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# Sealing of 300 mm boreholes KXTT3 and KXTT4 at Äspö HRL

Report of Subproject 3 of Borehole sealing Project

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November 2011

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## Summary

Sealing of two moderately dipping 300 mm diameter holes that had been bored from a niche adjacent to the ramp in the Äspö URL in conjunction with earlier geohydrological investigations (TRUE Project, Winberg et al. 2000) was successfully completed in early 2010. The work was preceded by a pilot test on the ground surface for investigating if a simple version of sealing by pressing down clay pellets into clay mud is feasible. The borehole was simulated by a steel tube that was filled with smectite mud in which a cage filled with clay pellets was moved down. The experience from this experiment was that the mud used was somewhat too stiff and that strong vibration significantly reduced its viscosity but not sufficiently much. The predicted average density at water saturation was 1,780 kg/m<sup>3</sup> (dry density 1,240 kg/m<sup>3</sup>).

The finally selected sealing method comprised casting of concrete plugs where the inflow of water was significant (up to 30 l/min), and to install precompacted blocks of smectite-rich clay where the rock was "dry". The finally matured clay plugs will have a density of around 2,000 kg/m<sup>3</sup> (dry density 1,580 kg/m<sup>3</sup>) and a hydraulic conductivity of less than E-12 m/s, which is estimated to be at least one order of magnitude lower than the bulk conductivity of the surrounding rock mass (Pusch 2008). This provides excellent sealing of all parts of the holes deeper than about 1–2 m below the tunnel floor. Groundwater flow in the rock around the clay plugs can cause some minor erosion but this effect is deemed negligible considering the coagulating effect on released clay particles by the brackish Äspö water.

Inspection of the closed upper borehole ends showed accumulation of some very dilute mineral suspension after a few days. A careful analysis of the mineral content showed that it did not contain smectite particles, hence certifying that erosion of the clay plugs in the holes had not taken place. The particles were concluded to represent fracture minerals.

# Sammanfattning

Denna rapport för Delprojekt 3 ingår i "Sealing of investigation Boreholes, Phase 4 TU02". Project Decision approved 2011-01-28.

Förslutning av två lutande 300 mm hål som borrats från en nisch vid rampen i Äspölaboratoriet för tidigare geohydrologiska undersökningar (TRUE-projektet) genomfördes framgångsrikt i början av 2010. Arbetet föregicks av ett försök för undersökning av möjligheten att på ett enkelt sätt försluta borrhål genom att pressa ned en stålkorg med lerpellets i lermudd. Borrhålet representerades av ett stålrör som fylldes med en mudd av smektitlera i vilken en cylindrisk korg med lerpellets pressades ned. Erfarenheten från försöket var att mudden hade för hög skjuvhållfasthet och att stark vibrering påtagligt men inte tillräckligt mycket reducerade den. Den teoretiska medeldensiteten var 1,780 kg/m<sup>3</sup>.

Den slutgiltigt valda metoden innebar gjutning av betongpluggar där vatteninflödet var stort, dvs upp till 30 l/min, och installation av förkompakterade block av smektitlera där berget var "torrt". Efter fullständig vattenmättnad och homogenisering kommer lerpluggarna att ha en densitet av ca 2,000 kg/m<sup>3</sup> och en hydraulisk konduktivitet som är lägre än E-12 m/s, som kan uppskattas vara åtminstone tio gånger lägre än medelvärdet hos omgivande berg. Det ger en utmärkt avtätning av alla delar av borrhålen som är belägna 1–2 m under tunnelgolvet. Grundvattenflödet runt lerpluggarna kan ge viss erosion men dess betydelse bedöms vara försumbar på grund av det bräckta Äspövattnets koagulerande inverkan på avlossade lerpartiklar.

Besiktning av de förseglade övre håländarna visade ansamling av en mycket utspädd suspension av finpartiklar. Noggrann analys av mineralinnehållet visade att den inte innehöll smektitpartiklar och att det därför inte fanns något spår av erosion av lerpluggarna i hålen. Partiklarna bedömdes representera sprickmineral.

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### 1 Introduction

### 1.1 Background

This report describes sealing of the two 300 mm boreholes KXTT3 and KXTT4 at Äspö early 2010, AP TD TU02-09-070. It also deals with a preliminary test that was carried out in late 2007. In the preliminary test the plug design was based on the use of pellets for cost and time reasons. It was concluded that, taking the rock structure in consideration, a sandwiched plug construction had to be constructed with both concrete and clay. The techniques used for sealing was developed in the course of the Borehole Sealing Project (Pusch and Ramqvist 2007) i.e. to tightly seal those parts of boreholes where the rock has few fractures and a low hydraulic conductivity, and filling of those parts that intersect permeated fracture zones with physically stable material that does not need to be low-permeable. Tight sealing is obtained by inserting highly compacted clay blocks while physically stable fillings are made by casting concrete.

