



## Minutes from the meeting with SKB regarding QA in SKB's Copper Corrosion Experiments

### Place and time:

SKB Offices, Stockholm  
29 August 2014

### Participants:

Christina Lilja (SKB)  
Johannes Johansson (SKB)  
Allan Hedin (SKB)  
Bo Strömberg (SSM)  
Clara Anghel (SSM)  
Tim Hicks (Galson Sciences Ltd)

### 1. Agenda

08:30 – 08:45 Agenda and Introduction  
08:45 – 09:45 Follow-up of the QA review from 2010  
09:45 – 12:00 QA review – 2014  
12:00 – 13:00 Lunch  
13:00 – 16:00 QA review – 2014

### 2. Introduction

SKB welcomed the Swedish Radiation Safety Authority (SSM) and its consultant to its offices, after which SSM introduced the rationale for the meeting (see presentation in Appendix 1).

SSM has completed the initial phase of its review of the SR-Site safety assessment produced by SKB that subsequently entered its main phase, with assignments targeted on prioritised tasks and issues, and aimed at supporting SSM's compliance judgments. As part of the main review, SSM has tasked Galson Sciences Limited to undertake an assessment of SKB's documentation and quality assurance (QA) of selected copper corrosion experiments:

- Miniature Canister (MiniCan) Experiment 3, focusing on the corrosion analysis of the copper coupons and canister.
- The Long Term Test of Buffer Material (LOT project) A0 parcel tests, focusing on the analysis of the copper coupons.
- The LOT project A2 parcel tests, focusing on the analysis of the copper tube.



- Copper corrosion tests in oxygen-free pure water.
- The atmospheric and aqueous copper corrosion tests undertaken at the Äspö Hard Rock Laboratory (HRL).

The overall aim of the assignment was to check the existence and application of appropriate procedures during all stages of these corrosion experiments, including analysis of the experiments and use of results. As part of the QA assessment, this meeting was scheduled with staff involved in SKB's copper corrosion experiments and SSM staff and its consultant involved in the QA review.

The aim of the QA meeting was to review QA procedures and evidence of their application in the selected copper corrosion experiments. Also, the meeting aimed to understand the links between the assumptions about corrosion rates made in the SR-Site licence application and the experiments that support those assumptions. SKB gave some general comments on the status of the copper corrosion experiments and gave a presentation on the use of the corrosion experiment data in the licence application (see presentations in Appendix 2).

In order to facilitate this review process, prior to the meeting, SKB had been provided with a list of QA issues relating to the design, running, analysis and use of results of the copper corrosion experiments (SSM 2011-2306-22). SKB developed responses to these issues for presentation at the meeting. The list of issues and SKB's responses provided the focus of discussions at the meeting. SKB's responses are provided in Appendix 3 (including revisions made subsequent to the meeting in response to clarification and discussion of the issues).

The meeting also looked back at the findings of a previous QA review of SKB's copper corrosion experiments reported in 2010 (Baldwin and Hicks, 2010). The previous review focused on copper corrosion experiments associated with the LOT project and the MiniCan experiment, and highlighted a number of important issues relating to QA of the experiments. The present QA review is re-examining these issues and investigating the measures/changes implemented by SKB to address them. The meeting began with a discussion of these issues.

Subsequent to the meeting, the QA review assignment included an assessment of published documents relating to the copper corrosion experiments. The assessment will draw on the findings of the QA review meeting. The final product of this QA review will be a report published in the SSM technical note series.

### 3. Follow-up of Viewpoints Expressed in SSM's 2010 QA Review

The initial discussion of the previous QA review of SKB's copper corrosion experiments focused on the status of experiments that include copper corrosion tests, as listed in Section 3 of Baldwin and Hicks (2010). SKB provided a brief update of the copper corrosion experiments (no prepared presentation), and noted that reference should be made to its 2013 "RD&D Programme" (SKB, 2013) for a more detailed description of the status of the experiments. The discussion of the experiments is summarised in Table 1. Publication details have been extracted from SKB (2013, §24.2) subsequent to the meeting, but references to the experiments published after SKB (2013) are not included in Table 1.





**Table 1:** Update of the status of SKB's copper corrosion experiments that were ongoing or planned at the time of the QA review reported by Baldwin and Hicks (2010, §3.3).

Experiment	Status
<b>Experiments on copper corrosion in a sulphide/water environment</b>	
Tests to understand the rate determining step(s) in the formation of sulphide films and their properties for experiments involving copper in sulphide solutions and different concentrations of chloride. The analysis involves electrochemical impedance and spectroscopic methods.	This work is being carried out by researchers in the Department of Chemistry of the University of Western Ontario, Canada. Published results up to SKB (2013) include Chen <i>et al.</i> (2012).
Tests to repeat the experiments of Taniguchi and Kawasaki (2008), who observed stress corrosion cracking (SCC) of copper in sulphide solutions. Copper has been subjected to slow strain rate testing (SSRT) and constant strain rate testing, and a range of techniques has been used to analyse the test results.	This work was carried out by researchers in the Department of Chemical Engineering and Applied Chemistry of the University of Toronto in Canada. The study has been completed and the results have been published by Bhaskaran <i>et al.</i> (2013).
<b>Experiments on copper corrosion in a bentonite environment</b>	
Investigation into the potential for sulphate-reducing bacteria (SRB) growth and biofilm formation on copper in compacted and saturated bentonite. Copper rods were embedded in compacted bentonite, which was saturated with groundwater from Äspö. At the end of the experiment, the metal rod surfaces were analysed for bacterial coatings.	The research was carried out by Microbial Analytics Sweden AB. The project has been completed and the results published (Persson <i>et al.</i> , 2011). However, further experiments are being undertaken to determine the sensitivity of SRB growth and sulphide production to bentonite compaction.
Electrochemical studies of copper corrosion in a compacted bentonite environment. The research has involved the analysis of copper electrodes that were exposed in the LOT A2 test parcel at the Äspö HRL. Electrical resistance and electrical impedance spectroscopy measurement techniques were used to analyse corrosion rates over time.  Also, copper profiles in the clay next to the copper tube (in the LOT A2 test) were analysed to estimate corrosion rates.	The corrosion analysis work for the LOT A2 test parcel was carried out by researchers at the Division of Surface and Corrosion Science at KTH, Stockholm, and the Slovenian National Building and Civil Engineering Institute, Ljubljana, Slovenia. The work has been completed and published (Rosborg <i>et al.</i> , 2012).  The compilation of data from the analysis of the copper tube was undertaken by Gruner Ltd, Switzerland (Wersin, 2013)
<b>Experiments in a repository-like environment</b>	
The MiniCan experiment is being carried out to study how corrosion of the cast iron insert would develop in the case of a defect in the copper canister. Copper coupons have been included in the tests. A canister from the MiniCan project was retrieved and analysed in 2011 (Experiment 3).	SKB is now leading the MiniCan project with the support of an SKB steering group; the experiments had previously been managed by AMEC (formerly Serco Technical Services). Results of the analysis of Experiment 3 have been reported by Smart <i>et al.</i> (2012a;b; 2013) and Hallbeck <i>et al.</i> (2011).
Tests to study SCC of copper in groundwater containing ammonium. This work uses SSRT and spectroscopy and electrochemical measurements to identify SCC.	This study was carried out by VTT, Finland, in co-operation with Posiva (Kinnunen and Varis, 2011).



