

# **International Progress Report**

**IPR-04-26**

## **Äspö Hard Rock Laboratory**

### **Prototype Repository**

#### **Chemical measurements in buffer and backfill; sampling and analyses of gases**

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June 2004

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*Keywords:* Prototype Repository, microorganisms, microbial processes, gas, buffer, groundwater, oxygen

This report concerns a study which was conducted for SKB. The conclusions and viewpoints presented in the report are those of the author(s) and do not necessarily coincide with those of the client.



# **PROTOTYPE REPOSITORY**

## **Deliverable D 24**

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# Abstract

The Prototype Repository project is a full-scale model of the planned deep repository for Swedish spent nuclear fuel. A monitoring programme has been planned (IPR-99-34) with the aim to establish microbial activities and chemical conditions in the different parts of the Prototype Repository. This will allow:

- following the function of the Prototype Repository, and
- understanding the processes occurring well enough to be able to predict reliably the performance of a future nuclear repository.

One of the specific aims will be to monitor the consumption of O<sub>2</sub> in the Prototype Repository. Oxygen is hypothesized to be consumed by certain bacteria within a relatively short geologic time span (i.e. years), as opposed to longer time spans predicted by abiotic processes.

A method for taking samples of gases in buffer and backfill and analysing them has been tested *in situ* in the Prototype Repository. The gases have been sampled in evacuated glass bottles. An extractor system boiled off dissolved gas which was transferred into a syringe for measurement of volume. Then, the gas was analysed by direct injection into an automatic sampling loop installed on two different gas chromatographs. Dilution of the gas could be performed if necessary. Analysis was preformed for the following gases hydrogen, carbon mono-oxide, carbon dioxide, methane, and C<sub>2</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>4</sub> and oxygen.

A good correlation was observed between methane and carbon dioxide. The oxygen concentrations grouped in three intervals ranging from below and close to detection, around 1 % and close to atmospheric concentrations. There was a reverse correlation between oxygen and the groundwater gas methane. This indicates that groundwater gases release from the surrounding groundwater and pushes away the tunnel air that was closed in during the installation process. Alternatively, it is also possible that biogeochemical processes in the Prototype Repository already locally consume oxygen.

The data indicate that there is a significant gas exchange going on between the prototype repository and the surrounding tunnels. Such a process may occur if there are fluctuating pressure differences between the inside and outside of the Prototype Repository. Simply put, the prototype may be breathing! Consequently, the disappearance of oxygen, as shown by the data is not due to microbial processes. This is reasonable, because microorganisms need water to be metabolically active and consume oxygen. As the repository still is very dry, such processes have yet not started. The oxygen is presently being exchanged with groundwater gas.

# Sammanfattning

Prototypförvaret simulerar i fullstor skala en del av ett framtida djupförvar för använt kärnbränsle. Ett övervakningssystem har planerats (IPR-99-34) och installerats för att bestämma mikrobiologiska aktiviteter och kemiska förhållanden i olika delar av Prototypförvaret. Detta system möjliggör:

- Löpande uppföljning av Prototypförvarets funktion
- Inhämtande av tillräcklig förståelse av processer som sker för att med stor noggrannhet kunna förutsäga ett framtida djupförvars utveckling

Ett speciellt syfte är att studera förbrukningen av syre i Prototypförvaret. Syre antas förbrukas av vissa bakterier inom en i geologiska sammanhang relativt kort tidsperiod (några år), jämfört med långa tidsperioder, som förutspås med abiotiska processer.

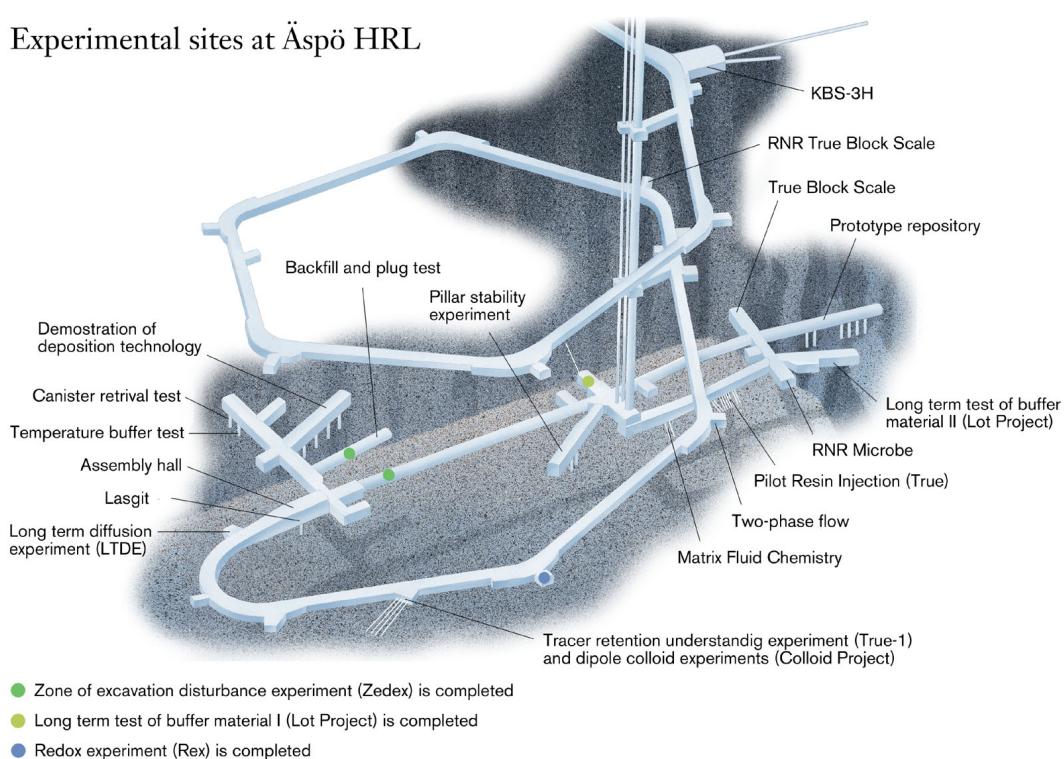
En metod för att ta prover av gaser i buffert och återfyll samt analysera dem har provats *in situ* i Prototypförvaret. Gaserna har provtagits med evakuerade glasflaskor. Ett extraktionssystem kokade av gasen som överfördes till en spruta för mätning av volymen. Sedan analyserades gasen genom direkt injicering i en automatisk provhanterare, som installerats på två olika gaskromatografer. Gasen kan spädas ut, om så erfordras. Analyser gjordes med avseende på följande gaser: väte, CO, CO<sub>2</sub>, CH<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>4</sub> och syre.

