Technical Report

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Isostatic compaction of beaker shaped bentonite blocks on the scale 1:4

Lars-Erik Johannesson
Clay Technology AB

Sven Nord
Ifö Ceramics AB

Roland Pusch
Geodevelopment AB

Rolf Sjöblom
ÅF-Energikonsult AB

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Svensk Kärnbränslehantering AB
Swedish Nuclear Fuel
and Waste Management Co
Box 5864
SE-102 40 Stockholm Sweden
Tel 08-459 84 00
+46 8 459 84 00
Fax 08-661 57 19
+46 8 661 57 19

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Executive Summary

The purpose of the present work is to test, on a scale of 1:4, the feasibility of manufacturing bentonite blocks by isostatic compaction for application as a buffer material in a repository for spent nuclear fuel. In order for the tests to be sensitive to any weaknesses of the method, the blocks were shaped as beakers.

The scope included the following:

1. **Preparation of powder**
    a. mixing of the bentonite and addition of water in predetermined amounts,
    b. sieving to remove any lumps generated.
2. **Isostatic compaction**
    a. establishment of a separate laboratory for the handling of bentonite powder (weighing, mixing, filling, sampling and machining),
    b. development and design of equipment and procedures for compaction of bentonite to beaker-shaped specimens,
    c. compaction process operation,
    d. visual inspection.
3. **Sampling and characterisation**
    a. extraction of samples from the blocks made,
    b. determination of water content,
    c. determination of density,
    d. determination of strain at maximum stress by means of bending tests,
    e. determination of tensile strength by means of bending tests,
    f. determination of geometries of the blocks prepared.
4. **Post-treatment by means of machining**
    a. machining of blocks made,
    b. visual inspection.
5. **Evaluation**

The work went very smoothly. No significant obstacles or unexpected events were encountered.

The conclusions are as follows:

- The conclusions drawn in this report from work on the (linear) scale of one to four are very relevant to the full scale.
- Mixing of bentonite powder as well as moistening can be carried out on a pilot scale with a good homogeneity and with maintained good quality of the press powder.
- The compaction of bentonite can be carried out in a similar manner to the present operation at Ifö Ceramics AB.

- This implies a very efficient handling as well as a very efficient use of the time in the press which may account for a large proportion of the total cost.
- The blocks could readily be produced to reproducible dimensions and relatively even surfaces.
- The blocks could readily be produced to desirable void ratio and density.
- The bentonite blocks produced had a high homogeneity.
- No fractures could be observed visually. Microcracks could be identified on strong magnification, as might be expected.
- The mechanical properties recorded were good and the spread in the values low for all blocks.
- No special difficulty was encountered which was related to the complex shape of the blocks (beaker-like). The complex shape influenced the design of the bag and filling system, as well as the procedure for filling.
- The method of machining presently used at Ifö Ceramics AB for their ordinary production works excellently for compacted bentonite as well.
- A coarse estimate indicates that the machining of the blocks for one entire deposition hole may take on the order of a small number of hours.
- The production went very well for all the combinations of production parameters used. Variation of production parameters might be utilised to adjust some of the properties of the blocks.
- It should be noted that in the processes, no substances are introduced into the bentonite other than pure water.
- No limitations have been identified regarding the proportions of the blocks manufactured (e.g. diameter/height ratio).
- Although very positive conclusions are drawn on essentially all points above, further development work is necessary before a process for full scale operation can be established.

Sammanfattning

Syftet med det arbete som redovisas i denna rapport är att testa, i skala 1:4, möjligheterna att tillämpa isostatisk kompaktering för tillverkning av bentonitblock för användning som buffert i ett slutförvar för använt kärnbränsle. För att testerna skulle bli så känsliga som möjligt med avseende på eventuella svagheter hos metoden tillverkades de i formen av en bägare.

Uppdraget innefattade följande delar:

1. **Pulverberedning**
   a. blandning av bentonit samt tillsats av förutbestämda mängder vatten,
   b. siktning samt avskiljning av eventuella klumpar.
2. **Isostatisk kompaktering**
   a. etablerande av ett laboratorium för hantering av bentonitpulver (vägning, blandning, fyllning av form, provtagning och avverkning),
   b. konstruktion och tillverkning av utrustning samt utveckling av förfarande för kompaktering av bentonit till bägarformiga kroppar,
   c. utförande av kompakteringsprocessen,
   d. okulär undersökning.
3. **Provtagning och karakterisering**
   a. uttag av provkroppar ur blocken,
   b. bestämning av vatteninnehåll,
   c. bestämning av densitet,
   d. bestämning av graden av deformation vid maximal påkänning i samband med böjprovning,
   e. bestämning av draghållfastheten i samband med böjprovning,
   f. bestämning av blockens geometri.
4. **Efterbehandling i form av avverkning**
   a. avverkning (svarvning),
   b. okulär undersökning.
5. **Utvärdering**

Utförandet av arbetet fortgick utan särskilda svårigheter. Inga hinder eller oväntade problem uppkom.

Slutsatserna är som följer:

- Slutsatserna från föreliggande arbete i skala ett till fyra (linjärt) är väl tillämpbara även i full skala.
- Blandning av bentonitpulver samt befuktning kan utföras i pilotskala med erhållande av en god homogenitet samt en bibehållen god kvalitet hos pressmassan.

- Kompaktering av bentonit kan utföras på liknande sätt som vid den löpande driften vid Ifö Ceramics AB.

- Detta innebär att hantering av bentonit samt utnyttjande av tiden i pressen kan ske på ett mycket effektivt sätt. Tiden i pressen förväntas utgöra en stor andel av den totala kostnaden för blocken.

- Blocken kunde utan särskilda svårigheter tillverkas till reproducerbara dimensioner samt relativt jämna ytor.

- Blocken kunde utan någon särskild svårighet tillverkas till önskade portal och densiteter.

- Blocken som tillverkades hade en hög homogenitet.

- Inga sprickor kunde observeras med blotta ögat. Mikrosprickor kunde (som väntat) observeras under stark förstoring.

- De mekaniska egenskaper, som registrerades, var goda och spridningen i värdena var låg för samtliga block.

- Inga särskilda svårigheter uppkom vilka kunde relateras till blockens komplicerade form (bägarform). Den komplexa formen påverkade utformningen av gummibagen samt system och procedur för påfyllning av pulver.

- Den metod för avverkning som för närvarande tillämpas på Ifö Ceramics AB i samband med den ordinarie produktionen fungerar utmärkt även för block av kompakterad bentonit.

- En grov uppskattning indikerar att avverkning av block för en deponeringsposition kan ta ett fåtal timmar.

- Produktionen förlöpte mycket väl för samtliga kombinationer av produktionsparametrar som använts. Variationer av produktionsparametrarna kan tänkas utnyttjas för att justera vissa av blockens egenskaper.

- Det bör noteras att inga ämnen utom rent vatten tillförs bentoniten under hanteringen.

- Inga begränsningar har identifierats beträffande blockens proportioner (t ex höjd/diameter förhållande).

- Trots att mycket positiva slutsatser dras beträffande i stort sett samtliga punkter ovan erfordras ändå ytterligare utvecklingsarbete innan en process för drift i full skala kan etableras.

Content

1 Introduction and background

The work described in the present report has been carried out on behalf of the Swedish Nuclear Fuel and Waste Management Company (SKB) as a part of their programme /1, 2/ for the management of nuclear waste in Sweden. According to the reference method KBS-3, the spent nuclear fuel will be enclosed in canisters of steel and copper and deposited in special drill holes at a depth of about 500 meters in crystalline rock. Between the canisters and the rock, there will be a bentonite buffer with an outer diameter of about 1.75 meters and a thickness of about 0.35 meters.

In order for appropriate materials properties /1, 2/ to develop on water saturation in the deposition hole, the dry density of the emplaced bentonite needs to be sufficiently high. Early in the programme of SKB, blocks with the desired density were prepared by isostatic compaction /3/. During the decade 1990–1999, the main efforts have concerned uniaxial compaction, but the work performed has included also isostatic compaction techniques.

Isostatically compacted blocks with a diameter of 700 millimeters were prepared /3/ already in 1982 as a part of the *Buffer Mass Test* at Stripa. This value actually refers to “halves” of cylindrical blocks cut in a plane through the main symmetry axis. The diameter of the cylindrical specimens exiting the press was thus considerably less than 700 millimeters. The purpose of the test was not primarily to develop a method for compaction or manufacturing of large blocks, but to fabricate blocks for the actual tests.

The feasibility of various aspects of isostatic compaction may at present be assessed as follows.

- Test methods are available for most of the needs. Testing on a small scale can conveniently be carried out using uniaxial pressing. The relevance of small scale testing for large scale phenomena need to be assessed, however.
- Methods are available for powder preparation including:
  - crushing, sieving and air classification,
  - intensive mixer granulation (Eirich).
- The Eirich granulated powder (intensive mixer) has larger and nearly spherical granules while the crushed powder has a more irregular shape and contains more fines. The former fills the die more evenly while the latter gives a higher mechanical strength in the compacted material.
- The mecanical strength of the blocks can be substantially increased by increasing the pressure as well as the hold time at maximum pressure. A typical pressure during the compactions is 100 MPa. Only for some parameter combinations.
- Compacted bentonite with dry densities in the range desired can be prepared using powders with water ratios in the range 10–18%.
- The dry density of compacted material decreases with an increase in water ratio (at 100 MPa).
- Machining may be required. Such machining may be carried out under dry conditions using standard tools for hard materials.
- Moistening of bentonite prior to compaction may be carried out using a Forberg type of mixer. The parameters can be adjusted so that an essentially lump-free product is generated.

One of the main merits of bentonite in conjunction with nuclear waste disposal is its ability to swell and to fill the space available. When the bentonite blocks are to be prepared and handled, this property needs attention, since the uptake or loss of moisture is associated with changes in volume and the formation of cracks. When moisture is lost, cracks form on the surface, and when moisture is absorbed, they can be assessed to form on the inside. These effects may appear in synergy with strains and fractures formed during the compaction as well as with inhomogeneities of various kinds in the powder before compaction. It is generally believed that the potential for compaction induced irregularities is substantially lower for isostatic compaction than for uniaxial.

Detection of fractures may significantly improve the progonsis for a non-problematic handling of a block. Thus, various techniques may be applied to this end, including visual inspection using an ocular, indication liquid and impact echo techniques. The ocular method was applied on the blocks prepared as a part of the present work, see section 7.2.

It has recently been reported /4/ that blocks have been prepared in the scale 1:5 by a semi-isostatic technique. These findings are in agreement with those in the present report.

2 Purpose and scope

2.1 The rationale for the present approach

It was concluded in section 1, that previously obtained results show that the isostatic technique appeared to be a very feasible one for the manufacturing of full-size blocks.

The uniaxial technique has, however, been selected for the manufacturing of the blocks for the Äspö Hard Rock Laboratory full scale tests primarily due to the lower cost of the tool for the pressing (a die in the case of uniaxial compaction) together with the precise dimensions obtained. However, the isostatic technique has potential advantages in terms of higher homogeneity and associated reduced potential for fracturing as well as absence of additives. In the case of uniaxial compaction, a lubricant has to be used in order to reduce the friction between the bentonite and the die. Moreover, for new production facilities, the manufacturing cost for blocks is significantly less for the isostatic technique.

Thus, the selection of the uniaxial technique for the manufacturing of the full-scale blocks needed for the experiments in the Äspö Hard Rock Laboratory has no implications for the selection of the technique to be used for the manufacturing of the blocks for the repository for the spent nuclear fuel.

Moreover, both techniques should be regarded as candidates for the reference technique as well as for the reserve alternative. Thus, the continued development work includes both techniques.

The previous results summarised briefly in section 1 above indicate that continued development work on isostatic compaction should include further tests on the laboratory scale as well as on a pilot scale. The present paper deals exclusively with the latter.

The techniques and equipment at Ifö Ceramics AB offer a great flexibility as regards the size and shape of the specimens to be prepared. The maximum dimensions possible for a bentonite block are limited geometrically by a cylinder with a diameter of about one meter and a height of about three meters. These limits were derived from a volume reduction factor for bentonite of about 2.0 together with the dimensions of the press at Ifö Ceramics AB. See section 5.2. The diameter is about 1.3 meters and the height is about 4 meters. Some of the diameter is "lost" due to the exchange of pressure medium (from oil to water) through a rubber membrane along the cylinder surface.

