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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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 cm^3 (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.

a. Bulk shrinkage corresponds to an external volume decrease.
b. Bulk expansion corresponds to an external volume increase.
c. Inner shrinkage corresponds to an intake of fluid.
d. 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:

a. The sample is covered in an impermeable container.
b. 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 ($CaSO_4$, $2H_2O$). 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 spaces have no

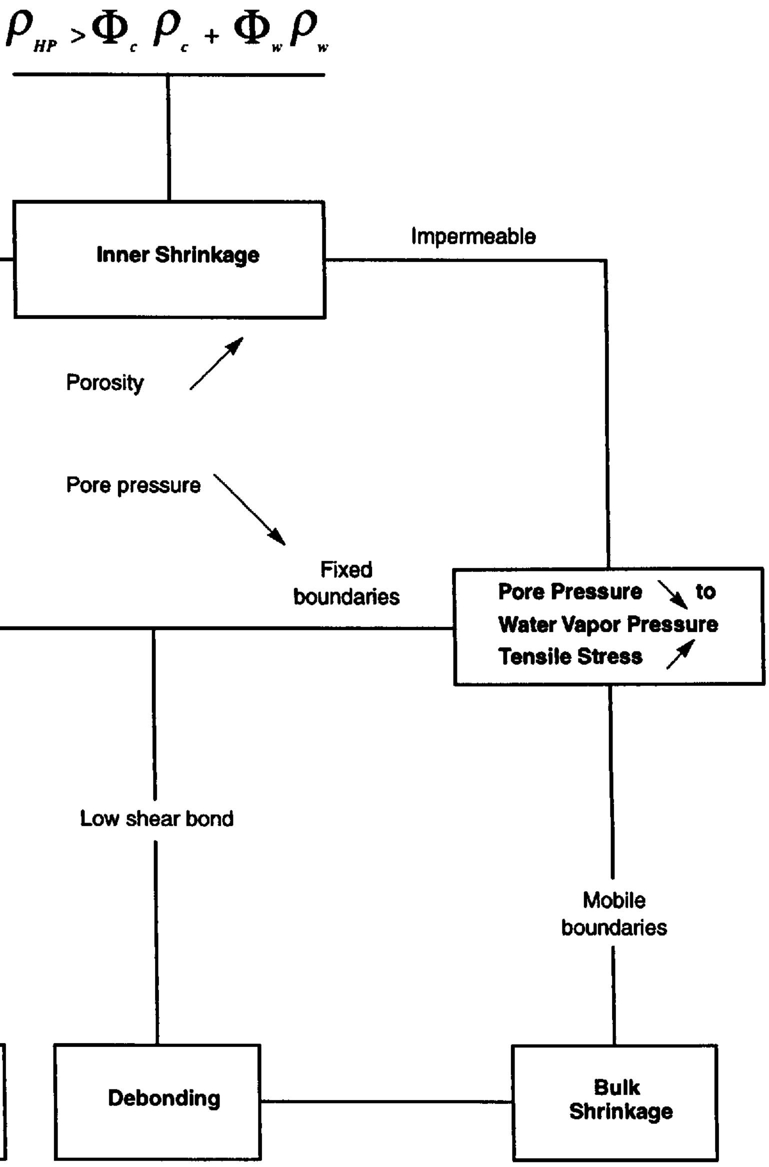


Figure 1—Cement Hydration

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:

a. It is suggested that the words *shrinkage* and *expansion* refer to the volumetric change of cement during hydration.
b. 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.
c. 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:

a. 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.
b. The cylindrical sleeve also operates under conditions of permeability to water. It allows the measurement of the bulk expansion of cement.
c. The membrane test allows the measurement of either bulk expansion or bulk shrinkage under impermeable conditions.
d. 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:

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

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

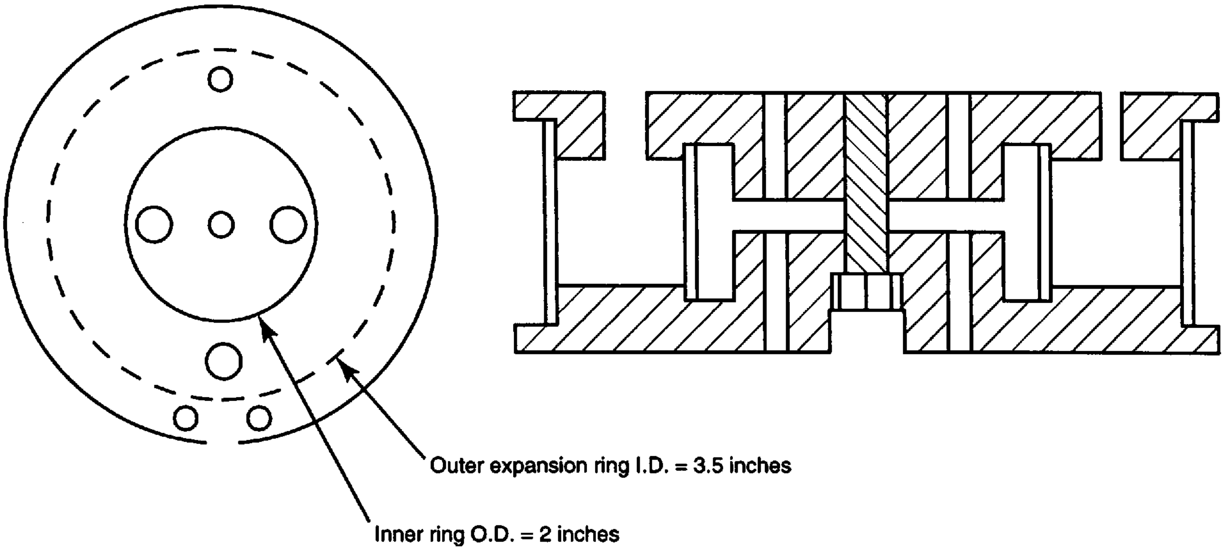


Figure 2—The Annular Expansion Mold

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:

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

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.

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

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

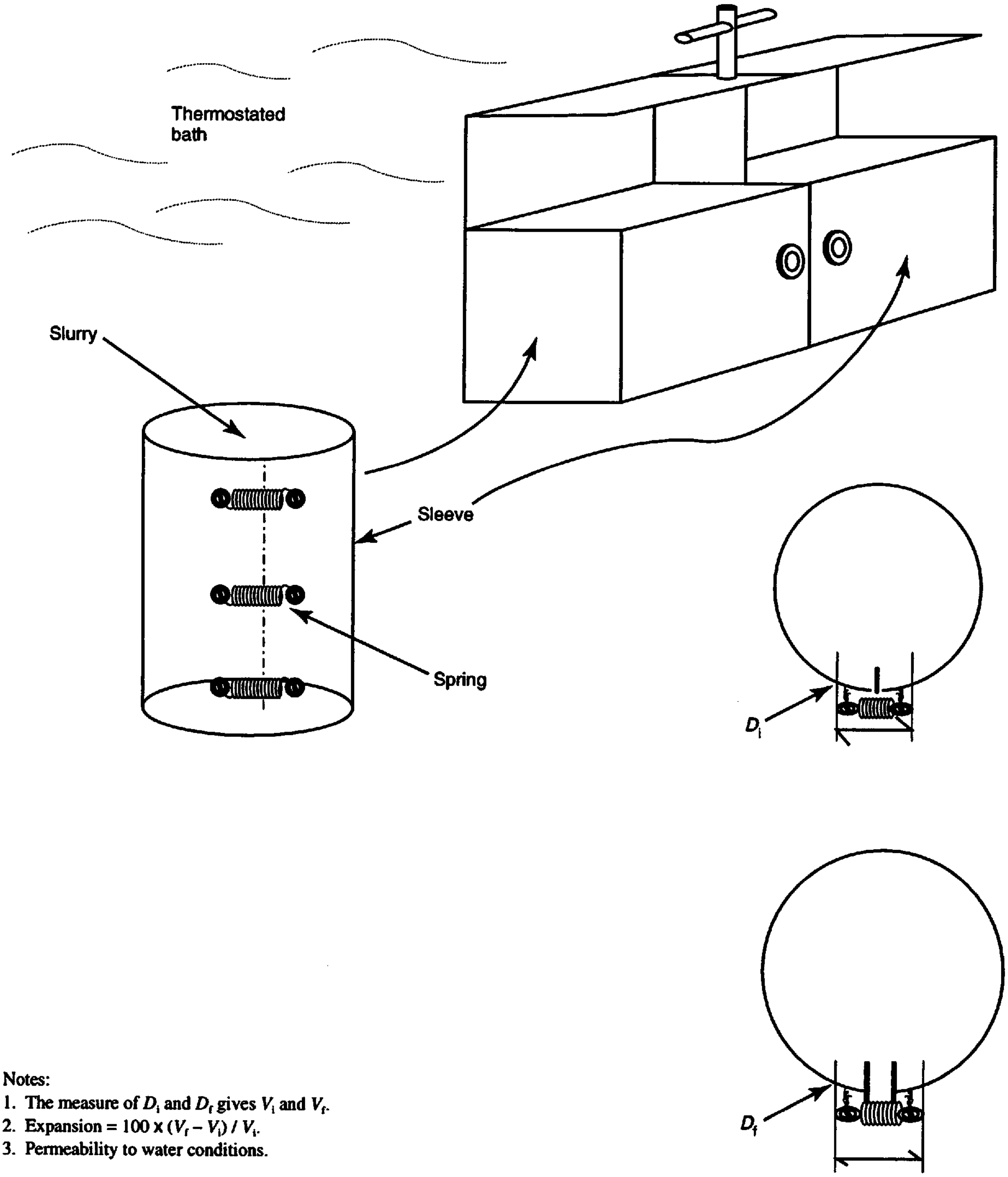
Where:

h = height of the sleeve.

The expansion becomes:

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

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



Notes:
1. The measure of D_i and D_f gives V_i and V_f.
2. Expansion = 100 x $(V_f - V_i) / V_i$.
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:

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

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

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

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.

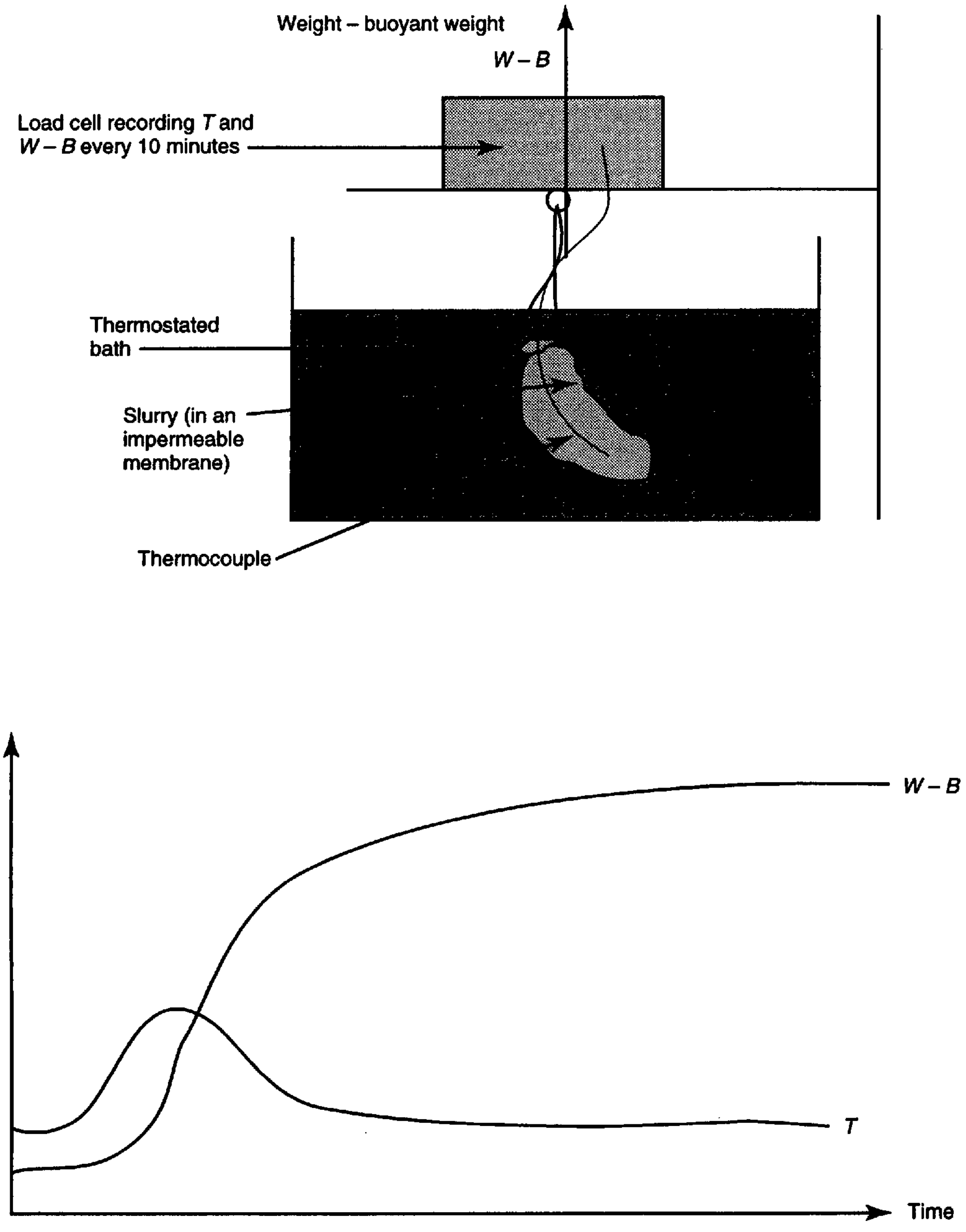
During the experiment, one can also measure the pressure at the top of the cell. This pressure slowly decreases until the temperature starts to increase. Then, pressure sharply decreases to a lower limit. At room temperature, the lowest value for the pressure in the cell is around 7 psi (0.5 bar). If 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 = $100 \times (V_f - V_i) / V_i$.
4. Impermeability to water conditions.

Figure 4—Measurement of Cement Shrinkage (or Expansion)

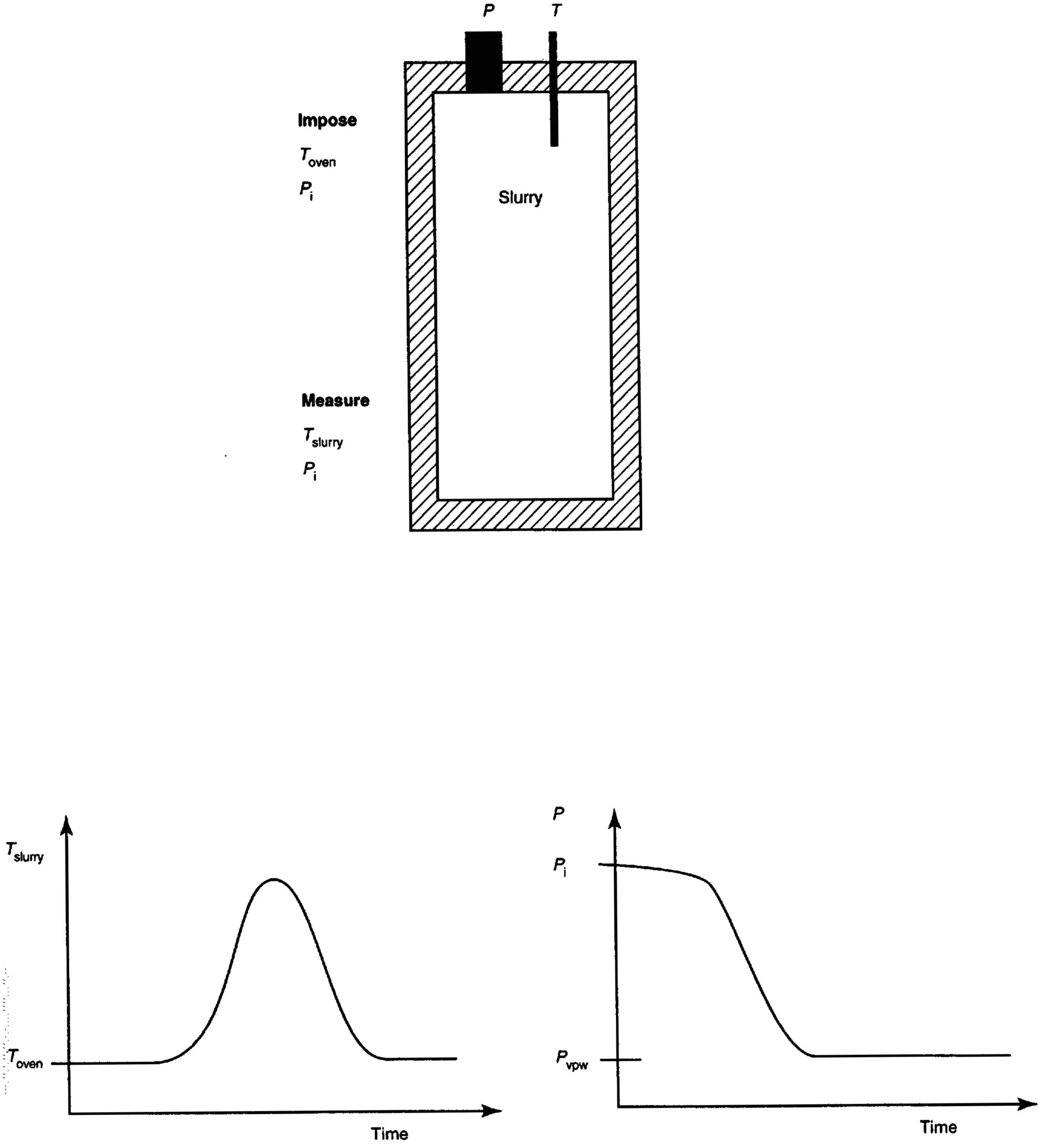


Figure 5—Experiment 1

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 centimeters^3, 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.

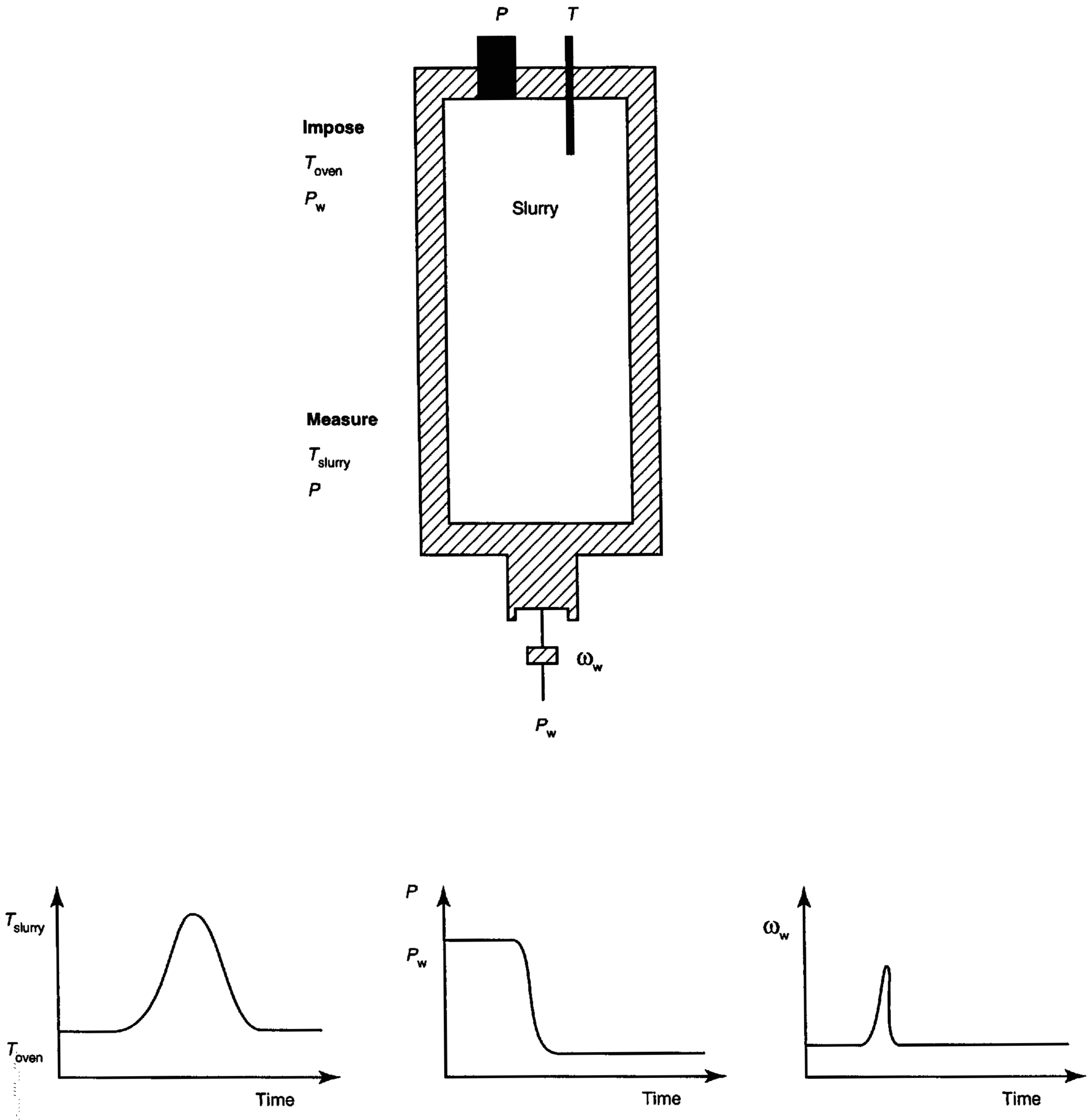
A chart recorder displays and plots the following parameters against time as the test progresses: piston rate, gas rate, temperature, and pressure. The control panel can also be connected to a computer.