Experiment	Status
<b>Experiments in oxygen-free water</b>	
Work to develop a kinetic model for the copper/electrolyte interface for copper in deoxygenated water using potential measurements and electrical impedance spectroscopy.	The experiments are being carried out at the University of Chemical Technology and Metallurgy in Sofia, Bulgaria. The experiments are ongoing, but results have been reported by Bojinov <i>et al.</i> (2010) and Betova <i>et al.</i> (2013a;b).
Research to look for unknown compounds of copper with oxygen and hydrogen. Spectroscopic studies and X-ray diffraction have been used to study CuH produced from a copper solution. The thermodynamic stability of different compounds and the synthesis of CuOH are also being investigated.	The experiments are ongoing. Results to date have been published by Korzhavyi and Johansson (2010), Korzhavyi <i>et al.</i> (2011; 2012) and Soroka <i>et al.</i> (2013).
Experiments to test hypotheses on hydrogen production from copper in oxygen-free water. The experiments involve placing copper strips in glass test tubes and analysing the gases generated.	The experiments are being undertaken by Microbial Analytics Sweden AB. The study methodology has been published by Bengtsson <i>et al.</i> (2013).
Copper corrosion experiments under anoxic conditions, similar to the long-term tests reported by Hultquist <i>et al.</i> (2009) that indicated the presence of a copper corrosion product that contains hydrogen. The experiment involves placing copper foils in anoxic deionized water in Erlenmeyer conical glass flasks in a reducing environment. The analysis included a reference test to investigate the effects of exposure to air atmosphere.	The work was carried out by the VTT Technical Research Centre of Finland. Results from the experiments have been reported by Ollila (2013).
<b>Copper corrosion experiments planned in 2010</b>	
Experiments on copper corrosion in ultra-pure, oxygen-free water using ultra-high vacuum equipment. These experiments aimed to either confirm or refute the hypotheses put forward by other researchers (e.g., Szakálos <i>et al.</i> , 2007) at KTH that copper corrodes at a non-negligible rate with pure water as the oxidant.	Researchers at Uppsala University's Ångström Laboratory in the Department of Chemistry were commissioned by SKB to undertake these experiments. The experiments are ongoing, but initial results have been reported by Boman <i>et al.</i> , (2013; 2014).
Analysis of copper wires that had been placed in water in test tubes as part of an experiment on copper corrosion in oxygen-free water initiated some 20 years ago. The test tubes had been sealed with palladium membranes and had been stored by SP, the Technical Research Institute of Sweden, ever since.	The results of the analysis have been reported by Möller (2012).





SKB noted that the copper corrosion reference group that was planned for at the time of the previous QA review (Baldwin and Hicks, 2010, §3.1) was established to guide the corrosion experiments and identify future research requirements. In addition to the experiments listed in Table 1, SKB noted that experiments had been set up to assess the effects of gamma radiation on copper corrosion. The experiments involve irradiating copper in pure water and analysing the results using spectroscopic methods. The experiments and their analysis are being undertaken as a PhD study at KTH (SKB, 2013, §24.2). SSM asked if any copper corrosion analysis was being undertaken as part of the Prototype Repository test at the Äspö HRL. SKB stated that there are some data from the test: electrochemical methods have been used to make real-time measurements of corrosion using copper electrodes embedded in the bentonite buffer (Rosborg, 2013a;b; SKB, 2013, §24.2).

Subsequent discussion focused on the copper corrosion experiments being undertaken at Uppsala University, the LOT copper corrosion tests and the MiniCan project, and the issues raised in the previous QA review (Baldwin and Hicks, 2010, §5).

SKB noted that the experiments on copper corrosion in ultra-pure, oxygen-free water at Uppsala University have been run as an academic research project using techniques similar to those used by researchers at KTH. However, the design of the experiments had been discussed with a dedicated reference group involving SKB and external groups. With regard to QA of the work, SKB stated that the experiments are not subject to an industry standard QA system and that this is the general case for university-run projects. SKB was asked if uncertainties about the QA of the work influenced how the results of the work are used. SKB replied that the research findings would be used as one strand of evidence in support of its understanding of copper corrosion processes; a lot of research from published literature is used in this way. SSM commented that understanding the details of the experiments may be important and so it is important to have control over the experiments. SKB agreed, but considers that it is not always possible for them to have total control over the experiments performed at a university.

SSM asked if SKB is provided with all of the data from the experiments. SKB replied that it does not have access to the primary data, but the data could be obtained if necessary. SKB expects that the results of the research will be published in peer-reviewed scientific journals and in SKB reports; the SKB reports could include raw data from the experiments. SKB considers that this complementary approach ensures an appropriate level of peer review as well as comprehensive reporting of the details of the experiments. This is consistent with the views expressed in the previous QA review, which stated that it is important for SKB to produce regular comprehensive technical reports of its experiments as well as publications in specialised journals (Baldwin and Hicks, 2010, §5.5). SKB also noted that it is now possible to publish "e-data", such as large databases, which would provide a method of disseminating raw data and the results of detailed analysis from the corrosion experiments.

SKB highlighted that Microbial Analytics (Micans) in Sweden had developed an alternative way of conducting the investigations made at Uppsala University by performing experiments in another type of set-up. The work is on-going.

SKB reported that there has been no activity on the LOT test since the publication of the results of the LOT A2 test analysis. The LOT test is not currently running as an SKB project. A project would be set up when SKB decides to recover the next test parcel for analysis, although there is no firm schedule or plan for this work. Also, the LOT test is primarily a buffer project and those involved in buffer research would make decisions about the next phase of the test.





Johannes Johansson of SKB is now leading the MiniCan project. There had been plans to recover Experiment 4 and Experiment 5 this year, but the budget to do so is not available. The work is now planned for next year pending budget decisions. The work would include analysis of the copper corrosion coupons used in the experiments. SKB mentioned that there would be less value in analysing the MiniCan copper tubes, because the tubes were not examined in detail before setting up the experiments; there would be few reliable data on initial conditions with which to make comparisons. Decisions on when to recover Experiments 1 and 2 have yet to be made.

The previous QA review raised a concern about the lack of transparency in data publication for the MiniCan experiment (Baldwin and Hicks, 2010, §5.3). It had been found that some corrosion monitoring data had not been reported in the publicly available SKB technical report. These data suggested corrosion rates several orders of magnitude higher than expected values. Although the high corrosion rates suggest problems with the measurement technique, the reasons for excluding the data were not documented and published. At the meeting, SKB stated that all corrosion rate data from the MiniCan Experiment 3 analysis have now been published (Smart *et al.*, 2012a); the report includes data that imply high corrosion rates, with explanations as to why the data are considered to be artefacts of the experiment and measurement methods. In some cases the derived corrosion rates are so high that they imply complete corrosion of the electrodes, but visual inspection of the electrodes found little corrosion. SKB commented that, for all experiments, checks are now made that all data are included in discussion and analysis of the results.

