



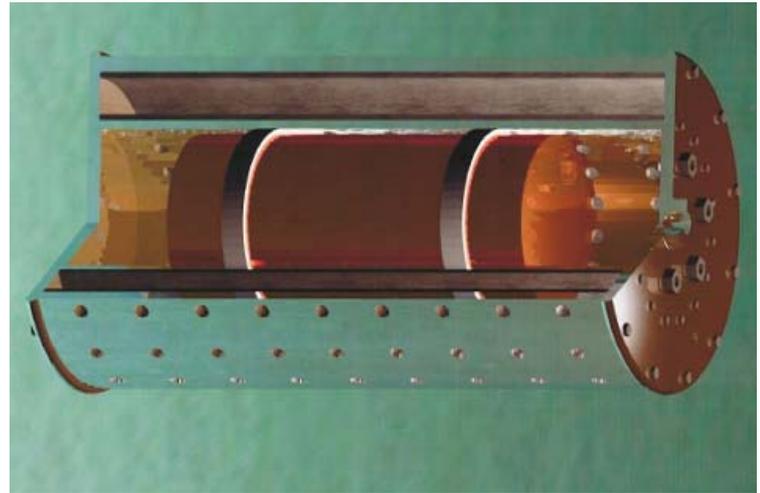
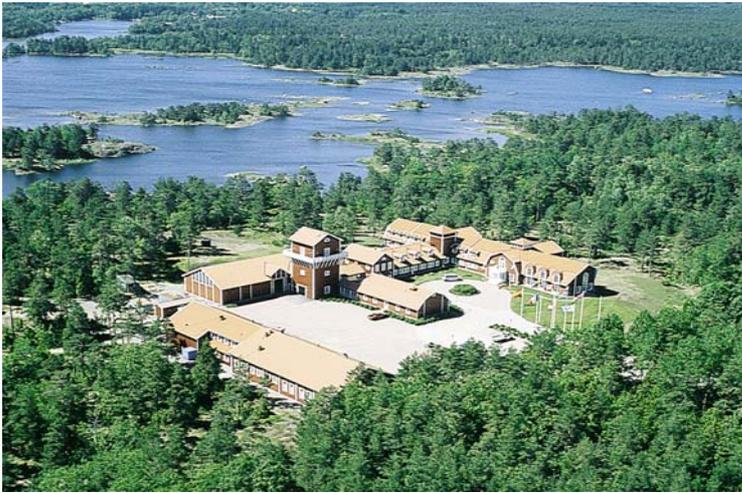
Internal information

Plan

Document ID 1275538	Version 1.0	Status Preliminary	Reg no	Page 1 (1)
Author Nick Smart			Date 2010-09-15	

## Plan for the Proposed Removal of MiniCan Experiment 3 from Borehole KA3386A04

# Plan for the Proposed Removal of MiniCan Experiment 3 from Borehole KA3386A04



**Prepared for** SKB  
**Prepared by** Serco  
**Your Reference** 3468  
**Our Reference** SERCO/TCS/004427.02/001 Issue 1  
**Classification** SERCO COMMERCIAL  
**14 September 2010**



**Title** Plan for the Proposed Removal of MiniCan Experiment 3 from Borehole KA3386A04

**Prepared for** SKB

**Your Reference** 3468

**Our Reference** SERCO/TCS/004427.02/001 Issue 1

**Confidentiality, copyright & reproduction** SERCO COMMERCIAL

This report is submitted by Serco Technical Consulting Services (hereafter referred to as Serco) in connection with a contract to supply goods and/or services and is submitted only on the basis of strict confidentiality. The contents must not be disclosed to third parties other than in accordance with the terms of the contract.

To minimise our impact on the environment, Serco uses paper from sustainable sources

**Contact Details**

Serco  
 F7 Culham Science Centre  
 Abingdon  
 Oxfordshire OX14 3DB  
 United Kingdom

T +44 (0) 1635 280385  
 F +44 (0) 1635 280389  
 E technical.services@serco.com

www.serco.com/tcs

	Name	Signature	Date
<b>Author(s)</b>	N.R. Smart, A.P. Rance, B. Reddy and R.J. Winsley		14.09.10
<b>Reviewed by</b>	P. Fennell		10.09.10
<b>Approved by</b>	N.R. Smart	 p.p.	14.09.10

## Contents

<b>1</b>	<b>Introduction and background to work</b>	<b>4</b>
<b>2</b>	<b>Removal of Experiment 3 for analysis</b>	<b>4</b>
2.1	Removal of assembly from the borehole	5
2.2	Transport	5
2.3	Opening and sectioning the model canister assembly	6
2.4	Summary of the proposed process	6
2.5	Analysis	7
2.6	Pre-work testing	11
2.7	Retention of Samples	11
<b>3</b>	<b>Reference</b>	<b>12</b>
<b>4</b>	<b>Tables and Figures</b>	<b>13</b>
<b>Appendix 1</b>	<b>Water analysis activity plan</b>	<b>22</b>

## 1 Introduction and background to work

To ensure the safe encapsulation of spent nuclear fuel rods for geological disposal, SKB of Sweden is considering using a system that consists of an outer copper canister and a cast iron insert. In 2007 Serco completed the set up of five model canister experiments at SKB's Äspö laboratory and monitoring has continued since.

The original aim of the model canister experiments was to examine how corrosion of the cast iron insert inside a copper canister would evolve with time, if leakage through a small defect in the copper canister were to occur. Serco has arranged manufacture and installation of five miniature copper canisters containing cast iron inserts, with 1 mm defects in the copper shell. We have also designed and procured electrochemical equipment to monitor the corrosion behaviour of the model canisters in situ. The detailed design is described in reference [1] and the set up and results obtained to date are summarised in references [2-4].

In addition to maintaining the existing experimental work programme, Serco Technical Consulting Services have prepared a detailed plan to remove one of the experiments (Experiment 3, borehole KA3386A04), as agreed at the last expert advisory group meeting in June 2009. Experiment 3 was originally installed at SKB's Äspö laboratory in December 2006. This document is the plan for removal and analysis of Experiment 3 and should be read in conjunction with the accompanying commercial proposal for carrying out the necessary activities [5].

This document presents the philosophy behind the proposed methods for removal and analysis of Experiment 3 and presents details of the corresponding equipment design and procedures. It sets out the analyses that will be conducted once the experiment has been removed from Äspö and transported back to Serco's Culham Laboratories within the UK. A previous draft of this document that was circulated to SKB was accepted in August 2010. It is currently anticipated that work will start on this removal programme in the October 2010; the proposed schedule is shown in Figure 1.

## 2 Removal of Experiment 3 for analysis

In view of the high corrosion rates currently being measured in some of the experiments it has been suggested that one of the 'MiniCan' experiments should be removed to investigate the extent of any corrosion, both on the coupons and on the model canisters itself. This will also give an opportunity to characterise any surface biological activity (e.g. the presence of biofilms, type of bacteria colonising the surfaces, etc.) and assessing the condition of the bentonite in the experiments. The experiment could then be replaced with a new MiniCan specimen, using a refurbished support cage. Some consideration would need to be given to the timing of replacement; for example it may be beneficial to wait until the analysis of the removed canister has been completed, so that the information gained can be used to identify improvements in the experimental design for subsequent tests. It is recommended that automated monitoring of the remaining four experiments should continue, using the current monitoring protocol.

At a planning meeting held in June 2009 it was agreed that Experiment 3 should be removed for investigation [6]. Following initial discussions, a plan for removal and analysis of the MiniCan Experiment 3 is presented below. The overriding aim of the proposed procedure is to use a method that will allow removal of the experiment while avoiding exposure to the atmosphere, in order to minimise changes in the composition of any air-sensitive corrosion products or other materials. Having removed the experiment from the borehole the support cage and the miniature model canister will be transported to the U.K. for analysis at Serco's Laboratories. The main stages of the proposed procedure are described below.

