

COMBINED RESISTIVITY, POROSITY, BRINE  
SATURATION, AND CAPILLARY PRESSURE  
MEASUREMENTS ON POORLY CONSOLIDATED SAMPLES

by

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ABSTRACT

Laboratory procedures were developed to obtain accurate resistivity, porosity, and capillary pressure measurements on poorly consolidated core. The essential change from conventional approaches is that all sample preparation, brine saturation and desaturation are done in a single pressure vessel at a constant pressure and temperature. Unconsolidated core, stabilized by resin casting in a PVC or fiberglass liner, is kept refrigerated and samples are cut by a plunge cutter designed to minimize damage. The pressure vessel is built to allow sample emplacement without damage. Samples are cleaned and brine saturated in the pressure vessel, by miscible displacement with solvents. Desaturation is done by displacing brine with humidified nitrogen or air with or without the use of a capillary plate at the downstream end. When a capillary plate is used, we simultaneously obtain capillary pressure and resistivity versus saturation, but no brine permeability. Without the plate, we get brine permeability and one or two saturation points before nitrogen/air breakthrough. Porosity is determined at the end of the resistivity index sequence by first removing brine and salts using solvents; then drying the samples and measuring the pore volume in place with a helium porosimeter, and finally removing the sample for grain volume determination. The advantage of the method is that all the petrophysical measurements are made without the samples being damaged by pressure cycling or heating.

Our primary focus was to obtain measurements on unfrozen core but with minor modification, the method is applicable to both frozen and well consolidated samples.

## INTRODUCTION

We were concerned initially that freezing would expand poorly consolidated core and alter its physical properties. We observed what we interpret to be core damage from freezing using X-ray CT scans of cores. Consequently, we began a program to develop core handling and preservation methods at the wellsite. Our goals were to obtain core which was unaltered by the coring and preservation processes and did not require freezing for transport to the laboratory. A resin casting method (Gidman et al. 1987) was devised which resulted in core preserved in PVC core liners or in fiberglass core barrels and chilled but never frozen. Development and use of the resin casting method required close cooperation between the well site geologists and drilling and core service company personnel.

The next step was to slab and sample the core without causing damage to it. A horizontal band saw was redesigned to cut along the upper third of the core sleeve/barrel. This allowed the removal of the slab without damage to the remainder of the core (Worthington et al., 1987). We then found (Worthington et al., 1987) that undistorted plugs could be obtained efficiently using a plunge cutter.

Samples were best placed directly in a Hassler-sleeve pressure vessel without packaging. All our attempts to package the plugs proved unsuccessful. Once in the pressure cell each sample was maintained at restored stress for the complete solvent cleaning and measurement process.

## PETROPHYSICAL MEASUREMENTS

We measure the physical properties: formation factor, brine permeability, porosity, brine saturation, resistivity index and air permeability. When a capillary plate is used, we can no longer measure permeability. However, we do gain the ability to measure air/brine capillary pressure versus brine saturation. The capillary plate also allows us to get more resistivity index measurements than we can without a capillary plate.

All measurements are made at restored pressure (net effective stress). Details of the procedures used to make these measurements are in the appendix and a flow chart of the measurement steps is given as Figure 1. Briefly, we plunge cut and trim the samples, bring them to room temperature and formation pressure (net effective stress), and then solvent clean and brine saturate the samples using miscible displacement. If we use a capillary plate, we can only measure the resistivity and determine formation factor. Without a capillary plate we have the added measurement of brine permeability. We then flow humidified air or nitrogen to drive the sample to lower brine saturations. When a capillary plate is not used, we can usually measure one and possibly two saturations before gas breakthrough, at which point no further desaturation is possible. When a capillary plate is used, we can get resistivity index and capillary pressure measurements at multiple brine saturations.

We then solvent clean and dry the sample and measure pore volume using a helium porosimeter. If a capillary plate is not used we also measure air permeability. Finally, we remove the sample, which is usually a loose pile of sand grains, and measure the grain volume. From the pore and grain volumes we calculate the porosity and brine saturation. These are used with the resistivity measurements to determine saturation and cementation exponents. This covers the general aspects of our measurements. Some of our experiences in handling and measuring the properties of unconsolidated core follow.

Our initial work was done without a capillary plate. This allowed the measurement of brine permeability and, typically, one resistivity index measurement. Two problems were recognized which we might have missed had we started with the capillary plate in place. The first was the tendency of salt to precipitate in methanol-brine mixtures and the second was fines migration localized at the outlet end of one sample during desaturation by humidified air.

### Salt Precipitation in Methanol-Brine Mixtures

To brine saturate samples without passing a potentially damaging, high energy interface through them, we pass miscible mixtures of methanol and brine. We start with a 90% methanol/10% brine mixture and increase the brine percentage in successive flows. We found that the solubility of salt at low brine fractions was lower than it was in the water alone. We observed salt precipitation both in the methanol-brine mixtures and in one core plug. Our original clue to the problem was that the permeability on this plug increased with flow volume i.e. precipitated salt was apparently being dissolved as more and more brine passed through the plug. By making up the methanol-brine mixtures and allowing them to stand overnight, we found which of the low brine fraction mixtures had salt precipitation. Where salt precipitation occurred, we poured off the liquid and added distilled water (approximately 10% of the brine volume). We used this diluted mixtures in the brine saturation step and had no further problems with salt precipitation. Note, if we had a capillary plate in place, we would not have been able to measure permeability and may not have seen the problem.

### Fines Migration

During desaturation of one sample, we obtained brine saturations down to approximately 20% without air breakthrough. In fact, the crossplot of desaturation pressure versus brine saturation looked like a normal capillary pressure curve (Figure 2) even though a capillary plate was not used in the measurements. When we removed the sample, we observed fines plugging the silver mesh end screens. The fines had the same composition as the bulk rock which indicated that they were generated locally at or near the outlet end of the sample (Figure 3). Apparently, the plugged end screen acted like a low permeability mudcake. This allowed us to desaturate the sample to lower than usual brine saturations before air breakthrough. Interestingly, the resistivity index data for this sample appeared similar to that for other samples (Figure 4). This consistency further leads us to believe that the movement of fines was local to the end screen. To stabilize the clay minerals and minimize future fines migration, we changed from a NaCl brine similar to the formation brine to an equivalent concentration KCl/CaCl<sub>2</sub> brine. This

change in brine composition appeared to solve the problem and no further indications of fines migration were observed.

