PHASE 2 INITIAL BOREHOLE DRILLING AND TESTING, IGNACE AREA

WP04F Data Report: Measurement of Surface Area and Cation Exchange Capacity of Core Samples for IG_BH06

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REPORT

PHASE 2 INITIAL BOREHOLE DRILLING AND TESTING AT IG_BH04/05/06

WP04F Data Report - Measurement of Surface Area and Cation Exchange Capacity of Core Samples for IG_BH06

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WP4F DATA REPORT – MEASUREMENT OF SURFACE AREA AND CATION EXCHANGE CAPACITY OF CORE SAMPLES FOR IG_BH06

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1.0 INTRODUCTION

The Initial Borehole Drilling and Testing project in the Wabigoon Lake Ojibway Nation (WLON) – Ignace Area, Ontario is part of Phase 2 Geoscientific Preliminary Field Investigations of the Nuclear Waste Management Organization's (NWMO) Adaptive Phased Management Site Selection Phase. This project includes the drilling and testing of six deep boreholes at the Revell site, as well as additional on-going studies, located within the northern portion of the Revell batholith.

This project involves testing of deep borehole IG_BH04 and the drilling and testing of deep boreholes IG_BH05 and IG_BH06 in the Revell site within the identified Potential Repository Area (PRA) as shown on Figure 1. The work comprises a total of eleven work packages and was carried out by a team led by WSP Canada Inc. (WSP) on behalf of the NWMO. The IG_BH06 program is described in a Borehole Characterization Plan (BCP) for IG_BH06.

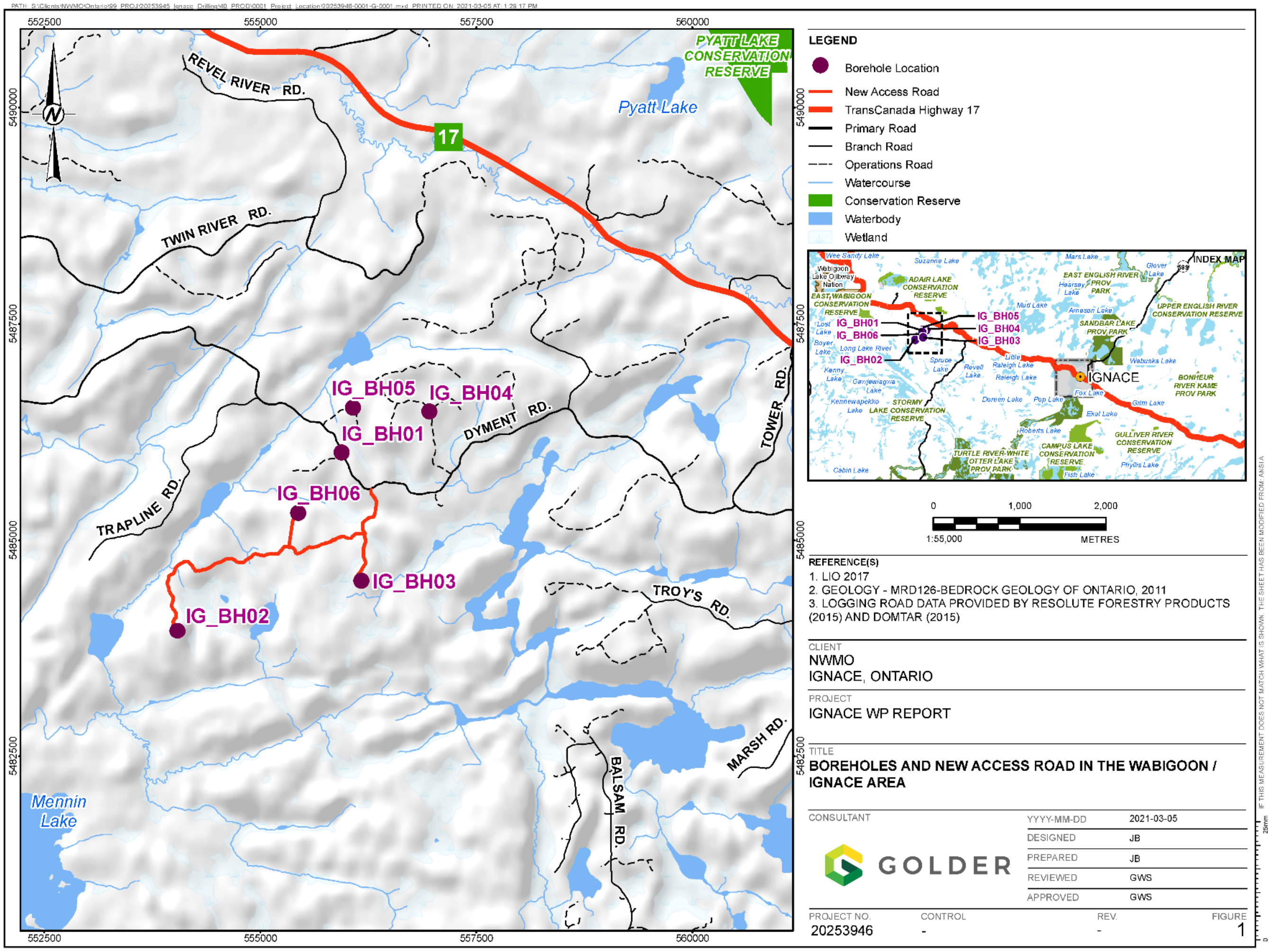


Figure 1: Location of IG_BH06 in relation to the Revell site

This data report describes the methodology, activities, and results for Work Package 4F (WP04F): Measurement of surface area and cation exchange capacity of core samples, on rock core samples recovered from borehole IG_BH06.

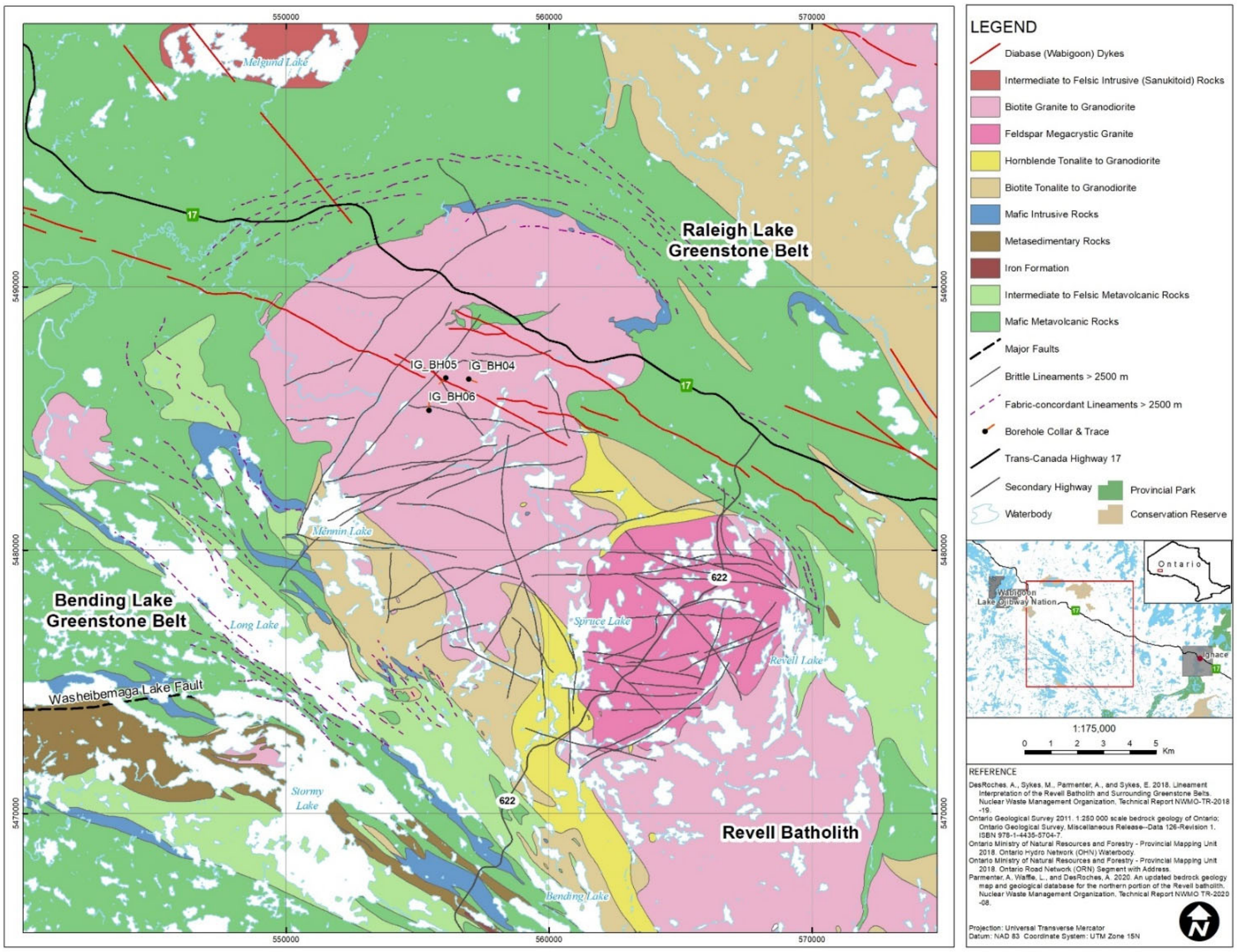
1.1 Geological Setting

The approximately 2.7 billion years old Revell batholith is located in the western part of the Wabigoon Subprovince of the Archean Superior Province. The batholith is roughly elliptical in shape trending northwest, is approximately 40 km in length, 15 km in width, and covers an area of approximately 455 km^2. Based on geophysical modelling, the batholith is approximately 2 km to 3 km thick through the center of the northern portion (SGL 2015). The batholith is surrounded by supracrustal rocks of the Raleigh Lake (to the north and east) and Bending Lake (to the southwest) greenstone belts (Figure 2).

IG_BH06 is located within an investigation area of approximately 19 km^2 in size, situated in the northern portion of the Revell batholith. Bedrock exposure in the area is generally very good due to minimal overburden, few water bodies, and relatively recent logging activities. Ground elevations generally range from 400 to 450 m above sea level. The ground surface broadly slopes towards the northwest as indicated by the flow direction of the main rivers in the area. Local water courses tend to flow to the southwest towards Mennin Lake (Figure 1).

Four main rock units are identified in the supracrustal rock group: mafic metavolcanic rocks, intermediate to felsic metavolcanic rocks, metasedimentary rocks, and mafic intrusive rocks (Figure 2). Sedimentation within the supracrustal rock assemblage was largely synvolcanic, although sediment deposition in the Bending Lake area may have continued past the volcanic period (Stone 2009; Stone 2010a; Stone 2010b). All supracrustal rocks are affected, to varying degrees, by penetrative brittle-ductile to ductile deformation under greenschist- to amphibolite-facies metamorphic conditions (Blackburn and Hinz 1996; Stone et al. 1998). In some locations, primary features, such as pillow basalt or bedding in sedimentary rocks are preserved, in other locations, primary relationships are completely masked by penetrative deformation. Uranium-lead (U-Pb) geochronological analysis of the supracrustal rocks produced ages that range between 2734.6 +/-1.1 Ma and 2725 +/-5 Ma (Stone et al. 2010).

Three main suites of plutonic rock are recognized in the Revell batholith, including, from oldest to youngest: a Biotite Tonalite to Granodiorite suite, a Hornblende Tonalite to Granodiorite suite, and a Biotite Granite to Granodiorite suite (Figure 2). Plutonic rocks of the Biotite Tonalite to Granodiorite suite occur along the southwestern and northeastern margins of the Revell batholith. The principal type of rock within this suite is a white to grey, medium-grained, variably massive to foliated or weakly gneissic, biotite tonalite to granodiorite. One sample of foliated and medium-grained biotite tonalite produced a U-Pb age of 2734.2+/-0.8 Ma (Stone et al. 2010). The Hornblende Tonalite to Granodiorite suite occurs in two irregularly-shaped zones surrounding the central core of the Revell batholith. Rocks of the Hornblende Tonalite to Granodiorite suite range compositionally from tonalite through granodiorite to granite and also include significant proportions of quartz diorite and quartz monzodiorite. One sample of coarse-grained grey mesocratic hornblende tonalite produced a U-Pb age of 2732.3+/-0.8 Ma (Stone et al. 2010). Rocks of the Biotite Granite to Granodiorite suite underlie most of the northern, central and southern portions of the Revell batholith. Rocks of this suite are typically coarse-grained, massive to weakly foliated, and white to pink in colour. The Biotite Granite to Granodiorite suite ranges compositionally from granite through granodiorite to tonalite. A distinct potassium (K)-Feldspar Megacrystic Granite phase of the Biotite Granite to Granodiorite suite occurs as an oval-shaped body in the central portion of the Revell batholith (Figure 2). One sample of coarse-grained, pink, massive K-feldspar megacrystic biotite granite produced a U-Pb age of 2694.0+/-0.9 Ma (Stone et al. 2010). The bedrock surrounding IG_BH06 is composed mainly of massive to weakly foliated felsic intrusive rocks that vary in composition between granodiorite and tonalite, and together form a relatively homogeneous intrusive complex. Bedrock identified as tonalite transitions gradationally into granodiorite and no distinct contact relationships between these two rock types are typically observed (SRK and Golder 2015; Golder and PGW 2017). Massive to weakly foliated granite is identified at the ground surface to the northwest of the feldspar-megacrystic granite. The granite is observed to intrude into the granodiorite-tonalite bedrock, indicating it is distinct from, and younger than, the intrusive complex (Golder and PGW 2017).



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Projection: Universal Transverse Mercator
Datum: NAD 83 Coordinate System: UTM Zone 15N

Figure 2: Geological Setting and Location of Boreholes IG_BH04, IG_BH05, and IG_BH06 in the Northern Portion of the Revell Batholith

West-northwest trending mafic dykes interpreted from aeromagnetic data extend across the northern portion of the Revell batholith and into the surrounding greenstone belts. One mafic dyke occurrence, located to the northwest of IG_BH06, is approximately 15-20 m wide (Figure 2). All of these mafic dykes have a similar character and are interpreted to be part of the Wabigoon dyke swarm. One sample from the same Wabigoon swarm produced a U-Pb age of 1887+/-13 Ma (Stone et al. 2010), indicating that these mafic dykes are Proterozoic in age. It is assumed based on surface measurements that these mafic dykes are sub-vertical (Golder and PGW 2017).

Long, narrow valleys are located along the western and southern limits of the investigation area (Figure 1). These local valleys host creeks and small lakes that drain to the southwest and may represent the surface expression of structural features that extend into the bedrock. A broad valley is located along the eastern limits of the investigation area and hosts a more continuous, un-named water body that flows to the south. The linear and segmented nature of this waterbody's shorelines may also represent the surface expression of structural features that extend into the bedrock.

Regional observations from mapping have indicated that structural features are widely spaced (typical 30 to 500 cm spacing range) and dominantly comprised of sub-vertical joints with two dominant orientations, northeast and northwest trending (Golder and PGW 2017). Interpreted bedrock lineaments generally follow these same dominant orientations in the northern portion of the Revell batholith (Figure 2; DesRoches et al. 2018). Minor sub-horizontal joints have been observed with minimal alteration, suggesting they are younger and perhaps related to glacial unloading. One mapped regional-scale fault, the Washeibemaga Lake fault, trends east and is located to the west of the Revell batholith (Figure 2). Ductile lineaments, also shown on Figure 2, follow the trend of foliation mapped in the surrounding greenstone belts. Additional details of the lithological units and structures found at surface within the investigation area are reported in Golder and PGW (2017).

1.2 Technical Objectives

Within the characterisation of the Revell Site, one core sample was taken from borehole IG_BH06 for the measurement of cation exchange capacity (CEC) and specific surface area (SSA) (by BET method) at different grain size fractions. The CEC measurements were conducted by Hydroisotop GmbH (Hydroisotop). The BET measurements were performed at the University of Bern. The work was carried out in accordance with the WP04F Test Plan, and the results of the experimental measurements are documented in the following sections of this report, with the analytical raw data provided in Appendix A.

2.0 SAMPLING AND SAMPLE PREPARATION

2.1 Sampling

One core sample from borehole IG_BH06 (sample ID: IG_BH06_SA001) with a length of 0.46 m was taken at a depth of 822.7 mbgs (downhole) on October 27, 2021 for the determination of CEC (cation exchange capacity) and specific surface area (SSA) using the BET method (Table 1). Sampling was conducted by Golder according to the instructions provided by Hydroisotop. After recovery from the borehole, the core sample was photographed and immediately packed in a plastic bag, evacuated and sealed airtight. This procedure was repeated for a second plastic bag and a final Al-coated plastic layer. The sample was stored in a refrigerator on site and then sent to Hydroisotop, Germany, in a cooler.

The sample was sent by Golder on October 29, 2021 and arrived at the Hydroisotop lab on November 05, 2021. The sample was well packed and arrived in the lab with preserved vacuum. At Hydroisotop the sample was stored in the fridge at 4 °C. The rock sample was identified as biotite granodiorite tonalite based on the mineralogy information from borehole IG_BH06.

2.2 Sample Preparation

The core sample was prepared for the CEC and BET measurements on February 3, 2022. The core sample was unpacked, photographed (photos of the rock core and core slice are shown in Appendix B) and immediately wrapped into Parafilm™ and cut by dry-sawing into full-diameter sections of 1 to 2 cm thickness. After sawing the core sections were crushed using a steel mortar and the crushed rock material was sieved using a vibration sieving tower (Retsch AS 450) and analytical stainless-steel sieves with mesh sizes of 0.065, 0.15, 0.2, 0.3, 1.0, 2.0, 4.0, 10 and 20 mm. The obtained crushed rock material with grain size fractions of 10-20 mm, 2-4 mm, 1-2 mm, 0.15-0.3 mm, 0.065-0.2 mm and < 0.065 mm was stored in PE bottles. After the first step of crushing and sieving, sufficient rock material of the largest grain size fraction 10-20 mm was achieved. Crushing and sieving was repeated until sufficient rock material with a grain size fraction of 2-4 mm was obtained and than the same procedure for grain size fraction 1-2 mm. Afterwards the grain size fraction of 0.3-1 mm was milled with a zirconium oxide grinding beaker and balls (Retsch AS 450) (300 rpm, 1 min) and sieved again to obtain the smaller grain size fraction (0.15-0.3 mm and 0.065-0.2 mm) needed for CEC and BET experiments.

One core section with a thickness of about 2.5 cm and a mass of 189.0 g was retained for BET experiments on a rock cylinder. Before sending this core slice and about 100 g of each grain size fraction to the University of Bern for BET measurements, the rock materials were dried at 105 °C for two weeks.

Table 1: Overview of the core samples taken from IG_BH06 for the CEC/BET measurements

Sample description		Depth				Sampling				
Sample ID	Hydro-isotop Lab-No.	From	to	Ave.	Length	Test Type	Date sampled	Time Recovered	Date sent	Date received
		[m]	[m]	[m]	[m]			[hh:mm]		
IG_BH06_ SA001	374206	822.43	822.89	822.66	0.46	BET surface area and CEC	2021/10/27	18:21	2021/10/29	2021/11/05

2.3 Core Logging Depth Correction

During the core logging of borehole IG_BH06, the logged depths were not reconciled with the depths obtained by the drilling supervisor calculated from measurement of the drill string and drill rod stick-up. This resulted in the WP03 core logging depths and core sample depths to be recorded as being slightly deeper than the actual sample depths. Refer to the WP03 Report for IG_BH06 for details of the correction applied to the core logging data.

All sample depths referred to in this report and appendices are the original, uncorrected sample depths in metres below ground surface (mbgs) downhole.