## 2.1 Removal of assembly from the borehole

The first stage of the procedure would be to build a transfer tank around the flange/entrance to the borehole. The transfer tank would be filled with anoxic water from the borehole itself. It is proposed that whilst the operations are being conducted the transfer tank water should be continuously purged with nitrogen, so as to ensure that dissolved oxygen levels are minimised (Such a procedure may well necessitate additional local monitoring of the air oxygen levels for safety reasons, if required breathing apparatus could be an option). The transfer tank would be designed and set up by Äspö staff, with design input contributed by Serco Staff. Preliminary discussions have already been held on this subject and an initial design has been proposed and prepared by SKB, which is shown in Figure 2 and Figure 3. Further design work on the transfer tank is ongoing and this will be completed early in the programme (see Figure 1). It is envisaged that the basic process of removing the assembly and transferring it into the tank, which is still being finalised by SKB and Serco staff, would involve a number of steps which are listed in bullet point form later in this document.

In order to further minimise contact with air, the borehole would also be purged with nitrogen while the assembly was being removed from the borehole. It would probably be necessary to move some of the equipment currently situated near the boreholes to provide sufficient working room (e.g. the emergency evacuation vehicle store). It will be necessary to ensure that sufficient lifting equipment is in place to lift the experiment into a transfer flask, although the existing lifting beams that were used to install the experiments originally may be sufficient.

## 2.2 Transport

It is planned that once the assembly has been removed from the borehole, and it is underwater in the transfer tank, it will be transferred into a sealed transfer flask for shipping back to the UK. All of these removal and packaging steps will take place underwater in the transfer tank, to minimise exposure to oxygen.

The shipping transfer flask will be designed and fabricated by Serco. Its purpose is to ensure the safe and oxygen free transport of MiniCan Experiment 3 from Sweden to Serco's Laboratories in Culham (UK). Initial designs for the transport flask have been considered and a current working design is shown in Figure 4; this is currently be converted into engineering drawings that can be provided to the manufacturing workshop, which is based at the Culham Science Centre. Once manufactured the transport flask would be shipped to Sweden in advance of the removal of MiniCan Experiment 3. A pressure test will be carried out on the flask before use to ensure that it is leak tight.

In brief the current working design is that the transfer flask will be slightly larger in diameter than the support cage / model canister assembly. After removal of the support cage assembly from the borehole (into the borehole transfer tank designed by SKB) the assembly would be loaded into the transfer flask containing groundwater and purged with nitrogen. An oxygen getter material would be placed in the transfer chamber (e.g. iron filings or steel wire) to minimise any remaining residual oxygen. The transfer flask would then be sealed with a bolted flanged lid and be transported to the U.K., using a specialist transport company. It would be necessary to ensure that there are no customs issues or restrictions that would impede or prevent transport of the experiment to the U.K. It is proposed that SKB help facilitate this process.

It is also proposed that the temperature of Experiment 3 is monitored during transport using a small compact temperature logger that could travel with the transfer flask.

A schematic diagram illustrating the proposed loading of Experiment 3 into the transfer flask underwater within the transfer tank is shown in Figure 5.

## 2.3 Opening and sectioning the model canister assembly

Once in the U.K. at Serco's Culham Laboratories the transfer flask would be placed in a specially constructed inert gas-purged glovebox, which would include a lifting device to enable the model canister to be withdrawn from the transfer flask. A cutting system, consisting of a rotary saw, would also be mounted inside the glovebox, so that the model canister assembly could be sectioned in a controlled manner (where possible this will be conducted without the use of lubricating fluids that would contaminate the surface). Detailed discussions have been held with numerous suppliers. The result of these discussions is that such a glovebox, lifting and cutting systems are available on a commercial basis. A design drawing for the two part glove box required for the proposed work programme is shown in Figure 6. Once procured, this equipment would be available to SKB for dismantling other experiments in the future.

In brief, it is proposed that the glove box designed and constructed for this work programme would be largely as follows;

- Special Glovebox 3000 mm width, 1200 mm depth, 2100 mm high
- Door on left hand side
- 3 sets of standard gloveports
- Special hoist for lifting and moving canisters
- Gloveports on both sides of saw section
- Internal connections for saw
- Removable plate for feedthroughs
- Interlocked door will only open when oxygen level is higher than 18%
- Dust cleaner for cleaning of glovebox and removal of particles/dust
- Gas purification system for closed cycle circulation of glovebox for removal of oxygen and moisture, <1 ppm
- Unit for removal of solvent contaminations from the glovebox atmosphere
- Electrochemical Oxygen analyzer. Range: 1 to 1000 ppm and 0 to 25%

## 2.4 Summary of the proposed process

In summary the main steps concerning removal of MiniCan Experiment 3 and its transfer to Serco's Culham Laboratories are as follows;

- Bolt transfer tank into place;
- Part fill and deaerated transfer tank;
- Remove flange from borehole and allow tank to fully fill;
- Disconnect cables and tubing from flange (seal up tubing to prevent oxygen ingress);
- Continuously purge tank and borehole water with nitrogen;
- Begin to remove assembly using pull rod already secured to it (if angles aren't conducive, the pull rod can be cut off in sections);
- When assembly reaches the mouth of the borehole secure strop to take the weight, then fully remove assembly;
- Lower assembly deeper into the tank using strop;
- Push assembly into pre-positioned transfer flask already submerged in tank water;
- Remove pull rod from assembly and bolt on lid of transfer flask, sealing flask. (Purge flask with nitrogen if required);
- Package and prepare transport flask for shipment to the UK;
- Transfer transport flask into purpose built glove-box at Culham laboratories in UK;
- Open transport flask and when ready remove Experiment 3.

## 2.5 Analysis

It is suggested that a further campaign of water analysis should be carried out on all five experiments before removal of Experiment 3. A detailed activity plan for the next phase of water analysis has been prepared by Ann-Sofie Karlsson, SKB, and this is included in Appendix 1. The proposed water analysis activity plan covers all of the species already analysed to date, as described in references [2-4]. In addition to this it is also proposed that microbial characterisation and dissolved gas analysis should be conducted on Experiment 3 by Microbial Analytics.

The surface of the support cage will be swabbed for microbial analysis immediately after removal from the borehole and further samples for microbial analysis will be taken once the experiment is opened up in the U.K..

From initial discussions, Microbial Analytics are of the opinion that the microbial analysis conducted when Experiment 3 is removed from the borehole and cut up for analysis in Culham, should focus on the presence of biofilms and particularly the presence of sulfate reducing bacteria on the metal surfaces.

Biofilms are not expected on the full size canisters in the repository due to the restrictions imposed from the bentonite, but it is thought possible that biofilms are present on the model canister metal surfaces, and also potentially inside the model canisters. Sulfide corrosion may then have also occurred in Experiment 3. It is suggested that biofilm samples are taken from the metal surfaces in an attempt to quantify the amount of sulfate-reducing bacteria per surface area. By quantifying the sulfate-reducing bacteria, one can relate sulfide corrosion to their activity. It is thought important to be able to conclusively show that metal corrosion by sulfide was caused by microbial activity that will be absent with fully compacted buffer present (Microbial Analytics are currently preparing a paper demonstrating this fact). The water samples that will be taken before Experiment 3 is removed, will account for sulfide production by unattached sulfate reducing bacteria. It is proposed that molecular DNA methods should be utilised for the quantification of sulfate reducers in the biofilms (this is similar to the techniques used previously in past water sampling campaigns conducted on the MiniCan experiments).

After transferring Experiment 3 to the U.K., a number of analyses will be carried out on the metal components and the bentonite in order to extract as much information as possible from them. Where possible all dismantling and preparation of the samples will take place under an inert gas atmosphere, in order to prevent alteration of the samples before analysis. Samples may be transferred from the purpose-built dismantling glovebox to an existing glovebox at Culham using small transfer flasks or glove bags, for further sample preparation. It is suggested that the analytical steps on the samples would include the following, working from the outside of the support cage to the inside:

### 2.5.1 Stainless steel support cage

- Overall visual examination, photographic record.
- Cut samples for sectioning and optical microscopic examination, to determine the extent of any pitting or possible microbial corrosion attack. If areas of interest are observed further detailed metallographic examinations are an option, using techniques such as SEM/EDX.