### 1.2 Scope and objective

Large-diameter boreholes in the repository area need to be effectively sealed and the experiment showed how this can be made. Two boreholes with 15–20 m length and 300 mm diameter were plugged so that no axial water flow can take place through them. The boreholes have been used for tracer experiment as part of the TRUE-1 project (Winberg et al. 2000). Concrete cannot provide the required tightness but sufficiently dense smectite-rich clay will serve acceptably and was therefore used for the sealing. The water pressure could be about 3 MPa, which would exert an axial force of about 20 t (200 kN). This made it necessary to seal the upper ends of the holes with durable concrete anchored to the rock in a reamed recess. The distribution of the inflow of water is illustrated in Figure 1-1, which is also an indication of the rock structure. One concludes that the inflow is relatively uniform except for peaks at about 3, 10, 13 and 20 m distance from the upper ends of the holes. Two "dry" intervals can be assumed, one at 10–12 m distance from the upper ends of both holes, and one above 2 m depth.



Figure 1-1. Measured inflows in the two boreholes KXTT3 and KXTT4 (TRUE-1 volume (Winberg et al. 2000).

### 1.3 Location and conditions on site

The two 300 mm holes extend from a niche excavated from the access ramp to the Äspö HRL at Section 2,950 and oriented largely WNW/ENE as indicated in Figure 1-2, which illustrates that the holes intersect a number of identified fractures grouped as shown in Figure 1-2. A schematic perspective view of the location and orientation of the holes is provided as well.



*Figure 1-2. Upper:* Horizontal section at Z=-400 (reference depth) showing structural model based on identified conductive geological structures in the TRUE-1 volume (Winberg et al. 2000). Lower: Perspective view.

### 2 Concepts

### 2.1 General

The very good sealing potential of moderately dense smectite-rich clay in rock with brackish groundwater was the basis of the initially proposed sealing concept. It implied use of dense smectite pellets embedded in a smectite mud mixed with silt of nonexpandable rock material for minimizing erosion and loss of clay material into the fractures (Pusch 2008). The viscosity of the mixed mud turned out to be somewhat too high and a second concept was selected, implying placement of highly compacted clay blocks in fracture-poor parts of the holes and casting of concrete where they intersected waterbearing fracture swarms.

### 2.2 First concept – pilot test

### 2.2.1 Planning

The planned sealing is illustrated in Figure 2-1. It comprised filling of smectite-rich, dense pellets in the entire holes. The pellets were to be placed in cages brought down in the holes in the partly mud-filled holes in 0.5 to 1 m long units.

The fill of densely compacted montmorillonite-rich pellets was expected to have an ultimate dry density of 1,000 kg/m<sup>3</sup>, i.e. about 1,650 kg/m<sup>3</sup> at water saturation, which corresponds to a hydraulic conductivity and swelling pressure of around E-10 m/s and 100 kPa, respectively, for Äspö ground-water (Pusch and Ramqvist 2007). With the voids in the pellet mass filled with a mud with a density of about 1,200 kg/m<sup>3</sup> the average density is raised to a somewhat higher value, i.e 1,750 kg/m<sup>3</sup> (Pusch 2008). The homogeneity of the fill would be fairly uniform since the mud was expected to be consolidated by the expanding pellets.



Figure 2-1. First concept.

The pellets, marketed under the name CEBOGEL, were made of soda-activated bentonite with 80% montmorillonite and 16% water content and delivered by Clay Technology AB, Lund. It is a grey/ green material delivered in the form of extruded cylindrical rods with 6.5 mm diameter and 5–20 mm length, appearing as illustrated by Figure 2-2.

The risk of erosion and loss of clay material into the fractures was known by experience and through early modelling attempts (Pusch 1983, 2008). Hence, if a fracture has an aperture exceeding about 100  $\mu$ m, smectite clay will expand into it and the process may ultimately involve substantial loss of clay to the rock if groundwater percolates the fracture at a sufficiently high rate, especially if the water is poor in electrolytes. The idea was therefore to submerge the clay plug in a mud with larger particle size or with a particle shape that can serve to form a filter-cake at the fracture aperture for retarding or halting migration of smectite particles released from the plug (Figure 2-3).

### 2.2.2 Construction

For placing the pellets in the holes it was planned to use cages and a pilot test was made in August 2007 for finding out if the technique is feasible. The experiment was made at Äspö using a 2 m high tube of stainless steel for simulating a 300 mm diameter borehole. A metal cage with 10 mm wide openings was filled with clay particles and lowered into the partly mud-filled tube. The general design principle is shown in Figure 2-4.



Figure 2-2. CEBOGEL "pellets".



*Figure 2-3.* Clogging of fractures by quartz grains forming arches. The right picture shows a hole in rock with strongly anisotropic stress field that causes widening of a steep intersecting fracture into which clay can move (Pusch 2008).



Figure 2-4. Schematic drawing of pellet cage.

