

Shrinkage and Expansion in Oilwell Cements

API TECHNICAL REPORT 10TR2
FIRST EDITION, JULY 1997

REAFFIRMED, SEPTEMBER 2002



AMERICAN PETROLEUM INSTITUTE

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Exploration and Production Department

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FOREWORD

Dimensional change after placement in oil and gas well cements, a phenomenon often referred to as shrinkage, has often been used to explain various problems (e.g., microannulus leading to bad cement bond logs, interzonal communication leading to costly remedial jobs and also lack of a seal to the gas of cement inflatable packers). Attempts have been made to find additives to decrease shrinkage, but it seems that shrinkage (as well as the exothermal reaction) are closely related to the hydration of the cement. The best solution for this shrinkage so far has been the identification of additives that favor the expansion of the cement. However, even if cement expands dimensionally, it still will shrink internally. In this case, the bulk expansion of the cement sample is simply superposed on an inner shrinkage that will affect the porosity of the sample. Shrinkage is not just one property of cement, such as rheology or thickening time that can be defined by one measurement. This is probably why little consensus has been reached today in the industry on this important issue, in spite of a large amount of work devoted to the subject.

In 1994, API proposed to open a work group on shrinkage. This report is an attempt to bring more understanding to this important area of cementing. One of the objectives of the work group was also to propose equipment to investigate shrinkage and expansion in oilwell cements. This report will address the objectives of that task group, which were:

- To develop standardized test procedures to measure expansion/shrinkage.
- To investigate possible impacts on invasion of wellbore fluids (gas, water).
- To standardize definitions of terms for cement hydration.

This report is a summary of all the background information and actual data that were generated to meet the objectives outlined above.

The equipment described in the report is the minimum recommended to measure the parameters in question.

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Suggested revisions are invited and should be submitted to the director of the Exploration and Production Department, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.

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Shrinkage and Expansion in Oilwell Cements

1 Introduction

A flawless cementation of the casing strings is fundamental not only for safety reasons, but also to keep drilling and production costs at a minimum level.

A cement job has to fulfill multiple conditions, but its main function is to prevent fluid and gas flow through the annulus by ensuring an impermeable bond and exhibiting sufficient compressive and shear bond strength.

Various factors can affect the sealing, such as poor mud removal, as well as bad slurry properties and borehole conditions. It is also recognized that the volumetric changes of cement during hydration substantially contribute to these problems. Bulk and inner volume changes produce bonding and permeability changes, respectively.

In this work, volume changes were better understood by measuring them on different slurries. Three different setups that correspond to three different testing conditions were used.

2 References

A list of suggested references may be found in Appendix A.

3 Definitions and Symbols

3.1 DEFINITIONS

For the purposes of this report, the following definitions apply:

3.1.1 cement: when the word is used alone, it refers to the material in any of the states from cement slurry to hard cement.

3.1.2 cement slurry: a mixture of Portland cement, water, and additives at a consistency below 50 Bc.

3.1.3 gel cement: cement slurry between the time at 50 Bc and the time at which the transit time of ultrasonic compressional P waves starts to decrease (UCA equipment).

3.1.4 gel cement under plastic state: a gel cement that can still deform to match the shape of its container.

3.1.5 hard cement: a material where the transit time of ultrasonic compressional P waves has decreased compared to the initial base line.

3.1.6 Portland cement: the powder as received from the supplier.

3.2 LIST OF SYMBOLS

The following symbols are used in this report:

- V_i = initial volume of cement
- V_f = final volume of cement
- T = temperature of the cement

- P = pore pressure inside the cement
- M_t = micrometer measurement after curing
- M_i = initial micrometer measurement
- $\%Lex$ = defined by equation [1], linear expansion measured with the annular ring
- V_i = initial volume of the sleeve
- V_f = final volume of the sleeve
- $\%Bex$ = defined by equation [3], bulk expansion measured with the sleeve
- D_i = 1.65 inches (initial diameter of the sleeve)
- D_f = final diameter of the sleeve
- C_i = initial circumference of the sleeve
- C_f = final circumference of the sleeve
- h = height of the sleeve
- ΔC_{12} = difference between C_{12} final and C_{12} initial
- $\%Bch$ = defined by equation [4], bulk volume change measured with the membrane
- V_p = volume compensated by the piston movement
- V_g = volume of gas entered in the slurry
- V_i = 392.5 cm³ (estimated by filling up the cell with water)
- $\%Ish$ = defined by equation [5], inner shrinkage measured with the CHA

4 Discussion of Shrinkage and Expansion

4.1 CAUSE AND RESULTS OF SHRINKAGE AND EXPANSION

Shrinkage and expansion in cement result from the formation of hydration products having a different density from the compounded density of the reaction components. This can result in:

- a. Change in pore volume.
- b. Change in pore pressure.
- c. Change in sample dimensions.
- d. Change in internal stress.

The change in sample dimensions will be referred to as bulk shrinkage or bulk expansion. The change in pore volume will be referred to as inner shrinkage.

Shrinkage and expansion of cement refer to the result of the measurement of a volume change in cement. This will be expressed in percent by volume. The volume to which all volume changes are related, is the volume of the slurry immediately after mixing and placement in the experimental equipment. The general expression for shrinkage and expansion is:

$$100 \frac{V_f - V_i}{V_i}$$

Where:

V_i = initial volume of slurry.

V_f = final volume of cement.

4.1.1 Some equipment measures the external volume change. Other equipment measures the volume of fluid intake or expulsion during the hydration. Measurement of V_f and V_i must be performed at the same temperature. In the measurement of an external volume change, the external stress applied to the material has to be constant during the experiment. For a correct measurement of fluid intake or expulsion, the pore pressure at the end of the test has to be the same as at the beginning.

- Bulk shrinkage corresponds to an external volume decrease.
- Bulk expansion corresponds to an external volume increase.
- Inner shrinkage corresponds to an intake of fluid.
- Inner expansion corresponds to fluid expelled from the slurry.

In a slurry made of cement and water only, one could observe bulk and inner shrinkage. In a slurry containing aluminum powder, one might notice hydrogen production, which could lead, under appropriate boundary conditions, to an inner expansion. In a slurry containing carbon or magnesium oxide, one could measure a bulk expansion.

