1988 SCA Conference Paper Number 8807

AN IMPROVED LABORATORY SYSTEM FOR MEASURING THE ELECTRICAL RESISTIVITY OF SMALL CORE SAMPLES AT RESERVOIR CONDITIONS

BY

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ABSTRACT

Considerable attention has been given, during the past three years, to problems associated with measurement of the electrical properties of core samples. Attendant research efforts have recently begun producing significant improvements in the equipment and methods employed in these measurements. This paper describes one of these new test systems and presents examples of its use in defining temperature and pressure effects on the electrical properties of cores.

The analytical equipment discussed can provide two and four electrode measurements at reservoir conditions. Operating capabilities include confining pressure to 10,000 psi and test temperatures to 300°F. The system can handle both consolidated and unconsolidated cores. Sample desaturation is achieved in the test vessel and the level of desaturation determined without removing the sample from the vessel. This equipment is also capable of making capillary pressure determinations at elevated temperature and pressure.

Unique design features of this equipment correct causes for most of the electrical interferences encountered in operation of conventional systems. Other sources of error eliminated include: grain loss and redistribution of pore fluids which occurred upon removal of the sample from the test vessel for saturation determinations and evaporation of pore brine during these operations.

Data from studies with this equipment reflect the expected trends in sample resistivity with increasing temperature and pressure. In addition, the data suggest that the rate of these changes are affected by a number of factors, including the porosity, permeability, and composition of the sample.

Results from this work are expected to produce better correlations with downhole logs and better understanding of electrical properties at formation temperature and pressure. These studies are also expected to provide a broad basis for future research efforts in electrical properties technology.

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INTRODUCTION

The use of electric well logs in petroleum reservoir evaluation has become widely accepted since Archie's basic work with clean sandstone formations in the 1940's(1,2). Correlations between these logs and formation characteristics defined at that time were later extended to shaly sandstone reservoirs by Hill, et al,(3), Waxman and Smits(4), and Waxman and Thomas(5). In 1986, Givens(6) expanded these correlations to cover most other types of reservoir rock.

Effective use of electric well logs in reservoir evaluation, however, depends heavily upon the accuracy with which electrical property measurements can be made on related core samples in the laboratory(1). Laboratory measurements of this type have historically been complicated by a number of factors. Some of these problem areas were discussed by Worthington(7) in 1975. A number of others were defined in a broad study conducted by the Society of Core Analysts(8) in 1986 and 1987. More recent studies in our own laboratory have revealed additional sources of errors involving both the methods and equipment commonly employed in making the measurements. Some of these problems are of such magnitude that they can preclude the generation of reliable electrical data. The more important of problems and available solutions to them are discussed in detail in this paper.

Using results from this series of studies, an improved electrical properties measuring system has been developed for laboratory use. Details of this system are included in this paper. Also included are examples of data produced by the system in tests at elevated temperature and pressure.

EXPERIMENTAL

For some time prior to this study, we had been concerned about occasional erratic behavior of our electrical properties test equipment and the associated production of suspect test data. One particular set of highly questionable data prompted an in depth examination of this equipment and the test procedures employed. Early results revealed a number of subtle and previously unrecognized electrochemical and mechanical problems that we felt demanded further investigation. The test equipment employed in these initial studies was considered typical of that used by most of the petroleum industry at that time. It lacked the flexibility needed, however, to support the planned study. As a result, the prototype system shown schematically in Figure 1 was put together to continue this work. Figure 2 gives details of the core holder/ test cell designed for use in this study. This prototype system is a modified version of equipment employed earlier by Waxman and Thomas(5), Swanson(9), and Longeron, et al(10), in their investigations.

Capabilities of this new test equipment included confining pressures to 10,000 psig and sample temperatures to 300°F. Core sizes up to 1.5 inches in diameter and 3.0 inches in length could be accommodated, as well as consolidated and unconsolidated samples. Sample desaturation could be achieved and the level of sample saturation level determined for each data point without removing the sample from the test cell. This very important capability was made possible in part through use of porous ceramic disks in the drainage end of the sample holder. A sensitive collection system for fluid displaced from the core sample during the desaturation process completed this modification. Disks which permitted capillary pressure determinations to 220 psig were employed. In addition, the sample holder was totally electrically isolated (no common ground) from the pressure vessel to insure the integrity of the test signal. Two and four electrode resistivity measurement capabilities were included in the sample holder design. It was felt that this combination of performance capabilities would allow testing of samples under most of the reservoir conditions normally encountered in case studies of this type.

The sample preparation and electrical property test procedures employed in this study were typical of those currently employed by the petroleum industry. Samples to be tested were first cleaned by Dean-Stark or toluene/carbon dioxide extraction techniques. Rock matrix characteristics of the clean samples were determined by conventional means. Sample saturation with brine prior to electrical testing was achieved through use of the vacuum evacuation/pressure cycling technique. Brine saturation pressures up to 3,000 psig were required in some cases to achieve desired saturation levels. In all of our tests, brine was employed as the wetting phase and nitrogen as the nonwetting, displacing phase.

Samples prepared in this manner were placed between end caps of the appropriate size and sealed in a Viton sleeve. The assembled sample holder was subsequently placed in the pressure vessel and subjected to the desired confining pressure and test temperature. Confining pressure was supplied by a high boiling point oil pumped into the confining vessel by a small positive displacement pump. Pressure was maintained at the desired level through a sensitive pressure transducer feedback to the pump. After temperature and pressure equilibrium had been achieved in the sample at the desired test levels, Formation Factor (FF) determinations, if desired, were made. Sample desaturation was then initiated by increasing nitrogen pressure on brine in the pore system of the core sample as shown in Figure 1. Nitrogen pressure increases were in controlled increments as shown in Figure 3. After brine displacement ceased (pressure/displacement equilibration achieved) at each selected displacement pressure, electrical resistance and sample saturation measurements necessary for Resistivity Index (RI) calculations were made. This process was repeated until the desired test limits had been achieved. The volume of brine displaced from the test core was monitored continuously throughout these incremented pressure steps and was employed to calculate needed core saturation levels at each of the test pressures. As noted earlier, it was not necessary to remove the sample from the pressure vessel to determine its level of desaturation.