En god korrelation erhölls mellan CH<sub>4</sub> och CO<sub>2</sub>. Syrehalterna grupperades i tre kategorier: noll till detektionsnivå, omkring 1% samt upp till atmosfärhalt. En omvänt korrelation erhölls mellan syre och CH<sub>4</sub> i grundvatten. Detta indikerar att gas i grundvattnet frigörs och trycker bort luft i tunneln, vilket blev instängt i samband med installationen. Alternativt är det också möjligt att biokemiska processer i Prototypförvaret redan förbrukar syre lokalt.

Data indikerar att ett betydande byte av gas pågår mellan Prototypförvaret och omgivande tunnlar. En sådan process kan förekomma om varierande tryckdifferenser finns mellan Prototypförvarets insida och utsida. I praktiken betyder detta att Prototypförvaret ”andas”! En följd av detta skulle vara att det ej är mikrober som orsakar den sjunkande halten syre, vilket synes rimligt med hänsyn till att mikroorganismer behöver vatten för att bli metaboliskt aktiva och förbruka syre. Då Prototypförvaret fortfarande är torrt, har sådana processer ej startat ännu. Syre håller för närvarande på att bytas ut mot gas i grundvattnet.

# 1 Introduction

The Prototype Repository project is an international full scale model project of the planned deep repository of Swedish nuclear fuel. It is conducted at the Äspö Hard Rock Laboratory, HRL, in crystalline rock at approximately 450 m depth. Figure 1-1 gives an overview of Äspö HRL and the location of the Prototype Repository.



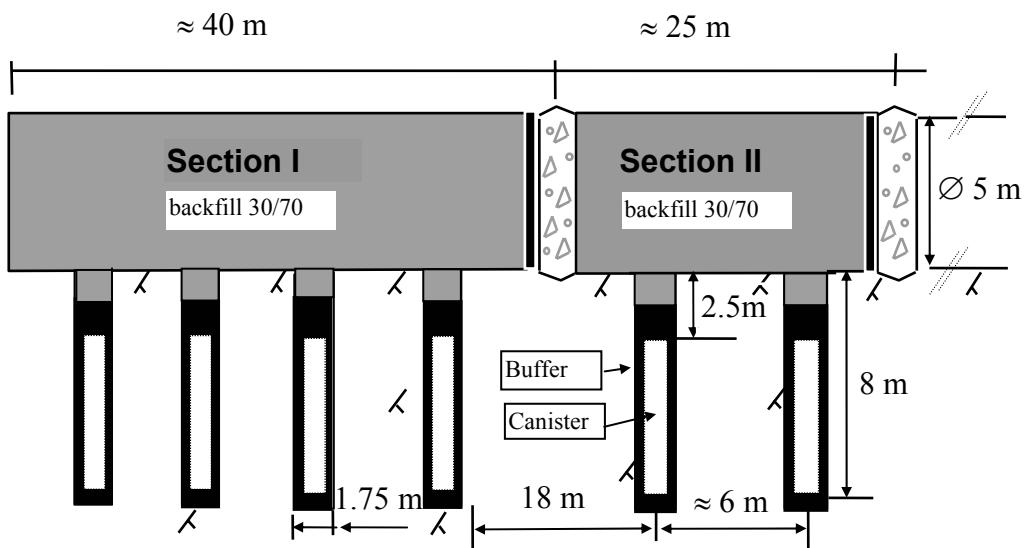
**Figure 1-1.** Schematic view showing the location of the Prototype Repository (lower left of the figure) in Äspö HRL.

A monitoring programme was planned (IPR-99-34), with the purpose of investigating the effect of microbial activity on the gas composition in different parts of the Prototype Repository. One of the specific aims will be to monitor the microbial consumption of O<sub>2</sub>. Oxygen is hypothesized to be consumed by bacteria within a much shorter time span (i.e. weeks to years), as opposed to longer time spans predicted by abiotic processes.

Sampling and analysis of gases on a regular basis is one of the activities that will be performed in order to monitor biogeochemical processes that take place in the Prototype Repository. A method for sampling and analysing gases in buffer and backfill has been tested *in situ* (AP TD F63P1-04-012). The results and evaluation from the *in situ* measurements are presented in this report.