The magnitude of these possible effects depends on the cement powder, slurry design, and curing conditions (temperature, pressure).

The slurry's permeability, mechanical properties, and stress applied to the boundaries control the partition between these effects.

If the cement is infinitely permeable, then a volume change in the pores simply requires a net flux of fluid into the sample and, thus, no bulk shrinkage is measured. The inner shrinkage is measured by the amount of fluid entering the cement.

If the cement is impermeable, then a volume change in the pores results in a pore pressure change and, subsequently, a bulk shrinkage or expansion governed by the mechanical properties of the slurry.

If the slurry is contained between fixed, impermeable boundaries, no bulk expansion can occur. Inner shrinkage results first in pore pressure reduction and then in creation of internal tensile stress that could lead either to debonding from the walls or to failures, and eventually both. In this situation, dramatic pore pressure decrease is observed down to the vapor pressure of the water at the temperature where the test is conducted. In turn, expansion creates compressional stresses. Depending on the mechanical properties of the slurry, the additional stress (tensile or compressive) can result in failures.

A slurry is impermeable if either of the following conditions exist:

- The sample is covered in an impermeable container.
- The permeability is sufficiently low or the sample size sufficiently large to prohibit fluid flow from compensating for volume changes on an appropriate timescale.

Most cementing scenarios fall somewhere between these two conditions.

Inner shrinkage always occurs when cement powder reacts with water. This might not be seen if an additive, e.g., aluminum powder, reacts with the slurry to produce gas. The addition of specific additives—e.g., plaster of Paris, calcium oxide, magnesium oxide, or a combination of these—may result in bulk expansion.

In a slurry containing a high C_3A content cement, plaster of Paris first hydrates to form gypsum ($CaSO_4 \cdot 2H_2O$). Gypsum then reacts with tricalcium aluminate ($Ca_3Al_2O_6$) to form a calcium trisulfoaluminate hydrate mineral called ettringite. The reaction occurs at temperatures not exceeding 170°F (77°C). The product of the reaction forms voluminous crystals which will exert stress inside the cement. Calcium oxide and magnesium oxide react to form hydroxides. The kinetics of these reactions are different, therefore, expansion will also depend on the strength of the cement at the time when the products of expansion develop.

If a sample of slurry containing an expansive agent is open to water or gas, initial shrinkage is first compensated for by fluid intake. Eventually, the permeability becomes small enough to isolate the pores and further shrinkage reduces the pore pressure. However, this is quite late into the hydration exotherm and the solid frame has sufficient strength to resist being compressed.

Little or no bulk shrinkage occurs in this situation and compensation for the inner shrinkage maintains pore pressure.

At later stages, when the expansion products are developing, the increase in pore pressure can exceed the applied total stress and bulk expansion of the sample will occur.

However, under impermeable conditions, fluid flow cannot compensate for inner shrinkage and the pore pressure is immediately reduced. Because the cement initially has little compressive strength, the sample collapses and bulk shrinkage is measured. This process continues until the cement has sufficient compressive strength to withstand the applied stress (which is early in the hydration isotherm). Then, additional shrinkage results in a dramatic reduction of the pore pressure.

At later stages of hydration, deposition of expanded products will increase the pore pressure. Thus, more products should be formed before the pore pressure increases above the original applied stress level.

To summarize, if one assumes that the chemistry is the same in all cases, samples in which the influx of fluids compensates for volume reduction before the permeability becomes sufficiently small to isolate the pore space, have no