Table I contains a description of core samples selected for use in this research effort. All of these samples were subjected to routine electrical property tests using the equipment and procedures described in the preceding paragraphs. Anomalous equipment behavior and other possible causes for suspect data generation were investigated during these tests. Results are presented in the following paragraphs.

TABLE I

BASIC PROPERTIES OF SAMPLES USED IN STUDY

SAMPLE ID	POROSITY (%)	PERMEABILITY TO NITROGEN (mD)	GRAIN DENSITY (g/cc)	TYPE OF FORMATION
1	22.17	336.69	2.650	Berea Sandstone
2	19.09	116.09	2.639	Berea Sandstone
3	18.77	103.47	2.643	Berea Sandstone
4	21.46	237.80	2.642	Berea Sandstone
5	19.01	92.75	2.652	Berea Sandstone
A	21.97	382.37	2.631	Berea Sandstone
B	19.79	159.32	2.642	Berea Sandstone
C	21.40	391.66	2.640	Berea Sandstone

RESULTS - TEST EQUIPMENT DEVELOPMENT

Several potential causes for problems observed in conventional laboratory resistivity measurements on core samples were discovered. Most were electrical in nature and inherent in the basic design of the test system. Among the more important were: (1) junction potentials generated by contact between dissimilar metals in the sample holders; (2) rather large battery effects which resulted from the immersion of these dissimilar metals in the test brine; (3) the presence of DC riders on the AC signal used in making resistance measurements on the test samples; (4) electrolysis of the brine, sample, and sample holder components caused by these DC voltages, and (5) solubility of the brine displacement gas (nitrogen) in the brine saturating the test samples.

Items (1), (2), and (3) produced direct currents between the electrodes of the sample holder. Combined DC voltages in excess of 2.4 volts were observed in some cases. The major effects of this DC voltage on the test system were the problems noted in (4), electrolyses of the brine and degradation of sample and sample holder. Electrolysis of the brine, as would be expected, produced chlorine and oxygen at the anode and hydrogen at the cathode of the test cell. The free chlorine and oxygen gases chemically attacked all reactive materials with which they came in contact. An example of the high level of chemical reaction involved is given in Figure 4. The Series 316 stainless steel screen shown in this picture was almost completely destroyed in less than three weeks of RI testing. The test sample in this case, a carbonate, also obviously suffered severe damage. This type of damage was found to be quite common, though at somewhat lower levels, in most of the tests performed in this phase of our work.

Not only did these gases produce chemical damage within the test sample, but also caused displacement of brine from the sample, itself, and from the brine exit tubing of the test cell. This excess brine, at first, was collected and erroneously included in the considered volume displaced from the test sample by nitrogen during RI and capillary pressure determinations. Errors produced in the resulting sample saturation calculations were quite significant, in some cases approaching 10% of total sample saturation. The presence of these electrolysis gases in the test cores also caused another type of error. Their displacement of the test brine in the cores altered the conductivity of these samples. It is likely that all of the errors of these two types would probably go undetected in conventional RI and capillary pressure tests, and lead to the erratic and erroneous data often observed.

In addition, DC riders were found on the AC signals of even the best of AC signal generators available for use in this type of electrical property testing. These DC riders, though small, often become significant when compared with the 1.0 VAC normally employed in these tests. This DC rider problem is complicated even further by the fact that the value of the rider voltage changes considerably as the AC frequency selected for use in these tests is varied from a few Hz to several MM Hz. Effective filtering of the DC signal is possible but difficult in this application because of the low AC and DC voltages normally involved.

As noted in error source (5) listed above, the solubility of the brine displacing gas (nitrogen) in the test brine also produced significant errors in the test data obtained with conventional equipment. This error arises from the fact that the amount of gas dissolved pore pressure in the brine increases with increasing (nitrogen) on the test sample. This level of solution approaches equilibrium with time under a given set of pressure/temperature conditions. Each time the sample is removed from the pressure chamber of a conventional test system to determine by weight, X-ray or NMR methods its level of saturation, some of the dissolved gas flashes within the brine solution because of the reduced pressure (atmospheric) to which the sample is subjected. This flashing of the nitrogen expels additional brine from the test core. Resulting saturation errors of this type were found to amount to several percent on a cumulative basis during RI determinations. The rate of brine loss to this cause obviously would depend upon the test temperature, the existing level of nitrogen saturation, and the basic porosity and permeability properties of the test sample.

Dissolved gas flashing (breakout) could also cause problems even in systems of the type developed for use in this research study, systems in which sample saturation levels are determined without removing the sample from the pressure vessel. As the brine exits the test cell in normal pressure displacement, for example, it may be allowed to enter a collector maintained at a pressure below the test cell pore pressure. In these cases, nitrogen gas may flash in the exit tubing of the test cell, displacing brine from part or all of this tubing. This brine from the tubing then becomes a part of the volume of the collected fluid as the nitrogen displaces it. The amount of flashing (brine displacement) cannot be readily determined during sample testing. We found, however, that this flashing error could be eliminated by maintaining a slight backpressure on the brine exit tubing of the test cell.

RESULTS - FF AND RI DATA FROM RESERVOIR CONDITION TESTS

After further modification of the prototype test system to eliminate the electrical and mechanical errors discussed in the previous section, this equipment was used to measure the effects of elevated temperature and pressure on the electrical properties of selected core samples. Given in Figure 5 are FF data obtained by increasing confining pressure on five different samples. These data are consistent with the expected loss of brine (pore volume reduction) from the conducting path within the sample as pressure on the sample is increased.

Figure 6 shows the effects of increasing temperature on measured FF data. These data suggest that several factors are probably contributing to the observed results. Reductions in pore volume are known to accompany increases in sample temperature at reservoir pressure(10). Data from our own efforts to measure this effect, shown in Figure 7, agree with this conclusion. Note the large differences in the brine volume expected to be displaced from the test sample by simple thermal expansion of the brine and the displaced volume actually observed. Increased test temperatures are also known to decrease the resistivity of brine solution(11,12). ARPS data(12) and results of our own measured data for the test brine employed are given in Figure 8. These two factors, and possibly others, combine to give the decrease in sample FF shown in Figure 6.