### 2.5.2 Bentonite in annulus of support cage

The support cage around the model canister would be dismantled (by removing the top lid) and photographed. If cutting of the cylindrical filters is required to establish the extent of any corrosion it would probably be necessary to remove the support cage from the glovebox (it is anticipated that the stainless steel would not be adversely affected by exposure to oxygen). Where possible, the homogeneity of the bentonite in the annulus will be examined (e.g. to determine whether 'channels' have formed in the bentonite during water sampling). Samples of the bentonite will be removed for analysis. This analysis will initially concentrate on visual observation to assess whether any

alteration appears to have taken place in the bentonite. If required, a range of analytical techniques could be applied to the bentonite, such as those used in the recent NF-PRO project [7]. Further details of such techniques are described below; and detailed in [8].

- Electron microprobe analysis (EMPA) to examine if, and how far iron has penetrated into the bentonite matrix from the interface between the bentonite and the metal;
- Analysing the bentonite using XRD to determine the effect of iron taken into the bentonite on the lattice parameter and to characterise any new minerals;
- Measuring the effect of absorbed iron on the cation exchange capacity of the bentonite;
- Exchangeable cation analysis;
- Fourier Transform Infra-red spectroscopy (FTIR) to examine the determine oxidation state of iron present in the bentonite after the corrosion experiments;
- TEM / EDX / EELS – to examine the mineralogical composition of grains of bentonite containing iron produced by corrosion (TEM), the local concentration of iron in the bentonite (EDX) and the oxidation state of the iron;
- Hydraulic conductivity;
- Swelling pressure;
- Total chemical composition;
- Application of the sequential chemical extraction technique (Chemometric Identification of Substrates and Element Distributions, CISED).

It is suggested that a decision on the exact analyses applied to the bentonite is postponed until the condition of the bentonite can be assessed visually. It may also be of interest to characterise microbial activity within the bentonite. Any analyses would be carried out following discussion with SKB.

### 2.5.3 Corrosion coupons

The main aim of the analyses of the corrosion coupons will be to measure weight loss, in order to derive a value of corrosion rate, for comparison with the electrochemically measured values. There is one weight loss specimen for copper and one for iron in each model canister experiment. These coupons will be used for characterising the surface chemistry using non-destructive techniques prior to making the weight loss measurement. After the weight loss measurements have been completed the samples will be sectioned and examined microscopically for signs of localised corrosion. The techniques used for characterising the coupons (both iron and copper) would be as follows:

**Visual examination and photographs** - In order to assess the extent of any corrosion of the corrosion coupons, macro scale visual examination will be carried out, using a Canon IXUX digital camera equipped with a range of optical lenses. Any areas of corrosion on the metal surface will be noted and photographed. More detailed micrographs will also be collected for each sample where visible corrosion is observed (from the above macro screening test).

**Weight gain** - Weight gain measurements will be recorded for each corrosion coupon removed from Experiment 3 using a calibrated A&D HM-202EC balance with an instrument resolution of  $\pm 2 \times 10^{-5}$  g.

**Identification of surface chemistry** - Raman spectroscopy will be conducted on each corrosion coupon removed from Experiment 3. Coupons for analysis will be sealed within inert gas-filled glass containers and Raman spectroscopy will be conducted to identify the composition of the corrosion product (oxidation state of the metal).

**SEM/EDX** - examination of corrosion coupons and their surface films will be conducted following Raman analysis (however this will require the samples to be exposed to the atmosphere). If the surface films are found to be too thin to measure using SEM/EDX, surface analysis techniques such as X-ray photoelectron spectroscopy could be used to characterise the surface specific composition of the films and to measure film thickness by depth profiling, for example using AFM, SIMS or XPS.

**Weight loss measurement** - weight loss measurements will be recorded for each corrosion coupon sample using a calibrated A&D HM-202EC balance with an instrument resolution of  $\pm 2 \times 10^{-5}$ g. To increase accuracy all specimens will be weighed in triplicate at each stage and a mean weight will be calculated. Prior to weight loss testing it will be necessary to remove any corrosion product or scale formed on the samples, using a chemical cleaning agent (as recommended by the ASTM standard G1-90). Weight loss testing will only be conducted once all the required visual and chemical data have been obtained from each individual sample.

The above mentioned cleaning procedures will be selected with the aim of removing any accumulated corrosion product, whilst minimising any dissolution of the base metal. Because of this it will probably be necessary to use a different cleaning solution on the steel and copper samples. In order to increase accuracy, the effects of the cleaning procedure will be determined and the weight loss will be corrected accordingly.

The initial total surface area of the specimen and the mass lost during the test will be determined. From these weight loss values it will then be possible to calculate the average corrosion rate using the following formula. It should be noted however that this equation assumes uniform corrosion; therefore caution is required when deriving corrosion rates in this manner.

- Corrosion Rate =  $(K \times W) / (A \times T \times D)$

where:

- $K$  = a constant,
- $T$  = time of exposure in hours,
- $A$  = area in  $\text{cm}^2$
- $W$  = mass loss in grams, and
- $D$  = density in  $\text{g/cm}^3$ .

**Attack morphology underneath corrosion product** - Following the removal of any corrosion product, it will then be possible to section through (if required) any areas of interest on either sample and use optical microscopy to determine the depth of general or localised corrosion. In addition to basic optical microscopy, we could make use of our in-house confocal microscopy equipment at our Rislely laboratories, to perform non-destructive measurements of pit depths without sectioning the specimens. It is proposed that this work should be conducted on both the copper and cast iron coupons following weight loss testing. The data will be used to calculate nominal pit growth rates.

#### 2.5.4 Corrosion coupons (electrode samples)

As well as the corrosion coupons mentioned above, Experiment 3 also contains two identical corrosion coupons (again one cast iron and one copper) that have connections welded to them. These electrode samples are the ones that have periodically been used for electrochemical monitoring experiments.

**Repeat electrochemical testing** - Initially it is proposed that after the transfer flask is opened (in the glovebox at Culham) we will repeat the electrochemical tests using a new Ag/AgCl reference electrode. In order to conduct repeat linear polarisation resistance (LPR) and AC impedance measurements on the copper and cast iron electrodes, it will be necessary to remove the lid of the transfer flask and the top end cap from the support cage (to gain access). Using this approach it should be possible to conduct these electrochemical tests in the borehole water that should still be filling the transfer flask and Experiment 3. If additional solution is needed (i.e. it has drained away) it can be added as required. From these additional electrochemical tests it would then be possible to calculate a corrosion rate and compare it to those measured previously for Experiment 3 (i.e. whilst it was still located within the borehole).

The protocol for analysis of the coupons would be similar to that for the weight loss coupons but it would not be possible to measure the weight changes on the electrodes because wires are already attached to the coupons. Analysis of the composition of the films on the surface of the coupons will aid interpretation of the electrochemical data. For example, if it can be shown that the corrosion product films on the surface of the coupons are composed of metal sulphides, it may be possible to construct equivalent circuit models based on the assumed structure of the films that will enable a better interpretation of the AC impedance data.

The analytical techniques detailed above for the standard corrosion coupons could well be conducted on these electrode corrosion coupons (with the exception of weight loss testing) or vice versa, if the need arose.

### 2.5.5 Copper-cast iron-copper sandwich specimens

The dimensions of the specimens will be checked using Vernier callipers and then carefully dismantled in an inert atmosphere to examine the extent of corrosion and the thickness of any corrosion products. Samples of corrosion product will be analysed using Raman spectroscopy and both the copper and cast iron specimens will be sectioned and examined using optical microscopy to investigate the extent of any localised corrosion. It is proposed that we could make use of our in-house confocal microscopy equipment at our Risley Laboratories, to perform non-destructive measurements of pit/crevice depths without sectioning these specimens.