An Addendum to the Laboratory Report summarizing the original and corrected core depths for each sample is provided in Appendix D.

3.0 EXPERIMENTAL SET-UP AND ANALYTICAL METHODS

The specific surface areas for different grain size fractions were determined at the University of Bern. CEC experiments were conducted at Hydroisotop. Unless otherwise specified, the analytical work was conducted at Hydroisotop GmbH, Germany.

3.1 Determination of Specific Surface Area Using the BET Method

The specific surface area (SSA) was determined on the four grain size fractions (crushed rock) and a 9 mm diameter core approximately 25 to 30 mm thick drilled out of the intact core by nitrogen adsorption isotherms at -196 °C, using a BELSORP miniX surface analyser at the University of Bern (Dr. Urs Eggenberger).

A mass of 3 – 24 g of crushed rock material (Table 2; maximum volume 18 cm^3) was weighed (to an accuracy of 0.001 g) in a glass container until $^1/_3$ to maximum $^2/_3$ of the container was filled. After inserting the glass container containing the rock sample into a BELPREP vac III, the vacuum pump was started, and the sample was heated up to 105 °C overnight. Hereby the rock sample was desorbed of primary adsorbed gases under vacuum before measuring the adsorption isotherm. After the glass container cools down for a minimum of six hours, the vacuum pump was stopped, and the valves of the glass container were closed. To determine the weight loss, the glass container was weighed before heating and after cooling.

Table 2: Used masses of crushed rock material for the determination of specific surface area by BET method (analytical error of the BET-measurement is ± 30 %)

Grain size fraction	Sent masses [g]	Used masses [g]
< 0.065 mm	app. 100 g	3.698
0.065-0.2 mm	app. 100 g	3.082
0.15-0.3 mm	app. 100 g	5.936
1-2 mm	app. 100 g	6.559
2-4 mm	app. 100 g	7.101
10-20 mm	app. 100 g	5.575
Core slice (9 mm diameter and 25-30 mm thickness)	189.013 g	5.850

For the BET measurement, the neck of the glass container was fixed in the BELSORP-miniX surface analyzer, and the measurement was started using the BELSORP-miniX software. After approximately three hours the measurement was finished. The specific surface area, expressed as m^2/g, was then calculated from the amount of adsorbed N_2 and the sample weight according to the BET method (Brunauer et al. 1938) for a relative pressure of P/P_0 (measured vacuum pressure to the atmospheric pressure) from 0.05 to 0.3.

Certified standard materials (four different BET-standards with specific surface areas from 0.18 to 214 m^2/g) were used for calibration and quality checks. The reproducibility for BET surface area is < 2 % CV (Coefficient of Variation) at 1 m^2 measured area.

Raw data are included in the lab report in Appendix C.

3.2 Determination of Cation Exchange Capacity (CEC)

The CEC is determined by the nickel-ethylenediamine (Ni-en) method originally developed for clay-rich sedimentary rocks (Baeyens and Bradbury 1994; Bradbury and Baeyens 1997, 1998) and adapted to crystalline rocks according to Eichinger et al. (2023). This method is based on the premise that Ni, as a strong sorbent, will exclusively displace (and replace) all cations from the exchange sites in sheet silicates (the concentrations of the displaced cations will increase in solution, while the Ni concentration will accordingly decrease, allowing determination of the cation exchange capacity).

The CEC measurements were conducted on three crushed rock size fractions (0.065 - 0.2 mm, 2 – 4 mm and 10 – 20 mm). Ni-en extractions were performed in duplicate at four different solid to liquid mass ratios (1:10, 1:4, 1:2, 1:1). In addition, a blank was carried out for each experimental run as a control. The blank sample is the experimental approach without rock material, which is treated in the same way as the cation exchange experiments with rock material. This means that this sample is prepared in the same way (solution only), shaken, decanted, filtered and measured for its elements. In this way, possible sources of error or contamination caused by the performance or handling can be checked and, ideally, excluded.

The experimental stock solution (0.0005 molar Ni-en solution) was prepared by adding ethylenediamine to a $Ni(NO_3)_2$ solution. The Ni-en solution was prepared using degassed, oxygen- and CO_2-free water that was prepared in the glove box by boiling and N_2 bubbling for 30 minutes.

For the determination of the CEC values, the batch volumes were selected according to the grain size fraction and the experiment duration. Table 3 gives an overview of the respective rock and test solution masses for the individual experiments.

Table 3: Overview of the experimental set-ups for CEC measurements

Rock core sample/test solution mass ratio		1:1	1:2	1:4	1:10	Blank
Experiment 0.065-0.2 mm (48 h) and 2-4 mm (96 h)						
rock material mass	[g]	40	20	10	10	0
test solution mass	[g]	40	40	40	100	75
Experiment 10-20 mm (app. 27 weeks or 185 days*)						
rock material mass	[g]	60	30	15	10	0
test solution mass	[g]	60	60	60	100	75

*until equilibrium (nearly) reached

The experimental procedures were carried out in an oxygen free glove box, which was continuously flushed with nitrogen and held on slight nitrogen overpressure. Oxygen concentrations in the glove box were measured in the inner atmosphere. The oxygen concentration was lower than 5 % related to air.

The weighted rock material was added into a 100 mL polyethylene bottle. To start the experiment, the experimental solution was added to the rock material in the polyethylene bottle in the glove box using a graduated cylinder. Immediately after addition of the solution, the pH and redox potential of the experimental solution were measured with a WTW Multi 3620 IDS with a SenTix-ORP-T900 redox-electrode and a SenTix-940 pH-electrode. The bottles were closed and continuously shaken upside down.

The samples from the experiments with the smallest (0.065-0.2 mm) and the middle grain size fraction (2-4 mm) were continuously shaken end-over-end in the polyethylene bottles. The polyethylene bottles of the large grain size fraction (10-20 mm) experiments were only shaken one to two times per day, to avoid too strong erosion of the material as shown by the experiments of the IG_BH04 core sample (IG_BH04_SA001). The reaction time depended on the sizes of the crushed rock sample. The smallest grain size fraction (0.065-0.2 mm) samples were shaken for 48 h, the grain size fraction 2-4 mm samples for 96 h. The largest grain size fraction (10-20 mm) samples were shaken until stable Ni-concentrations in the solution were achieved. After the nickel concentration was stable, an aliquot of 1.0 mL sub-sample was taken at 8, 16, 40, 56, 82, 101, 157 and 171 days. The experimental solutions were sampled after 8, 16, 40, 56, 82, 101, 157 and 171 days as sub-samples until the final sampling at day 185.

A 1 mL sub-sample was taken from the polyethylene bottle with a pipette and filled into a 1.5 mL reaction tube. The reaction tube was centrifuged until the solution was clear. The solution was then carefully removed with a pipette. 0.1 mL solution was directly added to a 10 mL centrifuge tube containing 9900 µL of 2 % ultrapure HNO_3 for Ni concentration measurement (1:100 dilution). The remaining solution was transferred to a clean 1.5 mL reaction tube for the cation concentration analysis by IC.

For the final sampling of all experimental set-ups, the pH value and the redox potential of the final solution were measured in the glove box. The solution was then decanted into a 50 mL centrifuge tube and centrifuged until the solution was clear. The solution was divided into 3 vials in the glove box for concentration analysis.

1. For the photometer and IC measurements, a partial sample (vial 1) was filtered with a 0.45 µm and finally a 0.2 µm syringe filter and filled into a tube.

2. For the total iron (Fe_{tot}) analysis, a 2nd aliquot of the sample (vial 2) was filled unfiltered (0.1 mL) into a 10 mL centrifuge tube containing 9.9 mL of 2 % HNO_3.

3. For all cations measured by the AAS and Fe^{2+}, the 3rd subsample (vial 3) was filtered with a 0.45 µm and a 0.2 µm syringe filter and filled into a 50 mL tube and added concentrated HNO_3 (0.1 %).

All samples were immediately analyzed after the experiment was finished.

The Ni-en solution was analyzed for Ni^{2+} concentration before and after extraction by AAS (Analytic Jena contra 800) with a detection limit of 0.01 mg/L. The analytical error of the AAS is ± 10 % based on multiple measurements of high-grade, commercial check-standard solutions.

For the sub-samples, Ni^{2+}, $Ca^{2+}$and Mg^{2+} concentrations were determined by AAS and K^+, Na^+ and NH_4^+ concentrations by IC (Thermo Scientific Dionex Integrion HPIC).

For the final samples, Ni^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+}, Ba^{2+}, Fe^{2+}, Fe^{3+}, Al^{3+} concentrations were determined by AAS. Na^+, K^+, Cl^-, SO_4^{2-}, F^-, NO_3^- and NH_4^+ concentrations were measured by IC.

The cation exchange capacity (CEC) was then derived from the Ni consumption, i.e., the difference between the Ni concentration in the initial and the final extract solutions. The cumulative error of the entire procedure (i.e., extraction and analysis) is approximately 10 %. The cation concentrations determined before and after the experiments are used to calculate the cation exchange capacity. The exchange of cations can always take place in both directions. As long as cations (except nickel) are brought from the rock into the solution, the value is positive, meaning it is released from the rock. If cations from the solution are bound in the rock, meaning a backward reaction takes place, the exchange factor is negative.

This means that the forward reaction (from the rock into the solution) has positive values and the backward reaction (from the solution into the rock) has negative values. For nickel, the forward reaction (positive values) was defined as solution into the rock and the backward reaction (negative values) from rock into solution.

In order to confirm the CEC value obtained by measuring the nickel concentration, major and trace cation (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Sr^{2+}, Ba^{2+}, Fe^{2+}, Fe^{3+}, Al^{3+}) concentrations in the final extract solution were also determined. The nickel, major and trace cation concentrations were measured in duplicate. pH was measured in the individual solutions before and after the experiments using a Schott Titroline alpha system.

All CEC experiments were conducted under anoxic conditions, so that the concentrations of redox specific ions like Fe^{2+}, Fe^{3+} and NH_4^+ are obtained. Total iron (Fe_{tot}) and Fe^{2+} concentrations were measured by AAS, afterwards the Fe^{3+} concentration was calculated by the concentration of Fe_{tot} minus concentration of Fe^{2+}. The redox potential of the solution was analyzed before and after the experiments for all CEC solutions using a WTW Multi 3620 IDS with a SenTix-ORP-T900 electrode.

4.0 RESULTS

The results of the BET and CEC experimental measurements of the core sample (IG_BH06_SA001) taken from borehole IG_BH06 are explained in the following sections.

4.1 BET Specific Surface Area

The results of the BET measurements are summarized in Table 4 and Figure 3. The accuracy of the measurement for the grain size fractions (<0.065 mm, 0.065-0.2 mm, 0.15-0-3 mm, 1-2 mm, 2-4 mm and 10-20 mm) and the core slice is ± 30%. The specific surface area of the grain size fraction 2-4 mm is 0.04 m^2/g, of the grain size fraction 10-20 mm is 0.03 m^2/g, and of the core slice (with 9 mm diameter, 2.5 to 3 cm high) is 0.02 m^2/g. For the grain size fraction < 0.065 mm, the specific surface area is 2.49 m^2/g, for the grain size fraction 0.065-0.2 mm is 3.76 m^2/g, for the grain size fraction 0.15-0.3 mm is 0.73 m^2/g and for the grain size fraction 1-2 mm is 0.10 m^2/g.

Within the same sample and rock type, the specific surface area generally increases with decreasing grain size.

Table 4: Results of the specific surface area measured at different grain size fractions by the BET method (analytical error = ± 30 %)

Grain size fraction	Specific surface area [m^2/g]
< 0.065 mm	2.49
0.065-0.2 mm	3.76
0.15-0.3 mm	0.73
1-2 mm	0.10
2-4 mm	0.04
10-20 mm	0.03
Core (9 mm diameter and 25-30 mm thickness)	0.02

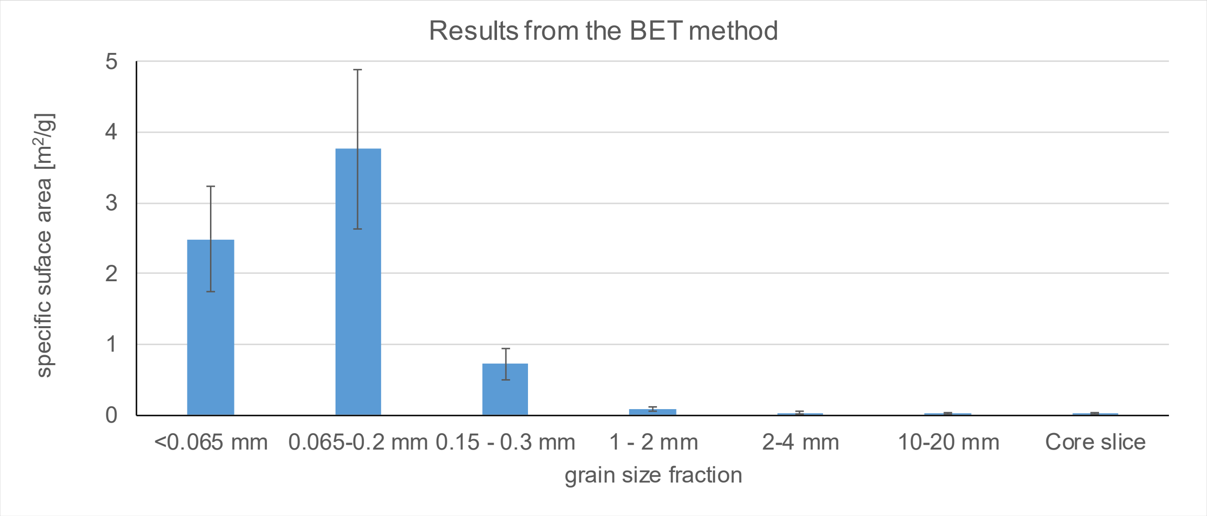


Figure 3: The specific surface area measured of IG_BH06 at different grain size fractions and at a 9 mm diameter core using the BET method

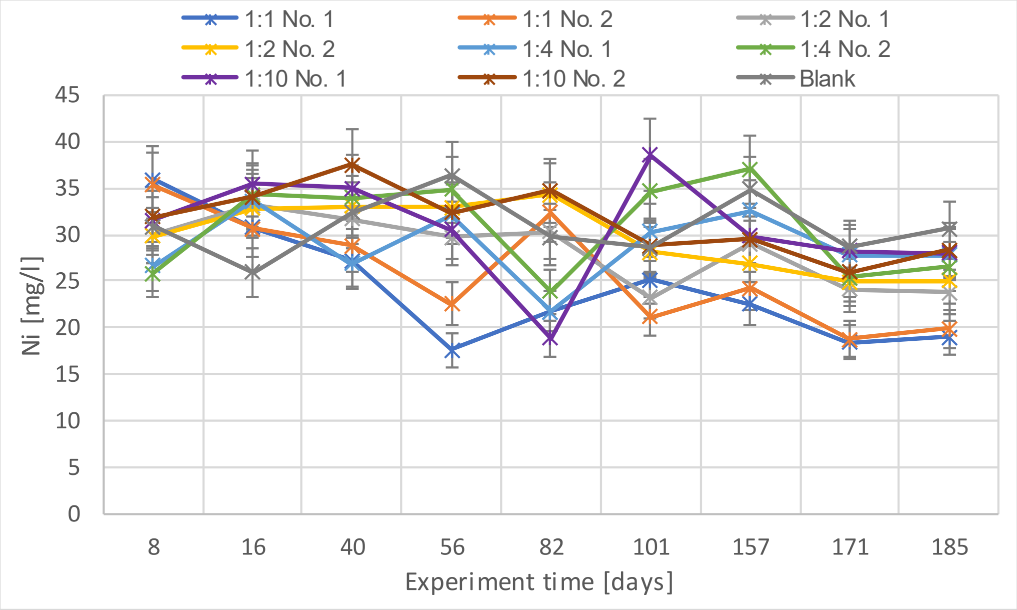
4.2 Cation Exchange Capacity

4.2.1 CEC_{Ni}

The initial Ni concentration of the Ni-en stock solution was on average for all experiments (including all start, sub and end samples of the Ni-en-Solution) 29.6 mg/L with a variation of± 3.2 mg/L. Each experiment was performed in duplicate indicated as No. 1 and No. 2 in the following tables and figures.

Nickel concentrations of all experimental approaches were analyzed for the periodically taken sub-samples. The element concentrations of the samples for the time-series CEC measurements are compiled in Appendix A, Tables A-3 to A-10.

Figure 4 shows the nickel concentrations and the resulting CEC_{Ni} values during the course of the experiment for the largest grain size fraction (10-20 mm) versus experimental time. The nickel concentration in the solution decreases with the experimental time because nickel exchanges with the cations on the rock surface. As a result, the calculated CEC value increases with time. The equilibrium of Ni exchange with cations on the rock surface was defined as being reached when the nickel concentration between two subsamples did not change by more than ± 10 %. The time-series show that the time period of reaching Ni-equilibration depends on the rock core sample (solid)/test solution (liquid) ratio. The experiments with high solid/liquid (s/l) mass ratio (1:1) reach Ni-equilibrium faster than those with lower s/l mass ratios (1:2, 1:4, 1:10). As a result, the nickel concentration levels off at a constant lower value, confirming the cation exchange equilibrium within the experimental time (Figure 4). For all experiments cation exchange equilibrium was achieved with respect to the Ni-concentrations. The equilibrium time appears to depend on the s/l ratio. For the experiments conducted with a s/l ratio of 1:1 equilibrium was achieved after 171 days, whereas for those with a s/l ratio of 1:2, 1:4 and 1:10 equilibrium was nearly reached after 185 days. This can be explained by the higher availability of nickel at higher s/l ratios and so to the higher reactive surface in relation to the Ni concentration.



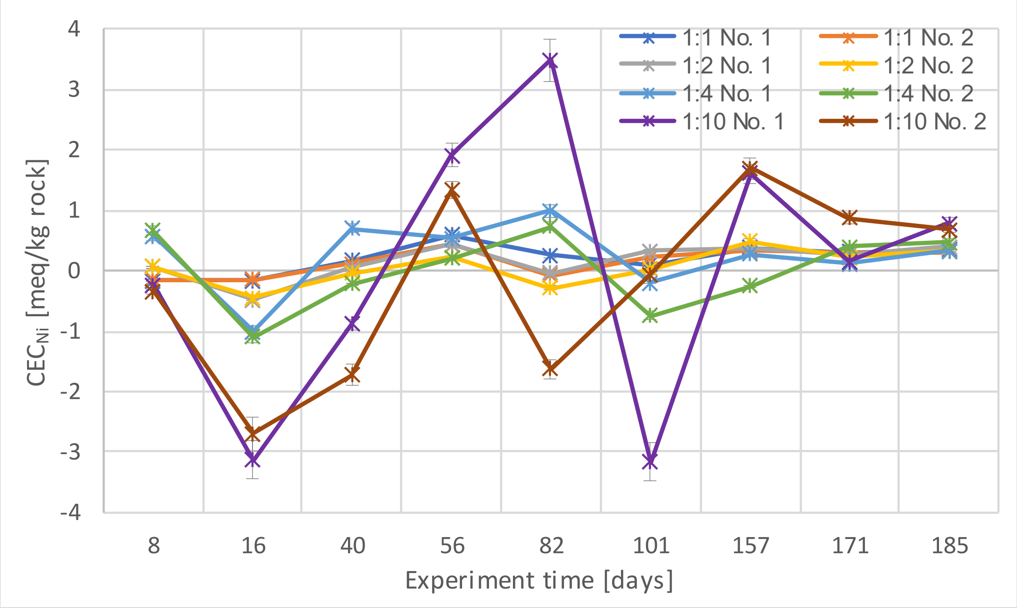


Figure 4: Nickel concentration in test solution (upper) and CEC$_{Ni}$ values (bottom) measured for the largest grain size fraction 10-20 mm over experimental time. Experiments ran for 185 days.