As with the salt solubility problem discussed previously, we may not have observed the fines migration had we started with a capillary plate in place. We are not arguing that a capillary plate should not be used however. The capillary plate allows us to obtain multiple brine saturations to much lower values than can be achieved without it. We are simply issuing a cautionary note on the measurement of the petrophysical properties of unconsolidated core.

## SUMMARY

Over the past few years our colleagues and ourselves have been developing means of obtaining, preserving and measuring the physical properties of unfrozen, unconsolidated core. This paper represents the status of our work on the physical properties of such core. Our data on clastics appears consistent with measurements on less friable, homogeneous samples. Formation factor-porosity crossplots yield cementation exponents( $m$ ) less than approximately 1.7 (Figure 5) and our porosities are consistent with density log measurements (Figure 6). The narrow range of the porosities of the core plugs, however, precluded an accurate determination of the transform between formation factor and porosity. The laboratory procedures we developed are described in the appendix. These procedures have been applied largely to unconsolidated core but have also been useful in measurements on consolidated but friable core. We believe that the prime advantage of the method is the minimization of the alteration of the physical properties of unconsolidated core due to core damage. The core is never frozen or heated, solvent cleaning and brine saturation are by miscible displacement, handling and stress cycling are kept to a minimum, and the passage of high energy interfaces through the core (as occurs with drying) is done only at the end of the measurement cycle during the porosity determination.

## REFERENCES

Gidman, J., Worthington, A. E., and Newman, G. H., 1987, Well-site Handling of Poorly Consolidated Core, Inglewood Field, Los Angeles Basin, SEG Annual Convention Expanded Abstracts.

Worthington, A.E., Gidman, J., Newman, G. H., 1987, Reservoir Petrophysics of Poorly Consolidated Rocks, I. Well-Site Procedures and Laboratory Methods, SPWLA Twenty-Eighth Annual Logging Symposium, Paper BB.

