

**P-14-07**

## **KBS-3H**

### **Manufacturing of buffer and filling components for the Multi Purpose Test**

Lars-Erik Johannesson, Clay Technology AB

January 2014

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*Keywords:* KBS-3H, MX-80, Delivery control, XRD, CEC, Mixing of bentonite, Compaction of bentonite blocks.

This report concerns a study which was conducted for SKB. The conclusions and viewpoints presented in the report are those of the author. SKB may draw modified conclusions, based on additional literature sources and/or expert opinions.

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## Abstract

The so called Multi Purpose Test (MPT) is carried out at the Äspö HRL starting 2011 and is part of the KBS-3H project development. The MPT is also part of the LucoeX project and is partly funded by the European Commission.

The MPT includes the key KBS-3H component, the Supercontainer, consisting of a canister surrounded by buffer blocks inside an outer perforated metal shell, distance blocks and a metallic plug with its transition zone (blocks and pellets), Figure 1-2.

This report describes the manufacturing of the buffer components including, delivery control of the bentonite (MX-80), adjusting of the water content of the bentonite, compacting of the blocks, machining of the blocks and controlling of the finished blocks.



Large Underground Concept Experiments

This Project has received funding from EuroAtom/FP7 under grant agreement n°269905

# Sammanfattning

Det så kallade Multi Purpose Test (MPT) genomförs på Äspölaboratoriet under perioden 2011–2014 och ingår som en del i utvecklingsarbetet av KBS-3H-konceptet. MPT-försöket är också en del av LucoeX-projektet och finansieras delvis av EU-kommissionen.

I MPT-försöket ingår alla huvudkomponenter från KBS-3H-konceptet, dvs Supercontainern innehållande en kapsel omgärdad av buffertblock placerad inuti ett perforerat metallskal, distansblock och en metallplug med tillhörande övergångszon (block och pelletar), se figur 1-2.

I denna rapport beskrivs tillverkningsprocessen av buffertblocken inklusive leveranskontrollen av bentoniten (MX-80), blandningen av bentoniten med vatten, samt kompaktering, bearbetning och kontroll av blocken.



Large Underground Concept Experiments

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# 1 Background

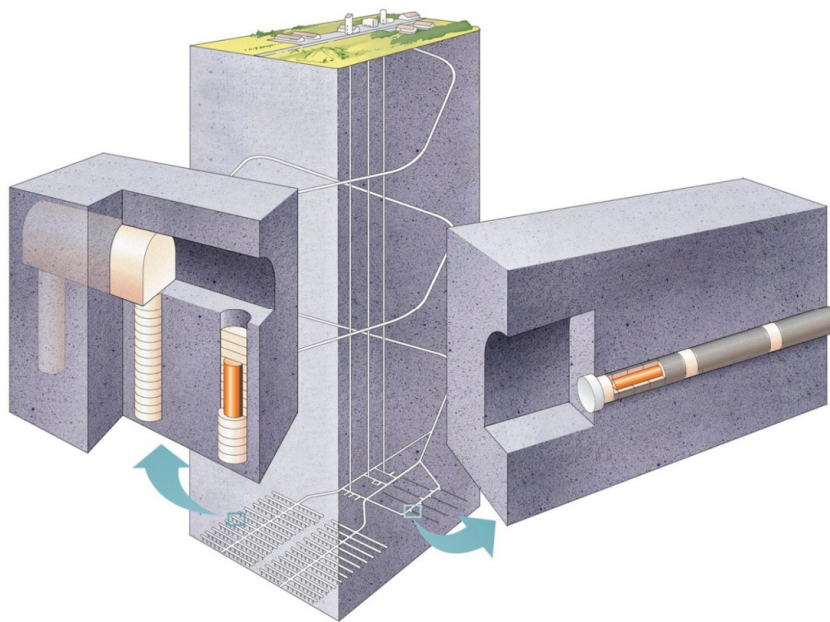
## 1.1 General

SKB and Posiva are planning for disposal of spent nuclear fuel from Swedish and Finnish nuclear power plants at depth in crystalline bedrock to ensure the safety of human beings and the environment for long periods of time. The method selected for the final repository is the KBS-3 method, Figure 1-1. The reference design is KBS-3V employing vertical disposal of the waste canisters, where horizontal disposal of the canisters, KBS-3H, is a possible alternative which is being elaborated by the two organisations. SKB's and Posiva's current programmes for KBS-3 are detailed in SKB's RD&D Programme 2013 (SKB 2013) and in Posiva's TKS-2009 (Posiva 2010).

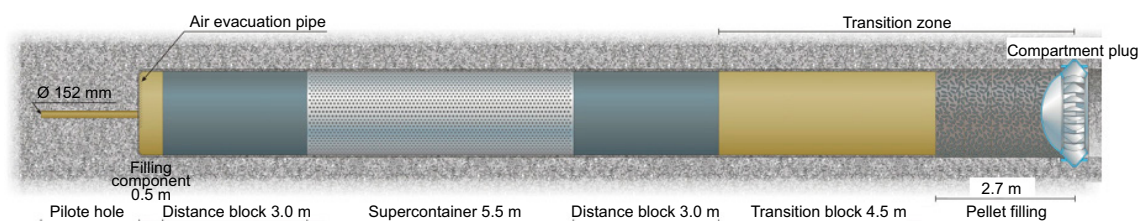
The so called Multi Purpose Test (MPT) is carried out at the Äspö HRL starting 2011 and is part of the KBS-3H project development (SKB 2012). The MPT is also part of the LucioeX project and is partly funded by the European Commission.

The test is basically a shortened non-heated installation of the KBS-3H reference design, Drainage, Artificial Watering and air Evacuation (DAWE), and includes the main KBS-3H components, see Figure 1-2.

The test installation is carried out according to DAWE after which the test conditions are monitored. Dismantling and analysis will be carried out at a later stage and the timing for this will be dependent on the measured data.



**Figure 1-1.** Schematic illustration of the KBS-3 method with its three barriers: the canister, the buffer and the rock. The vertical reference design is illustrated to the left and the horizontal alternative to the right.



**Figure 1-2.** Schematic illustration of the MPT layout. In the KBS-3H reference design multiple Supercontainers separated by distance blocks would be placed in a 300 m long drift.

## 1.2 Buffer components and manufacturing

The MPT includes a Supercontainer, Figure 1-3, consisting of a canister surrounded by buffer blocks inside an outer perforated steel shell. The Supercontainer is pre-assembled in an assembly hall. In the KBS-3H reference design the shell is made of titanium but in the MPT a carbon steel shell is used. Additionally, the MPT rings around the canister are not as thick due to factory limitations, 10 instead of 4 rings are used inside the Supercontainer.

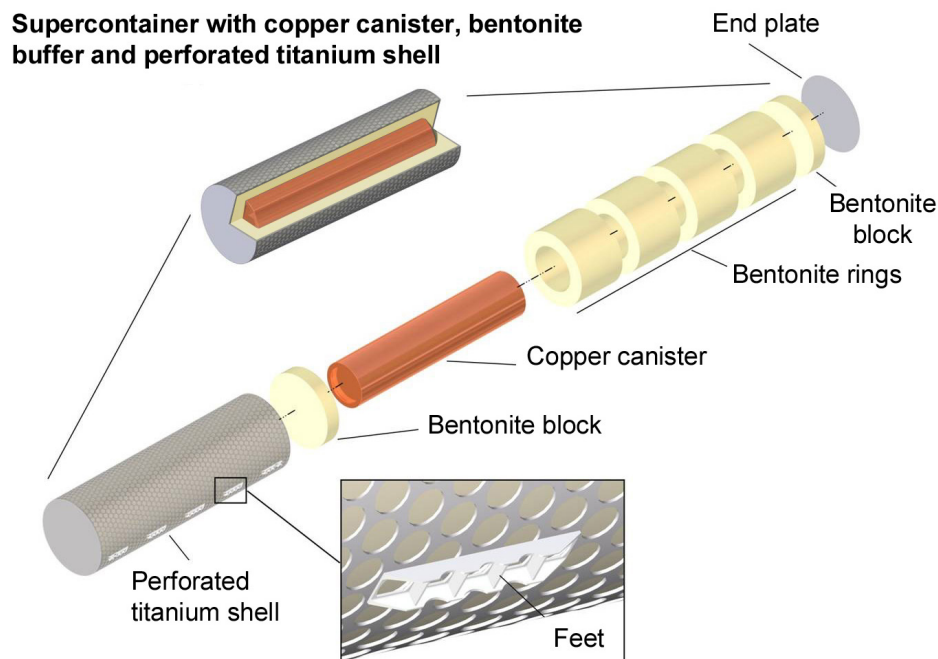
The Supercontainers are separated by cylindrical distance blocks. The purpose of the distance block is to restrict the temperature (not applicable in the MPT), and to seal off each canister position from the next, and to prevent water and bentonite transport along the drift. Their axial length will range roughly from 2 to 6 m dependent on the type of canister and surrounding rock.

The reference buffer material is bentonite clay with the material composition specified in Table 1-1.

The reference designs of the blocks are presented in Table 1-2 and Figure 1-4. The densities are given as dry densities and are described with a nominal value together with an accepted variation. The water contents of the material are given as an accepted interval and an accepted variation for the chosen water content.