Baldwin and Hicks (2010, §5.4) also observed that reports from the MiniCan and LOT experiments provided little information on the sources or quantification of data uncertainty, or the level of confidence that can be assumed in the results. In particular, understanding when conditions are oxidic and when they are anoxic is of key importance in real-time copper corrosion tests. The QA review found that it was not clear how well redox conditions are understood in the vicinity of the copper corrosion tests in the MiniCan and LOT experiments. At the meeting, SKB stated that little more work had been done on real-time corrosion tests beyond what had been published by (Rosborg *et al.*, 2012) for the LOT test and Rosborg (2013a;b) for the Prototype Repository experiment. SKB commented that all corrosion products were observed to be oxidic, which indicates that the corrosion occurred under aerobic conditions.

#### 4. Use of Corrosion Experiment Data in the SR-Site Licence Application

SKB gave a presentation on how data from the copper corrosion experiments were used in the SR-Site licence application and in support of SKB's response to SSM's requests for supplementary information as part of the SR-Site review (see Appendix 2). All documents are listed in a database, which includes information about where the documents are cited in the licence application and in responses to SSM's requests for supplementary information. SKB's presentation focused on entries in the database relating to copper corrosion that had been included since the publication of SR-Site as well as in response to SSM's review (Table 1 in Appendix 2).

SKB summarised how different copper corrosion processes were treated in SR-Site and discussed in responses to SSM's review. SKB also described how results of the MiniCan and LOT copper corrosion tests, the corrosion tests in oxygen-free pure water at Uppsala University, and the corrosion tests under atmospheric and aqueous conditions at Äspö had been used.





The issue of corrosion in pure water was discussed in the context of a status report SKB prepared for SSM in 2013 and compared with the results presented in SSM report (Macdonald and Sharifi-Asl, 2011) on the subject. SSM mentioned that the recent thermodynamic data (Macdonald and Sharifi-Asl, 2011) improved knowledge regarding corrosion of copper in oxygen-free water and brought more light to the controversy by explaining the conditions for hydrogen gas to occur. Documenting this issue clearly is also important from a QA perspective. SSM asked SKB to take into consideration the latest findings and include them in the safety assessment. SKB noted this and will take them into account in future reporting.

In discussion of the analysis of the copper tube from the LOT A2 test parcel, SSM commented that, from a traceability perspective, it is important to show that corrosion rates from observations are consistent with the modelling. However, this is difficult if it is not known how long oxic conditions persist. SKB noted that work had been undertaken to estimate corrosion rates over time through analysis of copper electrodes from the LOT A2 test parcel, but the results of this work had not been used directly in its responses to SSM's review.

SKB reported that certain experiments at Äspö to investigate corrosion in chloride- and sulphide-rich groundwater had been terminated after 2.5 years because the groundwater flow rate from the tunnel wall was too low (Taxén, 2009). The system actually dried out and oxygen could have been affecting corrosion. SKB mentioned that in the atmospheric corrosion experiments the heater didn't work for periods of weeks, and this has been noted in the publication of the results of the experiments (Taxén 2004).

In conclusions to the presentation, SKB pointed out that the MiniCan and LOT tests at Äspö had been designed with a focus on understanding processes other than copper corrosion (i.e. corrosion of the cast iron insert and behaviour of the bentonite buffer). SKB also confirmed that in the SR-Site safety assessment, corrosion depths had been calculated based on mass balance or mass transport limitations on specific corrodants at the canister surface, rather than derived from measured corrosion rates. SKB noted that no results from the experiments obviously contradict the treatment of copper corrosion in the safety assessment.

SSM asked why more controlled experiments had not been set up that were dedicated to understanding copper corrosion processes. To include copper corrosion coupons in experiments on other processes introduces too many process couplings and uncertainties in the analysis. SKB replied that corrosion experiments would not be undertaken as part of other experiments in the future. Current practice is to design carefully controlled corrosion experiments, such as the experiments on stress corrosion cracking of copper.

SSM commented that a key issue regarding QA in corrosion experiments is the demonstration that the geochemical and hydrological environment intended for the experiment has been achieved. SKB replied that it is not straightforward to ensure that this is done when the experiments are carried out by different contractors and universities, sometimes in different countries. SSM asked if SKB has control over who does experiments at universities. SKB replied that the contract would be with a professor in the research department. SKB would have only a small influence over who does the work, especially if it is a PhD project. SKB would not have a great deal of influence on how the project is conducted, but could provide advice and suggestions for the research.

SSM suggested that if a researcher were to show results that contradicted SKB's understanding of corrosion processes, then SKB would need to set up its own research study to address the issue. SKB stated that the researcher may be required to undertake



more work if it was deemed necessary to clarify understanding of the process. Further, it may be judged necessary to repeat the experiments, which may take many years. SKB also commented that it is valuable to have university researchers contributing new ideas to the analysis and modelling of corrosion processes.

SSM asked if SKB considers that it has identified all copper corrosion processes. SKB replied that this is a difficult question to answer, which is why SKB undertakes 'what-if' calculations in the treatment of copper corrosion. Also, SKB's RD&D Programme does try to evaluate the importance of different processes and understand the state of knowledge of the processes, which helps to prioritise research. SKB concluded that it is undertaking the research it considers necessary to address the current understanding of copper corrosion issues.

### 5. Discussion of QA Review Issues

The QA review of copper corrosion experiments and analysis is focusing on the following experiments:

- MiniCan Experiment 3 (analysis of copper coupons and canister).
- The LOT project A0 parcel tests (analysis of copper coupons).
- The LOT project A2 parcel tests (analysis of copper tube).
- Copper corrosion tests in oxygen-free pure water.
- The atmospheric and aqueous copper corrosion tests.

SKB had been provided with this list of issues prior to the meeting (SSM 2011-2306-22). The list of issues and SKB's responses for each of the above-noted copper corrosion experiments are provided in Appendix 3. The responses include revisions made subsequent to the meeting in response to clarification and discussion of the issues.

SSM noted that SKB's responses to the list of issues for each copper corrosion experiment will inform the review of the experiment documentation in the next stage of the QA review task.

The meeting notes are approved by:

Clara Anghel (SSM)

Christina Lilja (SKB)





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## Appendix 1 – SSM's Presentation at the Copper Corrosion QA Review Meeting



**Strål  
säkerhets  
myndigheten**  
Swedish Radiation Safety Authority

### **QA in SKB's Corrosion Experiments Meeting with SKB**

*Tim Hicks (Galson Sciences Ltd), Bo Strömberg(SSM), Clara Anghel (SSM)*

2014-08-29



### **Agenda**

08:30 – 08:45	Agenda & Introduction
08:45 – 09:45	Follow up of the QA review from 2010
09:45 – 12:00	QA review – 2014
12:00 – 13:00	Lunch
13:00 – 16:00	QA review - 2014

2014-08-29



## Background

- As part of the SR-Site main review, on behalf of SSM, Galson Sciences Limited undertakes an assessment of SKB's documentation and quality assurance (QA) of selected copper corrosion experiments.
- The overall aim of the assignment is to check the existence and application of appropriate procedures during all stages of the corrosion experiments, including analysis of the experiments and use of results.

