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CARACTERIZACIÓN HIDROGEOQUÍMICA DE EMPLAZAMIENTOS

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# GMT: Material Tests on Specimens of Kunigel VI Bentonite/Sand (20/80) Mixture

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INDEX

# 1. Introduction/ Rationale

## 1.1 Scope

After the GMT Co-ordination Meeting, held on Wednesday, June 30, 1999, at the Nagra's Office, CIEMAT presented a proposal for a test procedure to be applied for the high pressure gas permeability experiment. The proposal was reviewed by UPC (Prof. E. Alonso) and NAGRA, which prepared a modification to CIEMAT's Test Plan, dated September 15, 1 999.

The final set-up and experimental procedures for the permeability tests were defined after reviewing and suggestions of the authors of the initial NAGRA's proposal (September 15, 1999) and further discussion with UPC (Prof. E. Alonso).

## 1.2 Primary objectives of the lab experiments

The experiments have been designed to enhance the understanding of gas migration processes through the Kunigel/sand mixture for a given range of well-defined experimental conditions. The boundary and initial conditions (e.g. initial water saturation, confining pressure) should match those of the mock-up and in-situ experiments as closely as possible.

The aim has been to investigate a range of flow regimes with different dominating processes by gradually increasing the inlet gas pressure and monitoring flow rates and pressure.

## 1.3 Contents

This report describes the mass flow measurement system set-up and the experimental procedure, the data obtained by CIEMAT for the GMT up to December 1999on the characterisation test of gas migration in a sand/clay mixture under different pressures and the set of calibration data sheets of the instrumentation.

The basic physical characterisations of the clay and mixture have also been performed and presented in a previous report including the first results of the gas permeability tests at low injection pressures.

# 2. Preliminary characterisation

## 2.1 Materials: clay and sand

The tested clay is a Japanese bentonite of reference Kunigeru V1, received in CIEMAT on December 1998, supplied by Obayashi (Tokyo, Japan).

Instituto Eduardo Torroja supplied the sand from Segovia. It has a quartz content higher than 98 %. Several calibrated sizes of sand were provided and the appropriated quantities have been mixed to obtain the size distribution proposed by the Japanese team. The proportion of each size is shown in Table I and the granulometric curve obtained is shown in Figure 1. Sand mixtures of 160 g were prepared separately to avoid size segregation.

The samples were prepared by mixing the appropriate quantity of sand and clay to obtain 200 g of sand/clay mixture of 80/20 proportion in a dry weight basis. Then distilled water was added and mixed to obtain the required saturation state. The samples were prepared by static uniaxial compaction. The temptative dimensions were diameter 3.81 and height 7.63 cm. Latex membranes protected the compacted samples.

At the end of the compaction, the final dimensions of the specimen obtained were checked, and after testing, its water content was determined by oven drying.

The dry density is defined as the relationship between the dry mass of solid and the volume of the sample The water content is calculated on a weight basis as defined in soil mechanics: the ratio of weight of water to weight of solids within a sample or, equivalently, the ratio of the mass of water to the mass of solid (Chen, W.F.: "The Civil Engineering Handbook", CRC Press, 1995). The mass of solid is the sample weight after 24 hours oven drying at 110ºC.

Table I: Granulometric distribution of the quartz sand

| **Size (mm)** | 2.5 | 2.0 | 1.5 | 1.18 | 0.6 | 0.3 | 0.15 | 0.074 |
|---|---|---|---|---|---|---|---|---|
| **Passing (%)** | 98 | 90 | 80 | 70 | 38 | 14 | 4 | 1 |

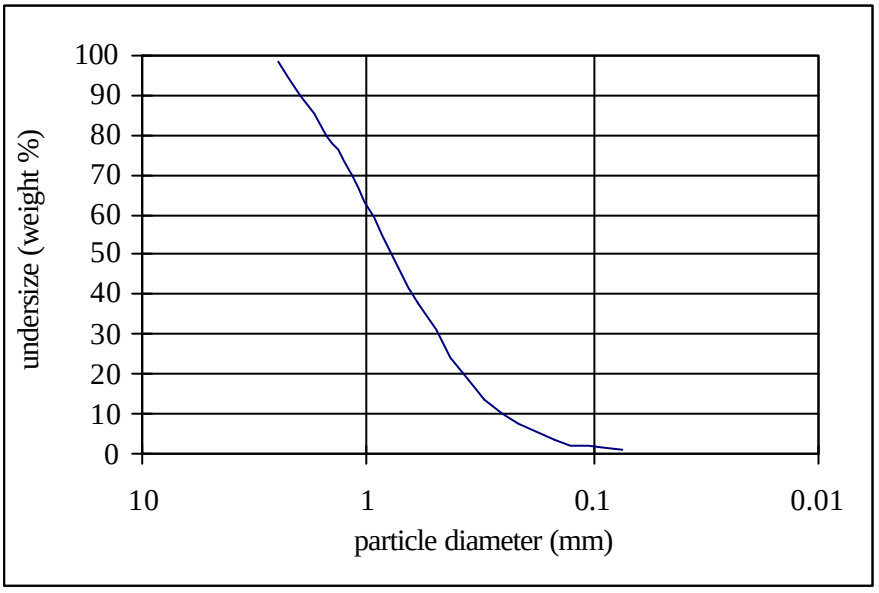



Figure 1: Particle size distribution of the quartz sand

## 2.2 Laboratory tests

Some tests were performed to identify the main clay characteristics. The results are shown in the following section. The rest of the tests were done on sand/clay mixtures.

### 2.2.1 Identification test: clay

*Water content*

Clay water content (7.4 %) was determined as defined above, as it came from Japan. Clay was kept in a closed plastic bag to preserve this initial water content constant. This initial water

content has no repercussion on the subsequent results, as water is added to the sand/bentonite mixture in the appropriate quantity to obtain the desired water content.

The specific weight of Kunigeru clay is 2.64, determined at CIEMAT laboratories.

*Atterberg limits*

The Atterberg limits have been determined according to the standards UNE 103-103 and UNE 103-104 (corresponding to ASTM D 4318). The sample was prepared 48 hours before the determination. For the liquid limit the Casagrande's method has been followed. The Kunigeru values are $W_L$=369%, $W_P$=43% and $I_P$=326%.

2.2.2 Gas permeability: low saturation mixture (Sr=70%)

Gas permeability has been measured at low pressure on some of the specimens prepared according to the procedure described above with low saturation (Sr=70%). The cylindrical sample is covered by two latex membranes with silicone paste between both, and porous stones on top and bottom.

It is placed in a triaxial cell where a confining pressure of 1.2 MPa is applied to the cell chamber, to assure the perfect contact between the sample and the membrane and avoid the gas flow through this interface.

The bottom of the sample is connected to a hermetic deposit of known volume in which nitrogen gas has been previously injected to a pressure slightly higher than the atmospheric one. The upper outlet of the cell is open to the atmosphere.

This equipment works as a variable head permeameter. During the test, the air in the deposit is allowed to flow through the sample, while the pressure decrease in the deposit is monitored. The test must be performed at constant temperature.

The gas permeability is calculated according to the following expression (Yoshimi & Osterberg 1963):

$$k = 2.3 \times \frac{V \times h \times m_a}{A \times \left( P_{atm} + P_0 / 4 \right)} \times \frac{-\mathrm{Log}_{10}\left( P(t) / P_0 \right)}{t - t_0}$$

where k is the intrinsic permeability ($m^2$), V is the volume of the deposit ($m^3$), h is the height of the sample (m), A is the section of the sample ($m^2$), $\mu_a$ is the air dynamic viscosity ($N \cdot s/m^2$), $P_{atm}$ is the atmospheric pressure ($N/m^2$), $P_0$ is the excess pressure in the deposit over the atmospheric one for time $t_0$ (s) and P(t) is the excess over the atmospheric pressure for time t.

The volume of the deposit is $2.21 \cdot 10^{-2}$ $m^3$. All the tests are performed with nitrogen gas, whose dynamic viscosity has been taken as $1.79 \cdot 10^{-5}$ Pa·s. The pressure in the deposit, at the beginning of the tests, is around 1.03 bar.

Taking into account the dynamic viscosity of nitrogen, and assuming a gas density of 0.04 mol/l (CRC Handbook), the following relationship between the intrinsic permeability measured with nitrogen gas (k, $m^2$) and the relative permeability ($K_r$, m/s) can be established:

$$K_r = \frac{r \times g}{m} \times k = 6.2 \cdot 10^5 \times k$$

Every specimen has been run twice. The average results obtained up to now are shown in the Table 2. The intrinsic permeability value obtained must be a function both of the dry density and of the water content; the higher degrees of saturation, the lower permeability values.

