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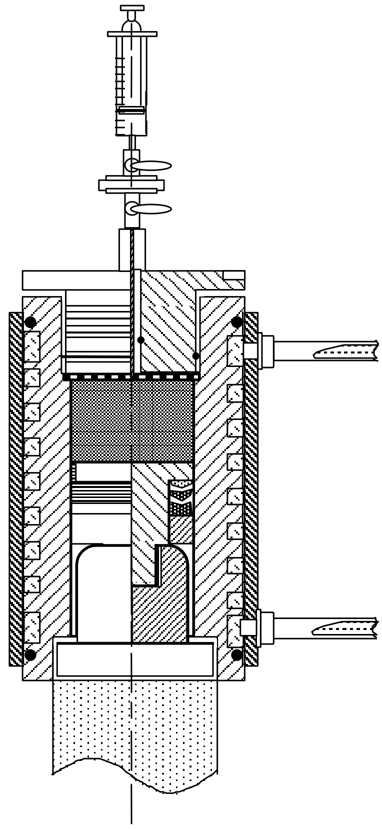
Extraction and Characterisation of Interstitial Pore-Waters from Ruprechtov

Environmental Protection Programme

Commissioned Report CR/04/151N



BRITISH GEOLOGICAL SURVEY

COMMISSIONED REPORT CR/04/151N

Extraction and Characterisation of Interstitial Pore-Waters from Ruprechtov

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Summary

This report describes work carried out by the British Geological Survey as part of a contract (Ref GA/02F/107) with Gesellscahft für Anlagen und Reaktorsicherheit (GRS) to carry out the practical extraction and characterisation of interstitial clay pore-waters from the Ruprechtov site. The contract is a continuation of earlier work carried out by the BGS in 1999/2000 (Reeder *et al*. 2000)

Seven samples were tested for pore-water extraction by mechanical squeezing. These were tested in two phases: the first three in December 2002, and the second four in the Spring of 2004. The report describes the methods used to carry out these tests and summarises all the data produced.

1 Introduction

This report describes work carried out by the British Geological Survey as part of a contract (Ref GA/02F/107) with Gesellscahft für Anlagen und Reaktorsicherheit (GRS) to carry out the practical extraction and characterisation of interstitial clay pore-waters from the Ruprechtov site. The contract is a continuation of earlier work carried out by the BGS in 1999/2000 (Reeder *et al.*, 2000)

Seven samples were tested for pore-water extraction by mechanical squeezing. These were tested in two phases: the first three in December 2002, and the second four in the Spring of 2004. Upon pore-water extraction, 35 water samples were produced by mechanical squeezing (including a deionised blank). The sample codes, test conditions and number of fractions produced by each squeezing test are detailed in Table 1.

Table 1 Sample Code and Test Details

Sample Name	Test Type	Number of Fractions
NA6 37.32-37.42	Pseudo Anaerobic	5
NA7 11.15-11.25	Pseudo Anaerobic	5
NA7 15.35-15.45	Anaerobic	3
NA 10 10.40-10.50	Pseudo Anaerobic	5
NA 12 48.55-48.61	Pseudo Anaerobic	5
NA 13 54.90-55.00	Anaerobic	5
NA 13 55.90-56.00	Pseudo Anaerobic	6

Definitions

Anaerobic	These samples were prepared and squeezed anaerobically within the confines of the anaerobic chamber. See Section 2.2.2.
Pseudo-anaerobic	These samples were prepared in the anaerobic chamber and transferred under anaerobic conditions to minimise oxidation, but squeezed in cells outside the anaerobic chamber. See Section 2.2.3.

2 Pore-Water Extraction by Mechanical Squeezing

2.1 BACKGROUND

The extraction of pore-water from rocks, soils and clayey material by squeezing is analogous to the natural process of consolidation, caused by the deposition of sediments during geological time. A heavy duty squeezing apparatus was developed by the British Geological Survey from the designs of a number of Soviet workers, notably Kriukov (1947), and is capable of applying pressures up to 70 MPa (Manheim 1966). Brightman *et al.* (1985) reviewed the extraction of pore-water by squeezing and described the early work of the BGS. A more detailed description of the technique and its application to a wide range of samples is given by Entwisle and Reeder (1993).

2.2 SAMPLE PREPARATION

Under ideal circumstances, particularly when time and cost are not constraints, all samples would be squeezed undisturbed and anaerobically. According to the original proposal, the samples were to be tested under anaerobic and 'pseudo anaerobic' conditions, however, it should be noted that *all* samples were prepared inside the anaerobic chamber. Samples squeezed in the anaerobic chamber are denoted as 'Anaerobic'; those squeezed outside are denoted as 'Pseudo-anaerobic' (see Sections 2.2.2 and 2.2.3).

2.2.1 Undisturbed Material

All the undisturbed samples were prepared in the anaerobic chamber in an atmosphere of less than 100 ppm O_2. The samples were cut to the required dimensions (less than 75 mm diameter and less than 100 mm height) with a 75 mm stainless steel cutting ring and a large knife. Potentially contaminated or oxidised material within an approximate 10 mm annulus of the block was discarded. A separate sub-sample was also taken for moisture content determination.

2.2.2 Anaerobic Tests

The prepared material was weighed and placed into the anaerobic chamber squeezing cell and squeezed as described in section 2.4.

2.2.3 Pseudo-Anaerobic Tests

The prepared and weighed sample was double-bagged in re-sealable plastic bags in the anaerobic chamber. A plastic extender was attached to the squeezing cell located outside the anaerobic chamber, ensuring that the top was about 100 to 150 mm above the top of the cell and the lower part of the extender was sealed on the side of the cell. The inside of the cell and extender were flushed with argon, which was selected because it is denser than air. The double-bagged sample was transferred out of the anaerobic chamber and the inner bag flushed with argon. The sample was then carefully placed into the squeezing cell while maintaining the argon flush. The paper filter was placed into position and the stainless steel filter was flushed with argon as it was emplaced (see Figure 1). The plastic extender was folded back and the top platen was screwed into the cell after the pore-water pipe was flushed. An initial stress was added rapidly to remove most of the gas remaining in the cell. The syringes were also flushed with argon several times just before use.

2.3 SQUEEZING APPARATUS

The squeezing apparatus utilises available technology based on pumps producing a maximum output stress of 70 MPa. A diagram of the cell body is given in Figure 1.

The metal parts of the cell are made of Type 316 stainless steel, which was selected because of its resistance to corrosion and high tensile strength. The sample chamber is 75 mm in diameter and has a height of about 100 mm.

One cell is contained within an anaerobic chamber for true anaerobic tests while a further three cells are located outside the chamber.