Thus, full size diameter cannot be achieved directly.

In the Stripa experiments, blocks were manufactured in the following way /3/. Solid cylinders were manufactured by isostatic compaction. These were subsequently sawed to generate specimens with a plane parallel cross section the normal of which was orthogonal to the previous rotational symmetry axis. Semi-cylindrical mantle surfaces were then sawed with their radii of curvature perpendicular to the just mentioned normal. In this way, two blocks made up a cylindrical volume or a volume corresponding to that of a short piece of pipe.

This method was used in order to obtain a buffer for a long-term test. It was not assessed to be a feasible method for the manufacturing of blocks on any larger scale. Since the blocks for the Äspö experiments were to be produced by uniaxial technique, little or no reason remained for pursuing this alternative. It will therefore not be discussed further in the present report.

Three alternatives were considered for the pilot scale tests:

1. Blocks with a shape similar to a slice taken from a cylindrical cake (in the following referred to as “cake slices”).
2. Cylindrical blocks together with blocks shaped as pipes or rings (in the following referred to as “pipes”).
3. Blocks shaped as a pipe with one end open and the other end closed (in the following referred to as “beakers”).

Alternative 1 above, in which the blocks are produced in a cake slice shape, offers the possibility of preparing blocks which could be put together to make up a full size buffer. (For instance, by six blocks of the same size and shape constituting each plane). Such blocks could be manufactured in the present facilities at Ifö Ceramics AB. It was assessed, however, that the shape of a cake slice (especially the corners) introduced unwanted and unnecessary complexities and unnecessary potential difficulties. Moreover, the primary objective was not to necessarily come up with fullsize specimens, but to test the isostatic technique on a sufficiently large scale in order for the results to be expected to be relevant for the full scale.

Alternative 1 was thus discarded on these grounds.

Alternative 2 might then appear to be the obvious choice since the specimens generated would closely resemble those considered for the main alternatives in the KBS-3 family /5/.

On closer examination and consideration, it was realised, however, that the new information expected to be gained from such tests would be limited. Isostatically compacted blocks with a high quality had been generated already in the Stripa tests/3/. This conclusion has been further verified in the introductory stages of the present work. Thus, moderate difficulty was expected in obtaining blocks with cylindrical or pipe-like shapes.

It was also realised that if blocks with a beaker-like shape could be prepared with good results, then blocks with the simpler cylindrical or pipe-like shapes could most certainly be prepared as well with even better results.

Blocks with beaker-like shapes are of interest in conjunction with certain other alternatives within the KBS-3 family. Knowledge of the prerequisites for preparing beaker-shaped blocks is thus of interest for SKB as a basis for repository system development and alternative selection activities.

Thus, alternative 2 above – “cylinders” and “pipes” – was also discarded and alternative 3 – “beakers” – was selected for the pilot scale tests.

The above mentioned constraints on dimension in the press allows a maximum scale of 1:2 to be used in the pilot tests. However, it was considered desirable that a number of tests should be conducted with different parameter combinations. Moreover, the experience at Ifö Ceramics AB clearly indicates that isostatic compaction is grossly independent of scale. It was therefore decided to use the scale of 1:4 in order for the scale to be sufficiently large to be relevant to the questions asked, but at the same time sufficiently small in order for the efforts and costs to be reasonable.

It was also decided to make the walls of the beakers – before machining – considerably thicker than that which corresponds to a straight scaling. This was a precautionary measure intended to enable appropriate dimensions to be obtained through machining even if the shapes of the beakers would for some reason become very distorted during compaction.

One of the issues of interest in conjunction with isostatic compaction is the homogeneity of the powder in the bag and amongst other items its apparent density. If the apparent density varies throughout the specimen-to-be, the shrinkage during compaction will be uneven and the shape of the body distorted – e g "banana-like" shape if one side of a high specimen has an apparent density which is different from the other. The even filling of the bag is more difficult to achieve for a beaker-like shape than for a cylindrical one. Thus, with the selection of alternative 3 – "beakers" – above, this issue must be dealt with in the present work.

2.2 Purpose

The purpose of the present work is to investigate the following issues:

1. **Addition of moisture and separation of lumps in the press powder**
2. **Isostatic compaction process**
3. **Process parameters: moisture content and press cycle**
4. **The properties of the blocks**
    a. visual inspection,
    b. moisture content, density, etc,
    c. mechanical properties,
    d. dimensions.
5. **Post-treatment by machining to precise dimensions**

The purpose is also to assess the feasibility of the isostatic compaction technique for the manufacturing of bentonite blocks for use as a buffer in a repository for spent nuclear fuel. The assessment should be based on the present as well as on previously obtained results.

Addition of moisture and separation of lumps (point 1 above) should be carried out in such a way that the powder becomes homogeneous. Inhomogeneities in the moisture content might lead to differential compaction with the possible consequences of fracturing together with residual stresses. Inhomogeneities in the moisture content might also lead to migration of moisture after compaction and the development of the associated stresses. The presence of lumps – such lumps usually have a higher moisture content than the rest of the powder – might give rise to defects, some of which may constitute starting points for fractures.

Thus, the investigation will also deal to some extent with the homogeneity of moisture content, the presence and separation of lumps together with any associated possible influences on the properties of the blocks.

The isostatic compaction technology at Ifö Ceramics AB has changed and improved considerably throughout the years. Also, the records from the previous tests carried out more than 15 years ago /3/ may not be sufficiently detailed for the present purposes. Furthermore, the generation of a specimen in the shape of a beaker requires a somewhat different technique in comparison with the present factory practice.

Thus, the work is aimed at resulting in the establishment of a process for generation of beaker-shaped blocks on the scale of 1 to 4. The knowledge base for the process should include some variations in the following parameters: moisture content, maximum pressure and hold time at maximum pressure.

The present work is also aimed at resulting in the presentation of some of the most relevant characteristics of the blocks generated, namely the presence of defects, geometry, homogeneity and mechanical properties.

Since isostatic compaction generates blocks with less precise geometry compared with uniaxial compaction, it is also the purpose to find out how the predetermination and precision might be improved by means of machining.

2.3 Scope

The scope includes the following four separate activities:

1. **Preparation of powder**
    a. mixing of the bentonite and addition of water in predetermined amounts,
    b. sieving to remove any lumps generated.
2. **Isostatic compaction**
    a. establishment of a separate laboratory for the handling of bentonite powder (weighing, mixing, filling, sampling and machining),
    b. development and design of equipment and procedures for the compaction of bentonite to beaker-shaped specimens,
    c. compaction process operation,
    d. visual inspection.
3. **Sampling and characterisation**
    a. extraction of samples from the blocks made,
    b. determination of water content,
    c. determination of density,
    d. determination of strain at maximum stress by means of bending tests,
    e. determination of tensile strength by means of bending tests,
    f. determination of geometries of the blocks prepared.
4. **Post-treatment by means of machining**
    a. machining of blocks made,
    b. visual inspection.

The work has been carried out by the four organisations directly involved in the present project: Clay Technology AB (Clay), Geodevelopment AB (Geo), Ifö Ceramics AB (Ifö) and ÅF-Energikonsult AB (ÅF-Ene) jointly and in collaboration. Nonetheless, the main parts of items 1 and 3 above have been carried out by Clay, item 4b by Geo, items 2 and 4a primarily by Ifö and the reporting largely by ÅF-Ene.

2.4 The arrangement of the material in the present report

The structuring of the items in the previous section, 2.3 scope, deviates from that of section "2.2 purpose" for practical reasons. For instance, the homogeneity of the bentonite in a block is achieved during the mixing and maintained during the filling of the bag while the actual measurement of moisture which is used to determine the homogeneity is carried out on the samples taken from the compacted blocks.

The structuring of the bulk of the report follows the structure of the work performed. Thus, the establishment of a laboratory for powder handling is dealt with in section 3, powder preparation in section 4 and isostatic compaction in section 5. The dimensions of the blocks, the experiments on machining as well as an analysis of the need for machining are presented in section 6. Visual observations are dealt with in section 7. Sampling and characterisation relevant to the work presented in sections 4 and 5 are presented in section 8.

The discussion and conclusions in section 9 relate to the issues presented in section 2.2 "purpose" above.

3 Establishment of a laboratory for powder handling

Ifö has large facilities at its disposal at its plant at Bromölla in south Sweden, including a large press for cold isostatic compaction. The press has a cylindrical volume available for compaction with the following dimensions: diameter 1.3 meters and height about 4 meters. The maximum pressure is 115 MPa.

The press is of the Quintus type which means that tensile forces are assumed by steel wire wound around the press cylinder as well as around the frame of the yoke for the end plugs of the press cylinder. In this way, any fracturing due to tensile forces will have a small effect since the friction between the wires is large so that the breakage of one wire does not affect the others to any significant extent.

The facilities also include amongst others laboratories and equipment for machining.

The handling in question could, in principle, readily be carried out in many parts of these facilities. However, contamination by bentonite must be avoided since even low levels of impurities from this substance may have a detrimental effect on the quality of the products made. Therefore, it was decided that all open handling of bentonite should be carried out in a special secluded area only.

Thus, a large room of about 250 m^2 was allocated for the purpose and services were installed, as needed. When ready, the room contained the following:

- A flat concrete floor with a high bearing strength so that transports could be made using a hand-propelled fork-lift.
- Three-phase electricity, 380 Volts.
- A hoist block with a sufficient lifting capacity.
- A vertical turning lathe.
- A large vacuum cleaner with a high capacity.
- Tap water.
- A high quality scale with a capacity on the order of one metric tonne.
- Containers for interim storage of moisture adjusted bentonite.
- Funnels and other equipment needed for very even filling of the press bag.

The room was furnished in such a manner that no open handling of bentonite needed to be carried out outside its premises.

In addition, a Forberg mixer was rented from the manufacturer. It was used for moistening the bentonite to the appropriate moisture content, see futher below.

4 Powder preparation

The bentonite used in all experiments was obtained from Volclay Ltd through their agent in Sweden, Askania AB and the quality used was MX-80.

MX-80 consists of natural sodium bentonite which has been dried and crushed. Most of the finest material from the crushing has been removed by "aspiration". Crushed material has a high internal friction and relatively poor flow properties.

The material was used as delivered, in which case the water ratio was around 10%, as well as with higher contents of water.

The addition of water was carried out using a Forberg type of mixer supplied with a spray nozzle which was used for the addition of water.

The functioning of the mixing and moistening processes was as follows. Two axes with platelets on short arms were installed at the bottom of the mixing container with their axes parallel. When the equipment was in operation these axes were rotated in reverse relative to each other, and in such a manner that the grains in the powder coming down towards the bottom hit the platelets and were thrown up again into new trajectories. In this way, each powder particle spent the very most of its time in the air.

The spray nozzle was positioned in such a manner that the spray formed almost exclusively hit the particles in their trajectories in the air. This arrangement allowed for the microdroplets in the spray to be absorbed into the surface area of the particles before they hit each other or the platelets.

The operation of the equipment was as follows. The mixer was filled with a predetermined (weighed) amount of bentonite powder. The appropriate amount of water was determined based on the desired and existing moisture content of the bentonite to be mixed. This amount was metered by volume and supplied to the inlet side of a pump feeding the spray nozzle. After these preparations, the lid was closed and the mixing started. While the mixing was going on, the pump of the spray system was started and operated until the appropriate amount of water had been added. After this moment, the mixing was continued in order to make the mixing complete.

Initially, a spray nozzle with too high a capacity was used. This led to unwanted clogging and the formation of scale. These problems disappeared almost completely after a spray nozzle with an appropriate size had been installed. As expected, most care was needed when high water ratios were to be achieved.

Thus, the design and operation of the equipment was such that only a small part of the material mixed formed larger aggregates in the form of lumps or scale on the platelets and other surfaces in the mixer.

Although this process gave rise to a moistened powder with only on the order of 2% of lumps by weight, it was felt that their presence might still influence the mechanical properties of the blocks. Since bentonite is a relatively brittle material, it can be expected that the overall mechanical strength is highly dependent on the occurrence of discontinuities (character, orientation and location relative to volumes under tensile stresses).