**Table 1-1. Reference buffer material.**

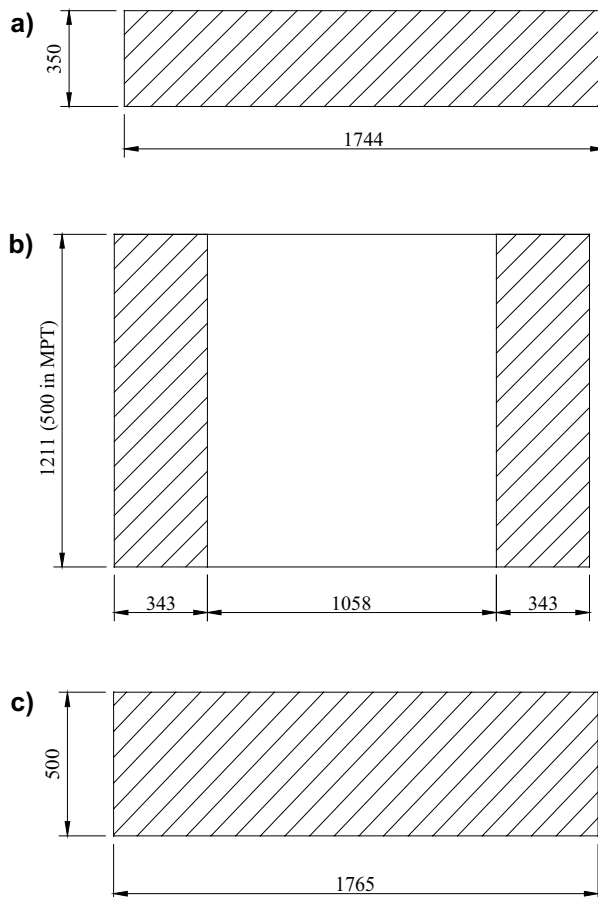
Design parameter	Nominal design [wt-%]	Accepted variation [wt-%]
Montmorillonite content	80–85	75–90
Sulphide content	limited	< 0.5
Total sulphur content (including the sulphide)	limited	< 1
Organic carbon	limited	< 1



**Figure 1-3.** The buffer and canister are embedded in a perforated shell (with solid end plates) forming a Supercontainer in the KBS-3H design.

**Table 1-2. Reference buffer blocks for both inside and outside the Supercontainer.**

Design parameter	Nominal design	Accepted variation
<i>Solid blocks inside the Supercontainer</i>		
Dry density (kg/m <sup>3</sup> )	1,753	±20
Water content (%)	10–17	±1
Dimensions (mm)	Height: 350	±1
	Outer diameter: 1,740	+1/-2
<i>Ring shaped blocks inside the Supercontainer</i>		
Dry density (kg/m <sup>3</sup> )	1,885	±20
Water content (%)	10–12	±1
Dimensions (mm)	Height: 1,211 (500 in MPT)	±1
	Outer diameter: 1,740	+1/-2
	Inner diameter: 1,058	±1
<i>Solid blocks outside the Supercontainer (distance blocks)</i>		
Dry density (kg/m <sup>3</sup> )	1,712	±20
Water content (%)	21	±1
Dimensions (mm)	Height: 500	±1
	Outer diameter: 1,765	



**Figure 1-4.** Schematic drawing of the blocks inside the Supercontainer a) solid blocks, b) ring shaped blocks around the canister and c) outside the Supercontainer (distance blocks).

A mould has been constructed and manufactured where blocks can be compacted with a maximum diameter of 1,800 mm and with a maximum height of 500 mm. The mould has been used for production of all blocks used in the MPT. A drawing of the mould set up for producing ring shaped blocks is shown in Figure 1-5.



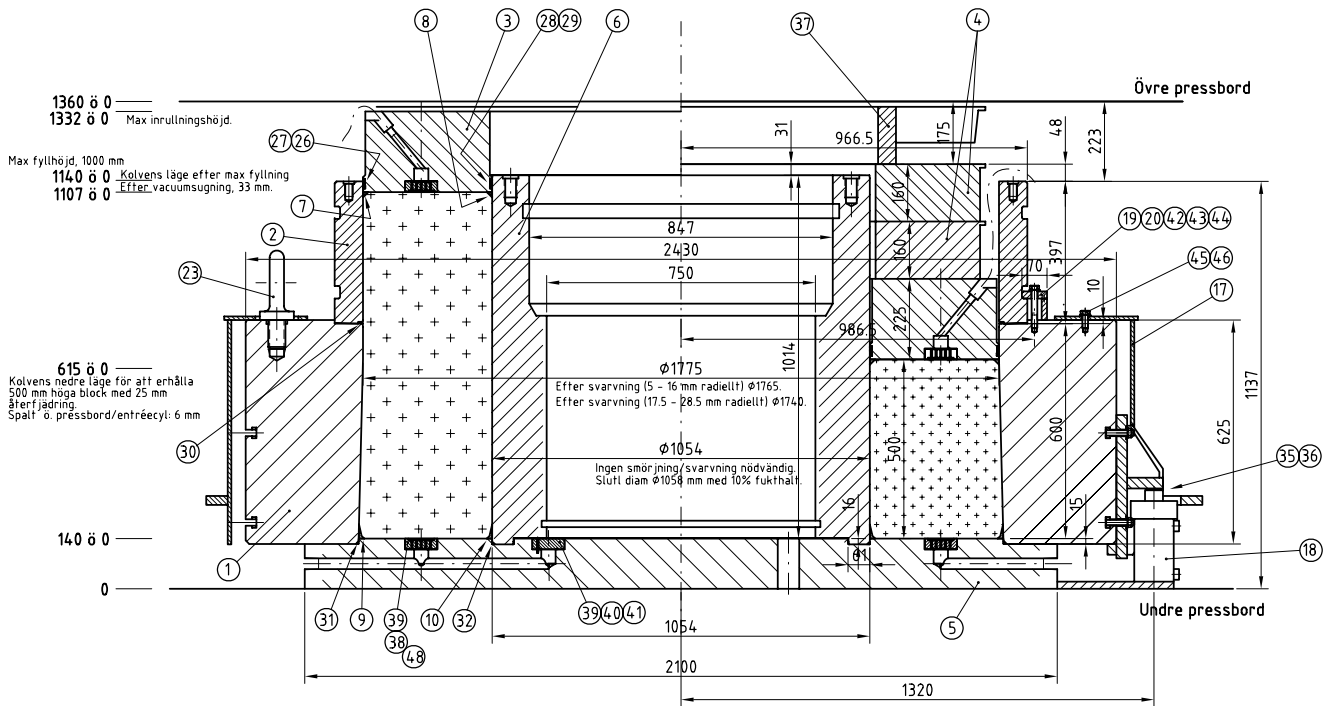


Figure 1-5. A drawing of the mould prepared for compacting ring shaped blocks.

### 1.3 Description of the activities

The activities included in the manufacturing of the buffer blocks are listed in Table 1-3. The activities are listed in the order they were executed. In the following section are the activities described in detail.

Table 1-3. A list over activities included in the manufacturing of the bentonite blocks.

Activity	Description
Control of delivered bentonite	Samples from some of the delivered big-bags of bentonite are taken and investigated with laboratory tests.
Mixing of bentonite	Water is added to the bentonite to yield the right water content
Compaction of bentonite	Bentonite powder is compacted to blocks with a maximum compaction pressure of about 65 MPa.
Investigation of the compacted blocks	A visual inspection of the compacted blocks is made. The weight and the dimensions of the blocks are measured.
Machining of the blocks	The compacted blocks are machined to the stipulated shape
Investigation of the machined blocks	A visual inspection of the machined blocks is made. The weight and the dimensions of the blocks are measured.

## 2 Delivery control of the bentonite material

A delivery of bentonite, approximately 150 tons was purchased by SKB for different large scale experiments at Äspö during the winter 2012. The bentonite was originally from Wyoming and of type MX-80 and produced by AMCOL International Corporation USA. Furthermore the bentonite was delivered in big-bags to Äspö (altogether 167 big-bags). A smaller amount of MX-80 from 2013 was also delivered and used by SKB at the production of the blocks.

A delivery control of the bentonite was made in June 2012 and in June 2013. The delivery control included measurements/tests of the following parameters:

- Compaction properties (only on the delivery from 2012)
- Normalized free swelling
- Liquid limit
- CEC (Cation Exchange Capacity)
- EC (Exchangable Cations)
- XRD (only on the delivery from 2012)
- Chemical composition
- Swelling pressure and hydraulic conductivity

The number of samples and the results from the different investigated parameters are presented in the following sections. The different sections include a short description of the used investigation technique.

### 2.1 Compaction properties

Since most of the bentonite was used for producing highly compacted buffer blocks, it is of great importance to know its compaction properties in advance in order to be able to choose the right compaction pressure for the blocks. These properties were investigated by compacting the material in a small mould ( $\varnothing$  50 mm, height 20 mm). The bentonite was mixed to ten different water contents in a small Eirich mixer and compacted at five different compaction pressures. After the compaction, the sample was removed from the mould and its water content and density were determined.

The determination of the water content was made in the following way:

1. The balance was checked with reference weights before the starting of the measurements.
2. A small baking tin of aluminum was placed on the balance and the weight ( $m_{bt}$ ) was noted in a protocol.
3. The sample was placed in the baking tin and the weight of sample and tin is noted in a protocol ( $m_{bt} + m_{bulk}$ ).
4. The tin with the sample was placed in an oven with a temperature of 105°C for 24 h.
5. After the drying the weight of the baking thin and the sample ( $m_{bt} + m_{solid}$ ) was measured and noted in a protocol.

The mass of water dried from the sample was determined according to Eqn. 2-1:

$$m_{water} = m_{bulk} - m_{solid} \quad (2-1)$$

and the water content ( $w$ ) was calculated according to Eqn. 2-2.

$$w = \frac{m_{water}}{m_{solid}} \quad (2-2)$$

The bulk density of the samples was determined by weighing the samples both in air and immersed in paraffin oil with known density. The determination was made as follows:

1. A piece of thread was weighed.
2. The sample was weighed hanging on the thread underneath the balance ( $m_{bulk}$ ).
3. The sample was then submerged into the paraffin oil with the density  $\rho_{paraffin}$  and the weight ( $m_{paraffin}$ ) was noted.

The volume of the sample ( $V_{bulk}$ ) and the density ( $\rho_{bulk}$ ) were calculated according to Eqns. 2-3 and 2-4.

$$V_{bulk} = (m_{bulk} - m_{paraffin}) / \rho_{paraffin} \quad (2-3)$$

$$\rho_{bulk} = \frac{m_{bulk}}{V_{bulk}} \quad (2-4)$$

The dry density ( $\rho_{dry}$ ) and the degree of saturation ( $S_r$ ) can be calculated according to Eqns. 2-5 and 2-6.

