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Quality Assurance in SKB's Copper Corrosion Experiments

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SSM perspektiv

Bakgrund

I Sverige planeras slutförvaring av använt kärnbränsle. Metoden som har utvecklats kallas för KBS-3 och den bygger på tre skyddsbarriärer; kopparkapslar, bentonitlera och det svenska urberget. I den aktuella utformningen för KBS-3 kommer det använda kärnbränslet att placeras i en insats av gjutjärn vilken är placerad i en 50 mm tjock kopparkapsel. Kapseln ska i sin tur deponeras i ett kristallint bergförvar i Forsmark på ett djup av ca 500 m. Gjutjärnsinsatsen ger mekanisk hållfasthet och strålskydd medan kopparkapselns roll är att skydda mot korrosion. I utvärderingen av det planerade KBS-3-systemet är förståelsen för långtidsutveckling av processer som kan påverka kapseln, inklusive degradering via korrosion, mycket viktig.

Strålsäkerhetsmyndigheten (SSM) granskar Svensk Kärnbränslehantering AB:s (SKB) ansökningar enligt lagen (1984:3) om kärnteknisk verksamhet om uppförande, innehav och drift av ett slutförvar för använt kärnbränsle och av en inkapslingsanläggning. Som en del i granskningen ger SSM konsulter uppdrag för att inhämta information och göra expertbedömningar i avgränsade frågor. I SSM:s Technical note-serie rapporteras resultaten från dessa konsultuppdrag.

Projektets syfte

Det övergripande syftet med projektet är att ta fram synpunkter på SKB:s säkerhetanalys SR-Site för den långsiktiga strålsäkerheten för det planerade slutförvaret i Forsmark. Det specifika syftet är att få en förståelse för, och en bedömning av, SKB:s kvalitetssäkring av deras försök angående kopparkorrosion. Kunskap om korrosion av kopparkapsel i slutförvarsmiljö har avgörande betydelse för att förstå den långsiktiga integriteten av slutförvarssystemet. Korrosionsförsök är förhållandevis komplexa och har genomförts i flera år av ett antal medverkande. I samband med SSM:s bedömning av ansökansunderlaget är det därför av intresse för SSM att skapa sig en förståelse för SKB:s kvalitetssystem och hur det har tillämpats i SKB:s kopparkorrosionsförsök.

Författarens sammanfattning

Denna QA-granskning har fokuserat på olika kopparkorrosionsexperiment som ingår i MiniCan- och LOT-projekten vid Äspö-laboratoriet (HRL), tester av kopparkorrosion i rent syrefritt vatten som genomförts vid Uppsala universitet samt atmosfärisk och kopparkorrosionstester i vattenlösningar som genomförts vid Äspö-laboratoriet. För att förstå QA-aspekterna för de valda experimenten har personal från SKB som arbetar med försöken rådfrågats vid ett granskningsmöte. Rapporterna som beskriver experimenten, genomföranden och resultaten av experimenten kontrollerades.

Förutsättningarna för kopparkorrosionsexperiment är komplexa och återhämtning av de exponerade proverna är en komplicerad process. Således finns det ett behov av att sådana experiment är noggrant förberedda, med grundlig karaktärisering av proverna före exponering samt att korrekta initiala förhållanden måste säkerställas. Dessutom kan övervakning, kontroll och analys av experiment vid HRL vara problematiska, alltifrån misslyckanden av

värmaren i de tidiga atmosfäriska korrosionstesterna och kontrollproblem av grundvattenflöden i de vattenbaserade korrosionstesterna, till elektrod- och datormisslyckanden i MiniCan-projektet.

Dessutom är det vanligt att det finns långa perioder mellan upptäckt av sådana systemfel och korrigering av problemet. Systemövervakning, kontroller och oförutsedda händelser bör byggas in i utformningen av experimenten i syfte att säkerställa att värdefull data inte går förlorade. Det är viktigt att dra lärdomar från experimenten som genomförts och ta hänsyn till det i planeringen av framtida korrosionsexperiment.

Användningen av elektrokemiska metoder för att mäta korrosionshastigheter i realtid har haft begränsad framgång och de mekanismer, hastigheter och den spatiala fördelningen av syreförbrukningen i experimenten är inte väl förstådd. Endast genom att utföra experiment på ett isolerat koppar- och bentonitprov, har korrosionsövervakning i realtid kunnat ge rimliga indikationer på att kopparkorrosionshastigheter utvecklas, enligt oxiska förhållanden. Trots detta visades korrosion efter provundersökningar vara ojämn och lokaliserad och att den uppskattade allmänna korrosionshastigheten kunde ha överskattats.

Osäkerhet kvarstår angående möjligheten för kopparkorrosion i vatten under anoxiska förhållanden (i avsaknad av aggressiva ämnen som sulfider och klorider). SKB:s korrosionsförsök för att undersöka hur koppar korroderar under dessa förhållanden har inte kunnat peka på källan till vätgas som genererades i försöken, trots att hög standard på QA och kvalitetskontroll antogs.

Endast ett begränsat antal resultat från kopparkorrosionsexperiment används av SKB för att få fram korrosionshastigheter som underlag för slutförvarets säkerhetsanalys. Istället använder SKB resultaten av dessa experiment på ett mer kvalitativt sätt, det vill säga att SKB använder experimenten för att stödja förståelsen av kopparkorrosionsprocesserna under relevanta förhållanden för slutförvaret.

SKB har kommit fram till att resultaten i dagsläget inte motsäger behandlingen av kopparkorrosion i säkerhetsbedömningen SR-Site, även om det noteras att SKB inte hänvisar till den icke-uniforma/ lokala korrosionen som observerades under oxiska förhållanden i realtid i korrosions-övervakningsexperiment.

Denna QA-granskning pekar på att kvaliteten på det senaste experimentella forskningsarbetet är av tillräckligt hög standard och att SKB:s bedömningar har gjorts på ett tillförlitligt sätt. SKB:s senaste rapporter om kopparkorrosionsfrågor visar i allmänhet större uppmärksamhet för QA-relaterad information och en mer övergripande strategi för att rapportera uppgifter än i äldre rapporter eller artiklar från SKB.

Projekt information
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Diarienummer: SSM2013-5714
Aktivitetsnummer: 3030012-4069

SSM perspective

Background

The Swedish plan for disposal of High-Level Nuclear Waste (HLNW) implies the encapsulation of spent nuclear fuels and deposition of the canisters holding the spent fuel in a crystalline bedrock repository at a depth of about 500 m. In the current KBS-3 design, the spent fuel will be emplaced in an inner cast iron insert that is contained in a copper canister with a 50 mm wall thickness. The role of the cast iron insert is to provide mechanical strength as well as radiation shielding, while the copper canister's role is to provide corrosion protection. The evaluation of the performance of the KBS-3 system, understanding of the long-term development of the processes that can affect the canister including degradation via corrosion is very important.

The Swedish Radiation Safety Authority (SSM) examines under The Act on Nuclear Activities (1984:3), the applications of the Swedish Nuclear Fuel and Waste Management Co's (SKB) for the construction, ownership and operation of a final repository for spent nuclear fuel and of an encapsulation plant. As part of the review process, SSM commissioned external expert consultants to gather information and make expert judgments regarding specific issues. The results of these consulting assignments are then published in SSM Technical Note series.

Objective

The overall aim of the project is to gather information and make expert judgments of SKB's safety analysis SR-Site for the long term safety of the planned repository in Forsmark. The specific aim is to gain understanding and make expert evaluation of SKB's quality in their copper corrosion tests. Knowledge of corrosion of the copper canister in the repository environment is crucial to ensure the long-term integrity of the repository system. Corrosion tests are relatively complex and have been conducted for several years by a number of participants. In conjunction with SSM's assessment of the application, it is therefore of interest to SSM to get an understanding of SKB's quality control process and how it has been applied in SKB's copper corrosion tests.

Summary by the author

The quality assurance (QA) review focused on various copper corrosion experiments that form part of the MiniCan and LOT projects at the Äspö hard rock laboratory (HRL), tests of copper corrosion in oxygen-free pure water being undertaken at Uppsala University, and atmospheric and aqueous copper corrosion tests that were undertaken at Äspö. In order to understand the QA status of the selected experiments, SKB staff involved in the experiments were consulted at a review meeting and reports describing the setup, conduct and results of the experiments were checked.

The exposure conditions for experiments at the HRL are complex and the retrieval of the exposed samples is complicated. Thus, there is a need for such experiments to be prepared carefully, with thorough pre-characterisa-

tion of the samples before exposure, and for the correct initial conditions to be ensured. Also, monitoring, control and analysis of experiments at the HRL is typically problematic, from the heater failures in the early atmospheric corrosion tests and the groundwater flow control problems in the aqueous corrosion tests, to electrode and computer failures in the MiniCan project. Also, it seems usual for there to be long periods between such system failures and the detection and correction of the problem. System monitoring, controls and contingencies should be built into the design of the experiments with the aim of ensuring that valuable data are not lost. It is important that lessons are learnt from the experiments undertaken to date in the planning of future corrosion experiments.

The use of electrochemical techniques to measure real-time corrosion rates has been of limited success, and the mechanisms, rates and spatial distribution of oxygen consumption in the experiments are not well understood. Only by conducting experiments on an isolated and controlled copper and bentonite sample, has real time corrosion monitoring been able to give reasonable indications of evolving copper corrosion rates under oxic conditions. Even so, post-test examinations revealed corrosion to be uneven and localised and showed that the estimated general corrosion rate could have been overestimated.

Uncertainties remain about the possibility of copper corrosion in water under anoxic conditions (in the absence of aggressive species such as sulphide and chloride). SKB's experiments dedicated to establishing an understanding of the behaviour of copper under such conditions have been unable to pin-point the source of hydrogen generated in the experiments or the small concentrations of copper detected in the water, despite the high standard of QA and quality control adopted.

Only a limited number of the results of the copper corrosion experiments have been used by SKB to derive copper corrosion rates for use in the repository safety assessment. Instead, SKB generally uses the results of these experiments in a more qualitative way. That is, SKB uses the experiments to support its understanding of copper corrosion processes under repository conditions. SKB has concluded that the results to-date do not contradict its treatment of copper corrosion in the SR-Site safety assessment, although it is noted that SKB has not made reference to the non-uniform/localised corrosion observed under oxic conditions in the real-time corrosion monitoring experiments. This QA review has found that the quality of the most recent experimental research work is of a sufficiently high standard that SKB's judgments can be made reliably. Indeed, SKB's most recent reports on copper corrosion issues generally show greater attention to recording QA-related information and a more comprehensive approach to reporting data than in older reports and papers.

Project information
Contact person SSM: Clara Anghel and Bo Strömberg
Reference: SSM2013-5714
Activity number: 3030012-4069

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Contents

1. Introduction

The Swedish Radiation Safety Authority (SSM) has completed the initial phase of its review of the SR-Site safety assessment produced by the Swedish Nuclear Fuel and Waste Management Company (SKB). The review subsequently entered its main phase, with assignments targeted on prioritised tasks and issues and aimed at supporting SSM's compliance judgements. As part of the main review, SSM tasked Galson Sciences Limited to undertake an assessment of SKB's documentation and quality assurance (QA) of selected copper corrosion experiments. This report presents the results of the QA review.

The review coverage has ranged from copper corrosion experiments that have been included as components of long-term tests of engineered barrier system evolution being undertaken at SKB's Äspö Hard Rock Laboratory (HRL), to laboratory experiments that have been designed specifically to gain further understanding of copper corrosion processes under controlled chemical conditions. The review focused on the following copper corrosion experiments:

- The Miniature Canister (MiniCan) experiment at the Äspö HRL, focusing on the extraction of Experiment 3 and the subsequent analyses of the copper weight loss coupons and copper canister.
- The Long Term Test of Buffer Material (LOT project) at the Äspö HRL, focusing on:
    - The analysis of the heated copper tube from the A2 parcel test.
    - The analysis of the copper coupons from the A0 parcel test.
- Copper corrosion tests in oxygen-free pure water undertaken at the Ångström Laboratory of Uppsala University's Department of Chemistry.
- Atmospheric and aqueous copper corrosion tests undertaken at the Äspö HRL.

The overall aim of the assignment was to check the existence and application of appropriate procedures during all stages of these corrosion experiments, including analysis of the experiments and use of results. In order to achieve this aim, the QA review involved two main components:

- A review meeting with SKB staff to discuss QA in the copper corrosion experiments.
- A review of documentation relating to the copper corrosion experiments.

The QA review meeting took place at SKB's offices in Stockholm on 29th August 2014; see SSM2011-2306-22 (2015) for minutes of the meeting. The status of SKB's various experiments on copper corrosion was discussed at the meeting as summarised in Section 2. However, the main aim of the meeting was to review QA procedures and evidence of their application in the selected copper corrosion experiments. Also, the meeting aimed to understand the links between the assumptions about corrosion rates made in the SR-Site licence application and the experiments that support those assumptions. In order to facilitate the review process, prior to the meeting, SKB had been provided with a list of QA questions relating to the design, running, analysis and use of results of each copper corrosion experiment. Findings from the review meeting and SKB's responses to the list of issues for each experiment are referred to in this report.

Following the QA review meeting, SKB reports, contractor reports and published papers relating to the copper corrosion experiments were reviewed in order to understand the reliability of results, especially those that support SKB's understanding of copper corrosion as expressed in the SR-Site safety assessment and subsequent interactions with SSM during SSM's safety assessment review process. The review findings are presented in Section 3 for each copper corrosion experiment and overall conclusions of the review are presented in Section 4. Appendix 1 lists the SKB reports that have been reviewed and Appendix 2 summarises QA issues relating to each experiment, based on SKB's responses at the QA review meeting.

2. SKB's Copper Corrosion Experiments

The status of SKB's copper corrosion experiments was discussed at the QA review meeting (SSM2011-2306-22, 2015) and the results of the discussions are summarised in Table 1. Full details of the experiments are given in SKB's 2013 "RD&D Programme" (SKB, 2013). The RD&D programme is published every 3 years.

In addition to the experiments listed in Table 1, at the QA review meeting, SKB noted that other experiments had been set up to assess the effects of gamma radiation on copper corrosion. The experiments involve irradiating copper in pure water and analysing the results using spectroscopic methods. The experiments and their analysis are being undertaken as a PhD study at KTH (SKB, 2013, §24.2). SKB also noted that some corrosion data are available from the Prototype Repository test at the Äspö HRL. Electrochemical methods have been used to make real-time measurements of corrosion using copper electrodes embedded in the bentonite buffer (Rosborg, 2013a;b; SKB, 2013, §24.2).