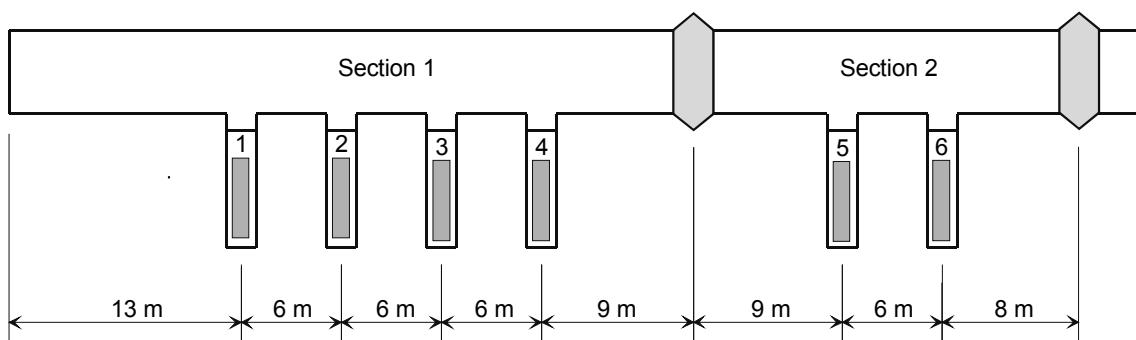
## 1.1 Design of the Prototype Repository

The Prototype Repository has six deposition holes in full scale distributed in two sections as shown in Figure 1-2. The inner section contains four deposition holes that will be left for 20 years, while the outer section contains two boreholes which will be decommissioned after about 5 years. A full size canister with electrical heaters surrounded by bentonite has been placed in each deposition hole.



**Figure 1-2.** Schematic view of the Prototype Repository. From IPR-99-34.

## 1.2 Sampling points and sample collectors



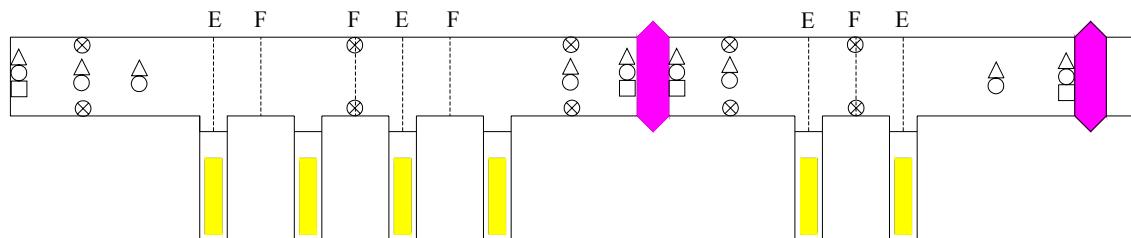
**Figure 1-3.** The deposition holes in the Prototype Repository. From IPR-04-13

The instrumented deposition holes in Section 1 are denoted DA3587G01 and DA3575G01, hole number 1 and 3 respectively according to Figure 1-3. Eight sample collectors have been installed (AP TD F63-02-054) for continuous hydrochemical sampling in Section 1 (Table 1-1); six in the backfill (Figure 1-4), one in top of deposition hole DA3587G01 and one in the top of deposition hole DA3575G01.

**Table 1-1. Sample collectors in Section 1.**

Deposition hole/backfill	Mark	Block/section
Backfill	KBU10001	Inner part
Backfill	KFU10002	Inner part
Backfill	KBU10003	Between dep. hole 2 and 3
Backfill	KBU10004	Between dep. hole 2 and 3
Backfill	KBU10005	In front of plug
Backfill	KBU10006	In front of plug
DA3575G01	KBU10008	C4 (hole 3)
DA3587G01	KBU10007	C4 (hole 1)

- ⊗ hydrochemical sampling
- pore water pressure + temp
- total pressure + temp
- ×
- △ relative humidity + temp
- E, F measuring sections



**Figure 1-4.** Figure showing the IPR-02-03.

The instrumented deposition holes in Section 2 are termed DA3545G01 and DA3551G01, hole number 5 and 6 respectively according to Figure 1-3. For section 2 (AP TD-F63-02-040) (Table 1-2) four sample collectors are placed in the backfill (Figure 1-4), two in the interface rock/bentonite at the top of deposition hole DA3545G01 and two in the interface rock/bentonite at the top of deposition hole DA3551G01.

**Table 1-2. Sample collectors in section 2.**

Deposition hole/backfill	Mark	Block/section
Backfill	KFA01	Inner part
Backfill	KFA02	Inner part
Backfill	KFA03	Between dep. hole 5 and 6
Backfill	KFA04	Between dep. hole 5 and 6
DA3545G01	KB613	C4 (hole 5)
DA3545G01	KB614	C4 (hole 5)
DA3551G01	KB513	C4 (hole 6)
DA3551G01	KB514	C4 (hole 6)

The exact positions (coordinates) of the titanium cups in the buffer and backfill is given in AP TD F63-01-054 and AP TD-F63-02-040.

The sample collectors consists of a titanium cup with a titanium filter on their top and with tubes (made of PEEK) connected to the bottom of the cups. The length of each tube is approximately 79 m and the inner diameter is 2 mm.

## 2 Methods

### 2.1 Evacuation and sampling

Junctions with a valve and stopcock have been mounted on the PEEK tube to all sample collectors. Prior to this operation all PEEK tubes stood open to the tunnel atmosphere. The standing gas volume in the PEEK tubes was evacuated with a vacuum pump before sampling. This was done to ensure that the gas sample was representative for the interior of the Prototype Repository. The time for the evacuation was kept at about 15 minutes, which was enough to evacuate the tubes. The evacuation was done via a closed glass bottle to avoid that water reached the vacuum pump. Some sample collectors already delivered water, which could be registered in the glass bottle. The volume water extracted was measured with a graduated measuring cylinder. When the evacuation cycle was completed, the stopcock was closed and the vacuum pump was switched off.

Figure 2-1 shows the equipment setup for sampling. Before sampling, a needle was mounted on the valve. The needle was inserted through the butyl rubber membrane of a 120 mL serum bottle. The stopcock was opened so that gas and water could pass. The sample bottle had been evacuated and flushed with nitrogen in three cycles and left fully evacuated. As the bottles were under vacuum, gas would move from the Prototype Repository into the bottles. The bottles were left in contact with the respective Prototype Repository collector for 20 minutes. Subsequently, the stopcock was closed and the needle removed from the bottle.



