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**Laboratory Evaluation of
Polyurethane Foam for
Geothermal Lost-Circulation
Plugging**

MASTER

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LABORATORY EVALUATION OF POLYURETHANE FOAM
FOR GEOTHERMAL LOST-CIRCULATION
PLUGGING

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Edited by B. C. Caskey and J. R. Kelsey

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ABSTRACT

To evaluate polyurethane foam formulations for use in plugging hot (such as geothermal) formations when lost circulation is encountered during drilling, laboratory tests of three foams were performed. Foams were mixed at high temperature (up to 300°F) and high pressure (up to 900 psig); the mechanical and fluid loss properties of the foams were subsequently determined. The results show that polyurethane foams can be mixed at downhole conditions and yield acceptable plugging properties.

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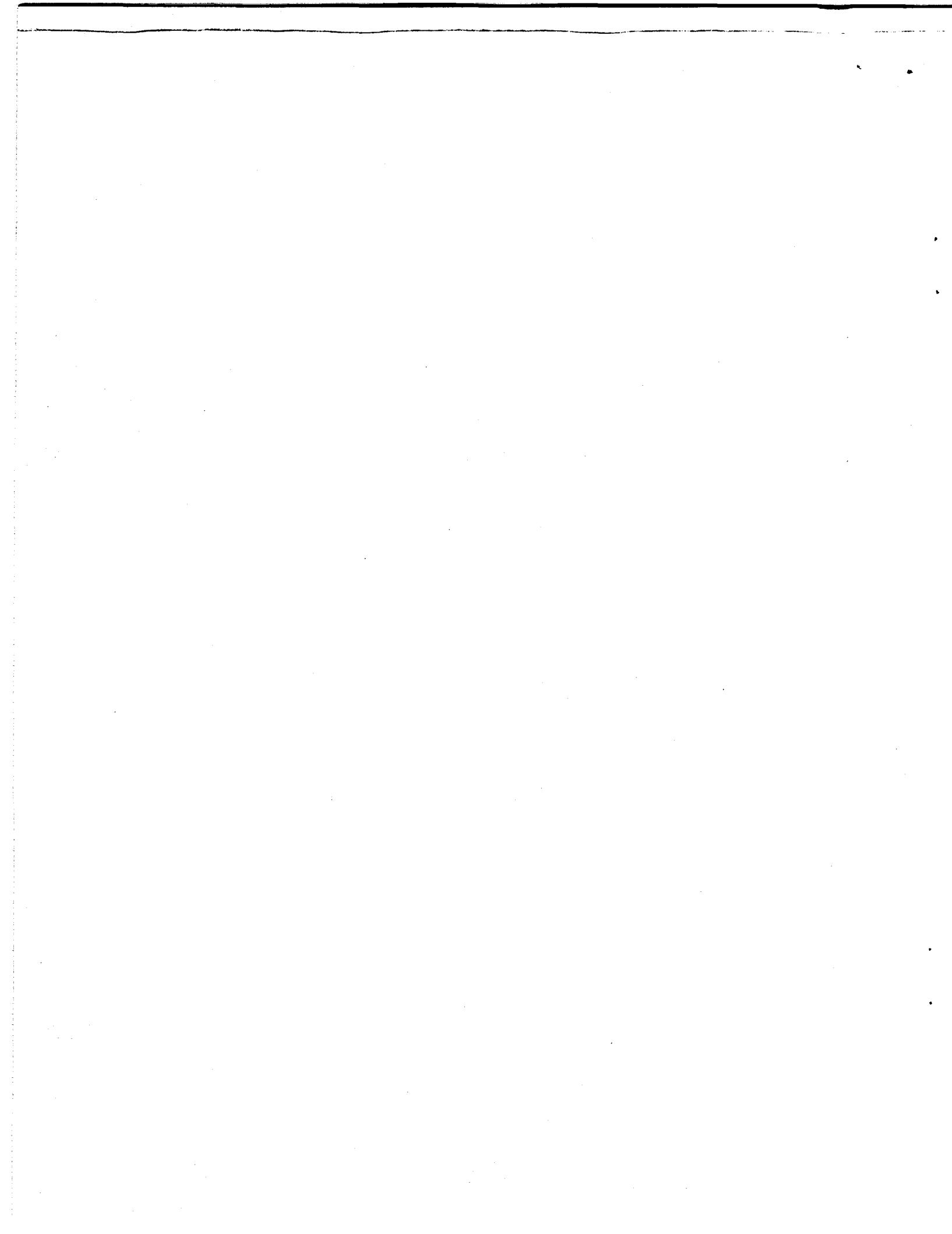
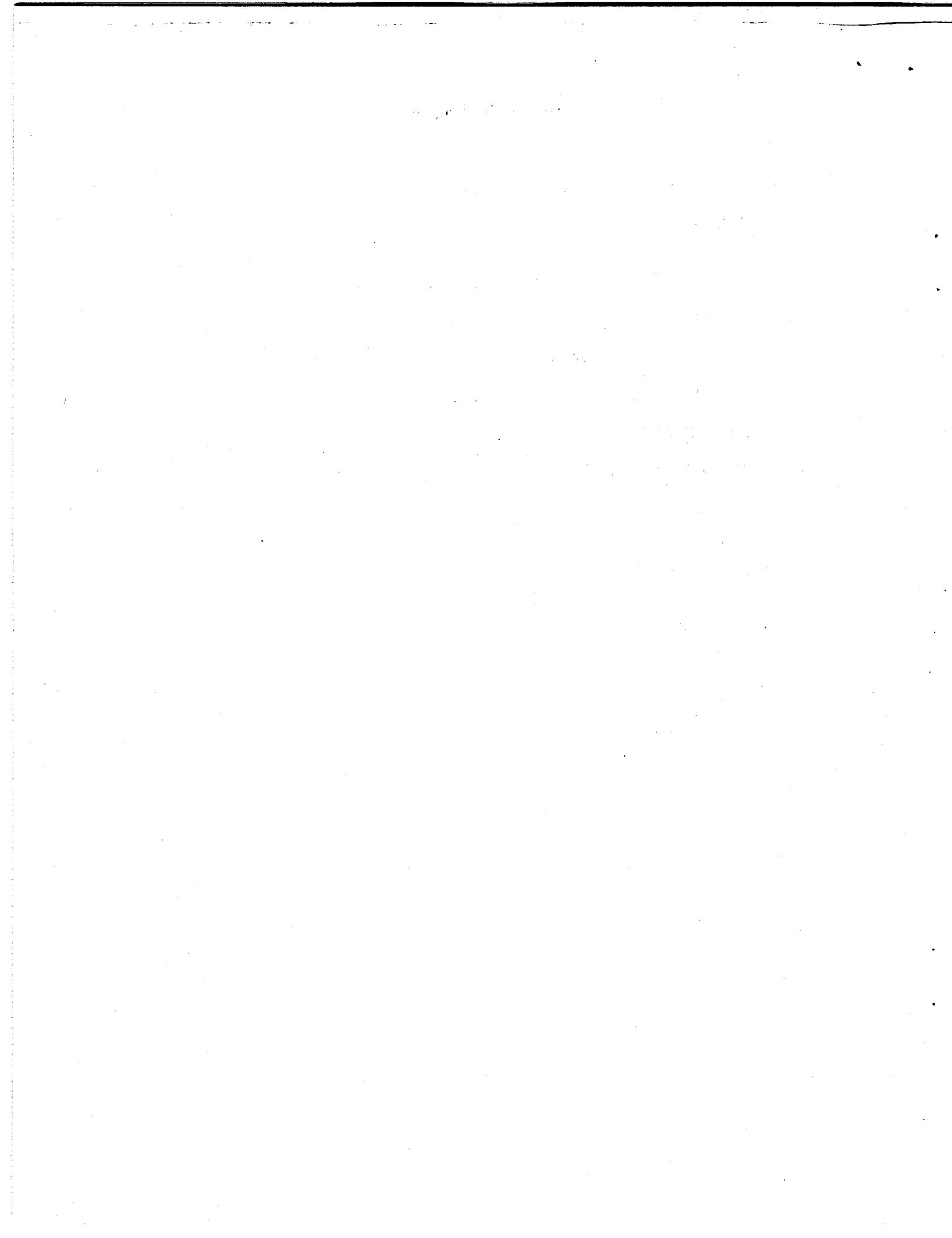


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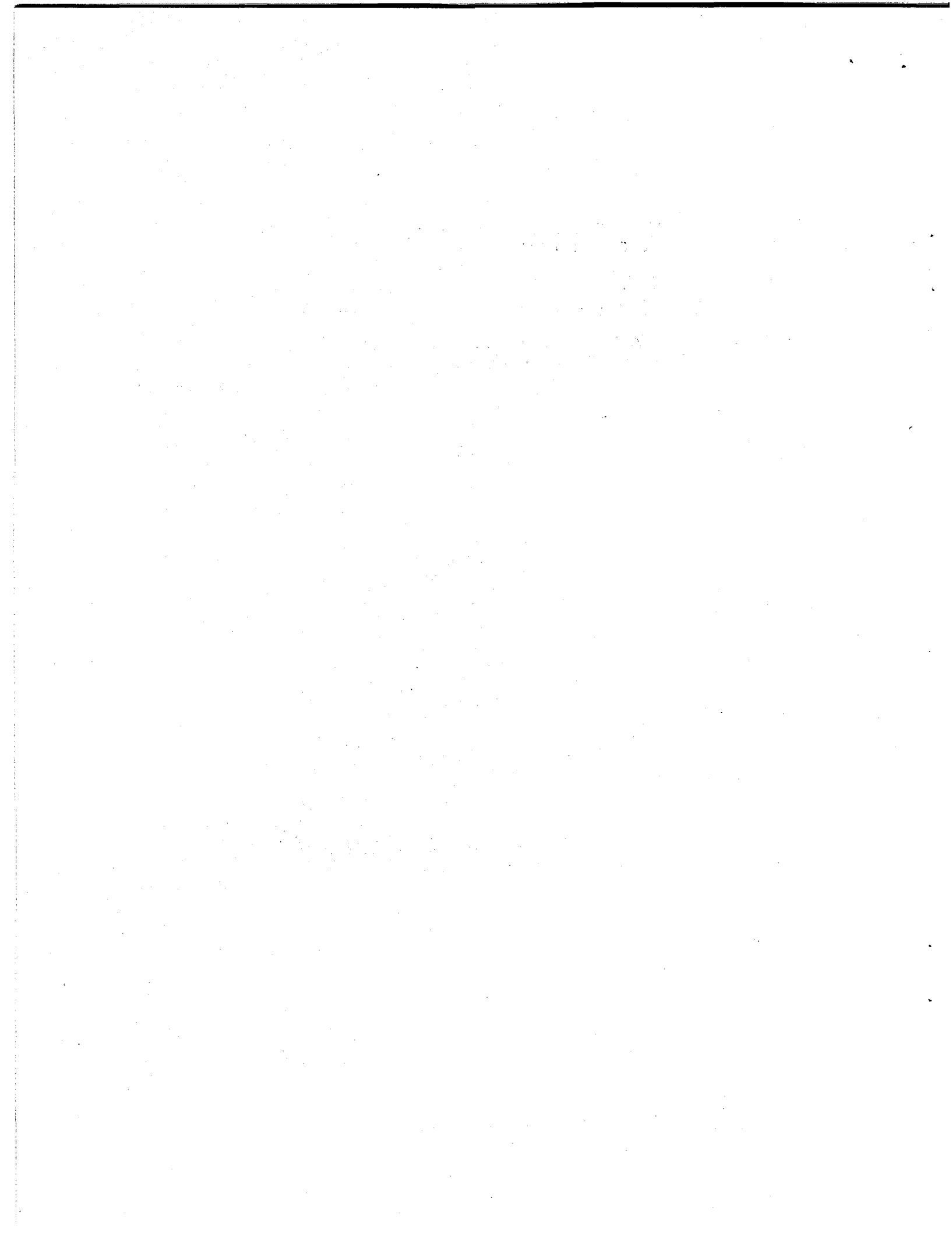
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I. INTRODUCTION

One of the major impediments to the widespread development of geothermal energy is the high cost of drilling wells. A significant contributor to this cost is the problem of lost circulation. This problem arises when the preferential flow path of the drilling fluid is into the fractured formation rather than up the wellbore to the surface. Due to the large fracture sizes and high temperatures associated with geothermal reservoirs, conventional plugging agents are not a reliable solution.

