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**KG²B, a collaborative benchmarking exercise for
estimating the permeability of the Grimsel granodiorite
– Part 1: measurements, pressure dependence and
pore-fluid effects**

C. David, C Wassermann, Marc Fleury, Guillaume Berthe, Alexandra Amann-Hildenbrand, Bernhard Krooss, Joel Billiotte, Catherine Davy, Pierre Delage, Philipp Braun, et al.

► **To cite this version:**

C. David, C Wassermann, Marc Fleury, Guillaume Berthe, Alexandra Amann-Hildenbrand, et al.. KG²B, a collaborative benchmarking exercise for estimating the permeability of the Grimsel granodiorite – Part 1: measurements, pressure dependence and pore-fluid effects. *Geophysical Journal International*, 2018, 215 (2), pp.799-824. 10.1093/gji/ggy304 . hal-01992587

HAL Id: hal-01992587

<https://ifp.hal.science/hal-01992587v1>

Submitted on 12 Feb 2019

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2 **KG²B, a collaborative benchmarking exercise**
3 **for estimating the permeability of the Grimsel granodiorite:**
4 **measurements, pressure dependence and pore-fluid effects**
5

6 **C. David¹, J. Wassermann², and the KG²B Team^{3*}**
7

8 ¹Université de Cergy-Pontoise, Laboratoire GEC, Cergy-Pontoise, France.

9 ²Université de Cergy-Pontoise, Laboratoire L2MGC, Cergy-Pontoise, France.

10 ³Complete list detailed in Appendix A.
11
12

13 Corresponding author: Christian David (christian.david@u-cergy.fr)
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15 **Key Points (140 characters max):**

- 16
- 17 • A benchmarking exercise involving 24 laboratories was organized to measure the permeability of the Grimsel granodiorite
 - 18 • The influence of pore fluid, sample size, pressure sensitivity and methods (steady-state, transient pulse, oscillation) are discussed
 - 19 • The average permeability measured with gas is larger than that measured with liquid
 - 20 (mostly water) by a factor 2
 - 21
 - 22

23 Abstract

24 A benchmarking exercise involving 24 laboratories was organized for measuring the
25 permeability of a single low permeability material, the Grimsel granodiorite, at a common
26 effective confining pressure (5 MPa). In total 39 measurements were collected that allowed us to
27 discuss the influence of (i) pore-fluid, (ii) measurement method, (iii) sample size and (iv)
28 pressure sensitivity. Discarding some outliers from the bulk data set (4 out of 39) an average
29 permeability of $1.11 \cdot 10^{-18} \text{ m}^2$ with a standard deviation of $0.57 \cdot 10^{-18} \text{ m}^2$ was obtained. The most
30 striking result was the large difference in permeability for gas measurements compared to liquid
31 measurements. Regardless of the method used, gas permeability was higher than liquid
32 permeability by a factor ~ 2 ($k_{\text{gas}}=1.28 \cdot 10^{-18} \text{ m}^2$ compared to $k_{\text{liquid}}=0.65 \cdot 10^{-18} \text{ m}^2$). Possible
33 explanations are that (i) liquid permeability was underestimated due to fluid-rock interactions (ii)
34 gas permeability was overestimated due to insufficient correction for gas slippage and/or (iii)
35 gases and liquids do not probe exactly the same porous networks. The analysis of Knudsen
36 numbers shows that the gas permeability measurements were performed in conditions for which
37 the Klinkenberg correction is sufficient. Smaller samples had a larger scatter of permeability
38 values, suggesting that their volume were below the REV. The pressure dependence of
39 permeability was studied by some of the participating teams in the range 1 to 30 MPa and could
40 be fitted to an exponential law $k=k_o \cdot \exp(-\gamma P_{\text{eff}})$ with $\gamma=0.093 \text{ MPa}^{-1}$. Good practice rules for
41 measuring permeability in tight materials are also provided.

42

43 1. Introduction

44 Permeability is a property of a given porous medium which quantifies its ability to allow
45 fluid flow. Since the introduction of Darcy's phenomenological law (Darcy, 1856), permeability
46 characterization usually involves pressure gradient and flow measurements of a single fluid
47 phase. In the field, such measurements may only provide apparent permeability estimates for
48 rock masses including pore, crack and fracture networks which are usually saturated or partially
49 saturated with several fluids (Zinszner & Pellerin, 2007). Estimates of the single phase (or
50 intrinsic, or absolute) permeability (hereafter simply referred to as permeability) are typically
51 made by laboratory testing of core samples, following a saturation or a drying procedure for,
52 respectively, liquid or gas phase testing. In the case of liquids, fluid saturation can be assessed
53 from the evolution of poroelastic parameters such as the isotropic Skempton coefficient
54 (Makhnenko & Labuz, 2013) which is very sensitive to residual air, during a step by step back
55 fluid pressure increase to dissolve trapped air bubbles (Wild et al., 2015a). For tight rocks, the
56 sample preparation and saturation procedures can be particularly long and may disturb the
57 original pore network. For example saturation can change the equilibrium between solid and
58 fluid phases naturally present in clay rocks (Pearson et al., 2011; Wild et al., 2015b). The drying
59 procedure can also have dramatic effects in the presence of clay minerals, causing desiccation
60 cracks (Wild et al., 2015). Many observations have been made regarding perturbations and
61 modifications of rock properties due to sampling processes and stress release effects during
62 coring (Blümling et al., 2007; Schild et al., 2001).

63 Measuring the permeability of tight formations, which can potentially serve as seals for
64 nuclear waste repositories and/or strata for geological sequestration of CO₂, for instance, poses a

65 number of challenges. In addition to the traditional sources of errors that affect more permeable
66 formations (e.g., sample selection, non-representative specimens, disturbance introduced during
67 sample acquisition and preparation), rocks that are particularly tight and prone to solid-fluid
68 interactions can be more sensitive to the methods, procedures and techniques used to acquire
69 permeability data. In low permeability rocks, classical steady-state flow measurements may be
70 very difficult to perform because of slow variations of the measured quantities (pore pressure,
71 flow rate) and the long time needed for flow stabilization. Due to the long duration of flow
72 experiments, variations in external conditions (typically ambient temperature) may occur,
73 compromising the accuracy of permeability estimates. For this reason two other methods have
74 been developed: the transient pore pressure (or pulse) method and the pore pressure oscillation
75 method which is similar to a steady-state oscillatory method. The pulse decay method pioneered
76 by Brace et al. (1968) involves applying a pressure step increase in an upstream reservoir and
77 measuring the pressure variations with time in both upstream and downstream reservoirs
78 connected to the sample. As pressure diffusion occurs through the rock sample, permeability can
79 be estimated from decay of the differential pore pressure which follows a decreasing exponential
80 law. Further knowledge on transient pulse tests was gained from parametric analysis of pore
81 pressure diffusion processes in rocks by Hsieh et al. (1981) and Neuzil et al. (1981). The pulse
82 method has been widely and successfully used both in crystalline and shaly rock samples during
83 triaxial mechanical tests in the laboratory (Bourbie & Walls, 1982; Brace et al., 1968; Carles et
84 al., 2007; Selvadurai et al., 2005). Other transient methods, like the drawdown method or the
85 pressure build-up method (Martin, 1959) are particularly well adapted to use in the field in
86 boreholes (Bossart et al., 2002; Jakubick & Franz, 1993; Wassermann et al., 2011). Transient
87 methods can be applied step by step after re-equilibration periods during loading tests, providing
88 discrete measurements of permeability. Continuous measurements have been developed in order
89 to investigate loading effects on low permeability rocks and are more representative of the
90 evolution of in situ conditions during reservoir activities. Such methods are based on continuous
91 oscillatory flow and analysis of sinusoidal signals of pore pressure at both ends of a sample
92 through phase lag and amplitude ratio (Fischer, 1992; Kranz et al., 1990; Song & Renner, 2007).

93 All the above methods assume Darcy flow and more or less steady state conditions during
94 the measurements. The measured quantities in the pulse decay and steady-state flow experiments
95 are more sensitive to ambient temperature variations than the phase shift or amplitude ratio
96 continuously measured in the oscillatory method (Kranz et al., 1990). The pulse decay method
97 has the advantage of being relatively easy to perform but requires appropriate selection of the
98 reservoir volume compared to the pore volume of the tested sample (Hsieh et al., 1981).
99 Derivative techniques have been developed to face such issues linked to the experimental
100 apparatus (Lin, 1982; Trimmer et al., 1980). The oscillation technique also requires some
101 experimental adjustments concerning mainly: i) the frequency of the forcing pore pressure signal
102 — as pointed out by Song & Renner (2007), the frequency dependence of hydraulic properties
103 could be a way to define scaling parameters of the pore structure and ii) the peak-amplitude of
104 the forcing waveform which has to be small enough to avoid local poroelastic and fluid
105 compressibility effects. Sometimes it is also a technical challenge to maintain a sinusoidal
106 forcing signal. In addition to permeability, the oscillatory method allows estimation of other key
107 parameters such as diffusivity and specific storage capacity (Song & Renner, 2007).
108 Theoretically, permeability depends only on the pore structure of the material, and should be
109 independent of the nature of the pore fluid used for the measurement (Muskat & Wyckoff, 1937).
110 However, differences have been reported in the literature between water and gas permeability

111 measurements. Gas permeability estimations must take into account the gas compressibility, and
112 the measured permeability is an apparent value that needs to be corrected for the so-called gas
113 slippage effect: the “liquid equivalent” permeability can be obtained by applying the
114 Klinkenberg correction (Klinkenberg, 1941) to gas permeability measurements made at different
115 mean pore pressures. When the pore size is of the same order as the gas molecule mean free path,
116 additional corrections have to be done to account for Knudsen diffusion (Anez et al., 2014;
117 Ziarani & Aguilera, 2012). Whatever the fluid used, permeability measurements require also a
118 sufficiently low flow rate to avoid inertial effects, otherwise the Forchheimer correction has to be
119 applied (e.g. Rust & Cashman, 2004).

120 In order to bring a know-how about accurate transport property characterization in low
121 permeability material, we proposed a benchmarking exercise involving 24 laboratories around
122 the world using both direct (steady-state, transient, oscillatory) and indirect methods (pore and
123 crack network imagery, modeling) to study fluid flow. Such an extensive benchmarking effort in
124 rock physics has not been done before to our knowledge: the FEBEX benchmark study
125 numerically modeled the results of a fluid injection test in the Grimsel Test Site (Alonso et al.,
126 2005) and, within the framework of the SAFOD project, an inter-laboratory benchmark of
127 physical rock properties measurements involving 20 research organizations was organized
128 several years ago. In the SAFOD effort, measured rock properties were permeability, ultrasonic
129 wave speed, electrical resistivity, friction and strength; however very few of the participating
130 laboratories provided results, so that the outcome of this benchmark was never published
131 (Lockner et al., 2009).

132 The selected material for the present benchmarking exercise came from a single meter
133 scale rock volume in a well-known underground rock laboratory, the Grimsel Test Site (GTS) in
134 Switzerland. The GTS consists of several meter long galleries of 3.5 meters diameter excavated
135 at 450 m depth in 1983 in granite and granodiorite of the Central Aar massif in the Swiss Alps in
136 order to perform in situ experiments in the context of nuclear waste storage research in
137 Switzerland (Lieb, 1989). Here the objectives and organization of the benchmark will be
138 presented, and then the permeability measurements data set will be analyzed and discussed.

139

140 **2. The KG²B Project**

141 Following a workshop on «The challenge of studying low permeability materials» that
142 was held at Cergy-Pontoise University in December 2014, a benchmark in which several
143 laboratories would estimate the permeability of a single material was proposed to the attendees.
144 The material to be selected for this benchmark had to fulfill different criteria such as availability,
145 homogeneity and scientific interest. Several options were examined until finally we selected the
146 Grimsel granodiorite (Switzerland). The benchmark was named the “KG²B” project, which
147 derived from “K for Grimsel Granodiorite Benchmark” where K stands for the symbol of
148 permeability. Fresh cores from the Swiss Grimsel test site, an underground research laboratory in
149 hard rock, were drilled during the coring campaign of a scientific project funded through the
150 Swiss Competence Center of Energy Research – Supply of Electricity (SCCER-SoE), that was
151 aimed at performing a series of demonstration experiments at various scales (up to 1 km) to
152 support implementation of deep geothermal energy in Switzerland. From published work on the

153 Grimsel Granodiorite (Ota et al., 2003), we expected a porosity of about 0.7%, and permeability
154 in the range of 0.1 to $1 \cdot 10^{-18} \text{ m}^2$.

155 2.1. The Objectives and Organization of the Benchmarking Exercise

156 Multiple objectives were defined for the benchmark: (i) to compare the results for a given
157 method, (ii) to compare the results between different methods, (iii) to analyze the accuracy of
158 each method, (iv) to study the influence of experimental conditions (especially the nature of pore
159 fluid), (v) to discuss the relevance of indirect methods and models, and finally (vi) to suggest
160 good practice for low permeability measurements. Guidelines were given to the participants, in
161 which they were requested to follow a number of mandatory instructions: (i) permeability should
162 be measured along the same direction, (ii) permeability should be measured at 5 MPa effective
163 pressure (a pressure high enough to prevent leakage, small enough to minimize crack closure),
164 and (iii) rock samples should not experience any effective pressure higher than 5 MPa before the
165 permeability measurement was done. Effective pressure was assumed to be the difference
166 between confining and pore pressure: indeed experimental evidence was found to support this
167 statement (see section on pressure dependence). No recommendations or requirements were
168 made concerning the pore fluid, confining and pore pressures, sample size and method to be used
169 for estimating permeability. However we requested that all of this information be reported on a
170 results spreadsheet (David et al., 2017). The benchmark was designed as a “blind-test”: other
171 results were not shared with the participants until after they had submitted their own results. The
172 participants were also encouraged, once the permeability at 5 MPa effective pressure was
173 obtained, to study the pressure dependence of permeability, in particular by reproducing the in
174 situ stress conditions (estimated effective pressure 30 MPa). Any additional data reported was
175 also welcome, such as porosity values. This complementary data set is discussed in the
176 companion paper.

177 2.2. The Participants

178 When the benchmark was announced, 30 laboratories from 8 different countries
179 volunteered to participate. Three groups were forced to withdraw participation for different
180 reasons (experimental setup not available, technical problems, work overload), one group was
181 not able to provide the results in due time, and two others did not respond to our further
182 solicitations. Ultimately, we received results from 24 laboratories that form the “KG²B Team”.
183 The complete list of participants who sent their results is given in alphabetic order in Appendix
184 A. A dedicated website <https://labo.u-cergy.fr/~kggb/> was created, including in particular a web
185 page where the progress of the project could be followed on the so-called “KG²B-wheel” (David
186 et al., 2017) which was updated as soon as results were received from any of the participants. It
187 took one year to collect all the results. Participants were regularly sent updates on the benchmark
188 progress, to encourage those who had not yet sent their results.

189 2.3. The Selected Material

190 Two cores of Grimsel granodiorite, each about one meter long and 85 mm in diameter,
191 were provided by our Swiss colleagues in September 2015. These cores were retrieved at a
192 distance of 4 to 6 meters from the tunnel where the borehole has been drilled; the sampling region
193 was expected to be sufficiently distant from the fracture network of the excavation damage zone

194 (EDZ) and the cores were therefore assumed to be free of tunnel induced damage. The cores
195 were cut into small blocks at lengths requested by each participant (2 to 10 cm). Foliations are
196 visible on the cores, at an angle of about 20-30° with respect to the core axis. The foliation is
197 related to compositional banding of alternating dark biotite layers and quartz-rich layers (Schild
198 et al., 2001). A thorough microstructural study is presented in the companion paper: the minerals
199 identified are quartz, feldspars (albite, plagioclase), micas (muscovite, biotite) and apatite.
200 Before blocks were sent to the participants, a quality check was performed on each block by
201 the organizers at Cergy-Pontoise University. After drying the samples at 60°C for 24h, the P-
202 wave velocity was measured at room conditions in three orthogonal directions. We observed that
203 the P-wave velocity in the core axis direction increased with the distance from the tunnel, and
204 decreased slightly with distance in the radial direction perpendicular to the foliation (David et al.,
205 2017). This trend may result from mineralogical changes along the borehole or from the
206 persistent influence of the excavation damage effects. A significant P-wave velocity anisotropy
207 was found, due to the foliation inclination with respect to the core axis. It was necessary to
208 require that all participants make permeability measurements in a common direction. This
209 common direction was chosen for convenience as the core axis direction. Some laboratories
210 performed additional permeability measurements in other directions, thus providing insight into
211 the permeability anisotropy in the Grimsel granodiorite (see the discussion in the companion
212 paper). The main result of this quality check was that reproducibility is acceptable.

213

214 **3. Permeability Measurements at Constant Effective Stress**

215 We will use the following convention for presenting the data set. Each lab was assigned a
216 number in increasing order based upon the distance between their sample and the borehole
217 mouth. Lab#01 worked on the sample closest to the borehole mouth (i.e. closest to the tunnel
218 wall), and Lab#24 on the farthest sample (i.e. the deepest from the tunnel wall).