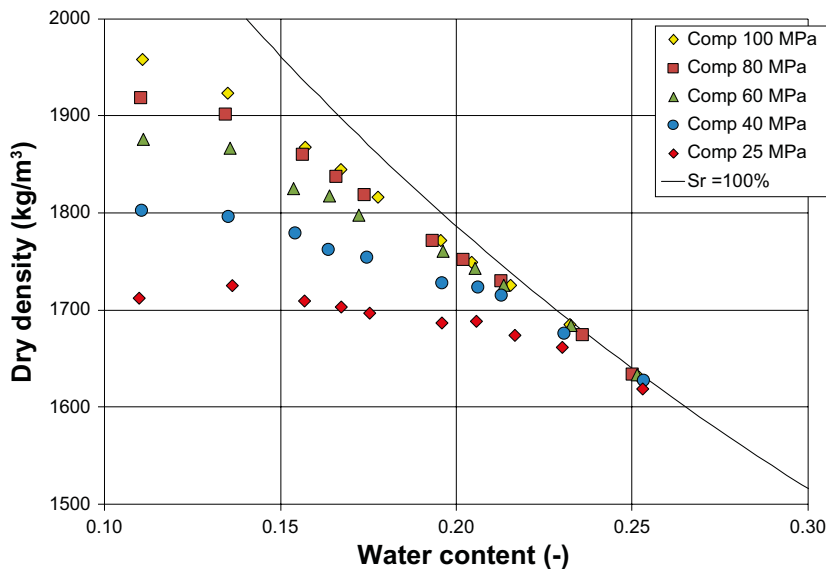
$$\rho_{dry} = \frac{\rho_{bulk}}{(1+w)} \quad (2-5)$$

$$S_r = \frac{w \times \rho_{bulk} \times \rho_s / \rho_w}{\rho_s \times (1+w) - \rho_{bulk}} \quad (2-6)$$

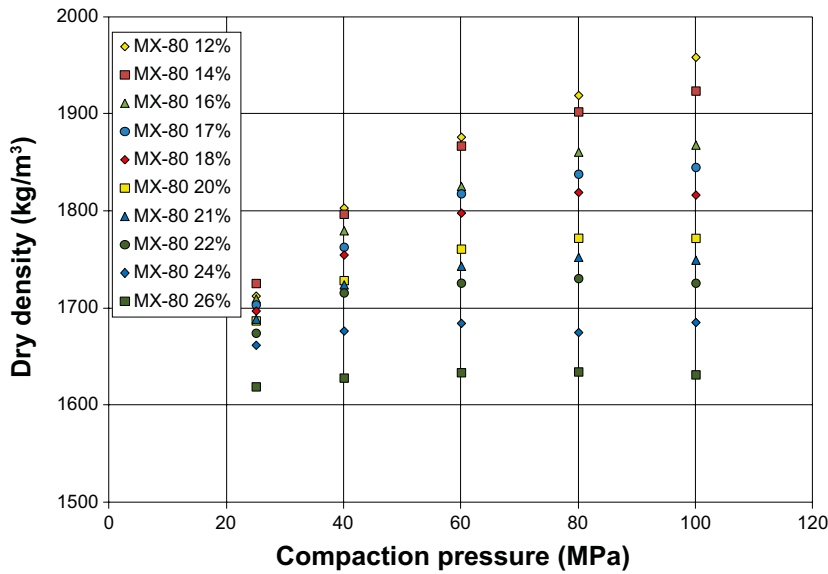
For calculating the degree of saturation, the values of the density of the solid particles  $\rho_s = 2,780 \text{ kg/m}^3$  (see Karnland et al. 2006) and the density of water to  $\rho_w = 1,000 \text{ kg/m}^3$  are used. The void ratio ( $e$ ) can be calculated according to Eqn. 2-7.

$$e = \frac{\rho_s - \rho_{bulk}}{\rho_{bulk} - \rho_w \times S_r} \quad (2-7)$$

At the test the compaction stress varied between 25–100 MPa and the water content of the bentonite varied between 11–25%. The results from all the tests are summarized in Figure 2-1 where the dry density of the samples is plotted as function of the water content together with the saturation curve for the bentonite assuming the density of the particles in the bentonite is  $2,780 \text{ kg/m}^3$  and the density of the water is  $1,000 \text{ kg/m}^3$ . The same data set is plotted in Figure 2-2 where the dry density for the samples is plotted as function of the compaction pressure.



**Figure 2-1.** The dry density of the samples as function of the water content for samples compacted at five different compaction pressures.



**Figure 2-2.** The dry density of the samples as function of the compaction pressure for samples with 10 different water contents.

## 2.2 Normalized free swelling

The swelling index for a bentonite is measured by pouring about 1 g bentonite into a measuring glass filled with 100 ml de-ionized water and after 24 hours measure the volume of the expanded clay. The volume of the clay is normalized to the dry weight of 1 g is considered to be the swelling index. The determination of the normalized free swelling ( $V_n$ ) was made in the following steps:

1. Determine the water content ( $w$ ) of the material, Eqn. 2-2.
2. Weigh approximately 1.1 g of the test material and note down the sample mass ( $m$ ) with mg resolution.
3. Calculate the solid mass ( $m_s$ ) of the sample, Eqn. 2-8.
4. Fill the graduated measuring glass with 100 ml de-ionized water.
5. Pour down the 1.1 g test material slowly, and small portions at the time, in order not to let the material stay at the water surface but fall down as individual grains.
6. Leave the sample over night.
7. Note down the volume ( $V$ ) of the swollen sample.
8. Calculate the normalized free swelling ( $V_n$ ), Eqn. 2-9.

$$m_s = \frac{m}{w + 1} \quad (2-8)$$

$$V_n = \frac{V}{m_s} \quad (2-9)$$

The results from altogether 154 determinations of swelling index are shown in Figure 2-3. The average value of the normalized free swelling was 12.9 ml/g with a standard deviation of 0.57 ml/g.

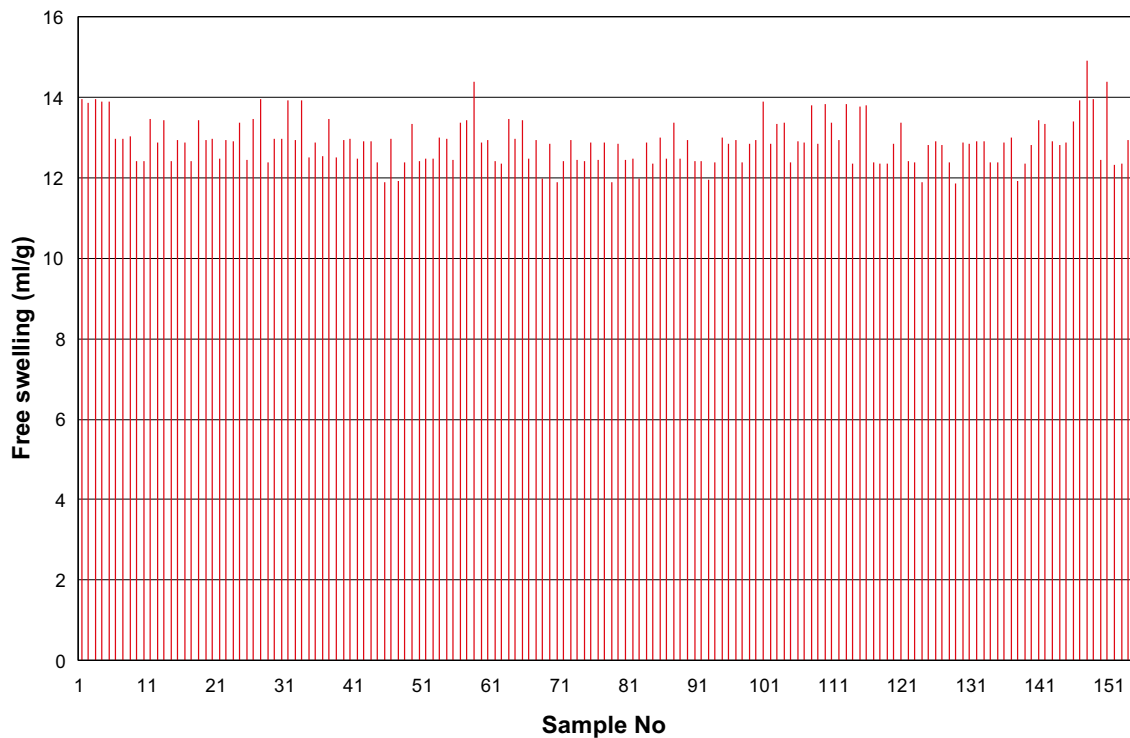


Figure 2-3. The evaluated normalized free swelling for 154 samples of MX-80.

## 2.3 Liquid limit

The liquid limit for a clay is defined as the water content ( $w_l$ ) at which the clay is changed from a plastic state to a liquid state. The liquid limit was determined on altogether 33 samples taken from every fifth delivered big-bags.