The results of the CEC experiments (concentrations of the final measurements when the experiment was completed) performed on the smallest grain size fraction (0.065-0.2 mm) are summarized in Table 5, the results measured for the middle grain size fraction (2-4 mm) are summarized in Table 6 and the results measured for the largest grain size fraction (10-20 mm) are summarized in Table 7.

The pH value and redox potential were measured at the beginning of the experiment right after adding the experimental solution to the grinded rock material and after the experiment (after 48 h, 96 h, 185 days for the grain size fractions 0.065-0.2 mm, 2-4 mm and 10-20 mm, respectively). During the experiments the pH values generally slightly increased or remained constant depending on the s/l ratio (Tables 5 to 7).

Table 5: Element concentrations of the final CEC experiment solutions using different rock material / test solution mass ratios conducted with the smallest grain size fraction (0.065-0.2 mm) after 48 h (sample name IG_BH06_SA001 0.065-0.2 mm)

Hydro Lab.-No.		382210	382212	382213	382214	382215	382216	382217	382218	382219	382220
Sample		Ni-en Solution	1:1 No. 1	1:1 No. 2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock/ test solution Mass ratio (s/l ratio)	unit	-	1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
Rock mass	g	-	39.999	39.997	19.995	19.996	10.002	9.999	10.002	10.001	0
Solution mass	g	-	38.849	39.239	39.123	39.191	39.270	39.414	99.021	99.446	73.807
pH value start		-	9.65	9.47	9.42	9.21	9.61	9.55	9.39	9.41	8.92
pH value end		-	10.09	10.06	10.06	10.06	9.96	9.95	9.73	9.73	8.91
Redox potential start	mV	-	-6.0	-10.8	7.4	-6.0	2.8	-3.4	30.5	14.0	29.7
Redox potential end	mV	-	-76.5	-75.5	-71.3	-80.9	-64.8	-83.8	-49.0	-41.1	-5.0

Hydro Lab.-No.		382210	382212	382213	382214	382215	382216	382217	382218	382219	382220
Sample		**Ni-en Solution**	**1:1 No. 1**	**1:1 No. 2**	**1:2 No. 1**	**1:2 No. 2**	**1:4 No. 1**	**1:4 No. 2**	**1:10 No. 1**	**1:10 No. 2**	**Blank**
Rock/ test solution Mass ratio (s/l ratio)	**unit**	**-**	**1:1**	**1:1**	**1:2**	**1:2**	**1:4**	**1:4**	**1:10**	**1:10**	**-**
Nickel (Ni^{2+})	mg/l	27.0	0.257	0.400	0.730	0.762	2.73	2.79	11.3	12.1	29.7
Barium (Ba^{2+})	mg/l	0.055	0.027	0.128	0.078	0.092	0.009	0.028	0.003	0.003	0.069
Strontium (Sr^{2+})	mg/l	0.033	0.005	0.031	0.011	0.016	0.008	0.009	0.010	0.012	0.033
Aluminum (Al^{3+})	mg/l	< 0.02	2.75	26.5	11.0	10.6	1.05	2.46	0.257	0.316	< 0.02
Iron tot. (Fe_{tot})	mg/l	< 0.01	14.1	14.0	4.46	6.31	1.37	1.69	< 0.01	< 0.01	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	1.19	14.3	3.004	3.236	0.448	0.815	0.013	0.010	< 0.01
Iron-III (Fe^{3+})	mg/l	< 0.01	12.9	< 0.01	1.45	3.086	0.923	0.873	< 0.01	< 0.01	< 0.01
Sodium (Na^{+})	mg/l	< 0.2	145	145	96.8	98.4	62.9	61.2	29.2	28.7	1.71
Potassium (K^{+})	mg/l	< 0.1	35.9	38.3	27.8	28.6	22.5	22.7	14.5	14.8	2.23
Calcium (Ca^{2+})	mg/l	8.81	2.21	4.25	2.83	1.73	2.27	2.49	5.47	4.55	8.75
Magnesium (Mg^{2+})	mg/l	0.424	0.557	2.59	0.957	1.01	1.03	0.924	0.961	0.592	0.769
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.15	0.16	0.24	0.23	< 0.1

Table 6: Element concentrations of the final CEC experiment solutions using different rock material / test solution mass ratios conducted with the medium grain size fraction (2-4 mm) after 96 h (sample name IG_BH06_SA001 2-4 mm)

Hydro Lab.-No.		**380975**	**380977**	**380978**	**380979**	**380980**	**380981**	**380982**	**380983**	**380984**	**380985**
Sample		**Ni-en Solution**	**1:1 No. 1**	**1:1 No. 2**	**1:2 No. 1**	**1:2 No. 2**	**1:4 No. 1**	**1:4 No. 2**	**1:10 No. 1**	**1:10 No. 2**	**Blank**
Rock/ test solution Mass ratio (s/l ratio)	**unit**	**-**	**1:1**	**1:1**	**1:2**	**1:2**	**1:4**	**1:4**	**1:10**	**1:10**	**-**
Rock mass	g	-	39.999	39.999	20.004	20.008	10.003	9.993	9.998	9.997	0
Solution mass	g	-	39.679	39.924	39.628	40.241	39.858	39.945	99.185	99.617	74.423
pH value start		-	8.99	8.98	8.96	8.96	8.96	8.96	8.97	8.95	8.95
pH value end		-	9.73	9.73	9.66	9.64	9.46	9.47	9.22	9.22	9.73
Redox potential start	mV	-	48.7	48.8	49.6	50.4	59.1	49.3	62.7	59.8	67.9
Redox potential end	mV	-	-145.8	-151.0	-109.3	-93.4	-55.2	-56.6	-36.9	-35.6	-145.8
Nickel (Ni^{2+})	mg/l	25.8	5.06	3.87	9.22	9.13	21.5	22.7	26.5	31.4	26.7
Barium (Ba^{2+})	mg/l	0.059	0.125	0.119	0.016	0.032	0.018	0.019	0.016	0.105	0.059
Strontium (Sr^{2+})	mg/l	0.023	0.045	0.044	0.029	0.039	0.048	0.049	0.038	0.057	0.026
Aluminum (Al^{3+})	mg/l	< 0.02	16.2	16.5	0.717	2.85	1.29	1.26	0.370	2.20	< 0.02
Iron tot. (Fe$_{tot}$)	mg/l	< 0.01	9.47	11.4	2.34	2.46	2.66	3.43	< 0.01	1.23	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	7.78	8.45	0.249	0.831	0.379	0.305	0.153	0.475	< 0.01
Iron-III (Fe^{3+})	mg/l	< 0.01	1.69	2.91	2.10	1.63	2.28	3.13	< 0.01	0.752	< 0.01
Sodium (Na^{+})	mg/l	< 0.2	29.9	31.5	19.1	17.2	10.2	10.1	3.95	3.77	2.28
Potassium (K^{+})	mg/l	< 0.1	27.1	28.4	20.9	19.7	12.1	13.0	4.22	4.49	1.25
Calcium (Ca^{2+})	mg/l	10.7	6.49	7.65	6.00	8.69	10.5	9.28	10.6	9.38	9.41
Magnesium (Mg^{2+})	mg/l	0.489	2.67	3.99	0.96	1.55	0.971	0.940	0.804	1.26	0.309
Ammonium (NH$_4^{+}$)	mg/l	< 0.1	0.15	0.19	0.14	0.13	< 0.1	< 0.1	< 0.1	< 0.1	0.11

Table 7: Element concentrations of the final CEC experiment solutions using different rock material / test solution mass ratios conducted with the largest grain size fraction (10-20 mm) after 185 days (sample name IG_BH06_SA001 10-20 mm)

Hydro Lab.-No.		**380687**	**388026**	**388027**	**388028**	**388029**	**388030**	**388031**	**388032**	**388033**	**388034**
Sample		**Ni-en Solution**	**1:1 No. 1**	**1:1 No. 2**	**1:2 No. 1**	**1:2 No. 2**	**1:4 No. 1**	**1:4 No. 2**	**1:10 No. 1**	**1:10 No. 2**	**Blank**
Rock/ test solution Mass ratio (s/l ratio)	**unit**	**-**	**1:1**	**1:1**	**1:2**	**1:2**	**1:4**	**1:4**	**1:10**	**1:10**	**-**
Rock mass	g	-	60.032	60.072	29.894	30.006	15.060	15.080	9.978	10.044	0
Solution mass	g	-	59.211	60.097	59.731	59.509	59.660	59.688	100.025	99.130	74.896
pH value start		-	9.02	9.01	9.02	9.02	9.01	9.01	9.00	9.00	8.98
pH value end		-	8.89	8.94	8.51	8.64	8.83	8.60	8.83	8.77	8.90
Redox potential start	mV	-	42.8	39.2	46.9	44.8	48.6	50.1	42.2	47.8	68.1
Redox potential end	mV	-	29.4	27.8	50.8	38.4	44.3	53.1	49.5	44.0	72.1
Nickel (Ni^{2+})	mg/l	27.0	19.0	19.9	23.8	25.0	27.8	26.5	28.1	28.4	30.6
Barium (Ba^{2+})	mg/l	0.045	0.042	0.081	0.040	0.022	0.037	0.041	0.033	0.027	0.027
Strontium (Sr^{2+})	mg/l	0.029	0.210	0.207	0.137	0.136	0.089	0.116	0.078	0.066	0.045
Aluminum (Al^{3+})	mg/l	< 0.02	0.037	0.043	0.026	0.048	0.080	0.023	0.014	0.017	0.017
Iron tot. (Fe_{tot})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Iron-III (Fe^{3+})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Sodium (Na^{+})	mg/l	0.28	14.5	13.9	5.15	3.76	2.52	4.73	3.18	1.97	1.53
Potassium (K^{+})	mg/l	< 0.1	2.63	2.35	2.56	2.25	2.59	2.87	0.81	1.12	1.92
Calcium (Ca^{2+})	mg/l	10.1	18.1	19.7	20.4	18.4	12.8	14.6	11.6	11.5	12.1
Magnesium (Mg^{2+})	mg/l	0.482	0.404	0.486	0.516	0.527	0.486	0.442	0.472	0.470	0.417
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1

Table 8 and Figure 5 give an overview of the calculated mean CEC values based on the nickel concentrations. Depending on the rock material / test solution ratio (s/l ratio) and the grain size fraction, the CEC_{Ni} value varies from -1.60 to 6.19 meq/kg rock. The results of the duplicates of the individual experiments agree well with each other (Table 8). This shows that the experiments are comparable and reproducible. An exception is the 1:10 experiment of grain size fraction 2-4 mm, where the replicates differ from each other. The reason is probably that the 10 g rock weight (in 100 g solution) corresponds to only a few rock grains and thus the reactive surface of the rock sample can differ significantly between the two replicates, and the change in the Ni concentrations were very low, in the range of the standard deviation.

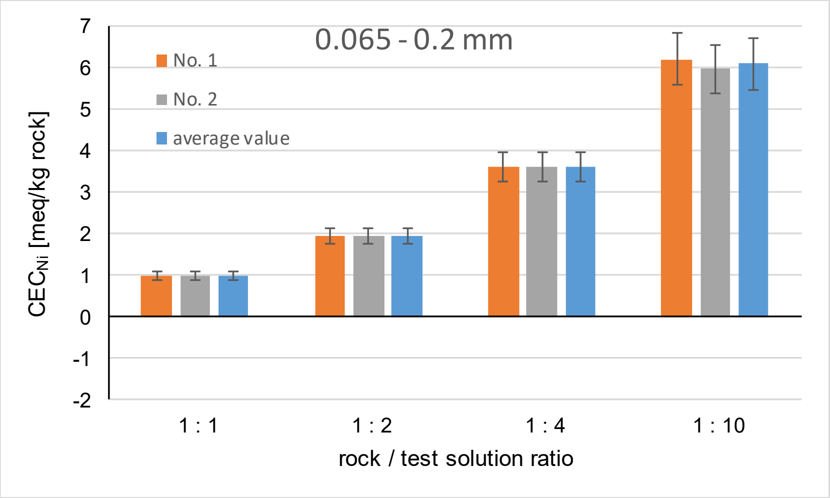
The results also show that the CEC value for each grain size fraction increases as the volume of solution increases in relation to the mass of rock. For the grain size fraction 0.0.65-0.2 mm the CEC_{Ni} values increase from 0.97-0.98 meq/kg rock at a s/l ration of 1:1 to 5.98-6.19 meq/kg rock at a s/l ratio of 1:10. The grain size fraction 2-4 mm shows greater variations and a significantly lower trend than the grain size fraction 0.065-0.2 mm. From the s/l ratio 1:1 to the s/l ratio 1:2, a slight increase from 0.73-0.78 meq/kg rock to 1.18-1.20 meq/kg rock can be seen, as for the other grain size fractions. However, the s/l ratio 1:4 again shows similar values (0.54-0.71 meq/kg rock) to the 1:1 ratio. As already mentioned above, the duplicates of the s/l ratio 1:10 show very large variations among each other, cause the Ni values changes are lower than the standard deviation. For the grain size fraction 10-20 mm the CEC_{Ni} values increase very slightly from 0.32-0.34 meq/kg rock at a s/l ration of 1:1 to 0.68-0.80 meq/kg rock at a s/l ratio of 1:10.

CEC_{Ni} values increase with decreasing grain size fractions for the experimental approaches with a s/l ratio of 1:4 and 1:10. For the experiments with a s/l ratio of 1:2 and 1:1 this trend is less pronounced (Table 8, Figure 5). At a s/l ratio of 1:1, CEC_{Ni} values of the individual grain size fractions range from 0.32 meq/kg rock (at 10-20 mm) to 0.98 meq/kg rock (at 0.065-0.2 mm), whereas at a s/l ratio of 1:10, CEC_{Ni} values range from < 0.01 meq/kg rock (at 10-20 mm) to 6.19 meq/kg rock (at 0.065-0.2 mm) (see Table 8).

Table 8: Calculated CEC_{Ni} values, mean CEC_{Ni} values and standard deviation from the CEC experiments with three grain size fractions 0.065-0.2 mm, 2-4 mm and 10-20 mm

Rock/test solution mass ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10
0.065-0.2 mm	CEC_{Ni} (meq/kg rock)	0.97	0.98	1.93	1.93	3.60	3.61	6.19	5.95
	Mean CEC_{Ni} (meq/kg rock)	0.98±0.01		1.93±0.01		3.61±0.01		6.07±0.17	
2-4 mm	CEC_{Ni} (meq/kg rock)	0.73	0.78	1.18	1.20	0.71	0.54	0.07	<0.01
	Mean CEC_{Ni} (meq/kg rock)	0.75±0.03		1.19±0.02		0.63±0.11		0.07±0.07*	
10-20 mm	CEC_{Ni} (meq/kg rock)	0.34	0.32	0.40	0.33	0.33	0.47	0.80	0.68
	Mean CEC_{Ni} (meq/kg rock)	0.33±0.02		0.36±0.05		0.40±0.10		0.74±0.08	

*based only on one batch



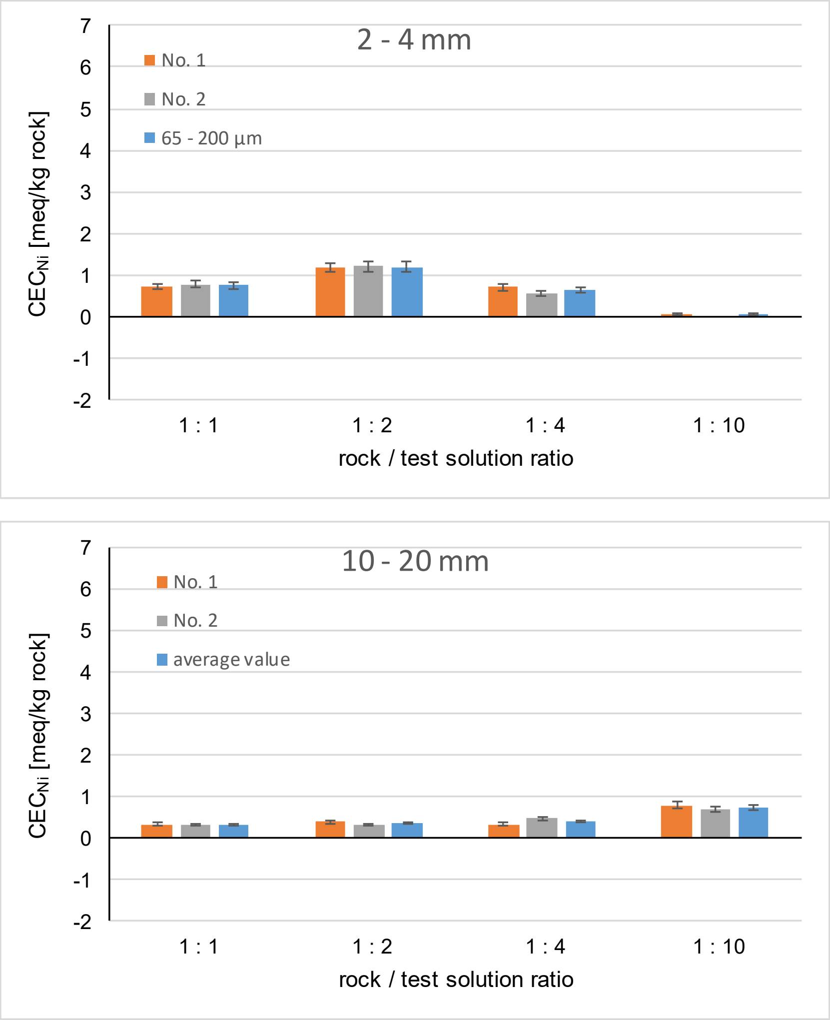


Figure 5: Results of the CEC_{Ni} values determined for the different grain size fractions (0.065-0.2 mm; 2-4 mm; 10-20 mm) and rock/test solution mass ratios based on calculation by nickel concentrations

4.2.2 $CEC_{Cations}$

To verify the measured CEC_{Ni} values calculated by nickel concentration measurement, the concentrations of major and trace cations (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe) in the final extract solutions are measured by ion chromatography and AAS. The concentrations of each cation and the CEC calculations based on the sum of cations ($CEC_{cations}$) are summarized in Table 9 to 11. The results of the CEC calculations based on the sum of all the cations are summarized in Table 12 and Figure 6. The CEC calculations based on the cation concentrations take the initial concentrations into account and are corrected for those.

