

Measuring the diffusion coefficient for He in Opalinus Clay

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1 Introduction

Deep clay-rich sedimentary formations are currently being investigated as a potential host rock for the geological disposal of high-level and long-lived radioactive waste. In different countries hydrogeological and geochemical research on these clay layers has been performed and is still on-going.

In clay-rich formations, transport properties in pore water can be constrained from natural tracer profiles which can be considered as long-term and large-scale natural experiments. Information obtained from lab-scale experiments and from experiments in underground research laboratories is restricted to spatial and temporal scales of typically 0.01 m to 1 m and 1 year. In contrast, information provided by natural tracer profiles considers scales of tens to hundreds of meters and 0.1 to 1 Ma. This information is very important for underpinning the safety assessment of a deep geological repository as it is an important tool for upscaling from lab scale experimental results. Moreover, natural tracer profiles represent an independent evidence for system understanding. Natural tracer profiles have been determined for anions (Cl^- , Br^- , I^-), water isotopes ($\delta^{18}\text{O}$, $\delta^2\text{H}$) and noble gases (mainly He) (Mazurek et al., 2011).

As natural tracers are present in deep clay layers for millions of years, and diffusion is considered to be the dominant transport mechanism, it should be possible to model these natural tracer profiles by using diffusive models. For these models, reliable diffusion coefficients have to be used. Diffusion coefficients for anions and water isotopes are widely available. The availability of reliable diffusion coefficients for He is limited.

In Switzerland, the Opalinus Clay is considered as a potential host rock for the disposal of high-level and long-lived nuclear waste. Also for this low-permeability clay layer, natural tracer profiles are important for the safety assessment of a repository. One of these natural tracers is helium, but in order to model the profile of helium in the Opalinus Clay a precise value of the diffusion coefficient of helium is needed. In the literature, diffusion coefficients for He on Opalinus Clay have been reported by Gomez-Hernandez (2000) and Rubel et al. (2002). Gomez-Hernandez performed an in-situ in- and out-diffusion experiment with He parallel to the bedding plane on Opalinus Clay in the Mont Terri underground laboratory. The best fit for $D_{\text{eff}}(\text{He}) = 2.1 \times 10^{-10} \text{ m}^2/\text{s}$ was obtained for an out-diffusion experiment but a porosity of 30% was used, which is double the typical reported porosity values for Opalinus Clay. This can be explained by the fact that during the drilling of the experimental borehole an EDZ was created, leading to a higher permeability and increased porosity (Gomez-Hernandez, 2000). However, this porosity value has not been confirmed by other measurements and is thus an estimate. As the fit for D_{eff} is based on this estimated porosity, the precision of D_{eff} is questionable. The other out- and in-diffusion experiments performed by Gomez-Hernandez (2000) could not confirm this value for D_{eff} without adapting parameters like the circulation volume or the initial concentration ^4He .

Rubel and coworkers (Rubel et al., 2002) fitted D_{app} for He ($D_{\text{app}} = 3.5 \times 10^{-11} \text{ m}^2/\text{s}$) in Opalinus Clay based on the natural profile of helium obtained at Mont Terri, Switzerland. However, according to (Mazurek et al., 2011), this D_{app} value is likely too small because of the use of an overly simplified model – in particular, of the unsubstantiated assumption of a steady-state situation in which out-diffusion of He is balanced by in-situ production.

In (Mazurek et al., 2011), the diffusion coefficient for He was estimated from the diffusion coefficient of HTO. The pore volume and pore geometry accessible to He is considered to be similar to that for water and therefore the diffusion coefficient for HTO can be taken as proxy for that of He if an appropriate scaling factor is applied that accounts for the different molecular masses. This scaling factor was set to 3, which is the ratio of the diffusion coefficients of He and HTO in free water.

The absence of reliable measurements of the diffusion coefficient for He in Opalinus Clay has been recognised as a knowledge gap, and this was the motivation for the experiment reported here, targeted at the measurement of the diffusion coefficient for He using the apparatus and methodology developed at SCK•CEN.

