

MEASUREMENT OF UNSATURATED
ROCK WATER POTENTIALS IN SITU

by

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With gratitude and love, this thesis is
dedicated to my parents.

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ABSTRACT

Characterizing water movement in unsaturated fractured rock is important for evaluating the isolation properties of waste repositories. By determining water potential gradients in situ, the direction and magnitude of groundwater flow can be established. This study evaluates four methods, originally designed for soil water potential measurements, for their ability to measure ranges of rock water potentials in situ. The downhole tensiometer, measuring potentials from 0 to -0.85 bars, worked well in laboratory simulations, but was not tested in the field. The osmotic tensiometer, with a measurement range of 0 to -3 bars, was successful in the laboratory but field experiments were inconclusive. Thermocouple psychrometers were not able to give precise water potential measurements, however, temporal trends revealed a range of water potentials from -2 to -50 bars. The absorber method, involving the equilibration of rock water potentials from -2 to -100 bars with filter papers, was also not able to give precise measurements. The four methods need further modifications to improve the accuracy of measurement of rock water potentials, especially for determining the hydraulic head gradient for flow calculations.

CHAPTER ONE

INTRODUCTION

The in situ measurement of water potential in geologic media is essential for the determination of the magnitude and direction of ground-water flow. Water potential is an expression of the potential energy of water within a geologic medium relative to that of water in a standard reference state. The standard reference state generally used is that of a hypothetical reservoir of pure water, at atmospheric pressure and at a specified temperature. Water movement in the medium is a result of differences in potential energy of the water with distance. Water in a geologic medium obeys the universal law which states that water moves in the direction of decreasing potential. The rate of decreasing potential with distance is the force causing flow.

Saturated geologic medium has hydrostatic pressures greater than atmospheric pressure (i.e. under a water table) and the potential level of the water may be greater than that of the reference state and thus considered to have positive potential. In unsaturated media, the water is constrained by various forces; most notably, capillary and adsorptive forces. The equivalent hydrostatic pressure of water in the unsaturated zone is less than that of the reference state, thus its potential is negative. The water in unsaturated geologic medium moves

from regions of higher water potential (less negative) to regions of lower potential (more negative).

Unsaturated geologic media is subject to many different force fields which cause its potential to differ from that of pure free water. The force fields are caused by the attraction of the matrix for water, the presence of solutes, and the pressure of external gas pressure and gravitation. The influence of all these force fields are manifested in the total water potential (ψ_t) which is the sum of the following potentials:

- 1) Elevation or gravitational potential (ψ_g);
- 2) Pressure potential (ψ_p) includes positive or submergence pressure;
- 3) Matric potential (ψ_m) associated with capillary and adsorptive forces, which determines the fractures and pores which are partially drained and also the thickness of water films along walls of drained pores.
- 4) Osmotic potentials (ψ_o) associated with solute concentrations within the water.

Many techniques have been developed for measuring water potentials of soil systems in situ. These methods include: thermocouple psychrometers (Spanner, 1951; Richards and Ogata, 1958), standard tensiometers (Richards and Gardner, 1936; Richard, 1965) and the osmotic tensiometer (Peck and Rabbidge, 1966, 1969).

The purpose of this study was to evaluate adaptations of some of the methods for the measurement of water potential in unsaturated fractured rock. Four methods used to measure various ranges of water

potentials in a fractured rock system were assessed and the laboratory and field results were evaluated. These methods included the:

- 1) Downhole tensiometer which works on the same principle as the standard soil tensiometer with modifications for use in a borehole;
- 2) Osmotic tensiometer is also based on the same principles as the standard tensiometer but with modifications that include filling the device with an osmotic solution and achieving contact with a borehole wall using a semipermeable membrane supported by a stainless steel core;
- 3) Thermocouple psychrometer indirectly measures soil water potential by measuring the dewpoint depression of the air in equilibrium with the sample; and
- 4) Absorber method involves the equilibration of filter papers with rock walls of a certain water potential and measuring the resulting water potential of the filter papers with the thermocouple psychrometer sample changer.

