

KG²B, a collaborative benchmarking exercise for estimating the permeability of the Grimsel granodiorite-Part 2: modelling, microstructures and complementary data

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2	KG ² B, a collaborative benchmarking exercise
3	for estimating the permeability of the Grimsel granodiorite:
4	modeling, microstructures and complementary data
5	
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7	
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14	Key Points:
15 16	• A benchmarking exercise involving 24 laboratories was organized to estimate the permeability of the Grimsel granodiorite
17 18	• The microstructures of the Grimsel granodiorite were analyzed and quantified using BIB-SEM, micro-CT scanning, MICP and NMR techniques
19 20 21	• Permeability predictions from different models using microstructure data as input parameters are in good agreement with measurements

22 Abstract

A benchmarking exercise involving 24 laboratories was organized for measuring and 23 modeling the permeability of a single low permeability material, the Grimsel granodiorite. To 24 complement the data set of permeability measurements presented in a companion paper, we 25 focus here on (i) quantitative analysis of microstructures and pore size distribution, (ii) 26 permeability modeling and (iii) complementary measurements of permeability anisotropy and 27 poroelastic parameters. BIB-SEM, micro-CT, MICP and NMR methods were used to 28 characterize the microstructures and provided the input parameters for permeability 29 modeling. Several models were used: (i) basic statistical models, (ii) 3D pore network and 30 effective medium models, (iii) percolation model using MICP data and (iv) free-fluid model 31 using NMR data. The models were generally successful in predicting the actual range of 32 33 measured permeability. Statistical models overestimate the permeability because they do not adequately account for the heterogeneity of the crack network. Pore network and effective 34 medium models provide additional constraints on crack parameters such as aspect ratio, 35 aperture, density and connectivity. MICP and advanced microscopy techniques are very 36 useful tools providing important input data for permeability estimation. Permeability 37 measured ~orthogonal to foliation is lower that ~parallel to foliation. Combining the 38 experimental and modeling results provides a unique and rich data set. 39

40

41 **1. Introduction**

Following a workshop on «The challenge of studying low permeability materials» 42 that was held at Cergy-Pontoise University in December 2014, a benchmark exercise in 43 which several laboratories estimate the permeability of a single material was proposed to the 44 attendees. The selected material was the Grimsel granodiorite (Switzerland) and the 45 benchmark was named the "KG2B" project, from "K for Grimsel Granodiorite Benchmark" 46 (David et al., 2017). Multiple objectives were defined: (i) to compare the results for a given 47 method, (ii) to compare the results between different methods, (iii) to analyze the accuracy of 48 each method, (iv) to study the influence of experimental conditions (especially the nature of 49 pore fluid), (v) to discuss the relevance of indirect methods and models, and finally (vi) to 50 suggest good practice for low permeability measurements. The permeability measurements 51 are presented in the companion paper. Here we will focus on item (v) and present the results 52 of microstructure analyses and permeability modeling. 53

Fluid flow processes in rocks are controlled by the geometrical properties of pore 54 and/or cracks and the topology of the pore/crack network. Linking permeability to 55 microstructural properties has always been a challenge in rock physics. A first step is to 56 acquire high quality data that allow thorough characterization of the pore space, preferably in 57 3D. As we are dealing with a crystalline rock, the focus is on cracks rather than pores. Cracks 58 in rocks can be approximated as planar features with small width or aperture, randomly 59 oriented or not in a 3D medium. Due to their limited resolution, optical microscopy 60 techniques are not well-suited for the study of cracks. SEM studies have been commonly 61 used to analyze cracks on thin-sections at high magnification. Ion beam milling is 62 recommended to avoid biased interpretation of the microcrack morphology and statistics 63 (Wong, 1982). Crack statistics provided by SEM studies can be from 2D analyses, from 64 which 3D parameters (like crack surface per unit volume) can be inferred using stereology 65 (Fredrich & Wong, 1986). Recent advances in ion polishing now allow improved images of 66 pore structures and crack networks to be obtained using BIB-SEM (Klaver et al., 2015), or 67 even 3D structures from FIB-SEM image stacks (Holzer et al., 2004). Wood's metal injection 68

into the pore space greatly enhance pore and crack detection and analysis on SEM images 69 (Hu et al., 2012; Klaver et al., 2015). High resolution micro-CT techniques have become 70 widely used to investigate the three-dimensional distribution of minerals and pores (Baker et 71 al., 2012; Godel, 2013). With improvement of technology and analytical tools, sub-micron 72 resolution can now be achieved with micro-CT imaging methods, but sometimes even this is 73 insufficient to identify tiny cracks in crystalline rocks. One major advantage of micro-CT is 74 75 that the technique is non-destructive and can be applied on centimeter scale plugs. Pore or crack size distributions can be obtained by image analysis on SEM images (2D analysis) or 76 micro-CT reconstructed volumes (3D analysis), and also by conducting mercury injection 77 78 capillary pressure (MICP) tests on small plugs. MICP is commonly used in petrophysical studies to obtain the throat size distribution and capillary breakthrough pressure by injecting 79 mercury under increasing pressure (Hu et al., 2015). The throat size distribution given by 80 MICP does not actually match the pore size distribution of the rock because of constrictions 81 and ink-bottle effects in the pore space (Abell et al., 1999) but provides a first-order 82 approximation that can be used in models. Other methods that provide insight into the pore 83 size distribution include the gas adsorption (or BET) method (Schull, 1948) and NMR 84 85 techniques (Josh et al., 2012).

Permeability models using microstructural data as input parameters have evolved 86 since the pioneering work of Kozeny in the 1920s (Kozeny, 1927). A main challenge of all 87 permeability models is to identify the characteristic length scale controlling permeability. 88 This general statement rises from the permeability having the unit of squared length, but 89 other factors like pore size variability and connectivity are also very important. Many 90 different approaches have been proposed (Guéguen & Palciauskas, 1994). Originally based 91 on the Kozeny-Carman equation (Kozeny, 1927), the equivalent channel model states that the 92 characteristic length scale is the hydraulic radius, defined as the ratio between the pore 93 volume and the pore surface area (Paterson, 1983; Walsh & Brace, 1984). In the equivalent 94 channel model, permeability depends on bulk properties related to the pore space (volume to 95 surface ratio, porosity, tortuosity - an ill-defined parameter related to the increased path 96 length in a "tortuous" pore space) that, with the exception of tortuosity, are measurable at the 97 sample scale. Statistical and effective medium models take advantage of the statistics of pore 98 or crack geometries. For example, Gueguen & Dienes (1989) proposed a statistical model for 99 crystalline rocks in which permeability depends on the mean crack aperture and radius, with 100 cracks modeled as penny-shaped objects, on the average distance between cracks and on the 101 fraction of connected cracks (which can be estimated from percolation theory). Only 102 ensemble averages are estimated with limited input of the crack network topology. In 103 104 contrast, network topology is taken into account in percolation and network models. For the percolation model proposed by Katz & Thompson (1986) the characteristic length scale is the 105 so-called critical conductance (linked to a critical crack size), defined as the smallest 106 conductance in the sample-spanning sub-network made of conductances larger than the 107 critical conductance. The critical length scale can be obtained from the breakthrough pressure 108 in MICP experiments using Washburn's equation (Hu et al., 2015). Percolation models are 109 supposed to work best when the pore size distribution is very wide. In heterogeneous porous 110 media, preferential flow paths (with similar properties as the critical percolation subnetwork) 111 are more likely to occur (David, 1993). 112

Pore network modeling has been widely used for permeability prediction (Bauer et al., 2012; Bernabé et al., 2003). In such models fluid flows in pipes or cracks forming the bonds of a 3D (or 2D) lattice with fixed topology (e.g. a cubic lattice). The geometrical properties of the conducting elements follow the pore/crack size statistics obtained by SEM analysis or

MICP (David et al., 1990). The flow equations are solved at each node and permeability is 117 directly derived from Darcy's law, so does not depend on statistical averages (De Boever et 118 al., 2012). Pore network modeling also allows the bond occupancy probability to be varied, 119 so that networks with different average coordination number (or connectivity) can be 120 considered for permeability estimation (David, 1993). Several of the permeability models 121 mentioned above were tested by Castelevn et al. (2011) on series of oolitic limestones from 122 the Paris basin. Since the pore size distribution of these rocks was not very heterogeneous, 123 hence statistical and network models were successful in matching the measured permeability, 124 while the percolation model underestimated the permeability by about one order of 125 magnitude. 126

One of the objectives of the KG²B benchmark was to conduct permeability modeling. Several models were selected by the participants. To achieve successful modeling, as discussed above, information is required about the rock microstructure (such as porosity, pore/crack aperture and length distribution, and connectivity). We will present the results of a thorough microstructural analysis and from NMR and MICP tests, as well complementary data on anisotropy and poroelastic parameters measured on a voluntary basis by some participants.

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135 **2. The KG²B Project: Summary**

In total 30 laboratories from 8 different countries volunteered to participate in KG²B. 136 and we received results from 24 laboratories that form the "KG2B Team". The complete list 137 of participants who sent results is given in Appendix A. A dedicated website https:/labo.u-138 cergy.fr/~kggb/ was created with information on the benchmark, including a web page where 139 the progress of the project could be followed on the "KG2B-wheel", which was updated as 140 soon as results were received from any of the participants. It took one year to collect all of the 141 results. In total we collected 45 permeability values, including 39 measured values and 6 142 results from modeling, on which this paper will focus. Statistical, network, percolation and 143 effective medium models were used. We add a seventh modeling result in which a rock 144 sample is treated as an RC (Resistance + Capacitor) low pass filter during pore pressure 145 oscillation tests. 146

The Grimsel granodiorite was obtained from the Swiss Grimsel test site, a 450 meter 147 deep Underground Research Laboratory (URL). The 950 meter long and 3.5 meter diameter 148 tunnels were excavated in 1983 by a full face Tunnel Boring Machine (TBM) in hard rocks, 149 mainly granite and granodiorite, at an altitude of 1730 m in the Central Aar Massif in 150 Switzerland. The TBM excavation method limited perturbation of the host rock, with a quite 151 small Excavation Damage Zone (EDZ) around the tunnel (Egger, 1989). Along the tunnel, 152 major damage zones are located in meter scale shear zones or widely spaced discontinuities 153 caused by regional deformation. Two cores of Grimsel granodiorite, each about one meter 154 long and of diameter 85 mm, were provided by our Swiss colleagues in September 2015. 155 These cores were retrieved at a distance of 4 to 6 meters from the tunnel of the Grimsel test 156 site, far away from the EDZ influence. The cores were cut into small blocks at lengths 157 158 requested by each participant (between 2 and 10 cm). A grain shape foliation is visible on the cores at an angle of about 20-30° with respect to the core axis. The foliation is related to 159 compositional banding of alternating dark biotite layers and quartz-rich layers (Schild et al., 160 2001). Natural and induced cracks have been observed in past studies (e.g. Smith et al., 161 2001). In particular there is a natural interconnected network of cracks producing about 1% 162

porosity in the granitic matrix. Stress release due to drilling and sample preparation outside the URL seems to be responsible for larger micro-crack apertures than those observed directly in situ (Schild et al., 2001). As some laboratories provided permeability measurements in directions other than that required of the participants (i.e. the core axis direction), we will also discuss the permeability anisotropy.

