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Quality Assurance Review of SKB's Copper Corrosion Experiments

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SSM Perspective

Background

SKB is preparing a license application for the construction of a final repository for spent nuclear fuel in Sweden. This application will be supported by the safety assessment SR-Site for the post-closure phase. The assessment of long-term safety is based on a broad range of experimental results from laboratory scale, intermediate scale and up to full scale experiments. It is essential that there is a satisfactory level of assurance that experiments have been carried out with sufficient quality, so that results can be considered to be reliable within the context of their use in safety assessment. The former named authority, SKI, has initiated a series of reviews of SKB's methods of quality assurance and their implementation.

This quality assurance review is focused on the work of copper corrosion being conducted in at SKB's Hard Rock Laboratory (HRL) in Äspö, LOT and Miniature canister (Minican) experiments. In order for the reviewers to get a broad understanding of the issue of copper corrosion both SKB reports as well as the viewpoint of MKG was collected prior to commencement of the actual review task.

Objectives of the project

The purpose of this project is to assess SKB's quality assurance with the view of providing input for the preparation of the SR-Site safety assessment. This has been achieved by examination of the corrosion part of the LOT and Minican experiments using a check list, visits to the relevant facilities, and meetings with contractors and a few members of the SKB staff. The same approach for quality assurance reviews has been used earlier in similar review tasks.

Results

During the quality review of the selected projects, several QA- related issues of different degree of severity was noted by the reviewers. The most significant finding was that SKB has chosen to present only selected real-time corrosion monitoring data in TR-09-20. This was surprising and SSM expect that SKB will analyse the reason for this thoroughly. The reviewers also made other observations which can be grouped as transparency related e.g. significant delays in reporting, lack of uncertainty evaluation for experimental data and too limited access to progress reports from research suppliers. Transparency and full accessibility of primary data is essential for upcoming licensing activities. SSM therefore encourage SKB to address the concerns in this review and provide a plan for improved transparency of field testing activities. However, it must be emphasised that this quality assurance review only covers limited aspects of two ongoing field experiments and the results should not be generalised. Other quality assurance reviews of SKB has not resulted in any severe comments, it can therefore not be excluded that the deficiencies reported here is of singular occurrence.

Effect on SSM supervisory and regulatory task

Quality aspects will be further analysed as part of the review of SKB's SR-Site safety assessment. Additional scrutiny of this subject will be needed also for the subsequent stages of SKB's program.

Project information

SSM project manager: Jan Linder
Project Identification Number: SSM 2009/3443 project number 1777 and SSM 2009/4300 project number 3037027-01.

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Executive Summary

SKB intends to submit to the Swedish Radiation Safety Authority, SSM, an application to construct a spent nuclear fuel repository at Forsmark. The SR-Site safety assessment will form part of the application. SR-Site will include quantitative analyses aimed at presenting an understanding of how the repository system will evolve and an evaluation of the potential risks of spent fuel disposal. Such a safety assessment must be underpinned by assurances that the development and application of models, and work to evaluate parameters and uncertainties, has been undertaken under appropriate quality management systems.

The copper canister provides an important corrosion-resistant barrier in SKB's repository concept. SKB's experiments on copper corrosion are of particular interest because some independent researchers have questioned SKB's understanding of corrosion processes under the anoxic conditions that are expected to persist in the repository in the long term. Therefore, SSM commissioned Galson Sciences Limited to undertake quality assurance (QA) reviews of some of SKB's experiments on copper corrosion.

The Long Term Test of Buffer Material (LOT) Project and the Miniature Canister (MiniCan) Project were selected for review because they include copper corrosion tests aimed at providing data on corrosion rates under repository conditions. These experiments are being conducted at SKB's Hard Rock Laboratory (HRL) in Äspö. Visits to the HRL and to SKB's offices in Stockholm provided opportunities to discuss QA aspects of these corrosion tests with SKB staff and contractors. LOT and MiniCan project reports and publications were also studied. Consistent with previous QA reviews of SKB's experiments, a checklist of quality-affecting issues was prepared to facilitate and document the LOT and MiniCan reviews, covering the framework, design, conduct, analysis and reporting of experiments, and the use of experimental results in the KBS-3 repository research programme.

Regarding quality management systems, the review found that both the LOT and MiniCan projects are being conducted under SKB's management procedures according to appropriate project and activity plans. Both projects are being led by suitably qualified contractors, who have produced project QA plans approved by SKB. The contractors undertake project work under their own accredited quality management systems. This approach to QA is consistent with approaches adopted by SKB for other tests and experiments currently being undertaken at the HRL, as noted in previous QA reviews conducted by Galson Sciences Limited on behalf of SSM (and previously SKI).

The detailed QA review of the LOT and MiniCan projects made several observations regarding the conduct, analysis and reporting of the experiments. The most significant finding was that the MiniCan technical report published by SKB in 2009 presents only selected real-time corrosion monitoring data, although the full data set has been included in internal project progress reports. No indication was given in the SKB technical report that some data had been excluded. The absence of selected data from the SKB report became apparent during the QA review. The published data were stated as being consistent with data reported in the literature, but the missing data indicate extremely high copper corrosion rates, which suggests that there are problems with the measurement technique.

It is unclear how or why the decision to exclude selected corrosion rate data from the published report was made (no record of the decision is available), but this decision does not reflect scientific best practice. It would have been more appropriate for the full data set to have been published, accompanied by a discussion of the reliability of the data, uncertainties, potential faults with the measurement technique, and the need for further analysis.

Other key QA-related observations from the review are as follows:

- The copper corrosion tests that form part of the LOT and MiniCan experiments are subsidiary tests to already planned experiments to investigate other processes. Experiments whose sole aim is to study copper corrosion in a repository-like environment would avoid the potential complication, constraints or influence of tests of other processes in the same experiment.

- SKB is undertaking or planning a diverse range of experiments that include copper corrosion tests, including future experiments dedicated to understanding copper corrosion. These experiments are spread across different organisations and countries, contractors and sub-contractors, and it is important that SKB's requirements for these experiments are communicated effectively.

- It is apparent that SKB places significant reliance on its external consultants for determining the scope of the copper corrosion experiments reviewed here. It is important that SKB fully understands the work carried out on its behalf and that it is of direct support to SKB's objectives.

- The copper corrosion tests in the reviewed experiments aimed to confirm SKB's understanding of corrosion rates in a repository-like environment. The review has noted that researchers infer that higher than expected corrosion rates reflect problems with the experiment. However, it is unclear how SKB would respond if it is shown that the corrosion rates are greater than hypothesised.

- The reports from the MiniCan and LOT experiments provide little information on the sources or quantification of data uncertainty, or the level of confidence that can be assumed in the results. Factors that influence data uncertainty should be identified, such as measurement detection limits, the problems in defining the length of time a sample is subject to certain geochemical conditions, and instrumentation problems, such as electrode degradation.

- Understanding when conditions are oxic and when they are anoxic is of key importance in real-time copper corrosion tests; it will be difficult to interpret corrosion measurements and long-term corrosion rates unless the evolution of geochemical conditions is understood. It was not clear, in this review, how well redox conditions are understood in the vicinity of the copper corrosion tests in the MiniCan and LOT experiments.

- There have been delays in the publication of SKB technical reports on the LOT project. This QA review acknowledges the time required to analyse and understand the data obtained both before and after parcel extraction, but timely publication of results is important. Publication of the results for the LOT A0 parcel, extracted in 2001, has been given a low priority by SKB, although results were presented at the QA review meeting and have been provided at other SKB meetings with SSM. The results for the A2 parcel, extracted in 2006, were not published until the end of 2009. There have been discussions of these experiments and their results at meetings and conferences, but such presentations do not justify the delay and/or lack of publicly available SKB reports.

- SKB stated during the QA review that it gives greater weight to publications in peer-reviewed journals than to SKB technical reports. The importance of publishing articles in specialised journals to support the evolving body of knowledge is recognised, but it is also important that SKB publishes its work in a more comprehensive form in easily accessible technical reports. Other stakeholders will not have easy access to specialised journals, and the publication conditions of such journals, in particular limited article length, mean that key technical details and data cannot be published.

SSM has continued to maintain awareness of issues and uncertainties regarding copper corrosion processes under repository conditions. In 2010, SSM decided to finance three experiments concerned with copper corrosion in anoxic environments, with the aim of enhancing SSM's knowledge of the subject. However, SKB remains responsible for acquiring the information on copper corrosion needed to support its repository safety assessment.

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1 Introduction

1.1 Background

Later this year, SKB intends to submit to the Swedish Radiation Safety Authority, SSM, an application to construct a spent nuclear fuel repository at Forsmark. The SR-Site safety assessment, to be based on the KBS-3 repository concept, will form part of the application. SR-Site will include quantitative analyses aimed at presenting an understanding of how the repository system will evolve and an evaluation of the potential risks of spent fuel disposal. Such a safety assessment must be underpinned by assurances that the development and application of models, and work to evaluate parameters and uncertainties, has been undertaken under appropriate quality management systems.

Previously, SSM (and SKI prior to the formation of SSM) commissioned Galson Sciences Ltd (GSL) to undertake a series of review tasks in order to understand SKB's approach to quality assurance (QA) and the application of QA procedures in SKB's work. These QA reviews were as follows:

- A review of the documentation and testing of a selection of the computer codes used by SKB in its repository research programme (Hicks, 2005).
- A comparison of SKB's approach to QA with QA programmes adopted in radioactive waste management projects in other countries, and a quality review of a selection of experiments on engineered barrier performance undertaken as part of SKB's repository research programme (Hicks, 2007).
- A review of how data and code quality assurance was addressed and reported in the SR-Can safety assessment (Hicks and Baldwin, 2008).
- A review of the QA procedures and instructions that SKB has prepared for the SR-Site safety assessment and further quality audits of key tests and experiments that may provide data for the safety assessment (Baldwin and Hicks, 2009).

SSM is continuing to review and provide feedback to SKB on the development of the SR-Site safety assessment. Examination of the quality of SKB's experimental investigations to understand the long-term evolution of the repository multi-barrier system continues to form an important part of SSM's review process.

The corrosion-resistant copper canister forms a key component of the KBS-3 barrier system, but SKB's understanding of copper corrosion processes and rates under repository conditions has been questioned by researchers who have conducted independent corrosion tests (see Section 2.2). In order to address some of the concerns raised by these researchers on the reliability of SKB's understanding of copper corrosion, SSM commissioned GSL to undertake a QA review of some of the copper corrosion tests undertaken by SKB. This report provides the results of the QA review.

1.2 Approach

Initially, a broad understanding of the key issues and uncertainties relating to copper corrosion was established. SKB reports and experiments on copper corrosion were identified, as were publications and reviews by other researchers on copper corrosion processes. In addition, MKG (the Swedish non-governmental organisation (NGO) office for Nuclear Waste Review) was consulted in order to understand its concerns regarding the quality of SKB's copper corrosion tests.

Subsequently, the main part of the project involved performing QA reviews of a number of experiments that SKB has conducted in order to determine the corrosion behaviour of copper under repository conditions. The Long Term Test of Buffer Material (LOT) Project and the Miniature Canister (MiniCan) Project were selected for review because they include copper corrosion tests aimed at providing data on corrosion rates under repository conditions. These projects are being conducted at SKB's Hard Rock Laboratory (HRL) in Äspö.

The LOT and MiniCan reviews included meetings with SKB staff and contractors at the HRL on 1 December 2009, and at SKB's offices in Stockholm on 10 and 11 March 2010. In order to facilitate the discussions at these meetings and the documentation of review findings, a checklist of quality-affecting issues was prepared covering the framework, design, conduct, analysis and reporting of experiments, and the use of experimental results in the KBS-3 repository research programme. Such a checklist has been used in previous experiment audits (Hicks, 2007; Hicks and Baldwin, 2008; Baldwin and Hicks, 2009). The findings of the review were documented on forms based on the above-mentioned checklist and are discussed in this report.

1.3 Report Structure

Section 2 provides a brief summary of SKB's evaluation of copper corrosion rates and alternative views of copper corrosion processes presented by other researchers. Section 3 outlines the review approach undertaken in this project. Section 4 presents the review of quality-related aspects of the copper corrosion tests performed as part of the LOT and MiniCan experiments. A discussion of review findings and conclusions is presented in Section 5. Appendix A comprises the completed QA checklists for each experiment reviewed.

2 Copper Corrosion Issues

This section provides a brief summary of key research undertaken by SKB on the corrosion resistance of copper canisters under repository conditions. In addition, concerns raised by other researchers regarding the potential for long-term corrosion under anoxic conditions are noted. The role of the present QA review in developing an understanding of the reliability of copper corrosion experiments undertaken by SKB is also noted.

2.1 SKB's Evaluation of Copper Corrosion Rates

In the KBS-3 spent fuel disposal concept, the 50-mm-thick copper canister is intended to provide a corrosion-resistant barrier that, in conjunction with other barrier components, serves to isolate the spent fuel for a one million year assessment period (SKB, 2006). SKB's research and development programme has included modelling studies and experiments that have aimed to confirm that the copper canister will provide the required corrosion resistance under repository conditions.

In 1994, SKB (Wersin *et al*., 1994) published a modelling study of copper corrosion under repository conditions that derived "conservative" corrosion rates of:

- 7×10^{-6} m/y for oxic conditions (with the rate limited by the rate of diffusion of dissolved oxygen towards the canister surface); and

- 2×10^{-8} m/y for anoxic conditions (with the rate limited by the rate of diffusion of dissolved sulphide towards the canister surface).

