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In situ experimental investigation of neon diffusion in the Boom Clay in HADES URL, Belgium

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Over the past decade, the Belgian nuclear research centre (SCK CEN) has gathered extensive laboratory data on diffusion coefficients of dissolved gases in water-saturated Boom Clay (BC). Since gas transport around a geological disposal facility occurs at metre scales, a key question is whether centimetre-scale laboratory values remain valid under in situ conditions. To address this, SCK CEN, EURDICE, and ONDRAF/NIRAS launched the NEMESIS (NEon diffusion in MEgaS In-Situ) experiment in September 2023 at the HADES Underground Research Laboratory in Belgium. Its purpose is to confirm and refine understanding of gas diffusion in BC at realistic scales. NEMESIS reuses four piezometers from the 1990s MEGAS experiment. Dissolved neon is injected into the clay through a vessel–microtube–filter system, and its migration is monitored at three observation filters. This paper outlines the experimental design, strategy, initial findings, and early interpretations. The setup has proven effective under in situ conditions and provides new insights into the coupling between diffusion and water flow. Preliminary best-fit diffusion coefficients from Phase 1 (in-diffusion) are broadly consistent with laboratory measurements. Phase 2 (through-diffusion), to be conducted over the coming years, will deliver additional data to validate results, refine interpretations, and strengthen the characterisation of gas diffusion in Boom Clay.

Keywords: in situ testing/radioactive waste disposal/waste management and disposal

Introduction

Geological disposal is the preferred option for the long-term management of high-level radioactive waste and spent fuel in most countries (IAEA, 2022). Clay formations are considered by several national programmes as host rock or engineered barrier material because of their favourable properties for radionuclide confinement (ANDRA, 2005; Maes *et al.*, 2024).

Within a geological repository, significant amounts of gas, mainly hydrogen, can be generated through anaerobic corrosion of metals in the waste itself, waste packaging, or structural components of the repository (Levasseur *et al.*, 2024; Norris, 2015). Although gas production is expected to be slow, spanning over centuries, millennia or more, the extremely low permeability of a clay host rock, its very fine pore structure and a relatively low solubility of most gases in water can limit

gas evacuation. Also, engineered barrier components, such as clay-based buffers and seals, may hinder gas evacuation. This may lead to a build-up of gas pressure within the repository (Levasseur *et al.*, 2024). Initially, gas dissolves in pore water and diffuses away. But if the rate of gas production exceeds the diffusion of dissolved gas, a free gas phase may develop, potentially leading to pressure-driven fracturing or pathway creation, which could compromise the barrier function of the host rock (Capouet *et al.*, 2015; Jacops *et al.*, 2013; Norris, 2015).

The first fundamental mechanism for gas transport in the host rock of a repository system is thus diffusion of dissolved gases through pore water. If gas generation is sufficiently slow, and diffusion through the host rock is sufficiently rapid, the generated gases may remain fully dissolved; no free gas phase will form that could mechanically perturb the barriers. The balance between gas

generation and removal depends on gas solubility, described by Henry's law (Henry, 1803), and diffusion, which is governed by Fick's law and characterised by the diffusion coefficient (Jacops *et al.*, 2013). Therefore, accurate determination of diffusion coefficients for dissolved gas is essential to assess whether a free gas phase and overpressures may develop in the geological repository.

Previous studies have measured diffusion coefficients for various gases (e.g. He, Ne, Ar, H₂, CH₄, Xe, and C₂H₆) in fully saturated Boom Clay (BC) using laboratory-scale (centimetre-scale) experiments (Jacops *et al.*, 2017a, 2017b, 2020a, 2020b). These tests used a reliable double through-diffusion apparatus developed by Jacops *et al.* (2013), yielding valuable data under controlled conditions. The experimental protocol is currently well established and robust and has been extended to a wide range of clayey materials under various mechanical conditions. Comprehensive data sets of diffusion coefficients of seven dissolved gases in the BC, Callovo-Oxfordian claystone (COx), and Opalinus Clay (OPA) are summarised in Table 4-1 of Levasseur *et al.* (2024). Like hydraulic conductivity, the diffusion coefficient exhibits anisotropy due to better interconnection of pore spaces parallel to the bedding planes compared with those orthogonal to them.

However, a key open question remains: are diffusion coefficients derived from laboratory experiments still valid at a larger (metre) scale and under in situ conditions? This upscaling challenge has long been recognised in the context of radionuclide transport. Historically, migration experiments for radionuclides were first conducted in the laboratory on samples of only a few centimetres in size. To address the scale gap, several in situ tracer experiments have been carried out in the high-activity disposal experimental site (HADES) Underground Research Laboratory (URL), which is located at a depth of 225 m within the BC in northeastern Belgium. Successful examples include the CP1 and Tribicarb-3D in situ experiments, which studied the transport of tritiated water (HTO) and H¹⁴CO₃ + HTO, respectively. These studies demonstrated good agreement between lab-scale and in situ-derived transport parameters (Aertsens *et al.*, 2023). By first deriving diffusion coefficients in the laboratory and then validating them through in situ testing, we establish a cross-scale verification process that strengthens confidence in their accuracy and relevance.

To evaluate the diffusion coefficient of dissolved gas at metre-scale and under in situ conditions, a large-scale in situ diffusion experiment named NEon diffusion in MEgaS In Situ experiment (NEMESIS) was initiated in September 2023 by the Belgian nuclear research centre (SCK CEN), the European Underground Research Infrastructure for Disposal of nuclear waste In a Clay Environment (EURIDICE), and the Belgian Agency for Radioactive Waste and Enriched Fissile

Materials (ONDRAF/NIRAS) at the HADES URL in Mol, Belgium. The experiment uses dissolved neon to study gas transport behaviour in the BC. The primary objectives of NEMESIS are (i) to validate diffusion coefficients and anisotropy for diffusion of dissolved gas previously measured in the laboratory on small-scale samples and (ii) to enhance the understanding of gas diffusion processes in the BC at repository-relevant scales. NEMESIS is considered as the first in situ experiment conducted to measure gas diffusion coefficients in the BC, serving as a critical step in bridging the gap between laboratory-scale findings and large-scale behaviour.

Diffusion data for neon in the other clay-based materials, such as compacted bentonite, COx, and OPA, are reported in Jacops (2018). All data are derived from lab-scale experiments on samples between 20 and 60 mm in diameter and 10 and 30 mm in length. No other experiments, focusing on upscaling of diffusion of dissolved gases, have been set up. A summary of all gas diffusion experiments on OPA is available in Levasseur *et al.* (2024). Several reported values suffer from experimental issues and problems with data interpretation. On top of this, samples taken from different depths, and thus with variable porosity and pore size distribution, are used. Hence, direct comparison between lab and in situ data is impossible. Hence, upscaling of diffusion of gas in the other host rocks remains also an open issue.

This paper is organised as follows: the 'Material and in situ conditions' section introduces the HADES URL and describes briefly the relevant properties of the BC. The 'Methodology' section outlines the experimental design and workflow of the NEMESIS test. The 'Results of Phase 1' section presents and interprets findings from Phase 1 (helium in-diffusion tests). The 'Results of Phase 2' section presents and interprets findings from Phase 2 (neon through-diffusion tests). Some uncertainties and discussions are given before the final conclusions.

Materials and in situ conditions

In Belgium, no formal decision has been taken yet, but for R&D purposes, ONDRAF/NIRAS considers BC as a potential natural barrier for the disposal of its high-level and long-lived intermediate-level radioactive waste. In the 1980s, the HADES URL was constructed in the middle of BC at a depth of 225 m below SCK CEN's site in Mol, Belgium. Over the past four decades, the HADES URL has played a crucial role in providing arguments and the safety and feasibility statements of the Belgian programme (Li *et al.*, 2023). A summary overview of the experiments conducted in the HADES URL can be found in Table 1 of Van Geet *et al.* (2023).