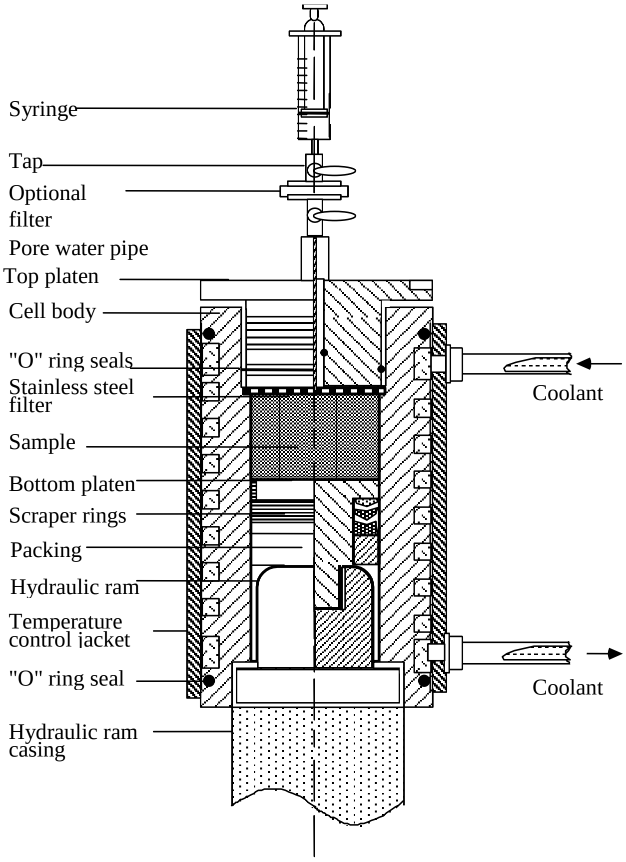

Figure 1 A schematic of the squeezing cell as described by Entwisle and Reeder (1993).

2.4 SQUEEZING PROCEDURE

A pump pressure of about 2 to 3 MPa was applied initially to remove most of the gas from the cell and allow the sample to ‘bed in’. The system was left to stabilise at the ambient temperature of about 16°C. The syringe tap and labelled syringe of known weight (argon flushed syringe for the pseudo-anaerobic tests) were pushed into the top of the pore-water collection pipe.

Applied stress was gradually increased throughout the extraction. The stepped increase in stress during testing is dependent on the physical characteristics of the material tested and the volume of water required. A particular stress may be maintained for a few hours or for several days depending on the rate of pore-water flow. Details of the extraction tests, including the stresses applied, the duration of tests and the volumes of pore-water obtained, are given for each sample in Appendix 1.

2.5 DETERMINATION OF MOISTURE CONTENT

Moisture content of the original sample was determined as soon as possible after opening the preserved material. A subsample of material was accurately weighed then dried at 105ºC for at least 24 h. Moisture content values quoted in this report have been calculated using the following equation:

$$MC = \frac{M_w - M_d}{M_d} \cdot 100$$

Where: MC is the moisture content (%)

M_w is the mass of wet sample (g) and

M_d is the mass of dry sample (g)

3 Analytical Techniques

All analyses carried out by the BGS laboratories have been completed in accordance with the relevant UKAS procedures. A small number of analytes fall outside of UKAS accreditation; where this is the case, it has been stated in the Data Quality section of the relevant technique.

All BGS analytical techniques are subjected to rigorous quality control. Quality control standards (QCs) are analysed at the beginning and end of each run and after not more than a set number of unknown solutions, as described in each procedure. All BGS methods used for the analysis of the pore-waters are also subject to proficiency testing using the Aquacheck scheme.

3.1 DETERMINATION OF MAJOR AND TRACE CATIONS IN AQUEOUS SAMPLES BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

3.1.1 Instrumentation

The determination of major and trace metals, S, P, Si and B was carried out on a Varian/Vista AX CCD simultaneous instrument with dedicated Varian SPS-5 Autosampler. The instrument views the plasma along its axis and is equipped with a high resolution Echelle polychromator with a Vista Chip^{TM} image mapped Charge Coupled Device (CCD) solid-state detector covering a wavelength range of 167 to 785 nm. The sample is introduced via a peristaltic pump into a glass concentric nebuliser connected to a cyclonic action spray chamber. The system is controlled by a dedicated PC running software supplied by the instrument manufacturer.

3.1.2 Theory

The nebulised sample is swept into the central channel of the inductively coupled plasma in a stream of argon carrier gas. On introduction to the plasma, the sample undergoes desolvation, evaporation, atomisation/ionisation and excitation. The atoms or ions decay from their excited state through radiative energy transitions, emitting light of specific energies (or wavelengths). The light emission from the plasma is separated into a 2-dimensional array of the component wavelengths by the Echelle polychromator and the CCD detector simultaneously measures the intensity of each of the measured wavelengths. The intensity of the light emitted at the characteristic wavelength for a particular element is proportional to the amount of the element in the sample being sprayed into the plasma. The instrument is calibrated against standards containing known amounts of the elements being analysed against which samples of unknown concentrations can be quantified.

3.1.3 Analytical Method

Major cations (Ca, Mg, Na, K), trace cations (Ba, Sr, Mn, Fe, Al, Li), total S, total P and Si are determined directly by ICP-AES. Analysis was carried out on samples acidified to 1% with respect to concentrated HNO_3. The instrument was re-calibrated after not more than every 20 samples. Two quality control standards, high and low, were analysed after each calibration, during the run after no more than ten unknown solutions and at the end of each run to check for drift. Blanks, calibration standards and quality control standards were matrix matched to the samples to be analysed. All reported measurements are based on the average of three replicate analyses.

3.1.4 Data Quality

Potassium (K) falls outside of UKAS accreditation, but the quality of the data are nevertheless considered to be as good as for those species that are within the scope of accreditation.

All species preserved by acidification are considered to be stable over an appreciable length of time and certainly within the period between sampling and analysis. Wavelengths and conditions of analysis are optimised to ensure that interference effects are minimised for all elements; samples with complex matrices are diluted prior to analysis. The accuracy of the cation (and anion) determinations can be assessed by considering the ionic charge balances (see Appendix 1).

3.2 DETERMINATION OF MAJOR AND TRACE ANIONS BY ION CHROMATOGRAPHY

3.2.1 Instrumentation

The analysis of major and trace anions was carried out on a Dionex DX-600 Ion Chromatograph system consisting of the following modules: GP50 gradient pump, ED50A electrochemical detector, AD20 absorbance detector, AS50 autosampler and AS50 thermal compartment. The whole system is controlled and data captured by a dedicated computer, installed with the Chromeleon Software (instrument) version 6.40, which is connected to the instrument via a Netgear® Ethernet hub. Separation is performed using an AG14 guard column and an AS14 analytical column using an injection volume of 100 µl.