2014-08-29

## Follow up of the QA review from 2010 SSM 2010:17

- 5.1 Quality Assurance
- 5.2 Design of Experiments
  - Update on the performed corrosion test that were planned or on-going in 2010 (see section 3.3, p. 6-9)
  - What is the schedule for the remaining tests (for ex. MiniCan Experiment 4 and test S2 from the LOT experiment?).
  - Are there some plans for the phase out of these long term experiments as well as post test examination plans?
- 5.3 Data Reporting Issues for the MiniCan Experiment
  - Have SKB done some analysis of the high corrosion rate results?
- 5.4 Analysis of Uncertainties
- 5.5 Publication and Use of Results
  - Yearly reports from the Äspö lab. (LOT tests?)
  - IPR-reports (last one from 2011) – good, needed and requested yearly reports

2014-08-29



## QA 2014: issues to be discussed

- MiniCan Experiment 3, focusing on the corrosion analysis of the copper coupons and canister.
- LOT project, the A0 parcel tests, focusing on the analysis of the copper coupons.
- The LOT A2 test parcel tests, focusing on the analysis of the copper tube.
- The copper corrosion tests in oxygen-free pure water.
- Atmospheric and aqueous copper corrosion tests at Äspö HRL.

2014-08-29





## Appendix 2 – SKB's Presentations at the Copper Corrosion QA Review Meeting



### Review meeting on corrosion experiments - some general comments

2014-08-29

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#### New organisation of earlier projects

- Minican
  - new project leader: Johannes Johansson, SKB
  - steering group at SKB
  - reference group – internal SKB group meeting regularly ("sulphide forum")
- LOT
  - currently not a "project" within SKB (just under administration)
  - a new project will be set up when further dismantling is to be done





## Participation at review meeting

- Broad range of projects to be covered
- Several contractors have been working
  - SKB will answer the question list
  - not possible to invite all organisations without more details for the meeting
  - if needed, the contractors can be contacted for more specific questions



## The experiment at Uppsala U.

- The experiment on copper corrosion in anoxic water is financed by SKB.
- The task given to the contractor (Uppsala University) is to investigate claims by a research group at KTH of evidence for copper corrosion in anoxic water
- The group at Uppsala university was given freedom to investigate the fundamental question, using techniques that are similar to those developed by the group at KTH
  - The design of the experiments at Uppsala have been discussed continuously with SKB, and, until the autumn of 2013, in a dedicated reference group.
- The work is carried out as a research project in academia, to result ultimately in publications in the peer reviewed scientific literature. It is hence not controlled by QA plans as other SKB specific projects







## Context for corrosion experiments in licence application

2014-08-29

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### Supporting documents

- References in safety assessment SR-Site (top documents) and license application
  - compiled in database with notation of where they are used
- Search for the list of reports from SSM + a few related reports
  - listed in Table 1 (next slide)
- Use of the results – Tables 2-5
  - type of results
  - related to which process in corrosion assessment – Fig 6-1 in TR-10-66 used for comparison





Table 1: Reports, SSM list + [ ] added by SKB

Project	Report	Reference in
Minican	TR-12-09 (Analysis of Exp 3)	SKBdoc 1398013, Feb 2014
	P-12-13 (progress report 4)	SKBdoc 1398013, Feb 2014
	R-13-35 (metallographic)	SKBdoc 1398013, Feb 2014
	P-12-01 (microbial analysis)	-
LOT	TR-09-31 (A0 test parcel)	SKBdoc 1415886, Dec 2013
	TR-13-17 (compilation A2)	SKBdoc 1416862, Dec 2013
	[R-13-15 (electrodes from A2)]	-
	WM'02 Tucson (LOT Pilot)	-
Uppsala U.	R-13-31 (exp. design)	SKBdoc 1418966, Dec 2013
"Äspö exp."	WM'02 Tucson (exp. design atm+aq)	-
	[MRS 2004, atmospheric)	TR-10-46, TR-10-67
	[unpublished report from Kimab]	-



## The "SKBdoc" documents in Table 1

- **SKBdoc 1398013, ver 3.0:** Svar till SSM på begäran om komplettering avseende degraderingsprocesser för kapseln. Svensk Kärnbränslehantering AB.
- **SKBdoc 1415886, ver 1.0:** 4.2 Analys av i vilken mån en långsam återmättnad kan förvärta försämringar av buffertens materialegenskaper pga kemiska och strukturella omvandlingar i förhållande till ett fall med snabb återmättnad. Tillgängliga observationer och pågående studier. Clay Technology, November 2013.  
(Appendix 9 i SKBdoc 1385067, ver 3.0 Svar till SSM på begäran om komplettering rörande lång återmättnadsfas. Svensk Kärnbränslehantering AB.)
- **SKBdoc 1416862, ver 1.0:** Interaktion mellan kopparkorrosionsprodukter och bentonit. Svensk Kärnbränslehantering AB.
- **SKBdoc 1418966, ver 1.0:** Lägesrapport om kopparkorrosion i syrgasfritt vatten december 2013. Svensk Kärnbränslehantering AB.







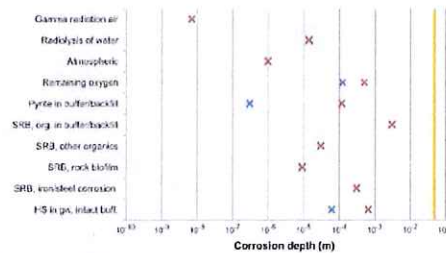
## Added reports

- Rosborg B, Kranj A, Kuhar V, Legat A, The corrosion rate of copper in a bentonite test package measured with electric resistance sensors, SKB R-13-15, Svensk Kärnbränslehantering AB.  
[Published results for these experiments were asked for in SSM 2010:17.]
- Taxén C, 2004. Atmospheric Corrosion of Copper 450 Metres Underground. Results From Three Years Exposure in the Äspö HRL. Materials Research Society Symposium Proceedings Volume 807, p 423-428
- Taxén C, Exposure of Copper in Äspö Groundwaters, Swerea Kimab, Kimab ref 708048 (also as SKBdoc 1455253).