Originally, MX-80 pellets were planned to be used in the pilot test and in subsequent sealing of the two boreholes but lack of material led to selection of CEBOGEL grains, which consist of 6.5 mm diameter bentonite particles delivered by a Dutch company, CEBO Holland. This material is described in Appendix 1 to this report<sup>1</sup>. The mud was prepared by mixing MX-80 clay with 50% silica flour and tap water to yield a density of about 1,200 kg/m<sup>3</sup>. The silica flour, consisting of quartz particles with a size of 0.010 to 2 mm, would clog rock fractures and prevent clay particles from migrating into the fractures in significant amounts. The construction work included the following activities:

- The tube was filled with crushed rock (0–4 mm) to 250 mm from the bottom for reducing the volume.
- The steel cage, which was surrounded by a plastic sheet that would be pulled up parallel to submerging the cage, was filled with CEBOGEL grains with some light compaction using a wooden stock before inserting it in the steel tube.
- Preparation of mud was made by mixing 0.7 kg rock flour with 2.8 kg silica powder M300, (size of mesh) adding 3.5 kg clay powder to it. Tap water was added in steps to reach a viscosity that was deemed suitable, corresponding to a net density of 1,250 kg/m<sup>3</sup>. The mixture was stirred with a concrete mixer.
- A wire was placed around the cage for carrying it in the placement phase. The idea was to remove the plastic sheet parallel to the submergence but the wire made this impossible.
- The cage was brought up again for removal of the plastic sheet by which a small number of clay grains fell out.
- The cage was submerged again and it sank quickly some centimetres. A 250 kg weight was applied to make it move further down but the load was too small. Increasing the weight to 500 kg and vibrating the tube by the aid of a concrete vibrator brought the cage further down to a level somewhat below the top of the tube by the aid but it could not be moved sufficiently deep down.

### 2.2.3 Results

The conclusions from test were:

- 1. The mud was somewhat too stiff for allowing the cage to move down without difficulties.
- 2. A concrete vibrator was found to significantly reduce the viscosity but the effect was not sufficient to bring the cage down.
- 3. The mud penetrated the pellet fill to a few centimetres depth in the upper part of the cage and to about 10 cm in its lowest part (Figures 2-5 and 2-6).
- 4. The dry density of the pellet fill was about 1,000 kg/m<sup>3</sup> and that of the mud about 250 kg/m<sup>3</sup>. Had the mud completely filled the voids of the pellet fill the net dry density would have been 1,250 kg/m<sup>3</sup> and the density at saturation 1,780 kg/m<sup>3</sup>.

<sup>&</sup>lt;sup>1</sup> The construction team consisted of Roland Pusch, Gunnar Ramqvist, Lars Liw, Elinor Örtendahl, Nils Göran Myrén och Peder Karlsson.



*Figure 2-5.* The tube and cage cut open for sampling. The clay grains are seen in the upper part while they are embedded in mud in the lower part.



Figure 2-6. The water content distribution in the fill after termination of the test<sup>2</sup>.

<sup>&</sup>lt;sup>2</sup> The planned fill of MX-80 pellets was changed to Cebogel grains in the actual construction. The bottom fill of gravel was changed to crushed rock (0–4 mm).

### 2.3 Final concept – Sealing of the boreholes

### 2.3.1 Planning

The difficulties in placing cages with clay pellets and the relatively poor net density of the clay seals called for simpler and safer plugging techniques and the finally selected and applied way of plugging the two 300 mm boreholes is illustrated in Figure 2-7. It follows the general proposed principle of plugging boreholes, i.e. to fill them with physically stable material, concrete, where the holes intersect water-bearing fracture zones and install dense clay blocks where the rock is poor in fractures. This concept means that no mud is required because the density of the clay-plugged parts will be high as calculated from the known diameter and dry density of the clay blocks. The ultimate density would be about 2,000 kg/m<sup>3</sup>.

The central copper tubes served to discharge water flowing in from the lower ends of the holes throughout the construction period and to provide support to the clay blocks stacked around them. After completing the sealing campaign the tubes were sealed with concrete in conjunction with casting the upper concrete plug. The whole arrangement represents a version of the "Couronne" concept (Pusch and Ramqvist 2007).



*Figure 2-7.* The finally selected sealing principle. *P* (*P* stands for mechanical packer) is flange for making the upper surface of the concrete perpendicular to the axia os the holes.

### 2.3.2 Materials

#### Concrete

The concrete recipe used in preceding full-scale boreholes sealing projects in Finland ("KR-24" at Olkiluoto) and in construction of concrete plugs in 200 mm boreholes at Äspö (Pusch and Ramqvist 2007) is shown in Table 2-1. Results from CBI's testing and from the last mentioned field experiment had indicated that the uniaxial compressive strength would be about 1 MPa after one day of curing and at least 10 MPa after one month (Pusch and Ramqvist 2007).

Components	Amount (kg/m³ concrete)	Manufacturer
White cement	60.0	Aalborg Portland
Silica Fume	60.0	Elkem
Fine ground α-quartz M300	200.0	Sibelco
Fine ground cristobalite M6000	150.0	Sibelco
Superplasticizer Glenium 51	4.375 (dry content)	Degussa
Granitic aggregates 0–4 mm	1,700.0	Jehanders grus
Water	244.27	local

Table 2-1. Concrete (low strength) recipe for plugging of boreholes (CBI), [2].

### Clay

Two materials were used for block preparation: CEBOGEL manufactured by the Dutch company CEBO Holland, that supplied the granular material ("pellets") in the pilot test, and G001-L, a Turkish bentonite manufactured by BIROLA Mineral Nakliye Hayvan Malzemeleri San Tic Ltd.Sti, provided by Nyberg Commercial AB, Drottninggatan 111 60 Stockholm. The first material, in pellet form, is being used in SKB's BACLO experiment, the other is a candidate for use as top and bottom liners in waste landfills by Ragn-Sell AB.