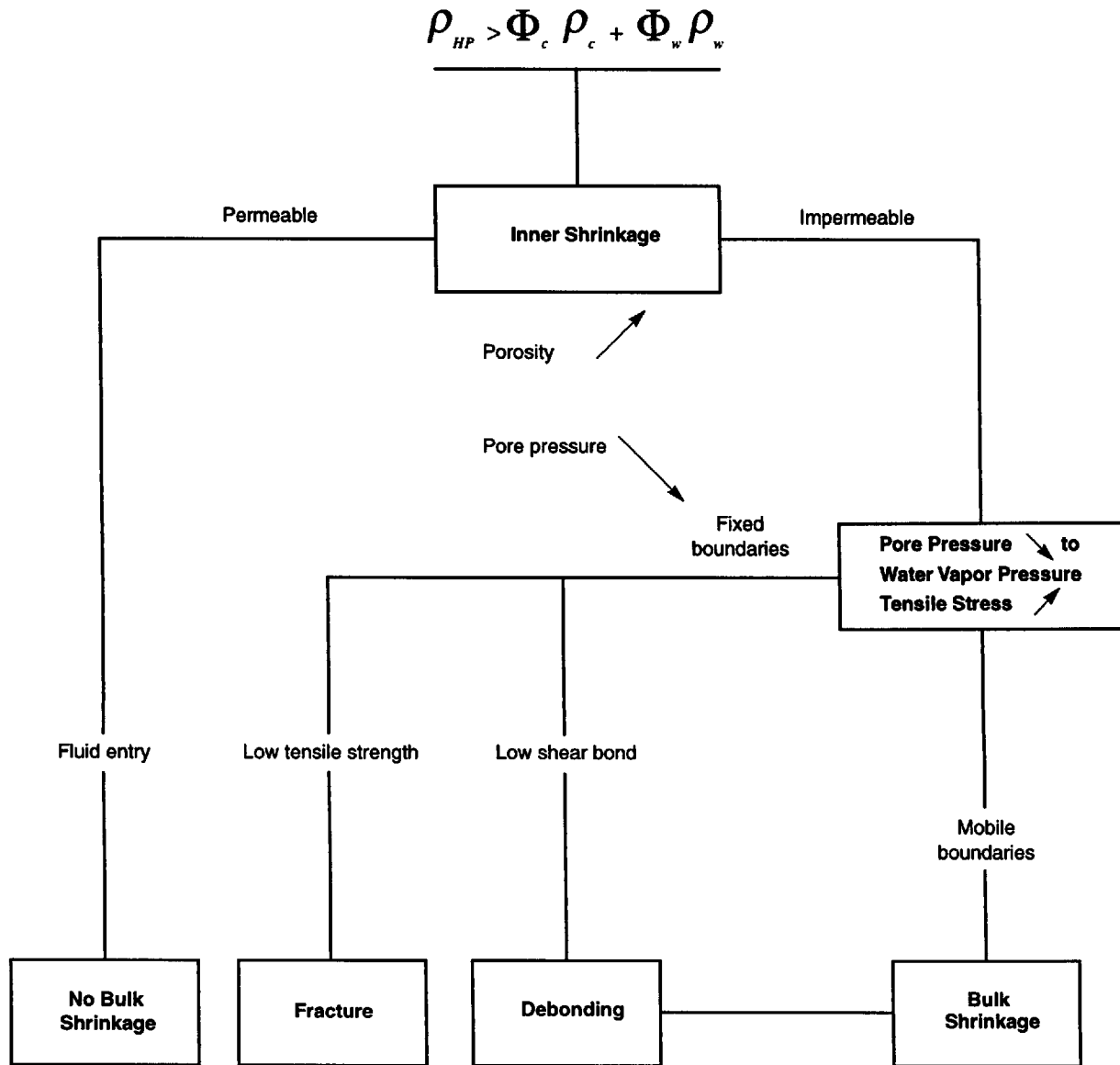


Figure 1—Cement Hydration

room for expanded products. So the samples must either expand or constrain a high pore pressure.

In those samples where shrinkage is not compensated for earlier, room is available for the deposition of the expansion products, and the subsequent rise in pore pressure may be insufficient to cause an expansion.

The relevant timescales for shrinkage, as well as expansion, are functions of permeability and length scale.

In conclusion:

- It is suggested that the words *shrinkage* and *expansion* refer to the volumetric change of cement during hydration.
- It is proposed that these words contain the adjective *bulk* in reference to an external volumetric change, or *inner* in reference to an internal volumetric change.
- The reported value depends on the conditions under which the test is performed. This leads to the procedures outlined in Section 5 that are currently used to perform the measurements.

5 Experimental Procedure

Four pieces of equipment were used to measure the cement volume changes under different boundary conditions:

- The annular ring expansion mold operates under conditions of free access to water entry. This equipment is devised to measure the linear expansion of an annular ring of cement.
- The cylindrical sleeve also operates under conditions of permeability to water. It allows the measurement of the bulk expansion of cement.
- The membrane test allows the measurement of either bulk expansion or bulk shrinkage under impermeable conditions.
- The Cement Hydration Analyzer (CHA) operates with a total stress (286 psi [20 bar] typically) applied to the bottom part of a 8-inch high, 2-inch diameter column of slurry. It allows the measurement of inner shrinkage under permeability-to-gas conditions.

All four pieces of equipment exert some stress on the cement. The expandable ring, the three springs on the cylindrical sleeve, the fixed wall of the CHA cell, and even the membrane resist the bulk expansion of the cement somewhat. Ideally, except for the CHA, this resistance should be minimized. In this report, the resistance of the walls to cement bulk expansion is neglected.

5.1 THE ANNULAR EXPANSION RING: A MEASUREMENT OF LINEAR EXPANSION UNDER CONDITIONS OF FREE ACCESS TO WATER

5.1.1 Apparatus and Procedure

The annular expansion mold (Figure 2) is a device to measure the expansion properties of cement compositions when placed into an annular mold simulating the annulus of a well.

The slurry is poured into the large hole at the outer portion of the tip of the ring mold. The small hole in the top of the mold is for venting air from the mold during filling. The mold is filled until the slurry exits the small hole. On some thicker slurries, the mold may need to be tapped or vibrated to ensure it is completely filled.

Once the slurry is poured, the mold is placed into a thermostated water bath. Slurry is in contact with water during the entire test. Water entry will compensate for any inner shrinkage as long as the cement matrix is permeable. If the cement expands during the hydration period, the outside diameter of the annulus will expand.

This mold can also be placed in a pressurized vessel for curing under higher temperature and pressure.

5.1.2 Expansion Measurement

Before curing, an initial measure is taken with a micrometer for calibration of the annular ring. The micrometer is opened and placed on the smooth, flat surface beside the mold to measure the distance between the outside of the steel balls attached to each side of the split on the expandable ring.

After curing, the sample is cooled to below 170°F. Measurement is taken in the same manner as the initial measurement was done. The distance between the two steel balls is to be measured with a micrometer. This measurement must be performed very quickly to prevent the specimen from sufficiently cooling and causing an erroneous measurement. If other measurements are to be determined, the specimen is placed back into the curing bath or autoclave, and curing continues at the test temperature and pressure conditions.

5.1.3 Calculations

One can only measure linear expansion with this test. This is noted as %Lex. The equation to be used is:

$$\%Lex = (M_t - M_i) \times 10.95 \quad (1)$$

Where:

M_t = micrometer measurement after curing.

M_i = initial micrometer measurement.

Because of the small distance between the measurement balls, no correction is needed for extrapolating from the initial measurement of the mold at room temperature to the measurement at the actual test temperature.

5.2 THE CYLINDRICAL SLEEVE: A MEASUREMENT OF BULK EXPANSION UNDER CONDITIONS OF FREE ACCESS TO WATER

5.2.1 Apparatus and Procedure

The slurry is poured into two cylindrical sleeves placed in standard 2 x 2 x 2-inch brass molds (Figure 3). The sleeves



