CADE/CAODC SPRING DRILLING CONFERENCE
April 19, 20, 21, 1995
Calgary, Alberta Canada

TITLE: TESTING SYSTEM TO EVALUATE THE RESISTANCE OF CEMENT SLURRIES TO GAS MIGRATION DURING HYDRATION

PAPER NO. 94-405

AUTHOR(S): John Jamth, Veba A/S, Stavanger, Harald Justnes, SINTEF Structures and Concrete, Trondheim, Nils Egil Nødland, STATOIL, Stavanger, Pål Skalle, U. of Trondheim, Jostein Sveen, SINTEF, Safety & Reliability, Trondheim

ABSTRACT

Gas leaking in the annulus after cementing is still a problem, especially in shallow permeable gas sands.

To examine the ability of different cement slurries to withstand gas migration during hydration, a new test rig was developed over a period of 5 years. The purpose of this rig is to simulate different conditions which might be encountered in oil and gas well, under different pressures, temperatures and angles. The cell consists of a 2 m long pipe with 102 mm ID which can be run at up to 200°C and 69 bars). Hydrostatic pressure decline, heat evolution and gas flow into and out of the slurry are recorded. Recorded data and slurry characteristics are used for interpretation.

Several slurries have been tested in the rig. The slurries were choosed for their gas migration potential, ranging from very simple leaking slurries to gas tight commercial slurries. Also, variations of the amount of additives were tested. The rig proved to be able to determine if a slurry is leaking.

The rig has already been used by several oil and service companies with success to determine the gas flow hazard of slurries. When exploring for oil and gas, this is a guarantee for operators that the cement slurry is satisfactory. The rig offers the oil industry a test process which simulates the conditions expected when cement slurry is used for borehole cementing .

INTRODUCTION

A severe problem in cementing gas wells is gas migration through cement immediately after cementation, in particular in shallow permeable sands. Reasons and mechanisms of gas migration are presented among others by Levine et al[1], Sabins et al[2] and Cheung & Breirute[3]. Annular gas flow may occur when a) the hydrostatic pressure falls below the formation pore pressure due to shrinkage, fluid loss or gel strength build up and b) if the cement slurry stays or becomes permeable for some reason; initial gas forms channels, created micro annuli or stress created micro fractures before the slurry has reached a certain strength. Thus, the phenomenon of gas

migration is believed to occur during the transition state between initial and final set of the cement when the pressure transferred by the cement slurry column changes from being given by the density of the cement slurry to the density of the percolating water phase, or even less.

Many attempts have been made in trying to simulate self induced flow of gas from the formation into the slurry. Trying to reproduce field conditions in the laboratory is very difficult due to the ratio aspect of a well's height vs. its diameter, which governs the pressure decrease in the well.

The objective of building a physical model of a cemented annulus was to study the slurry's ability to withstand gas intrusion and migration during hardening under HTHP conditions, by monitoring gas out and into the slurry, when and how much gas is flowing, hydrostatic pressure behaviour and temperature evolution.

EXPERIMENTAL SET-UP

Test equipment

The HTHP gas migration system shown in Fig. 1 and 2, consists of a tube with a length of 2000 mm and an inside diameter of 102.26 mm, and a wall thickness of 6.02 mm, certified for 2800 psi (19.3 MPa) internal pressure. The tube is placed inside a pipe with larger diameter through which hydraulic oil is circulated for heating the inner tube. The heating temperature ranges up to 200°C, while the upper operating pressure limit is 1005 psi (6.93 MPa). During all tests the cement column was pressurized to 1000 psi (6.90 MPa). The test tube may be inclined from 90 to 0°.

Prior to this work, Kalvenes et al[4] reported on tests made on the Aker rig which was the processor of this rig. The Aker rig was not so advanced in many ways, as there were problems with pressure and temperature control, and also the insulation at the ends of the cell were insufficient. In spite of the controlling problems, the Aker tests gave valuable information. When designing the new HTHP gas migration system the problem of low cell height (2m) was solved by controlling the differential pressure in a separate control line and by applying a differential pressure transducer, allowing control at extremely low differential pressures (0.05 psi/0.3 kPa). Differential pressure (up to 15 psi/0.1 MPa) was controlled at the nitrogen gas inlet, situated 200 mm above the bottom of the test tube leading into a 300 mm high, porous, radial gas-distributing filter. The accuracy of the differential pressure transducer was +/-0.05 psi (0.3 kPa). Temperature was controlled according to a chosen scheme simulating the cementing job. Data measurement were logged each 10th second and the parameters were stored on PC-files. After each test the data were printed out automatically as a report, including trend curves.

Calibration and testing procedures

After mixing the slurry with a controlled amount of shear, si-

mulating both mixing and pastly pumping through the casing, the slurry is pumped into the pipe and heated according to the appropriate API schedule. The cell was calibrated using water and neat cement slurry, the latter case shown in Fig. 3. The pressure stayed relatively constant until initial set was reached. Then it dropped quickly, proceeded by a small pressure increase. As soon as the cement slurry hydrostatic pressure falls below the gas pressure, gas starts to flow into the slurry, and the magnitude of the flow is proportional the pressure difference. At the top of the cement column, gas is coming out through the slurry. The slurry was proven untight against gas intrusion.

Cement slurries used

In this work gas tight cement slurries were tested. Gas tight slurries were defined by Hibbeler et al[5] as:

1. Low gel strength < 10 lb/100 ft²
2. Fluid loss control < 50 ml/30 min
3. Low free water = 0%
4. Main transition (40-70 Bc) < 20 min (right angle set)
5. Staggered thickening time > 1 hour diff. between lead and tail slurry

Additional design criteria are Stability (BP setting test) and restrictions in amount of gas entering or leaving the slurry during the test described in this paper. To obtain such slurries typical remedies are to add certain amounts of Microblock or Latex.

After calibration with water and neat cement slurry, 9 different gas tight cement slurry compositions for HTHP conditions were formulated An overview of the slurries is shown in Table 1.