Given in Figure 9 for comparison are results from two and four pole electrode FF measurements on the same sample made with the prototype equipment. The agreement between these sets of data appear to be quite good, providing increased validity for the electrical measurements involved.

Figure 10 contains an example of RI data generated with the new test system. Confining pressure in these tests was 6,000 psig. Test temperature was 250°F. Figure 3 contains the actual desaturation profile data observed for these particular tests. It is obvious that capillary pressure data, if desired, could be determined simultaneously with the RI data at each test point.

These examples of performance data are presented simply to demonstrate the utility of the new electrical properties measuring system at elevated temperature and pressure. More complete case studies on a variety of test samples are the subject of another paper now being prepared.

CONCLUSION

This research effort has resulted in an improved electrical properties measuring system for use with core samples in the laboratory. The system is capable of two and four electrode measurements. Formation Factor, RI, and capillary pressure measurements can be made at elevated temperature and pressure (reservoir conditions). Several electrical and mechanical problems experienced with conventional test systems have been eliminated. The most important of these problems, erroneous saturation and resistivity data and damage to sample and test cell resulting from electrolysis, have been defined and controlled.

In addition, sample desaturation is achieved within the test cell and desaturation levels determined without removing the sample from the pressure vessel. This capability has eliminated problems (grain loss, pore fluid loss, and brine redistribution within the sample) caused by frequent removal from the test vessel to determine saturation levels. The new system is also designed to handle both consolidated and unconsolidated test cores with equal ease.

Results from this research effort have demonstrated the feasibility of making electrical property measurements on core samples in the laboratory under reservoir conditions. This capability is expected to provide better understanding of changes in electrical properties caused by elevated temperatures and pressures, and to generate data which better correlates with downhole logs. These results are also expected to provide a broad basis for future research efforts in electrical properties technology.

