P-05-79

Forsmark site investigation

Hydrochemical characterisation in KFM05A

Results from an investigated section at 712.6–722.0 m

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March 2005

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ISSN 1651-4416 SKB P-05-79

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Keywords: Groundwater, Measurements on line, In-situ sampling, Measurements in-situ, Redox potential, Dissolved gas, Humic and fulvic acids, Colloids, Microbes, Chemical analyses, Isotope determinations, AP PF 400-04-84.

This report concerns a study which was conducted for SKB. The conclusions and viewpoints presented in the report are those of the authors and do not necessarily coincide with those of the client.

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Abstract

Complete chemical characterisation is the most extensive chemical investigation method performed in core drilled boreholes. The method entails pumping, measurements on-line and regular water sampling for chemical analyses in isolated borehole sections during approximately three weeks per section at a flow rate of between 50 and 200 mL/min.

The method has been used in one section of borehole KFM05A at 712.6–722.0 m. At the completion of the investigation, it was suspected that water from above the upper packer intruded into the test section during the pumping and measurement period. The indications were; 1) the pressure measurements did not show any drawdown, 2) the water yield was unexpectedly large and 3) the chloride concentration in the water samples was unexpectedly low. Further testing was performed using modified packer positions. The section length was increased to 20 m in order to include a more fractured part of the borehole and two alternative positions of the packers where tested. However, the same pressure pattern was obtained. After three unsuccessful attempts to investigate the water bearing zone at approximately 720 m, the equipment was lifted to the water-yielding zone at 265 m. Despite a determined hydraulic transmissivity of 1.9×10^{-8} m²/s from the differential flow logging /6/, for the zone at 265 m, the pumped flow was not more than a few mL/min at a drawdown of approximately 50 m and the investigation was interrupted.

The collected water samples and the conducted measurements at 712.6–722.0 m in KFM05A are not regarded as representative for the sampled water-bearing section. Some results are however obtained, including on-line measurements of redox potential, pH, dissolved oxygen, electric conductivity and water temperature in the borehole section as well as chemical analyses of major constituents. Gas content and composition, microbes, inorganic colloids as well as humic and fulvic acids were not investigated. The water composition was stable during the entire pumping and sampling period. The chloride concentrations amounted to approximately 4,500 mg/L, which is lower than the expected value (> 5,500 mg/L). However, the redox potential measurements show reducing conditions and it can be concluded that oxygen is consumed rapidly in the water column in the borehole also during open-hole conditions. All the six redox electrodes stabilised at similar values.

Sammanfattning

Fullständig kemikarakterisering är den mest omfattande kemiska undersökningsmetoden för kärnborrhål. Metoden innebär pumpning, mätning on-line och regelbunden vattenprovtagning för kemiska analyser i avgränsade borrhålssektioner under cirka tre veckor per sektion och vid ett pumpflöde på mellan 50 och 200 mL/min.

Metoden har utförts i en sektion av borrhålet KFM05A vid 712.6–722.0 m. Under genomförandet uppstod misstanken att vatten från vattenpelaren i borrhålet ovanför den övre manschetten hade dragits in i testsektionen under pump- och mätperioden. Indikationer på detta förhållande var 1) att tryckmätningarna inte visade på någon avsänkning, 2) vattentillgången var oväntat stor och 3) kloridhalten i vattenproven var lägre än förväntat. Ytterligare två tester gjordes med förändrade manschettplaceringar. Sektionslängden utökades till 20 m för att inkludera ett mera uppsprucket avsnitt av borrhålet inom sektionen. Tryckbilden blev emellertid densamma. Efter de två mindre lyckade försöken att undersöka den vattenförande zonen vid ca 720 m, lyftes utrustningen till en vattenförande zon vid 265 m. Trots att den hydrauliska transmissiviteten bestämts till 1.9×10^{-8} m²/s genom differensflödesloggning /6/ för zonen vid 265 m, erhölls ett flöde på endast några mL/min vid en avsänkning på 50 m och undersökningen fick avbrytas.

De vattenprov som har tagits och de mätningar som har genomförts i sektionen 712.6–722.0 m i KFM05A bedöms inte vara representativa för den provtagna vattenförande zonen. Vissa resultat har dock erhållits som omfattar mätningar on-line av redoxpotential, pH, löst syre, elektrisk konduktivitet och vattentemperatur i borrhålssektionen liksom kemiska analyser av huvudkomponenter. Gasmängd och sammansättning, bakterier, oorganiska kolloider samt humus- och fulvosyror i grundvattnet undersöktes däremot inte. Vattensammansättningen var stabil under pump/provtagningsperioden. Kloridkoncentrationen uppgick till cirka 4 500 mg/L, vilket är lägre än förväntat för representativa prov (förväntat värde > 5 500 mg/L). Redoxpotentialmätningarna däremot, påvisar reducerande förhållanden vilket visar att syret förbrukas snabbt i vattenpelaren i borrhålet även under "öppet hål" förhållanden. Alla sex elektroderna visade stabila och överensstämmande värden.

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1 Introduction

This document reports performance and results of the activity: "Complete chemical characterisation in KFM05A" within the site investigation programme at Forsmark /1/. The report presents hydrogeochemical data from the borehole section at 712.6–722.0 m. The field work was carried out during October 2004. The SKB internal controlling documents for the activity are listed in Table 1-1. The obtained data from the activity are reported to SICADA, see Table 1-2.

Borehole KFM05A is the fifth deep (1,002.7 m) telescopic borehole drilled at Forsmark /2/. The location of the borehole and the current drill sites for deep telescopic boreholes within the investigation area are shown in Figure 1-1, whereas Figure 1-2 is a detailed map of drill site DS5 with KFM05A and nearby situated percussion drilled boreholes in rock and soil. The borehole has an inclination of 60° from the horizontal plane and is directed towards east. The borehole section between 0–100 m is percussion drilled and has a stainless steel casing with an internal diameter of 200 mm, whereas the 100.4–1,002.7 m interval is core drilled with a diameter of 77 mm. The design of the borehole is presented in Appendix 1.

The borehole is of the so-called SKB chemical-type and the drilling was performed according to method descriptions MD 620.003 (Method description for drilling cored boreholes) and MD 610.003 (Method description for percussion drilling). An SKB chemical-type borehole requires cleaning procedures to be carried out on all equipment to be used in the borehole, both during and after drilling, according to level 2 in the cleaning instructions outlined in MD 600.004 (Instruktion för rengöring av borrhålsutrustning och viss markbaserad utrustning). The method descriptions and instructions are SKB internal controlling documents.

Activity plan	Number	Version
Fullständig kemikarakterisering med mobilt fältlaboratorium i KFM05A.	AP PF 400-04-84	1.0
Method descriptions	Number	Version
Metodbeskrivning för fullständig kemikarakterisering med mobilt fältlaboratorium.	SKB MD 430.017	1.0

Table 1-1. Controlling documents for performance of the activity.

Table 1-2. Data references.

Subactivity	Database	Identity number
Water sampling series	SICADA	Field note Forsmark 422
Chemmac measurement	SICADA	Field note Forsmark 422



Figure 1-1. The investigation area at Forsmark including the candidate area selected for more detailed investigations. The core boreholes, KFM01A to KFM08B, as well as the percussion borehole HFM13 (supply well for flushing water to the drilling of KFM05A), are marked with circles.



Figure 1-2. Location of the cored borehole KFM05A, the percussion boreholes and monitoring wells at drill site DS5.