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.

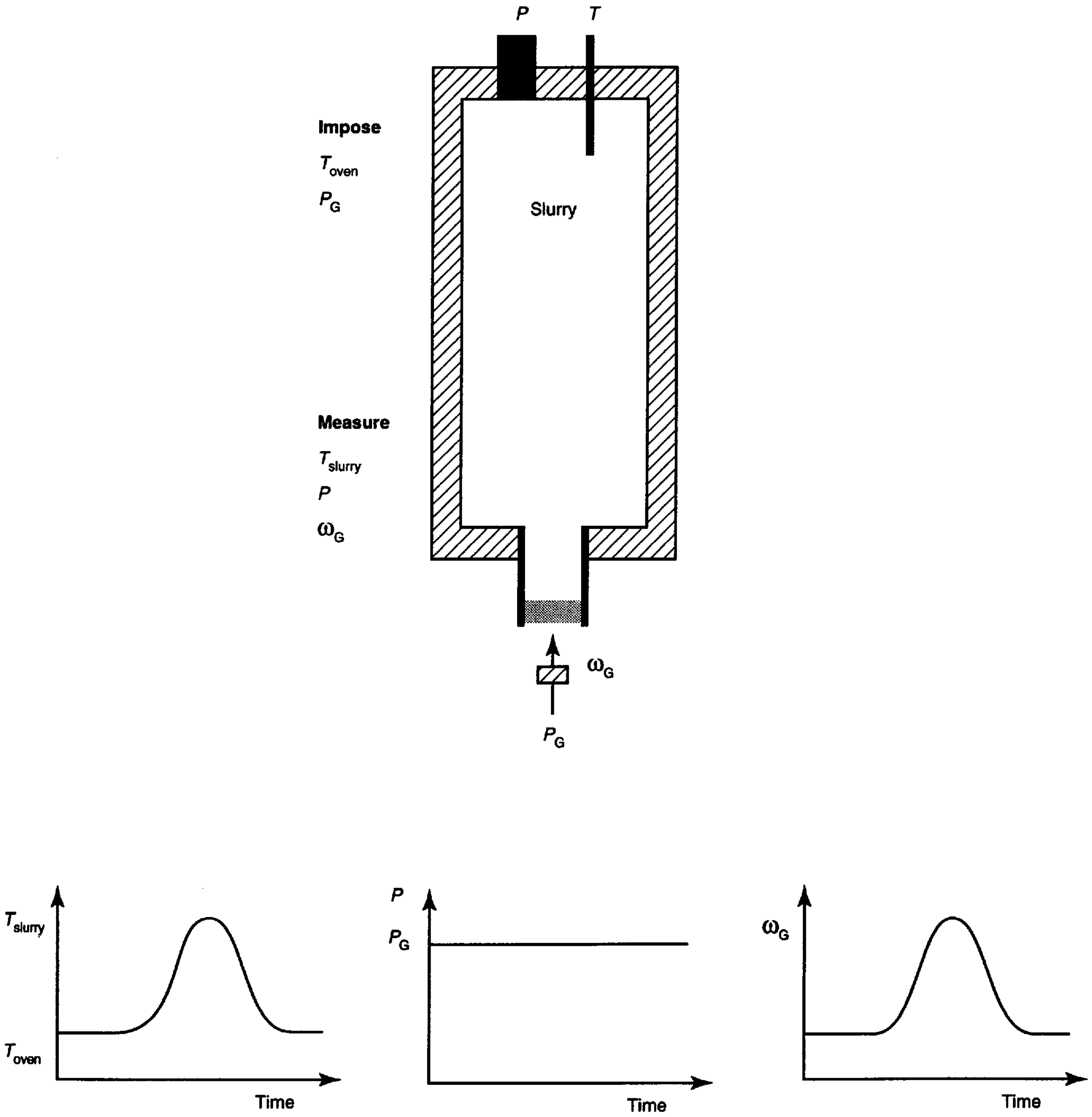
In the gas procedure, the volume of gas that enters the slurry is an estimate of the inner shrinkage. It is equal to the total shrinkage, if there is no debonding from the walls of the cell.

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

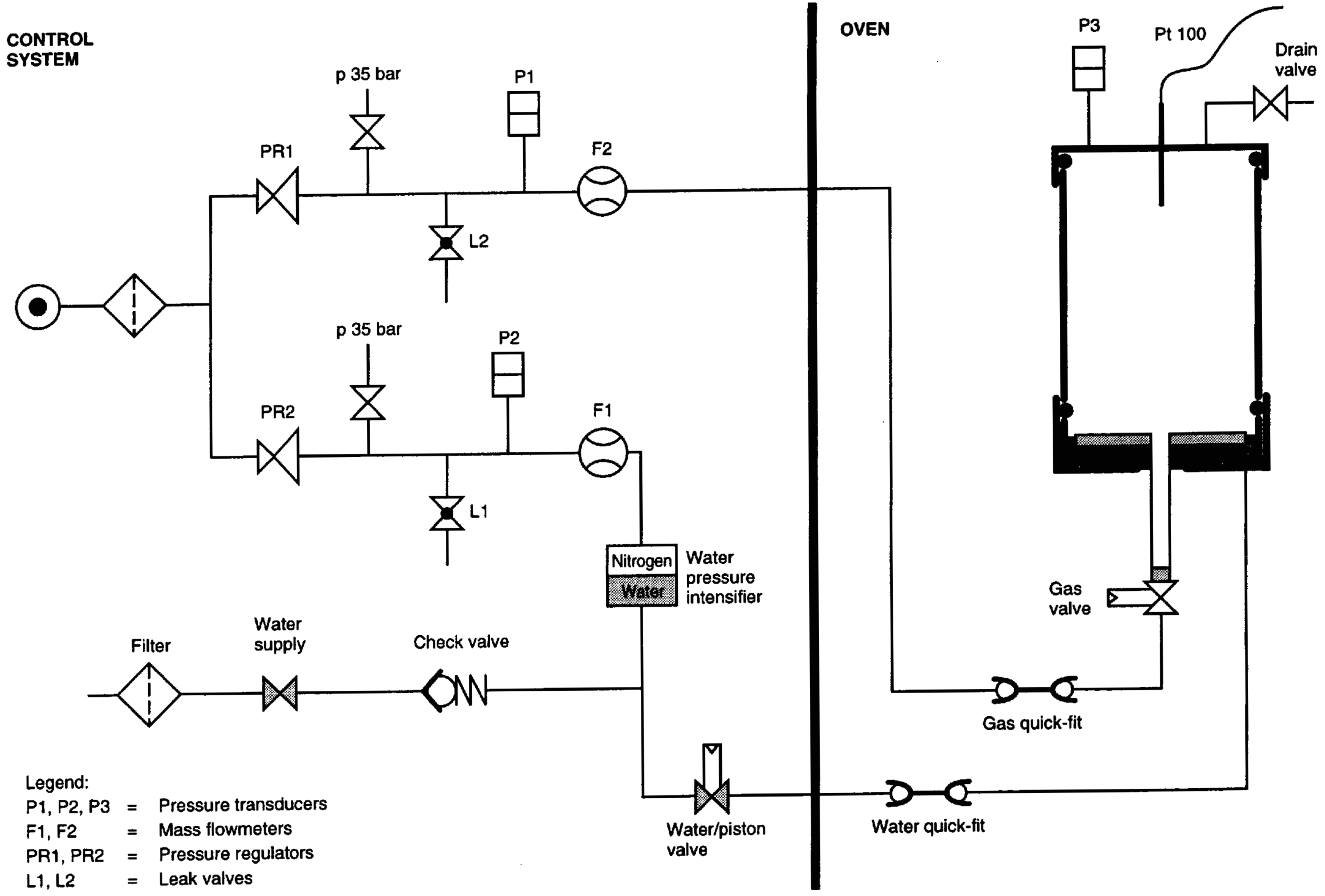


Figure 8—Cement Hydration Analyzer

Because the bulk shrinkage measured with the piston is due to the inner shrinkage of the slurry, the results for the total shrinkage should be similar if measured either using the gas or piston/gas procedure.

5.4.6 Calculations

When the final pore pressure is equal to the gas pressure and when the final temperature is equal to the initial temperature, either the integral of the gas rate curve in procedure Number 1, or the integral of the piston advancement rate curve plus the integral of the gas flow rate curve in procedure Number 2 give a good approximation of the inner shrinkage of the slurry.

Because measurements are made using mass flowmeters, data obtained are expressed in standard cubic centimeter per minute. Conversions taking into account pressure and temperature of the test must be applied to determine the exact volumes compensated for by gas entry and piston movement.

The shrinkage is calculated using the following equations:

$$V_f = V_i - V_p - V_g$$

$$\%Ish = 100 \frac{V_p + V_g}{V_i} \qquad (5)$$

Where:

V_p = volume compensated for by the piston movement.
V_g = volume of gas entered in the slurry.
V_i = 392.5 milliliters (estimated by filling up the cell with water).

6 Data Analysis

6.1 ANNULAR RING AND LINEAR BAR

The linear expansion of cement was compared using the annular ring and the linear mold as used by the ASTM. The slurry was a 16.4-ppg Class H cement containing 5 percent BWOC 60:40 CaO:MgO expanding agent. The test was run up to 7 days at 180°F (82°C) in a water bath with free access of the water to the cement. Results are presented below.

Time	Annular Ring Mold	Linear Bar Mold
1 day	0.68	0.54
3 days	1.14	1.00
7 days	1.24	1.10

It can be seen that both methods produce roughly the same results. Other laboratories should repeat these tests with different cement formulations under different conditions of temperature and pressure before any recommendation can be made.

6.2 SLEEVE AND MEMBRANE TESTS

A total of 17 slurries were tested on the sleeve and on the membrane (two were Slag-Mix). Eight additional tests were run with the membrane by measuring the initial and final pseudo weights only.

The slurries were mixed according to the API specifications, and time zero was set at the end of the 20-minute conditioning. Both sleeve and membrane tests were conducted with the same slurry at the same time and in the same thermostated bath. One sleeve and membrane test was made at 167°F (75°C); all other tests were made at 95°F (35°C).

With the sleeves, expansion values from 0.03 percent up to 0.31 percent were obtained for slurries without an expansive agent, and up to 2.16 percent for slurries with the expansive agent. With the membranes, the external volume reduction—from –0.54 percent to –4.61 percent—was always measured except for one slurry with an expansive agent, which gave an expansion of 0.99 percent.

See Table 1 for an outline of the results.

Each slurry tested on the sleeve and on the membrane is presented on a specific page showing:

a. Composition, theoretical density according to the composition and porosity.
b. Results of the membrane test, i.e., the evolution of the buoyant weight (or pseudo weight) and of the temperature during the test. (The final shrinkage is also calculated.)
c. The initial volume and the density calculated with the final real weight (i.e., the weight of the sample measured at the end of the test) are also provided.
d. The results of the sleeve test (Tables 2 to 18).

See Tables 19 to 22 for the complementary tests run with the membrane.

The calculated density appears to be different from the theoretical density. This phenomenon can have several of the following causes that also influence the result on shrinkage:

a. The real density of the slurry poured into the membrane can be different from the theoretical density. Settling can occur between the end of the conditioning and the time when the slurry is poured into the membrane. In that case, a denser slurry will be in the membrane. This was observed with the two XE tests (composition not optimized) with a theoretical density of 16.2 ppg, and a calculated density of 17.5 and 17.4 ppg.
b. Air can be trapped in the slurry. In that case, the density obtained is lower than the theoretical density. This was observed in tests with a foaming agent and also some tests where latex was used. Practically, when one can see an expansion (i.e., a decrease of the buoyant weight) while the cement slurry has not yet reached the temperature of the bath, it may correspond to the thermal expansion of the air which has been trapped. In this case, the lower value for the buoyant weight is taken as the initial pseudo weight.

c. If W, V = weight and volume of the slurry.
W_m, V_m = weight and volume of the membrane.
W_n = weight of the net.
V_n = volume of the immersed part of the net.
M = what the balance measures,

then:

$$M = W - V + W_m - V_m + W_n - V_n.$$

To calculate the initial and final volumes, and then the shrinkage, one assumes that the terms $W_m - V_m$ and $W_n - V_n$ are very small. Therefore:

$$M = W - V.$$

Taking into account the buoyancy of the membrane and the net, the real initial volume would be superior to the calculated volume and the real density would be inferior to the calculated density.

d. The water evaporation in the bath is not critical at 95°F (35°C). It becomes significant at higher temperatures. This can be observed during the test at 167°F (75°C) and the 10 percent latex slurry test made at 95°F (35°C). (Tables 10 and 15.) A reference level is chosen for the water bath, and water is regularly added. Each time water is added, one can observe an increase in the temperature and a decrease in the buoyant weight.

The temperature increases because the added water is cooler than the water in the bath; consequently, the temperature regulator produces extra heat to bring the temperature back to its initial value.

The decrease in the buoyant weight corresponds to an increase in the immersed volume. This can be attributed to thermal expansion, but more likely is due to the increase of the water level, which increases the amount of the net immersed in the water. Practically, the water should be at its reference level precisely at the beginning and at the end of the test. For the test made at 167°F (75°C), the value of the final pseudo weight corresponding to this reference level was extrapolated.

6.3 CHA TESTS

In this study, the CHA has only been used twice in direct comparison with the sleeves and the membrane. CHA tests were performed with the same slurry (Slag-Mix 14.6 ppg) using the two procedures of gas only and piston/gas (Figures 9 and 10).

a. Sleeve test: 0.05 percent bulk expansion (that is, no expansion).

b. Membrane test: –1.51 percent of bulk shrinkage.

c. CHA gas only: –2.09 percent inner shrinkage.

d. CHA piston + gas: $(-0.66\%)_{\text{piston}} + (-1.45\%)_{\text{gas}} = (-2.11\%)$ total shrinkage.

The results call for a few comments.

Using both procedures (gas-only and piston-plus gas), the total room compensated for by gas entry is equal to the total room compensated for by piston displacement plus gas. Inner shrinkage measured by the CHA is greater (in absolute value) than bulk shrinkage measured with the membrane. This makes sense because cement in the membrane cannot totally collapse while gas can enter into at least all connected pores.

Two other tests are presented in this study (Figures 20 and 21). Both are run with the same cement at the same density (16.5 ppg) and the same temperature, 150°F (65°C). One system did not contain any expanding agent while the other contained 5 percent BWOC 60:40 CaO:MgO expanding additive. After 24 hours, System 1, without an expanding agent, had developed almost –4 percent shrinkage, while System 2, with an expanding agent, had developed only –3 percent shrinkage.

Once again two comments are to be made about these results. First that the products of expansion may fill part of the porosity created by the shrinkage and exert stress on the cement matrix. The second comment is that here again one can see that regular cement develops more shrinkage than slags under similar testing conditions.

7 Results Interpretation

7.1 GENERAL COMMENTS ABOUT THE EXPANSION

Expansion measurements using the sleeves are done under conditions where water can freely enter the slurry. On all slurries studied, a slight bulk expansion was measured to be inferior to 0.31 percent, except with the cement containing an expansive agent. These systems will be separately studied.

These results substantiate the fact that, if the entire inner shrinkage is compensated for by fluid entry, no bulk shrinkage should occur.

7.2 NEAT SLURRIES

Four different slurries were first studied in the membrane. These slurries had the same density of 16.8 ppg. The only difference was the brand of cement. Experimental results are detailed in Table 19 and are summarized below:

Cement Brand	Bulk Shrinkage
Holnam H	–4.39%
Holnam H (1 month later)	–3.77%
Lone Star	–3.28%
Lafarge Balcones	–3.77%
Lafarge Joppa	–4.61%

The last four tests were performed at the same time in the same water bath at 95°F (35°C). Values were taken after 5 days.

Results indicate that for the cements tested and under the test conditions, the bulk shrinkage of a neat 16.8-ppg Class H slurry varies in the range of –4.00 ± 0.75 percent.

The same cement (Holnam H) was tested under the same conditions except that the density was changed. Detailed results are presented in Table 20.

Density	Porosity	Bulk Shrinkage
15.6 ppg	60.0%	–4.24%
17.0 ppg	52.5%	–3.77%
17.6 ppg	49.0%	–1.99%

A test was made on a neat 15.6-ppg Lone Star A slurry, giving a shrinkage of –3.83 percent (Table 3). This is quite similar to what was measured on Class H slurries.

Shrinkage is not significantly different between a Class A and a Class H under the same tests conditions. It was observed that in a neat slurry, the more water (i.e., the less cement) there is in the slurry, the higher the shrinkage. This result was also observed in the Slag-Mix (Tables 17 and 18).

Density	Porosity	Bulk Shrinkage
13.0 ppg	69.6%	–2.50%
14.6 ppg	59.7%	–1.51%

The 14.6-ppg slag system was one of the few systems studied under the same temperature conditions with the membrane and with the CHA (Figures 9 and 10).

These results call for several comments:

a. Under the membrane test conditions, the same behavior was observed for the Slag-Mix as for the Class H cement: more slag per unit volume leads to less bulk shrinkage.

b. This conclusion is somewhat contrary to intuition. One could believe that the more cement there is in a given volume of slurry, the more bulk shrinkage will exist. A possible explanation could be that more cement leads to a more rigid structure with a reduced tendency to collapse. Intuition and reality can be reconciled if it is observed that more cement per unit volume leads to more inner shrinkage under permeable conditions. This was not investigated in this study.

7.3 INFLUENCE OF FOAMING AGENT AND ENTRAPPED AIR

A total of 0.1 gallons per sack of foaming agent was added to the neat 16.8 Holnam H slurry and the test was repeated twice (Tables 4 and 5). This value is now very similar to what was observed on the neat system (–4.39 percent).

The first test leads to a calculated density of 13.1 ppg, showing that foam had been produced (22 percent of the total volume). A very low bulk shrinkage of –0.54 percent (to be compared to –4.39 percent) and an expansion of 0.10 percent were obtained. The test was repeated after deaerating the slurry and a much lower shrinkage (–4.35 percent) was obtained.

7.4 CONCLUSION

These tests show that the foaming agent had no chemical effect on bulk shrinkage. In turn, if air is entrapped inside the slurry, its physical effect can be drastic.

This phenomenon can be easily explained. When inner shrinkage develops, pore pressure decreases. Because the air compressibility is much higher than the water compressibility, pore pressure will not decrease as much when air is present. Hence, much less bulk shrinkage is produced.

Therefore all shrinkage experiments should be run on deaerated slurries.

7.5 ACTION OF AN EXPANDING AGENT

Two tests were performed to observe the influence of a 60:40 CaO:MgO expanding agent. One test was run with 5 percent BWOC expanding agent (Table 6) and the other with 10 percent (Table 7). Respectively, there was 0.61 and 2.16 percent of bulk expansion in the sleeve and –1.32 and +0.99 percent of bulk shrinkage in the membrane. This is a perfect example that the same slurry might show bulk shrinkage under impermeable conditions and still expand under permeable conditions.

