Appendix 3

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Arbeitsbericht NAB 14-55

FEBEX - Assessment of Redox Conditions in Phase 2 before Dismantling

July 2014

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Nationale Genossenschaft
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radioaktiver Abfälle

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KEYWORDS

FEBEX, bentonite, EBS, redox conditions, gas analysis, microbiology, corrosion

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1 Introduction

1.1 Objectives

An assessment of the current redox state of the FEBEX Phase II experiments is part of the preparation of the dismantling of the second heater, planned to occur in 2015. The oxido-reduction state inside the FEBEX experiment setup controls the chemical reactions affecting metal corrosion and mineral phase alteration and precipitation. Particularly important are the microorganism colonies which are as well strongly dependent on the oxygen concentration of their environment and which control many corrosion processes.

The objective of assessing the present conditions inside the experiment is to provide sufficient information to allow selecting efficient sampling and dismantling procedures. The final goal is to obtain and analyse samples from the various parts of the experiment that are as close as possible to the in-situ conditions before the start of the dismantling operations, and to provide a support to subsequent discussions on the potential alteration or contamination that may have affected the samples as compared to their "undisturbed" in-situ conditions.

The information available today includes the knowledge of the environment before closure of the experiment by a concrete plug, i.e. the conceptual design of the experiment, the chemistry and mineralogy of the equipment and the bentonite buffer, the hydro- and geological settings, etc. Other important sources of information are the physical and chemical data collected during the operational phase of the heater, including water and gas samples as well as data collected from the numerous sensors placed inside the experiment. Finally, the dismantling of Heater 1 also provides a wealth of information on what conditions to expect in the excavation of Heater 2.

1.2 Factors Controlling Redox Conditions

The redox conditions are mostly controlled by:

- the O_2 present in the experiment before closure
- redox equilibria in the surrounding rock
- the corrosion of metals present in the experiment
- microbiology
- inflow of O_2 from the access gallery into the experiment (including through the gas sampling pipes)

Some of these factors are interdependent. The first two, the conditions in the experiment at the time of closure and the conditions in the host-rock can be fairly well understood and constrained. The latter three factors are difficult to assess without actually dismantling the experiment. What is well known is that the groundwater at Grimsel is O_2-free (Frick et al. 1992) and that the degradation of organic matter in the experiment will very likely lead to an anoxic environment. This natural evolution is balanced by the possible O_2 flow into the experiment from the access gallery, through the plug, along cables and pipes, or through the EDZ. In this case, the O_2 ingress will strongly influence the microbial activity in the experiment and the rates and types of corrosion.

Some insight on these processes can be gained from gas analyses performed on samples from within the experiment during Phase 1 and Phase 2 (Jockwer & Wieczorek 2008), and from the dismantling of the first heater (Huertas et al. 2006, and references therein).

2 Results from Heater 1 Excavation

2.1 Experiment Design of Phase 1

2.1.1 Concrete Plug

The concrete plug constructed to seal the FEBEX experiment from the GTS access tunnel was made of plain concrete (Fig. 1), without any reinforcement, in order to facilitate dismantling (Huertas et al. 2006). The design is specific to the FEBEX experiment, since the reference concept does not include a design of the plug (Fuentes-Cantillana & García-Siñeriz 1998). Most importantly for the chemical behaviour of the system, no specification was included for the water tightness or gas tightness of the concrete plug; the only requirement for the plug was to withstand the mechanical stress resulting from the swelling pressure of the bentonite.

2.1.2 Cable Pass-through

Four 200 mm diameter PVC tubes were inserted in the plug as pass-through for the >600 cables and pipes linking the experiment to the access gallery. The tubes were later filled with polyurethane foam in the outer part and with slightly expansive fine mortar in the interior part (Fuentes-Cantillana & García-Siñeriz 1998). The sealing, however, never proved to be complete, especially for the right lateral tube, where water leaks were still observed after sealing (op. cit.).