The conservative cases assumed high rates of uniform and pitting corrosion compared to "realistic" cases presented. Wersin *et al*. (1994) also estimated an upper bound timescale of 280 years for oxic corrosion, implying a maximum corrosion depth of 22 mm in one million years (substantially less than the canister thickness).

King *et al*. (2001) reviewed a range of studies on copper corrosion processes under repository conditions. Studies by Werme *et al*. (1992), the Swedish Corrosion Institute (1983) and Johnson *et al*. (1996), as well as the results reported by Wersin *et al*. (1994), were found to support the expectation that canister lifetimes will exceed one million years.

2.2 Copper Corrosion under Anoxic Conditions

SKB's SR-Can safety assessment drew strongly on the findings of the King *et al*. (2001) review of corrosion processes (SKB, 2006), and, as discussed in Section 4, a number of corrosion tests undertaken by SKB have aimed to confirm that realistic corrosion rates are less than the conservative values presented by Wersin *et al*. (1994). However, independent researchers at the Royal Institute of Technology (KTH, Kungliga Tekniska Högskolan) in Stockholm have published articles on copper corrosion experiments that challenge the conventional understanding of copper corrosion under anoxic conditions. In particular, Szakálos *et al*. (2007) published experimental results in support of the proposition that copper can corrode by extracting oxygen from water molecules even under anoxic conditions. Such a process would allow general copper corrosion to proceed under repository conditions in the long term.

The Szakálos *et al*. (2007) experiments, and other KTH publications that support the view that copper corrodes in water under anoxic conditions, have been the subject of several reviews and discussions. MKG considered the issues that have been raised in the copper corrosion debate and concluded that the long-term experiments demonstrate that corrosion of copper in oxygen-free water can occur (MKG, 2009). However, reviews by Apted *et al*. (2009), on behalf of SSM, and King (2009) have questioned the evidence for such a corrosion process.

2.3 Aims of the QA Review of Copper Corrosion Experiments

The QA review presented in this report has not aimed to make judgments on the evidence for copper corrosion in water under anoxic conditions. Instead, the review has examined the design, conduct, analysis and reporting of key copper corrosion experiments undertaken by, or on behalf of, SKB, with the aim of gaining an understanding of the reliability of data used by SKB in support of its view of copper corrosion processes. In particular, the review has included consideration of the reliability of controls on geochemical, hydrological and thermal conditions during the experiments and of measurements of corrosion rates under evolving conditions. The availability of reliable measurements of time-dependent corrosion rates under known geochemical conditions would build confidence that long-term copper corrosion processes are understood.

The approach to and scope of the QA review is discussed in Section 3 and the findings of the review are presented in Section 4.

3 QA Review Approach

Following discussion with SSM, two SKB experiments (LOT and MiniCan) being undertaken at the Äspö HRL were selected as the focus for the detailed QA review of copper corrosion tests:

- In the LOT experiment, copper tubes containing heater elements surrounded by bentonite blocks were placed in boreholes at the HRL with the primary aim of investigating bentonite buffer properties and mineral stability in a repository-like environment (SKB, 2000; Rosborg and Werme, 2008; and SKB, 2009a). However, copper coupons, ^{60}Co tracers, bacteria and other materials were embedded in various bentonite blocks in order to investigate other processes. In particular, the copper coupons were included in order to investigate copper corrosion under repository conditions.
- In the MiniCan experiment, a number of small-scale copper canisters with cast iron inserts have been placed in boreholes at the HRL (SKB, 2009b). Holes were pre-drilled in the canisters to simulate leaks, thereby enabling investigation of the effects of corrosion of the cast iron insert. The canisters are either surrounded by bentonite in the boreholes or are exposed to unconditioned groundwater. Similar to the LOT experiment, the opportunity was taken to measure copper corrosion under repository conditions by including corrosion coupons. Electrochemical measurement devices were also included to enable measurement of real-time corrosion.

The QA reviews of these experiments were centred on meetings with SKB staff and contractors at the HRL and at SKB's offices in Stockholm, as discussed in Sections 3.2 and 3.3. The detailed review findings are discussed in Section 4.

Checklists of quality-affecting issues were used in these meetings to ensure comprehensive coverage and documentation of issues. However, as noted in Section 2.2, MKG has expressed concerns regarding SKB's understanding of copper corrosion processes under repository conditions. Thus, it was considered important to contact MKG prior to the QA reviews, to ensure that any concerns regarding the quality of SKB's corrosion tests were captured by the review and documented on the checklists. Key points of the discussion with MKG are described below.

3.1 Discussion with MKG

A discussion of SKB's copper corrosion tests was held with Johan Swahn of MKG on 25 November 2009 (teleconference). The following comments were made:

- It is not clear that SKB fully understands the evolving geochemical conditions in the LOT experiment, particularly the changing oxygen content of the system. It is possible that anoxic conditions develop rapidly in the LOT tests, possibly on the scale of days or weeks, in which case the reported copper corrosion would be due to some kind of anoxic corrosion mechanism. Alternatively, the observed corrosion may occur early in the experiment when oxygen is present and then reduce significantly under anoxic conditions but MKG considers this unproven.

- Bacteria may be responsible for consuming oxygen very quickly under repository conditions, but this process needs to be better understood.

- It is not clear why SKB has not studied corrosion on the central copper tube in each of the LOT test parcels in any detail.

- MKG would like access to the raw data from SKB's copper corrosion experiments but it is not clear if these data can be made available.

- The QA review should cover the analysis undertaken to measure the extent of corrosion of the LOT copper coupons.

- SSM (or an independent expert) should perform properly controlled long-term experiments on copper corrosion.

These issues were noted and raised during the review meetings.

3.2 Meeting at the Äspö HRL

A review meeting focusing on the LOT experiment was held at the Äspö HRL on 1 December 2009, attended by staff from SKB, Clay Technology (who manage the LOT experiment), SSM and GSL. Additionally, Dr Hans-Peter Hermansson, who is a corrosion expert, attended this meeting on behalf of SSM.

The LOT experiment and its findings so far concerning copper corrosion were discussed, facilitated by a presentation by Ola Karnland (Clay Technology), the LOT project manager. This discussion was followed by a visit to the LOT experiment in the HRL. Subsequently, a discussion of QA in the LOT experiment took place, using the QA checklist as a focus. The checklist covered the framework, design, conduct, analysis and reporting of the LOT experiment, and the use of results.

Section 4.1 summarises the LOT experiment and the findings from this review. The completed QA checklist is presented in Section A.1.

3.3 First Meeting at SKB's Offices in Stockholm

Meetings were held on 10 and 11 March 2010 at SKB's offices in Stockholm. The meeting on the first day focused on the MiniCan experiment, and was attended by Nick Smart (Serco Technical Services), the project manager for the MiniCan experiment, and staff from SKB, SSM and GSL.

Prior to the discussion of the MiniCan experiment, Christina Lilja (SKB) provided a brief overview of the copper corrosion experiments that are being conducted or planned by SKB. These include:

- Ongoing experiments on copper corrosion in a sulphide/water environment:

- Tests to determine the rate determining step(s) in the formation of sulphide films and their properties. This work is being carried out by Dave Shoesmith at the University of Western Ontario, Canada, and uses electrochemical and spectroscopic methods.
- Attempts to repeat the experiments of Tanaguchi and Kawasaki (2008), who observed stress corrosion cracking (SCC) of copper in sulphide solutions. This work is being carried out by Roger Newman at the University of Toronto, Canada, and uses slow strain rate tests (SSRT) and electrical impedance spectroscopy.

- Ongoing experiments on copper corrosion in a bentonite environment:
  - The potential for the formation of a sulphide-reducing bacteria (SRB) biofilm on copper in a compacted bentonite environment is being investigated. This work is being carried out by Karsten Pedersen of Microbial Analytics Sweden AB using compacted bentonite in cells and groundwater from Äspö, and microbial analysis techniques. This work should be finalised in 2010 and it is intended to support the SR-Site assessment.
  - Electrochemical studies of copper corrosion in a compacted bentonite environment are being carried out, using copper electrodes exposed in the LOT A2 parcel as well as new electrodes. This work is assessing electrical resistance, electrical impedance spectroscopy and potential measurement techniques, and is being carried out by Bo Rosborg (affiliated with KTH, Stockholm) and Andraz Legat of the Slovenian National Building and Civil Engineering Institute, Ljubljana, Slovenia.
- Ongoing experiments in a repository-like environment:
  - 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. This is being managed by Nick Smart of Serco Technical Services and uses water and gas analyses, microbial analyses, weight loss coupons, potential measurements, electrochemical measurements of corrosion rate and electrical resistance measurements using assemblies mounted in boreholes in the Äspö HRL.
  - Tests are being carried out to study SCC of copper in groundwater containing ammonium. This work is being carried out by VTT, Finland, in co-operation with Posiva and uses SSRT and electrochemical techniques.
- Ongoing experiments in oxygen-free water:
  - An experiment is studying a kinetic model for the copper/electrolyte interface for copper in deoxygenated water using potential measurements and electrical impedance spectroscopy. The work is being carried out by Martin Bojinov at the university of Chemical Technology and Metallurgy, Sofia, Bulgaria.

    - Spectroscopic studies of Cu(I) species are being carried out to obtain data to support first principles calculations. The techniques used include spectroscopic studies, x-ray diffraction of CuH and Cu_2O, and tests of synthesis methods for CuOH. This work is being carried out by Inna Soroka, Uppsala University.
    - Experiments are being carried out to test hypotheses on gas production from copper in oxygen-free water. This work started in 2010 using copper plates in glass test tubes and will use gas analyses techniques. The work is being carried out by Karsten Pedersen of Microbial Analytics Sweden AB.
    - Copper foils in water in Erlenmeyer, or conical, flasks in a reducing environment are being studied to consider the influence of the atmosphere outside the flask. The work is being carried by Kaija Ollila of VTT, Finland, in co-operation with Posiva, and uses water analysis, surface analysis and gravimetric techniques.
- A new copper corrosion project will be established with a reference group to steer future copper corrosion experiments. Two experiments that will be managed by this group have been identified so far:
    - It is intended to repeat the KTH pressure gauge experiment (Szakálos *et al*., 2007) to consider possible interpretations of results. Who will undertake this experiment and how it will be performed are yet to be decided.
    - A 20-year-old test tube containing a copper sample with a Pd membrane, which was part of a 1995 SKI study, has been found. Analysis of the test tube will be performed by SP Technical Research Institute of Sweden.

The 2010 SKB RD&D programme report is currently being drafted and will discuss early, current and planned projects in the context of the knowledge available about copper corrosion.

Following Christina Lilja's overview, Nick Smart gave a presentation on the MiniCan experiment, its objectives and approach, and the results obtained so far. The presentation was followed by a discussion of the experiment, using the QA checklist as a focus. Section 4.2 summarises the MiniCan experiment and the findings from the QA review. The completed QA checklist is presented in Section A.2.

3.4 Second Meeting at SKB's Offices in Stockholm

The second meeting in Stockholm (11 March 2010) focused on the analysis of copper coupons undertaken following their extraction from the LOT A2 test parcel. The meeting was attended by Bo Rosborg (Rosborg Consulting) and staff from SKB, SSM and GSL.

Discussion of the measurement techniques was facilitated by a presentation from the project manager, Bo Rosborg. Findings from this meeting are recorded in the discussion of the LOT experiment in Section 4.1 and in the LOT QA checklist in Section A.1.

Bo Rosborg also discussed the real-time corrosion monitoring experiments - involving the use of copper electrodes - which he is managing via his affiliation with KTH University in collaboration with a team at the Slovenian National Building and Civil Engineering Institute, Ljubljana, Slovenia. Although the experiments were discussed at the meeting, insufficient information is available at this stage to assess the QA of the work in detail. Instead, a brief summary of the experiment is presented in Section 4.1.5 and several QA issues are identified.

4 Review of QA in Selected Experiments

As discussed in Section 3, the QA review focused on the long term test of buffer material project (LOT) and the miniature canister project (MiniCan). The main review findings are discussed in the following sections. The detailed checklists of quality-affecting issues for each experiment are reported in Appendix A.

4.1 LOT Experiment

4.1.1 Background

The LOT project was primarily developed to investigate bentonite buffer properties and mineral stability in a repository-like environment. However, considering the resource invested in such an experiment and the time required for test conditions to develop, further investigations to consider copper corrosion, cation diffusion and bacterial behaviour were combined with the primary experiment. The QA review carried out here considers QA in the LOT project with respect to the copper corrosion tests. QA issues associated with the LOT tests of bentonite behaviour were reviewed and reported previously by Hicks (2007).

The aims of the copper corrosion tests in the LOT experiment are to test the hypothesis that the mean copper corrosion rate is less than $7x10^{-6}$ m/year under initial oxic conditions, as indicated by corrosion modelling (see Section 2.1), and to identify possible pitting and corrosion products (SKB, 2000; SKB 2009a).

The LOT experiment consists of copper tubes containing heater elements surrounded by bentonite blocks and placed in boreholes at Äspö. There are two types of experiment in which the bentonite and copper tube test parcels are exposed to different conditions:

- standard or S-parcels (S1, S2, and S3) that are exposed to expected repository conditions, with temperatures of about 90°C imposed at the copper tube surface; and
- adverse or A-parcels (A0, A1, A2, and A3) that are exposed to adverse repository conditions, with temperatures of about 130°C imposed at the copper tube surface in order to accelerate reactions.