CEBOGEL is a soda-activated bentonite with 70–80% montmorillonite and 16% water content. G001-L consists of granular material appearing as MX-80. G001-L has an XRD spectrum in that shows that the clay is dominated by smectite minerals but that also quartz, kaolinite (tubular, i.e. halloysite), and andesine, and labradorite are present. Chemical and geotechnical data are collected in Appendix 1 for the two materials. CEBOGEL and G001-L have properties similar to those of MX-80. However, G001 delivered for preparation of blocks has somewhat less good expandability as exemplified by the lower liquid limit ( $w_L$ =250%) than that of CEBOGEL ( $w_L$ =420%). It is believed to have less potential to migrate into fractures than MX-80 and CEBOGEL but the importance of this was not assessed in the project. The cost of the G001L clay is significantly lower than that of CEBOGEL.

The blocks were prepared by compaction of the clay material in a steel form as illustrated in Figure 2-8a and b. It had a slightly conical shape and was manufactured by Livinstone AB for easy release of the blocks. This company also made the block compaction using a compressive force of up to 250 t (2.5 MN, pressure = 22 MPa). Table 2-2 is a compilation of the block data, the density of the saturated clay referring to the expanded state, i.e. when the blocks have expanded from 285 to 300 mm diameter.

nark
erical" aggregate particles with verage diameter of 0.5–3 mm
s with 5 mm diameter and ) mm length
ht 120 mm ght 14.57 kg
ht 9.8 cm ght 12.12 kg

Table 2-2. Material specification of granular materials and blocks, which were prepared by uniaxial compression under 22 MPa pressure.

Assuming the same smectite content the swelling pressure of the CEBOGEL block material should hence be 1% higher than for the G001-L block material. The difference is larger, however, indicating that the compressibilities are different. The higher swelling pressure of the CEBOGEL material provides better expandability and this brand was therefore used for preparation of the blocks for sealing the two 300 mm diameter holes.



*Figure 2-8a.* Compaction of blocks. G001-L bentonite block with 285 mm diameter and 120 mm height. The black colour is molybdene disulphide for lubricating the form. Density  $2,035 \text{ kg/m}^3$ .



*Figure 2-8b.* Compaction of blocks. CEBOGEL bentonite block with 285 mm diameter and 100 mm height. Density 2,060 kg/m<sup>3</sup>.

# 3 Sealing of holes

### 3.1 General

The principle of sealing is shown in Figure 2-7. The ingredients of the concrete and the properties of the investigated clays of which CEBOGEL was used for preparing the blocks are reported in the Appendices 1 and 2. G001 was not further investigated or used but represents a back-up. They were measured individually with respect to the height and weight and were tightly wrapped in plastic for transport to the construction site where they were temporarily stored close to the boreholes. Installation of the seals followed the procedure described in detail below. The blocks were stacked around a central copper tube served as drainage in the construction phase.

### 3.2 Procedure

### 3.2.1 Preparative work, specification of activities step by step

- 1. Preparations on site
  - Recording of water inflow.
  - Recording of water pressure in surrounding boreholes.
  - Opening of surrounding holes 2 weeks prior to the sealing operation for lowering the *in situ* water pressure <sup>3</sup>.
- 2. Cleaning of the 300 mm holes.
- 3. Construction of funnel for moving clay blocks into the holes and lifting objects weighing up to 250 kg.
- 4. Permissions
  - Checking of permission of personnel to be in the area respecting possibly remaining radioactivity from preceding projects (Sr etc).
  - Steps to get access to the fenced-in construction site.
  - Access to electric power (380 V), tap water and compressed air.
  - Transport conditions for bringing clay blocks, concrete mixers, pumps, pipes etc to the site.
- 5. Installation steps
  - Filling of bottom parts by sand/gravel.
  - Casting of the lower concrete plug with a copper tube with 72 mm inner diameter in the center and a copper plate fixed to the tube as upper form. Quality control of the concrete by compression testing.
  - Placement of the lower clay plug of 5 m total length with a total number of clay blocks of **50** (Figure 3-1) one day after casting the lower concrete plug.
  - Casting of the central concrete plug with a copper tube with 72 mm inner diameter in the center and a copper plate fixed to the tube as upper form. Quality control of the concrete by compression testing.
  - Placement of the upper clay plug total length 1 m, total number of clay blocks 10 after casting the central concrete plug.
  - Mounting of steel box anchored to the rock by 3 m bolts at the upper end of the holes.
  - Filling the central copper tube with CEBOGEL pellets.
  - Mounting of expander plug of copper in the copper tube at its upper end for sealing the tube.
  - Casting of the upper concrete plug (about 2 m) with the through-going copper tube in the center. The upper end of this plug coincides with the tunnel floor.

<sup>&</sup>lt;sup>3</sup> This activity was an attempt to reduce the groundwater pressure in the area.