Table 2: Gas permeability tests performed with quartz/clay mixtures

| Clay | $\rho_d$ ($g/cm^3$) | w (%) | $S_r$ (%) | k ($m^2$) | K (m/s) |
|---|---|---|---|---|---|
| Kunigeru | 1.88 | 10.8 | 70 | 1.8E-14 | 1.1E-08 |

2.2.3 Hydraulic tests: saturated mixture

Hydraulic tests were performed on saturated samples. A confining pressure (1.2 MPa) was applied to the external surface of the membranes while water was injected from the bottom of the sample at 0.6 MPa pressure. Water flow rates were measured and saturation is assumed once a steady state flow is reached.

Water level flow was measured from the differences in water levels vs time, and permeability was calculated from the formula for Darcy's linear flow of an incompressible liquid phase under steady state conditions.

The hydraulic conductivity was measured on a constant head permeameter that supplies a head of 1000 cm. The samples were prepared according to the method given above.

The sample, protected by the latex membranes, was placed into a triaxial cell, confined on top and bottom by porous stones. The cell was pressurised to 7.5 bar to assure the perfect contact between the membrane and the sample, to avoid water flow through this interface.

The sample was saturated by top and bottom at a pressure of 6 bar during, at least, 24 hours. Then, the pressure at the bottom of the sample was increased in about 1000 cm, resulting in a hydraulic gradient of about 125. The hydraulic conductivity was calculated by applying Darcy´s law once the flow has stabilised.

At the end of the test, the dimensions of the specimens were measured with a calliper and the water content of the sample was determined by oven drying.

To avoid the density variation caused by the swelling/collapse of the sample during saturation, another testing method was checked. The specimens are compacted and introduced in a stainless steel jacket that confines the sample, with porous stones at top and bottom.

The saturation of the clay in the permeability cell is assured by injecting water to obtain a steady state water flow. Afterwards, the injection pressure at the bottom of the specimen is increased, and the water outflow is automatically measured as a function of time. The hydraulic conductivity is calculated by applying Darcy's law. The values obtained ranges about $10^{-11}$ m/s at a 1.8 dry density.

## 3. Preliminary testing

Some preliminary test were perform with the non-modified equipment, to establish the actual conditions we could expect during testing of samples, both saturated and unsaturated. From these experiments, we can extract the following conclusions:

- The injection pressure increments produce transient peaks in the inlet flow that does not correspond to the outlet flow.
- When the injection pressure values get closer to the confining pressure, maintained constant, it produces the separation of the membrane characterised by a gas flow of the same value than the maximum flow of the pressure controller. The pressure difference is about 0.5 bar.
- Decreasing of the injection pressure produces the gas flow to disappear.
- The first indication of gas flow appears after 225 hours, with the same tendency in the following two pressure steps.
- As part of this previous test, the pressure injection was increased during the last phase by pressure steps of 1 bar, without major variations in flow. The injection pressure decrease causes the immediate decreasing of flow, while confining pressure is maintained.

## 4. Experimental Set-up and Procedure

### 4.1 Specimen preparation & size

Two specimens were tested at two different degrees of initial saturation: 90 and 100%, respectively; they are standard triaxial samples compacted with the sand-bentonite mixture at the required water content. Samples are 3.81 cm in diameter and 7.68 cm in length.

In the saturated sample, a 6 bar back pressure was applied to the sample to achieve a steady state water flow, in order to assure a complete saturation; then the sample was installed in the gas transport apparatus.

Sand, bentonite and water were weighed to ensure that specimens conform to fixed dry density and a fixed water content (target saturation). The specimens were manufactured by uniaxial compaction of those materials in a mould.

The specimens were extracted from the mould by a light, mechanically applied pressure. The specimen surface is covered with vacuum grease in order to finish it softly and prevent the formation of preferential pathways.

The sample was placed between two end-caps, each one with a ceramic porous disc completely dried, and jacketed in two latex sheaths, one thin-walled and another thick-walled, to exclude confining fluid and prevent diffusion losses of gas. By this way, we ensure gas flows through the sample with no peripheral pathways. The space between the membranes was also filled with vacuum grease. The elastic properties of the latex and the combination of sizes guarantee interference fit between the sample and the sheath.

The sample set was introduced in a modified commercial triaxial cell to perform the gas permeability and the hydraulic conductivity tests. The tests are not performed under real triaxial conditions (Figure 2).

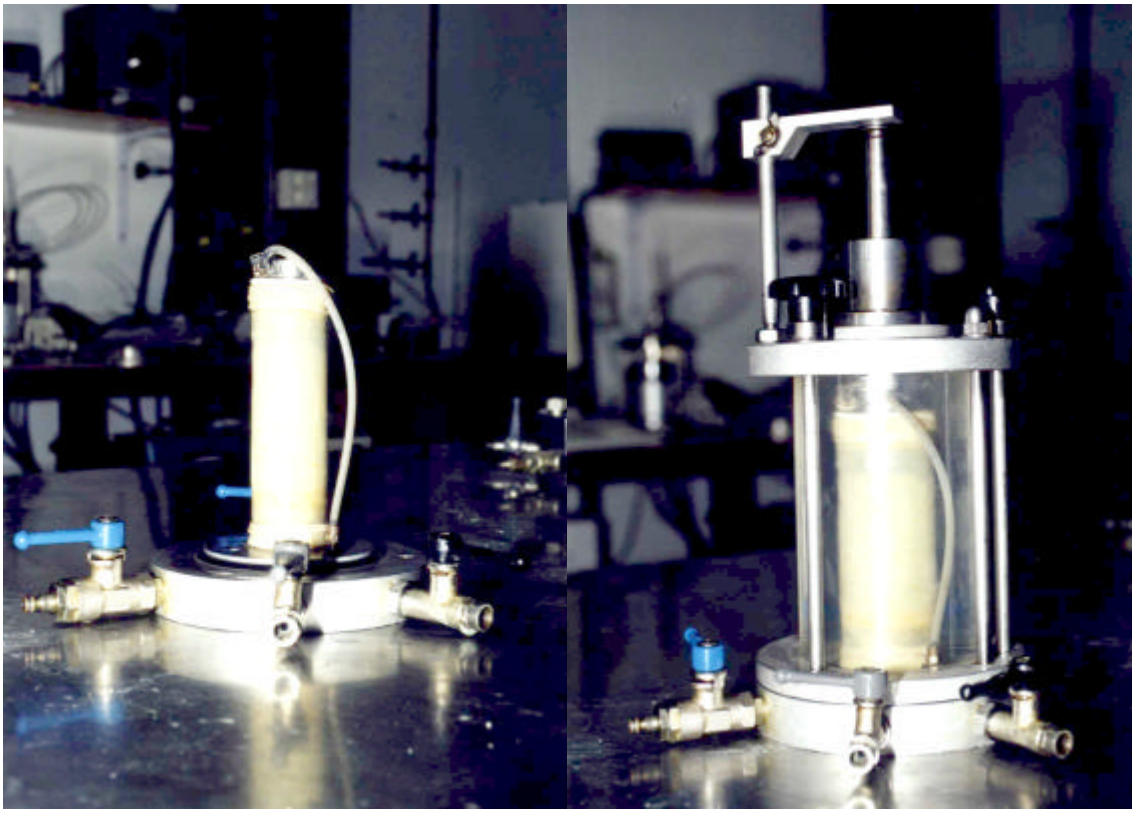


Figure 2: Installation of the sample in the triaxial cell.

### 4.2 Experimental set-up description

The experimental set-up consists of a modified transparent triaxial cell connected to the fittings of the measurement system (Figure 3). The equipment works like a constant head permeameter, with the possibility to change the head value and measure directly the gas flow value. The system applies the pressures to the sample and registers flow and pressures from the measurement devices. In and outflow gas rates, up and downstream pressure, and the confining pressure are monitored.

A high-pressure cylinder (300 cm3) acts as gas buffer, upstream the sample, to give the required gas flow in case the sample breaks. A high-pressure gas drier is located downstream the sample to prevent wrong measurements from the outlet flowmeters, caused by moisture. Additionally, a second cell with a rigid stainless steel jacket has been used for measuring, under low confining pressure (swelling pressure major component), the transport of gas.

Figure 3: Measuring system: general view.

The injected fluid flow rates were measured using three pairs of HITECH mass flowmeters with different measurement flow range: 0.2-10, 2-100 and 20-1000 STP cm3/min. It means that the minimum and maximum values measured without uncertainty in the system is around 0.003 and 16.7 $cm^3/s$, respectively.