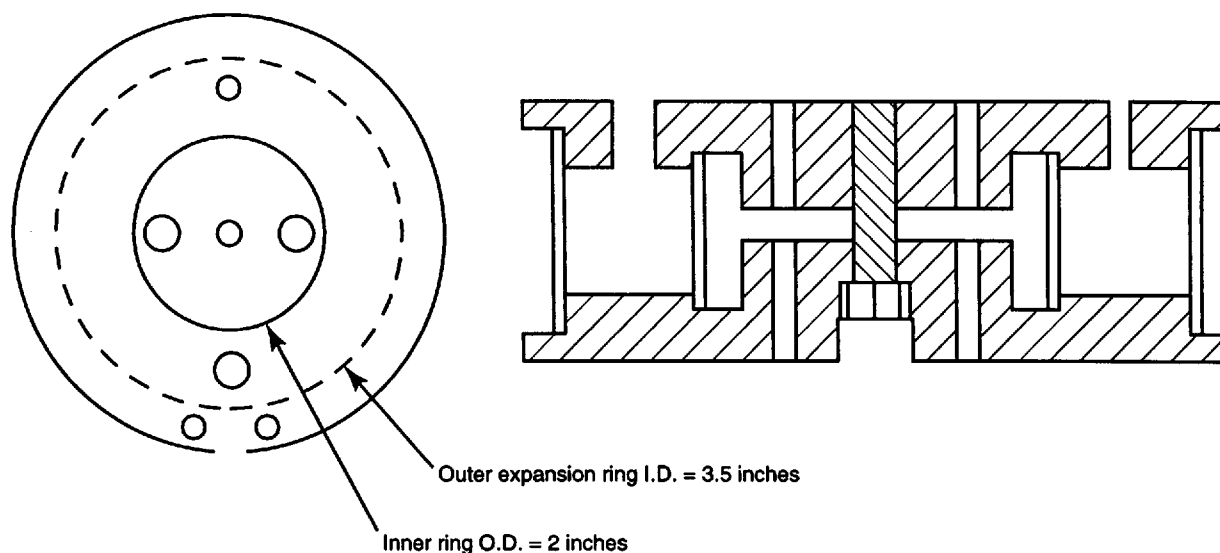


Figure 2—The Annular Expansion Mold

are opened on both sides and have a vertical slit therein. They are kept closed by three springs attached to three sets of pins mounted on the outside. Once the slurry is poured, the molds are closed and placed into a thermostated water bath. The slurry is in contact with water during the entire test. Water entry will compensate for any inner shrinkage as long as the cement matrix is permeable. If the cement expands during the hydration process, the diameter of the molds will increase. These tests can be run at a higher pressure and temperature in any curing chamber.

5.2.2 Expansion Measurement

Before the slurry is introduced, the sleeves are in a closed position and a zero reading of the distance between each set of pins is taken with a micrometer, giving D_i . After 120 hours (the duration of the test), the measurement is repeated, giving D_f .

5.2.3 Calculations

One can only measure bulk expansion with the sleeve test. This will be noted as %Bex.

For each sleeve, an average is made of the differences ($D_f - D_i$) measured on the three springs, giving C_1 and C_2 (two sleeves are used per test). C_1 and C_2 are assimilated to the circumference increase of the sleeves and an average is made over the two sleeves, giving $C_{1,2}$.

The expansion is calculated according to the equation:

$$\%Bex = 100 \frac{V_f - V_i}{V_i} \quad (2)$$

Where:

V_i = initial volume of the sleeve.

V_f = final volume of the sleeve.

$$C_i = \pi D_i$$

$$C_f = \pi D_f = \pi D_i + \Delta C_{1,2}$$

Where:

D_i = 1.65 inches (initial diameter of the sleeve).

D_f = final diameter of the sleeve.

C_i = initial circumference of the sleeve.

C_f = final circumference of the sleeve.

$$V_i = \frac{\pi}{4} D_i^2 h$$

$$V_f = \frac{\pi}{4} \left(D_i + \frac{\Delta C_{1,2}}{\pi} \right)^2 h$$

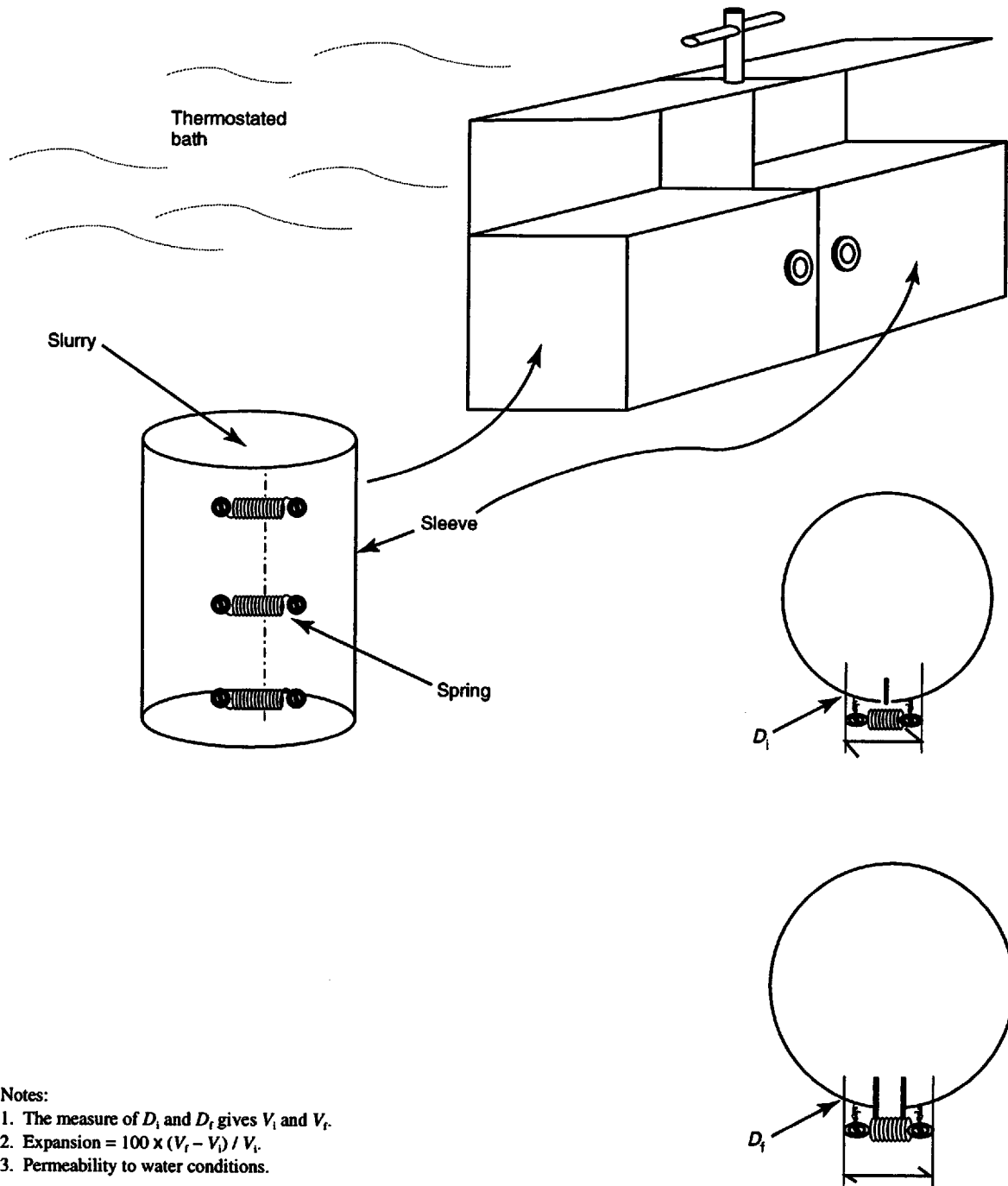
Where:

h = height of the sleeve.