*Figure 3-1. Placement of clay blocks. Upper: Funnel for letting the blocks slide down. Lower: Block column with copper plate and fixed central tube passing through.* 

### 3.3 Function of seals

### 3.3.1 Completion of the sealing operation

The construction work was completed in agreement with the plan in AP TD TU02-09-070, except for the clay plug in KXTT3 that get stuck at 11.62 instead of 12.5 meters. During the connection of the center pipe, there was some deformation in the end of the pipe. As a consequence of that, the decision was taken to fill the center pipe with bentonite pellet instead of compacted blocks. The operation is described in accordance with rutines in the AP TD TU02-09-070.

The field work with installations of the plugs started early December and was finished in about two weeks time.

Water samples were taken at the construction site on January 14th 2010 and later.

### 3.3.2 Observations

No leakage was seen at the closed upper borehole ends but some very dilute clay suspension had accumulated near the concrete seals after ending the work. It attenuated stopped after a few weeks. A sample of the dilute suspension had been taken after a couple of days for analysis at the Geological Dept. of Greifswald University with respect to the mineralogical composition. The outcome of the analysis was that the larger part of the very small amount of minerals consisted rock fracture minerals and not smectite particles (cf. Appendix 3). It is hence concluded that no smectite had migrated out from the clay plugs.

### 3.3.3 Expected performance of matured plugs

The finally matured clay plugs will have a density of around 2,000 kg/m<sup>3</sup> (dry density about 1,550 kg/m<sup>3</sup>) and a hydraulic conductivity of less than E-12 m/s, which is estimated to be at least one order of magnitude lower than the bulk conductivity of the surrounding rock mass. This provides excellent sealing of the holes deeper than about 1–2 m below the tunnel floor. Groundwater flowing in the rock around the clay plugs can cause some minor erosion of the clay where it is in contact with minor water-bearing fractures but this effect is deemed negligible considering the coagulating effect of the Äspö brackish water on possibly escaping smectite particles.

# 4 Conclusions

The following conclusions are drawn:

- The sealing operation was successful, but the pretest was not successful and materials and method used in it had to be changed during the project.
- The project serves as an example of how boreholes of 300 mm or smaller dimensions, oriented in any direction except upwards can be sealed even where water inflow is strong. The principle can be applied also to deposition tunnels.
- The performance of the plugs is expected to be very good although two mechanisms may have a degrading effect in a long term perspective:
  - Groundwater flowing in the rock around the clay plugs can cause some minor erosion of the clay where it is in contact with water-bearing fractures. When the site is finally abandoned and the groundwater level in the area restored the regional hydraulic gradients will be so low that the risk of erosion will be negligible.
  - Chemical interaction between clay and concrete may lead to degradation of both. This matter is presently being investigated by analysis of contacting clay and concrete in boreholes on the 220 m level at Äspö. These materials are of the same type as those used in the 300 mm holes.

The experiment was intended to demonstrate the construction of plugs in large diameter holes under realistic conditions. Water samples collected from the walls near the upper ends of the holes were analysed with respect to the content of possibly released clay particals from the plugs but no such contamination was found. Leakage was checked for a couple of months. The entire hydrological function is continuously monitored in the Äspö HMS (Hydo-monitoring system).

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## Appendix 1

### **Preparation of concrete**

Original data from the preparation of concrete.

Bag 1–5 describe water content in ballast before mixing.

Mixed volume(litre)Water content in aggregate(%)		50 Total batch. Type in water content in bag to compensate for the variations in ballast.						
	kg/m³ c	of cement	50 litre con	crete				
White cement (Aalborg Portland) Water Superplasticizer 51 Silica Fume Fine ground quartz M300 Fine ground cristobalit quartz M6000	60 141.8 12.5 60 200 150	60.0 124.8 12.5 60.0 200.0 150.0	3.0 6.24 0.625 3.0 10.0 7.5	kg liter kg kg kg kg	Red field gives the			
Aggregate 0–8 mm	1,700	1,717.0	85.85	kg	recipe for that volume.			
Bag 1 4 9%								

Bag 14.9%Bag 24.0%Bag 35.5%Bag 45.2%Bag 55.1%

### Investigation of clay materials

#### A2.1 Chemical composition

A2-1. Chemical data (MX-80 included for comparison).

Clay	SiO <sub>2</sub>	$AI_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	K <sub>2</sub> O	Na₂O
MX-80	63.6	19.8	5.0	3.2	3.1	1.0	2.8
G001-L	59.8	14.0	6.1	3.3	4.1	1.6	2.0
CEBOGEL	53.3	17.1	5.5	2.0	5.9	0.5	2.8

### A2.2 Mineralogy

The CEBOGEL clay has been used in a number of practical experiments made by Clay Technology AB for SKB and reference is made to this company respecting its mineralogical composition. For the G001-L clay X-ray Diffraction (XRD) was one of the techniques used for characterization. A typical XRD spectrum is shown in Figure A-1.



Figure A2-1. XRD diagram of G001-L.

### A2.3 Geotechnical properties of the clay materials

The evolution of the hydraulic conductivity and swelling pressure of the two clay types investigated by oedometer testing is illustrated by Figures A-2 and A-3 for samples with a dry density of 1,425 kg/m<sup>3</sup> (1,995 kg/m<sup>3</sup> at saturation) as functions of time and the density at saturation with distilled water.