The injection pressure of the upstream fluid and the confining pressure were controlled using HITECH forward pressure controllers. The pressure of the downstream fluid was not controlled (atmospheric pressure), but measured with 100-mbarg DRUCK transmitters. Other three DRUCK transmitters, 40 bar a, monitor the injection pressure, the outgoing pressure and the confining pressure.

The Data Acquisition System (DAS) that records and controls the whole set of parameters includes the hardware (acquisition boards, the sensor conditioning and the personal computer interface) and the application software.

A complete description is documented in ANNEX 1 and ANNEX 2.

### 4.3 Test of the gas tightness of the equipment

Prior to start the tests, the inlet system is tested for leaks: different pressure steps were imposed on the injection lines while the system is closed. The system works with continuous gas supply and does not need to monitor small pressure drops. These values are documented through several phases of the tests. In addition, all fitted connections have been tested with a leak spray at the highest pressure of the system (2 MPa).

### 4.4 Experimental set-up improvement

The considered improvements of the apparatus are related to incremental pressure steps, water saturation of the injection gas, water expelled out the sample, confining pressure vs rigid jacket, and use of $H_2S$ as tracer gas. This needed some extra time.

Other improvements of the apparatus (e.g. reduction of the downstream chamber volume) were made to refine the low flow measurements.

#### 4.4.1 Incremental pressure steps scheme

Modification of the injection pressure-steps to the new scheme has been incorporated to the application software without major problems.

#### 4.4.2 Injected gas and wetting-drying processes in the samples

Due to the high saturation of the sample, the injection gas (dried $N_2$) is water saturated to inhibit the sample to dry. The gas bubbles through a saturation vessel, partially filled with water, located between the outlet gas flow meter and the upstream side of the sample.

The water level in the vessel cannot be monitored, due to the volume of gas bubbles.

#### 4.4.3 Water expelled out of the sample

To the volume of water expelled out of the sample by changes in the water level, a device was added to the sample outlet. Since the gas outlet is connected to atmosphere, it was not considered necessary to set under backpressure the measuring system connected to the downstream end of the sample.

The main problem observed is the presence of gas bubbles in the line with continuous variation of the water level, even with complete displacement of water in the line to the maximum value of the system (around 100 mbar g). This makes impossible the measurement of the water level.

#### 4.4.4 Confining pressure

Kunigel/sand mixture swelling pressure is estimated to range from 1 to 3 bar. A preliminary experiment with a saturated sample and a confining pressure of 6 bar (initial saturation >98%) was performed. After more than 100 hours with pressure injections higher than 4 bar (maximum value 5.5 bar), no clear indications of gas flow through the sample were observed.

Increasing the gas injection pressure above the confining pressure would give a “balloon effect” in the elastic rubber membrane. This was avoided by using a rigid cell. However, the formation of preferential pathways to gas flow along the cell wall has to be considered.

#### 4.4.5 $H_2S$-gas as path-flow tracer

$H_2S$-gas is not compatible with the o-rings installed in the gas flow meters. The gas corrosive properties force to major modifications in the whole system (gas flow meters, rubber membrane, etc) with an additional time delay.

Moreover, by bubbling the gas through the water an important quantity of gas will be lost by dissolution until an equilibrated saturated liquid is reached (which will be oxidised to sulphate), without possibility of control.

As a temptative solution, a test has been performed in an alternative way. A deposit with a mixture of $N_2/SH_2$ (1%) at the last test injection pressure with gas flow is connected to the inlet of the triaxial test. The set is placed in an evacuation chamber. The gas mixture must flow through the sample and SH2 reacts with the lead acetate within the sand/bentonite mixture almost saturate.

## 4.5 Test procedure

Minor modifications were made to CIEMAT's proposal that includes multi-step constant pressure tests, with short pressure steps (1 hour) and small pressure increments. Increasing gas pressure steps (0.15 bar) were applied on the sample until gas flow was detected at the outlet of the sample. The maximal gas pressure was estimated to be in the range of 20 bar (comparable to the expected gas pressure in the in-situ experiment). All pressure values are absolute.

The selected test procedure emphasises the characterisation of an injection pressure that is much lower than the confining pressure. The time for one entire test run was estimated to last approximately 30 days.

This option was performed with the improvements of the experimental device described above. The procedure was slightly modified in comparison to the one proposed by NAGRA

### 4.5.1 Phase 1: Initial increasing of the injection pressure

Prescribed pressure increases are applied until gas flow is detected at the sample inlet:

- Start injecting gas with an initial pressure of 1.15 bar a. Confining pressure is 6 bar g.
- If no gas flow is detected at the inlet after about 1 hour, increase the injection pressure by 0.15 bar. This step may be repeated up to get a value of injection pressure close to initial confining pressure.

### 4.5.2 Phase 2: Maintaining of the injection pressure

Prescribed pressure step at the end of phase 1 is continued (max. 2 working days) until gas flow is detected at the sample outlet:

- Continue the measurement of gas flow at the sample inlet.
- Observe the gas flow at the outlet.
- Observe the water level at the outlet of the saturated sample.

### 4.5.3 Phase 3: High increasing of the injection pressure

If no gas flow and water level changes are detectable at the outlet after 2 working days of phase 2, increase the injection pressure by 2 bar every day. Accordingly, confining pressure must be also increased in the same rate.

In total, 7 working days are required to increase the pressure up to the value of 20 bar absolute, which is the maximal pressure expected to be used in the experiment.

#### 4.5.4 Phase 4: Steady state gas flow

Injection pressure is constant; gas flow is observed at sample inlet and outlet:

- Continue the injection of gas at a constant pressure until a constant trend is identified in the observed flow rates (max. 1 working day).
- Add $SH_2$ into the inflow to aid the visualisation of the flow process.

#### 4.5.5 Phase 5:

No gas is injected; observation of recovery:

- Close the valve at the inlet port.
- Monitor the recovery to atmospheric pressure at the inlet port (max. 1 working day).

The samples must be visually inspected after the completion of the experiments. No major emphasis is made on the determination of the gas flow paths.

After the completion of the test, measurement of the specimen state is conducted. The specimen is sliced and a profile of water content along the gas path determined by standard laboratory techniques.

The measurements of porosity by resin impregnation on small sliced pieces are not considered.

## 5. Results

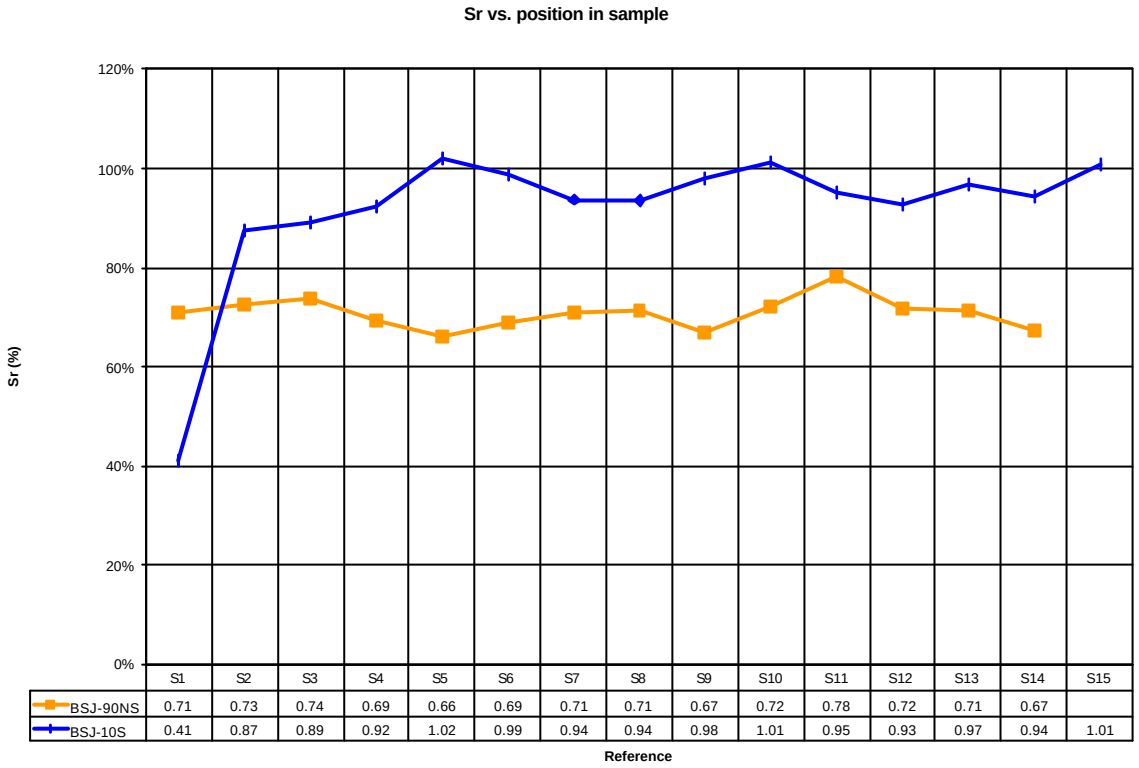
No interpretation of the data in terms of conceptual models or hydraulic parameters is provided. Due the amount of data points, filtering has been applied to the data set to plot the flow-pressure/time curves, but not factor correction. These plots are presented here.