Each parcel includes additives and sensors, such as copper coupons, ^{60}Co tracers, bacteria and chemicals, embedded in the bentonite blocks surrounding the copper tube. At the end of each experiment, the test parcels are extracted and various laboratory tests are performed on the bentonite blocks (measurements of bentonite properties, tracer analysis, analysis of bacteria populations, and measurements of copper coupon corrosion).

The pilot test parcels, A1 and S1, were emplaced in the boreholes at Äspö in 1997 and 1998, respectively, and left for a year. Work on five more tests (S2, S3, A0, A2 and A3) was started in 1999 and parcel A0 was retrieved after one year in 2001. Parcel A2 was retrieved in early 2006, after just over six years of operation. The remaining parcels (A3, S2 and S3) are still in place and a LOT project meeting will be held in 2010 to discuss their extraction.

The results of the two pilot tests (A1 and S1) were reported by SKB (2000). The results of the analysis of parcel A0 remain under review. The results of the analysis of parcel A2 were recently reported by SKB (2009a).

Clay Technology (Ola Karnland) is leading the LOT experiment on behalf of SKB and Posiva. The analyses of each recovered parcel have been undertaken by various organisations, including:

- Bentonite mineralogy and physical properties – Clay Technology AB, BGR Laboratory (Germany), University of Bern (Switzerland), and the G2R and LEM Laboratories (France)
- Pore water chemistry – VTT (Finland)
- Bacterial behaviour – University of Gothenburg and Microbial Analysis Sweden AB
- Cation diffusion – Royal Institute of Technology (KTH)
- Copper corrosion – Rosborg Consulting (using Clay Technology and Studsvik Nuclear AB facilities)

The LOT QA review is concerned with the copper corrosion analysis performed by Rosborg Consulting.

4.1.2 Corrosion Analysis

Pilot Parcels (S1 and A1) Copper Coupons

Twelve copper coupons were used for the pilot study: four in S1, four in A1 and four were retained as a reference. Supplied by Outokumpu Poricopper OY, Finland, the copper specification for the coupons was chosen in order to correspond to the copper proposed for the canister material (SKB, 2000, §8.2). The A1 bentonite blocks containing the copper coupons were damaged during extraction of the parcel and so were not analysed.

Copper coupon A from ring S122, which was heated in the parcel to 50°C, and one of the reference coupons were iteratively photographed, weighed, cleaned and dried, leading to calculation of a mean corrosion rate of 3×10^{-6} m/year (SKB, 2000, §8). SKB (2000) does not indicate the accuracy in this calculated corrosion rate and does not present a discussion of possible uncertainties.

Coupon D from ring S130 and a few centimetres of its surrounding bentonite were impregnated with resin for SEM/energy dispersive x-ray spectroscopy (EDS or EDX) analyses. There is no statement in the report (SKB, 2000) as to how the remaining two S1 copper coupons were analysed or what results were obtained.

Parcel A2 Copper Coupons

The four copper coupons in the A2 test parcel were placed in bentonite rings 22 and 30 (SKB, 2009a, p147). After the bentonite rings containing the copper coupons were cut apart from the rest of the parcel, they were immediately wrapped in plastic sacks which were evacuated using a vacuum pump and then transported to Studsvik Nuclear AB and stored. During the cutting process the copper coupons in bentonite ring A222 were damaged by the cutting wheel during retrieval, preventing accurate assessment of the coupon corrosion rate (SKB, 2009a, p147).

At Studsvik, the coupons were removed from the bentonite rings in a step-wise fracturing process. Similar to the pilot parcels, this was then immediately followed by cycles of photographing the coupons, scraping and cleaning, performing SEM, and drying and weighing (SKB, 2009a, p148). X-ray diffraction (XRD) was used to identify the corrosion products, gravimetric assessment (coupon weight loss) was used to calculate the corrosion rate, and EDS was used to assess the penetration depth of the copper into the surrounding bentonite block.

SKB (2009a, p148) states that it was intended to include a reference coupon through all the cleaning procedures for comparison but, because two of the four coupons in the test parcel were damaged during retrieval, it was decided to save the reference coupons for later investigations in the LOT project. However, SKB (2009) does not explain how this affects the uncertainty in the corrosion rate calculated – the calculation outlined by SKB (2000, Equation 8-1) includes the mass loss of the reference coupon through the cleaning process. It is unclear how the absence of this parameter when calculating the corrosion rate for the A2 parcel is accounted for.

The results from the A2 copper coupon gravimetric assessment (SKB, 2009a, Table A3-3) raise a number of questions. The copper coupons placed in bentonite ring A222 were cut during extraction of the parcel; no weight loss measurement has been carried out for coupon A222F but an estimate was achieved for coupon A222E. However, there is no discussion of how this was achieved or the assumptions made to perform such an assessment. In addition, the table of results records the original coupon weight and the weight loss for A222E, but does not record the final weight; this does not aid transparency.

An estimated average corrosion rate for coupon A230G of $<0.5\times10^{-6}$ m/year is recorded (A230G had the largest weight loss) (SKB, 2009a, Appendix 3). The exact calculated value is not presented and there is no indication or discussion of the uncertainty associated with this estimate, or factors that influence the accuracy of the measurement.

Uncertainties in Corrosion Measurements

Ola Karnland presented the calculated corrosion rates for the copper coupons from LOT test parcels S1, A0 and A2 (see Table 4.1) at the LOT review meeting on 1 December 2009, although no error estimates were provided.

Table 4.1: Calculated corrosion rates for the LOT copper coupon tests (Ola Karnland presentation, 1 December 2009).

Parcel and Coupon	Mass Loss ($x10^{-3}$ g)	Mean Corrosion Rate ($x10^{-6}$ m/year)
S122A	78	2.9
A022A	86	3.5
A030C	83	3.4
A230G	46	0.41
A230H	27	0.24

SKB (2009a, p155) acknowledges that the estimated average corrosion rate recorded for the A2 parcel coupons is considerably lower than those obtained for test parcels A0 and S1 - the observed weight loss for A2 coupons is less than for A0 and S1 coupons despite the additional four years exposure time for A2. This review acknowledges that a definitive reason for this difference may not be known, but it would be useful to include a discussion of the potential reasons for it, such as the different experimental conditions, or uncertainties in data recording or measurement techniques.

SKB (2000, §9.5) states for the S1 copper coupons that optical and scanning electron microscopy (SEM) did not reveal any signs of pitting corrosion, although the corrosion attack was uneven. Similarly, for the A2 coupons, the nature of the corrosion was described as "somewhat uneven general attack", without obvious signs of pitting (SKB, 2009a, Appendix 3). Whilst this review acknowledges the stated aim of the copper coupon analysis was to derive qualitative information about pit corrosion, the term "somewhat uneven" is unclear and could be better supported, for example using cross-section depth measurements to indicate how large the variation in the uneven corrosion is. A sample of such cross-section data was presented at the LOT meeting in Stockholm, on 11 March 2010, for the A2 coupons; it would be beneficial to include such data and discussion in SKB technical reports.

In addition, the defect presented in Figure A3-2d (SKB, 2009a), which is explained as one of a number of surface defects that "*are believed to originate from the manufacturing process rather than being a result of corrosion*", could possibly be viewed as a corrosion pit. It would aid transparency and verification if a similar defect could be shown on an unexposed sample.

It is observed that no quantification of data uncertainty or qualitative discussion of the sources of uncertainty is presented in any of the LOT copper corrosion analyses. It is recognised that sources of uncertainty may not be easily quantifiable but such sources should be discussed and considered so that an understanding can be gained of the confidence in the data presented and the areas of greatest uncertainty. Factors that influence data uncertainty should be identified. For example, whilst this is unlikely to be the largest source of data error, the detection limits and measurement accuracy of the experimental techniques used should be recorded in the technical report. The equipment used and its calibration date have been recorded on the project file but there is no record of uncertainties associated with the equipment. From the SKB reports (2000; 2009a) it is unclear if the same equipment is used to analyse the copper coupons from all the test parcels (which is unlikely given the timescales involved) or if the same person is performing the tests (some procedures such as the visual analysis are subjective and could be influenced by different investigators).

Through discussion at the review meeting in Stockholm it was identified that the key uncertainty in quantifying the copper corrosion rate is defining the start time of the experiment. The measured coupon weight loss corresponds to the total corrosion that has occurred from the point of coupon creation until final measurement. However, corrosion will have occurred at different rates depending on the conditions the coupon is subject to at any one time. To define the corrosion rate, the length of time the coupon is subject to a set of conditions is key, but it is unclear for the coupons in LOT what start time should be used. For example, the experiment start time could be assumed to be the time at which the parcel is emplaced, the time the applied heat reaches a stable temperature or the time at which the test parcel is fully saturated (if the latter, defining the time of full saturation is also uncertain).

There is some uncertainty regarding the geochemical conditions within the test parcel. The water analysis by VTT (SKB, 2009a, Appendix 5) indicates that reducing conditions are present in the A2 parcel but the estimated corrosion rate is approximately a factor of ten greater than the theoretically calculated corrosion rate of $2x10^{-8}$ m/year for anoxic conditions (Wersin *et al.*, 1994). However, it is possible that most of this corrosion took place under oxic conditions prior to the establishment of anoxic conditions. In addition, as discussed at the review meetings, it is possible that there are differing conditions along the length of the test parcel. Nonetheless, the lack of knowledge of the conditions for each copper coupon does not aid understanding of the calculated corrosion rates. It would aid transparency if the oxygen content in the vicinity of the corrosion coupons was discussed in the technical report when considering the corrosion results obtained.

4.1.3 Analysis of Copper Tubes

Instinctively it would be expected that copper corrosion analyses would be carried out on the large copper tube at the centre of each LOT test parcel, rather than just the four small copper coupons in each parcel. SKB (2000, §8.2) explains that the small copper coupons were used instead of the central copper tube because the coupons were manufactured from the same copper quality as proposed for the real canisters, they could be well characterised and allowed simple sampling for subsequent analyses. SKB (2000, §9.5) comments that a visual inspection of the copper tube and coupons from the pilot parcels before the cleaning treatment did not reveal any significant differences between the surfaces of the copper tubes and the copper coupons.

SKB (2000, §9.5) and SKB (2009, §9.2.2) did report measurements of copper concentrations in the bentonite adjacent to the copper tubes, revealing potential differences in corrosion rates associated with different temperature and saturation conditions, although no definitive explanation of differences was given. It would appear a lost opportunity not to carry out more detailed analysis of the copper tube itself.

4.1.4 Reporting

Both SKB and Bo Rosborg made clear at the review meeting in Stockholm on 11 March 2010 that they did not regard the copper corrosion work as part of the LOT project, but a separate project that uses the conditions available within the LOT parcels. This explains why the copper coupon corrosion analysis results are only presented in the appendix, not the main body, of the A2 parcel report (SKB, 2009a) and why the work on real-time corrosion monitoring (see Section 4.1.5) is not mentioned. However, to those not involved in these experiments, such tests appear part of the LOT project and discussion of them in the LOT technical reports is expected. It would aid traceability if the results of such experiments were included in the LOT report or references were provided to reports in which such work is discussed.

There have been delays in publication of SKB technical reports on the LOT project. This QA review acknowledges the time required to analyse and understand the data obtained both before and after parcel extraction, but timely publication of results is important. Publication of the results for the LOT A0 parcel, extracted in 2001, has been given a low priority by SKB, although, as discussed at the QA review meeting, results have been presented at meetings with SSM. The results of the A2 parcel, extracted in 2006, were not published until the end of 2009. There have been discussions of these experiments and their results at conferences, but such presentations do not justify the delay and/or lack of publicly available SKB reports.

During the LOT copper corrosion review meeting in Stockholm it was made clear that publications in peer-reviewed journals are given a greater weight than SKB technical reports. For example, whilst copper coupon analysis has been recorded by SKB (2000; 2009a), the real-time corrosion monitoring work has only been published in conference proceedings and academic journals[1]. This review recognises the importance of publishing articles in specialised journals to support the evolving body of knowledge, but it is also important that SKB publishes its work in a more comprehensive and easily accessible format. Other stakeholders are unlikely to have easy access to specialised journals and the publication conditions of such journals, in particular limited article length, mean that key technical details and data cannot be

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[1] According to the information provided at the March 2010 review meeting, eight conference presentations have been made by Bo Rosborg since 2001 on the SKB copper corrosion work, with a further four planned for this year. Journal articles published since 2005 include Rosborg and Werme (2008), Rosborg and Pan (2008) and Rosborg *et al.* (2005), with a further four planned for submission this year. During this period, no SKB technical reports have been published on the real-time corrosion monitoring work managed by Rosborg Consulting.

published. It is therefore important that SKB produces regular comprehensive technical reports recording the motivation, procedure, results and interpretation of the experiments they commission. In addition, it was indicated at the Stockholm review meeting that there is no internal SKB review procedure for articles intended for journal submission.

4.1.5 Real-Time Corrosion Monitoring

SKB is evaluating electrochemical techniques for real-time monitoring of copper corrosion in a bentonite/saline groundwater environment. The techniques applied include polarisation resistance, harmonic distortion analysis, electrode impedance spectroscopy, and electrochemical noise techniques (Rosborg and Werme, 2008, §5.4.1).

Although the real-time monitoring in the LOT experiment was discussed at the meeting, insufficient information was available to assess the QA of the work in detail and a QA checklist was not prepared. Instead, a brief summary of the experiment and follow-on tests is provided and several QA issues are identified.