The detailed analysis of the permeability measurements is given in the companion 168 paper. Let us recall the most important results. For the whole data set of 39 measurements, 169 the average permeability was $1.47 \ 10^{-18} \ m^2$; however 4 outliners were identified and removed, 170 leading to an average permeability of $1.11 \ 10^{-18} \ m^2$ and a standard deviation of $0.57 \ 10^{-18} \ m^2$. 171 A striking result was the large difference between measurements using gas or liquid as the 172 pore fluids: the permeability to gas was about twice as large as the permeability to liquid 173 $(k_{gas}=1.28 \ 10^{-18} \ m^2, k_{liquid}=0.65 \ 10^{-18} \ m^2)$. The model predictions presented in this paper will 174 be compared to those values. 175

We will use the same convention as in the companion paper for presenting the data set. Each lab was assigned a number in increasing order with respect to the distance between their sample and the tunnel. Lab#01 worked on the sample closest to the tunnel, and Lab#24 on the farthest sample.

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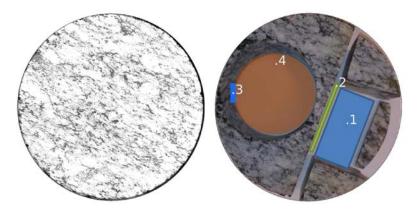
181 **3. Microstructure and porosity analyses**

182 3.1. Quantitative Microstructural Analysis

Here we describe efforts to determine the main fluid flow pathways at the centimeter scale, which is the relevant scale for the laboratory experiments. To this purpose, several direct imaging methods were used: automated optical microscopy of thin-sections, and Broad Ion Beam/Scanning Electron Microscopy (BIB-SEM) of intact or Wood's Metal (WM) impregnated samples.

188 *3.1.1 Methods*

189 Two adjacent blue dye impregnated thin-sections of standard size were prepared perpendicular and parallel to the core axis from the original core sample (Figure 1). Thin-190 sections were automatically scanned with the Virtual Petroscan (ViP) (Schmatz et al., 2010) 191 in plane polarized and crossed polarized light (PPL and XPL). Porosity was segmented from 192 the PPL image map (approximately 20,000 * 12,000 pixels, pixel size of 1.4 µm i.e. 2.8 cm * 193 1.68 cm \approx 4.7 cm²) by unsupervised iso-cluster classification and re-grouped into porosity 194 and matrix based on visual inspection followed by a boundary cleaning operation (dilation), 195 all in ArcGIS 10. 196



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Figure 1. Grimsel granodiorite core (left) and sampling (right) for thin sections perpendicular (1), and parallel
 (2) to the core axis, for BIB-SEM and WM (3), and plug for permeability measurements for Lab#23 (4).

From the same core sample, one subsample was prepared by BIB polishing to investigate the 200 microstructure by SEM. Details on this technique are given by Klaver et al. (2012). Four 201 areas were mapped at high resolution $(10,000 - 20,000 \times magnification)$ for quantitative 202 analyses of the pore space. Porosity was segmented by a seed-and-grow algorithm (Jiang et 203 204 al., 2015) and manually corrected where needed. Pore spaces with circularity below 0.2 and an axial ratio above 3 were automatically classified as cracks (including grain boundary 205 206 cracks). Average crack intensity (expressed in crack number/m) and average crack thickness were calculated based on each pixel row from every map. 207

Another sub-sample was injected at 500 MPa with Wood's metal (WM), which is a nonwetting alloy with wetting properties similar to mercury and which solidifies at room temperature. This method resembles Mercury Intrusion Porosimetry (Klaver et al., 2015). We expected insignificant damage to the pores due to the material strength.

212 *3.1.2 Visible porosity and pore size distributions*

Over 60,000 pores were segmented from the thin-sections and the largest pores were approximately 0.3 millimeter in equivalent diameter (Figure 2). The thin-sections show different visible porosities: 0.71% and 1.55% for the parallel and perpendicular sections, respectively. This difference most likely occurred because the thin-sections are not wholly representative at the centimeter scale regarding porosity. Alternatively, this contrast may owe to highly anisotropic pore shapes with large pore diameters parallel to the section and small diameters perpendicular to the section.

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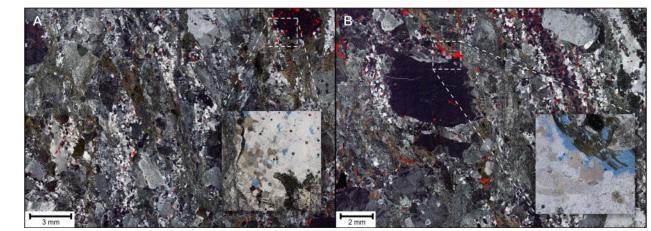
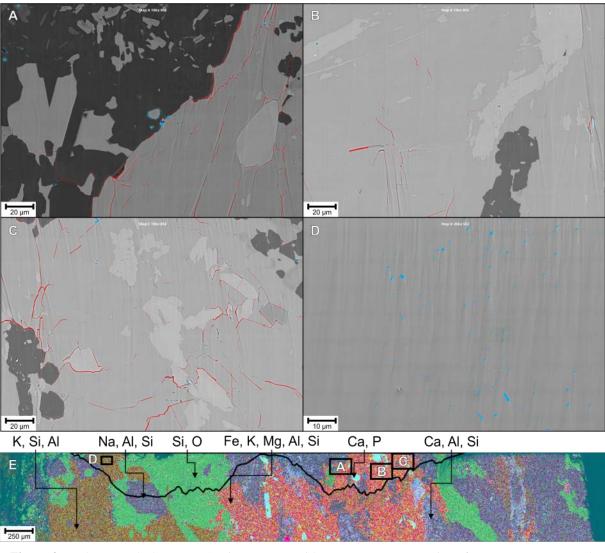


Figure 2. ViP XPL maps overlain by pore segmentation in red of the parallel thin-section (A) and perpendicular thin-section. The insets show the blue dye filled pores in PPL.

225 From SEM, the weighted average porosity in segmented maps perpendicular to the core direction is 0.45% with a porosity of 0.54%, 0.16%, 0.64% and 0.39% in maps A, B, C and 226 D, respectively (Figure 3A-D). However, most of the pore space (average 0.36%) is 227 associated with cracks, indicated in red in the figure. In map D, no cracks were counted, and 228 all pores are interpreted as isolated pores within a single phase consisting mainly of K, Si, Al 229 (Figure 3E) and interpreted as K-feldspar. Other pore space-mineral associations are: 1) 230 cracks at grain boundaries and within biotite (Fe, K, Mg, Al, Si); 2) minor pores and cracks 231 along albite/plagioclase (Na/Ca, Al, Si) grain boundaries; 3) pores and cracks at quartz (Si, 232 O) grain boundaries and fluid inclusions; and, 4) fluid inclusions in apatite (Ca, P). 233

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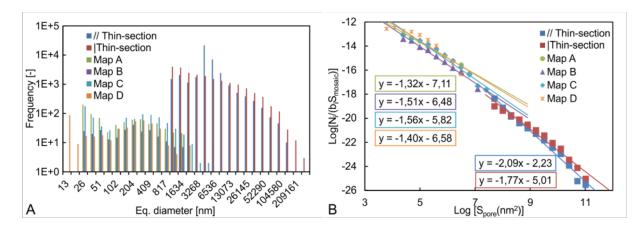


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Figure 3. Backscattered Electron (BSE) image maps with pore space segmentation of maps A-D. Interpreted
 cracks are in red and pores in cyan. The EDS (Energy Dispersive Spectroscopy) overview map shows the
 locations of the maps with respect to BIB cross-sections and elemental compositions (E).

The pore size distributions (PSDs) of the imaged thin-sections show a clear increase in pore frequency with decreasing equivalent diameter to about 6 μ m (Figure 4A). The PSDs of the BIB-SEM maps show a clear peak at 200 - 300 nanometer equivalent diameter and another apparent increase below 100 nanometers. These smaller segmentations are below 18 pixels in size. They are interpreted as noise and hence excluded from the analyses in Figure 4B, which

- shows normalized frequencies (number of pores divided by the imaged area and bin width).
- Taking into account both pores and interpreted cracks, only maps B and C show comparable best fits. The fact that the normalized PSDs do not show uniform best fits indicates that pore
- space may have been underestimated due to the large grain sizes and other heterogeneities.
- 247 space may have been underestimated due to the large grain sizes and other neterogenetices. 248 Considering only the interpreted cracks in red (Figure 3A-D), the average crack thickness is
- 248 283 nm, within the visible range in Figure 4A. The average crack intensity over maps A-C is
- 250 14749 cracks/m.
- 251



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Figure 4. A) PSDs of segmented porosity in the thin-sections and BIB-SEM maps. B) Plot showing the PSDs,
 normalized over the imaged area for all segmented pores above 18 pixels in size, i.e. 6.5 μm, 144 and 72 nm for
 the thin-sections, map A-C and map D respectively.

3.1.3 Pore connectivity

The WM-filled cross-section is shown in Figure 5; the minerals are mostly biotite, albite, plagioclase and quartz (Figure 5A). Most of the WM is located in the cracks. Most of the WM-filled cracks seem to be associated with biotite (Figure 5B), and have widths of approximately 0.2-1 μ m (Figure 5C).