7.5.1 Shrinkage

As can be seen on both curves, the expanding agent is active during the exothermal part of the cement hydration. Then the slurry comes back to a more normal behavior, a progressive shrinkage, similar to what was observed with the neat slurry (Table 2).

With 10 percent expanding agent, a negative shrinkage could ultimately be possible. However, for the duration of the test (5 days), the late shrinkage did not compensate for the initial expansion.

7.5.2 Expansion

The sleeve tests show that in permeable-to-water conditions, the expanding agent leads to a significant expansion.

The bulk expansion measured on the sleeves was plotted as a function of the bulk shrinkage measured on the membrane (Figure 11).

7.5.3 Conclusion

The 60:40 CaO:MgO additive produces expansion under permeability-to-water conditions. Under impermeability-to-water conditions, the additive reduces bulk shrinkage and can also lead to a bulk expansion.

7.6 POROSITY EFFECT

In 7.2, it was noted that on increasing the cement content, less bulk shrinkage was observed. Cement content was increased by increasing the slurry density. In this paragraph, the effect of porosity without changing the density is studied.

The effect of porosity was tested using a latex additive. This additive is inert, has the same density as water, and was used to decrease the porosity by 5 and 10 percent respectively (Tables 8 and 10) without changing the cement content. The 10 percent test was repeated twice because air was trapped in the first formulation (Tables 9 and 10).

Shrinkage was plotted as a function of porosity (Figures 12 and 13). The 5-percent reduced porosity test was made with the Holnam H cement and, consequently, should be compared to the Holnam neat slurry test. The 10 percent reduced porosity test should be compared to the Lone Star neat slurry test.

In the first case, the decrease of porosity by 5 percent produces less bulk shrinkage, –3.74 percent instead of –4.39 percent. In the second case, the decrease of porosity by 10 percent produces more bulk shrinkage, –3.93 percent instead of –3.28 percent. The trends are inverse, but in any case, the changes are rather small.

Concerning the expansion, there seems to be the same correlation as for the neat slurry:

a. 0.03 percent bulk expansion for the neat.
b. 0.07 percent when porosity is reduced by 5 percent.
c. 0.17 percent when porosity is reduced by 10 percent.

Other tests were performed using latex and expansive agent additives. At 5-percent BWOC expansive agent, a 5-percent porosity reduction increases the bulk shrinkage from –1.32 percent to –1.63 percent (Tables 6 and 11). At 10-percent BWOC expansive agent, a 5-percent porosity reduction increases the bulk shrinkage from –0.99 percent to –1.48 percent (Tables 7 and 13).

No immediate conclusion can be made on the effect of the slurry porosity when the cement content is unchanged.

7.7 RESEARCH OF POSSIBLE TRENDS

7.7.1 Water-to-Cement Ratio

For comparable systems, the shrinkage was plotted as a function of the water-to-cement ratio (Figures 17 to 19).

Two inverse trends are observed because there is an increase of the shrinkage with this ratio for neat slurries, but a decrease of it from a 10-percent porosity-reduced slurry to a neat slurry and to the XE test.

Conclusion:

No general trend was observed between the shrinkage and the water-to-cement ratio.

7.7.2 Effect of Dispersion

If it is assumed that the presence of latex has no important effect on the shrinkage, then 1-percent BWOC dispersant added to the system with 10 percent BWOC expanding agent increased the shrinkage from 0.99 percent to –1.48 percent. This effect was confirmed on the system with 5 percent BWOC expanding agent, adding latex so as to reduce the porosity of 5 percent and 1 percent dispersant BWOC (Table 12).

The shrinkage was also increased here because it was –2.33 percent instead of –1.32 percent (Figure 14).

Conclusion:

It appears that cement shrinkage increases with slurry dispersion. What seems to be important is the available surface of cement to be hydrated.

A microcement was studied (Table 22). The result, –2.28 percent of shrinkage, does not allow any conclusion. It does not mean that the hypothesis is not checked; the relative amount of cement is much lower in the case of the microcement because the ratio of cement weight to total weight is 58.5 percent (compared to 73.7 percent).

8 Conclusion

The experimental study confirms the general theory about shrinkage developed in Section 4.

When a particle of cement reacts with water, the volume of the hydration product is less than the initial volume of cement plus water. In a cement sample, this phenomenon will occur everywhere inside the matrix. This phenomenon leads to an inner shrinkage.

If there is free access to an external fluid, fluid entry will compensate for this volume loss and no bulk shrinkage will be observed.

If fluid entry is not allowed, inner shrinkage leads to pore pressure reduction. The cement matrix will have a tendency to collapse, leading to bulk shrinkage.

If bulk shrinkage is not allowed due to a perfect bonding to the fixed boundaries, then the tensile stress will increase in the material, leading to possible fractures if the tensile strength is exceeded.

In the described tests, it was seen that a loose matrix with less cement and more water presents more bulk shrinkage than a strong matrix having more cement. It would be interesting to measure inner shrinkage under permeable conditions as a function of cement content. Logic suggests that inner shrinkage increases with cement content, as long as the minimum water necessary for hydration is available. This could easily be verified using the CHA.

It was also observed that if air is trapped inside the cement matrix, less bulk shrinkage will take place because air will expand, filling the newly created porosity.

Expansion proceeds from the growth of crystals which exert stress on the cement matrix. The bulk expansion does not prevent the cement from undergoing inner shrinkage.

In an oil or gas well, the following trends should be expected:

a. Less tendency to bulk shrinkage in front of a permeable zone (water or gas) if the mudcake does not create an impermeable barrier.

b. More tendency to bulk shrinkage or debonding or even fractures in front of impermeable zones.

c. Little bulk expansion in a closed packer compared to what could be expected from laboratory experiments run under permeable conditions.

d. Very low pressure, down to the vapor pressure of water at that temperature, right above an External Casing Packer set between casings.

Finally, it is the contention of the authors of this study that a cement laboratory interested in making tests on expansion and shrinkage could make use of the following equipment:

a. The annular ring to measure linear expansion under free access to fluid. The equipment is inexpensive and easy-to-run at any pressure or temperature.

b. The cylindrical sleeve to measure bulk expansion under free access to fluid. The equipment is inexpensive and easy-to-run at any pressure or temperature.

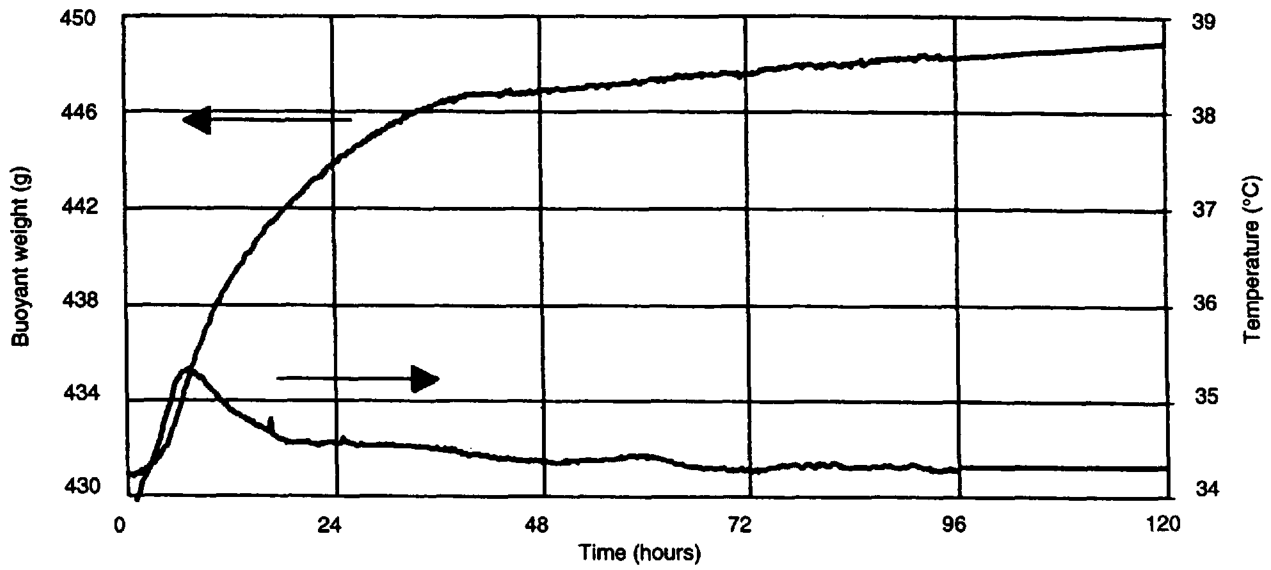
c. Flexible membranes to measure bulk shrinkage or expansion under impermeable conditions. The actual problem is that the membranes resistant to the temperature conditions existing in an oil well are not disposed of.

d. CHA-type equipment to measure inner shrinkage under conditions of free access to gas. This equipment requires a minimum of care to operate. At this time, such equipment is not designed to operate above 300°F and 600 psi.

Table 1—General Table

Test	Slurry Type	Theoretical Density ppg	Calculated Density ppg	Membrane %	Sleeves %	Porosity %	Liquid Total (volume) %	Water/ Cement (weight) %	Cement/ Total (volume) %
5	Neat (H) H	16.8	17.1	–4.39	0.03	53.0	53.0	35.6	47.0
(i1)	Neat (H) H	16.8	17.0	-3.77		53.0	53.0	35.6	47.0
(i4)	Neat Lone Star H	16.8	16.8	-3.28		53.0	53.0	35.6	47.0
(i5)	Neat LaFarge B H	16.8	17.0	-3.77		53.0	53.0	35.6	47.0
(i7)	Neat LaFarge J H	16.8	16.9	–4.61		53.0	53.0	35.6	47.0
13	Neat (L) A	15.6	15.9	-3.83	0.15	59.7	59.7	46.8	40.3
8	Foaming Agent (H)	16.8	13.1	–0.54	0.10	63.4 (52.9)	41.2	34.7	36.6
9	Foaming Agent (H)	16.8	16.7	–4.35		53.2 (52.9)	52.6	34.7	46.7
7	Latex (5%) (H)	16.8	16.6	-3.74	0.07	48.2 (47.6)	47.1	32.1	46.6
15	Latex (10%) (L)	16.8	15.7	–4.37	0.15	46.2 (42.5)	39.7	28.7	44.1
16	Latex (10%) (L)	16.8	16.7	-3.93	0.17	42.9 (42.5)	42.2	28.7	46.8
2	CaO:MgO (5%) (H)	17.1	17.2	-1.32	0.61	50.1	50.1	33.7	47.0
3	CaO:MgO (10%) (H)	17.5	18.0	0.99	2.16	47.2	47.2	31.8	47.1
4	Latex (5%)+CaO:MgO (5%) (H)	17.1	17.7	-1.63	0.64	44.8	44.8	30.2	47.0
18	Latex (5%)+CaO:MgO (5%) (H)	17.1	17.3	-2.33	0.53	44.3	44.3	30.2	46.6
17	Latex (5%)+CaO:MgO (10%) (H)	17.5	18.0	-1.48	1.46	41.6	41.6	28.4	46.5
(i2)	Neat (H) H + Dispersant	16.7	16.7	-3.24		52.4	52.4	25.6	46.6
(i6)	Microcement	13.4	13.4	-2.28		67.0	67.0	71.0	33.0
10	Blend (35C) (L)	16.2	17.5	-2.86	0.31	47.4	47.4	82.5	18.2
11	Blend (75C) (L)	16.2	17.4	-1.81	0.21	47.1	47.1	82.5	18.1
(i3)	Neat (H) H 15.5 ppg	15.5	15.6	–4.24		60.0	60.0	47.5	40.0
(i8)	Neat (H) H 17.5 ppg	17.5	17.6	-1.99		49.0	49.0	30.4	51.0
19	AMOCO Latex (D) G 23 ppg	23.0	22.8	-3.61	0.15	31.3 (30.7)	30.4	32.8	26.1
9a	Slag-Mix 13 ppg	13.0	13.2	-2.50	0.04	69.6	69.6	87.7	27.4
14	Slag-Mix 14.6 ppg	14.6	14.8	-1.51	0.05	59.7	59.7	53.0	38.8

Table 2—Neat Slurry (H) Test, 16.8 ppg



Slurry:

Holnam H	890.6 g	—	**Calculated Density**	17.1 ppg
DI Water	315.2 g	315.2 ml		
Antifoam	2.0 g	2.0 ml	**Porosity**	52.9%

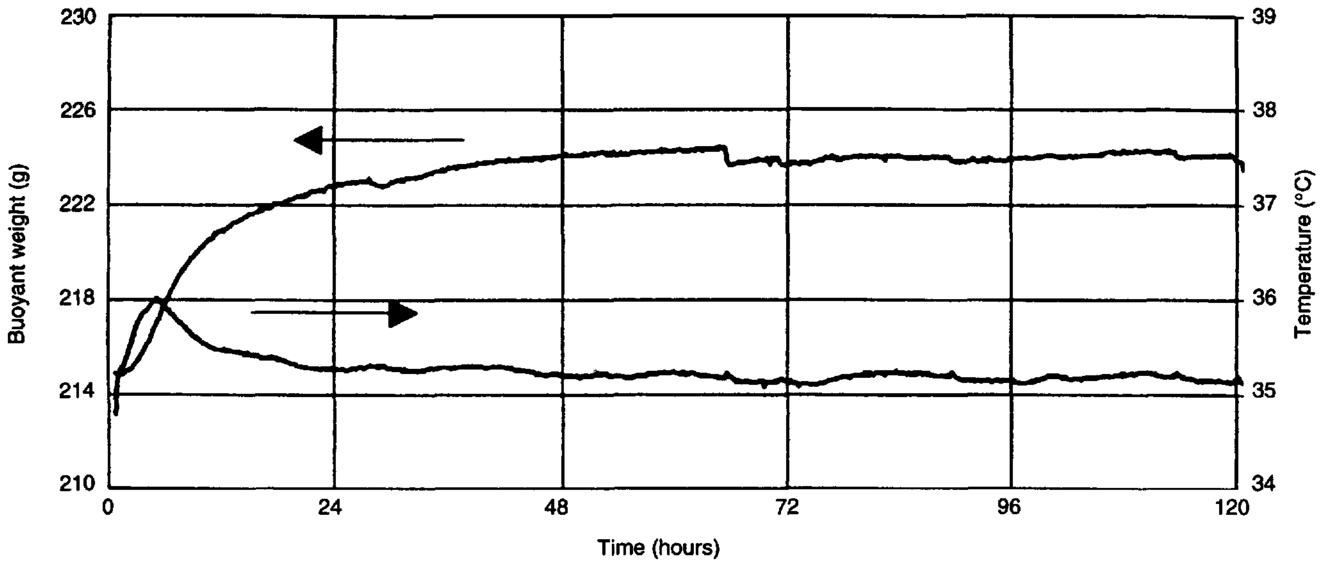
Membrane:

Initial pseudo weight	430.92 g		
Final pseudo weight	448.91 g		
Final real weight	841.00 g		
Initial volume	410.08 cm^3		
Final volume	392.09 cm^3	**Shrinkage**	–4.39%

Sleeve:

Initial 5	Final 5	Average		
0.915 in.	0.915 in.			
0.916 in.	0.917 in.	0.0003 in.		
0.912 in.	0.912 in.			
Initial 9	**Final 9**	**Average**		
1.028 in.	1.030 in.			
1.014 in.	1.016 in.	0.0010 in.		
1.039 in.	1.038 in.		**Expansion**	0.03%

Table 3—Neat Slurry (A) Test, 15.6 ppg



Slurry:

Lonestar A	764.6 g	—	**Calculated Density**	15.9 ppg
DI Water	358.1 g	358.1 ml		
Antifoam agent			**Porosity**	59.7%

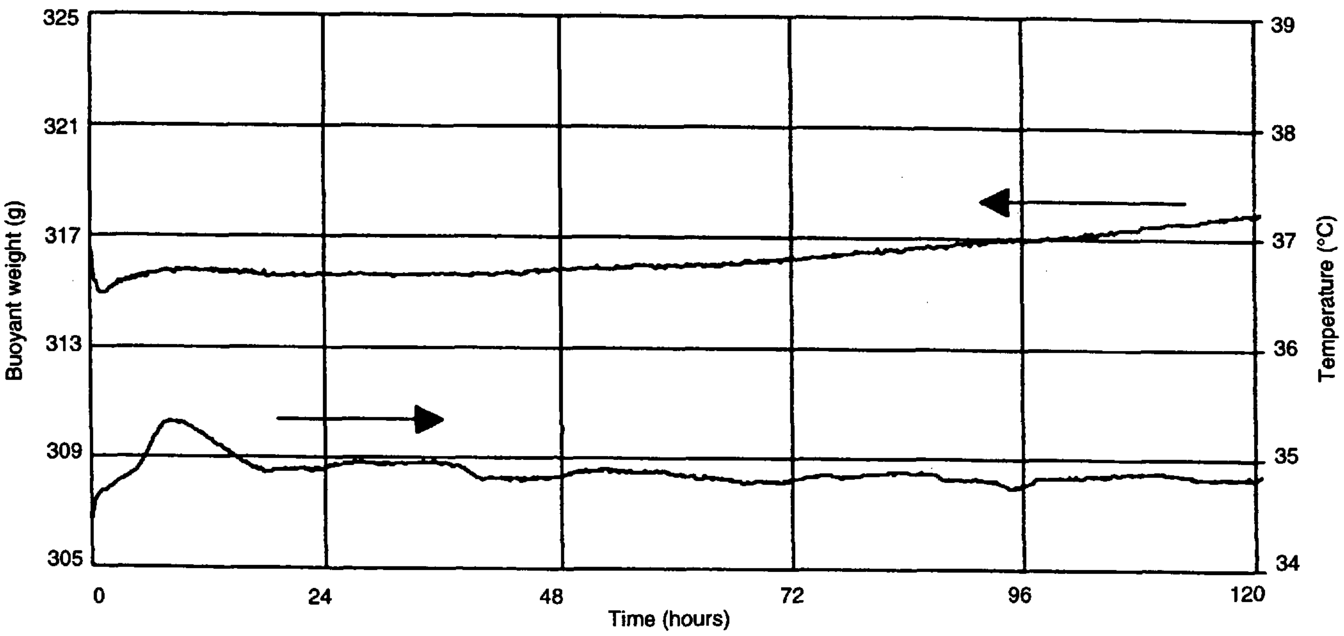
Membrane:

Initial pseudo weight	214.89 g		
Final pseudo weight	223.87 g		
Final real weight	449.50 g		
Initial volume	234.61 cm^3		
Final volume	225.63 cm^3	**Shrinkage**	−3.83%

Sleeve:

Initial 5	Final 5	Average		
0.914 in.	0.915 in.			
0.922 in.	0.925 in.	0.0027 in.		
0.921 in.	0.925 in.			
Initial 20	**Final 20**	**Average**		
0.920 in.	0.925 in.			
0.912 in.	0.916 in.	0.0050 in.		
0.896 in.	0.902 in.		**Expansion**	0.15%

Table 4—Foaming Agent Test, 16.8 ppg



Slurry:

Holnam H	890.6 g	—	**Calculated Density**	13.1 ppg
DI Water	309.3 g	309.3 ml		
Foaming agent	8.3 g	7.9 ml	**Porosity**	63.4% (52.9%)
			Air:	172.5 ml

Membrane:

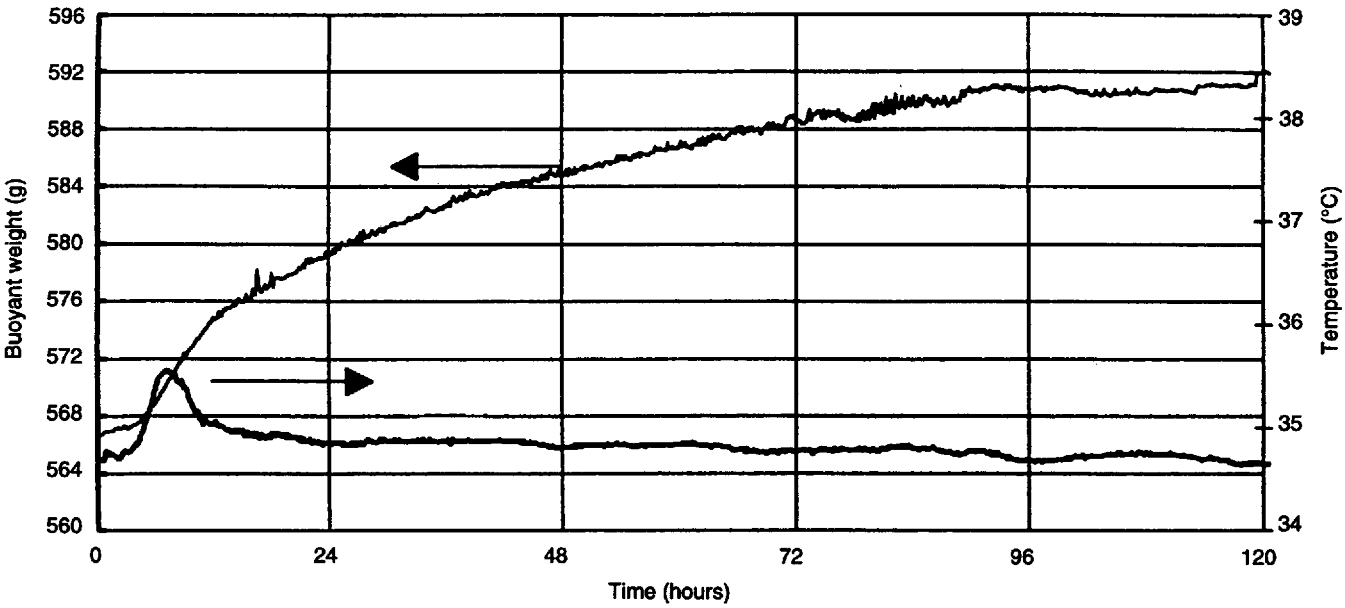
Initial pseudo weight	314.97 g		
Final pseudo weight	317.95 g		
Final real weight	868.90 g		
Initial volume	553.93 cm³		
Final volume	550.95 cm³	**Shrinkage**	–0.54%

Sleeve:

Initial 5	Final 5	Average		
0.913 in.	0.915 in.			
0.917 in.	0.919 in.	0.0020 in.		
0.912 in.	0.914 in.			