One bentonite ring (36) in the LOT A2 test parcel included three copper electrodes for real-time copper corrosion monitoring (see Figure 4.1). The A2 parcel was emplaced in 1999 and retrieved in early 2006. During this period real-time corrosion measurements were recorded with a commercially available SmartCet corrosion monitoring system using a three-electrode system (Rosborg and Werme, 2008, §5.4.1).

Figure 4.1: Extraction of the LOT A2 test parcel and retrieval of the exposed copper electrodes for further real-time monitoring.

Upon retrieval of the parcel, the bentonite ring containing the exposed electrodes was removed and placed in a bucket, a new copper electrode was added on 20 June 2006 and the bucket sealed with paraffin (see Figure 4.1). This system was then subject to further real-time corrosion monitoring of the copper electrodes. Datalogging is performed automatically using the portable SmartCET apparatus and the data are transferred to a computer. The experiment is being undertaken in collaboration with a team at the Slovenian National Building and Civil Engineering Institute, Ljubljana, Slovenia.

A corrosion rate of 1.5×10^{-6} m/year was recorded on 5 December 2005, just prior to retrieval of the A2 test parcel. The same electrodes indicated a corrosion rate of 2.2×10^{-6} m/year on 9 May 2007 (after placement in the bucket). The new electrodes experienced a corrosion rate of 1.6×10^{-6} m/year on 9 May 2007. The observed corrosion rates suggest that there is oxygen present in the system or, if the environment is anoxic, that some other corrosion process is occurring.

An additional test using an electrical resistance technique has also been implemented in the bucket experiment. The test device consists of thin copper wires printed on a circuit board and placed in the bentonite in the bucket with the other electrodes. As the copper corrodes, the resistance of the wire changes and can be measured, although Bo Rosborg noted that the selection of the wire thickness is important to the success of such a technique due to the small changes to be measured. Whilst one of the four tests appears to be faulty, the remaining sensors display decreasing corrosion rates, which, after 1000 days exposure, have tended to rates of 1.5×10^{-6} m/year (two sensors) and 6.9×10^{-6} m/year (one sensor). Such results are consistent with the results obtained through the first technique.

No SKB reports have been published on this work although a number of peer reviewed journal articles have been produced (e.g., Rosborg and Werme, 2008; Rosborg and Pan, 2008). SKB intends to publish a technical report on this work in 2010.

SKB and Bo Rosborg do not regard these tests as part of the LOT experiment. The copper coupon weight loss measurements were carried out using Studsvik Nuclear facilities and implementing its QA procedures. The electrochemical experiments being carried out do not implement a defined QA system for the project and rely on peer review of journal publications for quality assurance, although the laboratory at Ljubljana is quality assured and the facility itself is a certification body.

During the review, it became apparent that SKB places significant reliance on its contractors; Bo Rosborg has an important role in deciding the direction and scope of the LOT corrosion experiments. The extent to which SKB controls or influences the aims and design of the experiments it funds to ensure that they meet SKB's requirements is not clear.

4.2 MiniCan Experiment

4.2.1 Background

The MiniCan experiment is being undertaken as a further step in developing understanding of the likely performance of the canister in a repository environment. The experiment is focused on obtaining information about corrosion of the cast iron insert and its effects following a leak in the copper canister (SKB, 2009b, p7). As for the LOT experiment, due to the resources invested in such a project and the timescales involved, additional experiments to study copper corrosion have been included.

In the MiniCan experiment, five small-scale model canisters are used to simulate the main features of the SKB canister design (SKB, 2009b). The model canisters consist of outer copper bodies fabricated from 150 mm outer diameter copper tubing, of the same grade of copper as that used for full size canisters, and end caps fabricated from the lid material used for full-scale canister assemblies. The end caps were electron-beam welded and at least one 1 mm defect (a drilled hole) was introduced in each canister in the copper body near the weld area.

Five boreholes, one for each model canister, were drilled at a 10° slope to the horizontal at locations in the Äspö HRL where the groundwater supply is large. Using a support cage, the first three model canisters are surrounded by low density bentonite and the fourth surrounded by high-density compacted bentonite, whilst the fifth canister is exposed directly to unconditioned groundwater. Each canister support cage contains a range of sensors (e.g., reference electrodes, E_h electrodes, copper and iron electrodes), weight loss corrosion coupons (copper and cast iron) and stress corrosion test pieces.

Serco Technical Services (Nick Smart) is leading the MiniCan experiment on behalf of SKB. The copper and cast iron corrosion data are analysed by Serco and microbial activity analysis is undertaken by Microbial Analytics Sweden (Karsten Pederson). Water sampling and on-site experiment monitoring are undertaken by staff at Äspö. There is also a project advisory group, consisting of Serco and SKB staff, and experts as required.

Stage 1, consisting of design work for the MiniCan experiment, started in 2004/05. Stage 2, project procurement and set-up, took place between September 2006 and February 2007, whilst experiment monitoring, forming Stages 3 and 4, extended to 2009. The project is currently in Stage 5, continued monitoring and staged removal of each miniature canister. It is intended that miniature canister 3, with low density bentonite, will be extracted during 2010/11.

4.2.2 Corrosion Analysis

Corrosion Coupons

Five types of corrosion coupon were mounted within the support cage used for the model canister experiments, at the top of the support cage on a nylon support rack (SKB, 2009b, §4.6), as follows:

- Each of the model canister experiments contains plain corrosion coupons of copper and iron so that corrosion rates can be determined through weight loss measurements. No miniature canisters have yet been extracted and so no weight loss measurements are reported.
- Coupons of copper and cast iron electrically connected to the exterior are designed to allow the real-time corrosion potential of the electrodes to be measured. With a platinised titanium gauze electrode used as the counter electrode in a conventional 3-electrode electrochemical cell, it is also possible to carry out electrochemical measurements of the real-time corrosion rate using linear polarisation resistance (LPR), AC impedance (ACI) and electrochemical noise (ECN) techniques.
- Model canisters 2 and 5 contain copper electrical wire resistance probes. These were set up to measure the real-time corrosion rate of copper using a technique proposed by VTT. Each consists of a coiled 112.5 cm length of 1 mm diameter copper wire divided into three sections, the end sections sheathed in heat-shrinkable, adhesive-lined polymer tubing. The screened lengths act as reference resistances and the change in the resistance of the exposed length, processed by an ACM Field Machine electrochemical unit, enables calculation of the corrosion rate.
- To assess stress corrosion, four Wedge Opening-Loaded (WOL) specimens, machined from a copper lid and pre-cracked to give a range of stress intensity factors, were mounted in the boreholes. Four U-bend samples were also manufactured from the same copper lid material. Two of each specimen were mounted in the boreholes for model canisters 3 and 4 by loosely suspending them from the stainless steel push rod using plastic connectors and are therefore exposed directly to the groundwater. The specimens will be examined for stress corrosion cracking upon the removal of each miniature canister.
- To investigate crevice corrosion, galvanic corrosion and expansive corrosion, copper-iron-copper sandwich specimens were mounted on the nylon support rack at the top of the canister support cage. These consist of a sheet of copper clamped against a block of cast iron using a ring of nylon bolts; the cast iron used is the same type as for the model canister insert. The specimens allow investigation of the effect of separation distance between mating surfaces by including a series of steps machined into the surface of the cast iron. The specimens will be examined upon removal of each miniature canister.

The project plans to conduct a detailed examination of all of the materials in the experiment, including the copper canister, when the experiments are extracted from the boreholes.

Uncertainties in Corrosion Measurements

SKB (2009b) does not quantify data uncertainty or discuss the sources of uncertainty. Graphs of results are published without error bars or discussion of the confidence with which the data should be used. Also, the detection limits of the techniques used are not recorded.

A key uncertainty in the MiniCan experiment is the validity of the real-time corrosion rate measurements, which cannot be confirmed until after canister extraction – the electrochemical measurements could be affected by degradation of the electrode insulation. The copper electrode electrochemical measurements could also be affected by the formation of a copper sulphide film. Additionally, there is a potential issue with the electrical resistance measurement sampling frequency, discussed at the review meeting, which appears to affect the results obtained; Serco has discussed this with the equipment manufacturers ACM Ltd and has been informed that this is typical for data obtained with this instrumentation. It would aid transparency if such issues were discussed in the technical report.

Some expected experimental parameters have not been recorded, such as the ground water flow rate in each borehole and the pyrite content of the bentonite. In addition, it was noted in the review meeting that other experiments nearby can disturb the water pressure and chemistry of the MiniCan experiment, although the experimenters believe that the water pressure is more affected by water loss through the tunnel walls. These factors could explain some of the observed differences between boreholes, but their impact has not been quantified.

4.2.3 Reporting

Serco has produced two progress reports for SKB that are retained on the internal project record (published in January 2010 and February 2010). SKB allows access to these reports in person at its offices in Stockholm. It is not clear why these reports cannot be made available for review outside SKB's offices – limited report availability hinders transparency.

Details of the experimental set up and results obtained during the first year of operation, up to May 2008, were reported by SKB (2009b). A Serco progress report is planned for the end of 2010, but a publicly available SKB report is not anticipated until one of the miniature canisters is extracted from its borehole and the associated data analysed. In addition, a conference paper in June 2010 is planned. A paper has been submitted to the 4th International Workshop on Long-Term Prediction of Corrosion Damage in Nuclear Waste Systems, to be held in Brugge during June 2010.

Generally, SKB (2009b) reports the MiniCan experimental set-up well. However, the QA review has identified concerns regarding the publication of data. SKB (2009b) purports to present the results of the MiniCan experiment up to May 2008.

Figure 6-33 of SKB (2009b) shows the corrosion rates obtained by AC impedance and LPR measurements for four of the miniature canisters; data from canister 2 are excluded from the graph without explanation. The presented corrosion rate data show some scatter but the maximum value is around $4.5x10^{-6}$ m/year. However, during the March 2010 review meeting, it became clear that additional corrosion monitoring data were available for these canisters and for canister 2 prior to May 2008, but these data had not been reported in the publicly available SKB technical report. The missing data indicated corrosion rates of up to $500x10^{-6}$ m/year for canister 2 and up to $15,000x10^{-6}$ m/year for canister 4. Data obtained post-May 2008 show corrosion rates that are several orders of magnitude higher than expected values for all but canister 5.

Clearly such high corrosion rates suggest that there are problems with the measurement technique. Degradation of the electrode insulation and formation of a copper sulphide film have been suggested by the experimenters as potential causes of the unexpected results. However, it will not be possible to investigate the issue further and confirm corrosion rates until the canisters are extracted and weight loss coupons can be analysed and sensor equipment checked.

The technical report (SKB, 2009b) gives no indication that only selected data were published. Indeed, it is stated clearly in executive summary of the report that "*the copper corrosion rate had a maximum value of 3.5 μm/year, which is consistent with data reported in the literature*" (SKB, 2009b, Executive Summary). This statement is not supported by all of the experimental data obtained up to May 2008.

Although all of the data were presented in the Serco progress reports to SKB, it is unclear how or why the decision to exclude the high corrosion rate data from the publicly available SKB report was made (no record has been kept on the project file), but this decision does not display scientific best practice. It would have been more appropriate for the full data set to have been published, accompanied by a discussion of the reliability of the data, uncertainties, potential faults with the measurement technique, and the need for further analysis.

It was stated at the March 2010 QA review meeting that an expert advisory group was formed to advise the project, although the existence of this group is not mentioned in the technical report (SKB, 2009b). Details of the advisory group meetings, along with the Serco progress reports, are saved in the project file and are viewable at SKB's Stockholm office. The group has met twice during the course of the project so far, although no record of the first meeting was kept by SKB. Notes of the second meeting, held on 22 June 2009, show attendees included Nick Smart (Serco), Andrew Rance (Serco), Christina Lilja (SKB), Lars Werme (SKB), Ola Karnland (Clay Technology), Fraser King (Integrity Corrosion Consulting Ltd), Claes Taxén (Swedish Corrosion Institute), and Karsten Pedersen (Microbial Analytics Sweden and Göteborg University). The record indicates that the advisory group discussed the fact that the electrochemical measurements showed corrosion rates that were much higher than expected and they concluded that results reflected apparent corrosion due to a sulphide film on the surface of copper. The meeting record included actions, but none were noted regarding the high corrosion rate observations.

Whilst this QA review recognises the relatively early nature of the real-time corrosion data and that they are subject to validation using the weight loss coupons following canister extraction, there is a lack of explanation and discussion of the results and their meaning. Such discussion would ensure that potentially erroneous data are not used out of context and would clearly indicate where there are problems and uncertainties that have yet to be clarified.

A number of (minor) mistakes are present in the technical report (SKB, 2009b), such as stating that the corrosion rate was less than 3.5×10^{-6} m/year in the executive summary, whilst Figure 6-33 displays corrosion rates of up to 4.5×10^{-6} m/year and including negative data points in a graph of total organic content (Figure 6-18).

5 Conclusions

SKB intends to submit the SR-Site safety assessment to SSM as part of an application to construct a spent nuclear fuel repository at Forsmark. SSM has identified a need to review quality-related aspects of some of the many tests and experiments instigated by SKB to obtain the data that are used to abstract or support conceptual understandings of repository evolution. Such QA reviews provide insights into the level of confidence and reliability that can be assumed in the data that underpin the safety assessment.