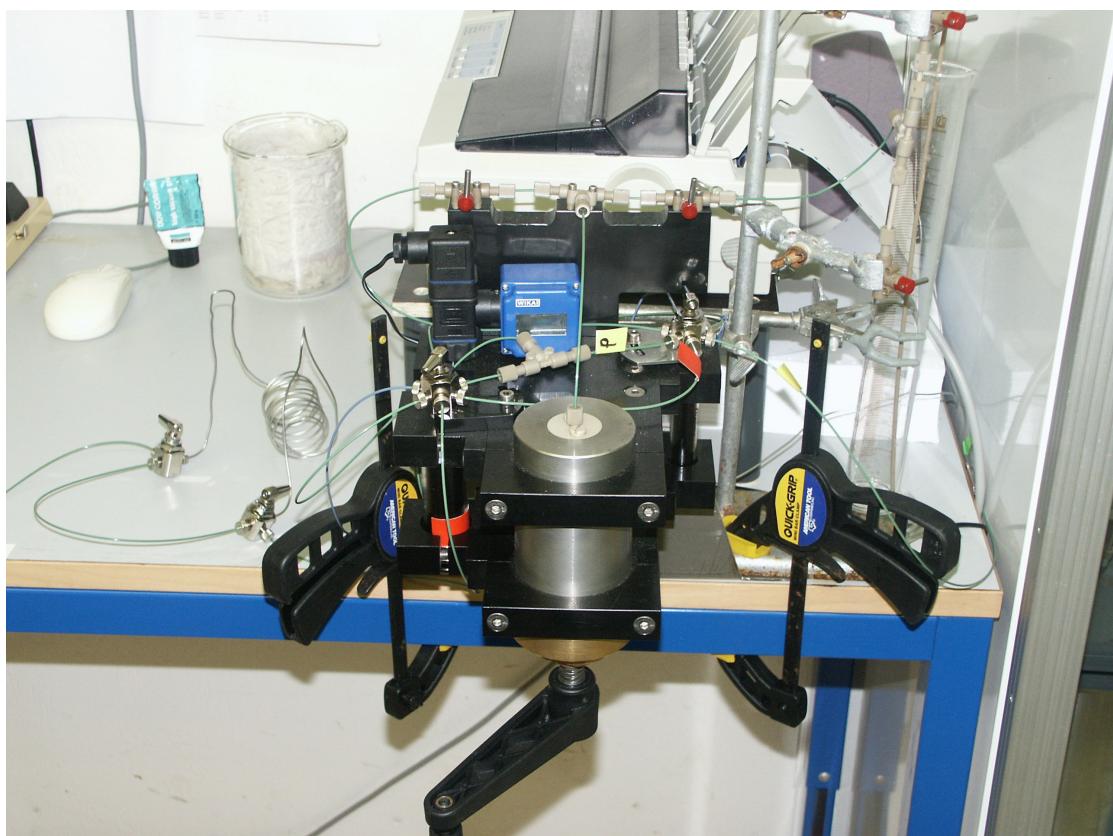
**Figure 2-1.** The sampling equipment. From left to right: junctions and valve with stopcock, glass flask, vacuum pump.

Sampling was done at two different occasions. The first sampling was done to develop and adjust the sampling methodology and technique. Three samples were collected during the 18<sup>th</sup> February 2004 and analysed the following day at the site, as described under 2.2.1.

A second, full sampling round was made 3<sup>rd</sup> March 2004. All sample collectors except one was successfully sampled. The samples were brought to the laboratory at Gothenburg University and analysed as described under 2.2.2.

## 2.2 Analytical procedures

The extraction of gas from the bottles and the measurement of the extracted volume of gas were done using equipment specially built and designed for analysis of gas in groundwater and the prototype. We have access to two similar tools. One is based underground, at the MICROBE laboratory in a tunnel next to the Prototype Repository tunnel (Figure 2-2). It has a 200 mL extraction volume. The second extractor is based at the laboratory at Gothenburg University and has an extraction volume of 500 mL.



**Figure 2-2.** Gas extractor for field analysis of gas content in deep groundwater at the 450 m level underground at Åspö Hard Rock Laboratory. The crank moves a piston up and down in the cylinder, creating the vacuum needed to extract the gas. The extracted gas volume is measured at a given pressure (blue gauge), possibly diluted and injected into the gas chromatograph sampling loop via a freeze trap for moisture with CO<sub>2</sub> pellets/ethanol or liquid nitrogen.

The extractor system boils off dissolved gas and transfers the extracted gas into a syringe for measurement of volume. Then, the gas is analysed by direct injection into an automatic sampling loop installed on the respective GC. Dilution of the gas can be performed if necessary. The unique features with this extraction system is that the sample is transferred through the extraction and analysis system in one isolated line, there is no possibility for oxygen or carbon dioxide contamination from the laboratory air.