The downhole tensiometer measures matric potentials in the range of 0 to -0.8 bars and the osmotic tensiometer increases the measurement range from 0 to as much as -15 bars. The water potential measured by the thermocouple psychrometer is the sum of the matric and osmotic potentials and has an effective range of -2 to -50 bars. The thermocouple psychrometer sample changer used in the absorber method also measures matric and osmotic potentials with an effective range of -2 to -1000 bars.

Interest in unsaturated fractured rock as a possible site for a

high level radioactive waste repository has led to the investigation into methods for determining rock water potentials in situ. Water movement in unsaturated fractured rock may be the principle mechanism for transport of radioactive contaminants from the repository to the accessible environment. Thus, by determining the water potential distribution in an unsaturated system, an understanding of magnitude and direction of water flow may be established.

As water potential in a fractured rock decreases, smaller and smaller fractures and pores will partially drain, and the total cross-sectional area through which liquids can flow will progressively be reduced. Consequently, water potential distribution in an unsaturated system influences not only the hydraulic gradient but also the hydraulic conductivity.

CHAPTER TWO

LITERATURE REVIEW

This chapter reviews the previous work and important criteria concerning the development and use of instruments for measuring water potentials in situ. Although the previous techniques used for measuring water potentials are applied to soil, the ultimate objective of this study is the application of these techniques for measuring rock water potentials.

2.1 Tensiometer

Numerous workers have contributed to the development of tensiometers. These workers have also developed various designs of tensiometers dependent on the materials available and measurement conditions. Gardner (1922) and his co-workers were among the first to use porous cups and vacuum gauges to directly measure capillary potentials of moist soils. The basic components of the tensiometer are a porous ceramic cup connected through a hollow tube to a vacuum-measuring device. The tensiometer is filled with water and inserted into the unsaturated soil. Good hydraulic connection between the porous cup and soil is crucial for accurate measurement of matric potentials. During the subsequent equilibration process, dissolved solutes move through the ceramic cup and water flows in or out of the cup in response to the soil matric potential. As the water tension or potential inside the cup

equilibrates with the surrounding soil, the tension is transmitted through the water-filled tube to an accessible vacuum gauge and recorded manually or automatically. The osmotic component of the total moisture potential is not measured because the ceramic cup is permeable to solutes. The effectiveness of the tensiometer is judged by the ability of water in the tensiometer to equilibrate through a porous cup with adjacent soil water and the accuracy of a vacuum gauge to measure suction or matric potential of the soil.

Many variations of the tensiometer design were described by Richards and Gardner (1936), and Hunter and Kelley (1946). To decrease the response time and increase sensitivity of the gauge readings, Klute and Peters (1962) used a strain gauge pressure transducer to record tensiometer soil potentials. The rapid-response tensiometer systems forms an essential part of the instrumentation of laboratory columns used to study unsteady flow problems in unsaturated media (Watson, 1965). Field application of the rapid-response automatic tensiometer scanning system was developed by Rice (1969). The multiple tensiometer system permits rapid-response measurement of a number of tensiometers with a scanning valve and only one pressure transducer. Precautions must be taken to avoid air leakage and pressure fluctuations within exposed tubing due to short term temperature variations within the tubing.

Improvements and variations of the rapid-response tensiometer scanning system have been described by numerous authors (Williams, 1978; Long, 1984). Marthaler and others (1983) developed a pressure transducer with attached syringe needle and digital meter. The needle

is inserted through the septum stopper which closes the upper end of the tensiometer. The pressure in the air below the septum, in equilibrium with the water potential, is read on the digital meter (calibrated in millibars). Upon withdrawal of the needle from the tensiometer, the septum stopper seals the tensiometer. Although many improvements have been made on the tensiometer design, the matric potential measurement range is limited to 0 and -0.8 bars because negative pressures are measured with reference to atmospheric pressure.