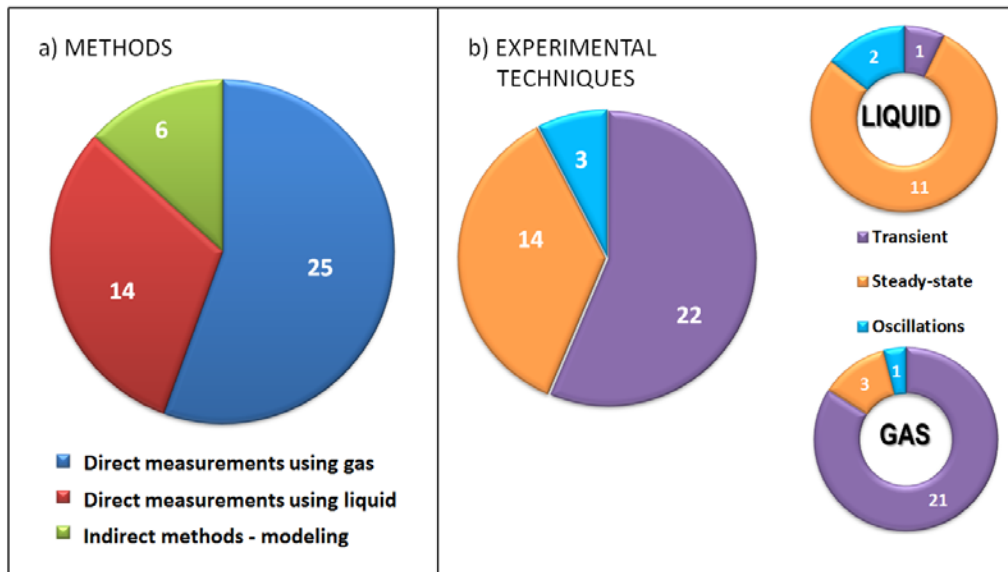
219 In the following analysis, the number of results is larger than the number of laboratories
220 in the KG²B team for several reasons: (i) some laboratories tested several small samples sub-
221 cored from the original core, (ii) some laboratories made different kinds of measurements on a
222 single sample. Before permeability measurements were made, the samples were systematically
223 dried before being saturated with the working fluid. Our benchmarking exercise specifically
224 excludes two-phase flow and relative permeability estimation which, although important, imply
225 higher order of complexity.

226

227 **3.1. General Characteristics of the Data Set**

228 In Table B1 of Appendix B we report the location of each sample (distance from the
229 tunnel), the size of the sub-cored samples on which permeability was measured, the method
230 applied and the fluid used to conduct the measurements. In total we collected 45 permeability
231 values from measurements (39) and modeling (6). Most of the results (56%) come from direct
232 measurements using gas as the pore fluid (Figure 1a), about 31% of the results come from direct
233 measurements using liquids (mostly water) as the pore fluid, and only six results (about 13%)

234 were collected from models using microstructural data to predict the permeability. Here we will
 235 only consider the experimental data set, while the modeling data set will be presented in the
 236 companion paper. Figure 1b summarizes the techniques which were used.

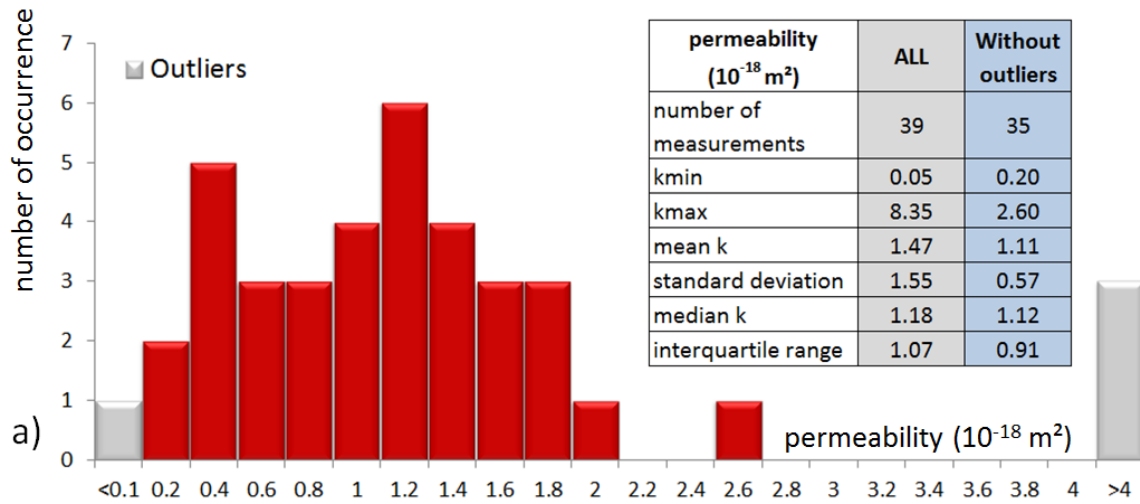


237
 238 **Figure 1.** a) Methods used in the benchmark and b) techniques used for the experiments, global distribution (left)
 239 and distribution by working fluid type (right).

240 Regardless of the nature of the pore fluid, the transient technique was the most used (56%)
 241 followed by the standard steady-state method (36%), a direct application of Darcy's law. A few
 242 laboratories used the pore pressure oscillation technique (8%). The distribution is, however, very
 243 different if one takes into account the fluid used to measure the permeability. In experiments
 244 with liquid as the working fluid, 78% of the results were obtained with the steady-state
 245 technique. In contrast, with gas as the working fluid, only 12% of the results were obtained with
 246 the steady-state technique and 84% of the results come from the pulse transient technique.
 247 Various gases were used: mostly nitrogen, but also argon, helium and air.

248 3.2. Statistical Analysis of the Raw Data Set

249 The complete data set is reported in Table B2 of Appendix B. Here we present the
 250 statistical analysis for all measured permeability values at 5 MPa effective pressure in the core
 251 axis direction only (no anisotropy effect included), regardless of the method, pore fluid or sample
 252 size (Figure 2). For the complete data set, permeability ranges between 0.05 and $8.35 \cdot 10^{-18} \text{ m}^2$,
 253 with an average value of $1.47 \cdot 10^{-18} \text{ m}^2$ and a high standard deviation of $1.55 \cdot 10^{-18} \text{ m}^2$. We
 254 identified four outliers (three in the last bin, one in the first bin in Figure 2) with permeability
 255 higher or lower by a factor three than the average permeability. It is, consequently, preferable to
 256 use the median ($1.18 \cdot 10^{-18} \text{ m}^2$) and interquartile range ($1.07 \cdot 10^{-18} \text{ m}^2$) as estimators of the
 257 permeability statistics. If we remove the four outliers, we find a range of permeability spanning
 258 one order of magnitude (between 0.20 and $2.6 \cdot 10^{-18} \text{ m}^2$) with a mean value of $1.11 \cdot 10^{-18} \text{ m}^2$ close
 259 to the median ($1.12 \cdot 10^{-18} \text{ m}^2$), and a standard deviation significantly reduced ($0.57 \cdot 10^{-18} \text{ m}^2$).
 260 These values are in good agreement with the Grimsel granodiorite values reported by (Schild et
 261 al., 2001).



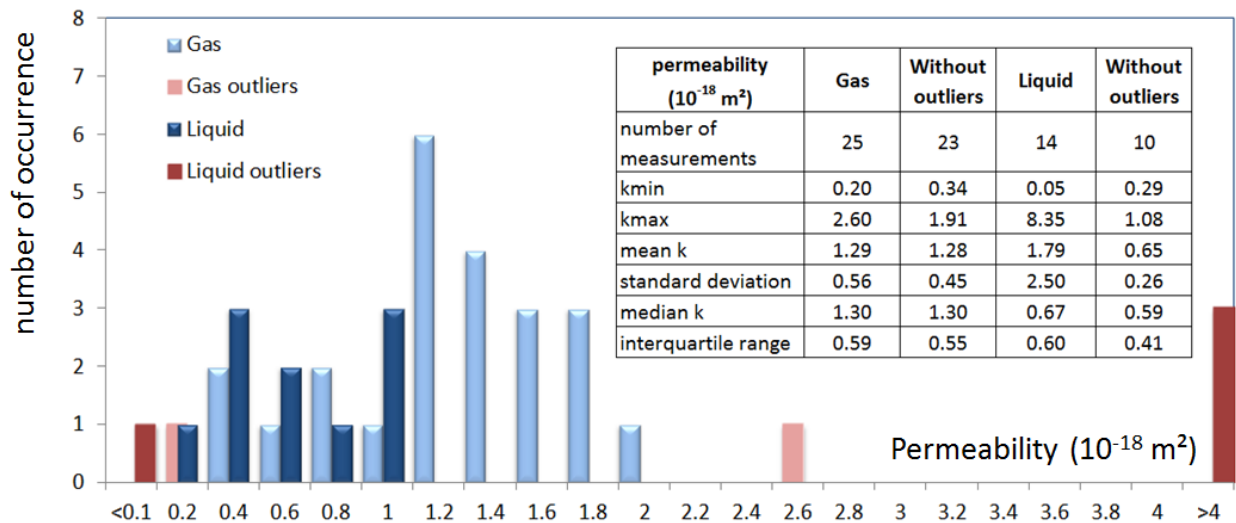
263

264 **Figure 2.** Statistical distribution of the raw data set for all methods, fluids and sample size. Each label gives
 265 the central value of each bin (e.g. bin 0.2 corresponds to the range [0.1, 0.3]).

266 **3.3. Influence of the Pore Fluid**

267 Considering only the measurements in the core axis direction (“axial” flow), the 39
 268 permeability values at 5 MPa effective pressure (Figure 2) can be divided into two sub-sets based
 269 on the nature of the pore fluid (gas or liquid) used to measure the permeability. The statistical
 270 distribution of these two subsets exhibits two overlapped distributions (Figure 3) and defines
 271 possible new outliers for the gas group: 2.6 10⁻¹⁸ m² (extrapolated value and poorly defined
 272 stresses) and 0.2 10⁻¹⁸ m². The influence of these possible gas outliers is low.

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Figure 3. Statistical distribution of measured permeability values as a function of the working fluid.

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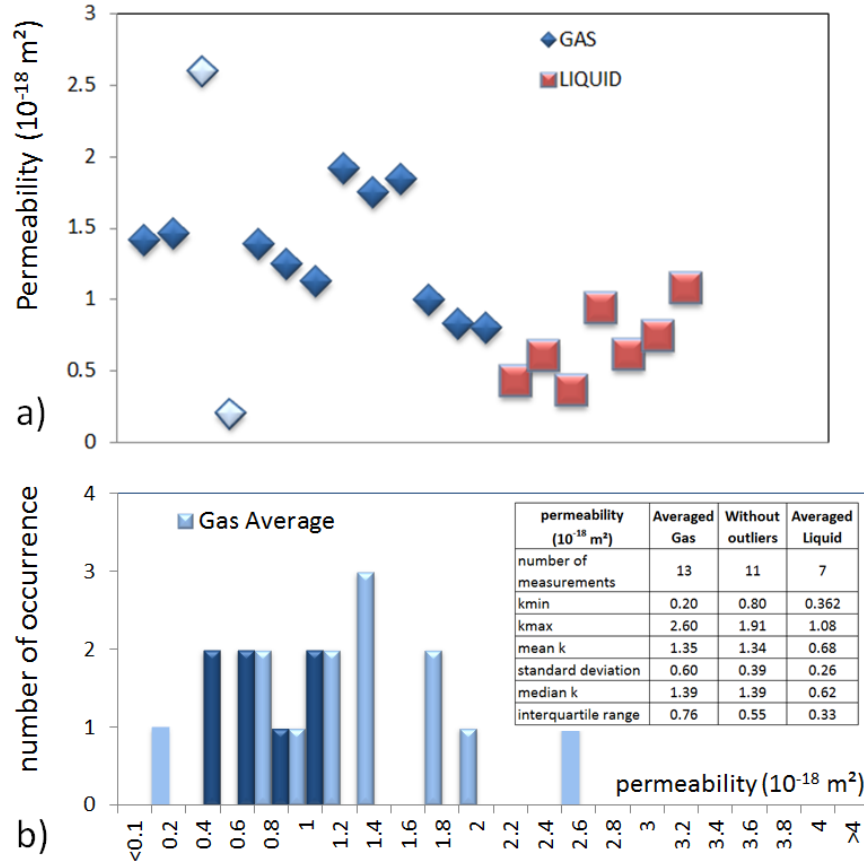
A first analysis shows that the statistical parameters of the gas sub-set are about twice those of the liquid sub-set without outliers. Whereas the lowest outlier value seems to reflect to a

279 bad measurement, the higher ones probably owe to anomalous samples.

280 Permeability measurements using gas and liquid on a single sample were carried out by
281 two labs with the following results: 1.3 & 0.94 10^{-18} m² @5MPa (Lab#09) and 34 & 28 nD
282 @27MPa (Lab#23) respectively, giving a 1.3 ratio between gas and liquid permeability values.
283 In both cases, the gas pressure was higher than 1.7 MPa and the Klinkenberg correction should
284 be small. However successive permeability measurements on a single sample induce a complex
285 history of loading and unloading that could generate irreversible changes in the porous space and
286 therefore variations in measured permeability values.

287 Each team described the protocol used to saturate the porous space of the tested samples.
288 There is no basis for associating low permeability values with incomplete saturation or to low
289 pore pressures. Each lab which used a low gas pressure, attempted accurate evaluation of the
290 Klinkenberg slip factor *b*.

291
292 Some labs provided several values of permeability by using the same sample with
293 different gases, pore pressures and confinement pressures at constant effective pressure. Some
294 labs used several sub samples from their initial piece of core. To remove the large weight of
295 repetitive measurements, a single value for each lab was obtained by replacing multiple values
296 by their average. The reduced data set contains 20 permeability values. Removing two outliers in
297 the gas subset (light blue diamonds), the set of data was further reduced to 18 values: 11 for the
298 gas subset and 7 for the liquid subset (Figure 4).



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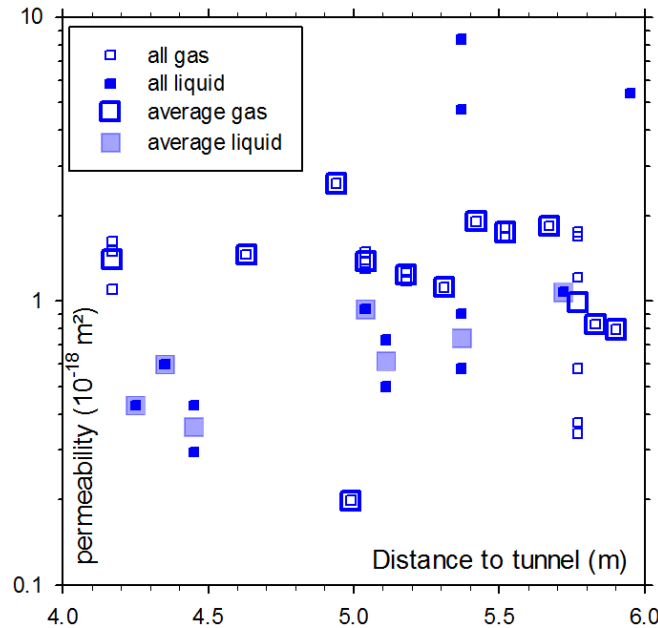
Figure 4. a) Average permeability values at a 5MPa effective pressure for each lab (data are ordered by lab number) and b) statistical parameters for these average values.

302 The overlapping of gas and liquid sub-sets is now reduced and this new representation of
 303 the data clearly confirms that measured gas permeability values tend to be larger than liquid
 304 permeability values by a factor of about two. This significant difference leads us to separate both
 305 subsets in the following discussion.

306 3.4. Influence of the Distance to the Tunnel

307 The permeability as a function of distance to the tunnel (Figure 5) shows no clear trend.
 308 David et al. (2017) reported P-wave velocity measurements at room temperature and unconfined
 309 conditions on the original core samples: they observed that P-wave velocity in the core axis
 310 direction increases with distance from the tunnel. The P-wave velocity increases linearly from
 311 ~ 4200 m/s at a distance of 4.2 m to ~ 4600 m/s at 5.7 m, and then jumps to a value close to 4800
 312 m/s from 5.7 to 6 m. Between 4.2 and 5.7 m, the average gas permeability seems to be relatively
 313 constant, whereas the average liquid permeability seems to increase slightly. The observed trend
 314 in P-wave velocity may result from changes in mineralogy or foliation orientation along the
 315 borehole. This trend could also be linked to a varying Borehole Damage Zone which induced
 316 different stress release patterns from the tunnel to the deepest part of the borehole. Between 5.7
 317 m and 6 m, the average gas permeability seems to decrease, which could be correlated with the
 318 observed 200 m/s jump in P wave velocity. Both observations could reflect a preexisting crack
 319 density (Fortin et al., 2011), which is lower for the samples located between 5.7 and 6 m.

320 However, only 3 samples are located in this interval, so that we cannot draw any firm conclusion
321 on the effect of sample location (distance to tunnel) or porosity (see companion paper).



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Figure 5. Measured axial permeability vs. distance to the tunnel from which the long cores were drilled.

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3.5. Influence of the Sample Size

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No recommendations were made with respect to sample size for permeability estimation. Most of the laboratories chose to sub-core the provided material (Table B1) and prepared small samples (volume $\sim 20 \text{ cm}^3$, length 2-5 cm) in order to reduce the time required for completing the permeability measurements (David et al., 2017). Two laboratories decided to work directly on the original cores without sub-coring, whereas one group used a special device designed to work on tiny samples or chips (Lenormand et al., 2010). Permeability measurements with gas (open symbols) or liquid (solid symbols) are plotted in Figure 6a as a function of sample length in the direction of flow. As mentioned previously there is a wide range of permeability values (about two orders of magnitude), and the scatter appears larger for smaller samples, while little variation is found for the longest ones. This size dependence can be linked to the magnitude of the Representative Elementary Volume (REV), which may be large in the case of the Grimsel granodiorite because the grain size can be up to 2 cm (Schild et al., 2001). In Figure 6b a density map obtained from micro-CT scanning reveals the size of mineralogical heterogeneities. Although the density map is not necessarily correlated with pore network heterogeneity, one may expect an REV size larger than a few centimeters.