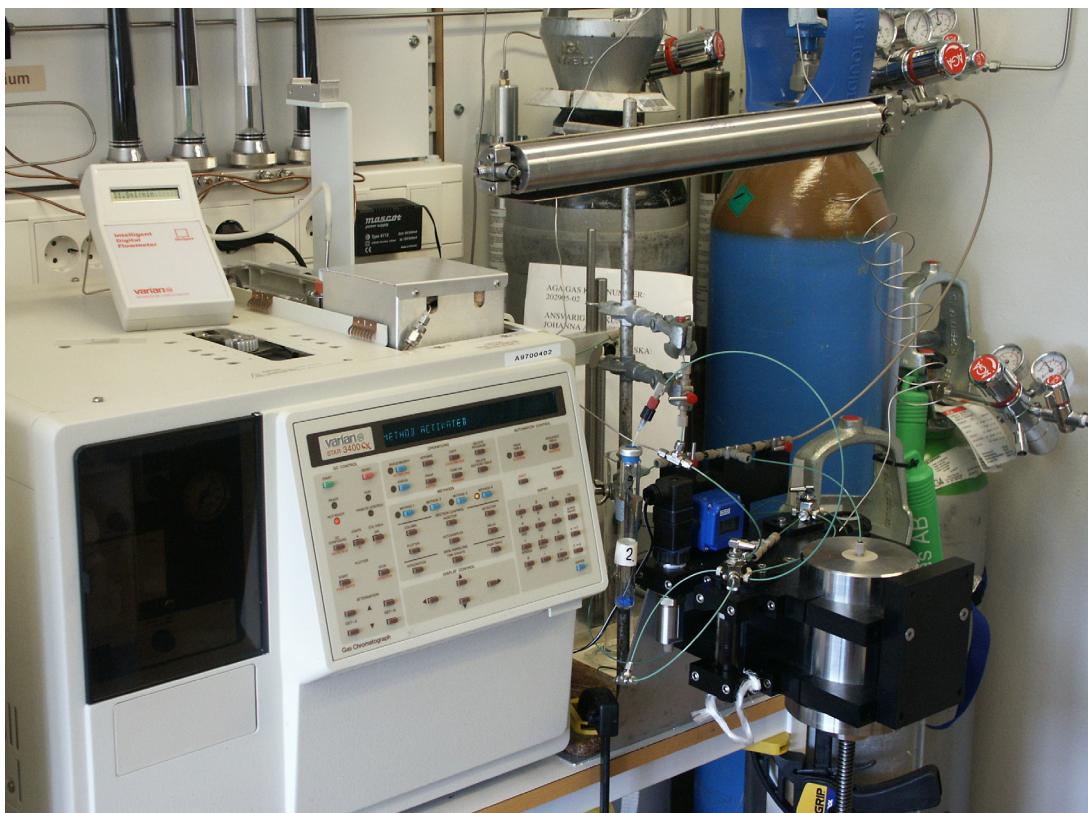
### **2.2.1 KAPPA-V gaschromatograph at the MICROBE site**

A first analysis of gases was done the day after sampling 18<sup>th</sup> February at the MICROBE site, 450 m underground. This laboratory is situated at the same level as the Prototype Repository is. A gas chromatograph (GC) named KAPPA-5 and a gas extraction system (Figure 2-2) have been installed. The chromatograph is equipped with a Flame Ionization Detector (FID) for carbon dioxide, methane, ethane and ethylene and a Reductive Mercury Detector (RMD) for hydrogen and carbon mono-oxide. Haysep and Molsikt columns are installed with a methanizer for carbon dioxide analysis in line. This system can analyse the gases with the following detection limits: hydrogen 1 ppb, carbon mono-oxide 1 ppb, carbon dioxide 1 ppm, methane 1 ppm, ethane 1 ppm and ethylene 1 ppm.

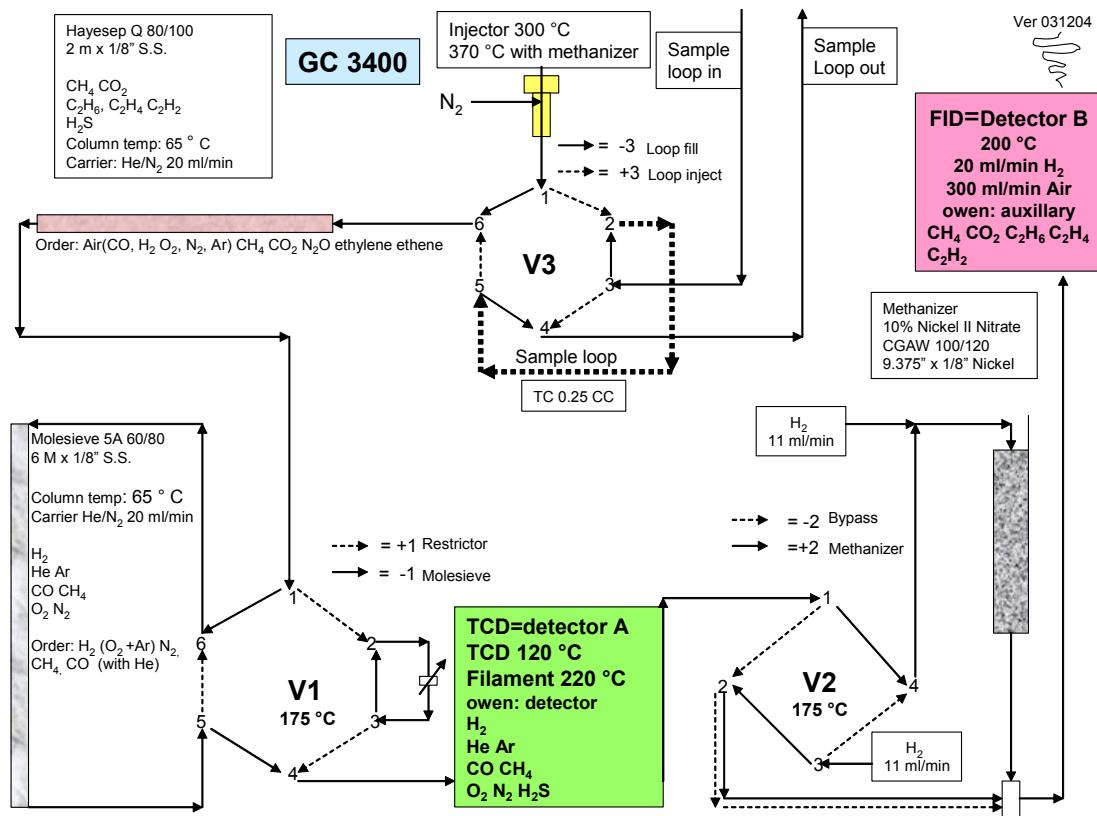
Samples from three collectors were analysed in triplicate. Extraction was first made via the extractor (Figure 2-2). Thereafter, the extracted gas sample was diluted with nitrogen before injection via a sample loop in the GC.