2 Objectives and scope

Complete chemical characterisation is the most extensive chemical investigation method performed in core drilled boreholes. The method is intended to provide as complete information as possible about the groundwater chemical conditions in individual waterbearing fractures or minor fracture zones. Considerable effort is put on obtaining representative samples from a limited rock volume. Careful pumping and continuous control of the pressure in the sampled borehole section, as well as above the section, is maintained in order to minimise the risk of mixing with water from other fracture systems.

The north-western part of the candidate area is given priority for the continuing investigation in Forsmark. In the investigation so far there are no chemical data from below depths of 200 m. Further, there are reasons to believe that very few water-yielding fractures will be found at depth in the forthcoming boreholes. Therefore, every opportunity to conduct chemical investigations at depth needs to be considered, despite that the hydraulic transmissivity may be at the lower practical limit as in the fracture zone at 720 m.

The analysis program is carried out according to SKB chemistry class 4 and class 5, including all options. Further, pH, redox potential (Eh) and water temperature are measured in flow-through cells in the pumped borehole section as well as at the surface. The flow-through cell at the surface also measures electric conductivity and dissolved oxygen. Normally, samples are also collected in situ in the borehole section for determination of gas content and composition, microbe content and characterisation as well as for determination of colloid content and composition. Due to sampling problems, see Chapter 5, these analyses were not conducted. For the same reason, the analyses following fractionation of organic acids and inorganic species (DOC and ICP) as well as after enrichment of the organic acids (∂^{13} C and pmC) were not performed.

3 Background

3.1 Flushing water history

The percussion drilled borehole HFM13 served as a supply well for the flushing water used to drill borehole KFM05A /3/. The chemical composition of the flushing water was checked before and during use. The chemical data from the supply well HFM13 are reported in /4/. The core drilling of the 1,000 m long borehole consumed 1,090 m³ of flushing water and the volume of returned water pumped from the borehole during drilling was 3,500 m³. The nominal concentration of the dye Uranine, added as a tracer to mark the flushing water, was 0.2 mg/L. Automatic dosing equipment for injection of Uranine was installed in the flushing water system. The Uranine concentration in the flushing water was checked regularly during drilling and a total of 125 samples were analysed. The average Uranine concentration in the entire sample series was 0.17 ± 0.07 mg/L. If the period of malfunction between 2004-03-12 and 2004-03-24 is omitted, the average is 0.20 ± 0.05 mg/L. The Uranine concentrations in the flushing water are presented in Figure 3-1. Further, the amount of Uranine added to the borehole via the flushing water and the estimated amount recovered in the return water is given in Table 3-1.

It can be concluded from the Uranine budget in Table 3-1 that the increased sampling frequency, compared with earlier boreholes, resulted in more reliable calculations. Further, the budget indicates that the major part of the flushing water added to the borehole was recovered.



Figure 3-1. Uranine concentrations in the flushing water and in the return water versus borehole length. The addition of Uranine was done using the automatic dosing equipment which is controlled by a flow meter. A period of malfunction occurred between 600 and 750 m borehole length.

Table 3-1. Amount of Uranine added to KFM05A via the flushing water during core drilling and the amount recovered from the mammoth pumping.

Uranine	(g)
Added, according to the log book.	170
Added, calculated from the average Uranine concentration and the total volume of flushing water.	185
Recovered, estimated from the average Uranine concentration and the total volume of return water.	175

As borehole KFM05A is of SKB chemical-type, the following special precautions were taken in order to minimise contamination via the flushing water:

- The supply well was also of SKB chemical-type.
- Borehole HFM13 was selected to supply flushing water because of the low concentration of total organic carbon (TOC). The concentration should preferably be below 5 mg/L and the concentration in the samples collected in HFM13 was showing a decreasing trend with time from 6 to 3 mg/L.
- A dosing equipment for Uranine was installed, thereby removing the need for an in-line flushing water storage tank after the UV-system.

Analyses of the microbe content in the flushing water, before and after the UV-system, was performed at four occasions during drilling; 1) at the drilling start just after cleaning the system, 2) when half the borehole was drilled before and 3) after cleaning the system and, 4) without cleaning, shortly before completing the borehole. The results showed that the amounts of algae and bacteria in the flushing water entering the borehole were low at all sampling occasions /5/.

3.2 Previous events and activities in the borehole

KFM05A is a SKB chemical-type telescopic borehole and thus specially intended for complete hydrochemical characterisation. Only those investigations that are necessary in order to select borehole sections for hydrochemical investigations are carried out in the borehole prior to the chemistry campaign. The more equipment that is used in the borehole, the greater is the risk of contamination and effects on the in situ microbiological conditions. The activities/investigations performed in KFM05A prior to the chemistry campaign are listed in Table 3-2 below.

Activities performed	Date of completion	Length or section (m)	Comment
Percussion drilling	2003-12-16	0–100.35	
Core drilling	2004-05-05 2004-02-17	100.35–1,002.71	HFM13 was the source of flushing water for drilling the cored part of KFM05A. HFM13 is an SKB chemical-type borehole /2, 3/.
Sampling of water pumped to the surface, class 3. The WL-sond was not used.		100–121.6	SKB sample no 8332, first strike water from the first water yielding fracture zone that was intersected during drilling.
Flushing water treatment	-	-	Automatic dosing of Uranine was used during drilling of KFM05A. In this way no in-line storage tank was needed after the UV-system /2/.
Geophysical logging	2004-05-08	110–1,000.4	_
DIFF-flow logging	2004-06-02	100–1,000	/6/
BIPS-logging	2004-06-04	108–995	Several logging occasions, the last occasion for logging of the entire borehole is given /7/.
Geophysical logging	2004-06-08	110–1,000	Radar directional antenna
Transducer installation	2004-06-11 2004-09-09		To register possible responses from the drilling of KFM06A.
Extensive microbe control	2004-02-17 2004-03-25 2004-03-26 2004-04-14	-	Check of microbe content in flushing water four times, at the start, in the middle and at the end of the drilling period /5/.
Hydrochemical logging, not performed	-	-	Not performed
Hydrogeochemical characterisation	2004-10-23	-	Presented in this report.

Table 3-2. Activities performed in borehole KFM05A prior to the chemical characterisation.

3.3 Choice of borehole sections

A very dominant water-bearing zone (108–125 m) was identified early during core drilling of borehole KFM05A. The water yield was very large which made it difficult to identify additional inflow zones further down in the borehole during drilling. However, results from the subsequent difference flow logging showed some minor water-bearing zones that could possibly be investigated, see Table 3-3 and Appendix 2.

A rather deep water bearing zone was indicated at 710 to 720 m, although the indication was uncertain. As water at depth is very rare in this part of the candidate area, it was considered very important to make all possible efforts in order to investigate this zone, despite that the calculated hydraulic transmissivity (T) from the difference flow logging did not quite reach the practical limit to permit sampling (the range $T = 10^{-8}$ to $T = 10^{-6}$ m²/s is optimal). The borehole wall showed fractures and did not allow a shorter section length than 10 m, resulting in a section at 712.6 to 722.0 m, see Appendix 2 and 3.

Further, the second deepest zone at 265 m was selected as a second borehole section for sampling. The calculated hydraulic transmissivity (T) was large enough according to the difference flow logging. The investigated section length was 4.9 m, 262.0–266.9 m.

Table 3-3. Water-yielding fractures/fracture zones identified from difference flow logging of KFM05A /6/. Selected fractures/fracture zones are given in bold text. Hydraulic transmissivity calculated from difference flow logging and from injection tests /8/ are compared. The injection tests were performed after the chemical investigation.