The flow-meter value must be corrected with calibration factors, as a function of the used gas, the temperature and the pressure of the system, by a HITECH's software application. The correction factors are includes in the ANNEX 1.

Table 3: Resume of the performed tests.

| Reference | Test duration (hours) | Confining pressure (bar a) | 1st gas flow indication (bar a/bar a) | 1st cycle B.P./T.P. (bar a/bar a) | 2nd cycle B.P./T.P. (bar a/bar a) | 3rd cycle B.P./T.P. (bar a/bar a) | Dd ($g/cm^3$) | Sr i (%) | Sr f (%) |
|---|---|---|---|---|---|---|---|---|---|
| BSJ-90NS | 90 | 7 | 3.5 | 4.3 / 3.3 | 2.6 / 2.2 | 2.4 / 2.2 | 1.84 | 0.9 | 0.71 |
| BSJ-10S | 160 | 7 | 3.3 | 4.5 / 2.2 | 2.3 / 2.2 | 2.2 / 1.8 | 1.82 | 1.0 | 0.92 |
| BSJ-10S2 | 96 | N/A | 1.4 | 1.8 / 1.4 | 1.6 / 1.6 | 1.6 / 1.6 | 1.79 | 1.0 | 0.96 |
| B.P.: Breakthrough Pressure T.P.: Threshold Pressure Dd: Dry density Sr: Saturation index i,f: initial, final | | | | | | | | | |

These results present data from three final tests (Table 3 and Figure 4): one unsaturated (BSJ-90NS) and two saturated (with elastic membrane, BSJ-10S, and with rigid jacket, BSJ-10S2). Minimal gas-outflow to consider gas breakthrough without uncertainty is 0.2 STP $cm^3$/min. All pressure units are bar absolute (bar a).




| | S1 | S2 | S3 | S4 | S5 | S6 | S7 | S8 | S9 | S10 | S11 | S12 | S13 | S14 | S15 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| BSJ-90NS | 0.71 | 0.73 | 0.74 | 0.69 | 0.66 | 0.69 | 0.71 | 0.71 | 0.67 | 0.72 | 0.78 | 0.72 | 0.71 | 0.67 | |
| BSJ-10S | 0.41 | 0.87 | 0.89 | 0.92 | 1.02 | 0.99 | 0.94 | 0.94 | 0.98 | 1.01 | 0.95 | 0.93 | 0.97 | 0.94 | 1.01 |

Figure 4: Measured saturation vs. position in sample (S1: inlet).

### 5.1.1 Sample at 0.9 saturation

The results in sample at 0.9 saturation are presented in Figure 5. The main aspects are:

- Duration of test is about 90 hours and includes three rounds on the sample.
- Confining pressure on the elastic membrane is about 7.
- In the first round, gas-outflow indication is observed at 3.5 pressure, while gas breakthrough pressure is 4.3, and gas out-flow blocking pressure is about 3.3.
- These values decrease in the two following test rounds to similar values for each category: gas breakthrough pressure between 2.6 and 2.45; blocking pressure about 2.2. They are close to the expected value of swelling pressure in the saturated mixture.
- $N_2/SH_2$-gas mixture flow was tried with this sample.
- Some drying was caused in sample caused by handling to slice it. It is considered that this drying process did not produce the observed phenomenon. The final average water content of the sample was about 0.71.