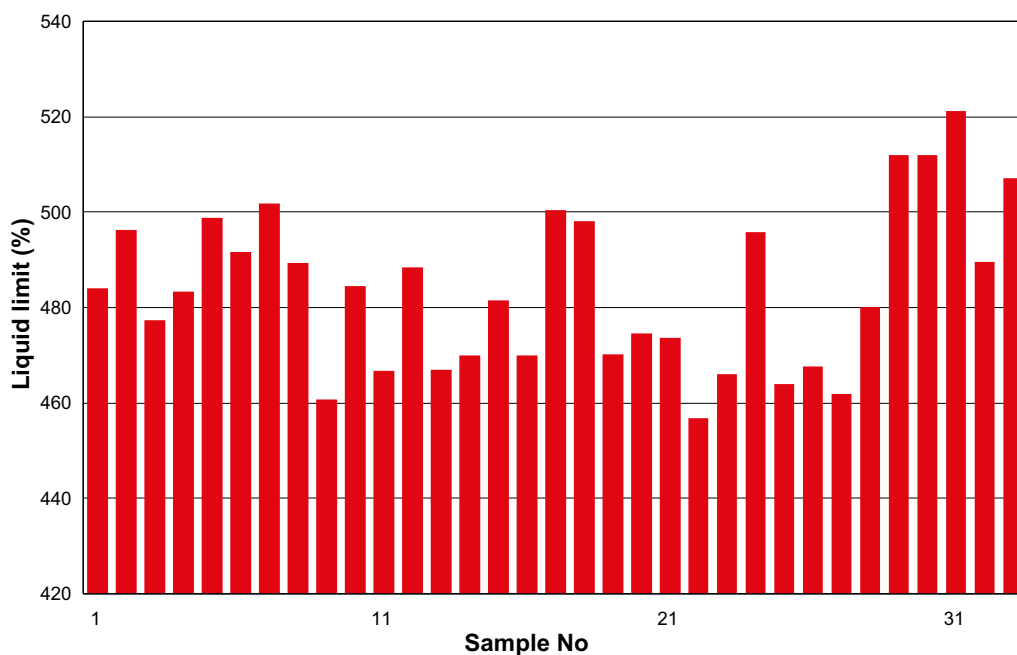
The determination is based on the one-point method for liquid limit determination according to the cone method as describe in the “Consistency Limits” part of the Laboratory manual series of the Swedish Geotechnical Society (SGF). The bentonite is not acting as ordinary clays which implied that the standard technique has to be changed with respect to the mixing procedure and the rest period after mixing. The determination was made according the following:

1. Weigh 125 g of pure water (de-ionised) in a glass beaker.
2. Stir the water by use of an electrical propeller mixer.
3. Slowly pour down approximately 28 g of bentonite material into the **stirred** water.
4. Mix thoroughly for another 2 minutes.
5. Fill a plastic cup with the bentonite paste and smoothen the surface.
6. Cover the bentonite paste in the beaker with a plastic bag, and the cup with a cap, and place it to rest for 24 h.
7. Drop the cone three times and read the penetration depth by use of the magnifying glass and calculate the average penetration depth, see Figure 2-4.
8. Take approximately 5 g of paste from the hit position and determine its water content ( $w$ ) according to Eqn. 2-2.
9. Determine the liquid limit according to the one-point method according to the “Consistency Limits” part of the Laboratory manual series of the Swedish Geotechnical Society (SGF).

The results from the made determinations are shown in Figure 2-5. The average value of the liquid limit was 483.7% with a standard deviation of 16.9%.



**Figure 2-4.** The cone apparatus used for determining the liquid limit of the bentonite.



**Figure 2-5.** The evaluated liquid limit for 33 samples of MX-80.

## 2.4 Cation exchange capacity analyses (CEC)

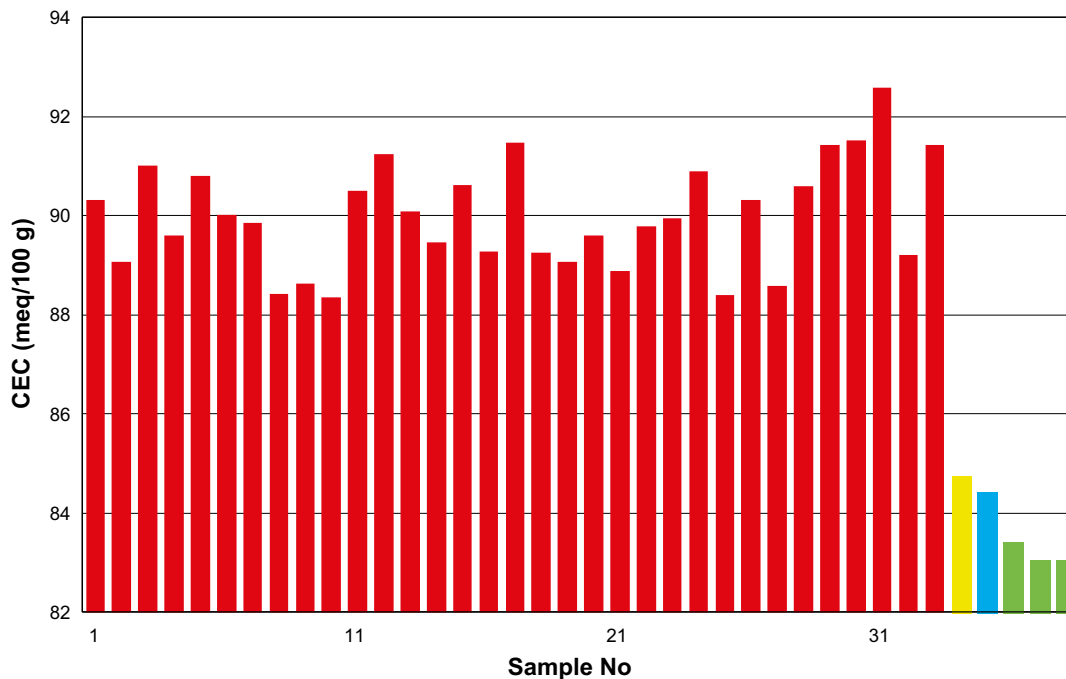
The CEC of the bulk materials was determined by exchange with the copper(II)-triethylenetetramine complex.

The specimens for the copper(II)-exchange methods were grounded and dried at 60°C overnight before analysis.

The procedure follows the recommendations of Meier and Kahr (1999). The absorbance at 620 nm of the Cu-trien-complex before and after the exchange reaction is determined by use of a spectrophotometer (Shimadzu) and CEC is calculated by the difference in the Cu-concentration. All determinations were duplicated.

CEC analyses were made on every fifth delivered big-bag of the delivery from 2012 during the summer of 2012. The results are plotted, marked in red, in Figure 2-6. The average value of the CEC was 90 meg/100 g with a standard deviation of 1.1 meg/100 g. These values were higher than expected. Another three tests were made on the delivery with similar results.

Supplementary investigations of the CEC on different deliveries of MX-80 were made during the summer of 2013. The results from these investigations are also plotted in Figure 2-6.



**Figure 2-6.** The evaluated CEC for 33 samples of MX-80 from the 2012 delivery marked with red, one sample from a delivery from 2001 marked with yellow, on sample from the delivery from 2012 marked with blue and three samples from a delivery from 2013 marked with green.

At the investigation CEC was determined on a sample from a delivery of MX-80 from 2001 (marked with yellow), on one sample from a delivery from 2012 (marked with blue) and three samples from a delivery from 2013 (marked with green). The CEC values determined 2013 were lower compared to the previous determined values. The results imply that there was something wrong with the CEC analyses made during 2012. Despite reviewing of the data from the investigations no explanation of the differences were found.

The variation in CEC between the three different deliveries were small.

## 2.5 Exchangeable cations analyses (EC)

The exchangeable cations of the bulk bentonite were determined by extraction into alcoholic ammonium chloride solution according to a procedure originally recommended for CEC determinations of gypsiferous/calcareous soils (e.g. Belyayeva 1967, Jackson 1975). An alcoholic solution was used to minimize dissolution of gypsum and calcite, which are soluble in aqueous solutions. Ideally, i.e. when there is a minimum of easily soluble salts, such as chlorides and carbonates of alkali metals, the sum of extracted cations should be equivalent to the CEC of the sample.

**Table 3-5. Exchangeable cations, extracted by exchange against  $\text{NH}_4^+$  in 80% alcohol solution.**

Sample id	Na meq/100 g	%	Ca meq/100 g	%	K meq/100 g	%	Mg meq/100 g	%	Sum meq/100 g
Delivery 2001	58.3	63	23.0	25	2.1	2	9.5	10	92.8
Delivery 2012 A	58.1	65	22.2	25	2.2	2	7.5	8	90.0
Delivery 2012 B	63.9	66	22.2	23	2.2	2	8.1	8	96.3
Delivery 2012 C	59.6	64	23.0	25	2.2	2	7.6	8	92.4
Delivery 2012 D	64.2	69	20.5	22	2.1	2	6.9	7	93.7
Delivery 2013 A	58.9	64	22.3	24	2.0	2	8.6	9	91.8
Delivery 2013 B	58.6	65	21.2	24	2.0	2	8.4	9	90.2
Delivery 2013 C	57.7	64	21.9	24	2.0	2	8.2	9	89.8

## 2.6 X-ray diffraction analyses (XRD)

The XRD-analysis of the bulk samples mainly aims at identifying the accessory minerals in the samples. In order to produce specimens with randomly oriented powders of the bulk material, the material was ground in an agate mortar to a grain-size  $< 10 \mu\text{m}$ . This preparation technique gives complete “three-dimensional fingerprints” of all types of minerals and is needed for a general characterization of the bulk materials and for quantitative evaluations. Prior to X-ray scanning, the samples were equilibrated at ambient relative humidity.

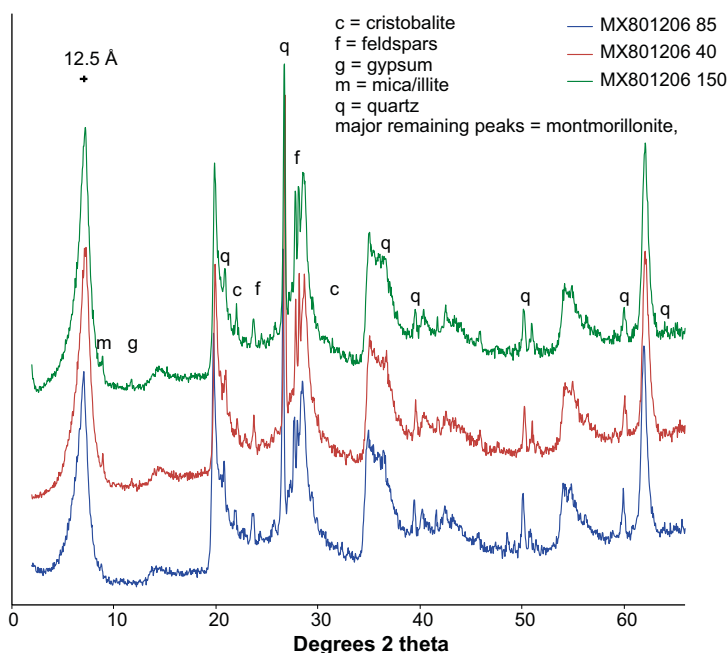
A Seifert 3000 TT X-ray diffractometer with  $\text{CuK}\alpha$  radiation and automatic slits was used for the X-ray diffraction analyses. The samples were scanned in the interval  $2$  to  $66^\circ 2\theta$ .