Table 1: Status of SKB's copper corrosion experiments.

Experiment	Status
Experiments on copper corrosion in a sulphide/water environment	
Tests to understand the rate determining step(s) in the formation of sulphide films and their properties for experiments involving copper in sulphide solutions and different concentrations of chloride. The analysis involves electrochemical impedance and spectroscopic methods.	This work is being carried out by researchers in the Department of Chemistry of the University of Western Ontario, Canada. Published results include Chen *et al*. (2012).
Tests to repeat the experiments of Taniguchi and Kawasaki (2008), who observed stress corrosion cracking (SCC) of copper in sulphide solutions. Copper has been subjected to slow strain rate testing (SSRT) and constant strain rate testing, and a range of techniques has been used to analyse the test results.	This work was carried out by researchers in the Department of Chemical Engineering and Applied Chemistry of the University of Toronto in Canada. The study has been completed and the results have been published by Bhaskaran *et al*. (2013).
Experiments on copper corrosion in a bentonite environment	
Investigation into the potential for sulphate-reducing bacteria (SRB) growth and biofilm formation on copper in compacted and saturated bentonite. Copper rods were embedded in compacted bentonite, which was saturated with groundwater from Äspö. At the end of the experiment, the metal rod surfaces were analysed for bacterial coatings.	The research was carried out by Microbial Analytics Sweden AB. The project has been completed and the results published (Persson *et al*., 2011). However, further experiments are being undertaken to determine the sensitivity of SRB growth and sulphide production to bentonite compaction.

Experiment	Status
Electrochemical studies of copper corrosion in a compacted bentonite environment. The research has involved the analysis of copper electrodes that were exposed in the LOT A2 test parcel at the Äspö HRL. Electrical resistance and electrical impedance spectroscopy measurement techniques were used to analyse corrosion rates over time.	The corrosion analysis work for the LOT A2 test parcel was carried out by researchers at the Division of Surface and Corrosion Science at KTH, Stockholm, and the Slovenian National Building and Civil Engineering Institute, Ljubljana, Slovenia. The work has been completed and published (Rosborg *et al*., 2012).
Also, copper profiles in the clay next to the copper tube (in the LOT A2 test) were analysed to estimate corrosion rates.	The compilation of data from the analysis of the copper tube was undertaken by Gruner Ltd, Switzerland (Wersin, 2013)
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. Copper coupons have been included in the tests. A canister from the MiniCan project was retrieved and analysed in 2011 (Experiment 3).	SKB is now leading the MiniCan project with the support of an SKB steering group; the experiments had previously been managed by Amec Foster Wheeler (formerly Serco Technical Services). Results of the analysis of Experiment 3 have been reported by Smart *et al*. (2012a;b; 2013) and Hallbeck *et al*. (2011).
Tests to study SCC of copper in groundwater containing ammonium. This work uses SSRT and spectroscopy and electrochemical measurements to identify SCC.	This study was carried out by VTT, Finland, in co-operation with Posiva (Kinnunen and Varis, 2011).
Experiments in oxygen-free water	
Work to develop a kinetic model for the copper/electrolyte interface for copper in deoxygenated water using potential measurements and electrical impedance spectroscopy.	The experiments are being carried out at the University of Chemical Technology and Metallurgy in Sofia, Bulgaria. The experiments are ongoing, but results have been reported by Bojinov *et al*. (2010) and Betova *et al*. (2013a;b).
Research to look for unknown compounds of copper with oxygen and hydrogen. Spectroscopic studies and X-ray diffraction have been used to study CuH produced from a copper solution. The thermodynamic stability of different compounds and the synthesis of CuOH are also being investigated.	The experiments are ongoing. Results to date have been published by Korzhavyi and Johansson (2010), Korzhavyi *et al*. (2011; 2012) and Soroka *et al*. (2013).
Copper corrosion experiments under anoxic conditions, similar to the long-term tests reported by Hultquist *et al*. (2009) that indicated the presence of a copper corrosion product that contains hydrogen. The experiment involves placing copper foils in anoxic deionized water in Erlenmeyer conical glass flasks in a reducing environment. The analysis included a reference test to investigate the effects of exposure to air atmosphere.	The work was carried out by the VTT Technical Research Centre of Finland. Results from the experiments have been reported by Ollila (2013).
Experiments on copper corrosion in ultra-pure, oxygen-free water using ultra-high vacuum equipment. These experiments aimed to either confirm or refute the hypotheses put forward by other researchers (e.g. Szakálos *et al*., 2007) at KTH that copper corrodes at a non-negligible rate with pure water as the oxidant.	Researchers at Uppsala University's Ångström Laboratory in the Department of Chemistry were commissioned by SKB to undertake these experiments. The experiments are ongoing, but initial results have been reported by Boman *et al*., (2013; 2014).
Experiments to test hypotheses on hydrogen production from copper in oxygen-free water. The experiments involve placing copper strips in glass test tubes and analysing the gases generated.	The experiments are being undertaken by Microbial Analytics Sweden AB. The study methodology has been published by Bengtsson *et al*. (2013).

Experiment	Status
Analysis of copper wires that had been placed in water in test tubes as part of an experiment on copper corrosion in oxygen-free water initiated some 20 years ago. The test tubes had been sealed with palladium membranes and had been stored by SP, the Technical Research Institute of Sweden, ever since.	The results of the analysis have been reported by Möller (2012).

3. QA Review of Copper Corrosion Experiments

As discussed in Section 1, the main QA review focused on the following copper corrosion experiments:

- The MiniCan Experiment 3 copper coupon and canister analyses (Section 3.1).
- The LOT project A2 parcel test copper tube analysis and the A0 parcel test copper coupon analysis (Section 3.2).
- Copper corrosion tests in oxygen-free pure water (Section 3.3).
- Atmospheric and aqueous copper corrosion tests (Section 3.4).

QA aspects relating to each experiment, as discussed at the QA review meeting, are summarised in Appendix 2.

In addition, a brief QA review is included of the real-time copper corrosion monitoring experiment undertaken using copper electrodes in a bentonite block from the LOT A2 parcel (Section 3.2), although the experiment was not subject to review at the QA review meeting.

3.1. MiniCan Experiment 3

3.1.1. Overview of MiniCan

The Miniature Canister (MiniCan) experiment at SKB's HRL in Äspö was designed to investigate how corrosion of the cast iron insert would develop if a defect were present in the outer copper canister. The experiment involved placing five small-scale model canisters in boreholes at the HRL to simulate the main features and conditions of the SKB disposal concept. The model canisters consist of outer copper bodies fabricated from the same grade of copper as will be used for full size canisters and a cast iron insert with four holes drilled to simulate fuel assembly channels (see Figure 1a). End caps were fabricated from the lid material that will be used for full-scale canisters and were electron-beam welded to the canisters. At least one 1 mm defect (a drilled hole) was introduced in each canister in the copper body near the weld area to allow groundwater to access the cast iron insert.

The model canisters were emplaced in support cages, with three of the support cages containing low density bentonite (Experiments 1, 2 and 3) (see Figure 1b), one containing high-density compacted bentonite (Experiment 4) and one containing no bentonite (Experiment 5). Where bentonite was included, it was intended to provide a groundwater chemistry representative of disposal conditions. Each canister support cage contains a range of sensors (e.g. reference electrodes, Eh electrodes, copper and iron electrodes), weight loss corrosion coupons (copper and cast iron) and stress corrosion test pieces (see Figure 1c). The model canisters in their support cages were placed in separate sub-horizontal boreholes in the HRL where the groundwater supply is large.

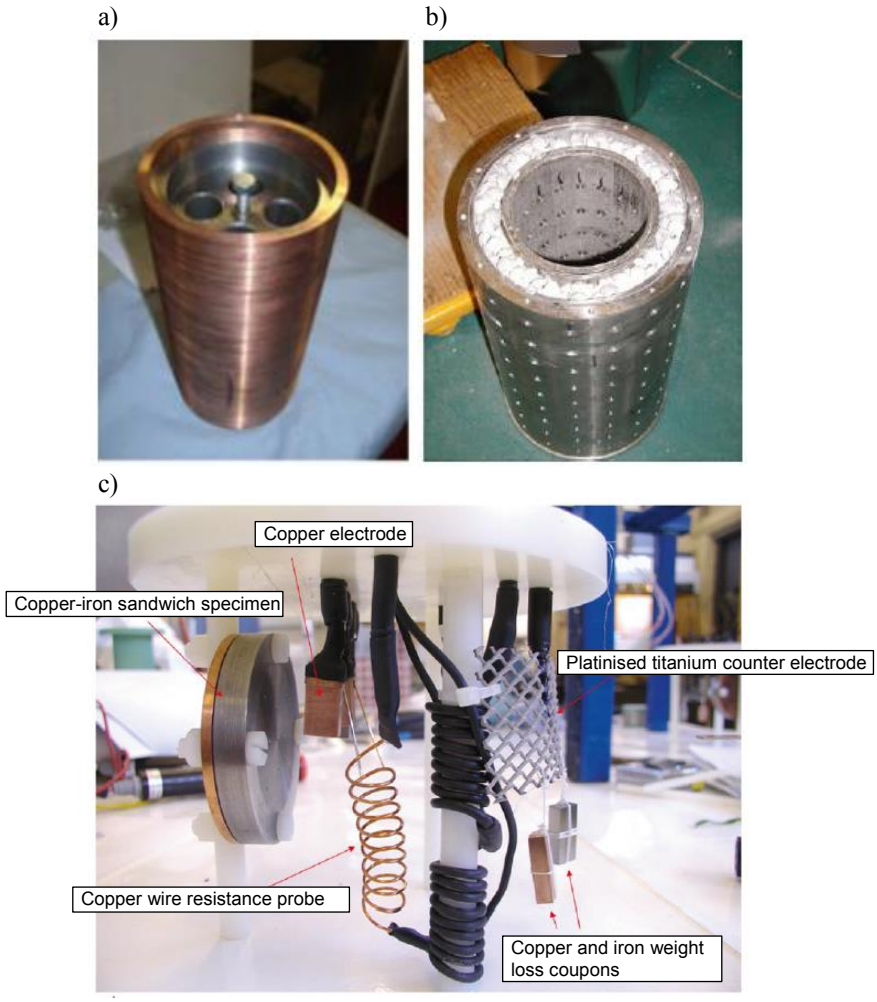

Figure 1: a) MiniCan model copper canister with cast iron insert (Smart and Rance, 2009, Figure 3-6); b) support cage for the copper canister, containing bentonite pellets (Smart and Rance, 2009, Figure 5-2); c) instrumentation included at the top of the support cage (Smart and Rance, 2009, Figure 4-2).

The canisters were installed in late 2006 and results of the experiments up until May 2008 were reported by Smart and Rance (2009). Experiment 3 was recovered in 2011 (after about five years of operation) and the results have been analysed (Smart *et al*., 2012a; Hallbeck *et al*., 2011; Smart *et al*., 2013). Progress on the MiniCan project up to the end of 2011 has been reported by Smart *et al*. (2012b).

3.1.2. Motivation for QA review of MiniCan Experiment 3

The QA review of SKB's copper corrosion experiments reported by Baldwin and Hicks (2010) included a review of the MiniCan experiment as reported by Smart and Rance (2009). At this time, none of the five miniature canisters had been extracted for analysis. However, as noted above, Experiment 3 has now been recovered and the results have been analysed. Thus, this QA review focuses on Experiment 3 and, in particular, the analysis of copper corrosion reported by Smart *et al*. (2012a) and Smart *et al*. (2013), and the microbial analysis reported by Hallbeck *et al*. (2011).

Relevant QA aspects of the MiniCan project up to the end of 2011, as discussed in the MiniCan progress (Smart *et al*., 2012b), are also considered. The findings of the previous QA review are referred to and updated as necessary, but detailed discussions about QA relating to the overall running and reporting of MiniCan (Baldwin and Hicks, 2010, §4.2, Appendix A.2) are not reproduced here.

At the QA review meeting (SSM2011-2306-22, 2015) and during the previous review Baldwin and Hicks (2010, §5.2), SKB acknowledged that the MiniCan experiment had been designed with a focus on understanding processes other than copper corrosion (i.e. corrosion of the cast iron insert and behaviour of the bentonite buffer). Furthermore, monitoring results from the MiniCan experiment relating to copper corrosion were not used directly in the SR-Site safety assessment. Corrosion depths were calculated based on mass balance or mass transport limitations on specific corrodants at the canister surface, rather than derived from measured corrosion rates (SKB, 2010). However, at the QA review meeting, SKB reported that no results from the MiniCan experiment obviously contradict the treatment of copper corrosion in the safety assessment and, in particular, the experiments confirm that reducing conditions would develop after a few months in the vicinity of a disposal canister and there is no evidence for localised corrosion processes. Such observations have been used to support SKB's interactions on copper corrosion with SSM during the safety assessment review process. Thus, it is important to understand the QA status of the MiniCan experiment and analysis in order to build confidence in the reliability of the MiniCan experiment findings in supporting SKB's position on copper corrosion behaviour.

3.1.3. MiniCan Experiment 3 QA review

Project management, QA plans and procedures

At the QA review meeting (SSM2011-2306-22, 2015), SKB explained that there had been changes in the management of MiniCan: SKB's Johannes Johansson is now project leader, with the support of an SKB steering group. The MiniCan project had previously been managed by Amec Foster Wheeler (formerly Serco Technical Services). In the previous QA review, Baldwin and Hicks (2010, §5.2) had observed that SKB placed significant reliance on its external consultants for determining the scope of the copper corrosion experiments, and the extent to which SKB controls or influences the aims and design of experiments such as MiniCan to ensure that SKB's requirements are met was not clear. It is concluded that the revised MiniCan project management arrangements should enable SKB to exercise greater control on the direction, analysis and reporting of its experiments.

The status and plans for the MiniCan experiments were also discussed at the QA review meeting. As noted above, Experiment 3 was recovered in 2011 and the results have been analysed. SKB plans to recover Experiment 4 and Experiment 5 in 2015, although this depends on budgetary decisions. Decisions on when to recover Experiments 1 and 2 have yet to be made. The MiniCan progress report (Smart *et al*., 2012b) does not discuss any project plans for recovery and analysis of Experiments 1, 2, 4 and 5. Instead, Smart *et al*. (2012b, §6) only provide broad "suggestions" for future work on the MiniCan experiments as well brief thoughts on other possible corrosion research and experiments. In the previous QA review, Baldwin and Hicks (2010, Appendix A.2) noted that MiniCan is subject to an SKB project plan and the plan is updated every 1 to 2 years to meet ongoing requirements. It would be helpful if plans for the MiniCan project and its role in SKB's broader repository research and development programme were documented

in progress reports, such that any developments in the significance of the experiment and its relevance to the repository development programme could be understood. However, it is acknowledged here that the programme is discussed in SKB's RD&D report (SKB, 2013, §24.2.5).