### 2.5.6 Miniature copper canister

One of the main objectives of the programme is to investigate the evolution of corrosion around the annulus between the cast iron insert and the outer copper canister. After a thorough visual examination of the intact canister, including accurate measurement of the outer dimensions to check for any expansion or contraction, the outer copper canister will be carefully cut using a mechanical saw mounted in the custom-built glovebox. The canister will be cut into slices and the copper will then be peeled away from the cast iron to reveal the distribution of any corrosion product found around the annulus in relation to the position of the defect in the copper container. The distribution of corrosion product in the annulus will be photographed and documented. The questions raised at the outset of the project will be addressed (see Section 1), namely:

- Does water penetrate into the annulus through a small defect?
- How does corrosion product spread around the annulus from the leak point?
- Does the formation of corrosion product in a constricted annulus cause any expansive damage to the copper canister?
- What is the effect of water penetration on the insert lid seal?
- Is there any detectable corrosion at the copper welds?
- Are there any deleterious galvanic interactions between copper and cast iron?

- Does corrosion lead to failure of the lid on the iron insert?

**Identification of surface chemistry** - Raman spectroscopy will be conducted on samples of both the copper and cast iron sectioned from the canister within Experiment 3. These samples sectioned for analysis will be sealed within inert gas-filled glass containers and Raman spectroscopy will be conducted on them to identify the composition of the corrosion product (oxidation state of the metal). Following Raman analysis further information could be gained on the composition and appearance of any surface films present using an SEM linked to an EDX detector.

**Investigating the possibility of preferential weld corrosion** - It is proposed that sections from the electron beam welds in the copper canister will be prepared so that they can be investigated to determine whether there are any indications of localised corrosion (e.g. pitting, cracking, intergranular corrosion etc.). Surface profilometry measurements will be used to characterise the roughness of the copper surface to determine whether any corrosion of the copper surface has taken place.

**Possibility of crevice corrosion around the gasket material** - The lid region of the cast iron inset will be carefully dismantled and examined for any signs of crevice corrosion around the gasket material. This will be conducted using a thorough visual examination and the collection of macro scale digital photographs, using a Canon IXUX digital camera equipped with a range of optical lenses. Any areas of interest could be sectioned out and studied in further detail.

### 2.5.7 Stress corrosion test specimens

The copper SCC Wedge Open Load specimens (WOL) present in the borehole of Experiment 3 will be examined to measure the length of the cracks, to determine whether exposure to the borehole water had led to any increase in crack length. Similarly, the U-bend specimens will be examined for indications of SCC initiation.

## 2.6 Pre-work testing

Due to the unique nature of this work programme a number of testing tasks are built into the working project plan in order to validate the techniques before the examination of Experiment 3 commences, specifically as follows:

- **Copper canister and cast iron insert** – We propose that we make up a trial test piece of similar thickness to the canister wall. This will allow us to test the cutting procedure in air, prior to cutting one for real under anoxic conditions within the glove-box.
- Repeat trials of cutting procedure inside the glovebox.

## 2.7 Retention of Samples

All of the stainless steel, cast iron and copper samples used or created through sectioning components of Experiment 3 would be uniquely and durably marked. All of the metal samples will be retained in a desiccator for a pre-determined period (agreed with SKB), following the completion of the proposed work programme. The use of a desiccator will help to preserve the specimens in a clean, dry, contamination-free environment. Those samples that require anoxic storage conditions could be stored within our glove box.

### 3 Reference

- 1 N.R. Smart, P.A.H. Fennell and G. Knowles, *Design of Model Canister Experiments*, SA/EIG/11080/C001, 2005.
- 2 N.R. Smart and A.P. Rance, *Miniature Canister Corrosion Experiment – Results of Operations to May 2008*, SERCO/TAS/E.001607.01, Issue 1, and SKB report TR-09-20, 2009.
- 3 N.R. Smart, A.P. Rance and B. Reddy, *Miniature Canister (MiniCan) Corrosion Experiment Progress Report 1 for 2008-9*, SERCO/TAS/E.003110.01, Issue 1, 2009.
- 4 N.R. Smart, A.P. Rance and B. Reddy, *Miniature Canister (MiniCan) Corrosion Experiment Progress Report 2 for 2008-9*, SERCO/TAS/E.003110.02, Issue 1, 2010.
- 5 *Plan for the Proposed Removal of MiniCan Experiment 3 from Borehole KA3386A04*, SERCO/TCS/004427.02/001 Issue 1, September 2010.
- 6 N.R. Smart, *Minutes of Model Canister Planning Meeting*, 2 June 2009, SKB offices, Stockholm.
- 7 N.R. Smart, F. Bate, L. Carlson, M.R. Cave, K. Green, T.G. Heath, A.R. Hoch, F. M. I. Hunter, O. Karnland, S.J. Kemp, A.E. Milodowski, S. Olsson, A. M. Pritchard, A. P. Rance, R.A Shaw, H. Taylor, B Vickers, L. O. Werme and C.L. Williams, *Interactions Between Iron Corrosion Products And Bentonite*, Serco/TAS/MCRL/19801/C001 Issue 2, 2008.
- 8 A.E. Milodowski, S.J. Kemp, C.J.B. Gowing, M.R. Cave, H. Taylor, K. Green and C. Richardson, *Mineralogical Investigations of the Interaction Between Iron Corrosion Products and Bentonite From the NF-Pro Experiments (Phase 3)*, British Geological Survey Commissioned Report CR/09/148, 2009.

## 4 Tables and Figures

**Table 1. Details of techniques for the analysis of the bentonite, corrosion coupons, electrode samples and sectioned samples from Experiment 3.**

Samples	Analytical Technique	Purpose
<b>Stainless steel support cage</b>	Visual observation	-
	Optical observation	-
<b>Corrosion coupons (Copper and cast iron)</b>	SEM	Morphology of corrosion product
	Raman spectroscopy	Identification of corrosion product
	X-ray diffraction	Identification of corrosion product
	Weight loss	Measuring corrosion rate
<b>Corrosion coupons (electrodes) (Copper and cast iron)</b>	Electrochemical analysis (LPR, ACI)	Measuring corrosion rates
	SEM	Morphology of corrosion product
	Raman spectroscopy	Identification of corrosion product
	X-ray diffraction	Identification of corrosion product
<b>Copper-cast iron-copper sandwich specimens</b>	Raman spectroscopy	Identification of corrosion product
	SEM	Morphology of corrosion product
	X-ray diffraction	Identification of corrosion product
	Optical	Presence of localised corrosion/pitting
<b>Copper canister and cast iron insert</b>	Raman spectroscopy	Identification of corrosion product
	SEM	Morphology of corrosion product
	X-ray diffraction	Identification of corrosion product
	Optical	Presence of localised corrosion/pitting
<b>Stress corrosion test specimens</b>	Optical	To measure the crack length
<b>Bentonite</b>	SEM/electron microprobe analysis - bentonite	Distribution of iron in bentonite
	Raman spectroscopy	Complimentary to FTIR
	X-ray diffraction	Characterisation of minerals, estimation of Fe content
	Exchangeable ion analysis and cation exchange capacity	Characterisation of bentonite
	FTIR	Study of Fe-mineral bond
	Hydraulic conductivity and swelling pressure	Effect of iron on properties of bentonite
	ICP-AES	Total chemical composition
	Sequential chemical extraction	To identify adsorbed species
	Porewater squeeze / analysis	Composition of porewater in bentonite