The expansion becomes:

$$\%Bex = 100 \left(1 + \frac{\Delta C_{1,2}}{\pi D_i} \right)^2 - 100 \quad (3)$$

D_i and D_f are measured with a precision of 0.001 inch.



Notes:

1. The measure of D_1 and D_2 gives V_1 and V_2 .
2. Expansion = $100 \times (V_2 - V_1) / V_1$.
3. Permeability to water conditions.

Figure 3—Measurement of Cement Expansion

Several approximations are made:

- The cement expansion along the axis of the sleeve is not considered.
- C_1 and C_2 are chords and not arcs, but because the difference is small, it can be neglected.
- This measure is taken at the pins' level, it could be corrected by the factor $L/(L+l)$ where L is the radius of the sleeve and l the length of the pin.

5.3 THE MEMBRANE: A MEASUREMENT OF BULK SHRINKAGE OR BULK EXPANSION UNDER IMPERMEABLE CONDITIONS

5.3.1 Apparatus and Procedure

The slurry is poured into an impermeable flexible membrane which is sealed by tying a knot at the top (Figure 4). Extreme care is taken so that no air is entrapped in the slurry or at the top of the membrane.

The membrane is then placed in a fine net attached to a balance hook and immersed into a thermostated water bath. The electronic balance (interfaced to a computer) records and plots the weight and the slurry temperature via a thermocouple immersed in the slurry. Data are recorded every 10 minutes.

5.3.2 Shrinkage or Expansion Measurements

The *pseudo weight*, as measured by the balance, is the weight of the membrane containing cement minus the buoyancy force acting on it ($W - B$). B is equal to the volume of the membrane containing cement multiplied by the specific gravity of water (1.00 sgu).

The evolutions of the sample volume and temperature are recorded during the test. An increase of the pseudo weight corresponds to a decrease of the volume.

After 120 hours, the hardened cement is removed from the membrane and weighed. This setup is used to measure bulk shrinkage or bulk expansion.

5.3.3 Calculations

Because the membrane test can result in either a decrease or an increase of the external volume, the volumetric change measured with the membrane is called *percent bulk change* and noted as $\%Bch$. A positive value corresponds to a bulk expansion, while a negative value corresponds to a bulk shrinkage. It is calculated using the following equation:

$$\%Bch = 100 \frac{V_f - V_i}{V_i}$$

$$V_i = W - (W - B_i) \quad (4)$$

$$V_f = W - (W - B_f)$$

V_i and V_f are measured with a precision of 0.1 gram.

5.4 THE CHA: A MEASUREMENT OF INNER SHRINKAGE UNDER FREE ACCESS TO GAS

5.4.1 Experiment Number 1

A cell is filled with liquid slurry and placed in a thermostatic oven (Figure 5). Hydraulic pressure is applied on the slurry until thermal equilibrium is reached between the slurry and the oven. Then the cell is closed. The temperature in the cell remains constant until there is a rapid rise caused by cement hydration. The temperature reaches a maximum and finally decreases, approaching the temperature of the oven.

The shape of the temperature curve depends on the heat produced by the hydration of the cement and on the test conditions. Under purely adiabatic conditions, temperature would increase until hydration was over and then would remain constant. Under purely isothermal conditions, the heat would immediately dissipate and the temperature would stay constant in the cell.

During the experiment, one can also measure the pressure at the top of the cell. This pressure slowly decreases until the temperature starts to increase. Then, pressure sharply decreases to a lower limit. At room temperature, the lowest value for the pressure in the cell is around 7 psi (0.5 bar). If the test is run at 302°F (150°C), the limit is around 70 psi (5 bar). These limits correspond to the vapor pressure of the interstitial water at the temperature of the test.