Sandia National Laboratories, as a part of the DOE Geothermal Technology Development Program, is investigating potential solutions to the lost circulation problem. One candidate solution is hard polyurethane foam formed from liquid constituents. The several liquids could be placed into the fracture system where they would react to form a solid which seals the borehole wall, preventing further lost circulation.

To verify the feasibility of using such foams in the geothermal environment, it is first necessary to determine if foams can be made to react at the borehole conditions (300°F and 1000 psi), and second, to confirm that the mechanical strength after reaction is sufficient to withstand the pressures associated with the drilling procedure. In order to make these determinations, Sandia National Laboratories, in a joint program with Poly Plug, Inc. of Houston, Texas, sponsored the testing

of several foam formulations (proprietary to Poly Plug, Inc.) at simulated geothermal conditions.

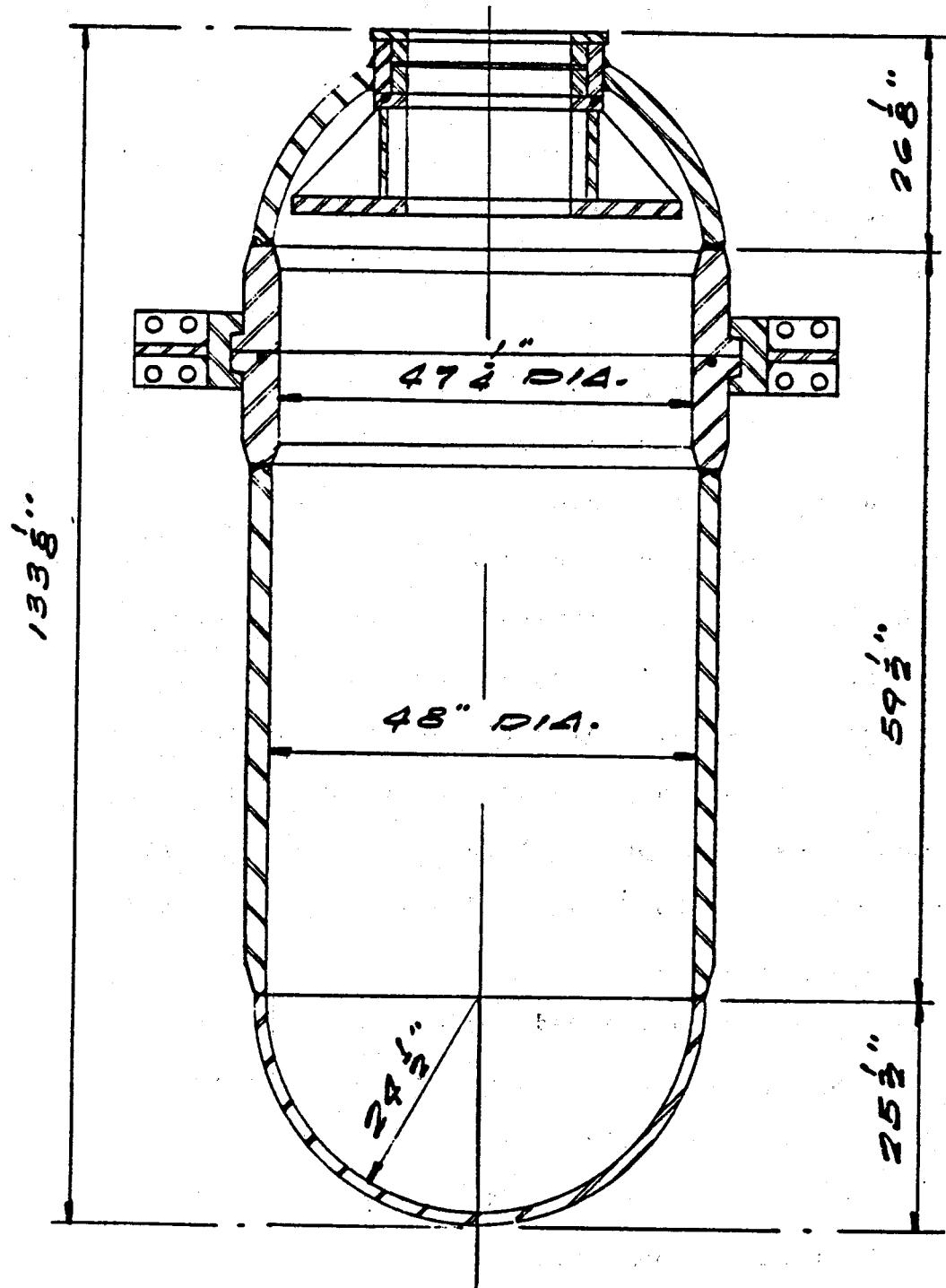
These tests were conducted at the Southwest Research Institute in San Antonio, Texas, and this report presents the results of those tests as well as the results of bench scale tests performed at Poly Plug, Inc.

Editing of this report was provided by B. C. Caskey and J. R. Kelsey of Sandia National Laboratories.

II. OBJECTIVE

The objective of this program is to establish that polymeric foam can be produced at temperature and pressure, and to evaluate the effects of these conditions on the density, compressive strength, closed cell structure, speed of reaction, fluid loss, and exotherm of the foams produced. The effect of the speed of reaction within the polymer was produced by testing a high temperature polyurethane foam system formulated with three reaction speeds provided by Poly Plug Incorporated. The effect of pressure was reproduced in the SWRI laboratory 48-inch diameter pressure vessel, of which a sketch is included as Figure 1. Temperature effects were reproduced with electric heaters and controllers along with suitable thermal insulation. Four different pressures (ranging from 15 psig to 900 psig) and four different temperatures (ranging from 100°F to 300°F) were identified by Sandia in a matrix of test conditions.

While the foam was being made under simulated well conditions of temperature and pressure, the exotherm temperature of the foam was measured continuously. Pressure in the test vessel was also measured continuously to establish the quantity of gas produced by the reacting polyurethane foam. After the finished foam product was removed from the simulated well conditions, physical properties of samples removed from the foam specimen were measured. These included density, compressive strength, closed cell structure, and fluid loss through cut samples.



48-inch-diameter DEEP Ocean Simulator

3750 psi

Water, saltwater, oil

Temperature > 32°F

Photography capability

NOTE: The closure plate may be changed to facilitate special penetration configurations necessitated by geometry of the item to be evaluated.

FIGURE 1. Sketch of the SwRI 48-Inch Diameter Pressure Vessel

III. EXPERIMENTAL PROGRAM

SwRI Tests

In order to meet the objectives of the test program the SwRI 48-inch diameter pressure vessel was adapted for temperature control with electric resistance heaters and temperature controllers. Two pressurization systems were required for these tests: one for pressurization of the SwRI 48-inch diameter pressure vessel (to simulate well conditions), and one to supply the necessary pressure to induce foaming of the two components of the polyurethane foam. Figure 2 is a schematic of the test setup showing both temperature control and pressurization systems. Table I lists critical components of the instrumentation and hardware used during the test.

Temperature within the pressurized environment was measured with iron-constantan thermocouples and electric temperature controllers listed in Table I. Leads from thermocouples pass through fittings which penetrated the pressure vessel boundary. Strip heaters which had been strapped to the bare wall of the outside of the 48-inch diameter pressure vessel were adequately insulated with two types of insulation to reduce power consumption.

In ordinary use the 48-inch diameter pressure vessel is pressurized using tap water after the vessel has been completely filled with water. However, during these tests it was desirable that the two components of polyurethane foam not be contaminated with water. Therefore, it was decided that an inert atmosphere of nitrogen would be the best environment in which to produce the foam so that undesirable chemical effects could be avoided.

Nitrogen Pressure to Move Platen and Begin Reaction

Reaction Pressure Tank Pressure 500 to 1,000 psi

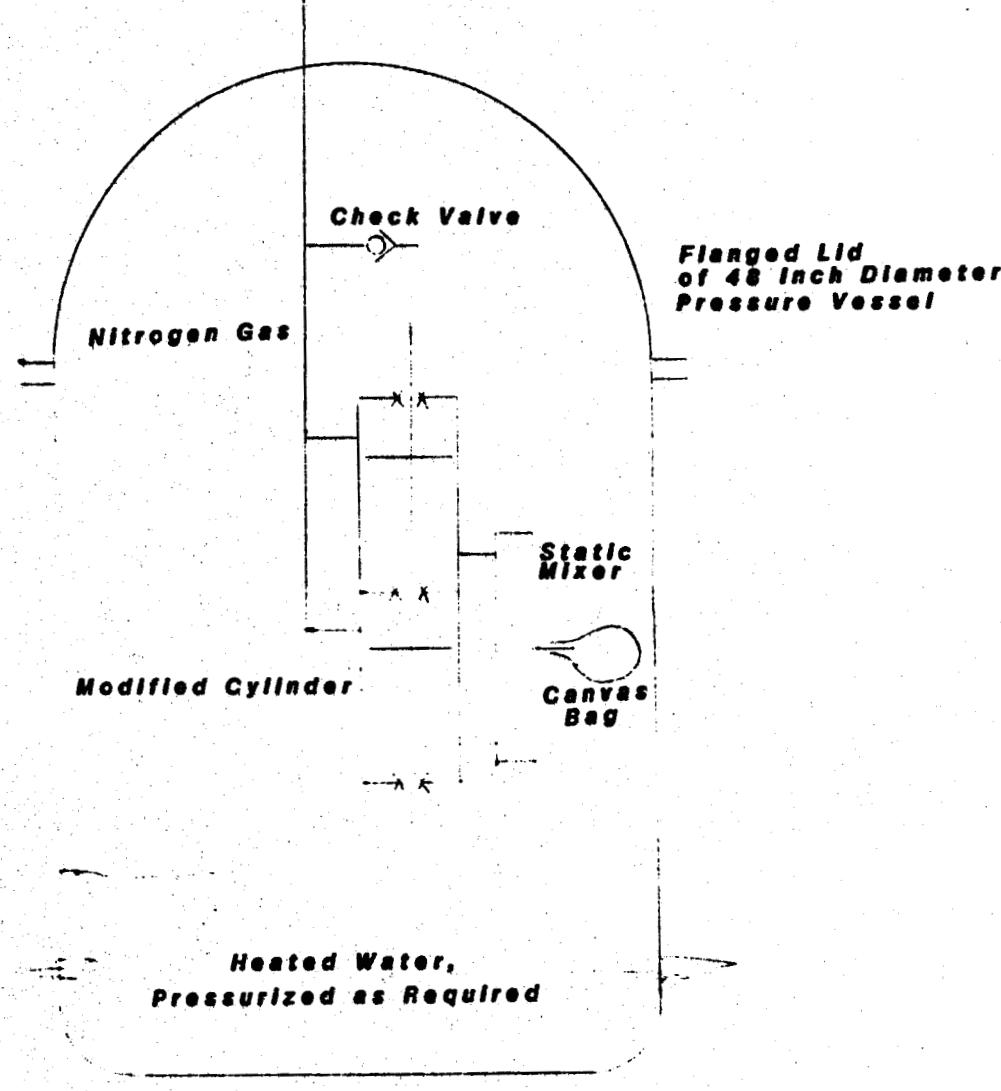


FIGURE 2. Simplified Schematic of Test Setup

TABLE I
INSTRUMENTATION AND HARDWARE
USED DURING THE TEST

Pressure Vessel: 48-Inch Diameter SwRI Pressure Vessel (see Figure 1)

Temperature Controller: Love Controls Corp. Model 49 Proportioning Controller, 0-400°F, 5°F increments.