## Corrosion assessment

- In SR-Site: corrosion depth is given for different processes, e.g. in Fig 6-1 in TR-10-66



- Corrosion by oxygen is handled in 2 processes
  - atmospheric, indoor air: estimated to < 1 µm based on Äspö exp. and comparisons to general atmospheric corrosion rates in Sweden
  - remaining oxygen in bentonite pores:
    - massbalance: 500 µm at the most
    - localised corrosion: allowance of ± 50 µm (included in the 500 µm above)





## Corrosion assessment (cont.)

- Corrosion by sulphide from ground water
  - handled by mass transport limitations, using data from Hydro DFN models and measured data for sulphide in groundwater
- Corrosion in ground waters with high chloride concentration
  - discussed in TR-10-46, further elaborated on in additional material to SSM in June 2013 (SKBdoc 1398013, with details in SKBdoc 1398014)
  - used as safety function indicator criteria, i.e. further analysis needed if  $\text{pH} < 4$  or  $[\text{Cl}^-] > 2\text{M}$
- Corrosion in pure water (Status report to SSM, Dec. 2013, SKBdoc 1418966)
  - not valid to use analysis with equilibrium pressure from experiments
  - still possible to do what-if calculations, using initial measured hydrogen gas "production" rates



### Table 2: Minican

	Type of result	Corrosion process	Used as ref for
TR-12-09 (Analysis of Exp 3)	Cast iron coupon completely corroded, corrosion rate $> 500 \mu\text{m/y}$ ; uniform corrosion of copper, rate $0.15 \mu\text{m/y}$ from weight loss measurement, results from electrochemical methods unreliable due to deposition of iron corrosion products, no indication of localised corrosion	Oxic corrosion initially, reducing with sulphide after some months	Observations: no cracks or sign of localised corrosion in U-bend specimens, no crack growth in precracked WOL specimens (though not correctly loaded)
P-12-13 (progress report 4)	Statements on water chemistry, microbial activity, potentials, measured corrosion rates, no expansion of the insert	Oxic corrosion initially, reducing with sulphide after some months	Reducing conditions after some months





Table 2: Minican (cont.)

	Type of result	Corrosion process	Used as ref for
R-13-35 (metallo-graphic)	No evidence for localised corrosion of any of the copper MiniCan components or stress corrosion test specimens examined, no increase in corrosion susceptibility in the EBW areas, although some porosity in the weld material was noted	Oxic corrosion initially, reducing with sulphide after some months	Observations: no cracks or sign of localised corrosion in U-bend specimens, no crack growth in precracked WOL specimens (though not correctly loaded)
P-12-01 (microbial analysis)	A succession in microbial population from a complex mix to mostly SRB, type of microbes in biofilms on steel cage and copper canister	-	-

Table 3: LOT

	Type of result	Corrosion process	Used as ref for
TR-09-31 (A0 test parcel)	Corrosion rate < 4 µm/y, somewhat uneven, no obvious signs of pitting	Oxic corrosion in bentonite	Observation of uneven distribution of chloride from ground water, and redistribution of relatively soluble secondary minerals in the buffer
TR-13-17 (compilation A2, tube)	Use of copper tube to study effect of copper on bentonite; exp. not designed for corrosion studies: corrosion rates from Cu profiles: 1.7 µm/y (hot blocks), 0.12 µm/y (cold blocks), also from weight loss	Oxic corrosion in bentonite, possibly also anoxic with sulphide	Summation of observations on corrosion, to answer questions on interaction between corrosion products and bentonite





Table 3: LOT (cont.)

	Type of result	Corrosion process	Used as ref for
[R-13-15 (electrodes from A2)]	Corrosion rates, measured with electrochemical techniques decreases with time to < 1 µm/y	Oxic corrosion in bentonite	-
WM'02 Tucson (LOT Pilot)	Corrosion rate 3 µm/y (one year, weight loss), 1.7 µm/y (LPR); uneven corrosion, no signs of pitting though EN indicate a slight tendency to localised corrosion	Partly under strongly oxidising conditions, uncertainty on redox conditions for EN electrodes	-



Table 4: Uppsala University

	Type of result	Corrosion process	Used as ref for
R-13-31 (exp. design)	<ul style="list-style-type: none"> <li>• Intermediate results from design stage of pressure experiments</li> <li>• Absence of corrosion products on copper surfaces exposed to anoxic water</li> </ul>	Corrosion of copper in pure water	<ul style="list-style-type: none"> <li>• Further development of the pressure experiment.</li> <li>• Status report to SSM, Dec. 2013.</li> </ul>





Table 5: "Äspö exp.: atmospheric and aqueous"

	Type of result	Corrosion process	Used as ref for
WM'02 Tucson (exp. design)	Brief description of start	see below	-
[MRS 2004, atmospheric]	Corrosion rate < 0.1 µm/y, less aggressive than outdoor in Sweden; statement on that deposits may aggravate corrosion and cause localised corrosion	Atmospheric + corrosion during saturation (remaining oxygen)	To estimate < 1 µm for atmospheric corrosion
[unpublished report from Kimab]	Sulphide water: mass loss and corrosion products consistent with formation of Cu <sub>2</sub> S Chloride water: corrosion by complexing with Cl or by traces of sulphide.	Corrosion in chloride and sulphide rich groundwaters. With too low flow of gw, oxygen could have been diffusing in via the outlet, and the exp. was terminated after 2.5 years.	-

## Conclusions on use of data in SA

- Most of the "corrosion experiments at Äspö"
  - were designed with another primary focus (Minican – corrosion of insert, LOT – behaviour of bentonite)
  - did experience oxidizing conditions (or not well-controlled redox conditions)
- Observations in the experiments of corrosion products and nature of corrosion (e.g. general vs localised) used in argumentation on processes
- In SA, corrosion depth during the assessment period (10<sup>6</sup> years) is mostly calculated with mass balances or mass transport limitations - not from measured corrosion rates
- No results from the experiments that obviously contradicts the handling of the processes in the safety assessment







## Appendix 3 – SKB's Answers to QA Review Questions Relating to Copper Corrosion Experiments

The following tables list SKB's responses to QA review questions relating to the following copper corrosion experiments:

- MiniCan Experiment 3 (analysis of copper coupons and canister).
- The LOT project A0 parcel tests (analysis of copper coupons).
- The LOT project A2 parcel tests (analysis of copper tube).
- Copper corrosion tests in oxygen-free pure water.
- The atmospheric and aqueous copper corrosion tests.

The QA review questions are organised in terms of issues relates to the design, running, analysis and use of results of the experiments, as follows:

### Procedures for experiment design and management

- How was the requirement for the experiment identified and how does it support the repository development programme?
- Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?
- What constraints or requirements are there on the location, scale and schedule for the experiment?
- How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?
- What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?

### Procedures for quality control of materials and use of instrumentation

- What quality controls are there on the materials used and the installation of the experiment?
- What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?
- How are measurement uncertainties and instrument detection limits reported and accounted for?
- What controls are there on material recovery for analysis at the end of the experiment?

### Procedures for running the experiments

- What procedures are used for ensuring that the conditions of the experiment (e.g., chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?
- What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?
- What procedures are used for checking records of ongoing results?
- What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?



#### **Procedures for material analysis**

- What procedures are used for calibration and testing of instrumentation used in the material analysis?
- What procedures are there for recording the results of the analysis, including uncertainties?
- What procedures are used to identify, evaluate and report outliers?
- How are measurement uncertainties and instrument detection limits recorded and taken into account?
- Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?
- What procedures are used for checking the results of the analysis?
- What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters?  
Which experimental data are used for validation of modelling results?