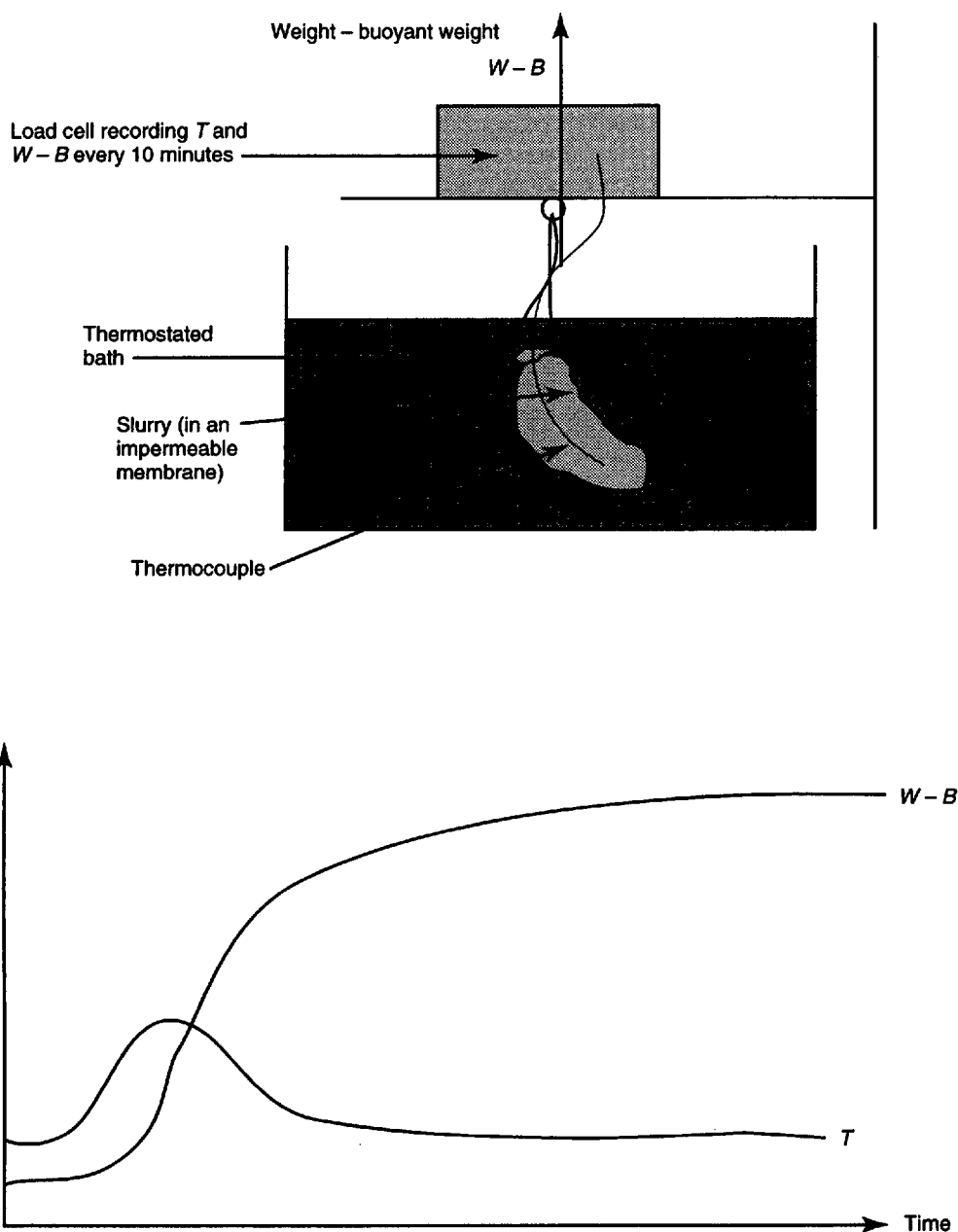
These last observations do not depend on the slurry formulation, the size of the cell, or the initial pressure in the cell. During hydration, the density of the hydration products becomes higher than the density calculated from the cement and water. In an impermeable environment (the cell is closed and the walls cannot deform), inner shrinkage develops. New porosity is created inside the cement, pressure decreases, and the interstitial water will expand to fill the extra porosity. Expansion of the water takes place until the water vaporizes. The lower pressure limit is the vapor pressure of water at the temperature of the test.

5.4.2 Experiment Number 2

A hydraulic piston is used to pressurize the slurry (Figure 6). This piston is located at the bottom of the cell to apply a total stress to the cement as soon as a solid structure starts to develop. If the piston were at the top, the tiny layer of free water (always present at the top of the slurry) would result in the application of a pore pressure, and not in a total stress on the cement matrix.

As gelation of the slurry increases, friction stress builds up at the wall of the cell opposing the movement of the piston. The pressure at the top of the cell decreases and the difference



**Notes:**

1. $B = V$, volume of slurry.
2. The variations of $W - B$ give the variations of V : $V = W - (W - B)$.
3. Shrinkage = $100 \times (V_i - V_f) / V_i$.
4. Impermeability to water conditions.

Figure 4—Measurement of Cement Shrinkage (or Expansion)

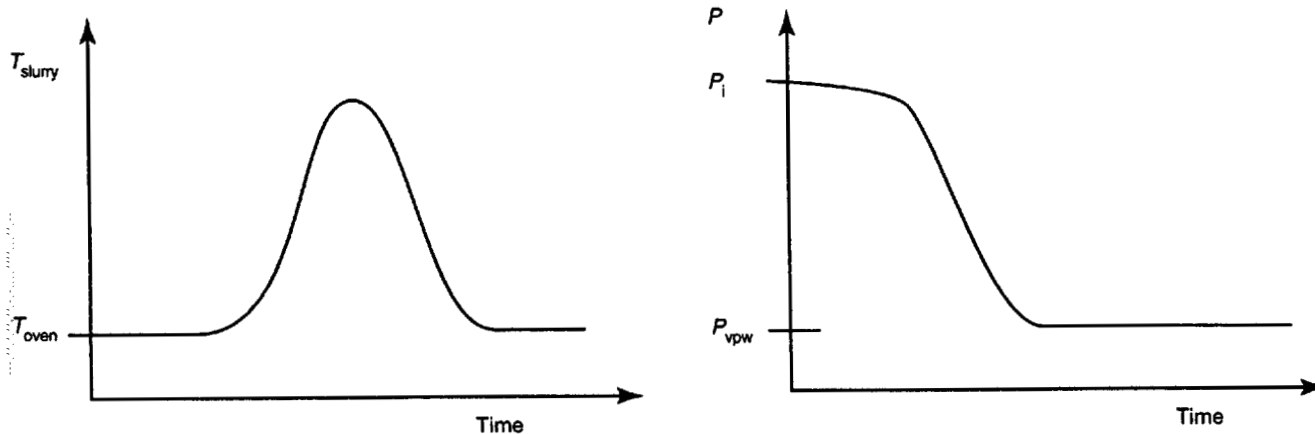
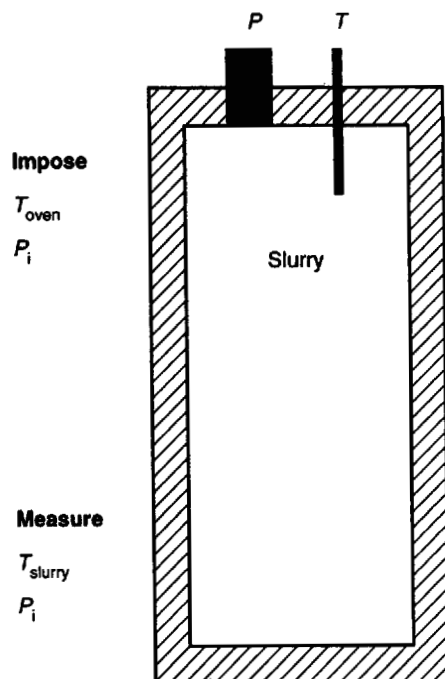


Figure 5—Experiment 1

between applied and top pressure can be calculated using the classical formula:

$$\Delta P = 4SGS \frac{L}{D}$$

Where:

SGS = static gel strength of the cement.