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Figure 1

SCHEMATIC OF PROTOTYPE RESERVOIR CONDITIONS RESISTIVITY AND CAPILLARY PRESSURE TEST SYSTEM

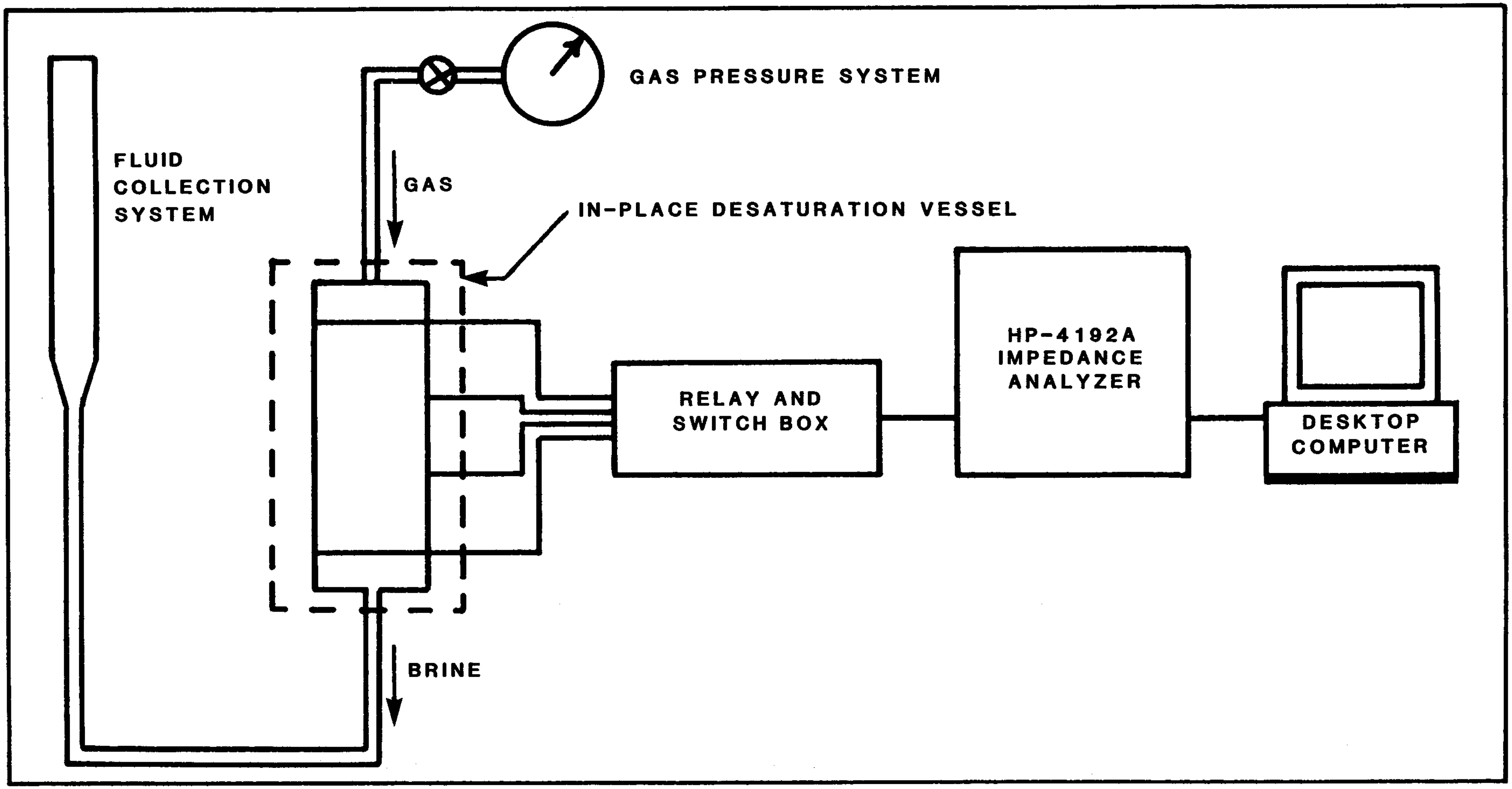

Figure 2

DETAILS OF CORE HOLDER DESIGNED TO IMPROVE TEST FLEXIBILITY