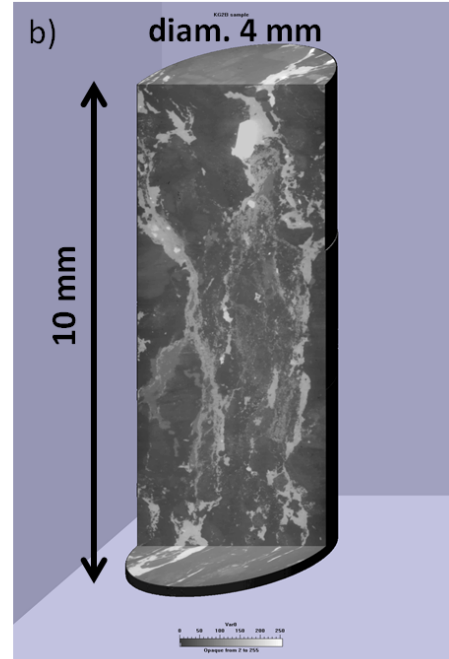
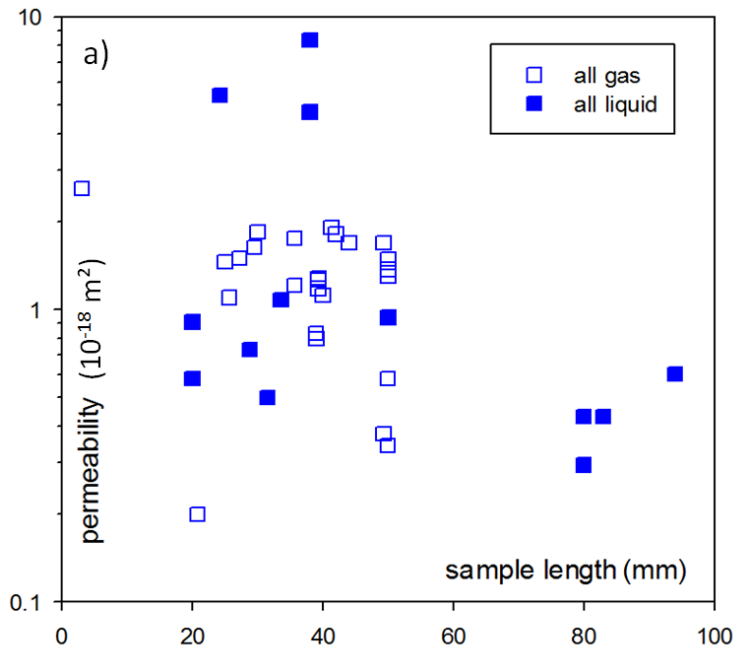


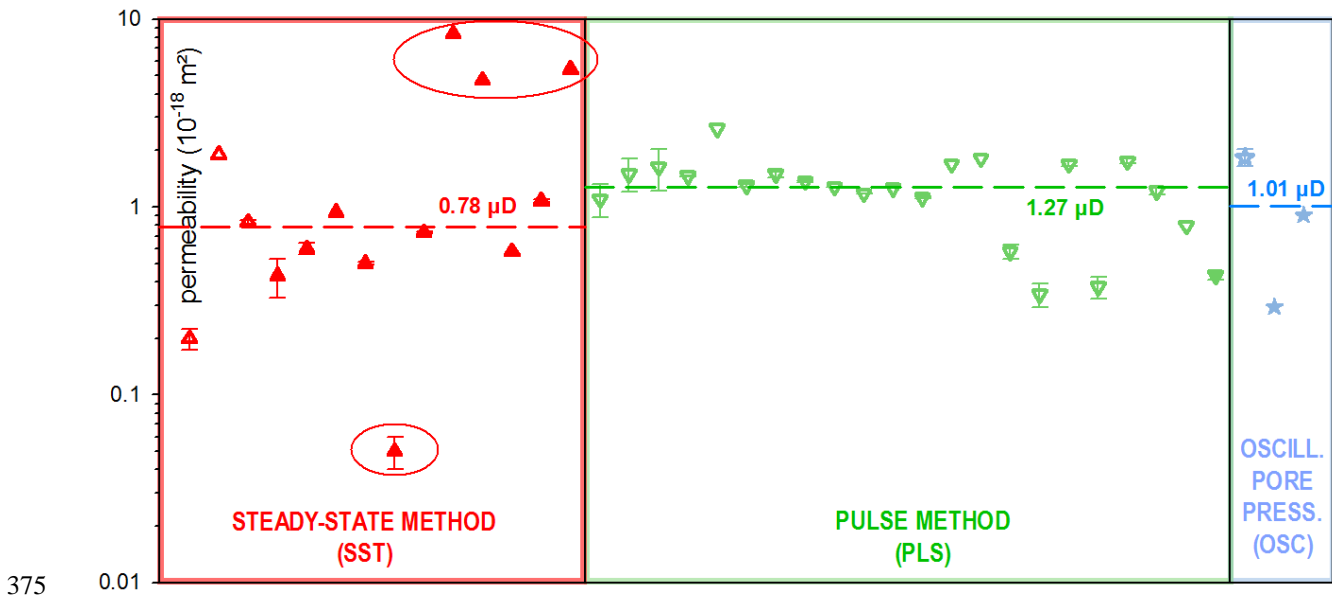
Figure 6. a) Measured axial permeability values vs. sample length in the flow direction. b) 3D reconstruction from micro-CT images obtained on a small volume of Grimsel granodiorite (voxel size 5 μm).

The sample length L has also a significant influence on the time required to establish steady-state flow conditions. Assuming no Klinkenberg effect, the duration of the transient scales as $(\mu C_f \phi / k) L^2$ with ϕ the porosity, k the permeability, C_f the fluid compressibility and μ the fluid viscosity. In addition there is a non-linear effect of mean pore pressure that can be quantified using numerical simulation. Taking $k=1.0 \cdot 10^{-18} \text{ m}^2$ and $\phi=1\%$ for the Grimsel granodiorite, the transient time at low fluid pressure ($< 1 \text{ MPa}$) is about 15 seconds for a one centimeter long sample, but increases to about 30 minutes for a ten centimeter long sample. At higher fluid pressure ($\sim 10 \text{ MPa}$) the transient time is shorter, about 2 minutes for a ten centimeter long sample. As stability is generally obtained after a few minutes, due to the relatively high permeability and low porosity, the transient is not likely to be a source of error for the KG²B measurements. However for a sample in the nanodarcies range (10^{-21} m^2), the stabilization time for a similar measurement at low pressure on a 10 cm plug would be multiplied by 1000 (20 days!).

3.6. Influence of the Experimental Method

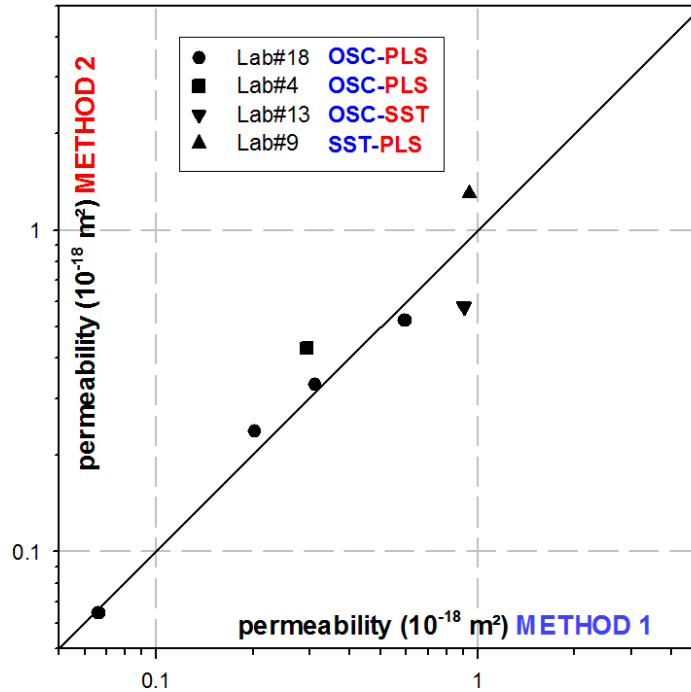
Steady-state measurements with gas were systematically corrected for the Klinkenberg effect. Not all transient measurements with gas were corrected for the Klinkenberg effect when the pore pressure was higher than 3 MPa. Several transient techniques were used to extract permeability from the recorded pressure decay data, including the standard transient pulse approximation (Brace et al., 1968), a complex transient inversion scheme that additionally provides specific storage (based on (Kranz et al., 1990)) and the more recent step decay method that provides the intrinsic permeability, porosity and Klinkenberg coefficient for gas

365 measurements in tight rocks (Lasseux et al., 2012). In Figure 7 the data set is divided into three
 366 groups according to the method used for measurements (SST for steady-state, PLS for transient
 367 pulse, and OSC for pore pressure oscillation). For each sub-group we also separate gas (open
 368 symbols) and liquid (solid symbols) measurements. For each subgroup, we estimated an average
 369 permeability value, omitting outliers. The average value for the transient pulse method ($1.27 \cdot 10^{-18}$
 370 m^2) is the highest, and that for the steady state method is the smallest ($0.78 \cdot 10^{-18} \text{m}^2$). For the
 371 pore pressure oscillation method, there are only three values available, so it is difficult to draw
 372 any statistically meaningful conclusion. Notice however that there is a great overlap between the
 373 division in terms of testing and the division in terms of working fluid (SST mostly liquids and
 374 PLS mostly gases).



375
 376 **Figure 7.** Measured axial permeability for the different techniques used with error bars when known (data
 377 points are ordered by lab number in each group). Open symbols correspond to measurements using gas. Dashed
 378 lines correspond to the average permeability value per method (without the outliers highlighted by ellipses).

379 To address this problem, we analyzed results provided by four teams who measured
 380 permeability on the same sample using different methods but the same pore fluid, sometimes at
 381 different pressure conditions (Lab#18). Figure 8 is a cross-plot of permeability using one
 382 particular method vs. permeability using another one. For this limited set of measurements, the
 383 permeability values are such that $k_{\text{SST}} < k_{\text{OSC}} < k_{\text{PLS}}$ which is the same order derived from
 384 statistical analysis on the three subgroups (Figure 7).



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Figure 8. Comparison of permeability values for the same sample under the same pressure condition but using two different techniques. OSC = oscillating pore pressure method; SST = steady-state flow method; PLS = transient pulse method.

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3.6.1. Example of Steady-state Flow Method for Permeability Determination

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[Contribution of Lab#19] Permeability was measured on a section of whole core using the steady-state flow technique at a series of effective pressures and pore pressure gradients. Normally, sub-cores would be prepared from the original samples for testing. However, since the starting material had relatively large grain size, we decided that the best determination of average permeability would be obtained if the entire sample were tested. In this case, the core circumference, which was smooth and even, was used without modification and faces were ground flat and parallel using a diamond wheel on a surface grinder. The resulting sample, shown in Figure 9a, had physical dimensions of diameter = 8.3348 ± 0.0008 cm and height = 3.3617 ± 0.0005 cm.

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Stainless steel end caps with 1.5 mm-diameter center holes and groove patterns on faces were placed on either end of the sample. Fine mesh stainless steel screen was placed between end caps and the sample to provide uniform pore pressure on sample faces. Shrink tubing covered the sample + end cap assembly which was then cast in 2-part polyurethane (approximately 5 mm thickness) to isolate the sample from the silicone oil confining fluid (Figure 9b). A coiled stainless return tubing provided pore fluid access to the bottom end cap.

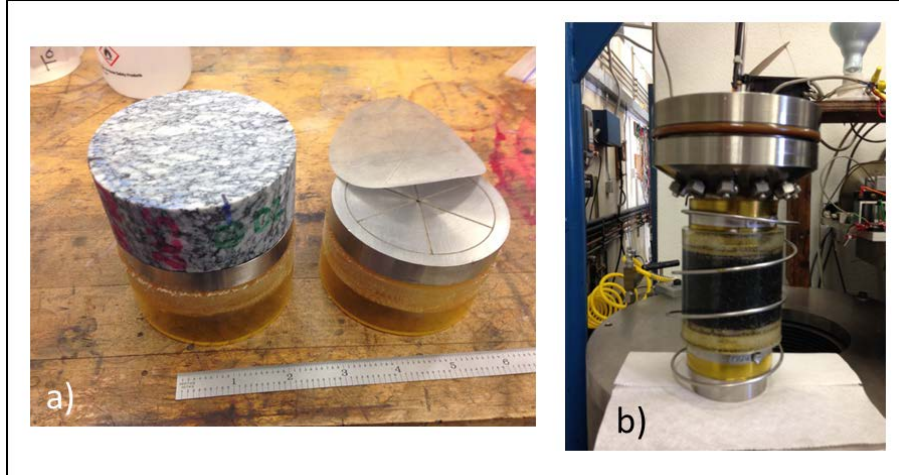


Figure 9. a) Whole core KG²B test sample with stainless steel end caps and stainless screen that assures uniform pore water access to sample faces. b) Sample assembly, including coiled return pore fluid line, is jacketed and ready to place in the pressure vessel.

This assembly was placed in a pressure vessel and an initial confining pressure, P_c , of 2 MPa was applied. The pore pressure system was evacuated and then pressurized to $P_p = 1$ MPa with deionized water. P_c and P_p were then increased together to assure that effective pressure ($P_{eff} = P_c - P_p$) never exceeded 2 MPa. The low-aspect-ratio cracks that provide flow paths in this crystalline rock are sensitive to effective pressure and have memory of past pressure history. Therefore, it is important that the target test pressure is not exceeded during sample pressurization.

Evacuation of the combined sample + pore pressure system, prior to saturation, is important in a low porosity sample to prevent air bubbles that would alter the fluid flow paths being trapped in the pore space. Conducting permeability tests at elevated pore pressure further reduces the risk of spurious measurements by compressing and dissolving remnant air bubbles that might remain trapped in pore space. The sample assembly in the pressure vessel, as well as the pore pressure pump and flow rate sensor were enclosed in a temperature-controlled chamber that maintained $23.5 \pm 0.1^\circ\text{C}$. For steady flow tests in low permeability samples, variations in ambient temperature can become the primary source of uncertainty in determining permeability, since room temperature changes produce fluid volume fluctuations that appear as transients in flow rate (Morrow et al., 2014, Figure A1)

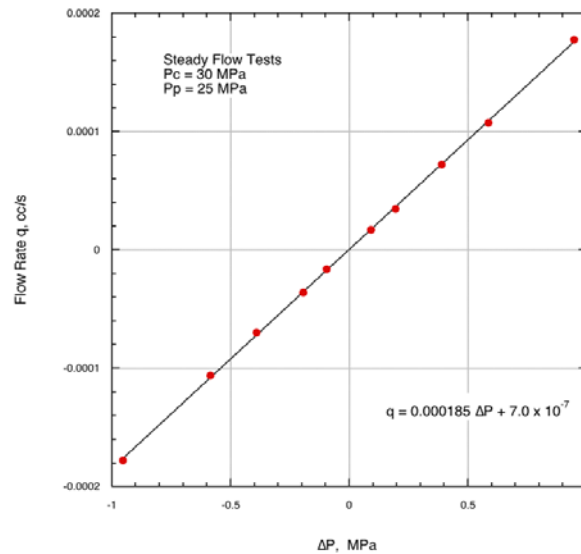
Confining pressure, up-stream (P_{UP}) and down-stream (P_{DOWN}) pore pressure were independently computer controlled. Reported effective pressure is defined as $P_{eff} = P_c - P_p = P_c - (P_{UP} + P_{DOWN})/2$. Pressure drop across the sample is just $\Delta P = P_{UP} - P_{DOWN}$. The pore pressure generator on the up-stream side of the sample recorded the change in pore volume (V_p) needed to maintain constant pressure with a precision of 10^{-5} cm^3 . This volume change was used to determine flow rate, $Q = dV_p/dt$. Once the pressure drop across the sample is established and a steady state flow condition is attained, permeability k can be calculated from Darcy's law

$$k = \left(\frac{\mu L}{A}\right) Q / \Delta P \quad (1)$$

442 where μ is dynamic viscosity and L and A are the length and cross-sectional area of the sample.
 443 Dynamic viscosity of water at 23.5°C is 0.921×10^{-3} Pa.s and L/A for this sample is 0.06161 cm^{-1} .
 444 As an example, a flow measurement at $P_c = 30$ MPa, $P_{eff} = 5$ MPa and $\Delta P = 0.946$ MPa resulted
 445 in $k = 1.068 \times 10^{-18} \text{ m}^2$.

446 This reported permeability is of limited use without estimating errors. We next consider
 447 different methods for determining confidence intervals for the permeability measurements. The
 448 first method is to take multiple measurements of k and compute a mean value and standard error.
 449 We performed a series of 10 flow tests at $P_{eff} = 5.0$ MPa and at varying pore pressure gradients
 450 that produced both forward and reverse flow (Figure 10). If we assume that the individual
 451 determinations of k are random samples from the same distribution, and the ‘true’ permeability is
 452 represented by the mean of the distribution, then uncertainty can be expressed as standard error
 453 of the measurements. In this case, permeability, based on ten measurements, is $k = (1.04 \pm 0.01)$
 454 $\times 10^{-18} \text{ m}^2$.

455



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458 **Figure 10.** Permeability tests at $P_{eff} = 5.0$ MPa. Flow rate is a linear function of pressure gradient, ΔP , and using
 459 equation (1) gives $k = (1.04 \pm 0.01) \times 10^{-18} \text{ m}^2$.

460

461 An equivalent method is to compute a least squares fit to the data in Figure 10 to provide the
 462 ratio $Q/\Delta P$. The result, including a calculation of the formal error is $\partial q/\partial \Delta P = 0.00185 \pm 0.00001$
 463 cc/MPa, and applying equation (1) leads to a similar estimate of permeability.

464 A more complete measure of uncertainty includes consideration of errors in all of the variables
 465 on the right-hand side of equation (1). In this case, an uncertainty of $\pm 0.1^\circ\text{C}$ for the
 466 environmental chamber implies an uncertainty of $\pm 0.002 \times 10^{-3}$ Pa.s in viscosity (*i.e.*, $\pm 0.2\%$).
 467 Uncertainties in physical sample dimensions (L/A) are estimated to be only $\pm 0.03\%$ and errors in
 468 ΔP are 0.001 MPa. Taken together, these contribute uncertainty of approximately $\pm 0.5\%$ in
 469 computing k . The final quantity to consider is the flow rate Q . The displacement transducer that
 470 measures V_P has a linearity of about 0.05% which limits the accuracy of determining Q . During a
 471 permeability test, V_P is sampled ten times per second and averaged at 1 s intervals. If we assume
 472 that the errors in measuring V_P are uncorrelated, we can divide the total measurement time into N
 473 intervals of equal length and estimate a Q_i for each interval. Then, the mean of the Q_i 's provides
 474 an estimate of Q and the standard error provides an estimate of the uncertainty in Q . Using this

475 approach, with the standard recording interval used in this study of approximately 2500 s,
 476 accuracy in determining q was typically $\pm 0.5\%$. Taken together with the uncertainties in the
 477 other variables, we estimate a total uncertainty in determining k of approximately 1%. This is in
 478 close agreement with the confidence interval quoted above and probably represents the best
 479 accuracy that we can expect to achieve with the current test system. Notice that the two largest
 480 contributions to uncertainties in determining permeability are variations in ambient temperature
 481 and uncertainties in determining Q . Some improvement in accuracy can be gained by sampling
 482 flow rate over longer intervals. However, accuracy in Q will improve, at best, as $(\text{time})^{1/2}$ and
 483 fluctuations in ambient temperature will ultimately limit accuracy. The importance of controlling
 484 ambient temperature cannot be overstated. The viscosity of water decreases by about $0.02 \cdot 10^{-3}$
 485 Pa.s/ $^{\circ}\text{C}$, so an error of 1°C in measurement temperature will result in a 2% error in calculated
 486 permeability.