The bentonite moistened as described above was therefore sieved through a semi-manual sieve so that lumps larger than a few millimeters were removed. This material was used for block number 8.

The various batches of powder prepared are presented in Table 1. The composite batches used for each block are also shown in the table.

Table 1. The various batches of powder prepared together with the composite batches used for each block

Weight of the bentonite kilograms	Water ratio %	Block number which the composite batch was used for
78.1	15.6	5
76.5	15.5	
76.9	15.7	
76.1	17.7	6 and 8
75.9	18.1	
77.5	18.4	
77.2	18.0	
77.2	18.3	
76.4	18.5	
77.4	18.2	
81.5	21.2	7
81.5	21.1	
86.3	21.0	
83.5	15.5	9 and 12
84.0	15.7	
84.1	15.3	
84.0	15.5	
84.6	15.5	
84.3	15.6	
80.1	18.2	10
86.1	18.3	
77.1	18.5	
85.8	21.7	11
84.7	21.3	
86.5	21.2	

5 Isostatic compaction

5.1 Background

For the most part, the isostatic press at Ifö compacts powder to objects with a cylindrical shape or the shape of a tube. Beaker-shaped specimens have not been produced previously in the Swedish programme.

Development and design work was therefore needed before a compaction procedure could be identified and operations take place. Most of the elements of the process could, however, be found in existing procedures and therefore only some supplementary development was needed.

Much of the know-how around isostatic compaction at Ifö is proprietary. However, Ifö regards the development work carried out by SKB positively, and supports their programme by also allowing proprietary system and process components to be used in the development work.

The level of detail in the process descriptions presented in this report is in concordance with these prerequisites.

5.2 The equipment and procedure used

The principle used for the isostatic compaction is briefly as follows. Bentonite is put into a rubber bag. The bag is evacuated in order for trapped air not to expand and possibly damage the specimen during pressure release. The evacuated bag is put into the pressure chamber and subjected to a high hydrostatic pressure after which the pressure is released and the bag removed from the press. Finally, the bag is removed from the compacted bentonite.

A number of provisions need to be made in order for the blocks to obtain the desired shape and to be handled simply and swiftly.

A schematic drawing of the bag system is shown in Figure 1.

For the preparation of beaker-shaped blocks, two bags were used, one on the outside of the "beaker" and the other on the inside. The outside bag is supported by a so-called canister which is a drum made of stainless steel with a large number of holes drilled through the surfaces. This canister should not be confused with the canister (in American English nomenclature container) containing the spent nuclear fuel in a repository. In the present report, both meanings are used in parallel. The inside bag is supported by a cylinder with closed ends. In order for the cylinder to position correctly, it is supplied with two guides which are directed by two pipes attached to the outside of the canister.

The bentonite is added through a special funnel, which provides for an even filling to a homogenous powder material inside the canister. One delicate part of the filling concerned the area below the inside cylinder. In order for this region to be filled in as much the same manner as elsewhere as possible, the canister was tilted in various directions a number of times immediately after the powder level had exceeded the level of the bottom of the inner cylinder.

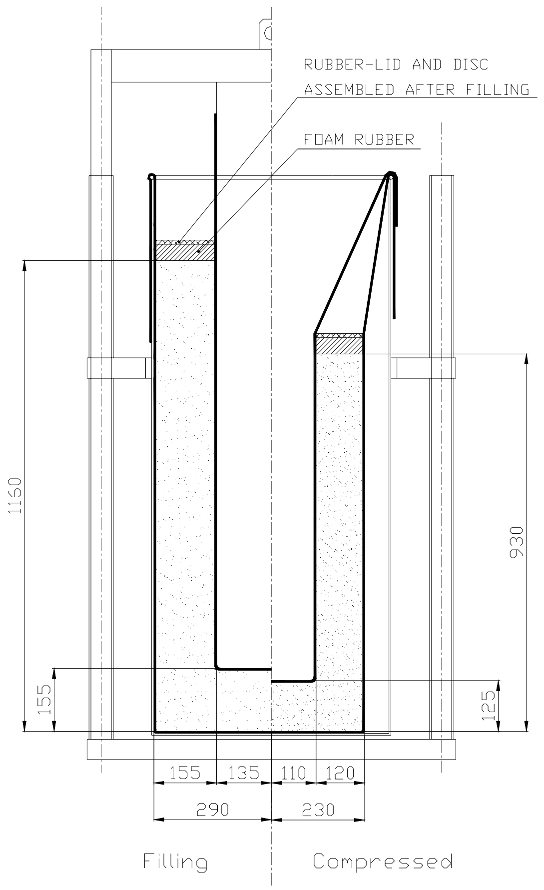

Figure 1. *A schematic drawing of the bag system.*

When the filling was complete, a pineapple-ring-shaped piece of foam rubber was put on top of the bentonite powder and the bags were then sealed using straps. The sealing system was designed in such a manner that the inner cylinder was not involved.

At this stage, the air in the powder was still connected to the air outside through a nipple with an appearance and a function similar to that of the nipple of a tyre.

The canister, thus prepared, was then moved to the area of the main press. Evacuation was achieved by connecting the nipple of the rubber bag to vacuum for five minutes. The vacuum inside the bag improves the friction between the grains in the powder considerably and at this stage, the powder inside the bag can be handled somewhat like a solid body.

After evacuation, the inner cylinder was removed using a hoist. In order for removal to take place swiftly, it was lubricated using a mixture of soap and water. The geometry during the removal is guided by means of the above mentioned guiding devices. The precision of the guiding of the inner cylinder was not high, and this might have influenced the dimensions of the blocks.

The removal of the inner cylinder is crucial for the operation. Isostatic compaction ideally implies that the object becomes smaller but retains its proportions. Thus, the diameter of the hole should decrease during compaction. If the cylinder is left inside the hole during compaction strong forces are likely to arise which can be expected to deform the cylinder unless it is designed to withstand the very high pressure of the press. (Moreover, the compaction would in this case not be isostatic).

The thus prepared canister was then put inside the pressure chamber using a hoist. The lid was swung back and the frame of the yoke was transferred over the presscylinder together with its lid.

When the press cycle had been completed – which took about five minutes – the press was opened, and the canister containing the compacted bentonite was lifted out of the press. Since the specimen is beaker-shaped (and oriented with the bottom down), the inside of the beaker is at this stage filled with the compaction medium used. The medium compacted in the pumps is oil, but this fluid is "converted" to water in the main cylinder by the action of a rubber membrane covering the inside of the press cylinder.

Thus, water needs to be removed before the bentonite block can be uncovered. At the press, water was removed only to the extent needed in order for the specimen to be transferred to the room specially equipped for operations involving the handling of bentonite.

After the transfer, the canister was positioned onto a vertical metal bar acting through a hole in the bottom of the canister onto a plate positioned between the bottom of the canister and the rubber bag. In this way, the compacted bentonite together with the bags were somewhat separated from the canister which came to rest at the base of the bar.

Next, most of the remaining water was removed by scooping. Then, air was allowed to enter the inside while at the same time remaining water was allowed to flush onto the floor. In order for this to occur without running the risk of the bentonite becoming wet it was imperative that the inner bag was lifted and water allowed to drain away before any large openings were made in the seals.

After the removal of all water, the entire seals were opened and the upper part of the beaker-shaped block was uncovered.

At this stage, loops of a rope were applied on opposite sides of the beaker-shaped block after which the block was lifted to allow the rest of the outer bag to be removed.

The block was placed on a plastic sheet on a pallet made of wood after which the plastic sheet was wrapped around the block in order to avoid drying (or uptake of moisture).

The volume reduction of the powder depends on the particular batch, the water ratio and the press cycle. Therefore, only a general figure can be given for overall considerations. Tests carried out in the introductory stages of the work reported here had shown that the volume reduction is not far from 50% which corresponds to a linear reduction of about 20.7%. Thus, the diameter of a compacted specimen was expected to be around 460 millimeters and the wall thickness around 123 millimeters.

It is expected that the surfaces may be somewhat uneven or perhaps also bent reflecting inhomogeneities in the powder and in the filling.

5.3 The compaction operations

The first compacted block was distorted because the rubber plate supporting the above mentioned rubber foam was too stiff. After change to a thinner rubber plate, the problem disappeared and the shape of the block was as expected.

When block number two was to be uncovered from the rubber bag, water accidentally flushed over the bentonite surface and became absorbed to an unknown extent. No measurements were therefore taken on this block.

Otherwise the operations went very smoothly and according to the procedure described in section 5.2.

The parameters used in the various compactions are given in Table 2.

Table 2. The parameters used in the different compactions

Block number	Water ratio % §	Maximum pressure, MPa	Hold time † minutes	Weight of block, kg
1	11.4*	112.5	0	251
2‡	10.0‡*	112.5	10	
3	11.3*	112.5	10	242
4	11.9*	74	0	255
5	14.1	112.5	0	255
6	16.9	112.5	0	255
7	19.9	112.5	0	255
8	17.0	112.5	10	
9	14.4	74	0	
10	17.5	74	0	
11	20.3	74	0	
12	14.4	112.5	10	

§ Measured after compaction (except for block number 2, see ‡).
† At maximum pressure.
* No addition of water was made in this case.
‡ This block was not used in the subsequent analyses for reasons explained in the text; the water ratio given is the intended one.

6 Dimensions of the blocks and machining

6.1 The dimensions of the blocks

Before the tests on machining, the turntable of the lathe was used to determine the dimensions of the blocks. This was carried out as follows.

1. The block to be measured was put on the turntable so that its symmetry axis coincided approximately with the axis of rotation of the turntable.
2. The block was fastened.
3. The distance between a vertically oriented fixed bar and the block was measured at 100 millimeter intervals starting at the upper end of the block.
4. The turntable was rotated 90° whereafter point 3 was repeated.
5. Operation 4 was repeated until measurements had been made for 0, 90, 180 and 270° rotation of the turntable and until measurements at 100 millimeter intervals vertically had been taken for each of these rotations.
6. Procedure 5 was repeated for the inside of the blocks.
7. The height of the blocks were measured at each of the orientations.

The full results of the measurements of the outer and inner surfaces are presented in Tables A-1 and A-2, respectively, in Appendix A. Summary data are presented in Table 3 together with the (radial) linear reduction factor obtained for the compaction. The initial (outer) radius is taken to be 288 millimeters which is half of the diameter as given in Figure 1 minus 2 millimeters which is the approximate thickness of the rubber bag.

Data on the average heights of the blocks are also given in Table 3.

The standard deviations given in Table 3 were calculated using the data for the diameters together with the following formula:

$$\sigma = \sqrt{\frac{n * \sum \mathrm{x}^2 - (\sum \mathrm{x})^2}{n(n-1)}} \tag{1}$$

The standard deviation is close to the root mean square value of the deviances from the average.

The standard deviation of the radius of the outer surface was around 1–3 millimeters (1%), except in one case of high water ratio in combination with a high pressure.

The standard deviation of the radius of the inner surface was around 2–4 millimeters (3–4%) except in two cases where the evacuated powder was disturbed during the removal of the inner cylinder after evacuation but before compaction.

The standard deviations obtained are somewhat optimistic but qualitatively correct. However, they do not adequately indicate the need for machining since the machining tool will have to sweep over any surface where there is something to remove somewhere on it. Therefore, an improved analysis is made in section 6.3.

Table 3. The summary results of the measurements of the blocks, cf text

Block number	Height, average, mm	Inner radius Average mm	Outer radius Standard deviation † mm	%	Average mm	Standard deviation † mm	%	Linear reduction, %, *cf* text
1	877	121	6	5	228	3	1	20.7
3	814	108	4	4	228	2	1	20.9
4	814	112	2	2	235	1	0	18.6
5	847	106	5	4	231	3	1	19.8
6	883	112	2	2	232	2	1	19.4
7	930	101	5	5	224	6	3	22.3
8	936	109	4	3	228	3	1	20.8
9	931	112	2	2	233	2	1	19.2
10	943	110	3	3	229	3	1	20.3
11	956	108	3	3	228	3	1	20.9
12	841	109	3	3	230	3	1	20.0
Average	888	110	3	3	230	3	1	20.3
Std dev	53	5			3			1.0

† The standard deviations are based on half of the diameters.