Borehole length (m)	Difference flow logging, T (m²/s) /6/	Injection test, T (m²/s) /8/	Comments
108–125	1×10 ⁻³	Section 116.5–126.5 m 2.5×10 ⁻⁶	Difference flow logging: Several fractures, yielding from 10 to 800 L/h each. A fracture with extremely high T-value $(1 \times 10^{-3} \text{ m}^2/\text{s})$ was found at 108.9 m /6/. The inflow from the fracture was somewhat languished at a small drawdown. This fracture was not measured during the injections tests.
			Injection test: Section length 20 m. Measurements above 116.5 m are not performed.
160–175	2.9×10 ⁻⁷	Section 156.5–176.5 m 1.0 ×10 ⁻⁶	Difference flow logging: Two fractures, giving about 3 L/h each, T \approx 3×10 ⁻⁷ m ² /s. The lower fracture is situated at 175.6 m /6/.
			Injection test: Section length 20 m.
262.0–266.9	1.9×10 ^{_8}	Section 254.0–274.0 m 1.4×10 ⁻⁸	<i>Difference flow logging</i> : 0.36 L/h. According to DIFF /6/ the fracture is situated at 264.4 m.
			<i>Injection test</i> : Section length 20 m. The fractures are situated between 264–269 m.
712.6–722.0	8.6×10 ⁻⁹	Section 706.5–806.5 m 1.0×10 ⁻¹⁰	<i>Difference flow logging</i> : 0.2 L/h. According to DIFF /6/ the fracture is situated at 720.0 m.
			<i>Injection test</i> : Section length 100 m. Only 100 m sections were measured at this depth.

4 Equipment

4.1 The mobile field laboratory

The mobile field laboratories used by SKB for water sampling and downhole measurements consist of a laboratory unit, a hose unit with downhole equipment and a Chemmac measurement system. The equipment is presented schematically in Figure 4-1. It is also possible to include a separate computer unit (MYC). The different parts of the system are described in the SKB internal controlling documents SKB MD 434.004, 434.005, 434.006, 434.007 and SKB MD 433.018 (Mätsystembeskrivningar för mobil kemienhet allmän del, slangvagn, borrhålsutrustning, Chemmac mätsystem och dataapplikation).

The Chemmac measurement facilities include communication systems, a measurement application and flow-through cells with electrodes and sensors at the ground surface (surface Chemmac) and in the borehole (borehole Chemmac).

The borehole equipment consists of inflatable packers, pump, borehole Chemmac and the in situ sampling unit (PVP) allowing measurement (borehole Chemmac) and sampling in situ in the borehole section (PVP sampling unit). The four sample portions collected with the PVP sampling unit maintain the pressure from the borehole section when lifted to the surface. The portions are used for colloid filtration, gas analyses and microbe investigations.

The mobile units used in borehole KFM05A, section 712.6–722.0 m, consisted of the hose unit S3, the laboratory unit L3 and the MYC 3 unit for computer work. The electric connections to the umbilical hose were modified and later on the entire umbilical hose was replaced.



Figure 4-1. The mobile chemistry laboratory including laboratory unit, hose unit and downhole equipment. The configuration of the downhole units in the borehole can be varied depending on desired section length. However, the in-situ water sampler must always be positioned first in the sample water path.

5 Performance

5.1 General

Complete chemical characterisation of two sections in borehole KFM05A was planned according to activity plan AP PF 400-04-84 following the method described in SKB MD 430.017 (Metodbeskrivning för fullständig kemikarakterisering med mobilt fältlaboratorium, SKB internal controlling document). The chemical investigation of the first section continued according to plan although it became obvious with time that, due to leakage, the pumped water consisted of water from the water column above the upper packer. Two more attempts were made in order to obtain representative samples from the zone at 720 m. First, the section length was increased to 20 m to include a fractured part of the borehole and second, the packer positions were changed one metre. However, no drawdown was noticed in either case. Pumping above the upper packer caused a pressure response in the section which indicated a connection between the water column above the packer and that of the borehole section. An overview of the investigation sequence is given in Table 5-1.

Start date/ Stop date	Investigation	Section (m)	Comment
2004-09-23/ 2004-09-27	Interrupted test, Complete chemical characterisation	712.6–722.0	Leakage in the electric connection to the umbilical hose.
2004-09-27/ 2004-10-19	Complete chemical characterisation	712.6–722.0	<i>In situ</i> sampling failed, only small water volumes in the PVB-containers. Water regarded as not representative. No drawdown and leakage of borehole water from above the upper packer was suspected. Poor quality of borehole wall. Pumped volume from section: 5.8 m ³ .
2004-10-20/	Interrupted test, repeated	702.5–722.2	20 m section. No drawdown. Pumping
2004-10-26	Complete chemical characterisation	(701.5–721.2)	was performed in the upper part of the borehole and a pressure response was noticed in the borehole section, see Appendix 5. Connection between water column above upper packer and borehole section. The packer positions were changed one metre but the results were the same. Pumped volume: 1.6 m ³ .
2004-10-26/ 2004-11-01	Interrupted test, Complete chemical characterisation	262.0–266.9	Limited flow of only a few mL/min and drawdown of 50 m. The pumping was interrupted due to low water yield. Pumped volume: 0.01 m ³ .

Table 5-1. Investigation sequence in KFM05A.

5.2 Overview of field work procedure

A short chronological summary of the different steps that constitute chemical characterisation of groundwater in a borehole section is given below.

The preparations conducted before the downhole equipment is lowered in the borehole include:

- The inside of the umbilical hose (the sample water channel) is rinsed with de-ionised, deoxygenated water. The sample water channel is then filled with de-ionised and deoxygenated water prior to lowering.
- Cleaning and preparation of the four sample containers (PVB) belonging to the in situ water sampling unit (PVP). The containers are cleaned using 70% denatured ethanol. One of the containers is used for microbe sampling and sterile conditions are desirable. The containers are purged with nitrogen gas and a small nitrogen gas pressure is maintained in the containers. The magnitude of the pressure depends on the depth of the section to be sampled.
- Calibration of the pH and redox electrodes in the borehole Chemmac.

The different downhole units are assembled during lowering of the equipment in the borehole and the following steps are taken:

- The outside of the umbilical hose is cleaned with 70% denatured ethanol (SKB MD 600.004).
- Calibration of the umbilical hose length is conducted at least once for each borehole. For this, a length mark detector unit (caliper) is mounted together with the regular downhole equipment. The length mark detector indicates length calibration marks milled into the borehole wall at almost every 50 m along the borehole /2/. At each indication, a reading is made of the corresponding length mark on the umbilical hose. The correct distance to each length mark is obtained from the SICADA database.

When the pump is started and the packers are inflated at the desired positions in the borehole, a pumping and measurement period begins. Typical measures taken and activities carried out during this period are:

- Calibration of the pH and redox electrodes as well as the electric conductivity and oxygen sensors in the surface Chemmac is conducted when the pumped water from the borehole section has reached the surface.
- Careful attention is paid in order to make sure that the packed off section is well isolated from the rest of the borehole. A significant drawdown in the section during pumping is one indication that the section is isolated. Leakage would cause pumping of water from the borehole column above and/or below the packers and not only from the fracture zone of interest. However, the drawdown in the borehole section must not be too large since the larger the drawdown, the larger the bedrock volume affected by the pumping and the risk of mixing with water from other fracture systems increases. The pumping flow rate is adjusted depending on the flow yield from the fracture or fracture zone (to between 50 and 300 mL/min) and maintained more or less constant during the pumping and measurement period.
- Water samples are collected regularly once or twice a week during the pumping period. Changes in water composition are monitored by conductivity measurements and by immediate analyses at the site.