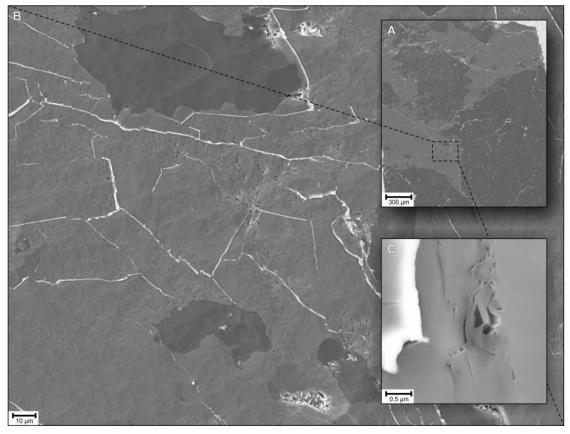


Figure 5. (A) Overview BSE image showing the WM intrusion (in white) at the sample scale. (B) Higher
 definition image map of the biotite dominated area shows connected crack networks in 2D. (C) WM-filled crack
 200 nm in width next to isolated pores.

266 3.1.4 Synthesis of microstructural analysis

Macroscopic investigation reveals minerals of several centimeters in size, indicating that microstructural investigations limited to two adjacent thin-sections are most likely not representative of porosity at the centimeter scale. The variability in porosity at the thinsection scale is significant (with values of 0.71% and 1.55%). However, the expected order of magnitude of porosity (about 1%) is attained. A greater number of realizations would be necessary to achieve representativeness in a statistical sense.

In addition ViP- BIB-SEM investigations provided PSDs, enabling comparison with bulk 273 measurements, and revealed pore-mineral associations which can help with up-scaling 274 scenarios. Most of the pore space is visible with optical microscopy, indicating that the 275 relevant pores for storage are in the sub-millimeter to micrometer range. However, the pore 276 connections are most likely in the submicron range as indicated by the BIB-SEM 277 278 investigations, which revealed significant cracks and grain-boundary features in that range. The WM-BIB-SEM investigations also indicate that crack flow is the most important 279 transport process. The WM-filled cracks tend to be wider and are connected in 2D, perhaps 280 opened due to the high pressures (while closing smaller cracks). This hypothesis is the 281 subject of ongoing research. 282

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This analysis is complemented by simplified calculations of permeability in Section 4, which assume that biotite is the main contributor to fluid flow, and that at room conditions the rock has an average porosity of 0.45%, a crack aperture of 283 nm, and a crack density of 14749 cracks/m. These estimates provide insights into the key factors controlling the transport properties and flow paths identified by microscopy. 289

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3.2. Microstructure study using micro-CT

A micro-tomography study was conducted at CSIRO Perth on a small sample of 291 Grimsel granodiorite with 4 mm diameter and 10 mm length. The micro-CT equipment is the 292 XradiaTM Versa microtomography system (XVRM126). This system is composed of an X-293 Ray source, a rotating sample holder and an X-Ray detection system. The source is generated 294 by the impact of a focused beam on a thin target; the spot size can vary from 1 to 5 µm 295 depending on the operating conditions. The diverging geometry of the X-Ray results in a 296 magnification of the object. The X-Ray source used allows application of voltage and power 297 ranges from 40 to 160 kV and from 4 to 10 W respectively. The X-Ray detector comprises 298 several lenses mounted on a turret and the detector itself picking up X-Ray images of the 299 sample. The mounted lens ranges from magnification level 0.4X to 40X covering resolutions 300 from few tens of µm to 0.7 µm in optimal conditions. The latter resolution can be obtained on 301 5 mm diameter samples. The images are generated by acquisition of a set of radiographs, 302 while rotating the sample stepwise through a 360° rotation. For the present study, the voxel 303 size was 5 µm, enough to identify tiny pores (as shown also in BIB-SEM analysis) but 304 insufficient to see the cracks, which have thicknesses dominantly in the sub-micron range. In 305 306 Figure 6a, four density maps with grey-scale coding are shown on cross-sections at different heights from top to bottom. The brighter areas correspond to denser minerals. Clearly the 307 rock appears very heterogeneous from the mineralogical viewpoint. The foliation oriented 308 from left to right on the images is visible. Magenta circles highlight the presence of tiny, 309 probably isolated pores, as discussed in the BIB-SEM section. 3D reconstructions of the 310 sample are shown in Figure 6b. Again heterogeneity is ubiquitous. The reconstructions 311 confirm that, at this scale, the investigated volume is below the REV (as discussed in the 312 companion paper). The pore space reconstruction (excluding cracks) shows that the tiny 313 pores are isolated and should not contribute significantly to macroscopic flow, unless 314 connected through the crack network. Generally the pores are uniformly distributed in the 315 rock, although clusters are sometimes observed (Figure 6b). As mentioned in the BIB-SEM 316 analysis, fluid flow is controlled by a 3D network of cracks that is mostly located at grain 317 boundaries or within biotite. Indeed such cracks not visible at the micro-CT scale were filled 318 319 with Wood's metal (Figure 5) after WM injection.

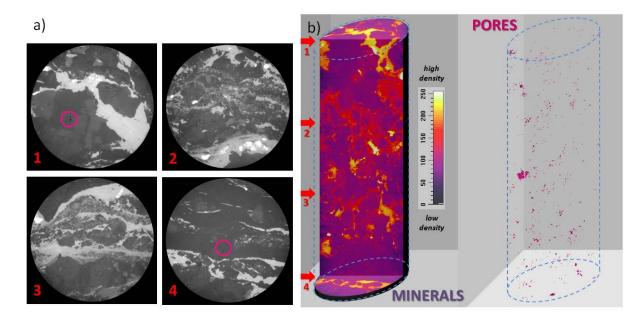
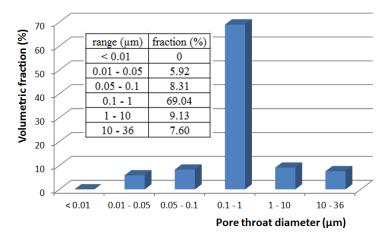


Figure 6. Micro-CT scan analysis of a small sample of Grimsel granodiorite (diameter 4mm, length 10 mm). a)
 Four sections at different heights from top to bottom; the pink circles highlight some pores (black spots). b)
 Left, 3D reconstruction of the matrix density map (8 bits color coding), with red arrows indicating locations of
 the four cross-sections; right, 3D map showing isolated or clustered tiny pores. The cracks evidenced by BIB SEM analysis could not be resolved by this technique.

326 3.3 Pore Structure Analysis with MICP and NMR

Mercury injection capillary pressure (MICP) can be used to measure pore structure 327 characteristics such as total pore area, bulk density, porosity, pore throat distribution, 328 permeability and tortuosity (Hu et al., 2015). Liquid mercury, which has a high surface 329 energy and is non-wetting, is forced into the pore space under increasing capillary pressure. 330 As mercury pressure increases, smaller pore throats are invaded. Mercury will only invade a 331 pore throat when a sufficient mercury pressure, inversely proportional to the throat diameter 332 is applied (Gao & Hu, 2013). This is expressed through the Washburn equation, which 333 assumes a cylindrical pore shape (Washburn, 1921). For a 1cm long cubic sample a typical 334 MICP test takes 3-4 hours to complete, with measurable pore-throat size ranging from 3 nm 335 to 36 µm for low-porosity (<5%) samples. Figure 7 shows the results obtained from MICP on 336 the Grimsel granodiorite. 337



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Figure 7. Throat size distribution derived from MICP on the Grimsel granodiorite.

The porosity provided by the mercury injection test at the highest capillary pressure is 0.59%, lower but close to the average porosity found by other teams using different techniques (0.77%). This is probably linked to the smaller size of the MICP plug compared to the permeability samples. A very strong peak is observed on the histogram, corresponding to a pore throat radius in the range between 0.1 and 1 μ m.

In Figure 8 we present the results of NMR spectroscopy conducted on five small 345 plugs (diameter 25 mm, length 22 mm) saturated with water under vacuum and 13 MPa 346 hydrostatic pressure, using a 2 MHz GeoSpec2 from Oxford-GIT Ltd. Low-field proton 347 NMR provides the transverse relaxation time T₂ from which bulk and bound water 348 distributions can be extracted (Dillinger & Esteban, 2014). For the saturated state (Figure 8a), 349 350 the results are very consistent, with one strong peak at $T_2=0.15$ ms and two weakest ones in the range 10 - 100 ms. Short relaxation times usually correspond to bound water (e.g. 351 capillary pore sizes and clay bound water) and long relaxation times correspond to free (or 352 mobile) water; Figure 8 shows that, in the Grimsel granodiorite, most of the water in the pore 353 space is bound water at 13 MPa hydrostatic pressure. 354

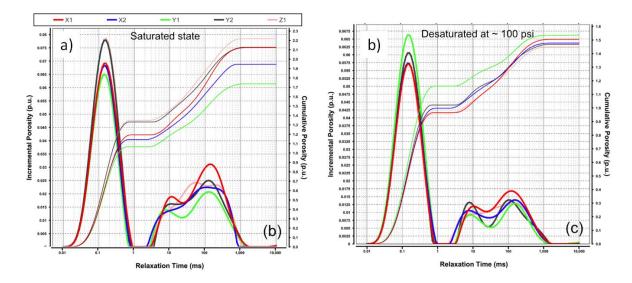


Figure 8: NMR transverse relaxation time T_2 spectra and associated cumulative porosity in five small core plugs of Grimsel granodiorite (a) under water saturated conditions, and (b) a desaturated state at ~ 6.9 bars.

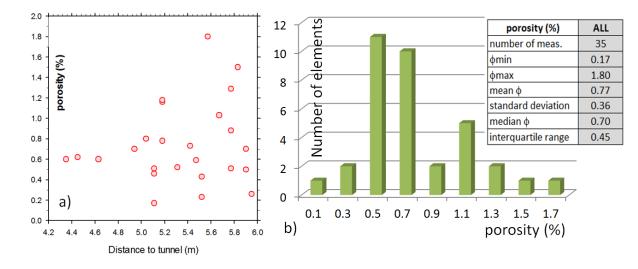
For these five plugs the NMR porosity ranges from 1.75 to 2.2 % (average 2.03 %) which agrees with the range found with direct measurements (see next section). The five samples were also desaturated by centrifuge to achieve an equivalent capillary pressure of \sim 6.9 bars, and NMR measurements were repeated (Figure 8b). Such experiments allow one to evaluate the relative amount of mobile water and irreducible water. Note that sample Y1 has a different behavior than the others.