Initial 7	Final 7	Average		
1.035 in.	1.041 in.			
1.013 in.	1.015 in.	0.0033 in.		
1.021 in.	1.023 in.		**Expansion**	0.10%

Table 5—Foaming Agent Test, 16.8 ppg



Slurry:

Holnam H	890.6 g	—	**Calculated Density**	16.7 ppg
DI Water	309.3 g	309.3 ml		
Foaming agent	8.3 g	7.9 ml	**Porosity**	5.32% (52.9%)
			Air:	4.21 ml

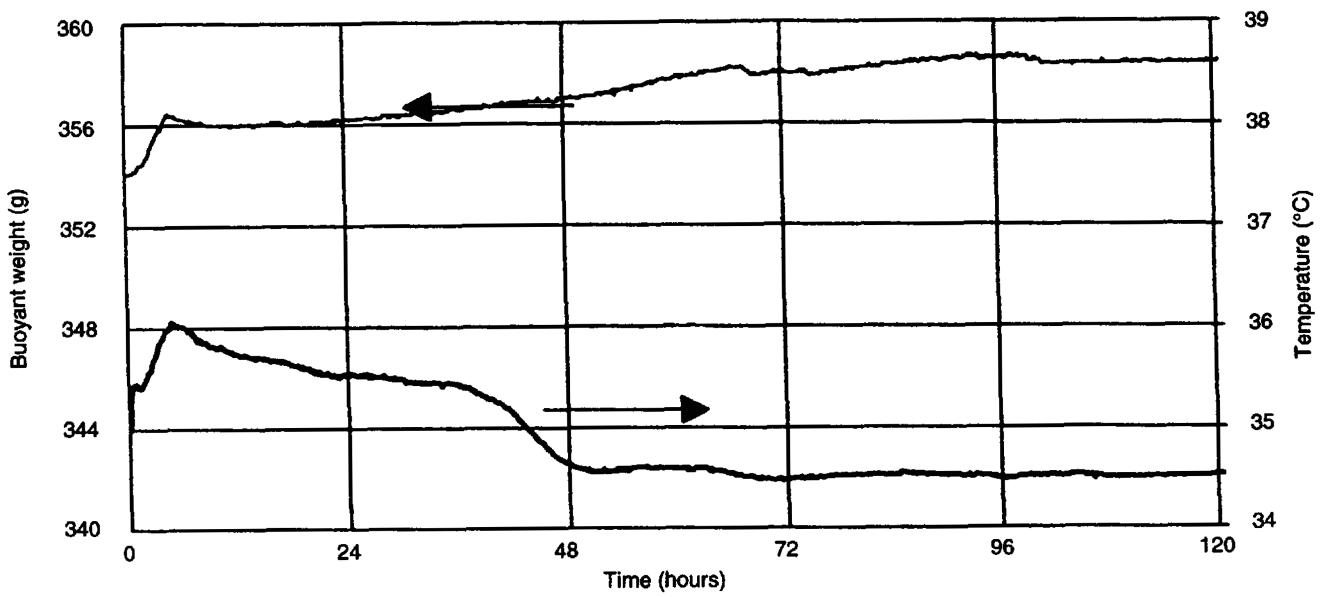
Membrane:

Initial pseudo weight	566.71 g		
Final pseudo weight	591.24 g		
Final real weight	1130.50 g		
Initial volume	563.79 cm³		
Final volume	539.26 cm³	**Shrinkage**	–4.35%

Sleeve:

Initial 5	Final 5	Average		
Initial 91	**Final 91**	**Average**		
			Expansion	%

Table 6—Expanding Agent Test, 17.1 ppg



Slurry:

Holnam H	890.60 g	—	**Calculated Density**	17.2 ppg
DI Water	298.10 g	298.1 ml		
Antifoam agent	2.00 g	2.0 ml	**Porosity**	50.0%
Expanding agent	44.53 g	17.1 ml		

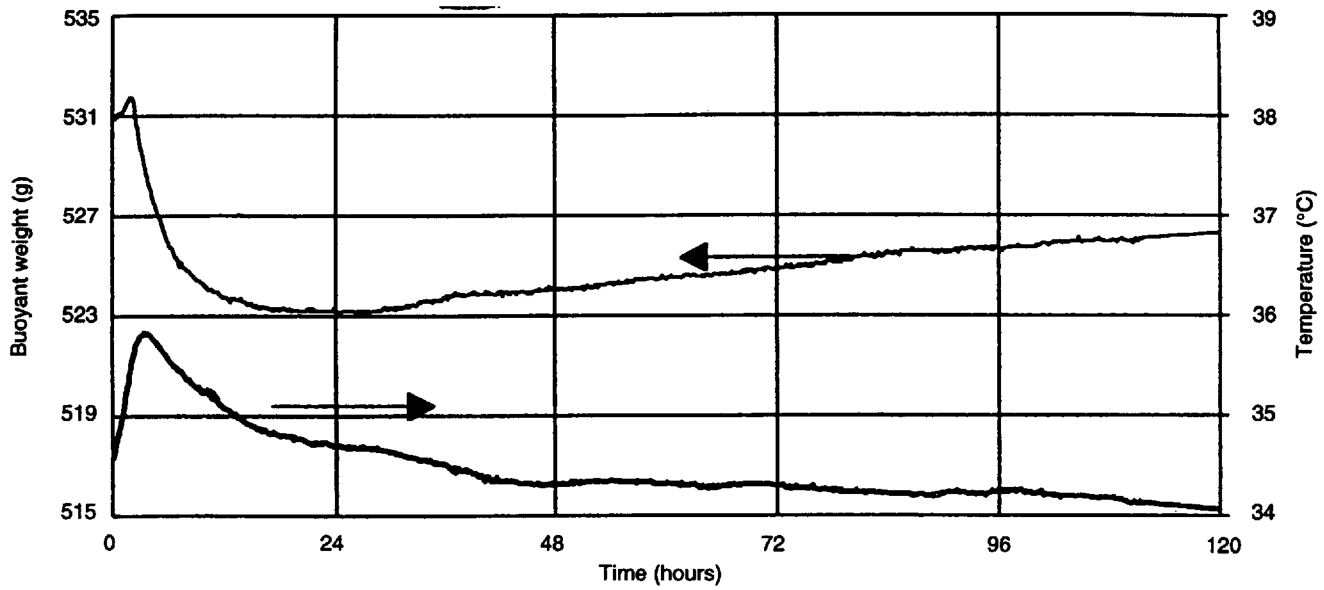
Membrane:

Initial pseudo weight	354.05 g		
Final pseudo weight	358.43 g		
Final real weight	686.60 g		
Initial volume	332.55 cm^3		
Final volume	328.17 cm^3	**Shrinkage**	−1.32%

Sleeve:

Initial 5	Final 5	Average		
0.914 in.	0.929 in.			
0.915 in.	0.931 in.	0.0157 in.		
0.911 in.	0.927 in.			
Initial 91	**Final 91**	**Average**		
1.026 in.	1.043 in.			
1.014 in.	1.029 in.	0.0157 in.		
1.038 in.	1.053 in.		**Expansion**	0.61%

Table 7—10 Percent Expanding Test, 17.5 ppg



Slurry:

Holnam H	890.6 g	—	**Calculated Density**	18.0 ppg
DI Water	280.9 g	280.9 ml		
Antifoam agent	2.0 g	2.0 ml	**Porosity**	47.2%
Expanding agent	89.1 g	34.3 ml		

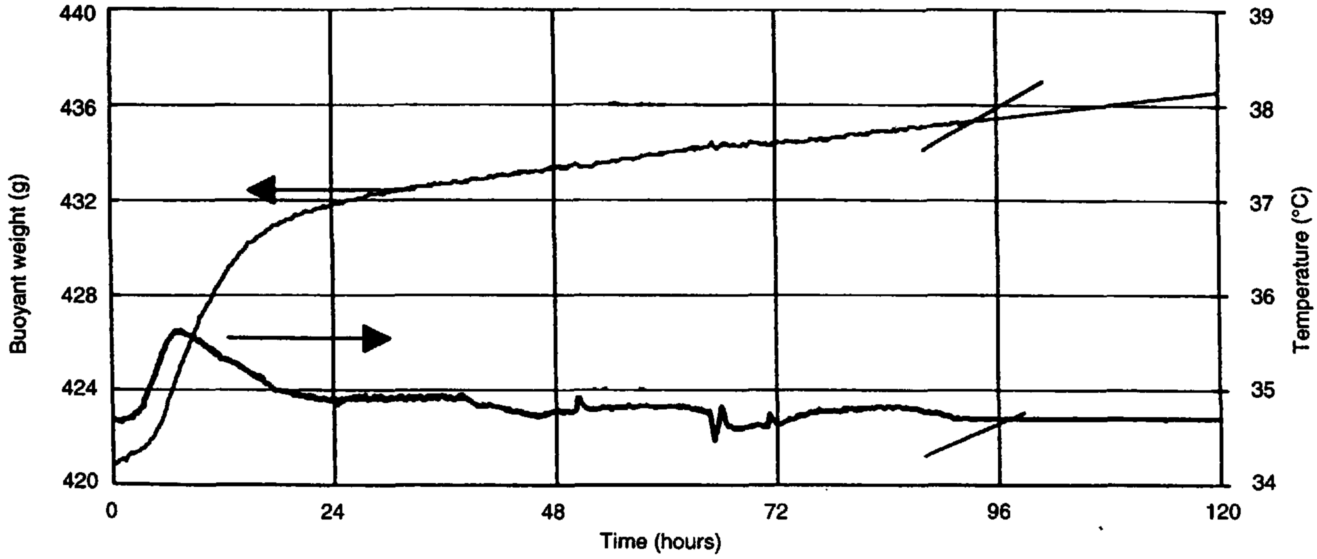
Membrane:

Initial pseudo weight	530.83 g		
Final pseudo weight	526.31 g		
Final real weight	985.30 g		
Initial volume	454.47 cm³		
Final volume	458.99 cm³	**Shrinkage**	0.99%

Sleeve:

Initial 5	Final 5	Average		
0.915 in.	0.978 in.			
0.917 in.	0.977 in.	0.0593 in.		
0.913 in.	0.968 in.			
Initial 91	Final 91	Average		
1.028 in.	1.085 in.			
1.017 in.	1.070 in.	0.0520 in.		
1.042 in.	1.088 in.		**Expansion**	2.16%

Table 8—5 Percent Latex Slurry Test, 16.8 ppg



Slurry:

Holnam H	890.6 g	—	**Calculated Density**	16.6 ppg
DI Water	251.8 g	251.80 ml		
Antifoam agent	2.0 g	2.00 ml	**Porosity**	48.2% (47.6%)
Latex	63.4 g	62.16 ml	Air:	7.58 ml

Membrane:

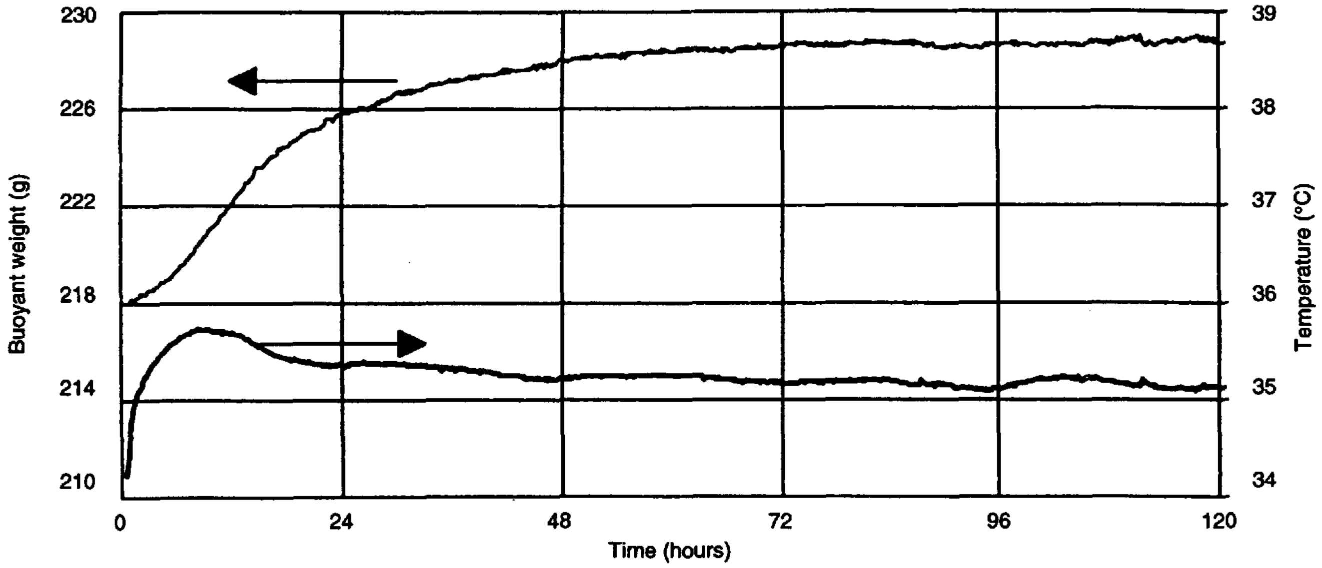
Initial pseudo weight	420.78 g		
Final pseudo weight	436.60 g		
Final real weight	844.10 g		
Initial volume	423.32 cm^3		
Final volume	407.50 cm^3	**Shrinkage**	–3.74%

Sleeve:

Initial 5	Final 5	Average		
0.914 in.	0.914 in.			
0.915 in.	0.917 in.	0.0007 in.		
0.913 in.	0.913 in.			

Initial 7	Final 7	Average		
1.038 in.	1.043 in.			
1.015 in.	1.016 in.	0.0030 in.		
1.022 in.	1.025 in.		**Expansion**	0.07%

Table 9—10 Percent Latex Slurry Test, 16.8 ppg



Slurry:

Lonestar H	890.6 g	—	**Calculated Density**	15.7 ppg
DI Water	190.9 g	190.9 ml		
Antifoam agent	1.0 g	1.0 ml	**Porosity**	46.2% (42.5%)
Latex	126.8 g	124.3 ml	Air:	41.68 ml

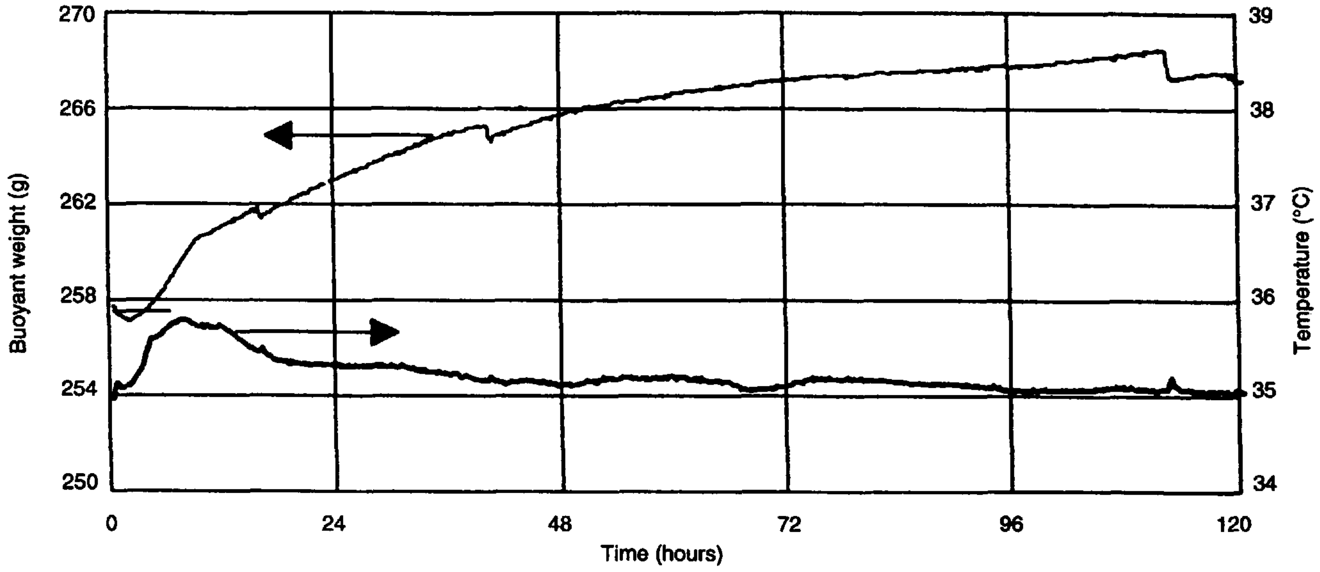
Membrane:

Initial pseudo weight	217.97 g		
Final pseudo weight	228.70 g		
Final real weight	463.50 g		
Initial volume	245.53 cm^3		
Final volume	234.80 cm^3	**Shrinkage**	–4.37%

Sleeve:

Initial 5	Final 5	Average		
0.9145 in.	0.9165 in.			
0.9220 in.	0.9260 in.	0.0037 in.		
0.9230 in.	0.9280 in.			
Initial 20	Final 20	Average		
0.900 in.	0.905 in.			
0.914 in.	0.918 in.	0.0040 in.		
0.922 in.	0.925 in.		**Expansion**	0.15%

Table 10—10 Percent Latex Slurry Test, 16.8 ppg



Slurry:

Lonestar H	890.6 g	—	**Calculated Density**	16.7 ppg
DI Water	189.9 g	189.9 ml		
Antifoam agent	2.0 g	2.0 ml	**Porosity**	42.9% (42.5%)
Latex	126.8 g	124.3 ml	Air:	4.18 ml