BC is a stiff, fully saturated, and plastic clay formation found primarily in northeastern Belgium. Its physical (e.g. porosity) hydro-mechanical properties (e.g. intrinsic permeability, elastoplasticity,

and self-sealing and healing), in situ hydromechanical conditions (e.g. in situ stresses), mineralogical composition, cation exchange capacity, and so on have been extensively studied in the past decades by field tests (Bastiaens *et al.*, 2006; Bernier *et al.*, 2007) and laboratory tests (Bésuelle *et al.*, 2014; Chen *et al.*, 2014; Yu *et al.*, 2013; Zeelmaekers *et al.*, 2015). BC has low hydraulic conductivity (on the order of 10^{-12} m/s), favourable self-sealing capacity, and a high degree of mineralogical and structural homogeneity. The low porosity and fine-grained structure of BC, together with a low hydraulic gradient over the host formation, results in a negligible water flow through the clay; hence, molecular diffusion is the dominant transport mechanism (Aertsens *et al.*, 2009; De Craen *et al.*, 2006; Yu *et al.*, 2013).

Methodology

The NEMESIS setup is based on a double through-diffusion apparatus originally developed for measuring gas diffusion under laboratory conditions (Jacops *et al.*, 2013). This section begins with an introduction of the double through-diffusion technique, outlining its key components and operation principles. It then describes how the apparatus was adapted for the NEMESIS experiment to accommodate the complexities of in situ conditions (Jacops *et al.*, 2020c). To ensure the success of the in situ experiment, extensive preparatory work was undertaken, as summarised in Figure 1, which provides a general overview of the NEMESIS workflow.

The double through-diffusion technique

A versatile method for determining precisely the gas diffusion coefficient for dissolved gases in BC was developed by Jacops *et al.* (2013). The method allows for the simultaneous diffusion of two different dissolved gases through a single clay sample. The setup consists of two water/gas vessels and two independent circuits, which are placed on opposite sides of a BC core (see Figure 2).

The saturated clay core is sealed within a constant-volume stainless steel cell and connected at both ends to stainless steel filters, which interface with the two external vessels. Each vessel is initially filled with 500 ml of degassed synthetic BC pore water and an equal volume of gas, with each vessel containing a different

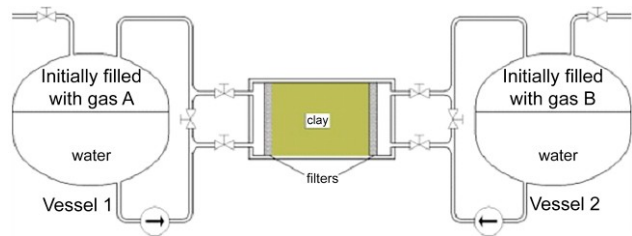


Figure 2. Schematic design of the setup to measure the diffusion of dissolved gases in the laboratory (modified from Jacops *et al.*, 2015)

type of gas. Both vessels are pressurised to the same level to ensure that gas transport occurs solely by diffusion of dissolved gases. The water in each circuit is continuously circulated over its respective filter using a pump. Once the water encounters the sample, dissolved gas diffuses from the high-concentration side (upstream vessel) to the low-concentration side (downstream vessel). In both vessels, the dissolved gas in the water equilibrates with the gas phase in the headspace. By measuring the gas composition in vessel 2 over time using analytical techniques such as gas chromatography, the diffusion coefficient of gas A can be determined; the same process applies to gas B. For detailed experimental and technical information, the reader is referred to Jacops *et al.* (2013). Given the good performance and robustness of the technique, the through-diffusion approach was adapted for the NEMESIS experiment.

The principle of the in situ gas diffusion experiment NEMESIS is like that of laboratory experiments. A gas (A) is dissolved in water within a source vessel, and a different gas (B) is filled within three target vessels. The water containing dissolved gas is circulated through micro-tubes and a piezometer filter embedded in the clay at a pressure as close as possible to the in situ pore pressure to minimise the advective flow of pore water into the filter. When circulation starts, dissolved gas begins to diffuse into the surrounding clay. After some time, dissolved gas A will be present in the water in target vessels through target filters, which are located at different distances from the source filter and in different directions relative to the natural bedding of the clay. By continuously

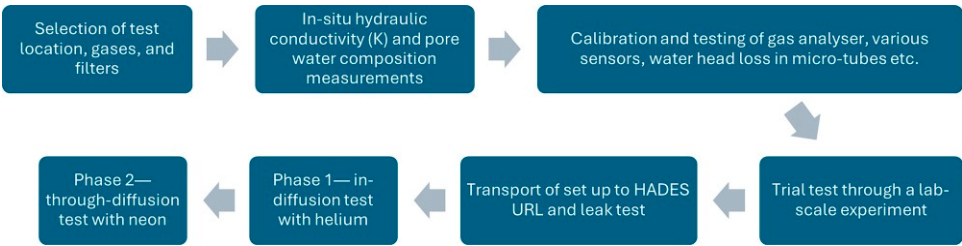


Figure 1. A general overview of the NEMESIS workflow

monitoring the concentration of gas A in the target vessels using online gas analysis, the diffusion parameters, including their anisotropy, can be determined with the support of numerical modelling.

Selection of test location

To study the undisturbed diffusivity of dissolved gas in BC under in situ conditions, it is essential to avoid preferential gas pathways in the formation. These pathways often result from unavoidable hydromechanical disturbances to the clay around piezometers during the drilling and subsequent installation. Such heterogeneities can provide easier routes for pore fluid transport compared with

the intact clay matrix, resulting in an overestimation of diffusion or permeability.

The existing Modelling and experiments on GAS migration in repository host rocks (MEGAS) E5 piezometers (Figure 3) were selected for use in the NEMESIS experiment. These piezometers were installed in 1992 as part of the MEGAS EC project (Ortiz *et al.*, 1997; Volckaert *et al.*, 1995). MEGAS E5 consists of four piezometer boreholes arranged in a three-dimensional configuration: three oriented horizontally and one inclined slightly upward (piezometer C, with a 3% angle of inclination) (Figure 4). Each piezometer extends 15 m from the underground gallery named

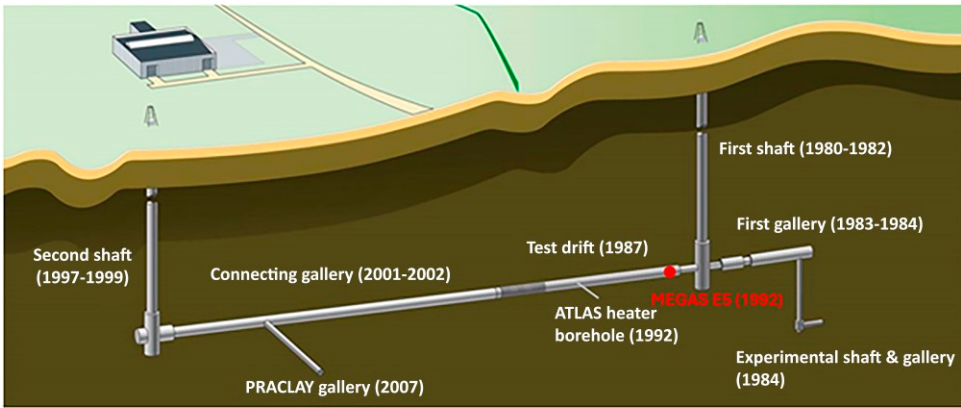


Figure 3. Location of the MEGAS E5 piezometer borehole entrances in the HADES URL