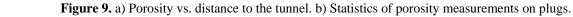
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365 3.3. Porosity measured on plugs

We collected 35 porosity values using different methods (helium pycnometry, triple weight method, mercury injection, NMR). As with permeability, no systematic trend was found when plotting porosity values as a function of the distance to the tunnel (Figure 9a). However more consistent values seem to occur in the first eighty centimeters. The average porosity is 0.77% (Figure 9b) with a standard deviation of 0.36%.





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These values measured on macroscopic samples are in good agreement with those derived from thin section analyses reported in section 3.

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4. Permeability Estimation from Models

About 13% of the permeability estimates collected during the benchmarking exercise were obtained from model predictions. Several models have been used and can be classified as statistical, percolation, free-fluid, pore network and effective medium models. In addition, we propose a model based on the analogy with an RC filter circuit to interpret the results from pore pressure oscillation experiments.

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4.1 Permeability Estimation from Statistical Models

Past research on natural or artificial geo-materials (Scherer et al., 2007; Song et al., 2015) has shown that the order of magnitude of fluid permeability may be assessed with simple statistical models. This requires a number of assumptions, the first being that the fluid does not interact with the solids.

The pore network in a crystalline rock can be approximated by a 3D array of orthogonal flat cracks with constant length and aperture 2w (*w* is defined hereafter as the half aperture). The pore space in the Grimsel granodiorite is considered with such model, in which the aperture is replaced by the average crack width obtained from the BIB-SEM analysis. The simplest model derived from Poiseuille's law for flow into straight parallel cracks gives:

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 $k = \phi w^2 / 3 \tag{1}$

394 395

In real materials, the pores are non-circular, intersecting and tortuous, so that the equation 396 above is oversimplified (Scherer et al., 2007). The BIB-SEM results in Section 3 yielded an 397 average porosity ϕ =0.45% and average crack aperture 2w=283 nm or 1 µm. This provides a 398 permeability prediction of 3 10^{-17} m² if the main pore width is 283 nm, and 3.7 10^{-16} m² if the 399 main pore width is 1 µm. As shown earlier, the order of magnitude of measured permeability 400 for the Grimsel granodiorite is 10^{-18} m². This suggests that the main pore size for transport is 401 sub-micrometric, rather on the order of 283 nm, taking into account that the permeability 402 measurements were done at 5 MPa effective pressure whereas the porosity measurements 403 404 were done on unstressed rock.

Alternatively, permeability can be estimated by a fracture-based relationship for laminar flow
 (Zimmermann et al., 2005):

407

$$k = 2\lambda_L w^3/3 \tag{2}$$

where λ_L is the linear frequency of fractures or cracks. Taking $\lambda_L = 14749 \text{ m}^{-1}$ and again 2w=283 nm results in a predicted permeability of 2.8 10⁻¹⁷ m². However, considering that most of the cracks relevant for flow are associated with biotite, and assuming a biotite content of 40%, the predicted permeability based on crack density is 1.1 10⁻¹⁷ m² (i.e. 11 10⁻¹⁸ m²), in agreement with the prediction of the previous model. This value is also larger than the average measured permeability by one order of magnitude, but again corresponds to the unstressed rock.

415 Although this analysis is quite simplistic, it provides useful insights into the location of fluid 416 pathways and relates permeability measurement to microstructure quantification. Further analysis could be done by 3D pore quantification and modeling, as done by (Song et al.,
2016) for a tight sandstone (with crack-like pores). Such analyses allow us to assess the
contributions of different pore (crack) sizes to transport, material anisotropy, and the effect of
stress on permeability variations.

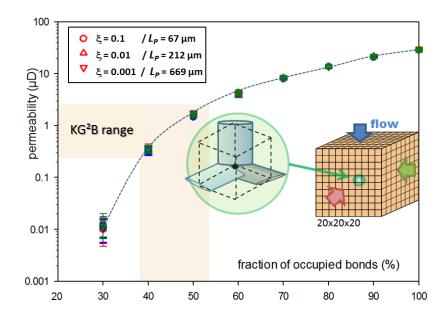
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422

4.2 Permeability Estimation from Pore Network Modeling

Permeability simulations were conducted using a 3D pore network model (PNM) 423 described in Casteleyn et al. (2011). The input data required for such modeling include (i) an 424 425 analytical description of the pore (or crack) size distribution, (ii) the average pore (or crack) shape, and (iii) the rock porosity. In the Grimsel granodiorite, fluid flows through a network 426 of cracks with low aspect ratio (Figure 5). In the PNM simulation, fluid flows through a 427 428 network of pipes with elliptical cross-section. For sake of simplicity all the pipes have the 429 same aspect ratio ξ and constant length L_P (David, 1993). MICP provides an estimate of the crack aperture distribution (equivalent to the throat size diameter in Figure 7) which 430 corresponds to the minor axis 2w of the elliptical pipes in the model; the semi-major axis R 431 given by $R=w/\xi$ in the model corresponds to the half-width of the cracks. The local 432 conductance of each bond is given by $\pi w^4/(4L_P\xi(1+\xi^2))$ (David, 1993). The experimental 433 crack aperture distribution (Figure 7) is modelled by a log-normal distribution in the range 434 (0.01µm, 30µm) with a peak centered at 0.5µm. The PNM is a cubic lattice with 20 nodes in 435 each direction (Figure 10); the pipes are located at the branches of the lattice. An algorithm 436 437 generates as many aperture values as pipes in the network (about 24000), following the lognormal distribution. These aperture values are randomly assigned to the pipes in the network. 438 The constant pipe length is derived from the "network porosity" which must match the rock 439 440 porosity. For sake of simplicity the network porosity was fixed at 1%, close to the average porosity value measured on plugs. Fluid flow is simulated by imposing a constant pressure 441 gradient across any pair of opposite faces of the network (David et al., 1990) and the 442 permeability is derived from the net flow rate at the outlet face using Darcy's law. The whole 443 process is repeated 10 times to obtain an average permeability and standard deviation. 444 Several simulations were conducted for three different values of the aspect ratio in the range 445 ξ =0.001, 0.01 and 0.1 (Figure 10). The simulations were done for different bond occupancy 446 ratios until permeability fell to zero (the percolation threshold); this can be achieved by 447 randomly removing pipes in the network until a selected value of bond occupancy is 448 achieved. 449

The results of PNM simulations show that (i) permeability decreases when the 450 fraction of pipes in the network decreases, with a sharp fall near the percolation threshold 451 (0.25 for a cubic lattice), (ii) permeability is the same in all three directions within numerical 452 errors, and (iii) permeability is not changed by the pipe aspect ratio. This last result shows 453 that permeability is essentially controlled by the crack aperture distribution which is the same 454 in all simulations. For 100% bond occupancy, the coordination number is equal to 6 and the 455 network permeability is 28 10^{-18} m². Such a high coordination number (and permeability) is 456 457 probably much too high for the Grimsel granodiorite.



458

Figure 10. Results of permeability simulation using a 3D pore network model on a 20x20x20 cubic lattice.
Cracks are represented by pipes with elliptical cross-sections with minor axes derived from MICP data, constant
aspect ratio and constant length. Three aspect ratios were considered: 0.1 (circles), 0.01 (upward triangles) and
0.001 (downward triangles). Colors red, blue and green define the directions in which permeability is calculated.
Error bars correspond to the standard deviation of permeability values for 10 network realizations with the same
statistical properties.

The experimental permeability range found in the benchmarking exercise is highlighted in 465 Figure 10. This range is consistent with a fraction of occupied bonds between 38% and 53%, 466 thus a mean coordination number probably lower than 3, a reasonable value for a hard rock in 467 which crack connectivity is expected to be low. The crack network in Figure 5 suggests an 468 average coordination number close to 3, although it is hard to imagine what the real 3D 469 coordination number is from 2D images. Given the crack lengths observed in Figure 5 (tens 470 of micrometers) and the PNM results (Figure 10), our simulations suggest that the crack 471 aspect ratio should range between 10^{-1} and 10^{-2} . As we tried to match the permeability 472 measured at 5 MPa effective pressure, the inferred microstructural properties (aspect ratio 473 and coordination number) correspond to that of the stressed rock. 474

475

476

4.3 Permeability Estimation from Effective Medium Modeling

Based on the microstructural data available, the Grimsel granodiorite is modeled as a 477 homogeneous and isotropic solid, an aggregate of randomly oriented and naturally fused 478 grains containing randomly oriented and spaced micro-cracks with finite diameter 2R and 479 aperture 2w. The number of micro-cracks per unit volume is N_V , and their aspect ratio is $\xi =$ 480 w/R. For sake of simplicity, the micro-cracks are modeled as oblate ellipsoids (thin cracks 481 with $\xi \ll 1$). They can overlap/intersect so as to allow hydraulic connectivity and fluid flow 482 through the rock at the macroscopic scale. The theoretical porosity of such a medium is given 483 484 by Garboczi et al. (1995):

487

489

$$\phi = 1 - e^{V_C N_V} \tag{3}$$

486 where V_C is the volume of a single ellipsoidal micro-crack,

$$V_C = \frac{4}{3}\pi\xi R^3 \tag{4}$$

488 In this context, the crack density $\rho_V = N_V R^3$ is (Sarout, 2012; Walsh, 1965)

$$\rho_V = -\frac{3}{4\pi\xi} \log(1-\phi) \tag{5}$$

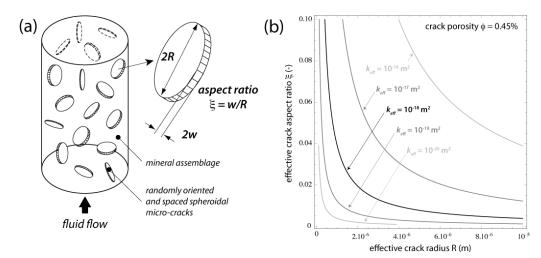
Let us assume that the network of micro-cracks in the Grimsel granodiorite is well above the hydraulic percolation threshold and that this network is the sole source of permeability (no background porosity). In this case, the permeability of the rock can be modeled using the concept of hydraulic radius (Gueguen & Dienes, 1989)

$$k \sim \alpha \phi m^2 \tag{6}$$

where $m = V_C / S_C$ is the hydraulic radius of ellipsoidal micro-cracks defined as their volumeto-surface ratio; and α is a dimensionless parameter derived from Poiseuille's law, related to the geometry of the hydraulically conducted network of micro-cracks, of the order of $\alpha \sim 1/3$ for a network of ellipsoidal micro-cracks (Sarout, 2012). The effective permeability of this cracked medium is explicitly related to its micro-structural parameters by (Sarout, 2012; Sarout et al., 2017)

501
$$k_{eff}(\phi,\xi,R) = \frac{16}{27} \frac{\phi R^2 \xi^2 (1-\xi^2)}{2\sqrt{1-\xi^2} + \xi^2 \log\left(\frac{2-\xi^2+2\sqrt{1-\xi^2}}{\xi^2}\right)}$$
(7)

This simple model explicitly relates the effective permeability of the micro-cracked rock to the crack porosity ϕ , the crack aspect ratio ξ and radius *R* or, equivalently, to the crack density ρ_V , ξ , and *R* (Figure 11). This is because ϕ and ρ are related through equation (5) once the geometry of the cracks is set in the micro-structural model (oblate ellipsoids with ξ <<1).