A project plan (including a task schedule) was developed for removal and post-test analysis of Experiment 3 (Smart *et al*., 2012a, Figure 1-1). The project plan was reviewed and agreed by the copper corrosion Reference Group, which included a number of independent researchers. The work was led by experts from Amec Foster Wheeler, who had performed similar and related work for SKB. Each task in the programme to remove Experiment 3 is described in detail by Smart *et al*. (2012a). There are no explicit references to QA procedures followed in the analysis, although it is acknowledged here that much of the work to recover and analyse Experiment 3 was novel and should provide good experience and learning when plans are developed for the recovery and analysis of the other MiniCan experiments. Also, as noted previously by Baldwin and Hicks (2010, Appendix A.2), it is understood that SKB checks that appropriate QA systems are used by its contractors for projects such as MiniCan.

The project plan reported by Smart *et al*. (2012a, Figure 1-1) for the post-test work does not include the groundwater and microbial analysis, beyond taking groundwater samples and microbial swabs. Instead, the microbial analysis was treated as a separate project that was undertaken by Microbial Analytics Sweden AB and reported by Hallbeck *et al*. (2011). Hallbeck *et al*. (2011) do provide references to relevant activity plans, which include descriptions of microbial sampling and analysis procedures. Also, the metallurgical analysis for Experiment 3 indicated in the project plan is reported separately by Smart *et al*. (2013). No mention of the project plan or project procedures for the metallurgical analysis is made by Smart *et al*. (2013).

Experiment 3 prior to recovery

A reliable understanding of how conditions evolved while Experiment 3 was running is of course important to the interpretation of the results of the experiment. Smart *et al*. (2012b) provide an update of the monitoring results for each of the MiniCan experiments up until the end of 2011. Regular water sampling was undertaken during the first few months of the experiments (during 2007), but no further samples were taken until late 2008 and late 2010. This reduction in sampling was apparently made in order to allow the water chemistry to stabilise, but it is not clear if this change in sampling was part of the original monitoring plan or whether plans had to be changed because the early sampling was found to have been influencing experimental conditions significantly (e.g. by introducing oxygen into the experiments). Ensuring an appropriate balance between obtaining sufficient monitoring data to understand the conditions of the experiment and limiting any perturbations to the hydro-chemical conditions that could influence the experiment is challenging. Decisions about monitoring frequency and reasons for any changes to a monitoring plan should be documented.

It is also important to ensure that monitoring and control systems are functioning as required during the experiment. Smart *et al*. (2012b, §4.1.7) note that there were many months of pressure gauge data losses owing to computer system failures; Smart *et al*. (2012b, Figure 4-12) indicate a gap in data for a period of about 10 months from mid-2007. No information is provided on the frequency with which checks are carried out on the functionality of monitoring equipment.

For all experiments, potential measurements revealed failures of the gold and platinum reference electrodes and the silver-silver chloride reference disc electrode

inside the support cage; the problem was resolved by switching to Silvion (silver-silver chloride) reference electrodes outside the support cage. Smart *et al*. (2012b, §4.1.7) suggest that the problem was caused by iron sulphide deposits on the electrodes. However, for all but Experiment 3, the electrodes were showing positive and increasing potential from the start of the experiments, which suggests that such an iron sulphide build-up would have been rapid (e.g. see results for Experiment 1 in Figures 4-13 and 4-14 of Smart *et al*., 2012b). In Experiment 3, a sudden failure appears to have occurred after 8,000 hours (Smart *et al*., 2012b, Figure 4-17) and the problem was resolved by switching to the Silvion reference electrode some three months later. Generally, in each experiment there appears to have been a long interval between an instrumentation failure and the response to modify the measurement system. Again, it is not clear how frequently checks are carried out that the monitoring equipment is functioning as expected.

The presentation of all corrosion rate data by Smart *et al*. (2012b) is welcomed. In particular, it is noted that the summary of AC impedance and LPR measurements and all the resulting copper corrosion rates for all five experiments are shown in Figures 4-26 and 4-27 of Smart *et al*. (2012b); high values of copper corrosion rates had been excluded from the previous MiniCan progress report (Smart and Rance, 2009, Figure 6-33), as noted by Baldwin and Hicks (2010). It is also noted that many figures relating to corrosion rate measurement data have been included in Appendix 1 of Smart and Rance (2009), although there is no narrative or explanation of any significant features of these figures. At the QA review meeting, SKB explained that, for all experiments, checks are now made that all data are included in discussion and analysis of results (SSM2011-2306-22, 2015).

Smart *et al*. (2012b) acknowledge that an increase in corrosion rate with time in all but Experiment 5 might have been caused by sulphide produced by sulphate reducing bacteria (SRB) in the system under anoxic conditions, but also suggest that the conductive iron sulphide layer deposited on surfaces in the support cage, as observed in the analysis of Experiment 3, could have resulted in misleadingly high corrosion rates being recorded. Smart *et al*. (2012b, §5.1) suggest that the SRB activity could also be responsible for the pH reduction with time observed in the experiments.

Clearly, information on sulphide concentrations and SRB populations in the groundwater during the experiments is important to understanding potential influences on copper corrosion and its measurement. Smart *et al*. (2012b, §4.1.1) note that the maximum sulphide concentration in the monitoring period was 0.22 mg/litre, but the maximum value shown in Table 4-1 of Smart *et al*. (2012b) is 0.084 mg/litre, which was measured in 2010 for Experiment 5. The maximum value for Experiment 3 is 0.059 mg/litre. The max value of 0.22 mg/litre was measured for Experiment 5 inside the cage in March 2007 (see Fig. 4-6, Smart *et al*. 2012b) but the fluctuations of the sulphide concentration between different sampling dates or analysis timelines are not clearly explained. ~~It is not clear if the value of 0.22 mg/litre is an error or relates to data obtained prior May 2007 that are not shown in the report.~~

Hallbeck *et al*. (2011) report information on evolving SRB populations while Experiment 3 was running (including measurement uncertainties), although Smart *et al*. (2012b) make little reference to the results of the microbial analysis. SRB populations under anoxic conditions are of particular interest, because oxidation of hydrogen by SRB acts concurrently with reduction of sulphate to hydrogen sulphide, which can cause copper corrosion (Hallbeck *et al*., 2011, §1.2). The hydrogen may have been produced by the corrosion of the cast iron and steel in the experiment. Large fluctuations in the population of SRB measured during the course of the

MiniCan experiment have been reported. The population decreased in the first year, but had increased by an order of magnitude after four years.

Generally, Smart *et al*. (2012b) have attempted to provide clear and consistent explanations of the copper corrosion and other monitoring results for all experiments. However, greater use of the results of the microbial analysis would have enabled a more comprehensive description and better understanding to have been documented of conditions while Experiment 3 was running. Of course, understanding conditions during the experiments has been made difficult because the experiments have been beset by problems and failures with monitoring instrumentation. In particular, a reliable method for the long-term monitoring of copper (and iron) corrosion rates under conditions such as those experienced by the MiniCan canisters appears not yet to have been found.

The recovery and analysis of Experiment 4 may provide data on copper corrosion under anoxic conditions that are more relevant to expected repository conditions. SKB points out that the corrosion rates measured electrochemically in Experiment 4 appear to be unreliable, probably because the swelling of the bentonite has led to deformation or damage to the electrical connections. SKB states that the microbial activity would be expected to be restricted under such conditions and sulphide production and any sulphide-induced corrosion would be limited and that the true corrosion rate in compacted bentonite will not be confirmed until Experiment 4 is dismantled. This observation points out the importance of Experiment 4 as it represents the closest analogy to the proposed SKB design, using fully compacted bentonite and thus it should be closely followed up and the retrieval plan and subsequent analysis should be carefully prepared so that valuable data will be preserved.

Analysis of Experiment 3

A key objective during the Experiment 3 extraction process and analysis was to minimise exposure of the materials to oxygen (Smart *et al*., 2012a). To this end, the canister was extracted from the borehole into a transfer tank full of deoxygenated groundwater, where it was placed in a transfer flask. The flask was transported to Amec Foster Wheeler's Culham Laboratories in the UK for analysis in a specially constructed inert-gas glovebox. This procedure ensured that the risk of contamination or atmospheric oxidation was minimal. Clear details of the design of the transfer tank, the transfer flask and the glovebox, as well as extraction, transfer and dismantling operations are provide by Smart *et al*. (2012a, §2 to §6).

Smart *et al*. (2012a, §7) provide details of much of the analysis of Experiment 3 samples. Details of the techniques used are included in Appendix 1 of the report and results of the scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis are presented in Appendix 2, although there is no narrative to explain the figures. Although it is not stated in the report, presumably the Experiment 3 data are stored in SKB's SICADA database with appropriate back-ups, as is the case for other MiniCan data (Baldwin and Hicks, 2010, Appendix A.2).

Smart *et al*. (2012a) do not discuss all of the Experiment 3 analysis in detail. Groundwater samples were taken during the Experiment 3 removal process and biofilm samples were taken from the surface of the copper canister as well as from other system components. The groundwater and biofilm samples were transferred to Microbial Analytic's laboratory in Mölnlycke, Sweden for treatment and analysis, as reported by Hallbeck *et al*. (2011). Also, some copper specimens were sent to Amec Foster Wheeler's laboratory in Risley, UK for metallurgical analysis, as reported by Smart *et al*. (2013). The metallurgical analysis aimed to investigate the extent of

any corrosion of the canister materials and to identify any possible microstructural features of interest. A more comprehensive understanding of copper corrosion processes could have been presented if the findings of all of the Experiment 3 analyses had been documented and assessed together.

In the main Experiment 3 report, Smart *et al*. (2012a, §7) provide clear descriptions of the analysis of the copper electrodes and weight loss specimen in the support cage. The copper electrodes were found to be covered in a black deposit (Smart *et al*., 2012a, §7.1.1). Raman spectroscopy was used to analyse the surfaces of the electrodes. The material was found to be non-crystalline, with indications of graphitic carbon and mixed oxide/sulphide species. The copper weight loss specimen was also covered in a black deposit. Again, Raman spectroscopy showed that the surface deposit was mainly amorphous (Smart *et al*., 2012a, §7.1.2). It was not possible to identify any specific materials, although the presence of copper sulphide or iron-copper sulphide was suggested, which indicates the presence of SRB. High concentrations of SRB were known to be present in the groundwater during the experiment.

A weight loss measurement was made on the copper specimen to determine the corrosion rate. Smart *et al*. (2012a, §7.1.2) indicate that great care was taken in weighing the sample at least three times at each stage of the cleaning process to determine the weight loss by corrosion. Smart *et al*. (2012a, §7.1.2) also provide information on measurement accuracy. An expression for the corrosion rate calculation is given and values for the terms in the expression are shown in Table 7-2 of Smart *et al*. (2012a). However, the duration of the experiment is not reported, although it is presumably about 33,200 hours (3.8 years) based on the results of the calculation. Also, the constant K in the corrosion rate expression is not discussed, but presumably it is a factor for converting from hours to years. Smart *et al*. (2012a, §7.1.2) calculated the corrosion rate for the copper weight loss specimen to be 0.15 ± 0.02 µm/year.

Smart *et al*. (2012a) attribute the relatively high corrosion rate for the weight loss specimen under anoxic conditions to a high sulphide content in the groundwater. However, as noted above, it was not possible to clearly identify the corrosion product on the specimen (presumed to be copper sulphide or iron-copper sulphide). No information on the sulphide content of the groundwater at the end of the experiment is given by Smart *et al*. (2012a), although a reduction in sulphate concentration is attributed to SRB activity.

Smart *et al*. (2012a, §7.3) also provide descriptions of the surfaces of the model copper canister, concluding that there is no indication of localised corrosion, although outer surface materials may include copper sulphide. One area of potential localised attack was attributed to machining damage, because no corrosion products were found to be present at the site.

Hallbeck *et al*. (2011, §4.1.2) noted that the black biofilm on the canister surface consists mostly of SRB; SRB prefer the stagnant water in the vicinity of the canister rather than flowing water in the support cage, which had only a thin biofilm of various groundwater bacteria. Hallbeck *et al*. (2011, §3.1) suggested that the SRB concentration may have increased because the rate of corrosion of the cast iron and steel support cage accelerated in the last year of the experiment, which increased the supply of hydrogen, thereby supporting SRB growth. The sulphate and hydrogen concentrations decreased and the ferrous iron concentration increased in the last year of the experiment. Curiously, the sulphide concentration in the groundwater showed only small variations during the experiment, increasing towards 2010 and 2011, with a small decrease in the final year of the experiment (Hallbeck *et al*., 2011, Table 3-4), although Hallbeck *et al*. (2011, §4.1.2) mistakenly report higher increase

between 2010 and 2011 than between the previous years (in Table 3-4 the highest increase in sulphide concentration has been reported between 2008 – 2010).

Smart *et al*. (2013) reported metallurgical analysis of Experiment 3 components. Copper U-bend specimens had been installed in the MiniCan experiment to assess the potential for stress corrosion cracking. Wedge Open Loaded (WOL) specimens had been included to investigate the susceptibility of copper to stress corrosion. The U-bend and WOL samples had been exposed directly to groundwater outside the Experiment 3 support cage. Weld areas of the canister were also analysed to assess the possibility of enhanced corrosion in the weld regions. The copper samples were stored for several months prior to analysis; Smart *et al*. (2013, §2.2) acknowledged the potential for oxygen ingress into the deaerated-water-filled and sealed boxes containing the samples during this period. A description of the analytical technique and equipment used in the metallurgical analysis is provided by Smart *et al*. (2013, §2.2).

Smart *et al*. (2013) reported that the WOL samples showed no signs of significant growth of the pre-crack on the samples (~1.5 mm long). However, no information is provided on the state of the cracks before the tests, or indeed the dimensions of the samples. Such information would have aided transparency in judgments about the results of the test. Also, Smart *et al*. (2013) reported that no additional loading had been applied to the WOL specimens during the experiment. That is, the samples were not actually stressed beyond residual crack stresses present; some form of tensile loading would be required to induce stress corrosion cracking. No reason for not loading the samples is given.