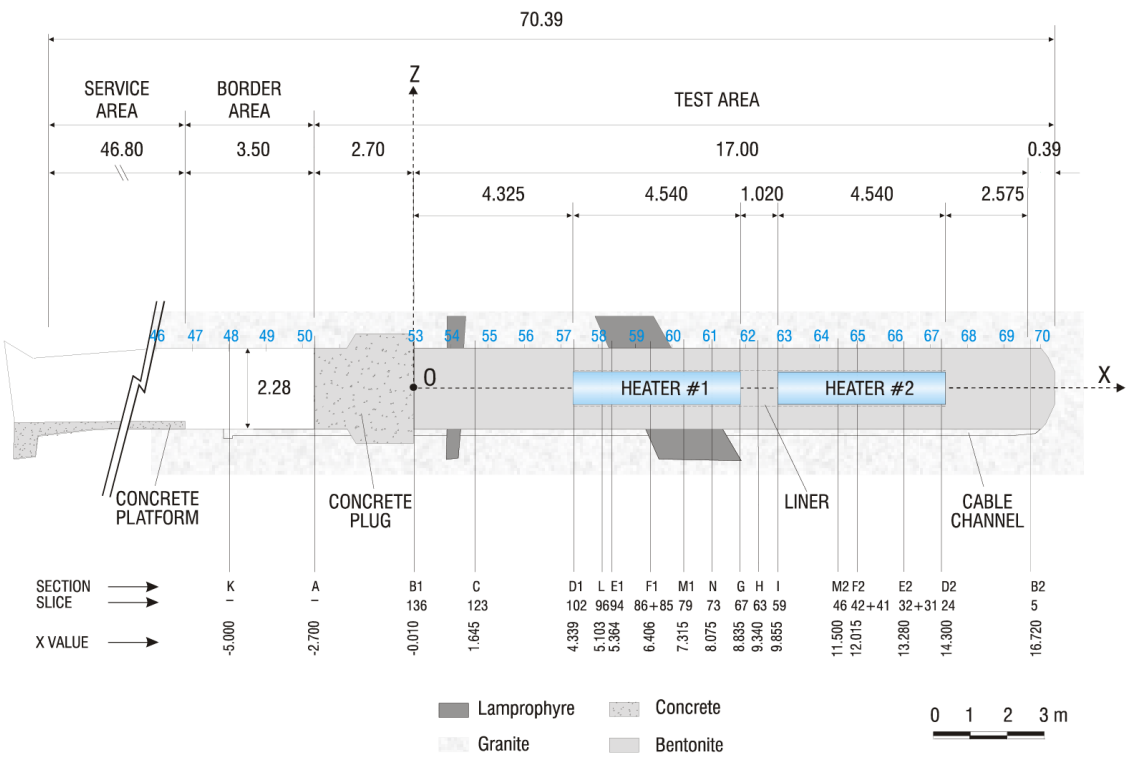

Fig. 1: Longitudinal section of the FEBEX tunnel operational phase I (FEBEX I) with the heaters, bentonite buffer, concrete plug, and surrounding rock (Huertas et al. 2006)

2.1.3 Gas Analyses

The Gesellschaft für Anlagen- und Reaktorsicherheit (GRS) analysed fluids during Phase I and II of the FEBEX experiment (Jockwer & Wieczorek 2008).

The gas phase present in the pore space of the bentonite buffer was sampled through ceramic filter pipes around heater 1 during Phase I. These draining pipes were emplaced to allow for gas content and permeability measurements. The system also allowed taking water samples if the pipes were located in a saturated part of the experiment.

The measured oxygen concentration decreases after closure of the experiment from an atmospheric concentration of 20 %v/v, to less than 2 %v/v. As a consequence, the N_2 concentration increases from about 80 %v/v to about 90 %v/v. The fact that O_2 was measured throughout the entire running of Phase 1 of the experiment shows either a slight air contamination of the samples, or an inflow of atmospheric gases through the plug, or both. Fig. 2 shows the O_2 concentration measured in samples collected from the filter pipes. Besides a general trend towards a decrease of the O_2 concentration, the great variability seems to indicate that some of the samples were contaminated by air, either in-situ or upon sampling.

The random pattern of the O_2 variation suggests that the high O_2 concentrations are a consequence of contamination of the sample by air at the time of sampling, or subsequently upon sample storage and/or handling. A study of N_2/Ar ratios in measured in the samples could provide some insight on the source of O_2. Argon has so far not been analysed in the in-situ gas sampling campaigns of the FEBEX experiment, but will be analysed in future campaigns.

During the two FEBEX phases, a similar pattern was observed in the gas concentrations with high H_2 concentrations shortly after the sealing of the plug, followed by a decrease (Fig. 3). This succession of high H_2 concentration shortly after construction of the plug and decrease has been interpreted as the result of anoxic corrosion of fresh metallic parts and escape of the H_2 through the non-gas-tight plug. However, H_2 escape towards the access gallery should have been even higher before plug construction. The most likely explanation for the decrease of H_2 is the activity of methanogen bacteria.

What this uncertainty implies, is that one cannot ascertain the presence or lack of oxygen in the system based on these analysis.

Fig. 4 shows the concentrations of O_2, CO_2, H_2, CH_4 and the sum of light hydrocarbons sampled in filter pipe GF-S-L-06 during Phase 1. Additionally to the concentration evolutions described above, one can observe opposite trends between CO_2 concentrations and hydrocarbons (including CH_4). This behaviour, together with the variation of H_2, suggests that gas concentrations are controlled by microbiological processes (Fernández et al. 2012).

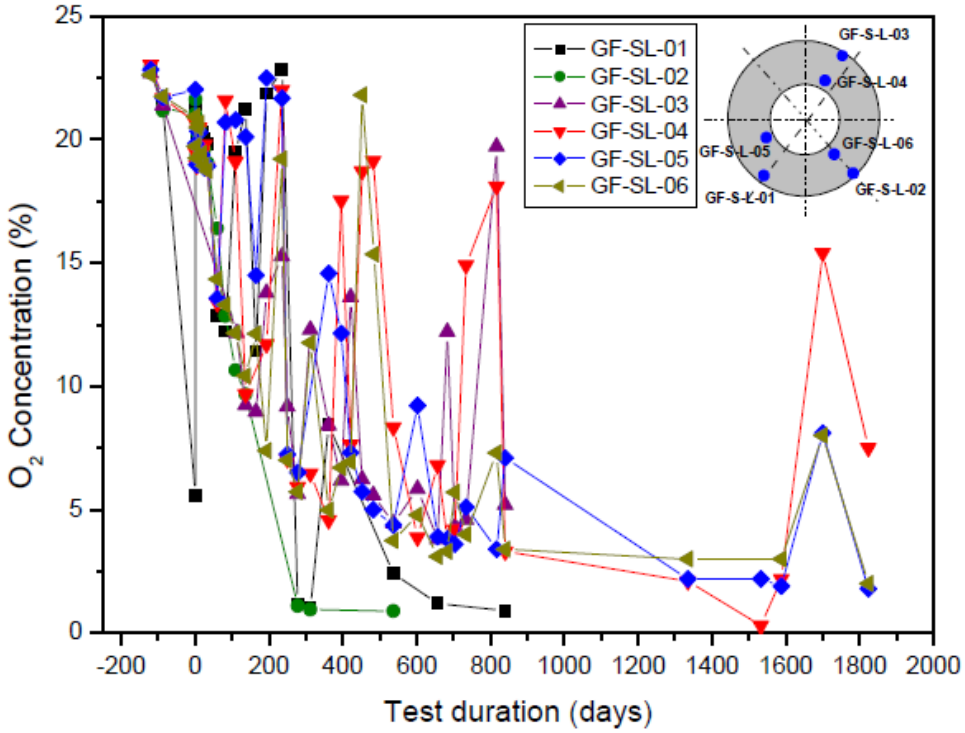

Fig. 2: O_2 concentrations (in % v/v) in samples collected from the different filter pipes during Phase 1 (Fernández & Wieczorek, in prep.)