3.2.2 Theory

A known quantity of unpreserved sample is injected into a stable flow of eluent maintained by the instrument's pump. The eluent and sample mixture pass though the suppressor, which reduces the conductivity of the eluent and increases the conductivity of the analyte by the electrolytic exchange of sodium and hydrogen ions across a cation membrane. Highly conducting sodium carbonate and sodium bicarbonate are converted into poorly conducting carbonic acids by the replacement of sodium for hydrogen; poorly conducting anionic salts in the sample are converted into more conductive anionic salts, thus improving the conductivity detector background/sample response ratio. The now 'suppressed' eluent and sample mixture passes through the guard column, the function of which is to protect the analytical column from contamination or damage. The mixture then passes though the analytical column. The various anions are differentially retarded on the column according to size and charge, which dictate their affinity for the stationary and mobile phases. The conductivities and UV absorbances of the separated anions eluted from the column are detected as transient peaks using both electrochemical and UV absorbance detectors. Quantification is subsequently achieved by comparing their areas to those of standards with known concentrations using the dedicated instrument software, Charlton *et al.* (2003).

3.2.3 Analytical Method

Major and trace anions (Cl^-, SO_4^{2-}, NO_3^-, NO_2^-, Br^-, HPO_4^{2-}, $S_2O_3^{2-}$ and F^-) are determined by ion chromatography as described by Charlton *et al.* (2003). The instrument was calibrated at the beginning of every analytical run using twelve standards, two of which are prepared manually, the other ten being prepared on line by the instrument. QC and blank samples are analysed at the start and end of each run and after not less than every 20 samples. A calibration drift check standard was run at the end of each run and after not less than every 50 samples. The analysis followed a pre-programmed schedule and the data were collected by the software with peaks identified by retention time. The method is limited by the number of exchange sites available within the column. With the injection loop and column used in this study, solutions with total anion concentrations of up to 1000 mg l^{-1} can be analysed; above this the column becomes overloaded, causing poor peak shapes, variable retention times and thus unreliable results. To overcome this problem, more concentrated solutions were diluted. Dilution was also used to bring the analyte concentration within the concentration range covered by the standards.

3.2.4 Data Quality

All determinands reported for pore-water analysis fall within the scope of UKAS accreditation. Thiosulphate ($S_2O_3^{2-}$) determination falls outside of UKAS accreditation, however no thiosulphate was observed in any sample.

Most anions are stable in solution for an appreciable length of time and certainly within the period between sampling and analysis. Nitrate and nitrite may, however, be modified by microbial activity and for this reason samples were analysed by ion chromatography as soon as possible after collection.

3.3 DETERMINATION OF pH AND ALKALINITY BY POTENTIOMETRIC TITRATION

3.3.1 Instrumentation

The analysis for this work was carried out on a Radiometer auto titrator consisting of a VIT 90 video controller module with an ABU 93 autoburette and a SAM 90 sample section. The VIT 90 is connected to a printer and results for each sample are printed upon completion of analysis.

3.3.2 Theory

pH is measured potentiometrically by immersing a hydrogen ion selective electrode and a reference electrode into the solution to be analysed, the resulting response is compared to that of known standards. Alkalinity is measured titrimetrically by monitoring the change in pH of the solution as a function of the volume of titrant added, the volume of titrant added is then converted into mg l^{-1} HCO_3^- by the instrument using molar calculations.

Total alkalinity may be defined as the amount of sulphuric acid required to titrate a sample to the equivalence point of bicarbonate at approximately pH 4.5, expressed as the equivalent mg l^{-1} of bicarbonate (HCO_3^-). It is actually a measure of the acid neutralising capacity of the sample but, as this is dominated in groundwaters by the carbonic acid equilibria system, the expression of total alkalinity in terms of bicarbonate is common practice.

3.3.3 Analytical Method

The instrument is calibrated for pH using pH 4 and pH 10 buffers after entering the laboratory temperature. This calibration is then checked with a pH 7 buffer for which the result must be within 0.02 pH units. The alkalinity titration is checked at the beginning and end of every run and not less than every 10 unknown samples using a 200 mg l^{-1} HCO_3^- QC solution. A known quantity of sample is placed in the titration vessel and the electrodes lowered in to the solution. The sample is allowed to stabilise before the pH reading is taken and the titration started. Once the titration has begun, the instrument adds small amounts of acid to the solution until the end point is reached at approximately pH 2.8. The instrument then calculates the concentration of HCO_3^-, this information is then printed and the electrodes and vessel rinsed and wiped before the next sample.

3.3.4 Data Quality

All data for this method are within the scope of UKAS accreditation.

The inflection point for samples that gave clearly defined end-points, was in the range pH 4.3 to 5.0. However, the determination of total alkalinity is complicated by the presence of species other than bicarbonate and carbonate (e.g. organic bases, silicates, borates, sulphides or phosphates) that buffer the titration and may cause higher than expected results. It is difficult to calculate the total buffering capacity to the neutralisation point because, in most cases, the end point is poorly defined. In cases where the total inorganic carbon content was very low, it was often found that total alkalinity could not be determined because of the ill defined end point.

3.4 DETERMINATION OF ORGANIC AND INORGANIC CARBON

3.4.1 Instrumentation

Measurements are made on a Shimadzu TOC-5000 total organic carbon analyser fitted with an ASI-5000 autosampler. A Parker Balston gas generator, model number 78-40-220, was also connected to the instrument as a carbon-free air supply.

3.4.2 Theory

For the measurement of total carbon, pure air carrier gas (containing no CO_2) flows at 150 ml min^{-1} through the total carbon (TC) combustion tube, which is packed with Pt-coated alumina catalyst maintained at 680°C by an electrically heated furnace. When the sample enters the TC combustion tube, all of the carbon is oxidised to carbon dioxide and swept out of the tube by the carrier gas into a non-dispersive infrared (NDIR) detector which measures the carbon dioxide concentration by energy absorption. The output analogue signal of the NDIR detector is displayed as a response peak. The area of the peak is proportional to the total carbon concentration. The total carbon in a sample is determined from the calibration curve prepared using standard solutions containing known amounts of organic and inorganic carbon.

For the measurement of inorganic carbon (IC), the sample is injected into the inorganic carbon reaction vessel which has carrier gas bubbling through a phosphoric acid reaction solution. The IC reacts with the acid to form carbon dioxide which is detected by the NDIR detector. The inorganic carbon content is the sum of the aqueous carbon dioxide, carbonate and bicarbonate present in solution.