6.2 The machining operation

The machining was carried out using a turning lathe. The tools used for the lathing were of the same design and quality as those used for the dry machining of the insulator green bodies in regular production. They were made of high alumina (aluminium oxide sintered at a very high temperature) which is a very hard material (related to sapphire).

After some testing, good parameters for the lathing could be identified. They comprise the following:

- a feed of half the width of the tool, that is about 11 millimeters,
- a depth of about 6 millimeters,
- a speed of rotation of about 100 revolutions per minute,
- a surface roughness < 1 millimeter (can be modified/improved).

The above numbers correspond to the following:

- a speed of machining of about 2.4 meters per second,
- a rate of machining of almost 100 m^2 per hour,
- a rate of machining of about 0.6 m^3 per hour.

In order to obtain an even surface, it is adviceable to make the machining in two steps. In such a case, the first step may be carried out with a greater depth if required.

It was observed that considerable amounts of dust were generated in the process. Thus, if the method is to be used in conjunction with serial production, provisions will have to be made in order to remove and separate the dust formed. However, such removal and filtration is practised routinely in the regular production at Ifö.

6.3 The need for machining

It was mentioned in section 6.1, that the standard deviations presented in Table 3, based on the data in Tables A-1 and A-2 in Appendix A, do not adequately describe the need for machining.

Instead, a hypothetical cylinder needs to be defined, and the differences taken between the data points and the cylinder surface.

A standard procedure to determine such a cylinder would be a least squares refinement. In such an analysis, the sum of the squares of the differences between the real positions on the block surface and the ideal ones would be minimised. The analysis would render a map of the differences in radii between observed and calculated values together with the best radius.

Statistically, one would obtain a general parameter on the goodness of the fit as well as a standard deviation of the calculated radius. The main underlying assumption would be that the deviances are statistical in nature and random.

It can be expected that the standard deviations of the radii obtained would be considerably lower than those presented in Table 3.

An analysis of the nature just indicated would, however, not correspond very well to the present needs. The question is namely how much machining is needed before an even surface can be obtained. Therefore, the following parameters are of interest.

1. The *smallest maximum thickness* of material necessary to machine for a given block in order to obtain an even mantle surface. Below, this will be referred to as **SMT**.
2. The average over the height of the block of the *smallest average maximum thickness* of material which would have to be machined. Below, this will be referred to as **SAMT**.

The above statements may require some elaboration. In a simple lathing device, and for a given height and radius, the time of lathing would be proportional to the difference between the initial distance between the tool and the rotation axis, and the end distance. The initial distance corresponds to the position of the block which is furthest away from the axis of rotation (assuming that it is the outer surface which is to be lathed), and the final position corresponds to the position of the block which is closest to the axis of rotation. The latter case corresponds to a completely lathed surface.

The difference between the initial and end distance corresponds to the maximum thickness of material necessary to remove. For most of the surface the actual thickness is smaller.

Obviously, the difference between the initial and the end distance depends on how well the object has been mounted onto the lathing equipment. The smallest distance obtainable – SMT – corresponds to the "best" possible mounting.

If the lathing device is more intelligent, it may be able to utilize the fact that the maximum distance which needs to be lathed varies with the lateral position (i e along the direction of the axis of rotation). Thus, the tool engages only when there is material to be removed somewhere along the circumference of the block. In this case, it is the average along the direction of the axis of rotation of the maximum thicknesses along the circumference which is of interest, namely the above mentioned SAMT.

The values of SMT and SAMT have been determined and estimated as follows.

A cylinder was refined (orientation as well as diameter) to the data points for each block in such a way that the distance to the point furthest away was minimised. At the same time, no point was allowed to be inside the cylinder in the case of the outer diameter, and outside the cylinder in the case of the inner diameter. These conditions were fulfilled in each case through iterative calculations. Only one solution was found in each case. The farthest distance between a point and the refined cylinder surface was then taken as SMT. Mathematically, the SMT was determined exactly.

The solutions found for the refinement of SMT were then utilised for the estimation of SAMT. The maximum thickness was determined for each position along the direction of the axis of rotation, and the average over these was taken as SAMT.

This procedure gives rise to (at least) two types of systematic error.

Firstly, the optimization of the mounting with regard to SAMT does not, in general, give rise to the best mounting with regard to SAMT. For thin machinings in comparison with the dimensions of the blocks, this error is not assessed to be very great. The error does, however, give rise to values of SAMT which are pessimistic.

Secondly, measurements were taken at 90° intervals which implies that any roughness between these angular positions is not included. This error gives rise to optimistic values. It will, however, not give rise to any qualitatively different results.

It is possible that the two types of error largely balance each other. The results of the analyses and calculations are shown in Table 4.

The indications from Table 3 are supported by the more solid analysis underlying the results in Table 4. In the cases of the highest water ratio in combination with the highest pressure, block 7, the need for removal is substantially higher than for the rest of the blocks. This might be related to the degree of saturation during compaction, which is considerably higher than at ambient pressure due to the elastic (part of the) deformation during compaction. A high degree of saturation might give rise to oversaturation locally, in which case flow may be induced by a high pore pressure. (Data on the degree of water saturation in the blocks after the compaction operation can be found in Table 2).

For blocks 1 and 5, the depth of removal is comparatively high for the inner surface. A close examination of the original (see Tables A-1 and A-2) as well as the difference data (observed minus calculated values, not included in any of the tables) indicates that the differences are attributable mainly to one of the planes analysed and for the most part only a fraction of it. In view of this observation, a plausible explanation might be that some alteration occurred in the powder body when the central bar was removed after evacuation but before compaction. There was a friction between the bar and the rubber bag, and at least in some cases, a lubricating solution had to be applied in combination with force.

It is therefore assessed that the data just mentioned might be excluded when an estimate of the real need for removal is to be evaluated. It can thus be estimated that it is possible to obtain a completely lathed surface without having to remove more than at most between 10 and 15 millimeters, and on average about half of that or slightly less.

Table 4. The results of the calculations (in mm) of the smallest maximum thickness to be removed for each block. Regarding SMT and SAMT, see text

Block number	Inner surface Radius after machining	Need for removal average	SMT	SAMT	Outer surface Radius after machining	Need for removal average	SMT	SAMT
1	137	16	26	23	222	6	13	11
3	115	7	14	11	222	6	12	8
4	117	5	10	8	232	3	10	6
5	118	12	20	16	227	4	12	7
6	118	6	10	7	226	6	14	10
7	110	9	29	17	216	8	22	13
8	121	12	19	14	224	4	13	7
9	116	4	8	6	228	5	9	6
10	117	7	14	10	226	3	11	6
11	115	7	16	10	222	6	14	8
12	115	6	11	9	224	6	13	8

This need might be illustrated using a fictitious example. Let us assume that one of the blocks should be lathed using the same equipment as in the present study but with computerized devices for optimal orientation as well as sweeping over the surface. In such a case, the volume to be covered should correspond to SAMT. According to the results presented in Table 4, the average of SAMT for the different blocks is 8 millimeters. Since the outer mantle surface of one of the blocks is about 1.28 square meters, this corresponds to a volume to be removed of about 10 liters. With a capacity of the lathing equipment of 0.6 cubic meters per hour, this corresponds to an operation time of about one minute per run.

It should be noted that the above analysis of the need for machining does not include the variability of the compaction. This topic is discussed in section 9 but it may be mentioned already at this point that – for blocks of the present size – this effect is assessed to be smaller than the effect of the unevenness of the surface.

6.4 Significance of process parameters

The subsequent analysis on the significance of the process parameters is based on the data presented in Tables 3 and 4. In order to facilitate the analysis, data from these tables have been extracted and restructured as presented in Table 5.

As discussed in the previous section, distortion of the inner surface occurred as a result of the removal of the inner cylinder after evacuation but before compaction. The subsequent discussion is therefore primarily based on the data for the outer surface.

The (radial) linear reduction ranges between 18.6% and 22.3% with an average of 20.3%. This may be compared with the value for the linear reduction of 20.7% given in section 5.2, which corresponds to a volume reduction of 50%.

The linear reduction taking place as a result of the compaction of the powder increases with increasing pressure and hold time. For the lowest pressure and no hold time the linear reduction increases with increasing water ratio.

The need for removal increases with increasing pressure. The highest value appears for the highest water ratio and the highest pressure, cf section 6.2.

Table 5. Compilation of results from the measurements on the blocks. Regarding SMT and SAMT, see text.

Process parameters			Cf section 8.2.1	Inner mantle surface				Outer mantle surface				
				Measured	Derived need for removal			Measured		Derived need removal		
Block number	Maximum pressure, MPa	Hold time, minutes	Water ratio %	Average radius, mm	Average mm	SMT mm	SAMT mm	Linear reduction %	Average radius mm	Average mm	SMT mm	SAMT mm
4	74	0	11.6	112	5	10	8	18.6	235	3	10	6
1	112.5	0	11.4	121	16	26	23	20.7	228	6	13	11
3	112.5	10	11.4	108	7	14	11	20.9	228	6	12	8
9	74	0	14.4	112	4	8	6	19.2	233	5	9	6
5	112.5	0	14.1	106	12	20	16	19.8	231	4	12	7
12	112.5	10	14.4	109	6	11	9	20.0	230	6	13	8
10	74	0	17.5	110	7	14	10	20.3	229	3	11	6
6	112.5	0	16.9	112	6	10	7	19.4	232	6	14	10
8	112.5	10	17.0	109	12	19	14	20.8	228	4	13	7
11	74	0	20.3	108	7	16	10	20.9	228	6	14	8
7	112.5	0	19.9	101	9	29	17	22.3	224	8	22	13
Average				110	8	16	12	20.3	230	5	13	8

7 Visual observations of the compacted specimens

7.1 Observations made using the naked eye

The observations immediately after manufacturing were as follows. All of the blocks prepared had the expected shape as well as smooth and even surfaces. (*Cf* section 5.3 regarding a deformed block that was prepared as a consequence of too stiff a rubber plate). The surface roughness was assessed to be considerably less than 1 millimeter. In no case could any fracture be observed by the naked eye.

Immediately after manufacturing, the blocks were wrapped with plastic sheet material in order to avoid loss or uptake of water and the subsequent fracturing. From time to time the blocks were uncovered and inspected. No fractures were observed on the stored blocks while they were still covered with plastic sheets.

Much of the material had been subjected to lathing before the more detailed inspection.

When the samples were taken for the characterization (*cf* section 8) the plastic sheets had to be removed. (By that time the machining tests described in section 6 had also been concluded.) The sampling meant that the blocks were divided into pieces, and these pieces were not wrapped in plastic once again but left in the open in the room commissioned for the handling of bentonite. These pieces fractured as a result of drying. In some instances this fracturing could be connected to features in the blocks.

In one case, material at what might be the initiation point of a fracture showed a different texture from the rest of the fracture surface. A possible interpretation is that the area of different texture corresponds to what might have been a lump before compaction.

In a few cases, fractures were observed, the normal of which made an angle to the main axis of rotation of the "beaker" of about 45 degrees. Moreover, fracturing in a direction perpendicular to the previously mentioned fracture indicated some sort of stratification.

It might be tempting to conclude that such a stratification occurred as a result of uneven filling of the bag, and the associated segregation of grains along the slide surface. This explanation could be ruled out, however, since the filling had been carried out whilst maintaining an approximately horizontal surface throughout.

The conclusion is that the observations made indicate that the blocks had excellent quality. It should be noted that this conclusion includes the parts around the bottom and the bottom edge where the presence of anomalies might not otherwise readily be excluded.

7.2 Observations made using an ocular

The investigation of the blocks using an ocular as well as the naked eye included as compacted as well as lathed surfaces. The ocular used had a magnification of 30 times. It had a scale such that the size of the patterns observed could be readily apprehended.

The observations include the following:

1. **Local, lens shaped inclusions with a diameter of about 0.1-0.5 millimeters and consisting of very fine grains in an uneven surface.** The frequency was about 10 per square centimeter. They were observed only on the lathed surfaces and were interpreted as damaged bentonite granules with such an orientation that their shape was distorted and position altered during the passage of the tool of the lathe.
2. **Fine fractures in the borders between the grains.** The approximate average length was assessed to be about 0.05–0.2 millimeter. The frequency was assessed to be about 1–5 per square centimeter. These defects were observed only on the lathed surfaces and were interpreted as small fracturings appearing as a result of small modifications in the positions of the grains caused by the passage of the tool of the lathe.
3. **Fractures with a length of 1-5 millimeters, more or less parallel to the end faces of the block.** The frequency was assessed to be between 1–10 per square centimeter within about 20 millimeters from the upper end of the block. No or very little such fracturing was observed on other surfaces. These fractures were interpreted to be caused by the stresses developed during the pressure relief.