2.2 Osmotic Tensiometer

Realizing the limited range of measurement of the standard tensiometer, many soil scientists have researched alternative field methods that would increase the measurement range. One method is the osmotic tensiometer developed by Peck and Rabbidge (1966). Their apparatus differed from a standard tensiometer in several important ways: 1) communication between the tensiometer and soil moisture was achieved through a semi-permeable membrane protected and supported by a porous ceramic disk; 2) the tensiometer was initially filled with an aqueous solution of large organic molecules which were retained by the semi-permeable membrane; and 3) positive hydrostatic pressures were maintained within the device at all times. The method is based on the ability to depress the reference free energy of water by a predetermined concentration of an organic solute.

Earlier research by Kemper and Evans (1963) showed that osmotic pressure and hydraulic pressure are equally effective in moving water through a membrane when solutes are completely restricted. To demonstrate this phenomenon, they used Visking dialysis membranes and various

concentrations of polyethylene glycol, commonly known as "carbowax".

As mentioned in Chapter 1, the total water potential is a function of pressure, temperature, composition, external and internal force fields. Previous researchers manipulated the pressure component to control soil water potential but Zur (1966) manipulated the composition (osmotic potential) to control matric soil water potential. His experiments consisted of a soil chamber separated by a cellophane membrane from solution chambers filled with an aqueous solution of Carbowax of a molecular weight of 6000. After 72 hours, the matric potentials of the soil water fully equilibrated with the osmotic potentials of the Carbowax solution. The soil moisture content was determined gravimetrically and a moisture release curve (MRC) was prepared for each soil type. Zur compared the MRC for soils equilibrated on a pressure plate and soils equilibrated with the osmotic method. The agreement between the two methods was very good at all pressures for various soil types. Leakage tests indicated that from 3 to 5 percent of the Carbowax had passed through the membrane after 10 to 15 days. After observing bacterial decomposition of the membrane, Zur treated the membranes with bacteriocides and disinfectants. The bacteriocides did not alleviate the problem, but sterilizing the soil before use did work for periods of 2 to 3 weeks.

Williams and Shaykewich (1969) also utilized Carbowax solutions to create known matric potentials in soils. A double thickness of Visking dialysis membrane was placed in direct contact with soil samples with solutions of Carbowax in adjacent chambers. Results obtained using Carbowax solutions of a molecular weight of 6,000 and 20,000 were

compared over a range of -0.5 to -15.3 bars. The authors concluded that either molecular weight solution can be successfully used for the osmotic control of matric potential.

Peck and Rabbidge (1969) reported the effects of changes in external capillary and osmotic potentials, temperature, and zero drift on the performance of the prototype osmotic tensiometer. Osmotic potentials ranging from 0 to -15 bars were created by solutions of a molecular weight of 20,000. Communication between the soil water and the Carbowax solution occurred through a disk of Visking dialysis membrane supported on a fine porous plate. Very good accuracy was reported. Reequilibration after a known change in external matric potential required less than 2 hours, while reequilibration to a change in external salt concentration required almost 10 days due to the slow diffusion of salt through the membrane. The authors observed temperature sensitivity of -1.5%/°C and zero drift of 1 to 2% per month.

2.3 Thermocouple Psychrometer

The thermocouple psychrometer indirectly measures soil water potential by measuring the dewpoint depression or the wet bulb depression of the air in equilibrium with the sample. The total water potential, ψ_t (J/Kg), is related to the relative humidity, p/p_0 by the equation:

$$\psi_t = (RT/M) \ln p/p_0 \quad (1)$$

where M is the molecular weight of water (.018 kg/mole), R is the ideal gas constant (8.31 J/K° mol), T is the Kelvin temperature of the liquid

phase, p and p_0 are the actual and saturated water vapor pressures.