507

508 **Figure 11.** (a) Microstructural model of the Grimsel granodiorite. (b) Effective permeability predictions as a function of crack porosity, effective aspect ratio, and crack radius.

510 The experimental and microstructural parameters derived from measurements are the 511 following:

- Measured average permeability @ $P_{eff} = 5$ MPa: 0.6 10^{-18} m² (liquid) to 1.3 10^{-18} m² (gas)
- Permeability extrapolated to $P_{eff} = 0$ MPa (room conditions): $k_{exp} = 1$ to $5 \ 10^{-18} \ m^2$
- Measured porosity @ $P_{eff} = 0$ MPa: $\phi_{exp} = 0.8\%$ (cracks only)
- Porosity from microstructure @ $P_{eff} = 0$ MPa: $\phi_{micro} = 0.45\%$ (cracks only)
- Crack half-aperture @ $P_{eff} = 0$ MPa: $w_{micro} = 140$ nm
- Linear crack number density @ $P_{eff} = 0$ MPa: $\lambda_{L-micro} = 14749$ m⁻¹

519 One model parameter can be inferred from these data, the volumetric crack density ρ_V 520 defined in equation (5) which is related to the surface crack number density λ_A through the 521 average of their squared crack radius $\langle R^2 \rangle$ (Hadley, 1976)

522
$$\rho_V = \frac{3}{4\pi} \lambda_A \langle R^2 \rangle \tag{8}$$

523 The linear and surface crack number density are related through (*Zimmermann et al.*, 2005)

524
$$\lambda_A = \frac{\pi}{2} \frac{\lambda_L}{\langle R \rangle} \tag{9}$$

525 Combining equations (8), (9) and (5) yields

526
$$\rho_V = \frac{3}{8} R \lambda_L \text{ and } \phi(\lambda_L, \xi, R) = 1 - e^{-\frac{1}{2}\pi R \xi \lambda_L} = \phi(\lambda_L, w) = 1 - e^{-\frac{1}{2}\pi w \lambda_L}$$
 (10)

527 so that the permeability in equation (7) can be rewritten as

528
$$k_{eff}(\lambda_L,\xi,w) = \frac{16}{27} \frac{w^2(1-\xi^2)}{2\sqrt{1-\xi^2} + \xi^2 \log\left(\frac{2-\xi^2+2\sqrt{1-\xi^2}}{\xi^2}\right)} \left(1 - e^{-\frac{1}{2}\pi w\lambda_L}\right)$$
(11)

529 The data inversion strategy consists of the following steps:

530 1. Using equation (7), and the measured porosity ϕ_{exp} and permeability k_{exp} , we first 531 define the effective crack radius function $R_{sol}(\xi)$ satisfying $k_{eff}(\phi_{exp},\xi,R_{sol}(\xi)) = k_{exp}$,

532
$$R_{sol}(\xi) = \frac{3}{4} \left[\frac{3k_{exp}}{\phi_{exp}\xi^2(1-\xi^2)} \left[2\sqrt{1-\xi^2} + \xi^2 \log\left(\frac{2-\xi^2+2\sqrt{1-\xi^2}}{\xi^2}\right) \right]^2 \right]^{1/2}$$
(12)

533 2. Noting that by definition $R_{def}(\xi, w) = w / \xi$, we equate $R_{sol}(\xi) = R_{def}(\xi, w_{sol})$ and 534 determine the effective crack half-aperture w_{sol} so that this equality is satisfied for all aspect 535 ratios ξ .

536 3. Using equation (10), and noting that $\phi(\lambda_{L-\text{sol}}, w_{\text{sol}}) = \phi_{\text{exp}}$, we determine the linear 537 crack number density $\lambda_{L-\text{sol}}$ satisfying this equality. 538 4. Finally, using equation (11), and setting $k_{eff}(\lambda_{L-sol}, w_{sol}/R_{sol}, w_{sol}) = k_{exp}$, we determine 539 the effective crack radius R_{sol} satisfying this equality.

540 5. Knowing w_{sol} and R_{sol} , we compute the effective aspect ratio of the cracks $\xi_{sol} = w_{sol}$ 541 / R_{sol} .

542 This strategy is implemented considering the permeability values estimated at room 543 conditions in the range 1 to 5 μ D and a porosity of either $\phi = 0.8\%$ (experimentally 544 measured) or $\phi = 0.45\%$ (determined from 2D microstructure). Table 1 summarizes the 545 results of the data inversion using these input parameters.

546

run #	$\phi(\%)$	$k (10^{-18} \text{ m}^2)$	w (nm)	$\lambda_L (\mathrm{m}^{-1})$	<i>R</i> (µm)	ξ
1	0.8	1	29	99009	none	none
2	0.8	5	65	44173	none	none
3	0.45	1	39	73622 (ρ _V ~ 0.032)	0.92	4.2 x 10 ⁻²
4	0.45	5	87	33003 (ρ _V ~ 0.025)	2.6	3.3 x 10 ⁻²

547

 Table 1. Results of the data inversion from effective medium modeling.

548

For the first two scenarios (run #1 and #2) in Table 1 we observe that no value of the effective crack radius *R* can satisfy $\phi = 0.8\%$ and $k = 1 \ \mu\text{D}$, or $\phi = 0.8\%$ and $k = 5 \ \mu\text{D}$. The derived aperture *w* and linear crack number density $\lambda_{\rm L}$ do not match the corresponding parameters estimated from 2D microstructural analysis ($w_{\rm micro} \sim 140 \ \text{nm}$ and $\lambda_{\rm L-micro} \sim 14724 \ \text{m}^{-1}$).

554 The two other scenarios (run #3 and #4) yield reasonable results, that is,

555 - An effective crack radius exists (R = 0.92 to 2.6 µm) that honors the measured 556 permeability ($k = k_{exp} = 1$ to 5 µD) and porosity ($\phi = \phi_{micro} = 0.45\%$)

557 - The inverted apertures ($w_{sol} = 39$ to 87 nm) do not match the corresponding parameter 558 estimated from 2D microstructural analysis ($w_{micro} \sim 140$ nm and $\lambda_{L-micro} \sim 14724$ m⁻¹). 559 However, out of all scenarios, #4 ($\phi = 0.45\%$ and $k = 5 \mu$ D) offers the value of half-560 aperture (w = 87 nm) closest to that determined from 2D microstructural analysis (w =561 140 nm).

562 - The inverted crack number densities (λ_L =33003 to 73622 m⁻¹) do not match the 563 corresponding parameter estimated from 2D microstructural analysis ($\lambda_L \sim 14724 \text{ m}^{-1}$). 564 However, out of all scenarios, #4 ($\phi = 0.45\%$ and $k = 5 \mu$ D) offers the value of crack 565 number density ($\lambda_L = 33003 \text{ m}^{-1}$) closest to the value determined from 2D 566 microstructural analysis ($\lambda_L = 14724 \text{ m}^{-1}$).

567 - The inverted crack aspect ratio ($\xi = 3.3$ to 4.2 10⁻²) reflects a realistic crack geometry 568 ($\xi \ll 1$).

In conclusion, scenario #4 is the most realistic in view of the available experimental and microstructural data. To generate this scenario, we have used as an input $k = k_{exp} = 5$ mD and 571 $\phi = \phi_{\text{micro}} = 0.45\%$. The model and data inversion strategy outputs are: an effective half-572 aperture $w \sim 90$ nm, an effective crack radius $R \sim 2.6 \,\mu\text{m}$, an effective aspect ratio $\xi \sim 3 \times 10^{-2}$ 573 and a crack number density $\lambda_{\text{L}} \sim 33003 \,\text{m}^{-1}$ (or crack density $\rho_{\text{V}} \sim 0.025$).

574 Although the inverted w, R, ξ , and λ_L are not exactly those determined from the 575 microstructural analysis, they are reasonably close, and most importantly, they yield the 576 expected porosity and permeability. The discrepancies can be explained as follows:

- The difference in crack aperture (90 nm versus 140 nm) could be due to (i) the resolution limits of the 2D image; (ii) an undesired inflation of the cracks after Wood's metal injection; and/or (iii) the use of 2D images to determine a 3D parameter.
- The difference in crack number density (33003 m⁻¹ versus 14724 m⁻¹) could be due to the heterogeneity of the rock and the fact that the images probe only a sub-volume (in fact a 2D surface) of the whole sample on which the porosity/permeability are measured.
- The difference between the measured porosity (0.8%), and the porosity determined from 2D microstructures (0.45%) could be due to: (i) the heterogeneity of the rock and the fact that the images probe only a sub-volume (in fact a 2D surface) of the whole sample on which the porosity/permeability are measured, and/or (ii) a resolution limit of the porosity measurement as this type of crack porosity is inherently very small.
- 591 _ The inverted crack radius $R \sim 2.6 \,\mu\text{m}$ does not seem to qualitatively reflect the scale of the cracks highlighted by Wood's metal injection in Figure 5; in the figure, the 592 cracks appear longer than 2.6 µm. However, the effective crack radius is determined 593 from the effective hydraulic permeability of the rock which hosts natural and jagged 594 cracks, perhaps with multiple contact points between asperities (see Sarout et al. 595 (2017)), so that the effective hydraulic radius is smaller than the cracks length 596 visualized in the 2D thin section. Other possible causes of discrepancy listed above 597 could also contribute to the discrepancy in the inverted crack radius. For instance, the 598 injection of Wood's metal could have inflated the crack network so that the cracks 599 appear thicker (larger aperture), and longer (less contacts at asperities). 600
- 601

602 *4.4 Permeability Estimation from Percolation Model (MICP)*

MICP results from the Grimsel granodiorite can be used with the Katz and Thompson equation (Katz & Thompson, 1986) as outlined in (Hu et al., 2015). This model is based on percolation theory and states that a critical pore (or crack) size controls permeability. The critical pore size can be determined from the inflection point of the MICP cumulative intrusion curve when mercury starts to percolate into the pore space. According to this model, the permeability *k* is given by:

$$k = \frac{1}{89} (d_{max})^2 \left(\frac{d_{max}}{d_C}\right) \phi S(d_{max})$$
(13)

610 where d_{max} is the pore throat diameter at which conductance is maximum, d_C is the critical

611 pore throat diameter at percolation threshold and $S(d_{max})$ is the mercury saturation at a 612 pressure corresponding to d_{max} (Hu et al., 2015). Using the throat size distribution given in 613 Figure 7 and the porosity derived from MICP on the same plug, a predicted permeability 614 value of 1.05 10⁻¹⁸ m² for the unstressed rock is obtained, suggesting that MICP captures the 615 correct characteristics of the fluid flow pathways at the sample scale.