Thermocouples (3 each): Iron-Constantan

Temperature Indicators (3 each): Analogic Model AN2572, Calibrated Range - 170°F to + 1400°F

Cylinder Pressure Regulators (3 each): Victor Compressed Gas Regulator UL Listed No. 5411, Serial No.'s 24123, 24133, and 24139.

Cylinder Pressure Gauges (3 each): 3D Instruments, Inc., No. 2047-0186-33, 0-3,000 psi, 20 psi increments.

Deadweight Tester (used for calibrating pressure gauges before test): Ashcroft Portable Deadweight Tester; Type No. 1305-B-100; Range: 2,000 to 10,000, Serial No, 83710010, Traceable to National Bureau of Standards (NBS),

The frame to which the foam mixing hardware was to be attached was suspended from the hemispherical lid of the pressure vessel (see Figure 3).

Figure 4 shows the underside of the lid of the 48-inch diameter pressure vessel through which pressure and thermocouple penetrations were made. Figure 5 shows thermocouple wires inside the lid of the pressure vessel. To measure exotherm temperature, a thermocouple was installed near the expected center of the bag into which the foam would be injected. In addition, a thermocouple was installed to measure ambient temperature of the pressurized nitrogen environment in which the foam was produced.

Before the test, the 48-inch diameter pressure vessel was filled to within inches of its capacity. For tests at elevated temperatures, the water within the pressure vessel was preheated to the desired test temperature. After the hemispherical pressure vessel lid with the foam mixing hardware attached was installed to the pressure vessel, the upper volume of the vessel (in which the foam was to be produced) was pressurized with nitrogen. A schematic of the nitrogen pressurization system is given in Figure 2. Figure 6 shows plumbing of the bank of nitrogen bottles which were used to pressurize the top of the 48-inch diameter pressure vessel as well as to drive the two components of foam through the mixing chamber and ultimately into the canvas bag. To speed up this operation, an accumulator (Figure 7) was installed so that line restrictions such as regulators and valves could be bypassed. Pressure was controlled via regulators and valves installed on a control panel (see Figure 8).

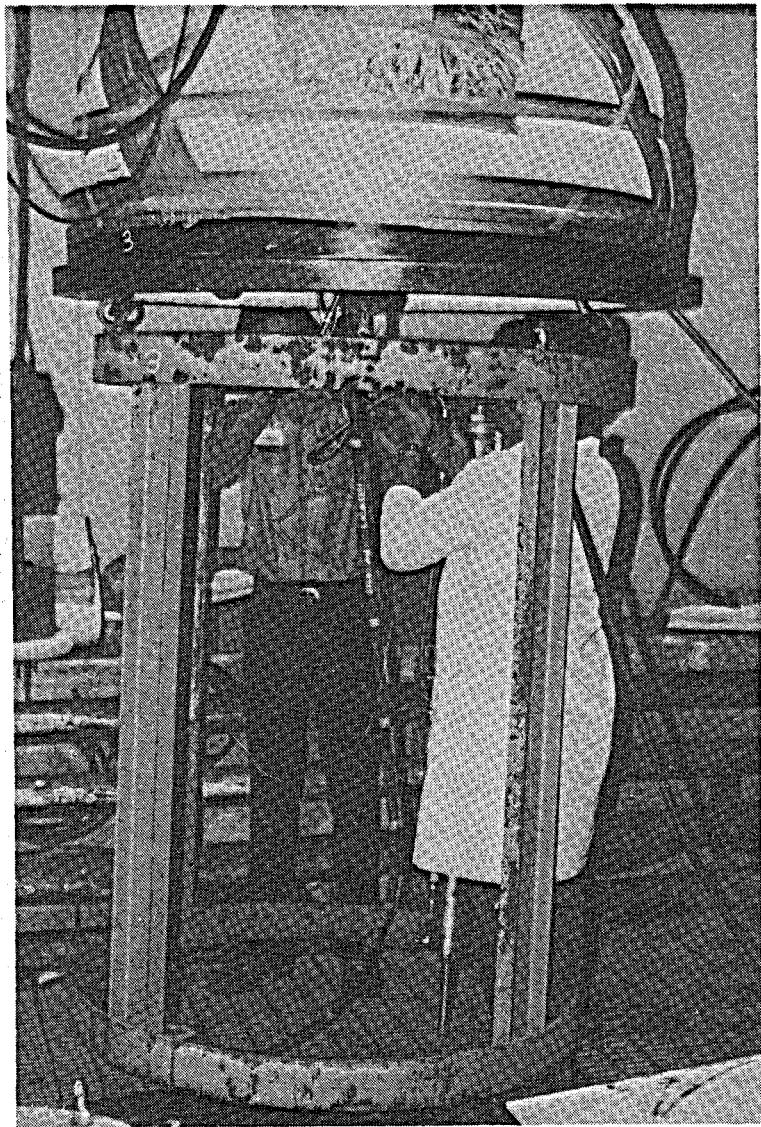
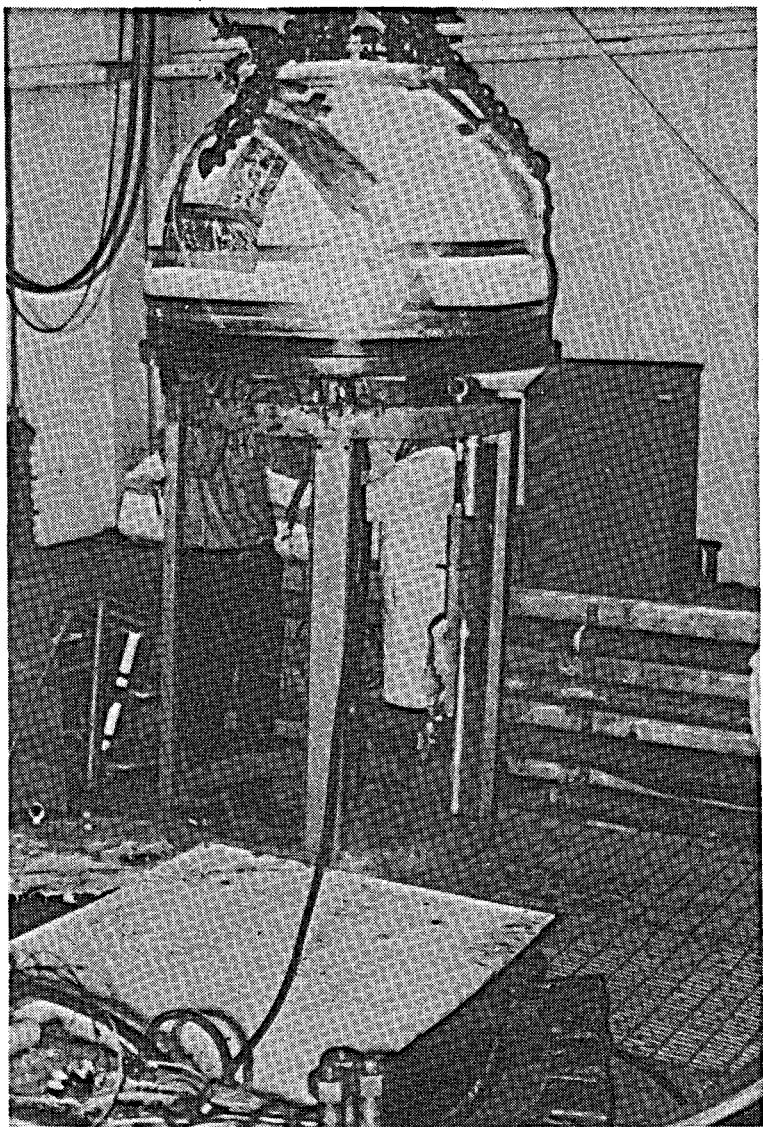


Figure 3. Installing the three foam cylinder assemblies to frame hanging beneath lid of 48-inch diameter pressure vessel prior to test.

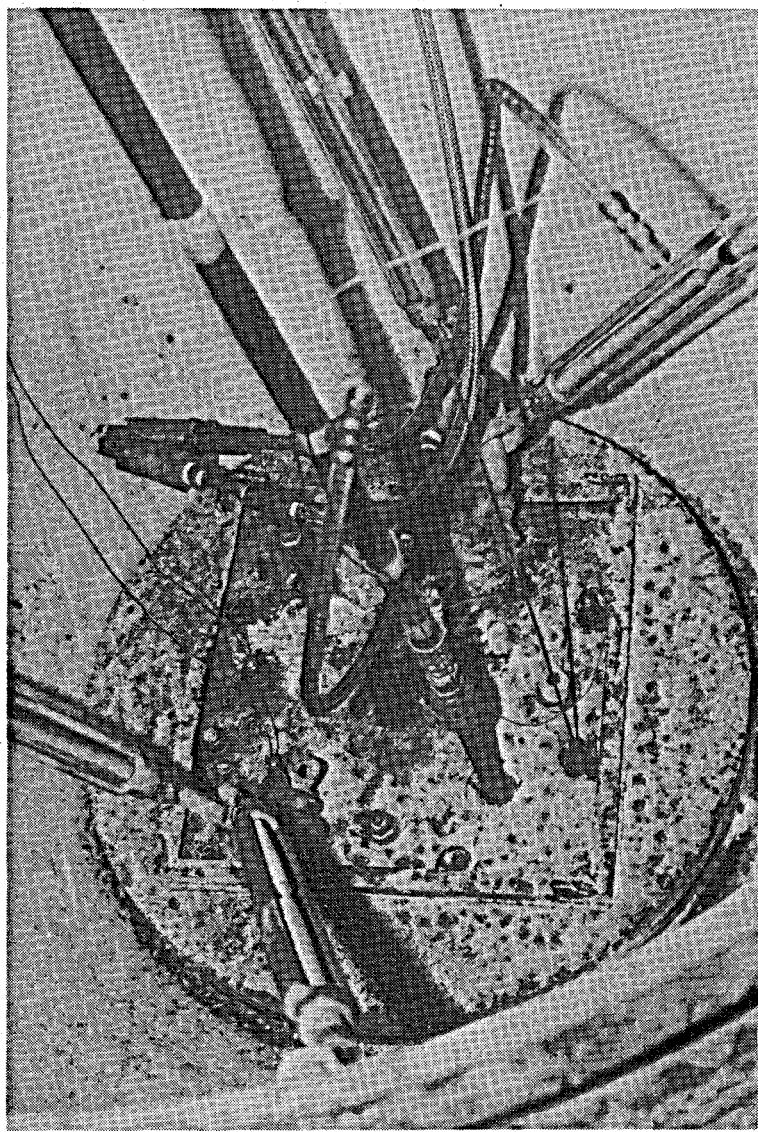


Figure 4. Underside of lid of 48-inch diameter pressure vessel through which pressure and thermocouple penetrations were made.