The development of an instrument to measure relative humidity of the pore air began with Spanner (1951). Spanner's psychrometer, like most of those presently used, consists of a stationary thermocouple, with measuring and reference junctions. The reference junction remains dry while the measuring junction is moistened. Two methods used to moisten the junction involve dipping it into a small reservoir of water (Richards and Ogata, 1958), or forcing a current through the thermocouple to cool the measuring junction to the dewpoint, thereby condensing a drop of water on the junction (i.e. Peltier effect). As water evaporates from the measuring junction, the junction is cooled and the temperature difference between the reference and measuring junction creates a voltage. The voltage is proportional to the difference between wet- and dry-bulb temperatures of the vapor being sampled. The rate of evaporation is primarily a function of the relative humidity of the chamber, but also varies with the diffusivity of water in air. The voltage produced becomes stable when equilibrium is attained and is assumed to be proportional to the wet-bulb temperature depression. When making psychrometer measurements, the major sources of errors are lack of thermal or vapor equilibrium, contamination and variation in wet-junction size and shape.

Several different types of psychrometers have been developed for field and laboratory measurements of soil water potential. A field-thermocouple psychrometer and a thermocouple psychrometer sample changer utilize two variations of the basic design described previously.

A typical thermocouple psychrometer consists of a thermocouple with .0025 cm diameter wires of copper-constantan (the reference junction) and chromel-constantan (the measuring junction). The thermocouple wires are welded together and protected by a cover of a ceramic cup or fine mesh wire screen. Psychrometer measurements are made utilizing control units/meters available from several manufacturers. These units provide the necessary switching and cooling circuitry and signal amplification.

Psychrometers measure relative humidity by either of two methods: 1) the psychrometric method (Merril and Rawlins, 1972; Brown and Johnston, 1976; and Daniel et al., 1981) or 2) the dewpoint method (Campbell et al., 1973). The psychrometric method involves the measurement of the temperature depression associated with the evaporation of water from the wet surface of the measuring thermocouple. The thermocouple surface is initially wetted by inducing condensation through Peltier cooling. The cooling is then discontinued and the junction with condensed water begins to return to ambient temperature. When the wet bulb temperature is reached, evaporation of water from the junction holds its temperature constant. The voltage signal from the thermocouple at this "plateau" is proportional to the relative humidity and therefore related to the water potential.

The dewpoint method utilizes circuitry within the control unit to maintain the temperature of the wetted measuring thermocouple at the dew-point corresponding to the ambient relative humidity. At the dew-point temperature, the condensation is equal to the evaporation of moisture from the thermocouple.

Details of the calibration of the field thermocouple psychrometers is presented in Appendix A. Briefly, the calibration procedure involves a series of measurements made over salt solutions having known osmotic potentials. Measurements are repeated at several temperatures covering the range anticipated in the field.

The sample changer psychrometer, developed for laboratory use, was first described by Campbell et al. (1966). Samples contained within six chambers drilled into a cylinder are rotated so that a single psychrometer is used for all samples. The whole apparatus is placed in a constant temperature bath for thermal equilibration. A thermocouple psychrometer sample changer SC-10A (Decagon Devices, Inc., Pullman, Washington) is a modification of the sample changer described by Campbell et al. (1966) and can be used in the field or laboratory. The SC-10A is constructed of machined and anodized aluminum body and does not require a constant temperature bath. The SC-10A consists of a ceramic bead thermocouple which is moistened by dipping into distilled water to provide the wet junction. Samples are contained within ten removable chromed brass cups and rotated to the single thermocouple for measurement. A lever and piston arrangement raises the sample and seals the chamber directly below the thermocouple in an isothermal chamber. The SC-10A can accommodate samples of soil or rock with volumes of approximately 1 cm³, as well as filter papers, leaf cuts, and salt solutions. Calibration procedures for the thermocouple psychrometer sample changer SC-10A are presented in detail in Appendix B.