## SOFT CORE PROCEDURES

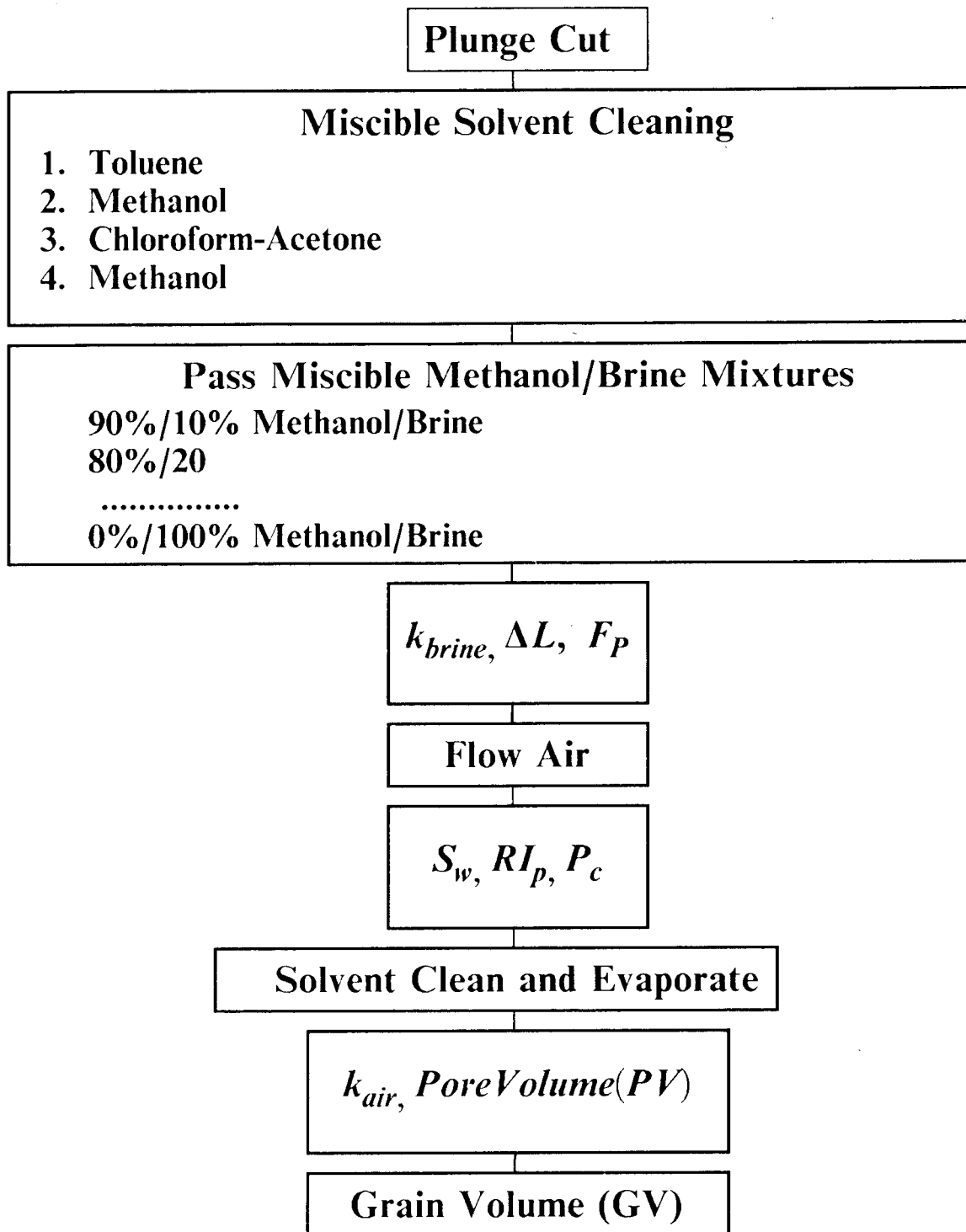
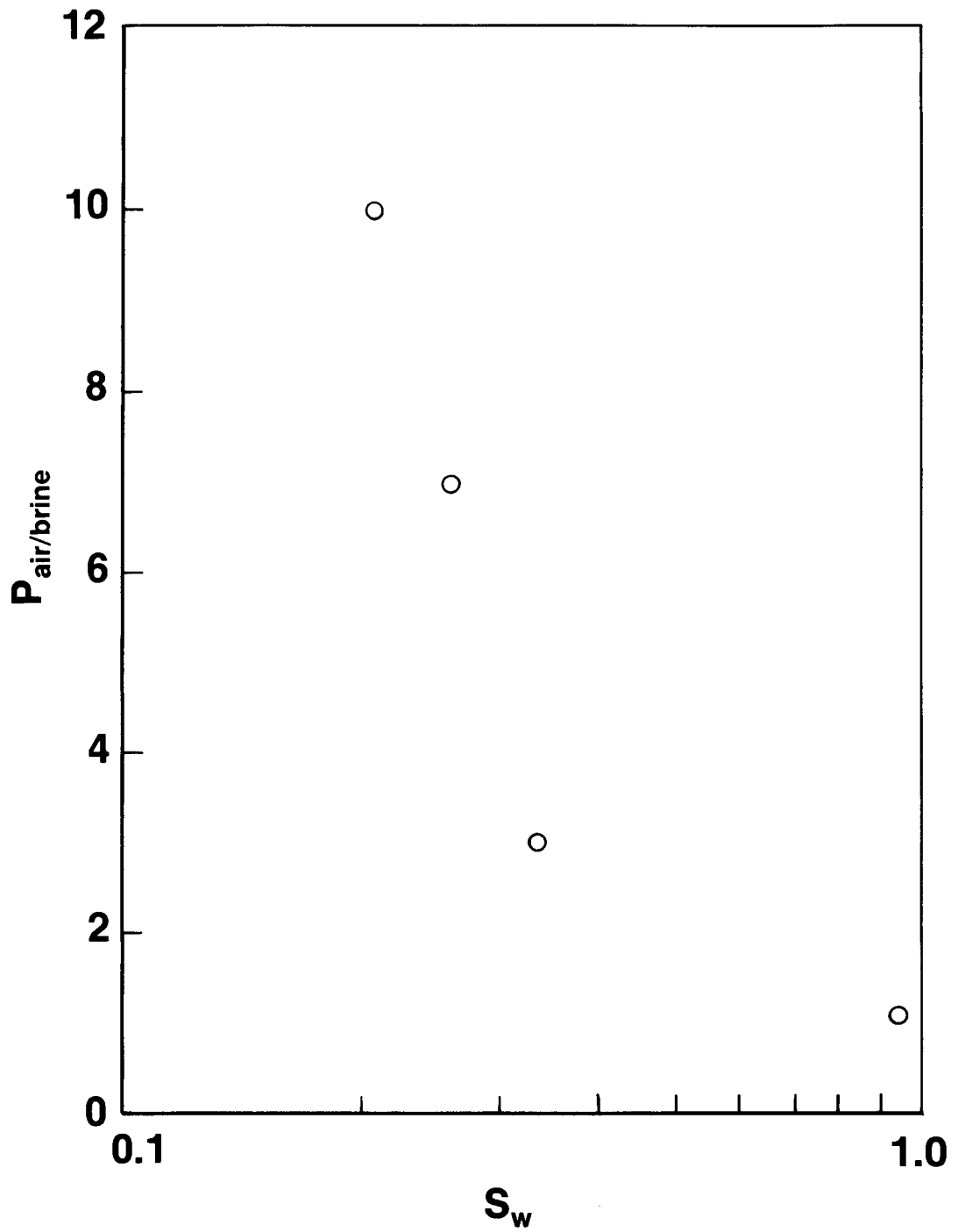
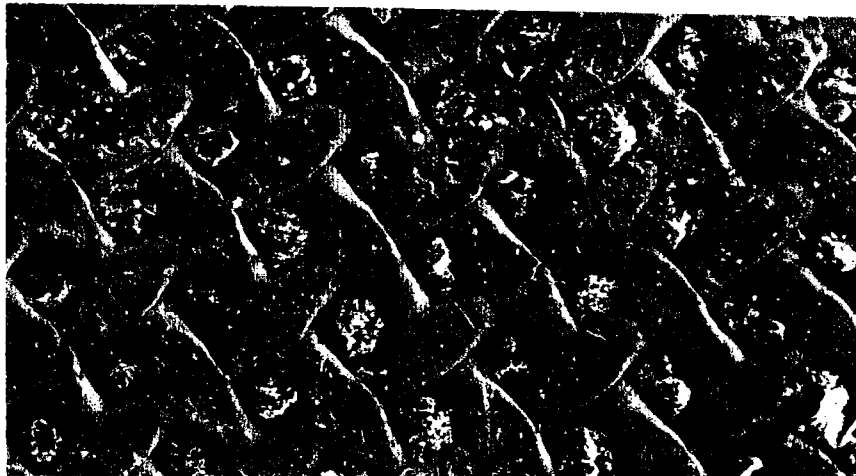


