the text (3 locations)	Tables renumbered through the report.

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23	Section 3.5.1.3	Estimation of corrosion rate based on [Cu] profiles	When estimating the corrosion rate based only on the [Cu] profiles in the bentonite, are you not under-estimating the amount of oxidised copper because you are not including in the calculation the oxide/corrosion products on the coupon.	In principle yes, but the thickness of the adherent oxide layer was very thin (<1µm) as seen in the SEM cross-sections in Appendix F. Clarified in 3.6.1.3 and in 4.2.
24	Table 9-12	Place in Appendix	There is a lot of detail in these tables. Would they be better in an Appendix?	The XRF data is regarded as very useful for different kind of readers and will stay in the chapter. The amount of calculations in the tables was however reduced. Details of the calculations are given in a new Appendix.
25	3.5.1.4, Tables 13 and 14	Use of EDS data	Another example of using qualitative EDS data to speculate about the presence of different copper sulfide phases.	The SEM/EDX is semi-quantitative and I would say good enough to discriminate between 1:1 and 2:1 CuS-phases (the scattering in the analysis done was rather small). Hence the statement "One can probably conclude that the corrosion product visible to the eyes, is a copper sulfide, with a Cu/S ratio of around 2" should be ok.
26	Figure 27	The line scans are too feint to see in my printed version		The figure was improved.

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27	3.5.1.5	XRD analysis	I note that there is no evidence for Cu ₂ S from the XRD.	Yes that is correct, as it says in the text: "No chalcocite was visible in the sample".
28	Figures 30, 31, 32	Shape of [Cu] profiles	As noted under comment #1 above, to me these profiles are clear evidence of strongly adsorbed Cu(II).	We agree that this is clear evidence that the Cu-phase(s) has no or limited mobility, but for example chalcocite is not a Cu(II) phase and has no mobility either. Since we have not determined the oxidation state or phase of the Cu in the bentonite, we have only mentioned the possibility of Cu(II) and that it would agree with earlier findings. See reply to comment 1.
29	Figure 32	Y-axis scale needs to be fixed		This was corrected
30	Tables 15-19	Move to appendix?	Should these tables be moved to an appendix?	The XRF data is regarded as very useful for different kind of readers and will stay in the chapter. The amount of text showing calculations was reduced. An appendix with details of the calculations was added.

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31	Tables 20 and 21	Move to an appendix?		The XRF data is regarded as very useful for different kind of readers and will stay in the chapter. The amount of text showing calculations is reduced.
32	Figs 34 and 35	Legends too small to read	The legends are too small to read and it is unclear which trace belongs to which interval.	The figure was improved.
33	General	Introduction of new information in the Results section	I was always taught that it was bad form to introduce new information in the Discussion section. Should much of the discussion in Section 4.1 be moved to the Results section? Figure 40, 41, 42, and 45 also appear to be new data.	Agree, moved former 4.1 to the results section so that it now is 3.1 and changed the caption to "Visual appearance...at retrieval." Moved figures from discussion to the corresponding result sections.
34	P 60	Last paragraph, section 4.1	You suggest that the similarity of appearance of the coupons is consistent with the similarity in the weight loss. Why should there be a correlation between visual appearance and weight loss?	Agree, deleted the statement regarding the gravimetric analysis.
35	P 61/62	Consumption of O ₂ by "other chemical processes"	What is the evidence for the consumption of some of the initial O ₂ by "other chemical processes"? What about microbial O ₂ consumption?	Changed to "other chemical processes and/or microbial consumption".

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36	P 62	<p>“Although sulfur is observed in relatively minor amounts on the surfaces and in the corrosion products, the sulfidation of cuprite, and possibly also the sulfide induced corrosion of copper, has been in progress for several years.”</p>	<p>I am not sure that you have presented the evidence to be able to claim this so definitively.</p>	<p>The text following this statement explains what is meant: “On the cross-sectioned samples of coupons S2/N and A3/K, the EDS analysis showed a tendency for higher concentrations of S in the outer parts of the corrosion products. Several examples of this trend are found in Appendix C.” After this follows a couple of examples of findings. I think there is no other explanation than sulfidation of Cu₂O, and/or possibly some sulphide induced corrosion, stated as another possibility. See also reply to comment 1 concerning the verification of Cu₂S or stoichiometrically nearby Cu-S phases. (S is found also at spots where there is no Ca or Fe.)</p>