2.4 Absorber Method

The use of paper as a sensor of moisture stress began with Hansen

(1926) at the University of Copenhagen. His research involved using blotting paper strips saturated with four different concentrations of sugar solutions and exposing them to soil samples in closed chambers. The paper strip that did not gain or lose weight was assumed to represent the soil stress level of the soil sample. Thus, the water potential of a soil sample was estimated by determining the osmotic potential of the sugar solution which had the same vapor pressure as the soil. Gradmann (1934) improved the method by using a single strip of blotting paper soaked in a salt solution, and then calibrated for weight versus stress. He also enclosed the papers with soil samples allowing equilibrium.

The first use of paper as a moisture stress sensor without a hygroscopic salt was reported by Gardner (1937). The method is based on the fact that a moist soil, in contact with an absorbant material (i.e., filter paper), exchanges water until equilibrium is achieved. If a capillary tension or MRC is determined in advance for the absorbing material in contact with a soil, the tension or potential of the soil is found by reference to the curve. Using Schleicher and Schuell, No. 589, White Ribbon, filter papers, Gardner calculated the MRC for the filter paper. Various methods were used for plotting different portions of the curve. Ultimately, the portions were combined, to describe the entire MRC. For the low-moisture concentrations (high water potential), both wet and dry papers were placed in desiccators over sulfuric acid of a known vapor pressure and allowed to equilibrate at a constant temperature. Several months later, the average moisture percentage of these filter papers were measured for the low moisture portion of the MRC.

The portion of the MRC covering the high-moisture concentrations (low water potential) was obtained with a centrifuge and specially constructed centrifuge tubes.

In the same paper, Gardner determined the MRC for the soil samples by separately placing a wet and a dry filter paper between layers of the soil of unknown moisture content. The samples were sealed for five to six days and stored in a constant temperature room. After this period, the filter papers were removed and weighed to determine the moisture percentage of the paper. The results of the initially dry and wet filter papers were averaged and the tension of the paper determined from the corresponding moisture percentage of the MRC. Gardner proposed that moisture potentials from -0.3 to -1000 bars could be estimated with this method.

The filter paper method was not given much recognition until Williams and Sedgley (1965) simplified Gardner's technique to a form suitable for a wide range of agronomic uses. They investigated the use of filter papers for estimating the critical 15-bar percentage of various soils. Following this work, Fawcett and Collis-George (1967) determined a MRC for Whatman No. 42 filter papers using conventional pressure membranes and plates (Richards, 1947) and vacuum desiccator (Thomas, 1921) methods. Fawcett and Collis-George emphasized the usefulness of the filter paper method for estimating a wide range of soil moisture potentials in place of more conventional methods.

McQueen and Miller (1968), using the Schleicher and Schuell, No. 589, White Ribbon, filter paper used by Gardner (1937), obtained a different calibration curve for this filter paper than the calibration

curve of the Whatman No. 42 filter paper of Fawcett and Collis-George. McQueen and Miller noted that filter paper in contact with a partially saturated soil absorbed moisture by capillary flow and measured only matric potential, whereas filter papers supported a small distance from the soil in a closed chamber measured both matric and osmotic potentials.

McQueen and Miller defined the filter paper MRC for high, medium, and low water potential conditions. Low water potentials (below -15 bars) involved the equilibration of filter papers with saturated salt solutions within a closed chamber. Soil samples equilibrated on a pressure membrane extractor were sealed in cans in good contact with wet and/or dry filter papers to measure medium water potentials (-0.1 to -15 bars). Calibration for high water potentials (above -0.2 bars) were determined from field samples obtained at known heights above a water table. All of the filter paper calibration data were plotted on semi-logarithmic paper. When plotted, the data were found to represent two straight line segments intersecting at 0.21 bars.