The conclusions of the observations are as follows.

The discontinuities of types 1 and 2 above have a very insignificant depth and are therefore assessed to have no significance for the stability of a block.

Defects of type 3 may occur not only at the surface and might therefore have a certain significance. It is assessed, however, to be very low since the frequency is low.

It is therefore assessed that the blocks are sufficiently homogenous to resist fracturing in conjunction with handling.

8 Sampling and characterization

8.1 Prerequisites for the sampling and characterization

The sampling and characterization programme was designed to reveal not only the materials properties in general but also any volumes in the blocks which might have properties which deviate from those in general.

The sampling was also designed to indicate any changes in the properties of the blocks or the material in the blocks, which would come about as a result of the changes in the parameters of preparation for the different blocks.

The sampling programme was dimensioned in such a manner that the data could be subjected to statistical analysis.

8.2 Sampling

The extraction of samples was made in accordance with the map presented in Figure 2. These positions were used for all determinations described in section 8, i.e. including measurements of density, water ratio, strain at maximum stress and tensile strength. The number of samples taken are also indicated in the figure.

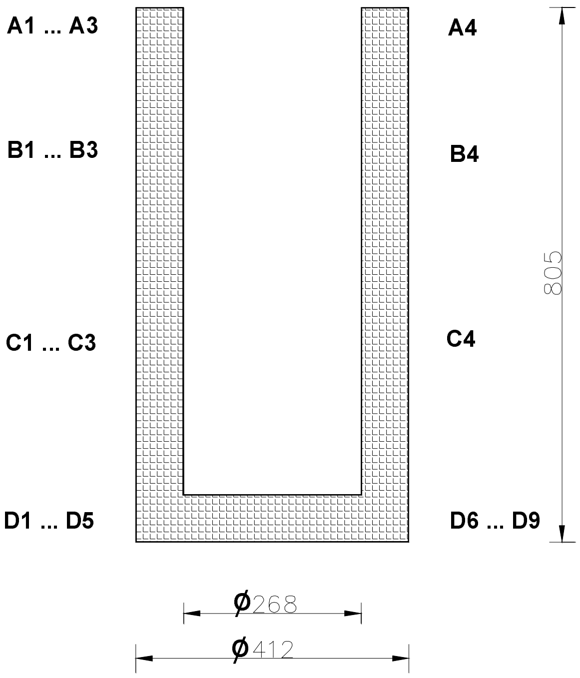

Figure 2. *The approximate positions of the sampling points in the blocks together with the number of samples taken at each point.*

8.3 Water ratio and density

Composite data for the water ratios and densities obtained are shown in Tables 6 and 7, respectively. Full data are presented in Table A-3 (water ratios) and Table A-4 (densities).

Data on void ratios are also shown for comparison. Composite data are shown in Table 8 and full data are presented in Table A-5. The data were derived from the original data on densities and water ratios as described in /6/.

The composite data was obtained by taking the average of the measurements of each sampling position (for each block). The number of samples used for the composite data are also shown in Tables 6–8 (and in more detail in Tables A-3–A-5).

Standard deviations were calculated on the composite data as well as on the full set of 21 values for each block and the results are also shown in Tables 6 and 7.

The standard deviations were calculated as described in section 6.1 (using formula 1).

Inspection and analysis of the data in Tables 6, 7, A-3 and A-4 indicates that the values are exceptionally stable and the variations are very small.

The differences between the corresponding mean values do not appear to be large in relation to the standard deviations.

Moreover, the composite standard deviations do not appear to be substantially smaller than the corresponding standard deviation of all data (on one property on one block).

Thus, a very simple statistical analysis of the data provides little basis for the preference of any model where the different sampling positions see Figure 2 are assumed to be different.

Furthermore, the simplest model which is compatible with the data is one in which the sampling positions are equivalent.

Accordingly, it is assessed that the latter model is the one to be preferred.

Table 6. Water ratio and standard deviations for the various blocks and different positions in the blocks (see Figure 2 in section 8.2)

Block number	Sampling position; number of samples in parenthesis A (4)		B (4)		C (4)		D (9)		Average and standard deviation of all	
	average water ratio	standard deviation	average water ratio	standard deviation	average water ratio	standard deviation	average water ratio	standard deviation	water ratio	standard deviation
1	0.1164	0.0008	0.1146	0.0023	0.1105	0.0072	0.1151	0.0030	0.1144	0.0040
3	0.1170	0.0063	0.1130	0.0005	0.1127	0.0005	0.1129	0.0008	0.1137	0.0031
4	0.1159	0.0003	0.1170	0.0007	0.1160	0.0002	0.1151	0.0020	0.1158	0.0015
5	0.1400	0.0009	0.1409	0.0007	0.1405	0.0008	0.1421	0.0018	0.1412	0.0015
6	0.1698	0.0016	0.1677	0.0032	0.1675	0.0010	0.1694	0.0028	0.1688	0.0025
7	0.1985	0.0028	0.2002	0.0012	0.1994	0.0007	0.1982	0.0017	0.1989	0.0018
8	0.1708	0.0010	0.1699	0.0009	0.1698	0.0007	0.1700	0.0009	0.1701	0.0009
9	0.1437	0.0005	0.1438	0.0005	0.1443	0.0010	0.1430	0.0009	0.1436	0.0009
10	0.1746	0.0007	0.1748	0.0014	0.1757	0.0008	0.1754	0.0028	0.1752	0.0019
11	0.2033	0.0007	0.2020	0.0009	0.2025	0.0014	0.2027	0.0013	0.2026	0.0011
12	0.1431	0.0032	0.1451	0.0013	0.1444	0.0010	0.1426	0.0007	0.1435	0.0018
Average	0.1539	0.0017	0.1536	0.0012	0.1530	0.0014	0.1533	0.0017	0.1534	0.0019

Table 7. Density and standard deviations in grams per cubic centimeter for the various blocks and different positions in the blocks (cf Figure 2 in section 8.2)

Block	Sampling position; number of samples in parenthesis								Average and standard deviation of all	
number	A (4)		B (4)		C (4)		D (9)			
	average density	standard deviation	average density	standard deviation	average density	standard deviation	average density	standard deviation	density	standard deviation
1	2.101	0.003	2.097	0.003	2.094	0.004	2.094	0.009	2.096	0.007
3	2.113	0.003	2.113	0.003	2.114	0.002	2.111	0.004	2.112	0.003
4	2.028	0.001	2.030	0.002	2.026	0.003	2.025	0.004	2.027	0.003
5	2.089	0.003	2.090	0.001	2.091	0.003	2.088	0.003	2.089	0.003
6	2.055	0.000	2.056	0.002	2.055	0.002	2.052	0.006	2.054	0.004
7	2.034	0.002	2.036	0.002	2.037	0.001	2.034	0.002	2.035	0.002
8	2.057	0.003	2.057	0.003	2.057	0.002	2.056	0.003	2.057	0.003
9	2.028	0.001	2.021	0.004	2.020	0.002	2.017	0.004	2.021	0.005
10	2.038	0.002	2.034	0.004	2.035	0.003	2.029	0.005	2.033	0.005
11	2.034	0.001	2.033	0.001	2.034	0.002	2.030	0.004	2.032	0.003
12	2.088	0.029	2.097	0.005	2.097	0.003	2.097	0.003	2.095	0.013
Average	2.0605	0.0045	2.0604	0.0027	2.0601	0.0024	2.0577	0.0041	2.0592	0.0046

Table 8. Void ratio and standard deviations for the various blocks and different positions in the blocks (cf Figure 2 in section 8.2). (Void ratio data are derived from data on water ratio and density)

Block	Sampling position; number of samples in parenthesis								Average and standard deviation of all	
number	A (4)		B (4)		C (4)		D (9)			
	average void ratio	standard deviation	average void ratio	standard deviation	average void ratio	standard deviation	average void ratio	standard deviation	pore ratio	standard deviation
1	0.4773	0.0031	0.4773	0.0038	0.4741	0.0105	0.4803	0.0038	0.4780	0.0056
3	0.4698	0.0089	0.4644	0.0024	0.4630	0.0014	0.4660	0.0030	0.4658	0.0048
4	0.5295	0.0007	0.5299	0.0019	0.5299	0.0015	0.5306	0.0032	0.5302	0.0023
5	0.5168	0.0032	0.5176	0.0012	0.5166	0.0028	0.5203	0.0038	0.5184	0.0034
6	0.5821	0.0021	0.5785	0.0042	0.5790	0.0021	0.5845	0.0056	0.5819	0.0048
7	0.6382	0.0050	0.6390	0.0034	0.6368	0.0018	0.6373	0.0033	0.6377	0.0033
8	0.5819	0.0039	0.5814	0.0032	0.5810	0.0008	0.5819	0.0022	0.5816	0.0025
9	0.5679	0.0012	0.5731	0.0033	0.5741	0.0019	0.5751	0.0027	0.5731	0.0036
10	0.6027	0.0024	0.6055	0.0044	0.6062	0.0026	0.6105	0.0076	0.6072	0.0061
11	0.6446	0.0016	0.6439	0.0013	0.6434	0.0018	0.6468	0.0036	0.6452	0.0029
12	0.5222	0.0234	0.5178	0.0050	0.5173	0.0036	0.5143	0.0024	0.5172	0.0102
Average	0.5575	0.0050	0.5571	0.0031	0.5565	0.0028	0.5589	0.0037	0.5578	0.0045

8.4 Mechanical properties

Composite data for the tensile strengths and maximum strains at failure obtained are shown in Tables 9 and 10, respectively. Full data are presented in Table A-6 (tensile strengths) and Table A-7 (maximum strains at failure).

The composite data were obtained by taking the average of the measurements of each sampling position (for each block). The number of samples used for the composite data are also shown in Tables 9 and 10.

Standard deviations were calculated in the same way as described in section 6.1 (using formula (1)).

Standard deviations were calculated on the composite data as well as on the full set of 21 values for each block and the results are also shown in Tables 9 and 10.

Inspection and analysis of the data in Tables 9, 10, A-6 and A-7 were conducted in the same way as in section 8.3 and the conclusions are as follows.

The tensile strength and maximum strain at failure have much higher standard deviations than the water content and the density.

A preferred model can be identified in the same way as in section 8.3. According to this model, the sampling points are equivalent.

Examples of stress versus strain curves obtained for two water ratios are shown in Figure 3 in section 8.4. It can be seen in the figure that the shapes of the stress versus strain curves vary with the water content. In Figure 3a, there is a relatively brittle behaviour for block number 3 which has a water content of 11.3%. In figure 3b, the material is more ductile in block number 7 which has a water content of 19.9%.