The copper canister provides an important corrosion-resistant barrier in SKB's KBS-3 spent fuel disposal concept. Reliable copper corrosion data are required to support demonstrations of safety of the KBS-3 concept. SKB's experiments on copper corrosion are of particular interest because some independent researchers have questioned SKB's understanding of corrosion processes under the anoxic conditions that are expected to persist in the repository in the long term. Therefore, SSM commissioned GSL to undertake QA reviews of some of SKB's experiments on copper corrosion.

The copper corrosion tests that form part of the LOT and MiniCan experiments that are being conducted at the Äspö Hard Rock Laboratory were selected for review. Visits to the Hard Rock Laboratory in December 2009 and to SKB's offices in Stockholm in March 2010 provided opportunities to discuss QA aspects of these corrosion tests with SKB staff and contractors. LOT and MiniCan project reports and publications were also studied as part of the review process. Consistent with previous QA reviews of SKB's experiments, a checklist of quality-affecting issues was prepared to facilitate and document the review, covering the framework, design, conduct, analysis and reporting of experiments, and the use of experimental results in the KBS-3 repository research programme. Conclusions of the QA review are presented below.

5.1 Quality Assurance

The review found that both the LOT and MiniCan projects are being conducted under SKB's management procedures according to appropriate project and activity plans. Both projects are being led by suitably qualified contractors, who have produced project QA plans approved by SKB. The contractors undertake project work under their own accredited quality management systems, which include appropriate data management and reporting procedures. This approach to QA is consistent with approaches adopted by SKB for other tests and experiments currently being undertaken at the HRL, as noted in previous QA reviews conducted by GSL on behalf of SSM (and previously SKI).

5.2 Design of Experiments

The copper corrosion tests that form part of the LOT and MiniCan experiments are subsidiary tests to already planned experiments to investigate other processes. Experiments whose sole aim is to study copper corrosion in a repository-like environment would avoid the potential complication, constraints or influence of tests of other processes in the same experiment.

SKB presented a list of ongoing and future experiments that include copper corrosion tests (see Section 3.3), including future experiments dedicated to understanding copper corrosion processes. It is noted that there are a number of disparate projects, and work is spread across different organisations and countries, contractors and sub-contractors. Potentially, communication of SKB's requirements for these experiments, including QA requirements, and control and monitoring of progress could be hampered by such a diverse programme.

It is apparent that significant reliance is placed by SKB on its external consultants for determining the scope of the copper corrosion experiments reviewed here. Whilst it is important that the knowledge of external experts is sought and utilised, it is also important that SKB fully understands the work carried out on its behalf and that it is of direct support to SKB's objectives. The extent to which SKB controls or influences the aims and design of some of the experiments it funds to ensure that they meet SKB's requirements is not clear.

5.3 Data Reporting Issues for the MiniCan Experiment

The QA review has raised a significant concern regarding lack of transparency in data publication for the MiniCan experiment. As discussed in Section 4.2.3, the MiniCan technical report (SKB, 2009b) presents only selected real-time corrosion monitoring data up to May 2008. The presented data show some scatter but the maximum value is around 4.5×10^{-6} m/year and the report concludes, somewhat erroneously, that "*the copper corrosion rate had a maximum value of 3.5 µm/year*" (SKB, 2009b, Executive Summary). However, during the March 2010 review meeting, it became clear that additional corrosion monitoring data were available prior to May 2008, but these data had not been reported in the publicly available SKB technical report. No indication was given in the SKB technical report that selected data had been excluded. The missing data indicated copper corrosion rates of up to 500×10^{-6} m/year for one canister and up to $15{,}000 \times 10^{-6}$ m/year for another. Data obtained post-May 2008 show corrosion rates for most canisters that are several orders of magnitude higher than expected values.

Clearly such high corrosion rates suggest there are problems with the measurement technique, perhaps associated with degradation of the electrode insulation or formation of a copper sulphide film. However, it will not be possible to investigate the issue further and confirm corrosion rates until the canisters are extracted and weight loss coupons can be analysed and sensor equipment checked.

Although all of the data were presented in the Serco progress reports to SKB, it is unclear how or why the decision to exclude selected data from the publicly available SKB report was made (no record has been kept on the project file), but this decision does not reflect scientific best practice. It would have been more appropriate for the full data set to have been published, accompanied by a discussion of the reliability of the data, uncertainties, potential faults with the measurement technique, and the need for further analysis.

The copper corrosion tests in the reviewed experiments aimed to confirm SKB's understanding of corrosion rates in a repository-like environment. The review has noted that researchers infer that higher than expected corrosion rates reflect problems with the experiment. However, it is unclear how SKB would respond if it is shown that the corrosion rates are greater than hypothesised.

5.4 Analysis of Uncertainties

In general, the reports from the MiniCan and LOT experiments provide little information on the sources or quantification of data uncertainty, or the level of confidence that can be assumed in the results. Whilst it is recognised that sources of uncertainty may not be easily quantifiable, they should be discussed and considered so that an understanding can be gained of the confidence in the presented data, and the areas of greatest uncertainty. Factors that influence data uncertainty should be identified, such as measurement detection limits, the problems in defining the length of time a sample is subject to certain geochemical conditions, or instrumentation problems, such as electrode degradation.

Understanding when conditions are oxic and when they are anoxic is of key importance in real-time copper corrosion tests; it will be difficult to interpret corrosion measurements and long-term corrosion rates unless the evolution of geochemical conditions is understood. It was not clear, in this review, how well redox conditions are understood in the vicinity of the copper corrosion tests in the MiniCan and LOT experiments.

5.5 Publication and Use of Results

There have been delays in publication of SKB technical reports on the LOT project. This QA review acknowledges the time required to analyse and understand the data obtained both before and after parcel extraction, but timely publication of results is important. Publication of the results for the LOT A0 parcel, extracted in 2001, has been given a low priority by SKB, although results were presented at the QA review meeting and have been provided at other SKB meetings with SSM. The results for the A2 parcel, extracted in 2006, were not published until the end of 2009. There have been discussions of these experiments and their results at meetings and conferences, but such presentations do not justify the delay and/or lack of publicly available SKB reports.

SKB stated during the QA review that it gives greater weight to publications in peer-reviewed journals than to SKB technical reports. For example, whilst LOT copper coupon analysis has been recorded by SKB (2000; 2009a), the real-time corrosion monitoring work has only been published in a number of conference proceedings and academic journals. The importance of publishing articles in specialised journals to support the evolving body of knowledge is recognised, but it is also important that SKB publishes its work in a more comprehensive form in easily accessible format. Other stakeholders will not have easy access to specialised journals and the publication conditions of such journals, in particular limited article length, mean that key technical details and data cannot be published. It is therefore important that SKB produces regular comprehensive technical reports recording the motivation, procedure, results and interpretation of the experiments they commission. In addition, there appears to be no clear internal SKB review procedure for articles intended for journal submission.

During the review meetings, SKB stated that the copper corrosion experiments are intended to aid understanding and to verify corrosion rates, and will not be used directly in the safety assessment. The lack of direct input to the safety assessment should not lessen the significance of the results obtained or enable experimental uncertainties to be overlooked.

5.6 SSM's Research on Copper Corrosion

SSM has continued to maintain an awareness of issues and uncertainties regarding copper corrosion processes under repository conditions. In 2010, SSM decided to finance three experiments concerned with copper corrosion in anoxic environments, with the aim of enhancing SSM's knowledge of the subject. However, SKB remains responsible for acquiring the information on copper corrosion needed to support its repository safety assessment.

6 References

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Appendix A - QA Reviews of SKB’s Experiments

Quality assurance (QA) reviews of two of SKB’s copper corrosion experiments have been carried out: the reviews considered the LOT project copper corrosion tests and the MiniCan experiment. The reviews were based on meetings at SKB’s Hard Rock Laboratory (HRL) at Äspö on 1st December 2009 and at SKB’s offices in Stockholm on 10th and 11th March 2010. Information was also extracted from SKB’s reports on the experiments.

A.1 Long Term Test of Buffer Material (LOT Project)

The LOT project was primarily developed to investigate bentonite buffer properties in a repository-like environment. However, considering the resource invested in such an experiment and the time required for test conditions to develop, further investigations to consider copper corrosion, cation diffusion and bacterial behaviour were combined with the primary experiment. Quality assurance in the LOT project with respect to the bentonite elements of the experiment was reviewed and reported by Hicks (2007). The information reported in Table A.1, below, considers quality assurance with respect to the copper corrosion tests that form part of the LOT project.

Table A.1: Copper corrosion tests as part of the long term test of buffer material (LOT project) at the Äspö Hard Rock Laboratory.

1. Framework of Experiment	
1.1 Purpose and objectives	
What is being investigated?	The main LOT tests are investigating bentonite buffer properties and mineral stability in a repository-like environment. However, the LOT configuration provided an opportunity to include a number of supplementary tests to investigate other processes (copper corrosion, cation diffusion and bacterial behaviour). This quality assurance checklist is concerned with the copper corrosion tests.
What experiment is being undertaken?	Experiments are being undertaken in which copper tubes containing heater elements are surrounded by bentonite blocks and placed in boreholes at Äspö. There are two types of experiment in which the bentonite and copper tube “parcels” are exposed to different conditions: - standard or S-parcels (S1, S2, and S3) are exposed to expected repository conditions, - adverse or A-parcels (A0, A1, A2, and A3) are exposed to adverse repository conditions with the aim of accelerating reactions. Figure A.1 shows a schematic illustration of the A2 test parcel that indicates the “additives” and sensors embedded in the bentonite blocks. The additives include copper coupons, ^{60}Co tracers, bacteria and chemicals. The pure copper coupons, manufactured by milling with one side then polished, had nominal dimensions of 60 x 15 x 1.5 mm (SKB, 2009a, p147). The copper coupons in the A2 test parcel were placed in bentonite rings 22 and 30.

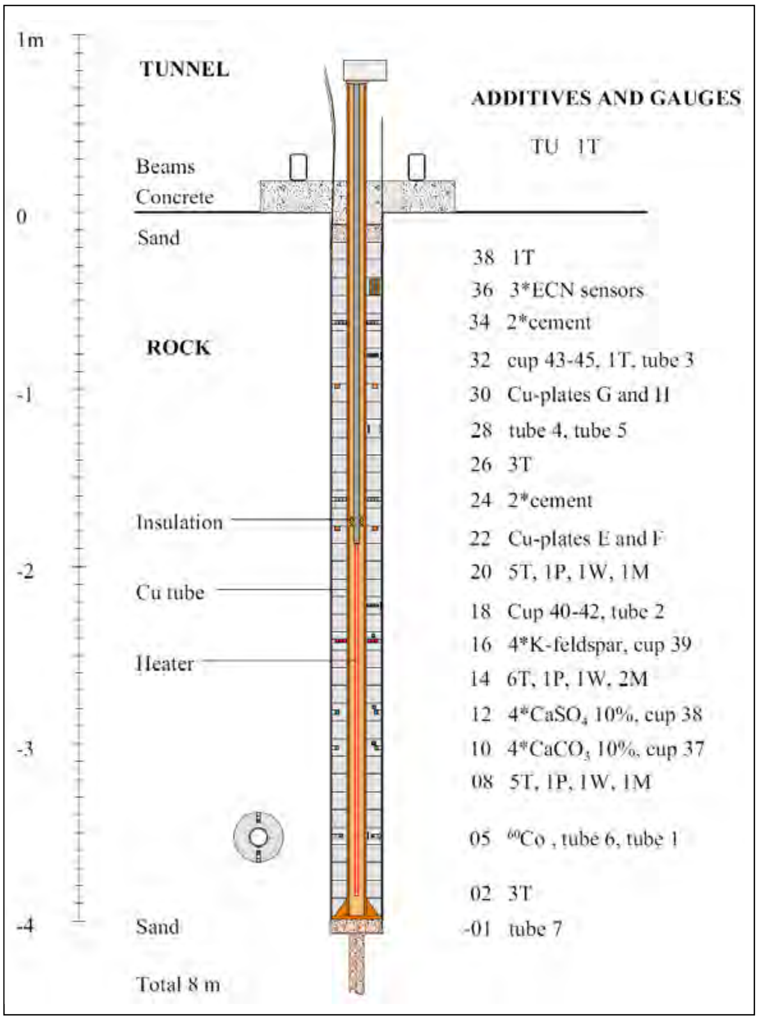

Figure A.1: Schematic drawing of the A2 test parcel (SKB, 2009a, Figure 3-3); copper test coupons (plates) are placed in bentonite blocks 22 and 30.

Additional groundwater from a dedicated borehole in the experiment vicinity was added through a small titanium filter tip in the upper part of the test parcel. Full saturation of the A2 test parcel was expected after less than one year with free access to water but moisture and pressure results suggest full water saturation was reached within two years (SKB, 2009a, p147).

At the end of each experiment, the test parcels are extracted and various laboratory tests are performed on the bentonite blocks (measurements of bentonite properties, tracer analysis, analysis of bacteria populations, and measurements of copper coupon corrosion).

Twelve copper coupons were used for the pilot study: four in S1, four in A1 and four were retained as a reference. Supplied by Outokumpu Poricopper OY, Finland, the copper specification for the coupons was chosen in order to correspond to the copper proposed for the canister material (SKB, 2000, §8.2). The A1 bentonite blocks containing the copper coupons were destroyed during extraction of the parcel and so were not analysed.