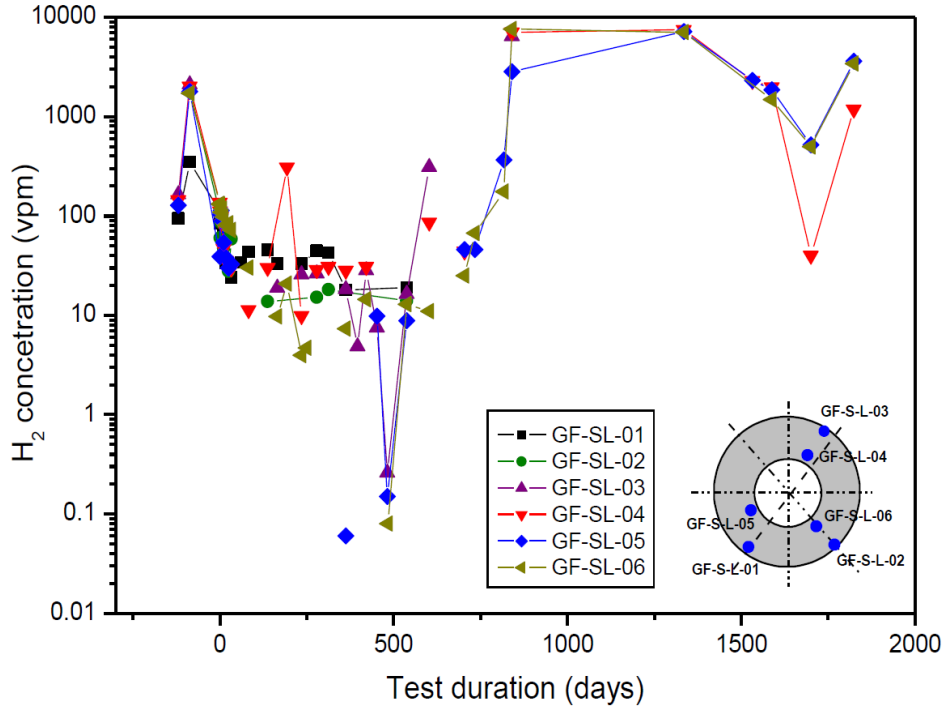

Fig. 3: H_2 concentrations (in vpm) in samples collected from the different filter pipes during Phase 1 (Fernández et al. 2012)

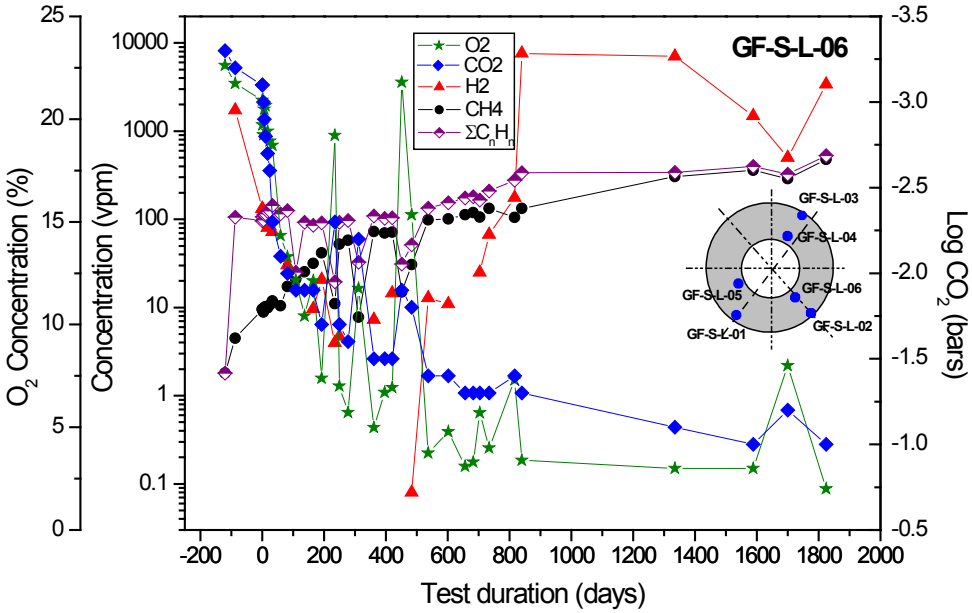

Fig. 4: Concentrations of O_2, CO_2, H_2, CH_4, and light hydrocarbons in samples collected from filter pipe GF-S-L-06 during Phase 1 (Fernández & Wieczorek, in prep.)

2.2 Sampling after Dismantling of Heater 1

2.2.1 Corrosion Study

INASMET studied corrosion on different components of the experiment after dismantling of Phase 1 (INASMET, 2003). The surfaces and elements studied include the heater, the liner hosting the heater in the experiment, corrosion coupons, sensors, and the chemistry and microbiology of the bentonite.

2.2.1.1 Metal Parts and Coupons

The heater and liner, made of carbon steel and steel, respectively, both showed a limited corrosion, thanks to the very low humidity in the immediate vicinity of the heater. No pitting or localized corrosion was observed.

Corrosion coupons of various metals had been emplaced in the Phase 1 experiment, close to the heater, in the lower part of the bentonite buffer. The metal coupons comprise carbon steel, stainless steel, various titanium alloys, pure copper, and two different cupronickel alloys. The interpretability to corrosion processes based on the analysis of the coupons after dismantling is limited by the general low corrosion observed on the samples. The carbon steel samples showed very low corrosion, with a thin layer (not measurable by XRD) of iron oxide at the surface, whereas stainless steel and titanium coupons showed no corrosion at all. Surprisingly, the corrosion rates were highest on the copper and cupronickel alloys. The only analysed corrosion product on the latter coupons is cuprite.