RESULTS

In Fig. 4 two representative tests of the 9 are selected and presented, showing the four most interesting parameters as a function of time:

- hydrostatic pressure
- temperature
- gas flow rate in
- gas flow rate out

Test 6 and 9 were running for 12.5 and 8.2 hours after mixing. The slurries gelled quickly and hydrostatic pressure stabilized at 0.5-3 psi (3-21 kPa) above the gas pressure in the bottom inlet. When the temperature gradient increased the curing started, occurring simultaneously with a drop in hydrostatic pressure. The pressure stayed relatively constant until it dropped quickly, proceeded by a small pressure increase. In test 9 some gas was seen coming out temporarily.

In Fig.5 the relative variation for all 9 tests are gathered in an overview graph. The maximum temperatures usually coincided with the maximum rate of heat evolution dominated by cement hydration.

In tests no. 1 and 4 a too high differential pressure resulted in early inflow of gas before the actual test started. In such situations the differential pressure had to be reduced in order to accurately counterbalance the pressure exerted by cement slurry column (i.e. ρgh), and gas flow was stopped immediately.

In test no. 1 the differential pressure was accidentally set just above the hydrostatic pressure of the cement. The flow stopped immediately when it was adjusted below this level. The flow in test no. 4 was caused by a fast decrease in the hydrostatic cement pressure which was not accompanied by a sufficient gel strength build up. In test no. 7 small amounts of gas entered the slurry and some time later it came out at the top at a maximum rate of approximately 10 ml/min.

DISCUSSION

The hydration process is in most cases a process with repeatable or predictive behaviour. When deviation from the expected behaviour is seen, an explanation should be sought. The knowledge gained through these studies may then later be applied to explain why some slurries were not gas tight.

1) Heat evolution

The slurry was gradually heated during the experiment, simulating temperature increase due to downhole pumping.

Slurries 1, 2, 3, 4 had a density of 2.05, while slurries 5, 6, 7, 8 and 9 had a density of 2.15 g/cm^3 and respectively different API temperature schedule. The resulting temperature profile for these two groups were distinctly different. The 2.05 group exhibited smooth curves with a distinguished maximum, the 2.15 group had a temperature-peak prior to a flat maximum. The flat temperature profile and the "false" T-peak may mislead an observer and cause the test to be ended too early. This, however, was double checked by testing the compressive strength of the cured cement after the test. In all cases it was found that hydration had proceeded satisfactorily.

Temperature peaks before the onset of the hydration process, like the ones seen in tests no. 5, 7, 8 and 9, are probably caused by the content of retarders exceeding what is required for the initial complex formation on the outer surface of the cement grains. When the first layer breaks up, a new retarding surface complex (exothermal reaction) is formed by the excess retarder in solution, rather than starting the hydration. Thus, this temperature profile may be typical for slurries with too much retarder.

The abnormal flat temperature profile or lack of the normal temperature peak seen in tests no. 5, 6, 7 and 8 may be caused by large amounts of silica powder. Silica powder reacts with alkalis and lime (products of the cement hydration). This is a slow reaction with a low exotherm heat evolution, and causes the silica to lower the overall hydration temperature due to the lesser cement content pr. slurry volume able to counteract the heat dissipation. Too much silica may therefore cause the slurry to have a low mechanical strength during the critical time gap. In spite of low strength, the permeability may be less if the silica particles are much smaller than the average cement grains (e.g. condensed silica fume). If a similar

amount of latex is used instead of silica, a much quicker temperature increase is observed, almost like a flash set. The latter effect may partly be due to an insulating effect caused by the low heat conductivity of polymers compared with minerals.

2) Hydrostatic pressure

From the experimental data it is seen that initially, the hydrostatic pressure is equal to the total pressure of the cement column plus the test pressure. The hydrostatic pressure decreases during the entire test. Pressure decline is a reaction of shrinkage and the gelation process is the normal behaviour. From these tests and previous tests it was seen that gas usually entered the slurry when the hydrostatic pressure started to fall dramatically. The hydrostatic pressure reduction in a cement slurry coincide with the hydration / gelation / shrinkage process (or immediately afterwards). In this period of the hydration process, the gas will enter a gas leaking slurry, while a "gas tight" slurry will resist gas intrusion. In these tests the initial differential pressure was always 2-4 psi (14-28 kPa).

In some slurries the pressure behaved abnormally by exhibiting a pressure increase just prior to reaching the maximum temperature. In almost all cases this was interpreted to be caused by temperature increase. The slurry sticks to the wall when the gel strength builds up creating compression when the cement expands thermally.

After the normal pressure decline has occurred, the pressure will sometimes be restored to a value below the original (see fig. 5). The most probable reason for this pressure equalization is a very small amount of gas flowing through:

- micro annulus formed at the outer boundary of the slurry
- micro cracks formed internally in the matrix
- pores within the slurry itself (hydraulic flow by percolation)

3) Gas in/gas out

Gas flow was seen in five tests. Two of them (1 and 4) produced in- and out-flow from the start of the test. In test no. 1 this was caused by applying a too high differential pressure just above the cement hydrostatic pressure of 5.05 psi (35 kPa). Lowering the differential pressure immediately stopped the flow. Density measurements after mixing of slurry no. 4 showed an average density of 1.99 instead of 2.05. This type of instability and gel build up made the cement pressure in the bottom lower than expected, and hence the flow started. Decreasing the differential pressure again prevented further flow. In addition, the slurry was unstable as free water was observed on the top of the column after the test.

Test no. 7 was the only slurry that showed a "normal" leaking behaviour as reported by Kalvenes et al[4]. The flow starts when the cement pressure decreases 5.5psi (38 kPa) below the bottom gas pressure. This point of time also coincide with the maximum slurry temperature. Gas out-flow is delayed by

approximately 100 minutes and after further 2 hours the maximum flow-rate (9ml/min) is reached for both flows. Slurry no. 7 and 8 were identical but test conditions were different. At an early stage in test no. 7, a forced gas flow test were performed, causing some gas to enter the slurry (see fig. 5). Slurry no. 8 is apparently gas-tight. The minimum cement pressures were 12 psi (83 kPa) and 8 psi (55 kPa) below the gas pressure for no. 7 and 8 respectively.