Figure 1



**Figure 2**  
**Air Pressure vs. Saturation**



I  
100 $\mu$

**Figure 3**  
**Fines Plugging End Screen**

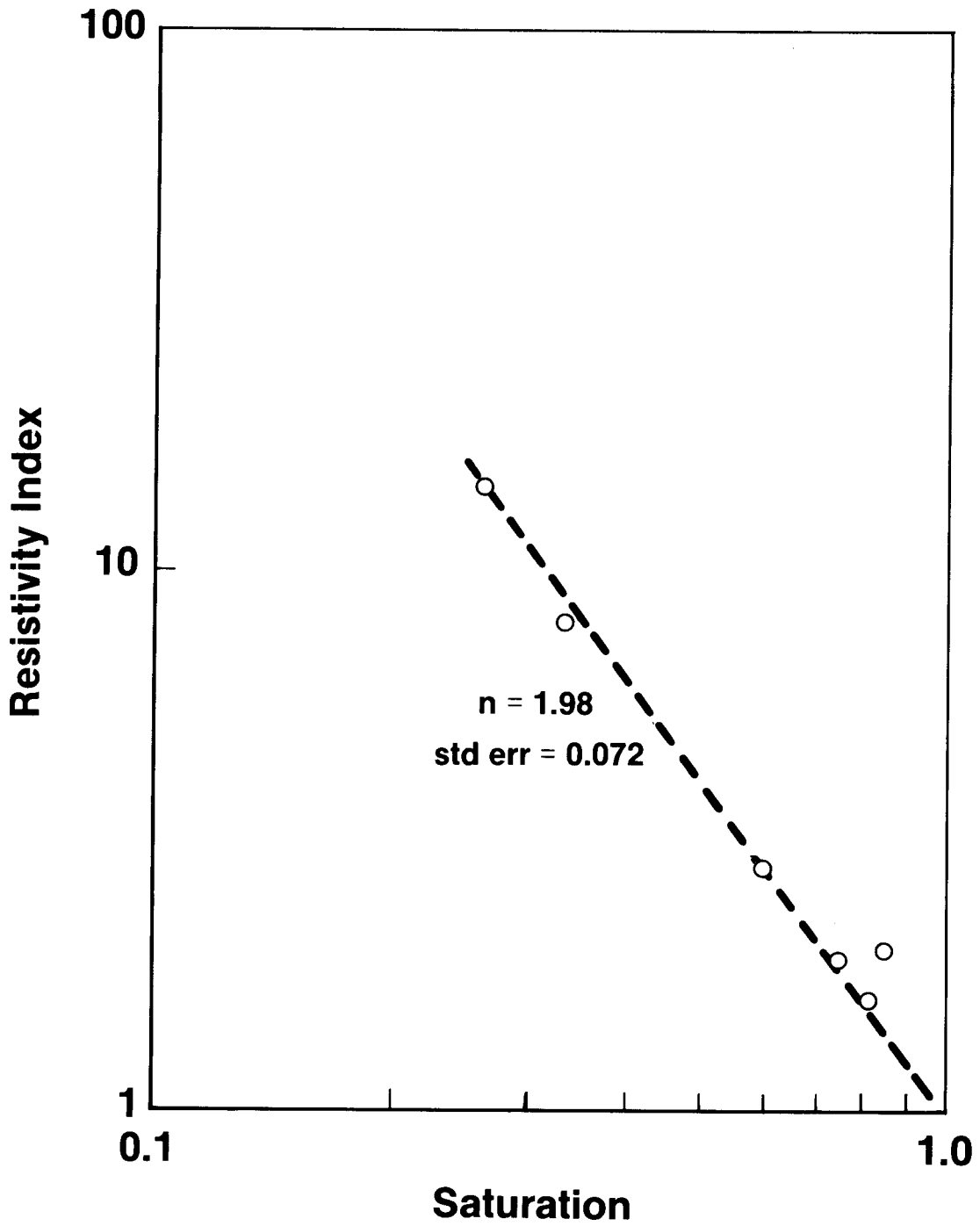


Figure 4  
Resistivity Index vs. Saturation



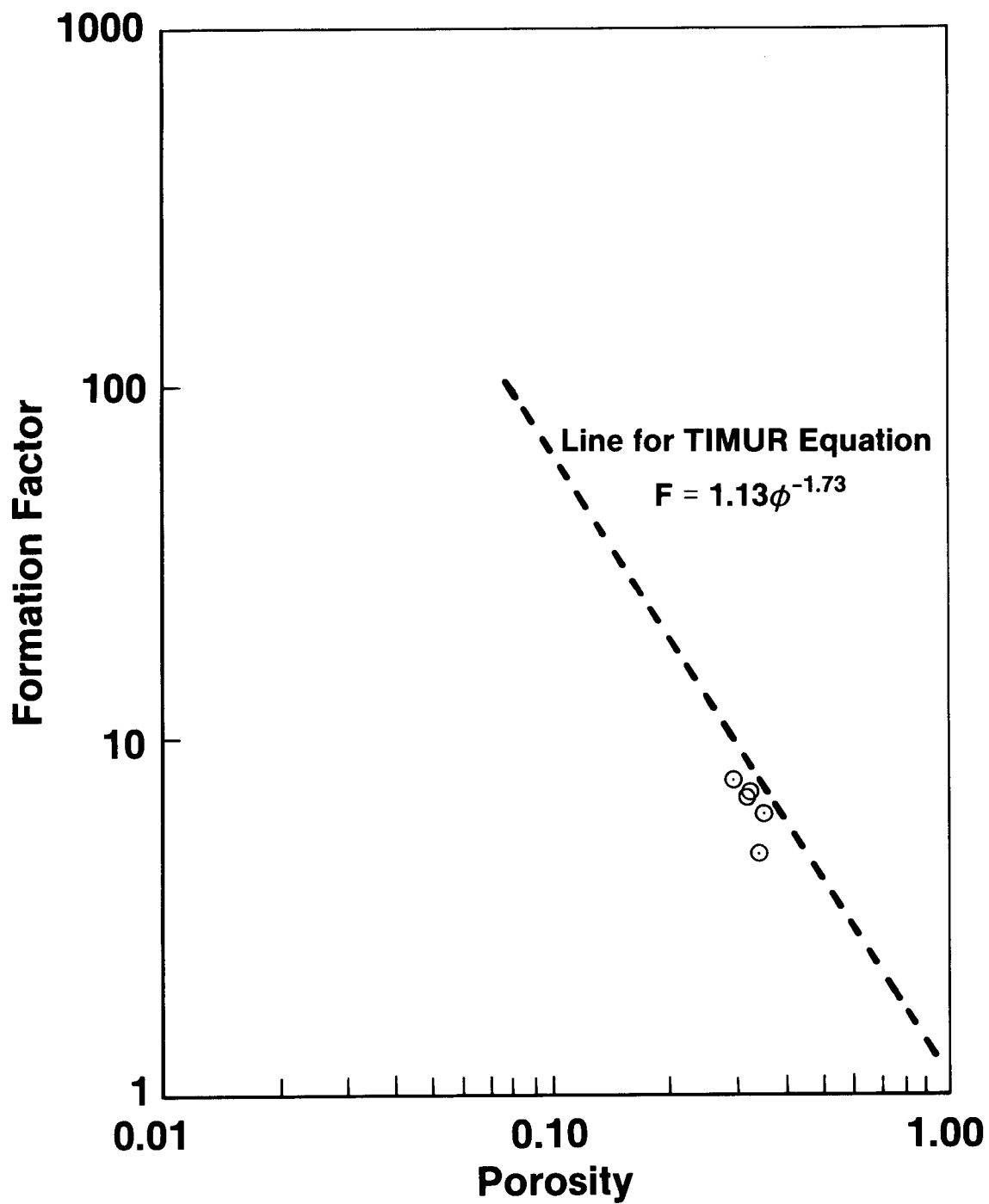
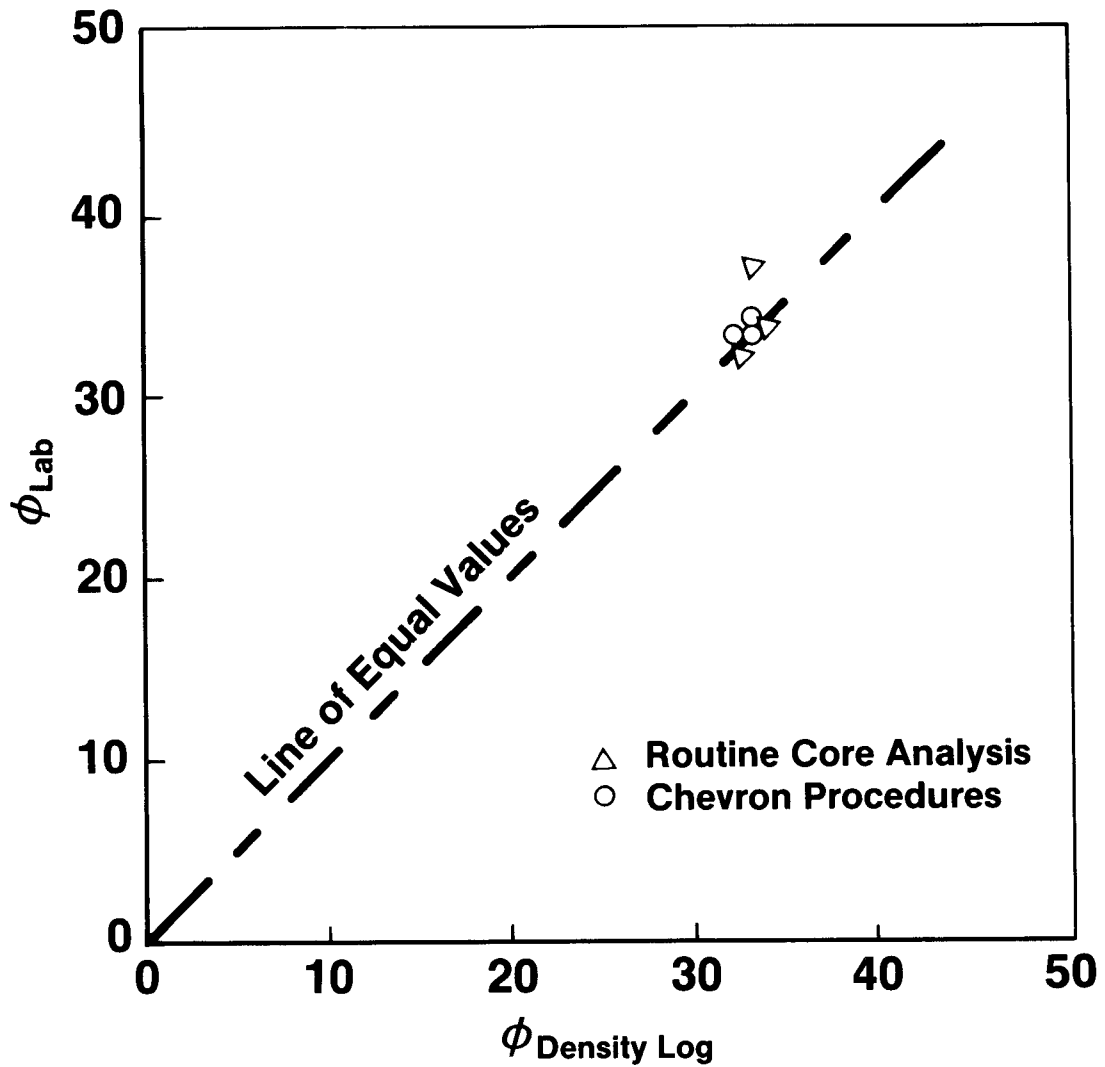


Figure 5  
Formation Factor vs. Porosity



**Figure 6**  
**Lab and Log Porosities Compare Favorably**

## APPENDIX ON EXPERIMENTAL PROCEDURES

### 1. Plunge Cutting Unfrozen Samples

Samples were obtained by plunge cutting. The plunge cutting operation is depicted in Figure A-1. We plunge cut each plug from unfrozen, but chilled, core using the apparatus and procedure described in Paper BB of the SPWLA Twenty-Eighth annual Logging Symposium, June 1987. The cutter is mounted in a drill press and slowly lowered into the core. No more pressure is used than is necessary to move the cutting edge through the core. The cutter is inserted by feel to prevent forcing it through large sand grains and pebbles which can damage both the core plug and the cutting tip. To avoid damaging its sharp edge, the cutter penetrates to just above the resin in the PVC core liner. The cutter is then raised out of the core which causes the plug, which is weak in tension, to break at the bottom and come out in the plunge cutter. The plunge cutter has a sharp cutting edge which is designed to deflect forces away from the plug as it cuts. Inside the plunge cutter is a teflon liner into which the plugs slide as they are being cut. We remove the teflon liner containing the plug and slide or push the plug to one end of the liner so that it can be cut square with a razor blade, using the square end of the liner as a guide. We then push the sample to the other end and cut it square. The length is measured while the sample remains in the liner. Harder core requires alternatives to plunge cutting such as hand carving.