Figure 5. Thermocouple wires inside the lid of the 48-inch diameter pressure vessel prior to test. Tube (at hand in photo) is exit passage of foam to which canvas bag was attached.

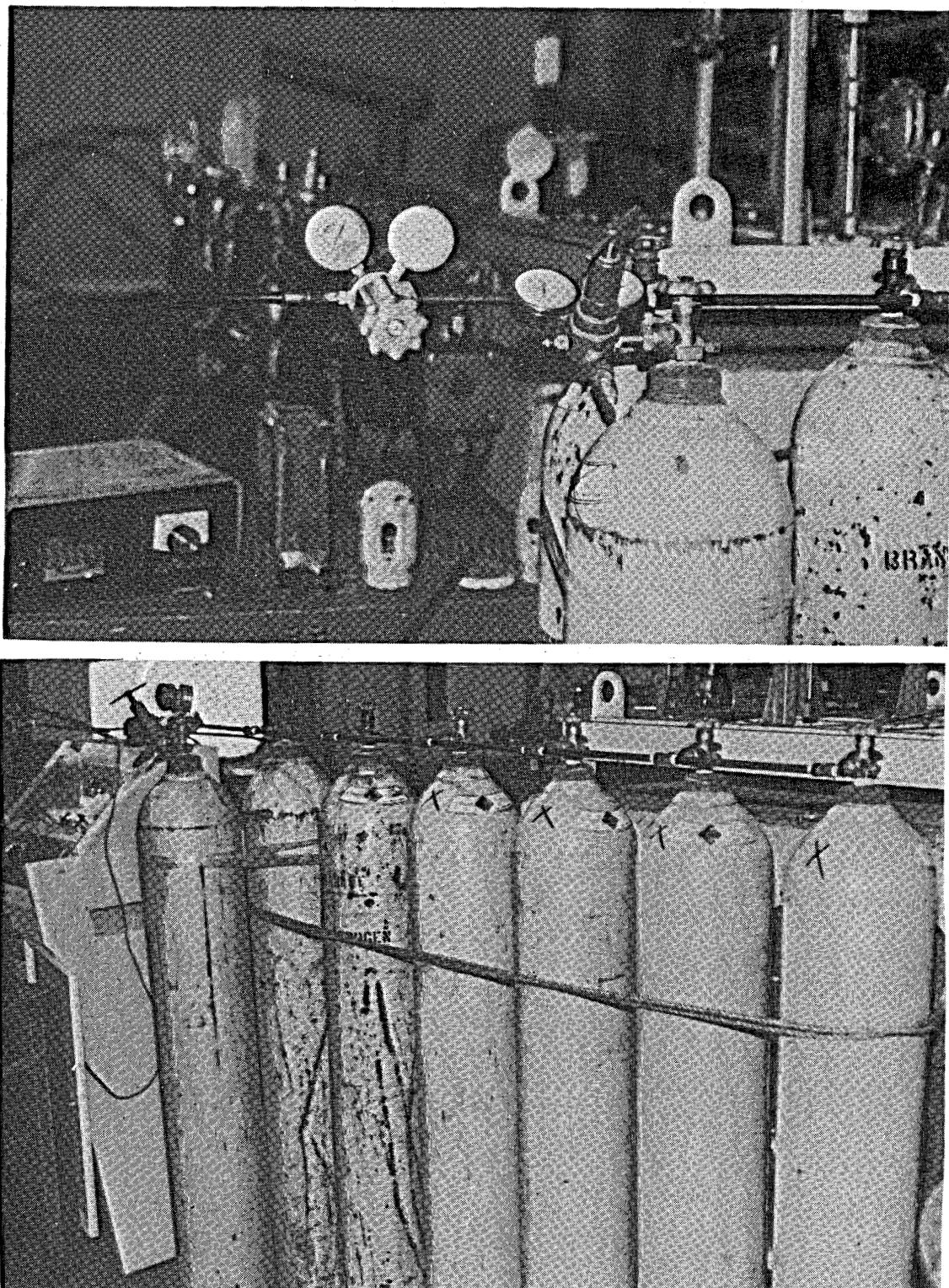


Figure 6. Bank of nitrogen bottles used to pressurize top of 48-inch diameter pressure vessel as well as to drive two components of foam into mix chamber and into canvas bags.

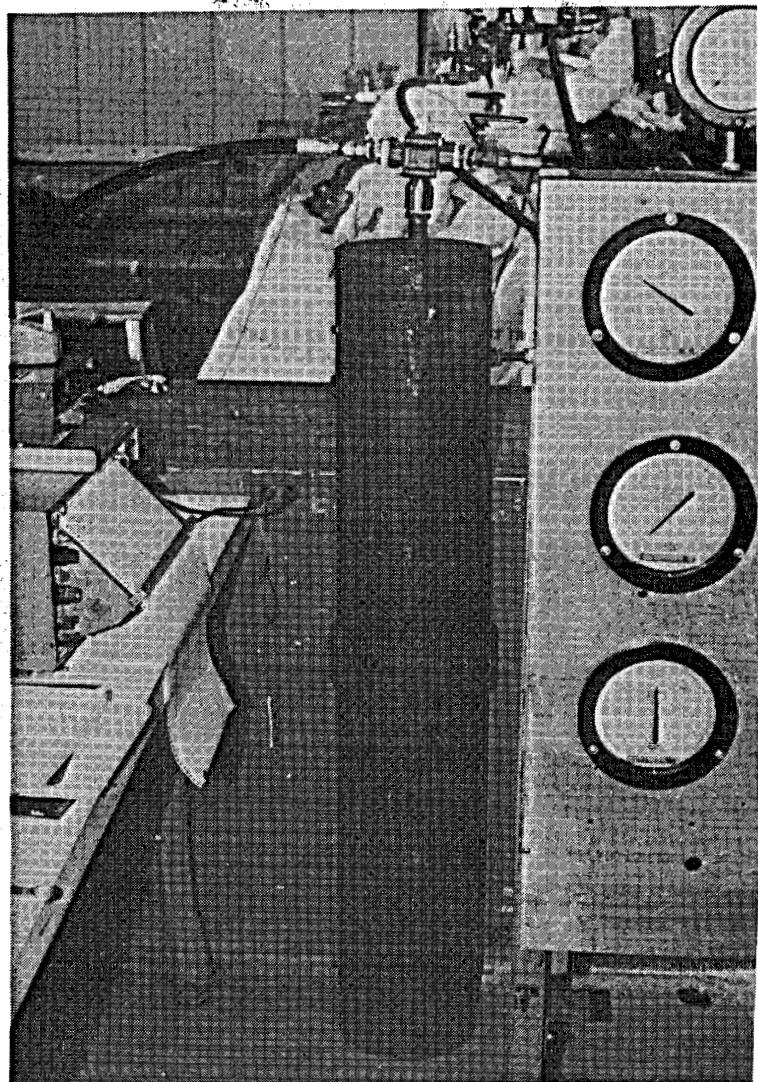


Figure 7. Accumulator installed between nitrogen bottles and foam samples to allow rapid discharge of nitrogen (to prevent premature foaming).

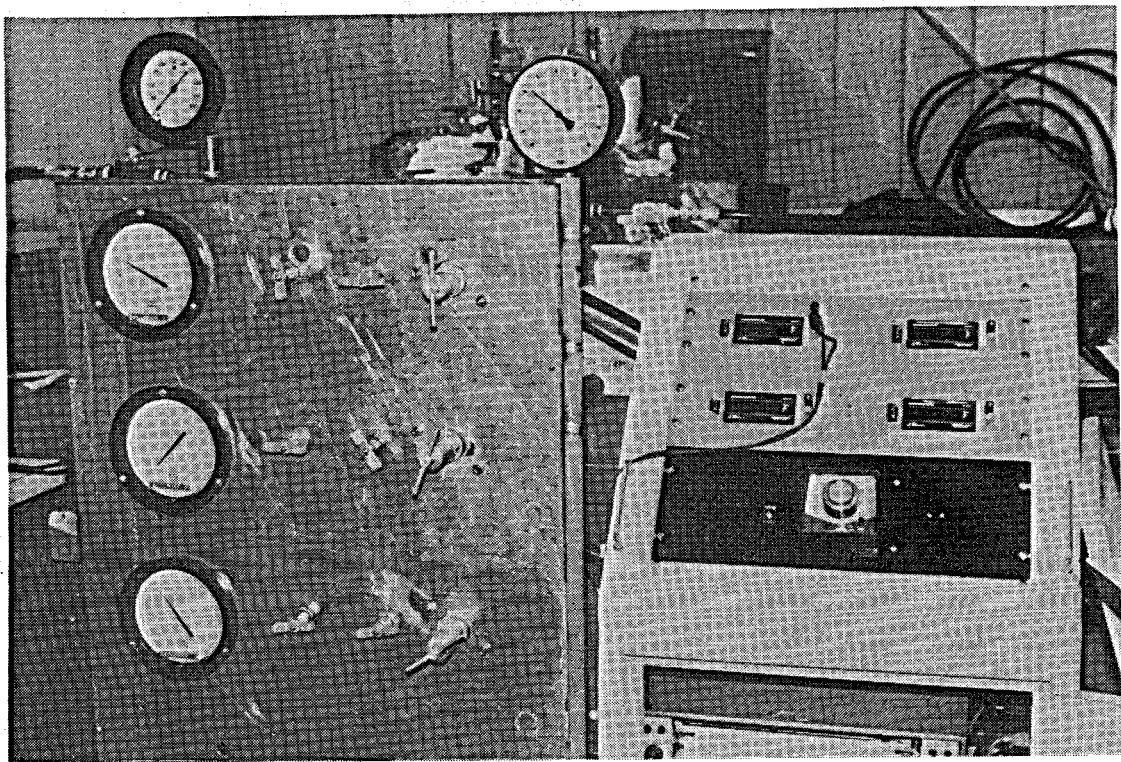


Figure 8. Control Panels - nitrogen pressure to each of 3 foam samples (left) and temperature control and readout for 3 samples and pressure vessel.

If necessary, the temperature of the pressure vessel was adjusted to the desired level after pressurization with nitrogen. Temperature was controlled using thermocouples and a temperature controller which powered the resistance heaters on the pressure vessel. Figure 8 shows the four temperature monitors (one for each of the three foam exotherms and one for the nitrogen environment in which foam was produced).