Smart *et al*. (2012a, §7.2.1) noted that manufacturer's tape had been left on the U-bend samples during the experiment. Presumably this did not affect the experiment conditions, although this possibility is not discussed by Smart *et al*. (2013). Smart *et al*. (2013) note evidence of surface roughening on the outside surface of the U-bend, which is attributed to the mechanical deformation introduced into the surface when the copper sheet was bent into the U-shape, rather than being due to any localised corrosion. There is no discussion of the condition of the samples prior to the experiment; pre-test surface analysis would have provided a reference for post-test analysis.

The analysis of the canister wall specimens and the electron beam weld components revealed a uniform surface corrosion product film, but no signs of localised corrosion (Smart *et al*., 2013, §3.3, §3.4). EDX analysis was reported for the inner surface of the canister sample, which showed the major elements present. It is not clear why there was no EDX analysis of the outer surface. At the QA review meeting (SSM2011-2306-22, 2015), SKB mentioned that there was not much value in analysing the MiniCan copper tubes, because the tubes were not examined in detail before setting up the experiments; there would be few reliable data on initial conditions with which to make comparisons. High resolution cross section microscopy analysis (including SEM) is usually used to be able to draw conclusions regarding the occurrence of local corrosion in form of pitting as well as for the characterization of the cracks resulted due to stress corrosion cracking.

Generally it can be concluded that as the exposure conditions are complex as well as the retrieval of the exposed samples, carefully prepared experiments and thorough pre-characterization of the samples before exposure as well as ensuring the correctness of the initial conditions would be needed for future corrosion experiments planned by SKB as well as follow-up and better monitoring during the in-situ exposure and more detailed after exposure characterization, using the lessons learned from the previously exposed and analysed samples. SKB should use all the results from the analysis of Experiments 1-5 and continue to verify if the results

from the MiniCan experiment are in agreement with the treatment of copper corrosion in the safety assessment.

3.2. The LOT Project

3.2.1. Overview of the LOT project

The Long-term Test of Buffer Material (LOT) project at SKB's Hard Rock Laboratory in Äspö was primarily developed to investigate bentonite buffer properties and mineral stability in a repository-like environment. However, SKB took the opportunity to include copper corrosion coupons in the bentonite with the aim of improving knowledge of copper corrosion under oxic conditions.

The LOT experiment comprises copper tubes containing heater elements surrounded by bentonite blocks and placed in boreholes at Äspö. There are two types of experiments 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.
- 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.

The parcels include copper coupons embedded in the bentonite blocks surrounding the copper tube. Based on the analysis of the corrosion coupons, the LOT test aims to determine the mean corrosion rate of copper and identify possible pitting corrosion and corrosion products, with the objective of testing the hypothesis that the mean corrosion rate under oxic conditions is less than 7 μm/year (e.g. Karnland *et al*., 2009).

The test parcels S1 and A1, and parcels A0 and A2 have been recovered and analysed. Copper corrosion analysis undertaken following extraction of parcels A0 and A2 are the focus of this QA review. In addition, a brief QA review is included of the real-time corrosion monitoring experiment undertaken using copper electrodes in a bentonite block from the LOT A2 test parcel, although the experiment is not formally part of the LOT project.

At the QA review meeting (SSM2011-2306-22, 2015), SKB reported that there has been no further activity on the LOT test and the test is not currently running as an SKB project. A project would be set up when SKB decides to recover the next test parcel for analysis, although there is no firm schedule or plan for this work. Also, because the LOT test is primarily a buffer project, those involved in buffer research would make decisions about the next phase of the test.

3.2.2. Motivation for QA review of the copper corrosion tests

QA issues associated with the LOT tests of bentonite behaviour were reviewed and reported previously by Hicks (2007) and the review of SKB's copper corrosion experiments reported by Baldwin and Hicks (2010) included a QA review of the

LOT project copper coupon tests based on information available at the time. SKB has published further results of the LOT project since the Baldwin and Hicks (2010) QA review. These publications include a compilation of corrosion data from the A2 test parcel (including data from the heated copper tubes), which was retrieved in 2006 after just over six years of operation (Wersin, 2013), and the final report on the A0 test parcel that was retrieved in 2001 after one year of operation (Karnland *et al*., 2011). This QA review is focused on these recent publications.

Baldwin and Hicks (2010) also commented on QA issues associated with the real-time corrosion monitoring that was being undertaken on a sample from the LOT A2 test parcel. No SKB reports had been published on this work at the time of that QA review. However, the results have now been reported by SKB (Rosborg *et al*., 2012) and a review of the report has been included.

As noted at the QA review meeting (SSM2011-2306-22, 2015), SKB has used the results of the A2 test parcel copper tube analysis in its interactions with SSM during the safety assessment review, but only in terms of observation on interactions between corrosion products and bentonite. The corrosion results from the A0 test parcel and the results from the real-time corrosion monitoring have not been used in such interactions.

3.2.3. A2 test parcel QA review

Project management, QA plans and procedures

The results of the analysis of parcel A2 were reported by Karnland *et al*. (2009). Baldwin and Hicks (2010) identified a number of QA issues regarding the A2 copper coupons relating largely to the lack of reporting of measurement accuracy and detection limits, unavailability of reference coupon data, uncertainties in corrosion rate estimates and the conditions and period over which corrosion occurred, and insufficient descriptions of the observed corrosion. Also, Baldwin and Hicks (2010) noted that, although SKB had made some observation of corrosion of the heated copper tubes at the centre of each LOT test parcel (Karnland *et al*., 2009, §9.2.2), more detailed analyses could be carried out. Finally, Baldwin and Hicks (2010) noted concerns over the quality, management and reporting of real-time corrosion monitoring, albeit in part relating to a test using copper electrodes in a bentonite ring that continued after retrieval of the A2 test parcel.

Wersin (2013, §2.3.1) does provide, for the A2 test parcel, estimates of the corrosion rate of copper based on measurements of copper concentration profiles in blocks of bentonite that had surrounded the heated copper tube (five blocks from hot areas and one from a colder area). The measurements were made by Clay Technology, Andra (France) and BGR (Germany) as discussed in detail in Karnland *et al*. (2009). As noted in the review by Baldwin and Hicks (2010), SKB stated that each organisation implemented its own laboratory QA procedures for the analysis.

A2 test parcel results

Corrosion rates were estimated based on the measured copper masses in the bentonite and the timescale of the test. Presumably the corrosion depth was estimated based on the assumption that corrosion occurred evenly over the exposed copper surface, although there is no discussion of this assumption. Wersin (2013,

§2.2) does comment on the condition of the copper tube at the start of the test, but there is no discussion of the condition of the copper tube surface after the test. Wersin (2013, §2.3.1) assumes that corrosion occurred at a uniform rate over five-years (rather than the six-year test period), but does not comment on uncertainties in this assumption or expectations regarding the persistence of oxic conditions during the test. A maximum copper corrosion rate of about 2 μm/year is reported based on the measurements on the bentonite from the hottest areas. The corrosion rate on the colder part of the tube was estimated to be about an order of magnitude lower.

QA issues relating to the analysis of the A2 test parcel copper corrosion coupons were identified previously by Baldwin and Hicks (2010, §4.1.2). Concerns were noted regarding the sufficiency of the discussions of uncertainty in the corrosion rate estimates. Wersin (2013, §2.3.1) notes that there are large uncertainties in the corrosion measurements, but does not elaborate on the nature of the uncertainties. Wersin (2013, §2.3.1) reports a maximum corrosion depth of 2.52 μm, but uncertainties in the period and conditions under which this corrosion occurred are not discussed. If the corrosion occurred over a five year period it would imply a corrosion rate of about 0.5 μm/year.

Wersin (2013, §2.5.1) discusses whether there is a sufficient inventory of oxygen in the test parcel to account for the amount of corrosion estimated to have occurred. The analysis indicates that, if the hottest parts of the system are last to resaturate and, as such, experience the longest period of exposure to air, then there would just have been sufficient oxygen in the parcel to explain the corrosion behaviour. There are several simplifying and averaging assumptions about equivalent corrosion depths in hot, warm and cold parts of the tube, but as general conclusion, a consistent and reasonably clear interpretation of an O_2-induced corrosion process resulted[1]. Clear descriptions are given of why there is considered to have been insufficient Fe(III) available to explain the copper corrosion (should such a hypothetical process occur) and why there would have been insufficient dissolved sulphide available to cause corrosion. It is important to mention that the calculated equivalent corrosion depths estimated by Wersin (2013) do not represent the total corrosion depths, as they were deduced only from the measurements of copper concentration profiles in the blocks of bentonite that had surrounded the heated copper tube. For the thickness of the corrosion products from the surface of the copper tubes after the removal from the bentonite, Wersin (2013) refers to corrosion estimates from the copper coupons placed in the warm (~ 75°C) block 22 and the cold (~ 30°C) block 30 obtained by weight loss measurements that were carried out by Bo Rosborg, see Appendix 3 in Karnland *et al.* (2009) .

3.2.4. A0 test parcel QA review

Project management, QA plans and procedures

The results of the A0 test parcel analysis had not been published at the time Baldwin and Hicks (2010) reviewed the LOT project QA process, even though the parcel was retrieved in 2001. At that time as noted by Baldwin and Hicks (2010, §4.1.4), the publication of results from the A0 test parcel analysis was given a low priority by SKB. However, the results of the corrosion coupon analysis were presented and discussed in a QA review meeting with SKB in 2010 (Baldwin and Hicks, 2010, §4.1.2).

[1] It is noted that the mass loss of copper in the cold blocks is give as 20.5 mmole rather than the correct 2.9 mmole.

Karnland *et al*. (2011) provide a general description of the LOT project and the installation, termination and recovery of the A0 test parcel. The description is similar to that provided by Karnland *et al*. (2009) for the A2 parcel test. The A0 test parcel included four copper coupons embedded in the bentonite blocks surrounding the copper tube. The results of the examination of the copper coupons are presented briefly in Appendix B of Karnland *et al*. (2011); the results are not discussed in the main text of the report.

Karnland *et al*. (2011) provide no reference to a QA plan for the A0 corrosion coupon analysis. However, in the QA review meeting (SSM2011-2306-22, 2015), SKB commented that the QA process was similar to that undertaken for the A2 corrosion coupons. As noted by Baldwin and Hicks (2010, Appendix A.1), this involved approval of a QA plan for the work by SKB and subsequent checks that the work was undertaken according to the plan.

The bentonite blocks containing copper coupons were sealed and delivered to Studsvik AB for analyses after most of the analyses of the bentonite had been performed by Clay Technology. This meant that the analysis of the coupons took place almost a year after retrieval of the bentonite blocks (Karnland *et al*., 2011, Appendix B3). Karnland *et al*. (2011) do not comment on whether this delay in sending the coupons for analysis could have affected the condition of the coupons significantly. The conditions under which the blocks containing the coupons were stored and the potential for corrosion to occur during the storage period are not discussed.

The experimental procedure for analysis of the copper coupons is listed in Karnland *et al*. (2011, Appendix B4), and includes various activities to photograph, clean, weigh and analyse the coupons. It is stated that the water from each step in the cleaning process was saved for possible later examination of loose corrosion products. Such analysis could enhance understanding of the corrosion reactions, but no information is given regarding plans or a schedule for the work.

Corrosion test results

Tests on copper coupons A022A (which had been exposed to a temperature of 80°C during the LOT project) and A030C (which had been exposed to a temperature of 35°C) are reported by Karnland *et al*. (2011, Appendix B). No explanation is given as to why coupons A022B and A030D were not tested. It is stated that coupon A022B was archived in its plastic container for later transport to Clay Technology, but no indication of the tests to be performed on the coupon is given (Karnland *et al*., 2011, Appendix B4).

The discussion of the results of the A0 corrosion tests in Karnland *et al*. (2011, Appendix B) is similar to that presented for the A2 parcel tests (Karnland *et al*., 2009, Appendix 3) and similar comments regarding QA issues can be made to those documented by Baldwin and Hicks (2010) on the A2 corrosion test results. In particular:

- There is no quantification of data uncertainty or qualitative discussion of the sources of uncertainty, and no discussion of detection limits, measurement accuracy and equipment calibration. Karnland *et al*. (2011, Appendix B4) does refer to a contractor report (Rosborg, 1998) (presumably publically available) for details of the actions undertaken in earlier analysis of other corrosion coupons, but full details of the A0 corrosion coupon measurement techniques, data and results do not appear to have been published.

- As for the A2 coupons, the A0 coupons were described as showing uneven corrosion attack, but no signs of pitting corrosion. However, again, no information on cross-section depth measurements was provided to indicate the extent of the variations in corrosion across the surfaces of the coupons.
- There is no discussion of why the weight loss of the A0 coupons is greater than that of the A2 coupons, despite the fact that the duration of the A0 test was several years less than that of the A2 test. The estimated corrosion rates of the A0 coupons are an order of magnitude greater than those of the A2 coupons.
- There is no discussion of the expected time-dependence and spatial distribution of redox conditions in the vicinity of the corrosion coupons, or of how variations in conditions might have affected corrosion rates. In particular, no explanation is given as to why, contrary to the A2 results, the estimated corrosion rates are independent of the temperature to which the coupons were exposed. Corrosion rates of 3.5 μm/year and 3.4 μm/year were estimated for coupons A022A and A030C, respectively, assuming uniform corrosion over the period of the heater test.
- No estimates have been made of copper corrosion rates based on analysis of copper in the bentonite adjacent to the heated copper tube used in the A0 test.

It is curious that micro-hardness indentation marks that had been made on the A0 and A2 test coupons could not be found after either of the tests (Karnland *et al*., 2009, Appendix 3; Karnland *et al*., 2011, Appendix B6). No explanation is offered regarding the processes that could have caused the marks to have disappeared.

A compilation of the A0 test parcel copper corrosion data similar to that published for the A2 test parcel (Wersin, 2013) would be informative. This could include an interpretation of the copper profiles in the bentonite adjacent to the copper tube shown by Karnland *et al*. (2011, Figure 8-4). These profiles indicate much greater corrosion in the hotter regions of the copper tube than in the cooler regions. The earlier report of the A2 test parcel (Karnland *et al*., 2009, §9.2.2) discusses the measured copper profiles for both the A2 and A0 parcels. The copper content of the bentonite in the hottest region of the six-year A2 parcel test was reported as being only 15% greater than in the comparable region of the ~one-year A0 parcel test. Karnland *et al*. (2009, §9.2.2) interpreted this finding as an indication that corrosion proceeds at a higher rate in the early stages of a test. A discussion of the evolving oxygen distribution and availability for corrosion similar to that presented by Wersin (2013) for the A2 test parcel would aid understanding of the A0 corrosion results and estimated corrosion rates under oxic conditions.