#### **Procedures for data management and control**

- How are data from the experiments stored, backed-up, accessed and controlled?
- What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?

#### **Procedures for reporting the results of the experiments**

- What procedures are used for reporting the experiments, analysis and results?
- What procedures are used for review and checking of reports?
- Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?
- Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?
- How is it ensured that the reported results are used appropriately and uncertainties are taken into account?
- How is it ensured that experimental results are not omitted from being reported?

SKB prepared responses to this list of QA issues for each copper corrosion experiment prior to the meeting and revised its responses following clarification of issues during the meeting. The revised responses are shown in the tables in this appendix.



## Answers to questions in SSM QA review August 29, 2014: Minican

3.1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	In the safety assessment SR 97 a case with a pin hole through the EB weld brought forward the questions on what would happen with the iron insert with such a hole. The change of welding method to FSW has diminished the importance of the investigations of the corrosion aspects of the iron insert.  More generally, the description of needs for experiments is given in the RD&D programmes, given every 3rd year.
3.1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	The plan for removal and post-test analysis was reviewed by the reference group for copper corrosion, including several independent researchers who had the chance to comment on the plan. An external expert was consulted before an agreement on the project plan was made. Documentation can be provided (if asked for).
3.1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	The specific question originally addressed, i.e. consequences of iron corrosion due to a minor leakage in the copper shell, should not depend qualitatively on the scale of the canisters.
3.1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	In general, technical consultants are contracted on basis of experience from earlier work and, to increasing extent, through competitive procurement. In the specific case, Nick Smarts team at Amec (earlier Serco) was given the contract since they had performed similar and related work for SKB. When contracting a consultant, SKB requires QA certificates for the company and CV for all persons involved in the work.
3.1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	Generally, the objectives are initially described in the tender sent out to the potential consultants. The proposal written by the consultants is then compared with the original tender and discussed within SKB and with the consultants. When an agreement is reached SKB places an order to the consultant. Finally, each report that is published by SKB is reviewed concerning QA as well as factual content.
3.2.1	What quality controls are there on the materials used and the installation of the experiment?	The miniature copper canister is made out of the same OFP copper quality that is going to be used in the KBS-3 repository. The composition of the copper, for example oxygen and phosphor content, is ensured by the manufacturer.
3.2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	All the electrodes installed in Minican were calibrated before the start of the experiments. Despite this, it was seen early after installation that some of the experiments had electrodes not working properly, which was seen as large and random variations in the signals. Several electrodes were therefore replaced and stable signals were obtained.
3.2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Uncertainties in experimental data are given in the published reports. See for example watercomposition in P-12-13 and weight loss analysis in TR-12-09.
3.2.4	What controls are there on material recovery for analysis at the end of the experiment?	When the first canister was retrieved it was taken out under water from the borehole and inserted to a transport flask. An inert-gas glovebox was constructed specially for handling the canisters from Minican. All specimens to be examined with metallographic methods were prepared inside the glovebox, which ensures that the risk for contamination or atmospheric oxidation of the surface was



		minimal. The water chemistry has been recorded regularly, both within the steel cage keeping the experiment canisters, as well as in the boreholes. Other parameters recorded regularly are for example hydrostatic pressure, redox potentials and pH. Several types of electrochemical measurements are applied (LPR, ACI, ECN, ohmic resistance) and data is recorded several times per year and stored in the SICADA database. A number of progress reports have been published. Electrochemical on-line measurements were performed four times annually. Since the variations observed today are small, and since it is known after the retrieval of canister 3 that some of the measurements are producing meaningless results, data will be recorded less often from 2015. When large variations in signals from the electrodes were encountered early after installation and start of the measurements (see 3.2.2) reference electrodes were replaced. When large changes in for example corrosion rates have been observed at a later stage of the experiment, no action has been undertaken. It is likely that the reasons for such behaviour will become apparent when the canisters are retrieved and analysed (as it was for canister 3).
3.3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	The electrodes used in Miminan were calibrated in the laboratory before installation. This procedure is described in the first report published for Miminan.
3.3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	All measurement data are recorded and stored in the SICADA database.
3.3.3	What procedures are used for checking records of ongoing results?	Outlying data are included in the tables/diagrams of the progress reports together with a note or explanation.
3.3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	In one of the early reports (P-11-40) data from one electrode was omitted, see further discussion in SSM 2010:17. Since then, these data have been reported in several reports.
3.4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	Measurement uncertainties are given in the reports. If a measured parameter gives values that are below detection limit for the instrument or method used this is stated (for example the oxygen pressure in the ground water has been reported to be below the detection limit in several Miminan reports).
3.4.2	What procedures are there for recording the results of the analysis, including uncertainties?	No particular procedures. The range of possible corrosion mechanisms considered is based on the composition of the ground water and thermodynamic data (Pourbaix diagrams).
3.4.3	What procedures are used to identify, evaluate and report outliers?	No procedures for checking the raw data is specified, but SKB is reading any reporting. The published reports are factually reviewed in a documented process before publication.
3.4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). No data from Miminan was used in the modelling in SR-Site.
3.4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	
3.4.6	What procedures are used for checking the results of the analysis?	
3.4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	

3.5.1	How are data from the experiments stored, backed- up, accessed and controlled?	SKB has a database (SICADA) for storing raw data as well as certain processed data of measurements.
3.5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	Is mainly handled by the factual review of the reporting (see below).
3.6.1	What procedures are used for reporting the experiments, analysis and results?	Raw data is stored in the SICADA database. Several reports have been published by SKB. All of these reports have gone through factual and quality review. Two peer reviewed papers on Minican have been published in scientific journals.
3.6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037. Within TS (Research and Safety assessment) a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document).
3.6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	Minican is a well-documented project. Several reports and peer reviewed papers have been published. The first report (TR-09-20) describes measurement details, experimental setup and materials in detail.
3.6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	The references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database.
3.6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
3.6.6	How is it ensured that experimental results are not omitted from being reported?	Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.  SKB is requiring that all results are reported.





## Answers to questions in SSM QA review August 29, 2014: LOT A0

3.1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	<p>The LOT experiment was designed primarily for investigation of the long-term testing of buffer materials.</p> <p>In LOT A0 the main aspects were to check that compaction, placement and water saturation did not significantly change the physical properties of the buffer (TR-09-31, section 2.1).</p> <p>Regarding the corrosion part the specific issues of interest were "Check calculated data concerning copper corrosion, and collect information regarding the character of possible corrosion products" (TR-09-31, section 2.1).</p> <p>More generally, the description of needs for experiments is given in the RD&amp;D programmes, given every 3rd year.</p>
3.1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	See SSM 2010:17, Q 3.1 in App A.
3.1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	<p>The coupons were embedded in the LOT A0 test parcel and final investigation could be done when the whole package was taken up.</p> <p>The size of the buffer rings were not considered important for the corrosion investigations.</p>
3.1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	<p>In general, technical consultants are contracted on basis of experience from earlier work. When contracting a consultant, SKB requires QA certificates for the company and CV for all persons involved in the work.</p> <p>See also SSM 2010:17, Q 1.3 in App A.</p>
3.1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	See SSM 2010:17, Q 1.3 in App A.
3.2.1	What quality controls are there on the materials used and the installation of the experiment?	The copper coupons was manufactured from plate material of canister quality (stated in TR-09-31, section B3).
3.2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	The techniques and equipment are commented in SSM 2010:17, Q 2.2 and 3.3 in App A.
3.2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Some details on the measurement procedures are given in the report TR-09-31.