Four Electrode System With Porous Disk

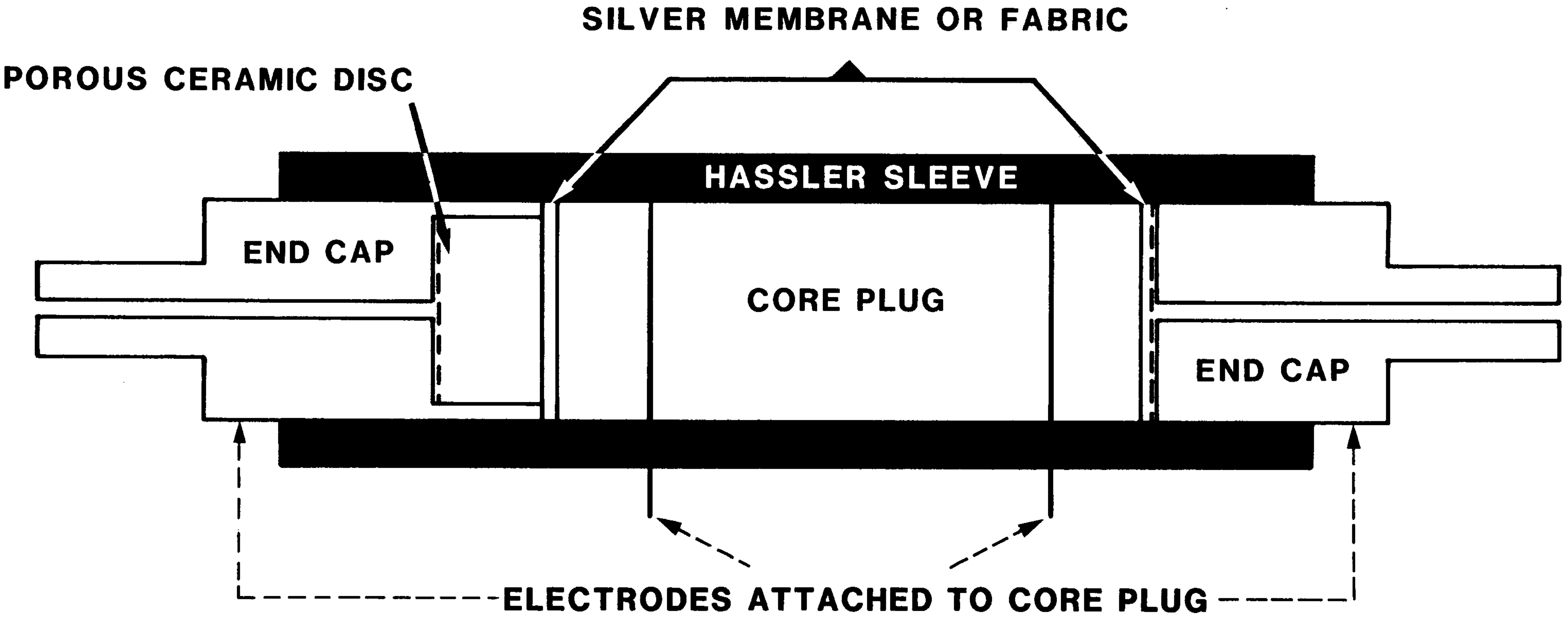

Figure 3

DATA FROM TYPICAL PORE PRESSURE/DESATURATION STUDY ASSOCIATED WITH RESISTIVITY INDEX DETERMINATIONS ON CORE SAMPLE

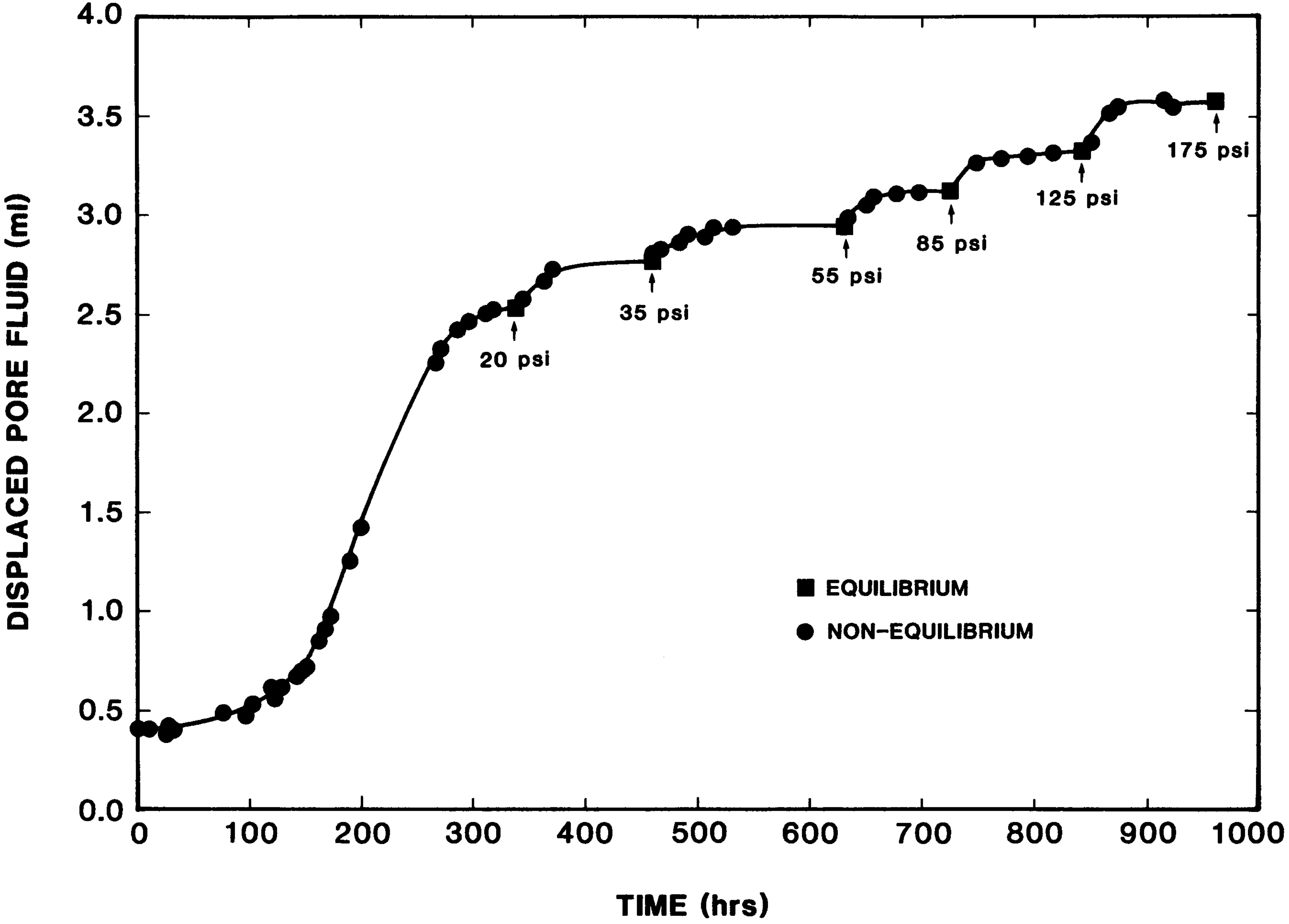

Figure 4