616

617 *4.5 Permeability Estimation from Free-Fluid Model (NMR)*

NMR analysis is also able to predict the permeability from the T₂ relaxation time 618 distribution (Josh et al., 2012) shown in Figure 8. In this analysis permeability prediction is 619 based on the free fluid model by Coates et al. (1991). As the five samples were first measured 620 saturated then desaturated after centrifuging, one can estimate the Free Fluid Index FFI 621 (corresponding to the water removed at 6.9 bars equivalent capillary pressure) and the Bound 622 Volume Index BVI (corresponding to irreducible water). Saturated and desaturated samples 623 help to define the T₂ cutoff that separate FFI from BVI as shown in the example on sample 624 X1 (Figure 12a). The five samples record a T₂ cutoff around 30 ± 10 ms, very close to values 625 found in the literature for quartz rich rocks (around 33 ms). 626

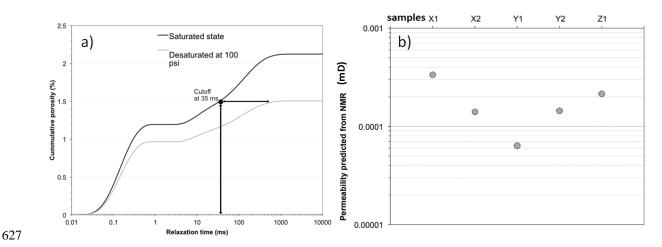


Figure 12. a) Example of NMR cumulative porosity of sample X1 under saturated and desaturated conditions to measure the T_2 cutoff that separates mobile and irreducible water. b) Predicted permeability from NMR in five small core plugs of Grimsel granodiorite using classical parameters from Coates.

As formulated in the Coates model (Coates et al., 1991), the NMR predicted permeability is given by:

633
$$k = \left(\frac{\phi}{\Gamma}\right)^4 \left(\frac{FFI}{BVI}\right)^2 \tag{14}$$

where Γ is a constant related to pore geometry. Using a standard value for Γ according to the 634 Coates model (Γ =10 when the permeability unit is mD (10⁻¹⁵ m²) and porosity is in %, the 635 five tested plugs have a predicted permeability ranging from 0.14 to 0.35 10^{-18} m² (average 636 $0.20 \ 10^{-18} \ m^2$) except for sample Y1, which has a lower permeability (0.063 $10^{-18} \ m^2$)(Figure 637 12b). These values are lower than the average permeability found in the benchmark (see 638 companion paper). However they were obtained at 13 MPa confining pressure whereas the 639 KG²B effective pressure target was 5 MPa. Taking into account the pressure dependence of 640 permeability shown in the companion paper, the NMR predicted permeability values are in 641

- 642 good agreement with the measured permeability range.
- 643
- 644

4.6 Permeability Estimation from RC Filter Analog

Here we report a new way to analyze the data generated by pore fluid pressure 645 oscillation experiments (see companion paper) based on modeling the rock as a RC filter. The 646 approach has been used by Mckernan et al. (2017) and Rutter and Mecklenburgh (2018). In 647 contrast to the four previous models, this model is based on a physical analog rather than 648 microstructural data. Oscillatory flow of fluid through the pores of a rock is analogous to the 649 flow of electricity through a resistor-capacitor network. A first order resistance-capacitance 650 (RC) filter is shown in Figure 13a. This corresponds to a rock sample (the resistive element) 651 of zero storativity (zero porosity), and the downstream reservoir corresponds to the capacitive 652 653 element. The transfer function or gain $G=V_{out}/V_{in}$ depends on the frequency f because of the 654 time required to charge the capacitor through the resistor. At low frequencies the capacitor is infinitely resistant so a waveform applied as V_{in} passes unimpeded (provided the output does 655 not draw current). Beyond the break frequency $f_{\rm B}$ the capacitor can conduct so the R and C 656 elements form the arms of a potential divider and the output is progressively attenuated as 657 frequency is increased. This is a low pass filter, because the unattenuated frequencies are low 658 frequencies. The high frequency waveform amplitude attenuation rate (gain) is always 20 dB 659 per decade; it has a slope of -1 on a plot of $\log G$ vs $\log f$. The linear prolongation of the high 660 frequency slope intersects the gain = 1 abscissa at a characteristic break frequency (or corner 661 frequency) $f_{\rm B} = 1/(2\pi R C)$. The output (across the capacitor) of an RC filter also has a 662 particular response to a step change in input voltage, with v_{out} decaying exponentially with 663 time. This was the basis of the widely-used pulse transient decay method proposed by Brace 664 et al. (1968) for the measurement of permeability of tight rocks. 665

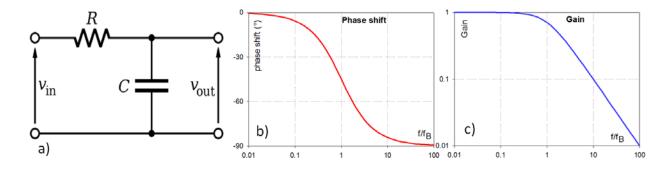




Figure 13. a) A first order electrical low pass filter analogous to fluid flow through a resistant rock R of zero
 storage capacity, with a capacitor C analogous to the downstream storage reservoir. Variation of b) phase shift
 and c) gain A with applied waveform frequency for a low pass electrical filter.

670

- In addition to progressively attenuating the output waveform, the filter progressively shifts its phase over the frequency range between the two linear segments, from 0° to 90° (Figure 13b). The gain *G* and phase shift θ can be expressed respectively as:
- 674

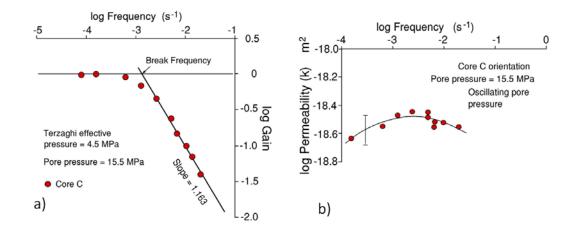
675

$$G = \frac{V_{out}}{V_{in}} = \frac{1}{\sqrt{1 + (f/f_B)^2}}$$
(15)

676
$$\boldsymbol{\theta} = -\tan^{-1}\left(\frac{f}{f_B}\right) \tag{16}$$

Higher-order low-pass filters can be formed by cascading first order filters to simulate the 677 behavior of more porous rocks (higher storativity). A rock might be imagined as a series of 678 such filters, with capacitive components corresponding to pore spaces connected by resistors 679 that combine to form the total resistance to flow. Each RC element in series can apply an 680 additional phase shift, but many such phase shifts will result in severe attenuation. Many 681 possible topologies of R and C combinations can be imagined, with the final capacitor 682 corresponding to the downstream volume of the permeameter. Analysis of such combinations 683 is beyond the scope of the present paper. Smaller ratios of rock storativity to downstream 684 storage translate to smaller phase shifts for a given gain, so that the behavior more closely 685 resembles that of a first-order filter. 686

This approach was evaluated on a Grimsel granodiorite sample cut at a high angle to the 687 foliation (called hereafter core C), to investigate how similar its behavior is to that of an RC 688 filter. Pore fluid pressure oscillation tests were conducted with a pressure cycling period 689 ranging from 50 to 12800 seconds (i.e. 7.8 10^{-5} s⁻¹ < $f < 2 \ 10^{-2}$ s⁻¹). Figure 14a shows a plot of 690 $\log G$ vs $\log f$ for the driving waveform when total confining pressure is 20.0 MPa and pore 691 pressure is 15.5 MPa. As expected the behavior is similar to that of an RC filter with $\log f_{\rm B}$ = 692 -2.869 (i.e. $f_{\rm B}=1.35 \ 10^{-3} \ {\rm s}^{-1}$). The slope in the frequency-dependent region is -1.16, slightly 693 greater than unity, as might be expected for the small degree of storativity (non-zero porosity) 694 within the rock specimen. 695





703

697Figure 14. a) Plot of log G versus log f for core C at 4.5 MPa effective pressure and 15.5 MPa pore pressure of
argon gas. This is typical of rock behavior as a first order filter with very small storativity in the rock sample
(slope of -1.16 close to unity). b) Frequency dependence of permeability calculated for the individual data. The
peak in the convex upward curve corresponds to the break frequency. The average of the log k data lying above
the break frequency is -18.52.

The fluid flow analogs of resistance R and capacitance C are:

$$\mathbf{R} = \frac{L\mu}{Ak} \text{ and } \mathbf{C} = \boldsymbol{\beta}_{\boldsymbol{D}}$$
(17)

where *L* and *A* are the length and cross-sectional area of the sample respectively, and β_D is the storage of the downstream reservoir (m³/Pa). Permeability can therefore be calculated from the break frequency provided that the frequency-dependence of gain is measured at constant confining pressure and pore pressure conditions:

$$k = 2\pi\mu(L/A)\beta_D f_B \tag{17}$$

This yields log k = -18.33 (i.e. $k=0.47 \ 10^{-18} \ m^2$) for the tested sample. Leaving aside the most 709 extreme values of very small or very large gain, the average of all the individual permeability 710 measurements is $\log k = -18.52 \pm 0.06$ (k=0.30 10⁻¹⁸ m²). The plot of $\log k$ vs $\log f$ (Figure 711 14b) shows slight upward convexity, similar to what was found for a sandstone by Song and 712 Renner, (2007). One of the KG²B labs (Lab#18, see companion paper) measured a 713 permeability of 0.501 10⁻¹⁸ m² on this sample with the standard approach for analyzing pore 714 pressure oscillation tests (Bernabé et al., 2006), and 0.582 10⁻¹⁸ m² using a transient pulse 715 716 test.