487 488 489 3.6.2. Example of Transient Method for Permeability Determination

490
491 [Contributions of Lab#11 and Lab#12] The transient (or pulse) method (Bruce et al.,
 492 1953) consists of instantaneously applying a pulse of differential pore fluid pressure across the
 493 sample that will re-equilibrate with time by fluid flow through the sample. An interpretative
 494 model was reported later by (Brace et al., 1968). The permeability is derived from the time-
 495 dependent decrease of the upstream pore fluid pressure $P_{UP}(t)$, which can be approximated by an
 496 exponential law:

$$497 \quad P_{UP}(t) - P_{\infty} \propto e^{-\alpha t} \quad \text{with} \quad \alpha = \frac{kA}{\mu C_f L} \left(\frac{1}{V_{UP}} + \frac{1}{V_{DOWN}} \right) \quad (2)$$

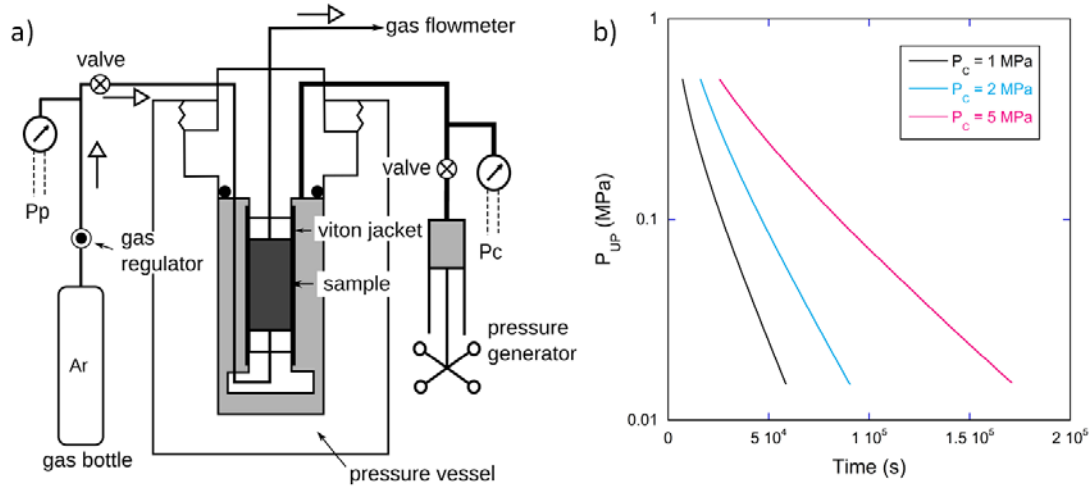
499 where P_{∞} is the final upstream pressure, k the permeability, L and A the sample length and cross-
 500 section area respectively, μ the fluid viscosity, C_f the fluid compressibility and V_{UP} and V_{DOWN}
 501 the volume of the tanks connected to the upstream and downstream end of the sample
 502 respectively. The permeability of a Grimsel granodiorite sub-core (length = 40.10 mm; diameter
 503 = 19.74 mm) was measured in a high pressure vessel (maximum confining pressure 50 MPa) at
 504 room temperature using water as the confining medium and argon as pore fluid (Figure 11a).
 505 Prior to experiments, the sample was vacuum dried at 40°C for 48 hours, a period beyond which
 506 no additional mass decrease was recorded. To apply the confining pressure to the sample and
 507 avoid any leak, the sample was inserted in a rubber sleeve clamped onto end-pieces.

508 The decay through time of the upstream gas pressure P_{UP} is monitored whereas the downstream
 509 gas pressure P_{DOWN} is kept constant at atmospheric pressure P_{ATM} , that is $P_{\infty} = P_{DOWN} = P_{ATM}$
 510 and the term $1/V_{DOWN}$ of equation (2) can be neglected. This configuration was shown to be the
 511 optimal one for a pulse-decay experiment (Jannot et al., 2007). Since the experiments are run at
 512 constant temperature ($T = 20^{\circ}\text{C}$) and low gas pressure (≤ 0.5 MPa), one may also assume that the
 513 gas compressibility C_f can be approximated by $C_f = 1/P_{MEAN}$, where $P_{MEAN} = (P_{UP} + P_{DOWN})/2$ is
 514 the mean gas pressure in the sample. Then, by calculating the time derivative of equation (2), one
 515 obtains the following equation relating the gas permeability k_{gas} to the evolution of the upstream
 516 gas pressure:

$$517 \quad k_{gas} = \frac{\mu_{Ar} L}{A} \frac{V_{UP}}{P_{MEAN} \Delta P} \left| \frac{dP_{UP}}{dt} \right| \quad (3)$$

518
519
520

521 where μ_{Ar} is the viscosity of Argon ($= 2.21 \cdot 10^{-5}$ Pa.s at 20°C) and $\Delta P = P_{UP} - P_{DOWN}$ is the gas
 522 pressure difference across the sample. Due to the narrow flow path in such a low permeability
 523 rock, the Klinkenberg correction must be carried out (see the section devoted to slip flow). Three
 524 confining pressure levels were tested: 1, 2 and 5 MPa. For each pressure level, the sample was
 525 allowed to rest in the cell for one night. The upstream inlet gas pressure is then increased to 0.5
 526 MPa, the outlet downstream gas pressure is maintained constant at atmospheric pressure and the
 527 outlet gas volume flow rate Q_{DOWN}^V is measured. Once the latter has stabilized, the inlet valve is
 528 closed and the upstream gas pressure is allowed to decrease. In Figure 11b, we show the pressure
 529 decay curves obtained at the various confining pressure levels. Note that in contrast to
 530 experiments using liquids (with constant compressibility), the pressure decay is not linear on the
 531 semi-log plot as expected from equation (2): this is so because the gas compressibility depends
 532 on the mean pressure which decreases with time.
 533
 534



535
 536 **Figure 11.** a) Experimental setup for measuring gas permeability in rock samples stressed up to 50 MPa
 537 hydrostatic confining pressure. b) Pressure decay curves on a semi-log plot obtained at confining pressures P_C of 1,
 538 2 and 5 MPa respectively.
 539

540 The time derivative of the pressure decay curve dP_{UP}/dt is calculated by applying a moving
 541 linear regression to the pressure decay curve over a constant number of points. The volume V_{UP}
 542 of the upstream gas circuit is either calibrated independently or by using the equation relating the
 543 outlet gas volume flow rate and the upstream pressure decay rate. At the upstream side of the
 544 sample, the inlet gas mass flow rate Q_{UP}^M is equal to:
 545

$$546 \quad Q_{UP}^M = \frac{M}{RT} V_{UP} \left| \frac{dP_{UP}}{dt} \right| \quad (4)$$

547
 548 where M is the molar mass of the gas, R is the universal gas constant, and T is the temperature.
 549 Assuming that the gas mass stored in the sample can be neglected, we have $Q_{UP}^M = Q_{DOWN}^M$, where
 550 Q_{DOWN}^M is the outlet gas mass flow rate. The latter quantity can be expressed as a function of the
 551 measured outlet gas volume flow rate Q_{DOWN}^V :
 552

$$553 \quad Q_{DOWN}^M = \frac{M}{RT} P_{DOWN} Q_{DOWN}^V \quad (5)$$

554

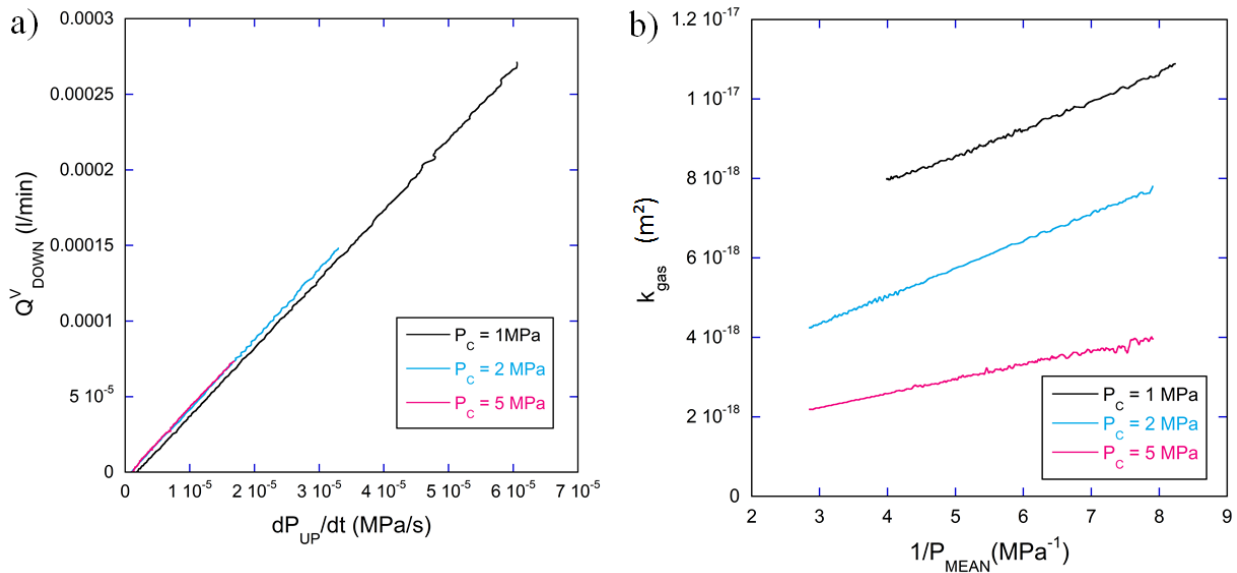
555 Combining equations (4) and (5) yields a relationship between the outlet gas volume flow
556 rate and the upstream gas pressure decrease rate that allows calculation of the upstream volume
557 V_{UP} :

558

559
$$Q_{DOWN}^V = \frac{V_{UP}}{P_{DOWN}} \left| \frac{dP_{UP}}{dt} \right| \tag{6}$$

560

561 In Figure 12a, the linear relationship between outlet flow rate and inlet pressure time-derivative
562 is shown for all three confining pressure levels. Assuming a constant atmospheric pressure
563 P_{DOWN} , the volume of the upstream gas circuit is derived from the slope of this linear relationship
564 following equation (6). Then the apparent gas permeability k_{gas} is estimated from equation (3) as
565 a function of time and finally Klinkenberg's correction is applied to the data set in order to derive
566 the true permeability k (see section 4.2). In Figure 12b, the gas permeability k_{gas} is plotted vs.
567 inverse mean gas pressure $1/P_{MEAN}$ for the three tested confining pressures. As one can see, the
568 linear trend is rather good, confirming that Klinkenberg's correction has to be applied. The true
569 permeability k is then taken as the intercept of the best linear fit to each curve, and the
570 Klinkenberg slip b factor is inferred from the slope. From the plot in Figure 12b the following
571 results are obtained: at 5 MPa confining pressure (effective confining pressure between 4.5 and 5
572 MPa close to the KG²B pressure target) $k=1.12 \cdot 10^{-18} \text{ m}^2$ and $b=0.33 \text{ MPa}$; at 2 MPa confining
573 pressure $k=2.26 \cdot 10^{-18} \text{ m}^2$ and $b=0.30 \text{ MPa}$; at 1 MPa confining pressure $k=4.99 \cdot 10^{-18} \text{ m}^2$ and
574 $b=0.15 \text{ MPa}$. The last point is to check the reproducibility, and hence the accuracy, of the
575 measurements. This has been done by doubling each measurement at each confining pressure
576 level. After the first measurement, the sample was allowed to rest at the prescribed confining
577 pressure for one night, before repeating the above mentioned procedure. Repeatability is very
578 good, with permeability variations lower than 2%.
579



580

581

582 **Figure 12.** a) Linear relationship between outlet flow rate and inlet pressure decrease obtained at a confining
583 pressure P_c of 1, 2 and 5 MPa respectively. b) Evolution of gas permeability k_{gas} with mean gas pressure for the
584 three tested confining pressure levels.

585

586 The well-known and widely employed transient method based on the pulse decay technique
587 described above has been extended recently (Lasseux and Jannot, 2011; Lasseux et al., 2012).
588 The main purpose for the development of this new method is to avoid repeating several
589 experiments at different values of P_{MEAN} to determine permeability k and slippage factor b . One
590 could consider using an inverse technique applied to the complete unsteady flow model for the
591 pulse-decay instead of the approximated analytical model of equation (1) so that these two
592 parameters (along with porosity ϕ) could be identified on a single upstream pressure decay
593 $P_{UP}(t)$ in the least square sense (Jannot et al., 2008). However, the sensitivity of the signal to
594 these parameters were shown to be insufficient in the general case for a reliable identification
595 (Lasseux et al., 2012). With the step-decay method, a downstream tank is introduced and both
596 $P_{UP}(t)$ and $P_{DOWN}(t)$ are recorded, the former being taken as the input for the history matching
597 that is carried out on the latter considered as the response. While $P_{UP}(t)$ can be modulated in any
598 convenient way to improve sensitivity (a simple choice is a succession of steps, giving the name
599 to the method of “step-decay”), it was shown that, for the interpretation: *i*) the volume of the
600 upstream tank does not need to be known; *ii*) the presence of a dead volume between the
601 upstream tank and the entrance of the porous sample, which represents a critical issue in the
602 pulse decay method, is of no consequence on the measurement and can be ignored, *iii*) any
603 irregularity on $P_{UP}(t)$, due to thermal effects or resulting from a leak at the upstream, will not
604 introduce any bias in the interpretation as it is part of the input signal. Moreover, it was shown
605 that, with this method, the three parameters, k , b and ϕ , can be simultaneously identified from a
606 single experiment (Lasseux et al., 2012).

607 The step decay experiment was run with nitrogen at 30°C on a Grimsel granodiorite sub-core
608 (length = 39.32 mm, diameter = 25.48 mm) that was first dried at 30°C for 3 weeks. The sample
609 was placed in a Hassler sleeve and a confining pressure of 5.5 MPa was applied. The volume of
610 V_{DOWN} was determined from 100 nitrogen pycnometry tests yielding $V_{DOWN}=8.38 \text{ cm}^3$ with a
611 standard deviation of 0.018 cm^3 . Three different tests were carried out for which the applied
612 average pore pressure difference over the different upstream pressure steps were 0.376 MPa
613 (Test1, 4 steps), 0.275 MPa (Test2, 7 steps) and 0.327 MPa (Test3, 4 steps). The experimental
614 recording times of $P_{UP}(t)$ and $P_{DOWN}(t)$ were 4h16min (Test1), 3h27min (Test2) and 1h10min
615 (Test3).

616 The interpretation, using an inverse technique, is performed with a complete model with no
617 assumption, except that the flow is isothermal and 1D in the x -direction within the sample which
618 upstream and downstream faces are at $x=0$ and $x=L$, respectively while the gas is supposed to
619 obey ideal gas law:

$$621 \quad \frac{\partial^2 \Pi}{\partial x^2} = \frac{\phi \mu}{k} \frac{1}{\sqrt{\Pi}} \frac{\partial \Pi}{\partial t}, \quad \Pi = \Pi(x, t) = (P(x, t) + b)^2 \quad (7)$$

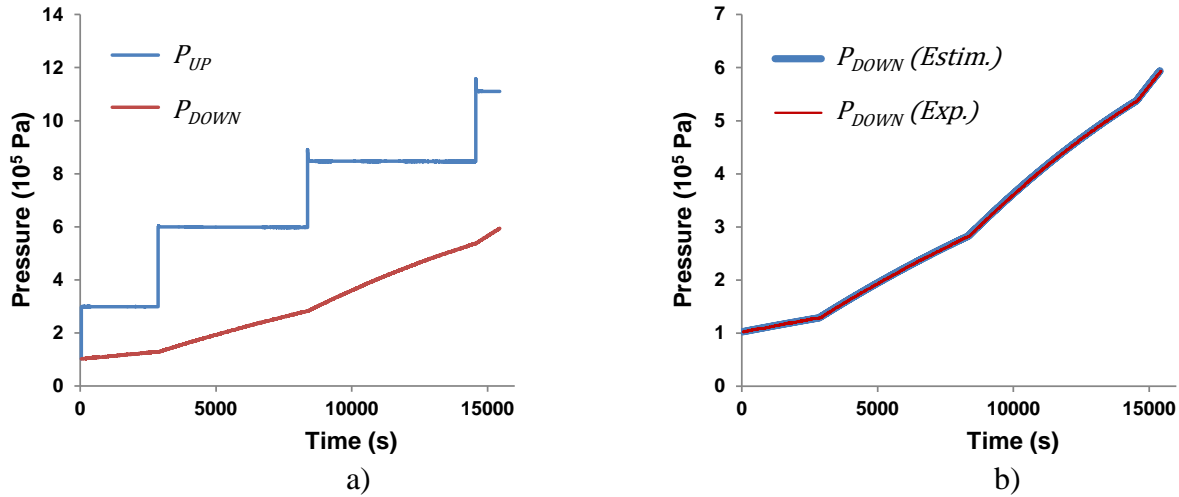
$$622 \quad \Pi(x, 0) = (P(x, 0) + b)^2, \quad 0 \leq x \leq L \quad (8)$$

$$623 \quad \Pi(0, t) = (P_{UP}(t) + b)^2, \quad t \geq 0 \quad (9)$$

$$624 \quad \left. \frac{\partial \Pi}{\partial t} \right|_{x=L} = \frac{kA}{\mu V_{DOWN}} \left(\sqrt{\Pi} \frac{\partial \Pi}{\partial x} \right)_{x=L}, \quad t \geq 0 \quad (10)$$

625
626 The recorded evolution of $P_{UP}(t)$ and $P_{DOWN}(t)$ for Test1 are reported in Figure 13a. For the
627 same experiment, the comparison between the measured signal $P_{DOWN}(t)$ and the signal

628 obtained at the end of the inverse procedure with the identified parameters k , b and ϕ in the least
 629 square sense is represented in Figure 13b, showing the excellent fit obtained with this procedure.
 630



631
 632
 633 Figure 13 a) Upstream ($P_{UP}(t)$) and downstream ($P_{DOWN}(t)$) pressure evolutions recorded during Test1. b)
 634 Downstream pressure signal measured during Test1 and obtained from the model with the fitted parameters k , b and
 635 ϕ at the end of the inverse procedure.