Organic carbon (OC) is calculated as the difference between the total carbon and the inorganic carbon contents.

3.4.3 Analytical Method

The instrument is calibrated at 10, 30 and 100 mg l^{-1} using separate organic and inorganic carbon standards. Mixed quality control standards containing 5, 10 and 50 mg l^{-1} organic and inorganic carbon were analysed at the beginning and end of each run and after not more than 20 unknown samples. Approximately 1 ml of unpreserved sample was used for each analysis. Samples exceeding the top calibration standard were automatically re-injected at a smaller volume to bring them into the calibration range. Data are stored by the instrument and sample analysis details, including concentrations, are printed automatically after each sample has been analysed.

3.4.4 Data Quality

All data for this method fall within UKAS accreditation.

TOC and TIC are stable for short periods of time if the sample is stored at <4°C, although every effort was made to analyse the samples as soon as possible after collection. The method is essentially interference free as long as the IC or OC is fully converted to CO_2 during the two different oxidation procedures. This has been thoroughly investigated by the instrument manufacturer and is also verified by the external check standards that are run.

3.5 DETERMINATION OF TRACE ELEMENTS IN AQUEOUS SAMPLES BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY

3.5.1 Instrumentation

The ICP-MS instrument used in this work was a VG Elemental ExCell in conjunction with a Cetac ASX-510 autosampler. The system is controlled by a pc through dedicated ICP-MS software. The ICP RF forward power used is 1350 W. Coolant, auxiliary and injector gas flow rates are set at 13, 0.9 and 0.94 l min^{-1} respectively. Data are acquired in peak jumping mode using three x 20 s acquisitions.

3.5.2 Theory

An ICP-MS consists of an ICP, which provides a source of positively charged ions, and a quadrupole mass spectrometer, which detects these ions, linked by an interface. Liquid samples are passed through a nebuliser, and the resulting sample aerosol and argon mixture passes through a Peltier-cooled spray chamber, to remove the larger droplets, before injection into the central channel of the ICP torch. At the high temperatures of the ICP (in excess of 6000°C) the sample is desolvated and substantially ionised.

The positively charged elemental ions are extracted from the plasma into the vacuum system of the mass spectrometer. The ions pass through a sampling orifice of 1mm diameter into a mechanically pumped vacuum system, where a supersonic jet forms. The central section of this jet is extracted through the skimmer orifice of 0.7 mm diameter and then focussed by a series of electrostatic ion lenses into the quadrupole mass analyser. DC and RF voltages are applied to opposite pairs of the four rods of the quadrupole. These voltages are varied such that only ions of a given mass:charge (m/z) ratio will have stable path through the rods and emerge from the other end. By varying the DC and RF voltages rapidly, the mass spectrometer is able to sweep across the mass range from 0 to 300 mass units in less than a second.

The ions transmitted by the quadrupole are detected using a discrete dynode electron multiplier. Counts for a particular mass are accumulated for a number of sweeps across the mass range and are proportional to the concentration of that element being aspirated. The response at any mass is calibrated against standards containing a known concentration of the element of interest. Although the quadrupole analyser has a relatively low resolving power (<400), it is sufficient to separate ion m/z from ion $(m/z)+1$ but not from a polyatomic ion with a very similar m/z.

3.5.3 Analytical Method

The isotopes selected for measurement were 232Th and 238U; Th is monoisotopic and 238U has a natural abundance of 99.3%. Because of the small volume of sample available, they were all diluted by a factor of 5 (1+4); further dilutions were necessary for samples containing very high concentrations of uranium.

The instrument was calibrated at the beginning of every analytical run using four standards in the range 0 to 50 µg l-1 prepared from certified Claritas PPT multielement standards purchased from

Spex CertiPrep®. In addition, mixed element standards at 10 µg l-1, plus a blank, were inserted at regular intervals throughout the analysis run and used to correct for any drift in instrument sensitivity via the instrument software. All working standards were prepared on the day of analysis.

An internal standard solution containing 10 µg l-1 indium was added to the samples in at a fixed ratio of approximately 1:1 via a T-piece. Any suppression of the instrument signal caused by the matrix was corrected by the instrument software using the response of the internal standard.

Multielement quality control (QC) check standards, containing the elements of interest at 10 µg l-1 were analysed at the start and end of each run and after no more than every 20 samples, and charted using QI Analyst® software. Raw concentration data were transferred to an Excel spreadsheet and corrected for dilution and reagent blanks. Each analytical run was independently verified by a different analyst, including post processing of the data for drift, dilution and collation.

Accuracy is monitored by regular participation in the Aquacheck interlaboratory proficiency testing scheme for waters, in which approximately 350 laboratories participate worldwide, as well as the inclusion of a certified reference water described below.

3.5.4 Data Quality

All data for this method fall outside the scope of UKAS accreditation.

All species preserved by acidification are considered to be stable over an appreciable length of time and certainly within the delay between sampling and analysis.

During the analysis of thorium, two out of a total of four thorium QC standards were approximately 10% high. However, because all the sample data were below the detection limit for this element, the blank data were considered to be much more important than the slope of the calibration line. Therefore these data have been reported. It was considered very doubtful whether better quality data for thorium could have been obtained by rerunning the samples.

4 Results

All pore-water extraction and subsequent physical and chemical testing was completed as set out in the proposal. Seven samples were provided for testing, as described in Table 1. The moisture content of the samples varied between 10.6% and 41.5% with respect to dry weight.

Pore-water extraction by conventional squeezing was successful for all seven tests. In order to obtain sufficient pore-water for chemical analysis of separate fractions, tests were carried out for between 1 h and 334 h. The maximum stress applied was 60 MPa. Between three and six pore-water fractions (each of approximately 5 g or greater) were successfully collected from each sample. The total volume of pore-water obtained per sample by mechanical squeezing varied between 1.68 ml and 21.44 ml.

Full suite chemical characterisation was possible on all of the 34 pore-water samples collected.

All but five of the 34 pore-water fractions had ionic charge balances within 10%, and all but ten were within 5%. The five samples for which ionic charge balances fall outside 5% are for fractions obtained from sample NA10 10.40-10.50 (10843-0018 to 10843-0022), which are low TDS (cationic and anionic totals less than 1 meq l^{-1}) and in which bicarbonate (and TIC) is less than the limit of quantification but could still be a major anionic constituent. These ionic charge balances are considered more than acceptable for these low TDS samples.