XRD was made on three samples (big-bag no 40, 85 and 150). The results from the analyses are presented in Figure 2-7. The mineralogical composition of the materials was evaluated from the XRD diffractograms (Figure 2-7) by use of the Siroquant quantitative XRD software. The modeling is based on the Rietveld refinement method of least squares fit of calculated to measured XRD profiles (Rietveld 1969). The method is described in general and used for montmorillonite in Taylor and Matulis (1994). The present analyses were made in a standardized way, including identification of present minerals by peak positions, and a subsequent modeling by use of the Siroquant mineral database.

The results from the analyses of the three samples are summarized in Table 2-1. The analyses indicate that the average montmorillonite content is about 90% and the variation among the three samples is small. The average montmorillonite content is within the acceptance criteria for a buffer material (cf. Table 1-1).

**Table 2-1. The mineralogical composition of 3 samples evaluated from the XRD diffractograms with the use of the Siroquant quantitative XRD software.**

Phase	MX801206 40		MX801206 85		MX801206 150	
	Weight (%)	Error of fit (%)	Weight (%)	Error of fit (%)	Weight (%)	Error of fit (%)
Montmorillonite, (CP)	90.3	1.7	89.7	1.5	90.5	1.9
Gypsum	0.5	0.3	0.3	0.3	0.7	0.4
Quartz	5.3	0.2	4.9	0.2	4.3	0.3
Calcite 1	0	0.3	0.2	0.2	0	0.3
Plagioclase	2.9	1	3.9	0.9	3.7	1.1
Muscovite	1	0.9	0.9	0.8	0.9	1



**Figure 2-7. X-ray diffractograms of 3 samples of MX-80.**



## 2.7 Chemical composition

The chemical composition of the bulk materials from three different deliveries of MX-80 was determined at a certified laboratory (ACME) Analytical Laboratories, Canada. Major and refractory elements, and rare earths were determined by ICP emission or mass spectroscopy using standard techniques for silicate analysis (LiBO<sub>2</sub>/Li<sub>2</sub>B<sub>4</sub>O fusion followed by nitric acid digestion). Precious metals and base metals (Mo, Cu, Pb, Zn, Ni, As, Cd, Sb, Bi, Ag, Au, Hg, Tl, Se) were determined by ICP mass spectroscopy in leachates of aqua regia. Loss on ignition (LOI) was determined as the difference in weight of the dried (at 105/60°C) and the ignited sample (at 1,000°C). Total carbon and sulfur were determined by evolved gas analysis (EGA) at the same laboratory by combustion of the samples in a Leco furnace, equipped with IR-detectors. Carbonate carbon was determined as CO<sub>2</sub> evolved on treatment of a sub-sample with hot 15% HCl. Sulfate was determined on separate samples pre-ignited at 550°C. The concentration of sulfide-S may be estimated by subtracting the sulfate-S concentration from the total concentration of S. Similarly, the concentration of organic C can be estimated by subtracting inorganic C from the total concentration of C.

The results from the analyses are shown in Table 2-2 indicating that the chemical compositions of the three different deliveries are similar.

**Table 2-2. Chemical composition of MX-80 samples delivered at three different occasions. DL = detection limit.**

Sample id	SiO <sub>2</sub> %	Al <sub>2</sub> O <sub>3</sub> %	Fe <sub>2</sub> O <sub>3</sub> %	MgO %	CaO %	Na <sub>2</sub> O %	K <sub>2</sub> O %	TiO <sub>2</sub> %	P <sub>2</sub> O <sub>5</sub> %	MnO %	Cr <sub>2</sub> O <sub>3</sub> %	LOI %	Sum %	tot C %	tot S %	
DL	0.01	0.01	0.04	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.002	-5.1	0.01	0.02	0.02	
Delivery 2001	62.02	19.62	3.61	2.38	1.20	2.09	0.54	0.14	0.05	< 0.01	0.006	8.1	99.81	0.32	0.29	
Delivery 2012	60.76	20.61	4.00	2.46	1.34	2.21	0.67	0.17	0.05	0.02	0.005	7.5	99.81	0.37	0.29	
Delivery 2013 A	60.33	19.71	4.37	2.37	1.56	2.19	0.68	0.22	0.09	0.03	0.006	8.3	99.81	0.43	0.28	
Delivery 2013 B	60.68	19.97	4.39	2.40	1.44	2.19	0.68	0.21	0.08	0.02	0.003	7.8	99.81	0.41	0.31	
Delivery 2013 C	60.35	19.97	4.50	2.41	1.55	2.20	0.69	0.22	0.08	0.03	0.006	7.8	99.80	0.41	0.25	
Sample id	Sc ppm	Ba ppm	Be ppm	Co ppm	Cs ppm	Ga ppm	Hf ppm	Nb ppm	Rb ppm	Sn ppm	Sr ppm	Ta ppm	Th ppm	U ppm	V ppm	W ppm
DL	1	1	1	0.2	0.1	0.5	0.1	0.1	0.1	1	0.5	0.1	0.2	0.1	8	0.5
Delivery 2001	0.4	301	< 1	1.9	0.4	29.5	7.3	27.9	13.1	8	282.4	2.6	42.4	14.3	26	< 0.5
Delivery 2012	0.5	302	< 1	1.3	0.5	31.3	9.0	28.9	17.0	9	301.5	3.3	46.4	15.3	20	< 0.5
Delivery 2013 A	0.7	357	1	4.6	0.7	27.5	7.8	26.1	18.5	9	329.4	2.9	40.0	14.4	28	< 0.5
Delivery 2013 B	0.7	301	2	3.4	0.7	27.7	8.4	26.5	17.8	9	314.1	3.3	40.5	14.8	24	0.6
Delivery 2013 C	0.6	353	1	5.0	0.6	26.5	8.2	26.6	17.6	9	332.2	2.8	41.3	14.4	26	0.6
Sample id	Zr ppm	Y ppm	La ppm	Ce ppm	Pr ppm	Nd ppm	Sm ppm	Eu ppm	Gd ppm	Tb ppm	Dy ppm	Ho ppm	Er ppm	Tm ppm	Yb ppm	Lu ppm
DL	0.1	0.1	0.1	0.1	0.02	0.3	0.05	0.02	0.05	0.01	0.05	0.02	0.03	0.01	0.05	0.01
Delivery 2001	210.4	43.9	56.4	119.8	13.78	52.4	10.59	0.69	9.77	1.36	9.20	1.52	4.08	0.57	3.82	0.51
Delivery 2012	200.3	44.9	59.4	121.7	14.01	47.3	11.12	0.77	9.59	1.43	8.42	1.54	3.86	0.63	4.09	0.57
Delivery 2013 A	201.2	46.5	54.0	117.4	13.48	50.3	10.55	0.80	10.09	1.42	8.82	1.60	4.35	0.66	3.80	0.55
Delivery 2013 B	210.5	45.1	53.3	110.9	13.00	48.6	10.71	0.79	9.46	1.39	8.56	1.44	4.06	0.59	3.97	0.59
Delivery 2013 C	202.6	46.4	53.9	108.7	13.01	50.5	10.65	0.81	9.58	1.43	8.99	1.58	4.12	0.67	4.06	0.59
Sample id	Mo ppm	Cu ppm	Pb ppm	Zn ppm	Ag ppm	Ni ppm	As ppm	Au ppm	Cd ppm	Sb ppm	Bi ppm	Hg ppm	Tl ppm	Se ppm	C/org %	SO <sub>4</sub> %
DL	0.1	0.1	0.1	1	0.1	0.1	0.5	0.5	0.1	0.1	0.1	0.01	0.1	0.5	0.02	0.02
Delivery 2001	2.5	5.4	42.9	99	0.2	3.1	11.5	0.9	0.4	0.5	1.0	< 0.01	0.2	0.9	0.05	0.59
Delivery 2012	2.9	4.6	42.4	63	0.2	2.6	12.4	0.8	0.2	0.4	1.0	< 0.01	0.3	< 0.5	0.12	0.62
Delivery 2013 A	3.0	10.1	42.7	94	0.1	5.3	10.7	1.2	0.3	0.4	1.1	< 0.01	0.2	< 0.5	0.13	0.68
Delivery 2013 B	2.8	9.0	39.7	75	0.1	4.2	10.3	1.3	0.3	0.4	1.0	< 0.01	0.2	< 0.5	0.10	0.82
Delivery 2013 C	3.3	9.5	40.9	68	0.2	5.8	10.6	5.1	0.2	0.4	1.0	< 0.01	0.2	< 0.5	0.10	0.76

## 2.8 Swelling pressure and hydraulic conductivity

A swelling pressure device was used to determine hydraulic conductivity and swelling pressure. The parameters are functions of the density of the sample and the chemical composition of the water and the buffer material.

The acid proof stainless steel test equipment used for hydraulic conductivity and swelling pressure measurements is shown in Figure 2-8. The specimens were confined by a cylinder ring with a diameter of 35 mm and stainless steel filters at the top and bottom. The test volume was sealed by o-rings placed between the bottom plate and the cylinder ring and between the piston and the cylinder ring. At test start, the height of the test specimen was approximately 15 mm. A load cell placed between the piston and the upper lid measured the axial force from the samples.

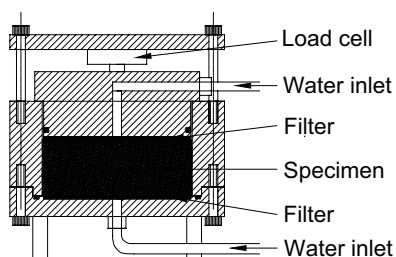
Three different deliveries of MX-80 have been tested. The deliveries are from 2001, 2012 and 2013. The delivery from 2001 was used for the compaction of the buffer blocks in the Prototype Repository project at Äspö Hard Rock Laboratory. The other two investigated deliveries were used for the production of the blocks KBS3H project.