The corrosion rates of the steel coupons were significantly lower than what had been measured in laboratory testing performed by INASMET in saturated bentonite. The cause of this discrepancy is mostly the low water-saturation level of the bentonite hosting the coupons and

the more anoxic conditions in the FEBEX experiment, compared to the laboratory experiment. However, the corrosion rates are similar in the laboratory and in the full-scale experiment for the Cu-bearing coupons.

The various sensors analysed for corrosion provide a richer variety of corrosion rates, due to the localization of some, or parts, of them in the saturated part of the experiment close to the tunnel wall. One type of sensors studied is the extensometers designed to measure the relative displacement of the heater to the tunnel wall. They are made out of stainless steel and attached to the liner at one end and to the host rock at the other end. The second type of sensors is steel thermocouples. The damage due to corrosion is very important in these sensors, with pitting leading to complete perforation in some parts of the sensor casings. Different corrosion products, mostly green or reddish in colour, were observed on the sensors and in the bentonite hosting the sensors. In almost all of the corrosion products, EDS analysis detected the presence of sulphur.

2.2.1.2 Bentonite

The microbiological analysis of the bentonite did not detect any microbial activity in the bentonite blocks in which the corrosion coupons were emplaced (INASMET 2003). On the other hand, the block hosting one of the extension sensors showed an important microbial activity, both of aerobic and anaerobic organisms (Tab. 1). The aerobic population consisted only of fungi, and the anaerobic population consisted of sulphate reducing bacteria (SRB). The corrosion of the extensometer does suggest an important influence of SRB. The authors of the report explain the coexistence of aerobic and anaerobic organisms by the growth of SRB under a biofilm of aerobic organisms, therefore protected from the O_2 present in the environment.

2.2.3 Bentonite Analysis

A thorough post-mortem bentonite analysis is presented in Villar et al. (2006) and Fernández et al. (2010). CIEMAT performed a series of bulk chemical analysis of the solid phase of the sampled bentonite, together with a piece of untreated bentonite block used as reference. The most significant difference in composition of the samples from FEBEX in-situ compared to the reference sample is a decrease in reduced species. For example, the concentration of FeO decreased, while that of Fe_2O_3, the more oxidised species, increased. Altogether, a decrease of divalent species was observed (Fig. 5).

Tab. 1: Microbiological characterisation of three samples of bentonite. c.f.u.: Colony forming units

Microbe group	**c.f.u./g count of sample**		
	Bentonite ref. SH-SD1-01 rock	**Bentonite ref. SH-SD1-01 liner**	**Bentonite ref. BS24-2**
Anaerobic bacteria	0	0	0
Aerobic bacteria	2×10^2*	4.23×10^3*	0
Sulphate-reducting	1.08×10^2	3.77×10^2	0
Iron-oxidizing	0	0	--
Sulphur-oxidizing	0	0	--

* The whole count consisted of fungi. Source: INASMET, 2003

Similarly, the chemical analysis of the solid phase of some bentonite samples carried out by INPL/LEM concluded to a large predominance of Fe^{+3} compared to Fe^{+2}.

The analysis from aqueous extracts from the bentonite showed a lixiviation of the most soluble components in the most hydrated parts of the experiment, i.e. closer to the tunnel wall, and a subsequent enrichment in the parts closer to the heater. Chloride is the most mobile species, followed by Na and SO_4. Calcite and dolomite are dissolved in the areas in contact with the granite, leading to a strong increase in bicarbonate (see also Samper et al., 2008). Data obtained on squeezed pore water of the bentonite showed similar patterns (Fernández et al. 2010).

During the gas sampling campaigns by GRS (Jockwer & Wieczorek 2008), water samples were collected in some of the sampling pipes and sent to CIEMAT for chemical analysis. Total Fe and Fe^{+2} were analysed in six samples from 2001 and 2002. In three of the samples iron is above the detection limit, and Fe^{+2} represents almost the total of the dissolved iron, which is a sign of a reduced water (see Table XXV in Villar et al. 2006, Fernández et al. 2007).