L = length of the cell.

D = diameter of the cell.

It is observed in most cases that the piston stops moving when the temperature of the slurry starts to increase. At that time, 0.5 to 1 percent shrinkage has been compensated for by the movement of the piston. When the piston stops moving, the pressure in the cell sharply decreases toward the vapor pressure of the water as in the previous experiment.

In this experiment, the amount of bulk shrinkage compensated for by the movement of the piston is controlled by the total stress applied to the piston as well as the relation existing between the amount of inner shrinkage and the wall shear stress development.

5.4.3 Experiment Number 3

The bottom of the cell is now connected to a gas source at a constant pressure (Figure 7). As shrinkage develops, gas enters into the cell to fill the space. If pressure stays constant in the cement, the volume of gas which enters is used to calculate the percentage of inner shrinkage. In most cases this amount corresponds to 4 percent of the volume of slurry.

With some slurries, it can be observed that pressure measured at the top of the cell drops and eventually very little gas enters into the slurry. Therefore, these slurries are impermeable to gas under the conditions of the test.

The same type of experiment can be run with water rather than gas. If the slurry is permeable to water, the volume of water entering into the slurry can also be used to calculate the percent of shrinkage. Note that during the course of the experiment, some slurries might be more permeable to gas than to water while others are more permeable to water than to gas.

The amount of fluid entry can be used to calculate inner shrinkage only if, at the end of the test, the final pore pressure measured at the top of the cell is equal to the fluid pressure applied at the bottom of the cell.

5.4.4 Introduction to the CHA

The CHA (Figure 8) was built to perform the previous experiments or any combination of these experiments (e.g. piston as in Number 2 and then gas as in Number 3).

The body of the cell is a section of common 2-inch schedule 80 carbon steel pipe. The ends of the pipe are faced square and smooth for a good seal as the pipe is clamped between

the end caps in a specialized press. The pipe is 7.9 inches (20 centimeters) long with an internal diameter of 1.87 inches (4.75 centimeters).

The total volume of the cell is 392.5 centimeters³, which requires less than one API mixing to fill up.

The top cap holds a pressure transducer (P3, 0 to 50 bar) to measure the pore pressure in the slurry. It also contains a temperature probe (Pt 100) to record the temperature evolution. There is a third hole in the top cap to purge the cell at the beginning of the test and to release any residual pressure at the end of the test.

The bottom cap contains a hydraulic piston to pressurize the slurry. Both caps contain O-rings to provide good seals on the flat ends of the cylindrical cell body.

The piston has a hole in the center to screw in a small pipe 5.12 inches (13 centimeters) long and 0.315 inches (0.8 centimeters) internal diameter. The small pipe is connected to an air operated valve. A porous stone (1.25 centimeters long and 0.8 centimeters outer diameter) is inserted at the bottom of the small pipe to prevent possible contamination of the air valve by the cement.

Because cement cures in both pipes used for the cell body, they are discarded after each test.

A specialized restraining press is used to support the cell. This assembly is designed to accommodate test pressures up to 600 psi (40 bar). The system can operate in the temperature range of 40° to 300°F (5° to 150°C). It is possible to run tests at a lower temperature, 14°F (-10°C), by using glycol instead of water to pressurize the piston. The entire cell should be put in a thermostatically-controlled oven that regulates the temperature during the test.

The control panel operates under ambient conditions. It is equipped with two pressure regulators to adjust the gas and water pressure. A rotary selector switch allows the operator to monitor the gas, water, or pore pressure on a digital display.

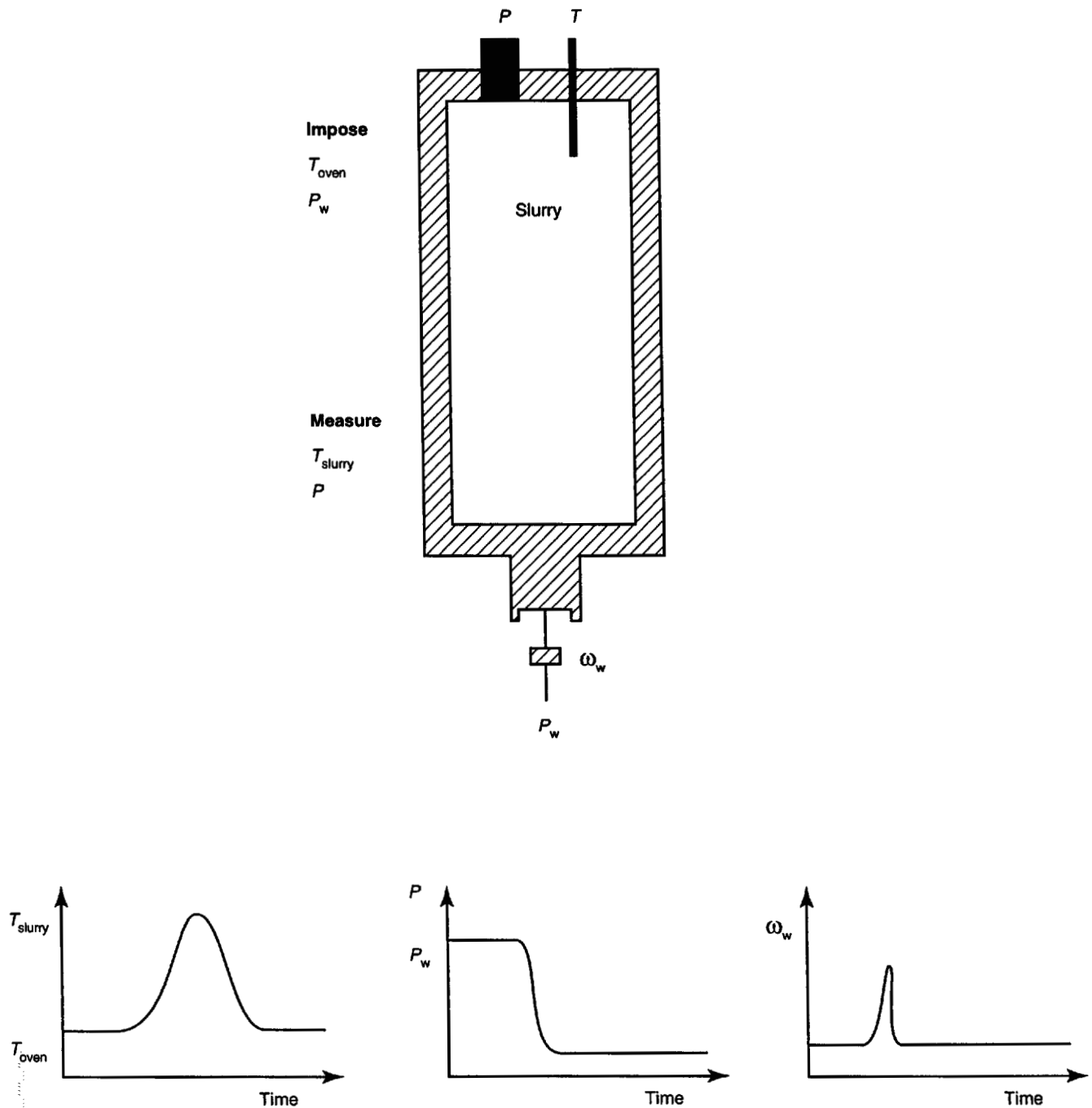
A chart recorder displays and plots the following parameters against time as the test progresses: piston rate, gas rate, temperature, and pressure. The control panel can also be connected to a computer.