Table 9: CEC values of each cation (Na^{+}, K^{+}, Ca^{2+}, Mg^{2+}, NH$_4^{+}$, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe) and CEC values of the sum of cations determined for the smallest grain size fraction (0.065-0.2 mm). Sample name IG_BH06_SA001 0.065-0.2 mm. All values in meq/kg rock

Hydro Lab.-No.	382212	382213	382214	382215	382216	382217	382218	382219
Sample	**1:1 No. 1**	**1:1 No. 2**	**1:2 No. 1**	**1:2 No. 2**	**1:4 No. 1**	**1:4 No. 2**	**1:10 No. 1**	**1:10 No. 2**
Rock/ test solution Mass ratio (s/l ratio)	**1:1**	**1:1**	**1:2**	**1:2**	**1:4**	**1:4**	**1:10**	**1:10**
Nickel (Ni^{2+})	0.97	0.98	1.93	1.93	3.60	3.61	6.19	5.95
Barium (Ba^{2+})	-5.92×10^{-4}	8.41×10^{-4}	2.35×10^{-4}	6.54×10^{-4}	-3.42×10^{-3}	-2.37×10^{-3}	-9.52×10^{-3}	-9.65×10^{-3}
Strontium (Sr^{2+})	-6.26×10^{-4}	-5.20×10^{-5}	-9.81×10^{-4}	-7.51×10^{-4}	-2.26×10^{-3}	-2.16×10^{-3}	-5.14×10^{-3}	-4.72×10^{-3}
Aluminum (Al^{3+})	0.20	1.93	1.59	1.55	0.31	0.72	0.19	0.23
Iron tot. (Fe$_{tot}$)	0.49	0.49	0.31	0.44	0.19	0.24	< 0.01	< 0.01
Iron-II (Fe^{2+})	0.04	0.50	0.21	0.23	0.06	0.12	4.58×10^{-3}	3.59×10^{-3}
Iron-III (Fe^{3+})	0.67	< 0.01	0.15	0.32	0.19	0.18	< 0.01	< 0.01
Sodium (Na^{+})	6.05	6.11	8.09	8.24	10.45	10.20	11.84	11.67
Potassium (K^{+})	0.84	0.91	1.28	1.32	2.04	2.06	3.11	3.20
Calcium (Ca^{2+})	-0.32	-0.22	-0.58	-0.69	-1.27	-1.23	-1.62	-2.08
Magnesium (Mg^{2+})	-0.02	0.15	0.03	0.04	0.09	0.05	0.16	-0.14
Ammonium (NH$_4^{+}$)	< 0.1	< 0.1	< 0.1	< 0.1	0.03	0.03	0.13	0.13
CEC$_{cations}$ (all cation/sum)	7.24	9.36	10.73	10.91	11.83	12.07	13.79	12.99
CEC$_{main\text{-}cations}$ (Na^{+}, K^{+}, Ca^{2+}, Mg^{2+})	6.56	6.95	8.83	8.92	11.30	11.08	13.48	12.64

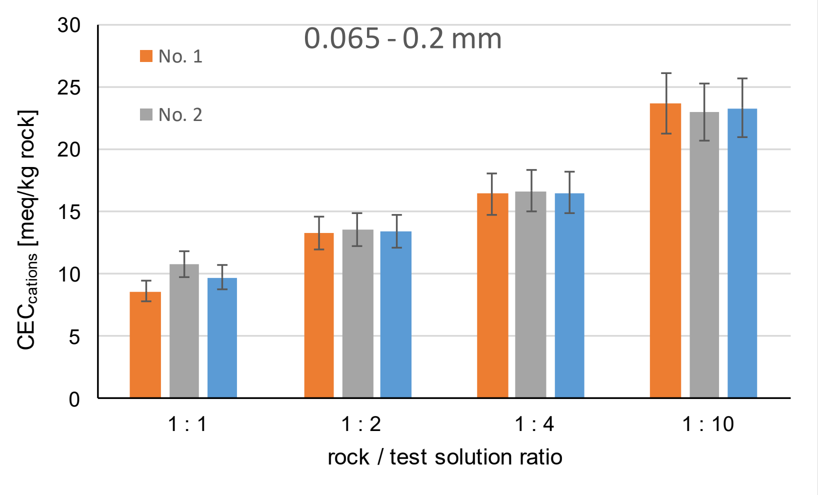
Table 10: CEC values of each cation (Na^{+}, K^{+}, Ca^{2+}, Mg^{2+}, NH$_4^{+}$, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe) and CEC values of the sum of cations determined for the medium grain size fraction (2-4 mm). Sample name IG_BH06_SA001 2-4 mm. All values in meq/kg rock

Hydro Lab.-No.	380977	380978	380979	380980	380981	380982	380983	380984
Sample	**1:1 No. 1**	**1:1 No. 2**	**1:2 No. 1**	**1:2 No. 2**	**1:4 No. 1**	**1:4 No. 2**	**1:10 No. 1**	**1:10 No. 2**
Rock/ test solution Mass ratio (s/l ratio)	**1:1**	**1:1**	**1:2**	**1:2**	**1:4**	**1:4**	**1:10**	**1:10**
Nickel (Ni^{2+})	0.73	0.78	1.18	1.20	0.71	0.54	0.07	-1.60
Barium (Ba^{2+})	9.53×10^{-4}	8.60×10^{-4}	-1.27×10^{-3}	-8.18×10^{-4}	-2.39×10^{-3}	-2.35×10^{-3}	-6.27×10^{-3}	6.58×10^{-3}
Strontium (Sr^{2+})	4.28×10^{-4}	4.06×10^{-4}	1.19×10^{-4}	5.91×10^{-4}	1.98×10^{-3}	2.06×10^{-3}	2.58×10^{-3}	6.94×10^{-3}
Aluminum (Al^{3+})	1.19	1.22	0.11	0.42	0.38	0.37	0.27	1.62
Iron tot. (Fe$_{tot}$)	0.34	0.41	0.17	0.18	0.38	0.49	< 0.01	0.44
Iron-II (Fe^{2+})	0.28	0.30	0.02	0.06	0.05	0.04	0.05	0.17
Iron-III (Fe^{3+})	0.09	0.16	0.22	0.18	0.49	0.67	< 0.01	0.40
Sodium (Na^{+})	1.19	1.27	1.45	1.31	1.37	1.36	0.72	0.65
Potassium (K^{+})	0.66	0.69	1.00	0.95	1.11	1.20	0.75	0.83
Calcium (Ca^{2+})	-0.14	-0.09	-0.34	-0.07	0.21	-0.02	0.58	-0.01
Magnesium (Mg^{2+})	0.19	0.30	0.11	0.21	0.22	0.21	0.40	0.78
Ammonium (NH$_4^{+}$)	2.20×10^{-3}	4.43×10^{-3}	3.29×10^{-3}	2.23×10^{-3}	< 0.1	< 0.1	< 0.1	< 0.1

Hydro Lab.-No.	380977	380978	380979	380980	380981	380982	380983	380984
$CEC_{cations}$ (all cation/sum)	3.43	3.81	2.49	2.99	3.67	3.61	2.73	4.31
$CEC_{main\text{-}cations}$ (Na^+, K^+, Ca^{2+}, Mg^{2+})	1.90	2.18	2.21	2.39	2.91	2.74	2.46	2.24

Table 11: CEC values of each cation (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe) and CEC values of the sum of cations determined for the largest grain size fraction (10-20 mm). Sample name IG_BH06_SA001 10-20 mm. All values in meq/kg rock

Hydro Lab.-No.	**388026**	**388027**	**388028**	**388029**	**388030**	**388031**	**388032**	**388033**
Sample	**1:1 No. 1**	**1:1 No. 2**	**1:2 No. 1**	**1:2 No. 2**	**1:4 No. 1**	**1:4 No. 2**	**1:10 No. 1**	**1:10 No. 2**
Rock/ test solution Mass ratio (s/l ratio)	**1:1**	**1:1**	**1:2**	**1:2**	**1:4**	**1:4**	**1:10**	**1:10**
Nickel (Ni^{2+})	0.34	0.32	0.40	0.33	0.33	0.47	0.80	0.68
Barium (Ba^{2+})	1.88×10^{-4}	6.77×10^{-4}	3.15×10^{-4}	-1.22×10^{-4}	5.13×10^{-4}	6.84×10^{-4}	8.27×10^{-4}	-1.84×10^{-4}
Strontium (Sr^{2+})	3.21×10^{-3}	3.20×10^{-3}	3.63×10^{-3}	3.56×10^{-3}	3.43×10^{-3}	5.55×10^{-3}	7.00×10^{-3}	4.42×10^{-3}
Aluminum (Al^{3+})	1.28×10^{-3}	1.64×10^{-3}	1.13×10^{-3}	3.91×10^{-3}	1.61×10^{-2}	1.37×10^{-3}	-2.15×10^{-3}	-3.36×10^{-4}
Iron tot. (Fe_{tot})	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Iron-II (Fe^{2+})	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Iron-III (Fe^{3+})	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Sodium (Na^+)	0.48	0.47	0.27	0.17	0.15	0.48	0.66	0.17
Potassium (K^+)	0.02	0.01	0.03	0.01	0.06	0.08	-0.26	-0.19
Calcium (Ca^{2+})	0.25	0.33	0.71	0.53	0.11	0.43	-0.24	-0.28
Magnesium (Mg^{2+})	-9.03×10^{-4}	4.93×10^{-3}	1.41×10^{-2}	1.56×10^{-2}	1.95×10^{-2}	7.08×10^{-3}	4.16×10^{-2}	3.99×10^{-2}
Ammonium (NH_4^+)	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
$CEC_{cations}$ (all cation/sum)	0.75	0.81	1.03	0.74	0.36	1.00	0.21	-0.25
$CEC_{main\text{-}cations}$ (Na^+, K^+, Ca^{2+}, Mg^{2+})	0.75	0.81	1.02	0.73	0.34	0.99	0.20	-0.25



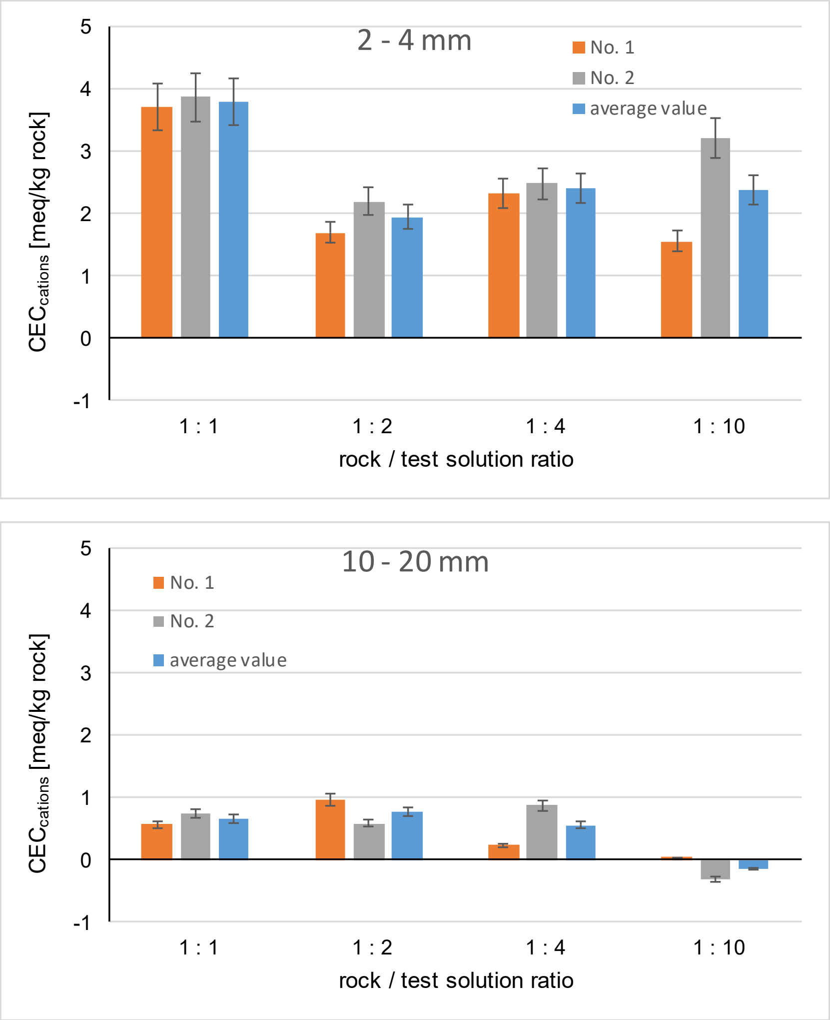


Figure 6: Results of the $CEC_{Cations}$ values determined for the different grain size fractions (0.065-0.2 mm, 2-4 mm, 10-20 mm) and rock material / test solution mass ratios based on calculation by the sum of concentrations of all the cations in the solution

Table 12: Calculated $CEC_{Cations}$ values, mean $CEC_{Cations}$ values and standard deviation from the CEC experiments with three grain size fraction 0.065-0.2 mm, 2-4 mm and 10-20 mm

Rock/test solution mass ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10
0.065-0.2 mm	$CEC_{Cations}$ (meq/kg rock)	7.24	9.36	10.73	10.91	11.83	12.07	13.79	12.99
	Mean $CEC_{Cations}$ (meq/kg rock)	8.30±1.50		10.82±0.13		11.95±0.17		13.39±0.57	
2-4 mm	$CEC_{Cations}$ (meq/kg rock)	3.43	3.81	2.49	2.99	3.67	3.61	2.73	4.31
	Mean $CEC_{Cations}$ (meq/kg rock)	3.62±0.27		2.74±0.36		3.64±0.04		3.52±1.12	
10-20 mm	$CEC_{Cations}$ (meq/kg rock)	0.75	0.81	1.03	0.74	0.36	1.00	0.21	-0.25
	Mean $CEC_{Cations}$ (meq/kg rock)	0.78±0.04		0.88±0.21		0.68±0.45		-0.02±0.32	

The CEC_{cation} values significantly increased during the experiment time of the largest grain size fraction (10-20 mm) (Appendix A, Tables A-3 to A-11).

The results of the experiment duplicates (No. 1 and No. 2) show that the CEC_{cation} values calculated by the sum of all cations are relatively comparable (Table 12).

The smallest grain size fraction (0.065-0.2 mm) results show that with the decrease of the solid to liquid ratio, the CEC_{cation} values decrease significantly from 100% by s/l ratio 1:10 with 13.39 meq/kg rock to 62 % by s/l ratio 1:1 with 8.30 meq/kg rock. Due to less available reactive surface area for cation adsorption in relation to the volume of solution, this trend can be observed. In contrast the CEC_{cation} values for the middle (2-4 mm) and largest grain size (10-20 mm) fractions determined at all s/l ratios are in a similar range.

As the grain size increases, the surface area of the rock samples decreases in relation to the solution. This is reflected in a decrease of the CEC_{cation} values with increasing grain size. At the largest grain size fraction, the difference of the determined CEC_{cation} values between the individual rock to test solution ratios is very small as shown by the s/l ratios 1:1, 1:2 and 1:4. As shown by the s/l ratio 1:10 the variations between the replicates were large and so the CEC values are shown also high variations, this is reasoned probably that the 10 g rock weight (in 100 g solution) corresponds to only a few rock grains and thus the reactive surface of the rock sample can differ significantly between the two replicates.

The results show that the CEC_{cation} values calculated by the sum of all the cations (measured by the s/l = 1:1, 1:2 and 1:4 experiments) (0.68-11.95 meq/kg rock) are 1.7 to 8.5 times (170-850%) higher than the CEC_{Ni} values (0.33-3.61 meq/kg rock) calculated from the nickel concentrations. The same counts for the grain size fraction 0.065-0.2 mm and 2-4 mm for the s/l ration 1:10 with 2.2 to 4.6 (220-460 %) times higher CEC_{cation} values (3.52-13.39 meq/kg rock) than the CEC_{Ni} values (-0.76-6.07 meq/kg rock). Only by the s/l ration 1:10 experiments of the largest grain size fraction (10-20 mm) the CEC_{cation} values are clearly lower than the CEC_{Ni} values.

Some elements (Ba, Sr, Ca, Mg) show slightly negative CEC values. This means that the initial concentrations are higher than the final concentrations after the experiments. This can be caused by an oversaturation of the individual elements in the experimental solution and a precipitation of those elements. The negative concentrations are taken into account for the sum of concentrations. This leads to a negative CEC_{cation} sum value for the s/l ratio 1:10 of the grain size fraction 10-20 mm. Due to the general low cation exchange capacity of the rock and the larger grain size fractions, especially, the effects of the cation decrease during the experiments is pronounced.

The experiment solutions at the end of the experiments did not show as high cation concentrations as that for the CEC measurements for the core sample IG_BH04_SA001 (also biotite granodiorite tonalite, APM-RP-01332-0355), because possible abrasion and erosion caused by end-over-end shaking of the previous experiments was avoided by normal daily shaking of the large grain size experiments and so an overestimation of the calculated CEC_{cation} values was avoided.

4.2.3 “Zero Exchange Experiment”

To evaluate the influence of possible disturbing effects, resulting in the overestimation of exchanged cations, aqueous extraction experiments, following the same procedures as the Ni-exchange experiments were conducted without addition of Ni-en.

The mineralization of a leach solution is the sum of: (i) the constituents originally dissolved in the porewater, (ii) the constituents present in fluid inclusions, and (iii) elements brought in by water-rock interactions during the leaching process. Thus, the aqueous leach solution represents a complex composition in rocks with abundant fluid inclusions, porewater components and reacting mineral phases.

Fluid inclusions, a high salt content in porewater and the presence of carbonates, such as calcite and dolomite (Ca^{2+} and Mg^{2+}) and other minerals, for example, feldspars (K^+, Na^+ and Ca^{2+}) in the rock material can induce an overestimated $CEC_{cations}$ value. Carbonates with concentrations > 1 wt.% are not expected in crystalline rocks from the Ignace site.

K^+ and Na^+ cations might originate from porewater in the Ni-en complex solution. Hence, the measured cation concentration has to be corrected for the porewater composition (Waber et al., 2003). Therefore, a blank experiment has to be realized parallel to the CEC experiments. Exchangeable cations that are not analyzed also may lead to an underestimated CEC value (e.g. NH_4^+, H^+) (Waber et al., 2003).

To evaluate these effects, a “zero exchange experiment” was conducted. The aim was to determine the basic dissolution of elements from the rock by rock-water interaction processes during the experiments and to use this as a basic value for the CEC calculation ($CEC_{cation\ corr.} = CEC_{cation} - CEC_{cation\ zero}$; $CEC_{cation\ zero}$ is the CEC measured by the “zero exchange experiment”, $CEC_{cation\ corr.}$ is the CEC after the correction). For this purpose, rock material was added to ultrapure water following the procedures for the normal CEC-experiment (same grain size fraction for a certain period of time: 0.065-0.2 mm for 48 h, 2-4 mm for 96 h, 10-20 mm for app. 185 d) for one rock material / test solution ratio (see Table 13). Therefore, the highest s/l ratio (1:1) was taken, since by the largest reactive surface area and thus the maximum cation exchange was to be expected with this ratio. The “zero exchange experiments” run parallel to the CEC experiments under the same conditions and time period. All “zero exchange experiments” were sampled and analyzed followed the same protocols as the CEC experiments.

Table 13: Overview of the experimental set-ups of the “zero exchange experiment”

Grain size fraction	Rock/test solution mass ratio	Reaction time	Mass (rock material)	Mass (solution)
			[g]	[g]
0.065-0.2 mm	1:1	48 h	40	40
0.065-0.2 mm Blank	-	48 h	0	75
2-4 mm	1:1	96 h	40	40
2-4 mm Blank	-	96 h	0	75
10-20 mm	1:1	185 days	60	60
10-20 mm Blank	-	185 days	0	75

The cation concentration and so also the $CEC_{cation\ zero}$ values slightly increased during the experiment time of the largest grain size fraction (10-20 mm) (Appendix A, Tables A-3 to A-11).

The sum of cation concentration of the “Zero Exchange Experiments” are 217 mg/l (0.065-0.2 mm), 221 mg/l (2-4 mm) and 29 mg/l (10-20 mm). In contrast at the normal experiments (1:1) the cation sum concentrations were 216 mg/l (0.065-0.2 mm), 100 mg/l (2-4 mm) and 56 mg/l (10-20 mm).

The results of the experiment duplicates (No. 1 and No. 2) show that the CEC$_{\text{cation zero}}$ values calculated by the sum of all cations are comparable (Table 14).

It can be seen that the highest CEC$_{\text{cation zero}}$ values were observed by the middle grain size fraction (2-4 mm).