	For the A2 parcel, after the bentonite rings containing the copper coupons were cut apart from the rest of the parcel, they were immediately wrapped in plastic sacks which were evacuated using a vacuum pump and then transported to Studsvik Nuclear AB and stored. During the cutting process the copper coupons in bentonite ring A222 were damaged by the cutting wheel during retrieval, preventing accurate assessment of the coupon corrosion rate (SKB, 2009a, p147). At Studsvik, the coupons were removed from the bentonite rings in a step-wise fracturing process. This was then immediately followed by cycles of photographing the coupons, scraping and cleaning, performing scanning electron microscopy (SEM), and drying and weighing (SKB, 2009a, p148). The copper coupons were then stored in a desiccator.
Why is the experiment being undertaken?	The aims of the copper corrosion tests are to verify that the mean copper corrosion rate is less than $7x10^{-6}$ m/year under initial oxic conditions (as indicated by corrosion modelling) and to identify possible pitting and corrosion products (SKB, 2000; SKB 2009a).
What is the role of the experiment in the repository programme?	The data obtained from the copper corrosion tests will not be used directly in the SR-Site safety assessment, but the findings from the analysis of the retrieved parcels (A0, A1, A2 and S1) support SKB's understanding of copper corrosion rates and mechanisms under oxic conditions.
1.2 Resources and schedule	
Where is the experiment being conducted?	SKB's Hard Rock Laboratory at Äspö near Oskarshamn.
Who is conducting the experiment?	Clay Technology (Ola Karnland) is leading the LOT experiment on behalf of SKB and Posiva. The analyses of each recovered parcel have been undertaken by various organisations: • Bentonite mineralogy and physical properties – Clay Technology AB • Pore water chemistry – VTT (Finland) • Bacterial behaviour – University of Gothenburg and Microbial Analysis Sweden AB • Cation diffusion – Royal Institute of Technology (KTH) • Copper corrosion – Rosborg Consulting (using Clay Technology and Studsvik Nuclear AB facilities) Bentonite mineralogy/chemistry for the A2 parcel was also analysed by independent laboratories: BGR in Germany, University of Bern in Switzerland, and G2R Laboratory and LEM from Nancy University in France. This additional work was financed by the collaborating organisations BGR, Nagra and Andra, respectively.
What is the schedule for the experiment?	Two one-year pilot tests (A1 and S1) were conducted in 1997 and 1998. Work on five more tests (S2, S3, A0, A2 and A3) was started in 1999 and parcel A0 was retrieved after one year in 2001. Parcel A2, emplaced on 29 October 1999, was retrieved in early 2006 after just over six years of operation. The remaining parcels (A3, S2 and S3) are still in place and a LOT project meeting will be held during the spring of 2010 to discuss their extraction.
When will results be available?	The results of the two pilot tests (A1 and S1) were reported by SKB (2000). The results of the analysis of parcel A0 remain under review. The results of the analysis of parcel A2 were recently reported by SKB (2009a).

What constraints do resources such as cost and timing place on experimental planning and design?	The time for resaturation constrains the scale of the test; full saturation would not occur in one year in a full-scale test. The smaller parcel size also facilitates extraction of the parcels in one piece. The copper coupon tests were designed to take advantage of the opportunity offered by the LOT project to study copper corrosion under repository conditions. The design of the coupons and the number of coupons used were constrained by the availability of bentonite blocks, given the need to accommodate other tests and gauges. SKB considered that the use of four copper coupons in two different bentonite blocks within each test parcel would be sufficient for the purpose of verifying copper corrosion behaviour.
1.3 Quality assurance	
What QA system and standards are used in the planning, design, execution, analysis, and reporting of the experiment?	The early LOT experiments were not performed under SKB's present QA process. Work is now carried out under SKB's management procedures according to project and activity plans. For example, extraction of the A2 parcel was carried out in accordance with activity plan SKB AP TD F62-06-012 (SKB, 2009a, p147). SKB describes the requirements for any work to be carried out by contracting organisations. The contractor provides a QA plan for the work, which is discussed and approved by SKB. Once the work is finished, SKB confirms with the contractor that the work was carried out according to the QA plan. This process was followed for the A2 copper corrosion measurements undertaken by Rosborg Consulting (SKB, 2009a). For the work financed by collaborating organisations BGR, Nagra and Andra, the contracting organisations are given access to LOT materials in order to undertake their analyses, but their procedures are not reviewed; appropriate QA procedures are assumed to be implemented. Copper coupon measurements carried out at Studsvik AB's laboratory followed Studsvik's QA plan, which has ISO 9001 accreditation.
How is the expert team selected/trained for the experiment?	SKB proposed the research groups to work on the experiments and asked Clay Technology (Ola Karnland) to design the experiments. Selection of the team was based on experience and skills known to be available at the research groups.
2. Design of Experiment	
2.1 Variables	
What are the dependent variables (i.e. those being observed)?	During the experiment the water content, water pressure, total pressure, and temperature distributions in the bentonite are monitored. The extent of copper corrosion under the observed conditions is measured following extraction of the coupons from the bentonite blocks. The temperature measurements are important with regard to interpreting the copper corrosion test results.
What are the independent variables (i.e. those that are varied to cause change in the dependent variables) and how are their values selected?	Heat sources maintain the copper tube surface temperature at about 90°C (S-parcels) and about 130°C (A-parcels), and generate a temperature gradient across the bentonite. The two A2 bentonite blocks containing the copper coupons were exposed to temperatures of 30°C (A2, ring 30) and 75°C (A2, ring 22); coupons in S1 were exposed to temperatures of 25°C (S1, ring 30) and 50°C (S1, ring 22). Initially the temperature was set using a temperature control, before changing to input power control. The emplacement positions of the copper coupons in the parcels determine the temperature that each coupon experiences. Full temperature lasted from September 2000 to 5 December 2005. Therefore, the coupon exposure time in the bentonite was more than 6 years, whilst the time exposed at full

	temperature was 5 years and 3 months (SKB, 2009a, p147). The groundwater pressure in the rock and the rate of inflow to the copper coupons in the bentonite vary depending on conditions local to each test hole, resulting in different saturation levels around each coupon. The oxygen and chloride content of the bentonite around the coupons are also independent variables, although not controlled, and there is no knowledge of the rate of oxygen depletion.
What are the control variables (i.e. those that are held constant) and how are their values selected?	The same batch of MX-80 bentonite was used to produce each block used in each parcel. Therefore, similar initial pyrite concentrations were present in each block containing copper coupons.
2.2 Experimental techniques	
What experimental techniques and instruments are being used?	Experimental procedure: - the parcels, each comprising a copper tube surrounded by bentonite blocks, were lowered into 4-m long, 30-cm diameter boreholes; - the copper tubes contain heater elements over the lower 2-m length of the borehole (e.g., 600 W in the S1 parcel, 1000 W in the A1 parcel and 2000 W in the A2 parcel); - copper plates, cement, tracers (^{134}Cs and ^{60}Co), bacteria or additives were included in some bentonite blocks; - about 40 sensors (relative humidity, water pressure, total pressure and temperature sensors) were placed at different locations in the bentonite blocks in each parcel to allow continuous monitoring; - the system was pressurised until the end of the experiment, being fed with water from a nearby fracture. Parcel extraction: - the pilot parcels A1 and S1 were extracted using core drilling, but this required water cooling which flushed away some of the A1 bentonite and, therefore, parcels A0 and A2 were extracted using percussion drilling; - the bentonite rings containing the coupons were cut from the test parcels and were wrapped in plastic sacks from which the air was evacuated (due to cutting through some coupons and destroying them at this stage, instructions for later test parcel extractions will require that the cutting leaves more bentonite around the coupon); - the samples were transported to Studsvik and stored prior to analysis; Analysis of copper coupons (SKB, 2009a): - the bentonite blocks were cut apart or fractured to extract the copper coupons; - copper corrosion was analysed by SEM, EDS, XRD, microscopy and weighing; - measurements were made of the copper corrosion rate and type (pitting and uniform), the corrosion product formed, and the copper distribution in the bentonite. Note that the copper tube in each parcel does not form part of the experiment. Traces of copper corrosion products in the bentonite blocks next to the copper tube have been measured using ICP/MS, but these observations have not been used to estimate copper corrosion rates.

Are they standard techniques?	A lot of the equipment was newly constructed. The heaters were specially designed. Generally, standard components and sensors have been used, although titanium was used instead of the usual steel in some sensors to avoid corrosion. The laboratory copper coupon analysis methods are standard.
Are acceleration methods used?	The parcel diameter is smaller than in a canister deposition hole to shorten the resaturation time.
Have the techniques been validated and documented?	Results of the two pilot tests (A1 and S1) guided the design of the later tests. Descriptions, results and analyses of the pilot tests are provided in SKB (2000) and the results of the A2 parcel are documented in SKB (2009a). The results of the A0 parcel test have not yet been published.
Are the techniques being used under normal conditions?	Equipment such as sensors is used under normal conditions and is expected to be reliable. However, some sensors have failed (including relative humidity sensors). The copper corrosion analysis techniques performed at the laboratory, after parcel extraction, are used under normal conditions.
Has equipment been calibrated and checked?	The copper tubes were checked for leaks when sealed. Equipment is calibrated before use and checked after use.
2.3 Uncertainty	
What are the key uncertainties in the experiment?	A key uncertainty is the timescale required to achieve the resaturated conditions needed to verify the chemistry model. The amount and distribution of oxygen in the system and the timescale for consumption of the oxygen are also important uncertainties for the copper corrosion analysis. It is thought that the warmest parts of the system are the last to be saturated and therefore the last to contain a gas phase (SKB, 2009a).
2.4 Risks to success of experiment	
What are the risks to the success of the experiment and how are they mitigated?	The key risks to success of the corrosion analysis in the LOT experiment are: - Lack of control of resaturation during the LOT test. Rapid resaturation is preferred and water is fed throughout the experiment to the parcel. Saturated conditions are essential for verification of the chemistry model. - Risk of equipment failure (e.g., temperature control and/or sensor). Alarms are used in the monitoring system with associated response actions. - Damage to the copper coupon during its extraction from the bentonite block. All four copper coupons in the A1 parcel were damaged during the extraction process and were not analysed. Two coupons in the A2 parcel were damaged, although a partial analysis was undertaken of one of them. - Disturbances to final conditions after parcel extraction. In particular, the coupons could be exposed to oxic conditions resulting in further corrosion after extraction of the parcels. To reduce the opportunity for such corrosion, the copper coupons are retained in the saturated bentonite blocks. The blocks are only exposed for 10 minutes after extraction before being sealed in a nitrogen-rich environment and then transported to the laboratory. Generally, the corrosion analysis takes place within a month, although there is no maximum time in the QA plan. The coupons are extracted from the bentonite in the laboratory. Staff turn-over is also a potential risk, but experiments are well documented to mitigate against this risk.

What are the critical decisions in the experiment?	The critical decision is when to terminate the tests.
Is there duplication in the experiment?	There is duplication in blocks and between blocks - experiments are always over-specified. Copper coupons were placed in pairs in the bentonite blocks.
3. Conduct of Experiment	
3.1 Data collection and quality control	
How are data collected?	Data are recorded hourly and data collection is also event-triggered. The commercial and widely-used Orchestrator data acquisition software is used, which was checked at installation.
How are data stored (e.g., filing, indexing)?	Data are stored on a local project computer, with monthly transmission to Clay Technology. Clay Technology processes the data using Microsoft Excel, stores the data on CD-Rom and submits it to the SKB SICADA database. An indexing system is used for identifying tests, sensors, bentonite blocks and bentonite test sample locations. The raw copper corrosion measurement data were recorded on a Microsoft Excel spreadsheet and then entered on the SICADA database.
How are data checked (e.g., independently)?	Data collection is checked using a monitoring system with alarm functions. Data are checked by two independent SKB reviewers and Clay Technology (Ola Karnland) must approve the data before it can be entered into the SICADA database. No independent measurements are made.
How are data backed-up?	Regular backups are made onto a separate hard disk.
What quality control procedures are used?	Non-conformance reports are prepared when deviations occur. Quality checks are made on data entered into the SICADA database.
3.2 Records of experiment	
Are notebooks being used for the experiments?	Field notes, daily logs and database entries are made for the LOT Project. The copper coupon analysis was recorded in a Microsoft Excel spreadsheet directly and log books are held by Bo Rosborg.
Are notebooks checked independently?	No.
Are planning, execution and analysis correspondence kept (e.g., emails)?	Important correspondence is kept and stored by Clay Technology or at Äspö. Activity plans have been used for extraction of the A0 and A2 test parcels. The SKB document handling system gives every document a unique number.
Are copies of records kept?	No.
3.3 Equipment	
Is equipment tested, inspected, and maintained?	The copper coupon corrosion analysis was performed at Studsvik Nuclear facilities and the equipment there is tested and re-calibrated as required.