- Enrichment of humic and fulvic acids is conducted for as long time as possible in each section. The time needed depends on the carbon concentration in the water and the flow rate through the ion exchanger. Generally, a period of at least two weeks is needed to collect the amount of carbon required to determine $\partial^{13}C$ and pmC.
- Fractionation of humic and fulvic acids, as well as inorganic species to determine the size distribution, is performed at the end of the pumping period.
- A decision when to terminate the sampling work in the section is made during a suitable stage of the pumping and measurement period. The investigation might be prolonged if the concentration of flushing water exceeds 1% or if the redox potential measurements have not reached stable values. A final SKB Class 5 sample including all options is collected the day before termination.

Completion of the investigation in the section and lifting of the borehole equipment entails:

- Collection of in situ sample portions prior to lifting the equipment. The valves to the PVB sampling containers in the borehole section are opened in order to rinse the system and fill the containers. After a few hours, the valves are closed and the water sample portions for analyses of colloids, dissolved gases and microbes are secured.
- Following stopping of the borehole pump and deflation of the packers, the equipment is lifted and the different downhole units are de-assembled.
- Calibration of the electrodes in the borehole Chemmac and surface Chemmac. The final calibration for a section can be used as the initial calibration for the next section.

5.3 Performance in section 712.6–722.0 m

The investigations in section 712.6–722.0 m were performed using the following configuration of the downhole equipment, from the top: umbilical hose, length mark detector, borehole Chemmac, upper packer, borehole pump, in situ water sampler (PVP), extension dummy and lower packer, see Appendix 4. The pressures above and within the section were measured by the borehole Chemmac unit and the PVP water sampling unit, respectively.

The pumping was performed at a flow rate of about 190–200 mL/min. No drawdown was observed at the initial stage, only a pressure fluctuation caused by the work of the piston in the borehole pump. A diagram showing the pressures within and above the borehole section and the flow rate during the pumping/measurement period is given in Appendix 5, Figure A5-1. The events during the investigation are listed in Table 5-2.

Date	Events Improvement/deviation	SKB sample no
040915	Borehole equipment (S3) repair due to failure of electric communication; the umbilical hose was replaced.	
040921	Level indicator; identification of faults and reparation.	
	Calibration of borehole Chemmac.	
040922	In situ water sampler, identification of faults and repair.	
040923	Lowering of downhole equipment (712.55–722.02 m).	
	Start of Chemmac measurements.	
040924	Calibration of surface Chemmac.	
	Water sampling: SKB class 4	8643
040927	High electric power consumption.	
	Lifting.	
	Lowering of downhole equipment (712.55–722.02 m).	
040928	Water sampling: Uranine only.	8645
	Humic and fulvic acids; enrichment start.	
040930	Water sampling: SKB class 4.	8646
041004	Electrical failure at drill site 5; momentary pump stop	
041005	Water sampling: SKB class 5.	8648
041008	Water sampling: SKB class 4.	8649
041011	Water sampling: SKB class 5.	8679
041015	Change of flow rate from about 200 mL/min to 80 mL/min.	
	Humic and fulvic acids; fractionation 1 kD and 5 kD.	8681
041018	Water sampling: SKB class 5, all options.	8681
	PVP-sampler: opening of valve at 17:55.	
041019	PVP-sampler: closure of valve at 05:10.	
	End of Chemmac measurements.	
	Lifting.	
	No sampling of dissolved gas, microbes or colloids.	
	Calibration of borehole Chemmac.	
	Humic and fulvic acids; enrichment eluation.	8681

Table 5-2. Events during the pumping/measurement period in section 712.6–722.0 m.

5.4 Performance in section 702.5–722.2 (701.5–721.2) m

During the investigation in the extended section between 702.5–722.2 m, the configuration of the downhole equipment in the borehole was, from the top; umbilical hose, upper packer, length mark detector, borehole Chemmac, borehole pump, in situ water sampler (PVP), extension dummies and lower packer. The pressure within the section was measured by the borehole Chemmac unit and the PVP water sampling unit.

Pumping was performed at a flow rate of about 200 mL/min and no drawdown was observed, despite the fact that the fractured and uneven part of the borehole wall seemed to be within the section. A pump was then installed in the upper part of the borehole, causing a drawdown above the packer. A pressure response was observed in the borehole section which verified the connection between the water column above the packer and the section. A diagram showing the pressures in the borehole section and the flow rate during the pumping/measurement period is presented in Appendix 5, Figure A5-2. Further, the effect of the pumping performed in the upper part of the borehole is demonstrated in Figure A5-3. The pressures in section 701.5–721.2 are shown in Figure A5-4.

The events during the investigation are listed in Table 5-3.

Date	Events Improvement/deviation	SKB sample no
041020	Lowering of downhole equipment (702.50–722.18 m).	
	Start of Chemmac measurements.	
	Calibration of surface Chemmac.	
041022	Water sampling: SKB class 4.	8693
041025	Stop of borehole pump due to pump test.	
	Lowering of an additional pump, in the upper part of the borehole, to approximately 15 m.	
	Start of pump, in the upper part of the borehole, at 10:48, flow about 100 mL/min.	
	Lowering of equipment for measurement of ground water level.	
	Ground water level at 10:52; 9.00 m from top of casing.	
	Ground water level at 10:54; 9.16 m from top of casing.	
	Pump, in the upper part of the borehole, stopped at 10:54.	
	End of Chemmac measurements.	
	Lifting.	
041025	Lowering of downhole equipment (701.50–721.18 m).	
	Start of Chemmac measurements.	
041026	End of Chemmac measurements.	
	Lifting.	

Table 5-3.	Events during the pumping/measurement period in section 7	02.5-722.2
(701.5-721	1.2) m.	

5.5 Performance in section 262.0–266.9 m

The investigations in section 262.0–266.9 m were performed using the following configuration of the downhole equipment, from the top; umbilical hose, borehole pump, borehole Chemmac, upper packer, in situ water sampler (PVP) and lower packer. The pressures above and within the section were measured by the borehole Chemmac unit and the PVP water sampling unit, respectively.

The pumping was performed at a flow rate of from 40 mL/min down to 2 mL/min during the first two days and below 1 mL/min the remaining three days before terminating the investigation in the section. A diagram showing the pressures in and above the borehole section and the flow rate during the pumping/ measurement period is presented in Appendix 5, Figure A5-5.

The events during the investigation are listed in Table 5-4.

5.6 Water sampling, sample treatment and analyses

The pumped water from the borehole section is led into the laboratory unit where sampling and sample filtration is carried out. Filtration of sample portions is performed on-line by connecting the filter holders directly to the water outlet. A water sample is defined as water collected during one day and consists of several sample portions, labelled with the same sample number.

An overview of sample treatment and analysis methods is given in Appendix 7. The routines are applicable independently of sampling method or type of sampling object.

In situ water samples were collected but not sent to the laboratories because they were not considered representative for the sampled fracture zone. For the same reason, neither enrichment of humic and fulvic acids for carbon isotope determinations nor fractionation of inorganic species and organic acids were performed.