### **2.2.2 VARIAN 3400 gas chromatograph at Gothenburg University**

A second run of analyses was performed the weeks after the second sampling campaign the 3<sup>rd</sup> of March. Samples were taken out as described in section 2.1 and transported to the laboratory in Gothenburg. They were analysed on a Varian 3400 GC equipped with a flame ionisation detector (FID). A thermal conductivity detector (TCD) was used for the analysis with nitrogen, helium or argon as carrier gas (Figure 2-3). Haysep and Molsikt columns are installed with a methanizer for carbon dioxide analysis in line. Analysis can also be executed for hydrogen, helium, argon, carbon mono-oxide, methane, and C<sub>2</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>3</sub>H<sub>8</sub>, C<sub>3</sub>H<sub>6</sub>, H<sub>2</sub>S and acetylene with nitrogen as carrier. Nitrogen, oxygen and argon are analysed with helium as carrier, Argon and oxygen can be analysed with helium as carrier. The respective column and detectors in use is shown in Figure 2-4. Here, we analysed for the gases carbon mono-oxide, carbon dioxide, methane C<sub>2</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>4</sub>, and oxygen.



**Figure 2-3.** The Varian 3400 GC with the 500 mL extraction unit.



**Figure 2-4** Schematic view of the Varian 3400 configuration.

### 3 Results and evaluation

Only two collectors delivered water at this time. They were KBU10002 and KBU10008. This resulted in too little extracted gas for the oxygen analysis. In the future, more parallels must be sampled to avoid this problem.

#### 3.1 Analysis at the MICROBE site

All gases analysed for were present except for C<sub>2</sub>H<sub>4</sub> (Table 3-1). There were high amounts of groundwater gases in the sample from the inner buffer section of section 1. There were also significant traces of groundwater gases in the other two samples. The reproducibility of the extraction and analysis procedure was generally very good. Hydrogen, methane in high concentration and particularly the presence of C<sub>2</sub>H<sub>6</sub> and C<sub>2</sub>H<sub>4</sub> are all indicative of groundwater gases. Those gases occur only in low concentrations or not at all in the tunnel atmosphere. The analysis at the MICORBE site went very well. This procedure will be particularly useful when time series or other types of analyses are to be performed, where a rapid analysis result is needed for the understanding of how an experiment should be developed.

Table 3-1 Analysis results of gas extracted from the Prototype Repository 18<sup>th</sup> February 2004. Each measurement is mean of extractions from three independent bottles.

Sample code	Position of collector	H <sub>2</sub> ppm	CO ppm	CH <sub>4</sub> ppm	CO <sub>2</sub> ppm	C <sub>2</sub> H <sub>4</sub> ppm	C <sub>2</sub> H <sub>6</sub> ppm
KBU10001	1 buffer inner	2.32±0.54*	0.52±0.06	4765±313	3147±178	0±0	1.95±0.30
KBU10003	1 buffer 2-3	1.87±0.55	0.36±0.11	43.1±17	442±15.7	0±0	0±0
KBU10008	1 C4 buffer	0.24±0.13	0.073±0.036	34.0±7.09	557.6±108	0±0	0±0

\* Standard deviation of three samples

#### 3.2 Analysis of samples at the laboratory in Gothenburg

The analysis results are represented in Table 3-2. The reproducibility between the repeated extractions was very good (a and b), except for KFA02. There was a span in oxygen from close to atmospheric concentration to concentrations below the detection limit which at present is about 0.1 %. There was also a very large variation in the concentrations of all other gases analysed, depending of analysed section.

All section 2 buffer samples showed very low oxygen concentrations and they also had relatively high concentrations of groundwater gases. This implies that gas is released from the surrounding groundwater and reaches the Prototype Repository. Hole no. 3 already seems to receive water. With some variation, there were high concentrations of oxygen in the backfill.

The highest methane concentration was found in front of the inner plug. This could indicate a groundwater flow close to the plug that releases its gas due to the pressure decrease it meets. This assumption can be tested against the pressure measurements when reported.

**Table 3-2 Results from analysis of gas from the Prototype Repository sampled the 3<sup>rd</sup> March. Letters a and b represents independent extractions and injections from the respective sample bottle.**