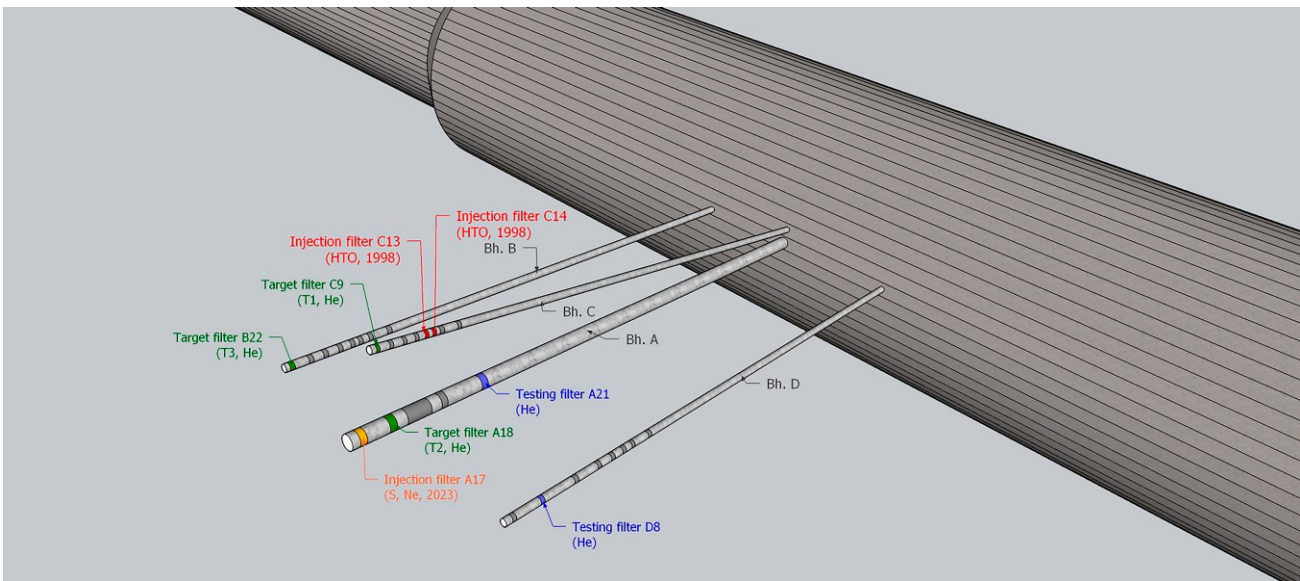


Figure 4. Overview of the four MEGAS piezometers (A–D) and their filters (1–29), with indication of the filters used in the NEMESIS diffusion experiment (not to scale)

‘Test drift’ and is equipped with multiple filter intervals. The setup was first utilised in 1994 for a gas breakthrough experiment involving helium injection into filter A20. In 1998, a second gas breakthrough test was conducted in filters C13 and C14, followed by a tracer diffusion experiment in which HTO was injected into filter C13 and monitored in the surrounding filters (Ortiz *et al.*, 1997, 2002; Volckaert *et al.*, 1995).

The excellent self-sealing capacity of BC, combined with over 25 years of convergence, ensures that the clay formation around the MEGAS E5 piezometers has sufficiently recovered, closing any potential preferential pathways (Jacops *et al.*, 2023). As a result, the BC surrounding the MEGAS E5 piezometers is considered to have been restored to an undisturbed state, making it ideal for the objectives of the NEMESIS diffusion experiment.

Selection of gases

In the NEMESIS experiment, neon was selected as the tracer gas. Although hydrogen (H_2) is the primary gas of interest under repository conditions, it was excluded from the study for several reasons. First, hydrogen is prone to leakage. Second, it can be converted into methane by microbes, which interferes with accurate measurement and interpretation (Jacops *et al.*, 2015). In many other in situ gas transport experiments, such as those conducted by Vinsot *et al.* (2014) and Volckaert *et al.* (1995), Ortiz *et al.* (1997), helium is used instead of hydrogen. As helium is naturally present in the BC and has already been used in previous MEGAS breakthrough experiments, helium cannot be used in the current gas diffusion experiment because of the existing high background concentrations. Neon, on the contrary, has a molecular size comparable with that of hydrogen (Jacops, 2018a), is chemically inert, and is not subject to microbial activity. These properties make neon the most appropriate proxy for hydrogen in the NEMESIS experiment. Helium is used in the target vessels because of its good compatibility with the gas analyser (CGC4 compact GC from Interscience). The latter uses a thermal conductivity detector (TCD) with helium as a carrier gas, allowing good detection of neon in a bulk volume of helium.

The diffusion coefficients of neon in BC were measured in the laboratory using the through-diffusion technique in Jacops *et al.* (2017b). Experiments were conducted on two samples: one oriented parallel to the bedding plane and the other perpendicular to it. The effective diffusion coefficients, D_e , determined at laboratory scale by fitting one-dimensional transport models, are $1.8 \times 10^{-10} \text{ m}^2/\text{s}$ ($D_e(\perp, \text{Ne})$, perpendicular to bedding plane) and $2.3 \times 10^{-10} \text{ m}^2/\text{s}$ ($D_e(\parallel, \text{Ne})$, parallel to bedding plane).

Selection of filters

A total of 29 filters are available on the MEGAS E5 piezometers (see Figure 4). Each filter is connected to double micro-tubes with an inner diameter of 2 mm, allowing for water circulation. Three

main criteria guided the selection of filters for the NEMESIS experiment: (i) a spatial configuration that enables the investigation of diffusion anisotropy; (ii) a reasonable distance to ensure the diffusion front progresses sufficiently for reliable monitoring over 5 years; (iii) location outside the contaminated zone.

In the 1998 HTO tracer test (Ortiz *et al.*, 2002), HTO was introduced into filter C13. From a safety perspective, filters with HTO activity below 10 Bq/mL were preferred (Jacops *et al.*, 2018). HTO activity was measured again in 2019. Among the 29 filters, 10 showed HTO activity levels exceeding 10 Bq/mL, including filters along piezometer C and four filters near filter C13 on the other piezometers (Jacops *et al.*, 2018). After evaluating various source and detection filter configurations through transport scoping calculations, filter A17 was selected as the source filter for neon introduction, and filters C9, A18, and B22 were chosen as target filters to monitor dissolved neon. Filters A17 and A18 have a diameter of 8.9 cm, a length of 9 cm, and a circumferential surface area of 0.025 m^2 . Filter C9 and B22 have a diameter of 5.56 cm, a length of 6 cm, and a circumferential surface area of 0.01 m^2 . These filters are positioned across both horizontal and vertical planes relative to A17: A18 is 0.750 m away horizontally, B22 is 0.782 m away horizontally, and C9 is 0.615 m away vertically.

Adaption of double-diffusion technique for in situ application

The NEMESIS setup comprises four independent circuits: one source circuit and three target circuits. A typical circuit is illustrated in Figure 5. Each circuit includes a vessel, a pump, and a filter, interconnected by two micro-tubes. One tube directs pore water from the filter to the vessel, while the other returns water from the vessel back to the filter by pumping. This closed-loop design enables continuous circulation of water in the circuit and monitoring of major variables within the system. The four vessel systems, together with associated pumps and sensors, are put together in one cabinet in the HADES URL.

The source circuit, hereafter referred to as A17-S, is connected to filter A17 and is responsible for introducing dissolved neon into the surrounding clay. Vessel A17-S is filled with a mixture of synthetic BC pore water and neon gas. The three target circuits – C9-T1, A18-T2, and B22-T3 – are connected to filters C9, A18, and B22, respectively. Each target vessel is filled with a mixture of synthetic BC pore water and helium gas. To accelerate the homogenisation of dissolved gas in the circuit, water is continuously circulated using the pump throughout the duration of the test.