2 Methodology

SCK•CEN has developed a versatile method to measure the diffusion coefficient of dissolved gases in low-permeability materials. The method is based on the through diffusion technique (Put and Henrion, 1988; Shackelford, 1991; Van Loon et al., 2003) with two dissolved gases placed on opposite sides of a test core (Figure 1). In a classical through-diffusion test, the porous medium is placed between 2 vessels, one containing a known concentration (high concentration compartment) of the diffusant while the other compartment is free of the diffusant (low concentration compartment). From the evolution of the diffusant concentration in both compartments, the diffusion characteristics of the diffusant can be obtained.

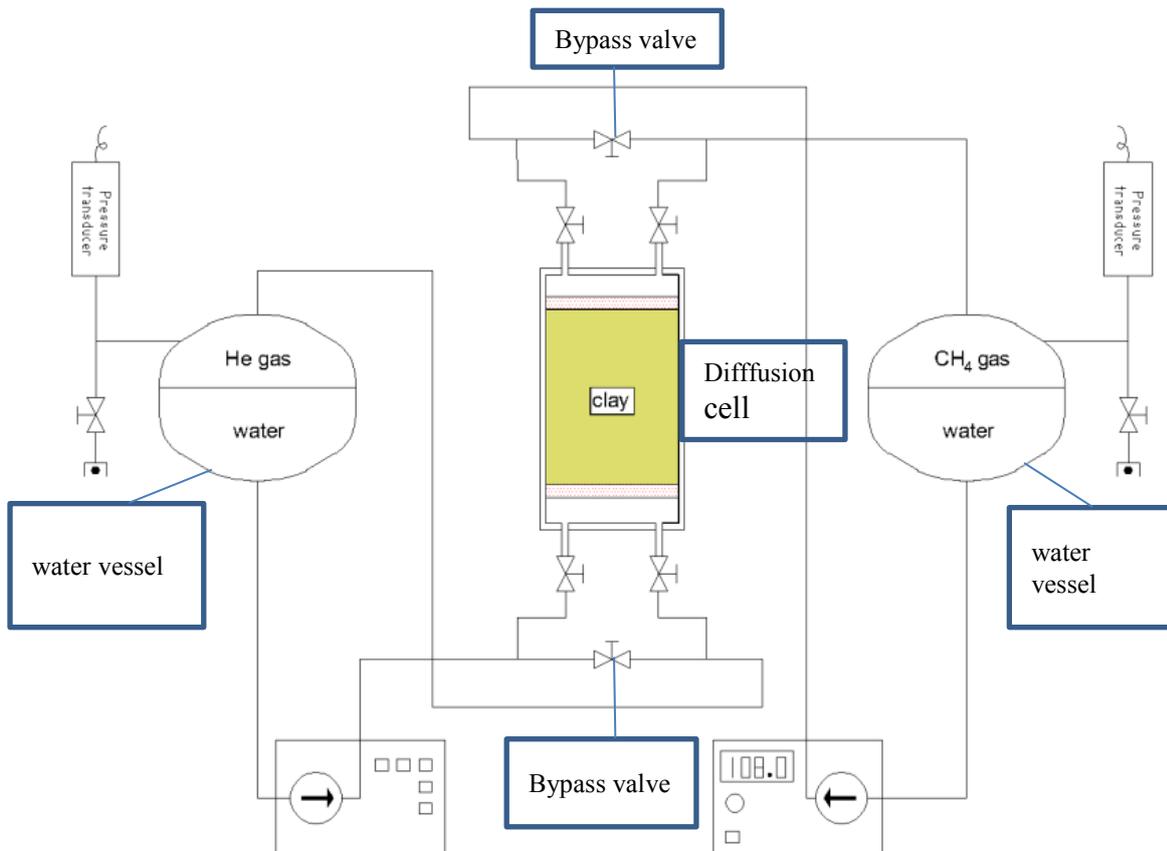


Figure 1: set-up to measure the diffusion coefficient of dissolved gas in low-permeability materials

The set-up consists of a diffusion cell, 2 water vessels with a volume of 1 liter, 2 pumps and 2 pressure transducers (Figure 1). During the selection of the different components, special attention was paid to the gas tightness. A more detailed description can be found in (Jacops et al., 2013).