### 2. Mounting Samples in the Pressure Vessel

We place either silver screens on each end of the plug or a silver screen on one end and a capillary plate on the other. The sample is then slowly raised into the pressure cell where the Hassler sleeve is expanded under vacuum to minimize grain loss and/or sample distortion (Figure A-2). We use Kalrez sleeving (made by DuPont for both 1" and 1.5" diameter samples) which has essentially the same flexibility as neoprene and does not dissolve in chloroform-acetone. The pressure cells are constructed so that the lower electrode can be removed without disturbing the elastomer sleeve. This construction requires that the length of the core plug be known to within better than 0.1 inch at the time the sleeve assembly is installed. The apparatus once set up is used for successive plugs of the same nominal length without having to remove the sleeve or to drain the hydraulic oil.

Insulation integrity of the sleeve and the electrode fittings is tested before making resistivity measurements. This is done by inserting a solid, nonconducting cylinder of nylon or teflon into the brine-filled or brine-wet sleeve, applying the operating hydraulic pressure to the sleeve, and measuring the resistance between the two electrodes. We require a minimum resistance of one megohm.

### 3. Net Effective Stress

Once the sample is at room temperature, we raise the confining pressure to net effective stress. The sample is kept at this stress throughout all of the following procedures. Length changes are recorded at this point and throughout the subsequent operations.

### 4. Flow-through Solvent Cleaning

Each plug is miscibly cleaned to a water wet condition using the solvent sequence: toluene, methanol, chloroform/acetone azeotrope (80% chloroform 20% acetone by volume). Flow rate through the plug is automatically limited to 0.15 ml/min to minimize the potential for movement of fines. Production of fines at any stage in the procedures is noted and reported.

Flow is pressure limited at 30 to 50 psi above the pressure required to flow solvent through the ceramic disc alone at approximately 0.15 ml/min. The direction of flow for both solvent cleaning and brine saturation is from the bottom up, i.e. through the porous plate and then through the plug. As soon as the toluene effluent appears to be clean, the flow is stopped and the sample soaked for 20 to 30 minutes. Flow is resumed until approximately one half a pore volume has flowed through the sample. If the toluene is not discolored, we then switch to methanol. If the toluene is discolored, we continue flow until it appears clear, then begin another soak period.

Our experience is that as the core becomes cleaner, the remaining organic matter takes longer to remove from the core either because it is less soluble and/or because it must diffuse through low permeability regions of the plug to reach the flushed pores. Accordingly, we lengthen the successive soak periods. Soak periods have not been longer than can be accommodated in an 8-hour shift, however, for even the most difficult to clean cores. For a single shift operation, a minimum of two flush periods and two soak periods was accomplished in each 24 hours. This could be increased to three flushes and three soaks per 24 hours in a two or three shift operation. Even the most difficult cores became clean within a few days.

Salts were cleaned from the core with methanol using the same flow times and soak cycles as for toluene. Inorganic salts do not color methanol, but some coloration of the methanol may occur from soluble organic compounds. Following each soak period, about one half a pore volume was passed through the samples and the effluent methanol was checked with silver nitrate to detect the presence of trace chloride. The methanol flow was ended when no chloride was detected in the effluent.

We then flow chloroform/acetone azeotrope, checking for coloration during a series of flow-soak cycles as with toluene. We then miscibly displace the chloroform-acetone with methanol. Note, the chloroform-acetone cleaning may not be necessary if the cores contain no black oil and if surface-active agents were not introduced to the core in the drilling mud filtrate.

The volume of liquid through the plug during the cleaning and saturating of these cores is kept as small as possible in order to minimize damage by movement of fines. Therefore, all flow procedures are limited to that required to clean and saturate the samples.

### 5. Brine Saturation

Our objective was to saturate the core with brine while avoiding the creation of a methanol-brine interface within the core and the precipitation of salts at the contact between solutions of different methanol content. This was accomplished by following the methanol with a series of miscible mixtures of brine and methanol; starting with 80% methanol, 20% brine and shifting in steps of 20% until pure brine is passing through the core. No more than one pore volume of each brine-methanol mixture was used.

The solubility of salts decreases as methanol is added to a brine, reaching a minimum in pure methanol. Therefore salts may precipitate in the core as the advancing front of each solution interacts with the higher methanol content solution which preceded it. If this happens and a severe blockage results, it can be removed by prolonged soaking and flowing of pure methanol. If the blockage is only partial, it can be dissolved by continuing to flow the brine-methanol mixture which caused the blockage. Fresh water must not be used as it could seriously damage clays in the rock. If it is necessary to return to pure methanol, then the next round of miscible displacements should be based on successive dilutions of brine in methanol differing by only 10% instead of 20%.

We found that brine-methanol mixtures were best prepared at least 24 hours in advance and allowed to stand to see if any salt precipitates out. When precipitation did occur, we decanted the clear solution and added an amount of pure water which corresponds to 10% by volume of the actual water content of that solution. This procedure eliminated an early problem we had of the precipitation of salt at the mixing fronts between the miscible mixtures.

### 6. Formation Factor

We begin monitoring the electrical resistance during the miscible displacement of methanol with brine. When full brine saturation is reached, brine is flowed until the temperature-corrected resistance is constant within the accuracy of the measurement. Each sample is soaked for several hours or overnight before resuming brine flow. We continue with alternate periods of flowing and soaking until the temperature-corrected electrical resistance does not change significantly between soak periods, recording all resistance and temperature measurements. The formation factor is calculated from the final stable values, the brine resistivity, temperature and sample dimensions. The sample length at stress is calculated from the initial plug length and the recorded length changes in the pressure vessel. The diameter at stress is estimated from the diameter of the unmounted plug multiplied by the ratio of the stressed to the unstressed length. This assumes that the sample behaves isotropically.