Also, an interpretation of the conditions to which the A0 copper coupons were exposed during the LOT tests and during the subsequent storage period would help understanding of the high corrosion rates estimated for the coupons. The length of time that copper is subject to a set of conditions is a key parameter in estimating corrosion rates. Note that Karnland *et al*. (2011, Appendix B5) report a 498-day exposure time at full temperature, but also give a total exposure time of 710 days; it is unclear what this total exposure time is referring to.

In conclusion, valuable information is still possible to be obtained from the LOT experiments which could improve the knowledge of the in-situ behaviour of the copper canisters embedded in bentonite. The experimentally obtained corrosion rates should be considered to improve the safety assessment.

3.2.5. Real-time Corrosion Monitoring QA review

Project management, QA plans and procedures

Rosborg *et al*. (2012) describe the electrical resistance measurements undertaken using copper electrodes in a bentonite block from the LOT A2 test parcel following its retrieval from the HRL. After retrieval of the parcel in 2006, the bentonite ring containing the electrodes was removed and placed in a plastic container with a copper sheet (counter electrode) and two reference electrodes. The container was sealed with paraffin. In 2007, copper and platinum electrodes were added. Initial electrochemical measurements were performed at KTH in Stockholm, before the package was transported to the Slovenian National Building and Civil Engineering Institute in Ljubljana, Slovenia, for installation of electrical resistance sensors. The experiment ran until 2011.

Rosborg *et al*. (2012) make no reference to a QA plan for the analysis, and the objectives and requirements of the experiment and its role in SKB's research and development programme are not clear. However, the experiment is referred to in SKB's RD&D programme report (SKB, 2013, §24.2.8).

Also, Rosborg *et al*. (2012) make no formal references to QA procedures followed in conducting the experiment, but thorough descriptions of the instrumentation used, measurement techniques and procedures followed are included in Section 3 of the report and appendices. Measurement data are included in the appendices, although the basis for selecting data for inclusion is not discussed (e.g. selected corrosion potentials are shown in Table A-1).

Real-time corrosion monitoring results

Rosborg *et al*. (2012, §3) provide clear descriptions of the set-up of the experiment, conditions at the start of the experiment, electric resistance (ER) and electrochemical measurement techniques and post-test examination.

Rosborg *et al*. (2012, §3.2) note that corrosion rate estimates using ER sensors assume that corrosion is uniform; uneven corrosion influences the calculated corrosion rate. However, the post-test examination revealed corrosion to be uneven and localised, which led to overestimates of the corrosion rate. Indeed, highly localised corrosion caused full penetration of the exposed electric lead of one sensor and resultant failure of the sensor. Rosborg *et al*. (2012, §4.4.2) note that the effects of uneven corrosion on recorded corrosion rates is the subject of a university research project in Slovenia. It would be of interest to determine if it is possible to derive a method for detecting uneven corrosion in ER measurements and to correct the derived corrosion rates accordingly. Rosborg *et al*. (2012, §4.4.1) found that electrical impedance spectroscopy (EIS) appears to give results that are more consistent with the findings of post-test examination.

From a QA perspective, other minor observations are:

- Measurement uncertainty is recorded for some data in the form of ranges (e.g. EIS measurement data in Table 4-3). However, such reporting of uncertainties has not been done consistently for all data.
- There appear to be some gaps in monitoring data that are not explained. For example, the reason for the break in ER measurements between March 2010 and January 2011 is not given.
- Rosborg *et al*. (2012, §3.3.3 and §3.3.4) express doubts on the suitability of the SmartCET corrosion monitoring system and the Electrochemical Frequency Modulator (EFM) technique, respectively. Although some

corrosion rate results using these techniques are presented by Rosborg *et al*. (2012, §4.5.2 and §4.5.3), there is no discussion of the reliability of the methods and results.

Rosborg *et al*. (2012, §5.1) concluded that the conditions during the experiment were oxic and the dominant corrosion species were oxygen and chloride (in the groundwater), which was confirmed by analysis of the corrosion products. The corrosion was uneven and localised. Disturbances at the time the parcel was removed and as a result of interventions to examine and maintain the reference electrodes are likely to have introduced oxygen, which would have influenced the corrosion rate. The electrical impedance spectroscopy, EIS, analysis shows that the corrosion rates started high (of the order 10 μm/year), but fell to of the order 1 μm/year by the end of the experiment.

In conclusion, the real-time corrosion monitoring was quite complex, disturbances during retrieval process occurred but overall the results are important for building an understanding for the in-situ behaviour of the copper canisters embedded in bentonite. Novel real-time monitoring techniques for doing in-situ corrosion measurements have been implemented but further development for using them for this specific application would be needed. Important result regarding the corrosion behaviour was obtained via post-test examinations that revealed corrosion to be uneven and localised and that the estimated general corrosion rate could have been overestimated. The inclusion of high resolution SEM/FIB microscopy cross section of one of the exposed samples is welcomed. This type of analysis can reveal valuable information in case more localized corrosion takes place as well as when the general corrosion is uneven.

3.3. Copper corrosion tests in oxygen-free pure water

3.3.1. Overview of copper corrosion tests

SKB commissioned researchers at the Ångström Laboratory of Uppsala University's Department of Chemistry (Boman *et al*., 2013; 2014) to undertake experiments aimed at investigating claims made by other researchers (e.g. Szakálos *et al*., 2007) that copper could corrode in water in an anoxic environment. The evidence for such claims derives from observations of hydrogen gas production during copper corrosion experiments using pure water; the hydrogen gas has been interpreted as arising from copper corrosion (e.g. Szakálos *et al*., 2007). The experiments being undertaken by Boman *et al*. (2013; 2014) represent the focus of substantial work to understand copper corrosion mechanisms that is being carried out as part of SKB's research programme (SKB, 2013, §24.2.8).

The main copper corrosion experiment has involved placing pieces of copper foil in a Duran glass (borosilicate) beaker filled with ultrapure water. Boman *et al*. (2014, §2.3) indicate that eight pieces of copper (47 x 12.5 mm) were placed in the beaker and these were attached to a quartz glass holder. A Duran glass sheet was also placed in the beaker. The beaker was placed in a stainless steel reaction chamber. A copper gasket was used to seal the stainless steel lid. The reaction chamber was connected to a vacuum chamber, with a 23-mm-diameter, 0.1-mm-thick palladium foil seal between the reaction chamber and vacuum chamber; palladium is only permeable to hydrogen gas. The vacuum chamber included pressure-gauge equipment to measure the hydrogen gas pressure as an indication of the amount of copper assumed to have oxidised.

Two reference experiments were also set-up that were identical to the main experiment, except for the exclusion of copper pieces (i.e. each reaction chamber only held a Duran glass sheet) and in one case the use of a silver-plated copper gasket to seal the reaction chamber lid.

In addition, five long-term tests were included in which the reaction chambers were not connected to pressure-gauge equipment. The reaction chambers included palladium seals to allow any hydrogen generated to escape, thereby avoiding pressure build up that might influence reaction kinetics. These experiments were intended to be stopped and the samples and water analysed progressively (after one, three, six, nine and twelve months). Seven copper foils 20 x 10 mm were used in these five experiments and a Duran glass sheet was included in each experiment.

The experiment set-ups are illustrated in Figure 2. The experiments were set up in a glove box with a nitrogen atmosphere and temperature was controlled by inclusion of a heating jacket around each reaction chamber (see Figure 3).

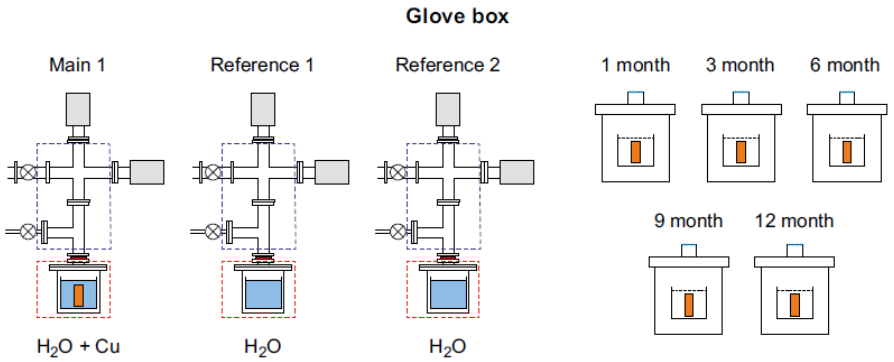

Figure 2: Set-up of main and reference experiments with pressure monitoring and five experiments without pressure monitoring (for analysis at different times as indicated) (Boman *et al.*, 2014).

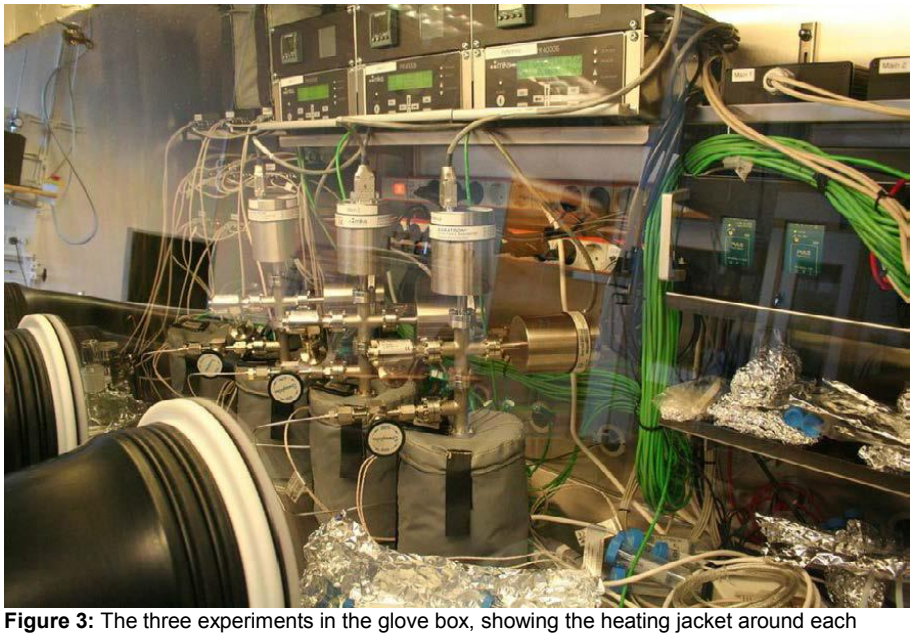

Figure 3: The three experiments in the glove box, showing the heating jacket around each reaction chamber and the vacuum chamber with pressure gauge above the reaction chamber (Boman et al., 2014).

3.3.2. Motivation for QA review of the copper corrosion tests

In recognition of the debate on the potential for copper corrosion in pure water under anoxic conditions, SKB (2010, §5.4) evaluated the effects of such corrosion as a 'what-if' calculation in support of the SR-Site safety assessment. The what-if calculation found that the depth of corrosion by this mechanism on a 5-cm-thick copper canister would be, at most, on the millimetre scale over 10^6 years. The analysis was based on hydrogen equilibration pressure data from the experiments reported by Szakálos *et al*. (2007) (which may not be appropriate for disposal conditions) and the assumption that the rate of corrosion is limited by the rate of transport of dissolved H_2 through bentonite away from the canister once the equilibration pressure has been reached. In a review of SKB's treatment of copper corrosion mechanisms in the SR-Site safety assessment, Scully and Hicks (2012, §2.1.1) observed that controlled experiments and improved diagnostic methods were needed to understand the anoxic copper corrosion process and the view that, if such a process occurred, it would be limited by the rate of H_2 transport from the copper surface (SKB, 2010, §5.4).

Also, a review of copper corrosion processes undertaken by Macdonald and Samin (2011) on behalf of SSM confirmed that copper corrosion in oxygen-free water is possible thermodynamically, but only when the concentration of the corrosion product Cu^+ and the hydrogen pressure are very low. Thus, the rate of copper corrosion is controlled by the rate of transport of the corroding species (H^+) to the copper surface and the rate of transport of the corrosion products (Cu^+ and H_2) away from the copper surface. Macdonald and Samin (2011) concluded that the lack of agreement between different copper corrosion experiments reflects differences in the initial states of the experiments in terms of hydrogen partial pressure and hydrogen equilibrium pressure.

Clearly, a high standard of QA, quality control and reporting of copper corrosion tests in pure water under anoxic conditions is extremely important in ensuring the

reliability of the corrosion test results. This QA review focuses on the QA aspects of the experiments reported by Boman *et al*. (2013; 2014)

3.3.3. Copper corrosion test QA review

Project management, QA plans and procedures

Although there is no explicit QA plan for the experiment, comprehensive details of the planning, design and conduct of the experiments are provided by Boman *et al*. (2014), as would be expected in a QA plan for such research. During the project QA review meeting (SSM2011-2306-22, 2015), SKB noted that a preliminary study was undertaken prior to the decision to carry out the experiment and much of the planning and design was done in the preliminary study. SKB and its copper corrosion Reference Group have overseen the experiments through regular meetings, discussions and review of reports.

Boman *et al*. (2014) aimed to undertake similar experiments to those reported by Szakálos *et al*. (2007) and others using the purist copper and cleanest water practicable, and the means by which this has been achieved is well documented in the report. In particular, clear details are provided on:

- The purity of the copper and surface cleaning.
- Purification of the water (especially removal of dissolved gases).
- The basis for selection of borosilicate glass beakers (Duran glass) to minimise leaching of elements from the glass and cleaning of the glass before the experiments.

The inclusion of analysis certificates (Boman *et al*., 2014, Appendix A) to show the composition of the copper, palladium and water provide confidence in the purity of the materials. Also, the detailed drawings and equipment descriptions provided by Boman *et al*. (2014, Appendix A) give confidence in the reproducibility of the experiments. Minor observations are that:

- The thickness of the copper foil used in the main experiment is not stated clearly in the report, but is presumed to be 0.25 mm based on the figure shown in Appendix C1 of Boman *et al*. (2014).
- Details of the dimensions of the Duran glass sheets used in the experiments are not provided.
- It is not clear from the report how the seven copper foils were distributed in the five long-term experiments without pressure monitoring or what the thickness of the foil was (presumably 0.50 mm).

Information is provided on the systems for controlling the experiments and logging monitoring data, including the precaution of installing a power supply back-up (Boman *et al*., 2014, §3.1.3). However, it is not clear if the raw data from the experiments have been documented or made available in a form that would be publically accessible.