Data are summarized below the figures.



Evolution of hydraulic conductivity

*Figure A2-2.* Evolution of the hydraulic conductivity. Serie 1 represents CEBOGEL and Serie 2 G001-L that had a higher maturation rate because of its somewhat lower smectite content.





*Figure A2-3.* Evolution of the swelling pressure. Serie 1 represents CEBOGEL and Serie 2 G001-L that gave a lower pressure because of its somewhat lower smectite content

Summary of lab data of CEBOGEL and GOO1-L clay materials saturated and percolated with distilled water.  $w_L$  is about 500% for MX-80, 420% for CEBOGEL and 250% for G001-L.

Dry density,	Density at saturation,	Hydraulic conductivity,	Swelling pressure, kPa				
kg/m³	kg/m³	m/s	For 3.5% CaCl <sub>2</sub>	For distilled water			
990	1,610	4.3E-11/3E-12*	45	300			
1,335	1,850	8E-13/5E-13*	1,200	1,000			
1,640	2,046	2E-13/7E-14*	4,500	5,500			

A2-2. CEBOGEL geotechnical data. MX-80 data taken to be representative of CEBOGEL.

\*MX-80 data according to Clay Technology AB.

#### A2-3. G001-L geotechnical data.

Dry density,	Density at saturation,	Hydraulic conductivity,	Swelling pressure, kPa			
kg/m³	kg/m³	m/s	For 3.5% CaCl <sub>2</sub>	For distilled water		
715	1,450	E-10	80	80		
875	1,550	5E-11	250	200		
1,110	1,700	E-12	580	500		
1,715	2,120	< E-12	3,200	15,000		

### **Appendix 3**

### ERNST MORITZ ARNDT UNIVERSITÄT GREIFSWALD



Universität Greifswald, Inst. für Geographie und Geologie, D17487 Greifswald, Germany

#### Institut für Geographie und Geologie

Georg Grathoff, Ph.D.

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19 March, 2010

To: Dr. Roland Push

RE: Analyses of Sample O by TEM: by morphology and chemical data

Overall the sample contained a lot of Ca, Na, K, and Cl in solution and therefore was difficult to analyze. As soon as we dried the suspension, salts precipitated. Only after dialyzing the sample more than 10 times the salts were removed.

Main results of the dialyzed samples:

1) There was no evidence of smectite.

2) Three phases were identified based on chemical data and morphology:

- a. Tubular mineral with an Al/Si ratio of about 0.9 and low Fe. This mineral could be potentially halloysite, which has a Al to Si ratio of 1.
- b. Platey minerals with High Fe
- c. Platey minerals with High Al

Morphologically a. and b. were indistinguishable. We suspect non sheet silicates, because of the low Silica concentration. All three phases are crystalline and have some spots on electron diffraction pattern.

The chemistry also showed a positive correlation with Na- Cl, suggesting that the Na was bound with Cl and not in any other phases such a expandable clay minerals.

Georg Grathoff

# Results of analyses of rock water sample provided by Roland Pusch on February 11, 2010



Figure A3-1. Overview of the analyzed areas (12, 13, 14, 15).



Figure A3-2. Close-up of Area 12 including the areas (A to N) that were analyzed chemically by TEM.



Figure A3-3. Close-up of Area 13 including the areas (A to F) that were analyzed chemically by TEM.



*Figure A3-4a.* Close-up of Area 14 including the areas (A to H) that were analyzed chemically by TEM.



Figure A3-4b. Electron Diffraction pattern of grain H in Figure A34a).



Figure A3-5. Closeup of Area 15 including the areas (A to E) that were analyzed chemically by TEM.



Figure A3-6. Closeup of grain 16 including the area that were analyzed chemically by TEM.

#### EDX-analysis (atomic-%) of Transmission Electron Microscope JEOL JEM1210

	WaterP	robe0									atomic-%	
Element	Water	S2A	Water\$2B	WaterS2C	WaterS2D	WaterS2E	WaterS2F	WaterS2G1	Water\$2G2	WaterS2H	WaterS2I	WaterS2K
0	64,16		67,67	64,90	63,41	68,00	64,89	62,87	66,26	70,12	65,69	63,02
Na		0,79	0,64	2,46	0,67	0,75	0,20	0,09	0,71	0,18	0,26	0,13
Mg		0,15	0,03	0,10	0,30	0,09	0,98		0,18		0,46	0,17
AI	13,49		14,04	12,64	15,62	13,63	13,53	34,17	13,04	12,98	11,64	15,80
Si	14,91		15,55	7,58	9,99	14,88	14,01	1,68	10,72	15,39	16,21	16,71
P		0,48	0,20	1,78	1,32	0,10	0,55		1,05	0,09	0,48	0,63
s	8	0,30		0,57		0,34	0,24	0,02	0,40	0,40	0,15	
CI	0,77		0,30	0,80	0,42	0,60	0,17	0,33	0,52	1,01	0,36	0,36
к	0,42		0,39	1,00	0,43	0,50	0,59	0,06	0,63	0,43	0,32	0,22
Ca		0,13	0,07	0,27	0,16	0,01	0,14	0,16	0,33		0,08	0,25
Ti		0,18	0,03	0,23	0,17	0,07	0,03	0,03	0,30	0,16	0,26	0,17
Zr										10 0000000	. <u>63</u> .	12. 18. 1
Mn			0,06	2,73	0,65	0,12		0,00			0,00	0,30
Fe	4,12		1,15	4,54	6,78	1,02	4,64	0,59	5,75	0,25	3,99	1,82