Accurate temperature measurement is necessary for reliable resistivity data. We use a miniature, pressure proof, platinum resistance thermometer mounted in the hydraulic fluid in the overburden cell, immediately adjacent to the core sleeve. Temperature is recorded to 0.1 degC precision and accuracy which corresponds to a 0.2% uncertainty in resistivity.

### 7. Resistivity Index and Capillary Pressure

We desaturated each sample in three to four steps. The steps are roughly equally spaced on a logarithmic scale between 80% and 20% saturation.

We used humidified nitrogen or air to drive the desaturation. The gas was admitted to the top of the sample and displaced the brine through the porous plate on the bottom. The volume of brine displaced was measured as accurately as possible, because small errors in measuring the brine produced can lead to large errors in brine saturation. Care was also taken to ensure that the volume of produced brine was not reduced by evaporation. Equilibrium at each stage was determined from the stability of the resistance after brine flow. The temperature corrected, electrical resistance had to remain constant over a period of several hours, preferably overnight.

The porous plate was selected, based on experience, to yield as wide a range of saturations as possible while retaining enough permeability to allow solvent and brine flows at pressure drops that do not exceed approximately 80% of the confining stress on the sample.

We set up the equipment to measure pressure in the gas simultaneously with the resistance and saturation equilibrium determinations. This allowed us to construct a capillary pressure curve as well as a resistivity index plot.

The total gas volume of the system was as small as possible to minimize water vapor condensation at the cool spots in the plumbing. Such condensation can cause errors in both electrical resistivity and saturation because of the resulting lost water from the sample to replace the water condensed from the vapor.

### 8. Final Solvent Cleaning and Drying

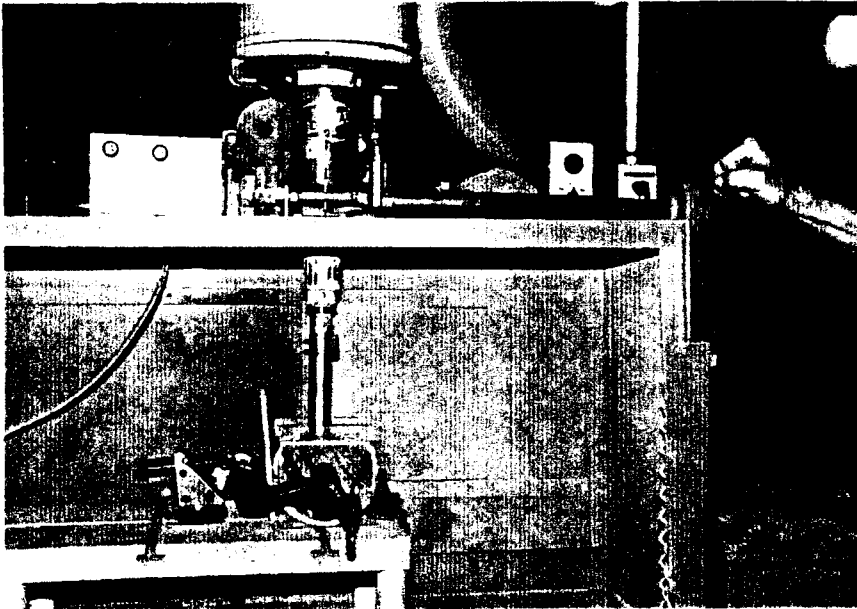
Following the last step of desaturation and resistivity index determination, each sample was cleaned by flowing approximately one pore volume each of the brine/methanol mixtures in the reverse order, starting with the 20/80 methanol/brine mix and proceeding to 100% methanol. The methanol was then followed by a couple of pore volumes of acetone, and the acetone by dry gas. Gas flow is continued until the sample is free of acetone. The soak-and-flow technique can be used to maximize the acetone concentration in the exit stream where it can be sensed with any suitable gas monitor. At least the last several pore volumes of gas used for drying should be helium, in order to prepare the sample for pore volume measurement.

### 9. Pore Volume

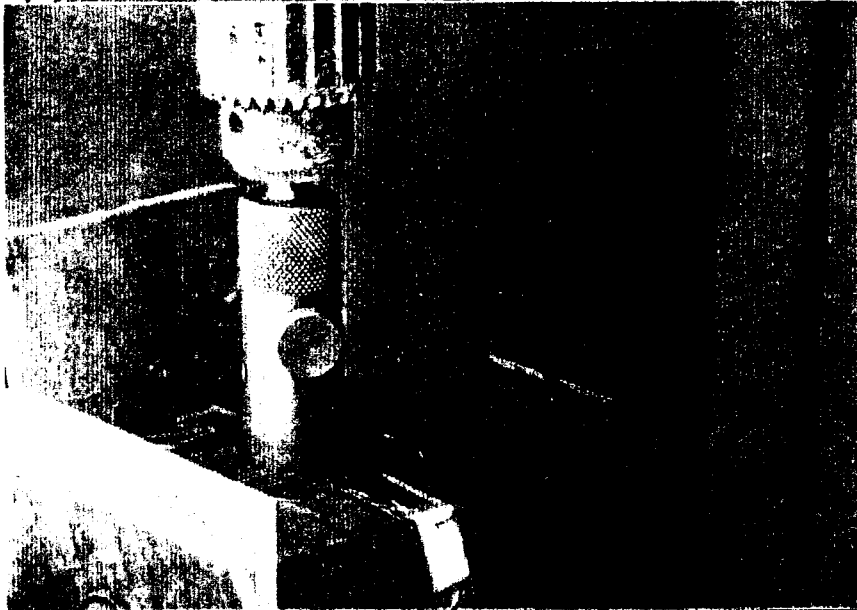
We used a helium porosimeter to measure the pore volume of the sample including the porous plate and the plumbing. The pore volume of the plate and the plumbing were measured independently at the same confining stress.