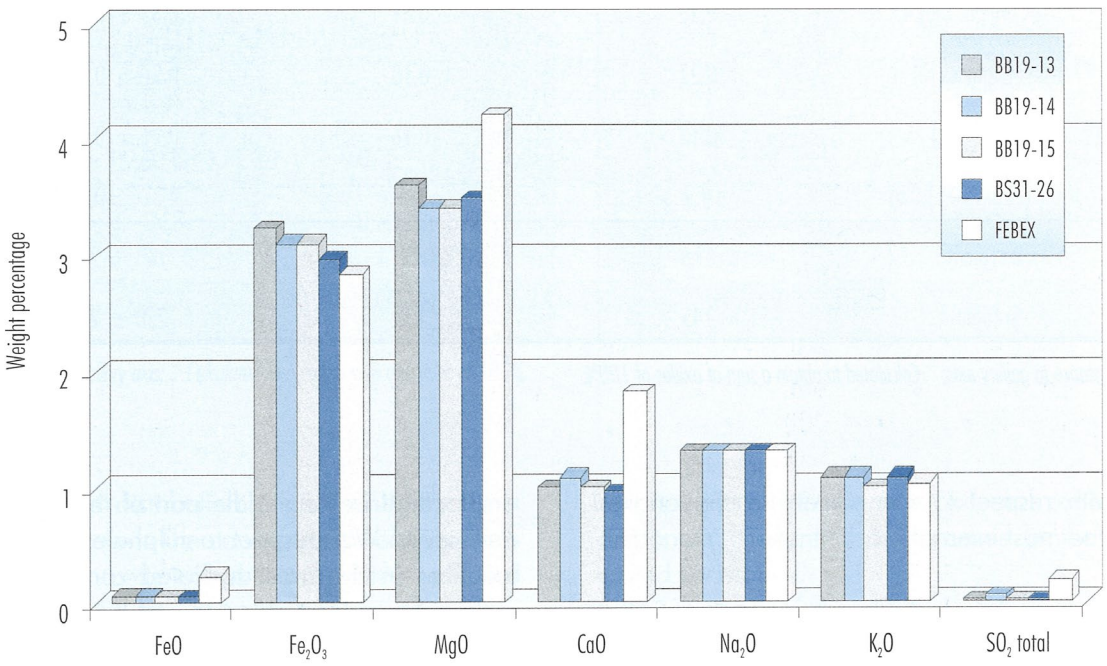

Fig. 5: Concentration of some species in the bentonite solid-phase from four different samples from the experiment Phase 1 and from a reference samples (from Villar et al. 2006)

Bentonite pore water obtained by squeezing was also analysed by VTT in Finland from samples at the interface with the concrete plug (Villar et al. 2006). The sampling and sample handling procedures were selected in order to minimize the contact of the bentonite sample with atmosphere. Both sulphide and ferrous iron were below the (relatively high) detection limit of 0.2 mg/l, while high concentrations, higher than 1000 mg/l of SO_4^{-2} were measured. The redox potential in the same samples gave values between -18 and -115 mV. Despite the low reduced sulphur and iron species concentrations, the negative Eh values indicate a rather reducing environment. It should also be kept in mind that these bentonite samples were taken in contact with the non-gas tight concrete plug, and that this area of the experiment was the most prone to air contamination from the access gallery.

3 Assessment of Current Redox Conditions

3.1 Experiment Design Specific to Phase 2

3.1.1 Concrete Plug

The sealing of the second part of the experiment, designed to keep heater 2 running for several more years (Fig. 6), was realised with a shotcrete plug, constructed in two phases (Fig. 7). A first, one meter thick plug allowed for installation of new sensors and instrument connections. This plug was constructed in 23-24 July 2002 (Huertas et al. 2006).

In June 2003, i.e. 11 months later, a layer of MASTERSEAL X345 was applied on top of the first temporary plug. This material is designed to improve water and gas tightness of the plug. Subsequently, a second section of 2 m thickness of shotcrete was applied to bring the total thickness of the plug to close to 3 m (Fig. 7).

Regarding permeability to gases, we can therefore expected relatively important exchanges through the plug in the period July 2002 to June 2003, while the experiment was only sealed by a 1 m shotcrete plug. At the end of June 2003, the gas permeability has strongly decreased, as a consequence of both the application of an impermeable layer, and the construction of additional 2 m of shotcrete plug.

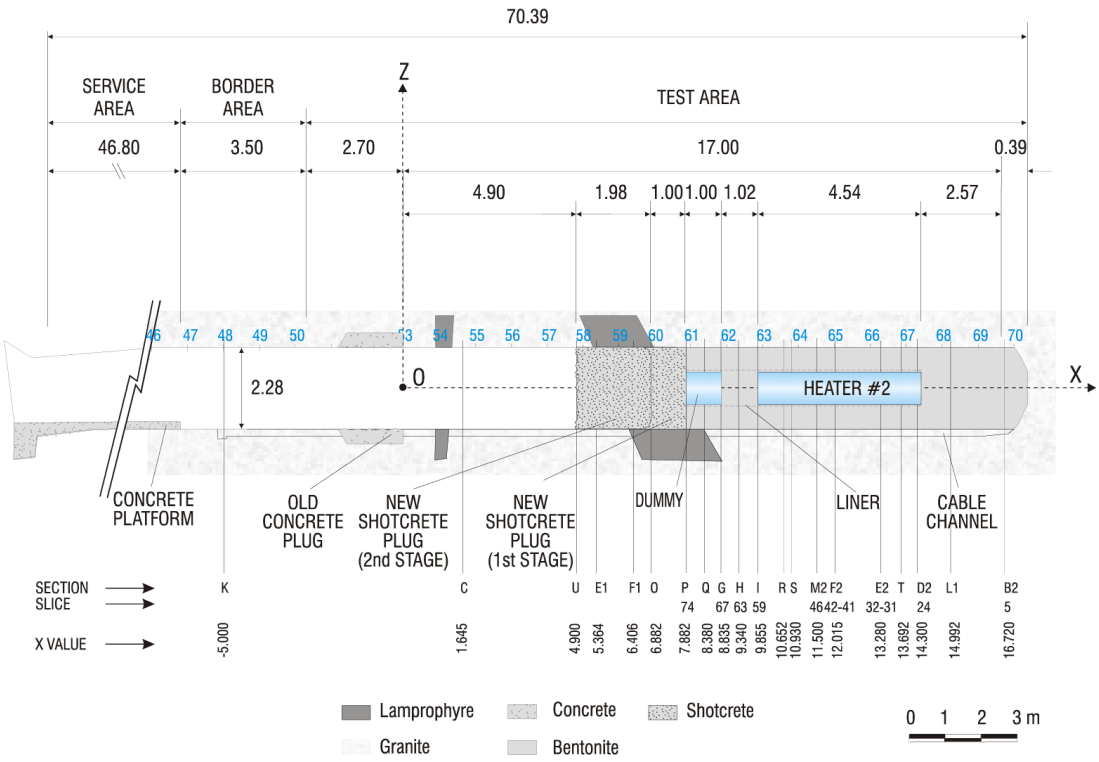

Fig. 6: Longitudinal section of the FEBEX tunnel operational phase I (FEBEX I) with the heaters, bentonite buffer, concrete plug, and surrounding rock (Huertas et al. 2006)