Legend: < 2\*sigma

	WaterProbe0				(						
Element	WaterS2J	WaterS2L	WaterS2M	WaterS2N	Water\$3A	Water\$3B	WaterS3C	WaterS3D	Water\$3E	WaterS3F	WaterS4A
0	33,43	62,66	65,84	64,80	64,27	65,29	71,65	62,73	66,65	65,05	64,92
Na	10,70	2,99	0,20	0,89	0,17	0,95	0,19	0,59	0,06	0,38	0,19
Mg	7,73	0,59	0,51	0,19	0,74	0,22		0,33			0,05
AI	6,01	4,14	12,08	9,64	11,95	13,70	18,13	23,77	14,50	31,63	32,54
Si	11,88	22,53	17,66	8,85	14,12	14,88	4,82	3,92	16,38	1,04	1,65
P		0,66	0,11	1,36	0,75	0,33	0,71	1,03	0,01		10.00
S	2,06	1,70	0,11	0,20	0,32	0,43	0,12	0,26	0,11		
CI	21,37	2,56	0,14	0,51	0,53	1,18	0,54	0,55	0,97	0,38	0,28
к	8,00	1,12	0,27	0,36	0,59	0,41	0,27	0,38	0,37	0,24	0,10
Ca	9,15	0,37	0,12	0,08	0,17	0,50	0,14	0,17	0,27		0,05
Ti		0,18		0,46	0,38	0,08	0,19	0,21	0,18	0,13	0,04
Zr		69638		80							
Mn			0,14	0,00	0,04	0,30	0,17	0,03			0,04
Fe	0,94	0,48	2,77	12,31	5,91	1,64	2,99	5,90	0,38	1,67	0,14

	WaterPro	obe0									
Element	WaterS4B	WaterS4C	WaterS4D	WaterS4E	WaterS4F	WaterS4G	WaterS4H	WaterS5A	Water\$5B	Water\$5C	Water\$5D
0	67,25	67,53	66,86	66,56	64,98	64,63	66,72	69,02	63,11	62,01	63,76
Na	0,19	1,07	1,31	1,65	0,53	0,84	0,93	7,48	0,79	0,36	0,38
Mg	0,12		0,16	0,39		0,14		0,63	0,00	0,17	0,10
AI	13,96	10,75	12,49	6,67	29,62	23,47	11,42	1,37	28,68	33,21	32,53
Si	16,87	8,70	10,71	2,62	2,67	5,81	12,71	3,31	3,24	2,05	1,48
P	0,19	0,87	1,07	1,42	0,05	0,30	0,36	1,96	0,42	0,19	0,09
S	0,00	0,52	0,37	0,15	0,06	0,26	0,24	3,58	0,31		0,21
CI	0,41	0,93	1,07	1,12	0,38	0,57	0,90	7,74	0,36	0,22	0,43
к	0,29	0,80	0,62	0,54	0,24	0,41	0,69	2,03	0,25	0,15	0,14
Ca		0,12	0,31	0,50	0,16	0,36	0,06	1,90	0,04	0,10	0,28
Π		0,20	0,24	1,98	0,14	0,11	0,03		0,16	0,06	0,00
Zr											
Mn					0,18	0,14	0,29		0,19	0,07	0,07
Fe	0,78	8,76	4,83	16,31	0,93	2,93	5,62	0,71	2,40	1,37	0,44
Interpretation											

Polytype

Legend:

< 2\*sigma

WaterProbe0	WaterPro	obe0						
Element	Water\$5E	WaterS6 s						
0	60,61	66,86	0	66,86				
Na	5,01	1,20	Na	1,20				
Mg	0,44	0,42	Mg	0,42				
AI	11,28	12,07	AI	12,07				
Si	5,16	10,49	Si	10,49				
P		0,99	P	0,99				
s	1,38	0,21	S	0,21				
CI	11,88	1,01	CI	1,01				
к	1,53	0,90	K	0,90				
Ca	0,75	0,42	Ca	0,42				
Π	0,06	0,14	Ti	0,14				
Zr			Cr	0,14				
Mn	0,69	0,11	Mn	0,11				
Fe	1,97	5,05	Fe	5,05				

### PM

2012-03-21

Torbjörn Sandén Ola Karnland Clay Technology AB

### Limited mineralogical investigation of Cebogel QSE

### A4.1 Background

Pellets made of a sodium activated bentonite, Cebogel QSE, have been used in several large scale tests performed at Äspö HRL. In order to check that the material data provided from the manufacturer is correct, see Appendix 1, a limited mineralogical investigation has been made. The following analyses were performed:

- X-ray diffraction analysis.
- Element analysis.
- Exchangeable cation analysis.