3.2.4	What controls are there on material recovery for analysis at the end of the experiment?	Some details on the measurement procedures are given in the report TR-09-31.
3.3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	Described in the reporting from the LOT project (TR-09-31). Some notes on the temperatures are given in the corrosion part (App. B).
3.3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	No on-line measurements of corrosion.
3.3.3	What procedures are used for checking records of ongoing results?	No on-line measurements of corrosion.
3.3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	No on-line measurements of corrosion.
3.4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	See SSM 2010:17, Q 1.3 and 2.2 in App A.
3.4.2	What procedures are there for recording the results of the analysis, including uncertainties?	No particular procedures have been encountered.
3.4.3	What procedures are used to identify, evaluate and report outliers?	No particular procedures have been encountered.
3.4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	Not documented as far as been recognised.
3.4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	No particular procedures. The range of possible corrosion mechanisms considered is based on the general knowledge of the composition of the pore water and on thermodynamic data (Pourbaix diagrams).
3.4.6	What procedures are used for checking the results of the analysis?	No procedures for checking the raw data is specified, but SKB is reading any reporting. The published reports are factually reviewed in a documented process before publication.
3.4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). No data from LOT was used in the modelling. Observations regarding the lack of localised corrosion have been used in supporting documents.
3.5.1	How are data from the experiments stored, backed-up, accessed and controlled?	See SSM 2010:17, Q 3.1 in App A.
3.5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are	Is mainly handled by the factual review of the reporting (see below).

	recorded and understood)?	
3.6.1	What procedures are used for reporting the experiments, analysis and results?	See SSM 2010:17, Q 4.2 in App A.
3.6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037. Within TS (Research and Safety assessment) a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document).
3.6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	No specific procedures were set up.
3.6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	The references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database.  Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
3.6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
3.6.6	How is it ensured that experimental results are not omitted from being reported?	SKB is requiring that all results are reported.





## Answers to questions in SSM QA review August 29, 2014: LOT A2

3.1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	<p>The LOT experiment was designed primarily for investigation of the long-term testing of buffer materials.</p> <p>In LOT A2 the main aspects were to check that the repository temperature and geochemical conditions after water saturation do not significantly change the physical properties of the buffer (TR-09-29, section 2.1).</p> <p>Regarding the corrosion part the specific issues of interest were "Check of calculated data concerning copper corrosion, and collect information regarding the character of possible corrosion products" (TR-09-29, section 2.1).</p> <p>More generally, the description of needs for experiments is given in the RD&amp;D programmes, given every 3rd year.</p>
3.1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	See SSM 2010:17, Q 3.1 in App A.
3.1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	<p>The scale of the experiment, with copper tubes and bentonite rings smaller than the plans in KBS-3, was to shorten the saturation period, to get a higher temperature gradient and to facilitate dismounting of the package.</p> <p>The size of the buffer rings were not considered important for the corrosion investigations.</p>
3.1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	In general, technical consultants are contracted on basis of experience from earlier work. When contracting a consultant, SKB requires QA certificates for the company and CV for all persons involved in the work.
3.1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	See SSM 2010:17, Q 1.3 in App A.
3.2.1	What quality controls are there on the materials used and the installation of the experiment?	The materials in the tube are specified in (TR-13-17, section 2.2). Further details of the installation etc is described in TR-09-29.
3.2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	See SSM 2010:17, Q 1.3 in App A.



3.2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
3.2.4	What controls are there on material recovery for analysis at the end of the experiment?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
3.3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	Described in the reporting from the LOT project (TR-09-29).
3.3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	No on-line measurements of corrosion.
3.3.3	What procedures are used for checking records of ongoing results?	No on-line measurements of corrosion.
3.3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	No on-line measurements of corrosion.
3.4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
3.4.2	What procedures are there for recording the results of the analysis, including uncertainties?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
3.4.3	What procedures are used to identify, evaluate and report outliers?	No particular procedures as far as has been recognised.
3.4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
3.4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	No particular procedures. The range of possible corrosion mechanisms considered is based on the general knowledge of the composition of the pore water and on thermodynamic data (Pourbaix diagrams).
3.4.6	What procedures are used for checking the results of the analysis?	No procedures for checking the raw data is specified, but SKB is reading any reporting. The published reports are factually reviewed in a documented process before publication. The data for copper was checked by later considerations (TR-13-17) of mass balances.
3.4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). No data from LOT was used in the modelling. Observations regarding the lack of localised corrosion have been used in supporting documents.
3.5.1	How are data from the experiments stored, backed-up, accessed and controlled?	See SSM 2010:17, Q 3.1 in App A.

3.5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	Is mainly handled by the factual review of the reporting (see below).
3.6.1	What procedures are used for reporting the experiments, analysis and results?	See SSM 2010:17, Q 4.2 in App A.
3.6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037. Within TS (Research and Safety assessment) a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document).
3.6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	See SSM 2010:17, Q 1.3 in App A.
3.6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	The references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database. Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
3.6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
3.6.6	How is it ensured that experimental results are not omitted from being reported?	SKB is requiring that all results are reported.



## Answers to questions in SSM QA review August 29, 2014: Uppsala U. experiment

3.1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	Following the publication of alleged evidence of copper corrosion in anoxic water by Hultquist and co-workers, SKB needed to perform some kind of repetition of the experiments.
3.1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	The experiments are expected to increase the knowledge base of the behaviour of copper in water. - A pre-study was carried out prior to the decision to carry out the experiment. Much of the planning and design was done in the pre-study. - The experimental plan is included in the project tender to SKB, where the outcome of the pre-study is referenced. - There is no explicit, overall QA plan for the experiment
3.1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	The location was given by the contractor's lab. The scale was intended to be close to the experiment by Gunnar Hultquist. The envisaged time scale was initially about 1.5 years, but has been extended. All the above is specified in the contract between UU and SKB.
3.1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	The chemistry group at the Ångström laboratory, Uppsala university was selected based on its members' competence and broad network of contacts in various, required areas at the university.
3.1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	The Uppsala group used their expertise and network of contacts at the university to select experts for the various types of analyses involved in the experiments. SKB has been following the work by the Uppsala group, as has the Reference Group that was set up for this experiment (and some other).  The means organised by SKB has been: - regular meetings with SKB staff, telephone and e-mail conversations - peer review of reports - the Reference group
3.2.1	What quality controls are there on the materials used and the installation of the experiment?	The performed controls of materials are documented in R-13-31 (in Swedish) and R-14-07 (English translation of R-13-31).
3.2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	To some extent described in R-13-31 and R-14-07 (English translation of R-13-31). Additional data stored at and available from the Uppsala group.
3.2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Described in R-13-31/R-14-07. (The ERDA results cited in the report have been re-evaluated after publication of report and deemed as less useful. To be reported.)
3.2.4	What controls are there on material recovery for analysis at the end of the experiment?	Described in R-13-31/R-14-07. Most of the experiment was performed in a glovebox with a controlled N <sub>2</sub> atmosphere. Transfer of copper samples after exposure to anoxic water to analysis instruments outside the glovebox was done