Normally if the grain size fraction increases, the CEC$_{\text{cation zero}}$ values will decrease, since less reactive surface area of the rock sample is available for cation exchange in relation to the volume of solution. The CEC$_{\text{cation zero}}$ value determined by the largest grain size is clearly smaller, than that of the other two smaller grain size fractions.

The results show that the CEC$_{\text{cation zero}}$ values without any Ni-cation-exchange calculated by the sum of all the cations range from 0.35 to 11.06 meq/kg rock (Table 14).

For the grain size fractions 0.065 – 0.2 mm and 2-4 mm the corrected CEC$_{\text{cation}}$ values are negative, which means that the CEC$_{\text{cation,zero}}$ (zero exchange experiments) are higher than the CEC$_{\text{cation}}$ (Ni-en experiment) values. This is caused by the generally low cation exchange capacity of the rocks and the low concentrations of ions brought in solution. For the grain size fraction 0.065 – 0.2 mm both values are within the determined error ranges (Table 14). For the grain size fraction 2-4 mm the CEC$_{\text{cation,zero}}$ value is three times as high as the CEC$_{\text{cation}}$ value. This can be caused by heterogeneities and the influence of nickel in the CEC experiment.

The experiment solutions at the end of the experiments did not show as high cation concentrations as that for the CEC measurements for the core sample IG_BH04_SA001 (also biotite granodiorite tonalite, APM-RP-01332-0355), because possible abrasion and erosion caused by end-over-end shaking during the previous experiments was avoided by normal daily shaking of the large grain size fraction experiments and so an overestimation of the calculated CEC$_{\text{cation}}$ values was avoided.

Table 14: Calculated CEC$_{\text{cation zero}}$ values (mean CEC-cation values) based on all cation concentrations from the zero exchange experiments with the three grain size fractions 0.065-0.2 mm, 2-4 mm and 10-20 mm at the end of the experiments. The CEC$_{\text{cation}}$ values and the CEC$_{\text{cation coor.}}$ values after correction with the CEC$_{\text{cation zero}}$ values

		CEC$_{\text{cation zero}}$ (zero exchange experiment)		CEC$_{\text{cation}}$		CEC$_{\text{cation corr.}}$
Rock/test solution mass ratio		1:1	1:1	1:1	1:1	1:1
0.065-0.2 mm	(meq/kg rock)	8.95	8.68	7.24	9.36	-
	Mean (meq/kg rock)	8.81±0.19		8.30±1.50		-0.51±1.50
2-4 mm	(meq/kg rock)	11.06	10.20	3.43	3.81	-
	Mean (meq/kg rock)	10.63±0.61		3.62±0.27		-7.01±0.61
10-20 mm	(meq/kg rock)	0.36	0.35	0.75	0.81	-
	Mean (meq/kg rock)	0.36±0.01		0.78±0.04		0.43±0.04

4.2.4 Comparison of determined CEC results

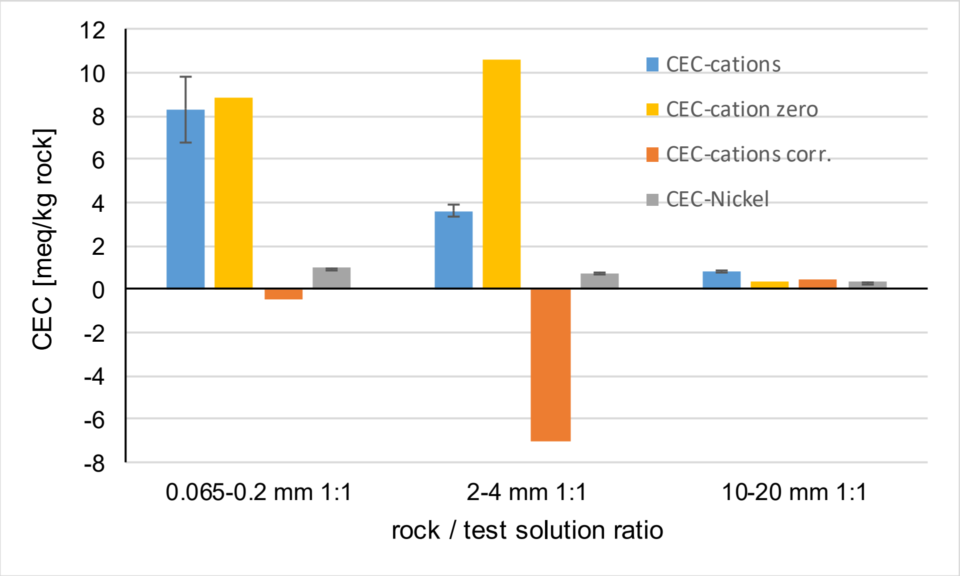


Figure 7: Results of the CEC values of the different grain size fractions (0.065-0.2 mm, 2-4 mm, 10-20 mm) and rock / test solution ratios calculated by the Ni-concentration (CEC_{Nickel}, grey bar), the sum of all cations in the solution (CEC_{cation}, blue bar) and corrected with the zero exchange experiment values ($CEC_{cation\ corr.}$; orange bar)

The comparison of the $CEC_{cations}$ and the $CEC_{cations\ zero}$ values (Figure 7) shows that the largest part of the cation concentration in the test solution is not by an exchange with nickel from the solution, but by the above-described processes (e.g. mineralization of leach solution, fluid conclusions, porewater; see chapter 4.2.3). Therefore, it is absolutely necessary to determine the $CEC_{cation\ zero}$ value and to correct the CEC_{cation} value with it.

It can be seen that the highest CEC_{Ni} and CEC_{cation} values were determined at a rock material to test solution ratio of 1:1. If the rock to test solution ratio decreases, the CEC_{Ni} and CEC_{cation} values also decrease significantly, since less reactive surface area of the rock sample is available in relation to the volume of solution for cation exchange. Also, as the grain size increases the surface area of the rock sample decreases in relation to the solution. This is also reflected in a decrease of the CEC_{Ni} and CEC_{cation} values with increasing grain size fraction. At the largest grain size fraction (10-20 mm), the difference of the CEC_{Ni} and CEC_{cation} values measured at the individual rock sample to test solution mass ratios is very small.

After the correction, the CEC_{cation} values are in a similar or even lower value as the CEC_{Ni} values (Figure 7). This could be caused by different rock-solution interactions or by an individual variation of the used rock particles (pore water, reactive surface) in the experiments.

5.0 SUMMARY

The investigations of the surface area by the BET method and the cation exchange capacity conducted on a crystalline core sample IG_BH06_SA001 (biotite granodiorite tonalite) from borehole IG_BH06 can be summarized as follows:

- In general, the specific surface area increases with decreasing particle size.
- The specific surface area of the grain size fraction < 0.065 mm is 2.49 ± 0.75 m^2/g.
- The specific surface area of the grain size fraction 0.065-0.2 mm is 3.76 ± 1.13 m^2/g.
- The specific surface area of the grain size fraction 0.15-0.3 mm is 0.73 ± 0.22 m^2/g.
- The specific surface area of the grain size fraction 1-2 mm is 0.10 ± 0.03 m^2/g.
- The specific surface area of the grain size fraction 2-4 mm is 0.04 ± 0.011 m^2/g.
- The specific surface area of the grain size fraction 10-20 mm is 0.03 ± 0.009 m^2/g.
- The specific surface area of the core slice (9 mm thickness and about 25-30 mm diameter) is 0.02 ± 0.008 m^2/g.
- Depending on the rock material / test solution mass ratio and the grain size fraction (reactive surface area), the CEC_{Ni} values vary from <0.01 to 6.19 meq/kg rock.
- The results also show that the CEC_{Ni} value increases as the volume of test solution increases in relation to the mass of rock (except exp. 2-4 mm 1:10).
- The CEC_{cation} values calculated from the sum of all cations (Na^+, K^+, Mg^{2+}, Ca^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe_{tot}) vary between -0.02 and 13.39 meq/kg rock depending on the rock material / test solution ratio and are of a factor 1.7 to 8.5 higher than the CEC_{Ni} values calculated from the nickel concentrations.
- CEC_{cation} values of the largest grain size fraction (10-20 mm) significantly increased over the experiment time.

It can be summarized that the measured CEC_{Ni} value depends on or is affected by various factors and processes, which include the particle size of rock sample (reactive surface), the rock sample / test solution ratio, the mineralogical composition of the sub-samples and the experimental setup (e.g., over head shaking caused abrasion).

On the other hand, the $CEC_{Cations}$ value also depends on or is affected by various factors and processes, which include the release of porewater, the opening of fluid inclusions, the particle size of rock sample (reactive surface), the rock sample / test solution ratio, the mineralogical composition of the sub-samples and the experimental setup (e.g. over head shaking caused abrasion).

Besides the calculation of the CEC value from the nickel concentration, the calculation from the sum of the cations ($CEC_{cations}$) is used to check the CEC_{Ni} values. Here it is important to note which cations (elements) are included in the calculation, as this can change the CEC value significantly.

It was also shown that the grain size fraction and consequently the reactive surface of rock sample as well as the rock sample / test solution ratio have a large influence on the measured CEC value.

Consequently, the CEC_{Ni} values should be used, but always taking into account the previously discussed factors and checking the CEC_{cation} values in order to know all possible influencing factors and to take them into account if relevant.

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APPENDIX A

Analytical Raw Data

CEC experiment 0.065-0.2 mm

Appendix Table A-1: Element concentrations of the final CEC experiment solutions with the smallest grain size fraction (0.065-0.2 mm) after 48 h. Sample name IG_BH06_SA001 0.065-0.2 mm.

Hydro Lab.-No.		382210	382212	382213	382214	382215	382216	382217	382218	382219	382220
Sample		Ni-en solution	1:1 No. 1	1:1 No. 2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock/ test solution Mass ratio		-	1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
Rock mass	g	-	39.999	39.997	19.995	19.996	10.002	9.999	10.002	10.001	0
Solution mass	g	-	38.849	39.239	39.123	39.191	39.270	39.414	99.021	99.446	73.807
pH value start		-	9.65	9.47	9.42	9.21	9.61	9.55	9.39	9.41	8.92
pH value end		-	10.09	10.06	10.06	10.06	9.96	9.95	9.73	9.73	8.91
Redox potential start	mV	-	-6.0	-10.8	7.4	-6.0	2.8	-3.4	30.5	14.0	29.7
Redox potential end	mV	-	-76.5	-75.5	-71.3	-80.9	-64.8	-83.8	-49.0	-41.1	-5.0
Nickel (Ni^{2+})	mg/l	27.0	0.257	0.400	0.730	0.762	2.73	2.79	11.3	12.1	29.7
Barium (Ba^{2+})	mg/l	0.055	0.027	0.128	0.078	0.092	0.009	0.028	0.003	0.003	0.069
Strontium (Sr^{2+})	mg/l	0.033	0.005	0.031	0.011	0.016	0.008	0.009	0.010	0.012	0.033
Aluminum (Al^{3+})	mg/l	< 0.02	2.75	26.5	11.0	10.6	1.05	2.46	0.257	0.316	< 0.02
Iron tot. (Fe_{tot})	mg/l	< 0.01	14.1	14.0	4.46	6.31	1.37	1.69	< 0.01	< 0.01	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	1.19	14.3	3.004	3.236	0.448	0.815	0.013	0.010	< 0.01
Iron-III (Fe^{3+})	mg/l	< 0.01	12.9	< 0.01	1.45	3.086	0.923	0.873	< 0.01	< 0.01	< 0.01
Sodium (Na^{+})	mg/l	< 0.2	145	145	96.8	98.4	62.9	61.2	29.2	28.7	1.71
Potassium (K^{+})	mg/l	< 0.1	35.9	38.3	27.8	28.6	22.5	22.7	14.5	14.8	2.23
Calcium (Ca^{2+})	mg/l	8.81	2.21	4.25	2.83	1.73	2.27	2.49	5.471	4.55	8.75
Magnesium (Mg^{2+})	mg/l	0.424	0.557	2.59	0.957	1.01	1.03	0.924	0.961	0.592	0.769
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.15	0.16	0.24	0.23	< 0.1
Hydrogen carbonate (HCO_3^{-})	mg/l	30.5	190	186	134	134	101	100	42.1	42.1	3.70
Chloride (Cl^{-})	mg/l	< 0.2	60.7	64.5	33.8	32.9	17.6	17.7	6.92	7.66	2.84
Sulfate (SO_4^{2-})	mg/l	< 0.5	13.3	13.3	6.47	6.61	3.65	3.52	1.41	1.47	< 0.5
Nitrate (NO_3^{-})	mg/l	68.1	71.0	73.6	73.5	74.6	74.2	73.7	75.7	75.2	< 0.2
Fluoride (F^{-})	mg/l	< 0.1	2.69	2.87	1.42	1.46	0.77	0.73	0.53	0.53	< 0.1

CEC experiment 2-4 mm

Appendix Table A-2: Element concentrations of the final CEC experiment solutions with the middle grain size fraction (2-4 mm) after 96 h. Sample name IG_BH06_SA001 2-4 mm.

Hydro Lab.-No.		380975	380977	380978	380979	380980	380981	380982	380983	380984	380985
Sample		Ni-en solution	1:1 No. 1	1:1 No. 2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock/ test solution Mass ratio		-	1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
Rock mass	g	-	39.999	39.999	20.004	20.008	10.003	9.993	9.998	9.997	0
Solution mass	g	-	39.679	39.924	39.628	40.241	39.858	39.945	99.185	99.617	74.423
pH value start		-	8.99	8.98	8.96	8.96	8.96	8.96	8.97	8.95	8.95
pH value end		-	9.73	9.73	9.66	9.64	9.46	9.47	9.22	9.22	9.73
Redox potential start	mV	-	48.7	48.8	49.6	50.4	59.1	49.3	62.7	59.8	67.9
Redox potential end	mV	-	-145.8	-151.0	-109.3	-93.4	-55.2	-56.6	-36.9	-35.6	-145.8
Nickel (Ni^{2+})	mg/l	25.8	5.06	3.87	9.22	9.13	21.5	22.7	26.5	31.4	26.7
Barium (Ba^{2+})	mg/l	0.059	0.125	0.119	0.016	0.032	0.018	0.019	0.016	0.105	0.059
Strontium (Sr^{2+})	mg/l	0.023	0.045	0.044	0.029	0.039	0.048	0.049	0.038	0.057	0.026
Aluminum (Al^{3+})	mg/l	< 0.02	16.2	16.5	0.717	2.85	1.29	1.26	0.370	2.20	< 0.02
Iron tot. (Fe_{tot})	mg/l	< 0.01	9.47	11.4	2.34	2.46	2.66	3.43	< 0.01	1.23	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	7.78	8.45	0.249	0.831	0.379	0.305	0.153	0.475	< 0.01
Iron-III (Fe^{3+})	mg/l	< 0.01	1.69	2.91	2.10	1.63	2.28	3.13	< 0.01	0.752	< 0.01
Sodium (Na^+)	mg/l	< 0.2	29.9	31.5	19.1	17.2	10.2	10.1	3.95	3.77	2.28
Potassium (K^+)	mg/l	< 0.1	27.1	28.4	20.9	19.7	12.1	13.0	4.22	4.49	1.25
Calcium (Ca^{2+})	mg/l	10.7	6.49	7.65	6.00	8.69	10.5	9.28	10.6	9.38	9.41
Magnesium (Mg^{2+})	mg/l	0.489	2.67	3.99	0.96	1.55	0.971	0.940	0.804	1.26	0.309
Ammonium (NH_4^+)	mg/l	< 0.1	0.15	0.19	0.14	0.13	< 0.1	< 0.1	< 0.1	< 0.1	0.11
Hydrogen carbonate (HCO_3^-)	mg/l	30.5	48.8	58.0	53.1	52.5	53.1	56.1	31.728	31.7	53.7
Chloride (Cl^-)	mg/l	0.10	13.8	15.0	8.25	8.44	5.86	5.85	2.16	2.27	1.82
Sulfate (SO_4^{2-})	mg/l	< 0.5	2.98	2.36	1.83	1.45	0.79	1.09	< 0.5	0.82	< 0.5
Nitrate (NO_3^-)	mg/l	68.2	69.1	71.5	74.7	73.1	72.5	74.0	75.5	74.1	73.3
Fluoride (F^-)	mg/l	< 0.1	0.69	0.8	0.37	0.38	0.16	0.17	< 0.1	< 0.1	< 0.1

CEC experiment 10-20 mm

Appendix Table A-3: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 8 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
380963	1:1 No. 1	35.9	4.51	1.14	8.77	< 0.01
380964	1:1. No. 2	35.4	2.81	1.76	9.88	< 0.01
380965	1:2 No. 1	30.0	1.73	1.25	11.8	< 0.01
380966	1:2 No.2	29.9	1.22	1.39	13.1	< 0.01
380967	1:4 No. 1	26.6	0.56	1.21	12.7	< 0.01
380968	1:4 No 2	25.9	1.60	0.86	10.6	0.52
380969	1:10 No. 1	31.5	0.94	0.47	9.14	< 0.01
380970	1:10 No. 2	31.9	0.44	0.65	9.84	1.00
380971	Blank	30.8	0.12	0.88	11.2	< 0.01
380972	Zero 1:1 No. 1	< 0.001	5.82	0.76	13.6	< 0.01
380973	Zero 1:1. No. 2	< 0.001	6.28	0.46	29.9	< 0.01
380974	Zero-Blank	< 0.001	< 0.2	1.18	< 0.05	< 0.01

Appendix Table A-4: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 16 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
381648	1:1 No. 1	30.7	6.78	0.76	15.7	< 0.01
381649	1:1. No. 2	30.6	7.10	0.42	16.1	< 0.01
381650	1:2 No. 1	33.1	2.01	1.29	13.4	< 0.01
381651	1:2 No.2	32.9	1.43	1.51	11.8	1.19
381652	1:4 No. 1	33.7	0.59	1.34	11.0	< 0.01
381653	1:4 No 2	34.3	1.87	0.83	11.0	< 0.01
381654	1:10 No. 1	35.4	1.21	0.41	8.03	7.51
381655	1:10 No. 2	34.1	0.52	0.69	9.86	0.85
381656	Blank	26.0	0.30	0.97	9.31	1.19
381657	Zero 1:1 No. 1	< 0.001	5.09	0.91	7.79	< 0.01
381658	Zero 1:1. No. 2	< 0.001	3.40	1.76	< 0.05	0.85
381659	Zero-Blank	< 0.001	0.22	1.33	< 0.05	0.73

Appendix Table A-5: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 40 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
382236	1:1 No. 1	27.2	7.97	0.81	16.3	0.55
382237	1:1. No. 2	28.9	8.08	0.47	15.1	< 0.01
382238	1:2 No. 1	31.7	2.20	1.23	15.3	< 0.01
382239	1:2 No.2	33.0	1.64	1.47	15.4	< 0.01
382240	1:4 No. 1	26.9	0.59	1.28	12.0	< 0.01
382241	1:4 No 2	34.0	1.97	0.83	12.8	< 0.01
382242	1:10 No. 1	35.	1.29	0.40	14.2	0.70
382243	1:10 No. 2	37.5	0.58	0.74	11.8	0.92
382244	Blank	32.3	< 0.2	0.99	12.1	1.16
382245	Zero 1:1 No. 1	< 0.001	5.76	0.98	8.14	< 0.01
382246	Zero 1:1. No. 2	< 0.001	3.71	1.74	8.45	0.57
382247	Zero-Blank	< 0.001	< 0.2	1.41	< 0.05	0.90