Before starting the diffusion experiment, the diffusion cell has to be loaded with the sample. This diffusion cell consists of a cylindrical stainless steel body with an internal diameter of 90 mm. As the plasticity/swelling capacity of the Opalinus Clay is limited compared to Boom Clay, the Opalinus Clay sample has to be embedded in a resin in order to seal the interface clay sample – cell. First, the Opalinus Clay sample is processed on a lathe into a cylinder of 80 mm diameter and 35 mm height. Next, the diffusion cell is glued to a bottom flange. In this bottom flange, a cut-away of 2.5 mm deep is foreseen which allows to place the sample exactly in the middle of the diffusion cell. After the sample has been placed in the cell, the remaining void between the sample and the cell (thickness 5 mm) is filled with resin (Sikadur 52 Injection Normal). During the curing process, the cell is placed on a heating plate, set at 30°C. Due to the heating, polymerisation starts at the bottom and then propagates upwards. This should minimize cracking due to shrinkage of the resin. After 1 day, the resin has hardened and the cell is machined again on a lathe in order to remove the "disturbed" parts of the sample: the bottom flange and the lower 2.5 mm of the diffusion cell are removed, even as the upper 5 mm of the sample on top of the cell until a remaining height of 25 mm is obtained. The top- and bottom flanges (including a sintered stainless steel filter of 2 mm thickness, diameter 80 mm and porosity 40%) are welded to the diffusion cell. Both flanges are provided with a circulation loop to ensure good contact between the water containing the dissolved gas and the clay sample.

As a good sealing between the sample, the resin and the inner wall of the diffusion cell is essential for a correct interpretation of the data, this sealing is verified by performing an hydraulic conductivity (K) measurement. The obtained value also gives a good indication on the status of the sample: large cracks or other anomalies will influence K. A scheme of the used set-up is given in Figure 2.