All squeezing test and chemical analysis data are contained on a sample by sample basis in Appendix 1

Appendix 1 Geotechnical Test and Chemical Analysis Data Sheets

Summary of Squeezing and Chemical Analysis Test Data For Sample NA6 37.32-37.42

Client	GRS
Core Sample Number	NA6 37.32-37.42
BGS Pore-Water Numbers	10497-0001 to 10497-0005
Test Type	Psuedo Anaerobic

Geotechnical Test Data

Date squeezing started		17/12/2002
Initial sample weight	g	522.45

Fraction		1	2	3	4	5
BGS Pore-Water Number		10497-0001	10497-0002	10497-0003	10497-0004	10497-0005
Test time	h	70	72	168	170	70
Total time	h	70	142	310	480	550
Minimum stress	MPa	2	13	20	25	35
Maximum stress	MPa	13	13	25	35	35
Initial moisture content	%	27.8	24.2	21.4	17.1	14.3
Final moisture content	%	24.2	21.4	17.1	14.3	11.8
Volume extracted	ml	14.60	12.01	17.01	10.57	5.82
Total volume extracted	ml	14.60	26.61	43.62	54.19	60.01

Chemical Analysis

pH		7.87	8.07	7.85	7.97	8.07
Calcium	mg l^{-1} Ca	131	126	136	145	124
Magnesium	mg l^{-1} Mg	65.9	69.2	82.4	89.3	81.6
Sodium	mg l^{-1} Na	33.8	30.3	25.9	24.5	12.4
Potassium	mg l^{-1} K	24.1	14.5	7.02	4.60	<2.50
Total alkalinity	mg l^{-1} HCO$_3$	204	199	212	268	256
Chloride	mg l^{-1} Cl	4.17	2.71	1.77	2.15	2.52
Sulphate	mg l^{-1} SO$_4$	473	463	488	474	381
Nitrate	mg l^{-1} NO$_3$	2.43	<0.20	<0.20	<0.40	0.47
Bromide	mg l^{-1} Br	<0.15	<0.15	<0.15	<0.30	<0.30
Nitrite	mg l^{-1} NO$_2$	0.63	0.10	0.05	<0.10	<0.10
Orthophosphate	mg l^{-1} HPO$_4$	<2.50	<2.50	<2.50	<5.00	<5.00
Fluoride	mg l^{-1} F	1.27	1.26	1.42	1.40	1.20
Total organic carbon	mg l^{-1} C	20.1	16.21	7.98	9.95	9.81
Total inorganic carbon	mg l^{-1} C	39.2	39.3	45.3	52.9	51.4
Total phosphorus	mg l^{-1} P	0.18	0.07	0.10	0.12	0.13
Total sulphur	mg l^{-1} S	165	161	172	167	135
Silicon	mg l^{-1} Si	8.52	9.66	10.7	12.5	13.0
Silicate	mg l^{-1} SiO$_2$	18.2	20.7	22.9	26.8	27.8
Barium	mg l^{-1} Ba	0.059	0.057	0.053	0.064	0.051
Strontium	mg l^{-1} Sr	0.944	0.902	0.985	1.07	0.90
Manganese	mg l^{-1} Mn	0.132	0.142	0.173	0.216	0.201
Total iron	mg l^{-1} Fe	0.037	0.039	0.038	0.057	0.229
Aluminium	mg l^{-1} Al	0.174	0.048	0.048	0.203	0.207
Lithium	mg l^{-1} Li	0.277	0.298	0.313	0.283	0.180
Uranium	µg l^{-1} U	6.74	5.09	5.26	5.63	4.33
Thorium	µg l^{-1} Th	<0.10	<0.10	<0.10	<0.20	<0.20
Ionic charge balance	%	2.50	2.64	4.10	4.87	4.77

Summary of Squeezing and Chemical Analysis Test Data For Sample NA7 11.15-11.25

Client	GRS
Core Sample Number	NA7 11.15-11.25
BGS Pore-Water Numbers	10497-0006 to 10497-0010
Test Type	Pseudo Anaerobic

Geotechnical Test Data

Date squeezing started		17/12/2002
Initial sample weight	g	507.00

Fraction		1	2	3	4	5
BGS Pore-Water Number		10497-0006	10497-0007	10497-0008	10497-0009	10497-0010
Test time	h	24	49	72	168	168
Total time	h	24	73	145	313	481
Minimum stress	MPa	2	8	13	18	23
Maximum stress	MPa	8	13	13	23	23
Initial moisture content	%	41.5	38.7	34.4	29.5	23.3
Final moisture content	%	38.7	34.4	29.5	23.3	20.2
Volume extracted	ml	9.45	14.32	17.26	21.44	9.24
Total volume extracted	ml	9.45	23.77	41.03	62.47	71.71

Chemical Analysis

pH		6.09	6.43	6.21	6.26	6.07
Calcium	mg l^{-1} Ca	214	183	168	182	203
Magnesium	mg l^{-1} Mg	64.1	61.3	63.7	74.1	84.7
Sodium	mg l^{-1} Na	28.8	25.5	23.6	20.5	19.1
Potassium	mg l^{-1} K	7.88	5.73	3.97	1.83	1.29
Total alkalinity	mg l^{-1} HCO$_3$	<44	<44	<44	<44	<44
Chloride	mg l^{-1} Cl	8.80	4.86	4.19	3.73	3.66
Sulphate	mg l^{-1} SO$_4$	837	718	691	766	873
Nitrate	mg l^{-1} NO$_3$	6.65	1.59	<0.40	<0.40	<0.40
Bromide	mg l^{-1} Br	<0.30	<0.30	<0.30	<0.30	<0.30
Nitrite	mg l^{-1} NO$_2$	<0.10	<0.10	<0.10	<0.10	<0.10
Orthophosphate	mg l^{-1} HPO$_4$	<5.00	<5.00	<5.00	<5.00	<5.00
Fluoride	mg l^{-1} F	<0.50	0.54	<0.50	0.61	0.55
Total organic carbon	mg l^{-1} C	40.3	17.9	8.57	8.00	9.02
Total inorganic carbon	mg l^{-1} C	<6.00	<3.00	<3.00	<3.00	<3.00
Total phosphorus	mg l^{-1} P	0.05	0.03	0.03	<0.01	0.03
Total sulphur	mg l^{-1} S	286	250	242	266	296
Silicon	mg l^{-1} Si	12.4	12.8	13.7	15.2	16.4
Silicate	mg l^{-1} SiO$_2$	26.4	27.3	29.3	32.5	35.1
Barium	mg l^{-1} Ba	0.056	0.065	0.062	0.048	0.044
Strontium	mg l^{-1} Sr	1.21	1.11	1.09	1.19	1.37
Manganese	mg l^{-1} Mn	15.2	14.5	15.3	17.7	20.0
Total iron	mg l^{-1} Fe	3.22	16.7	27.0	38.6	46.4
Aluminium	mg l^{-1} Al	0.054	0.064	0.074	0.031	0.089
Lithium	mg l^{-1} Li	0.542	0.565	0.574	0.495	0.529
Uranium	μg l^{-1} U	<0.20	<0.10	<0.10	<0.10	<0.20
Thorium	μg l^{-1} Th	<0.20	<0.10	<0.10	<0.10	<0.20
Ionic charge balance	%	-0.21	4.05	5.91	6.44	5.82