An air cylinder was adapted for use as both reservoir for the isolated foam components prior to mixing, as well as the pump by which the two components were transferred to the mixing chamber. A schematic describing this operation of the modified air cylinder (cartridge) is shown in Figure 9. Figure 10 shows the mixing chamber being attached to the modified cylinder. After the foam was produced, the pressure within the 48-inch pressure vessel was vented (see Figure 11) and canvas bags containing foam samples (see Figures 12 and 13) were removed for physical properties tests.

The schematics of Figures 2 and 10 describe the test setup used after several changes to variations of this setup had evolved.

One high-temperature polyurethane foam system was used throughout the testing. Initially this system was formulated with three reaction speeds. They were identified as follows: Formula No. 1 with an initiation time of 10 seconds at 77° F, Formula No. 2 with an initiation time of 20 seconds at 77° F, and Formula No. 3 with an initiation time of 30 seconds at 77° F.

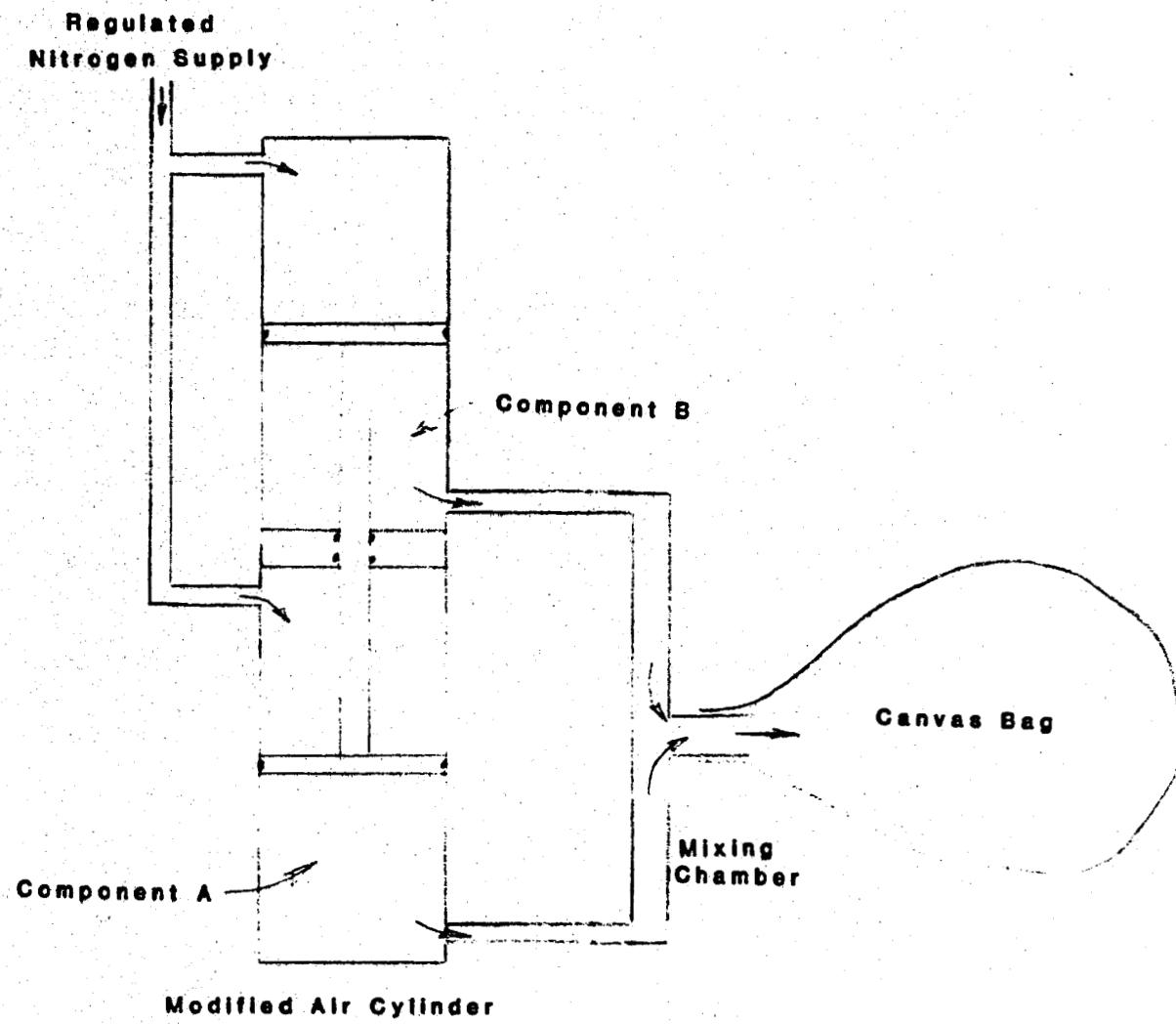


FIGURE 9. Schematic Showing Operation of the Modified Air Cylinder

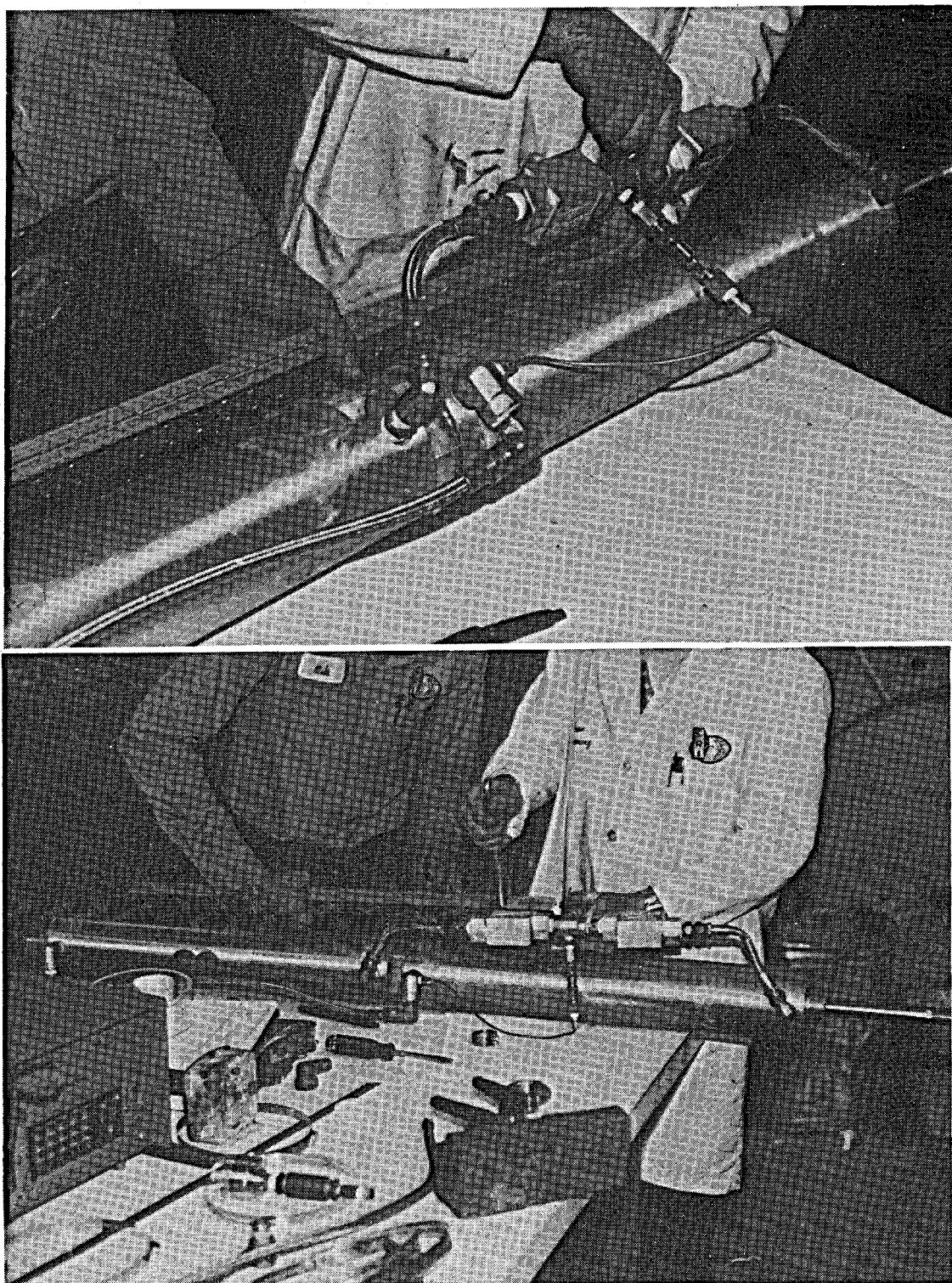


Figure 10. Two-chambered cylinder in which components were separately kept prior to foaming. During foaming, nitrogen pressure forced the two components into the mixing chamber (being attached in bottom photo) through which it travelled into the canvas bag.