Appendix Table A-6: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 56 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
382952	1:1 No. 1	17.6	8.81	1.23	19.2	< 0.01
382953	1:1. No. 2	22.6	9.32	0.93	19.4	< 0.01
382954	1:2 No. 1	29.7	2.65	1.55	15.1	< 0.01
382955	1:2 No.2	32.9	1.69	1.70	18.0	< 0.01
382956	1:4 No. 1	32.1	0.63	2.09	12.4	1.95
382957	1:4 No 2	34.8	2.17	0.99	14.2	< 0.01
382958	1:10 No. 1	30.5	1.43	0.44	11.3	1.12
382959	1:10 No. 2	32.3	0.61	0.78	11.3	< 0.01
382960	Blank	36.4	< 0.2	1.04	13.3	< 0.01
382961	Zero 1:1 No. 1	< 0.001	6.07	1.06	12.8	< 0.01
382962	Zero 1:1. No. 2	< 0.001	4.02	2.11	12.5	< 0.01
382963	Zero-Blank	10.3	< 0.2	1.49	< 0.05	< 0.01

Appendix Table A-7: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 82 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
383759	1:1 No. 1	21.7	9.96	1.02	14.1	< 0.01
383760	1:1. No. 2	32.4	10.1	0.88	15.9	< 0.01
383761	1:2 No. 1	30.3	2.77	1.70	11.7	< 0.01
383762	1:2 No.2	34.3	1.97	3.81	15.4	< 0.01
383763	1:4 No. 1	21.7	0.88	1.45	8.69	< 0.01
383764	1:4 No 2	23.9	2.85	0.89	12.0	< 0.01
383765	1:10 No. 1	18.8	3.05	1.80	10.3	< 0.01
383766	1:10 No. 2	34.7	0.73	0.69	10.2	< 0.01
383767	Blank	29.7	0.23	1.05	9.99	< 0.01
383768	Zero 1:1 No. 1	< 0.001	7.36	1.41	5.99	< 0.01
383769	Zero 1:1. No. 2	< 0.001	5.08	2.19	8.43	< 0.01
383770	Zero-Blank	< 0.001	< 0.2	0.14	5.63	< 0.01

Appendix Table A-8: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 101 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
384330	1:1 No. 1	25.2	10.3	1.15	13.9	1.52
384331	1:1. No. 2	21.2	10.3	0.68	16.3	< 0.01
384332	1:2 No. 1	23.2	3.32	1.58	14.8	< 0.01
384333	1:2 No.2	28.1	2.13	1.81	15.5	< 0.01
384334	1:4 No. 1	30.3	0.75	1.69	10.8	< 0.01
384335	1:4 No 2	34.7	2.71	0.92	10.1	< 0.01
384336	1:10 No. 1	38.5	1.83	0.39	7.01	< 0.01
384337	1:10 No. 2	28.8	0.95	0.83	7.75	< 0.01
384338	Blank	28.6	0.30	0.99	8.71	< 0.01
384339	Zero 1:1 No. 1	< 0.001	-	1.23	8.02	< 0.01
384340	Zero 1:1. No. 2	< 0.001	4.66	1.96	8.49	< 0.01
384341	Zero-Blank	< 0.001	< 0.2	1.46	< 0.05	< 0.01

Appendix Table A-9: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 157 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
387006	1:1 No. 1	22.6	11.5	1.24	20.6	< 0.01
387007	1:1. No. 2	24.2	11.3	0.71	21.8	< 0.01
387008	1:2 No. 1	29.0	2.83	1.24	18.1	< 0.01
387009	1:2 No.2	26.8	1.78	1.62	20.2	< 0.01
387010	1:4 No. 1	32.6	0.74	1.30	17.5	< 0.01
387011	1:4 No 2	37.0	2.70	0.76	19.8	< 0.01
387012	1:10 No. 1	29.8	1.83	0.36	16.7	< 0.01
387013	1:10 No. 2	29.5	0.80	0.88	15.8	< 0.01
387014	Blank	34.9	< 0.2	0.95	15.7	< 0.01
387015	Zero 1:1 No. 1	0.163	7.69	1.27	14.4	< 0.01
387016	Zero 1:1. No. 2	< 0.001	4.93	1.87	17.1	< 0.01
387017	Zero-Blank	< 0.001	< 0.2	1.47	< 0.05	< 0.01

Appendix Table A-10: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 171 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
387325	1:1 No. 1	18.4	11.9	0.92	14.9	< 0.01
387326	1:1. No. 2	18.8	11.8	0.66	17.0	< 0.01
387327	1:2 No. 1	24.1	2.86	1.17	15.0	< 0.01
387328	1:2 No.2	25.0	1.84	2.37	13.6	< 0.01
387329	1:4 No. 1	27.8	0.71	1.31	13.2	< 0.01
387330	1:4 No 2	25.4	2.68	0.72	12.2	< 0.01
387331	1:10 No. 1	28.2	1.87	0.36	10.1	< 0.01
387332	1:10 No. 2	25.9	0.72	0.59	9.60	< 0.01
387333	Blank	28.7	< 0.2	0.84	9.79	< 0.01
387334	Zero 1:1 No. 1	< 0.001	7.56	1.08	9.68	< 0.01
387335	Zero 1:1. No. 2	< 0.001	5.05	1.85	10.8	< 0.01
387336	Zero-Blank	< 0.001	< 0.2	1.37	< 0.05	< 0.01

Appendix Table A-11: Element concentrations of the final CEC experiment solution with the largest grain size fraction (10-20 mm) after 185 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.		380687	388026	388027	388028	388029	388030	388031	388032	388033	388034
Sample		**Ni-en Solution**	**1:1 No. 1**	**1:1 No. 2**	**1:2 No. 1**	**1:2 No. 2**	**1:4 No. 1**	**1:4 No. 2**	**1:10 No. 1**	**1:10 No. 2**	**Blank**
Rock/ test solution Mass ratio		**-**	**1:1**	**1:1**	**1:2**	**1:2**	**1:4**	**1:4**	**1:10**	**1:10**	**-**
Rock mass	g	-	60.032	60.072	29.894	30.006	15.060	15.080	9.978	10.044	0
Solution mass	g	-	59.211	60.097	59.731	59.509	59.660	59.688	100.025	99.130	74.896
pH value start		-	9.02	9.01	9.02	9.02	9.01	9.01	9.00	9.00	8.98
pH value end		-	8.89	8.94	8.51	8.64	8.83	8.60	8.83	8.77	8.90
Redox potential start	mV	-	42.8	39.2	46.9	44.8	48.6	50.1	42.2	47.8	68.1
Redox potential end	mV	-	29.4	27.8	50.8	38.4	44.3	53.1	49.5	44.0	72.1
Nickel (Ni^{2+})	mg/l	25.8	19.0	19.9	23.8	25.0	27.8	26.5	28.1	28.4	30.6
Barium (Ba^{2+})	mg/l	0.059	0.042	0.081	0.040	0.022	0.037	0.041	0.033	0.027	0.027
Strontium (Sr^{2+})	mg/l	0.023	0.210	0.207	0.137	0.136	0.089	0.116	0.078	0.066	0.045
Aluminum (Al^{3+})	mg/l	< 0.02	0.037	0.043	0.026	0.048	0.080	0.023	0.014	0.017	0.017
Iron tot. (Fe_{tot})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Iron-III (Fe^{3+})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Sodium (Na^{+})	mg/l	< 0.2	14.5	13.9	5.15	3.76	2.52	4.73	3.18	1.97	1.53
Potassium (K^{+})	mg/l	< 0.1	2.63	2.35	2.56	2.25	2.59	2.87	0.81	1.12	1.92
Calcium (Ca^{2+})	mg/l	10.7	18.1	19.7	20.4	18.4	12.8	14.6	11.6	11.5	12.1
Magnesium (Mg^{2+})	mg/l	0.489	0.404	0.486	0.516	0.527	0.486	0.442	0.472	0.470	0.417
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Hydrogen carbonate (HCO_3^{-})	mg/l	31.1	34.8	33.6	41.5	34.2	29.3	37.8	30.5	29.9	27.5
Chloride (Cl^{-})	mg/l	0.3	18.6	17.7	8.32	9.98	5.27	7.56	2.81	2.73	2.26
Sulfate (SO_4^{2-})	mg/l	< 0.5	2.70	3.34	1.16	1.00	0.69	0.81	0.51	< 0.5	< 0.5
Nitrate (NO_3^{-})	mg/l	61.6	75.6	77.4	79.6	81.2	79.4	52.7	76.5	77.8	79.6
Fluoride (F^{-})	mg/l	< 0.1	0.31	0.26	0.36	0.27	< 0.1	0.37	< 0.1	< 0.1	< 0.1

CEC zero experiment 0.065-0.2 mm

Appendix Table A-12: Element concentrations of the final CEC zero experiment solutions with the smallest grain size fraction (0.065-0.2 mm) after 48 . Sample name IG_BH06_SA001 0.065-0.2 mm.

Hydro Lab.-No.		382211	382221	382222	382223
Sample		Zero Start Solution	Zero 1:1 No. 1	Zero 1:1 No. 2	Zero Blank
Rock/ test solution Mass ratio		-	1:1	1:1	-
Rock mass	G	-	40	40.005	0
Solution mass	G	-	39.551	39.514	73.468
pH value start		-	10.27	10.18	7.45
pH value end		-	10.17	10.14	8.00
Redox potential start	mV	-	-3.7	30.2	56.3
Redox potential end	mV	-	-22.8	-120.8	9.4
Nickel (Ni^{2+})	mg/l	< 0.0001	0.003	< 0.001	< 0.001
Barium (Ba^{2+})	mg/l	< 0.001	0.057	0.122	0.0018
Strontium (Sr^{2+})	mg/l	< 0.002	0.017	0.006	< 0.002
Aluminum (Al^{3+})	mg/l	< 0.02	19.3	12.4	< 0.02
Iron tot. (Fe_{tot})	mg/l	< 0.01	27.3	29.9	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	4.70	2.92	< 0.01
Iron-III (Fe^{3+})	mg/l	< 0.01	22.6	27.0	< 0.01
Sodium (Na^{+})	mg/l	< 0.2	134	136	1.58
Potassium (K^{+})	mg/l	< 0.1	32.3	33.4	2.04
Calcium (Ca^{2+})	mg/l	< 0.05	3.00	2.55	0.079
Magnesium (Mg^{2+})	mg/l	0.014	1.30	1.79	< 0.01
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1
Hydrogen carbonate (HCO_3^{-})	mg/l	2.38	204	211	64.7
Chloride (Cl^{-})	mg/l	< 0.2	62.3	68.9	2.52
Sulfate (SO_4^{2-})	mg/l	< 0.5	7.89	13.2	< 0.5
Nitrate (NO_3^{-})	mg/l	< 0.2	0.22	< 0.2	69.2
Fluoride (F^{-})	mg/l	< 0.1	1.90	4.91	< 0.1

CEC zero experiment 2-4 mm

Appendix Table A-13: Element concentrations of the final CEC zero experiment solutions with the middle grain size fraction (2-4 mm) after 96 h. Sample name IG_BH06_SA001 2-4 mm.

Hydro Lab.-No.		380976	380986	380987	380988
Sample		Zero Start Solution	Zero 1:1 No. 1	Zero 1:1 No. 2	Zero Blank
Rock/ test solution Mass ratio		-	1:1	1:1	-
Rock mass	G	-	40.011	39.998	0
Solution mass	G	-	39.468	39.442	74.146
pH value start		-	8.98	8.92	7.79
pH value end		-	9.95	10.03	8.05
Redox potential start	mV	-	103.4	104.8	67.3
Redox potential end	mV	-	-53.4	-55.7	-23.1
Nickel (Ni^{2+})	mg/l	< 0.0001	< 0.001	< 0.001	< 0.001
Barium (Ba^{2+})	mg/l	< 0.001	0.280	0.262	< 0.01
Strontium (Sr^{2+})	mg/l	< 0.002	0.103	0.071	< 0.002
Aluminum (Al^{3+})	mg/l	< 0.02	93.5	65.2	< 0.02
Iron tot. (Fe_{tot})	mg/l	< 0.01	50.0	78.4	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	44.7	36.6	0.010
Iron-III (Fe^{3+})	mg/l	< 0.01	5.31	41.8	< 0.01
Sodium (Na^{+})	mg/l	< 0.2	31.0	33.8	2.28
Potassium (K^{+})	mg/l	< 0.1	19.3	29.5	1.24
Calcium (Ca^{2+})	mg/l	1.46	13.4	10.3	< 0.05
Magnesium (Mg^{2+})	mg/l	< 0.01	9.46	7.40	0.018
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1
Hydrogen carbonate (HCO_3^{-})	mg/l	2.44	85.4	84.2	24.4
Chloride (Cl^{-})	mg/l	< 0.2	16.2	24.2	2.08
Sulfate (SO_4^{2-})	mg/l	< 0.5	2.36	2.71	< 0.5
Nitrate (NO_3^{-})	mg/l	< 0.2	< 0.2	< 0.2	< 0.2
Fluoride (F^{-})	mg/l	< 0.1	0.70	0.70	< 0.1

CEC zero experiment 2-4 mm

Appendix Table A-14: Element concentrations of the final CEC zero experiment solutions with the largest grain size fraction (10-20 mm) after 185 days. Sample name IG_BH06_SA001 10-20 mm.

Hydro Lab.-No.		380688	388035	388036	388037
Sample		Zero Start Solution	Zero 1:1 No. 1	Zero 1:1 No. 2	Zero Blank
Rock/ test solution Mass ratio		-	1:1	1:1	-
Rock mass	g	-	60.013	59.963	0
Solution mass	g	-	59.284	59.51	73.832
pH value start		-	6.98	6.96	6.39
pH value end		-	8.59	8.56	8.23
Redox potential start	mV	-	156.8	153.8	183.1
Redox potential end	mV	-	50.8	66.7	79.9
Nickel (Ni^{2+})	mg/l	< 0.0001	< 0.001	< 0.001	0.0012
Barium (Ba^{2+})	mg/l	< 0.001	< 0.01	0.010	< 0.01
Strontium (Sr^{2+})	mg/l	< 0.002	0.097	0.114	< 0.002
Aluminum (Al^{3+})	mg/l	< 0.02	0.311	0.273	0.007
Iron tot. (Fe_{tot})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01
Iron-II (Fe^{2+})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01
Iron-III (Fe^{3+})	mg/l	< 0.01	< 0.01	< 0.01	< 0.01
Sodium (Na^{+})	mg/l	< 0.2	9.56	7.11	1.4
Potassium (K^{+})	mg/l	< 0.1	4.10	7.72	2.58
Calcium (Ca^{2+})	mg/l	0.076	12.2	15.4	0.096
Magnesium (Mg^{2+})	mg/l	< 0.01	0.115	0.124	0.011
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1
Hydrogen carbonate (HCO_3^{-})	mg/l	2.56	29.9	28.7	1.83
Chloride (Cl^{-})	mg/l	< 0.2	17.7	21.5	2.86
Sulfate (SO_4^{2-})	mg/l	< 0.5	2.40	2.31	< 0.5
Nitrate (NO_3^{-})	mg/l	< 0.2	< 0.2	< 0.2	< 0.2
Fluoride (F^{-})	mg/l	< 0.1	1.25	0.86	< 0.1

APPENDIX B

Photo Documentation

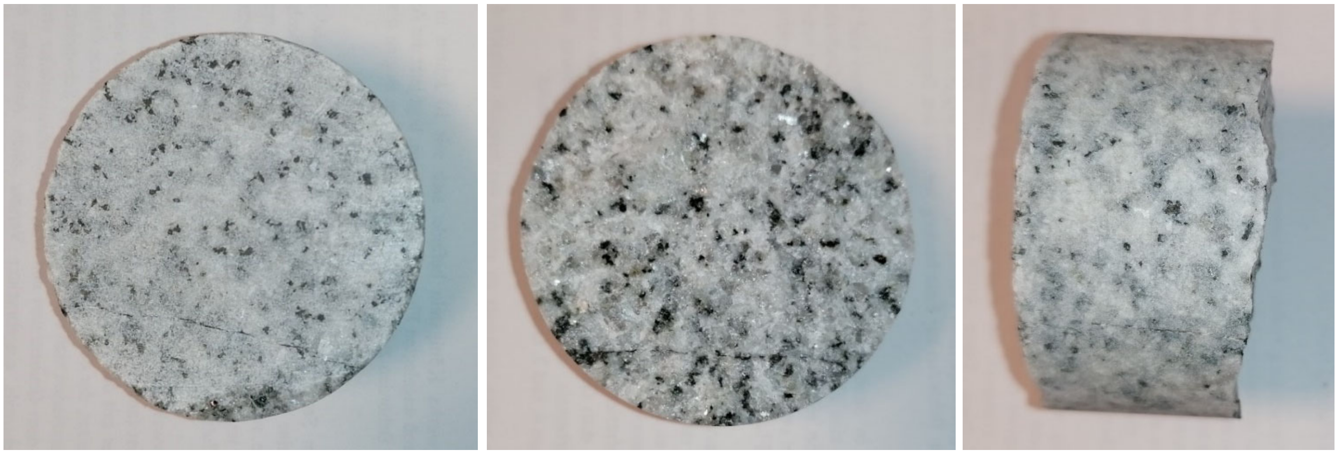
Photos of the core

Golder Project Number	20253946	Project Title	Phase 2 Initial Borehole Drilling and Testing Ignace Area (IG_BH06)
Photo ID	IG_BH06 SA001_B	Date (yy/mm/dd)	21/10/27
Core Condition	WET	Depth Interval	822.43 m - 822.89 m
Sample ID	IG_BH06 SA001		

Golder Project Number	20253946	Project Title	Phase 2 Initial Borehole Drilling and Testing Ignace Area (IG_BH06)
Photo ID	IG_BH06 SA001_P	Date (yy/mm/dd)	21/10/27
Core Condition	WET	Depth Interval	822.43 m - 822.89 m
Sample ID	IG_BH06 SA001		

Golder Project Number	20253946	Project Title	Phase 2 Initial Borehole Drilling and Testing Ignace Area (IG_BH06)
Photo ID	IG_BH06 SA001_F	Date (yy/mm/dd)	21/10/27
Core Condition	WET	Depth Interval	822.43 m - 822.89 m
Sample ID	IG_BH06 SA001		