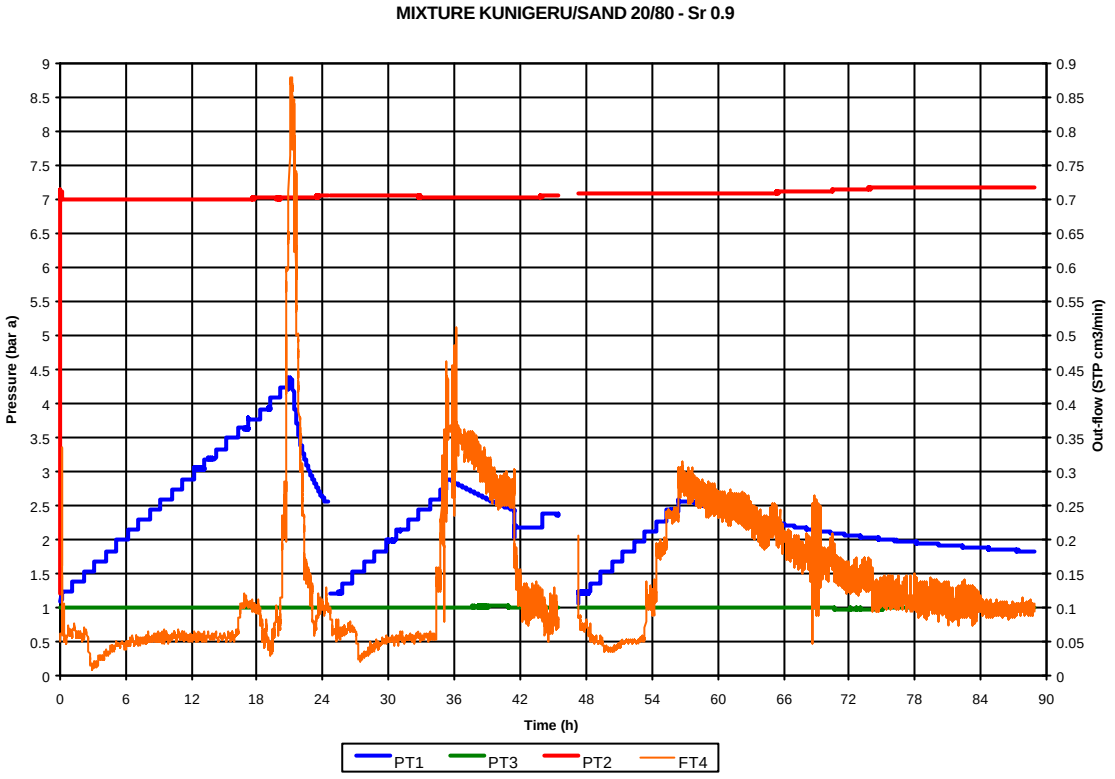



Figure 5: Pressure / Out-flow versus time for 0.9 saturation sample

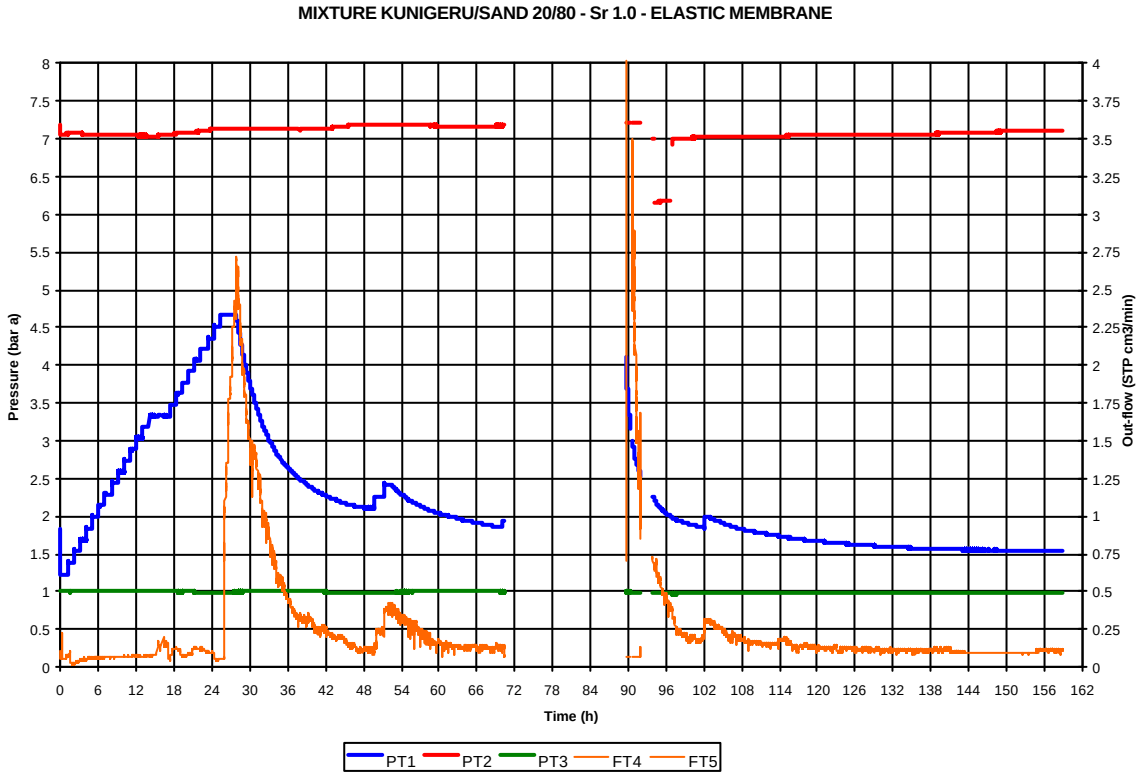



Figure 6: Pressure / Out-flow versus time for saturated sample within elastic membrane

### 5.1.2 Sample at complete saturation confined into an elastic membrane

The results in confined sample at full saturation, into an elastic membrane, are presented in Figure 6. The main aspects are:

- Duration of test is about 160 hours and includes three rounds on the sample. A power supply failure affected the test during 20 hours.
- Confining pressure on the elastic membrane is about 7.
- In the first round, gas-outflow indication is observed at 3.3 pressure, while gas breakthrough pressure is 4.5, and gas out-flow blocking pressure is about 2.2.
- These values decrease in the two following test rounds to similar values for each category: gas breakthrough pressure about 2.2; blocking pressure between 2.2 and 1.8. They remain similar to the values from previous sample but are closer to the expected value of swelling pressure in the saturated mixture, as could be expected.
- Due to the small amount of gas through the sample, it is considered that drying does not produce the phenomenon. The final average water content of the sample was about 0.92.

### 5.1.3 Sample at complete saturation confined into a rigid jacket

The results in confined sample at full saturation, into a rigid jacket, are presented in Figure 7. The main aspects are:

- Duration of test is about 96 hours and includes three rounds on the sample.
- Confining pressure is not applicable here. Swelling pressure of mixture seals the sample.
- In the first round, gas-outflow indication is observed at 1.4 pressure, while gas breakthrough pressure is 1.85, and gas out-flow blocking pressure is about 1.6.
- These values decrease in the two following test rounds to similar values for both categories: gas breakthrough pressure and blocking pressure about 1.65. They remain similar but lower than the values from previous sample, and more homogeneous respect to the expected value of swelling pressure in the saturated mixture, as could be expected.
- Expelled water volume is strongly related to the gas outflow but not good volume measurement has been possible, due to the full fill of the installed capillary.
- Due to the small amount of gas through the sample, it is considered that drying does not produce the phenomenon. The final average water content of the sample was about 0.96.

### 5.1.4 H2S-gas as path-flow tracer

One test has been made to see the gas flow pathways in a sample at 0.9 saturation. The pictures show the progress of the $N_2/SH_2$-gas mixture flow within the sample sections (Figure 9) and the $PbS_2$ profile at the surface of the sample (Figure 10 and Figure 8). The slides are dried to know their water content and plot the water content profile.

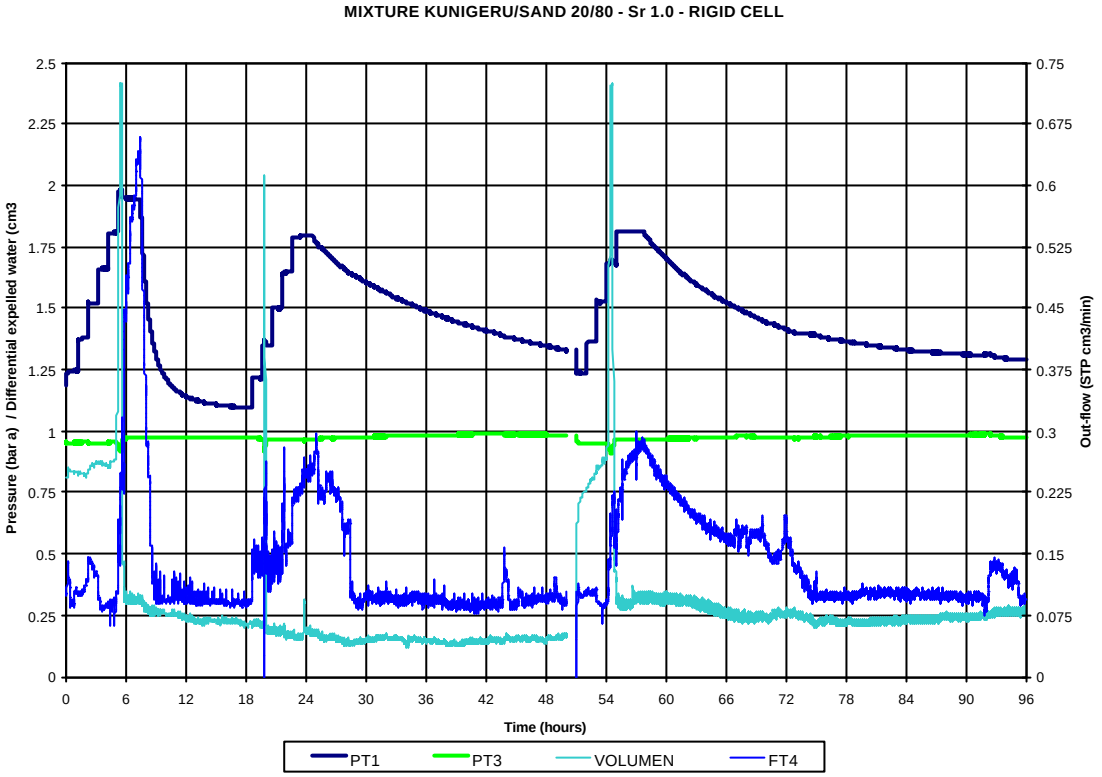



Figure 7: Pressure / Out-flow versus time for saturated sample within rigid jacket

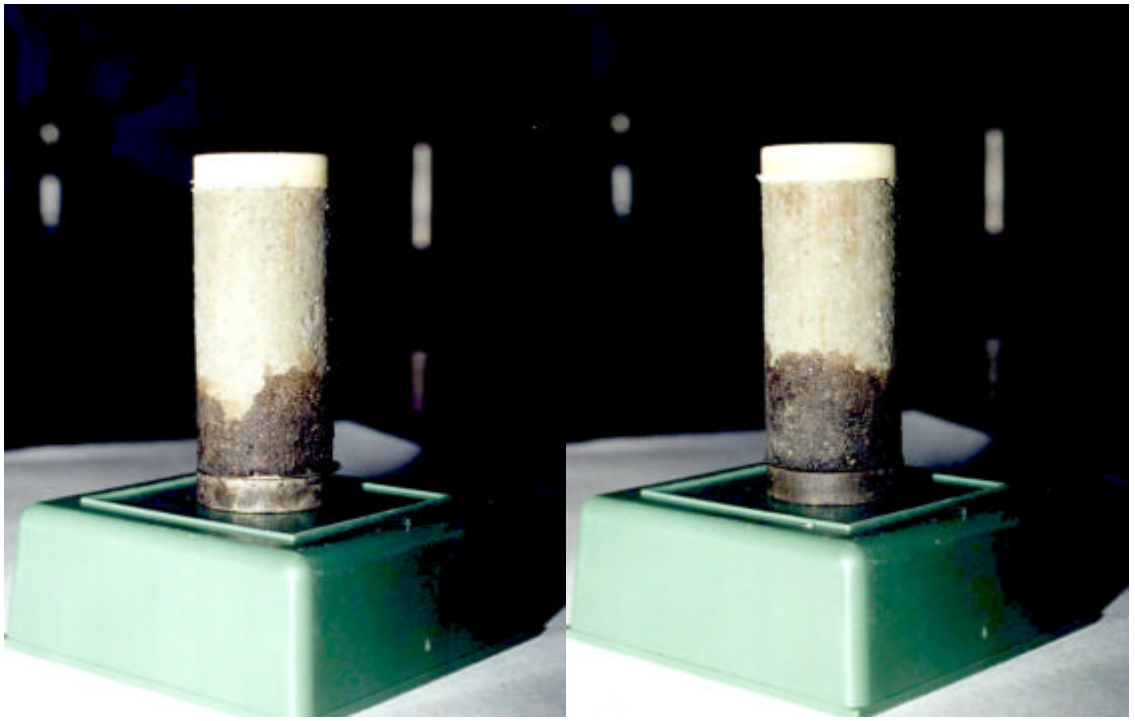


Figure 8: Sample exposed to $SH_2$ flow: views at 180º before cutting.