Monitoring system

Each circuit is equipped with two pressure sensors, one located upstream of the pump and near the vessel, referred to as PBP (pressure before pump), and the other positioned just downstream

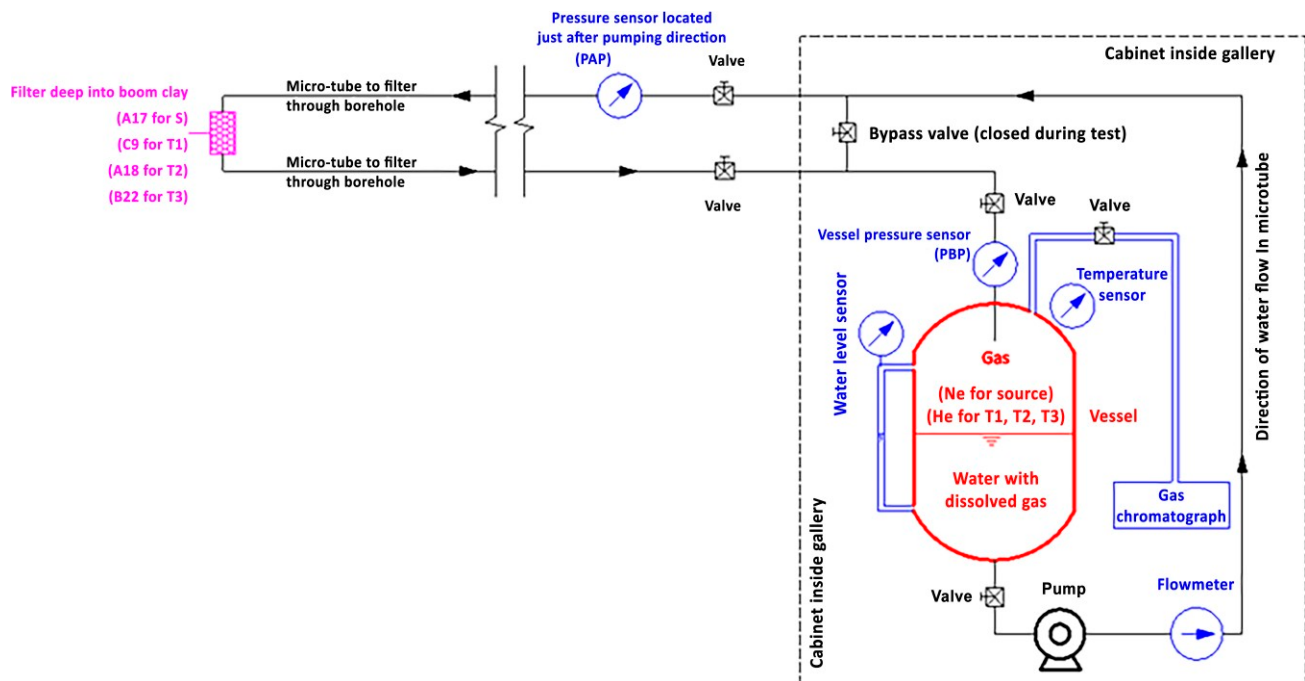


Figure 5. Illustration of the filter-microtube-vessel system used in the NEMESIS test

of the pump, referred to as PAP (pressure after pump). PBP reflects the pressure in the vessel, while PAP is equal to PBP plus the additional pressure imposed by the pump to circulate the water in the tube. Because the in situ filters are located very deep in the piezometer borehole, the total circuit extends over 30 m in length. The pressure difference between PAP and PBP must be designed to compensate for the pressure loss resulting from water flow through the circuit. A flow metre (Keyence, FD-XC8M) measures the water circulation rate within each circuit. A water level sensor (Sick, LFP0300-G1NMB) is installed in each vessel to track changes in water mass in the vessel. The sensor uses time domain reflectance technology – a process for determining the time of flight of electromagnetic waves. The time difference between the sent pulse and the pulse, which is reflected when it reaches the water level, is used to generate a level signal. The electrical signal is converted to percentage of level, which is, after applying a calibration equation, transferred into the volume of water inside the setup. Temperature is also monitored at the outer surface of each vessel because it was not possible to install the sensor inside, as this would lead to a technical modification of a certified pressure vessel. The top part of each vessel is connected to a CGC4 Compact Gas Chromatograph (Interscience, The Netherlands). This chromatograph is equipped with both a Molsieve 5A and a Carboxen 1010 column, enabling the separation of neon. A TCD is used to quantify neon concentrations. Helium is used as a carrier gas. The gas analyser also features a multi-position valve that facilitates automated gas sampling, enhancing the precision and reproducibility of measurements.

Table 1 provides an overview of the primary characteristics of the sensors.

In situ hydraulic conductivity (K) measurements

To verify that the BC surrounding the MEGAS piezometers has recovered to its undisturbed state, hydraulic conductivity (K) was estimated based on the average outflow rate recorded during HTO sampling. In addition, eight in situ permeability tests were conducted at four NEMESIS filters (A17, A18, B22, and C9), as well as at the four filters closest to the source filter A17 (Jacops *et al.*, 2023). The results show that K values at all MEGAS piezometer filters range between 3.2×10^{-12} and 4.6×10^{-12} m/s, except for filters D1 and C10, which have values of 2.0×10^{-12} and 6.1×10^{-12} m/s, respectively. At the four NEMESIS filters, K values range between 3.6×10^{-12} and 3.9×10^{-12} m/s. These consistently low and homogeneous K values confirmed that the BC surrounding the MEGAS piezometers has restored to its undisturbed state after over 25 years of recovery.

Other preparatory work

The NEMESIS system was first tested using a laboratory-scale diffusion experiment to verify the circuit functionality and assess whether the gas analyser could accurately capture temporal changes in gas concentration. The laboratory diffusion test, as illustrated in Figure 2, was conducted on a clay sample by filling two vessels with neon/water and helium/water mixtures, respectively. This test allowed for evaluation of both the circuits and the measuring system. The resulting diffusion coefficient measured

Table 1. An overview of the primary characteristics of the sensors

Parameter	Brand	Type	Range	Accuracy	Resolution
PBP	Druck	UNIK 5000	0–1.8 MPa (abs)	0.2% FS	—
PAP	Druck	PTX 1400	0–2.5 MPa (abs)	0.2% FS	—
Flow	Keyence	FD-XC8M	0–3000 ml/min	0.3% FS	0.1 ml/min
Level	Sick	LFP0300-G1NMB	0–300 mm	±5 mm	2 mm (12 ml)
Temperature	Digikey	JS8746B-0.15	–40°C–125°C	0.1°C	—

for neon, $D_e = 1.59 \times 10^{-10} \text{ m}^2/\text{s}$ was consistent with values obtained from previous laboratory experiments, confirming that the system functions correctly and is ready for deployment in the NEMESIS experiment (Jacops *et al.*, 2023).

The setup was then moved to the HADES URL and tested under in situ conditions. One of the most critical challenges in conducting in situ gas experiments is ensuring the leak-tightness of the experimental system. Even minor leaks in the tubing, connections, or vessels can introduce significant uncertainties in gas pressure measurements and compromise the reliability of the results (Jacops *et al.*, 2015). A leak test was conducted on each vessel system with the vessel filled with an artificial pore water/helium gas mixture and pressurised to the level equal to in situ pore water pressure of the corresponding filter. The pressure in the closed vessel system was monitored over five days and proved to be leak tight. However, the functionality of the filter system, including the double tubing within the piezometers and the in situ filters, cannot be checked.