A clear description of the procedures used to establish initial pressure conditions in the vacuum chamber is provided in Boman *et al*. (2014, §3.2). The pressure reductions in the vacuum chambers when the glove box atmosphere was changed to nitrogen gas (before the vacuum chambers were connected to the reaction chambers) appear to have been unexpected. These pressure reductions are attributed to hydrogen gas transfer into the glove box through the vacuum chambers' palladium seals, although there appears to be some uncertainty about this process.

Boman *et al*. (2014, §4) provide helpful descriptions of the methods for analysing the solid samples (including analysis depth of non-destructive techniques and details of melting analysis), the gas phase (mass spectrometry), and the water. Information on measurement detection limits is provided.

At the QA review meeting, SKB noted that it does not have access to the primary data from the experiments, but the data could be obtained if necessary. SKB reports could include raw data from the experiments. Also, SKB considers that the publication of the results of the experiments in peer-reviewed scientific journals and in SKB reports should ensure an appropriate level of peer review as well as comprehensive reporting of the details of the experiments.

Corrosion test results

The main experiment and two reference experiments all record hydrogen gas generation, with most hydrogen generated in the main experiment that included copper and least generated in the experiment that included no copper and a silver-plated copper seal. Boman *et al*. (2014, §5.1) reported that it was only possible to confirm that hydrogen was the major species in gas generated after redesigning the equipment to connect a mass spectrometer. It is not clear why the mass spectrometer was not included at the start of the experiment, when it was included and whether its installation affected the experiment in any way.

In each experiment the gas pressure increased to a maximum before gradually falling. Boman *et al*. (2014, §5.1) noted that it is not possible to say where the hydrogen gas generation takes place, because the hydrogen can pass though the palladium seal in either direction. In each test, the gas pressure decreases after attaining a maximum, rather than reaching a steady state, which implies that there is hydrogen leakage from each system. This behaviour was interpreted by Boman *et al*. (2014) as being evidence of a small lateral leakage of hydrogen through the palladium seal coupled with a reduction in the kinetics or the rate of change of gas production.

A clear discussion of the results of the test analyses at one, three and six months is provided in Section 5 of Boman *et al*. (2014). Key findings are:

- There is no observable CuO or CuO_2 on the copper surfaces at any time.
- The copper surface adsorbs hydrogen, carbon and oxygen, which is attributed to adsorbed water and hydrocarbon molecules. These findings are similar irrespective of the length of the test and are similar for copper that has not been used in the tests, which indicates a constant hydrogen content at the surface.
- Traces of copper were detected on the Duran glass samples.
- There are small increases in the copper content of water with time, which is interpreted as deriving from the glass beaker. This interpretation is supported by the observation that the iron concentration in the water increases proportionally; the glass also contains iron.

Thus, Boman *et al*. (2014) observed hydrogen production, but not copper corrosion. The rate of hydrogen generation was found to be similar whether or not copper is present. Boman *et al*. (2014, §7) argued that the hydrogen could derive from stainless steel in the system or from residual hydrogen in the pressure gauges, and the copper in the water could derive from the glass. With reference to Macdonald and Samin (2011), Boman *et al*. (2014) acknowledged that the equilibrium pressure of hydrogen gas in a copper-water system is much lower than the gas pressures measured in the closed experiments, but noted that hydrogen gas was allowed to

leave the experiments with open systems so that any corrosion reaction would be able to proceed in those experiments.

In conclusion, the corrosion experiments in oxygen free water performed by Boman et al. (2014) that were initially planned to be similar with the experiments performed by Szakálos et al. (2007) have introduced some additional uncertainties and thus the hydrogen release is difficult to be explained. A good approach was to start with very pure copper, pure water and the choice of the Duran glass that leaches very little impurities into the water but to control of the leakages in and out from the system are critical for understanding the processes that take place.

3.4. Atmospheric and aqueous copper corrosion tests

3.4.1. Overview of atmospheric corrosion tests

SKB has included experiments to investigate the corrosion behaviour of copper under atmospheric conditions as part of its research programme being undertaken at the Äspö HRL (Werme *et al*., 2002). The experiment involved exposing copper coupons to the underground atmosphere at 450 m depth in the Äspö HRL with the aim of understanding the corrosion behaviour of a copper canister before saturation of the deposition hole after disposal. The results of the experiment have been published in a short paper by Taxén (2004), but full details have not been published.

Ten coupons were placed in each of three different set-ups at the HRL: (a) suspended inside a cylinder with an open base, which allowed exposure to the humid sulphide-containing atmosphere, but provided protection from convection and water; (b) as (a) but with the cylinder surrounded by bentonite blocks to control humidity; and (c) as (a), but with the cylinder heated to about 75°C. The chambers were covered to avoid any influence of light on copper corrosion. The experiments began in 1999 and were intended to run for three years.

3.4.2. Motivation for QA review of atmospheric corrosion tests

The copper corrosion tests at Äspö provided the opportunity for SKB to gain an understanding of atmospheric corrosion behaviour under repository-like conditions. As discussed at the QA review meeting, the results of the tests have been used by SKB to support the view that atmospheric corrosion of a copper canister would be less than 1 µm in a repository (SSM2011-2306-22, 2015). Therefore, it is important to understand the reliability of the results by reviewing how QA issues were addressed during the experiment.

3.4.3. Atmospheric corrosion test QA review

Taxén (2004) reported that the heater failed to function for periods of weeks, because of power failures, although no details of the temperature fluctuations are provided. No indication is given regarding the frequency with which the control systems were checked. Also, some coupons fell into the bentonite and some coupons were collected for analysis after three and a half years. Taxén (2004) lists the cleaning and analysis methods, but few details of the methods or procedures followed are provided.

The surface of one specimen from each set-up was analysed and only the heated coupon was found to include chloride. The corrosion rate was estimated to be of the order 0.1 μm/year for the heated sample and slightly less for the unheated samples. Taxén (2004) noted that changes between wet and dry conditions could cause salt deposit to occur which could result in localised corrosion under humid conditions.

Little information about QA aspects of the experiments is provided by Taxén (2004) and so, in this respect, it is difficult to make judgments on the reliability of the work. The results of the experiment have not been published in an SKB report. However, the problems associated with running long-term tests in a repository-like environment were apparent for this early test. It seems to be typical in these experiments for there to be a long period between a system failure and the detection and correction of the problem.

3.4.4. Overview of aqueous corrosion tests

Taxén (2009) provides a summary of the aqueous corrosion tests in which copper coupons were exposed to natural, reducing groundwater at two depths at the Äspö HRL. At one location (at a depth of about 300 m) the groundwater was chloride-rich and at the other location (at a depth of about 150 m) the groundwater was sulphide-rich. The copper coupons were placed in pressure vessels (six coupons per vessel) through which the groundwater could flow; water was fed into the vessels from sealed-off fractures in the rock.

The tests began in 2001 and were intended to run for three years. However, the tests were stopped after two and a half years because flow into the vessels was observed to be decreasing, with the potential for oxygen to diffuse into the system and cause corrosion.

3.4.5. Motivation for QA review of aqueous corrosion tests

The aqueous corrosion tests provided the opportunity for SKB to gain an early understanding of copper corrosion in chloride- and sulphide-rich groundwater under repository-like conditions. The results of the tests have not been used by SKB to support the SR-Site safety assessment, but comment is anyway made here on QA issues associated with the experiment.

3.4.6. Aqueous corrosion test QA review

Taxén (2009) presents data on the composition of the groundwater sampled at each experiment location at different times leading up to the start of the tests, but no information is provided on groundwater composition during the tests. Although it had been intended to measure the corrosion potential of one coupon and the redox potential and pH of the water during the experiment, the measurements were found to be erratic and non-systematic. Taxén (2009) did not present the results of the measurements and no explanations of why the measurements were unreliable were offered.

Taxén (2009) reported the results of surface observations and weight loss measurements made on three of the copper coupons from each pressure vessel. No reason is given as to why the other coupons were not analysed. The observed copper corrosion in sulphide-rich water (at a mean rate of 0.560 μm/year) was judged to be as expected and caused by the sulphide. Corrosion in the chloride-rich water (at a mean rate of 0.301 μm/year) was judged to be caused either by the chloride or possibly by low concentrations of sulphide (below detection) in the groundwater. The corrosion products on the surfaces of the coupons do not appear to have been analysed beyond visual inspection.

Generally, it is difficult to judge the QA status of these experiments because of the lack of information on any QA plan for the experiments, on procedures followed at each stage of the set-up, running and analysis of the experiments, on quality control of materials used in the experiments (such as the copper coupons), on instrumentation used (including detection limits and calibration), and on recording and storing data. At the QA review meeting (SSM2011-2306-22, 2015), SKB acknowledged a lack of knowledge of such QA information, although it was noted that the copper was known to have derived from the manufacturing of a full-scale canister. SKB noted that, because the experiments had not run as planned in terms of flow conditions, less effort had been put into the final analysis. The results of the experiment have not been published in an SKB report. Also, the results of the experiments were not used to support the SR-Site safety assessment or to support subsequent interactions with SSM on copper corrosion issues.

4. Conclusions

The assessment of QA in SKB's copper corrosion experiments has involved a review meeting followed by a detailed review of reports on the following selected corrosion experiments, focusing on QA and quality control aspects:

- MiniCan Experiment 3 (analysis of copper coupons and canister).
- The LOT project A2 parcel tests (analysis of copper tube).
- The LOT project A0 parcel tests (analysis of copper coupons).
- Copper corrosion tests in oxygen-free pure water at Uppsala University.
- Atmospheric and aqueous copper corrosion tests at the Äspö HRL.

The broad conclusions from each experiment and a number of general conclusions are set out in the following sub-sections. Conclusions on the real-time copper corrosion monitoring experiment relating to the LOT A2 parcel are included.

4.1. MiniCan Experiment 3

Generally, the MiniCan project reports provide clear and consistent explanations of the project and, in particular, operations to recover Experiment 3 from its borehole at the HRL for analysis. Key observations relating to the QA of MiniCan Experiment 3 are as follows:

- Much of the work to recover and analyse Experiment 3 was novel and should provide good experience and learning when plans are developed for the recovery and analysis of the other MiniCan experiments.
- Future plans for the MiniCan project and the relevance and importance of the experiment to SKB's ongoing repository development programme are not clear; it would be helpful if such information was documented in progress reports as well as in SKB's RD&D programme report.
- Understanding conditions during the MiniCan experiments has been made difficult because of problems and failures with monitoring instrumentation during the experiments, and in many cases, there appears to have been a long period between the time of monitoring failure and the response to address the failure. SKB should ensure that checks on the functionality of monitoring equipment are carried out with sufficient frequency that important data are not lost.
- The presentation of all corrosion rate data in the latest MiniCan progress report is welcomed; high values of copper corrosion rates had been excluded from the previous progress report.
- A reliable method for the long-term monitoring of copper corrosion rates under conditions such as those experienced by the MiniCan canisters appears not yet to have been found. Electrochemical measurements generally appear to overestimate corrosion rates. In particular, in the MiniCan experiments, iron sulphide films appear to dominate the electrochemical response, leading to substantial and ever-increasing overestimates of corrosion rates.
- In setting up experiments such as MiniCan in the future, it would be beneficial to characterise all components, such as model copper canisters and copper coupons prior to the experiments in order to provide clear references for post-test analyses.

- SKB should ensure that experiments are set up and verified carefully to build confidence that intended conditions (such as stress conditions on stress corrosion samples, the presence or absence of oxygen during exposure) are imposed correctly.

Of course, the MiniCan experiment was not designed specifically to understand copper corrosion processes. As discussed at the QA review meeting (SSM2011-2306-22, 2015), to include copper corrosion coupons in experiments on other processes introduces too many process couplings and uncertainties in the analysis. SKB stated that corrosion experiments would not be undertaken as part of other experiments in the future; current practice is to design carefully controlled corrosion experiments. However, despite the difficulties in monitoring, controlling and understanding conditions during the MiniCan experiment, the analysis and reporting of Experiment 3 appear to be of sufficient quality that they reliably support SKB's qualitative judgment that the results do not contradict the treatment of copper corrosion in the SR-Site safety assessment, although it is noted that the non-uniform/localised corrosion processes should be more carefully investigated by SKB. The recovery and analysis of Experiment 4 may provide data on copper corrosion under anoxic conditions that are more relevant to expected repository conditions. SKB should continue to closely follow-up this experiment and prepare the retrieval plan and subsequent analysis using the lessons learned from the previous experiments. SKB should continue to monitor and use all the results from the analysis of Experiments 1-5 to verify if the results are in agreement with the treatment of copper corrosion in the safety assessment.

4.2. The LOT A2 test parcel

In the previous QA review of copper corrosion experiments, Baldwin and Hicks (2010) noted that analysis of the heated copper tubes at the centre of each LOT test parcel might improve the knowledge base on copper corrosion under disposal conditions. Thus, the estimates of corrosion rates based on measurements of copper concentration profiles in the bentonite that had surrounded the heated copper tube in the A2 test parcel is welcome.

It is accepted that the calculation of the corrosion rate needs to be based on a number of assumptions about evolving conditions (such as oxygen distributions) during the test. However, the uncertainties in these assumptions should be explored more fully than reported in order to demonstrate understanding of the range of possible corrosion rates.

Even so, it can be concluded that the analysis clearly adds to the understanding of copper corrosion under oxic conditions, although it is understood that the corrosion rate estimates have not been used by SKB in support of safety assessment assumptions.

4.3. The LOT A0 test parcel

The publication of results from the A0 test parcel analysis was given a low priority by SKB. The A0 test parcel was retrieved in 2001, but the analysis report was not published until 2011. The low priority of this test is reflected in the fact that the

copper corrosion results have not been used by SKB to support interactions with SSM during the SR-Site safety assessment review.

Many decisions (such as which copper coupons to test) and assumptions (such as about the period of corrosion) were made in the analysis that have not been explained in full. Also, observations about unexpected differences in results from the A0 and A2 corrosion coupon analyses have not been explored in great detail. An interpretation of the conditions to which the A0 copper coupons were exposed during the LOT tests and during the subsequent storage period would help understanding of the high corrosion rates estimated for the coupons.

A compilation of the A0 test parcel copper corrosion data (similar to that produced for the A2 test parcel), including an interpretation of the copper profiles in the bentonite adjacent to the copper tube and the evolving oxygen distribution and oxygen availability for corrosion, would be informative.