DAMAGE TO SERIES 316 STAINLESS STEEL SCREEN
AND TEST SAMPLE CAUSED BY ELECTROLYSIS IN THE SAMPLE CELL

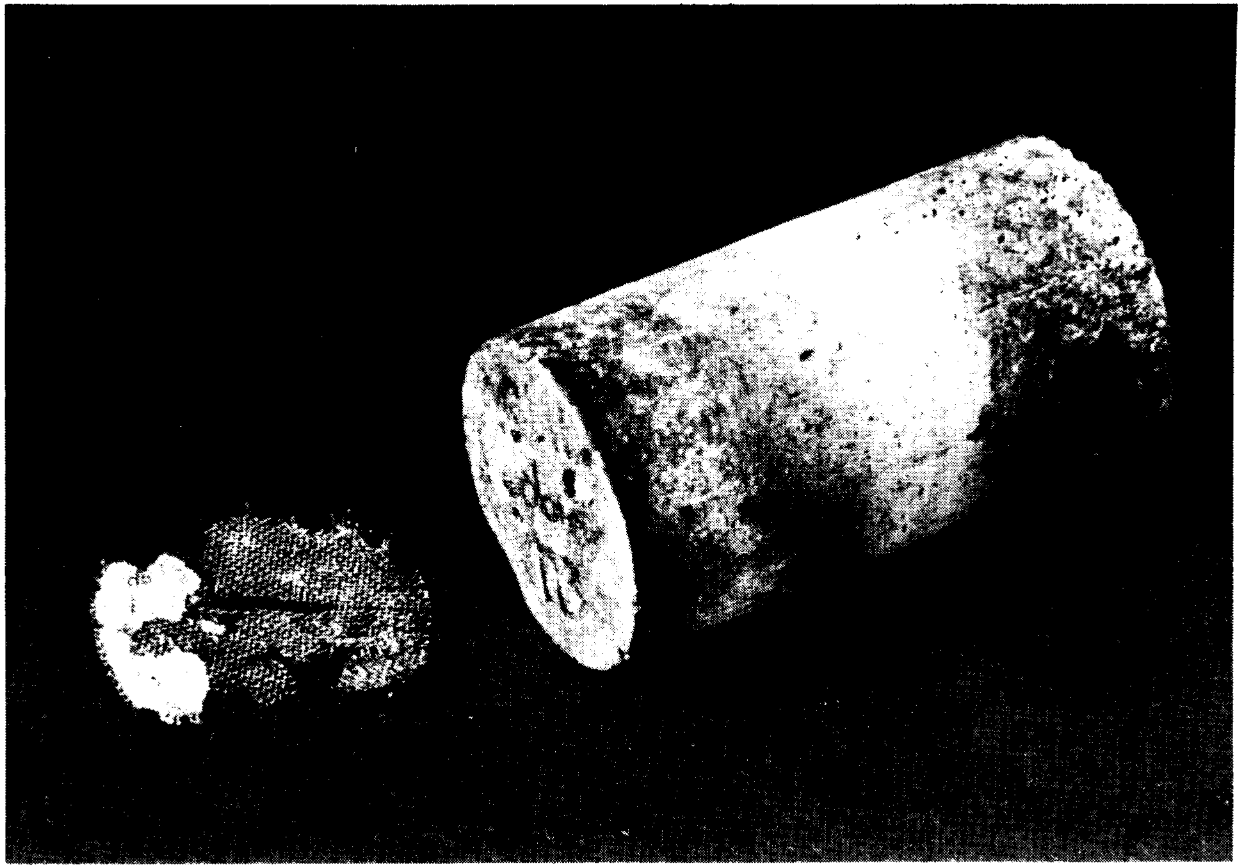

Figure 5

EFFECTS OF INCREASED CONFINING PRESSURE ON FORMATION FACTORS

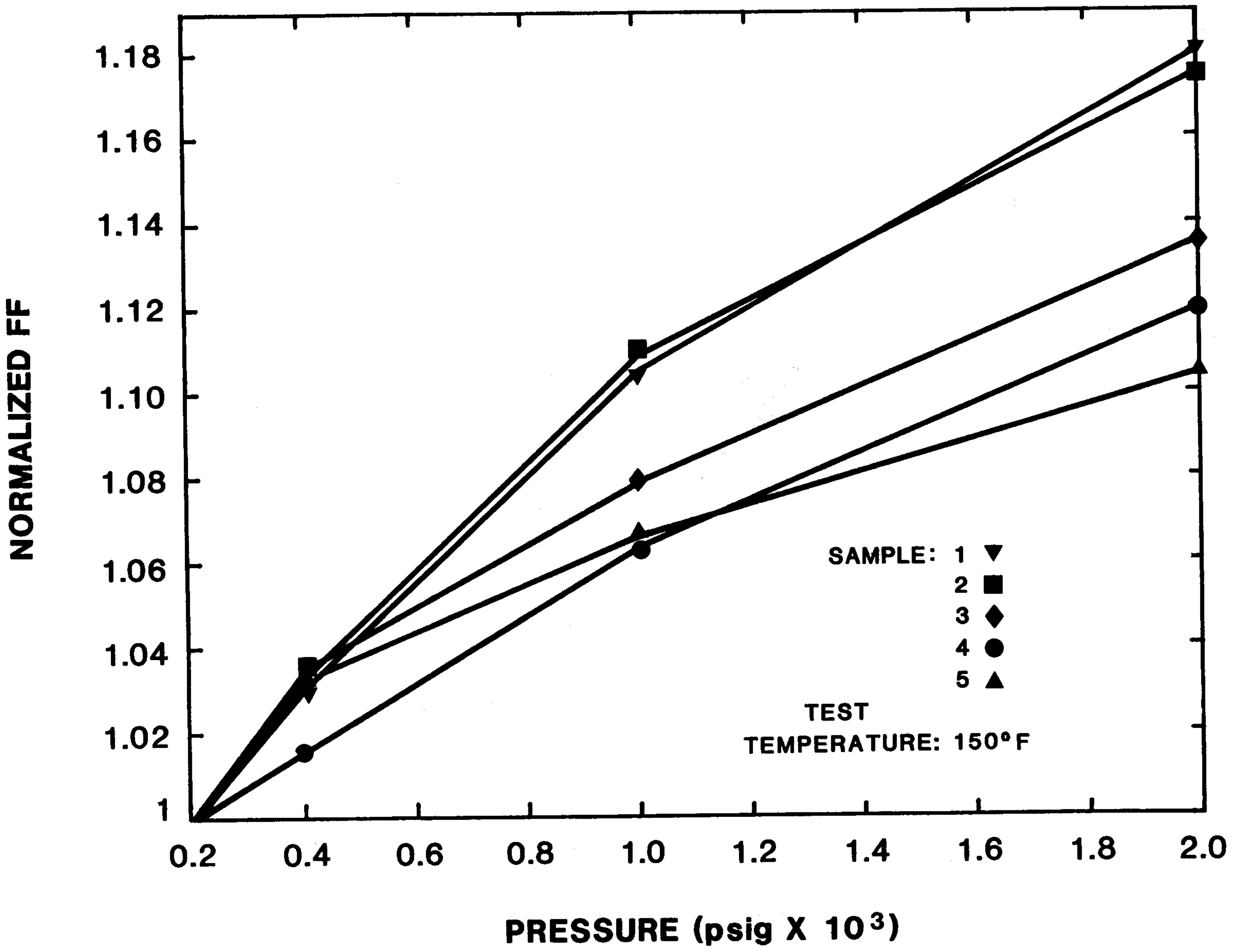

Figure 6