### A4.2 X-ray diffraction analysis (XRD)

The mineralogical composition of the material was evaluated from the XRD diffractograms by use of the Siroquant quantitative XRD software. The method is described in detail in Karnland et al. (2006). The results from the quantitative analysis are provided in Table A4-1.

#	ID	Phase	Weight%	Error of Fit
1	8137	Montmorillonite, (CP)	90	0.9
2	1	Quartz	1.4	0.4
3	37	Cristobalite	0.7	0.1
4	10	Calcite 1	3.6	0.4
5	31	Dolomite	3.1	0.6
6	29	Pyrite	1.1	0.1

Table A4-1. Results from the Siroquant analyses of the Cebogel QSE bentonite.

### A4.3 Element analyze

The bulk material was analyzed for major elements using standard ICP-AES technique at an ISO 9002 accredited laboratory (Acme Analytical Labs). Loss of ignition (LOI) was determined gravimetrically by ignition to 1,000°C. The results are provided in Table A4-2.

Table A4-2. The chemical composition expressed as weight percent of major oxides of the bulk material.

SiO <sub>2</sub>	$AI_2O_3$	$Fe_2O_3$	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	$P_2O_5$	LOI	С	S	Sum
52.3	16.9	5.0	3.8	4.9	3.6	0.6	0.7	0.1	11.7	1.5	0.4	99.8

### A4.4 Exchangeable cations, EC

The composition of extractable cations was determined by exchange against ammonium ions in an alcoholic solution. The method is described in detail in Karnland et al. (2006). The investigation included one duplicate. The results are provided in Table A4-3.

Table A4-3.	Table showing th	e distribution	of exchangeable	ions in the bulk	material.
	rasio ononing a	o aloti isation	of ononangeasie	10110 111 1110 10 4111	

	Ca %	K %	Mg %	Na %
Cebogel 1	5.2	1.8	2.6	90
Cebogel 2	6.2	1.9	2.6	89

### A4.5 Conclusions

In the manufacturer's data sheet regarding Cebogel QSE, a typical value of the montmorillonite content is declared to be 80%. The results from the performed XRD analyze gives a montmorillonite content of 90% (which is somewhat higher).

The results from the element analysis are typical for a bentonite with high content of montmorillonite.

The high sodium content detected in the EC-analyze, shows that the material is converted to a sodium state, as declared in the data sheet.

The results from the performed analyses are judged to be in fair agreement with the data provided in the data sheet for Cebogel QSE.

## PRODUCT DATA



### CEBOGEL QSE

#### Use

The large swelling capacity makes CEBOGEL QSE suitable for: The complete repair of drilled-through or damaged clay layers Securing spring-loaded charges in the ground for seismological study Making dams, dykes and water barriers non-water-permeable Rapidly sealing damaged wells, etc..

Careful and even dosing are required for an optimal result. Bridge formation can occur in the event of dosing too rapidly.

#### Description

Cylindrical bentonite rods (granules)made from 100 % activated sodium bentonite. A characteristic of CEBOGEL QSE is its considerable water absorption capacity, as a result of which it swells up considerably when in contact with water. The QSE quality is KIWA certified in the field of toxicological aspects.

#### Advantages

- The assurance of a strong, virtually watertight layer which can only be achieved using a pure sodium bentonite
- Has extra swelling capacity for sealing irregularities in the borehole wall or difficult to reach cavities
- Certified according to KIWA-ATA, therefore absolutely safe for use in drinking water areas
- Easy to apply
- Absolutely environmentally-friendly

#### Specification

Complies with the requirements set in BRL-K20236/01 for borehole day for sealing boreholes in bottom layers with poor water permeability

Supplied with KIWA certificate for Toxicological Aspects (ATA), which guarantees an environmentally-friendly product

Parameter	Method	Requirement	Typical Value
Water absorption capacity	ASTM E946-	≥ 600 % (BRL-	800 %
after 24 hours	92	265/01)	

Cebo Holland BV Westerduinweg 1 NL-1976 BV IJMUIDEN P.O. Box 70 NL-1970 AB IJMUIDEN Tel.: +31 255546262

Fax: 431 255546202 e-mail : sales@cebol olland.com www.oaboholland.com

In so far as we can ascertain the above-stated information is correct. However, we are unable to provide any guarantees with regard to the results that you will achieve with this. This specification is provided on the condition that you determine yourself to what degree it is suitable for your purposes.

Page 1 of 2



#### Typical values

Montmorillonite level	X-ray diffraction	80 %
Moisture content	DIN 18121	16 %

#### Chemical and physical properties

Composition	High-quality activated sodium bentonite		
Colour	Grey green		
Form	Cylindrical rods		
Dimensions	Diameter 6.5 mm Length 5 – 20 mm		
Density	2100 kg/m <sup>3</sup>		
Bulk density	1100 kg/m <sup>3</sup>		

Packaging

1000 kg packed in 25 kg polyethylene bags on a pallet with shrink film
1000 kg big bags

Cebo Holland BV Westerduinweg 1 NL-1976 BV 1DMUIDEN P.O. Box 70 NL-1970 AB 1DMUIDEN

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Revision date : 10-07-2003 Document no : CQ03IP

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