4.3.1. Real-time Corrosion Monitoring

SKB's report of the real time copper corrosion monitoring experiment provides a comprehensive description of the experiment and its analysis. However, it is not clear how the results of this work will be used by SKB. The corrosion rates were high throughout the experiment and consistent with expectations for copper corrosion under oxic conditions. The electrical impedance spectroscopy, EIS, method appears to give reasonable estimates of copper corrosion rates under such conditions. The electric resistance, ER, measurements are less reliable because of the effects of uneven and localised corrosion.

4.4. Copper corrosion tests in oxygen-free pure water

SKB commissioned researchers at Uppsala University to undertake experiments aimed at investigating claims made by other researchers that copper could corrode in water in an anoxic environment. The experiments represent the focus of substantial work to understand copper corrosion mechanisms that are being carried out as part of SKB's research programme.

SKB stated at the QA review meeting that the experiments are not subject to an industry standard QA system (as is the general case for university-run projects). Also, SKB does not always have total control over experiments performed at a university. Even so, the consultation with the copper corrosion Reference Group and the clear documentation of the experiments demonstrates a high level of expertise applied to the research and attention to QA and quality control. However, although every effort has been made to control conditions at the start of the experiment and during the experiment, in practice, such control has proved challenging. It has proved difficult to attribute hydrogen generation or the observation of small concentrations of copper in water to any particular process.

The suggested redesign of equipment to eliminate leakage and keep background levels of hydrogen gas to a minimum, if successful, should support understanding of the hydrogen generation rate and equilibrium pressure. However, the source of hydrogen gas would also need to be identified in order to underpin conclusions about copper corrosion.

Publication of the raw data from the experiments would enable other researchers to evaluate and review the findings. SKB reports could include raw data from the experiments.

Also, the experiments being performed by Microbial Analytics in Gothenburg (see Table 1) to check the interpretation and conclusions made by Boman *et al*. (2014) should help to build confidence in the understanding of how copper behaves in pure water under anoxic conditions.

4.5. Atmospheric and aqueous copper corrosion tests

SKB included experiments to investigate the corrosion behaviour of copper under atmospheric and aqueous conditions as part of its research programme being undertaken at the Äspö HRL. It is difficult to judge the QA status of these experiments because of the lack of information on any QA plan for the experiments or on procedures followed at each stage of the set-up, running and analysis of the experiments. The results of the experiments have not been published in SKB reports.

4.6. General Conclusions

Only a limited number of the results of the copper corrosion experiments is used by SKB to derive copper corrosion rates for use in the repository safety assessment. Instead, SKB generally uses the results of these experiments in a more qualitative way. That is, SKB uses the experiments to support its understanding of copper corrosion processes under repository conditions. SKB has concluded that the results to-date do not contradict its treatment of copper corrosion in the SR-Site safety assessment, although it is noted that SKB has not made reference to the non-uniform/localised corrosion observed under oxic conditions in the real-time corrosion monitoring experiments. This QA review has found that the quality of the most recent experimental research work is of a sufficiently high standard that SKB's judgments can be made reliably. Indeed, SKB's most recent reports on copper corrosion issues generally show greater attention to recording QA-related information and a more comprehensive approach to reporting data than in older reports and papers.

However, uncertainties remain about the possibility of copper corrosion under anoxic conditions (in the absence of aggressive species such as sulphide and chloride). SKB's experiments dedicated to establishing an understanding of the behaviour of copper under such conditions have been unable to pin-point the source of hydrogen generated in the experiments, despite the high standard of QA and quality control adopted.

Monitoring, control and analysis of experiments at the HRL are typically problematic, from the heater failures in the early atmospheric corrosion tests and the groundwater flow control problems in the aqueous corrosion tests, to the electrode and computer failures in the MiniCan project. In particular, the use of electrochemical techniques to measure real-time corrosion rates has been of limited success, and the mechanisms, rates and spatial distribution of oxygen consumption in the experiments are not well understood. Only by conducting experiments on an isolated and controlled copper and bentonite sample, has real time corrosion

monitoring been able to give reasonable indications of evolving copper corrosion rates under oxic conditions.

Also, it seems usual in experiments at the HRL for there to be a long period between a system failure and the detection and correction of the problem. System monitoring, controls and contingencies should be built into the design of the experiments with the aim of ensuring that valuable data are not lost. Not surprisingly, SKB has stated that it now aims to design more carefully controlled, dedicated corrosion experiments.

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APPENDIX 1

Coverage of SKB reports

Table A1-1: Main reports and papers checked in the QA review of copper corrosion experiments.

Reviewed report	Reviewed sections	Comments
TR-12-09: Analysis of SKB MiniCan Experiment 3.	Entire report	
P-12-01: Microbial analyses of groundwater and surfaces during the retrieval of experiment 3, A04, in MINICAN.	Entire report	
P-12-13: Miniature Canister (MiniCan). Corrosion Experiment Progress Report 4 for 2008-2011.	Entire report	
R-13-35: Metallographic Analysis of SKB MiniCan Experiment 3.	Entire report	
TR-13-17: LOT A2 Test Parcel. Compilation of Copper Data in the LOT A2 Test Parcel.	Entire report	
TR-09-31: Long Term Test of Buffer Material at the Äspö HRL, LOT Project. Final Report on the A0 Test Parcel.	Appendix B	
R-14-07: Corrosion of copper in ultrapure water.	Entire report	
Taxén (2004): Atmospheric Corrosion of Copper 450 Metres Underground. Results from Three Years Exposure in the Äspö HRL	Entire paper	A conference paper
Taxén (2009): Exposure of Copper in Äspö Groundwaters.	Entire report	A contractor report

QA reviews of SKB experiments

A copper corrosion experiment QA review meeting was held at SKB's offices in Stockholm on 29th August 2014 (SSM2011-2306-22, 2015). In order to facilitate the review process, prior to the meeting, SKB had been provided with a list of QA questions relating to the design, running, analysis and use of results of each copper corrosion experiment. The QA review questions are organised in terms of issues relating to the design, running, analysis and use of results of the experiments, as follows:

1. Procedures for experiment design and management

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

2. Procedures for quality control of materials and use of instrumentation

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

3. Procedures for running the experiments

1. What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?
2. What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?
3. What procedures are used for checking records of ongoing results?

4. What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?

4. Procedures for material analysis

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

5. Procedures for data management and control

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

6. Procedures for reporting the results of the experiments

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

SKB prepared responses to this list of QA questions for each copper corrosion experiment (SSM2011-2306-22, 2015). The following tables list the findings of the QA review in terms of the QA questions, based on SKB's responses to the QA review questions relating to the following copper corrosion experiments:

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

Table A2-1: MiniCan Experiment 3.

No.	QA question	Response
1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	In the SR 97 safety assessment, a scenario was considered in which a pinhole defect penetrated the electron-beam weld. This scenario raised the question of what would happen to the cast iron insert if groundwater penetrated such a hole. The change of welding method to friction stir welding has diminished the importance of the cast iron insert corrosion investigations.
1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	The plan for removal of Experiment 3 and post-test analysis was reviewed by the Reference Group for copper corrosion, including several independent researchers who had the chance to comment on the plan. An external expert was consulted before an agreement on the project plan was made. Documentation of the review process is available on request.
1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	The specific question relating to the consequences of iron corrosion due to minor leakage in the copper shell does not depend on the scale of the canister.
1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	In general, technical consultants are contracted on basis of experience from earlier work and, to increasing extent, through competitive procurement. In the specific case, Nick Smart's team at Amec Foster Wheeler (earlier Serco) was given the contract because they had performed similar and related work for SKB. When contracting a consultant, SKB requires QA certificates for the company and CVs for all personnel to be involved in the work.
1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	Generally, the objectives are initially described in the tender sent out to the potential consultants. The proposal written by the consultants is then compared with the original tender and discussed within SKB and with the consultants. When an agreement is reached SKB places an order to the consultant. Finally, each report that is published by SKB is reviewed with regard to QA as well as factual content.
2.1	What quality controls are there on the materials used and the installation of the experiment?	The miniature copper canister is made using copper of the same quality as that to be used in the KBS-3 repository. The composition of the copper (e.g. oxygen and phosphor content) is ensured by the manufacturer.
2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	All of the electrodes installed in Minican were calibrated before the start of the experiments. Despite this, soon after installation the electrodes in some of the experiments were found not to be working properly; large and random variations in the signals were seen. Several electrodes were therefore replaced and stable signals were obtained.
2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Uncertainties in experimental data are given in the published reports. See for example water composition data in P-12-13 and the weight loss analysis in TR-12-09.

No.	QA question	Response
2.4	What controls are there on material recovery for analysis at the end of the experiment?	When the first canister was retrieved it was taken out under water from the borehole and inserted into a transport flask. An inert-gas glovebox was constructed specially for handling the canisters from Minican. All specimens to be examined with metallographic methods were prepared inside the glovebox, which ensured that the risk of contamination or atmospheric oxidation of the surface was minimal.
3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	The water chemistry was recorded regularly, both within the steel cage holding the experiment canisters, as well as in the boreholes. Other parameters recorded included hydrostatic pressure, redox potentials and pH.
3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	Several types of electrochemical measurements were used (LPR, ACI, ECN, ohmic resistance) and data were recorded several times per year and stored in the SICADA database. A number of progress reports have been published.
3.3	What procedures are used for checking records of ongoing results?	Electrochemical on-line measurements were performed four times annually. Since the variations currently observed are small and, following retrieval of Experiment 3, it is known that some of the measurements are producing meaningless results, data will be recorded less often from 2015.
3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	When large variations in signals from the electrodes were encountered soon after installation and the start of the measurements, the reference electrodes were replaced. When large changes in, for example, corrosion rates were observed at a later stage of the experiments, no action was taken. It is likely that the reasons for such behaviour will become apparent when the canisters are retrieved and analysed (as was the case for Experiment 3).
4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	The electrodes used in MiniCan were calibrated in the laboratory before installation. This procedure is described in the first report published for MiniCan.
4.2	What procedures are there for recording the results of the analysis, including uncertainties?	All measurement data are recorded and stored in the SICADA database.
4.3	What procedures are used to identify, evaluate and report outliers?	Outlying data are included in the tables/diagrams of the progress reports together with a note or explanation. In one of the early MiniCan progress reports (P-11-40) data from one electrode were omitted, see further discussion in SSM 2010:17. Since then, these data have been published in several reports.
4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	Measurement uncertainties are given in the reports. If a measured parameter gives values that are below detection limit for the instrument or method used this is stated (for example the oxygen pressure in the ground water has been reported to be below the detection limit in several MiniCan reports).
4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	There are no particular procedures. The range of possible corrosion mechanisms considered is based on the composition of the ground water and thermodynamic data (Pourbaix diagrams).

No.	QA question	Response
4.6	What procedures are used for checking the results of the analysis?	There are no procedures for checking the raw data, but SKB checks all reports. Reports are factually reviewed in a documented process before publication.
4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Procedures of data selection for the SR-Site safety assessment are described and used in the Data report for SR-Site (TR-10-52). No data from MiniCan were used in the modelling in SR-Site.
5.1	How are data from the experiments stored, backed-up, accessed and controlled?	SKB has a database (SICADA) for storing raw data as well as certain processed measurement data.
5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	Ensuring correct use of data is mainly handled by factual review of the reports.
6.1	What procedures are used for reporting the experiments, analysis and results?	Raw data are stored in the SICADA database. Several reports have been published by SKB. All of these reports have gone through factual and quality review. Two peer reviewed papers on MiniCan have been published in scientific journals.
6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037. Within the Research and Safety assessment group a checklist has been developed for the implementation of this routine: SKBdoc 1394728 (an internal SKB document).
6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	MiniCan is a well-documented project. Several reports and peer reviewed papers have been published. The first report (TR-09-20) describes measurement details, experimental setup and materials in detail.
6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	The references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database. Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
6.6	How is it ensured that experimental results are not omitted from being reported?	SKB requires that all results are reported.

Table A2-2: LOT A2 parcel test.

No.	QA question	Response
1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	The LOT experiment was designed primarily for investigating the long-term testing of buffer materials. In the LOT A2 test, the main aspects were to check that the repository temperature and geochemical conditions after water saturation do not significantly change the physical properties of the buffer (TR-09-29, Section 2.1). Regarding copper corrosion, the specific issues of interest were “Check of calculated data concerning copper corrosion, and collect information regarding the character of possible corrosion products” (TR-09-29, Section 2.1).
1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	See SSM 2010:17, Question 3.1 in Appendix A.1.
1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	The scale of the experiment, with copper tubes and bentonite rings smaller than planned for KBS-3, was intended to shorten the saturation period, to get a higher temperature gradient and to facilitate recovery of the package. The size of the buffer rings was not considered important for the corrosion investigations.
1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	In general, technical consultants are contracted on basis of experience from earlier work. When contracting a consultant, SKB requires QA certificates for the company and CVs for all personnel involved in the work.
1.5	What QA procedures are in place for management of contractors’ work and ensuring that the objectives of the experiment and analysis are met?	See SSM 2010:17, Question 3.1 in Appendix A.1.
2.1	What quality controls are there on the materials used and the installation of the experiment?	The materials in the tube are specified in TR-13-17 (Section 2.2). Further details of the installation, etc. are given in TR-09-29.
2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	See SSM 2010:17, Question 1.3 in Appendix A.1
2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
2.4	What controls are there on material recovery for analysis at the end of the experiment?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.

No.	QA question	Response
3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	Described in the report from the LOT project (TR-09-29).
3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	No on-line measurements of corrosion.
3.3	What procedures are used for checking records of ongoing results?	No on-line measurements of corrosion.
3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	No on-line measurements of corrosion.
4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
4.2	What procedures are there for recording the results of the analysis, including uncertainties?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
4.3	What procedures are used to identify, evaluate and report outliers?	SKB is not aware of any particular procedures.
4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	Some details are given in the report TR-09-29, in the appendices with the reports from the different contractors.
4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	There are no particular procedures. The range of possible corrosion mechanisms considered is based on the general knowledge of the composition of the pore water and on thermodynamic data (Pourbaix diagrams).
4.6	What procedures are used for checking the results of the analysis?	There are no procedures for checking the raw data, but SKB checks any reports. The reports are factually reviewed in a documented process before publication. The data for copper were checked by later considerations (TR-13-17) of mass balances.
4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). No data from LOT were used in the modelling. Observations regarding the lack of localised corrosion have been used in supporting documents.
5.1	How are data from the experiments stored, backed-up, accessed and controlled?	See SSM 2010:17, Question 3.1 in Appendix A.1.