EFFECTS OF TEMPERATURE ON FORMATION FACTORS

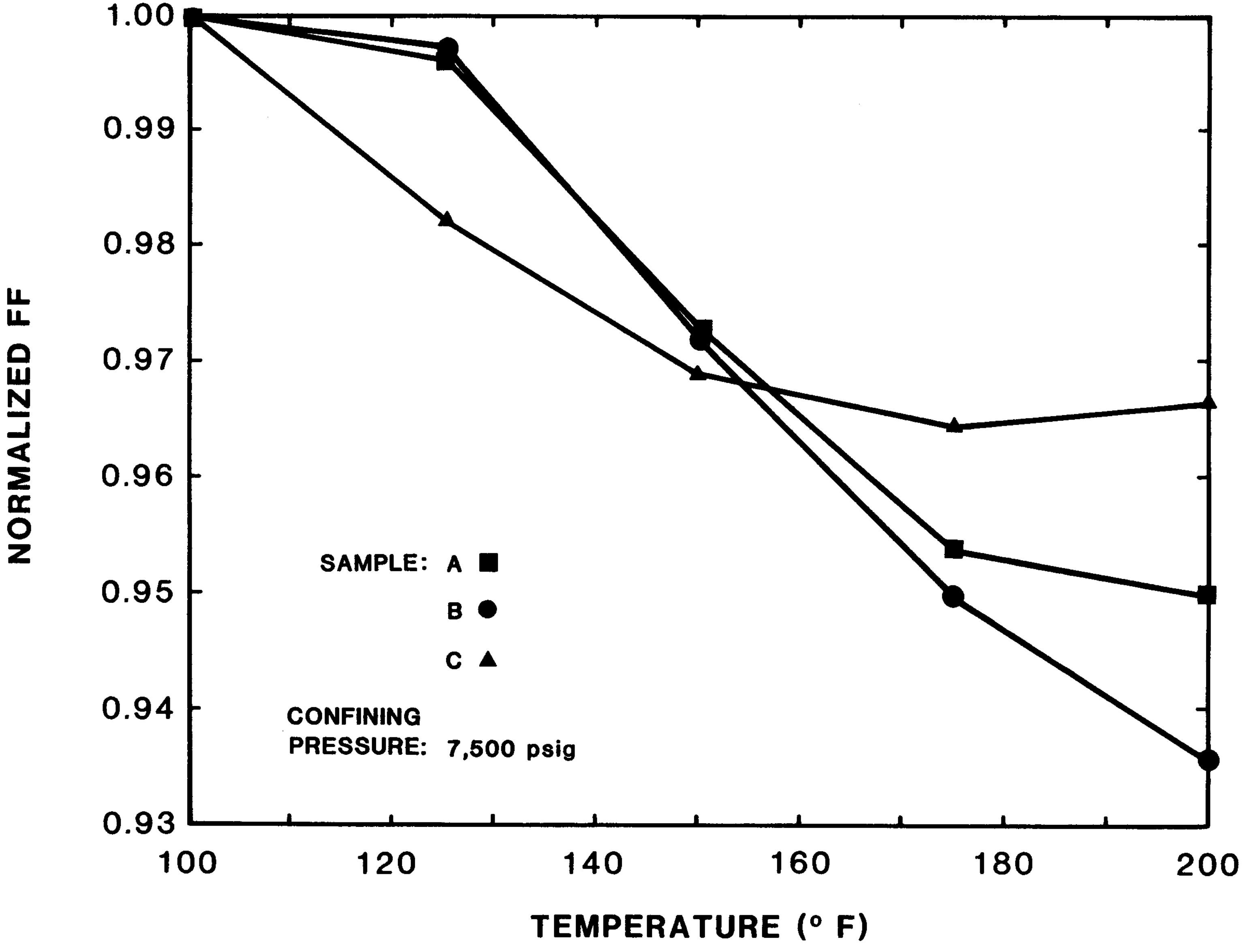

Figure 7

EFFECTIVE PORE VOLUME REDUCTION IN TEST SAMPLE
CAUSED BY INCREASED TEMPERATURES
AT RESERVOIR PRESSURE

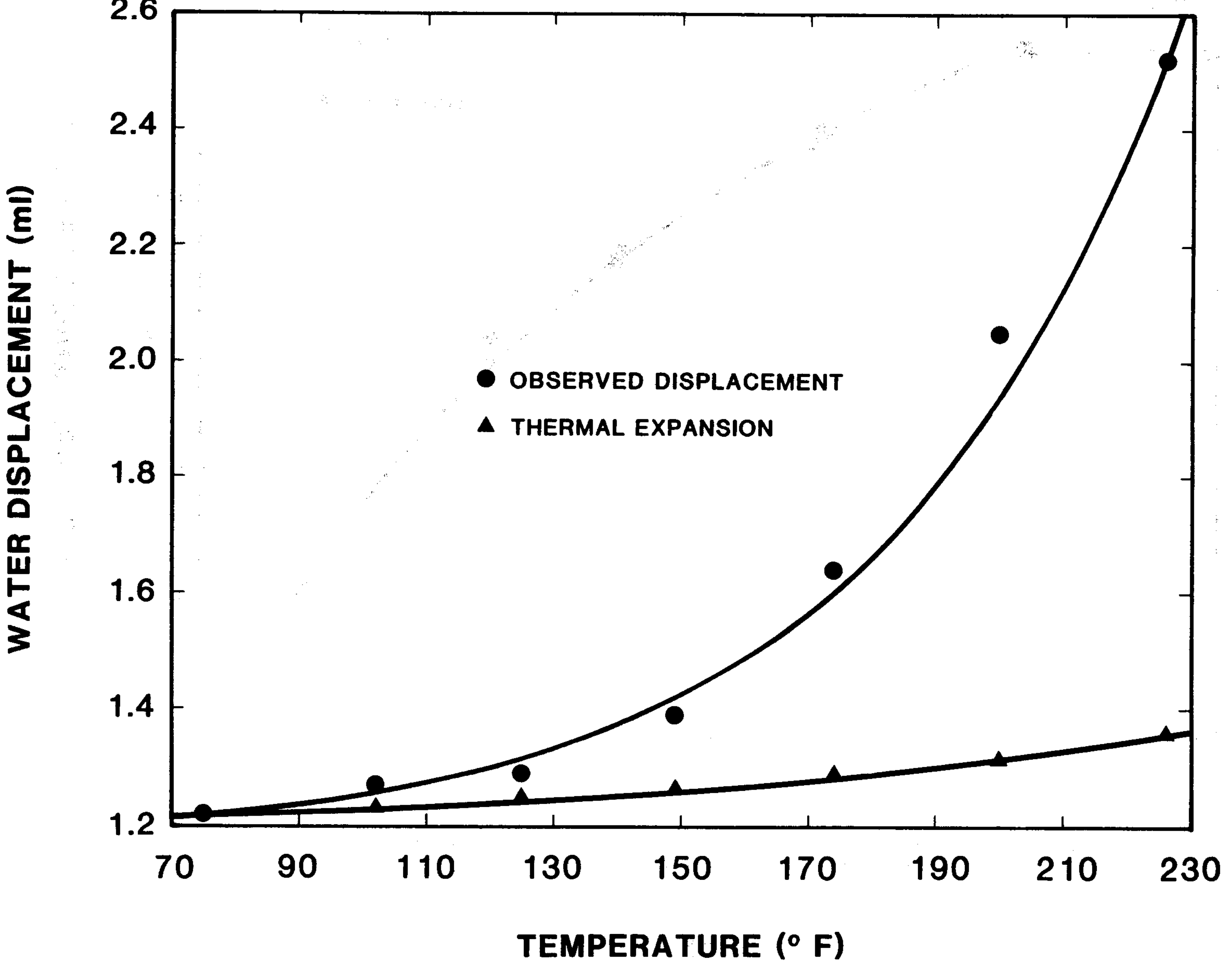

Figure 8