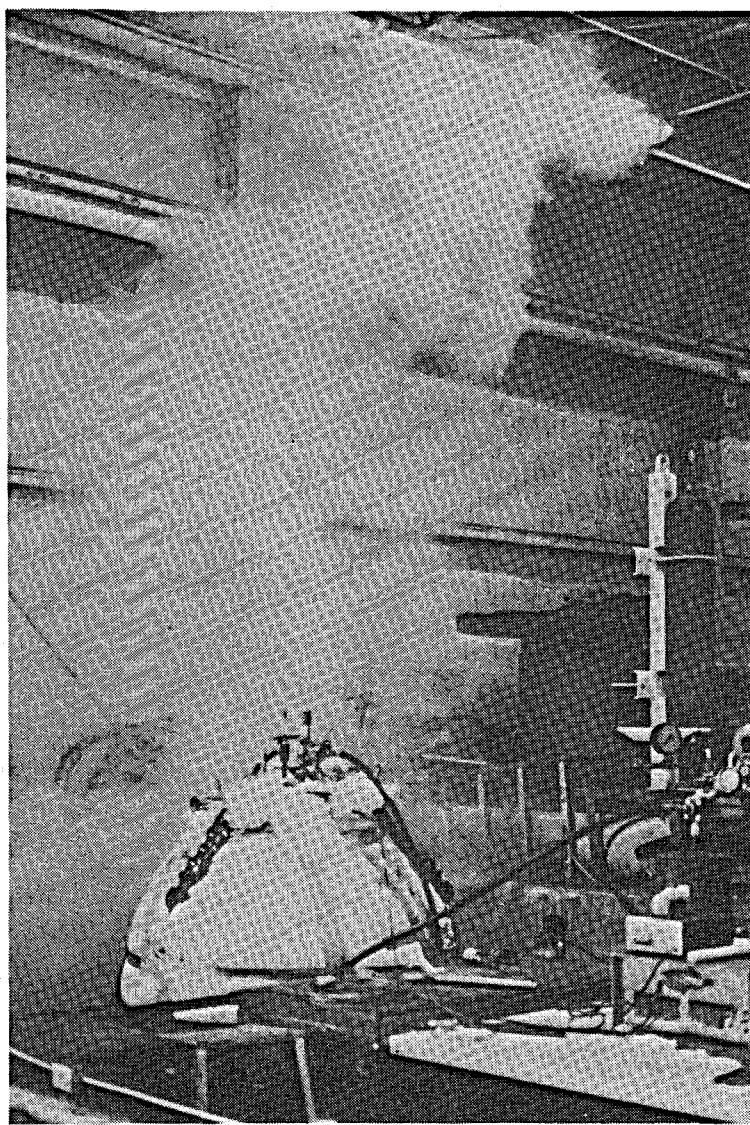
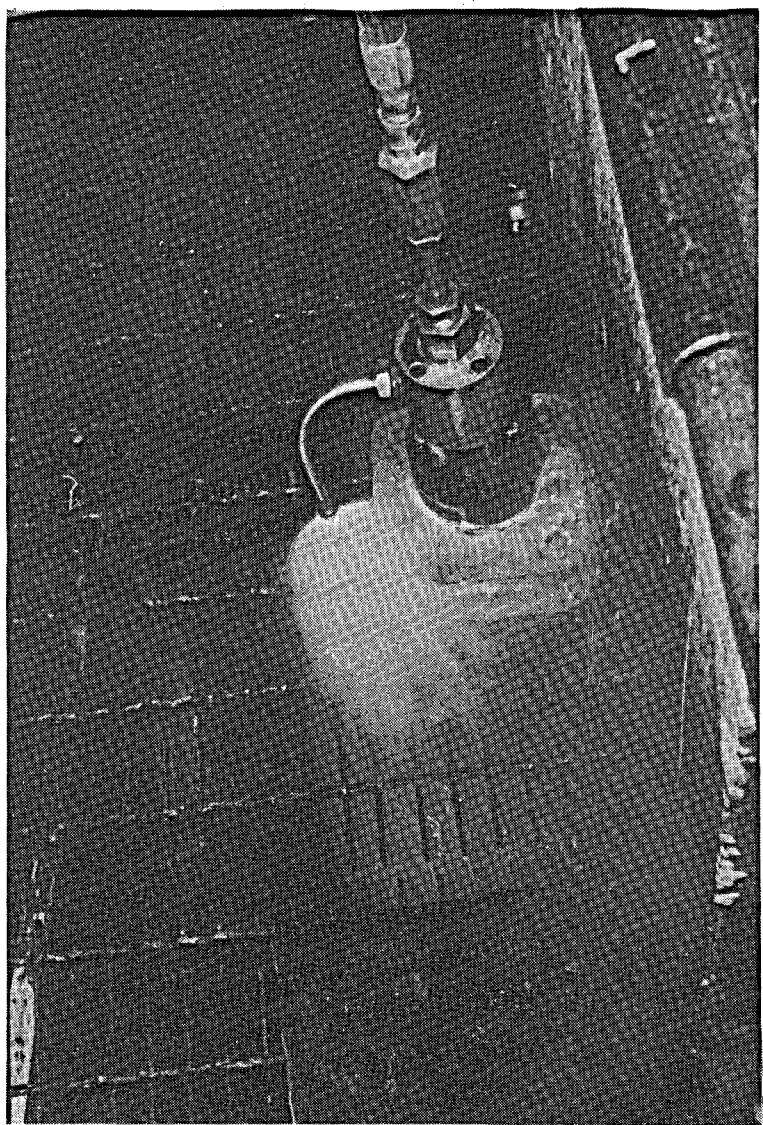


Figure 11. Venting the pressure vessel after a test.

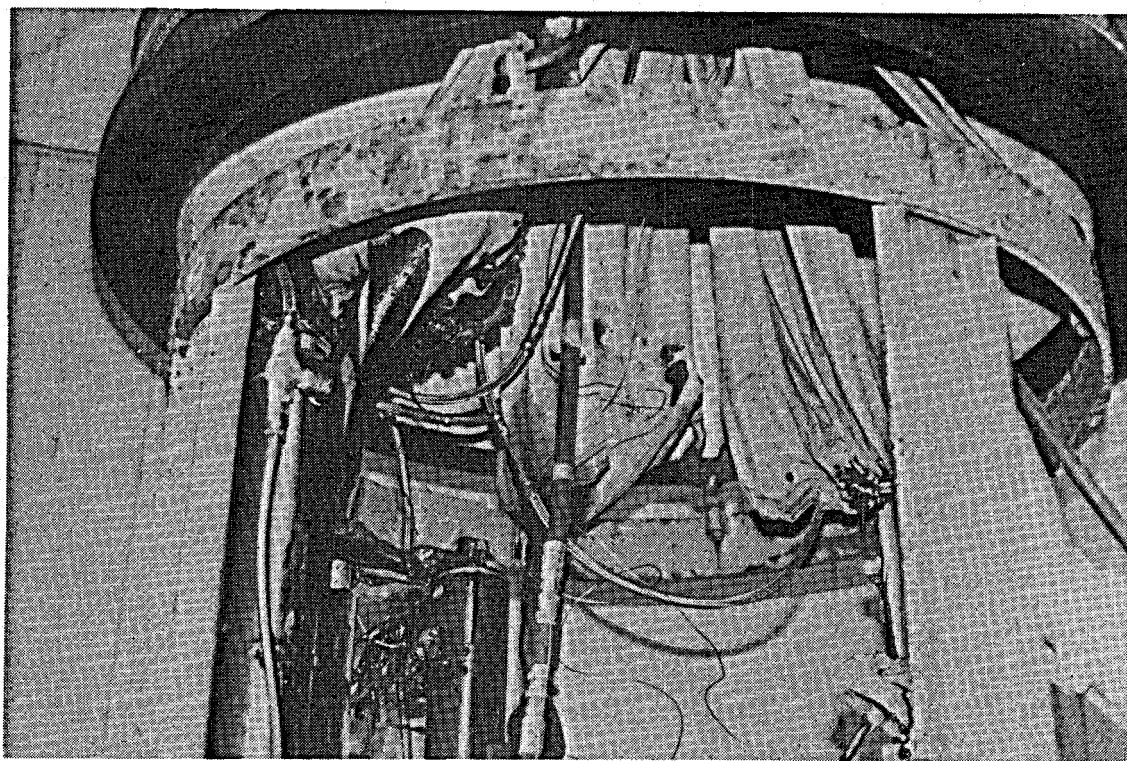


Figure 12. Canvas bags containing foam samples after a test.

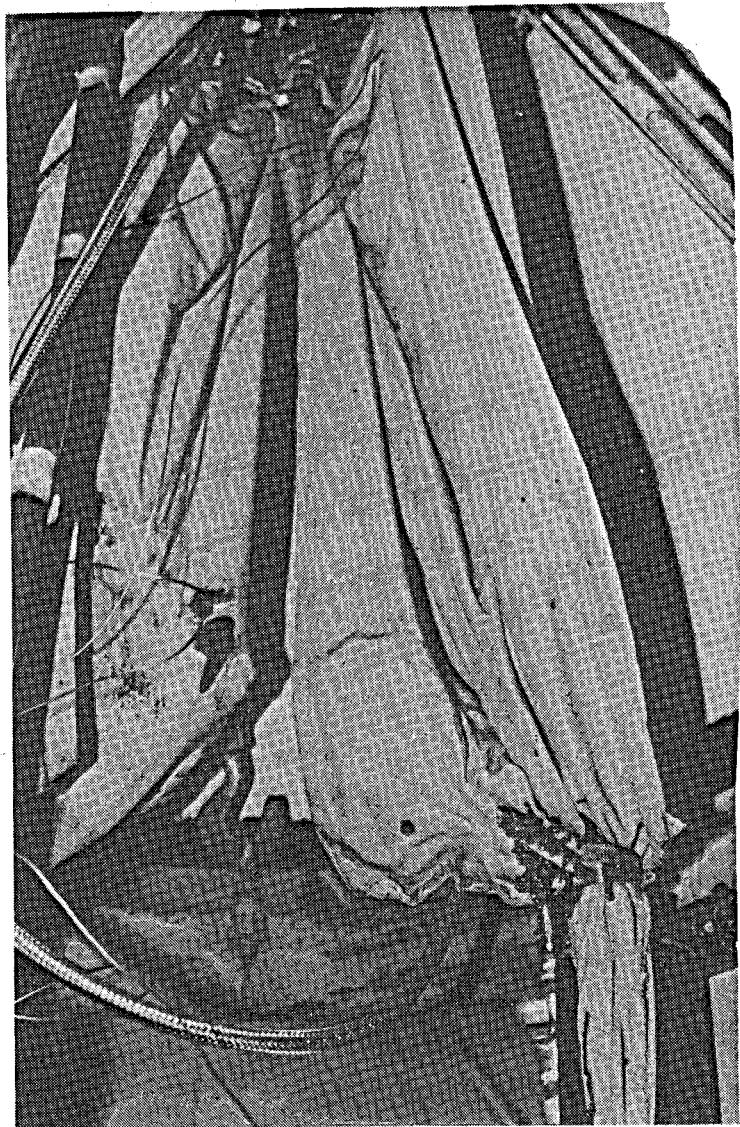


Figure 13. Canvas bags containing foam samples after a test.

It was established after evaluating the physical properties of the samples produced at 100°F, that the physical properties of Formula 3 were superior. Formula No 3 was used for the remainder of the tests as indicated in Tables II and III.

The sequence in which the Poly Plug cartridges were activated are as follows: At 100°F and 0 psig, Formula Nos. 1, 2, and 3 were placed in individual cartridges installed within the test vessel. Cartridges were activated in sequence one through three with all data being recorded between each cartridge being activated. At 100°F and pressures 300 psig through 900 psig, Formula No. 3 was placed within all three cartridges and installed in the test chamber. Nitrogen gas was used to increase the pressure within the test vessel to 900 psig. One cartridge was activated at this pressure (900 psig) and pressure was maintained for 15 minutes. The pressure was reduced to 600 psig by the removal of nitrogen gas. One of the remaining cartridges was activated at the pressure of 600 psig and held there for 15 minutes. Pressure was reduced to 300 psig by again removing nitrogen gas. The remaining cartridge was activated and pressure of 300 psig was held for 15 minutes. The pressure was reduced to 0 psig and the foam samples were removed. This procedure was repeated at temperatures of 200°F, 250°F and 300°F.