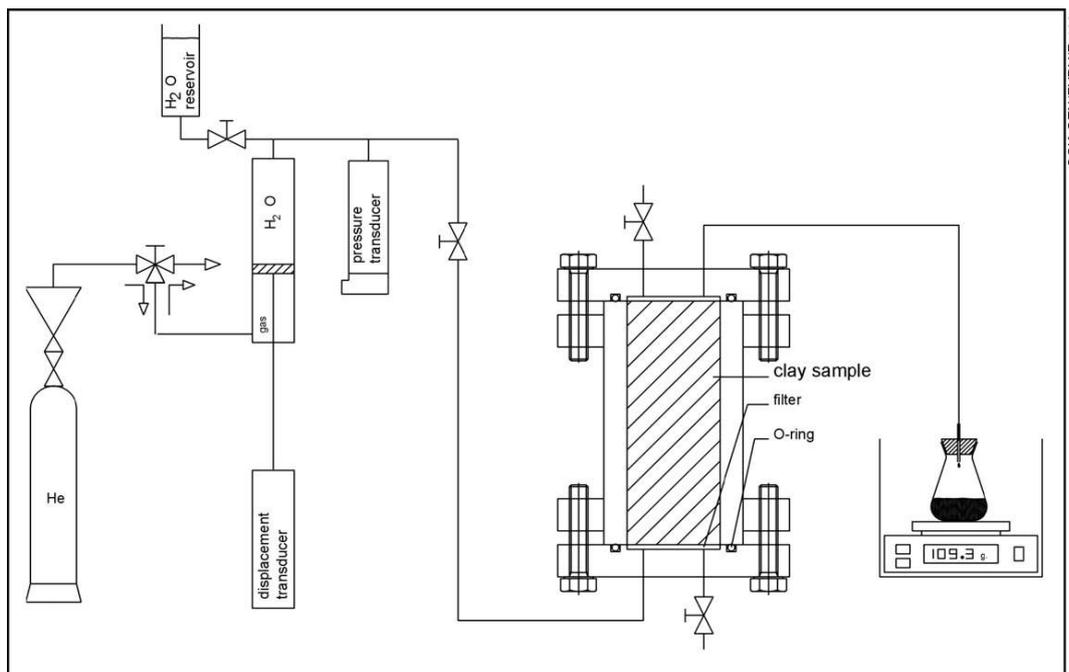


Figure 2: Scheme of the set-up to measure hydraulic conductivity

The design of the experimental set-up and the methodology have been developed within an internal R&D project of SCK•CEN. A detailed description can be found in (Wemaere et al., 2008). After a permeameter cell is loaded, it is installed in the experimental set-up (Figure 2). In our experiment, the diffusion cell is used instead of a permeameter cell. Water is injected under constant pressure at the bottom of the sample with a cylinder (piston) that is connected to a displacement transducer. The applied pressure is controlled by a pressure reducer (connected to a He gas bottle) and is measured by means of a pressure transducer (relative pressure). The water flowing out of the cell is collected in a flask (at atmospheric pressure) that is placed on a precision balance. During the experiment, the water inflow (displacement transducer), water outflow (balance) and applied pressure are registered.

The hydraulic conductivity K (m/s) is determined by applying Darcy's law:

$$K = (Q \cdot dl) / (S \cdot dh)$$

where Q : in-flow rate (m^3/s), S : surface area perpendicular to the flow rate (m^2), dh : pressure gradient (m water column) and dl : length of the sample (m).

In the described experiment, the water used to measure the hydraulic conductivity was low salinity Pearson water (composition in table 1), at a pressure gradient of 0.55 MPa. The measurement lasted 27 days.

Table 1: composition of low salinity Pearson water (Pearson et al., 2002)

	mg/l
NaCl	6132
Na ₂ SO ₄	1632
CaCl ₂ ·2H ₂ O	1020
MgCl ₂ ·6H ₂ O	1004
SrCl ₂ ·6H ₂ O	84
KCl	60
NaHCO ₃	50

Once the hydraulic conductivity has been measured and the sample is considered to be ready for the gas diffusion experiment, the diffusion cell is mounted in the gas diffusion set-up. The pumps are calibrated at a flow rate of 5 ml/min by shortly pumping milliQ water over the diffusion cell (valves of the diffusion cell are open, bypass valve is closed) in order to mimic the real situation during the diffusion experiment (taking into account resistance due to filters, tubing etc.). After calibration, the valves of the diffusion cell are closed and the bypass valve is opened. Now air is removed by performing at least 5 cycles of evacuation and refilling with gas (He for the left He-side and CH₄ for the right methane side). After the last flushing cycle, the set-up is evacuated and ready to be filled with 500 ml low salinity Pearson water.

The water (MilliQ) used to prepare the low salinity Pearson water is degassed to remove all atmospheric oxygen and stored in a glovebox with N₂ atmosphere. After preparation of the low salinity Pearson water, 500 ml is transferred into a clean, previously evacuated vessel, called "the transfer vessel". As this water is still in equilibrium with N₂, the transfer vessels are flushed respectively with He or CH₄ for at least 5 times in order to remove as much dissolved N₂ as possible.

Each transfer vessel is now connected to the corresponding (same gas) water vessel of the set-up, and the water is transferred from the transfer vessel into the water vessel of the set-up by

applying a pressure gradient. The amount of transferred water is determined by weighing the transfer vessels before and after filling the set-up.