Date	Events Improvement/deviation	SKB sample no
041026	Lowering of downhole equipment (262.00-266.85 m).	
	Start of Chemmac measurements.	
041028	Bore hole pump stopped.	
041101	End of Chemmac measurements.	
	Calibration of surface Chemmac.	
	Lifting.	
	Calibration of borehole Chemmac.	

Table 5-4. Events during the pumping/measurement period in section 262.0–266.9 m.

6 Nonconformities

During the hydrochemical investigation of borehole sections in KFM05A, a number of problems occurred and no representative data were obtained. The following points are worth noting:

- Sampled water is not representative of the water bearing fracture zone at 712.6 to 722.0 m. During the sampling and measurement period in the first section, it became clear that water from the borehole column above the upper packer intruded into the test section. The indications were; 1) the pressure measurements did not show any drawdown, 2) the water yield was unexpectedly large and 3) the chloride concentration in the water samples was unexpectedly low. Two more tests were performed with different packer positions. The section length was increased to 20 m in order to include a more fractured part of the borehole and two different positions of the packers were tested. However, the same pressure patterns were obtained. The hydraulic connection between the borehole above the upper packer and the test section was verified by pumping in the upper part of the borehole while measuring the pressure in the test section. The analytical data from section 712.6–722.0 m are registered in the database SICADA and a new water type notation is introduced saying; not representative water. The measurement data are stored and the unsuccessful pumping is commented.
- The section at 262.0–266.9 m did not yield enough water. The pumping was interrupted because the pumped flow rate was merely a few mL/min at a drawdown of 50 m which is the maximum allowed value (50 m) for the equipment.
- The allowed upper limit for flushing water content, 1%, was exceeded. However, the flushing water situation in the borehole was improved compared with previous boreholes. The samples collected in sections 712.6–722.0 showed a decreasing trend from 4 to 2%. These values are low, considering that most of the water originated from the present water column in the borehole and not from the fracture zone.
- The enrichment and fractionation of humic and fulvic acids were performed in the section at 712.6 to 722.0, but the samples were not sent for analysis due to that the water in the section was not representative. No pmC, ∂¹³C or fractionation data will therefore be available from this borehole.
- Gas content and composition, microbes and inorganic colloids were not determined.

7 Data handling and interpretation

7.1 Chemmac measurement data

The processing of Chemmac data are described in SKB MD 434.007-02 (Mätsystembeskrivning för Chemmac mätsystem, SKB internal controlling document, in progress). The following routines for Chemmac measurement data are generally applied.

7.1.1 Data file types and calculation software

The on-line measurements in a borehole section produce the following types of raw data files:

- Calibration files from calibration measurements (*.CRB) and corresponding comment files (*.CI). The files are used for calculation of calibration constants (pH and Eh) and the calibration factor (electric conductivity). For surface Chemmac ten *.CRB and ten *.CI files are produced, and for borehole Chemmac six *.CRB and six *.CI files.
- Raw data file containing the logged measurements from the borehole section and the surface (*K.MRB) as well as a corresponding comment file (*.MI). The logged voltage values need to be converted to pH and Eh values (also in mV) using the calibration constants obtained from calibration.
- Measurement file including equipment and environment parameters (*O.MRB), such as power consumption in the downhole Chemmac unit and temperature inside the hose unit.

The original raw data files are stored in the SICADA file archive. These data are further evaluated to obtain pH and redox potential values and to correct the electric conductivity values using the specially designed calculation software (Hilda). The resulting files containing calculated and evaluated values as well as comments on the performance are:

- A file *constants.mio containing all of the calculated calibration constants (one constant for each electrode in each buffer solution). The file is stored in the SICADA file archive and is useful for following the development of single electrodes.
- A file *measurements.mio containing the calculated and evaluated measurement values (pH, redox potential, electric conductivity and water temperature). The data from the file are exported to the data tables "redox" and "ph_cond" in SICADA. As the file also contains some measured parameters that are not included in the tables mentioned above (e.g. pressure registrations), the complete file is also stored in the SICADA file archive.
- A file *comments.mio containing comments on the fieldwork and the calculation/ evaluation. The comments in the file are imported as activity comments in SICADA.

7.1.2 Calculation and evaluation of pH and redox potential

The redox potential is measured by three electrodes at the surface and three in the borehole section. In addition, pH is measured by two electrodes at the surface and two in the borehole section. The redox and pH registrations are logged each hour during a measurement period of approximately three weeks and a calibration is performed before and after the measurement period. The treatment of the raw data includes the following steps:

- Calculation and choice of calibration constants.
- Calculation of one pH and one redox potential sequence for each electrode (i.e. three or six redox electrodes and two or four pH electrodes).
- Determination of representative pH and redox potential values as well as estimated measurement uncertainties for the investigated borehole section.

One calibration constant is selected for each electrode using one of the following alternatives:

- Case 1: Calculation of the average calibration constant value and the standard deviation. The initial and the final calibration measurements results in four constants for each redox electrode (in pH 4 and pH 7 buffer solutions) and six constants for each pH electrode (in pH 4, 7 and 10 buffer solutions).
- Case 2: The calibration constant obtained from the initial calibration measurement at pH 7 is selected since it is closest to the pH of the borehole water. This alternative is chosen if the calibration constants obtained in the different buffers show a large variation in value (generally a difference larger than 20 mV between the highest and the lowest value). The standard deviation is calculated in the same way as in Case 1.
- Case 3: If the final calibration constants turn out to be very different (more than 20 mV) from the initial constants, a linear drift correction is needed. The reason is most often a drift in the reference electrode. The average values and standard deviations are calculated for the initial and the final calibration constants separately and a linear correction is made between the selected initial and the selected final constant. The higher of the two standard deviation values is used in the estimation of the total measurement uncertainty.

The values in the measurement raw data file are converted to pH and Eh measurement sequences for each pH and redox electrode using the calibration constant selected as stated above.

The next step is to choose a logging occasion in a stable part of the measurement period and select a representative result for each electrode. The average values are calculated for each electrode group in order to obtain one representative value of redox potential, pH (borehole Chemmac) and pH (surface Chemmac), respectively. Obviously erroneous electrodes are omitted. The corresponding total measurement uncertainties are estimated using the standard deviations of the calibration constants and the standard deviations of the Eh and the pH values obtained by the different sets of electrodes. It is useful to evaluate pH at the surface and pH in the borehole section separately, since pH in the pumped water might differ from the pH measured in the borehole section. This is due to changing gas pressure conditions which affects the carbonate system.

Factors considered when evaluating the measurement uncertainties in pH and redox potential (Eh) values are:

- Difference in calibration constants for each electrode and calibration/buffer solution.
- Drift in calibration constants between the initial and the final calibration.
- Stability in voltage value during the final part of the on-line measurement. A successful measurement shows no tendency of a slope.
- Agreement between the different pH and redox electrodes on the surface and in the downhole borehole Chemmac.
- Number of electrodes showing reasonable agreement. Obviously erroneous electrodes are excluded from the calculation.

7.2 Water analysis data

The following routines for quality control and data management are generally applied for hydrogeochemical analysis data, independently of sampling method or sampling object.

Several components are determined by more than one method and/or laboratory. Moreover, duplicate analyses by an independent laboratory are performed as a standard procedure on each fifth or tenth collected sample. All analytical results are stored in the SICADA database. The applied hierarchy path "Hydrochemistry/Hydrochemical investigation/ Analyses/Water in the database" contains two types of tables, raw data tables and primary data tables (final data tables).