TABLE II
DATA COLLECTED AT SWRI

FORM. NO.	TEMP. °F	PRESS. PSIG	WATER TEMP. °F	NITROGEN TEMP. °F	INCREASE TANK PRESS. PSIG	MAXIMUM EXOTHERM TEMP. °F	TIME M.E.T. OCCURS MIN.
1	100	Ambient	104	98	5	372	0.21
2	100	Ambient	105	96	5	N/A	0.33
3	100	Ambient	104	98	5	222	0.425
3+	100	300	100	96	8	165	0.33
3*	100	600	100	98	10	249	0.35
3	100	900	102	96	20	142	1.00
3*	200	Ambient	200	200	N/A	325	0.40
3	200	300	202	175	30	342	0.45
3	200	600	201	170	40	355	1.00
3	200	900	202	178	40	365	1.30
3	250	300	250	250	25	378	0.33
3	250	600	250	217	20	347	0.35
3	250	900	249	217	40	400	0.35
3	300	300	299	289	40	562	3.30
3	300	600	300	296	10	314	2.80
3	300	900	302	290	40	308	0.30

Notes:

+ Foam produced at Poly Plug and at SwRI
* Foam produced at Poly Plug

TABLE III
SUMMARY OF DATA MEASURED AT POLY PLUG
FOAM PHYSICAL PROPERTIES

FORMULA NO.	TEMP. °F	PRESSURE PSIG	DENSITY PCF	CLOSED CELL %	COMP. STR. AMBIENT	COMP. STR. 200°F PSI	4 HR. FLUID LOSS, 100 PSI ML
1	100	ambient	7.2	42	160	160	125
2	100	ambient	6.9	54	175	165	84
3	100	ambient	7.4	47	180	175	55
3	100	300	17.7	72	285	285	104
3	100	600	17.6	66	290	290	84
3	100	900	18.3	62	295	295	72
3	200	ambient	6.6	74	185	185	44
3	200	300	13.6	64	230	230	77
3	200	600	9.7	53	215	215	60
3	200	900	9.4	66	205	205	77
3	250	300	7.9	64	200	195	73
3	250	600	9.1	76	210	210	64
3	250	900	8.6	57	190	180	48
3	300	300	9.9	63	290	290	63
3	300	600	11.2	78	270	270	29
3	300	900	8.1	72	285	285	22

Physical Properties Test

Samples were first visually inspected for physical defects such as cracks, burns, shrinkage or poor external texture. Findings showed all of the samples were of very good quality externally and stable as a mass.

Density was then checked by removing specimens from four samples (approximately one cubic foot each) and testing them by the water displacement method. Smaller specimens were then taken from the same four samples and tested by ASTM Standard No. 1564-58T for comparison. Findings showed the smaller specimens tested were comparable. The densities listed on Table III were established by ASTM Standard No. 1564-58T. Specimens tested were approximately six cubic inches in volume.

To establish the closed cell percentage of each specimen, the specimens used to establish density with known properties were submerged in water for a period of 24 hours. Samples were removed, weighed, and their water absorption was calculated to establish the closed cell content of each specimen. Each sample was crosscut by a bandsaw into one-half inch thick slices. The specimens were inspected for defects such as internal exotherm burns. Compressive strength at ambient and 200°F was established by ASTM No. C165-54 standard test procedure on the one-half inch cut specimens.

Three inch diameter discs were cut from the one-half inch specimens for fluid loss testing. Standard fluid loss testing apparatus and procedures were used. The sketch of Figure 14

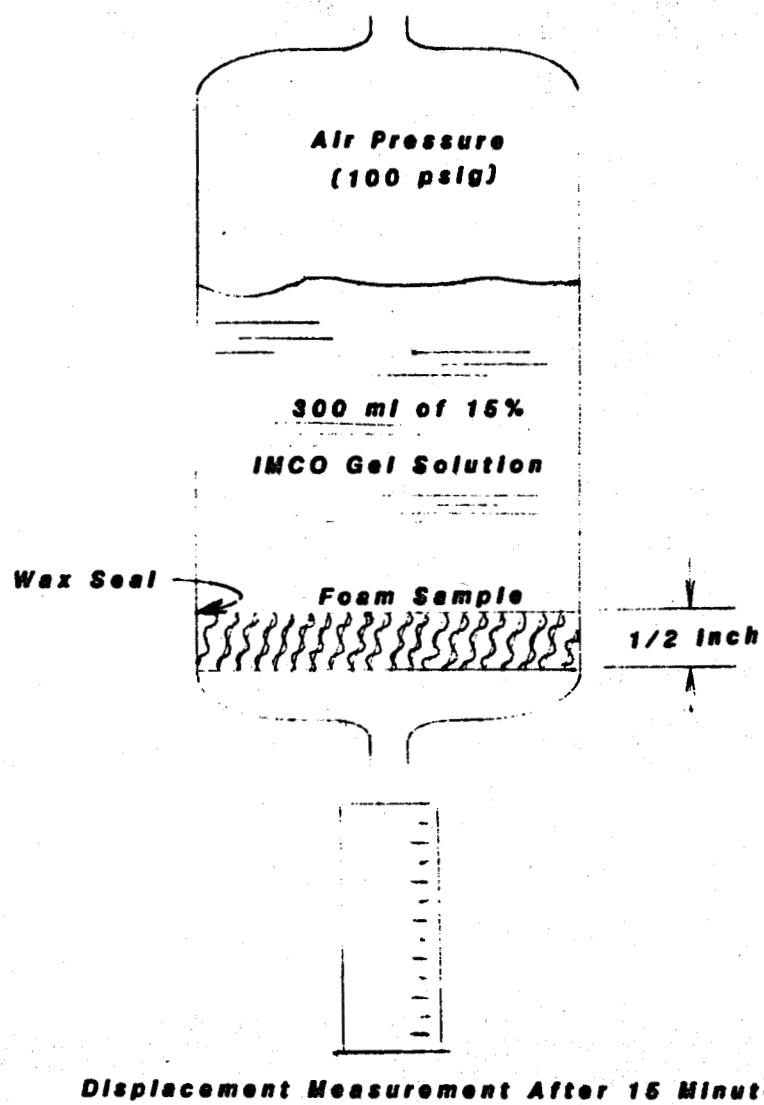


FIGURE 14. Schematic of Fluid Loss Test

depicts the setup for fluid loss tests. The one-half inch thick by three inch diameter discs replaced the filter paper normally used in testing fluid loss. Melted wax was used to seal any pin holes that extended through the specimens and seal its perimeter to prevent irrelevant fluid loss. A water solution containing 15 percent IMCO gel was prepared. The test procedure consisted of placing 300 ml of the gel solution over the foam specimen. Air pressure over the gel solution was increased from 0 psig to 100 psig over a period of two minutes. Fluid loss was checked at 15 minutes for excessive flow. No excessive flow was present in any specimen and testing continued until no flow existed. Fluid loss through specimens subsided well within the four hour test period.

It should also be noted that three foam samples were produced at Poly Plug in Houston to complete the test matrix from results previously obtained at SwRI. Four hundred (400) grams of each of the two components were mixed under elevated temperature and pressure conditions by mechanical mixing for 15 seconds. Table II identifies which foam samples were produced at Poly Plug.

IV. RESULTS

Results of the tests conducted at SwRI to produce the foam samples are given in Table II. Table III summarizes the physical properties data measured by Poly Plug, Inc., after those tests. No exotherm burns were apparent on any of the samples tested at Poly Plug. There was no apparent correlation between closed cell and fluid loss data.

It is interesting to note that, considering the density, compressive strength, and fluid loss properties of all samples, one of the best results was obtained from the 300°F, 900 psig sample. This is the most severe environmental condition under which foam was produced, and it was also one of the last samples made at SwRI.

This test program established that high temperature polyurethane can be expanded to a foam state from a free rise density of approximately 7 lb/ft³ at a temperature of 100°F and ambient pressure and retain a density as low as 8.1 lb/ft³ at a temperature of 300°F and a pressure of 900 psig. The density of the polymer constituents before foaming is 75 lb/ft³. From samples produced, physical properties were established and evaluated for the effects of temperature and pressure. The evaluation establishes that properly designed, high temperature polyurethane produces an acceptable finished product at all temperatures and pressures as set out in the matrix of this test.

Pressure affects the urethane foam by increasing the density which also inherently increases the compressive strength. These changes occur in the initial pressure increase from 0 psig to 300 psig. (See Figures 15 and 16.)

Physical properties of foam produced under pressures of 300 psig through 900 psig have no noticeable difference.

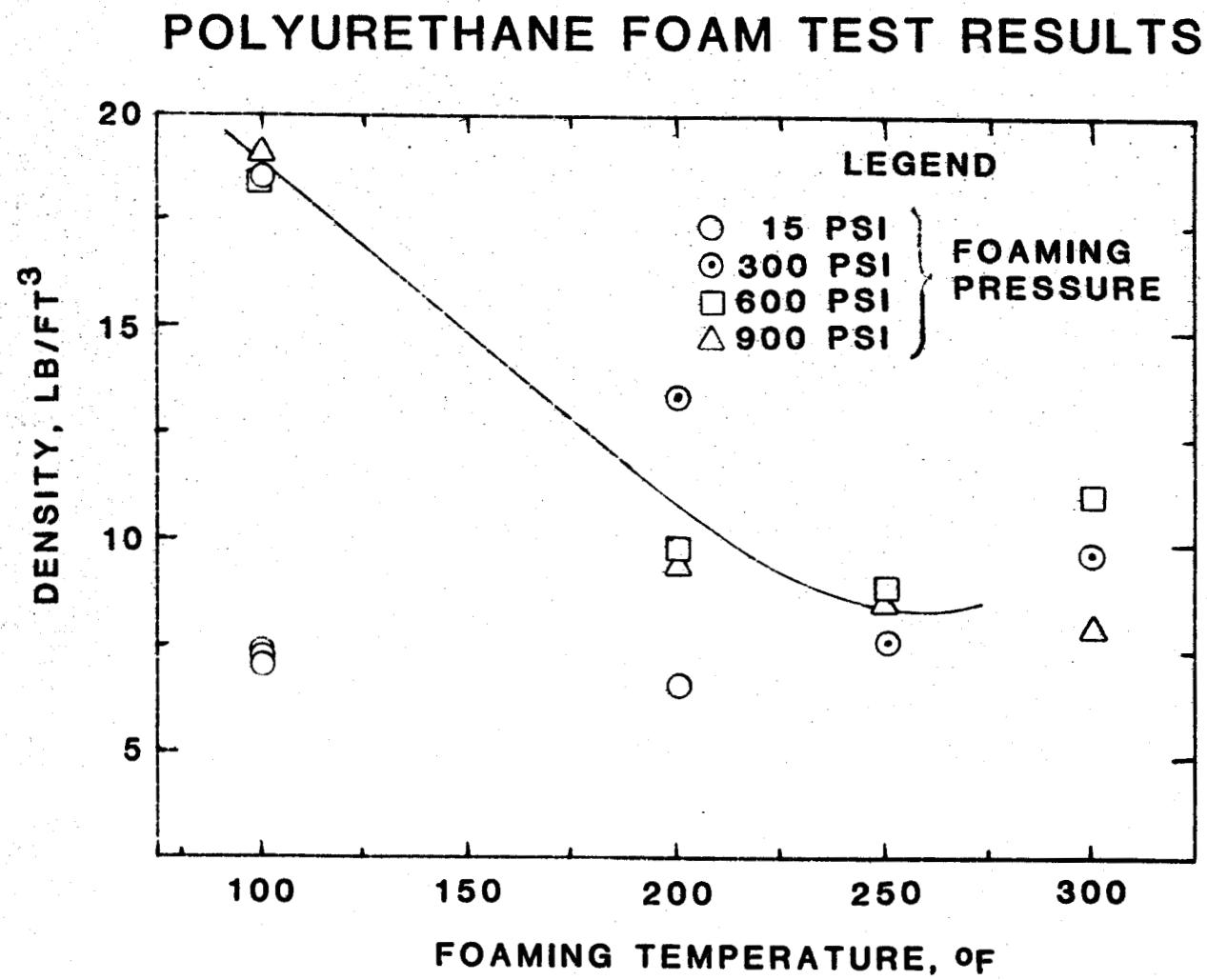
This effect is more noticeable at 100°F than at 200°F.