Next, the water vessels of the set-up are pressurised with He (left side) and CH₄ (right side) at 15 bar. During 3 days, the gas pressure is monitored in order to investigate the gas tightness. The right side of the set-up filled with CH₄ turned out to be gas-tight. The pressure at the side filled with He decreased during 3 days, pointing to a leak. This leak was caused by a damaged screw thread of the pressure transducer. The pressure transducer was replaced, and the gas phase was flushed again a few times to remove atmospheric oxygen which contaminated the gas phase during the replacement of the pressure transducer. A new leak test was performed, and the entire set-up was found to be gas-tight.

In order to exclude contamination of the gas phases, a gas chromatography analysis is performed prior to the start of the diffusion test. As no He is found in the vessel with CH₄ and vice versa, the experiment is ready to be started. The pressure is set at 10 bar for both sides of the set-up, the bypass valves are closed, the valves of the diffusion cell are opened and the pumps are started. The gas phases are sampled approximately every 2 weeks and samples are analysed with a micro-GC (CP4900 – Varian), equipped with a Molsieve 5A column and a TCD detector.

3 Sample

The Opalinus Clay sample originates from the Schlattigen borehole in northeastern Switzerland and was cored at a depth 860.32 m below surface. The sample axis is oriented perpendicular to the bedding plane. The composition and some basic physical parameters of the sample were determined from the remainders of the sample after filling the diffusion cell and the results are represented in Table 2.

Table 2: Mineralogical composition, bulk density, grain density and calculated physical porosity for the Opalinus Clay sample. The methodology used to obtain these parameters is described by Wersin et al., (2012)

Quartz [wt. %]	K-feldspar [wt. %]	Plagioclase [wt. %]	Calcite [wt. %]	Dolomite / Ankerite [wt. %]	Siderite [wt. %]	Pyrite [wt. %]	C(org) [wt. %]	Clay minerals [wt. %]	Bulk dry density [g/cm ³]	Grain density [g/cm ³]	Physical porosity [vol. %]
28	3	b.d.	8	b.d.	b.d.	0.6	1.0	59	2.446	2.705	9.6

4 Modelling

The tests are interpreted with a simple diffusive transport model. This model represents the transport equation in a 1D geometry. The model is based on the first and second Fick's laws for diffusive transport in porous media.



Figure 3: Geometry used in the diffusive transport model. Blue cells indicate where the evolution of the dissolved gas concentrations is recorded

In the numerical implementation of this model, the clay core is represented by 30 elements (see Figure 3). At one end, a gas concentration was imposed as boundary condition, corresponding to the dissolved fraction at the prevalent pressure in the vessel following Henry's law. At the other end, the concentration of the considered gas is assumed to be 0. The diffusion-accessible porosity is set at 0.12 (Wersin et al., 2012). As output, fluxes at both faces are calculated with Fick's first law, as well as concentration profiles at regular time intervals and concentration in selected elements (shown in blue in Figure 3). The modelling accounts for reduction in pressure in both vessels because of sampling. All calculations are performed with COMSOL multiphysics version 3.5a, Earth science module. COMSOL Multiphysics is a commercial finite element based analysis and modelling software package, used for various applications in physics and engineering. The diffusion coefficients are obtained by using a least squares fitting procedure to the experimental data with the MATLAB Optimization Toolbox. This toolbox uses a Levenberg-Marquardt method on a Trust region, which is specifically optimized for least square problems. In fact, the model calculates the best fit for 2 factors: $D_{\text{pore}} \times \eta$ and $R \times \eta$. As η is set at 0.12, the values for D_{pore} and R are directly obtained.

5 Results

The measured hydraulic conductivity normal to the bedding plane was $1.2 \cdot 10^{-13}$ m/s with a standard deviation of $0.7 \cdot 10^{-13}$ m/s.

All gas samples are analysed with a micro-GC. Based on the obtained results and the diffusive model, the diffusion coefficient (D_{pore}) of He was calculated: $D_{\text{pore}} = 5.4 \cdot 10^{-10}$ m²/s. The 95% confidence interval was $5.1 \cdot 10^{-10} - 5.6 \cdot 10^{-10}$ m²/s.