636
 637 The fitted values of the parameters are respectively $k=1.28 \cdot 10^{-18} \text{ m}^2$, $b=0.257 \text{ MPa}$ and $\phi=0.012$
 638 for Test1; $k=1.18 \cdot 10^{-18} \text{ m}^2$, $b=0.304 \text{ MPa}$ and $\phi=0.008$ for Test2; $k=1.26 \cdot 10^{-18} \text{ m}^2$, $b=0.279 \text{ MPa}$
 639 and $\phi=0.012$ for Test3. These values are consistent, in their trend, with the expected variations
 640 due to the difference between the confining pressure and the actual average pore pressure in the
 641 three different tests. Moreover, repeatability tests showed few percent of error on the above
 642 values.

643 3.6.3. Example of Oscillating Pore Pressure Method for Permeability Determination

644
 645
 646 [Contribution of Lab#18] Initially proposed by Turner (1958), the oscillation method was
 647 first applied to rocks by Kranz et al. (1990) and Fischer (1992). It uses a fixed-frequency,
 648 sinusoidally oscillating pore pressure signal applied at one end of the sample. The resultant
 649 (downstream) signal maintains the same period as the upstream signal, but is amplitude-
 650 attenuated and phase-shifted (Figure 14).

651 Bernabé et al. (2006) re-analyzed the oscillating pore pressure method and defined two
 652 independent dimensionless material parameters; η (dimensionless permeability) and ξ
 653 (dimensionless storativity ratio) which are functions of permeability (k , m²) and specimen
 654 storativity (β , Pa⁻¹) respectively and are defined:

$$655 \eta = \frac{A\tau k}{\pi L \mu \beta_D} \quad (11)$$

$$656 \xi = \frac{A L \beta}{\beta_D} \quad (12)$$

657
 658
 659 where A is the sample cross-sectional area (m²), τ is the oscillation period (s), L the sample
 660 length (m), β_D the downstream reservoir storage (m³ Pa⁻¹) and μ the fluid viscosity (Pa s).
 661 Bernabé et al. (2006) improved upon the solutions presented by Kranz et al. (1990) and Fischer

662 (1992) by defining ζ and η such that each would be dependent on only one material parameter of
 663 the rock, thus allowing them to be assessed as independent material properties. In terms of ζ and
 664 η the solution to the diffusion equation is:

$$665 \quad Ge^{-i\theta} = \left(\frac{1+i}{\sqrt{\xi\eta}} \sinh \left[(1+i) \sqrt{\frac{\xi}{\eta}} \right] + \cosh \left[(1+i) \sqrt{\frac{\xi}{\eta}} \right] \right)^{-1} \quad (13)$$

666
 667 G is the ratio of downstream to upstream wave amplitude (Gain) and θ is the phase shift between
 668 the upstream and downstream waveforms. Solving equation (13) to find G and θ using a range of
 669 valid values of η and ζ defines the region in which physically meaningful values of G and θ can
 670 be found. The region is limited by the lines $\zeta=0$ and $\zeta \rightarrow \infty$ (Bernabé et al., 2006). Sample
 671 storativity (β) is directly proportional to porosity (ϕ) and is given by:

$$672 \quad \beta = \phi(C_f + C_p) \quad (14)$$

673
 674 where C_f is the pore fluid compressibility and C_p is the compressibility of the porosity in
 675 response to changes in pore pressure at constant confining pressure. As $C_f \gg C_p$ the $C_f \phi$ term
 676 will dominate. Thus iso- ζ paths are nominally lines of constant porosity for a given value of
 677 downstream storage volume provided the compressibility of the pore fluid and the pores remain
 678 constant, there are no adsorption-desorption effects, and the sample behaves isotropically.

679
 680 When the permeating fluid is a liquid its compressibility is small and varies only slowly
 681 with pressure, but for an ideal gas the compressibility is $1/P_p$, where P_p is the pore fluid pressure.
 682 At low temperatures T and high pressures gases become non ideal, expressed by the gas
 683 deviation factor Z in the gas law for a single mole:

$$684 \quad P_p V = ZRT \quad (15)$$

685
 686 where V is the gas volume and R is the universal gas constant. The gas compressibility is
 687 modified thus

$$688 \quad c_f = \frac{1}{P_p} - \frac{1}{Z} \left(\frac{dZ}{dP_p} \right)_T \quad (16)$$

689
 690 For argon gas, for example, Gosman et al. (1969) show how Z varies with pressure, from
 691 which $C_f(P_p)$ can be calculated. Viscosity of the pore fluid must also be known as a function of
 692 pressure and temperature. For liquids the viscosity varies only slowly with pressure and
 693 temperature, but larger variations apply for gases. Data for argon are provided in Michels et al.
 694 (1954) and Younglove and Hanley (1986).

695
 696 Applying this method, it is usual to work with short core plugs, for example 25 mm in
 697 length and of similar diameter. Samples are jacketed in rubber tubing sealed to end pistons
 698 bearing a narrow (1mm diameter) hole to carry the pore fluid. Sintered metal discs are placed at
 699 each end of the specimen to diffuse the pore fluid across the whole diameter of the specimen. It
 700 is important that the jacket be pressed uniformly against the outer surface of the specimen to
 701 prevent short-circuit fluid paths. A steel blank can be used in lieu of a specimen to ensure that
 702 the experimental arrangements do not permit any unwanted fluid flow and for calibration of the
 703
 704
 705

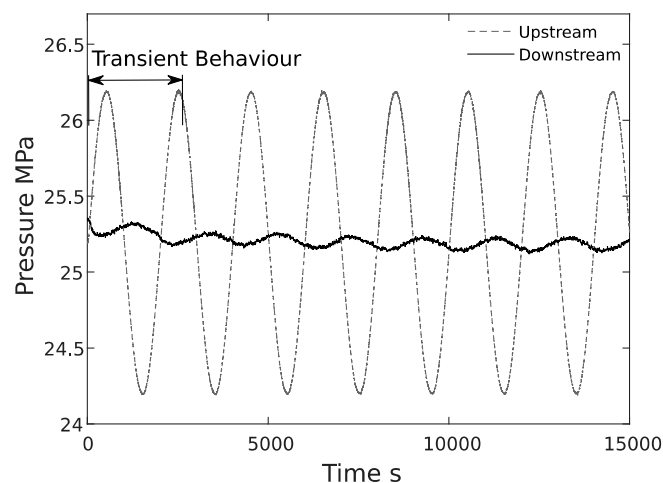
706 downstream volume. It is important to avoid any contamination of the specimen with liquid
707 when gas is being used as a permeant. Liquid contamination will generally reduce apparent
708 permeability.

709 The downstream volume (including pipework, downstream sintered plate and pressure
710 transducer) must be determined as accurately as possible, and for very low permeability
711 materials this volume will usually be as small as is feasible. Filler rods can be inserted into the
712 pipes to minimize the volume further. For good resolution of low permeabilities a downstream
713 volume of less than 500 mm³ is desirable. The downstream pressure transducer must have high
714 sensitivity (typically 0.02 MPa) and good stability.

715 A servo-controlled piston-cylinder pressure generator/volumometer is used to generate
716 and control the pore pressure. This can be used to determine the downstream volume by first
717 establishing an upstream pore pressure, then opening access to the downstream volume and
718 measuring the volume of fluid that must be accepted to bring the downstream volume to the
719 same pressure.

720 Experiments are typically carried out over a sequence of confining pressures at a fixed
721 pore pressure in order to investigate the sensitivity of permeability to effective pressure. Initially
722 it is important to raise the confining and pore pressures together such that the minimum desired
723 effective pressure is not exceeded, to avoid permanent changes to permeability before
724 permeability measurement at low effective pressure. The desired mean pore pressure and
725 confining pressures are allowed to stabilize, with the open bypass valve linking upstream and
726 downstream reservoirs. The bypass valve is closed slowly, to prevent buildup of unequal pore
727 pressures and the upstream oscillation is started. The amplitude of the oscillation will typically
728 be 1 MPa or less, to avoid violation of Darcy's law and pressure transients due to adiabatic
729 heating and cooling. After any initial transient effects a downstream waveform at a constant
730 mean pressure will develop, after which data from ~ 10 cycles will be collected (Figure 14). The
731 period of the forcing waveform can be varied between about 60 seconds and several thousand
732 seconds, in order to obtain a satisfactory gain ratio, ideally smaller than about 0.7.

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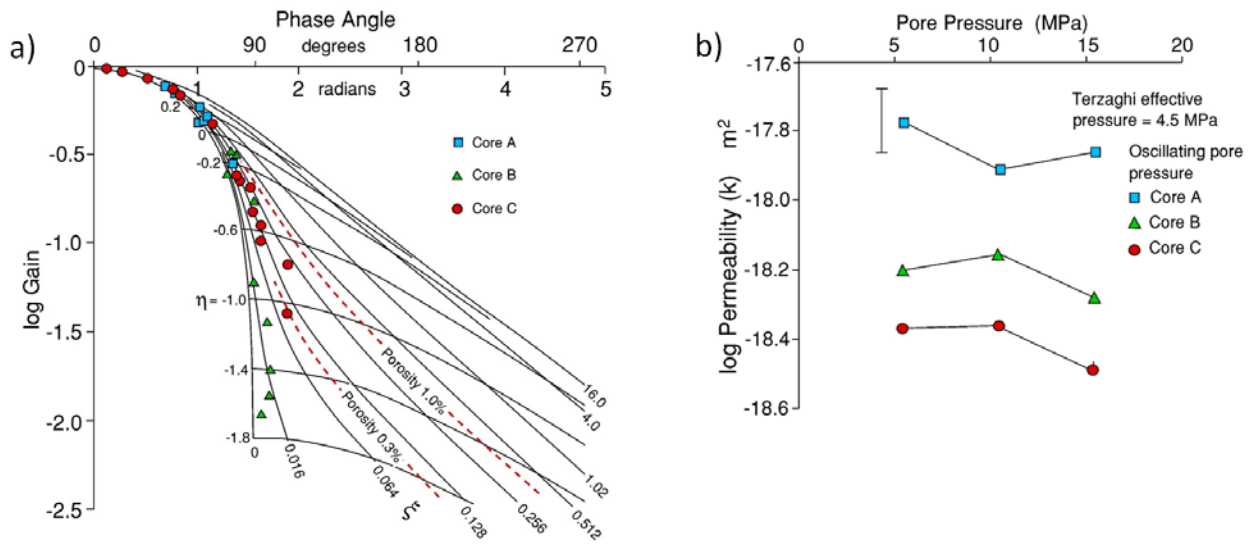
735 **Figure 14.** Example of oscillating pore pressure behaviour, showing amplitude attenuation of the forcing
736 waveform and the phase shift of the downstream wave.

737

738 From these data the gain (ratio of downstream to upstream wave amplitudes) and phase shift
739 must be determined. This can be done in several ways (i) from the Fourier transforms of the two
740 waveforms (e.g. Faulkner and Rutter, 2000; Bernabé et al., 2006; Song et al., 2007), (ii) from the

741 parameters (orientation and axial ratio) of the Lissajou ellipses linking the two waveforms (e.g.
 742 Song et al., 2007) or (iii) by applying inverse amplitude ratio and phase shifts to match the two
 743 waveforms. The permeability is found by solving equation (13) iteratively for both η and ζ , from
 744 which permeability and storativity can be calculated. This can be done using a numerical
 745 equation solver. Initial values of ζ and η are obtained from a look-up table containing the values
 746 plotted in Figure 15a. The algorithm then seeks the values of ζ and η that simultaneously solve
 747 the modulus and argument of equation (13) that correspond to the measured amplitude ratio G
 748 and phase shift θ respectively.

749 Core plugs with three different orientations were taken from the main core section; the
 750 gneiss foliation plane is oriented 16° to the axis of the main core. Core A is parallel to the large
 751 core axis, Core B is normal to the main core axis and parallel to the foliation, and core C is
 752 perpendicular to the first two, nearly normal to the foliation. Helium pycnometry for four short
 753 core plugs yielded porosity $1.028 \pm 0.011\%$. Permeability for each core orientation was measured
 754 at a constant effective pressure (P_{eff}) of 4.5 MPa, at each of three different pore pressures, 5.5,
 755 10.5 and 15.5 MPa (Figure 15b). Anisotropy is low, with the foliation-normal orientation
 756 displaying the lowest permeability (see section on anisotropy in the companion paper).
 757



758 **Figure 15.** a) Solution space of equation (13) bounded by curves for $\zeta = 0$ and 16, showing how gain and phase
 759 angle relate to η and ζ , with experimental results for KG²B cores A, B and C. b) Permeability vs. pore pressure at
 760 constant effective pressure (4.5 MPa) for the three cores.
 761

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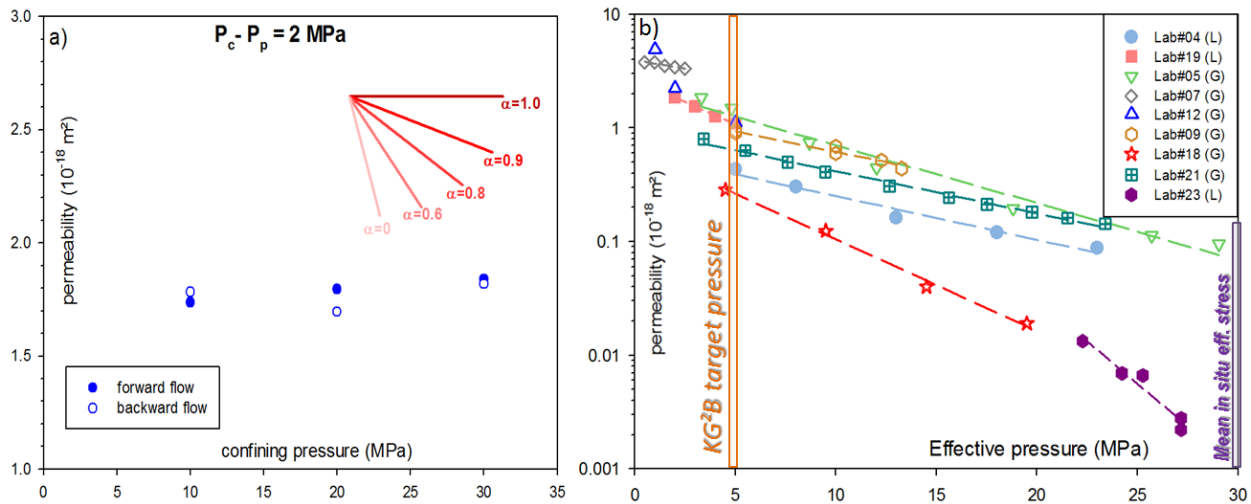
764 The experimental data in Figure 15a plot well to the left of the expected trend for a
 765 porosity of 1.0%, and imply that flow does not access all pore space with equal facility. The
 766 sample storativity calculated using ζ from the oscillation technique is commonly found to be
 767 lower than the total storativity of the sample, calculated from porosity (ϕ), the known
 768 compressibility of the pore fluid using equation (12) (Fischer, 1992) and the downstream storage
 769 of the experimental setup. This is evident from the plot of $\log G$ versus phase shift θ for the
 770 Grimsel granodiorite (Figure 15a). The data lie along a track expected for a porosity of 0.3 %
 771 or smaller, compared with the track expected for the measured porosity. There is a weak indication
 772 that the deviation is greater for foliation-parallel flow than foliation-normal flow. Stronger

773 deviation is seen for anisotropically textured rocks (e.g. Mckernan et al., 2017), and suggests that
 774 for one-dimensional fluid flow a reduced fraction of the pore space is readily accessible. In
 775 contrast, porosity measurement when all faces of the specimen are equally accessible to
 776 permeating gas allows the full porosity to be measured.

777
 778

779 **4. Pressure Dependence of Permeability**

780 The main target of the benchmarking exercise was permeability of the Grimsel
 781 granodiorite samples at 5 MPa effective pressure; in addition, on a voluntary basis, measurement
 782 at in situ effective pressure (30 MPa) was also encouraged. Several teams provided
 783 measurements at several pressures. Therefore we have: (i) single permeability values at 5 and 30
 784 MPa (2-point analysis, 4 teams), (ii) multiple permeability values over an extended pressure
 785 range of 1 to 30 MPa (multi-point analysis, 9 teams). The complete data set is given in Table 1
 786 and the results for the second data set are shown in Figure 16. Note that the effective pressure is
 787 defined here as the difference between confining and pore pressure, corresponding to an effective
 788 pressure coefficient equal to 1, in good agreement with the effective pressure law found by one
 789 participating lab (Figure 16a) showing that permeability measurements are nearly constant at
 790 fixed confining pressure minus pore pressure (2 MPa). It should also be noted that these data do
 791 not allow separation of pressure sensitivity in response to variations of effective pressure by
 792 varying total confining pressure at constant pore pressure, compared with varying pore pressure
 793 at a constant total confining pressure. Differences in behavior in this respect have been discussed
 794 for various rock types by several authors, (e.g. Heller et al., 2014; Kwon et al., 2001; Mckernan
 795 et al., 2017)



796

797 **Figure 16.** a) Test on effective pressure law by Lab#19 showing permeability measurements are consistent
 798 with $\alpha=1$; b) Permeability vs. effective pressure. Solid symbols: measurements with liquid; open symbols:
 799 measurements with gas. All the measurements were made in the core axis direction.