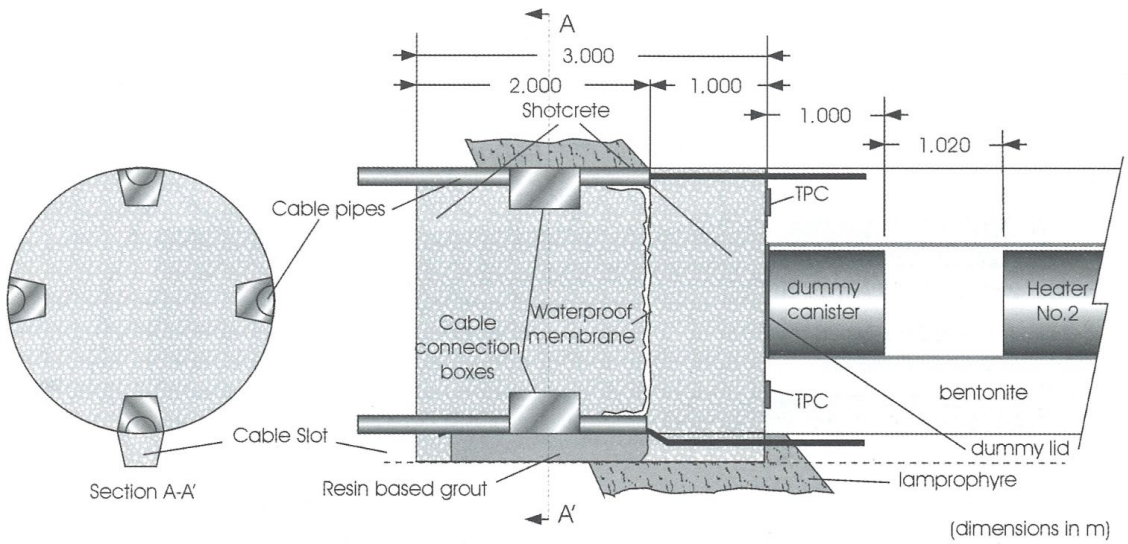

Fig. 7: Front and profile view of the shotcrete plug sealing Phase 2 of the experiment (Huertas et al. 2006)

3.1.2 Sensors and Sampling Pipes

After construction of the shotcrete plug to seal the part of the experiment remaining for Phase II, three new gas injection and collection pipes were installed in the buffer, in April 2003. The pipes were made of plastic with one three meter long sintered stainless steel sampling segment (Jockwer & Wieczorek 2008).

3.1.3 Fluid Analyses during Phase II

Similar to Phase 1, the O_2 concentrations measured in the samples taken from the sampling pipes around heater 2 and shown in Fig. 8 show large variations with time (Jockwer and Wieczorek, 2008, and CIEMAT, unpublished data). The higher values are the probable consequence of air contamination of the samples upon sampling and/or sample storage and handling. If we omit data from the two sampling campaigns showing very high values, the O_2 concentrations are much lower than the initial atmospheric concentration encountered at the time of closure of the experiment, even for the first sampling.

Measured H2 concentrations shortly after construction of the plug reach concentrations of up to 6,000 vpm but later decrease to less than 1,000 vpm (results for one of the sampling lines shown in Fig. 9). In one of the sampling ports (FP-1-C), after a decrease of H2 concentrations to less than 100 vpm, extremely high values have been recorded during at least three sampling campaigns, with concentrations exceeding 60,000 vpm, which corresponds to 6 %v/v (op. cit.).

The other gases show comparable trends to the gas data from the first Phase (Fig. 9), suggesting at least a strong influence of microorganisms on gas concentrations, linked to a probable ingress of O_2 from the access gallery. However, the reduced number of sampling lines (3 sampling lines compared to 6 in Phase 1) and the lack of sampling over a long period of time make interpretation risky.

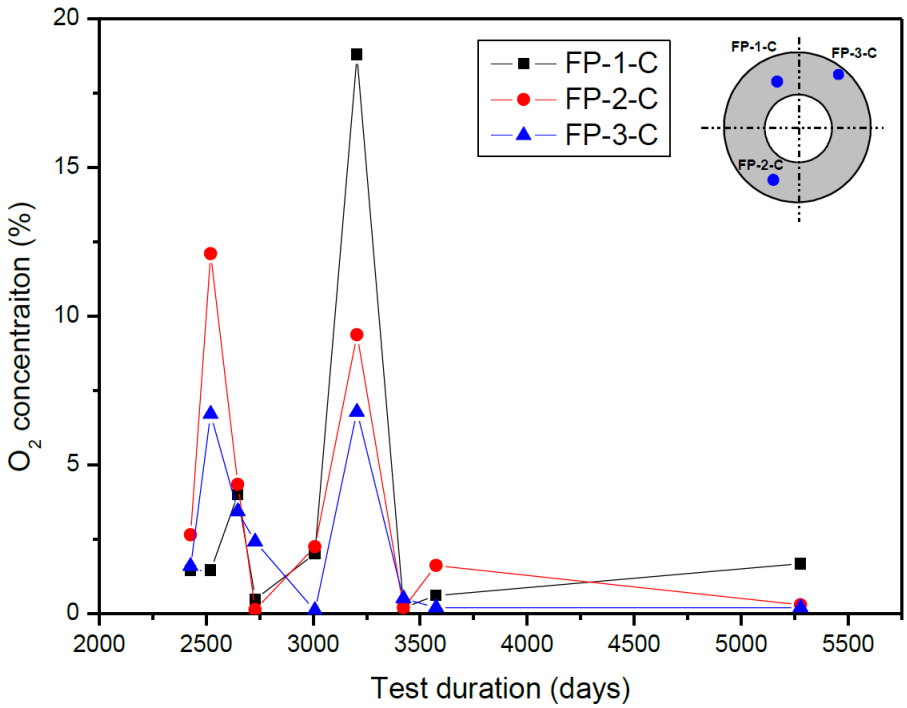

Fig. 8: O_2 concentrations (in % v/v) in samples collected from the different sampling pipes during Phase 2 (Fernández & Wieczorek, in prep.)