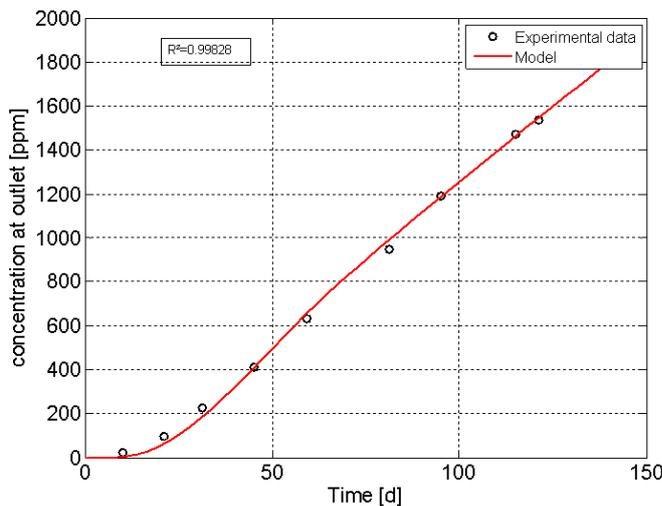


Figure 4: fit for D_{pore} of He

The value obtained for R by curve fitting is 12.3. However, for noble gases R is expected to be 1 as these gases do not react or sorb. This retardation ($R = 12.3$) can be explained by a failing pump during the first weeks of the experiment: when one of the pumps fails, the diffusion process is retarded because the pumps are needed for mixing the gas and water phase. After replacement of the pump body (without disturbing the experiment), the problem was solved and water was continuously pumped around. As the retardation affects only the offset of the curve (0 – 25 days), and not the linearly increasing part (25 – 150 days) from which is D_{pore} is calculated, the obtained value for D_{pore} should not be affected by the problem with the pump.

In order to compare the measured diffusion coefficient to other values, we tried to estimate the diffusion coefficient of He in Opalinus Clay from alternative methods. For 2 unretarded tracers (I and HTO), the ratio of their D_{pore} in Boom Clay vs. Opalinus Clay \perp to the bedding plane can be calculated (Table 3). As we know D_{pore} for He in Boom Clay ($12.2 \cdot 10^{-10} \text{ m}^2/\text{s}$) (Jacops et al., 2013), we can estimate D_{pore} for He in Opalinus Clay considering that this ratio remains the same. When using data for Opalinus Clay from Mont Terri, D_{pore} (He-predicted) becomes $4.4 \cdot 10^{-10} \text{ m}^2/\text{s}$. When using data for Opalinus Clay from the Benken borehole, D_{pore} (He-predicted) becomes $1.7\text{--}2.8 \cdot 10^{-10} \text{ m}^2/\text{s}$. Both numbers are close to the measured value for the sample from the Schlattingen borehole. The latter value is thus plausible, given the fact that the comparison is based on different core materials and that Opalinus Clay has some degree of mineralogical and textural heterogeneity. A more direct comparison based on measured D_{pore} (I, HTO) from the Schlattingen borehole will be made once the data become available.

Table 3: comparison of D_{pore} values for I and HTO in Boom Clay and Opalinus Clay

	I	HTO
D_{pore} Boom Clay (m^2/s) Samples from Mol	$1.4 \cdot 10^{-10}$ [1]	$2.3 \cdot 10^{-10}$ [2]
D_{pore} Opalinus Clay (m^2/s) Samples from Mt. Terri	$5.0 \cdot 10^{-11}$ [3]	$8.2 \cdot 10^{-11}$ [4]
D_{pore} Opalinus Clay (m^2/s) Samples from Benken	$1.9 \cdot 10^{-11}$ [4]	$5.3 \cdot 10^{-11}$ [4]
Ratio D_{pore} BC/ D_{pore} OPA Mt. Terri	2.8	2.8
Ratio D_{pore} BC/ D_{pore} OPA Benken	7.4	4.3

[1] (Bruggeman et al., 2010)

[2] (Bruggeman et al., 2009)

[3] (Van Loon et al., 2003)

[4] (Van Loon et al., 2004)

A comparison between the obtained diffusion coefficient and the data available in literature is made in Table 4.