BRINE RESISTIVITY VS TEMPERATURE

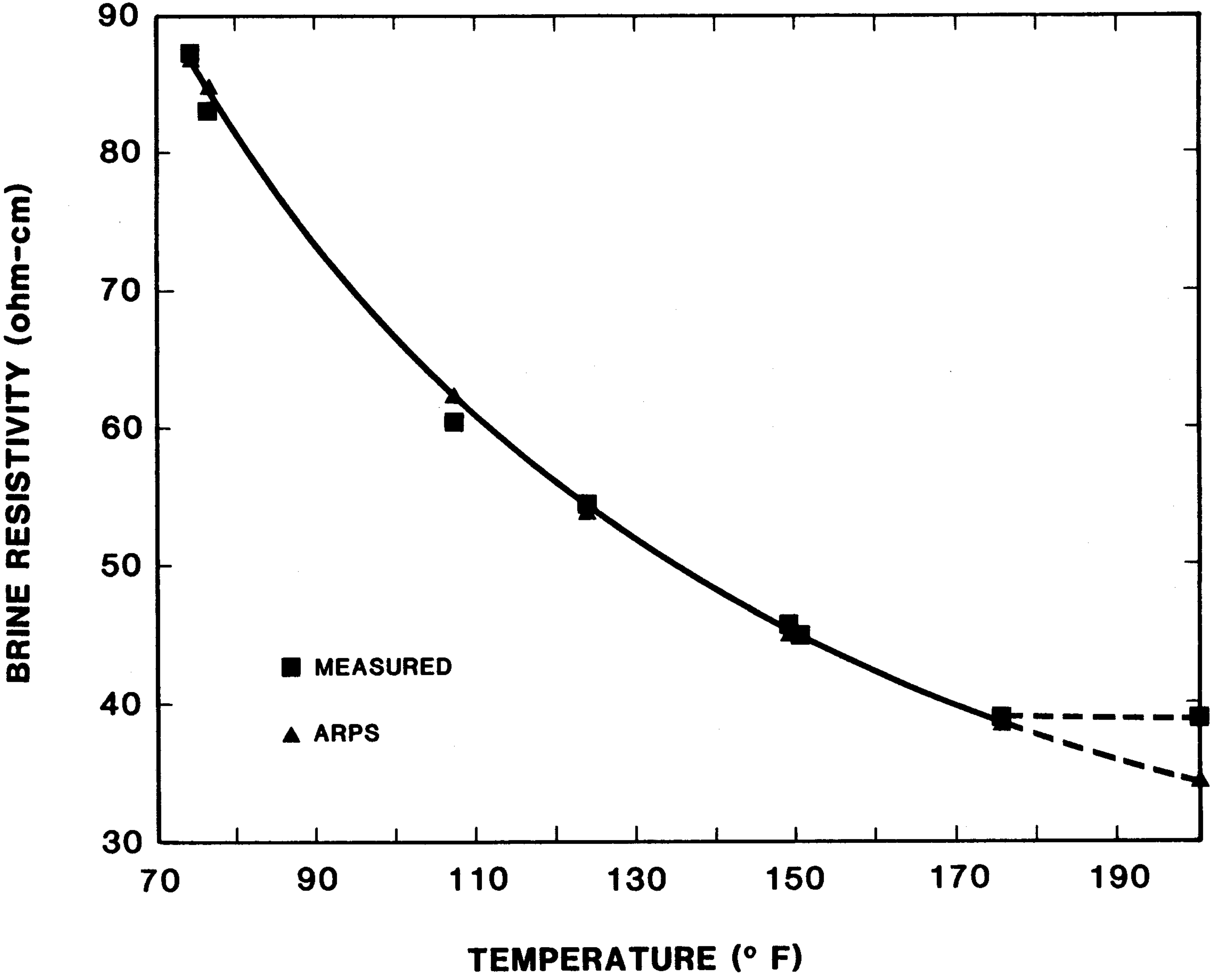

Figure 9

COMPARISON OF TWO AND FOUR ELECTRODE FF DATA GENERATED WITH NEW TEST SYSTEM

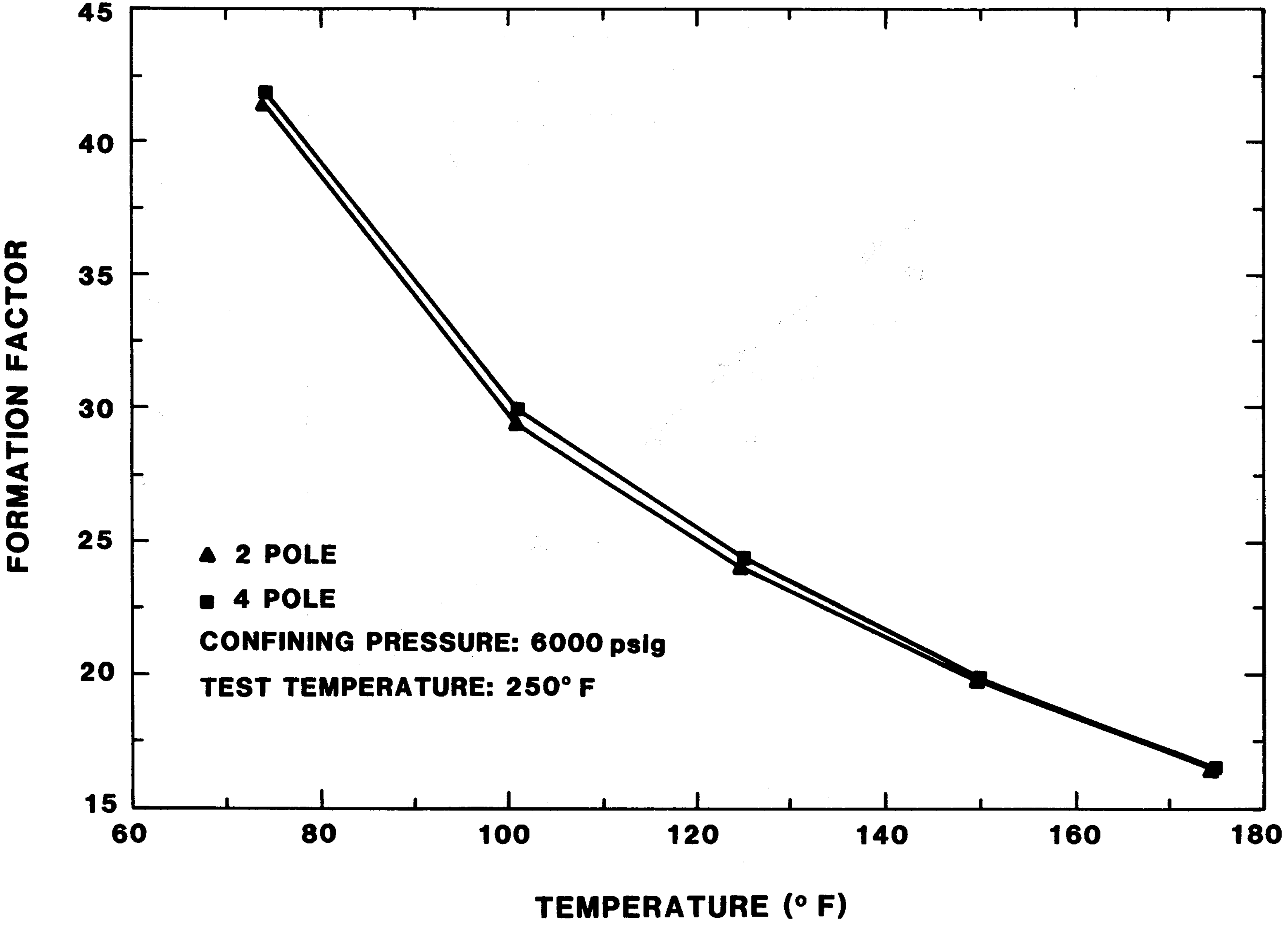

Figure 10

RESISTIVITY INDEX DETERMINATIONS AT RESERVOIR CONDITIONS