		in inert atmosphere.
3.3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	Described in R-13-31/R-14-07.
3.3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	Described in R-13-31/R-14-07. Temperature and pressures were recorded.
3.3.3	What procedures are used for checking records of ongoing results?	Described in R-13-31/R-14-07 (ongoing measurements continuously monitored, logged and backed-up).
3.3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	To some extent described in R-13-31/R-14-07.
3.4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	To some extent described in R-13-31/R-14-07.
3.4.2	What procedures are there for recording the results of the analysis, including uncertainties?	Such procedures are, for the analyses of the copper samples after exposure to water, to some extent described in the R-13-31. Additional data stored at and available from the Uppsala group.
3.4.3	What procedures are used to identify, evaluate and report outliers?	No particular procedures. All results are reported.
3.4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	Described in R-13-31/R-14-07.
3.4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	No particular procedures. Not really applicable since the experiment aims at checking whether an alleged corrosion mechanism exists in the first place. No evidence of corrosion has, so far, been found in the experiments.
3.4.6	What procedures are used for checking the results of the analysis?	The results from the Uppsala group were closely followed by both SKB and the Reference group. Experiments to check interpretation and conclusions from the Uppsala group has been performed at Micans in Gothenburg (see a recent status report from SKB to SSM, in Swedish). The published reports are factually reviewed in a documented process before publication.
3.4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Not applicable, since no evidence of corrosion was found.
3.5.1	How are data from the experiments stored, backed-up, accessed and controlled?	Double back-up systems with UPS. (Also through continuous deliveries of raw data of pressure and temperature measurements to SKB).
3.5.2	What procedures are used for ensuring that the data	Described in R-13-31/R-14-07. As described in the report, extreme measures are taken to check the

	are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	Cu material investigated and to control the environment in which the measurements are done.
3.6.1	What procedures are used for reporting the experiments, analysis and results?	<ul style="list-style-type: none"> <li>- Reporting is required in the contract with SKB.</li> <li>- Progress was reported to and discussed in a dedicated Reference group from the start of the project until the autumn of 2013, several times a year.</li> <li>- More frequent reports to and discussions with SKB.</li> <li>- Work carried out up to Spring 2013 is published in R-13-31/R-14-07, additional reporting to appear.</li> <li>- Ultimately, the aim is to report the results in a peer reviewed journal.</li> </ul>
3.6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037.
3.6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	Within the TS section at SKB (Research and Safety assessment) a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document).
3.6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	No particular procedures, but this is a general aim when describing scientific experiments in academia.
3.6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	Generally, the references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database. These specific experiments were performed after submitting the licence application. The progress of this particular issue has been directly reported to SSM in a number of status reports to SSM, in addition to the reporting in R-13-31/R-14-07. Studies with the alternative method developed at Mïicans (see response to Q 3.4.6) is an important complement to the studies at Uppsala.
3.6.6	How is it ensured that experimental results are not omitted from being reported?	SKB is requiring that all results are reported. The Reference Group was one attempt to give extra insight into the experiment.





## Answers to questions in SSM QA review August 29, 2014: Atmospheric and aqueous experiments at Äspö

3.1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	The experiments are expected to increase the knowledge base of the behaviour of copper in repository-like environments. More generally, the description of needs for experiments is given in the RD&D programmes, given every 3rd year.
3.1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	Not as far as been recognised.
3.1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	The idea was to use the atmosphere and the ground water naturally coming from the rock wall in the Äspö tunnel respectively, which thus set the environmental conditions.
3.1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	In general, technical consultants are contracted on basis of experience from earlier work. In this specific case, Claes Taxén was given the contract as he had been involved in earlier corrosion studies for SKB.
3.1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	No formal procedures have been evidenced.
3.2.1	What quality controls are there on the materials used and the installation of the experiment?	Stated in publication (Taxén 2004) that the copper material was delivered by SKB and from left over after manufacturing of a full-scale canister.
3.2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	No documentation of procedures as far as been recognised.
3.2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Not given in the reporting.
3.2.4	What controls are there on material recovery for analysis at the end of the experiment?	Some details are given in the reporting, but no formal procedures as far as been recognised.
3.3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	Atmospheric exp.: the temperature was measured periodically, and evidenced in the report for the first year. The set-up was provided with a dark cover to avoid influence of light. As noticed in (Taxén 2004) the heater didn't work for periods of weeks. Aqueous exp.: groundwater was fed off from fractures in the rock into a pressure vessel.
3.3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	No on-line measurements of corrosion.



3.3.3	What procedures are used for checking records of ongoing results?	No on-line measurements of corrosion.
3.3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	Atmospheric exp.: problems with the heater were recognised. All ten samples from the heated experiment was analysed as some had fallen down. Aqueous exp.: the experiment was stopped as the ground water flow decreased too much and ingress of oxygen not could be excluded.
3.4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	No formal procedures are documented as far as been recognised.
3.4.2	What procedures are there for recording the results of the analysis, including uncertainties?	No formal procedures are documented as far as been recognised.
3.4.3	What procedures are used to identify, evaluate and report outliers?	No formal procedures are documented as far as been recognised.
3.4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	Not documented as far as been recognised.
3.4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	No particular procedures. The range of possible corrosion mechanisms considered is based on the composition of the ground water and thermodynamic data (Pourbaix diagrams).
3.4.6	What procedures are used for checking the results of the analysis?	No particular procedures. After failing of part of the experiments less efforts were put into evaluation of the results.
3.4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The measured corrosion rates in the atmospheric experiment were used in the evaluation of the atmospheric corrosion during initial storage (before deposition).
3.5.1	How are data from the experiments stored, backed-up, accessed and controlled?	The results from the experiments were reported in a conference paper (atmospheric exp.) and as an internal report to SKB (the aqueous ex.).
3.5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	Is mainly handled by the factual review of the reporting (see below).
3.6.1	What procedures are used for reporting the experiments, analysis and results?	No particular procedures. The results from the experiments were reported in a conference paper (atmospheric exp.) and as an internal report to SKB (the aqueous exp.).
3.6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037.
3.6.3	Are there procedures to ensure that the documentation provides enough detail for the	Within TS (Research and Safety assessment) a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document). Not as far as been recognised.

3.6.4	<p>experiment to be repeated?                  Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?</p>	<p>The references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database.</p>
3.6.5	<p>How is it ensured that the reported results are used appropriately and uncertainties are taken into account?</p>	<p>Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.</p>
3.6.6	<p>How is it ensured that experimental results are not omitted from being reported?</p>	<p>Procedures of data selection for the SR-Site safety assessment is described and used in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.</p> <p>SKB is requiring that all results are reported.</p>