Figure 9: Sample exposed to $SH_2$ flow: Details of the sections.

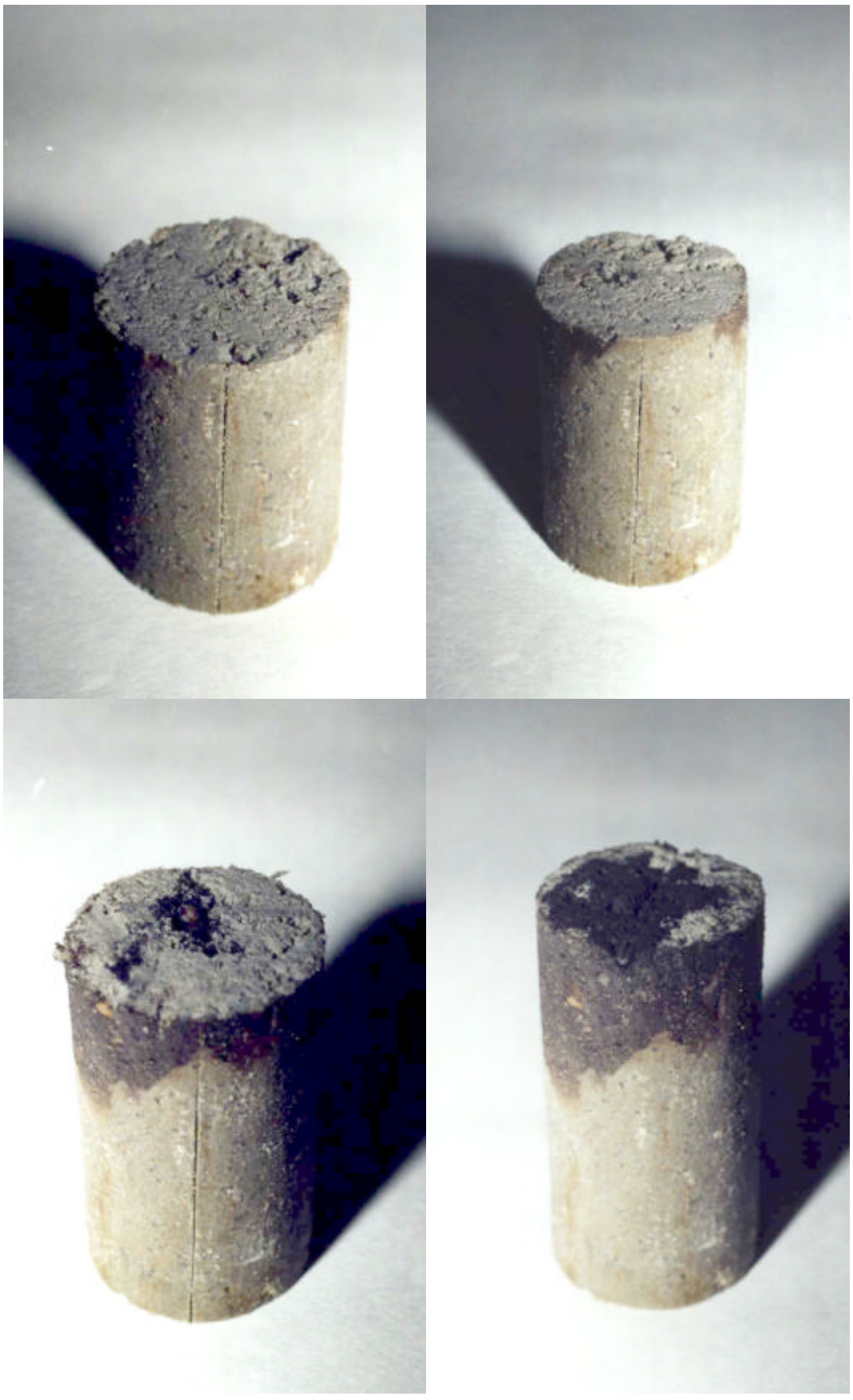


Figure 10: Sample exposed to $SH_2$ flow: Details of the cutting sequence.

No more tests have been made because it considers that we really observe the diffusion phenomenon of $SH_2$ in the water of the sample, not the actual gas pathways within it.

## 6. Summary and conclusions

### 6.1.1 Materials

- **Sand size distribution was reconstructed** to obtain the needed sand/clay mixture. **Distilled water** has been added to achieve the required water content to give the target saturation state of the sample after compaction.
- The **uniaxial static compaction method** has been used to manufacture the samples.
- **Sand/clay mixtures of proportion 80/20 and 1.8 dry density** have been tested to analyse some aspects related to its gas migration behaviour.

### 6.1.2 Hydraulic characterisation

- Similar **hydraulic conductivity** values have been obtained with all the mixtures tested, and all of them **are in the range measured by Hokari et al. (1997).**

### 6.1.3 Gas permeability at low saturation:< 0.7

- The **gas permeability of the specimens is a function both of the dry density and of the water content**, decreasing significantly as the degree of saturation increases.

### 6.1.4 Gas injection test at high saturation: 0.9 and full saturation

- Some tests have been performed prior to the testing of the two selected saturation states, what has shown that **high gas-flow could dry the sample**, increasing in turn the gas flow and drying the sample further. As well, **breakthrough pressures seem to be related to the confining pressure for the saturated samples**.
- The **tests performed under confining pressure**, both in the saturated and in the quasi-saturated (0.9) samples, **show similar initial gas-breakthrough values, higher than the expected swelling pressure**. The following test rounds give rise to lower gas-breakthrough pressure values, in the order of the swelling pressure. On the contrary, the **unconfined sample shows an initial gas-breakthrough value similar to the swelling pressure**, that decreases slightly in the subsequent rounds.
- The "threshold pressure" below which no gas-flow occurs after breakthrough is lower in the saturated samples, especially in the unconfined one. This could be explained by the fact that **the only limitation to gas-flow in the unconfined sample is its own swelling pressure**. This threshold value is somewhat higher in the first cycle of the test performed in the unsaturated sample.
- **The behaviour of the unconfined saturated sample is more homogeneous** throughout the test, than that of the confined samples. This indicates **that the confining pressure could modify the reaction of the mixture**.

- Due to the small amount of gas through the sample, it is considered that drying does not produce all the previous phenomena.

6.1.5 $SH_2$-gas tracer

- The phenomenon observed in the test carried out with lead acetate added to the mixture, is in fact the **$SH_2$ diffusion in the water of the sample** and **not the actual gas pathways** within it.

## 7. REFERENCES

Chen, W.F.: The Civil Engineering Handbook. CRC Press, 1995.

Yoshimi, Y.& Osterberg, J.O. (1963): Compression of partially saturated cohesive soils. J. Soil Mechanics and Foundations Division. ASCE 89, SM4:1-24.

Hokari, T.; Okihara, M.; Ishii, T.; and Kojima, K. (1997): Experimental study of self-healing of bentonite/sand mixture and its impact on hydraulic permeability. Mat. Res. Soc. Symp. Proc. Vol 465.

# ANNEX 1: CHARACTERISTICS OF THE MEASUREMENT EQUIPMENT

This annex includes the description of the test cell and the general layout of the measurement equipment with the location of the sensors and the actuators.

**Tests cell:**

The test cell is a modification of a commercial triaxial cell. The cell walls are manufactured from transparent plastic and are capable of withstanding pressures to 2700 kPa. Each cell has four take-off positions drilled in the base; 1 for top drainage/back pressure; 2 for bottom drainage/pore pressure, and 1 for confining pressure.

Four no volume changes valves are connected to the ports and an anvil fits the cell head.

**Tubing, fitting and valves:**

All the SWAGELOK fitting materials and valves are made from stainless steel, SS316. The SANVICK tubing material is SS316 1/8". The WHITEY gas cylinders are from SS304 or SS316. The maximum leakage rate that manufacturer assures at each valve packing is around 0.1 cm3/min at 68 bar g.