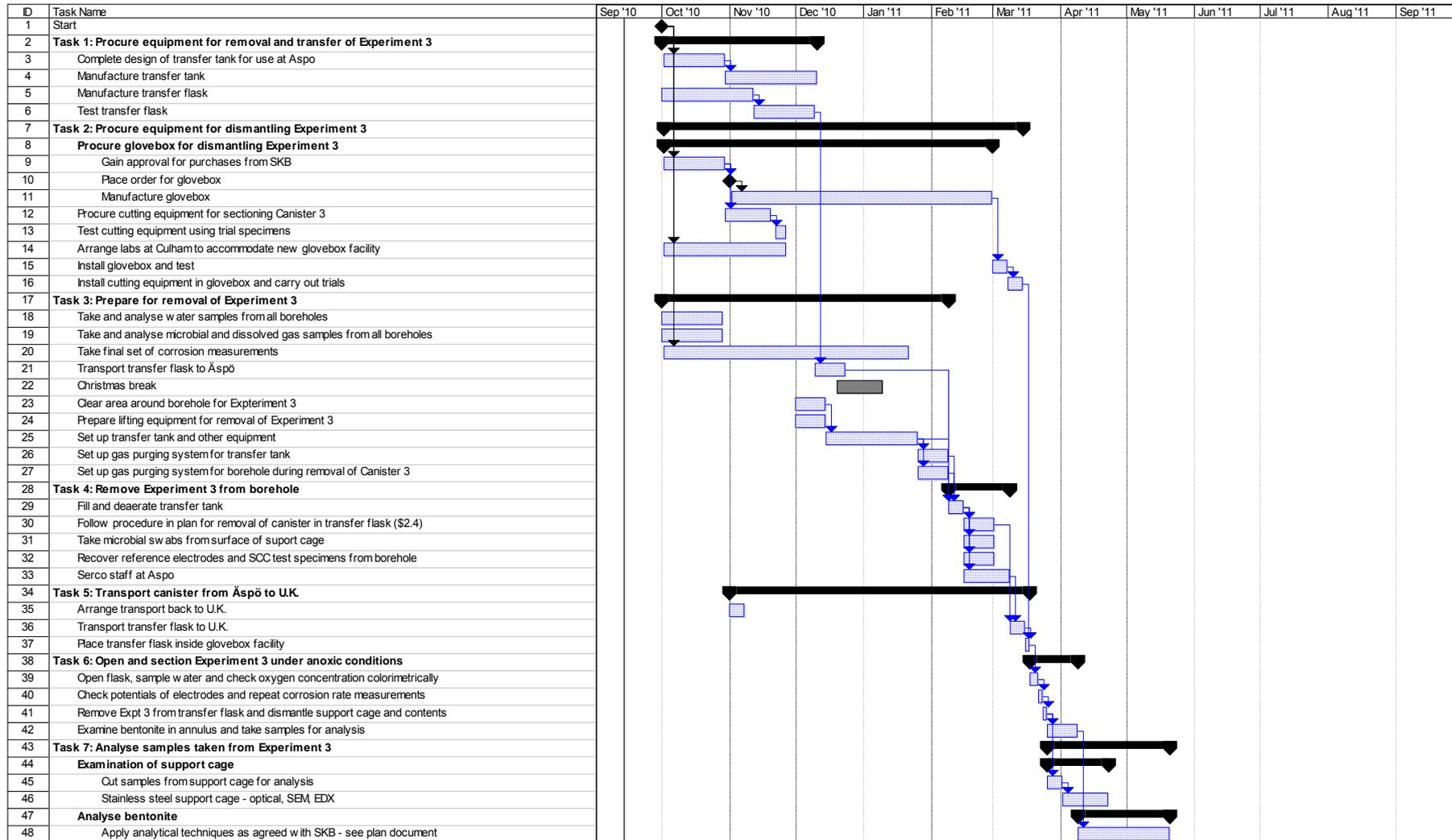


Figure 1. Proposed schedule for removal and analysis of Experiment 3.