In two of the tests, 8 and 9, there were gas out-flow almost from the start which, however, stopped after some hours. Most probably this was caused by an incompletely filled cell, i.e. a few centimetres at the top were nitrogen filled and this gas expanded as the cell was heated. This explanation is based on the fact that the flow begin some time after the heating starts and stops when the heating oil reaches BHST. As there is no information on this gas volume, it may be that in the 7 first tests the cell were completely filled. Even if this is the main contributor, there exist several other explanations that may vary from one test to another:

- Air bubbles trapped in the slurry during mixing and the solubility of air in water decreases with increasing T. When the temperature increases some of the slurries give off gas temporarily for several hours.
- Same as the above but amplified by effects through the hydration process. Water is a solvent for air. Thus, when water is consumed during hydration the gas-phase concentration increases.
- Water evaporated from the free water on top of the slurry column.
- Organic additives liberate hydrocarbon gases when heated. This has been checked by separate heating tests followed by an analysis of the gases coming out of the slurry. All gases were of organic origin. However, the test temperature was then higher than the usual HTHP test temperature.
- Condensed silica fume may contain small amounts of different metals such as Si, Al etc. These metals react with the alkalies from the cement and produce hydrogen gas. Any rests of metal could be detected through simple tests (boiling in NaOH). As an indication of the above, an expansion effect is seen when silica is added in the slurry. The slurry´s surface tended to create a convex surface (seen after end of test).
- Large amounts for condensed silica fume (e.g. 35%) may in particular at elevated temperatures temporarily form an unstable, expanding silica hydrate gel low in calcium partly due to the lower solubility of calcium hydroxide (cement hydration product) at increasing temperatures (anomaly).

In the previous cell, Kalvenes et al[4] observed that in some cases gas entered the slurry but did not come out. This may indicate a partially leaking slurry and be related to:

- Shrinkage and pressure decrease are compensated for

by gas flowing into the slurry. The driving force is thus eliminated or insufficient to support any further gas influx.

- Gel strength is building up during the influx period (which may last from several minutes to hours) finally creating sufficient resistance to flow.
- Local micro annulus and/or channels generated on the slurry surface may store some gas volume before they close.

4) General observations

The 9 slurries were remixed and run through standard API tests by Øvrebakk and Nødland[6]. In addition they found;

Complicated slurry mixtures with 6 - 8 different additives may easily lead to mixing problems and difficulties in predicting the effect of small variations in amount of additives. For instance, some of the retarders gave large setting time variation for small changes in amount. This is unacceptable, since this demands high accuracy in the weighting procedures. Some additives should therefore be easier to handle if they were supplied in diluted solutions.

Some retarders did not function satisfactory above 100 °C although the service company claimed they should. Mixing procedures and additives gave unacceptable levels of air/gas, and some slurries showed tendencies of settling. The BP-settling test is recommended for gas tight slurries above 100 °C.

POSSIBLE TEST IMPROVEMENTS

Based on the above discussion there seems to be a need for improvement both with respect to the test procedure itself and by supporting the results with additional tests in order to improve the understanding of observed phenomena's.

Procedures

It was not practical to simulate the actual mixing/pumping time in the HTHP gas migration system. Instead "Added shear during mixing" at 1450 RPM was calculated. Temperature increase during mixing was also recorded.

In order to simulate the real cementing process, which is the combination of a dynamic and a static process, it may be necessary to simulate pumping either through shearing or pumping. The setting process of a static slurry is significantly different from that of a continuously sheared slurry. This fact is seen from the consistometer data; the 70 Bc thickening time is usually reached long before the hydration is completed in the static case. Standard/controlled procedures have been worked out consisting of a detailed timetable for the whole mixing/pumping/static process.

Additional tests

Through the present study it was found that additional parameters are of importance when gas leaking slurries are to be characterized;

A test to determine the external and total chemical shrinkage[7] would help explain the decline of the hydrostatic pressure; i.e., determine the volume of contraction pores (internal chemical shrinkage) in order to estimate the gas capacity of the slurry.

A simple test to determine organic gas production or metal reminents in the condensed silica fume will point out if there is any chance of gas being developed in the slurry. This gas may act as an initiator for channel generation.

All tests that give additional information of the hydration and processes related to gas migration phenomena must be performed in order to predict the gas migration hazard.

Also, the reproducibility should be checked by running e.g. every 10th experiment twice.

The evolution of tensile strength and permeability provided useful additional information[8].

CONCLUSIONS

A new HTHP gas migration test facility has been developed to simulate the interaction between cement slurries and gas bearing formations in deep wells. The facility were applied to study gas migration in cement slurries.

Of the 9 slurries that were supposed to be gas-tight, only one was partially leaking. This is probably caused by the cement pressure decreasing so much that the resistant forces in the cement (gel strength/tensile strength/friction forces in the capillary system) are exceeded and gas breaks into the cement.

In two of the tests, adjusting the differential pressure down caused flow to stop. This indicates that the two slurries are able to heal themselves to withstand later in-flow. The two last tests showed an outflow from the start. Apart from gas expansion, this may be explained by mixed air and production of organic gases inside the cement. This may partly be a cause of gas migration. Slow reactions caused by the effect of silica or other major additives replacing cement will probably lead to a weak cement during the critical time gap.

The test procedure could be improved through adjusting the mixing procedure to also simulate the pumping process. Additional tests like shrinkage behaviour, gas formation within the slurry and calorimetric investigation of retarder effects could reveal otherwise unexplainable test behavior.

ACKNOWLEDGEMENT

The authors would like to extend their thanks to the sponsors of this work: the Royal Norwegian Council for Scientific and Industrial Research (NFR), Norsk Hydro as, and Saga Petroleum as and their representatives Svein Joakimsen, Øystein Kalvenes and Jan Erik Olvin for their personal engagement and contributions.