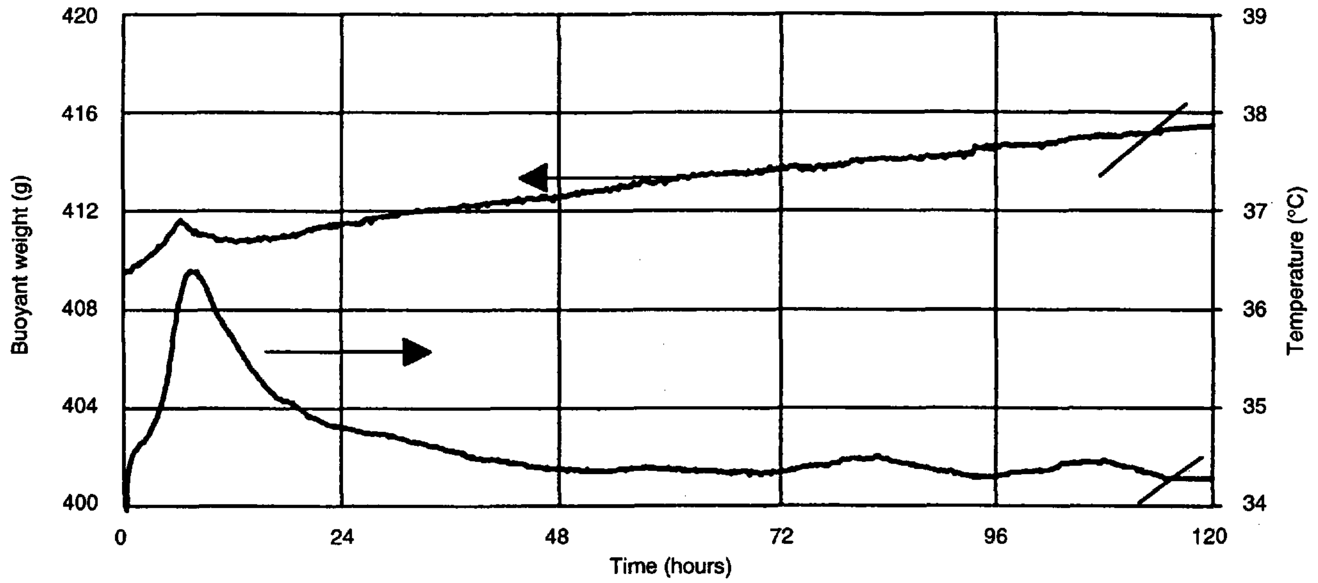
Membrane:

Initial pseudo weight	257.12 g		
Final pseudo weight	267.17 g		
Final real weight	512.90 g		
Initial volume	255.78 cm^3		
Final volume	245.73 cm^3	**Shrinkage**	–3.93%

Sleeve:

Initial 5	Final 5	Average		
0.9150 in.	0.915 in.			
0.9235 in.	0.927 in.	0.0027 in.		
0.9215 in.	0.926 in.			
Initial 20	Final 20	Average		
0.921 in.	0.927 in.			
0.914 in.	0.919 in.	0.0063 in.		
0.896 in.	0.904 in.		**Expansion**	0.17%

Table 11—5 Percent Expanding Plus Latex Test, 17.1 ppg



Slurry:

Holnam H	890.60 g	—	**Calculated Density**	17.7 ppg
DI Water	234.70 g	234.70 ml		
Antifoam	2.00 g	2.00 ml	**Porosity**	44.7%
Expanding agent	44.53 g	17.13 ml		
Latex	64.70 g	63.40 ml		

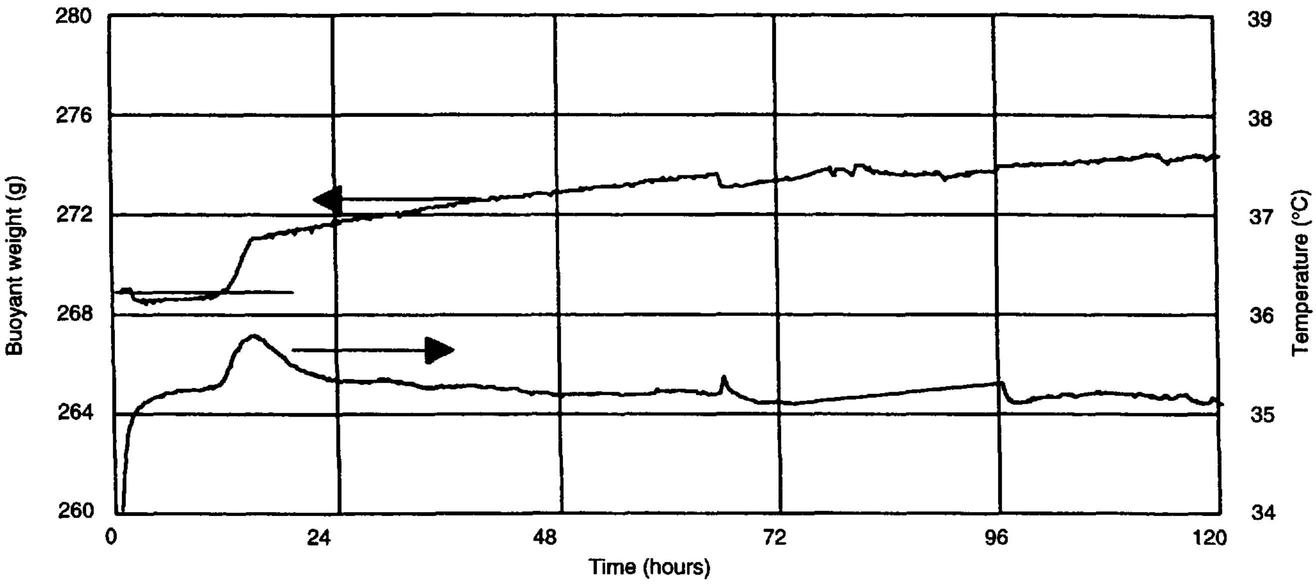
Membrane:

Initial pseudo weight	409.60 g		
Final pseudo weight	415.48 g		
Final real weight	771.20 g		
Initial volume	361.60 cm^3		
Final volume	355.72 cm^3	**Shrinkage**	−1.63%

Sleeve:

Initial 5	Final 5	Average		
0.915 in.	0.934 in.			
0.920 in.	0.937 in.	0.0183 in.		
0.912 in.	0.931 in.			
Initial 9	Final 9	Average		
1.027 in.	1.043 in.			
1.017 in.	1.032 in.	0.0150 in.		
1.039 in.	1.053 in.		**Expansion**	0.64%

Table 12—5 Percent CaO:MgO + Latex (5 Percent) + Dispersant (1 Percent) Test, 17.1 ppg



Slurry:

Holnam H	890.60 g	—	**Calculated Density**	17.3 ppg
DI Water	234.70 g	234.70 ml		
Antifoam agent	2.00 g	2.00 ml	**Porosity**	44.3%
Expanding agent	44.53 g	17.13 ml		
Latex	64.70 g	63.40 ml		
Dispersant	8.91 g	6.23 ml		

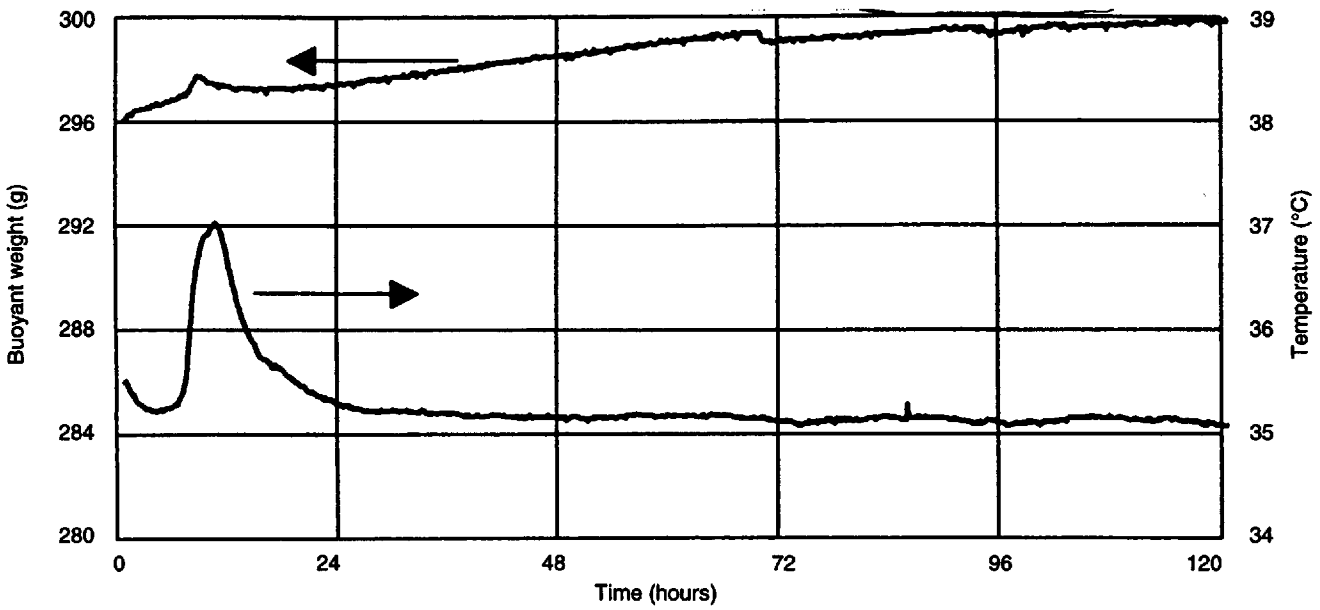
Membrane:

Initial pseudo weight	268.58 g		
Final pseudo weight	274.39 g		
Final real weight	517.50 g		
Initial volume	248.92 cm^3		
Final volume	243.11 cm^3	**Shrinkage**	-2.33%

Sleeve:

Initial 5	Final 5	Average		
0.919 in.	0.9310 in.			
0.918 in.	0.9300 in.	0.0122 in.		
0.911 in.	0.9235 in.			
Initial 20	Final 20	Average		
0.923 in.	0.936 in.			
0.916 in.	0.931 in.	0.0153 in.		
0.904 in.	0.922 in.		**Expansion**	0.53%

Table 13—10 Percent CaO:MgO + Latex (5 Percent) + Dispersant (1 Percent) Test, 17.5 ppg



Slurry:

Holnam H	890.60 g	—	**Calculated Density**	18.0 ppg
DI Water	218.50 g	218.50 ml		
Antifoam agent	2.00 g	2.00 ml	**Porosity**	41.6%
CaO:MgO	89.06 g	34.25 ml		
Latex	64.70 g	63.40 ml		
Dispersant	8.91 g	6.23 ml		

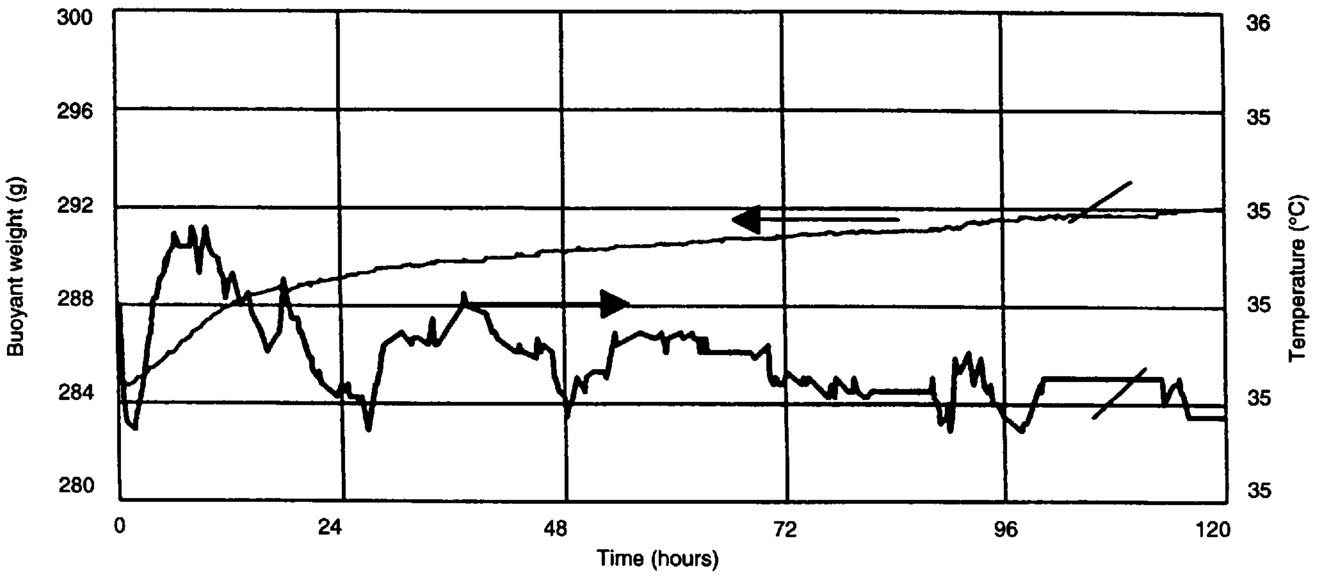
Membrane:

Initial pseudo weight	296.04 g		
Final pseudo weight	299.83 g		
Final real weight	551.80 g		
Initial volume	255.76 cm³		
Final volume	251.97 cm³	**Shrinkage**	–1.48%

Sleeve:

Initial 5	Final 5	Average		
0.919 in.	0.954 in.			
0.919 in.	0.955 in.	0.0363 in.		
0.910 in.	0.948 in.			
Initial 20	Final 20	Average		
0.923 in.	0.958 in.			
0.918 in.	0.955 in.	0.0390 in.		
0.901 in.	0.946 in.		**Expansion**	1.46%

Table 14—Proprietary Blend Test, 16.2 ppg



Slurry:

Blend	—	—	**Calculated Density**	17.5 ppg
DI Water	284.3 g	284.3 ml		
			Porosity	47.4%

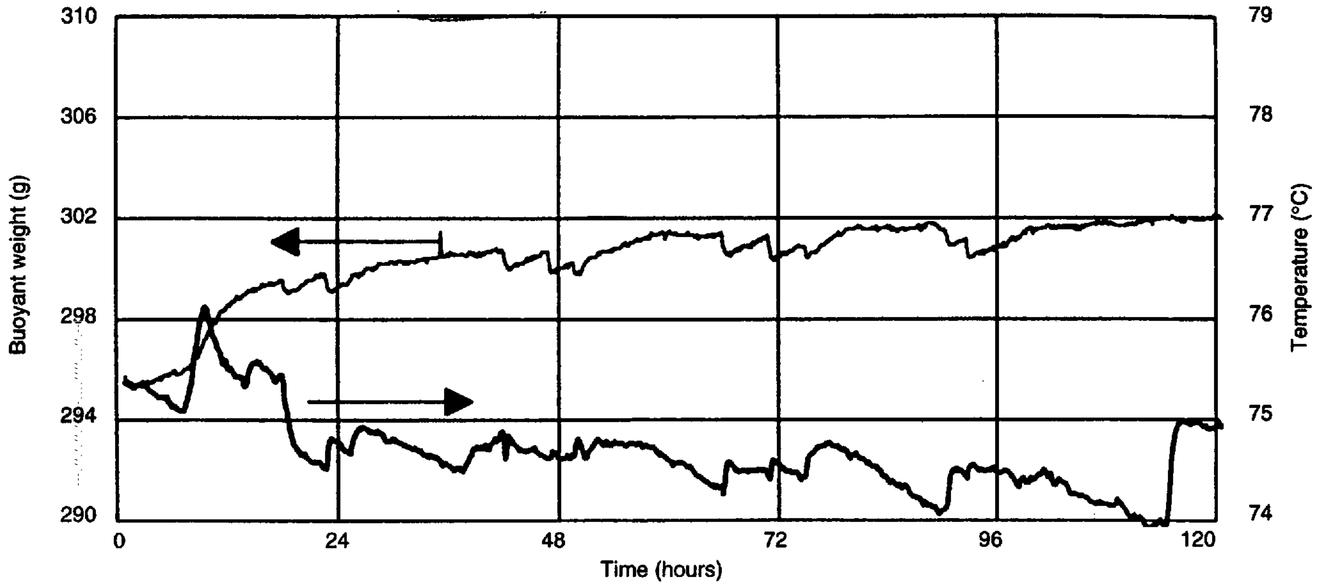
Membrane:

Initial pseudo weight	284.70 g		
Final pseudo weight	292.10 g		
Final real weight	543.20 g		
Initial volume	258.50 cm^3		
Final volume	251.10 cm^3	**Shrinkage**	–2.86%

Sleeve:

Initial 5	Final 5	Average		
0.912 in.	0.913 in.			
0.917 in.	0.923 in.	0.0063 in.		
0.911 in.	0.923 in.			
Initial 20	Final 20	Average		
0.912 in.	0.921 in.			
0.904 in.	0.913 in.	0.0097 in.		
0.886 in.	0.897 in.		**Expansion**	0.31%

Table 15—Proprietary Blend Test, 16.2 ppg



Slurry:

Blend	—	—	**Calculated Density**	17.4 ppg
DI Water	284.30 g	284.30 ml		
Fluid Loss	4.42 g	3.25 ml	**Porosity**	47.1%
Dispersant	0.44 g	0.31 ml		
Retarder	0.71 g	0.56 ml		
Antifoam				

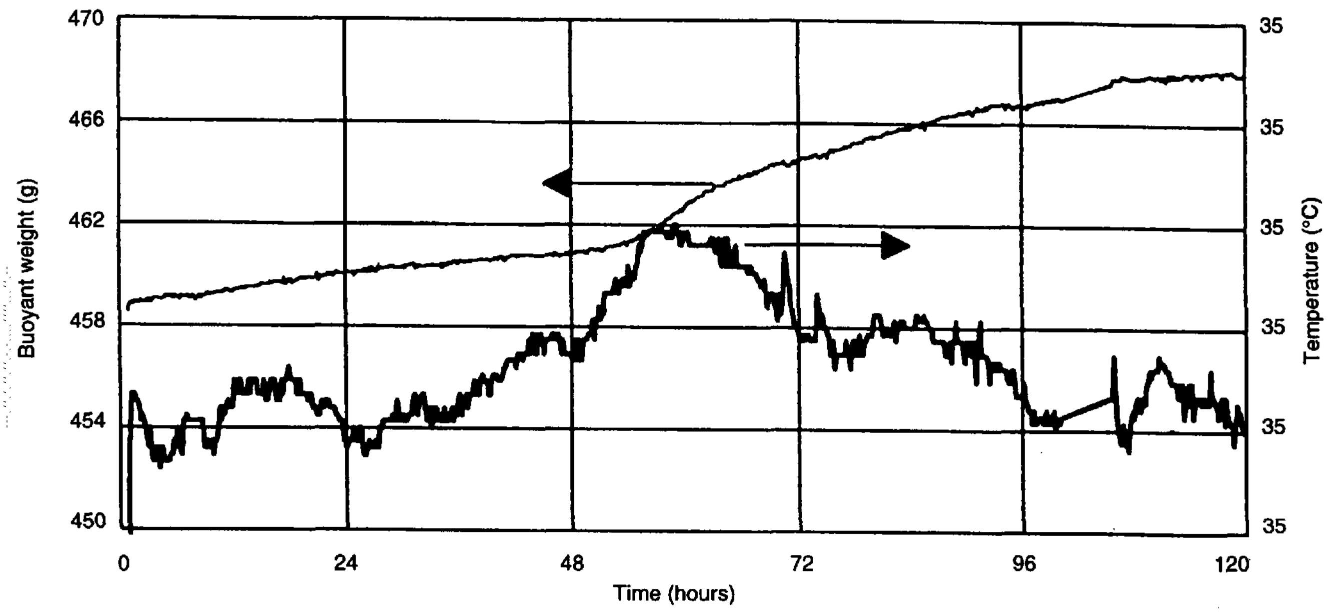
Membrane:

Initial pseudo weight	295.72 g		
Final pseudo weight	300.64 g		
Final real weight	566.90 g		
Initial volume	271.18 cm³		
Final volume	266.26 cm³	**Shrinkage**	−1.81%

Sleeve:

Initial 5	Final 5	Average		
0.914 in.	0.916 in.			
0.920 in.	0.925 in.	0.0053 in.		
0.915 in.	0.924 in.			
Initial 20	Final 20	Average		
		0.0000 in.		
			Expansion	0.21%

Table 16—Amoco Latex Test, 23.0 ppg



Slurry:

Blend	1413.0 g	—	**Calculated Density**	22.8 ppg
DI Water	108.6 g	108.6 ml		
Antifoam	2.2 g	2.2 ml	**Porosity**	31.3%
Retarder	3.6 g	3.1 ml		(30.7%)
Dispersant	17.5 g	14.5 ml	Air:	5.37 ml
Surfactant	5.6 g	5.3 ml		
Latex	103.0 g	101.0 ml		