The pump plays a critical role in the NEMESIS experiment. It not only circulates water within the circuit to ensure homogeneous concentration of the dissolved gas but also compensates for pressure losses caused by hydraulic head loss during water flow through the two tubing lines. To minimise water inflow from surrounding clay into the filter, the initial pressure at the filter location should be set equal to the in situ pore pressure. Accordingly, when specifying the initial vessel pressure, the pressure head losses along each circuit must be taken into consideration.

Two-phase strategy in NEMESIS

The NEMESIS experiment adopts a two-phase strategy to ensure the reliability of the test design and the quality of data collected under

in situ conditions. Before launching the full-scale through-diffusion experiment (Phase 2 test), preliminary in-diffusion tests (Phase 1 test) were conducted on two filters far from NEMESIS filters but employing the same experimental setup. The concepts behind these two experiments are simply illustrated and compared in Figure 6.

In the through-diffusion experiment (see also Figure 2 and its associated introduction), a clay sample is placed between two filters, each connected to a water reservoir. The upstream reservoir (on the left in Figure 6(a)), also referred to as the inlet, is initially doped with a known concentration of the tracer of interest (C_0), while the downstream reservoir (on the right in Figure 6(a)), or outlet, is initially tracer-free. The clay sample itself is also initially free of the tracer. Over time, the evolution of tracer concentration is monitored in the upstream reservoir (C_{in}) or/and downstream reservoir (C_{out}), and/or the concentration profile within the clay at the end of the experiment is measured. Diffusion parameters can be determined by solving appropriate transport models (Aertsens, 2011; Bourg and Tournassat, 2015).

In the in-diffusion experiment (Figure 6b), the clay sample is placed in contact with a single water reservoir containing the tracer at an initial concentration C_0 . The decrease in tracer concentration in the reservoir is measured (C_{in}) over time. At the end of the experiment, the tracer profile within the clay can also be analysed.

The Phase 1 – in-diffusion test in the NEMESIS experiment serves as a diagnostic phase, allowing verification of the functionality and integrity of the system, assessment of the imposed boundary conditions, and initial validation of the numerical modelling framework. It also provides an opportunity to fine-tune operational parameters and identify potential challenges in advance.

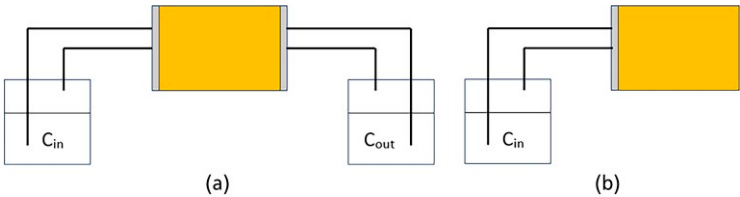


Figure 6. Schematic description of two methodologies commonly used to obtain diffusion coefficients in clay materials: (a) through-diffusion and (b) in-diffusion (after Bourg and Tournassat, 2015)

Phase 1 – helium in-diffusion experiment

The Phase 1 – helium in-diffusion experiment was conducted on October 24, 2022. Two filters far from the four NEMESIS filters, filters D8 and A21 (see Figure 4), were selected for testing. Each filter was connected to a vessel system, serving as the ‘upstream reservoir’ as depicted in Figure 6(b). The two vessels were initially filled with a mixture of synthetic BC pore water and helium gas, pressurised to match the in situ pore pressure at the respective filter locations. Water was continuously circulated with a constant flow rate to ensure homogenisation of dissolved helium within the circuit. Throughout the experiment, PAP, PBP, flow rate, temperature in each circuit, and water level in each vessel were closely monitored.

Phase 2 – through-diffusion experiment

Following the successful validation and experience in Phase 1, the full-scale through-diffusion experiment of Phase 2 was launched on September 5, 2023. Vessel A17-S was initially filled with 0.66 L of synthetic BC pore water (composition according to De Craen *et al.* (2004)) and 1 L of neon gas. To minimise the water advection from clay into the vessel, the initial pressure at the filter location should be as close as possible to the in situ pore pressure, for which the initial gas pressure in vessel A17-S was carefully set by considering the hydraulic head loss along the micro-tubes and the in situ pore pressure. Based on these considerations, the initial gas pressure in vessel A17-S was set to 1.508 MPa. The three target vessels – C9-T1, A18-T2, and B22-T3 – were each filled with ~0.85 L of synthetic BC pore water and 0.8 L of helium gas. Following the same considerations, their initial vessel pressures were also carefully set from 1.46 to 1.51 MPa. A constant flow rate, ranging between 25 and 27 ml/min, was used in each circuit. Throughout the experiment, high-resolution data of pressure, water flow rate, water level in each vessel, and temperature have

been continuously collected from all four circuits to analyse the spatial and temporal evolution of dissolved gas concentration in the BC.

Numerical modelling

The numerical model for the NEMESIS test was developed using the commercial software COMSOL Multiphysics V6. The model accounts for the coupled process of neon and helium diffusion (the module of transport of diluted species in porous media), as well as water exchange between the host rock and the circuit through the filter (the module of Darcy’s law). A brief overview of the modelling methodology is presented in the Appendix.

Results of Phase 1 – helium in-diffusion experiment

For the two helium in-diffusion tests of Phase 1, after helium started to diffuse into the surrounding BC, a gradual decrease in vessel pressure was observed over time, denoted as $\Delta p(t)$. The pressure drops recorded in the vessels connected to filters D8 and A21 are presented in Figure 7. Here, $\Delta p(t) = p_i - p(t)$, where p_i is the initial stabilised pressure just after the start of pumping, and $p(t)$ is the vessel pressure at time t .

As diffusion progressed, the pressure in each vessel gradually declined and approached a plateau. After ~5 months, the stabilised pressure drops reached around 0.016 and 0.028 MPa for the vessel connected to filters A21 and D8, respectively.

As the water with dissolved gas comes into contact with the surrounding host clay and the diffusion process starts, the gas pressure in the vessel starts to decrease, creating a pressure gradient between the vessel and the filter. This gradient drives clay pore water to flow into the vessel, which compresses the gas inside the

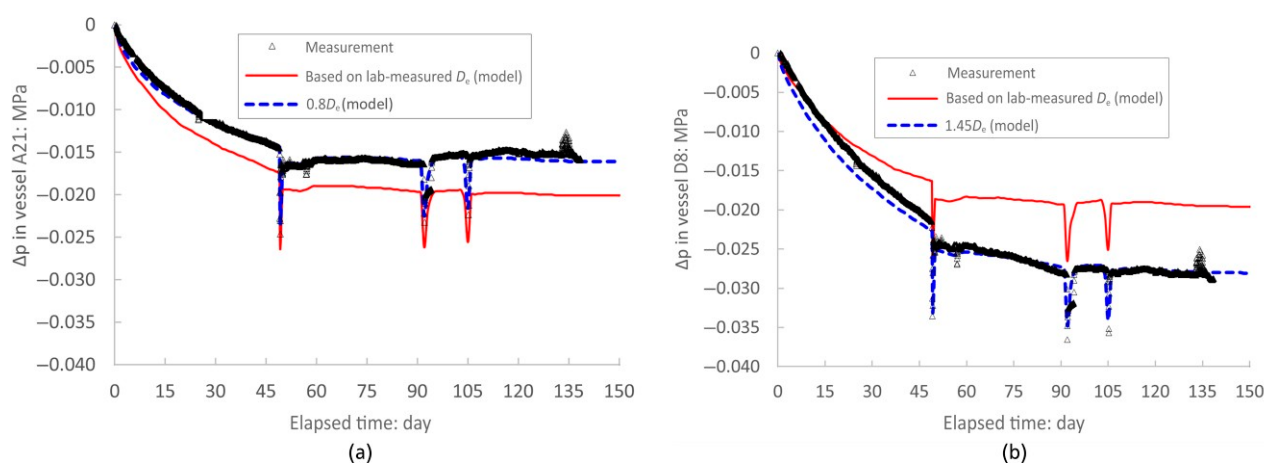


Figure 7. Results of Phase 1 – helium in-diffusion test. The figure shows the measured pressure drop Δp (black symbols), modelled results based on laboratory-measured diffusion coefficients D_e (red lines), and modelled results using best-fit diffusion coefficients (blue dashed lines)

vessel. The resulting gas compression leads to an increase in vessel pressure, partially counteracting the gas pressure drop due to diffusion. These two processes – diffusion of dissolved helium into the clay and inflow of pore water into the vessel – occur simultaneously and are dynamically balanced by the interaction between the gas and water phases within the vessel.