800 A striking result is that the permeability evolution with effective pressure is generally linear on
 801 the semi-log plot: therefore the pressure-dependence of permeability can be accounted for using

802 an exponential law:

803
$$k = k_o \exp(-\gamma P_{eff}) \quad (17)$$

804 Such an exponential decrease is in agreement with the data compiled by David et al. (1994) for
 805 sedimentary and hard rocks. The stress-sensitivity parameter γ and zero-pressure permeability
 806 parameter k_o are given in Table 1.

807

| 2-point analysis | | | | multi-point analysis | | |
|------------------|--|---|-------|--|-------------------------------|--|
| | perm (10^{-18} m ²) @5MPa | perm (10^{-18} m ²) @30MPa | ratio | k_o (10^{-18} m ²) | γ (MPa ⁻¹) | Comment |
| Lab#02 (L) | 0.43 | 0.030 | 14.3 | | | |
| Lab#13 (L) | 0.91 | 0.277 | 3.27 | | | |
| Lab#14 (G) | 1.91 | 0.189 | 10.1 | | | |
| Lab#16 (G) | 1.81 | 0.155 | 11.7 | | | |
| Lab#04 (L) | 0.43 | <i>0.043</i> | 10.0 | 0.609 | 0.0885 | fit on 5 points |
| Lab#19 (L) | 1.08 | <i>0.0118</i> | 91.4 | 2.65 | 0.180 | fit on 4 points, low pressure range |
| Lab#23 (L) | <i>Not relevant</i> | <i>0.00101</i> | - | 30.0 | 0.343 | fit on 5 points, high pressure range |
| Lab#05 (G) | 1.46 | <i>0.068</i> | 21.5 | 2.26 | 0.117 | fit on 7 points |
| Lab#07 (G) | <i>2.78</i> | <i>0.474</i> | 5.86 | 3.96 | 0.0707 | fit on 5 points, low pressure range, axial stress only |
| Lab#12 (G) | 1.12 | | | not exponential | | low pressure range |
| Lab#09 (G) | 0.92 | <i>0.115</i> | 8.03 | 1.43 | 0.0842 | fit on 6 points |
| Lab#18 (G) | 0.25 | <i>0.00257</i> | 98.7 | 0.666 | 0.185 | fit on 4 points, radial normal to foliation permeability |
| Lab#21 (G) | 0.83 | <i>0.0758</i> | 10.9 | 0.975 | 0.0851 | fit on 10 points |

808 **Table 1.** Permeability measured at different effective pressures. For the multi-point analyses, exponential
 809 laws with parameters k_o and γ have been determined. Bold numbers are measurements; italic numbers are
 810 extrapolated values from the exponential law. All data were obtained for the axial core orientation unless
 811 otherwise stated.

812

813 In the pressure range above 5 MPa, four experiments (Lab#4, 5, 9 and 21) found quite consistent
 814 results, with an average stress-sensitivity parameter $\gamma = 0.093 \pm 0.015$ MPa⁻¹ while two other

815 experiments found both lower permeability and stronger pressure dependence ($\gamma > 0.18 \text{ MPa}^{-1}$),
816 possibly because these samples might have lower crack density and with higher compliance
817 and/or because of the different sample orientation (Lab#18) . In their compilation for crystalline,
818 metamorphic and volcanic rocks, David et al. (1994) found that the stress-sensitivity parameter γ
819 ranged between 0.023 and 0.11 MPa^{-1} ; the Grimsel granodiorite is toward the higher end of this
820 range. In the pressure range below 5 MPa, the pressure dependence seems also to be larger
821 (except for Lab#07 who applied only axial stress). In this pressure range, one might both be
822 impacted by crack closure and possibly leakage flow at the sample surface. From the 2-point
823 analysis, we estimated the ratio $k(P_{eff} = 5 \text{ MPa}) / k(P_{eff} = 30 \text{ MPa})$ for both measured and
824 extrapolated values (Table 1). Except two large values close to 100, most of the ratios range
825 between 3.3 and 21 with an average value of 10.6. The Grimsel granodiorite exhibits a strong
826 pressure dependence of permeability which can be well described by an exponential law.

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833 **5. Discussion**

834 5.1. Outcome of the Benchmarking Exercise

835 Three main techniques were used to test sample permeability: steady-state flow that
836 satisfies Darcy's law, pulse-decay (Brace et al., 1968) and oscillating flow (Fischer & Paterson,
837 1992; Kranz et al., 1990). The steady-state flow technique is often the simplest and easiest to
838 interpret. Some rocks such as shales, clay-rich sandstones and fault gouge undergo time-
839 dependent relaxation in response to pressure changes or the introduction of pore fluid. The
840 steady-state method, which requires establishment of a constant flow rate, can be used to identify
841 when transient changes in pore geometry have ended and a reliable measurement of permeability
842 can be made. In some cases, the time needed for a sample to adjust to a new stress state can be in
843 excess of a day (Morrow et al., 2014). If the sample has low permeability, then the flow rate due
844 to an applied pressure gradient will be low, and water expelled as the sample compacts can result
845 in erroneous flow rate determinations. Reversing the flow direction can help identify when time-
846 dependent pore volume changes are important. Otherwise, it is best to confirm independently
847 that changes in porosity have ceased before starting the flow test. For low permeability samples
848 with small flow rates, both a high-accuracy flow sensor and a stable test chamber (especially
849 controlled temperature) are needed for accurate determination of permeability (see Section
850 3.6.1). In describing flow in porous media, effects are separated between fluid properties
851 (viscosity, μ) and pore geometry (permeability, k) with flow rate Q proportional to k/μ . Thus, for
852 low permeability samples, increased flow rate can be accomplished by using a low-viscosity
853 fluid, typically argon or nitrogen.

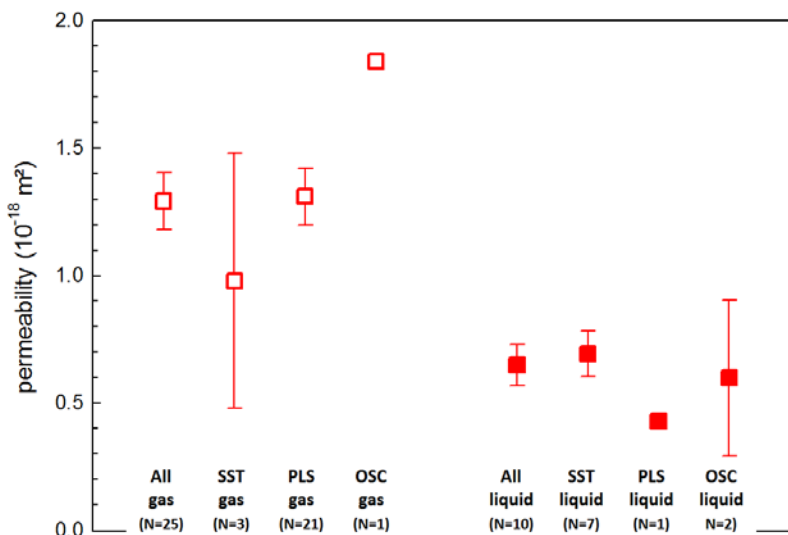
854 When testing low permeability crystalline rocks, accurate measurement of steady-state
855 flow rate can be technically challenging. Brace et al. (1968) presented a transient pulse-decay
856 technique that avoided this difficulty by measuring transient pressure changes rather than flow
857 rate. This can be a fast and reliable method for measuring low permeability. It requires that the
858 volume in closed chambers connected to the sample be optimized for the flow rate such that fluid
859 flow through the sample produces a measurable pressure change over a convenient time interval.
860 Similar to the steady-state technique, relaxation of pore volume in response to changes in stress
861 state can produce pressure transients that mimic pressure transients from the pulse-decay test.
862 Therefore, care must be taken to minimize these potential sources of error. On account of the
863 small pressurized volumes used, the technique is very sensitive to any slow pressure leaks.
864 Changes in ambient temperature can also lead to erroneous signals and need to be identified
865 when making pulse-decay measurements.

866 The oscillating flow technique introduced for rocks in the early 1990's (Fischer, 1992;
867 Fischer & Paterson, 1992; Kranz et al., 1990) represents a significant advance in the
868 measurement of flow properties of geologic materials. A time-independent oscillating pore
869 pressure (generally a sine wave) is applied on one side of the sample and the amplitude and
870 phase of the pressure in a chamber attached to the other side of the sample is recorded. The
871 signal is time-stationary and can be stacked over multiple cycles to improve accuracy. In this
872 case, both permeability and storativity (β) of the sample can be determined. There is a limited
873 range in the frequency of the pressure oscillation and the volume of the downstream chamber
874 over which accurate measurements can be made. In general, the downstream volume has to be
875 larger than the pore volume in the test sample if storativity needs to be measured, but not so large
876 as to affect adversely the sensitivity of the downstream pressure measurements. Then, the period
877 of the input signal that will produce a usable response will fall within a limited range that
878 depends on the sample permeability. Measurement of lower permeability generally requires
879 increased period of the sinusoid. If there is time-dependent relaxation of the sample, pressure on
880 the downstream side will show a steady drift that often can be separated from the oscillating
881 signal of known period. In the non-linear inversion procedure for analyzing the amplitude/phase
882 data, k is not determined uniquely. Rather, the ratio k/β is determined and errors in k and β are
883 correlated. Increasing the downstream reservoir solves this problem: permeability can then be
884 estimated accurately but not storativity.

885 All reported determinations of permeability for the KG²B core, measured at $P_{eff} = 5$ MPa
886 in the axial direction, are plotted in Figure 17 along with standard deviation. Many uncertainties
887 for individual measurements are smaller than the symbol size in the figure. Figure 5 shows that
888 there is no indication that k varies systematically with distance from the tunnel (the same holds
889 for porosity ϕ also, see companion paper). Pulse decay measurements are most abundant and
890 tend to be higher than steady-state measurements. The two values that deviate the most from the
891 mean value (both higher and lower) were steady-state measurements. Since measurements were
892 carried out in different laboratories, using different samples and different techniques, the outlier
893 values may be due to sample variability or test procedures. In the samples there is obvious
894 foliation, anisotropy and sample variability on a scale comparable to the individual sample
895 dimensions. A number of laboratories reported porosities of test samples spanning more than one
896 order of magnitude (see companion paper) implying that much of the variability in permeability
897 is the result of heterogeneity in the test samples.

898 Figures 3 and 4 show separate analyses of permeability determinations based on gas and
899 liquid (primarily water) pore fluids. Average permeability determined using gas is about twice

900 the average permeability based on water measurements. This is consistent with Figure 17, where
 901 mean values and standard errors are plotted for different types of tests and different fluids.
 902 Permeability determinations are grouped by technique and working fluid. Some results plotted in
 903 Figure 17 are not statistically significant since three of the sub-categories only contain one or
 904 two measurements.
 905



906
 907 **Figure 17.** Summary of all the permeability results in the axial direction at 5 MPa effective pressure per fluid and
 908 method. The averaged measurements with gas are systematically larger than those with liquids.
 909

910 Still, interesting trends can be seen. The left-most data point is the average permeability of all of
 911 the reported gas measurements ($k = 1.29 \cdot 10^{-18} \text{ m}^2$). Average values for the three techniques using
 912 gas are adjacent. On the right side of the plot, the average permeability for all liquid
 913 measurements is plotted ($k = 0.649 \cdot 10^{-18} \text{ m}^2$). Here the steady-state outlier values that were
 914 identified previously have been omitted. The correlation between permeability outliers and
 915 porosity values suggests that these samples were anomalous. Including them in the analysis has
 916 little effect on the mean value of permeability but increases uncertainty by about 3-fold. When
 917 the permeability values are separated into six sub-groups, there is no clear difference based on
 918 technique (steady-state, pulse decay or oscillating flow). However, a significant difference does
 919 exist in which gas permeability is about twice the permeability measured with liquid.

920 The choice of liquid or gas pore fluid can be problematic. For low permeability rocks and
 921 fault gouge, measurements can be completed much faster using gas, which has a relatively low
 922 viscosity. For some exceedingly tight samples, it may not be possible to obtain a usable flow rate
 923 with water as a pore fluid. At the same time, many samples have grain contacts and pore-filling
 924 minerals that are chemically reactive with water or brine. In this case, the choice of pore fluid
 925 becomes critical. Porosity filled with an inert gas may not have the same structure or pressure
 926 sensitivity as porosity filled with naturally occurring brine, or with water with which it is in
 927 chemical dis-equilibrium. This can cause water permeability to be up to 1-2 orders of magnitude
 928 lower than gas permeability. The reason for this phenomenon is not clear and various hypotheses
 929 are discussed in the literature, including core damage by clay plugging, clay swelling, structured
 930 water films on the mineral surfaces resulting in reduction of the effective transport volume, and
 931 electro-osmotic counter pressures (Faulkner & Rutter, 2000; Gray & Rex, 1966; Weber &

932 Stanjek, 2012). These issues may not be so important in crystalline rocks, but can be of major
933 concern when measuring shales or clay-rich fault gouge.

934 Many of the permeability measurements had reported uncertainties of 1 to 2 percent. This
935 appears to be a practical lower limit to the accuracy that can be obtained by any of the three
936 techniques used. When all reported measurements are included and estimates are based on
937 $\log(k)$, the standard error in estimating permeability is about 20%. The largest potential gain in
938 reducing uncertainty appears to be related to the systematic difference between liquid and gas
939 measurements. If this two-fold difference can be explained, the standard error might drop below
940 8%.

941 Two other important issues have been highlighted by the benchmarking exercise: the
942 effect of sample size and the pressure sensitivity of permeability. Results were obtained on a
943 large range of volumes, from 1 to 500 cm³. Whereas the results for the largest samples were
944 consistent, the permeability values for the smallest ones were scattered (Figure 6a) which may
945 indicate that the volume of the smaller samples is below the REV. In this regard the choice of the
946 Grimsel granodiorite, selected for its availability, convenience and relevance to geothermal
947 energy studies, probably was not optimal. Foliation and mineralogical heterogeneity (Figure 6b)
948 require to work on samples larger than the largest heterogeneity. Unfortunately this was not
949 systematically the case. Despite the size effect, consistent results were found regarding pressure
950 dependence, and showed that the Grimsel granodiorite is strongly pressure sensitive. The choice
951 of a common effective confining pressure was a key for the success of the benchmarking
952 exercise.

953

954

955

956 5.2 Offset between average gas and liquid permeability

957

958 Most of the permeability measurements were done using gas as the pore fluid (Figure 1),
959 so it is important to assess the corrections for gas slippage. Intrinsic (or absolute) permeability is
960 expected to be: i) determined only by the porous media structure, and ii) independent of the
961 (homogeneous) working fluid passing through it. Nevertheless differences between water and
962 gas permeability have been reported in literature for decades, and for several lithotypes (Muskat
963 et al., 1937) including shales/mudrocks, tight sandstones and carbonates (e.g. Busch and Amann-
964 Hildenbrand, 2013; Ghanizadeh et al., 2013; Amann-Hildenbrand et al., 2016). Klinkenberg
965 (1941) introduced a theory regarding slip flow and its microscale effect: the slippage of gas
966 molecules along capillary walls resulting in a non-zero wall velocity. He introduced a gas
967 slippage parameter (or Klinkenberg slip factor) b relating the apparent gas permeability k_{app} to
968 the mean (absolute) gas pressure P_{MEAN} :

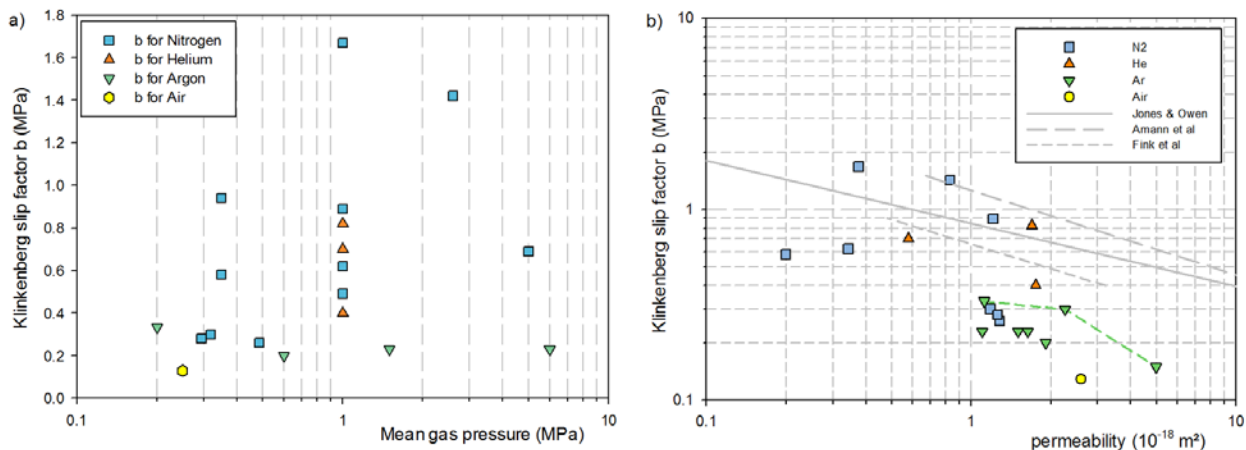
$$969 \quad k_{app} = k_{\infty} \left(1 + \frac{b}{P_{MEAN}} \right) \quad (18)$$

970 where k_{∞} is the permeability at infinite gas pressure (equivalent to the permeability k measured
971 using a liquid). The slip of gas near a solid wall was first studied by Maxwell (1867) and
972 Klinkenberg's concept of slippage was developed for gas flow within a bundle of constant radius
973 capillaries. Hence, this theory may only be applicable within certain boundary conditions. Its

974 validity for flow in tortuous pore systems including bulges and bottlenecks or in crack-like
975 porosity remains questionable.