REFERENCES

1. Levine, D. et al., "Annular Gas Flow After Cementing: A Look at Practical Solutions", SPE 8255 presented at 54th Ann. Fac. Techn. Conference in Las Vegas (Sept. 23-26, 1979)

2. Sabins, F., Tinsley, J. and Sutton, D.: "Transition Time of Cement Slurries between the Fluid and Set State", SPE 9285 presented at the 55th Annu. Fall Technical Conference (Sept. 21-24, 1980)

3. Cheung, P. R. and Beirute, R. M.: "Gas Flow in Cements", SPE (June 1985)

4. Kalvenes, Ø., Berge, L. and Tydal, T.: "HTHP-Gassmigrasjon", R-046338, Norsk Hydro, Research center, Bergen (Feb. 1991).

5. Hibbeler, J. C., DiLullo, G. and Thay, M,: "Cost-effective gas control: A case study of surfactant cement", paper SPE 25 328 presented at the SPE Asia Pacific Oil & Gas Conference, Singapore (Feb. 8-19, 1993)

6. Øvrebekk, R. and Nødland, N. E.: "Vurdering av sementblandinger for HTHP-sementering", Internal Statoil Document, B&B BRT (May 1993).

7. Skalle, P., Backe, K., Elvebakk, H., Justnes, H., Lile, O.B., Lyomov, S. and Sveen, J.: "Integrated testing programme for qualifying well cement slurries against formation gas intrusion", Paper available at IPT, NTH, Trondheim (1994).

8. Lile, O.B., Backe, K., Elvebakk, H., Lyomov, S. and Skalle, P.: "A new technique for meaasuring permeability and strength of a curing cement", Paper available at IPT, NTH, Trondheim (1994).

Table 1. Summary of slurry and control data

Test	CT/ST	SG	$t_{10''/10'}$	tset 30	40	70	FW	FL	PV/YP	
Cal	190	1.90								
1	105/140	2.05	-	5:53	5:58	6:00	0.0	40	-	3.5
2	105/140	2.05	-	2:29	2:30	2:32	0.0	35	90.0	15.0
3	105/140	2.05	2 4	3:58	4:08	4:09	0.0	-	-	3.0
4	105/130	2.05	6 17	2:40	3:12	3:18	< 0.1	24	31.6	2.5
5	140/180	2.15	1 2	4:49	4:50	4:51	-	28	-	3.0
6	140/180	2.15	-	3:02	3:17	3:18	0.0	14	58.5	11.5
7	140/180	2.15	9 50	3:23	3:24	3:25	0.0	20		2.25
8	140/180	2.15	9 50	3:23	3:24	3.25	0.0	20		2.25
9	120/140	2.15	9 19	5.25	5:26	5:26	< 0.1	30	43.2	10.9

CT/ST = Circulating Temperature Static Temperature (°C)
SG = Specific Gravity
$t_{10'' (10')}$ = 10 seconds (10 minutes) gel strength (lb/100 ft^2)
t_{set} = time (h) to reach a consistency of specified amount of B_C (Bearden Units)
FW = Free Water on top of glas cylinder filled with 250 ml slurry (ml)
FL = Fluid loss (ml)
PV/YP = Plastic Viscosity/ Yield Point (CP/lb/100 ft^2)

Fig. 1. The HTHP gas migration system.

Fig. 2. The HTHP gas rig flow diagram.

Fig. 3. Gas flow during calibration test (neat cement slurry). Flow out is not shown but is the same as flow in, only some minutes delayed.

Fig. 4. The results from tests 6 and 9.

Fig. 5. Hydrostatic pressure variation (schematic) in all 9 tests.