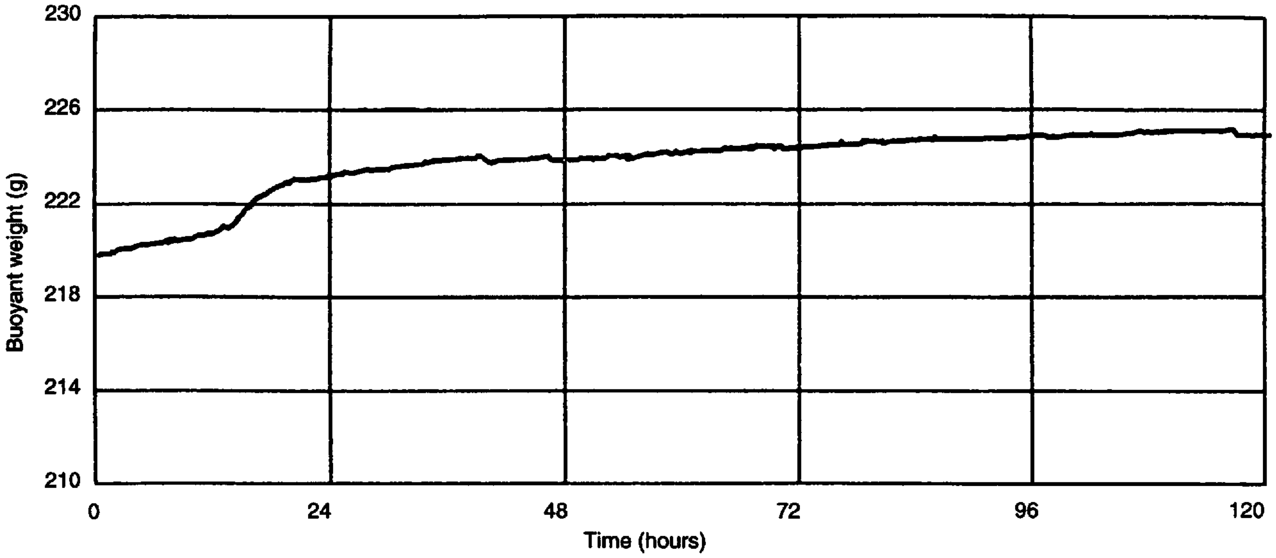
Membrane:

Initial pseudo weight	458.55 g		
Final pseudo weight	468.06 g		
Final real weight	722.10 g		
Initial volume	263.55 cm³		
Final volume	254.04 cm³	**Shrinkage**	–3.61%

Sleeve:

Initial 5	Final 5	Average		
0.919 in.	0.9210 in.			
0.918 in.	0.9205 in.	0.0018 in.		
0.912 in.	0.9130 in.			
Initial 20	**Final 20**	**Average**		
0.925 in.	0.929 in.			
0.918 in.	0.922 in.	0.0060 in.		
0.899 in.	0.909 in.		**Expansion**	0.15%

Table 17—Slag Mix Test, 13.0 ppg



Slurry:

Fresh water	417.7 g	417.7 ml	**Calculated Density**	13.2 ppg
Bentonite	11.9 g	4.5 ml		
BFS	476.2 g	164.2 ml	**Porosity**	69.6%
NaOH	21.4 g	10.0 ml		
Na2CO3	7.1 g	2.8 ml		
Spersene CF	1.2 g	0.8 ml		

Membrane:

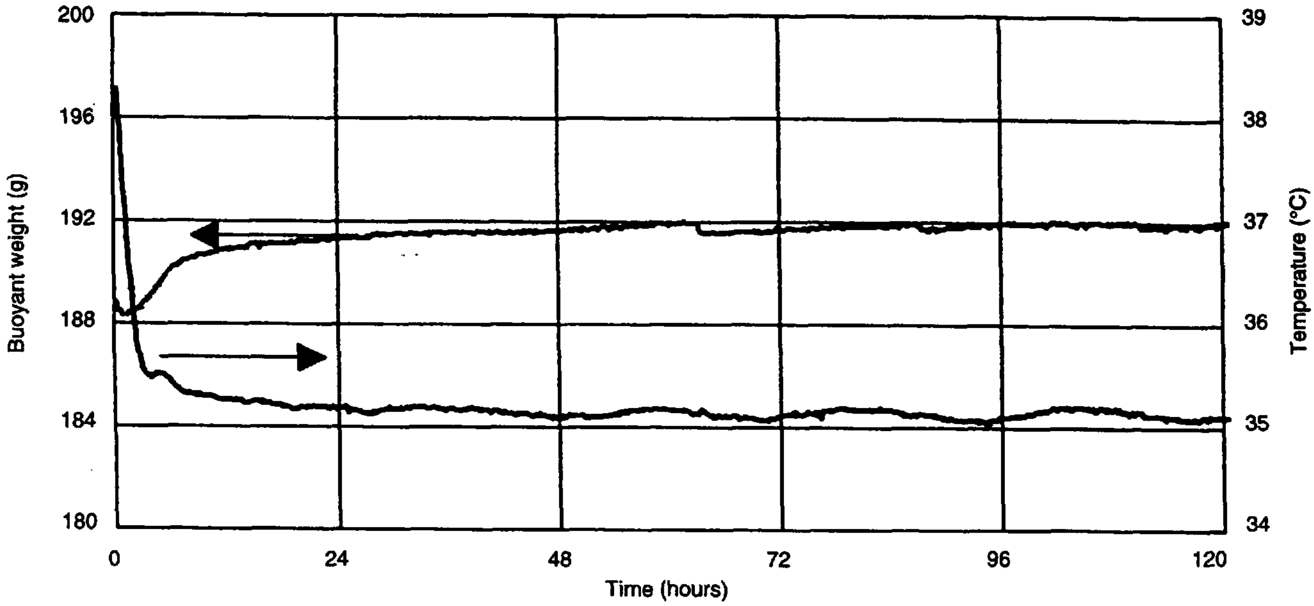
Initial pseudo weight	119.90 g		
Final pseudo weight	125.00 g		
Final real weight	323.70 g		
Initial volume	203.80 cm³		
Final volume	198.70 cm³	**Shrinkage**	–2.50%

Sleeve:

Initial 5	Final 5	Average		
0.912 in.	0.912 in.			
0.917 in.	0.918 in.	0.0010 in.		
0.915 in.	0.917 in.			

Initial 20	Final 20	Average		
0.914 in.	0.915 in.			
0.908 in.	0.909 in.	0.0010 in.		
0.891 in.	0.892 in.		**Expansion**	0.04%

Table 18—Slag Mix Test, 14.6 ppg



Slurry:

Fresh water	358.1 g	358.1 ml	**Calculated Density**	14.8 ppg
Bentonite	10.2 g	3.9 ml		
BFS	675.6 g	233.0 ml	**Porosity**	59.7%
NaOH	5.1 g	2.4 ml		
Na2CO3	5.1 g	2.0 ml		
Spersene CF	1.0 g	0.7 ml		

Membrane:

Initial pseudo weight	188.32 g		
Final pseudo weight	191.98 g		
Final real weight	430.60 g		
Initial volume	242.28 cm^3		
Final volume	238.62 cm^3	**Shrinkage**	−1.51%

Sleeve:

Initial 5	Final 5	Average		
0.914 in.	0.914 in.			
0.921 in.	0.922 in.	0.0010 in.		
0.919 in.	0.921 in.			
Initial 20	Final 20	Average		
0.922 in.	0.925 in.			
0.915 in.	0.915 in.	0.0017 in.		
0.900 in.	0.902 in.		**Expansion**	0.05%

Table 19—Neat H Slurries, 16.8 ppg

Slurry components:	
Cement - Class H	890.6 grams
DI Water	315.2 grams (315.2 ml)
Antifoam	2.0 grams (2.0 ml)

Porosity - **52.9%**

	Holnam	Lone Star	Lafarge- Balcones	Lafarge- Joppa
Calculated density (ppg)	17.0	16.8	17.0	16.9
Membrane:				
Initial pseudo weight (g)	207.2	215.3	279.2	271.8
Final pseudo weight(g)	214.7	222.2	289.3	283.9
Final real weight(g)	406.4	425.8	547.1	534.3
Initial Volume (cc)	199.2	210.5	267.9	262.5
Final Volume (cc)	191.7	203.6	257.8	250.4
Shrinkage	**–3.77%**	**–3.28%**	**–3.77%**	**–4.61%**

Table 20—Neat Holnam H Slurries

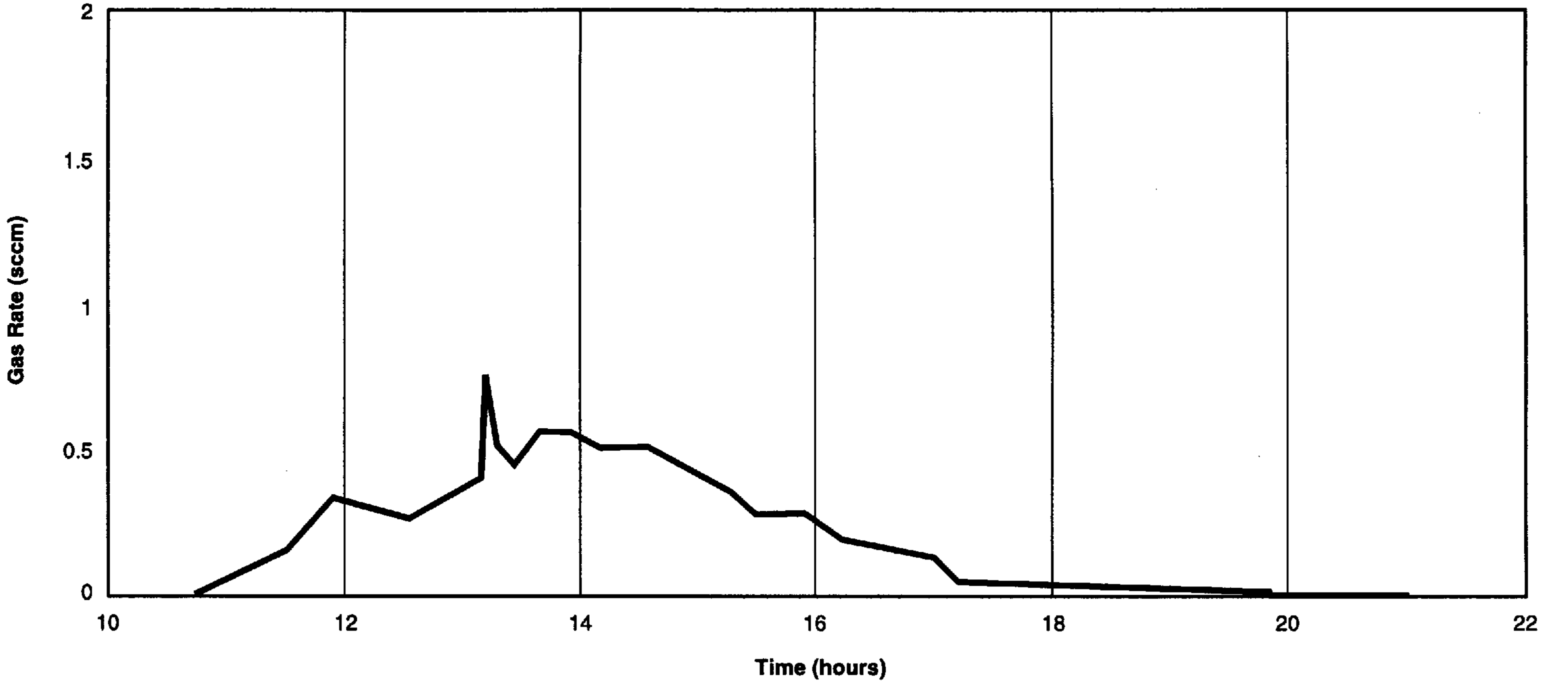
	15.5 ppg	16.8 ppg	17.5 ppg
Slurry:			
Holnam H (g)	758.4	890.6	967.0
DI water (g or ml)	358.0	315.2	292.0
Antifoam (g or ml)	2.0	2.0	2.0
Water/Cement (weight)	47.2%	35.4%	30.2%
Calculated density (ppg)	15.6	17.0	17.0
Membrane:			
Initial pseudo weight (g)	207.5	207.2	240.2
Final pseudo weight(g)	217.5	214.7	244.5
Final real weight(g)	443.6	406.4	456.4
Initial Volume (cc)	236.1	199.2	216.2
Final Volume (cc)	226.1	191.7	211.9
Shrinkage	**–4.24%**	**–3.77%**	**–1.99%**

Table 21—The Dispersion Effect on a Neat Slurry

	Neat	1% BWOC
Slurry Density (ppg)	16.8	16.7
Calculated density (ppg)	17.0	16.7
Cement/total (volume)	47.0%	46.6%
Slurry:		
Holnam H Cement (g)	890.6	890.6
DI Water (g or ml)	315.2	315.2
Antifoam (g or ml)	2.0	2.0
Dispersant (g, ml)	0, 0	8.91, 6.23
Membrane:		
Initial pseudo weight (g)	207.2	265.4
Final pseudo weight (g)	214.7	273.9
Final real weight (g)	406.4	527.4
Initial volume (cc)	199.2	262.0
Final volume (cc)	191.7	253.5
Shrinkage	**–3.77%**	**–3.24%**

Table 22—Microcement

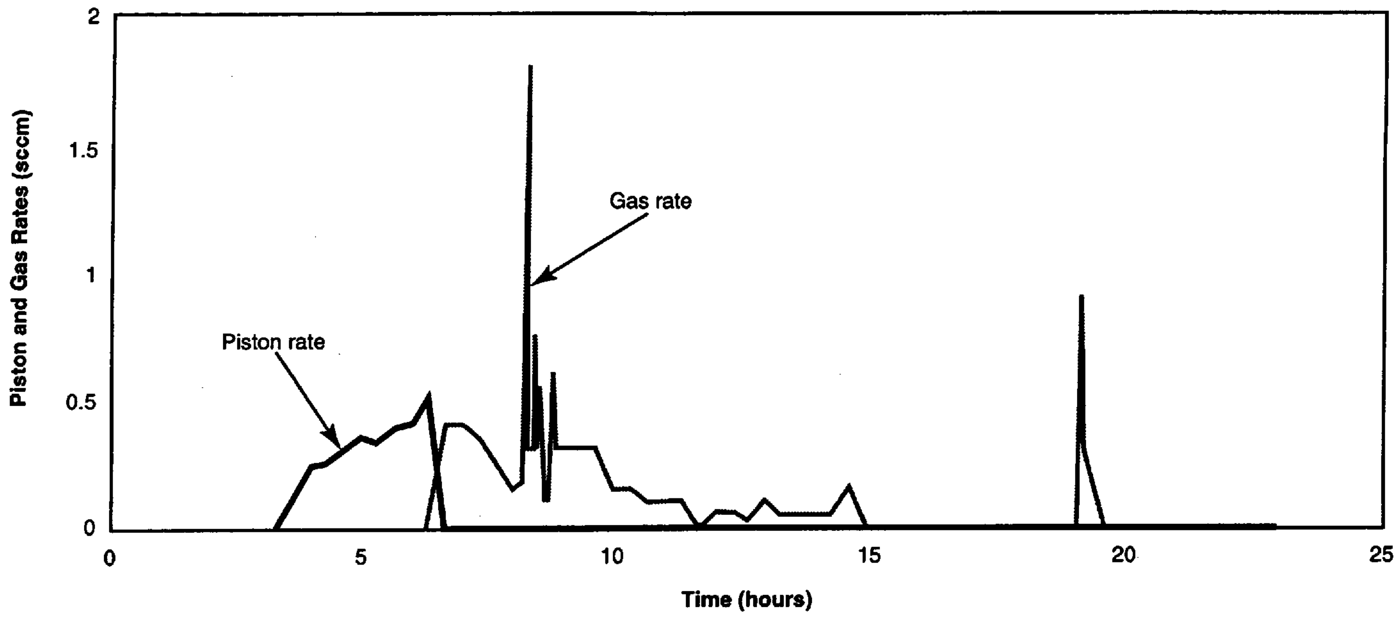
	Holnam H	Microcement
Slurry Density (ppg)	16.8	13.4
Calculated density (ppg)	17.0	13.4
Cement/total (weight)	73.7%	58.5%
Slurry:		
Cement (g)	890.6	566.3 (198 ml)
DI Water (g or ml)	315.2	400.0
Antifoam (g or ml)	2.0	2.0
Membrane:		
Initial pseudo weight (g)	207.2	153.0
Final pseudo weight (g)	214.7	158.7
Final real weight (g)	406.4	402.8
Initial volume (cc)	199.2	249.8
Final volume (cc)	191.7	244.1
Shrinkage	**–3.77%**	**–2.28%**