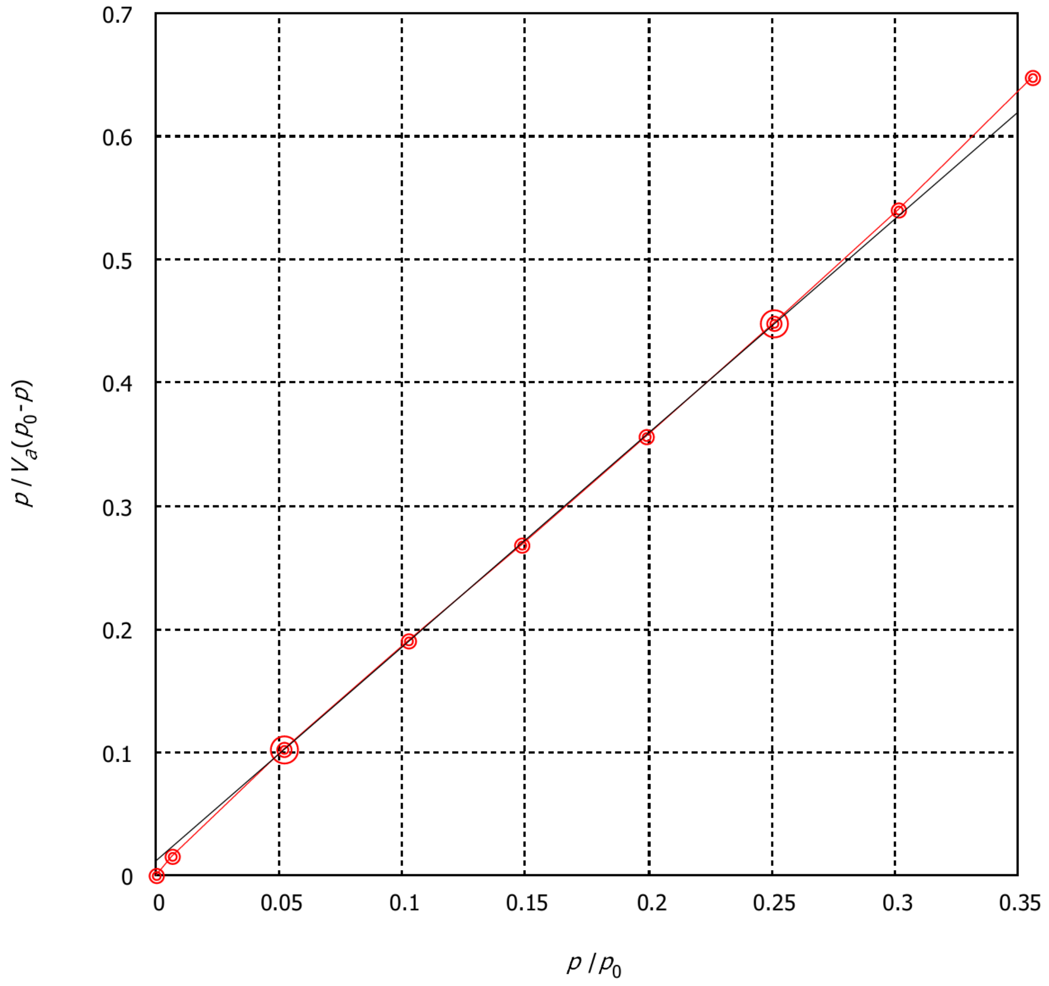
Photos of a core slice



APPENDIX C

Lab Report Uni Bern

Core size <0.065 mm



BET-Plot

Adsorptive N2

Adsorption temperature 77.350 [K]

◎ 65mu-Port1.DAT

, Vacuum degree before measurement:0.000E+0Pa

Information of Instrument	(S/N: 33,)		
Date of measurement	09/03/2022	Sample weight	3.6981 [g]
Time of measurement	2:01:19	Saturated vapor pressure	98.169 [kPa]

V_m	0.5724	[cm^3(STP) g^{-1}]	$a_{s,BET}$	2.4914E+00	[m^2 g^{-1}]
C	146.83		Total pore volume (p/ $_0$=0.356)	1.3192E-03	[cm^3 g^{-1}]
			Mean pore diameter	2.1181	[nm]

BET-Plot

(1/1)

Filename	65mu-Port1.DAT
COMMENT	, Vacuum degree before measurement:0.000E+0Pa

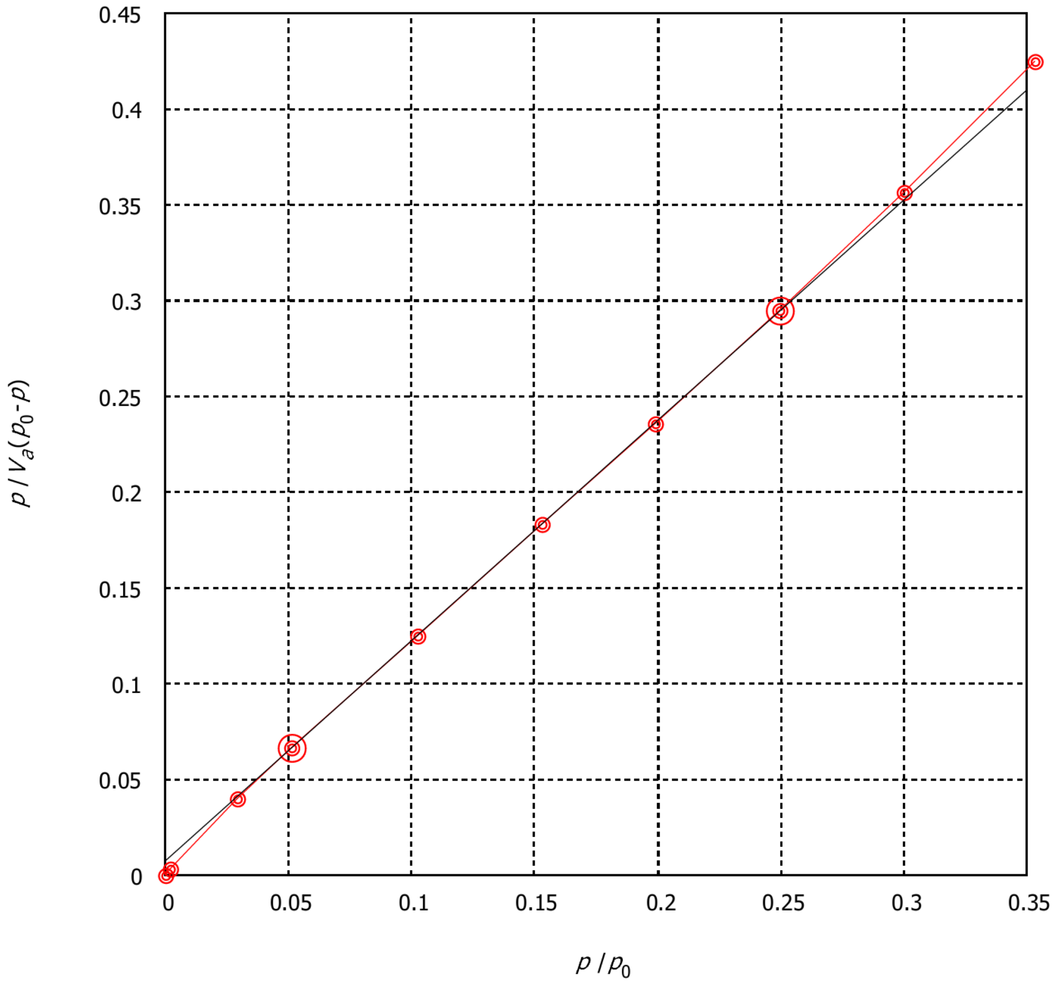
S/N	33	Vs_0	12.386
Date of measurement	09/03/2022	Time of measurement	2:01:19
Adsorptive	N2	Adsorption temperature	77.350 [K]
Sample weight	3.6981 [g]	Adsorbate molecules diameter	0.3540 [nm]
Saturated vapor pressure	98.169 [kPa]	Calculate mean pore diameter.	Do
Calculate mean particle size.	Do	Relative pressure for pore volume calculation	0.9900
Adsorptive molecular weight	28.013	Density	0.8080 [g cm $^{-3}$]
Adsorptive surface area	0.1620 [nm ^{2}]	Analytic method	Use pressure range setting.

S.P.	2	
E.P.	6	
Slope(Linear)	1.7350	
Intercept(Linear)	1.1897E-02	
Correlation coefficient	1.0000	
V_m	0.5724	[cm^3(STP) g^{-1}]
$a_{s,BET}$	2.4914E+00	[m^2 g^{-1}]
C	146.83	
Total pore volume (p/p_0=0.356)	1.3192E-03	[cm^3 g^{-1}]
Mean pore diameter	2.1181	[nm]

[Adsorption branch] Number of data point : 9

No	p/p_0	$p/V_a(p_0-p)$
0	0.0000	0.0000
1	6.1912E-03	1.5721E-02
2	5.2005E-02	0.1027
3	0.1027	0.1903
4	0.1485	0.2687
5	0.1992	0.3562
6	0.2508	0.4483
7	0.3012	0.5410
8	0.3561	0.6484

Core size 0.065-0.2 mm



BET-Plot

Adsorptive N2

Adsorption temperature 77.350 [K]

◎ 65-200mu-Port2.DAT

, Vacuum degree before measurement:0.000E+0Pa

Information of Instrument	(S/N: 33,)		
Date of measurement	09/03/2022	Sample weight	3.0815 [g]
Time of measurement	2:14:32	Saturated vapor pressure	98.123 [kPa]

V_m	0.8638	[cm^3(STP) g^{-1}]	$a_{s,BET}$	3.7597E+00	[m^2 g^{-1}]
C	154.46		Total pore volume ($p/_0$=0.353)	1.9867E-03	[cm^3 g^{-1}]
			Mean pore diameter	2.1137	[nm]

BET-Plot

(1/1)

Filename	65-200mu-Port2.DAT
COMMENT	, Vacuum degree before measurement:0.000E+0Pa

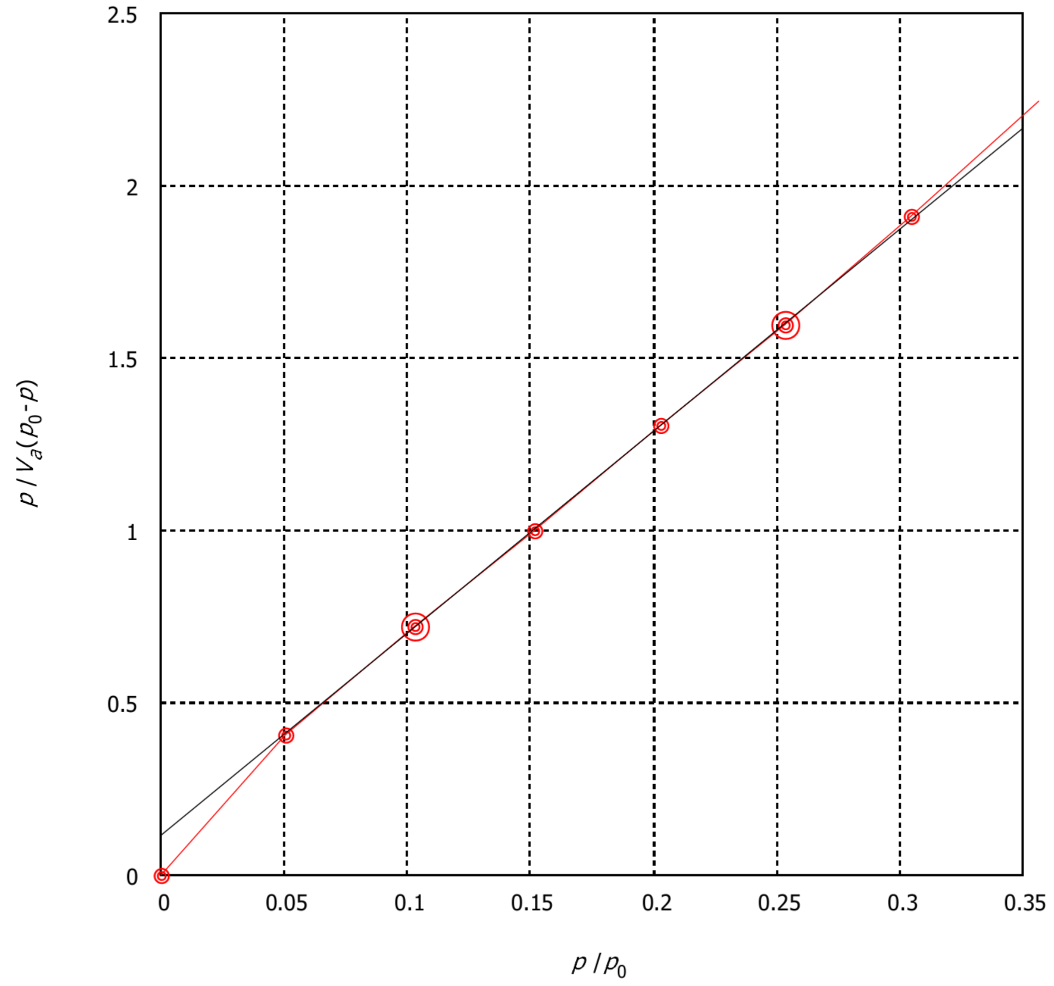
S/N	33	Vs_0	12.386
Date of measurement	09/03/2022	Time of measurement	2:14:32
Adsorptive	N2	Adsorption temperature	77.350 [K]
Sample weight	3.0815 [g]	Adsorbate molecules diameter	0.3540 [nm]
Saturated vapor pressure	98.123 [kPa]	Calculate mean pore diameter.	Do
Calculate mean particle size.	Do	Relative pressure for pore volume calculation	0.9900
Adsorptive molecular weight	28.013	Density	0.8080 [g cm $^{-3}$]
Adsorptive surface area	0.1620 [nm ^{2}]	Analytic method	Use pressure range setting.

S.P.	3	
E.P.	7	
Slope(Linear)	1.1501	
Intercept(Linear)	7.4948E-03	
Correlation coefficient	1.0000	
V_m	0.8638	[cm^3(STP) g^{-1}]
$a_{s,BET}$	3.7597E+00	[m^2 g^{-1}]
C	154.46	
Total pore volume (p/p_0=0.353)	1.9867E-03	[cm^3 g^{-1}]
Mean pore diameter	2.1137	[nm]

[Adsorption branch] Number of data point : 10

No	p/p_0	$p/V_a(p_0-p)$
0	0.0000	0.0000
1	1.7220E-03	3.3621E-03
2	2.9011E-02	3.9933E-02
3	5.1399E-02	6.6811E-02
4	0.1021	0.1252
5	0.1532	0.1832
6	0.1989	0.2357
7	0.2494	0.2949
8	0.3000	0.3565
9	0.3533	0.4253

Core size 0.15-0.3 mm



BET-Plot

Adsorptive N2

Adsorption temperature 77.350 [K]

◎ IG BH-06 0.15-0.3-Port1.DAT

, Vacuum degree before measurement:0.000E+0Pa

Information of Instrument	(S/N: 33,)		
Date of measurement	2/1/2023	Sample weight	5.9358 [g]
Time of measurement	1:39:26	Saturated vapor pressure	97.228 [kPa]

V_m	0.1675	[cm^3(STP) g^{-1}]	$a_{s,BET}$	7.2904E-01	[m^2 g^{-1}]
C	51.146		Total pore volume ($p/\ _0$=0.363)	3.8535E-04	[cm^3 g^{-1}]
			Mean pore diameter	2.1143	[nm]

BET-Plot

(1/1)

Filename	IG BH-06 0.15-0.3-Port1.DAT
COMMENT	, Vacuum degree before measurement:0.000E+0Pa

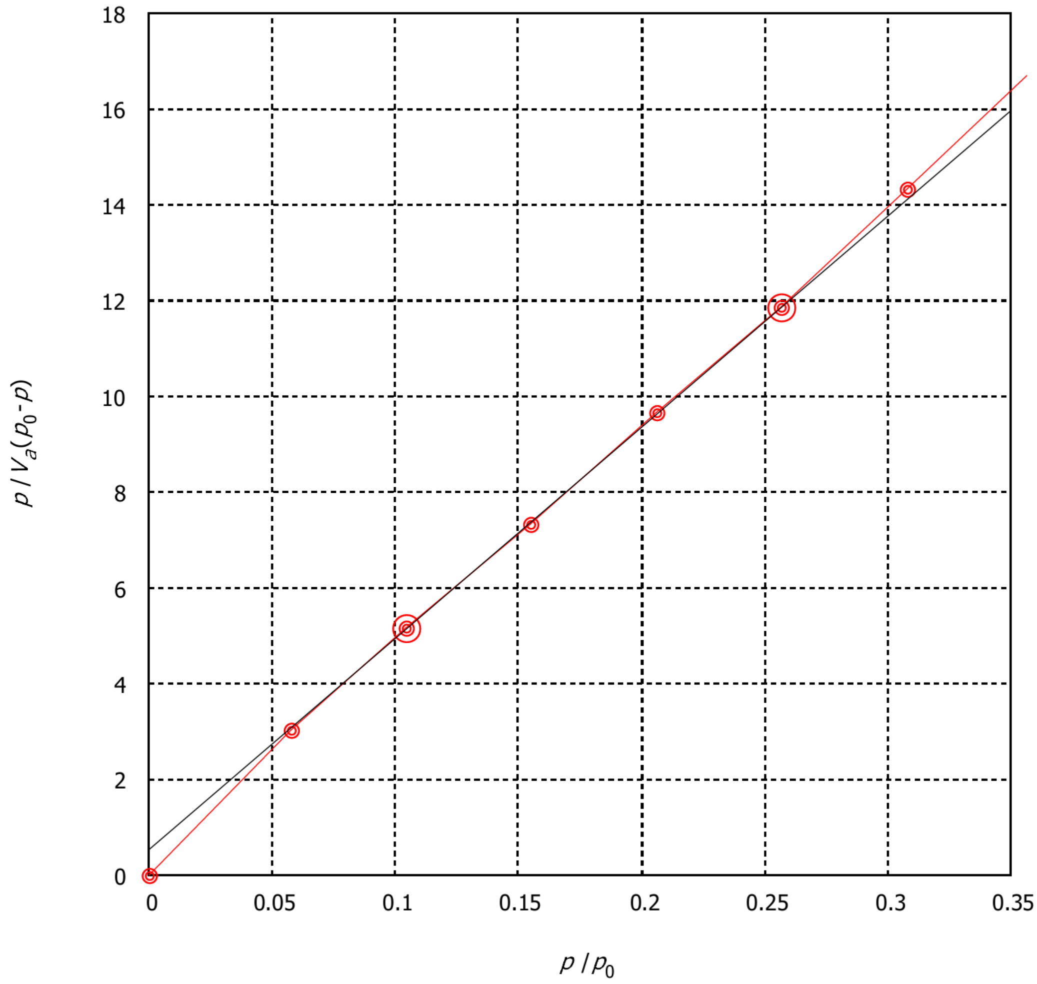
S/N	33	Vs_0	12.386
Date of measurement	2/1/2023	Time of measurement	1:39:26
Adsorptive	N2	Adsorption temperature	77.350 [K]
Sample weight	5.9358 [g]	Adsorbate molecules diameter	0.3540 [nm]
Saturated vapor pressure	97.228 [kPa]	Calculate mean pore diameter.	Do
Calculate mean particle size.	Do	Relative pressure for pore volume calculation	0.9900
Adsorptive molecular weight	28.013	Density	0.8080 [g cm $^{-3}$]
Adsorptive surface area	0.1620 [nm ^{2}]	Analytic method	Use pressure range setting.
Pressure range	0.1000-0.2500		

S.P.	2	
E.P.	5	
Slope(Linear)	5.8518	
Intercept(Linear)	0.1167	
Correlation coefficient	1.0000	
V_m	0.1675	[cm^3(STP) g^{-1}]
$a_{s,BET}$	7.2904E-01	[m^2 g^{-1}]
C	51.146	
Total pore volume (p/p_0=0.363)	3.8535E-04	[cm^3 g^{-1}]
Mean pore diameter	2.1143	[nm]

[Adsorption branch] Number of data point : 8

No	p/p_0	$p/V_a(p_0-p)$
0	0.0000	0.0000
1	5.0790E-02	0.4061
2	0.1033	0.7219
3	0.1515	1.0022
4	0.2028	1.3042
5	0.2533	1.5990
6	0.3048	1.9133
7	0.3629	2.2861

Core size 1-2 mm



BET-Plot

Adsorptive N2

Adsorption temperature 77.350 [K]

◎ IG BH-06 1-2-Port3.DAT

, Vacuum degree before measurement:0.000E+0Pa

Information of Instrument	(S/N: 33,)		
Date of measurement	2/3/2023	Sample weight	6.5592 [g]
Time of measurement	1:35:20	Saturated vapor pressure	98.801 [kPa]

V_m	2.2421E-02	[cm^3(STP) g^{-1}]	$a_{s,BET}$	9.7587E-02	[m^2 g^{-1}]
C	81.498		Total pore volume ($p/_0$=0.367)	5.2077E-05	[cm^3 g^{-1}]
			Mean pore diameter	2.1346	[nm]

BET-Plot

(1/1)