5.4.5 Shrinkage Measurements

The shrinkage measurement is possible only if the final pore pressure is equal to the gas pressure, i.e., if the slurry is permeable to gas.

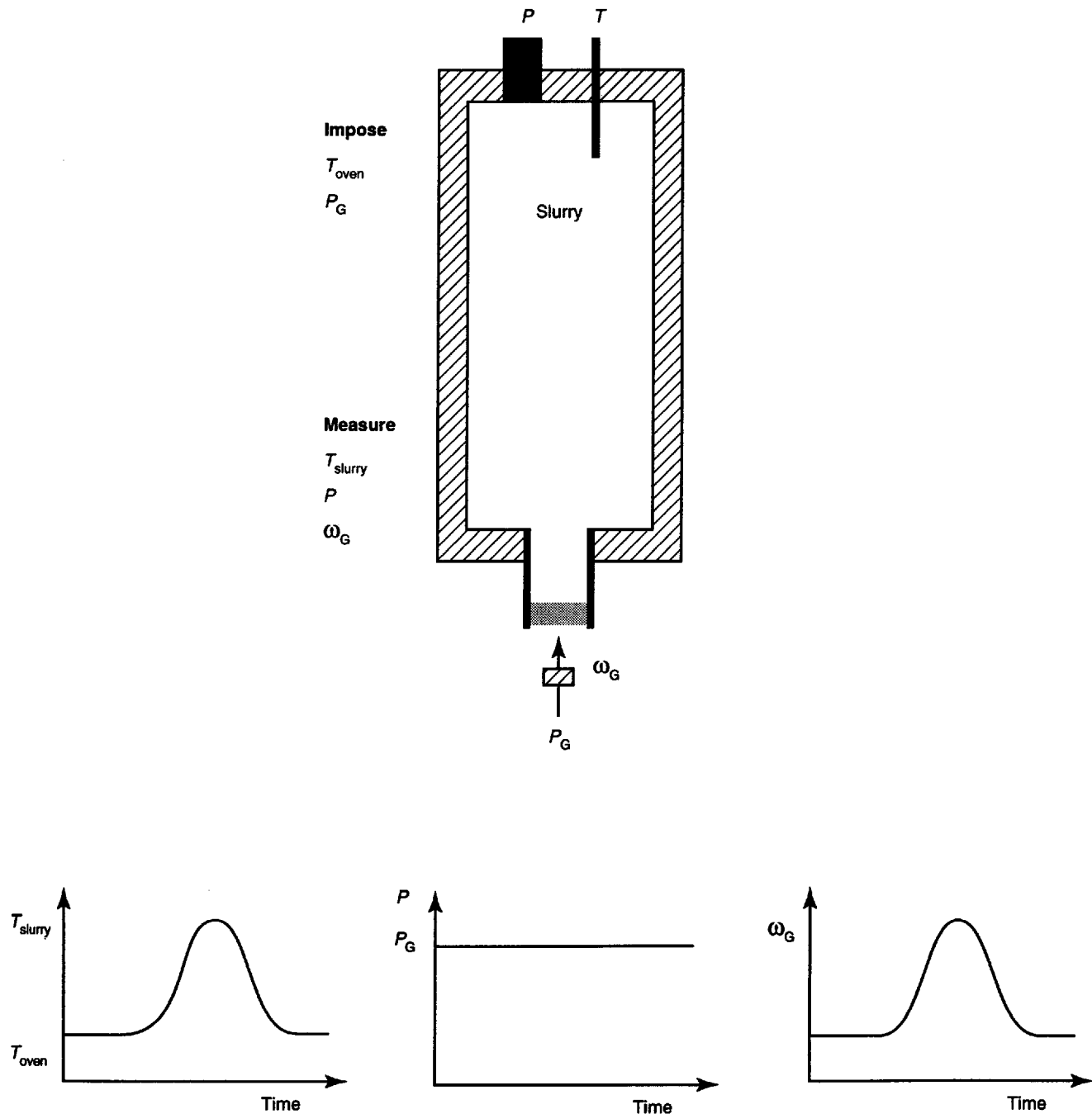
In the gas procedure, the volume of gas that enters the slurry is an estimate of the inner shrinkage. It is equal to the total shrinkage, if there is no debonding from the walls of the cell.

In the piston/gas procedure, the volume compensated for by the piston movement is an estimate of the bulk shrinkage. The volume of gas that enters the slurry is an estimate of the inner shrinkage under these operating conditions. The sum of both values is an estimate of the total shrinkage under these test conditions.



BULK SHRINKAGE

Figure 6—Experiment 2



INNER SHRINKAGE

Figure 7—Experiment 3