The specimens were compacted in a press to a certain density and then mounted in the swelling pressure device. The specimens were saturated by introducing water to the bottom and top side simultaneously. Water was added after evacuation of air from filters and tubes by a vacuum pump. For all specimens, except for two specimens, ground water from the Prototype test site was used. The main composition of the water used at the saturation of the specimens is shown in Table 2-3. For the two other specimens distilled water was used. During the saturation a minor water pressure of approximately 5 kPa was applied. The swelling pressure was measured continuously.

After saturation of the specimens, a pore pressure gradient was applied and the volume of the outflowing water measured until stable rate was observed. The hydraulic conductivity was then calculated according to Darcy's law. The gradient during the tests was between 2,500 and 10,100 m/m which corresponded to pore pressure differences of 400 kPa and 1,500 kPa over the samples. The measurements of the outflow were made during several days in order to get stable values of the evaluated hydraulic conductivity. The water pressure was thereafter reduced to zero and the test was terminated. The swelling pressure was evaluated just before the increase of pore pressure. The water content and density were determined for each specimen after the tests according to Section 2.1.1.

**Table 2-3. Main composition of the ground-water used for the laboratory tests.**

HD0025A Units	Na mM	K mM	Ca mM	Mg mM	HCO <sub>3</sub> mM	Cl mM	SO <sub>4</sub> mM	Br mM	F mM	Si mM	pH	E.C. mS/m
2002-09-17	92.6	0.26	49.9	2.00	0.63	195.0	4.78	0.554	0.08	0.18	7.42	1,907
2006-05-19	106.1	0.27	58.9	1.89	0.24	213.8	5.28	0.621	0.08	0.16	7.62	2,110
2006-10-02	98.7	0.26	56.6	1.90	0.21	219.2	5.57	0.617	0.08	0.21	7.62	2,170
2007-10-03	114.8	0.26	69.1	2.06	0.18	249.9	5.70	0.741	0.07	0.20	7.64	2,380
2008-09-16	–	–	–	–	0.19	225.9	5.54	0.582	0.09	0.00	7.79	2,230
2010-11-30	106.1	0.23	63.9	1.76	0.16	231.6	5.60	0.703	0.10	0.20	7.40	2,261

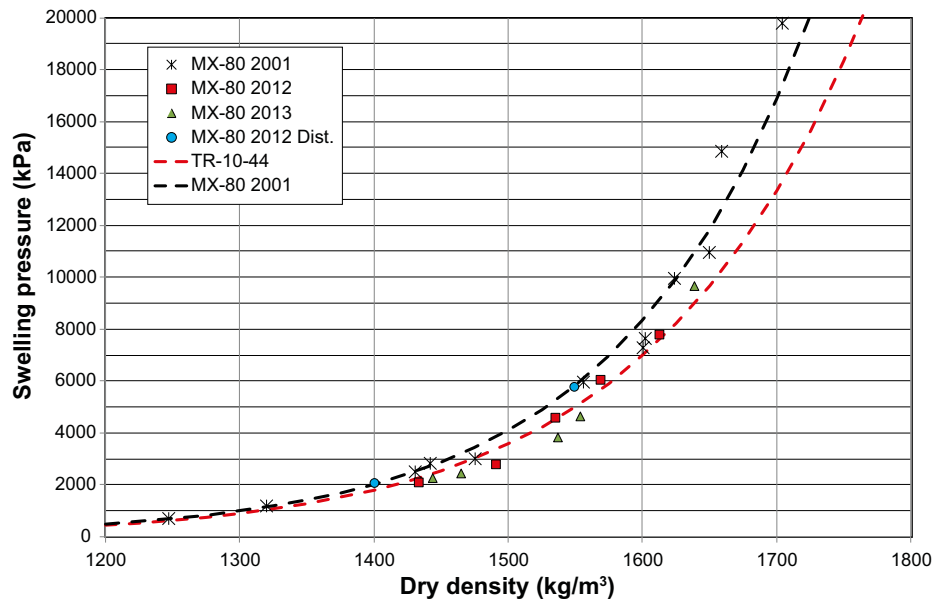


**Figure 2-8.** A picture and a schematic drawing of the swelling pressure device.

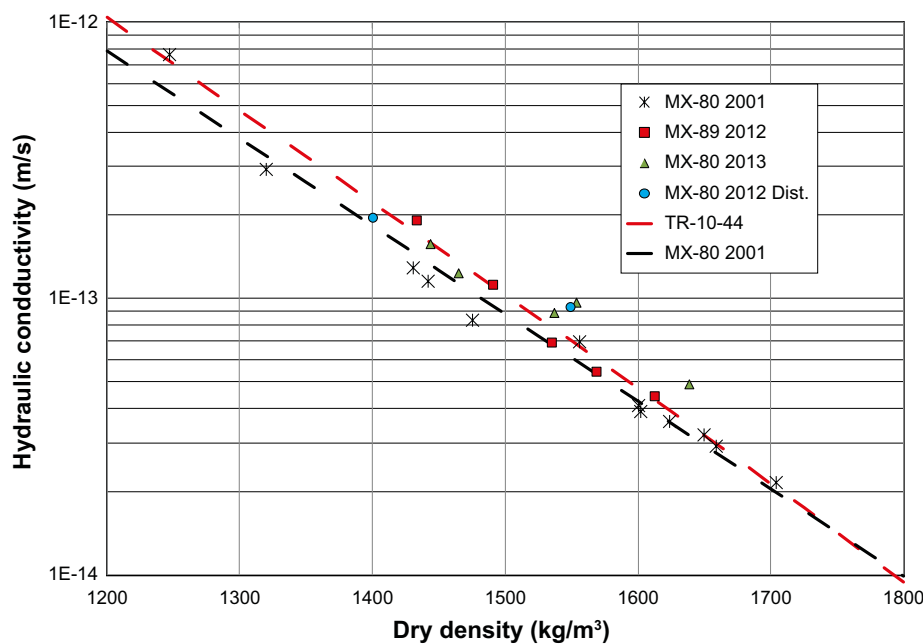
The results from measurements of swelling pressure and hydraulic conductivity are shown in Figure 2-9 and Figure 2-10. In the figures also the best fitting line (logarithmic) of the tests made on MX-80 2001 (black dotted line) and the relation presented in SR-Site Data report (Åkesson et al. 2010) (red dotted line) are plotted.

Figure 2-9 shows that the swelling pressure is similar for all the investigated deliveries of MX-80 although there is a small tendency that the deliveries from 2012 and 2013 have lower swelling pressure compare with the delivery from 2001.

Also for the hydraulic conductivity there is a small difference between the deliveries from 2012 and 2013 compared to the delivery from 2001.



**Figure 2-9.** The swelling pressure as function of the dry density for three different deliveries of MX-80 bentonite. The dotted lines are the best fitting line (logarithmic) of the tests made on MX-80 2001 (black dotted line) and the relation presented in SR-Site Data report (Åkesson et al. 2010) (red dotted line).



**Figure 2-10.** The hydraulic conductivity as function of the dry density for three different deliveries of MX-80 bentonite. The dotted lines are the best fitting line (logarithmic) of the tests made on MX-80 2001 (black dotted line) and the relation presented in SR-Site Data report (Åkesson et al. 2010) (red dotted line).

### 3 Mixing of bentonite

The bentonite used for the blocks had three different water content about 11%, 17% and 21%. In order to reach this water content, water was added to the bentonite in a mixer. The mixing of the bentonite was performed at Äspö Hard Rock Laboratory in a mixer. The mixer, see Figure 3-1, is an Erich mixer and the maximum batch that can be handled in this mixer is about 500 kg.

The bentonite of type MX-80, delivered in big-bags, was filled into a silo placed beside the mixer and then transported to the mixer with a conveyor screw. About 350 kg of bentonite was mixed in each batch. A small sample was taken from each delivered big-bag for determining the initial water content of the bentonite. This water content and the total amount of bentonite were used for calculating the amount of water needed in order to get the final water content. Each batch was mixed for about 3 minutes after the water was added. The final water content of the batches was determined by drying small samples in 105 C for 24 h. The average, the standard deviation and the 95% confidence interval for the water content after mixing for the big-bags (each of them consisting of 3 batches) are shown in Table 3-1 below. The clay was stored indoors after mixing. The value of the water content should be compared with the reference water content listed in Table 1-2. It is obvious that the 95% confidence interval for the mixtures with the expected water content of 21% is outside the accepted variation of  $21\% \pm 1\%$ .

**Table 3-1. Average water content and standard deviation for the big-bags of bentonite mixed for the KBS-3H project.**

Expected water content (%)	Number of big-bags	Average water content (%)	STDV water content (%)	95% confidence interval water content (%)
11	24	10.8	0.20	10.4–11.2
17	18	16.9	0.30	16.3–17.5
21	87	20.7	0.53	19.7–21.8



*Figure 3-1. The Erich mixer used at Äspö Hard Rock Laboratory.*

## 4 Block compaction

The mixed bentonite was transported in the big-bags with a covered lorry to GEA Heat Exchangers AB in Ystad for compaction. The compaction was made by personnel at GEA Heat Exchangers AB and from SKB in a press with a maximum capacity of 30.000 tons. The bentonite was filled into the mould by hanging the big-bags from an overhead crane over the mould and then slowly filling the mould, see Figure 4-1. The required amount of bentonite for one block was filled into the mould. The mould was placed in the press with a subsequent compaction of the bentonite. After removing the block from the mould it was placed on a pallet using specially designed lifting equipment, see Figure 4-4, and a cap was placed over the block in order to prevent the block from drying.

Table 4-1 shows the program for compacting altogether 43 blocks. In order to get a buffer with a homogenous density after saturation, the cylindrical blocks have to be compacted to a lower density than the ring shaped blocks. Hence the cylindrical blocks must be compacted with a lower pressure.