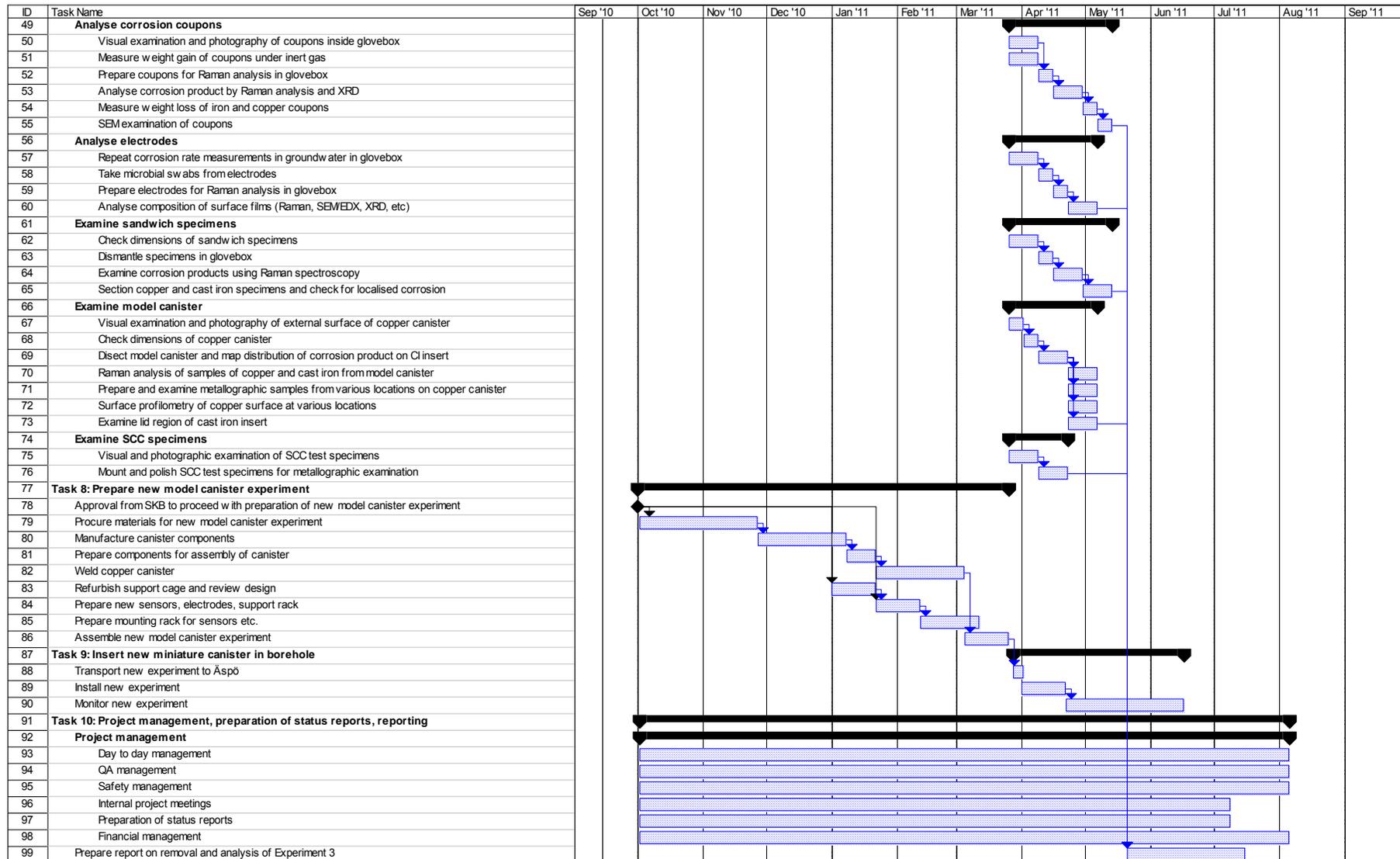
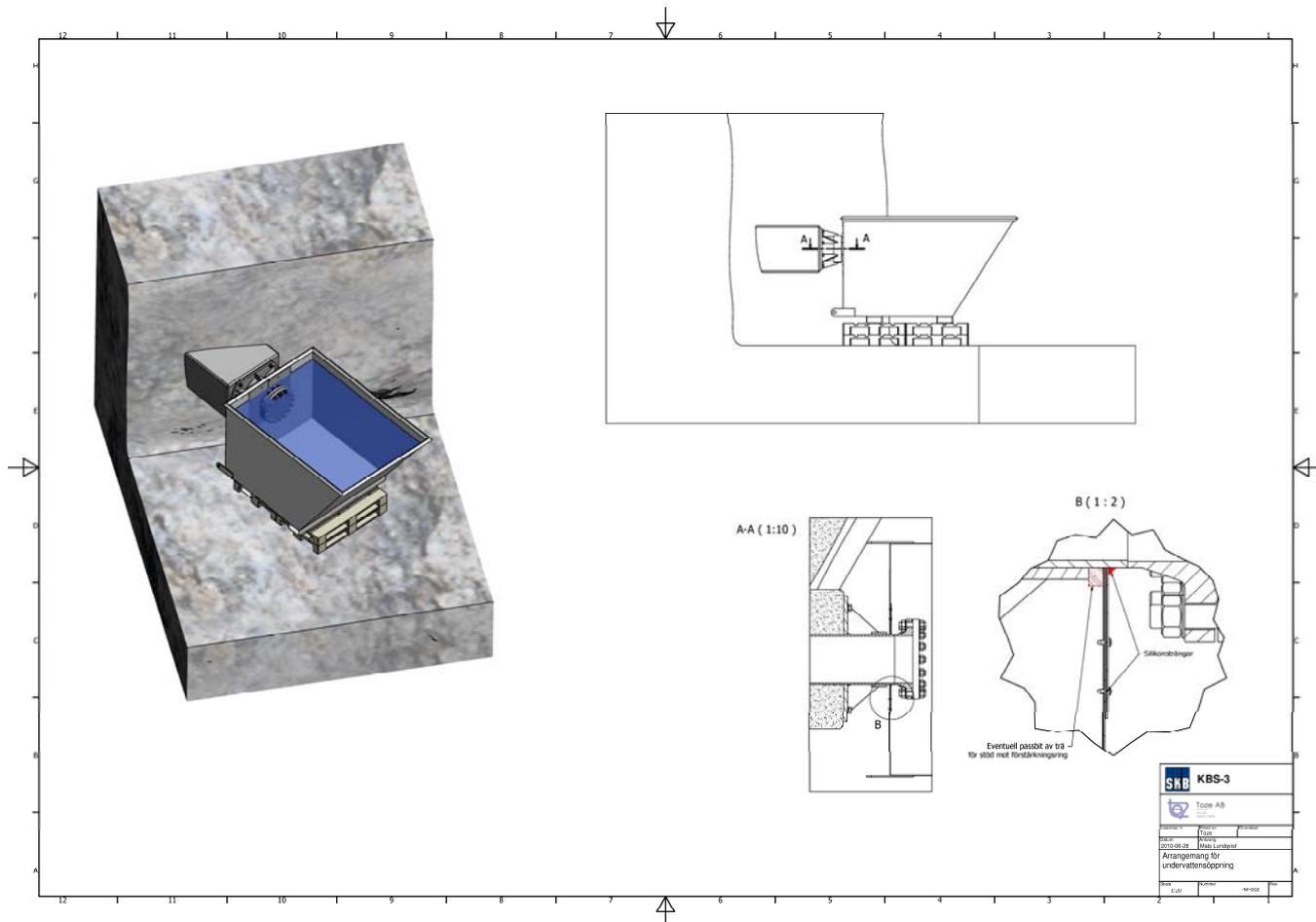
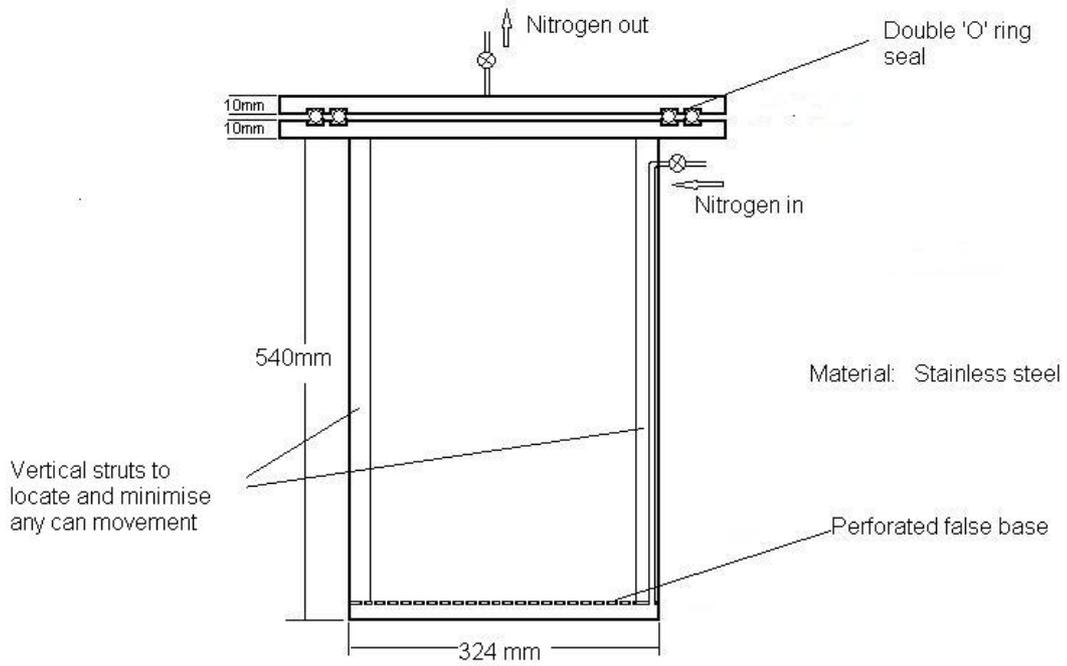


Figure 1 (contd). Proposed schedule for removal and analysis of Experiment 3.