976 Our study clearly shows differences depending on the fluid used. Gas permeability values
977 appear to be about twice the permeability values obtained using liquids (Figure 4). This
978 discrepancy is observed even after the Klinkenberg correction for gas slippage effects. Moreover
979 the type of gas used is expected to have an effect as well. Gas permeability (both apparent and
980 Klinkenberg corrected) has been observed to decrease in the order $\text{He} > \text{N}_2 > \text{CH}_4 > \text{CO}_2$, (e.g.
981 Han et al., 2010). In organic rich material (coals, shales) this phenomenon is linked to sorption
982 and swelling effects - in such cases a clear dependence upon total organic carbon can be
983 identified. In the absence of sorption the fluid dynamic characteristics of the different gases have
984 to be accounted for. Differences in molecule size and mean free path length result in different
985 slip flow characteristics and, for larger gas molecules, in size exclusion.

986 The Klinkenberg slip factor values for each of the gas permeability experiments are plotted
987 vs. the mean gas pressure in Figure 18a. Significant differences are observed between Helium,
988 Argon, Nitrogen and air. The lowest slip factors b are found for air and Argon, followed by
989 Helium. For Nitrogen, slip factor values span one order of magnitude, from 0.12 to 1.7 MPa,
990 without noticeable dependence on mean pressure. Although several parameters can disturb the
991 determination of slip factor b (accuracy of mean pressure value, lack of back pressure, inertia
992 effects, effective pressure effects), it is surprising that such a large range of values was found for
993 Nitrogen. Slip factor estimation is very sensitive to experimental procedures and several
994 measurements along large mean pressure values are needed to ensure a robust regression in order
995 to limit hazardous extrapolation for infinite mean pore pressure (McPhee and Arthur, 1991). In
996 Figure 18b the b values are plotted vs. permeability. A weak linear trend is observed, in
997 agreement with published results from tight sands, sandstones and shales (Jones & Owens, 1980;
998 Amann-Hildenbrand et al., 2016; Fink et al., 2017). The three Argon data points are linked with
999 a dashed line: these are measurements on a single sample at different confining pressures,
1000 showing a decreasing trend for b vs. permeability similar to published data on sedimentary rocks.
1001



1002 **Figure 18.** a) Klinkenberg slip factor b vs. mean pore pressure for gas permeability measurements with
1003 Klinkenberg correction. b) Klinkenberg slip factor b vs. permeability, grey lines are published data on gas sands
1004 (Jones & Owens, 1980), tight gas sandstones (Amann-Hildenbrand et al., 2016) and shale (Fink et al., 2017).
1005

1006

1007 Flow in porous media is generally modeled under the assumption that the fluid is slow,
1008 continuous and viscous, with negligible flow of molecules adjacent to the pore wall (Darcy flow
1009 conditions). As mentioned above, the use of the Klinkenberg slip factor b is related to the
1010 hypothesis of a slippage flow regime at the microscale along capillary walls. The presence of
1011 high-surface-area minerals in the Grimsel granodiorite, such as biotite and chlorite, and their
1012 micro-pore structures, enhances diffusion, adsorption, and reactivity to gases and liquids.
1013 Specifically, if the gas or liquid exhibits chemical affinity with the biotite/chlorite minerals, then
1014 adsorption onto clay platelets, swelling and particle mobilization may occur. Under certain
1015 pressure and temperature conditions, the mean free path λ of the gas molecules (i.e. the average
1016 distance travelled without molecular collisions, depending on the temperature, the reciprocal
1017 mean pore pressure and the nature of the gas (McPhee & Arthur, 1991)) will exceed the size of
1018 pores/cracks. In such conditions, molecule/molecule collisions become so rare that the concept of
1019 viscosity becomes irrelevant, rendering the concept of continuum and bulk flow inapplicable.
1020 Knudsen number is classically used to quantify the validity or failure of the Navier-Stokes flow
1021 regime, defined as $K_n = \lambda/H$ where λ is the mean free path and H a characteristic hydrodynamic
1022 length scale (Hadjiconstantinou, 2006). For sake of simplicity, we take for H the crack aperture.
1023 When K_n is high, wall friction is reduced which can be interpreted as a decrease in viscosity
1024 leading to an apparent increase of permeability (Allan & Mavko, 2013; Carrigy et al., 2012).
1025 Depending on the magnitude of K_n , several flow regimes can be identified (Schaaf & Chambre,
1026 1961; Wang et al., 2016). For example, when $0.01 < K_n < 0.1$ the flow is in the slippage flow
1027 regime and the Klinkenberg correction is applicable, but for $0.1 < K_n < 10$ the flow is in the
1028 transitional regime and the Klinkenberg correction may not be sufficient. In the latter case
1029 additional corrections need to be done to account for Knudsen diffusion flow. Following (Wang
1030 et al., 2016), the mean free path λ can be derived from a hard-sphere gas model and the Knudsen
1031 numbers are estimated using the following relation:

$$1032 \quad K_n = \frac{RT}{\pi\sqrt{2}(D_m)^2 r N_A P_{MEAN}} \quad (19)$$

1033
1034 where N_A is Avogadro's number, R the ideal gas constant, P_{MEAN} the average gas pressure, D_m the
1035 gas molecule diameter and T the absolute temperature of the gas. For the length scale H we take
1036 the average crack aperture obtained from microstructural analyses on the Grimsel granodiorite
1037 ($H = 283$ nm, see companion paper) and we allow this parameter to vary in the range 100 to 800
1038 nm (see Figure 4A in the companion paper) . Knudsen numbers are plotted vs. the mean pore
1039 pressure in Figure 19 for all the gas permeability measurements.
1040

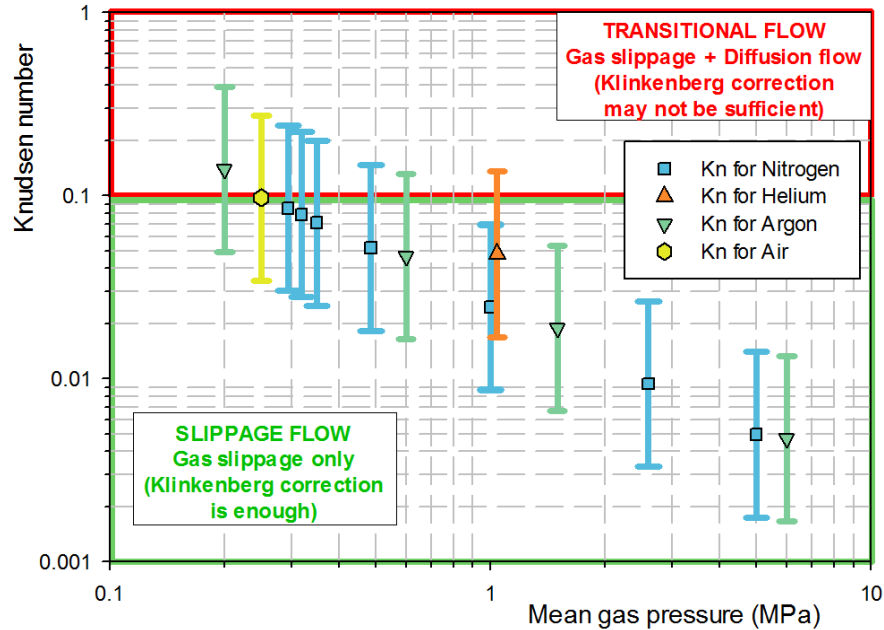


Figure 19. Knudsen numbers K_n vs. mean gas pressure for all the gas permeability experiments. The “error bars” correspond to a range of crack aperture from 100 to 800 nm, the symbols correspond to the average crack aperture (283 nm).

All the data points are located in the slippage flow region or close to the $K_n=0.1$ boundary. This suggests that the slippage flow has been correctly accounted for by the Klinkenberg correction. However complexity can arise from the pore size heterogeneity: in some pores the local Knudsen number may be very low while in others it may be high. Another assumption is that gases follow the ideal gas law, which might not always be true (e.g. in the event of water vapor contamination).

When gas transport in microporous rocks is dominated by gas diffusion through pores/cracks, the amount of gas adsorbed changes dynamically as pore pressure changes and is closely related to the properties of the adsorbate (viscosity and density) and solid adsorbent as well as the pore-space geometry (Cui et al., 2009; Silin and Kneafsey, 2012). In particular, since molecular collisions are controlled by the molecular kinetic energy, diffusion is controlled by pressure and temperature. Allan and Mavko (2013) show that a tortuous pore network with a static adsorbed layer experiences variable Knudsen diffusion as a function of pore pressure. Below a critical pore pressure, the effective permeability is significantly greater than the continuum prediction due to rarefaction of the gas and the onset of Knudsen diffusion. Above the critical pressure, the effect of Knudsen diffusion relative to adsorption is significantly reduced, resulting in effective permeability values up to 40% lower than the continuum prediction. It must also be noted that errors arise not only from measured values but also from computed ones.

Previous studies suggested that permeability tests should be performed using distilled water, because such water is expected to be inert. In fact, distilled water may cause sample leaching leading to the expansion of absorbed cations around clay particles and reducing hydraulic conductivity. Leaching can also mobilize particles due to either the expansion of diffuse double layers or the removal of cement (Wilkinson, 1969). This movement of particles results in ‘dynamic permeability reduction’ (Todd et al., 1978) caused by particle trapping at sub-critical pore throats. This dynamic permeability reduction can be regarded as non-reversible in the absence of dynamical stresses. Alternatives to distilled water include non-polar solvents, direct

1072 use of field-collected water and duplication of the original pore water as permeant. Another
1073 common source of measurement error in very tight formations is entrapped gas, or air dissolved
1074 in the permeant while injecting it into the sample at high pressure. As pressure in the flowing
1075 water decreases, air can exsolve, causing pore clogging and erroneous measurements. Loosveldt
1076 et al. (2002) showed that water permeability was systematically lower than gas permeability,
1077 whereas ethanol permeability was intermediate. However, when gas permeability was corrected
1078 for the Klinkenberg effect, ethanol and gas permeabilities were found to be of the same order. In
1079 presence of chemical activity induced by polar fluids, Loosveldt et al. (2002) suggest that the
1080 Klinkenberg effect is only a small contributor to observed differences between gas and water
1081 permeability: other processes such as rehydration, dissolution/precipitation, migration of fine
1082 elements, and water adsorption in the smallest pores of the matrix may be more important.

1083 Finally it may also be possible that liquids and gases do not probe the porous media in the
1084 same way: in such a situation, a common value of permeability is not expected at all between
1085 permeability measured with gases and liquids. Our data set suggests that in this case gases probe
1086 a more efficient pore network in terms of fluid transport than do liquids. Further studies are
1087 needed to support this viewpoint.
1088

1089 5.3. Source of Errors in Low Permeability Measurements

1090 We discuss here the most common experimental problems and mechanisms for sources of
1091 error in permeability measurements from tight formations. The first source of error is
1092 methodological and procedural diversity that, to a large extent, controls the degree of variability
1093 in the results. The pressure pulse decay method is often the standard technique for low
1094 permeability material, as the conventional steady state method may not work if flow rate and/or
1095 differential pressure are too low to measure accurately. McPhee and Arthur (1991) showed that
1096 the effect of pressure transducer error (± 0.69 kPa) on the derived slip factor becomes more
1097 pronounced ($\sim 73\%$) when measurements are performed under constant flow rate mode (rather
1098 than constant differential pressure). When utilizing the pressure pulse decay method, extreme
1099 care must be taken to ensure constant temperature over the experiment so that the measured
1100 pressure changes are associated only with flow through the pore space. In addition, it may be
1101 difficult to reconcile gas or liquid permeabilities measured by laboratories that use different
1102 sleeve specifications and/or confining pressure. The extent to which the radial pressure on the
1103 sleeve is effectively transferred to the specimen is a function of sleeve hardness and thickness. If
1104 measurements refer to ambient conditions, sleeve confining pressure should be sufficiently high
1105 (1.5-5.5 MPa) for the sleeve to laterally seal the sample by filling its surface irregularities, thus
1106 avoiding fluid bypass, and sufficiently low to avoid permeability reduction due to pore volume
1107 compaction. This issue may become particularly relevant in the presence of schistose
1108 microstructure and large amounts of soft (compressible) minerals. When comparing data from
1109 different laboratories, it is important to decide *a priori* whether to emphasize the
1110 data from 'virgin' (unseasoned) samples during their first loading or limit the investigation to
1111 elastic regimes by pre-stressing the specimen.

1112 The second source of error is associated with tight rock microstructure and solid-fluid
1113 interactions. The samples under investigation show visible foliation that relates to compositional
1114 banding (segregation of mineral phases). This mineralogical differentiation forms alternating
1115 layers of biotite and quartz (Schild et al., 2001), white mica and chlorite (Goncalves et al., 2012),
1116 and small amounts of chlorite/smectite (vermiculite), the latter resulting from alteration of biotite

1117 layers (Kralik et al., 1992). The analyses conducted in this study show that a significant part of
1118 the pore space resides within the biotite phase as a network of sub-micron cracks exhibiting an
1119 average fracture aperture of 283 nm (see companion paper). Both mineralogical and
1120 microstructural features lead to processes that change the macro-scale permeability measured in
1121 the laboratory and its sensitivity to pressure.
1122

1123 5.4. Good Practice for Low Permeability Measurements

1124 Experimental studies aim to determine of the “true” or “in-situ” permeability value
1125 and increase understanding of contributing processes. However, the measured permeability
1126 depends on various parameters and their interdependencies. In the context of this study, reported
1127 permeability coefficients varied by approximately 1-2 orders of magnitude. Systematic and
1128 random errors are considered irrelevant here, as the experiments were performed at controlled
1129 temperature and pressure conditions, and any erratic fluctuations were accounted for in data
1130 analysis. The most important factors influencing the experimental results for single-phase flow
1131 were a) effective stress history and loading time, including stress-release effects due to coring, b)
1132 the pore fluid (gas, water) used in the experiments and c) sample heterogeneity. The latter
1133 category includes intrinsic lithological/textural features but also those induced by plug
1134 preparation, transportation and the drying/saturation procedure. The impact of each factor will
1135 differ among rock types, especially where swelling processes in clays can modify the pore space,
1136 in which case the choice of measuring fluid becomes a critical issue. In order to account for these
1137 different effects, the design and protocol of the experimental procedure, and data management
1138 must be discussed beforehand. In this benchmark study, laboratories were asked to submit their
1139 results in a standard form (see Figure 3 in David et al. (2017)), that contained all information
1140 required for thorough knowledge of the permeability estimation process (method, fluid, pressure
1141 and temperature conditions). However, in many cases it was extremely important to receive
1142 additional information including:

1143 • Time information (absolute, relative) to identify whether the system had reached
1144 equilibrium with the applied pressure and temperature conditions and to investigate the effect of
1145 pressure cycling. The time required for pressure equilibration in low permeability material can be
1146 up to a month.

1147 • In the case of gas permeability tests, additional data at all pressure steps should be
1148 provided: (i) apparent permeability and slip factor, (ii) mean pore pressure, (iii) pressure
1149 difference, (iv) absolute pressure, (v) temperature and equation of state for the gas. We
1150 recommend against averaging values obtained with different gases.

1151 • Pressure history: the target effective pressure for a benchmarking exercise must never be
1152 exceeded during the loading stage prior to permeability measurement.

1153 Based on this additional information, detailed study of transport processes becomes possible and
1154 any deviation from the expected behavior can be analyzed.

1155

1156 6. Conclusion

1157 A benchmarking measurement exercise for low permeability material involving 24
1158 laboratories allows us to discuss the influence of (i) pore-fluid, (ii) measurement method, (iii)
1159 sample size, (iv) pressure sensitivity and (v) gas slippage effects on the permeability of the
1160 selected rock, the Grimsel granodiorite. A complementary data set on (vi) microstructures and
1161 pore size distributions, (vii) porosity and (viii) permeability modeling is presented in a
1162 companion paper. In measurements at 5 MPa effective confining pressure, an average
1163 permeability of $1.47 \cdot 10^{-18} \text{ m}^2$ was found, with a high standard deviation of $1.55 \cdot 10^{-18} \text{ m}^2$ which
1164 can be explained by the presence of few outliers (4 of 39 values). Discarding those outliers yields
1165 an average permeability of $1.11 \cdot 10^{-18} \text{ m}^2$ with a smaller standard deviation ($0.57 \cdot 10^{-18} \text{ m}^2$). The
1166 most striking result was the large difference in average permeability between gas and liquid
1167 measurements: independently of the method used, gas permeability is higher than liquid
1168 permeability by approximately a factor 2 ($k_{\text{gas}}=1.28 \cdot 10^{-18} \text{ m}^2$ compared to $k_{\text{liquid}}=0.65 \cdot 10^{-18} \text{ m}^2$).
1169 Possible explanations include (i) liquid permeability underestimated due to fluid-rock
1170 interactions (ii) gas permeability overestimated due to insufficient correction for gas slippage
1171 effects and/or (iii) gases and liquids do not probe exactly the same pore networks, and so there is
1172 no reason to expect a single permeability value. No decisive clue was found to favor one or the
1173 other explanation. However, the estimation of Knudsen numbers shows that all measurements
1174 using gas fell in the gas slippage regime and that no additional corrections are required to
1175 account for other gas flow. The larger scatter of permeability values for smaller samples seems to
1176 indicate that those samples have a volume below the REV, due to centimeter-sized mineralogical
1177 heterogeneities in the Grimsel granodiorite. Nevertheless our results are mostly self-consistent
1178 (except for few outliers) and in good agreement with other studies (Schild et al., 2001),
1179 especially the pressure dependence of permeability in the range 1 to 30 MPa. The permeability
1180 decrease with effective pressure can be described reasonably well with an exponential law,
1181 $k=k_o \cdot \exp(-\gamma P_{\text{eff}})$ with $\gamma=0.093 \text{ MPa}^{-1}$. Three examples of measurements are described in detail,
1182 using (i) the steady-state flow method, (ii) the transient pulse method and (iii) the pore pressure
1183 oscillation method: these experiments clearly show that many parameters need to be carefully
1184 controlled for successful permeability measurements in low permeability rocks. Another
1185 outcome of the benchmarking exercise was a set of good practice rules for measuring
1186 permeability in tight materials. A second round of benchmarking is currently under way with
1187 another tight material, the Cobourg Limestone. Additional challenges are expected in this
1188 benchmark (called KCL), as this rock has a permeability in the nano-Darcy range. With the
1189 experience gained with KG²B, the team is keen to take up this new challenge.