Data on *basic water analyses* are inserted into the raw data tables for further evaluation. The evaluation results in a final reduced data set for each sample. These data sets are compiled in a primary data table named "water composition". The evaluation is based on:

- Comparison of the results from different laboratories and/or methods. The analyses are repeated if a large disparity is noted (generally more than 10%).
- Calculation of charge balance errors according to equation (1). Relative errors within ± 5% are considered acceptable (in surface waters ± 10%).

Relative error (%) =
$$100 \times \frac{\sum cations(equivalents) - \sum anions(equivalents)}{\sum cations(equivalents) + \sum anions(equivalents)}$$
 (1)

• General expert judgement of plausibility based on earlier results and experience.

All results from *special analyses of trace metals and isotopes* are inserted directly into primary data tables. In cases where the analyses are repeated or performed by more than one laboratory, a "best choice" notation will indicate those results which are considered most reliable.

An overview of the data management is given in Figure 7-1.



Figure 7-1. Overview of data management for hydrogeochemical data.

8 Results

8.1 Chemmac measurements

Chemmac measurements are reported for the only investigated section, 712.6–722.0 m, see Appendix 6. The pumped water is not considered representative for the water-yielding fracture zone in the section and therefore the evaluation of the measurement data sequences is simplified compared with the description in Chapter 7. Neither linear correction of the calibration constants nor calculations of measurement uncertainties are conducted. The values of Eh, pH, electric conductivity and dissolved oxygen are selected from the middle part of the diagram just as the Eh starts to level out, see Figure A6-1. Selected data from the measurements are given in Table 8-1.

Table 8-1.	Chemmac measurement	results	in	KFM05A.
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Borehole section [m]	Electric conductivity* [mS/m]	pH (surface chemmac)	pH (borehole chemmac)	Eh (borehole chemmac) [∞] [mV]	Dissolved oxygen*** [mg/L]
712.6–722.0	1,380	7.8	7.5	-274	0.00 ± 0.01

*The electric conductivity is measured between 0–10,000 mS/m with a resolution of 1% of the measurement interval. Used measurement interval: 2,000 mS/m.

** Average value calculated from the three borehole electrodes.

*** Measuring interval 0-15 mg/L, resolution 0.01 mg/L.

8.2 Water analyses

Basic water analyses are reported to SICADA (Field note no 422) for the sample series collected in section 712.6–722.0 m and for one sample collected in section 702.5–722.2. Trace metal analyses and isotope determination have not been performed. The water type of the samples in SICADA is denoted as "not representative water". Although the usefulness of the analytical data is doubtful, some information can be obtained regarding the situation in the borehole. Therefore some of the results are presented and commented on below.

The basic water analyses include the major constituents Na, K, Ca, Mg, S, SO_4^{2-} , Cl⁻, Si, Sr and HCO_3^- as well as the minor constituents Fe, Li, Mn, DOC, Br, F, HS⁻, I⁻ and NH₄⁺. Another important parameter is the flushing water content in each sample. The charge balance errors give an indication of the quality and uncertainty of the analyses of major constituents. The error did not exceed the acceptable level of \pm 5% in any of the cases. Furthermore, batch measurements of pH and electric conductivity are compared with the corresponding on-line Chemmac measurement values for section 712.6–722.0 m in Appendix 6.

The flushing water contents and the concentrations of sodium, calcium and chloride in the sample series are presented in diagrams in Figures 8-1 and 8-2, respectively. The flushing water content was rather constant and surprisingly low for samples representing the water column standing in the borehole. The low and practically constant concentrations of Na, Ca and Cl, strongly indicate that the pumped water originates from the water column in the borehole.



Figure 8-1. Flushing water content versus sampling date in borehole section 712.6–722.0 m in KFM05A.



Figure 8-2. Sodium, calcium and chloride concentration trends from sample series at 712.6–722.0 m in borehole KFM05A.

9 Summary and discussion

The complete chemical characterisation in KFM05A did not produce representative chemical data due to lower hydraulic transmissivity in the two selected water-yielding fractures than indicated by the initial flow logging. However, some useful information was obtained and some conclusions from the investigation can still be made. These are summarised below:

- At the investigation of the section 712.6–722.0 m, a combination of three factors caused sampling of water from the water column above the upper packer; 1) misunderstanding considering interpretation of drawdown 2) unreliable flow logging data and 3) the borehole wall is uneven and fractured for quite a long distance in the vicinity of the fracture zone which made it difficult to isolate a test section.
- Unusually low flushing water content (4%) already from the start of the sampling and a quickly stabilising redox potential at a plausible value indicated that representative sampling conditions were obtained. Therefore the pumping/sampling continued despite that the high pumping flow rate did not result in any significant drawdown.
- The hydraulic transmissivity calculated from flow logging data and from pumping with the chemistry equipment are always compared. The flow logging data are not always reliable, but if the results diverge considerably, the reason has to be investigated thoroughly.
- A useful method to check if there is a hydraulic connection between the test section and the water column above the upper packer is to pump the upper part of the borehole and measure pressure responses in the test section.
- The redox potential measurement by the borehole Chemmac shows that Eh stabilises at a negative value, comparable in size to representative water from deep fractures, also when mixed water from the borehole column is measured.
- The low flushing water content (from 4% to 2%) in the borehole shows that the pumping carried out to clean the borehole was efficient compared to earlier boreholes.
- The salinity was stable but considerably lower than expected during the pumping and sampling period, which is an indication of not representative samples.

10 References

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Design of cored borehole KFM05A

Technical data Borehole KFM05A



Selected results of difference flow logging in KFM05A



Figure A2-1. Borehole KFM05A: Hydraulic head and hydraulic transmissivity along borehole KFM05A /6/.

Forsmark, Borehole KFM05A

Flow meas	urement 2004-05-11 - 2004-06-02
Δ	Without pumping (L=5 m, dL=5 m), (Flow direction = into the hole)
\bigtriangledown	Without pumping (L=5 m, dL=5 m), (Flow direction = into the bedrock)
\triangle	With pumping (L=5 m, dL=5 m), (Flow direction = into the hole)
	Without pumping (L= 5 m), 2004-05-24 - 2004-05-26
	With pumping (L= 5 m), 2004-05-27 - 2004-05-29
	With pumping (L= 1 m), 2004-05-29 - 2004-05-30

- Lower limit of flow rate



Figure A2-2. Borehole KFM05A: Differential flow measurements from 260–280 m including the water bearing fracture zone at 264.4 m /6/.

Forsmark, Borehole KFM05A



Figure A2-3. Borehole KFM05A: Differential flow measurements from 700–720 m including the possible water bearing fracture zones at 717 to 720 m /6/.

Forsmark, Borehole KFM05A

		ow measurement 2004-05-11 - 2004-06-0 △ Without pumping (L=5 m, dL=5 m), (Flow direct ○ Without pumping (L=5 m, dL=5 m), (Flow direction ○ Without pumping (L=5 m, dL=5 m), (Flow direction ○ Without pumping (L=5 m), 2004-05-24 - 2004-05-24 - 2004-05-27 - 2004-05-27 - 2004-05-29 - 2004-05-20 - 2004	2 tion = into the hole) tion = into the bedrock) n = into the hole) 05-26 -29 -30
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		Flow rate (mL/h)	Single point resistance (ohm)

Figure A2-4. Borehole KFM05A: Differential flow measurements from 720–740 m including the possible water bearing fracture zone at about 720.0 m /6/.



Pictures from BIPS logging in KFM05A

Figure A3-1. Borehole KFM05A: BIPS logging from 264.0–264.6 m.