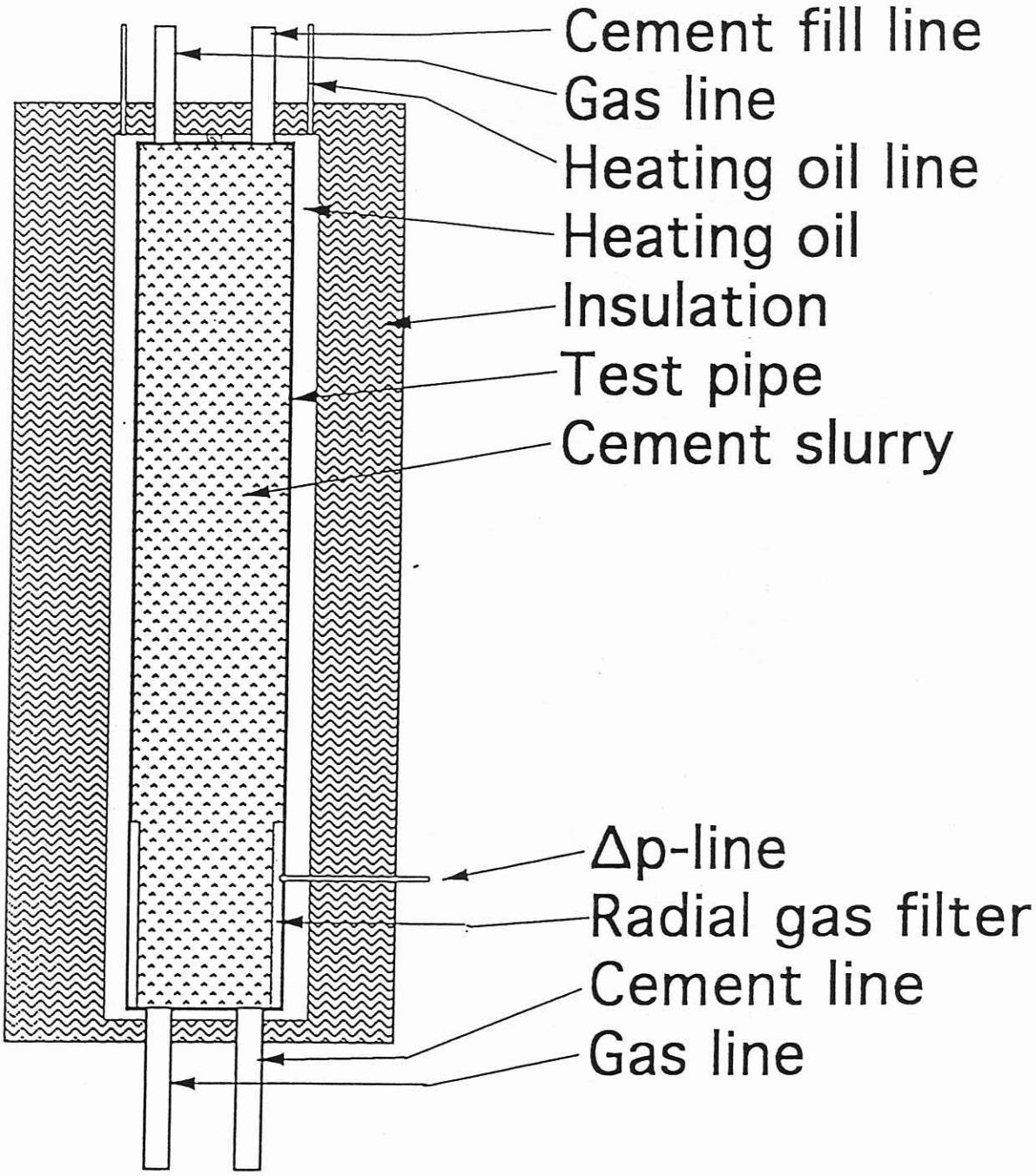

Cement fill line
Gas line
Heating oil line
Heating oil
Insulation
Test pipe
Cement slurry
Δp-line
Radial gas filter
Cement line
Gas line

Fig. 1

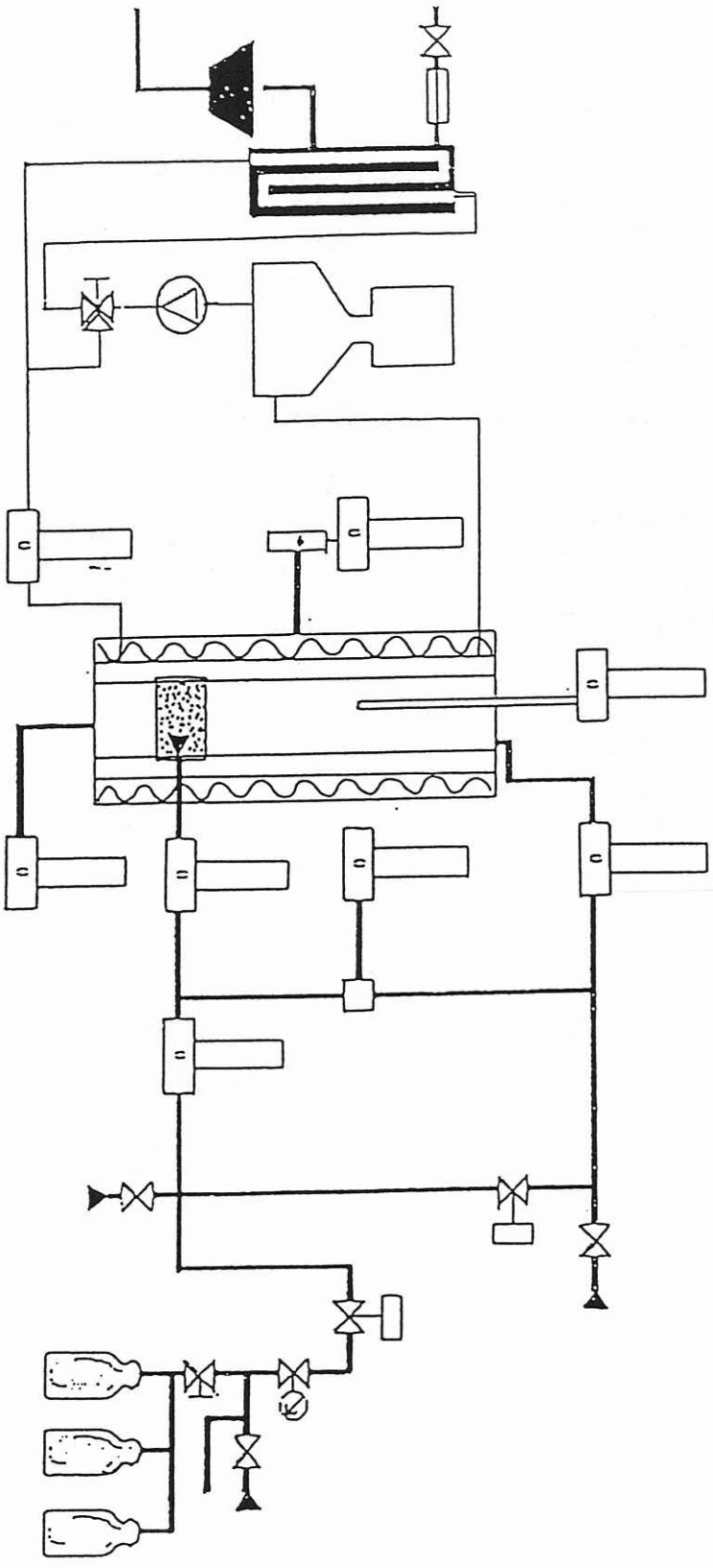


Fig. 2.

