Reviewer: Fraser King, Integrity Consulting

Type of review: Factual review

Review document: 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.

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Review plan: 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"

- 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.
- 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.

- 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).
- 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.
- 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.
- 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	Comment / Question	Corrections / Measure
		[Filled in by reviewer]	[Filled in by reviewer]	[Filled in by author]
[ID]	[Page,	[Content, e.g. text, figure, table,	[Specification of comment and/or question, including motivation. If needed, provide	[Brief description of how the
	headline,	etc. that the comment concerns.	advices, instructions and suggestion for improvements]	review comment will be
	section]	For example, text that is to be		handled]
		commented may be quoted.]		3742,

		T	T	1
1	General	Proposed mechanism based on aerobic/anaerobic transitional phase	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, Cu ₂ O formed under aerobic conditions is being transformed to Cu ₂ S by incoming sulfide. Unfortunately there is no clear indication of when initial O ₂ 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.	Changed description of O2 driven corrosion in 1.2, Cu(II) is now discussed. 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.
			I agree that the oxic phase may be over, but I think conditions may still be aerobic 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. 33, 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. 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).	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.

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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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	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. I wouldn't rule out the possibility of O ₂ 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.	I think its difficult to compute activation energies from these data since we are not sure how long time it took for O2 to be consumed. Furthermore, the time of O2 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 ploto of the coupons.

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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3	General	Evidence for Cu ₂ S	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 Cu ₂ S. However, I found the evidence for Cu ₂ S 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 Cu ₂ S, 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 Cu ₂ S is unclear to me. As discussed below, I think we should be careful about using the EDS data as evidence for Cu ₂ S in any of its forms.	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 come caution should be taken in the description of the results which I believe has been applied in the text.

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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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	We use it only in a semi-
			treated as qualitative, you then go on to repeatedly use it in a quantitative sense,	quantitative way. For example,
			even to the point of almost distinguishing digenite from djurleite from chalcocite	in 3.5.1.4 we say at the end
			(Section 3.5.1.4). Because of the quantitative manner in which you use the EDS	"This indicates that the visible
			data, I suggest re-phrasing the discussion in Section 2.2.	corrosion product, is a copper
			data, I suggest to pinusing the discussion in Section 2.2.	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 \circ
				measurement values cannot be
				strictly defined."

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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5	General	Microbial activity	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. I suggest adding some discussion about the possibility of in situ microbial activity	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.
			and sulfide production.	
6	General	More background information about the LOT test	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 ⁻]?	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). Changed 1.3 "Temperature" to "Physicochmical developmen" and added discussion on densities, pressure and humidity from Sandén and Nilsson (2020). Added Table 2, which contains chemical data for the supply water.

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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7	Title	Summary	A summary or abstract should be added.	Done.
	page			
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/O2 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 O2 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 H2S since it might be more conventional, although Im 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."

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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12	Page 6	Limitations of LOT, last bullet	Again, it might be useful to emphasize here that, although the time to consume the	Added: "However, as discussed
		point	initial O ₂ is unknown, it is believed that the experiment became O ₂ -free during the	above, it is implied by other
			course of the 20-year exposure period.	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	Fully agree but that's the way
			the A3 coupons!	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	Preparation of coupons	You might say a little more about how the bentonite was removed from the coupon	As explained in 3.1.1 we used
	2.2		surfaces, as I know UWO found they had to go to great lengths.	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 stil
				microscopic deposits left
				according to XRD and EDS.

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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15	Section	Samples selected from cross-	You might explain the rationale for selecting these two particular coupons for	Added: "Using 1 out of 4
	2.3	sectioning	cross-sectioning.	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	See reply to comment 4.
			is qualitative, then I think you should be more cautious.	Added: "From the EDS
				analysis, the sulfur rich
				particles and layers appear to
				have Cu:S ratios close to that of
				Cu ₂ S (chalchocite)." I changed
				"corresponding to" to "close
				to".
17	Table 5	Point of reference for pit	You should note in the text or table caption what point of reference you used to	The corroded surface. Added to
	and 6	depth measurements	estimate the pit depths (presumably the corroded surface rather than the original	the text about the pit depth $\frac{5}{2}$
			surface).	measurement method.

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No.	Section.	Concerning	Comment / Question	Corrections / Measure
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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 coupons in block 30 (OP
			the 5 deepest pits is greater for S2 than A3, and there are more of them on average.	and KL) had similar low
			More pitting at the lower temperature?	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	See reply to above comment.
			temperature (on the x-axis) instead?	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	The origin of pits is unclear,
			existence of pits. Is that what you mean to do?	but if its related to corrosion it
				is most probably the O2
				corrosion. Clarified in the
				discussion.
21	Figure 22	Visible unevenly distributed	The build-up of corrosion products in the corners is not clearly visible from the	I improved the figure by adding
		corrosion products	photo.	arrows, and added an additional
				closeup picture in black/white
				with enhanced contrast to
				further increase visibility.
22	Section	Table 9 and 10	Table 9 and 10 seem to be reversed in the text (3 locations)	Tables renumbered through the
	3.5.1.3			report.

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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23	Section	Estimation of corrosion rate	When estimating the corrosion rate based only on the [Cu] profiles in the	In principle yes, but the
	3.5.1.3	based on [Cu] profiles	bentonite, are you not under-estimating the amount of oxidised copper because you	thickness of the adherent oxide
			are not including in the calculation the oxide/corrosion products on the coupon.	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-	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
	12			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,	Use of EDS data	Another example of using qualitative EDS data to speculate about the presence of	The SEM/EDX is semi-
	Tables		different copper sulfide phases.	quantitative and I would say
	13 and			good enough to discriminate
	14			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 5
				visible to the eyes, is a copper \$\sqrt{5}\$
				sulfide, with a Cu/S ratio of
				around 2" should be ok.
26	Figure 27	The line scans are too feint to		The figure was improved.
		see in my printed version		Jokun

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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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	Shape of [Cu] profiles	As noted under comment #1 above, to me these profiles are clear evidence of	We agree that this is clear
	30, 31,		strongly adsorbed Cu(II).	evidence that the Cu-phase(s)
	32			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
				S
30	Tables	Move to appendix?	Should these tables be moved to an appendix?	The XRF data is regarded as
	15-19			very useful for different kind of
				readers and will stay in the
				chapter. The amount of text
				showing calculations was 5
				reduced. An appendix with
				details of the calculations was
				added.

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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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 appearanceat 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".

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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No. 36	P 62			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.) 0.1 Wisson (2,742,681 Quantum visual response of the control of

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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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 Cu2S 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)."		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.

No.	Section.	Concerning	Comment / Question	Corrections / Measure
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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 se 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	Appendi x 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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No.	Section.	Concerning	Comment / Question	Corrections / Measure
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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 Cu2S 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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No.	Section.	Concerning	Comment / Question	Corrections / Measure
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49	App G	S content of crack-like features	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? Having been critical about the overly quantitative use of the EDS data, here I am doing exactly the same!!	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.
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