Filename	IG BH-06 1-2-Port3.DAT
COMMENT	, Vacuum degree before measurement:0.000E+0Pa

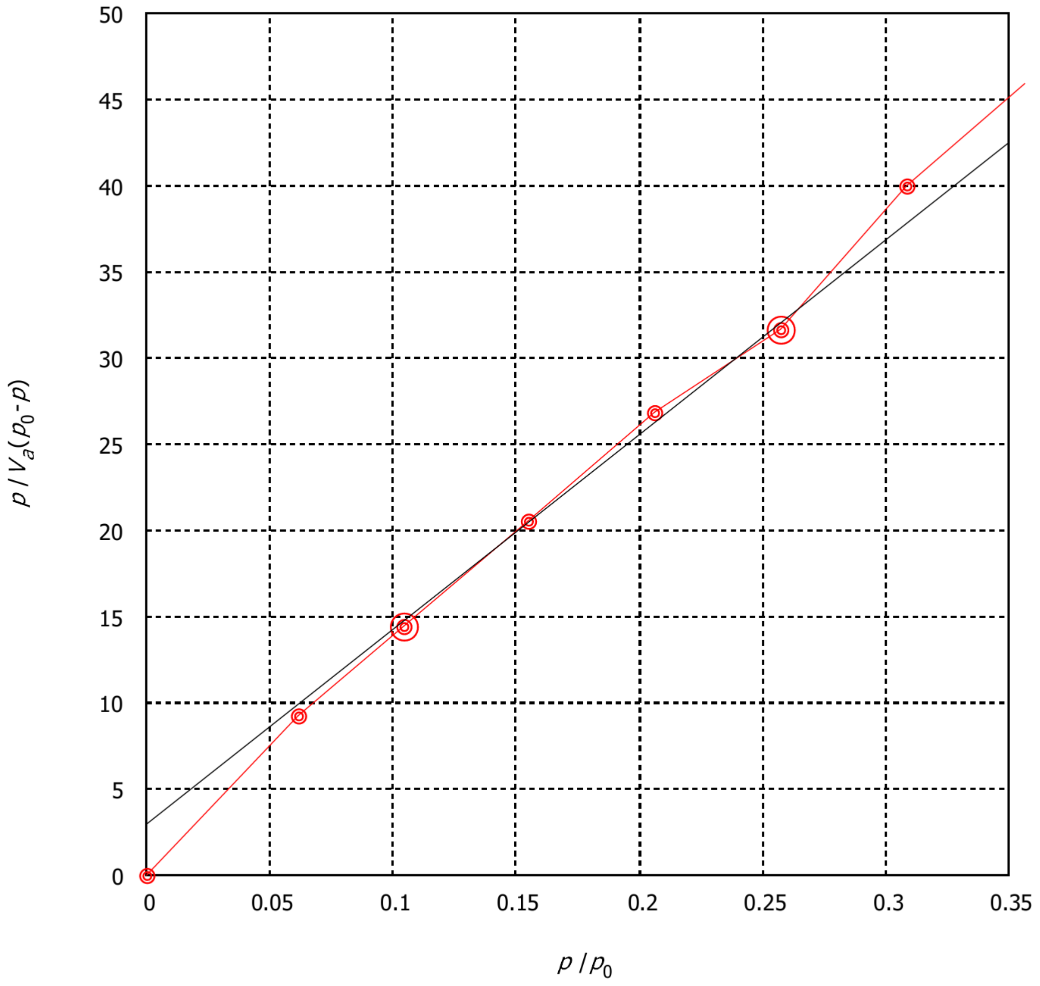
S/N	33	Vs_0	12.386
Date of measurement	2/3/2023	Time of measurement	1:35:20
Adsorptive	N2	Adsorption temperature	77.350 [K]
Sample weight	6.5592 [g]	Adsorbate molecules diameter	0.3540 [nm]
Saturated vapor pressure	98.801 [kPa]	Calculate mean pore diameter.	Do
Calculate mean particle size.	Do	Relative pressure for pore volume calculation	0.9900
Adsorptive molecular weight	28.013	Density	0.8080 [g cm $^{-3}$]
Adsorptive surface area	0.1620 [nm ^{2}]	Analytic method	Use pressure range setting.
Pressure range	0.1000-0.2500		

S.P.	2	
E.P.	5	
Slope(Linear)	44.053	
Intercept(Linear)	0.5473	
Correlation coefficient	0.9999	
V_m	2.2421E-02	[cm^3(STP) g^{-1}]
$a_{s,BET}$	9.7587E-02	[m^2 g^{-1}]
C	81.498	
Total pore volume (p/p_0=0.367)	5.2077E-05	[cm^3 g^{-1}]
Mean pore diameter	2.1346	[nm]

[Adsorption branch] Number of data point : 8

No	p/p_0	$p/V_a(p_0-p)$
0	0.0000	0.0000
1	5.7545E-02	3.0167
2	0.1043	5.1656
3	0.1550	7.3206
4	0.2062	9.6710
5	0.2569	11.858
6	0.3079	14.347
7	0.3669	17.216

Core size 2-4 mm



BET-Plot

Adsorptive N2

Adsorption temperature 77.350 [K]

◎ IG BH06 2 - 4 mm-Port3.DAT

, Vacuum degree before measurement:0.000E+0Pa

Information of Instrument	(S/N: 33,)		
Date of measurement	2/8/2023	Sample weight	7.1012 [g]
Time of measurement	1:34:43	Saturated vapor pressure	98.639 [kPa]

V_m	8.6201E-03	[cm^3(STP) g^{-1}]	$a_{s,BET}$	3.7519E-02	[m^2 g^{-1}]
C	39.390		Total pore volume ($p/_0$=0.369)	1.9052E-05	[cm^3 g^{-1}]
			Mean pore diameter	2.0312	[nm]

1 / 2

BET-Plot

(1/1)

Filename	IG BH06 2 - 4 mm-Port3.DAT
COMMENT	, Vacuum degree before measurement:0.000E+0Pa

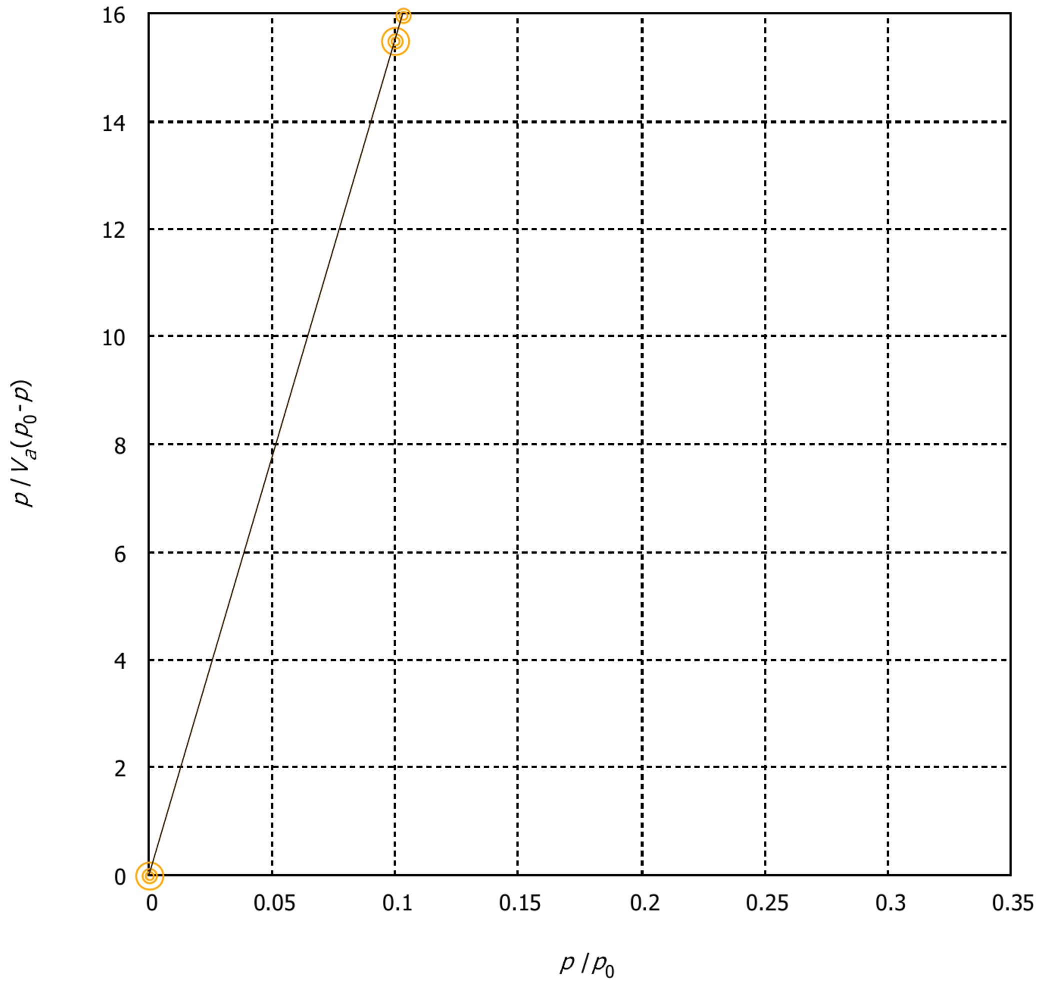
S/N	33	Vs_0	12.386
Date of measurement	2/8/2023	Time of measurement	1:34:43
Adsorptive	N2	Adsorption temperature	77.350 [K]
Sample weight	7.1012 [g]	Adsorbate molecules diameter	0.3540 [nm]
Saturated vapor pressure	98.639 [kPa]	Calculate mean pore diameter.	Do
Calculate mean particle size.	Do	Relative pressure for pore volume calculation	0.9900
Adsorptive molecular weight	28.013	Density	0.8080 [g cm $^{-3}$]
Adsorptive surface area	0.1620 [nm ^{2}]	Analytic method	Use pressure range setting.
Pressure range	0.1000-0.2500		

S.P.	2	
E.P.	5	
Slope(Linear)	113.06	
Intercept(Linear)	2.9451	
Correlation coefficient	0.9983	
V_m	8.6201E-03	[cm^3(STP) g^{-1}]
$a_{s,BET}$	3.7519E-02	[m^2 g^{-1}]
C	39.390	
Total pore volume (p/p_0=0.369)	1.9052E-05	[cm^3 g^{-1}]
Mean pore diameter	2.0312	[nm]

[Adsorption branch] Number of data point : 8

No	p/p_0	$p/V_a(p_0-p)$
0	0.0000	0.0000
1	6.1564E-02	9.2523
2	0.1042	14.460
3	0.1547	20.560
4	0.2064	26.830
5	0.2573	31.624
6	0.3083	40.008
7	0.3690	47.474

Core size 10-20 mm



BET-Plot(Single Point Method)

Adsorptive N2

Adsorption temperature 77.350 [K]

◎ IG BH-06 10-20mm-Port3.DAT

, Vacuum degree before measurement:0.000E+0Pa

Information of Instrument	(S/N: 33,)		
Date of measurement	5/19/2023	Sample weight	5.5748 [g]
Time of measurement	1:46:36	Saturated vapor pressure	97.216 [kPa]

V_m	6.4496E-03	[cm^3(STP) g^{-1}]	$a_{s,BET}$	2.8072E-02	[m^2 g^{-1}]
Total pore volume ($p/_0$=0.103)	1.1123E-05	[cm^3 g^{-1}]	Mean pore diameter	1.5849	[nm]
Analytical pressure [p/p_0]	0.1				

BET-Plot(Single Point Method)

(1/1)

Filename	IG BH-06 10-20mm-Port3.DAT
COMMENT	, Vacuum degree before measurement:0.000E+0Pa

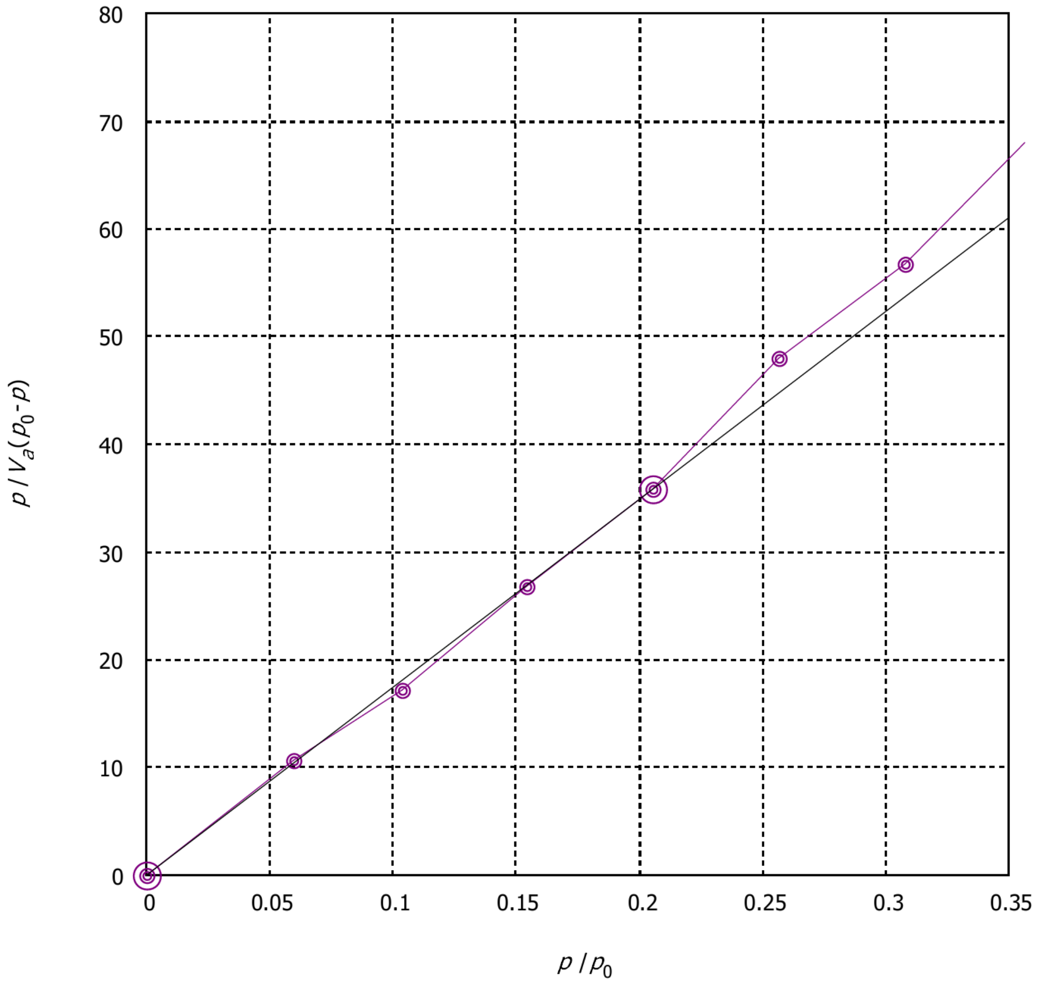
S/N	33	Vs_0	12.386
Date of measurement	5/19/2023	Time of measurement	1:46:36
Adsorptive	N2	Adsorption temperature	77.350 [K]
Sample weight	5.5748 [g]	Adsorbate molecules diameter	0.3540 [nm]
Saturated vapor pressure	97.216 [kPa]	Calculate mean pore diameter.	Do
Calculate mean particle size.	Do	Relative pressure for pore volume calculation	0.9900
Adsorptive molecular weight	28.013	Density	0.8080 [g cm $^{-3}$]
Adsorptive surface area	0.1620 [nm ^{2}]	Analytic method	Single Point Method.
Relative pressure	0.1000 [kPa]		

Analytical pressure	0.1	[p/p_0]
S.P.	0	
E.P.	1	
V_m	6.4496E-03	[cm^3(STP) g^{-1}]
$a_{s,BET}$	2.8072E-02	[m^2 g^{-1}]
Total pore volume (p/p_0 =0.103)	1.1123E-05	[cm^3 g^{-1}]
Mean pore diameter	1.5849	[nm]

[Adsorption branch] Number of data point : 3

No	p / p_0	$p / V_a(p_0 - p)$	
0	0.0000	0.0000	
1	0.1000	15.505	
2	0.1031	15.981	

Core slice



BET-Plot

Adsorptive N2

Adsorption temperature 77.350 [K]

◎ 2-4mm_PC-Port1.DAT

, Vacuum degree before measurement:0.000E+0Pa

Information of Instrument	(S/N: 33,)		
Date of measurement	04/03/2022	Sample weight	5.8500 [g]
Time of measurement	1:40:48	Saturated vapor pressure	96.843 [kPa]

V_m	5.7378E-03	[cm^3(STP) g^{-1}]	$a_{s,BET}$	2.4974E-02	[m^2 g^{-1}]
C			Total pore volume ($p/_{0}$=0.367)	1.2750E-05	[cm^3 g^{-1}]
			Mean pore diameter	2.0421	[nm]

BET-Plot

(1/1)

Filename	2-4mm_PC-Port1.DAT
COMMENT	, Vacuum degree before measurement:0.000E+0Pa

S/N	33	Vs_0	12.386
Date of measurement	04/03/2022	Time of measurement	1:40:48
Adsorptive	N2	Adsorption temperature	77.350 [K]
Sample weight	5.8500 [g]	Adsorbate molecules diameter	0.3540 [nm]
Saturated vapor pressure	96.843 [kPa]	Calculate mean pore diameter.	Do
Calculate mean particle size.	Do	Relative pressure for pore volume calculation	0.9900
Adsorptive molecular weight	28.013	Density	0.8080 [g cm $^{-3}$]
Adsorptive surface area	0.1620 [nm ^{2}]	Analytic method	Use pressure range setting.

S.P.	0	
E.P.	4	
Slope(Linear)	174.28	
Intercept(Linear)	0.0000	
Correlation coefficient	0.9995	
V_m	5.7378E-03	[cm^3(STP) g^{-1}]
$a_{s,BET}$	2.4974E-02	[m^2 g^{-1}]
C		
Total pore volume (p/p_0=0.367)	1.2750E-05	[cm^3 g^{-1}]
Mean pore diameter	2.0421	[nm]

[Adsorption branch] Number of data point : 8

No	p/p_0	$p/V_a(p_0-p)$
0	0.0000	0.0000
1	5.9352E-02	10.697
2	0.1035	17.189
3	0.1545	26.857
4	0.2056	35.824
5	0.2568	47.935
6	0.3079	56.756
7	0.3671	70.366

APPENDIX D

Lab Addendum Memo - Depth Correction

TECHNICAL MEMORANDUM

DATE August 4, 2024

Reference No. 20253946-6046-TM-001

TO File
WSP Canada Inc.

CC File

FROM George Schneider

EMAIL george.schneider@wsp.com

ADDENDUM TO LABORATORY REPORT FROM HYDROISOTOP FOR WORK PACKAGE WP04F - BOREHOLE IG_BH06

This addendum corrects the depths (along borehole) of the samples for BET / CEC analysis included in this report. The depth adjustment is the result of a correction applied to the core logging depths after reconciliation with the drilling depth records. Refer to the WP03 report for details and to the acQuire database for corrected depths.

Refer to Table 1 for the list of samples containing the original logged depths and the corrected depths after reconciliation between the core logging and drilling depths. This addendum shall always accompany the laboratory report when distributed to other parties.

Table 1: List of samples requiring depth correction

Sample ID	Test / Sample Type	Original Logged Depth (mbgs downhole)		Corrected Depth (mbgs downhole)	
		From	To	From	To
IG_BH06_SA001	BET / CEC	822.43	822.89	821.82	822.28

WSP Canada Inc.

George Schneider, M.Sc., P.Geo.

GWS/

Distribution: File

Attachments: N/A

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