Over time, the pressure drop in the vessel approaches a plateau, indicating a near-equilibrium between the two processes. The continued rise in water level within the vessel confirms the continuous water inflow.

Temperatures in the HADES URL remain relatively stable, ranging between 21°C and 23°C with minor seasonal variations. Short-term, abrupt pressure fluctuations observed in Figure 7 were linked to abnormal temperature variations in the HADES URL, primarily caused by external disturbances. For example, the sudden pressure drop on day 49 was triggered by a temporary power-off of ventilation at the HADES URL.

To reproduce this dynamic behaviour in this transient phase, the numerical model must incorporate both diffusion and Darcian water flow processes. In addition, the model must dynamically update the boundary conditions at the filter–clay interface based on the evolving interaction between the gas and water phases within the vessel. By analysing the temporal evolution of $\Delta P(t)$ in the vessel and incorporating known values for BC permeability, the diffusion coefficient of the BC was estimated through numerical modelling.

Based on the effective diffusion coefficients of helium obtained from laboratory tests: $D_e(\parallel, \text{He}) = 7.47 \times 10^{-10} \text{ m}^2/\text{s}$ (parallel) and $D_e(\perp, \text{He}) = 5.75 \times 10^{-10} \text{ m}^2/\text{s}$ (perpendicular) (Jacops *et al.*, 2017b, 2018), the numerical results capture the general trend of the measured pressure drops in both vessels, as shown in Figure 7. However, an offset around 0.005–0.01 MPa remains, as illustrated by the red curve in Figure 7. Improved agreement was achieved by slightly adjusting the diffusion coefficients, as indicated by the blue curves in Figure 7. The best fit was gained using diffusion coefficients of $0.8D_e$ for filter A21 and $1.45D_e$ for D8. These values fall within the expected uncertainty range.

The Phase 1 – helium in-diffusion experiment proves to be a crucial precursor to the subsequent Phase 2 – neon through-diffusion experiment. It verified not only the functionality of the experimental setup and the imposed boundary conditions but also provided an initial validation of the numerical model. The results suggest that the NEMESIS setup performs reliably and is well-suited to achieving the objectives of the full-scale experiment.

Results of Phase 2 – neon through-diffusion experiment

According to scoping calculations using the laboratory-measured diffusion coefficients, it is expected to take ~ 2.5 –3 years for neon

to diffuse from source filter A17 to the three target filters, assuming no preferential pathways are present (Jacops *et al.*, 2023). As a result, a complete analysis of the neon through-diffusion test is not yet feasible at the time of writing this paper (May 2025). Nevertheless, valuable insights can already be gained by analysing the vessel pressure responses during the initial transient phase. The early stage of Phase 2 is analogous to the Phase 1 experiment and provides an early indicator of system behaviour.

The circuits A17-S and B22-T3 have been operating smoothly, whereas C9-T1 and A18-T2 have experienced intermittent pumping and leakage issues. Water flow rates in circuits A17-S and B22-T3 are quite stable and range from 24 to 28 ml/min (see Figure 8(a)). Figure 8(b) shows the pressure variations (Δp) in vessels A17-S and B22-T3 from the test start to May 2025, in both vessels, a gradual decrease in pressure was observed over time, and both reached a plateau after ~ 440 days. The higher in situ pressure than the vessel pressure yields a continuous inflow of water into the vessels (Figure 8(c)). Comparing the vessel responses between B22-T3 and A17-S is not straightforward due to several factors: helium has a diffusivity ~ 3 times greater than that of neon; the circumferential surface area of filter A17 is 2.5 times larger than that of B22; and the initial gas volume in vessel A17-S is 1.25 times higher than that of B22-T3. Consequently, the same water inflow to the vessel does not have the same impact on vessel pressure. Temperature follows seasonal variations, generally ranging between 21°C and 23°C (Figure 8(d)), with occasional spikes primarily caused by temporary gallery ventilation failure.

Based on experience from the Phase 1 in-diffusion test and supporting scoping calculations, vessel pressure is expected to reach a quasi-steady state after several months. However, in vessels A17-S and B22-T3, a continuous pressure decrease has been observed even after 1 year. In comparison with the pressure drop observed in A21 with helium (black symbols in Figure 7(a)), the decline in A17-S occurs at a steeper rate and persists for a longer duration (see Figure 9(b)), although neon has a much lower diffusion coefficient than helium. These findings suggest that additional unidentified factors may be influencing the behaviour in circuit A17-S.

Uncertainties and discussions

Water exchange through the filter plays a crucial role in the pressure variations observed in the vessel. Although significant efforts were made prior to the test to investigate the influence of various factors on water pressure within the circuit, substantial uncertainties remain. Setting the initial vessel pressure to ensure that the initially set pressure at the filter location precisely matches the local in situ pressure is a challenge. After the pumping starts, the pressure along the circuit is not uniform due to pressure head loss along the circuit, and pressure at the filter location has to be assumed as an average of PAP and PBP; in addition, pumping

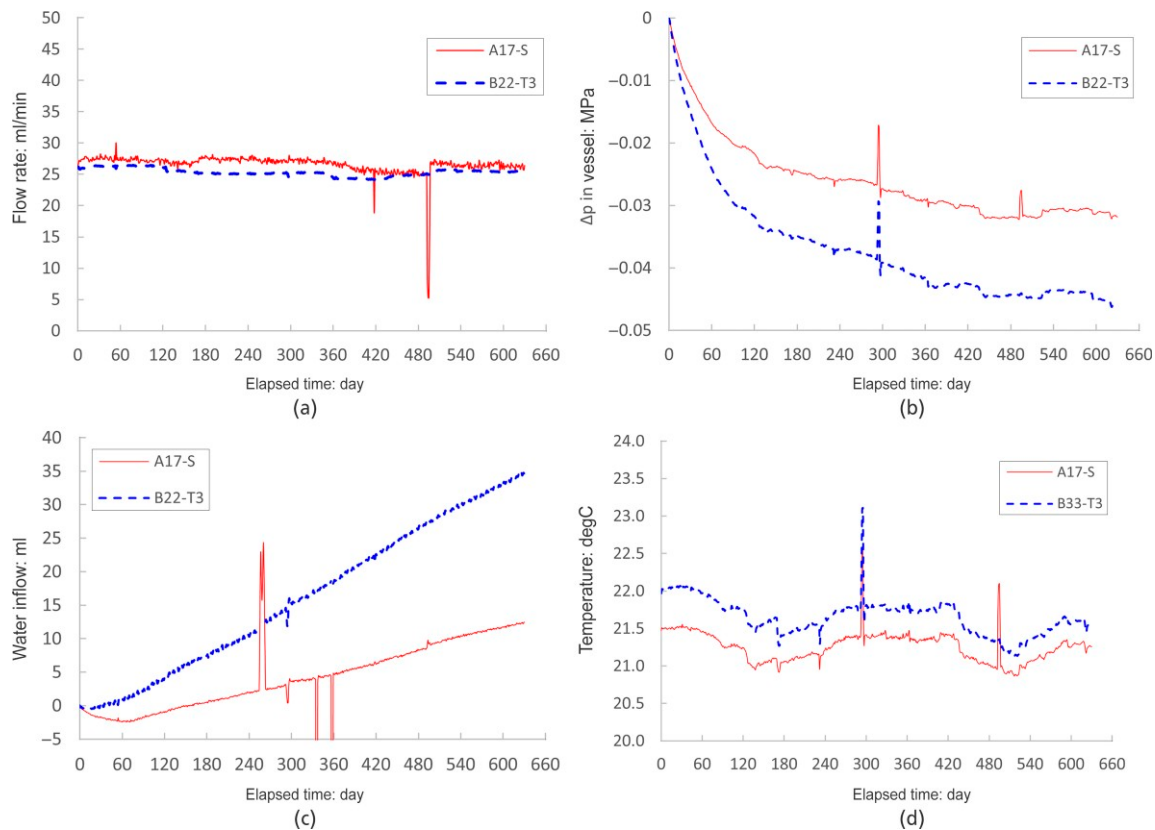


Figure 8. Measurements for A17-S and B22-T3 since the start of the experiment (September 2023) until May 2025