### 10. Grain Volume and Weight

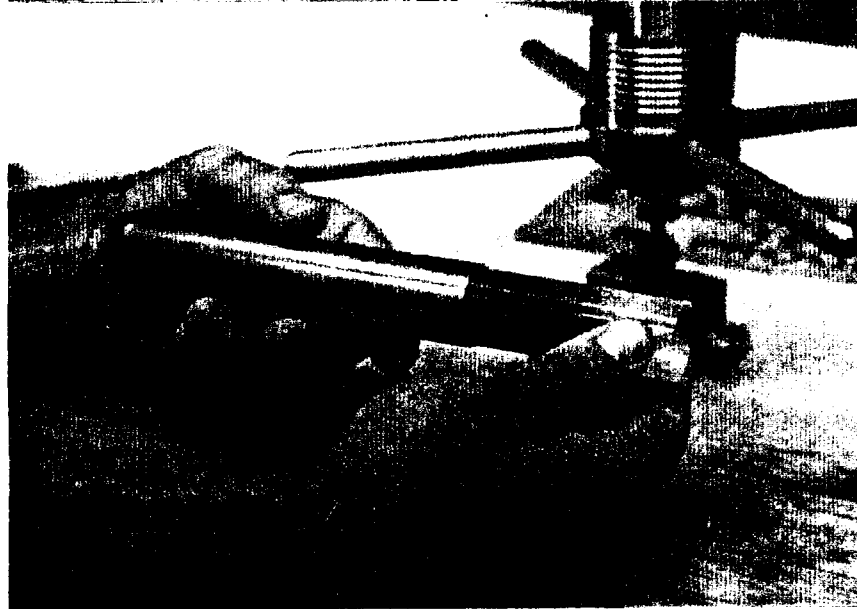
We slowly reduce the hydraulic stress to zero and remove the sample carefully. Samples disintegrate on handling. Dry samples are then placed in a helium porosimeter matrix cup to determine grain volume. Sample mass is measured and recorded.



**PLUNGE CUTTING**



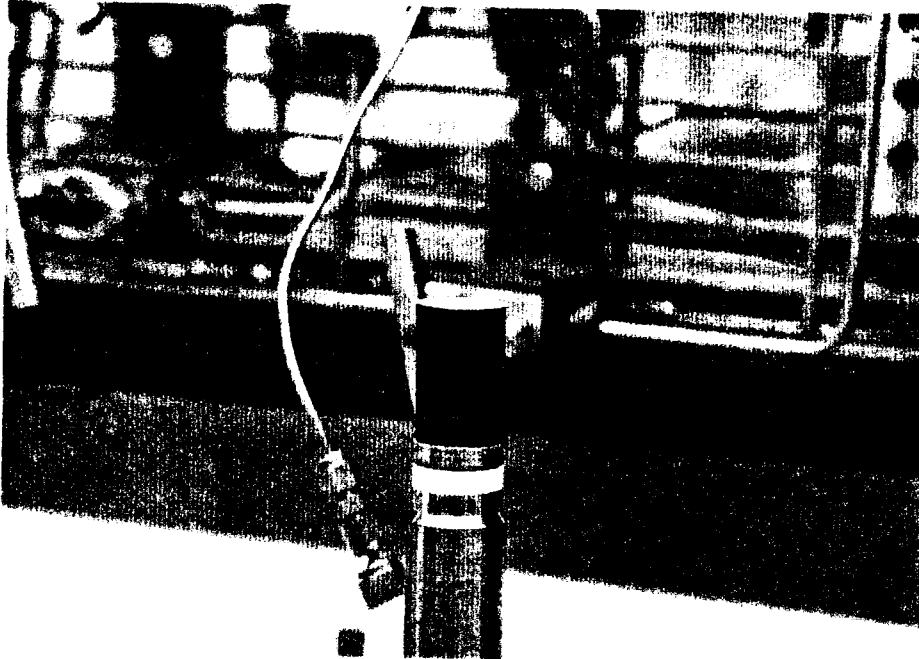
**PLUNGE CUTTER  
AT MAXIMUM  
PENETRATION**



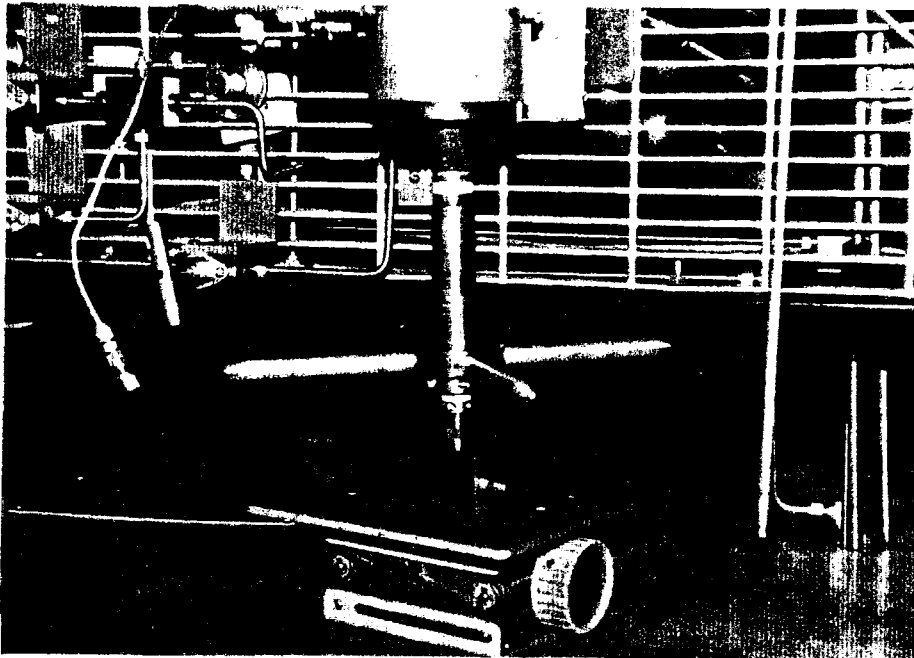
**REMOVING CORE  
PLUG FROM  
PLUNGE CUTTER**

**Figure A-1  
Plunge Cutting and Removing Plug**





**PLUG MOUNTED  
ON FIXED  
ELECTRODE  
(Silver screens on  
top and bottom)**



**RAISING PLUG  
INTO VESSEL**

**Figure A-2  
Placing Plug in Pressure Vessel**