Figure A3-2. Borehole KFM05A: BIPS logging from 713.5–715.0 m.



Figure A3-3. Borehole KFM05A: BIPS logging from 712.0–713.5 m.



Figure A3-4. Borehole KFM05A: BIPS logging from 715.0–716.5 m.

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Figure A3-5. Borehole KFM05A: BIPS logging from 716.5–718.0 m.



Mesurement information, section 712.6-722.0

Figure A4-1. Electrode configuration, section 712.6-722.0 m.

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Figure A4-2. Configuration of downhole equipment, section 712.6-722.0 m.



Figure A4-3. Length calibration, section 712.6-722.0 m.

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		Filer - inkal	librering		Filer - utka	librering	12				
CHEI	ммас	Data	Kon	nmentar	Data			Kommentar			
1	pH4	KFM05a0924IpH4Y.C	RB KEM	5a0924IpH4Y.CI							
	pH7	KFM05a0924IpH7Y.C	RB KEM	05a09241pH7Y.CI							
i	pH10	KFM05a09241pH10Y.	CRB KFM	05a0924IpH10Y.CI							
1.000	0.01M	KFM05a0924Ikcl01Y.	CR KFM	05a0924Ikcl01Y.CI							
	0.1M	KFM05a0924Ikcl1Y.C	RB KFM	05a0924Ikcl1Y.CI							
B	pH4	KFM05a0923IpH4B.C	RB KFM	05a0923IpH4B.CI							
RÅ	pH7	KFM05a09231pH7B.C	RB KFM	05a09231pH7B.C1							
RL	pH10	KFM05a09231pH10B.	CRB KFM	05a09231pH10B.CI							

Figure A4-4. Administration (041004), section 712.6-722.0 m

Appendix 5



Flow and pressure measurements

Figure A5-1. Pressure (P2V and PB) and flow rate (Q) measurements from the borehole section 712.6–722.0 m. The sensors P1V and P2V measure the pressure in the section and are both placed in the in situ sampling unit. P1V- and P2V-measurements coincide and P1V is omitted in order to make the diagram clearer. PB is the sensor in the borehole Chemmac and measures the pressure above the section. As can be seen, there is no drawdown except for the pressure fluctuations caused by the piston of the borehole pump.



Figure A5-2. Pressure (P1V) and flow rate (Q) measurements from the borehole section 702.5–722.2 m. In this case all three pressure sensors (P1V, P2V and PB) are measuring the pressure within the section. To make the diagram clearer, only one pressure sensor is included. As can be seen, there is no drawdown except for pressure fluctuations caused by the piston of the borehole pump. At the 25:th of October, pumping is performed in the upper part of the borehole and a pressure response is observed in the borehole section. The response is seen more clearly in Figure A4-3.



Figure A5-3. Pumping is performed in the upper part of the borehole and a pressure response corresponding to 2.5 m is observed in the borehole section 702.5–722.2 m.



Figure A5-4. Pressure (P2V) and flow rate (Q) measurements from the borehole section 701.5–721.2 m. Also in this case all three pressure sensors (P1V, P2V and PB) are measuring the pressure within the section. To make the diagram clearer, only the P2V sensor is included. There is no drawdown, except for pressure fluctuations caused by the piston of the borehole pump.



Figure A5-5. Pressure (P1V, P2V and PB) and flow rate (Q) measurements from the borehole section 262.0–266.9 m. P1V and P2V are pressure sensors placed in the in situ sampling unit and measure the pressure in the section. PB is the sensor in the borehole Chemmac and measures the pressure above the section. At a flow rate of a few mL/min the drawdown obtained was approximately 50 m.



Chemmac measurements (Eh, pH, electric conductivity, dissolved oxygen and temperature) section 712.6–722.0

Figure A6-1. Redox potential (Eh) measurements by platinum, gold and glassy carbon electrodes in the borehole section (EHPTB, EHAUB and EHCB) and at the surface (EHPTY, EHAUY and EHCY). The arrow shows the chosen representative Eh values for the borehole section.



Figure A6-2. Measurements of pH by two glass electrodes in the borehole section (PHB and PHIB) and two glass electrodes at the surface (PHY and PHIY). The laboratory pH in each collected sample (PHL) is given for comparison. The arrow shows the chosen representative pH values for the borehole section.



Figure A6-3. Electric conductivity measurements in the surface measurement cell (KONDY). The laboratory conductivity in each collected sample (KONDL) is given for comparison.



Figure A6-4. Dissolved oxygen measurements (O2Y) in the surface measurement cell.



Figure A6-5. Temperature of the water in the borehole section (TB).

Appendix 7

Sampling and analysis methods

Table A7-1. Sample handling routines and analysis methods.

			į				
Component group	Component/element	Sample container (material)	Volume (mL)	Filtering	Preparation/ Conservation*	Analysis method	Analysis within – or delivery time to lab
Anions 1 .	HCO ₃ pH(lab) cond (lab)	Plastic	250	Yes (not in the field)	No	Titration Pot. meas, Cond. meas	The same day – maximum 24 hours
Anions 2 .	Cl, SO4, Br', F-, I-	Plastic	100	Yes (not in the field)	No	Titration (Cl ⁻) IC (Cl ⁻ , SO4, Br ⁻ , F ⁻) ISE (F ⁻)	Not critical (month)
	Br, I	Plastic	100	Yes (not in the field)	No	ICP MS	Not critical (month)
Cations, Si and S according to SKB class 3	Na, K, Ca, Mg, S(tot), Si(tot), Li, Sr	Plastic (at low conc. acid washed bottles)	100	Yes (not in the field)	Yes (not in the field, 1 mL HNO $_3$)	ICP-AES ICP-MS	Not critical (month)
Cations, Si and S according to SKB class 4 and 5	Na, K, Ca, Mg, S(tot), Si(tot), Fe, Mn, Li, Sr	Plastic (Acid washed)	100	Yes (immediately in the field)	Yes (1mL HNO ₃)	ICP-AES ICP-MS	Not critical (month)
Fe(II), Fe(tot)	Fe(II), Fe(tot)	Plastic (Acid washed)	500	Yes	Yes (5 mL HCI))	Spectrophotometry Ferrozine method	As soon as possible the same day
Hydrogen sulphide	-SH	Glass (Winkler)	About 120×2	No	Ev 1 mL 1 M NaOH+ 1 mL 1M ZnAc	Spectrophotometry	Immediately or if conserved, a few days
Nutrient salts	NO ₂ , NO ₃ +NO ₂ , NH ₄ , PO ₄	Plastic	250	No	No	Spectrophotometry	Maximum 24 hours
Environmental metals	Al, As, Ba, B, Cd, Co, Cr, Cu, Hg, Mo, Ni, P, Pb, V, Zn	Plastic	100	Yes	Yes (1 mL HNO ₃)	ICP-AES ICP-MS	Not critical (month)