Volume compensated by gas entry: 8.21 cc

Shrinkage: –2.09%

Figure 9—CHA: Shrinkage Measurement—Test G4



Volume compensated by piston movement: 2.58 cc

Volume compensated by gas entry: 5.71 cc

Shrinkage: –2.11%

Figure 10—CHA: Shrinkage Measurement—Test P3

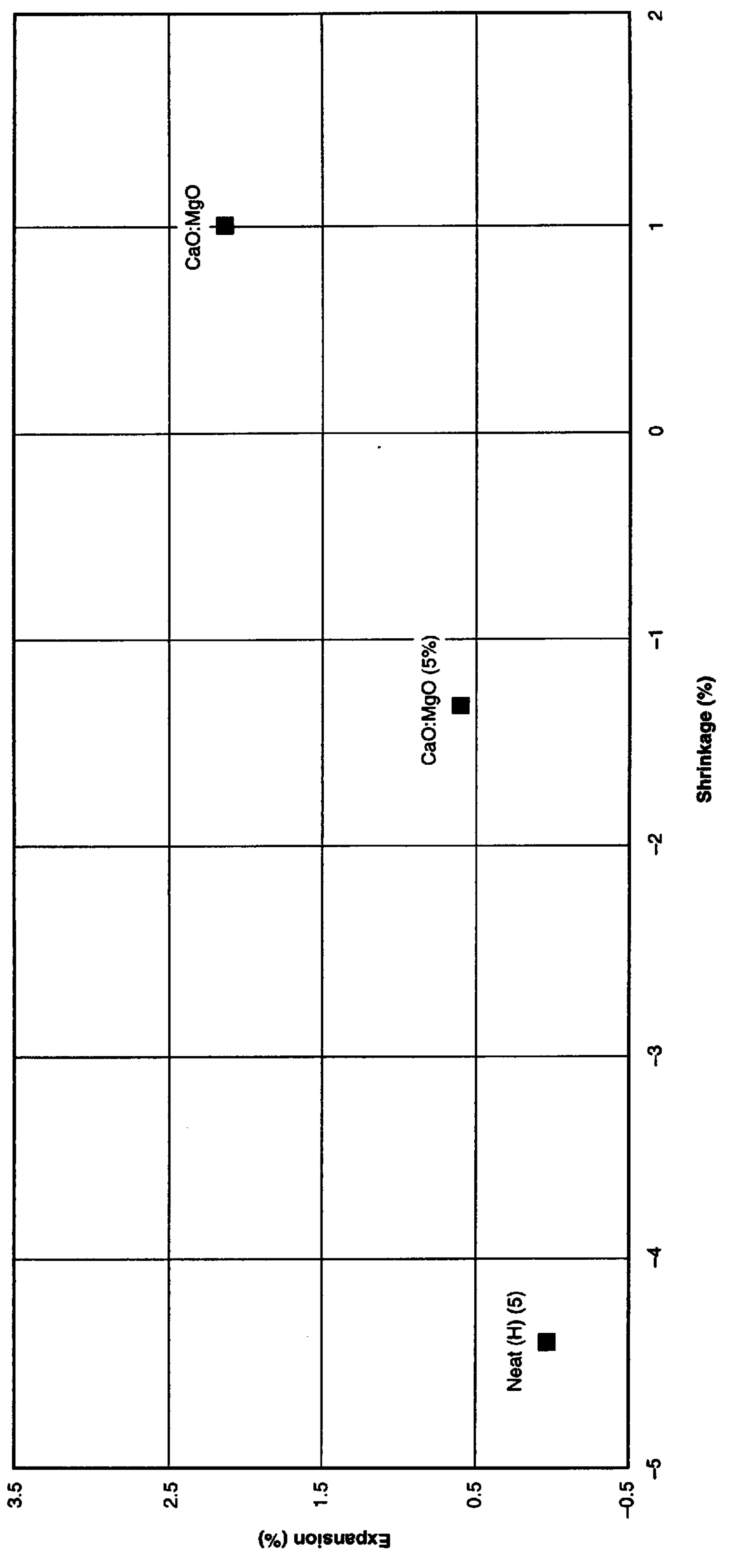


Figure 11—Action of Expanding Agent—Expansion as a Function of Shrinkage

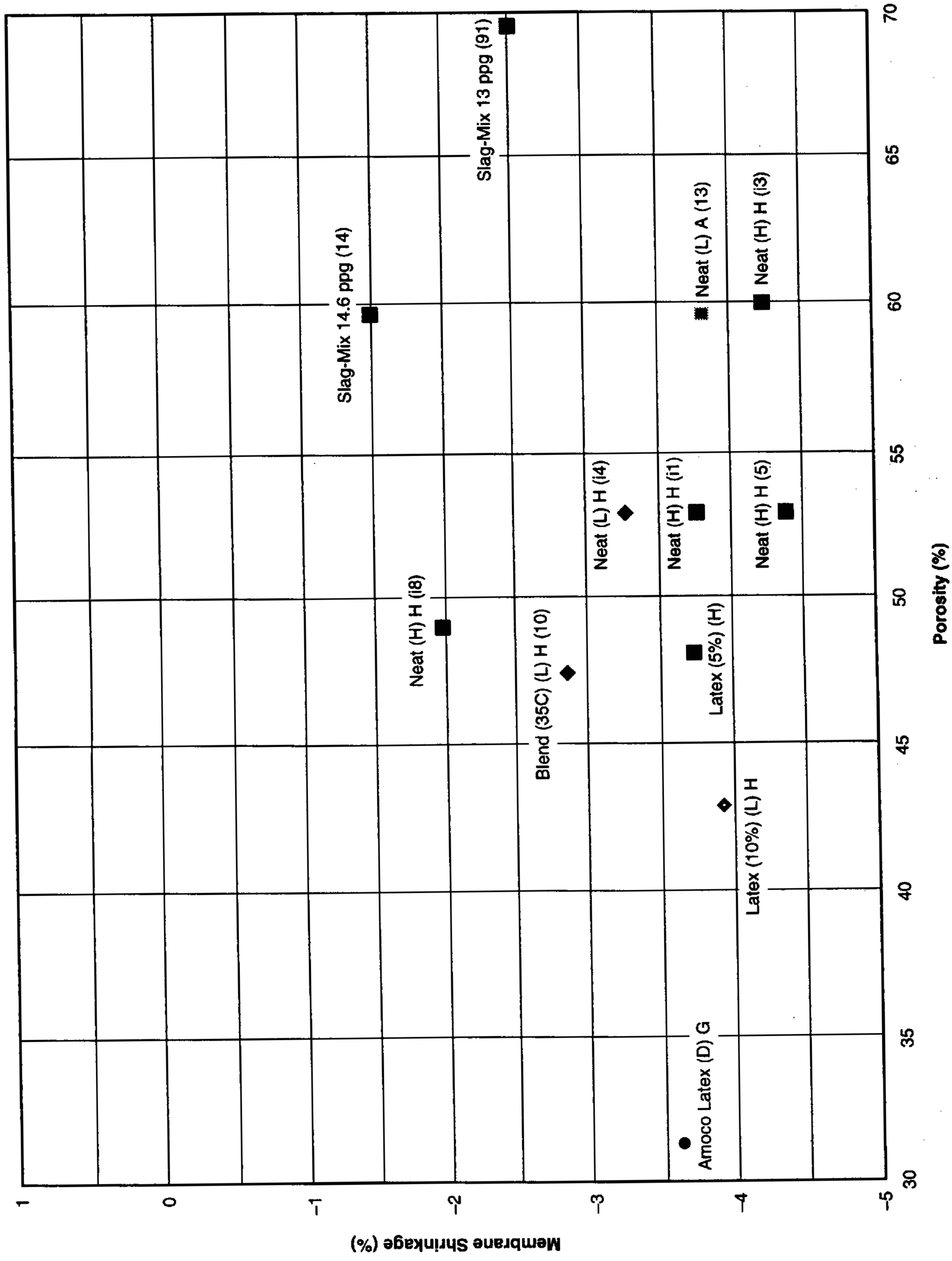


Figure 12—Porosity and Membrane Shrinkage

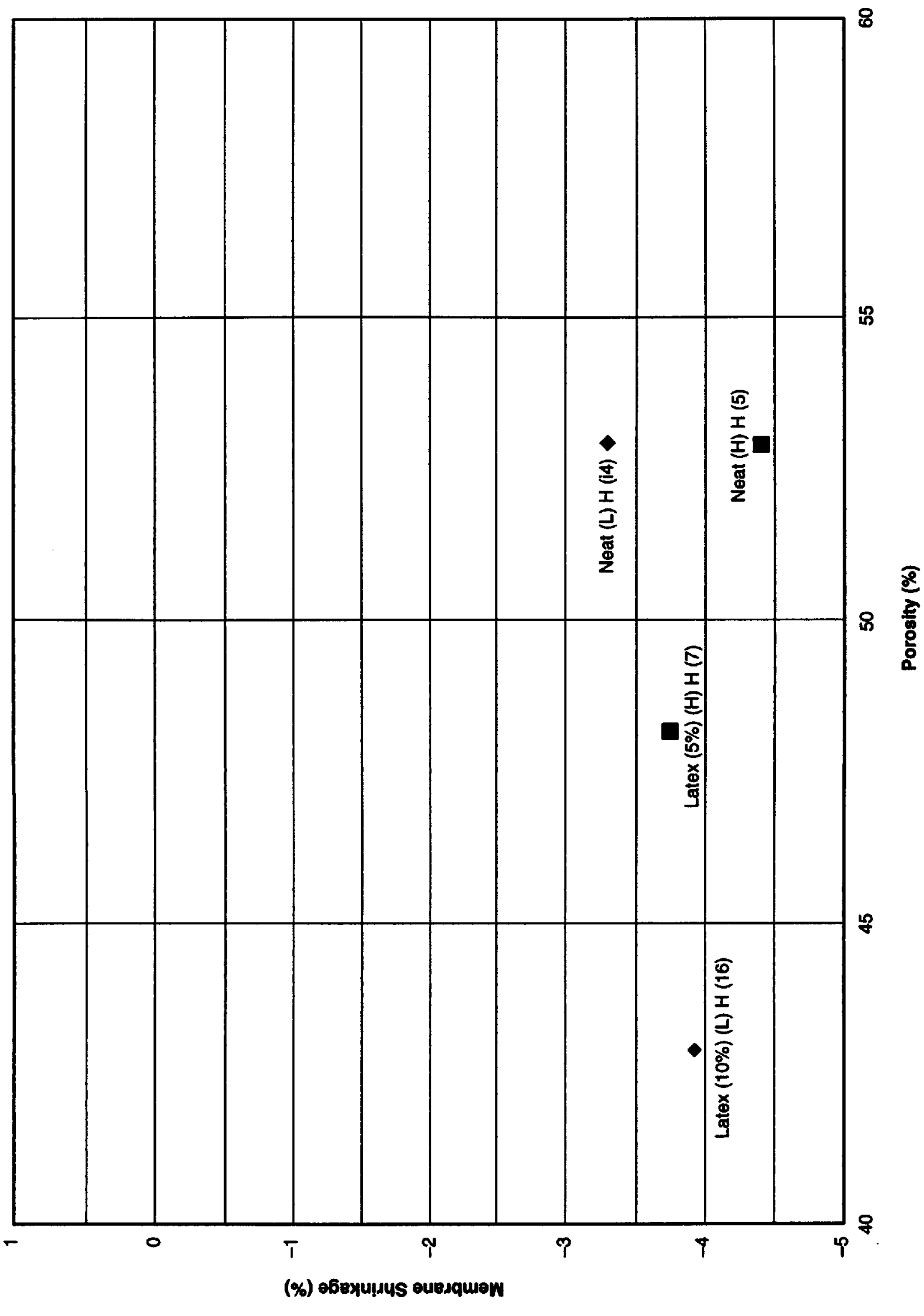


Figure 13—Porosity and Membrane Shrinkage

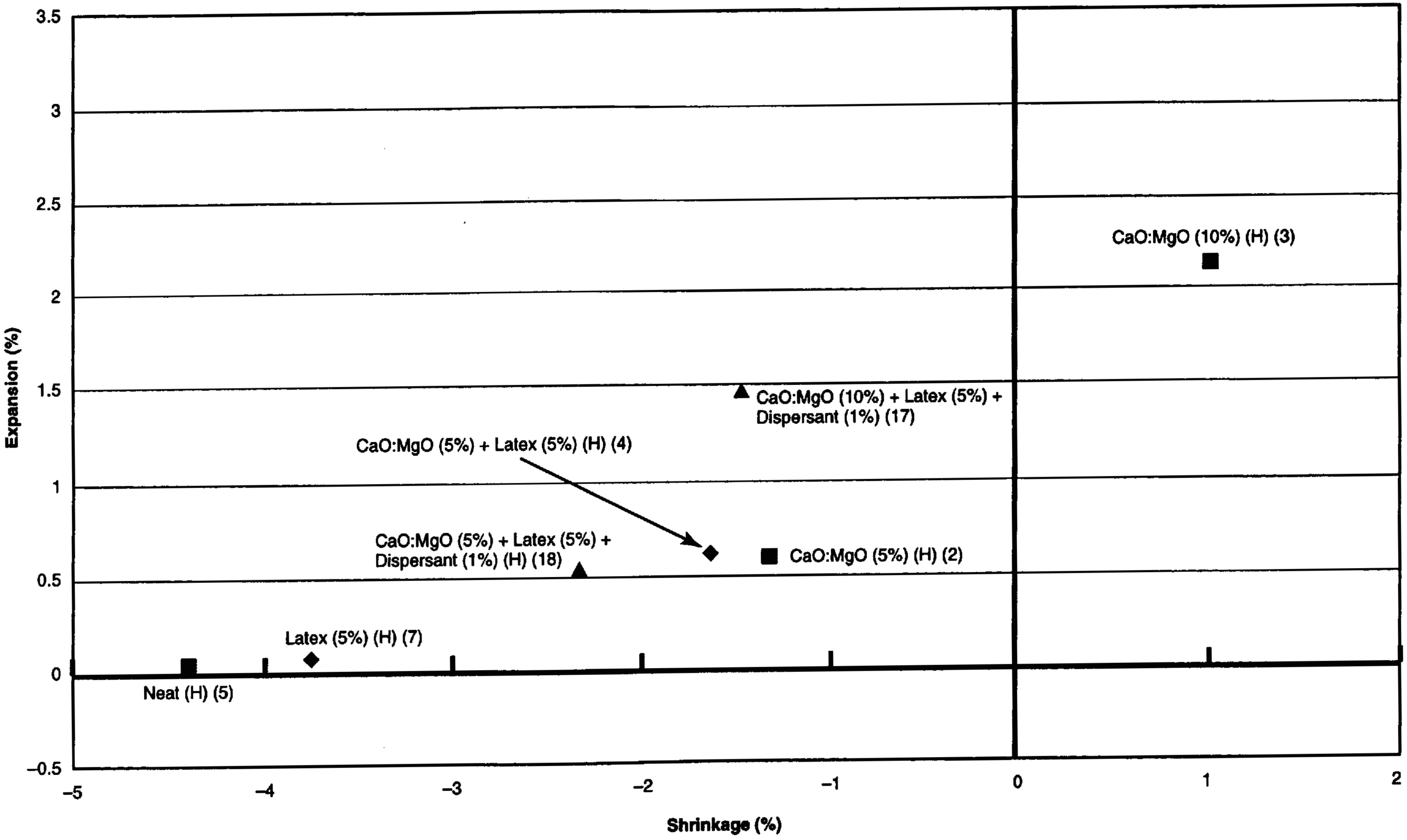


Figure 14—Action of Expanding Agent, Latex, and Dispersant

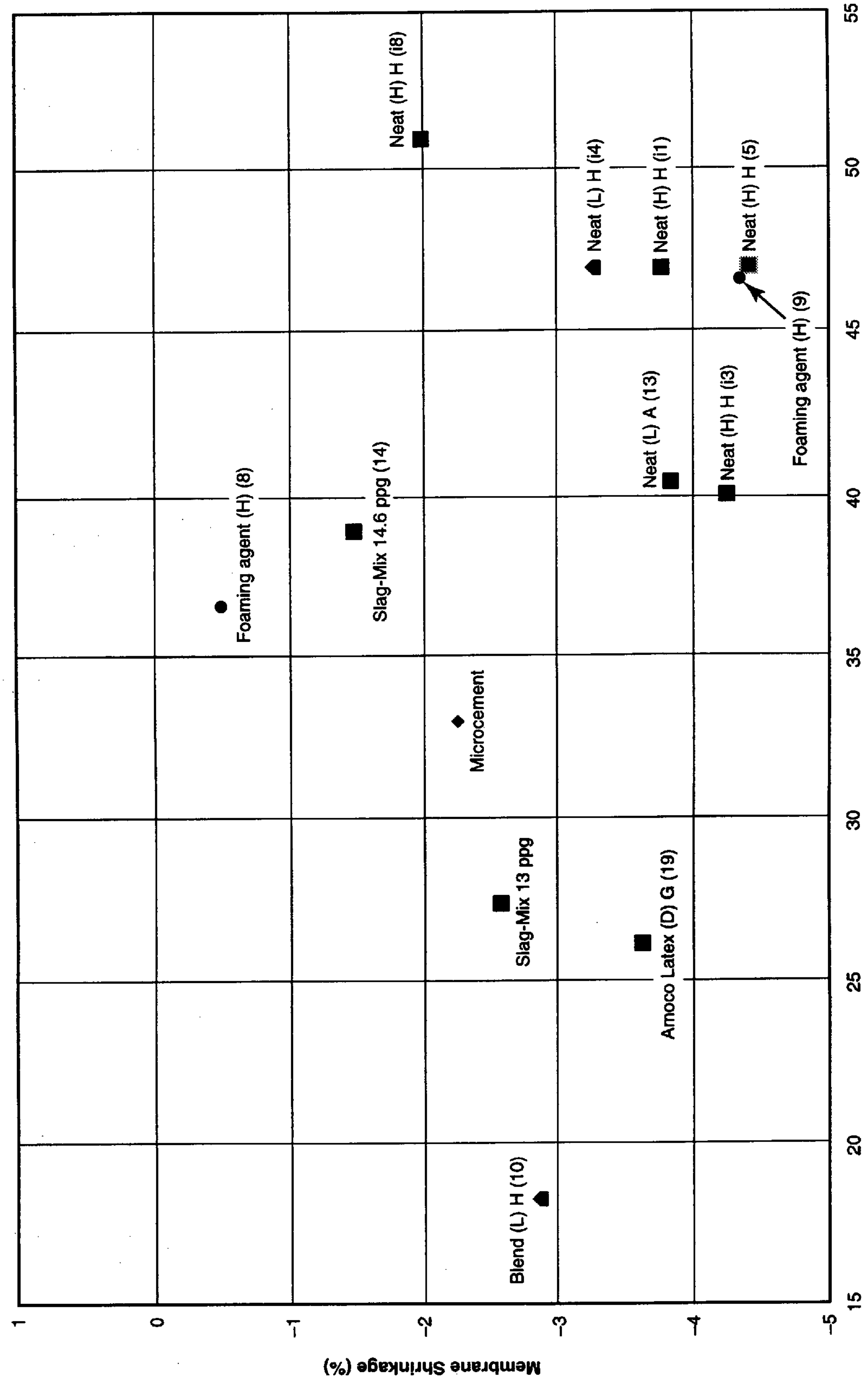


Figure 15—Cement Volume/Total Volume Ratio and Membrane Shrinkage