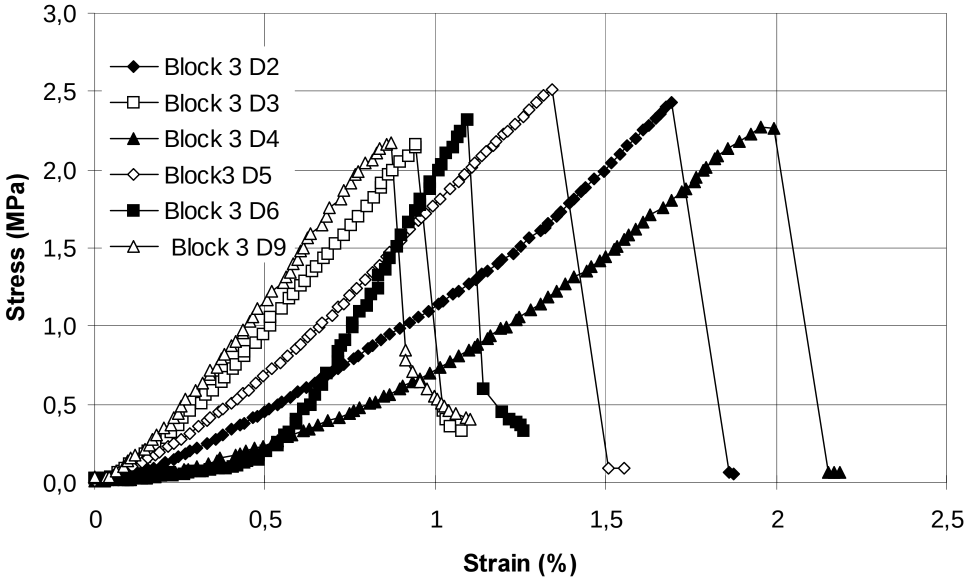

Figure 3a. *Stress (in MPa) versus strain curves for block number 3 which has a water ratio of 11.3%.*

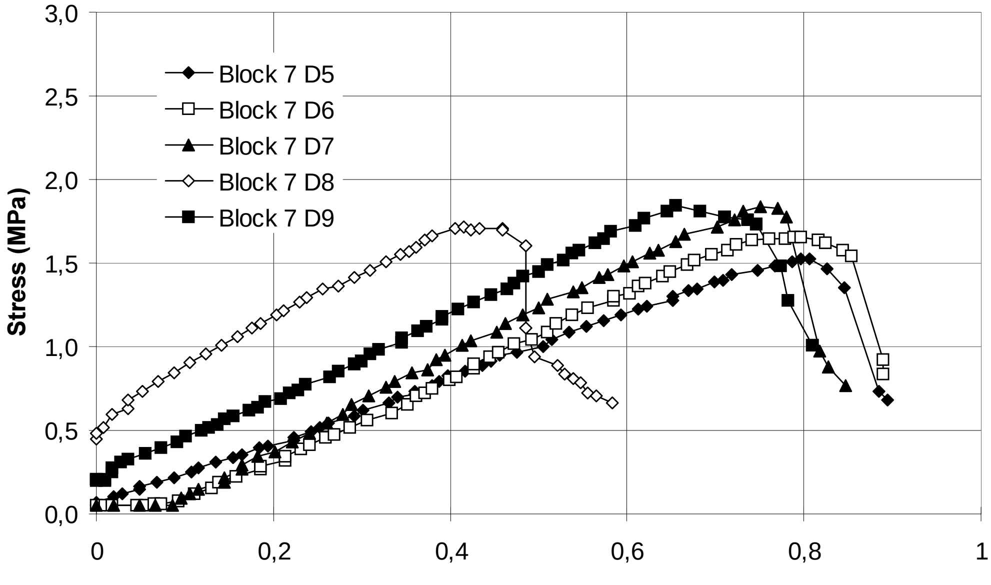

Figure 3b. *Stress (in MPa) versus strain curves for block number 7 which has a water ratio of 19.9%.*

Table 9. Tensile strength and standard deviations in MPa for the various blocks and different positions in the blocks (cf Figure 2)

Block number	Sampling position; number of samples in parenthesis								Average and standard deviation of all	
	A (4)		B (4)		C (4)		D (9)			
	average tensile strength	standard deviation	average tensile strength	standard deviation	average tensile strength	standard deviation	average tensile strength	standard deviation	tensile strength	standard deviation
1	1.24	0.04	1.62	0.73	1.89	0.29	2.04	0.47	1.83	0.52
3	2.00	0.28	2.38	0.37	2.27	0.62	2.31	0.14	2.24	0.34
4	1.71	0.29	1.34	0.32	1.72	0.48	1.27	0.08	1.47	0.34
5	2.48	0.44	2.23	0.19	2.41	0.18	2.80	0.45	2.56	0.42
6	2.61	0.28	2.22	0.15	2.27	0.34	2.65	0.29	2.50	0.33
7	1.38	0.15	1.52	0.13	1.80	0.36	1.67	0.13	1.61	0.23
8	1.98	0.11	2.02	0.08	2.02	0.13	2.39	0.56	2.17	0.41
9	1.93	0.11	1.82	0.12	1.86	0.12	2.06	0.31	1.95	0.24
10	1.96	0.65	1.65	0.33	1.70	0.18	1.86	0.26	1.80	0.33
11	1.71	0.29	1.97	0.26	1.91	0.22	1.67	0.22	1.78	0.25
12	2.46	0.51	2.37	0.30	2.37	0.28	2.31	0.66	2.36	0.49
Average	1.95	0.29	1.92	0.27	2.02	0.29	2.10	0.32	2.03	0.35

Table 10. Maximum strain at failure and standard deviations in percent for the various blocks and different positions in the blocks (cf Figure 2)

Block number	Sampling position; number of samples in parenthesis								Average and standard deviation of all	
	A (4)		B (4)		C (4)		D (9)			
	average maximum strain at failure	standard deviation	average maximum strain at failure	standard deviation	average maximum strain at failure	standard deviation	average maximum strain at failure	standard deviation	tensile strain at failure	standard deviation
1	0.82	0.11	0.94	0.17	0.85	0.21	1.01	0.17	0.94	0.17
3	0.99	0.45	0.81	0.13	1.04	0.38	1.32	0.43	1.07	0.40
4	1.10	0.22	0.88	0.01	0.76	0.12	0.98	0.10	0.94	0.17
5	0.59	0.02	0.60	0.06	0.72	0.14	0.71	0.08	0.67	0.10
6	0.69	0.10	0.66	0.06	0.54	0.17	0.78	0.14	0.70	0.15
7	0.57	0.08	0.99	0.63	0.86	0.17	0.71	0.12	0.77	0.31
8	0.71	0.11	0.63	0.03	0.74	0.12	0.73	0.07	0.71	0.09
9	0.62	0.07	0.58	0.03	0.56	0.14	0.80	0.16	0.68	0.16
10	0.69	0.13	0.59	0.12	0.74	0.15	0.66	0.04	0.66	0.10
11	0.78	0.19	0.87	0.16	0.82	0.11	0.70	0.08	0.77	0.13
12	0.63	0.10	0.80	0.03	0.70	0.06	0.65	0.20	0.68	0.15
Average	0.74	0.14	0.76	0.13	0.76	0.16	0.82	0.15	0.78	0.18

8.5 Significance of process parameters

It was concluded in sections 8.3 and 8.4 that the preferred model for the interpretation of all underlying data is that all points on each block are equivalent. This implies that composite data from all samples from each block can be used in comparisons between the blocks.

In order to facilitate such a comparison, all the main data from sections 8.3 and 8.4 (data from sampling and characterisation) have been compiled together with some process parameters from Table 2, and the result is shown in Table 11. Derived data on the degree of pore saturation as well as void ratio are also included in the table. The data is arranged in such a manner that an analysis of the influence of pressure, hold time and water ratio may be facilitated.

The following observations can be made.

The void ratio decreased with increasing pressure, but the effect was hardly significant for the higher water ratios.

A higher pressure implies that the tensile strength is higher while the maximum strain at failure is essentially invariant with regard to the pressures tested.

No variation in any of the properties can be detected as a result of variation in the hold time (except possibly for the lowest water ratio). It would therefore be tempting to conclude that hold time is of little significance for the pressures tested. Examination of the data indicates, however, that an influence of the hold time at the lowest pressure tested cannot be excluded since this was not tested.

The tensile strength was relatively invariant with respect to water ratio except for the highest pressure in combination with the highest water ratio and the lowest pressure in combination with the lowest water ratio.

The maximum strain at failure was lowest for intermediate water ratios, and highest for the lowest water ratios.

The void ratio increased with increasing water ratio.

Table 11. Compilation of results from the sampling and characterization activities

Process parameters			Data from sampling and characterisation				Derived data		
Block number	Maximum pressure MPa	Hold time, minutes	Water ratio, %	Density, grams per cubic centimeter	Tensile strength MPa	Maximum strain at failure, %	Density at full pore saturation grams per cubic centimeter †	Degree of pore saturation %	Void ratio, %
4	74	0	11.6	2.03	1.47	0.94	2.16	62	53
1	112.5	0	11.4	2.10	1.83	0.94	2.20	66	48
3	112.5	10	11.4	2.11	2.24	1.07	2.22	68	47
9	74	0	14.4	2.02	1.95	0.68	2.13	70	57
5	112.5	0	14.1	2.09	2.56	0.67	2.17	76	52
12	112.5	10	14.4	2.10	2.36	0.68	2.17	77	52
10	74	0	17.5	2.03	1.80	0.66	2.11	80	61
6	112.5	0	16.9	2.05	2.50	0.70	2.13	81	58
8	112.5	10	17.0	2.06	2.17	0.71	2.13	81	58
11	74	0	20.3	2.03	1.78	0.77	2.08	87	65
7	112.5	0	19.9	2.04	1.61	0.77	2.09	87	64

† The density of the hypothetical solid part of the bentonite was taken to be 2.78 grams per cubic centimeter.

9 Discussion and concusions

In the present report, various aspects of pilot scale isostatic compaction have been investigated in order to illuminate the potentially crucial issues which have been identified in the previous work. The experiments have been carried out on a scale of one to four (on a linear scale) in order to be reasonably relevant to the full scale.

The results from the various sub-tasks have been presented, analysed and discussed in their respective sections in the report. The main results and conclusions are also reiterated below in conjunction with some further analysis and discussion. In this way, a more integrated compilation can be made of the results. Also some more general conclusions can be drawn.

Crushed bentonite powder can be moistened and mixed with water using standard equipment which is readily commercially available. However, the mixing need to be intense and the equipment and procedure tested for the purpose. The product can be prepared to be very homogenous. Some care may be required in order to limit the formation of lumps which may cause inhomogeneities. Nonetheless, lumps will form but only to a minor extent. They can, if desirable, be removed using standard sieving equipment.

Bentonite blocks can be compacted with a swiftness and an efficiency which resemble those of the regular, optimized production.

Thus, evacuation can be carried out in a matter of five minutes for a block on the scale of 1:4. Similar evacuation times are used in the regular production at Ifö which involves much larger objects.

The compaction can be carried out within about the same amount of time as the evacuation plus the hold time. This implies that it is feasible to arrange the production in such a manner that the expensive press is being used only for the actual compaction. All other handling involves only very inexpensive equipment by comparison.

No problem was identified in operating a press for bentonite about as efficiently as for preparation of green bodies for large insulators.

The precision of the objects manufactured using isostatic compaction mainly depends on the homogeneity of the press powder in the bag. The homogeneity, in turn, is expected to be a result of the homogeneity of the press powder before filling, its flow properties and the filling operation.

In the present case, the homogeneity of the powder was checked and found to be good but not flawless. Each filling was composed of a few batches of mixed powder since the mixer used was not sufficiently large to mix all that which was needed for one block in one batch. The flow properties were poor in comparison with what is probably the case in comparable operations where spray dried powder is typically used. The filling operation was carried out carefully by hand which may be very good but may nonetheless be assessed to be no better than a special mechanical system.

It was found in section 8 that the influence of the hold time was relatively insignificant for relatively dry powder compacted at high pressure. Thus, blocks 1 and 3, blocks 5 and 8, and to some extent blocks 6 and 8 (cf Table 5) might be compared for reproducibility. It can be assessed that the reproducibility is probably better than 1%. It has been claimed that lower values than this can be obtained for optimized production.

Measurements made on the blocks together with mathematical evaluations indicate that even under relatively pessimistic assumptions, not more than about one centimeter would need to be removed by machining. It should be observed in this context that no special efforts have been made in order to minimize the variations in the raw surface and that there ought to be a potential for improvement.

It can be foreseen that the thickness to be removed is greater for a full size block, but the effect cannot be expected to be simply linear since the observations made during the course of the work indicate that there is a surface effect (e g associated with the rubber bag). It can be expected that less material would need to be removed than a linear assumption would indicate.

The results indicate that the dimensional stability may deteriorate in cases where the specimen approaches pore saturation during the compaction.

In order to illustrate the possible consequences of machining, an example is given of the types of calculations that can be made. It is assumed that the layer which will have to be removed is 30 millimeters. It is furthermore assumed that the total surface area of the blocks in a deposition hole will have to be treated. Only the cylindrical areas are included and are estimated to be about 60 m^2 in total.

It was mentioned in section 6.3 that in the present tests, an 8 millimeter thick layer could be removed from almost 100 m^2 in one hour. By going over the surface four times, 32 millimeters of material would be removed. (Actually the layer varies in thickness between 0 and 30 millimeters).