717

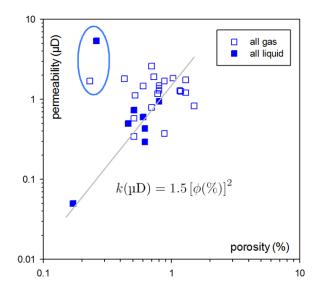
718 **5. Complementary Outcome of the Benchmarking Exercise**

719 In this section we present additional data produced by the KG²B team in their study of 720 the Grimsel granodiorite core samples. This data set is not as exhaustive as the permeability 721 data set because it was done on a voluntary basis with no specific instructions.

722 5.1. Permeability – Porosity Relationship

A log-log plot of permeability vs. porosity (Figure 15) shows a general trend with two 723 outliers and one isolated point aligned with the general cloud consistent with the expected 724 725 trend of permeability decrease with decreasing porosity. The correlation is not very strong, which is not really surprising as permeability is controlled by the geometrical properties (pore 726 size and shape, topology and connectivity) of the 3D pore or crack network and not simply by 727 728 the bulk porosity. Nevertheless, a simple power-law can be fitted to the data set (minus two outliers) with an exponent equal to 2 (Figure 15). The sample in the lower left corner was 729 730 considered as an outlier in the statistical analysis presented in the companion paper: its low 731 permeability can be explained by its porosity being much lower than all the others. A powerlaw relationship between permeability and porosity has often been invoked (e.g. David et al., 732 1994 and references therein). Wang et al. (2016) found an exponent between 4 and 5 in their 733 734 permeability-porosity correlation for two granite gneiss samples. In our KG²B experiments, porosity was measured at room conditions whereas permeability was measured at 5 MPa 735 effective pressure. If both properties were measured under the same pressure conditions, the 736 737 correlation would probably have been better.

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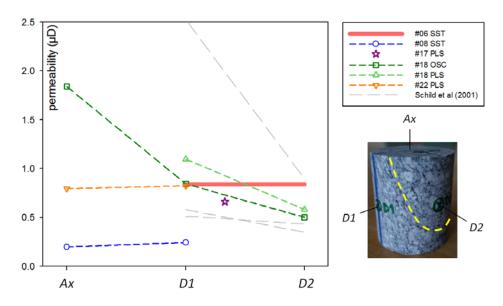


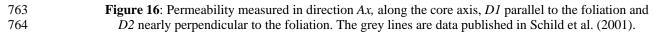
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5.2. Permeability Anisotropy

Three participants (Lab#8, Lab#18 and Lab#22) reported permeability measurements 742 in more than one direction in order to test for permeability anisotropy. These data sets include 743 the one presented in section 4.6. which was obtained by Lab#18 using the oscillating pore 744 pressure method and supplemented with measurements made in two other directions using 745 the transient pulse method. The two other labs used the steady state and the transient pulse 746 methods. All the anisotropy data was obtained using gas as the flowing fluid. The results are 747 748 compiled in Figure 16, together with those from (Schild et al., 2001). For the KG²B core, an orientation nomenclature was adopted whereby Ax represents the direction parallel to the core 749 axis, D1 is the direction perpendicular to Ax and parallel to the foliation; and D2 is 750 perpendicular to both Ax and D1. Note that D2 is nearly perpendicular to the foliation ($\sim 70^{\circ}$). 751 Schild et al., (2001) investigated permeability parallel and perpendicular to the foliation and 752 for the sake of comparison those directions were associated with directions D1 and D2, 753 respectively. Overall, anisotropy emerges from the comparison between directions D1 and 754 D2, where the permeability along the foliation consistently measures higher than the one 755 (quasi-) perpendicular to it (respective anisotropy coefficients of ~50% and ~60% for the 756 OSC and PLS measurements of Lab #18). This result is qualitatively consistent with the data 757 of Schild et al., (2001), albeit over a greater range of anisotropy coefficients. It also compares 758 759 well with the velocity data of Schild et al., (2001) and the velocity measurements made during our screening of the KG²B plugs (~30% P-wave anisotropy reported in (David et al., 760 761 2017)).





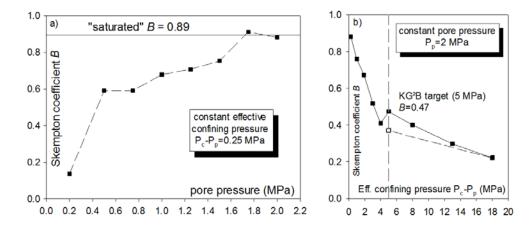
When both D1 and D2 plugs were tested, the permeability nearly perpendicular to foliation 765 (along D2) was systematically smaller that in the direction nearly parallel to foliation (along 766 D1 or Ax). Little permeability anisotropy was expected to arise from the measurements made 767 768 along Ax and D1, as Ax is relatively close to the foliation. This is confirmed by the results of Lab#08 and Lab#22. The value obtained along Ax by Lab#18 is not consistent with that 769 picture; it might be attributable to heterogeneity from sample to sample. More specifically, 770 771 since permeability is considered as being largely controlled by micro-cracking in the biotite grain fraction, slight changes in biotite content and grain size from sample to sample could 772 result in large baseline contrasts. A better assessment could be obtained if the same 773 774 representative elementary volume was measured along several directions as opposed to distinct plugs of various dimensions. Two additional participants (Lab#6, Lab#17) measured 775 the permeability only in radial directions: both values fall in the range of radial permeability 776 found by the others. The sample tested by Lab#17 was oriented at about 30° from the 777 foliation, and the radial permeability measured by Lab#06 is an average one (plotted with an 778 horizontal line) derived from a radial water flow experiment on a hollow cylinder parallel to 779 780 the core axis (Monfared et al., 2011) at 1.75 MPa effective confining pressure, lower than the KG2B target. Due to the limited number of samples, the anisotropy analysis is far from being 781 as convincing as the general KG²B data set. 782

783 5.3. Poroelastic Parameters

In situ rock masses include pore, crack, fracture networks which are usually saturated 784 or partially saturated with fluids, often with 2 or more fluid phases such as gas, water, or oil. 785 The degree of saturation, ranging between 0 and 1, is the ratio of volume of pore fluid in the 786 787 pore space to the pore volume. Controlling saturation during laboratory tests is important for two main reasons: i) to reproduce field conditions; ii) for intrinsic permeability estimates. For 788 the latter purpose, full saturation of specimens with fluid used for measurement is essential 789 (Zinszner & Pellerin, 2007). For measurements using gas, particular attention has to be paid 790 791 to sample preparation and drying. For permeability measurement using liquids, key issues include expelling trapped gas and checking for full saturation with liquid phase. For 792 measurement with water, flushing of de-aired water into the specimen, followed by a step by 793 step back pressure increase, have been recommended in order to avoid additional gas entry 794

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and to force trapped gas into solution (Black and Lee, 1973). This method was successfully 795 applied to a tight porous rock, the Opalinus clay (Wild et al., 2015) and also in the present 796 study by Lab#04 on a Grimsel granodiorite sample. Between each back pressure step, the 797 poroelastic response to hydrostatic confinement was checked in order to assess the degree of 798 saturation. The isotropic Skempton's coefficient measurements, defined as $B = \Delta P_p / \Delta P_c$, the 799 ratio of pore pressure change to confinement pressure change, should reach a plateau when 800 all trapped gas bubbles are dissolved in the solution. For the Grimsel granodiorite initially 801 filled with water under vacuum, a plateau is reached when the pore pressure exceeds about 802 1.8 MPa (Figure 17a), and the "saturated" Skempton's coefficient is B=0.89 at 0.25 MPa 803 effective confining pressure. 804





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Figure 17. a) Evolution of the Skempton's coefficient *B* vs. pore pressure. b) Evolution of the Skempton's coefficient *B* vs. effective confining pressure at constant pore pressure (2 MPa).

Once the "saturation" pore pressure was reached, the confining pressure was increased at constant pore pressure ($P_p=2$ MPa) to the KG²B pressure target ($P_{eff} = 5$ MPa) and beyond. The increase of effective confining pressure resulted in a sharp drop of the Skempton's coefficient (Figure 17b) to 0.47 at the target pressure, and even lower at higher pressures. This behavior is probably linked to the progressive closure of cracks in the rock sample.

Another poroelastic parameter was determined by one of the participants (Lab#19), the Biot-Gassmann effective pressure coefficient α . This coefficient was obtained from several permeability measurements under different pressure conditions (both P_p and P_c). The effective pressure law $P_{eff} = P_c - \alpha P_p$ was established for permeability, and it was shown that the effective pressure coefficient α was equal to 1 (see companion paper).

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819 6. Discussion

To complement the experimental permeability data set presented in the companion 820 paper, we present additional data from microstructural analyses using BIB-SEM and micro-821 822 CT, as well as permeability predictions from various models. High quality imaging with BIB-SEM technology allowed us to identify pores and cracks at the micrometer scale and their 823 relation with the rock mineralogy. Most of the cracks are located within biotite or at grain 824 boundaries. The Wood's metal injection technique, combined with SEM, provided detailed 825 and realistic images of the actual crack network, its connectivity (from which an average 826 coordination number can be estimated) and tortuosity. A statistical analysis provided relevant 827

data on pore size, crack length and aperture, and porosity obtained from more than 60,000 828 elements; the amount and quality of these data provided valuable information for 829 permeability modeling. Micro-CT imaging provided 3D volume rendering of the matrix 830 density as well as a 3D map of pore locations. Two important conclusions could be drawn: (i) 831 at the sample scale, the material appears to be very heterogeneous, with the size of 832 heterogeneities exceeding the sample size and (ii) the pores resolved by micro-CT are 833 isolated, confirming that fluid flow is controlled by a network of submicron cracks. It seems 834 clear that the size of the samples studied (core sample with 4 mm diameter and 10 mm 835 length) is well below the REV, which may explain the larger scatter in measured permeability 836 values for small samples shown in the companion paper. 837

Table 2 summarizes the results of permeability modeling using microstructural data as input parameters. For each model, a short description is provided, and the input parameters are given.