No.	QA question	Response
5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	This is mainly handled by the factual review of the report.
6.1	What procedures are used for reporting the experiments, analysis and results?	See SSM 2010:17, Question 4.2 in Appendix A.1.
6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037. Within the Research and Safety assessment group, a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document).
6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	See SSM 2010:17, Question 1.3 in Appendix A.1.
6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	The references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database. Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
6.6	How is it ensured that experimental results are not omitted from being reported?	SKB requires that all results are reported.

Table A2-3: LOT A0 parcel test.

No.	QA question	Response
1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	The LOT experiment was designed primarily for investigating the long-term testing of buffer materials. In the LOT A0 test, the main aspects were to check that compaction, placement and water saturation did not significantly change the physical properties of the buffer (TR-09-31, Section 2.1). Regarding copper corrosion, the specific issues of interest were to "Check calculated data concerning copper corrosion, and collect information regarding the character of possible corrosion products" (TR-09-31, Section 2.1).
1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	See SSM 2010:17, Question 3.1 in Appendix A.1.
1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	The coupons were embedded in the LOT A0 test parcel and the final investigation could only be done when the whole package was extracted. The size of the buffer rings was not considered important for the corrosion investigations.
1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	In general, technical consultants are contracted on basis of experience from earlier work. When contracting a consultant, SKB requires QA certificates for the company and CVs for all personnel involved in the work. See also SSM 2010:17, Question 3.1 in Appendix A.1.
1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	See SSM 2010:17, Question 3.1 in Appendix A.1.
2.1	What quality controls are there on the materials used and the installation of the experiment?	The copper coupons were manufactured from plate material of canister quality (stated in TR-09-31, Section B3).
2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	The techniques and equipment are commented on in SSM 2010:17, Questions 2.2 and 3.3 in Appendix A.1.
2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Some details of the measurement procedures are given in report TR-09-31.
2.4	What controls are there on material recovery for analysis at the end of the experiment?	Some details of the measurement procedures are given in report TR-09-31.

No.	QA question	Response
3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	Described in the report from the LOT project (TR-09-31). Some notes on the temperatures are given in the corrosion section (Appendix B).
3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	No on-line measurements of corrosion.
3.3	What procedures are used for checking records of ongoing results?	No on-line measurements of corrosion.
3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	No on-line measurements of corrosion.
4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	See SSM 2010:17, Questions 1.3 and 2.2 in Appendix A.1.
4.2	What procedures are there for recording the results of the analysis, including uncertainties?	SKB is not aware of any particular procedures.
4.3	What procedures are used to identify, evaluate and report outliers?	SKB is not aware of any particular procedures.
4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	SKB is not aware of any documentation of these issues.
4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	There are no particular procedures. The range of possible corrosion mechanisms considered is based on the general knowledge of the composition of the pore water and on thermodynamic data (Pourbaix diagrams).
4.6	What procedures are used for checking the results of the analysis?	There are no procedures for checking the raw data, but SKB checks any reports. Reports are factually reviewed in a documented process before publication.
4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). No data from LOT were used in the modelling. Observations regarding the lack of localised corrosion have been used in supporting documents.
5.1	How are data from the experiments stored, backed-up, accessed and controlled?	See SSM 2010:17, Question 3.1 in Appendix A.1.

No.	QA question	Response
5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	This is mainly handled by factual review of the report.
6.1	What procedures are used for reporting the experiments, analysis and results?	See SSM 2010:17, Question 4.2 in Appendix A.1.
6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037. Within the Research and Safety assessment group, a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document).
6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	No specific procedures were set up.
6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	The references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database. Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
6.6	How is it ensured that experimental results are not omitted from being reported?	SKB requires that all results are reported.

Table A2-4: Anoxic corrosion test using pure water.

No.	QA question	Response
1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	Following the publication of alleged evidence of copper corrosion in anoxic water by Hultquist and co-workers, SKB needed to repeat the experiments. The experiments are expected to increase the knowledge base of the behaviour of copper in water.
1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	A pre-study was carried out prior to the decision to carry out the experiment. Much of the planning and design was done in the pre-study. The experiment plan was included in the project tender to SKB, where the outcome of the pre-study is referenced. There is no explicit, overall QA plan for the experiment.
1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	The location was determined by the contractor's lab. The scale was intended to be close to the experiment by Gunnar Hultquist. The envisaged time scale was initially about 1.5 years, but has been extended. All the above is specified in the contract between Uppsala university and SKB.
1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	The chemistry group at the Ångström laboratory, Uppsala university was selected based on its members' competence and broad network of contacts in various, required areas at the university. The Uppsala group used its expertise and network of contacts at the university to select experts for the various types of analyses involved in the experiments.
1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	SKB has been following the work by the Uppsala group, as has the Reference Group that was set up for this experiment (and others). SKB has organised regular meetings with SKB staff, and telephone and e-mail conversations, peer review of reports, and interactions with the Reference Group.
2.1	What quality controls are there on the materials used and the installation of the experiment?	The controls of materials are documented in R-13-31 (in Swedish) and R-14-07 (English translation of R-13-31).
2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	To some extent described in R-13-31 and R-14-07 (English translation of R-13-31). Additional data are stored at and available from the Uppsala group.
2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Described in R-13-31/R-14-07. The ERDA results cited in the report have been re-evaluated after publication of the report and deemed to be less useful than expected. (To be reported.)
2.4	What controls are there on material recovery for analysis at the end of the experiment?	Described in R-13-31/R-14-07. Most of the experiment was performed in a glovebox with a controlled N_2 atmosphere. Transfer of copper samples after exposure to anoxic water to analysis instruments outside the glovebox was done in an inert atmosphere.

No.	QA question	Response
3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	Described in R-13-31/R-14-07.
3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	Described in R-13-31/R-14-07. Temperature and pressures were recorded.
3.3	What procedures are used for checking records of ongoing results?	Described in R-13-31/R-14-07 (ongoing measurements continuously monitored, logged and backed-up).
3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	To some extent described in R-13-31/R-14-07.
4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	To some extent described in R-13-31/R-14-07.
4.2	What procedures are there for recording the results of the analysis, including uncertainties?	Such procedures are, for the analyses of the copper samples after exposure to water, to some extent described in the R-13-31. Additional data are stored by and available from the Uppsala group.
4.3	What procedures are used to identify, evaluate and report outliers?	No particular procedures. All results are reported.
4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	Described in R-13-31/R-14-07.
4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	No particular procedures. Not really applicable since the experiment aims at checking whether an alleged corrosion mechanism exists in the first place. No evidence of corrosion has, so far, been found in the experiments.
4.6	What procedures are used for checking the results of the analysis?	The results from the Uppsala group were closely followed by both SKB and the Reference Group. Experiments to check interpretation and conclusions from the Uppsala group have been performed at Micans in Gothenburg. The published reports are factually reviewed in a documented process before publication.
4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Not applicable, since no evidence of corrosion was found.
5.1	How are data from the experiments stored, backed-up, accessed and controlled?	A double back-up system is used with UPS. Also there are continuous deliveries of raw data of pressure and temperature measurements to SKB.

No.	QA question	Response
5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	Described in R-13-31/R-14-07. Extreme measures are taken to check the Cu material investigated and to control the environment in which the measurements are done.
6.1	What procedures are used for reporting the experiments, analysis and results?	Reporting is required in the contract with SKB. Progress was reported to and discussed by a dedicated Reference Group several times a year from the start of the project until the autumn of 2013. There were more frequent reports to and discussions with SKB. Work carried out up to Spring 2013 is published in R-13-31/R-14-07, and additional reports will be published. Ultimately, the aim is to report the results in a peer reviewed journal.
6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037. Within the Research and Safety assessment section at SKB, a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document).
6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	No particular procedures, but this is a general aim to do so when describing scientific experiments in academia.
6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	Generally, the references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database. These specific experiments were performed after submitting the licence application.
6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	The progress of this particular issue has been directly reported in a number of status reports to SSM, in addition to the reporting in R-13-31/R-14-07. Studies with the alternative method developed at Micans is an important complement to the studies at Uppsala.
6.6	How is it ensured that experimental results are not omitted from being reported?	SKB requires that all results are reported. The Reference Group was one attempt to give extra insight into the experiment.

Table A2-5: Atmospheric and aqueous corrosion experiments.

No.	QA question	Response
1.1	How was the requirement for the experiment identified and how does it support the repository development programme?	The experiments are expected to increase the knowledge base of the behaviour of copper in repository-like environments.
1.2	Is there a QA plan for the experiment? Does the QA plan cover planning, design, running, analysis and reporting of the experiment?	SKB is not aware of any particular plan.
1.3	What constraints or requirements are there on the location, scale and schedule for the experiment?	The idea was to use the atmosphere at Äspö and the ground water coming naturally from the rock wall in the Äspö tunnel respectively, which thus set the environmental conditions.
1.4	How are organisation(s)/expert teams selected to undertake the experiments and analyse the results? How is it ensured that appropriate QA/Quality Control (QC) procedures are followed by the contractors and that the necessary expertise is available for the work?	In general, technical consultants are contracted on basis of experience from earlier work. In this specific case, Claes Taxén was given the contract as he had been involved in earlier corrosion studies for SKB.
1.5	What QA procedures are in place for management of contractors' work and ensuring that the objectives of the experiment and analysis are met?	SKB is not aware of any particular procedures.
2.1	What quality controls are there on the materials used and the installation of the experiment?	It is stated in the publication (Taxén 2004) that the copper material was delivered by SKB and was left over after manufacturing a full-scale canister.
2.2	What procedures are used for instrumentation calibration and reliability testing for the experiment, instrumentation checking and maintenance during the experiment, instrumentation backup/duplication, and instrumentation checking at the end of the experiment?	SKB is not aware of any particular procedures.
2.3	How are measurement uncertainties and instrument detection limits reported and accounted for?	Not given in the report.
2.4	What controls are there on material recovery for analysis at the end of the experiment?	Some details are given in the report, but SKB is not aware of any particular procedures.
3.1	What procedures are used for ensuring that the conditions of the experiment (e.g. chemical and hydraulic) are controlled as planned and monitored and recorded during the experiment? How are uncertainties in conditions identified and recorded?	Atmospheric experiment: the temperature was measured periodically, and documented in the report for the first year. The set-up was provided with a dark cover to avoid the influence of light. As stated in (Taxén 2004) the heater didn't work for periods of weeks. Aqueous experiment: groundwater was fed off from fractures in the rock into a pressure vessel.
3.2	What procedures are there for recording any ongoing corrosion results and the conditions of the experiment at the time of measurements?	No on-line measurements of corrosion.
3.3	What procedures are used for checking records of ongoing results?	No on-line measurements of corrosion.

No.	QA question	Response
3.4	What procedures are used if the on-going corrosion tests show unreliable measurements or if the test conditions are changing unexpectedly and are not representative for the designed aim of the experiments?	Atmospheric experiment: problems with the heater were recognised. All ten samples from the heated experiment were analysed as some had fallen down. Aqueous experiment: the experiment was stopped as the ground water flow decreased too much and ingress of oxygen could not be prevented.
4.1	What procedures are used for calibration and testing of instrumentation used in the material analysis?	SKB is not aware of any particular procedures.
4.2	What procedures are there for recording the results of the analysis, including uncertainties?	SKB is not aware of any particular procedures.
4.3	What procedures are used to identify, evaluate and report outliers?	SKB is not aware of any particular procedures.
4.4	How are measurement uncertainties and instrument detection limits recorded and taken into account?	SKB is not aware of such issues having been documented.
4.5	Are there procedures for checking that the range of possible corrosion mechanisms has been considered when interpreting the results of the experiments and analysis?	There are no particular procedures. The range of possible corrosion mechanisms considered is based on the composition of the ground water and thermodynamic data (Pourbaix diagrams).
4.6	What procedures are used for checking the results of the analysis?	There are no particular procedures. After part of the experiment failed, less effort was put into evaluation of the results.
4.7	What procedures are used for selection of the data that are implemented further for modelling studies of long-term corrosion behaviour of copper canisters? Which experimental data are used for validation of modelling results?	Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The measured corrosion rates in the atmospheric experiment were used in the evaluation of the atmospheric corrosion during initial storage (before deposition).
5.1	How are data from the experiments stored, backed-up, accessed and controlled?	The results from the experiments were reported in a conference paper (atmospheric experiment) and as an internal report to SKB (the aqueous experiment).
5.2	What procedures are used for ensuring that the data are used appropriately and uncertainties taken into account (i.e. ensuring that the experimental conditions under which the data were acquired are recorded and understood)?	This is mainly handled by the factual review of the report.
6.1	What procedures are used for reporting the experiments, analysis and results?	There are no particular procedures. The results from the experiments were reported in a conference paper (atmospheric experiment) and as an internal report to SKB (the aqueous experiment).
6.2	What procedures are used for review and checking of reports?	SKB has an established routine for review, SD-037. Within the Research and Safety assessment section at SKB, a checklist has been developed for the implementation of this routine, SKBdoc 1394728 (internal SKB document).
6.3	Are there procedures to ensure that the documentation provides enough detail for the experiment to be repeated?	SKB is not aware of any particular procedures.

No.	QA question	Response
6.4	Are there procedures for ensuring that results presented in the licence application can be traced back to particular experiments, and sets of data?	The references used in the safety assessment report and its main references, as well as in further licence applications documents, are recorded in a database. Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
6.5	How is it ensured that the reported results are used appropriately and uncertainties are taken into account?	Data selection procedures for the SR-Site safety assessment are described in the Data report for SR-Site (TR-10-52). The Process report (TR-10-46) is intended to give the arguments for the handling of a specific process, including the references used.
6.6	How is it ensured that experimental results are not omitted from being reported?	SKB requires that all results are reported.

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The Swedish Radiation Safety Authority has a comprehensive responsibility to ensure that society is safe from the effects of radiation. The Authority works to achieve radiation safety in a number of areas: nuclear power, medical care as well as commercial products and services. The Authority also works to achieve protection from natural radiation and to increase the level of radiation safety internationally.

The Swedish Radiation Safety Authority works proactively and preventively to protect people and the environment from the harmful effects of radiation, now and in the future. The Authority issues regulations and supervises compliance, while also supporting research, providing training and information, and issuing advice. Often, activities involving radiation require licences issued by the Authority. The Swedish Radiation Safety Authority maintains emergency preparedness around the clock with the aim of limiting the aftermath of radiation accidents and the unintentional spreading of radioactive substances. The Authority participates in international co-operation in order to promote radiation safety and finances projects aiming to raise the level of radiation safety in certain Eastern European countries.

The Authority reports to the Ministry of the Environment and has around 300 employees with competencies in the fields of engineering, natural and behavioural sciences, law, economics and communications. We have received quality, environmental and working environment certification.

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