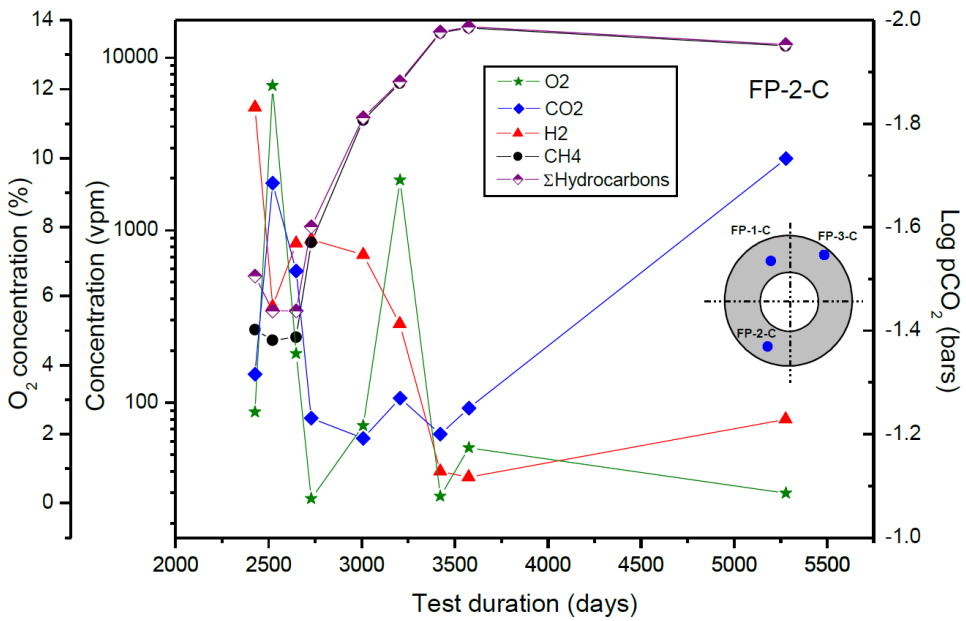

Fig. 9: Concentrations of O2, CO2, H2, CH4, and light hydrocarbons in samples collected from pipe FP-2-C during Phase 1 (Fernández & Wieczorek, in prep.)

Several water samples could be retrieved from the sampling pipes during the second operational phase (Fernández et al. 2007, 2010). One of the samples taken in 2005 could be collected in a gas-tight sampling bag and analysed in a glove box with very low O_2 (<1 ppm). This sample showed a strongly reducing redox potential of -284.2 mV.

Since the experiment is currently still running at GTS, and will be at least until spring 2015, when the heater will be switched off, the opportunity should be taken to perform 3 to 4 additional sampling and pore pressure measurement campaigns in order to improve the statistical significance of the gas data during Phase 2 and allow for a better assessment of the in-situ conditions at the time of starting the dismantling.

3.2 Potential O_2 Ingress from the Gallery

When the first heater was removed, no special protection was applied regarding chemical exchanges between the access gallery and the part of the experiment that was to remain in place for Phase 2. Subsequently, the a 1 m thick shotcrete plug was constructed and represented the only protection of the experiment to the atmosphere for about one year, before a gas-tight seal and the second part of the plug were constructed as well (see section 0). Gas exchanges have undoubtedly occurred during these two periods.

After the plug was completed, if one assumes that water and gas tightness of the plug material had been achieved, potential gas exchanges may still have been possible along sensor cables and pipes going through the plug, and through the EDZ. Tab. 2 summarizes the periods and location of potential O_2 contamination of the experiment.

Tab. 2: Possible gas exchange localization in time and space and estimate of the effect on the experiment O_2 content

Where	**When**
Open experiment front	During Heater 1 excavation, before first shotcrete plug construction
Through provisory shotcrete plug	Before plug sealing and construction of the final part of the plug
Through the final shotcrete plug	During Phase 2
Along sensor cables and pipes	During Phase 2
EDZ	During Phase 2

4 Experience from the SKB Prototype

The prototype repository at Äspö (Sweden), is a full-scale model of the Swedish concept KBS-3, of vertical emplacement of copper canisters. The prototype comprises 6 canisters (vertical emplacement concept) equipped with heaters, enclosed in a bentonite buffer. The access gallery is backfilled with sand/bentonite.

The chemistry and dissolved gases of pore water, the gas phase and the microorganisms are regularly analysed within the prototype. Additionally, the outer section of the prototype has been excavated and allowed for a more thorough sampling and analysis campaign.

The main outcome of the chemical monitoring and excavation of the SKB prototype at Äspö is that O_2 levels have decreased faster than would be expected from abiotic processes (Arlinger et al. 2013)

4.1 Gas Analyses

The gas samples collected through sampling pipes in the experiment show a great variety in composition (Lydmark 2011), similar to what is observed in the gas analyses of the FEBEX experiment. This reveals once more the difficulty in obtaining reliable gas samples. In particular, higher O_2 levels were measured in the backfill in 2010 than in 2009, which is explained by air contamination during sampling.

In samples that did not show any significant air contamination, the O_2 level in the gas phase had decreased to about 2 % from the atmospheric values, and N_2 had increased to about 98 %.The concentration of other gases remained low.

4.2 Microbiological Analysis

Altogether, the concentration of microorganisms (measured as ATP) was found to be 2 to 5 times higher in the prototype backfill and buffer than in the natural groundwater around the prototype.