Using these figures it can now readily be calculated that the blocks needed for one deposition hole could be machined in less than two and a half hours. This time does not include the time needed to load and unload the blocks and to handle them.

Although machining is still an extra step in the production and gives rise to the increased complexity of the repository system, no particular problem has been identified for the application of this technique.

It should be pointed out that the present report deals only with what can be obtained by the different techniques tested, and not on what should be required in a specification. At present, there are no specifications – at least no formal ones – on the precision needed in the dimensions of the blocks, and it cannot be excluded that the blocks can be used without any treatment of the surfaces after compaction.

In the twelve blocks produced, no crack could be identified by the naked eye. When an ocular was used which magnified about 30 times, microcracks were observed. Such cracks can be expected to be present since the grains deform and pore water relocates during compaction, and when the pressure is being released tensile forces are likely to appear in the microscale. In spite of their minute size the microcracks may well influence the mechanical strength measured since bentonite is a relatively brittle material and since fractures in such materials typically originate from discontinuities.

It is expected that bentonite fractures on drying, and the fact that fracturing took place at a later stage cannot be associated with the method used for the compaction. It is conceivable, however, that the pattern of fracturing actually obtained is related to the powder handling and compaction techniques. On the other hand, other types of factors may also have had an influence, for instance if the drying was not even over the entire surface.

The data from sampling and characterization indicate that the homogeneities of all the blocks were excellent, as reflected by the measurements of void ratio, density, strain at maximum stress and tensile strength. The supporting evidence includes not only the values themselves but also the spread in these values which was low or at least moderate. The high homogeneity observed is supported by the above mentioned absence of visible cracks and other discontinuities.

The high homogeneity was observed for different water ratios, compaction pressures and hold times, and it is concluded that isostatic compaction may be feasible for wide ranges of production parameters. The high homogeneity in all cases studied also implies that each block can possess specific and verifiable materials properties.

This means that the properties of the blocks may be somewhat engineered to fit the needs of the user.

Thus, a higher pressure implies that the block becomes somewhat smaller, especially for lower water ratios.

The need for machining may be smaller for lower pressures and lower water ratios.

An increase in pressure will lead to a lower void ratio (except perhaps for higher water ratios) and a higher tensile strength.

The hold time at high pressures appears to have little or no significance for the materials properties determined.

An increase in the water ratio leads to a higher void ratio. The maximum strain at failure may be lowest for intermediate water ratios. The tensile strength may to a large extent be invariant to variations in water ratio.

The magnitudes of the densities of the blocks are such that it can be assessed that pertinent densities can be achieved in the repository after water saturation.

It is important to note that the stress-strain curves have rather different appearances at high void ratios, compared to low, see Figure 3 in section 8.4. The material is quite ductile at high void ratios but brittle at low void ratios.

The general assessments which can be made based on the mechanical strength data is that all blocks contain material with a good mechanical strength and with relatively little spread in the data.

It can also be assessed that cracks with a high potential of causing fractures are unlikely to be present unless drying takes place. (Nonetheless, it can be expected that if fractures are to form, they will do so starting at a small crack). Thus, the present results provide no basis for questioning the mechanical integrity of a block even under handling.

However, the assessment of the performance of a block from a mechanical point of view involves other aspects as well which are outside the scope of the present study.

No special difficulty was encountered which was related to the complex shape of the blocks (beaker-like). The complex shape influenced the design of the bag and filling system, as well as the procedure for filling. It did not, however give rise to any inhomogeneities or discontinuities which could be detected by the techniques used in the present study.

It is beyond the scope of the work reported here to make comparisons with uniaxial compaction. It might nonetheless be mentioned that the isostatic technique implies that no substances are used other than bentonite and pure water, and that no limitations apply regarding the height / diameter ratio of the blocks.

One of the purposes of the present work was to try to identify issues where further difficulties might be foreseen on a larger scale. No such issues were identified apart for the one regarding the need for machining which is dealt with above. In addition, the extensive experience at Ifö indicates that processes involving cold isostatic compaction of powder readily lend themselves to scaling up. It is therefore assessed that no or very few difficulties are to be expected when one is going from the (linear) scale of 1:4 to full scale. The present results and conclusions are therefore assessed to be highly relevant also for the full scale.

It is nonetheless recommended that tests be carried out on a scale substantially larger than 1:4 in order to verify the present conclusions. In such a work, efforts should be made to try to identify effects and potential problems related to scale.

It is also recommended that the following issues should be dealt with in conjunction with the preparation of larger blocks.

- The need for machining
- Mechanical properties of large entities of bentonite
- Processes for the production of bentonite blocks. Special attention should be paid to design and removal of the central cylinder.

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A Appendix A, detailed data

This appendix includes the following tables showing detailed original data.

A-1 Outer surfaces of the blocks.
A-2 Inner surfaces of the blocks.
A-3 Water ratios.
A-4 Void ratios.
A-5 Densities.
A-6 Tensile strength.
A-7 Strain at failure.

Table A-1. The results of the measurements of the outer surfaces of the blocks. The measurements relate to the rotation axis which the block had when it was mounted in the lathing equipment

Angle °		Block number										
distance mm		1	3	4	5	6	7	8	9	10	11	12
0	0	232	227	226	229	233	246	259	239	247	237	234
0	100	234	223	225	219	238	235	253	237	237	230	230
0	200	232	221	228	224	235	234	248	237	233	228	229
0	300	237	219	227	225	235	235	244	235	232	229	229
0	400	236	218	227	225	236	236	241	235	233	229	230
0	500	234	218	228	228	236	239	238	234	232	230	231
0	600	234	217	227	233	237	242	235	234	234	230	233
0	700	231	222	227	237	238	246	234	233	233	230	234
0	800	229	216	229	236	239	248	232	233	232	230	
0	900			230			248	228	235	233	233	
90	0	216	225	227	219	230	252	223	221	252	251	224
90	100	213	222	223	214	227	239	219	221	245	242	217
90	200	215	222	225	214	226	235	217	219	240	239	214
90	300	219	223	227	216	227	233	217	217	238	237	215
90	400	222	226	229	218	230	235	218	220	237	235	220
90	500	227	229	232	222	232	233	218	222	237	234	222
90	600	232	233	234	223	235	231	219	225	236	232	226
90	700	237	237	237	225	238	230	219	227	237	230	228
90	800	238	226	240	220	240	231	220	230	233	228	
90	900			240			232	222	234	235	228	
180	0	227	236	245	248	233	225	211	232	227	234	238
180	100	222	233	242	240	229	212	202	230	221	226	232
180	200	221	233	240	238	227	207	204	230	219	224	230
180	300	221	232	241	236	225	204	207	230	220	225	231
180	400	223	236	241	233	226	202	211	232	222	226	229
180	500	224	236	241	232	224	202	217	232	223	227	227
180	600	227	239	242	230	225	203	222	232	227	226	227
180	700	230	242	242	229	226	199	226	233	228	226	226
180	800	237	241	243	228	227	206	231	234	229	224	
180	900			243			208	234	234	231	228	
270	0	233	236	247	254	244	225	243	254	221	220	249
270	100	232	236	244	245	241	225	240	246	213	211	243
270	200	231	232	243	242	236	221	236	242	213	209	240
270	300	233	229	241	242	233	213	235	240	215	210	238
270	400	228	226	238	241	233	208	234	239	216	213	239
270	500	228	225	237	239	229	203	233	238	217	217	240
270	600	229	225	235	238	227	203	233	236	221	219	236
270	700	228	225	233	237	228	206	235	237	223	223	228
270	800	226	219	231	235	230	209	235	236	226	227	
270	900			226			215	234	232	228	230	
Average	228	228	235	231	232	224	228	233	229	228	230	
Std dev	3	2	1	3	2	6	3	2	3	3	3	
Std dev %	1	1	0	1	1	3	1	1	1	1	1	

Table A-2. The results of the measurements of the inner surfaces of the blocks. The measurements relate to the rotation axis which the block had when it was mounted in the lathing equipment

Angle °		Block number										
distance mm		1	3	4	5	6	7	8	9	10	11	12
0	0	117	104	96	120	123	120	140	114	124	117	109
0	100	114	100	99	111	122	116	134	110	118	112	111
0	200	114	97	99	105	119	113	127	107	115	112	108
0	300	113	94	100	111	118	114	124	107	114	113	109
0	400	114	92	102	113	120	115	121	109	113	118	108
0	500	114	89	105	112	119	109	116	107	110	122	108
0	600	112	87	108	119	121	110	113	107	108	118	106
0	700	111		109	116	123	115	108	106	103	122	
0	800						123	110		108	114	
90	0	115	112	107	99	116	123	105	105	133	124	101
90	100	137	103	100	95	110	121	104	104	130	120	97
90	200	137	101	101	93	111	119	99	109	128	116	98
90	300	134	99	101	92	114	117	100	108	129	115	102
90	400	135	99	103	97	115	113	97	113	127	112	105
90	500	136	101	104	102	114	110	98	116	127	110	104
90	600	109	99	104	104	114	109	96	114	125	110	101
90	700	114		108	105	113	102	94	113	124	106	
90	800						109	88		133	94	
180	0	120	126	129	116	108	88	102	115	103	109	116
180	100	114	122	122	114	104	96	89	115	100	104	115
180	200	115	119	121	109	104	91	89	114	104	102	114
180	300	118	120	124	107	103	85	93	115	101	102	111
180	400	121	121	124	97	104	83	96	117	100	103	109
180	500	123	127	120	93	105	82	99	117	101	103	108
180	600	129	131	121	87	105	81	105	115	104	101	107
180	700	131		122	80	102	87	107	115	105	97	
180	800						86	112		109	95	
270	0	133	116	126	125	120	96	122	127	93	101	129
270	100	127	114	118	118	117	94	119	123	92	98	123
270	200	122	113	120	119	111	88	116	119	93	95	121
270	300	121	110	119	115	110	81	119	113	94	98	114
270	400	119	107	125	108	109	77	122	109	92	105	112
270	500	120	105	121	109	109	79	121	112	94	108	109
270	600	118	104	121	105	106	84	115	111	95	109	107
270	700	118		120	107	103	97	116	109	95	109	
270	800						110	124		101	109	
Average	121	108	112	106	112	101	109	112	110	108	109	
Std dev	6	4	2	5	2	5	4	2	3	3	3	
Std dev %	5	4	2	4	2	5	3	2	3	3	3	

Table A-3. The results of the measurement of the water ratios. The sample positions are given in Figure 2. Spurious data are given in parenthesis; they are not used in the statistical analysis

Sample position	Block number										
	1	3	4	5	6	7	8	9	10	11	12
A1	0.1165	0.1136	0.1155	0.1412	0.1692	0.1971	0.1694	0.1434	0.1741	0.2030	0.1440
A2	0.1152	0.1140	0.1160	0.1402	0.1719	0.1984	0.1705	0.1442	0.1741	0.2029	0.1454
A3	0.1172	0.1141	0.1163	0.1390	0.1697	0.1961	0.1713	0.1440	0.1751	0.2044	0.1445
A4	0.1166	0.1264	0.1157	0.1398	0.1682	0.2024	0.1717	0.1432	0.1753	0.2029	0.1383
B1	0.1143	0.1131	0.1173	0.1399	0.1673	0.2020	0.1701	0.1440	0.1735	0.2030	0.1454
B2	0.1145	0.1122	0.1176	0.1412	0.1652	0.1994	0.1695	0.1437	0.1737	0.2024	0.1440
B3	0.1120	0.1134	0.1172	0.1411	0.1659	0.2001	0.1690	0.1431	0.1758	0.2012	0.1441
B4	0.1176	0.1133	0.1160	0.1416	0.1724	0.1994	0.1711	0.1443	0.1762	0.2013	0.1468
C1	0.1048	0.1125	0.1162	0.1415	0.1683	0.1998	0.1687	0.1445	0.1765	0.2032	0.1443
C2	0.1038	0.1125	0.1158	0.1400	0.1676	0.2001	0.1701	0.1456	0.1758	0.2029	0.1431
C3	0.1178	0.1134	0.1159	0.1397	0.1661	0.1993	0.1701	0.1443	0.1747	0.2004	0.1448
C4	0.1156	0.1123	(0.1564)	0.1407	0.1680	0.1984	0.1702	0.1430	0.1759	0.2036	0.1455
D1	0.1080	(0.0840)	0.1125	0.1420	0.1729	0.1960	0.1680	0.1409	0.1708	0.2027	0.1418
D2	0.1156	0.1137	0.1163	0.1433	0.1720	0.1961	0.1701	0.1431	0.1770	0.2013	0.1417
D3	0.1177	0.1139	0.1169	0.1445	0.1708	0.1992	0.1702	0.1433	0.1777	0.2016	0.1432
D4	0.1164	0.1125	0.1174	0.1438	0.1704	0.2005	0.1708	0.1431	0.1804	0.2034	0.1431
D5	0.1141	0.1128	0.1174	0.1429	0.1685	0.2003	0.1701	0.1438	0.1762	0.2027	0.1430
D6	0.1177	0.1130	0.1128	0.1422	0.1654	0.1976	0.1694	0.1438	0.1749	0.2020	0.1425
D7	0.1164	0.1126	0.1135	0.1413	0.1646	0.1993	0.1700	0.1435	0.1745	0.2047	0.1438
D8	0.1156	0.1132	0.1136	0.1391	0.1687	0.1964	0.1704	0.1427	0.1734	0.2045	0.1418
D9	0.1141	0.1114	0.1157	0.1399	0.1708	0.1984	0.1714	0.1432	0.1738	0.2015	0.1426