Summary of Squeezing and Chemical Analysis Test Data For Sample NA7 15.35-15.45

Client	GRS
Core Sample Number	NA7 15.35-15.45
BGS Pore-Water Numbers	10497-0011 to 10497-0013
Test Type	Anaerobic

Geotechnical Test Data

Date squeezing started		16/12/2002
Initial sample weight	g	641.20

Fraction		1	2	3
BGS Pore-Water Number		10497-0011	10497-0012	10497-0013
Test time	h	16	120	334
Total time	h	16	136	470
Minimum stress	MPa	1	11	60
Maximum stress	MPa	11	40	60
Initial moisture content	%	10.6	9.25	7.52
Final moisture content	%	9.25	7.52	6.40
Volume extracted	ml	7.20	6.59	1.68
Total volume extracted	ml	7.20	13.79	15.47

Chemical Analysis

pH		7.36	7.11	7.51
Calcium	mg l^{-1} Ca	195	201	209
Magnesium	mg l^{-1} Mg	79.0	75.5	81.0
Sodium	mg l^{-1} Na	43.6	26.5	24.6
Potassium	mg l^{-1} K	40.0	16.6	13.6
Total alkalinity	mg l^{-1} HCO_3	66	48	<88
Chloride	mg l^{-1} Cl	8.05	7.17	8.10
Sulphate	mg l^{-1} SO_4	838	809	775
Nitrate	mg l^{-1} NO_3	3.57	<0.40	0.74
Bromide	mg l^{-1} Br	<0.30	<0.30	<0.15
Nitrite	mg l^{-1} NO_2	<0.10	<0.10	<0.05
Orthophosphate	mg l^{-1} HPO_4	<5.00	<5.00	<2.50
Fluoride	mg l^{-1} F	<0.50	<0.50	0.37
Total organic carbon	mg l^{-1} C	22.7	20.6	30.8
Total inorganic carbon	mg l^{-1} C	14.3	8.74	<12.0
Total phosphorus	mg l^{-1} P	0.08	<0.05	<0.20
Total sulphur	mg l^{-1} S	292	275	264
Silicon	mg l^{-1} Si	13.8	13.7	10.0
Silicate	mg l^{-1} SiO_2	29.6	29.3	21.5
Barium	mg l^{-1} Ba	0.062	0.057	0.111
Strontium	mg l^{-1} Sr	1.58	1.43	1.41
Manganese	mg l^{-1} Mn	13.8	16.8	17.1
Total iron	mg l^{-1} Fe	2.20	14.6	0.77
Aluminium	mg l^{-1} Al	0.209	0.075	0.325
Lithium	mg l^{-1} Li	0.59	0.63	0.762
Uranium	µg l^{-1} U	0.25	0.33	<2.00
Thorium	µg l^{-1} Th	<0.20	<0.20	<2.00
Ionic charge balance	%	1.51	2.53	6.59

Summary of Squeezing and Chemical Analysis Test Data For Sample NA10 10.40-10.50

Client	GRS
Core Sample Number	NA10 10.40-10.50
BGS Pore-Water Numbers	10843-0018 to 10843-0022
Test Type	Pseudo Anaerobic

Geotechnical Test Data

Date squeezing started		13/04/2004
Initial sample weight	g	536.50

Fraction		1	2	3	4	5
BGS Pore-Water Number		10843-0018	10843-0019	10843-0020	10843-0021	10843-0022
Test time	h	5	1	1	19	4
Total time	h	5	6	7	26	30
Minimum stress	MPa	1	3	3	7	16
Maximum stress	MPa	2	4	7	13	20
Initial moisture content	%	17.2	15.6	13.7	11.7	9.80
Final moisture content	%	15.6	13.7	11.7	9.80	8.10
Volume extracted	ml	6.16	8.13	8.35	8.50	6.88
Total volume extracted	ml	6.16	14.29	22.64	31.14	38.02

Chemical Analysis

pH		6.96	7.13	7.05	6.96	7.41
Calcium	mg l^{-1} Ca	5.75	3.18	1.79	2.57	2.20
Magnesium	mg l^{-1} Mg	3.09	1.55	0.799	1.19	0.909
Sodium	mg l^{-1} Na	7.54	4.51	3.50	4.41	4.15
Potassium	mg l^{-1} K	<5.00	<2.50	<2.50	<2.50	<5.00
Total alkalinity	mg l^{-1} HCO_3	<88	<88	<88	<88	<88
Chloride	mg l^{-1} Cl	2.19	1.11	0.576	0.525	0.516
Sulphate	mg l^{-1} SO_4	11.3	7.54	4.12	3.43	1.59
Nitrate	mg l^{-1} NO_3	0.728	0.535	0.247	0.133	0.300
Bromide	mg l^{-1} Br	<0.100	<0.100	<0.100	<0.100	<0.100
Nitrite	mg l^{-1} NO_2	0.227	0.165	<0.050	<0.050	<0.050
Orthophosphate	mg l^{-1} HPO_4	<0.500	<0.500	<0.500	<0.500	<0.500
Fluoride	mg l^{-1} F	0.336	0.216	0.143	0.151	0.190
Total organic carbon	mg l^{-1} C	38.7	17.5	6.49	6.44	<6.00
Total inorganic carbon	mg l^{-1} C	<3.00	<3.00	<3.00	<3.00	3.00
Total phosphorus	mg l^{-1} P	<0.100	<0.050	0.062	0.090	0.113
Total sulphur	mg l^{-1} S	7.70	4.37	2.57	2.49	<2.50
Silicon	mg l^{-1} Si	6.30	6.95	7.61	9.05	10.3
Silicate	mg l^{-1} SiO_2	13.5	14.9	16.3	19.4	21.9
Barium	mg l^{-1} Ba	0.049	0.043	0.057	0.072	0.101
Strontium	mg l^{-1} Sr	0.009	0.017	<0.010	0.011	<0.020
Manganese	mg l^{-1} Mn	0.058	0.043	0.027	0.044	0.036
Total iron	mg l^{-1} Fe	<0.100	<0.050	<0.050	<0.050	<0.100
Aluminium	mg l^{-1} Al	<0.100	<0.050	<0.050	<0.050	<0.100
Lithium	mg l^{-1} Li	<0.250	<0.125	<0.125	<0.125	<0.250
Uranium	µg l^{-1} U	4.75	3.43	1.67	2.92	2.00
Thorium	µg l^{-1} Th	<0.2	<0.2	<0.2	<0.2	<0.2
Ionic charge balance	%	44.94	39.02	46.23	62.68	70.88