circulates the colder pore water from the filter to the warmer cabinet, which reduces the vessel pressure set before pumping starts. Any imbalance between the pressure at the filter location and the local in situ pressure at the very beginning can trigger unintended water flow, which significantly influences the initial pressure drop in the vessel.

Throughout the test, the flow rate in the circuit is not constant (as shown in Figure 9(a)), leading to variable pressure head losses in the tubing. These factors collectively introduce uncertainties in the pressure boundary conditions at the filters, thereby affecting the accuracy of the numerical modelling.

The modelling results based on the laboratory-measured diffusivities of neon and helium show good agreement with the measured pressure drop in vessel B22-T3 but significantly underestimate the pressure drop in vessel A17-S, as presented in Figure 9(b). Various parameters were tested in the numerical model for A17-S, including diffusion coefficient, permeability, initial pressure imbalance between the filter and the vessel, and potential leakage of gas or water with the circuit. However, none of these factors could reproduce the continuous pressure drop observed in the vessel.

Several gas transport experiments reported microbial activity, which disturbed the experimental results (Jacops *et al.*, 2015; Vinsot *et al.*, 2014). As the NEMESIS experiment uses helium and neon, which are both noble gases, we expect no microbial activity related to these gases.

Uncertainties regarding the spatial coordinates of the filters must be carefully verified, as they directly influence the numerical interpretation of the through-diffusion test results in NEMESIS. The coordinates of the MEGAS filters were initially recorded in 1992, immediately following drilling. However, over time, the casings containing the integrated filters may have shifted from their original positions due to clay deformations. At present, it is not possible to survey through the interior of the casing to determine the precise filter locations, as the casing is occupied by tubes. A 2019 survey conducted at the casing entrances revealed deviations from the 1992 reference measurements, indicating the positional shifts have likely occurred. Improved coordinates are anticipated in the future. These will enhance the accuracy of the numerical modelling. Another point of attention is the fundamental difference between the in-diffusion and through-diffusion techniques. The in-diffusion process, on which the results and interpretations of Phase 1 are based, is a measurement technique where only the variation of pressure is measured. This pressure is very sensitive to changes in volume and temperature but

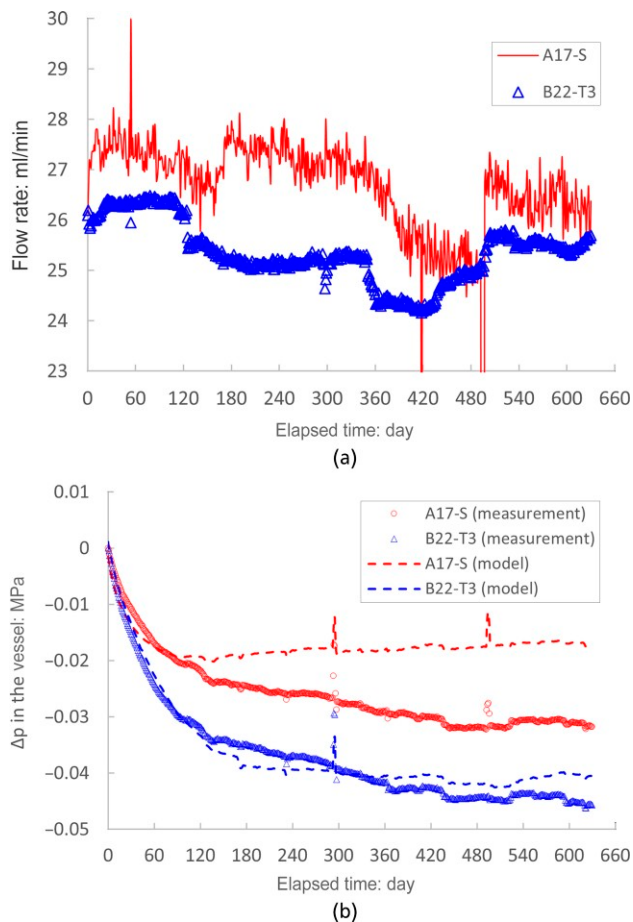


Figure 9. (a) Water flow rate measured in circuits of A17-S and B22-T3; (b) comparison between modelling results and measurements for A17-S and B22-T3 in Phase 2 – neon through-diffusion test

also to the functioning of the pump of the setup. When a pump is stopped because of technical issues (e.g. failure of the engine), the pressure in the circuit is disturbed, leading to abrupt changes in pressure which no longer allow a relevant assessment of in-diffusion. The through-diffusion process, which is the main objective of the NEMESIS experiment, is based on measuring the gas concentration change in time. Given the long time frame of through-diffusion, this process is less dependent on factors such as pumping or temperature variations, and short disruptions will have limited impact on the interpretation of the experimental results. Consequently, the through-diffusion part is the most relevant output of this experiment, while the in-diffusion data presented in this paper provide a first good indication of the diffusion process around the NEMESIS filters, allowing a first comparison of large-scale data compared with lab-scale data.

Conclusions

NEMESIS is considered the first in situ experiment conducted to measure gas diffusion coefficients in the BC, serving as a critical

step in bridging the gap between laboratory-scale findings and large-scale behaviour. Its primary objectives are (i) to validate diffusion coefficients and anisotropy for diffusion of dissolved gas previously measured in the laboratory on small-scale samples and (ii) to enhance the understanding of gas diffusion processes in the BC at repository-relevant scales.

The principle of this in situ gas diffusion experiment is based on the double through-diffusion technique used in the laboratory but adapted to accommodate the complexities of in situ conditions. To ensure the success of the experiment, extensive preparatory work was undertaken. NEMESIS follows a two-phase strategy to validate the test design and ensure high-quality data collection under in situ conditions. Before launching the full-scale through-diffusion experiment (Phase 2), a short-term in-diffusion test (Phase 1) was conducted using two filters.

During the Phase I helium in-diffusion experiment, reliable data were continuously collected over a five-month period. Pressure in the vessels gradually decreased as gas diffused out from the filter, resulting in a slow inflow of clay pore water into the experimental setup to ultimately reach a (quasi-)steady-state after ~3 months. This test suggested that the test setup performs reliably and is well-suited to achieving the objectives of the NEMESIS experiment. More importantly, the test provided valuable insights into how the coupling between diffusion and water flow into the setup evolves over time under dynamically regulated boundary conditions. The general trend of the measured pressure evolution in both vessels is in line with the expectations and is well captured by the numerical simulations. This validates the understanding of the behaviour of the experiment setup in general and of the diffusion process. The preliminary best-fit diffusion coefficient values of helium fall within the uncertainty range expected from the small-scale lab tests programme.