1190

1191 List of symbols

1192 $\Delta P = P_{\text{UP}} - P_{\text{DOWN}}$, pore pressure difference (Pa)

1193 μ , dynamic viscosity (Pa.s)

1194 A , sample cross-sectional area (m^2)

1195 b , Klinkenberg slip factor (Pa)

1196 BIB-SEM, broad ion beam – scanning electron microscopy

1197 C_f , pore fluid compressibility (Pa^{-1})
 1198 C_p , pore compressibility in response to pore pressure changes (Pa^{-1})
 1199 D_m , gas molecule diameter (m)
 1200 FEBEX, Full-scale Engineered Barrier EXperiment
 1201 G , gain, downstream to upstream wave amplitude ratio
 1202 GTS, Grimsel Test Site
 1203 k , permeability (m^2)
 1204 k_∞ , permeability at infinite gas pressure (m^2)
 1205 $k_{app}=k_{\text{gas}}$, apparent permeability measured with gas (m^2)
 1206 KG²B, K for Grimsel granodiorite benchmark
 1207 K_n , Knudsen number
 1208 k_o , permeability at zero effective pressure (m^2)
 1209 L , sample length (m)
 1210 MICP, mercury injection capillary pressure
 1211 N_A , Avogadro's number
 1212 P_∞ , pressure at infinite time in pulse test (Pa)
 1213 P_{ATM} , atmospheric pressure (Pa)
 1214 P_c , confining pressure (Pa)
 1215 P_{DOWN} , downstream pore pressure (Pa)
 1216 $P_{\text{eff}} = P_c - P_p$, effective pressure (Pa)
 1217 P_{MEAN} , mean pore pressure (Pa)
 1218 P_p , pore pressure (Pa)
 1219 P_{UP} , upstream pore pressure (Pa)
 1220 $Q = Q^V = dV_P / dt$, volume flow rate (m^3/s)
 1221 $Q^M = dM / dt$, mass flow rate (kg/s)
 1222 R , universal gas constant ($\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$)
 1223 REV, representative elementary volume
 1224 rH , hydrodynamic length scale / crack aperture (m)
 1225 SAFOD, San Andreas Fault Observatory at Depth
 1226 T , absolute temperature (K)
 1227 t , time (s)
 1228 V , gas volume (m^3)
 1229 V_{DOWN} , downstream tubing volume (m^3)
 1230 V_P , pore volume (m^3)
 1231 V_{UP} , upstream tubing volume (m^3)
 1232 Z , gas deviation factor
 1233 ζ , dimensionless storativity ratio
 1234 α , decay factor in pulse test (s^{-1})
 1235 β , sample storativity (Pa^{-1})
 1236 β_D , downstream reservoir storage (m^3Pa^{-1})
 1237 ϕ , porosity
 1238 γ , permeability pressure dependence factor (Pa^{-1})
 1239 η , dimensionless permeability
 1240 λ , mean free path of a gas molecule (m)
 1241 θ , phase shift between upstream and downstream waveforms (rad)
 1242 τ , period of oscillation (s)

1243

1244

1245

1246 **Acknowledgments and Data Availability Statement**

1247 This project was partially funded by a grant from the “Fondation de l’Université de Cergy-
1248 Pontoise”. We thank Belinda Godel for conducting the micro-CT study on a Grimsel
1249 granodiorite sample at CSIRO Perth. The KG²B project is supported by the GIS Géosciences
1250 Franciliennes (<http://www.geosciences-franciliennes.fr>) within the research group on “Low
1251 Permeable Media”. We thank Yves Bernabé and Steve Ingebritsen for their thorough early
1252 review of both companion papers. All the data presented in this paper can be made available
1253 upon request.

1254

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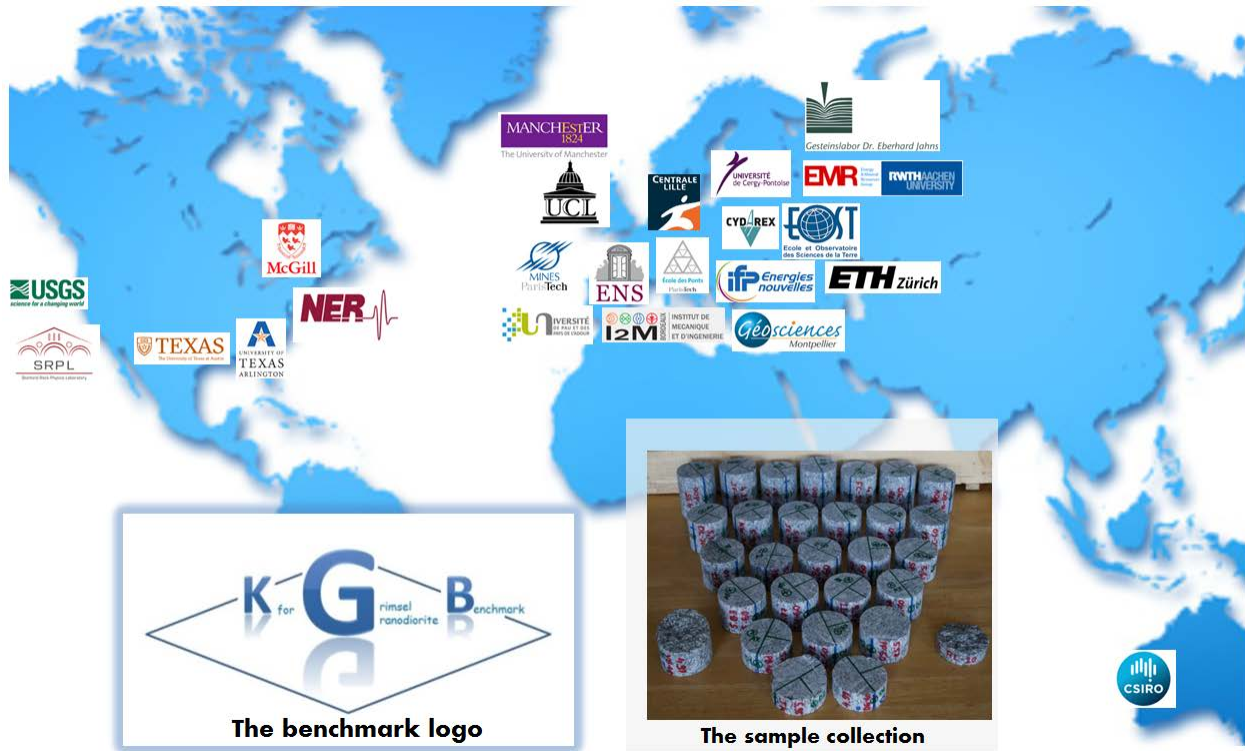
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APPENDIX A

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1471 ⁽³⁾**The KG²B Team:** the benchmark involved 24 rock physics laboratories around the
 1472 world. In Figure A1 the logo of each participating institution is shown on a world map, with the
 1473 benchmark logo and the collection of core samples sent to the participants. The name, e-mail
 1474 addresses and institution of each participant and co-author are given in Table A1.



1475

1476 **Figure A1.** World map with the participants' logos, the benchmark logo and the core sample collection sent to the
 1477 participants.

1478

| PARTICIPANTS (alphabetic order) | E-MAIL | INSTITUTION |
|---|------------------------------------|---------------------------------------|
| Alexandra AMANN HILDENBRAND / Bernhard KROOSS | alexandra.amann@emr.rwth-aachen.de | EMR group, Aachen University, Germany |
| Guillaume BERTHE / Marc FLEURY | guillaume.berthe@ifpen.fr | IFPen, France |
| Joël BILLIOTTE | joel.billiotte@mines-paristech.fr | École des Mines de Paris, France |
| Christian DAVID / Jérôme WASSERMANN | christian.david@u-cergy.fr | Université Cergy-Pontoise, France |
| Catherine DAVY | catherine.davy@ec-lille.fr | Ecole Centrale de Lille, France |
| Pierre DELAGE / Philipp BRAUN | delage@cermes.enpc.fr | ENPC, France |

| | | |
|--|-------------------------------|-------------------------------------|
| Jérôme FORTIN | fortin@geologie.ens.fr | ENS Paris, France |
| David GRÉGOIRE / Laurent PERRIER | david.gregoire@univ-pau.fr | Université de Pau, France |
| Qinhong (Max) HU | maxhu@uta.edu | University of Texas, Arlington, USA |
| Eberhard JAHNS | jahns@gesteinslabor.de | Gesteinslabor, Germany |
| Jop KLAVER | jop.klaver@emr.rwth-aachen.de | Aachen University, Germany |
| Didier LASSEUX/Yves JANNOT/Alain SOMMIER | didier.lasseux@u-bordeaux.fr | I2M TREFLE, Bordeaux, France |
| Roland LENORMAND | roland.lenormand@cydarex.fr | Cydarex, France |
| David LOCKNER | dlockner@usgs.gov | USGS Menlo Park, USA |
| Laurent LOUIS / Gregory BOITNOTT | llouis@ner.com | New England Research, Vermont, USA |
| Claudio MADONNA / Florian AMANN | claudio.madonna@erdw.ethz.ch | ETH Zurich, Switzerland |
| Philip MEREDITH / John BROWNING / Tom MITCHELL | p.meredith@ucl.ac.uk | UCL Earth Sciences, UK |
| Franck NONO / Didier LOGGIA | nono@gm.univ-montp2.fr | Université Montpellier II, France |
| Peter POLITO | peter.Polito@jsg.utexas.edu | University of Texas, Austin, USA |
| Thierry REUSCHLÉ | thierry.reuschle@unistra.fr | EOST Strasbourg, France |
| Ernie RUTTER | ernie.rutter@manchester.ac.uk | Univ. Manchester, UK |
| Joël SAROUT / Lionel ESTEBAN | joel.sarout@csiro.au | CSIRO, Perth, Australia |
| Patrick SELVADURAI | patrick.selvadurai@mcgill.ca | McGill University, Canada |
| Tiziana VANORIO / Anthony CLARK | tvanorio@stanford.edu | Stanford University, USA |

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Table A1: The KG²B Team: list of participants and co-authors

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APPENDIX B

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1483 Each participating laboratory was assigned a number following the core sample order, from
 1484 #01 for the sample closest to the tunnel (at 4.17 m) to #24 for the deepest sample in the borehole
 1485 (at 5.95 m). Table B1 provides sample location in the tunnel, the length and diameter of the sub-
 1486 core drilled from the original core, and the method and pore fluid used for permeability
 1487 measurements. Table B2 provides the permeability values measured at 5 MPa and (when
 1488 available) at in situ stress 30 MPa. Note that porosity and radial permeability values have also
 1489 been included in the table, although they are discussed in the companion paper.

1490

| | Distance from tunnel (m) | Sub-cored sample length (mm) | Sub-cored sample diameter (mm) | Method for permeability estimation | Fluid used for permeability measurement |
|--------|--------------------------|------------------------------|--------------------------------|---|---|
| Lab#01 | 4.17 | 25.6 | 25 | Transient pulse & Step-decay (transient) | GAS (Argon) |
| | 4.17 | 27.2 | 25 | | |
| | 4.17 | 29.5 | 25 | | |
| Lab#02 | 4.25 | 83 | 40 | Steady-state flow | LIQUID (water) |
| Lab#03 | 4.35 | 94 | 83 (no subcoring) | Steady-state flow | LIQUID (non degased water) |
| Lab#04 | 4.45 | 80 | 40 | Transient pulse, Pore pressure oscillation & Modeling | LIQUID (brine) |
| Lab#05 | 4.63 | 25 | 38 | Transient pulse & Modeling | GAS (Nitrogen) |
| Lab#06 | 4.78 | 86 | hollow cylinder 83/60 | Steady-state flow (radial flow) | LIQUID (distilled water) |
| Lab#07 | 4.94 | 1 to 5 | chips | Transient pulse | GAS (Air) |
| Lab#08 | 4.99 | 21.4 | 19.5 | Steady-state flow | GAS (Nitrogen) |
| | | 20.8 | 19.5 | | |
| Lab#09 | 5.04 | 50 | 40 | Transient pulse | GAS (Nitrogen) |
| | | | | Steady-state flow | LIQUID (deaerated tap water) |
| | | | | Transient pulse | GAS (Argon) |
| Lab#10 | 5.11 | 31.5 | 29.9 | Steady-state flow | LIQUID (water) |
| | 5.11 | 28.3 | 30 | | |
| | 5.11 | 28.8 | 30 | | |
| Lab#11 | 5.18 | 39.3 | 25.5 | Step-decay (transient) | GAS (Nitrogen) |
| Lab#12 | 5.31 | 40 | 20 | Transient pulse | GAS (Argon) |
| Lab#13 | 5.37 | 38 | 38 | Steady-state flow | LIQUID (deionised water) |
| | 5.37 | 20 | 20 | Steady-state flow | |
| | 5.37 | | | Pore pressure oscillation | |
| Lab#14 | 5.42 | 41.3 | 64.6 | Steady-state flow | GAS (Argon) |

| | | | | | |
|--------|------|--------------|---------------------|--|-----------------------------|
| Lab#15 | 5.47 | 15 | 15 (cube) | Microstructure analysis (MICP) & Modeling | NA |
| Lab#16 | 5.52 | 44 | 38 | Complex transient technique | GAS (Nitrogen) |
| | 5.52 | 42 | 38 | | |
| Lab#17 | 5.57 | 21.5 | 25.4 | Transient pulse | GAS (Argon) |
| Lab#18 | 5.67 | 30 | 26 | Pore pressure oscillation & Transient pulse | GAS (Argon) |
| | 5.67 | 30 | 26 | | |
| | 5.67 | 30 | 26 | | |
| Lab#19 | 5.72 | 33.6 | 83.3 (no subcoring) | Steady-state flow | LIQUID (deionized water) |
| Lab#20 | 5.77 | 49.9 | 25.4 | Transient pulse | GAS (Helium and Nitrogen) |
| | 5.77 | 49.3 | 25.3 | | |
| | 5.77 | 35.6 | 25.4 | | |
| Lab#21 | 5.83 | 38.9 | 39 | Steady-state flow | GAS (Nitrogen) |
| Lab#22 | 5.9 | 39 | 25.4 | Transient pulse | GAS (Argon) |
| | | 38 | 25.4 | | |
| | | 38.9 | 25.4 | | |
| Lab#23 | 5.95 | 24.2 | 38.3 | Steady-state flow | LIQUID (degassed tap water) |
| | | | | Steady-state flow & Transient pulse | GAS (Helium and Nitrogen) |
| Lab#24 | 5.95 | thin section | thin section | Microstructure analysis (BIB-SEM) & Modeling | NA |

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Table B1: List of samples with distance to the borehole mouth, length and diameter, and methods used for permeability estimation with corresponding fluids.

1493

| LAB# | Fluid | Method | Porosity (%) | Axial PERM@5_MPa (10^{-18} m^2) | Axial PERM@30_MPa (10^{-18} m^2) | Radial PERM@5_MPa (10^{-18} m^2) |
|------|--------|--------|--------------|---|--|--|
| | | | | | | |
| #01 | Gas | PLS | | 1.1 | | |
| | Gas | PLS | | 1.5 | | |
| | Gas | PLS | | 1.63 | | |
| #02 | Liquid | SST | | 0.43 | 0.03 | |
| #03 | Liquid | SST | 0.6 | 0.6 | 0.04 | |
| #04 | Liquid | PLS | 0.62 | 0.43 | 0.055 (*) | |
| #04 | Liquid | OSC | 0.62 | 0.294 | | |
| #05 | Gas | PLS | 0.6 | 1.46 | 0.064 (*) | |

| | | | | | | |
|-----|--------|-----|------|-----------|-----------|---------------|
| #06 | | SST | | | | 0.84 @1.75MPa |
| #07 | Gas | PLS | 0.7 | 2.6 (*) | | |
| #08 | Gas | SST | | | | 0.243 |
| | Gas | SST | | 0.199 | | |
| #09 | Gas | PLS | 0.8 | 1.3 | | |
| | Liquid | SST | 0.8 | 0.94 | | |
| | Gas | PLS | 0.8 | 1.49 | | |
| | Gas | PLS | 0.8 | 1.37 | | |
| #10 | Liquid | SST | 0.46 | 0.5 | | |
| | Liquid | SST | 0.17 | 0.05 (**) | | |
| | Liquid | SST | 0.51 | 0.73 | | |
| #11 | Gas | PLS | 1.16 | 1.28 | | |
| | Gas | PLS | 0.78 | 1.18 | | |
| | Gas | PLS | 1.18 | 1.26 | | |
| #12 | Gas | PLS | 0.52 | 1.12 | | |
| #13 | Liquid | SST | | 8.35 (**) | 2.06 (**) | |
| | Liquid | SST | | 4.73 (**) | | |
| | Liquid | SST | | 0.579 | | |
| | Liquid | OSC | | 0.906 | 0.277 | |
| #14 | Gas | SST | 0.73 | 1.91 | 0.189 | |
| #16 | Gas | PLS | 0.23 | 1.69 | | |
| | Gas | PLS | 0.43 | 1.81 | 0.155 | |
| #17 | | PLS | 1.8 | | | 0.66 |
| #18 | Gas | OSC | 1.03 | 1.84 | | |
| | Gas | OSC | 1.03 | | | 0.843 |
| | Gas | OSC | 1.03 | | | 0.501 |
| #19 | Liquid | SST | | 1.08 | | |
| #20 | Gas | PLS | 0.51 | 0.579 | | |
| | Gas | PLS | 0.51 | 0.342 | | |
| | Gas | PLS | 0.88 | 1.69 | | |
| | Gas | PLS | 0.88 | 0.375 | | |
| | Gas | PLS | 1.29 | 1.75 | | |

| | | | | | | |
|------------|--------|-----|------|----------|----------|-------|
| | Gas | PLS | 1.29 | 1.21 | | |
| #21 | Gas | SST | 1.5 | 0.83 | 0.07 (*) | |
| #22 | Gas | PLS | 0.7 | 0.795 | | |
| | Gas | PLS | 0.5 | | | 0.825 |
| #23 | Liquid | SST | 0.26 | 5.4 (**) | | |

Table B2: Permeability and porosity values. (*) extrapolated values; (**) outliers discarded from the global analysis (SST=Steady-state flow method, PLS=Transient pulse method, OSC=Oscillating pore pressure method).