Table A-4. The results of the determination of void ratios. The sample positions are given in Figure 2. Spurious values are given in parenthesis; they are not used in the statistical analysis

Sample position	Block number										
	1	3	4	5	6	7	8	9	10	11	12
A1	0.4788	0.4626	0.5294	0.5213	0.5816	0.6374	0.5775	0.5666	0.5999	0.6426	0.5116
A2	0.4729	0.4656	0.5289	0.5154	0.5850	0.6381	0.5810	0.5691	0.6017	0.6450	0.5149
A3	0.4772	0.4683	0.5305	0.5137	0.5816	0.6325	0.5820	0.5685	0.6037	0.6464	0.5568
A4	0.4801	0.4828	0.5293	0.5169	0.5801	0.6447	0.5870	0.5672	0.6054	0.6444	0.5057
B1	0.4745	0.4628	0.5327	0.5161	0.5762	0.6442	0.5814	0.5780	0.6018	0.6458	0.5205
B2	0.4758	0.4622	0.5295	0.5186	0.5773	0.6376	0.5796	0.5716	0.6038	0.6435	0.5136
B3	0.4759	0.4649	0.5289	0.5173	0.5759	0.6371	0.5787	0.5706	0.6044	0.6432	0.5136
B4	0.4830	0.4676	0.5286	0.5186	0.5847	0.6372	0.5859	0.5722	0.6118	0.6432	0.5236
C1	0.4690	0.4632	0.5316	0.5203	0.5816	0.6378	0.5817	0.5762	0.6069	0.6457	0.5171
C2	0.4624	0.4610	0.5294	0.5164	0.5769	0.6389	0.5811		0.6045	0.6429	0.5138
C3	0.4863	0.4645	0.5288	0.5134	0.5780	0.6355	0.5814	0.5734	0.6039	0.6413	0.5160
C4	0.4787	0.4630	(0.5895)	0.5163	0.5797	0.6350	0.5799	0.5726	0.6097	0.6437	0.5223
D1	0.4821	(0.4260)	0.5242	0.5213	0.5909	0.6355	0.5801	0.5777	0.5993	0.6452	0.5122
D2	0.4765	0.4681	0.5316	0.5246	0.5881	0.6349	0.5853	0.5745	0.6151	0.6464	0.5109
D3	0.4823	0.4667	0.5291	0.5222	0.5863	0.6367	0.5808	0.5794	0.6164	0.6455	0.5170
D4	0.4833	0.4600	0.5351	0.5238	0.5862	0.6425	0.5849	0.5704	0.6253	0.6506	0.5164
D5	0.4779	0.4690	0.5320	0.5201	0.5847	0.6406	0.5822	0.5763	0.6099	0.6411	0.5154
D6	0.4762	0.4661	0.5318	0.5236	0.5889	0.6356	0.5833	0.5734	0.6063	0.6510	0.5168
D7	0.4793	0.4663	0.5317	0.5175	0.5742	0.6389	0.5815	0.5735	0.6102	0.6508	
D8	0.4774	0.4684	0.5276	0.5145	0.5765	0.6318	0.5789	0.5742	0.6060	0.6478	0.5137
D9	0.4874	0.4631	0.5322	0.5151	0.5850	0.6391	0.5801	0.5766	0.6063	0.6425	0.5121

Table A-5. The results of the measurement of the densities (grams per cubic centimeter). The sample positions are given in Figure 2

Sample position	Block number									
	1	3	4	5	6	7	8	9	10	11	12
A1	2.099	2.117	2.028	2.085	2.055	2.032	2.061	2.029	2.040	2.036	2.104
A2	2.105	2.113	2.029	2.092	2.056	2.034	2.058	2.027	2.038	2.033	2.102
A3	2.102	2.109	2.028	2.092	2.056	2.037	2.058	2.028	2.037	2.034	2.044
A4	2.097	2.112	2.028	2.089	2.055	2.032	2.053	2.028	2.035	2.034	2.102
B1	2.101	2.115	2.027	2.090	2.059	2.032	2.057	2.015	2.037	2.032	2.094
B2	2.099	2.115	2.031	2.089	2.054	2.036	2.058	2.023	2.035	2.034	2.101
B3	2.095	2.113	2.031	2.091	2.057	2.038	2.059	2.023	2.037	2.032	2.101
B4	2.095	2.109	2.030	2.090	2.057	2.037	2.053	2.023	2.029	2.032	2.092
C1	2.091	2.114	2.026	2.087	2.054	2.037	2.054	2.019	2.035	2.032	2.097
C2	2.098	2.117	2.028	2.090	2.058	2.036	2.057		2.037	2.035	2.099
C3	2.091	2.114	2.029	2.094	2.054	2.039	2.057	2.022	2.036	2.033	2.099
C4	2.097	2.114	2.023	2.091	2.055	2.038	2.059	2.021	2.031	2.036	2.092
D1	2.078	2.113	2.029	2.087	2.050	2.033	2.055	2.010	2.035	2.032	2.099
D2	2.100	2.109	2.026	2.085	2.052	2.034	2.052	2.018	2.026	2.028	2.101
D3	2.096	2.111	2.031	2.090	2.052	2.037	2.058	2.012	2.025	2.030	2.095
D4	2.092	2.118	2.024	2.087	2.051	2.032	2.054	2.023	2.019	2.027	2.096
D5	2.096	2.106	2.028	2.090	2.050	2.034	2.056	2.017	2.031	2.037	2.097
D6	2.105	2.110	2.019	2.084	2.039	2.036	2.053	2.021	2.033	2.024	2.094
D7	2.098	2.109	2.021	2.091	2.057	2.034	2.057	2.020	2.028	2.029	
D8	2.099	2.108	2.027	2.091	2.061	2.038	2.061	2.018	2.031	2.032	2.097
D9	2.082	2.112	2.024	2.092	2.054	2.033	2.061	2.016	2.031	2.034	2.101

Table A-6. The results of the measurement of the tensile strength in (MPa). The sample positions are given in Figure 2. A spurious value is given in parenthesis; it is not used in the statistical analysis

Sample position	Block number										
	1	3	4	5	6	7	8	9	10	11	12
A1	(6.44)	1.75	1.68	2.24	2.80	1.43	1.98	1.99		1.28	2.92
A2	1.21	2.20	1.35	2.20	2.89	1.29	2.07	1.98	1.24	1.80	2.55
A3		1.76	1.78	2.99	2.47	1.57	1.82	1.77	2.48	1.87	2.66
A4	1.27	2.28	2.04		2.29	1.24	2.04	2.00	2.16	1.88	1.74
B1	1.13	2.89	1.26	2.24	2.17	1.55	2.06	1.70	1.83	1.64	2.21
B2	2.46	2.21		2.01		1.47	2.05	1.96	1.16	1.92	2.16
B3		2.37	1.07	2.19	2.39	1.37	1.91	1.88	1.76	2.09	2.31
B4	1.27	2.03	1.69	2.47	2.09	1.69	2.06	1.74	1.86	2.24	2.81
C1	1.72	2.95	2.31	2.42	2.71	1.28	2.20	1.83	1.54	1.81	2.76
C2		2.15	1.29	2.61	1.97	1.88	2.03	1.70	1.92	1.71	2.40
C3	1.72	1.72	1.36	2.18	2.04	1.98	1.88	1.93	1.56	2.21	2.20
C4	2.23		1.93	2.43	2.37	2.06	1.98	1.96	1.75	1.89	2.12
D1	1.56		1.32	3.28	2.80	1.62	2.22	1.60	1.87	1.82	2.48
D2	1.87	2.43	1.19	2.30	2.85	1.66	2.08	1.70	1.75	1.29	2.72
D3		2.16	1.37	2.15	2.61		2.32	1.97	1.54	1.82	0.62
D4	1.76	2.27	1.29	2.62	1.94	1.49	1.94	1.94	1.62	1.44	2.32
D5	1.69	2.52	1.19	2.35	2.82	1.53	1.60	2.13	1.64	1.64	2.20
D6	2.89	2.32	1.18	3.10	2.94	1.65	2.33	2.59	2.11	1.51	2.64
D7	1.91			3.30	2.61	1.84	3.50	2.01	2.25	1.94	2.63
D8	2.02		1.31	3.14	2.60	1.71	2.85	2.39	1.81	1.77	2.77
D9	2.63	2.17	1.34	2.97	2.72	1.84	2.68	2.23	2.18	1.84	2.45

Table A-7. The results of the measurement of the strain at failure (in percent). The sample positions are given in Figure 2

Sample position	Block number										
	1	3	4	5	6	7	8	9	10	11	12
A1	0.74	0.82	1.33	0.59	0.55	0.61	0.80	0.59		0.69	0.71
A2	0.78	1.61	0.79	0.57	0.70	0.44	0.60	0.62	0.54	1.07	0.56
A3		0.56	1.12	0.61	0.76	0.61	0.81	0.55	0.78	0.68	0.72
A4	0.95	0.96	1.17		0.74	0.60	0.63	0.72	0.75	0.69	0.54
B1	1.06	0.89	0.88	0.67	0.62	0.75	0.65	0.55	0.57	1.07	0.76
B2	1.02	0.70		0.59		0.66	0.67	0.56	0.45	0.75	0.83
B3		0.94	0.89	0.52	0.73	0.61	0.60	0.60	0.74	0.92	0.81
B4	0.75	0.69	0.87	0.60	0.63	1.94	0.62	0.60	0.58	0.73	0.82
C1	1.09	1.34	0.63	0.70	0.67	1.10	0.69	0.70	0.87	0.91	0.68
C2		1.18	0.69	0.93	0.71	0.68	0.65	0.53	0.65	0.91	0.64
C3	0.70	0.61	0.89	0.61	0.36	0.85	0.69	0.65	0.87	0.78	0.73
C4	0.78		0.84	0.65	0.44	0.81	0.92	0.38	0.58	0.69	0.77
D1	0.80		0.87	0.80	0.68	0.74	0.74	0.54	0.59	0.65	0.48
D2	0.95	1.69	1.02	0.71	0.72	0.83	0.70	0.78	0.61	0.72	0.76
D3		0.94	1.02	0.56	0.95		0.79	0.95	0.67	0.71	0.29
D4	0.79	1.95	0.92	0.73	0.57	0.66	0.63	0.86	0.64	0.66	0.44
D5	1.02	1.34	0.91	0.71	0.77	0.81	0.65	0.79	0.62	0.59	0.78
D6	1.07	1.10	0.88	0.84	0.79	0.79	0.83	0.60	0.68	0.87	0.90
D7	1.33			0.72	0.78	0.75	0.73	0.72	0.71	0.73	0.77
D8	1.04		1.13	0.68	0.73	0.46	0.66	1.05	0.69	0.74	0.72
D9	1.05	0.87	1.09	0.64	1.07	0.65	0.81	0.90	0.70	0.63	0.66