The Phase 2 full-scale neon through-diffusion experiment has been ongoing for more than 1 year and a half. Numerical modelling has been able to satisfactorily reproduce the pressure drop curve observed in vessel B22-T3, using diffusion coefficients for helium measured in the laboratory. However, for the source vessel A17-C, the causes of the continuous and steeper pressure drop remain unclear and require further investigation. Several uncertainties impacting the system will be addressed through additional analysis in the future. This highlights the complexity of in situ experiments, where a thermal, hydraulic, mechanical and chemical coupling is present.

The continued operation of the Phase 2 through-diffusion experiment will be crucial in the next stage, as it will provide more measurement data to validate the initial findings from Phase 1, improve our interpretation of NEMESIS test results, and enable a reliable characterisation of the gas diffusion properties of BC.

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Appendix

By May 2025, no neon had been detected in three target vessels. Therefore, four independent models were developed for each filter and its corresponding circuit system. The computational domain for each filter was defined as a clay cube with a side length of 5 m, centred on the filter (Figure 10). Within the clay domain, four hollow boreholes were included. The time-dependent conditions of the circuit system are imposed as boundary conditions on the filter surface. The domain was discretised into ~ 1 million quadratic tetrahedral elements. Modelling details for the source vessel (A17-S) are provided here as a representative example.

Initial conditions applied in the model

- Water pressure in the clay: set equal to the in situ water pressure measured at filter A17 (1.515 MPa).
- Neon concentration in the clay: initially zero.

Table 3. Transport parameters used in the model

Parameter	Value
Porosity	0.38
$K(//)$: m/s	4.5×10^{-12} (Yu <i>et al.</i> , 2013)
$K(\perp)$: m/s	2.1×10^{-12} (Yu <i>et al.</i> , 2013)
$D_e(//, \text{helium})$: m^2/s	74.7×10^{-11} (Jacops <i>et al.</i> , 2017a)
$D_e(\perp, \text{helium})$: m^2/s	57.5×10^{-11}
$D_e(//, \text{neon})$: m^2/s	22.9×10^{-11} (Jacops <i>et al.</i> , 2017b)
$D_e(\perp, \text{neon})$: m^2/s	17.6×10^{-11}

Boundary conditions

At the filter surface, the dissolved gas concentration is set equal to that in the vessel and is updated at each time step according to the vessel pressure:

$$C_{\text{vessel}}(t) = K_H P_{\text{vessel}}(t),$$

where $K_H = 0.00045$ mol/atm/L is Henry's coefficient for neon. The initial vessel pressure is 1.508 MPa, corresponding to a dissolved neon concentration of 6.79 mol/ m^3 .

The time-dependent water pressure at the filter, $P_{\text{filter}}(t)$, is prescribed and updated at each time step:

$$P_{\text{filter}}(t) = P_{\text{vessel}}(t) + \frac{1}{2} \Delta P_{\text{pump}}(t),$$

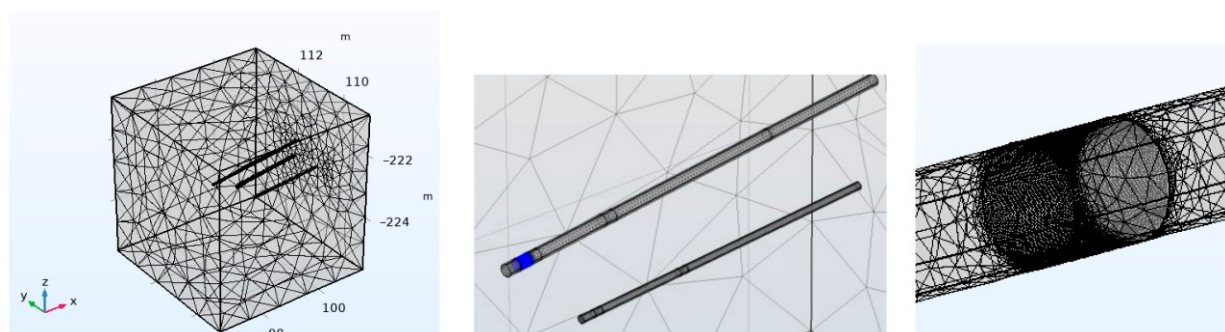


Figure 10. The computational domain (left); zoomed-in view of filter A17 (in blue) (middle); refined discretisation around the filter (right)

Table 2. Filter and vessel information

Filter	Gas	Length: mm	Outer/inner diameter: mm	Circumferential surface area: m^2	In situ water pressure: MPa	P_{vessel} : MPa	V_{gas} in vessel: mL	V_{water} in vessel: mL	V_{water} in tubing: mL
D8	He	60	55.6/50	0.01	1.470	1.455	750	750	166
A21	He	90	89/79	0.025	1.277	1.262	750	750	179
A17-S	Ne	90	89/79	0.025	1.515	1.508	1002	498	165
C9-T1	He	60	55.6/50	0.01	1.518	1.507	802	698	127
A18-T2	He	60	55.6/50	0.025	1.468	1.455	803	697	160
B22-T3	He	60	55.6/50	0.01	1.521	1.510	804	696	124

where $P_{vessel}(t)$ is the pressure in vessel A17-S, and $\Delta P_{pump}(t)$ is the overpressure applied by the pump to compensate for head losses induced by water circulation. At the start of the test, the flow rate in the circuit was 25 ml/min, corresponding to a head loss of 0.032 MPa. Using the equation above, the water pressure at the filter was estimated to be 1.524 MPa. This indicates that an initial overpressure of $1.524 - 1.515 = 0.009$ MPa was applied at the filter to the host Boom Clay, which in turn generated an initial water inflow. The pump-induced overpressure is updated proportionally to the flow rate (ml/min) according to:

$$\Delta P_{pump}(t) = flow_rate(t) \cdot \frac{0.032}{25}.$$

All other boundaries are subjected to no-flux conditions for both Darcy flow and diffusion models.

Water balance in the circulation system

The total water volume in the circulation system evolves as

$$V_{water}(t) = V_{0,water} + V_{water,in}(t),$$

where $V_{0,water} = 0.663$ l is the initial water volume (m^3) in the circulation system (vessel, tubes, filter, etc.) and $V_{water,in}(t)$ is the cumulative water volume entering from the clay into the circuit, obtained by integrating the water flux over the filter surface in both space and time.

Gas balance in the circulation system

The circulation system contains both dissolved gas and free (gaseous) gas. At time t :

- mass of dissolved gas:

$$M_{dissolved}(t) = K_H P_{vessel}(t) V_{water}(t),$$

- mass of free gas:

$$M_{gaseous}(t) = \frac{P_{vessel}(t) V_{gas}(t)}{RT(t)},$$

where $V_{gas}(t) = V_{0,gas} - V_{water,in}(t)$ is the gas volume in the vessel (initially $V_{0,gas} = 1.002$ L), $R = 8.314$ J/mol/K is the universal gas constant, and $T(t)$ is the vessel temperature (initially 21.75°C).

By mass conservation, the initial total gas in the circulation system satisfies

$$M_{0,gas} = M_{dissolved}(t) + M_{gaseous}(t) + M_{gas,out}(t),$$

where $M_{gas,out}(t)$ is the cumulative gas lost by diffusion to the surrounding clay. For the initial vessel pressure of 1.508 MPa in A17-S, the total neon in the circulation system $M_{0,gas} = 0.62$ mol, including 0.616 mol of gaseous neon and 0.0045 mol dissolved in water.

Governing pressure equation

Combining the above equations, the vessel pressure at any time t can be expressed as

$$P_{vessel}(t) = \frac{M_{0,gas} - M_{gas,out}(t)}{K_H V_{water}(t) + \frac{V_{gas}(t)}{RT(t)}}.$$

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