Temperature affects the urethane foam by improving the physical properties. These changes occur in the initial temperature increase from 100°F to 200°F. As the temperature is increased from 200°F to 300°F, there is no noticeable difference (see Figure 15). The initial increase in temperature represents the effect of temperature on foam produced, and any additional increase in temperature has no apparent effect.

The change produced by temperature is due to the fact that the viscosity of the urethane constituents is lowered by the temperature increase from 100°F to 200°F and above. The lower viscosity improves flowability of the constituents, thereby, the increase in temperature enhances the mixing ability of the static mixers used in this test.

Both temperature and pressure have an effect on fluid loss through the urethane foam. As pressure and/or temperature is increased during the formation of foam, its ability to stop fluid loss improves. This is illustrated by Figure 17.

FIGURE 15. DENSITY PROPERTIES OF THE FOAM



POLYURETHANE FOAM TEST RESULTS

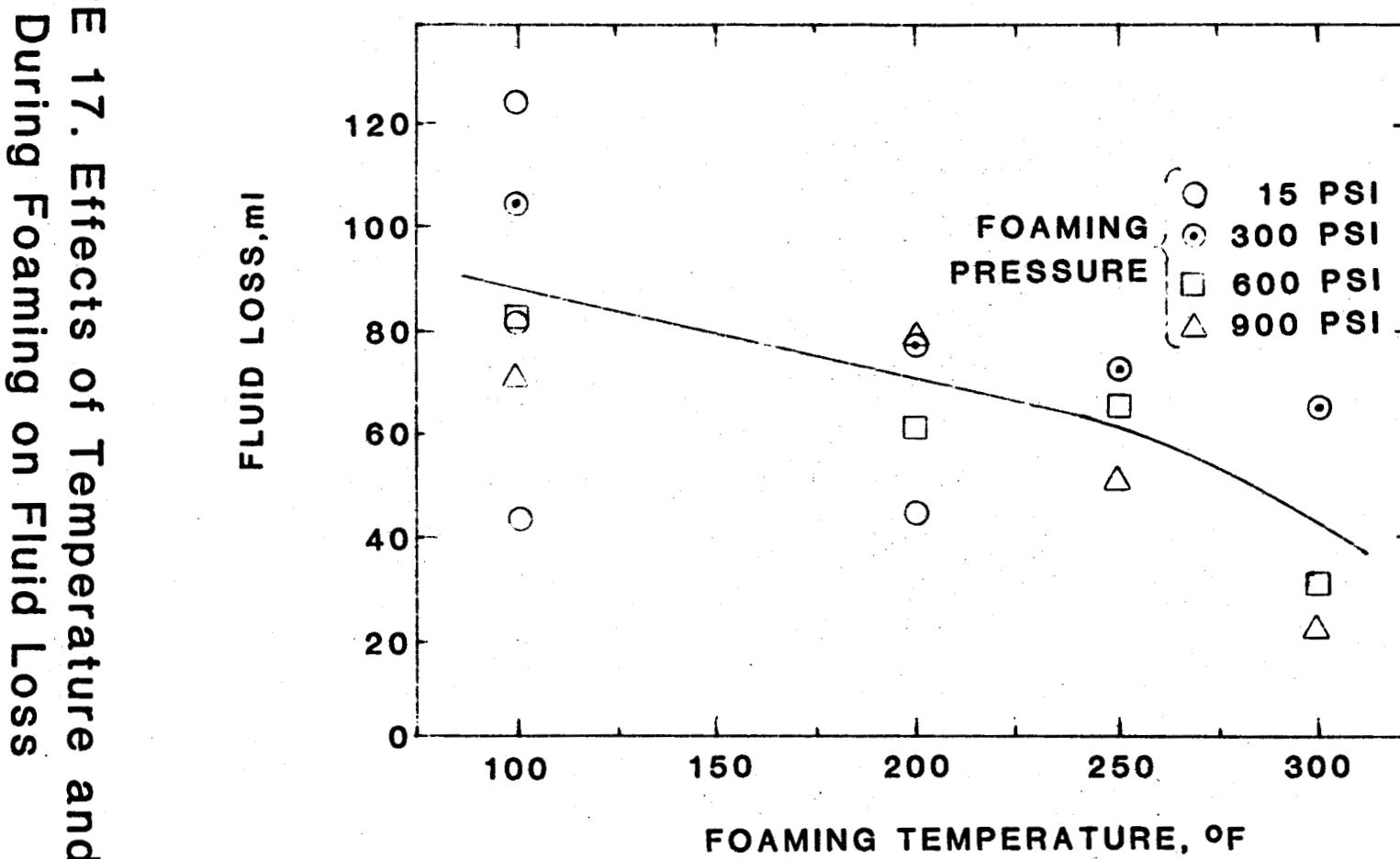
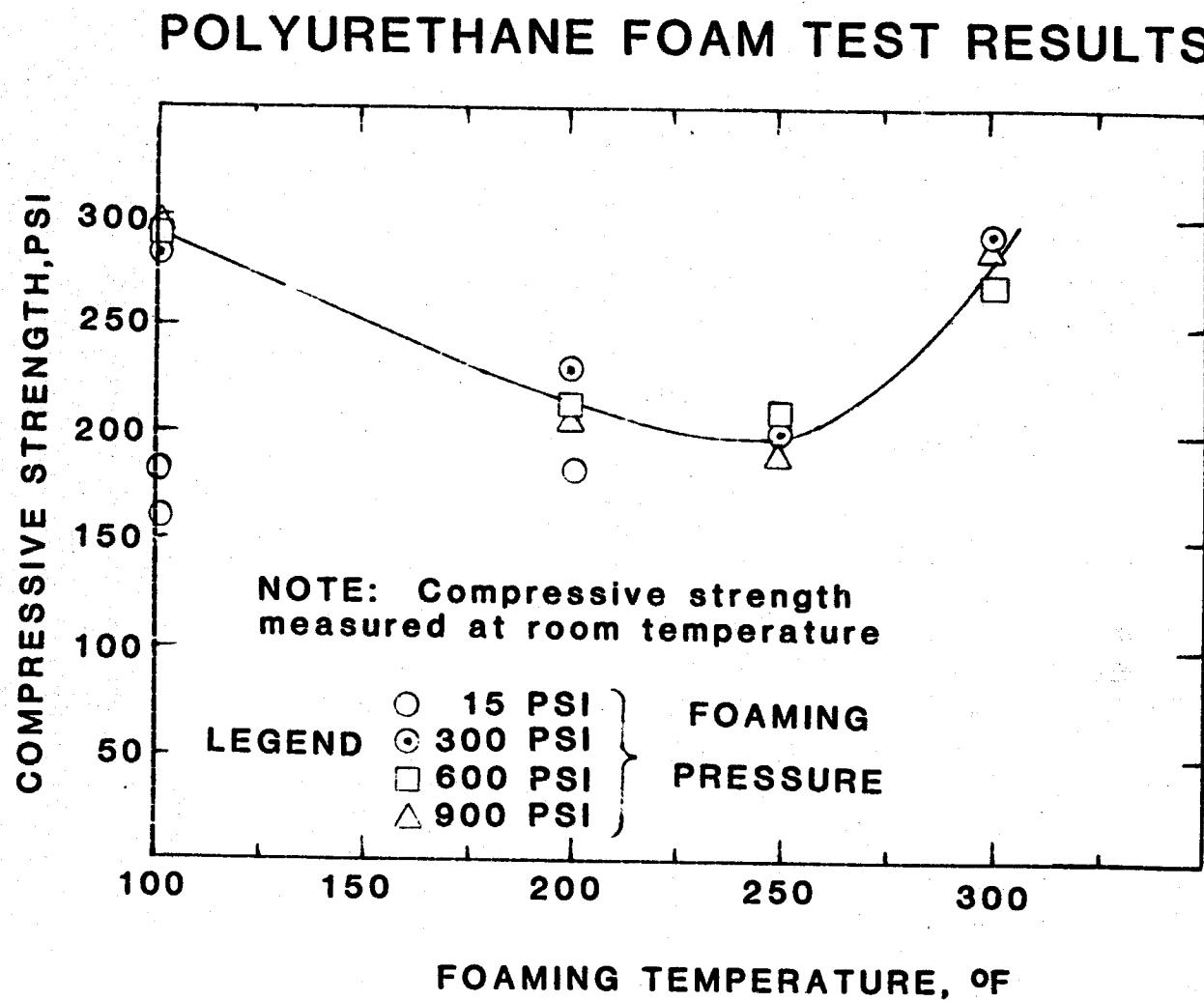


FIGURE 17. Effects of Temperature and Pressure During Foaming on Fluid Loss

FIGURE 16. COMPRESSIVE STRENGTH PROPERTIES OF THE FOAM



V. PROBLEM AREAS

By far the greatest difficulty encountered during the test at SwRI was premature mixing of a small portion of the two foam components resulting in clogging of the mixing chamber. This prevented mixing of an acceptable quantity of foam into the canvas bags. If the two mixing chambers of the cylinder are completely filled with the two components, when the cylinder is heated inside the pressure vessel, thermal expansion will cause a small quantity of the two components to mix in the mixing chamber. Although several cures for this problem were devised (orientation of check valves, purging lines with nitrogen, etc.), the most practical solution appeared to be as follows.

The two chambers of the cylinder were partially filled with the two constituents of the foam, and the rest of the volume of the two chambers was filled with inert gas. The inert gas, being a compressible fluid, was not nearly as sensitive to thermal expansion as the liquid constituents of the foam, and the problem was thereby resolved.

Another problem became apparent due to the fact that vapor pressure of constituent B of the foam was greater than the vapor pressure of constituent A. Acting on the same area within the modified cylinder, constituent B tended to displace a small quantity of constituent A into the mixing chamber as temperature increased. This problem was alleviated by installing constituent B into the rod end of the modified cylinder

while constituent A was on the cap end of the cylinder. Thus, constituent A, although it had a lower pressure than constituent B as temperature increased, acted on a larger area than constituent B. Forces inside the cylinder were thereby balanced and neither constituent was displaced prior to mixing time.

Some logistics problems were experienced in the laboratory that probably would not be experienced in the field. Downtime between tests due to the necessity of cleaning mix chambers and other components is one example. Also, thermal capacitance of the large 48-inch diameter pressure vessel required considerable time between tests in order to raise the temperature of the vessel.

VI. CONCLUSIONS

The results obtained through this testing program indicate that high temperature polymeric foam of low density and good physical properties can be produced under conditions equivalent to those experienced in drilling for geothermal energy. The objective with urethane foam, as with any lost circulation product, is to securely plug and stop fluid loss in cracks, holes, fissures, and porous stratas. Even though samples produced at all temperatures and pressures performed well when tested for fluid loss, as temperatures and pressures were increased, as would be experienced when drilling for geothermal energy, these properties improved consistently.

In field use, physical separation of the two foam constituents using positive sealing valves (as opposed to check valves) is recommended. Also, metering of the two foam constituents downhole at the proper rate might be accomplished using a flow control valve which incorporates a viscous damper in the design.

While laboratory results to date appear favorable, it is recommended that a field trial be instituted as further proof of the suitability of polyurethane foam systems in geothermal wells.

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