Summary of Squeezing and Chemical Analysis Test Data For Sample NA12 48.55-48.61

Client	GRS
Core Sample Number	NA12 48.55-48.61
BGS Pore-Water Numbers	10843-0001 to 10843-0003, 10843-0016 and 10843-0017
Test Type	Psuedo Anaerobic

Geotechnical Test Data

Date squeezing started		30/03/2004
Initial sample weight	g	403.91

Fraction		1	2	3	4	5
BGS Pore-Water Number		10843-0001	10843-0002	10843-0003	10843-0017	10843-0016
Test time	h	21	9	15	24	8
Total time	h	21	30	45	69	77
Minimum stress	MPa	3	8	15	20	40
Maximum stress	MPa	8	12	15	40	40
Initial moisture content	%	37.6	34.8	32.6	30.0	28.0
Final moisture content	%	34.8	32.6	30.00	28.00	24.2
Volume extracted	ml	7.45	5.82	6.52	5.57	10.13
Total volume extracted	ml	7.45	13.27	19.79	25.36	35.49

Chemical Analysis

pH		8.26	8.37	8.38	8.37	8.41
Calcium	mg l^{-1} Ca	138	113	103	99.6	95.1
Magnesium	mg l^{-1} Mg	63.8	54.6	53.5	56.6	59.5
Sodium	mg l^{-1} Na	35.2	32.7	29.8	26.7	17.8
Potassium	mg l^{-1} K	27.2	20.2	12.9	7.71	2.72
Total alkalinity	mg l^{-1} HCO_3	183	193	205	205	216
Chloride	mg l^{-1} Cl	4.48	4.35	3.56	3.32	2.78
Sulphate	mg l^{-1} SO_4	484	383	333	314	270
Nitrate	mg l^{-1} NO_3	0.204	0.122	<0.100	0.689	0.271
Bromide	mg l^{-1} Br	0.139	0.117	0.114	0.123	<0.100
Nitrite	mg l^{-1} NO_2	0.099	0.114	<0.050	<0.050	<0.050
Orthophosphate	mg l^{-1} HPO_4	<0.500	0.767	0.556	<0.500	<0.500
Fluoride	mg l^{-1} F	2.45	2.61	2.66	2.84	2.63
Total organic carbon	mg l^{-1} C	22.1	23.7	7.72	7.17	<6.00
Total inorganic carbon	mg l^{-1} C	35.4	33.5	38.0	37.8	41.1
Total phosphorus	mg l^{-1} P	0.075	0.101	0.062	<0.100	<0.050
Total sulphur	mg l^{-1} S	170	129	112	105	93.8
Silicon	mg l^{-1} Si	3.25	3.60	4.19	4.57	5.73
Silicate	mg l^{-1} SiO_2	6.94	7.69	8.96	9.79	12.3
Barium	mg l^{-1} Ba	0.169	0.121	0.115	0.061	0.072
Strontium	mg l^{-1} Sr	1.02	0.815	0.734	0.728	0.717
Manganese	mg l^{-1} Mn	0.073	0.062	0.071	0.076	0.088
Total iron	mg l^{-1} Fe	0.074	0.064	0.068	0.111	0.088
Aluminium	mg l^{-1} Al	<0.050	<0.050	<0.050	<0.100	<0.050
Lithium	mg l^{-1} Li	0.365	0.346	0.375	0.281	0.230
Uranium	µg l^{-1} U	32.4	21.1	19.7	18.6	17.8
Thorium	µg l^{-1} Th	<0.2	<0.2	<0.2	<0.2	<0.2
Ionic charge balance	%	3.94	3.01	3.27	4.16	5.85

Summary of Squeezing and Chemical Analysis Test Data For Sample NA13 54.90-55.00

Client	GRS
Core Sample Number	NA13 54.90-55.00
BGS Pore-Water Numbers	10843-0004 to 10843-0008
Test Type	Anaerobic

Geotechnical Test Data

Date squeezing started		24/03/2004
Initial sample weight	g	442.07

Fraction		1	2	3	4	5
BGS Pore-Water Number		10843-0004	10843-0005	10843-0006	10843-0007	10843-0008
Test time	h	28	19	99	30	19
Total time	h	28	47	146	176	195
Minimum stress	MPa	2	5	10	20	30
Maximum stress	MPa	5	8	18	25	35
Initial moisture content	%	31.1	28.9	26.2	20.9	18.7
Final moisture content	%	28.9	26.2	20.9	18.7	16.5
Volume extracted	ml	6.44	7.93	16.97	6.49	5.94
Total volume extracted	ml	6.44	14.37	31.34	37.83	43.77

Chemical Analysis

pH		4.11	3.96	4.06	4.09	4.16
Calcium	mg l^{-1} Ca	637	638	622	590	573
Magnesium	mg l^{-1} Mg	359	356	341	321	318
Sodium	mg l^{-1} Na	117	92.2	82.8	73.3	62.0
Potassium	mg l^{-1} K	65.4	59.0	41.2	25.7	14.2
Total alkalinity	mg l^{-1} HCO$_3$	<22	<22	<22	<22	<22
Chloride	mg l^{-1} Cl	2.34	3.24	2.69	0.533	1.43
Sulphate	mg l^{-1} SO$_4$	3677	3540	3313	3163	3117
Nitrate	mg l^{-1} NO$_3$	4.20	1.52	0.534	0.240	0.671
Bromide	mg l^{-1} Br	<0.100	<0.100	<0.100	<0.100	<0.100
Nitrite	mg l^{-1} NO$_2$	0.061	<0.050	<0.050	<0.050	<0.050
Orthophosphate	mg l^{-1} HPO$_4$	<0.500	<0.500	<0.500	0.545	0.611
Fluoride	mg l^{-1} F	4.18	4.15	3.55	3.01	2.76
Total organic carbon	mg l^{-1} C	36.7	22.0	14.1	15.0	15.9
Total inorganic carbon	mg l^{-1} C	<6.00	<3.00	<1.50	<6.00	<6.00
Total phosphorus	mg l^{-1} P	0.072	0.187	0.185	0.334	0.310
Total sulphur	mg l^{-1} S	1359	1184	1261	995	1111
Silicon	mg l^{-1} Si	66.5	63.3	73.3	63.7	79.6
Silicate	mg l^{-1} SiO$_2$	142	135	157	136	170
Barium	mg l^{-1} Ba	0.049	0.039	0.033	0.032	0.032
Strontium	mg l^{-1} Sr	4.46	4.18	3.84	3.50	3.36
Manganese	mg l^{-1} Mn	2.91	2.58	2.40	2.22	2.18
Total iron	mg l^{-1} Fe	200	178	165	155	158
Aluminium	mg l^{-1} Al	7.00	6.47	4.80	4.14	3.35
Lithium	mg l^{-1} Li	1.51	1.31	1.20	1.04	0.998
Uranium	µg l^{-1} U	457	797	613	482	375
Thorium	µg l^{-1} Th	<0.2	<0.2	<0.2	<0.2	<0.2
Ionic charge balance	%	1.46	1.63	2.57	1.63	1.05