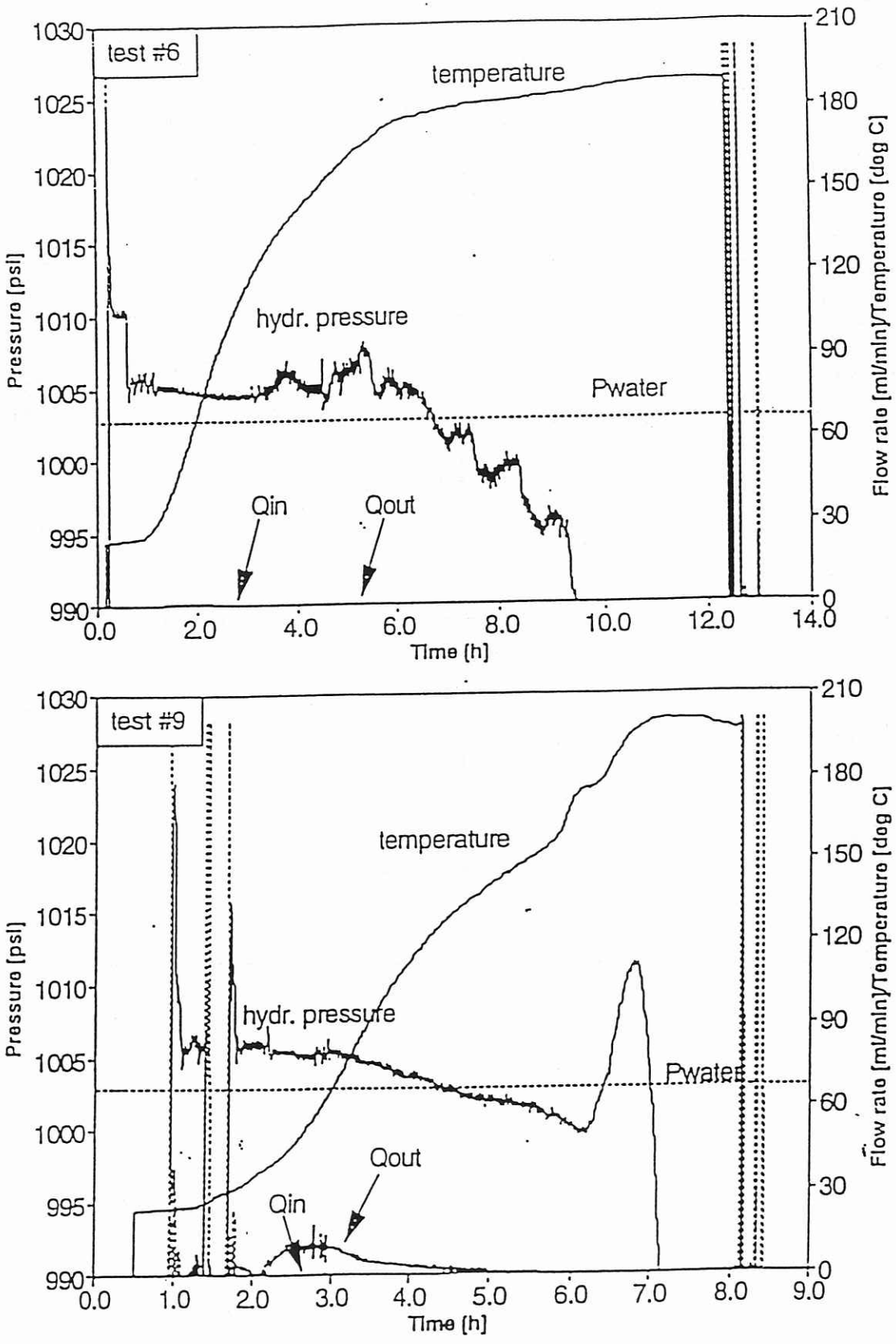
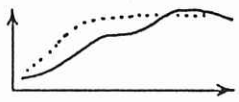
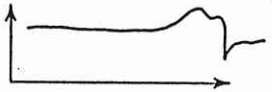
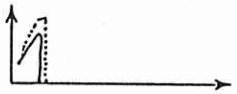
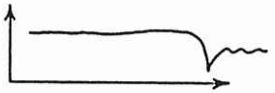
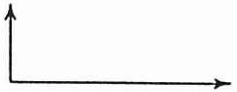
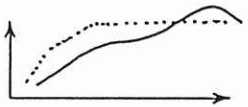
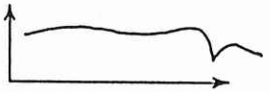
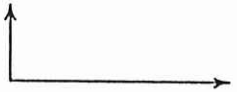
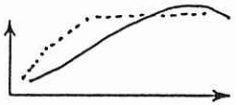
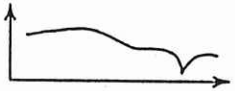
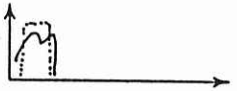
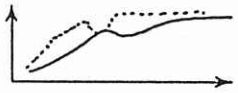
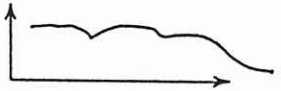
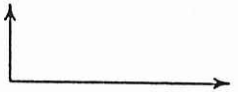
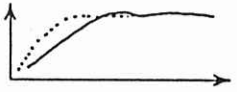
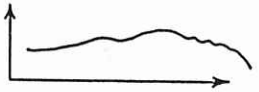
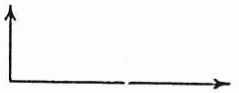
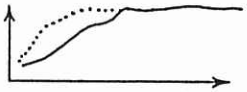
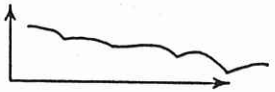
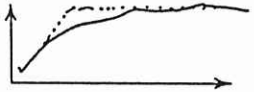
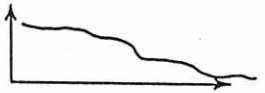
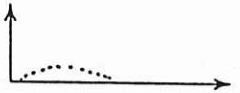
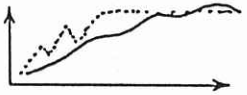
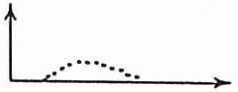