The number of aerobic bacteria follows the measured O_2 concentrations in the pore space, and tends to be higher in the gallery backfill than in the bentonite buffer around the heaters. The higher aerobic microorganism concentration was found close to the tunnel wall (Lydmark 2011). This distribution could be the consequence of O_2 seepage from the access tunnel through the EDZ, or through the instrumentation and sampling ports. The analyses after excavation showed very low concentrations of aerobic microorganisms in the bentonite buffer in the deposition holes.

The overall concentration in the pore water remained low in the samples extracted from the closed prototype. After excavation, on the other hand, the presence of viable sulphate reducing bacteria was detected close to the copper canisters, indicating the absence of oxygen in this area. Among the anaerobic bacteria, sulphate reducing bacteria were found to be more abundant than iron reducing bacteria (op. cit.).

4.3 Summary of the Äspö Experiment

The results from the prototype experiment in Äspö show us that large variations are observed in gas concentrations and microbiological analyses in the various parts of the experiment. This high variability is a consequence of the heterogeneity of the system, of the host rock, and of O_2 ingress from the outside of the experiment. A similar heterogeneity is likely to occur in the FEBEX Phase 2.

5 Summary

5.1 Redox Conditions

The sources of information for estimating the redox conditions prevailing in the FEBEX experiment Phase 2 analyses of water and solids done after the dismantling of Heater 1, as well as gas and water analyses completed during both Phases. Additionally, some information can be gained by studying the design of the concrete plug used in the two phases and the likelihood of O_2 ingress from the access gallery into the experiment.

The difficulty in assessing the redox conditions in general is due to the very high sensitivity of the redox potential to the contamination by atmosphere. Therefore, all sampling procedures that do not take into account the redox conditions severely limit the interpretability of the data, particularly if the samples have been in contact with the atmosphere. Secondly, even when the sampling procedure aims at hindering contact with the atmosphere, air contamination is difficult to exclude, and is also difficult to take into account when interpreting the analytical results.

The expected theoretical behaviour in a totally sealed experiment would be a change of redox potential towards strongly reducing conditions within a few weeks or months after the closure of the experiment. In FEBEX, as well as in other prototype experiments, this theoretical trend is disturbed mostly by the potential inflow of O_2 into the buffer through the plug, along cables and pipes, or through the EDZ.

From the analyses of Phase 1, it can be asserted that oxygen levels decreased rapidly after the closure of the experiment. The gas samples collected at regular interval show however a measureable concentration of O_2. The low measured concentrations can indicate the presence of O_2 in the experiment, but can also be the consequence of a small air contamination of the samples upon sampling or sample handling, and are therefore not considered as conclusive. The analysis of the bentonite and the bentonite pore water show mixed results. As the pore water seems to be mostly in a reduced state (predominance of Fe^{+2} in the total iron), a decrease of reduced species in favour of more oxidised species is observed in the solid phase as compared to a reference sample of FEBEX bentonite. The combined study of the corrosion of metallic components and of the microbiological activity in the bentonite showed a strong influence, respectively presence, of SRB (anaerobic) in the parts of the experiment where substantial corrosion had been observed. However, in the same samples, aerobic microorganisms were also detected, in similar or even greater counts. One should note that in some cases anaerobic organisms like the SRB can proliferate in aerobic conditions when protected under a biofilm of aerobic microorganisms and so both would be detected in subsequent analyses.

The gas analyses performed during Phase 2 of the experiment all show similar, low O_2 concentrations, even before the emplacement of the gas-tight membrane. However, the variability of the data during Phase 2 is even greater observed in Phase 1. This is likely attributable to air contamination before, during, or after sampling. Unfortunately, a gap of several years in the sampling of the gas in Phase 2 makes the interpretation of the data in the later stages of the experiment problematic. The only redox potential analysis performed on a water sample during Phase 2 showed a strongly reducing value of -284 mV.

The study of the plug design shows that special care has been taken to make the plug water- and gas-tight during Phase 2. However, while this may reduce the risk of air contamination through the plug, it does not allow us to exclude air contamination from the access gallery, whether through defects in the plug sealing, the EDZ, or the cable and pipe seals.

The available data do not allow ascertaining the exact redox conditions in the FEBEX experiment before dismantling of Phase 2. However, the low concentrations of oxygen in the gas phase, together with the presence of anaerobic bacteria in some parts of the bentonite and a predominance of Fe^{+2} in the total iron of the bentonite pore water bring us to the conclusion that the FEBEX experiment tends towards an anoxic, reducing, environment. There are, however likely some heterogeneities in the system due to possible inflow of O_2 through the plug, along the sensor cables and pipes, or even through the EDZ.

5.2 Recommendations for Dismantling

If we assume that the conditions are close to fully anoxic in the experiment, or at least in strong disequilibrium with the access gallery, special care should be taken in order to minimize the disturbance of the system and the preservation of the samples.

Before starting the dismantling, gas and water samples should be collected from the existing sampling tubes, and the gas pore pressure measured. These would be the last samples available before the disturbance induced by the dismantling operations.

One of the first dismantling steps will consist of sampling concrete and bentonite by core drilling through the plug. Drilling into bentonite implies air drilling instead of wet drilling. Keeping anoxic conditions during air drilling is not currently within the scope of this project. However, the contamination of the solid samples by O_2 can be limited if the samples are packed under vacuum or in an inert gas phase immediately after sampling.

The preservation of the in-situ redox conditions is critical for the study of corrosion products. Therefore, when possible, the bentonite blocks hosting the corrosion coupons should be extracted as much as possible as one piece and packed as described above. This method would allow extracting the coupons in a controlled environment (glove box), without exposing them to air. Unfortunately this will not be possible for other metallic parts like large sensors or the heater and liner, which did provide valuable corrosion data in Phase 1 of the experiment.

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