Geotechnical Test Data

Date squeezing started		25/03/2004
Initial sample weight	g	584.80

Fraction		1	2	3	4	5	6
BGS Pore-Water Number		10843-0009	10843-0010	10843-0011	10843-0012	10843-0013	10843-0014
Test time	h	2	2	16	10	62	24
Total time	h	2	4	20	30	92	116
Minimum stress	MPa	2	5	10	14	35	45
Maximum stress	MPa	3	7	11	25	35	55
Initial moisture content	%	15.9	14.6	12.9	11.2	9.90	8.00
Final moisture content	%	14.6	12.9	11.2	9.90	8.00	5.90
Volume extracted	ml	6.30	7.51	7.73	5.64	5.64	9.39
Total volume extracted	ml	6.30	13.81	21.54	27.18	32.82	42.21

Chemical Analysis

pH		6.75	6.45	6.82	6.65	6.72	7.24
Calcium	mg l^{-1} Ca	452	448	439	422	408	394
Magnesium	mg l^{-1} Mg	215	195	193	187	184	182
Sodium	mg l^{-1} Na	61.3	59.4	58.7	55.4	52.7	43.3
Potassium	mg l^{-1} K	62.8	63.2	55.8	46.7	37.2	20.9
Total alkalinity	mg l^{-1} HCO_3	<88	<88	<88	<88	<88	<88
Chloride	mg l^{-1} Cl	3.52	1.85	1.84	1.46	1.88	2.50
Sulphate	mg l^{-1} SO_4	2108	1989	1937	1869	1831	1706
Nitrate	mg l^{-1} NO_3	0.850	0.259	<0.100	0.361	<0.100	<0.100
Bromide	mg l^{-1} Br	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100
Nitrite	mg l^{-1} NO_2	0.561	0.208	0.114	0.091	0.073	<0.050
Orthophosphate	mg l^{-1} HPO_4	<0.500	<0.500	<0.500	<0.500	<0.500	<0.500
Fluoride	mg l^{-1} F	0.535	0.790	0.917	0.943	1.01	0.817
Total organic carbon	mg l^{-1} C	40.9	20.0	13.7	13.4	<24.0	12.5
Total inorganic carbon	mg l^{-1} C	<6.00	<3.00	<3.00	<6.00	<12.0	3.93
Total phosphorus	mg l^{-1} P	0.096	<0.050	<0.050	0.058	0.074	<0.058
Total sulphur	mg l^{-1} S	705	685	684	657	605	577
Silicon	mg l^{-1} Si	12.4	13.7	15.0	16.5	18.3	18.1
Silicate	mg l^{-1} SiO_2	26.5	29.3	32.0	35.3	39.1	38.7
Barium	mg l^{-1} Ba	0.056	0.063	0.064	0.065	0.054	0.046
Strontium	mg l^{-1} Sr	6.62	3.65	3.28	2.97	2.86	2.70
Manganese	mg l^{-1} Mn	3.56	4.80	5.14	5.30	5.16	5.41
Total iron	mg l^{-1} Fe	0.257	0.647	0.821	1.61	2.35	7.02
Aluminium	mg l^{-1} Al	0.103	0.159	0.169	0.349	0.304	0.128
Lithium	mg l^{-1} Li	1.19	1.18	1.22	1.24	1.28	1.40
Uranium	µg l^{-1} U	4.43	9.48	6.65	14.5	15.0	11.6
Thorium	µg l^{-1} Th	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Ionic charge balance	%	0.90	1.66	1.97	1.70	1.11	2.89

Summary of Squeezing and Chemical Analysis Test Data For Sample Blank

Client	GRS
Core Sample Number	Blank
BGS Pore-Water Numbers	10843-0015
Test Type	n/a

Geotechnical Test Data

Date squeezing started		n/a
Initial sample weight	g	n/a

Fraction		1
BGS Pore-Water Number		10843-0015
Test time	h	n/a
Total time	h	n/a
Minimum stress	MPa	n/a
Maximum stress	MPa	n/a
Initial moisture content	%	n/a
Final moisture content	%	n/a
Volume extracted	ml	n/a
Total volume extracted	ml	n/a

Chemical Analysis

pH		5.37
Calcium	mg l^{-1} Ca	<0.100
Magnesium	mg l^{-1} Mg	0.018
Sodium	mg l^{-1} Na	<0.350
Potassium	mg l^{-1} K	<0.500
Total alkalinity	mg l^{-1} HCO_3	<22
Chloride	mg l^{-1} Cl	<0.050
Sulphate	mg l^{-1} SO_4	<0.050
Nitrate	mg l^{-1} NO_3	0.029
Bromide	mg l^{-1} Br	<0.020
Nitrite	mg l^{-1} NO_2	<0.010
Orthophosphate	mg l^{-1} HPO_4	<0.100
Fluoride	mg l^{-1} F	<0.010
Total organic carbon	mg l^{-1} C	<1.00
Total inorganic carbon	mg l^{-1} C	<0.50
Total phosphorus	mg l^{-1} P	<0.010
Total sulphur	mg l^{-1} S	0.061
Silicon	mg l^{-1} Si	<0.075
Silicate	mg l^{-1} SiO_2	<0.16
Barium	mg l^{-1} Ba	<0.002
Strontium	mg l^{-1} Sr	<0.002
Manganese	mg l^{-1} Mn	<0.002
Total iron	mg l^{-1} Fe	<0.010
Aluminium	mg l^{-1} Al	<0.010
Lithium	mg l^{-1} Li	<0.025
Uranium	µg l^{-1} U	<0.05
Thorium	µg l^{-1} Th	<0.2
Ionic charge balance	%	51.68

References

Most of the references listed below are held in the Library of the British Geological Survey at Keyworth, Nottingham. Copies of the references may be purchased from the Library subject to the current copyright legislation.

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