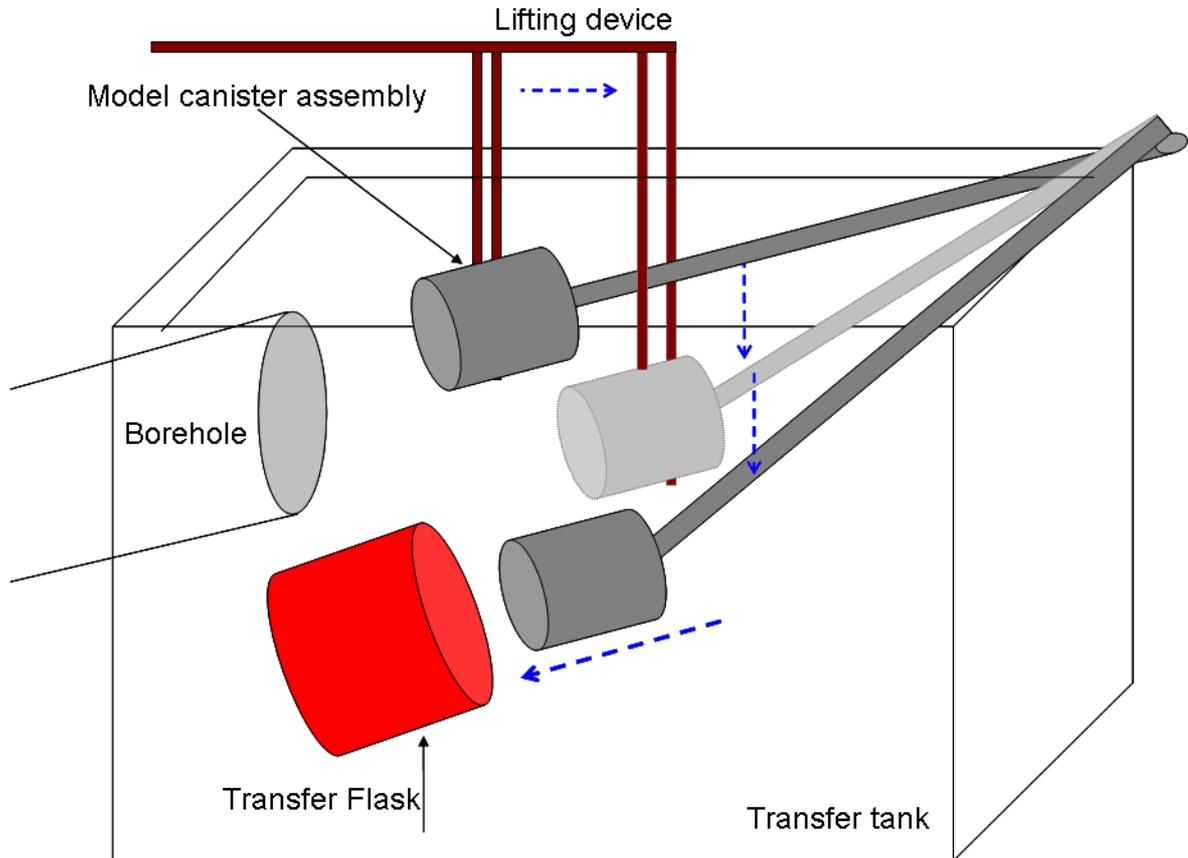




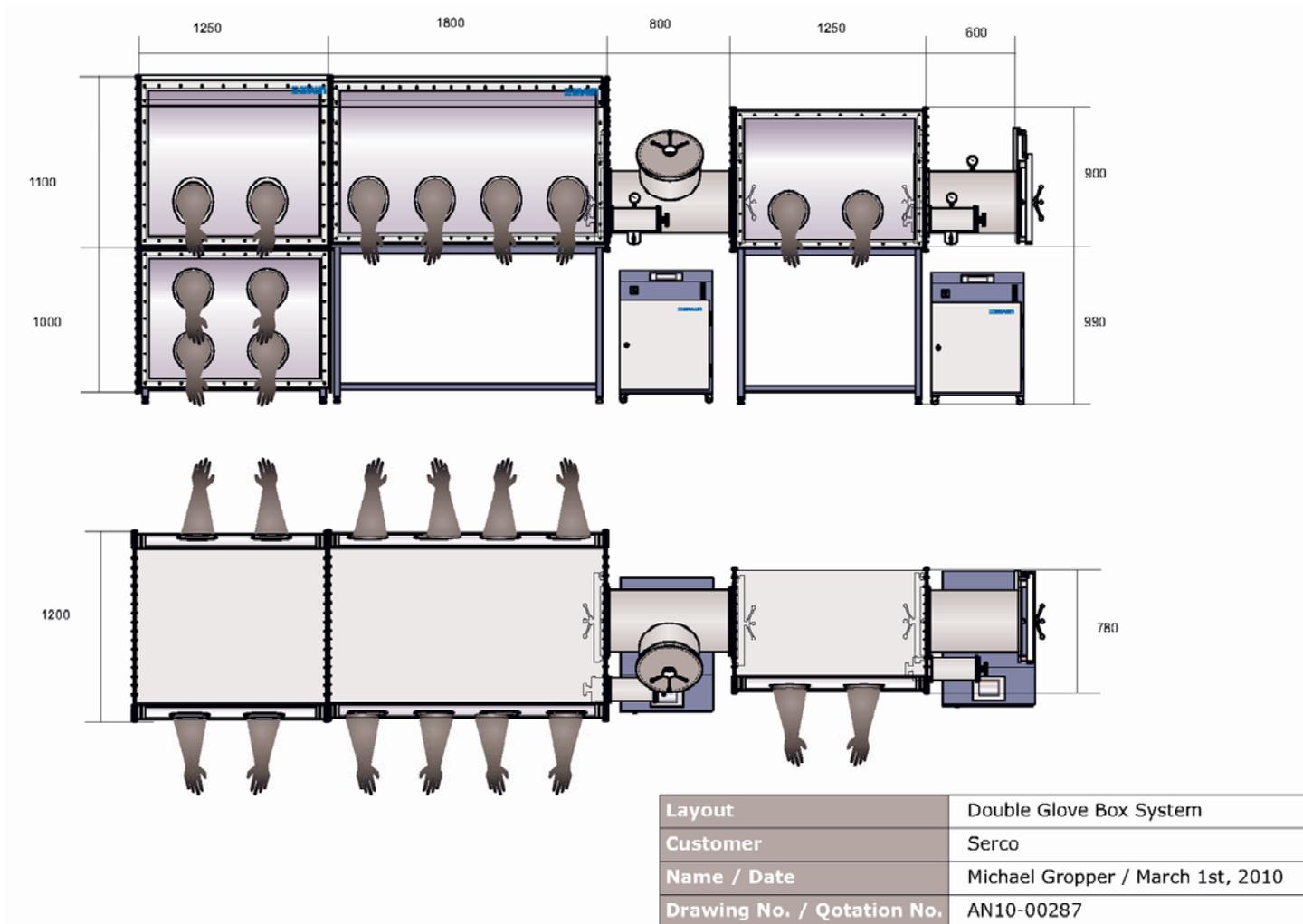
**Figure 3. Further details of the initial design of the borehole transfer tank proposed to remove MiniCan Experiment 3 (provided by SKB).**



**Figure 4. Initial design of the lidded transport flask planned to be used to anaerobically transfer Experiment 3 from the Äspö HRL to Serco's Culham laboratories in Oxfordshire, UK.**



**Figure 5. Schematic diagram illustrating the planned loading of Experiment 3 into the transport flask. It is envisaged that these operations will take place within the transport tank under deaerated water.**



**Figure 6. Proposed Double Glove Box system specifically designed (in collaboration with one of our suppliers) by Serco staff for use in disassembling MiniCan Experiments. The glovebox component on the left is for removing Experiment 3 from the transfer flask, the central part will contain the cutting equipment and the right had part would be used for preparation of specimens.**

# Appendices

## Contents

Appendix 1      Water analysis activity plan

# Appendix I

## Water analysis activity plan

### Contents

Title 1	Preliminary water analysis plan prepared by Ann-Sofie Karlsson TX 2010-07-05
---------	---

## Introduction

During 2005 and 2007 Äspö Chemistry Laboratory performed water sampling for the Mini-Can Project. In the spring of 2010 one of the canisters will be removed from the project site and before the removal, water sampling of the same parameters as before is of great importance. The sampling will be performed at all the five canisters, both from the inside and outside the support cage. The boreholes are situated at -420m level in Äspö HRL.

## Roles and responsibilities

Role	Responsibility
<b>Project leader</b> <i>Nick Smart</i>	Coordinates the project/process/operation including the activity. Approves the activity plan and final delivery. Takes decisions whether an activity is finished.
<b>Activity leader</b> <i>Ann-Sofie Karlsson</i>	Issues the activity plan. Conducts and control realisation of this activity. Responsible for control and archiving of produced results.
<b>Coordinator</b> <i>Mats Lundqvist</i>	Assists activity leader in management, realisation and control of the activity in field.
<b>Project administrator</b> <i>Siren Boen Mortelid</i>	Accounts for helping with documents and protocols.
<b>Subject representative</b> <i>Linda Alakangas</i>	Accounts for questions concerning respective subject areas and gives scientific support to the project, experiment and studies at Äspö HRL. Reviews the activity plan.

## Prerequisites

### Time Schedule

The sampling will be performed in the autumn 2010. It will take a maximum of two hours and will preferably be done early in the day to make time for analyses in the afternoon. The exact date will be decided later and depends on when other projects like Sulfidprojektet (KBP4001) are finished.

### Delivery of results

The parameters analysed at the Äspö Chemistry Laboratory will be reported as soon as possible. When all the results from external laboratories are delivered, the results will be quality controlled and the activity signed for in Sicada. After that a Report of the water sampling will be written and delivered to the project leader.

## Realization

### Sampling site

The water sampling will be performed at the project site in Äspö HRL at -420 m. The boreholes that will be sampled are:

- KA3386A02
- KA3386A03
- KA3386A04
- KA3386A05
- KA3386A06

All the boreholes listed above except KA3386A05 will also be sampled from the inside of the canister.