Table 4: comparison between different obtained diffusion coefficients for He in Opalinus Clay

SCK-CEN (Schlattingen)	Gomez-Hernandez (2000) (Mont Terri)	Rübel et al. (2002) (Mont Terri)
$D_{\text{pore}} = 5.4 \cdot 10^{-10}$ $D_{\text{eff}} = 0.65 \cdot 10^{-10}$ $D_{\text{app}} = 5.4 \cdot 10^{-10} *$	$D_{\text{eff}} = 2.1 \cdot 10^{-10} \text{ m}^2/\text{s}$	$D_{\text{app}} = 3.5 \cdot 10^{-10} \text{ m}^2/\text{s}$

* considering $R = 1$

During the diffusion experiment, no diffusion of CH₄ was observed. The concentration CH₄ in the gas phase of the He-vessel remained very low and was quasi constant (ca 40 ppm). Analyses of the gas phase of the CH₄ vessel indicated that mainly CH₄ is present (table 5), so no error occurred when the set-up was filled.

Table 5: composition of the gas phase in the vessel with CH₄

	Concentration
Ar/O ₂	0.8 % vol
N ₂	0.9 % vol
CO ₂	100 ppm
CH ₄	98 % vol

In order to exclude bacterial activity, ATP (adenosinetriphosphate) analyses (Wouters et al., 2013) performed on water samples taken from the diffusion experiment. An increased ATP value is an indicator for metabolic activity of microbes. The results are presented in table 6.

Table 6: results of ATP analyses

Sample codes	ATP [mol]	CFU* [#]
OPA1 Vat He	$9.17 \cdot 10^{-16}$	$9.17 \cdot 10^2$
OPA1 Vat CH ₄	$5.00 \cdot 10^{-15}$	$5.00 \cdot 10^3$
		(= Lower limit of detection for the applied methodology and nature of the samples)
Blanco (=H ₂ O)	$1.91 \cdot 10^{-16}$	
Blanco + extracellular ATP	$1.73 \cdot 10^{-16}$	(= quality check)
* Assumption: 1 CFU contains 10^{-18} mol ATP (CFU: colony forming unit)		

For the vessel with CH₄, ATP concentration is increased compared to both the blanco (water value) and the result for the He vessel. This is pointing to bacterial activity in the CH₄ vessel. As no DNA analyses has been performed up to now, the microbial population has not been identified.

Another reason could be the failure of the pump. But in this case, a warning would occur. After the experiment was stopped (5 months after the last sampling), the performance of the pumps was investigated. The left pump (He vessel) didn't work properly: the water flow was very limited and at higher speed (50 ml/min) the pump blocked completely and stopped working. But problems with this pump (warning/error on the display) were encountered earlier, in the period between the last sample (february 2013) and the stopping of the experiment (july 2013). So it is not clear whether the problems with the pump started during the experiment, or only after the last sampling. Thus it cannot be excluded that the absence of a diffusion profile for CH₄ is caused by the pump. With the right pump (CH₄ vessel), no problems were observed.

6 Conclusion

The diffusion coefficient of He in Opalinus Clay normal to bedding (sample from Schlattingen borehole) was determined : $D_{\text{pore}} = 5.4 \cdot 10^{-10} \text{ m}^2/\text{s}$ (or $D_{\text{eff}} = 0.65 \cdot 10^{-10} \text{ m}^2/\text{s}$, considering a porosity of 12%). The hydraulic conductivity was also obtained: $K = 1.2 \cdot 10^{-13} \text{ m/s}$.

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