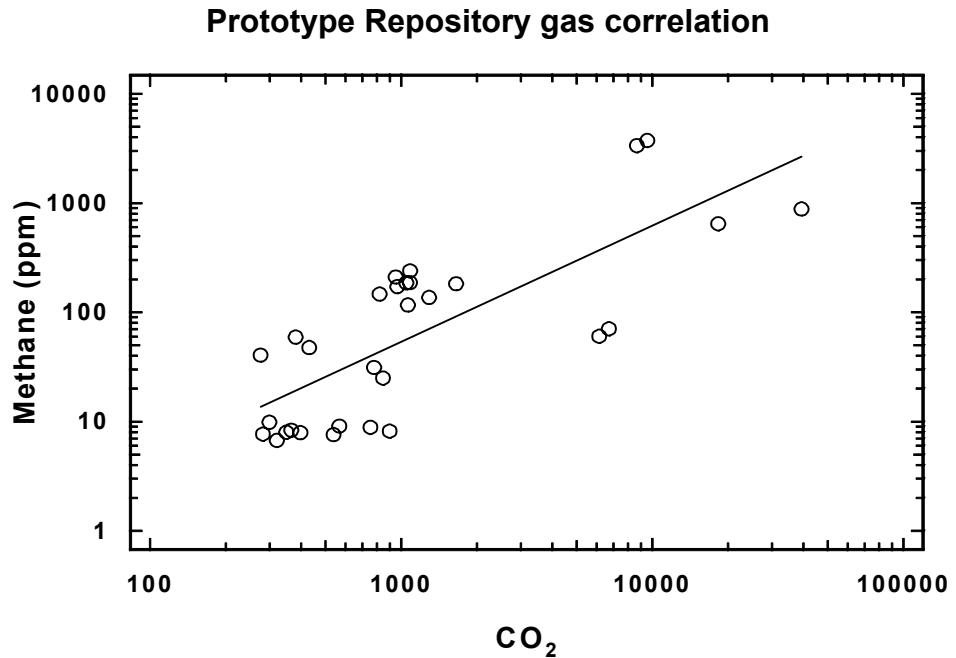
Sample code	Position of collector 1 and 2 = sections	CO ppm	CH <sub>4</sub> ppm	CO <sub>2</sub> ppm	C <sub>2</sub> H <sub>6</sub> ppm	O <sub>2</sub> %
KBU10001-a	1 backfill inner part	143,6	9,9	298,5	0,00	17,16
KBU10001-b	1 backfill inner part	120,4	8,0	348,9	0,00	17,60
KBU10002-a	1 backfill inner part	0,0	60,5	6142,0	0,00	n.a.
KBU10002-b	1 backfill inner part	11,8	70,7	6714,3	0,00	n.a.
KBU10003-a	1 backfill between holes 2-3	131,3	7,6	537,6	0,00	17,01
KBU10003-b	1 backfill between holes 2-3	127,9	9,1	567,0	0,00	18,94
KBU10004-a	1 backfill between holes 2-3	193,3	31,3	779,1	0,00	12,38
KBU10004-b	1 backfill between holes 2-3	186,4	25,1	845,8	0,00	16,61
KBU10005-a	1 backfill in front of plug	136,1	6,7	319,6	0,00	13,88
KBU10005-b	1 backfill in front of plug	130,9	8,3	364,8	0,00	17,13
KBU10006-a	1 backfill in front of plug	4,6	3359,5	8673,5	3,85	0,00
KBU10006-b	1 backfill in front of plug	5,5	3747,8	9546,3	3,50	0,41
KBU10007-a	1 C4 buffer, hole 1	123,3	8,9	753,9	0,00	15,81
KBU10007-b	1 C4 buffer, hole 1	138,2	8,2	899,2	0,00	18,39
KBU10008-a	1 C4 buffer, hole 3	57,6	647,1	18282,0	0,00	n.a.
KBU10008-b	1 C4 buffer, hole 3	104,4	883,2	39263,3	0,00	n.a.
KFA01-a	2 backfill inner part	104,2	7,7	281,3	0,00	15,09
KFA01-b	2 backfill inner part	123,3	7,9	396,9	0,00	18,42
KFA02-a	2 backfill inner part	74,3	47,6	430,1	0,00	11,61
KFA02-b	2 backfill inner part	262,8	136,8	1291,2	0,00	13,30
KFA03-a	2 backfill between holes 5-6	11,2	210,6	950,0	0,00	0,00
KFA03-b	2 backfill between holes 5-6	7,0	239,8	1084,7	0,00	1,04
KFA04-a	2 backfill between holes 5-6	120,6	40,6	275,3	0,00	16,56
KFA04-b	2 backfill between holes 5-6	193,9	59,4	379,9	0,00	18,85
KB513-a	2 C4 buffer hole 6	7,8	186,9	1083,4	0,00	0,00
KB513-b	2 C4 buffer hole 6	10,3	186,1	1047,9	2,75	0,64
KB514-a	2 C4 buffer hole 6	4,5	116,8	1063,2	0,00	0,00
KB514-b	2 C4 buffer hole 6	13,0	182,8	1653,9	0,00	1,37
KB613-a	2 C4 buffer hole 5	7,8	147,0	820,0	2,88	0,00
KB613-b	2 C4 buffer hole 5	8,3	171,9	964,3	0,00	0,79
KB614-a	2 C4 buffer hole 5	n.s.	n.s.	n.s.	n.s.	n.s.
KB614-b	2 C4 buffer hole 5	n.s.	n.s.	n.s.	n.s.	n.s.