**Water/nitrogen separator:**

An OLAER's pressure accumulator (to 330 bar) with an elastic membrane acts as separator between the nitrogen and the water phases of the confining pressure system.

**Gas buffer:**

A WHITEY gas sample cylinder (300 cm3), placed before the sample, eliminates the fluctuations introduced by the pressure controller in the flow measurement. Also, it permits to keep constant the expected flow even in case of pneumatic fracturing.

**High-pressure gas drier:**

An ALTECH high-pressure gas drier (130 bar g, 200 cm3) placed before the outlet flowmeters eliminates the moisture to avoid wrong measurement of flow. The gas drier removes moisture to 22 g $H_2O$ capacity, oil small aliphatics, HCl, $SO_2$, $NO_2$, $H_2S$ y $CO_2$, using a molecular sieve 5 Å.

**Gas Mass flowmeters:**

HI-TEC Gas mass flowmeters operate on a principle of heat transfer by sensing the temperature increment along a heated section of a capillary tube. They are calibrated to the consigned conditions: gas type He, pressure 20 bar a, and temperature 25ºC. The output signal is 4-20 mA. The models and S/N are shown in Table 4.

Application software, such as FLOW CALCULATIONS, enables to calculate accurate conversion factors from the calibration data, not only at 20ºC/1 atm (as shown in the conversion table, App.1, Instruction manual) but also at any temperature/pressure combination). This software has been used to calculate the conversion factors to be applied.

Table 4: Gas mass flowmeters.

| REFERENCE | MODEL | FLOW (cm3/min) | SERIAL No |
|---|---|---|---|
| FT1 | F-110C-HD-33-V | 0.2 - 10 | 9724208 A |
| FT2 | F-111C-HD-33-V | 2 - 100 | 9724208 C |
| FT3 | F-111C-HA-22-V | 20 -1000 | 99203188 C |
| FT4 | F-110C-HD-33-V | 0.2 - 10 | 9724208 B |
| FT5 | F-111C-HD-33-V | 2 - 100 | 9724208 D |
| FT6 | F-111C-HD-22-V | 20 -1000 | 99203188 D |

**Pressure controllers:**

HI-TEC Gas forward pressure controllers are calibrated to the consigned conditions: gas type He and temperature 25ºC, at different pressures and maximum flow capacities. The output and control signals are 0-5 V. The maximum differential pressure is 30 bar. The models and S/N are shown in Table 5.

The OUTPIC0 and OUTPIC1 controllers give the consigned gas injection pressure to the system. The OUTPIC2 controller regulates the internal pressure within the nitrogen/water separator, which water acts as confining medium on the sample membrane.

Table 5: Gas pressure controllers.

| REFERENCE | MODEL | FLOW (cm3/min) | PRESSURE | SERIAL No |
|---|---|---|---|---|
| OUTPIC0 | P-602C-FA-100A | 1000 | 75 bar a | 99203188 A |
| OUTPIC1 | P-602C-FA-100A | 1000 | 25 bar a | 99204464 A |
| OUTPIC2 | P-612C-FA-400A | 100 | 75 bar g | 9724208 E |

**Pressure transmitters:**

Associated with the pressure controllers, DRUCK pressure transmitters, PTX1400 series, have been placed at several points: the inlet port of the triaxial cell (injection pressure), the inlet port of the water/nitrogen separator (confining pressure), the outlet of the system (atmospheric pressure), and the base of the drainage water column (water level or volume drainage).

Transmitters range 40 bar a (0.25% BSL), except that which measures the water column (100 mbar g). The output signal is 4-20 mA. The models and S/N are shown in Table 5.

Table 6: Pressure transmitters.

| REFERENCE | MODEL | PRESSURE | SERIAL No |
|---|---|---|---|
| PT1 | PTX1400 | 40 bar a | PO1964/02 |
| PT2 | PTX1400 | 40 bar a | PO1964/01 |
| PT3 | PTX1400 | 40 bar g | PO1964/04 |
| PT4 | PTX1400 | 100 mbar g | RO2072/07 |

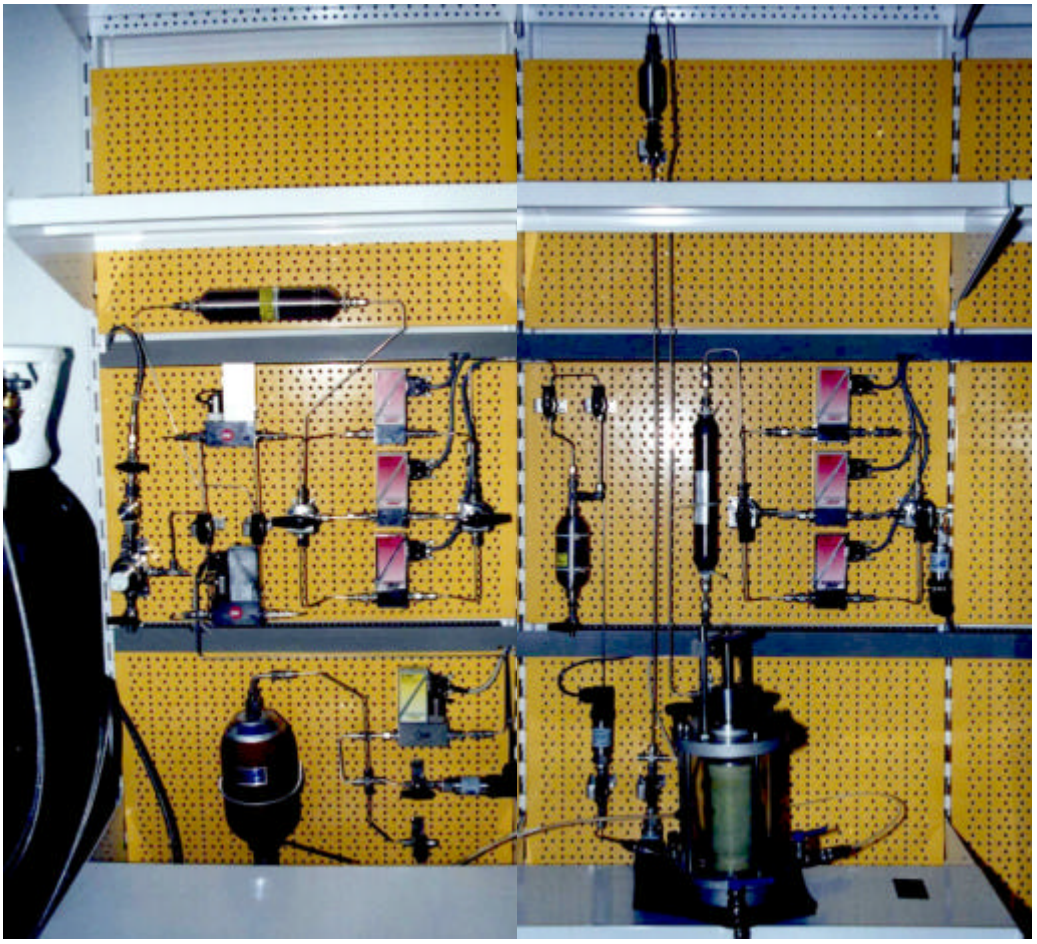


Figure 11: Measuring system: view of the hydro-pneumatic components.

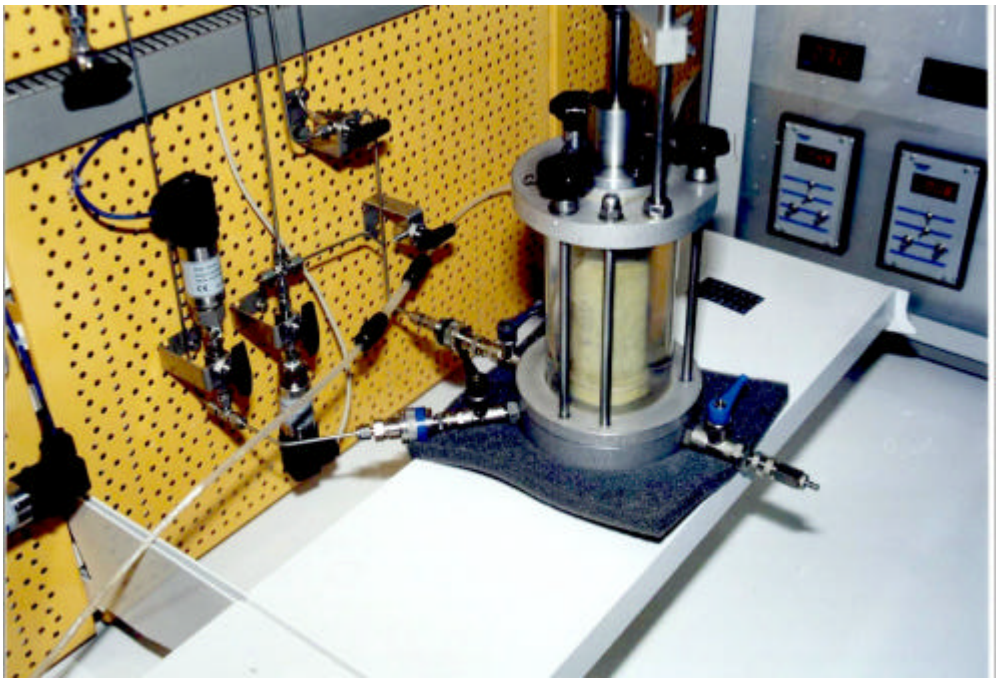


Figure 12: Installation of the sample: detail of the connection to the measuring system.