4. Analysis and Reporting of Experiment	
4.1 Data interpretation	
What data interpretation methods are being used (models, software packages, model simplifications)?	The measured coupon weight loss from the copper coupons has been used directly to infer the copper corrosion rate during the LOT test.
How are uncertainties and sensitivities analysed?	The copper corrosion analysis has been reported without any associated error discussion. It was identified at the review meeting in Stockholm that the key uncertainty in quantifying the copper corrosion rate is defining the start time of the experiment. Corrosion begins as soon as the coupon is produced and the measured coupon weight loss corresponds to the total corrosion that has occurred from the point of coupon creation until final measurement. However, corrosion will have occurred at different rates depending on the conditions the coupon is subject to at any one time. To define the corrosion rate the length of time the coupon is subject to a set of conditions is key, but it is unclear for the coupons in LOT what start time should be used. For example, the start time could be when the parcel is emplaced, as soon as the applied heat reaches a stable temperature or when the test parcel is fully saturated (if the latter, defining the time of full saturation is also uncertain). This uncertainty illustrates the potential benefits of successful real-time corrosion monitoring.
4.2 Reporting and review	
How are data and observations reported?	Many documents have been produced and are listed in a project document chart (an internal SKB document). Results have been published in scientific journals and in two PhD theses. SKB (2000) contains observations from the pilot tests and SKB (2009a) records the analysis of parcel A2. Results for the A0 parcel are yet to be published.
How are interpretations reported?	As above. SKB (2000) contains interpretations from the pilot tests and SKB (2009a) for parcel A2.
How are limitations on the use of data and results reported?	Limitations on the use of the data have not been discussed. However, as the copper corrosion tests are a verification experiment, the data will not be used directly in the SR-Site safety assessment calculations. The understanding derived from this work will be discussed in the SR-Site performance assessment.
How are reports reviewed (e.g. independently)?	Reports are reviewed and approved by SKB. An expert peer review of the A2 parcel report was performed and the review comments are recorded on the project folder. If contractors wish to publish data from SKB-funded work in peer reviewed journals or make conference presentations, SKB does not have a formal review process, although they may make comments on draft manuscripts.
How are review results managed/responded to?	The comment response process is managed by SKB.

5. Usability of Results	
5.1 Verification	
How are experimental outcomes checked against requirements of the experiment?	SKB performs checks and decides on whether further studies are required. The LOT copper coupon tests and weight loss measurements have provided the required information on copper corrosion. New copper corrosion experiments are underway and others are planned for the future, including laboratory experiments in which greater controls are exerted on conditions such as redox potential.
How are experimental results verified?	Observations are compared with expected results, such as from laboratory experiments or published data. The corrosion rates have also been compared with the model and calculated corrosion rates reported by Wersin *et al*. (1994).
5.2 Use of results	
How are results abstracted for use in the repository programme?	The experiments are being analysed or are ongoing. Whilst measurements such as the bentonite swelling pressure may be used directly in the future SR-Site safety assessment, the copper corrosion data obtained will only be used to support corrosion process understanding and verify a corrosion rate under oxic conditions of less than 7×10^{-6} m yr^{-1}.
Are results extrapolated for use on repository length and time scales?	The results are assumed to apply to repository time and length scales, although not directly.
What checks are made that data and results are used appropriately and within prescribed limitations?	The results are used to verify and validate an existing model. The model must be used appropriately. It would be made clear to anyone requesting the data that they should be cautious in their use and understand the uncertainties.

A.2 Miniature Canister (MiniCan) Experiment

The MiniCan experiment was designed to examine how corrosion of the cast iron insert would develop if a defect were present in the outer copper canister. However, measurements of copper canister corrosion and other copper corrosion measurements are included in the experiment. The information reported in Table A.2, below, considers quality assurance in the MiniCan experiment.

Table A.2: Copper and cast iron corrosion tests in the Miniature Canister (MiniCan) Experiment at the Äspö Hard Rock Laboratory.

1. Framework of Experiment	
1.1 Purpose and objectives	
What is being investigated?	The main aim of the work is to examine how corrosion of the cast iron insert would develop if a defect were present in the outer copper canister.
What experiment is being undertaken?	The experiment uses five small-scale model canisters that simulate the main features of the SKB canister design (SKB, 2009b). The model canisters consist of an outer copper body fabricated from 150 mm outer diameter copper tubing, of the same grade of copper as that used for the full size canisters, and end caps fabricated from the lid material used for full-scale canister assemblies. The end caps were electron-beam welded and at least one 1 mm defect (a drilled hole) was introduced in each canister in the copper body near the weld area. Five boreholes, one for each model canister, were drilled at a 10° slope to the horizontal at locations in the Äspö Hard Rock Laboratory with a plentiful supply of water. Using a support cage, the first three model canisters are surrounded by low density bentonite and the fourth with high-density compacted bentonite, whilst the fifth canister was exposed directly to unconditioned groundwater. Each canister support cage contains a range of sensors (e.g., reference electrodes, E_h electrodes, copper and iron electrodes), weight loss corrosion coupons (copper and cast iron) and stress corrosion test pieces (U-bend and Wedge Opening-Loaded (WOL) specimens). Two canisters are monitored using strain gauges. Plastic spacers are used between the steel support cage and the copper canister surface to prevent galvanic corrosion.
Why is the experiment being undertaken?	Reviews of the SR 97 safety assessment (SKB, 1999), which introduced the pin-hole canister failure model, identified a need for greater understanding of the processes involved. Subsequent mechanical modelling considered the production of bulges in the canister due to the creation of solid corrosion products but this required a number of assumptions about the properties of the corrosion products (Review meeting on 10/03/10). Whilst laboratory research has been carried out in this area, knowledge of the behaviour of the canister in a realistic repository environment is also required. Therefore, the MiniCan experiment is being undertaken as a further step in developing understanding of the likely performance of the canister in a repository environment, in order to obtain information about the internal canister corrosion evolution as a result of a leak in the canister (SKB, 2009b, p7). This is to address issues such as (SKB, 2009b, p7; Review meeting on 10/03/10): • Does water penetrate into the annulus through a small defect? • How do corrosion products spread around the annulus from the

	leak point? • Does the formation of corrosion products in a constricted annulus cause any expansive damage to the copper canister? • What is the effect of water penetration on the insert lid seal? • Is there any detectable corrosion at the copper welds? • Are there any deleterious galvanic interactions between copper and cast iron? • Does corrosion lead to failure of the lid on the iron insert? • Are there any effects of microbial corrosion on the canister? • What are the corrosion rates of copper and cast iron? • What is the risk of stress corrosion cracking of the copper?
What is the role of the experiment in the repository programme?	The experiments are to support understanding of the canister and insert corrosion behaviour.
1.2 Resources and schedule	
Where is the experiment being conducted?	Initial set up and sensor validation work was carried out in the Serco laboratory at Culham, UK, prior to installation of the experiments at at Äspö. Conducting the experiment at Äspö subjects the model canisters to realistic oxygen-free groundwater and natural microbial populations.
Who is conducting the experiment?	Serco Technical Services (Nick Smart) is leading the MiniCan experiment on behalf of SKB. The copper and cast iron corrosion data are analysed by Serco and microbial activity analysis is undertaken by Microbial Analytics Sweden (Karsten Pederson). Water sampling and on-site experiment monitoring are undertaken by staff at Äspö.
What is the schedule for the experiment?	Stage 1, consisting of design work for the MiniCan experiment, started in 2004/05. Stage 2, project procurement and set-up, took place between September 2006 and February 2007, whilst experiment monitoring, forming Stages 3 and 4, extended to 2009. The project is currently in Stage 5, continued monitoring and staged removal of each miniature canister. It is intended that miniature canister 3, with low density bentonite, will be extracted during 2010. The experiment is flexible and the project plan is updated every 1-2 years to reflect current requirements.
When will results be available?	Serco has produced two progress reports for SKB that are retained on the project record (published in January 2010 and February 2010). SKB allows access to these reports in person at their offices in Stockholm. Details of the experimental set up and results obtained during the first year of operation, up to May 2008, were reported by SKB (2009b). A Serco progress report is planned for the end of 2010, but a publicly available SKB report is not anticipated until one of the miniature canisters is extracted from its borehole and the associated data analysed. In addition, a paper has been submitted to the 4th International Workshop on Long-Term Prediction of Corrosion Damage in Nuclear Waste Systems, to be held in Brugge during June 2010.
What constraints do resources such as cost and timing place on experimental planning and design?	Cost and the available space at Äspö constrain the number of miniature canisters. The space available for instruments in each support cage also limits the range of measurements that can be made and means each canister does not include exactly the same combination of tests. In addition, the length of time required to plan and operate such an experiment means the design of the miniature canisters differs in some

	aspects from that of the actual planned canisters. For example, SKB have now selected Friction Stir Welding as the preferred canister weld technique, rather than Electron Beam Welding as is used to seal the miniature canisters.
1.3 Quality assurance	
What QA system and standards are used in the planning, design, execution, analysis, and reporting of the experiment?	SKB and Serco initially discussed the idea for this experiment and then Serco produced a project plan, which was discussed and approved by SKB. The experiment is being managed under the Serco QA system, which SKB has confirmed meets SKB QA requirements. Serco Technical and Assurance Services currently holds ISO 9001:2000 Quality Management Systems and ISO 14001:2004 Environmental Management Systems accreditation, awarded by Lloyd's Register Quality Assurance UK. For experiment and data analysis carried out by contractors (other than by Äspö), Serco is responsible for ensuring appropriate QA procedures are implemented and used. It was stated at the review meeting on 10/03/10 that an expert advisory group has been formed to steer the project. This is composed of SKB and Serco staff, and other specialised experts as required. The group has met twice during the course of the project so far, although the existence of this group is not mentioned in the SKB MiniCan report (SKB, 2009b). A record of the second meeting on 22 June 2009 is held on the SKB project file in Stockholm; SKB has no record of the first meeting.
What material quality controls are used?	The cast iron for the insert and the steel for the support cage were supplied directly to Serco and the material certificates are held on the project file. Clay Technology supplied the MX-80 bentonite, the copper tube was supplied by Outokumpu Poricopper OY, Finland, and SKB provided a full-size copper lid from which the end caps were manufactured; the bentonite and copper were of the same material specifications intended for use in the repository (SKB, 2009b, §3.2; Review meeting on 10/03/10).
How is the expert team selected/trained for the experiment?	This experiment evolved from previous corrosion work carried out by Serco on behalf of SKB. Selection of the team was based on experience and knowledge of the required skills, with additional expert advice sought as required.
2. Design of Experiment	
2.1 Variables	
What are the dependent variables (i.e. those being observed)?	During the experiment the electrochemical potential, water content, water pressure and outer copper surface canister strain are monitored. Real-time corrosion measurements possible during the canister monitoring period include copper and cast iron electrochemical measurements (linear polarisation resistance (LPR), AC impedance (ACI) and electrochemical noise (ECN)) and copper electrical resistance (SKB, 2009b, §4). Following extraction of each miniature canister, copper and cast iron coupon weight loss measurements will be made; these will also validate the real-time corrosion measurements. The four WOL and U-bend copper specimens will be assessed for stress corrosion cracking and the copper-cast iron-copper sandwich specimens will be examined for crevice, galvanic and expansive corrosion (SKB, 2009b, §4.6).

What are the independent variables (i.e. those that are varied to cause change in the dependent variables) and how are their values selected?	The bentonite density is varied between the different canisters (high, low and none), as are the number and positions of the applied canister defects. The low density bentonite selected was based on Ola Karnland's (Clay Technology) knowledge of bentonite permeability data (Review meeting on 10/03/10). The groundwater pressure in the rock and the rate of inflow to each MiniCan borehole varies depending on conditions local to each test hole. The oxygen and chloride content of the bentonite around the coupons are also independent variables.
What are the control variables (i.e. those that are held constant) and how are their values selected?	The bentonite position around each canister is controlled, with bentonite only placed around the canister sides, not the top and bottom. The experiment is carried out at ambient temperature (15°C) at the Äspö HRL (SKB, 2009b, p9), and experiences oxygen-free groundwater (below detection limit) and natural microbe populations. Water pressure in the system has reduced since the start of the experiment, potentially through the influence of other experiments nearby at Äspö, but mainly due to leakage through the tunnel walls, and so this is not a controlled variable (Review meeting on 10/03/10). The same batch of MX-80 bentonite was used for all the miniature canisters. Therefore, similar initial pyrite concentrations were present in each, although the pyrite content of the bentonite used was not known (Review meeting on 10/03/10).
2.2 Experimental techniques	
What experimental techniques and instruments are being used?	Stable reference electrodes are used to measure the overall corrosion potential of the model canisters and the redox potential of the environment. The electrochemical potentials are measured using commercial reference electrodes: two small disc silver-silver chloride reference electrodes, mounted inside the support cage, together with a large Silvion reference electrode outside the support cage but inside the borehole of each canister as a backup. The environment redox potential is measured using a gold wire and a platinum flag inside the support cage and an Eh sensor located outside the support cage. Water samples were taken for analysis, via stainless steel tubes that passed out through the borehole flange, at periodic intervals after installation of the experiment. Äspö staff carry out the water analysis or sub-contract it to appropriate laboratories as required. Water samples have not been taken during the last 18 months to allow the system to equilibrate (Review meeting on 10/03/10). PVB samplers, which take pressurised samples, were used for chemistry, gas and microbiological analyses by Microbial Analytics Sweden AB. The samples were taken when the experiments had not been drained for water sampling for at least four weeks to allow concentrations to build up (SKB, 2009b, §4.3). The microbial analysis was used to measure the total number of micro-organisms, the number of aerobic cultivable bacteria, the biomass (as adenosine-three-phosphate), and the most probable number of sulphate-reducing bacteria and autotrophic acetogens bacteria. The water pressure in the boreholes was initially measured using an analogue pressure gauge attached to the flanges on each borehole. This was later changed to an electrical pressure gauge attached to an outlet pipe on the flange, with the output recorded by the datalogging equipment (SKB, 2009b, §4.4). Standard strain gauge monitoring technology using bi-axial strain