n.a. not analysed, n.s. not sampled

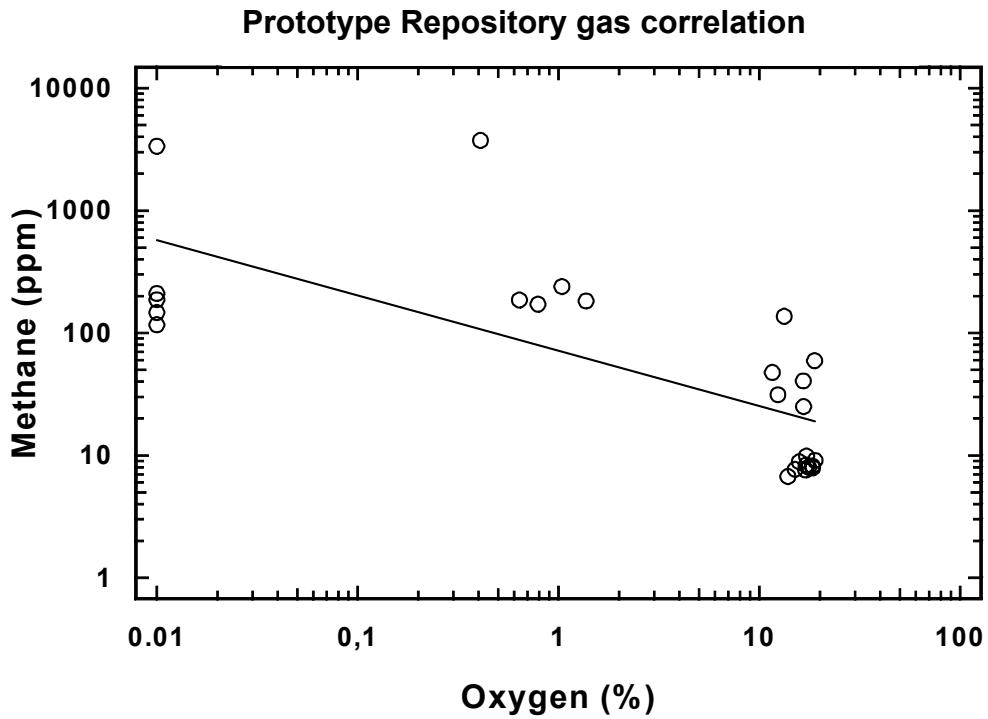
### 3.3 Evaluation of the results

In general, we have now a successful protocol for how to sample, extract and analyse gas in the Prototype Repository. However, the prototype has not yet been put under formation groundwater pressure, and some exchange of the gases inside the Prototype Repository with the tunnel environment cannot be excluded. It will, therefore, be very important to repeat this sampling and analysis just before closing of all connections between the tunnel and the Prototype Repository. That will be the baseline for future analysis and evaluation.

Already now, some compelling correlations between the analysed gases were observed. First, carbon dioxide and methane tend to correlate in analyses of groundwater gases (Pedersen, 2001, 2002). This was also observed for the gases analysed in the Prototype Repository. A good correlation was observed between methane and carbon dioxide (Figure 3-1).



**Figure 3-1.** Correlation between gas data from Table 3-2.

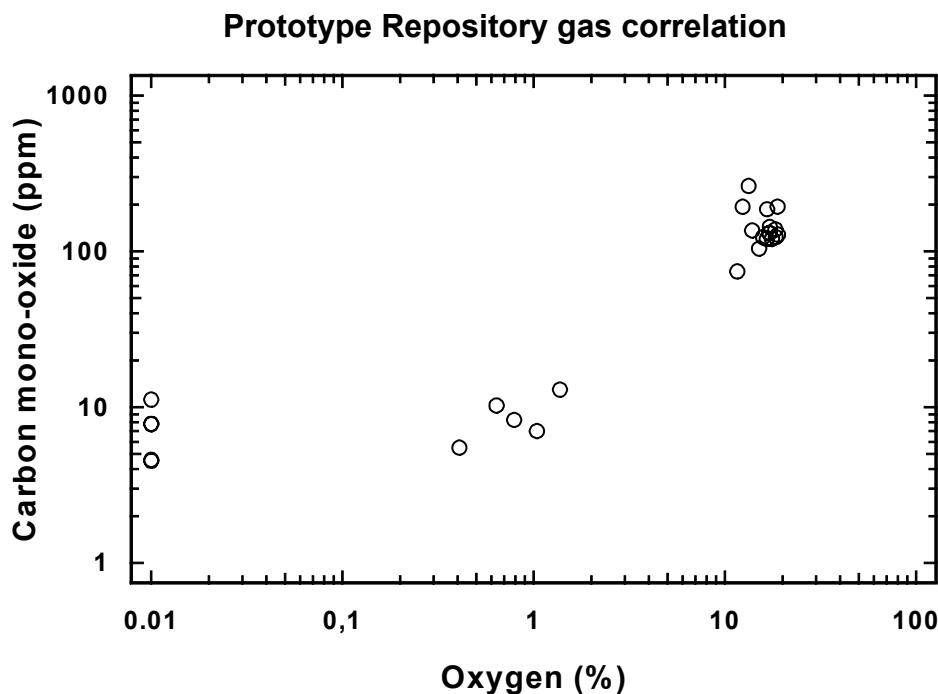


**Figure 3-2.** Correlation between gas data from Table 3-2.

The oxygen concentrations grouped in three intervals ranging from below and close to detection, around 1 % and close to atmospheric concentrations (Figure 3-2). There was a reverse correlation between oxygen and the groundwater gas methane. This indicates that 1) groundwater gases release from the surrounding groundwater and pushes away the tunnel air that was closed in during the installation process. Or 2) that biogeochemical processes in the Prototype Repository already locally consume oxygen.

The carbon mono-oxide showed not linear correlation with oxygen, but all samples with a high oxygen concentration also had a high carbon mono-oxide concentration (Figure 3-3). Common values for this gas in groundwater are a couple of ppm. Most of the carbon mono-oxide in the Prototype Repository seems to have its origin from the tunnel air. The tunnel environment is contaminated with this gas from the diesel trucks and cars that frequently pass the prototype tunnels. In samples with a low oxygen concentration, most of the carbon mono-oxide is gone, indicating that there is a significant gas exchange going on between the Prototype Repository and the surrounding tunnels. Such a process may occur if there are fluctuating pressure differences between the inside and outside of the Prototype Repository. Simply put, the Prototype Repository is most probably breathing!

Consequently, the disappearance of oxygen, as shown in Figures 3-2 is not due to microbial processes. This is reasonable, because microorganisms need water to be metabolically active and consume oxygen. As the Prototype Repository still is very dry, such processes have yet not started. The oxygen is presently being exchanged with groundwater gas.



## **4      References**

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