## Sampling

All the sampling is performed according to SKB MD 452.001 Provtagning och provhantering (Water sampling and handling of samples) and SKB MD 451.017 Provtagningstyper och felkällor (Sampling types and source of errors).

At the sampling the following equipments are used: valves, tecalantube, rustfree Swagelok-connections, pressure guage, on-line filter (Nucleopore) and high-capacity filter (Colly Company). Pore size on all filters are 0.45 µm.

Before sampling of the five boreholes outside the canisters, the boreholes will flow for approximately 10 minutes. Sampling from the inside of the canisters will start directly when the valve opens. These instructions were followed during the last campaign, new directives may come this time.

## Parameters

The parameters that will be analysed are the same as previous sampling campaigns:

Analyses	Volume	Comment
<b>Electric conductivity</b>	250 ml	
<b>pH</b>		Included in the bottle for conductivity.
<b>HCO<sub>3</sub><sup>-</sup></b>		Included in the bottle for conductivity.
<b>Cl<sup>-</sup></b>		Included in the bottle for conductivity.
<b>SO<sub>4</sub><sup>2-</sup></b>		Included in the bottle for conductivity.
<b>Br<sup>-</sup></b>		Included in the bottle for conductivity.
<b>F<sup>-</sup></b>		Included in the bottle for conductivity.
<b>Fe (total+II)</b>	250 ml	Was not analysed in the end of the last campaign
<b>NH<sub>4</sub>-N</b>	2*25 ml	Was not analysed in the end of the last campaign
<b>HS<sup>-</sup></b>	2*116 ml	
<b>Major cations<sup>A</sup></b>	100 ml	
<b>NO<sub>2</sub>, NO<sub>3</sub>, NO<sub>2</sub>+NO<sub>3</sub>, PO<sub>4</sub></b>	100 ml	
<b>Freeze stored back-up sample preserved</b>	2*100 ml	Can be removed if minimized sampling volume is important.
<b>Freeze stored back-up sample non preserved</b>	2*250 ml	Can be removed if minimized sampling volume is important.
<b>TOC (addition)</b>	15 ml	
<b>Environmental metals<sup>B</sup> (addition)</b>		Included in the bottle for major cations.

<sup>A</sup> = Major cations: Na, K, Ca, Mg, Si, Fe, Mn, Li, Sr, S

<sup>B</sup> = Environmental metals: Al, Ba, Cd, Co, Cr, Cu, Hg, Mo, Ni, P, Pb, V, Zn

It's defined as a SKB Chemistry Class 5 without DOC and uranin but with TOC and environmental metals as extra addition.

Required volume for the sampling above is approximately 1700 ml but the volume can be minimized by excluding the back-up samples (or just sampling one bottle instead of two).

## Laboratories

The laboratories that will analyse the parameters are:

Laboratory	Parameter (Method)
Äspö Chemistry Laboratory, Sweden	pH, conductivity, Cl <sup>-</sup> (potentiometric), HCO <sub>3</sub> <sup>-</sup> , Fe (tot+II), HS <sup>-</sup> , NH <sub>4</sub> -N, F <sup>-</sup> (ic + potentiometric), Br <sup>-</sup> (ic), SO <sub>4</sub> <sup>2-</sup> (ic)
ALS Scandinavia, Sweden	Major cations, environmental metals (pkt 4)
Department of System Ecology Stockholm University, Sweden	NO <sub>2</sub> , NO <sub>3</sub> , P-PO <sub>4</sub> , NO <sub>2</sub> +NO <sub>3</sub> , TOC

Äspö Chemistry Laboratory is accredited by Swedac since 2003. That means that the laboratory performs analyses with high quality and traceability.

## Results

Ni, Cu and Cr are of great interest for the project and will be reported despite the fact that these are metals that can be affected by the instruments and material in the boreholes.

## Quality and control, handling of data

The Äspö Chemistry Laboratory is responsible for delivery of quality assured results from the water sampling. Handling of data is to follow SDTD-217 Hantering av data vid Äspölaboratoriet (Management of data at the Deep Repository Technology unit).

Äspö Chemistry Laboratory has its own system with regulating documents and all the work is documented. Handling of all analyses and their protocols with results will be done according to SKB MD 451.008 Dokumenthantering och arkivering (Management of documents and archives). Quality control of all results will be done according to SKB MD 451.013 Rapportering av data till Sicada (Delivery of data to Sicada).

## Risk assessments, safety, health and environment

<b>Work that can be critical by working-environmental point of view</b>	<b>Yes</b>	<b>No</b>	<b>Work that can be critical by environmental point of view</b>	<b>Yes</b>	<b>No</b>
Occurring of noise		X	Discharge to the air		X
Occurring of dust		X	Discharge to water (drain/recipient)		X
Occurring of smells		X	Discharge of surplus heat		X
Occurring of vibrations		X	Occurring of wrapping waste		X
Work from ladder or platform		X	Occurring of hazardous water		X
Electricity safety		X	Occurring of other solid waste		X
Need of robe-off area		X			
<b>Work with special permit</b>	<b>Yes</b>	<b>No</b>	<b>Use of chemicals</b>	<b>Yes</b>	<b>No</b>
Work with radioactive tracers		X	Use of chemicals	X	
Work in boreholes or / enclosed spaces		X	Use of heavy metals		X
Blasting		X	Use of other dangerous substances		X
Hot work		X			
Work with truck/electric telfer/telfer line/sky lift		X			
Use of machines		X			
Use of fuel for machines		X	<b>Others</b>	<b>Yes</b>	<b>No</b>
			Need of robe-off area		X

The water sampling and the following analyses are regulated by the routines and instructions managed by the Äspö Chemistry Laboratory. The safety issues for this type of work are therefore taken care of.

## Controlling activity table

	<b>Activity</b>	<b>Input</b>	<b>Controlling Document</b>	<b>Results/Deliveries Reporting documents</b>	<b>Time</b>	<b>Responsible</b>	<b>Comments</b>
	<i>States what is to be executed</i>	<i>States what data are needed to be able to execute the activity</i>	<i>States reference number/chapter where it says how the activity is to be executed.</i>	<i>States what results are to be delivered after completion of the activity and how they are to be reported.</i>	<i>States timer period and/or estimated amount of time required of the activity.</i>	<i>States who is responsible of execution.</i>	
<b>1</b>	Water sampling	Clearance of the site and a decision for a suitable date.	This AP	Daily logs of the activity to Sicada.	4 h	ASS	
<b>2</b>	Analyses at Äspö Chemistry Laboratory	The samples	Äspö Chemistry Laboratory's methods of analyses, all included in the SKB's management system.	Results into Sicada.  Report of Äspö's analyses delivered to the project leader.	3 weeks from sampling	ASS	
<b>3</b>	Analyses at external laboratories	The samples		Reports with results delivered to Äspö Chemistry Laboratory. Results written in Sicada.	4 weeks from sampling	ASS	
<b>4</b>	Final Reporting	Quality controlled data in Sicada.	SKB MD 451.013	Chemistry Report to the project leader.	1 week when all results are delivered.	ASS	



## List of risks

	Risk	Probability	Consequence	Risk value	Action taken	Risk owner
1	Chemical contact with skin	3	3	9	Always use gloves when handling chemicals.	ASS
2	Chemical contact with eyes	2	4	8	Always use eye protection when handling chemicals.	ASS

Probability for the risk to happen
5 50 - 100%
4 25 - 50%
3 10 - 25%
2 1 - 10%
1 0,1-1%

Consequence from the damage
5 Serious
4 Less Serious
3 Severe
2 Less Severe
1 Insignificant

Risk value = Probability * Consequence
Risk value 10-25 means <i>high</i> risk
Risk value 5-9 means <i>medium</i> risk
Risk value 1-4 means <i>low</i> risk



## Time plan

The sampling will be performed in the autumn of 2010.