	gauges was applied to model canisters 1 and 4. This was to measure the strain on the outer surface of the copper canister due to the production of solid corrosion products from the corrosion of the cast iron insert (SKB, 2009b, §4.5). Five types of corrosion coupon were mounted within the support cage used for the model canister experiments, at the top of the support on a nylon support rack (SKB, 2009b, §4.6): • Each of the model canister experiments contain plain corrosion coupons of copper and iron so that corrosion rates can be determined through weight loss measurements. • Coupons of copper and cast iron electrically connected to the exterior are designed to allow the corrosion potential of the electrodes to be measured. With a platinised titanium gauze electrode used as the counter electrode in a conventional 3-electrode electrochemical cell, it is also possible to carry out electrochemical measurements of the corrosion rate using linear polarisation resistance (LPR), AC impedance (ACI) and electrochemical noise (ECN) techniques. • Model canisters 2 and 5 contain copper electrical wire resistance probes. These were set up to measure the corrosion rate of copper using a technique proposed by VTT. Each consists of a coiled 112.5 cm length of 1 mm diameter copper wire divided in to three sections, the end sections sheathed in heat-shrinkable, adhesive-lined polymer tubing. The screened lengths act as reference resistances and the change in the resistance of the exposed length, processed by an ACM Field Machine electrochemical unit, enables calculation of the corrosion rate. • To assess stress corrosion, four WOL specimens, machined from a SKB copper lid and pre-cracked to give a range of stress intensity factors, were mounted in the boreholes. Four U-bend samples were also manufactured from the same copper lid material. Two of each specimen were mounted in the boreholes for model canisters 3 and 4 by loosely suspending them from the stainless steel push rod using plastic connectors and are therefore exposed directly to the groundwater. The specimens will be examined for stress corrosion cracking upon the removal of each miniature canister. • To investigate crevice corrosion, galvanic corrosion and expansive corrosion, copper-cast iron-copper sandwich specimens were mounted on the nylon support rack at the top of the canister support cage. These consist of a sheet of copper clamped against a block of cast iron using a ring of nylon bolts; the cast iron used is the same type as for the model canister insert. The specimens allow investigation of the effect of separation distance between mating surfaces by including a series of steps machined into the surface of the cast iron. The specimens will be examined upon removal of each miniature canister.
Are they standard techniques?	Generally, standard components and sensors have been used. The water analysis uses standard techniques, as does the microbial analysis and coupon weight loss measurements (which use the relevant ASTM standard). The strain gauge technology used is also standard. However, whilst based on simple principles, the copper wire corrosion rate measurements, derived from electrical resistance differences, are not used routinely in such an environment by SKB.

Are acceleration methods used?	The boreholes were located at Äspö in areas of reasonably high fluid flow to ensure a plentiful supply of groundwater to the miniature canisters. This was achieved by reference to the Äspö HRL fracture mapping work and by carrying out test drillings (SKB, 2009b, §5.1). Using low density bentonite allows bentonite-conditioned groundwater to reach the miniature canister rapidly and allows it to wet the annulus between the inner surface of the support cage and the outer copper canister surface, in order to ensure water reaches the applied canister defect and anaerobic corrosion conditions are quickly established (SKB, 2009b, p9 & p22). The low density bentonite miniature canisters potentially experience a harsher environment than would be anticipated in the actual repository due to the easier access of groundwater to the canister surface and because the reduced bentonite density could enable greater microbial activity at the canister surface (Review meeting on 10/03/10). The bentonite used for the compacted bentonite model canister was cut from a pre-saturated block to minimise the time required to achieve full saturation and hence full swelling pressure (SKB, 2009b, p22). The fifth canister without any bentonite present was designed to examine whether a bio-film develops on the canister surface and to examine its effect on corrosion behaviour. Therefore, the canister was directly exposed to groundwater (SKB, 2009b, p22).
Have the techniques been validated and documented?	As none of the five miniature canisters have been extracted, the techniques used for this experiment have yet to be validated. However, as stated above, the majority of the measurements and analysis techniques applied are standard, although the use of electrochemical methods under repository conditions is novel. Details of the experiment and early data are documented by SKB (2009b). The electrical resistance technique is believed to be documented in the ACM Ltd field machine manual (Review meeting on 10/03/10).
Are the techniques being used under normal conditions?	The water and microbial analysis techniques are used under normal conditions. The datalogging equipment (e.g., the ACM Ltd field machine), whilst used normally, is used for a longer period than usual. The more rugged field machine version was selected to withstand the rigours of the experiment. Sensors were tested in the laboratory before selection and installation at Äspö. Sensor redundancy was built into the experiment, which allows for the sensor failures that have occurred so far.
Has equipment been calibrated and checked?	All the reference electrodes were calibrated in the UK and then tested before installation (SKB, 2009b, §4.2). Laboratory trials were carried out using the Eh probes, reference electrodes and strain gauges inside a Hastelloy autoclave containing Äspö groundwater pressurised to 7 MPa with nitrogen (SKB, 2009b, §4.2). The trials were run for a few weeks to demonstrate that the sensors would run reliably under the appropriate operating conditions. The experiment electrical wiring was checked and upgraded in June 2007 to enable easier access to the connectors (SKB, 2009b, p8).
2.3 Uncertainty	
What are the key uncertainties in the experiment?	Key uncertainties at this stage in the experiment are: • The potential for channelling in the bentonite, but this will not be known until the canisters are removed. • The groundwater flow rate in the boreholes is uncertain, as is the pyrite content of the bentonite.

	• A key uncertainty is in the validity of the real-time corrosion rate measurements, which cannot be confirmed until after canister extraction - electrochemical measurements could be measuring the corrosion of the electrode itself, rather than the sample. All connections were made using soldered joints which were then sheathed in heat shrink. SKB (2009b, §4.8) notes that the electrochemical measurements rely on the integrity of the sheathing system throughout the experiments but the success of the sheathing will only be confirmed when the experiments are dismantled and the sheathing can be examined. The electrochemical measurements could also be effected due to formation of a copper sulphide film. • There is currently uncertainty with regard to the electrical resistance measurement sampling frequency: the sampling frequency appears to affect the results obtained. Serco is in discussion with the datalogging field machine manufacturers, ACM Ltd, about this issue.
2.4 Risks to success of experiment	
What are the risks to the success of the experiment and how are they mitigated?	Key risks in the experiment include: • The monitoring reliability of the sensors and reference electrodes (backup electrodes were therefore included). • Damage to the sensors and weight loss coupons during miniature canister extraction. • Change in corrosion products formed when the miniature canisters are extracted from the Äspö environment. The extraction procedure details are still to be developed but the plan will be designed to eliminate oxygen intake (e.g., by placing the extracted canister in a nitrogen atmosphere as soon as possible).
What are the critical decisions in the experiment?	The critical decisions in this experiment are when and which miniature canisters to extract. The first is planned to be extracted this year and is expected to be miniature canister 3 because it is one of the three low density bentonite canisters and the other two such canisters contain strain gauge and electrical resistance tests that may yield useful results over a longer period in situ. A key initial decision was siting the experiment at Äspö HRL: the first location considered was too dry and it was important to ensure sufficient groundwater flow for the corrosion experiments (Review Meeting on 10/03/10).
Is there duplication in the experiment?	Multiple reference electrodes, sensors and tests have been included across the five model canisters. In addition, three of the model canisters contain the same (low) density bentonite.
3. Conduct of Experiment	
3.1 Data collection and quality control	
How are data collected?	An ACM Ltd Field Machine is used to carry out the electrochemical measurements of corrosion rate and measurements of electrical resistance of the copper wire electrodes. An Agilent datalogger is used to monitor the potential of the various electrodes and monitor the strain gauges (SKB, 2009b, §4.9). The datalogging equipment is located in a control room near the model canister boreholes and the data is then transmitted via the internet to Serco's Culham laboratory for analysis. The data logging system does not provide automatic notifications or alarms if it there is a problem; it must be manually checked to see if the has stopped working. Changes in the system are automatically logged

	in the data-recording system. The manual collection of water samples for analysis has not been carried out for the last 18 months to allow system equilibration (Review meeting on 10/03/10).
How are data stored (e.g., filing, indexing)?	Data are stored electronically on the computer at Äspö and, via the internet link to the UK, are also stored electronically at Serco's Culham laboratory. The water and microbial analysis data are stored in the SKB SICADA database.
How are data checked (e.g., independently)?	The data are reviewed by those involved in the project. The Äspö water analysis is added straight to SICADA without review by the project manager. Serco (Nick Smart) reviews the electrochemical data.
How are data backed-up?	The data are backed up on Äspö and Serco servers.
What quality control procedures are used?	The majority of the water sampling and analysis is carried out by staff at Äspö and is subject to Äspö HRL QA procedures. Some water analysis is carried out at other facilities via Äspö HRL but these have been checked to ensure they comply with the QA requirements of Äspö. The electrochemical and corrosion analysis by Serco is subject to Serco's QA procedures.
3.2 Records of experiment	
Are notebooks being used for the experiments?	A master notebook is used for all experiment changes and details of the experiment installation, maintenance visits and non-routine procedures are recorded.
Are notebooks checked independently?	The project manager (Nick Smart) reviews the experiment notebooks but the notebooks are not independently reviewed.
Are planning, execution and analysis correspondence kept (e.g., emails)?	A hardcopy of key project emails is placed on the project file and all the project emails are stored in Nick Smart's email account.
Are copies of records kept?	There is only one copy of the notebook. Project emails are backed up on servers at Serco.
3.3 Equipment	
Is equipment tested, inspected, and maintained?	There is an annual maintenance visit and equipment is tested at each visit. SKB staff at Äspö also monitor the experiment status. The data-recording computer system was installed in 2007 and has been reliable, with only one PC failure so far (Review Meeting on 10/03/10). The datalogging system does not have an automatic alarm so must be manually checked to ensure it is still operating correctly.
4. Analysis and Reporting of Experiment	
4.1 Data interpretation	
What data interpretation methods are being used (models, software packages, model simplifications)?	The ACM Ltd field machine includes integral software that interprets some of the raw data measured, although it uses standard techniques and so could be checked from first principles. The electrical potential measurements are recorded directly from the sensors with no human interpretation. The corrosion products and surface film information derived following extraction will be used to support corrosion models in the performance assessment.

How are uncertainties and sensitivities analysed?	Estimation of experimental and data analysis uncertainties and errors are yet to be considered. Values reported by SKB (2009b) are without associated error quantification. Areas of uncertainty include identification of the gradient in graphs of electrical impedance and measurement of electrode degradation (failure of the insulating heat-shrink will break electrical isolation from surrounding metals and there may also be crevice corrosion around the connection). Extraction of the model canisters will aid estimation of some uncertainties and help to verify real-time measurements. For example, coupon weight loss measurements will aid validation of the real-time electrochemical corrosion rates.
4.2 Reporting and review	
How are data and observations reported?	A progress report on all aspects of the experiment is produced by Serco and submitted to SKB for review. Conference presentations have been made and SKB (2009b) reports details of the experiment up to May 2008.
How are interpretations reported?	As above.
How are limitations on the use of data and results reported?	Limitations on the use of the data have not been discussed in SKB (2009b); the corrosion data published so far will not be confirmed until a model canister is extracted and its coupons and sensors analysed. However, data that indicated higher than expected corrosion rates were selectively excluded from SKB (2009b) (review meeting on 10/03/10). No indication was given in SKB (2009b) that such data had been excluded. As the copper corrosion tests are intended as a verification experiment, it is expected that the data will not be used directly in the SR-Site safety assessment calculations.
How are reports reviewed (e.g. independently)?	For Serco reports, Serco select a reviewer with relevant expertise who is independent of the project. The reviewer can be a Serco employee or an independent external expert. There is no formal route for approving SKB technical reports; SKB staff connected with the project review and approve the reports. In addition, the MiniCan advisory group commented on the draft technical report.
How are review results managed/responded to?	The Serco review process retains review comments on the project file but there is no requirement for the reviewer to see and agree the changes made, or approve the revised report. The SKB review process also keeps review comments on the project file.
5. Usability of Results	
5.1 Verification	
How are experimental outcomes checked against requirements of the experiment?	Data obtained so far through ongoing experiment monitoring cannot be confirmed until the miniature canisters are extracted from their boreholes. Once all the data are available and understood, SKB will decide if further experiments are required. New SKB copper corrosion experiments are underway and others are planned for the future, including laboratory experiments in which greater controls can be exerted on experiment conditions.
How are experimental results verified?	Verification of the real-time corrosion data will be through canister extraction and subsequent coupon weight loss measurements, and then by comparison of the results between the five miniature canisters. Correlation of the data from the different measuring techniques

	performed should aid verification. Observations will be compared with expected results, such as from published data and theoretically calculated values.
5.2 Use of results	
How are results abstracted for use in the repository programme?	The results of this experiment will be used to support understanding of the long-term evolution of the copper and iron canister. The copper corrosion data obtained from this experiment will be used to verify the corrosion process and demonstrate consistency with earlier results. Verified results from this experiment will not be available before the SR-Site licence application is submitted.
Are results extrapolated for use on repository length and time scales?	As the experiment was not heated it does not give information on the behaviour of real canister corrosion in the early period when the radioactive waste will be significantly heat-generating. Therefore, the information obtained from this experiment is assumed to indicate canister behaviour at a later time in the disposal programme after the waste has cooled.
What checks are made that data and results are used appropriately and within prescribed limitations?	The primary users of the experimental results, those in research and safety assessment, are already aware of the data limitations. SKB state it would be made clear to anyone requesting the data that they should be cautious in its use and understand the uncertainties in its derivation. It was stated that the applicable data range, constraints and uncertainties would be published with the data, although there is no discussion of uncertainties in the first MiniCan report (SKB, 2009b).

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