## ANNEX 2: CHARACTERISTICS OF THE DATA ACQUISITION SYSTEM (DAS)

A DAS records the values from the sensors and actuators in the measurement system.

### Signals:

The DAS registers 10 analogic signals: 6 signals (4-20 mA) from the flowmeters, 3 signals (4-20 mA) from pressure transmitters, and 3 signals (0-5 V) from the pressure controllers. The DAS also manages the pressure controllers by output signals (0-5 V).

### Hardware:

Two NATIONAL INSTRUMENTS's acquisition boards, with their accessories, installed in a personal computer perform the data recording and control. The whole set is composed by:

| | |
|---|---|
| Multifunction I/O board (16/2 ch): | AT-MIO-16E-10 |
| Analog output board (2 ch): | PC-AO-2DC |
| Connector block: | CB-68LP |
| Connector block: | CB-50 |
| Cable (68 pins, 1m): | R6868 |
| Cable (1m): | NB1 |

The set has 16 Singled Ended or 8 differential with programmable gain (12-bit resolution, 100 kS/s) inputs and 4 analogic outputs (12 bits). The free channels are available to future expansions.

A personal computer working under Windows 95, to run the specific software and precision resistances (250 Ω, 0.1%) at the 4-20 mA inputs complete the installed hardware.

### Software:

An specific runtime application developed for this test, with National Instruments' Labview, permits to manage the system in one of two ways: manual or automatic. It controls the data acquisition by user, showing the real time values of the sensors and their plots. Data are registered in ASCII format and then imported to MICROSOFT EXCEL.

In the manual mode, the user can modify the control values on the experiment and name the data file. In the automatic mode, the user can only modify the sampling frequency and name the data file, but he can see the plots of the time evolution. This mode does not permit to change the control parameters.

The following figures (Figure 13 to Figure 15) show some screens of the control application.


Figure 13: Main screen of the data acquisition system

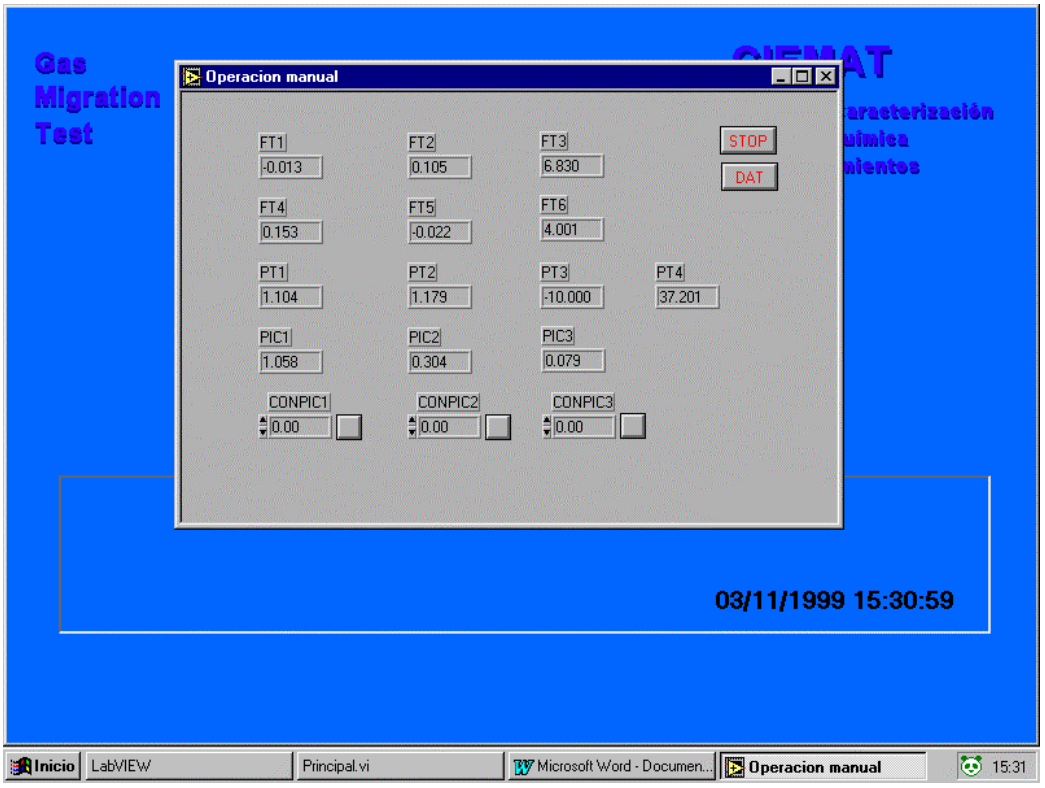



Figure 14: Main screen of the data acquisition system: manual test.

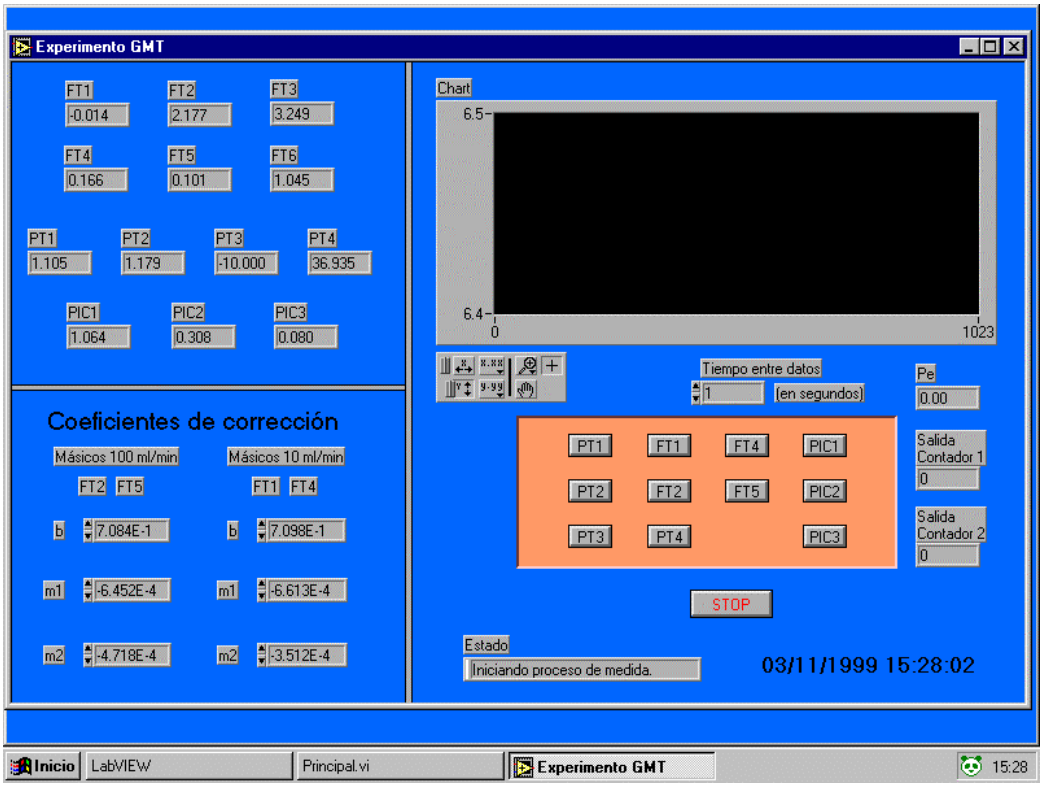



Figure 15: Main screen of the data acquisition system: automatic test