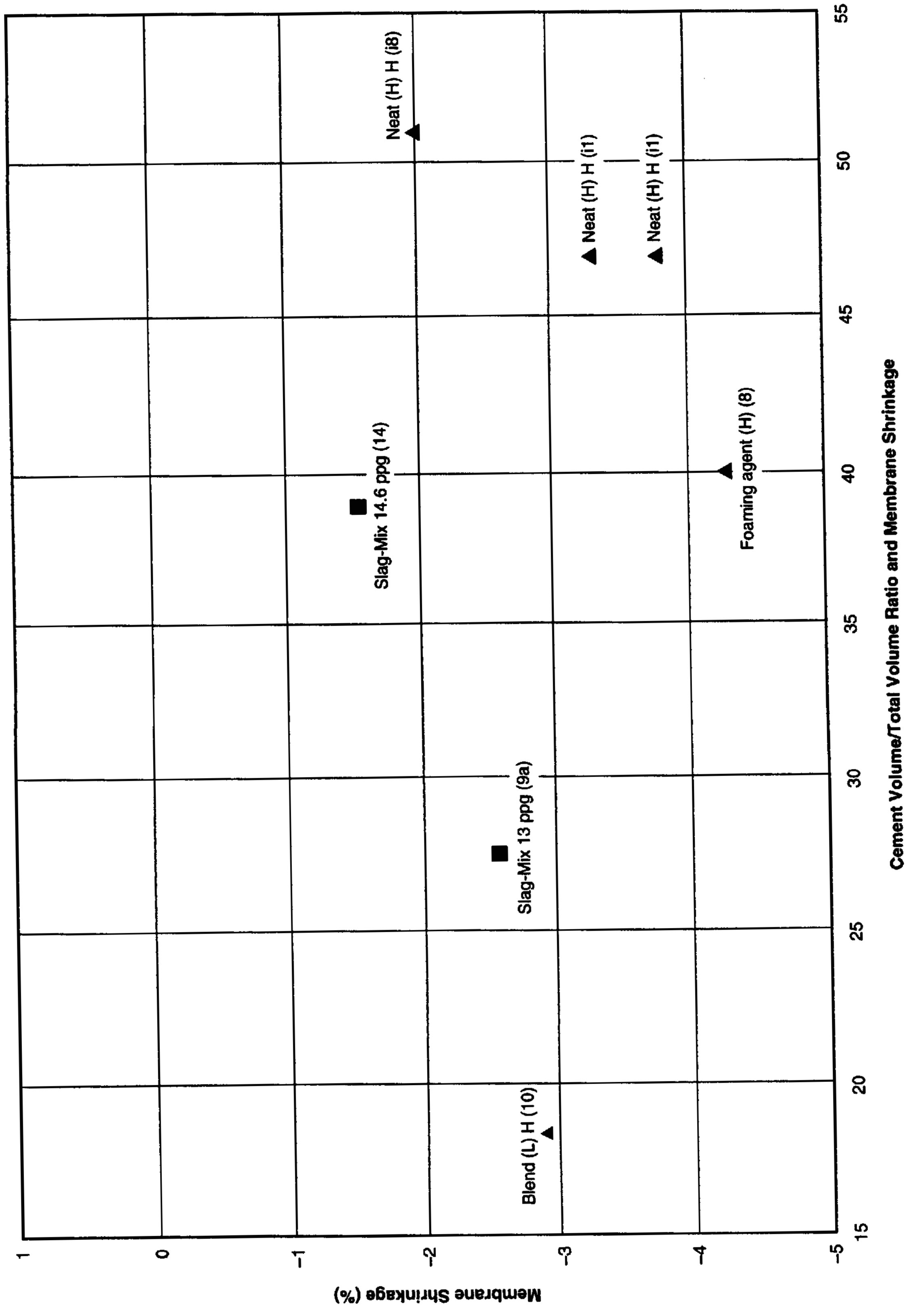


Figure 16—Cement Volume/Total Volume Ratio and Membrane Shrinkage

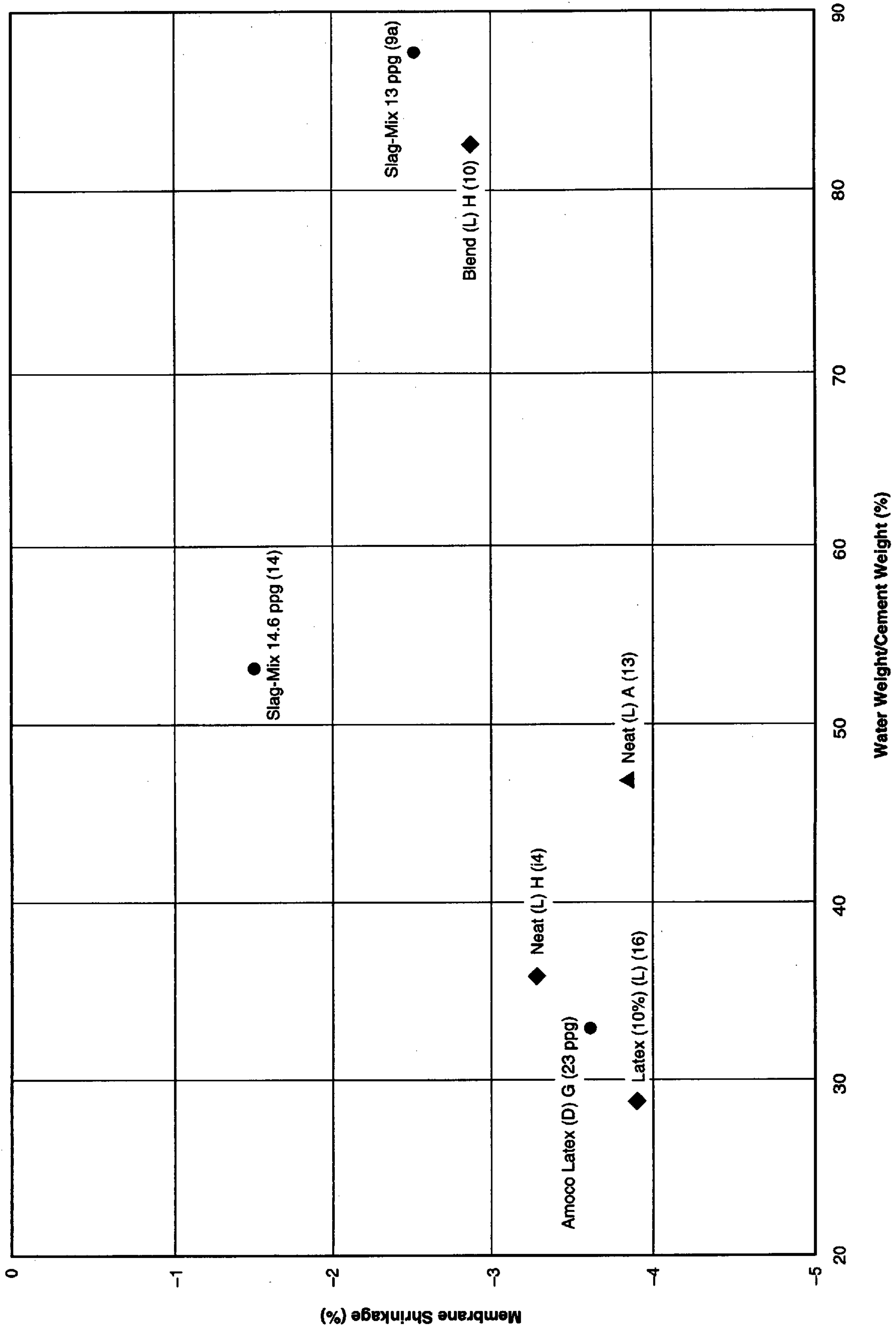


Figure 17—Water Weight/Cement Weight Ratio and Membrane Shrinkage

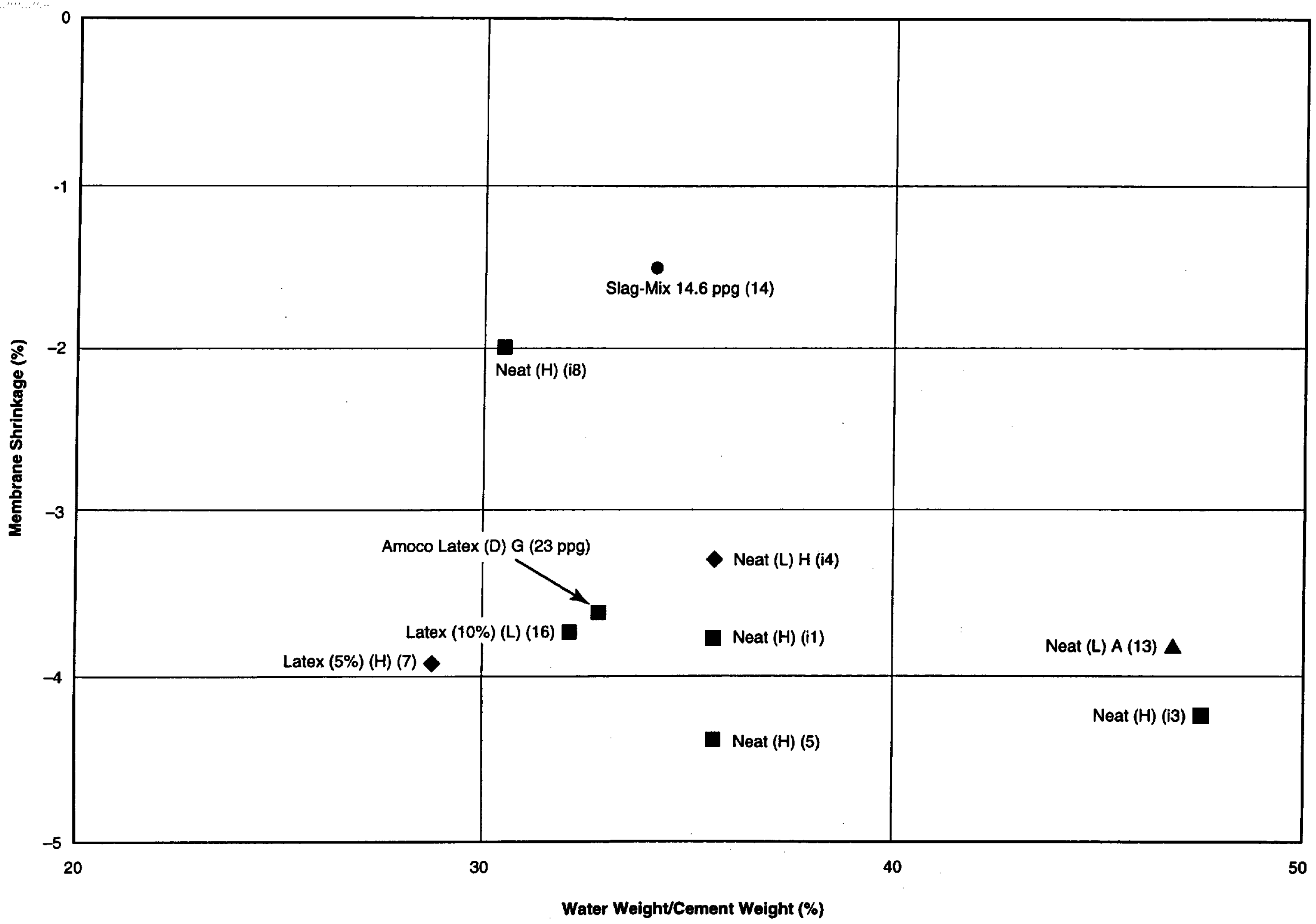


Figure 18—Water Weight/Cement Weight Ratio and Membrane Shrinkage

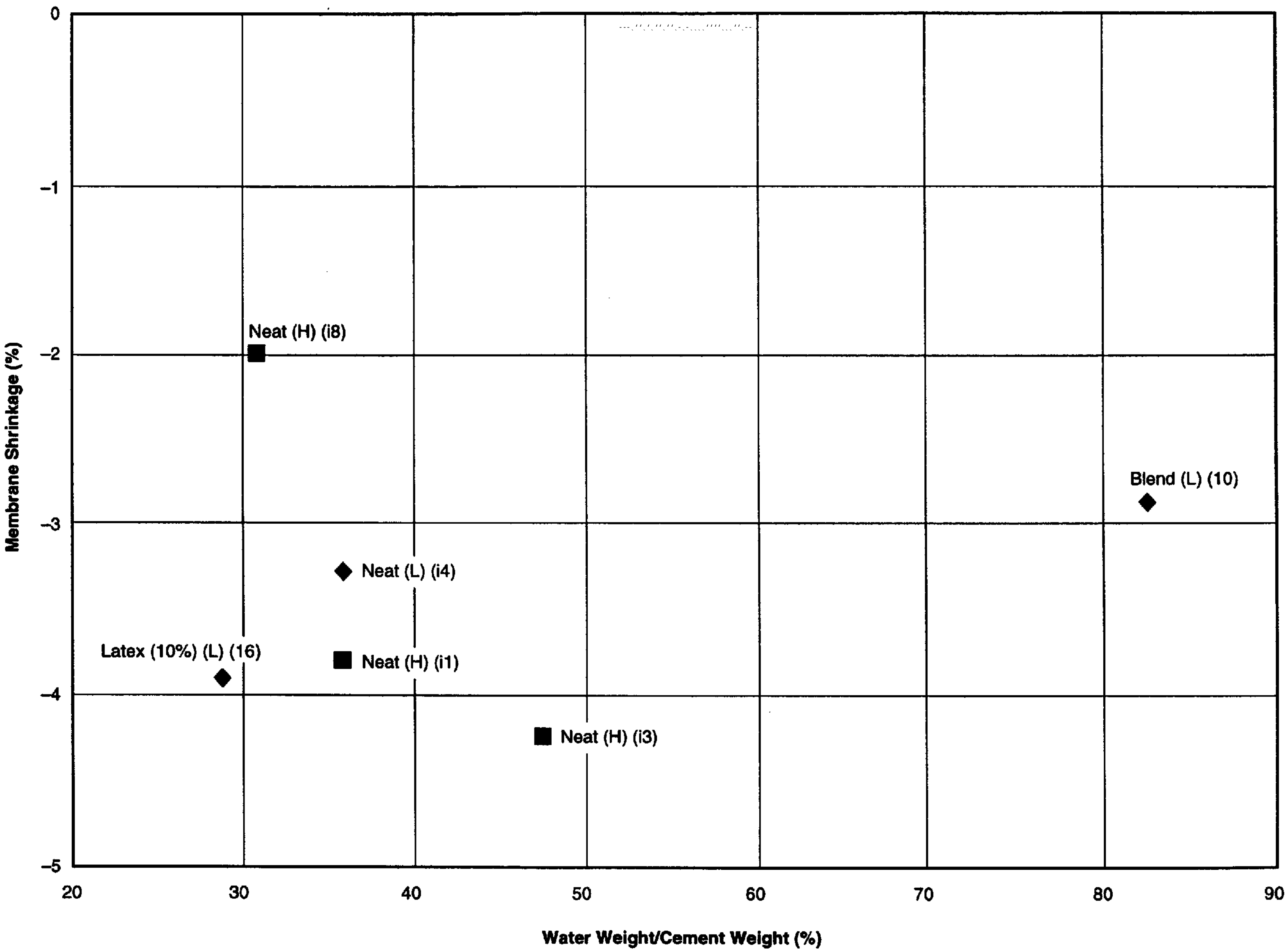


Figure 19—Water Weight/Cement Weight Ratio and Membrane Shrinkage

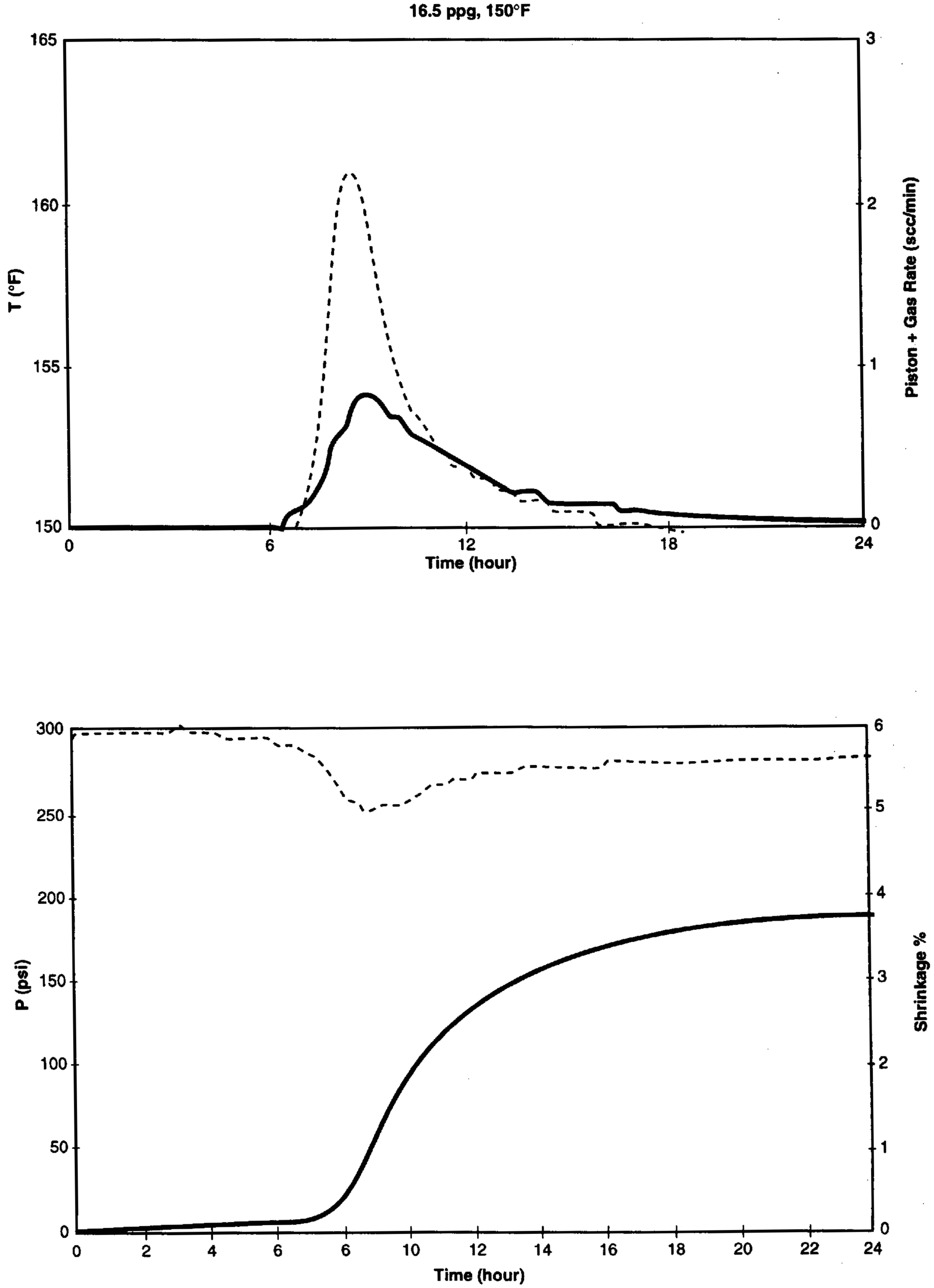
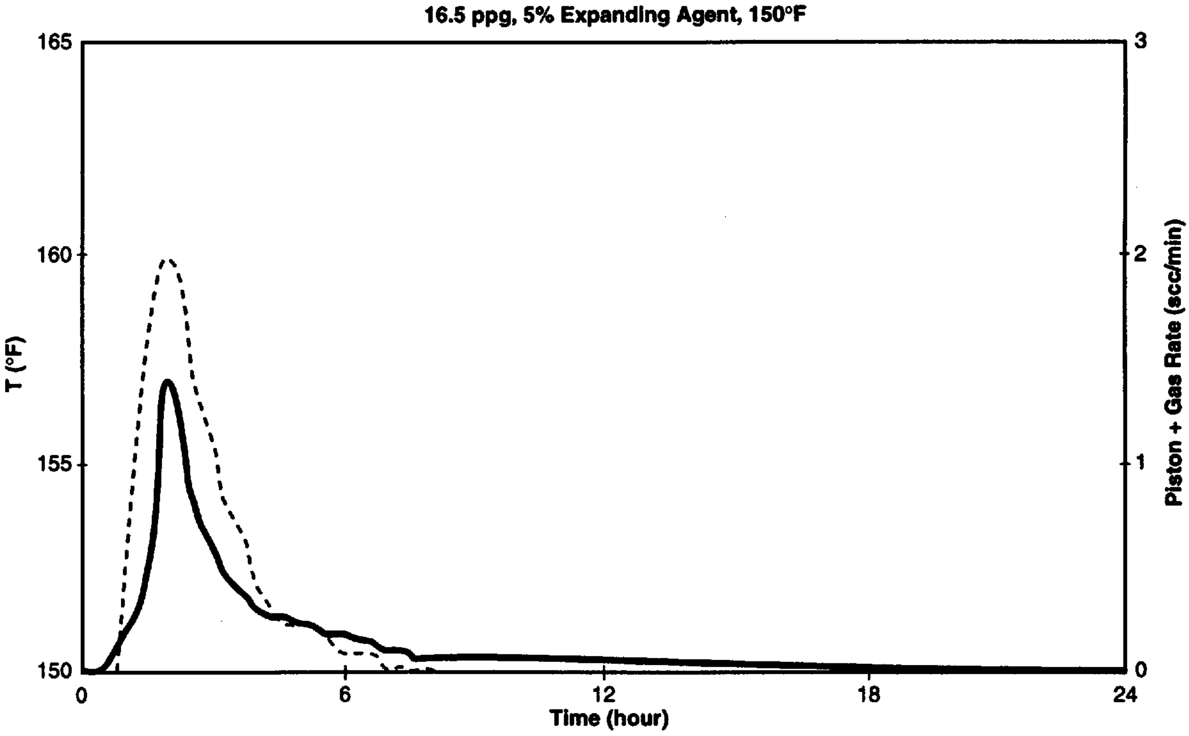


Figure 20—CHA Test, System 1



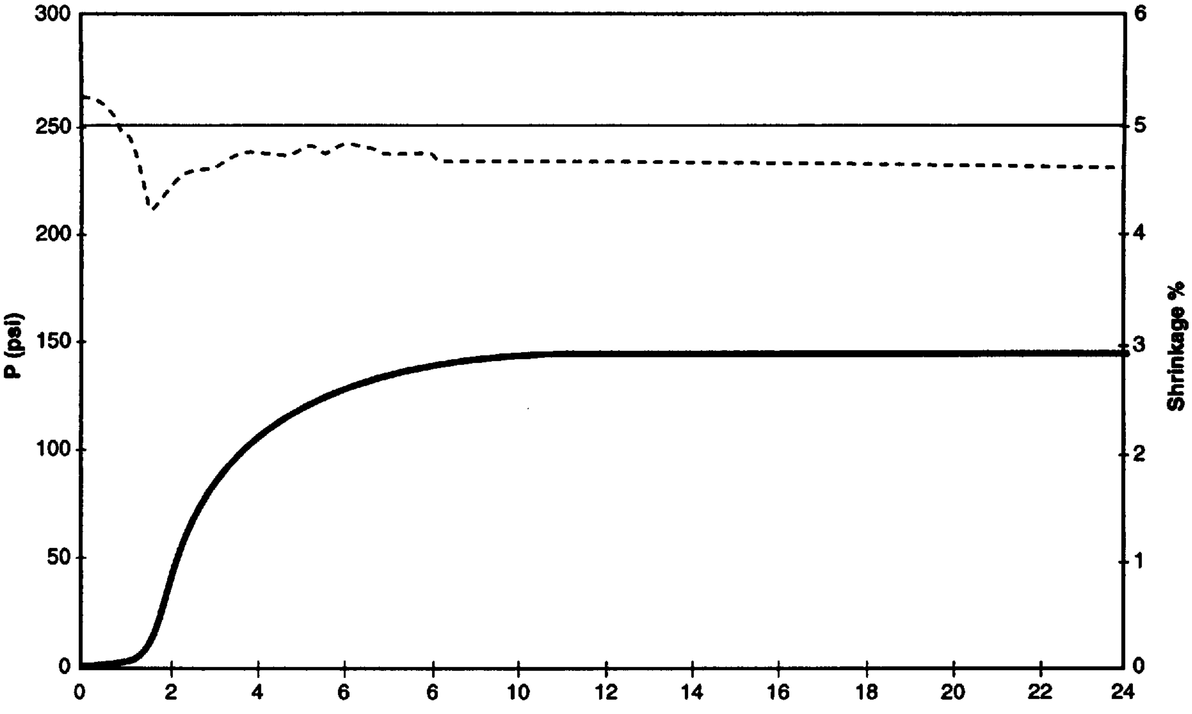


Figure 21—CHA Test, System 2

APPENDIX A—SUGGESTED REFERENCES

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