Fig. 4. The results from tests 6 and 9

#	T_{cem} ——— T_{oil} - - - - -	Pressure	Q_{in} ——— Q_{out} - - - - -
1			
2			
3			
4			
5			
6			
7			
8			
9			

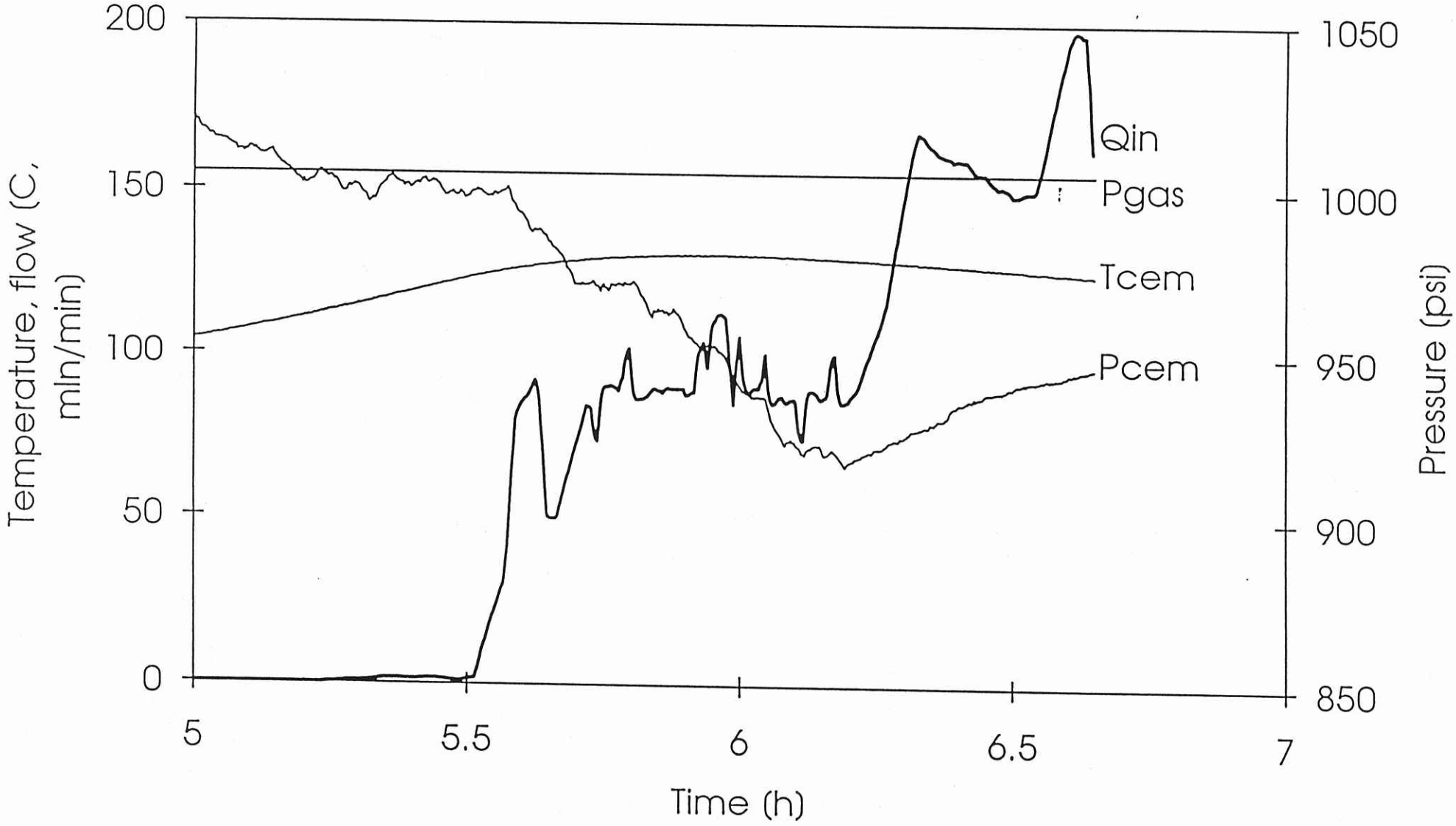

200
150
100
50
0
1050
1000
950
900
850
5
5.5
6
6.5
7
Time (h)
Temperature, flow (C, mln/min)
Pressure (psi)
Qin
Pgas
Tcem
Pcem