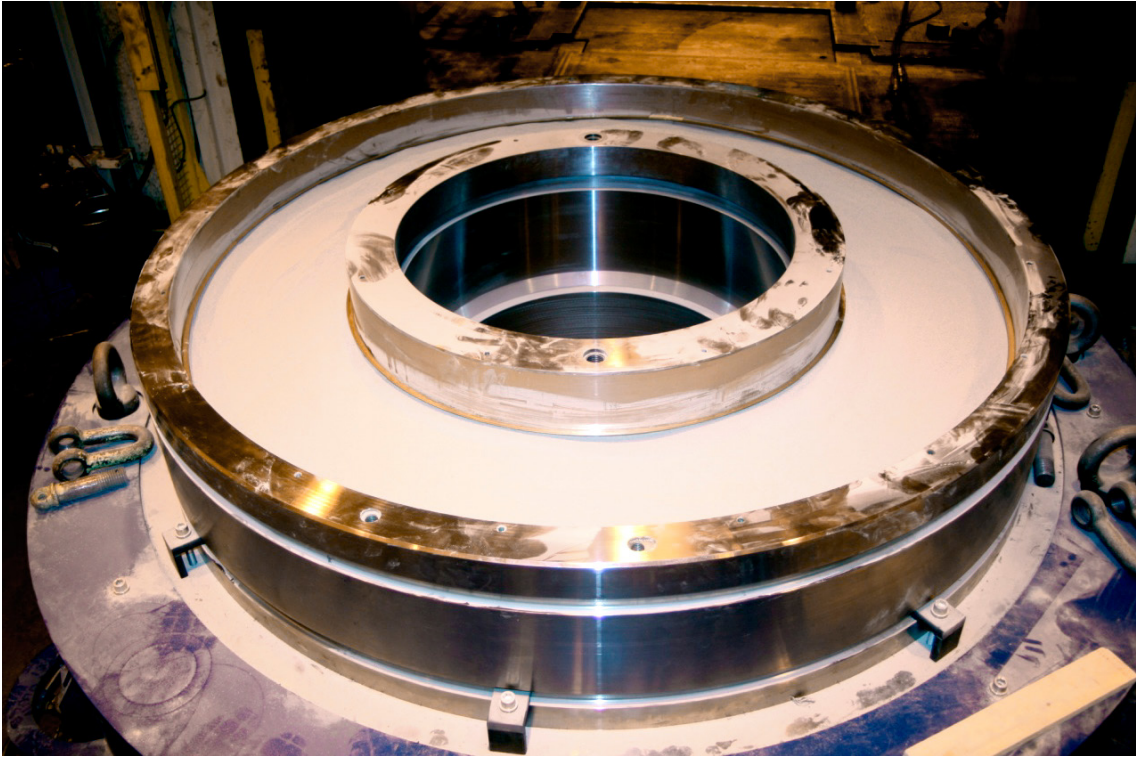
The bulk densities were expected to be 2,110 kg/m<sup>3</sup> for the ring shaped blocks and 2,070 kg/m<sup>3</sup> for the cylindrical blocks for placement inside the Supercontainer. The expected density for the distance blocks was 2,070 kg/m<sup>3</sup>.

The compaction was made in the following sequence:

- The mould was mounted outside the press and lubricated with MOLYKOTE BR 2 plus<sup>®</sup>, which is a lubricant for lubricating at high pressure.
- Material was portioned into the mould.
- A sample (about 3 kg) was taken from the bentonite in the mould and marked with the same number as the compacted block and the date of compaction.
- Since the gap in the press is small (see Figure 4-2) the compaction has to be made in three steps by placing small pistons on top of each other after each step. The first piston was placed on top of the bentonite in the mould and the mould was placed in the press. The tubes from the filters were connected to a vacuum pump and air was evacuated from the bentonite in the mould. The evacuation was retained through the whole compaction sequence. The bentonite was then compacted with the press as much as possible. Then the second piston was placed on top of the first piston and the compaction continued. The same procedure was repeated for the third piston. The total time for the compaction was about 10 minutes. The maximum load was then left on the piston for another 10 minutes (hold time 10 minutes).
- The reloading of the block took about 10 minutes. The mould with bentonite and the pistons were then lifted with jacks. Steel plates were placed between the mould and the bottom plate and the block was pushed out of the mould with the press.
- The mould and the block were then removed and the ring and pistons lifted off the block. The block was placed on a specially designed pallet with the lifting equipment.

**Table 4-1. Program for compacting blocks for the KBS3-H project.**

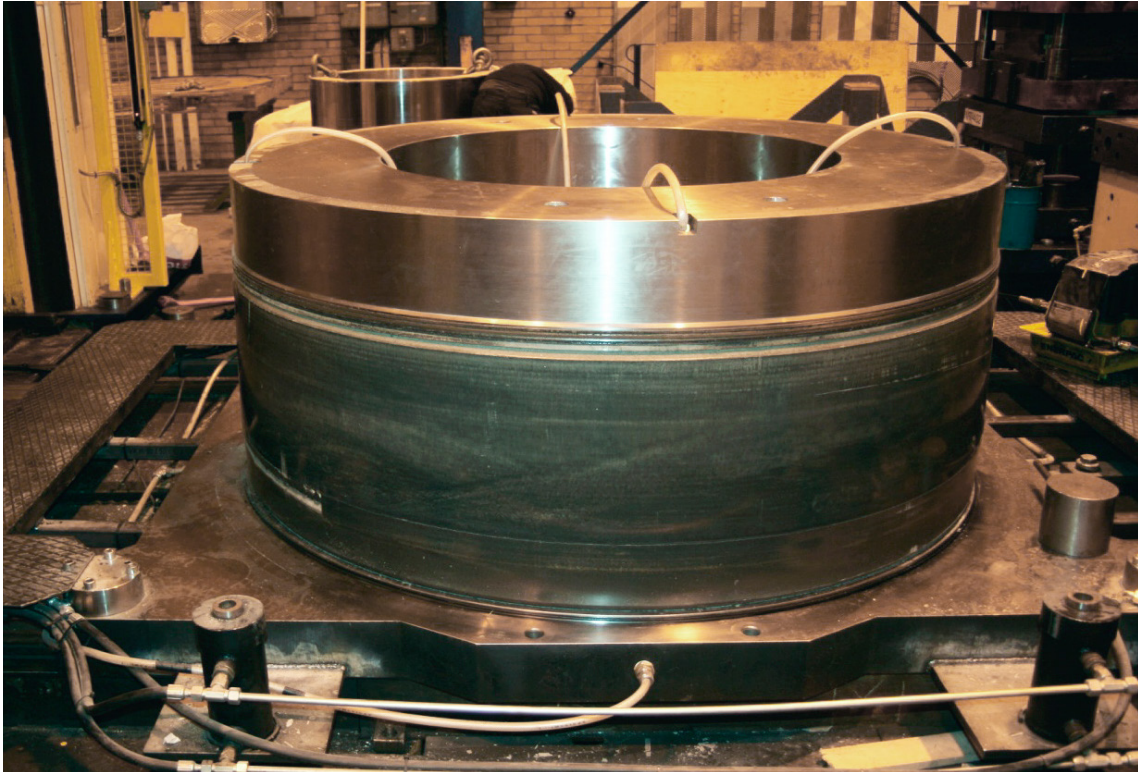
Shape	Number of blocks	Comp. pressure (MPa)	Comp. load (MN)	Water cont. (%)	Expected bulk density (kg/m <sup>3</sup> )
Ring	12	63	103	12	2,110
Cylindr. I	4	37	93	17	2,050
Cylindr. II	27	36	90	21	2,070



*Figure 4-1. The bentonite filled in the mould.*



*Figure 4-2. The mould placed in the press.*



*Figure 4-3. The bentonite block placed on the bottom plate with the piston on top.*



*Figure 4-4. The bentonite block lifted with the vacuum oak and placed on the special designed pallet.*

## 5 Investigation of the compacted blocks

The weight of bentonite used for the blocks, the time for compaction, the maximum compaction force, the time at the maximum load (hold time) and the time for reloading were noted in a protocol.

After compaction, the dimensions (height and diameters) of each block were measured. The height of the blocks was measured at 8 locations around the block. The used mould had a conical shape i.e. the upper outer diameter of the blocks was about 1,775 mm and the lower outer diameter was about 1,790 mm. The inner diameter of the ring shaped blocks was about 1,055 mm. Each diameter was measured in two perpendicular directions. All the dimensions of the blocks were determined with callipers. The weight of the blocks was also determined with an accuracy of  $\pm 2$  kg using a weighing machine hanging from an overhead crane. All these data were filled in the protocol. The blocks were also examined by eye. Any observed damages were noted in the protocol.

The water content of the sample taken from each block was determined by drying a sample at a temperature of 105°C for 24 hours as described in Section 2.1. Knowing density and water content it is possible to calculate the degree of saturation, void ratio and density at saturation, see Section 2.1. All these data are summarized in Appendix 1. In Table 5-1 below the average parameters from the three types of compacted blocks are listed.

**Table 5-1. Average parameters determined on the blocks for the KBS-3H project.**

Block type	Water cont	Bulk density (kg/m <sup>3</sup> )	Degree of saturation	Void ratio	Dry density (kg/m <sup>3</sup> )	Weight (kg)	Height (mm)
Ring	0.110	2,106	0.656	0.465	1,898	1,752	509.0
Cylindr. I (inside Supercont)	0.172	2,069	0.832	0.575	1,766	2,584	499.2
Cylindr. II (distance block)	0.206	2,071	0.924	0.619	1,718	2,606	504.2



## 6 Machining of the blocks

After the compaction all of the blocks were machined in order to get the exact dimensions shown in Figure 1-4 and Table 1-2. Since a lubricant was used at the compaction it is also important to remove this from surfaces of the compacted blocks before they are used in tests.

The blocks were transported in covered trucks to Sorvikivi in Finland where the machining was made. The machining was made in a milling machine, see Figure 6-1. All surfaces except for the bottom of the blocks were machined.

After the machining the weight of the blocks was determined. The data of the blocks after machining are shown in Appendix 2. The result from the weighting of 10 out of total 12 compacted ring shaped blocks gave an average dry density of 1,900 kg/m<sup>3</sup> with a standard deviation of 10.9 kg/m<sup>3</sup>, see Table 6-1. Corresponding values for the distance blocks were 1,728 and 9.2 kg/m<sup>3</sup> respectively. This data should be compared with the reference design of the blocks, see Table 1-2. The comparison shows that the 95% confidence intervals for both types of blocks are larger than the accepted variation of the dry density.