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37	P 65/66	“Although it was not possible to identify any crystalline Cu-S phase with XRD, quantification of Cu and S in the EDS spectra of selected small areas indicated a Cu/S ratio of 1.7 - 1.8. This can be compared to Cu/S ratios in common copper sulfides such as digenite (Cu _{1.8} S) djurleite (Cu _{1.9} S), and chalcocite (Cu ₂ S), as identified on the copper surfaces. “	Again, I would suggest that this is pretty speculative given the qualitative nature of the EDS and the absence of XRD evidence.	The cited text doesn't claim that the actual stoichiometry of the compound is 1.7, only that it is close to several copper sulphide phases, implying that any of these could match. See further reply to comment 4. The reason we didn't see Cu ₂ S in the XRD could be that the phase is amorphous.
38	P 67	“It is not known exactly how long it took before these initial gaps were closed, but reaching full saturation and swelling pressure of the bentonite clay took several years and for block 8 in LOT A3, near the hottest part of the copper pipe, the saturation was particularly slow, taking nearly 6 years to reach full saturation (Sandén and Nilsson 2020).”	I think this could usefully be moved to the Introduction or Experimental section.	The information is kept in the discussion but more general information on the development of pressures is added to the introduction, see replies to comment 6.

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39	P 72	“Observations of sulfur inside pits could mean that the sulfide is corroding the copper locally at spots where the surface is not protected by a Cu ₂ O film, a transient process that could proceed as long as the sulfidation process is not completed.”	This seems a bit speculative to me.	Agree and rephrased.
40	Figure A12		Could you indicate which end of the pipe sample was hotter? Is there a correlation between temperature and the appearance of precipitated calcium sulfate?	Perhaps we could study photos from different parts of the pipes and try to see if there seems to be a correlation but it has not been done.
41	Table B1	Reference to figure numbers	Some of the figures identified in the table show a single feature, whereas others show an entire cross section with many localised features.	Table refers to the deepest feature in a micrograph.
42	Appendix B		I know you want to demonstrate thoroughness and transparency, but I am wondering what the value is for including all of these SEMs?	I have removed any duplicate images between App B and C, and then SKB will reformat the sizes to fit more onto each page. The exception is the cross section images which are needed as they were used to estimate the corrosion depth.

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43	Figure C1		What are the peaks at 39°, 41.5°, 45.5°? The evidence for Cu ₂ S looks pretty thin.	There is no clear reason why we have got satellite peaks from this material, unfortunately we have not been able to find a reference for this phenomenon for XRD on Cu. Indications of Cu ₂ S comes also from EDS ratios, the reason for unclear XRD results could be that the phase is amorphous or nano crystalline.
44	App C	Tables of EDS data	Instead of showing the SEMs from App B again in App C, would it be possible to move the EDS tables from App C to App B?	See reply to comment 42.
45	App C5	Cross-references to Figures	For some reason, all of the cross references to figures repeat the figure number twice.	Fixed.
46	Table C1	Text below table C1	“reflection” instead of “reflex”?	Changed.
47	C.6	Summary	What do you mean by “natural movement in the clay?”	Reformulated.
48	App E	Significance of images in left-hand and right-hand columns	I assume the left-hand image is focussed on the pit bottom and the right-hand image is focussed on the surface in order to estimate the pit depth. If this is the case, perhaps it should be explained in the text.	Different light settings, I have removed one of the images for each pit to reduce the number of images where appropriate.

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49	App G	S content of crack-like features	<p>Is there any significance to the tendency for the S content to decrease with distance towards the tip of the crack-like features in Figures G23. G30, and G49?</p> <p>Having been critical about the overly quantitative use of the EDS data, here I am doing exactly the same!!</p>	<p>There is a general tendency for lower S towards the Cu surface. Although this is interesting, it is beyond the scope of this report. Presence of S in cracks is ongoing in other projects where exposure conditions are controlled. It may be useful to use the data in this report as reference in future SCC work however.</p>