Fig 2

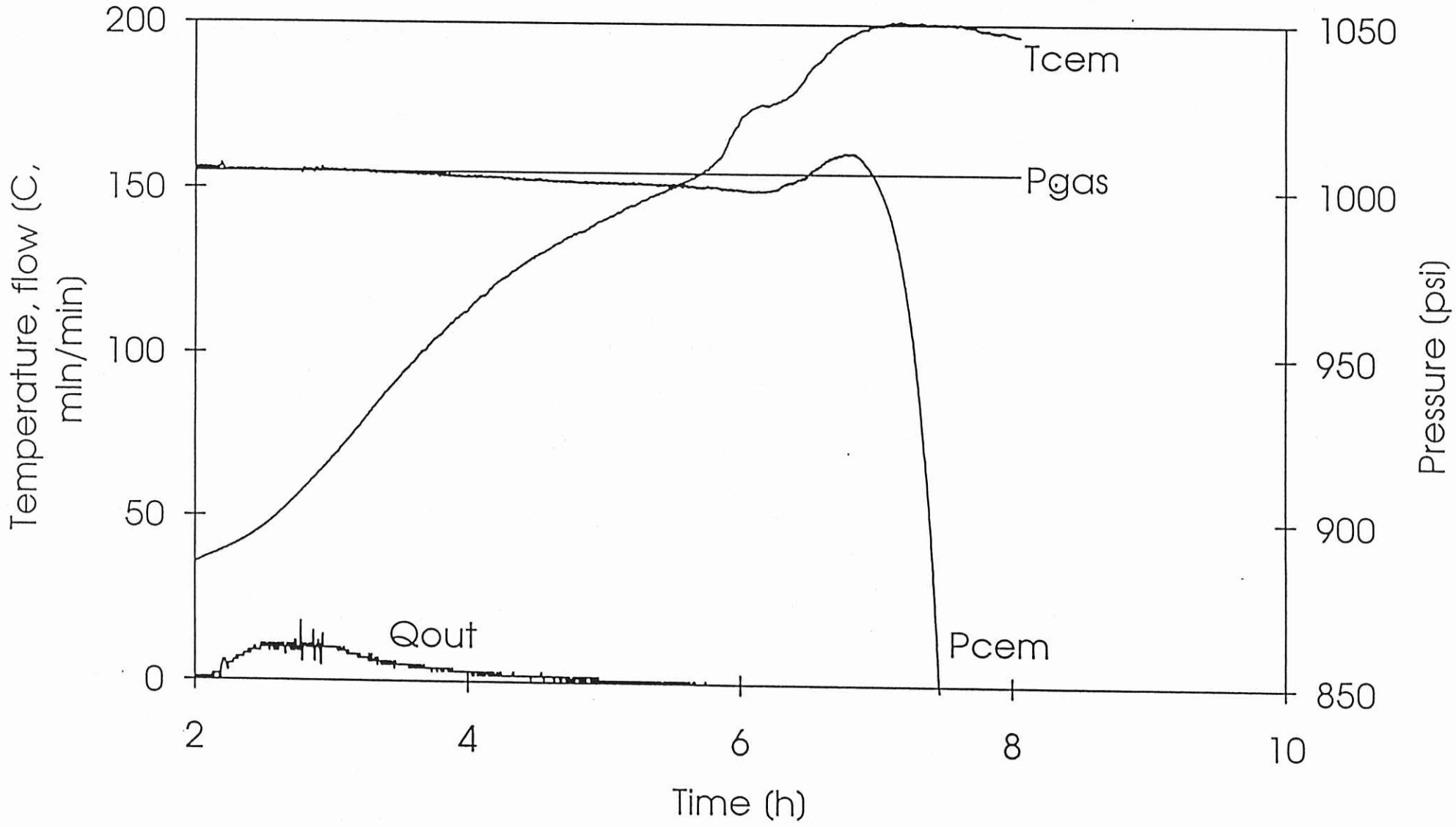

200
150
100
50
0
Temperature, flow (C, mln/min)
1050
1000
950
900
850
Pressure (psi)
Tcem
Pgas
Qout
Pcem
2
4
6
8
10
Time (h)

Fig. 4, test #9

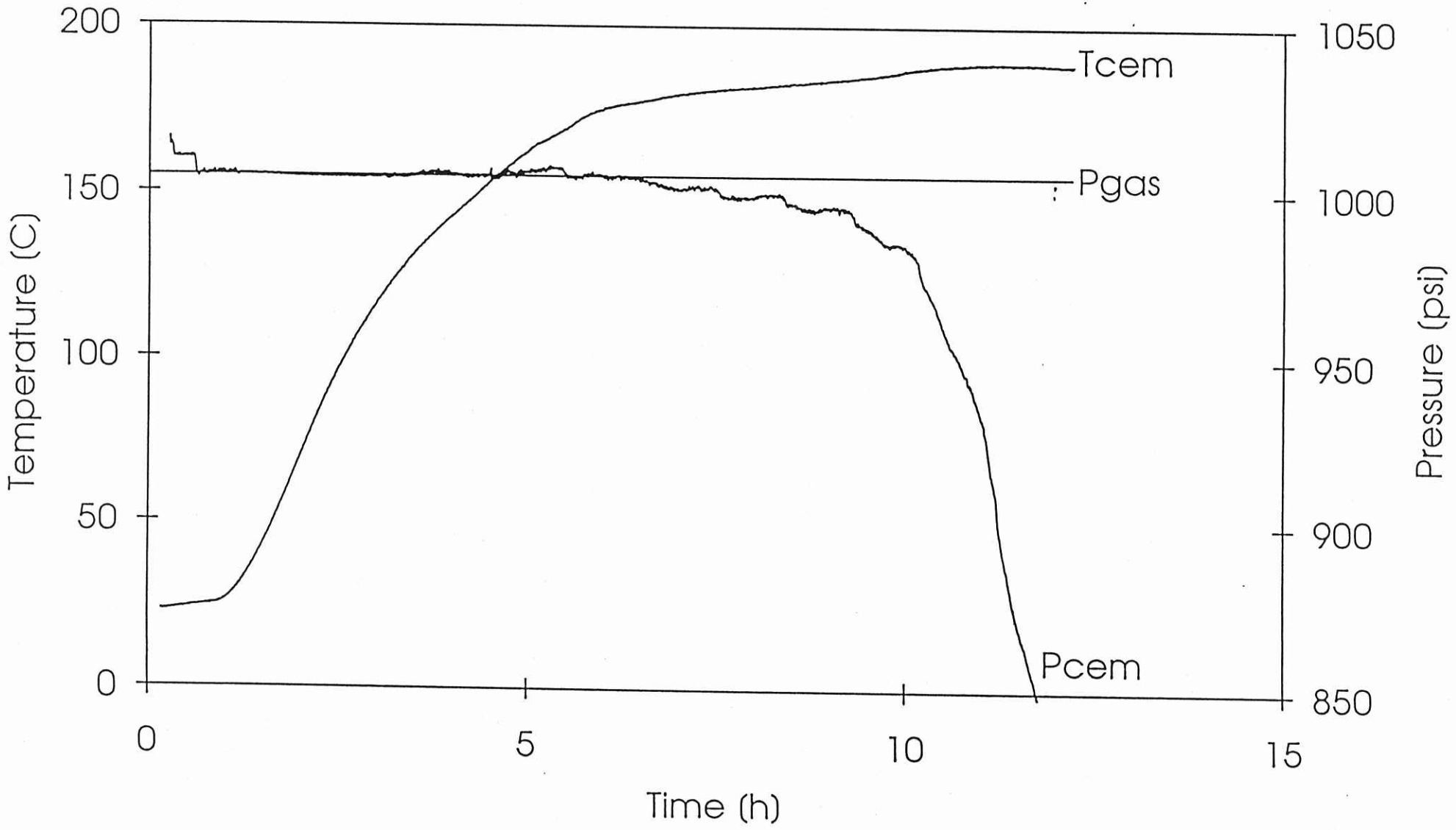

200
150
100
50
0
Temperature (C)
1050
1000
950
900
850
Pressure (psi)
Tcem
Pgas
Pcem
0
5
10
15
Time (h)

Fig. 4 test #6