**Table 6-1. Average parameters determined on the machined blocks for the KBS-3H project.**

Block type	Number of blocks	Average dry density (kg/m <sup>3</sup> )	STDEV dry density (kg/m <sup>3</sup> )	95% confidence interval dry density (kg/m <sup>3</sup> )
Ring shaped	10	1,900	10.9	1,879–1,921
Distance block	22	1,728	9.2	1,710–1,746



*Figure 6-1. The bentonite block machined with a milling machine.*

## 7 Conclusions

This report describes the production of the blocks for the Multi Purpose Test. The following conclusions can be drawn from the control of the bentonite and the quality of the produced blocks:

- The determined average montmorillonite content is within the acceptance criteria for a buffer material.
- The chemical compositions of the different deliveries of MX-80 are similar.
- The 95% confidence intervals of the water content after mixing are within the accepted variations, except for the material with the expected water content of 21% where the lower limit was determinate to 19.7%.
- The 95% confidence intervals of the dry densities of the produced blocks (ring shaped blocks and distance blocks) after machining are not within the accepted variations. However, the calculated average densities of the produced blocks are within the acceptable variation.

All the produced blocks were not within the accepted variation concerning the water content and dry densities. However, the judgment is that the blocks could be used for the Multi Purpose Test without affecting the expected outcome from the test in an essential way.

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## Data from the compaction of blocks

Block No	Date of comp.	w (%)	Bulk density (kg/m <sup>3</sup> )	Degree of sat.	Void ratio	Dry density (kg/m <sup>3</sup> )	Weight (kg)	Height (mm)
KBS312C1	2012-12-03	20.9	2,072	0.934	0.622	1,713	2,598	502.5
KBS312C2	2012-12-04	21.0	2,068	0.932	0.627	1,709	2,568	498.2
KBS312C3	2012-12-04	21.2	2,082	0.953	0.619	1,718	2,606	502.1
KBS312C4	2012-12-04	20.7	2,084	0.944	0.611	1,726	2,608	501.8
KBS312C5	2012-12-04	20.8	2,092	0.956	0.606	1,731	2,600	500.5
KBS312C6	2012-12-04	20.8	2,082	0.944	0.613	1,724	2,604	502.8
KBS312C7	2012-12-05	21.2	2,082	0.953	0.618	1,718	2,588	500.3
KBS312C8	2012-12-05	20.8	2,082	0.945	0.613	1,723	2,594	499.4
KBS313C1	2013-02-19	17.8	2,068	0.846	0.583	1,756	2,570	496.9
KBS313C2	2013-02-19	17.3	2,070	0.835	0.576	1,764	2,588	500.0
KBS313C3	2013-02-20	16.6	2,071	0.816	0.565	1,777	2,594	500.8
KBS313C4	2013-02-20	17.1	2,068	0.829	0.575	1,765	2,584	499.2
KBS313C5	2013-02-18	20.7	2,077	0.935	0.616	1,720	2,596	501.6
KBS313C6	2013-02-18	20.6	2,079	0.935	0.612	1,724	2,594	500.5
KBS313C7	2013-02-19	20.9	2,073	0.935	0.621	1,715	2,600	502.4
KBS313C8	2013-02-19	21.1	2,059	0.925	0.636	1,700	2,592	504.0
KBS313C9	2013-02-19	21.4	2,070	0.943	0.630	1,705	2,588	501.3
KBS313C10	2013-02-19	20.7	2,078	0.935	0.615	1,721	2,602	501.8
KBS313R1	2013-02-22	11.0	2,086	0.640	0.480	1,879	1,732	508.7
KBS313R2	2013-02-20	10.9	2,110	0.657	0.461	1,903	1,732	502.6
KBS313R3	2013-02-20	10.8	2,107	0.652	0.462	1,901	1,732	502.5
KBS313R4	2013-02-21	10.8	2,100	0.644	0.467	1,895	1,722	503.5
KBS313R5	2013-02-21	11.1	2,109	0.665	0.465	1,898	1,744	506.6
KBS313R6	2013-02-21	11.2	2,118	0.677	0.460	1,904	1,758	508.7
KBS313R7	2013-02-21	11.1	2,104	0.658	0.468	1,894	1,750	508.2
KBS313R8	2013-02-22	10.9	2,099	0.646	0.469	1,893	1,840	535.4
KBS313R9	2013-02-22	11.0	2,107	0.657	0.465	1,898	1,748	508.0
KBS313R10	2013-02-22	10.7	2,115	0.653	0.455	1,910	1,756	507.6
KBS313R11	2013-02-22	11.0	2,106	0.656	0.465	1,897	1,752	508.3
KBS313R12	2013-02-22	11.0	2,111	0.663	0.462	1,902	1,752	507.6
KBS313C11	2013-07-29	20.6	2,075	0.929	0.615	1,721	2,610	503.5
KBS313C12	2013-07-29	20.6	2,079	0.935	0.613	1,724	2,600	501.3
KBS313C13	2013-07-29	20.8	2,080	0.941	0.615	1,721	2,608	502.4
KBS313C14	2013-07-29	19.5	2,084	0.913	0.594	1,744	2,606	501.2
KBS313C15	2013-07-30	19.9	2,083	0.921	0.600	1,738	2,606	501.1
KBS313C16	2013-07-30	20.7	2,083	0.942	0.611	1,725	2,612	502.3
KBS313C17	2013-07-30	19.3	2,073	0.894	0.599	1,738	2,658	513.0
KBS313C18	2013-07-30	20.2	2,072	0.916	0.612	1,724	2,660	514.7
KBS313C19	2013-07-30	20.2	2,072	0.916	0.612	1,724	2,660	514.7
KBS313C20	2013-07-30	20.9	2,074	0.936	0.621	1,715	2,648	511.9
KBS313C21	2013-07-30	20.4	2,065	0.914	0.621	1,715	2,612	506.6
KBS313C22	2013-07-30	21.0	2,053	0.916	0.639	1,696	2,590	505.8
KBS313C23	2013-07-31	20.4	2,058	0.906	0.627	1,709	2,604	507.1
KBS313C24	2013-07-31	20.5	2,051	0.900	0.634	1,702	2,604	508.9
KBS313C25	2013-07-31	19.8	2,064	0.896	0.613	1,723	2,606	506.0
KBS313C26	2013-07-31	20.4	2,061	0.909	0.624	1,711	2,604	506.0
KBS313C27	2013-07-31	20.4	2,058	0.904	0.626	1,710	2,608	507.6
KBS313C28	2013-07-31	19.9	2,064	0.899	0.615	1,721	2,610	506.4
KBS313C29	2013-07-31	21.1	2,057	0.921	0.636	1,699	2,594	505.0
KBS313C30	2013-08-01	20.1	2,058	0.896	0.622	1,714	2,594	504.9
KBS313C31	2013-08-01	19.9	2,032	0.864	0.640	1,695	2,572	506.9

## Data from the machining of the blocks

Block No	Weight (kg)	Height (mm)	Outer diameter (mm)	Inner diameter (mm)	Bulk density (kg/m <sup>3</sup> )	Water content	Dry density (kg/m <sup>3</sup> )
KBS313C6	2,456	485.7	1,765.0		2,067	0.206	1,711
KBS313C7	2,479	486.1	1,765.3		2,084	0.209	1,720
KBS313C8	2,472	486.1	1,765.4		2,078	0.211	1,715
KBS313C9	2,482	486.2	1,765.7		2,085	0.214	1,718
KBS313C10	2,476	486.0	1,765.4		2,082	0.207	1,725
KBS313R1	1,526	486.3	1,740.0	1,058.0	2,094	0.110	1,886
KBS313R2	1,542	485.4	1,740.4	1,058.0	2,119	0.109	1,910
KBS313R3	1,540	484.5	1,740.1	1,058.0	2,120	0.108	1,913
KBS313R4	1,547	485.6	1,740.5	1,058.0	2,123	0.108	1,916
KBS313R5	1,535	485.3	1,743.3	1,058.0	2,098	0.111	1,888
KBS313R6	1,533	485.0	1,739.5	1,058.0	2,111	0.112	1,898
KBS313R7	1,528	485.2	1,740.2	1,058.0	2,101	0.111	1,891
KBS313R9	1,540	485.4	1,743.5	1,058.0	2,104	0.110	1,895
KBS313R11	1,542	485.4	1,740.9	1,058.0	2,116	0.110	1,907
KBS313R12	1,217	386.0	1,740.0	1,058.0	2,104	0.110	1,895
KBS313C12	2,469	485.3	1,765.0		2,080	0.206	1,724
KBS313C13	2,476	485.0	1,765.0		2,086	0.208	1,727
KBS313C14	2,473	485.0	1,764.9		2,084	0.195	1,744
KBS313C15	2,471	485.5	1,764.9		2,081	0.199	1,736
KBS313C16	2,476	485.0	1,764.5		2,088	0.207	1,730
KBS313C17	2,472	485.3	1,764.6		2,083	0.193	1,746
KBS313C18	2,476	485.6	1,764.0		2,086	0.202	1,736
KBS313C19	2,473	486.5	1,765.1		2,077	0.202	1,729
KBS313C20	2,472	485.2	1,764.8		2,083	0.209	1,723
KBS313C21	2,450	485.4	1,764.5		2,064	0.204	1,714
KBS313C23	2,471	484.5	1,764.2		2,086	0.204	1,733
KBS313C24	2,469	485.0	1,764.3		2,083	0.205	1,728
KBS313C25	2,462	486.4	1,764.6		2,070	0.198	1,728
KBS313C26	2,470	486.5	1,765.1		2,075	0.204	1,723
KBS313C27	2,473	485.0	1,764.7		2,085	0.204	1,732
KBS313C28	2,471	485.0	1,765.0		2,082	0.199	1,737
KBS313C30	2,469	485.5	1,764.5		2,080	0.201	1,732