MODEL	DESCRIPTION	INPUT PARAMETERS	PERMEABILITY PREDICTION	
Statistical model	3D array of orthogonal intersecting cracks with same length and aperture	porosity, mean crack aperture	$k_{pred} = 30 \ 10^{-18} \ \mathrm{m^2}$	
Statistical model (Zimmermann et al., 2005)	array of parallel cracks with the same aperture	linear density of cracks, mean crack aperture	$k_{pred} = 28 \ 10^{-18} \ \mathrm{m^2}$	
Percolation model (Katz & Thompson, 1986)	based on the estimation of the critical crack aperture at percolation threshold	MICP intrusion volume vs. pressure graph, pore throat and saturation at threshold pressure, porosity	$k_{pred} = 1.1 \ 10^{-18} \ \mathrm{m^2}$	
Free-fluid model (Coates et al., 1991)	based on NMR relaxation time distribution	NMR T ₂ spectrum, porosity, free and bound water fractions	$\begin{array}{c} 0.13 \ 10^{-18} \ \mathrm{m}^2 < k_{pred} < 0.33 \\ 10^{-18} \ \mathrm{m}^2 \qquad (@13 \ \mathrm{MPa}) \end{array}$	
Pore network model (David, 1993 Casteleyn et al., 2011)	3D cubic network of pipes with elliptical cross-section and constant length	crack aperture distribution from MICP, crack aspect ratio, porosity, fraction of occupied bonds χ	$\begin{array}{ccc} (\chi = 100\%) & k_{pred} = 28 \ 10^{-18} \\ \mathrm{m}^2 & (\chi = 53\%) & k_{pred} = \\ & 2.5 \ 10^{-18} \ \mathrm{m}^2 \\ (\chi = 38\%) & k_{pred} = \ 0.25 \ 10^{-18} \\ \mathrm{m}^2 \end{array}$	
Effective medium model (Sarout et al., 2017)3D random distribution of penny-shaped cracks		crack density, porosity, crack aperture, crack aspect ratio	$k_{pred} = 5 \ 10^{-18} \ \mathrm{m^2}$	

Table 2. Summary of permeability predictions obtained with 6 different models using input parameters based on
 microstructural data.

Both statistical models yield a permeability value of about 30 10⁻¹⁸ m², significantly larger than the mean outcome for the measured permeability data set (~1 10⁻¹⁸ m²). These models are based on an oversimplified representation of the pore space where heterogeneity is absent, so that analytical solutions for permeability can be calculated. The predicted values are likely higher because the microstructural data were obtained on stress-free samples, whereas permeability measurements were obtained at 5 MPa effective confining pressure. However, if one takes into account the pressure

dependence of permeability shown in the companion paper, extrapolated permeability at zero effective pressure would give a value in the range 2-10 10^{-18} m², still lower than the permeability predicted by both statistical models. This discrepancy suggests that the heterogeneous nature of the rock pore space is poorly accounted for in statistical models.

- In contrast, the percolation model proposed by Katz & Thompson (1986) takes 855 • advantage of the full mercury volume vs. pressure curve obtained in MICP 856 experiments. This model postulates the existence of a subnetwork spanning the rock 857 sample, consisting of highly conducting cracks with conductance larger than a critical 858 value. Heterogeneity is taken into account, the model prediction of the model (~1 10^{-1} 859 18 m²) is in good agreement with the measured permeability despite the fact that the 860 effective pressures do not match. MICP coupled with percolation modeling provides 861 the correct length scale for permeability estimation. 862
- Generally consistent results were obtained for NMR-predicted permeability using the free fluid model. This model is based on NMR detection of the fraction of bound water in the pore space. Although little information is captured regarding pore space geometry, the model was successful in predicting the correct range of permeability at the NMR operating confining pressure (13 MPa), based on the pressure dependence of permeability presented in the companion paper.
- The three-dimensional nature of fluid flow in porous rocks is accounted for in both • 869 the pore network model and the effective medium model. The difference between 870 these models is the topology of the crack network: cracks are located at bonds in a 871 cubic lattice for the former, and randomly distributed for the latter. For both models, 872 the number of input parameters is larger: porosity, crack aperture and aspect ratio, 873 length of pipes in the PNM and crack density in the effective medium model. Not all 874 of these parameters are well constrained either by MICP or microstructural data. 875 Therefore reasonable assumptions were made to find the best set of parameters to 876 match permeability measurements at 5 MPa effective pressure. Interestingly, both 877 models converge to a similar value of crack aspect ratio ($\sim 10^{-2}$), an apparently 878 reasonable value based on the micrographs in Figures 3 and 5. Another outcome of 879 the pore network model is that a permeability prediction consistent with the measured 880 value requires a coordination number close to three, again in agreement with the 881 micrographs in Figures 3 and 5. However, the models disagree regarding crack length, 882 of order 100 µm for the PNM, and 1 µm for the effective medium model. Whereas the 883 former value corresponds more or less to the actual crack length imaged in Figure 5, 884 the latter does not, and one may conclude that the effective medium model is unable 885 to match all our observations. Nevertheless, as discussed in section 4.3, the 886 discrepancy may be explained by the presence of asperities and the jagged nature of 887 observed cracks; in the effective medium model, an actual crack might be viewed as a 888 combination of smaller cracks relevant for fluid flow. 889
- 890

891 **7. Conclusion**

In the companion paper the complete data set of low-permeability measurements from a benchmarking exercise involving 24 laboratories was analyzed; here we present complementary results focusing on (i) quantitative analysis of microstructures and pore size

distributions, (ii) permeability modeling and (iii) measurements of permeability anisotropy and poroelastic parameters. BIB-SEM, micro-CT, MICP and NMR methods were used to characterize microstructures (both in 2D and 3D) and quantify pore size distribution. Wood's metal injection was used to image the crack networks on 2D images. All of these studies provided input parameters for permeability modeling using (i) basic statistical models, (ii) 3D pore network and effective medium models, (iii) a percolation model using MICP data and (iv) a free-fluid model using NMR data. A new method for simpler analysis of pore pressure oscillation tests, modeling the rock as an RC electrical circuit, was also described for the case of small sample storativity. The models were generally successful in predicting the observed range of measured permeability using microstructural, MICP and/or NMR data. Whereas statistical models overestimate the permeability due to lack of information on heterogeneity, percolation, pore network and effective medium models are more relevant and provide additional constraints on crack parameters such as aspect ratio, aperture, density and connectivity. This confirms that MICP and advanced microscopy techniques are potentially able to provide useful input data for permeability estimation. Additional results to complement the measured permeability data set show that (i) the average porosity measured on plugs is 0.77% + -0.36%, (ii) a weak power-law with exponent 2 relates permeability to porosity, (iii) permeability measured ~orthogonal to foliation is lower that ~parallel to foliation, and (iv) the Skempton's coefficient at 5 MPa effective pressure is about 0.5. A second round of benchmarking is currently under way, with another tight material, the Cobourg Limestone. Additional challenges are expected in this benchmark, project called KCL as the permeability is in the nano-Darcy range (10^{-21} m^2) .

918 List of symbols

- P_c , confining pressure (Pa)
- P_p , pore pressure (Pa)
- $P_{eff} = P_c P_p$, effective pressure (Pa)
- α , effective pressure coefficient
- *B*, Skempton's coefficient
- k, permeability (m²)
- k_{exp} , experimental value of permeability (m²)
- k_{pred} , predicted value of permeability (m²)
- k_{gas} , permeability measured with gas (m²)
- k_{liquid} , permeability measured with gas (m²)
- k_{eff} , predicted permeability given by the effective medium model (m²)
- T_2 , NMR transverse relaxation time (s)
- *φ*, porosity
- ϕ_{micro} , porosity obtained from BIB-SEM analysis
- *w*, average crack half aperture (m)
- w_{sol} , effective crack half-aperture, solution of the effective medium model (m)
- $\lambda_{\rm L}$, linear crack density (m⁻¹)
- λ_A , surface crack density (m⁻²)
- L_P , length of the pipes in the pore network model (m)
- χ , fraction of occupied bonds in the pore network model
- *R*, average crack radius (m)
- R_{sol} , effective crack radius, solution of the effective medium model (m)
- ξ , crack aspect ratio (=*w*/*R*)

- 942 ξ_{sol} , effective crack aspect ratio, solution of the effective medium model
- 943 N_V , number of cracks per unit volume (m⁻³)
- 944 V_C , volume of a single spheroidal crack (m³)
- 945 $\rho_V = N_V \cdot R^3$, crack density for effective medium model
- 946 *m*, hydraulic radius (m)
- 947 α , dimensionless parameter for Poiseuille's law (=1/3 for cracks)
- 948 *FFI*, free fluid index
- 949 *BVI*, bound volume index
- 950 Γ , constant linked to pore geometry in the free-fluid model
- 951 R, equivalent resistance in RC circuit
- 952 C, equivalent capacitor in RC circuit
- 953 $G=V_{out}/V_{in}$, gain
- 954 θ , phase shift (rad)
- 955 f, frequency (s⁻¹)
- 956 f_B , break frequency (s⁻¹)
- 957 *L*, sample length (m)
- 958 *A*, sample cross-sectional area (m²)
- 959 μ , dynamic viscosity (Pa.s)
- 960 β_D , downstream reservoir storage (m³Pa⁻¹)
- 961 KG²B, K for Grimsel granodiorite benchmark
- 962 BIB-SEM, broad ion beam scanning electron microscopy
- 963 ViP, Virtual Petroscan
- 964 PPL/XPL, in-plane / crossed polarized light
- 965 EDS, energy dispersive spectroscopy
- 966 PSD, pore size distribution
- 967 NMR, nuclear magnetic resonance
- 968 PNM, pore network modeling
- 969 WM, Wood's metal
- 970 MICP, mercury injection capillary pressure
- 971 REV, representative elementary volume
- 972 CT, computerized tomography
- 973

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APPENDIX A

⁽³⁾The KG²B Team: the benchmark involved 24 rock physics laboratories around the
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 in Table A1.

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