Component group	Component/element	Sample container (material)	Volume (mL)	Filtering	Preparation/ Conservation*	Analysis method	Analysis within – or delivery time to lab
Lantanoids, U, Th and so on.	Sc, Rb, Y, Zr, I, Sb, Cs, La, Hf, Tl, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, U, Th	Plastic	100	Yes	Yes (1 mL HNO ₃)	ICP-AES ICP-MS	Not critical (month)
Dissolved organic Carbon, dissolved inorganic Carbon	DOC, DIC	Plastic	250 25	Yes	Frozen, transported in isolated bag	UV oxidation, IR Carbon analysator Shimadzu TOC5000	Short transportation time
Total organic Carbon	TOC	Plastic	250 25	No	Frozen, transported in isolated bag	UV oxidation, IR Carbon analysator Shimadzu TOC5000	Short transportation time
Environmental isotopes	∂²H, ∂¹®O	Plastic	100	No		MS	Not critical (month)
Tritium, Chlorine-37	³ H (enhanced.) ∂ ³⁷ Cl	Plastic (dry bottle) Plastic	500 100	N N		LSC ICP MS	Not critical (month)
Carbon isotopes	∂ ¹³ C, pmC (¹⁴ C)	Glass (brown)	100×2	No	1	(A)MS	A few days
Sulphur isotopes	∂ ³⁴ S	Plastic	500- 1,000	Yes	1	Combustion, ICP MS	No limit
Strontium-isotopes	⁸⁷ Sr/ ⁸⁶ Sr	Plastic	100	Yes	1	TIMS	Days or week
Uranium and Thorium isotopes	234U, 235U, 238U, 232 Th, 230Th,	Plastic	50	No	I	Chemical separat. Alfa/gamma spectrometry	No limit
Boron isotopes	¹⁰ B/11B	Plastic	100	Yes	Yes (1 mL HNO_3)	ICP – MS	No limit
Radon and Radium isotopes	²²² Rn, ²²⁶ Ra	Plastic	500	No	No	EDA, RD-200	Immediate transport
Dissolved gas (content and composition)	Ar, N ₂ , CO ₂ , O ₂ , CH4, H ₂ , CO, C ₂ H2, C ₂ H4, C ₂ H6, C ₃ H6	Cylinder of stainless steel	200	No	No	GC	Immediate transport
Colloids	Filter series and fractionation (see below)	Polycarbonate filter	0.45, 0.2 and 0.05 µm	1	N ₂ atmosphere	ICP-AES ICP-MS	Immediate transport
Humic and fulvic acids	Fractionation	Fractions are collected in plastic bottles	250	I	N ₂ atmosphere	UV oxidation, IR (DOC)	Immediate transport

Component group	Component/element	Sample container (material)	Volume (mL)	Filtering	Preparation/ Conservation*	Analysis method	Analysis within – or delivery time to lab
Archive samples with acid	1	Plast (washed in acid)	100×2**	Yes	Yes (1 mL HNO_3)	. 1	Storage in freeze container
Archive samples without acid	I	Plastic	250×2**	Yes	No	1	Storage in freeze container
Carbon isotopes in humic and fulvic acids	∂ ¹³ C, pmC (¹⁴ C)	DEAE cellulose (anion exchanger)	1	I	1	(A)MS	A few days

* Suprapur acid is used for conservation of samples. ** Minimum number, the number of archive samples can vary depending on how many similar samples that are collected at the same occasion.

Abbreviations and definitions:

- IC ISE ICP-AES ICP-MS INAA MS MS (A)MS GC
- Ion chromatograph Ion selective electrode Inductively Coupled Plasma Atomic Emission Spectrometry Inductively Coupled Plasma Mass Spectrometry Instrumental Neutron Activation Analysis
- Mass Spectrometry Liquid Scintillation Counting (Accelerator) Mass Spectrometry Gas Chromatography

Component	Method	Detection limit	Reporting limit or range	Unit	Measurement uncertainty ²	"Total" uncertainty³
HCO ₃	Alkalinity titration	0.2	1	mg/L	4%	<10%
Cŀ Cŀ	Mohr titration	5 0.2	70 0.5	mg/L	5% 6%	<10% 10%
SO ₄	IC	0.2	0.5	mg/L	6%	15%
Br- Br-	IC ICP	0.2	0.7 0.001–0.010 ¹	mg/L	9% 15%	20%
F- F-	IC Potentiometri	0.2	0.6 -	mg/L	10% _	20% -
I-	ICP	_	0.001–0.010 ¹	mg/L	15%	20%
Na	ICP	_	0.1	mg/L	4%	10%
К	ICP	_	0.4	mg/L	6%	15%
Са	ICP	_	0.1	mg/L	4%	10%
Mg	ICP	_	0.09	mg/L	4%	10%
S(tot)	ICP	_	0.160	mg/L	10%	15%
Si(tot)	ICP	_	0.03	mg/L	4%	15%
Sr	ICP	_	0.002	mg/L	4%	15%
Li	ICP	_	0.2–2 ¹	µg/L	10%	20%
DOC	See tab. 1	_	0.5	Mg/L	8%	30%
тос	See tab. 1	-	0.5	Mg/L	10%	30%
δ²H	MS	-	2	‰ SMOW ⁴	1.0‰	_
δ18Ο	MS	-	0.1	‰ SMOW⁴	0.2‰	_
³Н	LSC	-	0.8 or 0.1	TU⁵	0.8 or 0.1 TU	_
δ ³⁷ Cl	ICP MS	-	0.2‰ (20 mg/L)	‰ SMOC ⁶	-	_
δ¹³C	A (MS)	-	>20 mg Carbon	‰ PDB ⁷	-	_
pmC (14C)	A (MS)	-	>20 mg kol	PmC ⁸	-	_
δ ³⁴ S	ICP MS	-	0.2‰	‰ CDT ⁹	0.2‰	_
⁸⁷ Sr/ ⁸⁶ Sr	MS	-	-	No unit (ratio) ¹⁰	0.000020	-
¹⁰ B/ ¹¹ B	ICP MS	-	_	No unit (ratio) ¹⁰	0,0020	_
²³⁴ U, ²³⁵ U, ²³⁸ U, ²³² Th, ²³⁰ Th	Alfa spectr.	-	0.0005	Bq/L ¹²³	0.05 Bq/L	Right order of magnitude
²²² Rn, ²²⁶ Rn	LSC	-	0.0005	Bq/L	0.05 Bq/L	11

Table A7-2. Reporting limits and measurement uncertainties.

1. Reporting limits at salinity \leq 0.4% (520 mS/m) and \leq 3.5% (3,810 mS/m) respectively.

2. Measurement uncertainty reported by consulted laboratory, generally 95% confidence interval.

3. Estimated total uncertainty by experience (includes effects of sampling and sample handling).

4. Per mill deviation¹² from SMOW (Standard Mean Oceanic Water).

5. TU=Tritium Units, where one TU corresponds to a Tritium/hydrogen ratio of 10⁻¹⁸ (1 Bq/L Tritium = 8.45 TU).

6. Per mill deviation¹² from SMOC (Standard Mean Oceanic Chloride).

7. Per mill deviation¹² from PDB (the standard PeeDee Belemnite).

8. The following relation is valid between pmC (percent modern carbon) and Carbon-14 age: pmC = 100×e^{((1950-y-1.03t)/8274)} where y = the year of the C-14 measurement and t = C-14 age.

9. Per mill deviation¹² from CDT (the standard Canyon Diablo Troilite).

10. Isotope ratio without unit.

 The following expressions are applicable to convert activity to concentration, for uranium-238 and thorium-232: 1 ppm U = 12.4 Bq/kg²³⁸U

1 ppm Th = 3.93 Bq/kg²³²Th

12. Isotopes are often reported as per mill deviation from a standard. The deviation is calculated as: $\delta^{y}I = 1,000 \times (K_{sample}-K_{standard})/K_{standard}$, where K = the isotope ratio and ${}^{y}I = {}^{2}H$, ${}^{18}O$, ${}^{37}CI$, ${}^{13}C$ or ${}^{34}S$ etc.

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