Al-Khafaf and Hanks (1974) developed a method wherein both matric and osmotic potentials could be obtained from the same soil sample. They evaluated the influence of the type of contact, temperature, and temperature variations on the relations of filter paper water content to soil water potential. They placed one Schleicher and Schuell No. 589 filter paper beneath the soil (in good contact for liquid and vapor flow) which measures only matric potential. A filter paper suspended above the soil, allowing vapor flow only, was used to measure matric and osmotic potentials. Evaluation of the factors that affect

predicted water potentials led to the conclusion that effects of absolute temperature were not as crucial as temperature variations. In their paper, Al-Khafaf and Hanks were the first to write about using a sample chamber psychrometer C-51 (Wescor Inc., Logan, Utah) to measure soil samples of high water potentials (-1 to -50 bars).

Using Whatman No. 42 filter papers, Hamblin (1981) compared calibration curves from two different batches of filter papers two years apart and found they were nearly identical. Hamblin emphasized the convenience and reliability of the filter paper method for measuring soil water potentials over a wide range of values for both in situ and laboratory research. He warns, however, that using a Peltier-type psychrometer (see Appendix A) to measure water potentials may be erroneous due to the water loss from the paper during cooling of the psychrometer thermocouple. More representative results were obtained when a small disc was stamped from the paper and introduced into a psychrometer leaf block apparatus for water potential measurement.

CHAPTER THREE

EXPERIMENTAL PROCEDURES AND RESULTS

This chapter describes the experimental procedures used to evaluate the methods used for determining in situ water potentials of a fractured rock. The absorber method in conjunction with the thermocouple psychrometer SC-10A was given the greatest emphasis in this study. The purpose of the experiments and discussion of the results are also found in this chapter.

3.1 Downhole Tensiometer

The downhole tensiometer is based on the same principles as standard tensiometers: a porous cup filled with water in good hydraulic contact with a medium, will lose water through the cup wall until the water tension inside the cup equals the tension of the surrounding medium. The tension or potential is transmitted through the water filled tube to a vacuum gauge (manometer or pressure transducer) and is recorded. As mentioned previously, many variations in tensiometer designs have been developed; this research involves the development of a specialized tensiometer for use in boreholes in fractured rock.

Criteria for the design of the downhole tensiometer are that good hydraulic contact is made with the borehole wall, it is easily moved within the borehole, and is attached to a pressure transducer that accurately measures negative pressures.

One of the most difficult tasks is to insure hydraulic contact between the porous cup and the borehole walls. Silica flour or finely crushed rock of the rock being measured can be used to make the hydraulic connection. The fine silica flour or rock powder slurry should be as close as possible to the initial water content of the rock being measured. Some researchers suggest using home insulating foam between the borehole walls and the silica flour which surrounds the tensiometer (Marvil, person. comm.). As the silica flour slurry dries out, it becomes a cement, making removal of the tensiometer difficult as well as contaminating the borehole. The use of insulating foam may aid in easy removal of the tensiometer at the completion of the experiment.

The tensiometer design developed for this study is presented in Figure 3.1. The tensiometer consists of a 1 bar, high flow ceramic cylinder 4 cm in diameter and 15 cm long (manufactured by Soilmoisture Equipment Corp., Santa Barbara, CA). The cylinder is sealed at both ends with stainless steel caps; one cap has an inlet for filling and attaching a pressure transducer. The Micro Switch pressure transducer model 246PC15GW (manufactured by Micro Switch, Honeywell division, Phoenix, AZ) measures pressures from +15 to -15 psi (1 bar). The pressure transducer was calibrated in a vacuum chamber at negative pressures ranging from 0 to -0.85 bars (see Figure 3.2).

The laboratory experiments involved sealing the tensiometer assembly in a plexiglass cylinder 10 cm in diameter and 50 cm long with an inlet for the vacuum pump and pressure transducer wires. A vacuum, regulated with a pressure gauge and release valve, was created within the plexiglass cylinder to simulate water potentials of 0 to -0.85 bars.

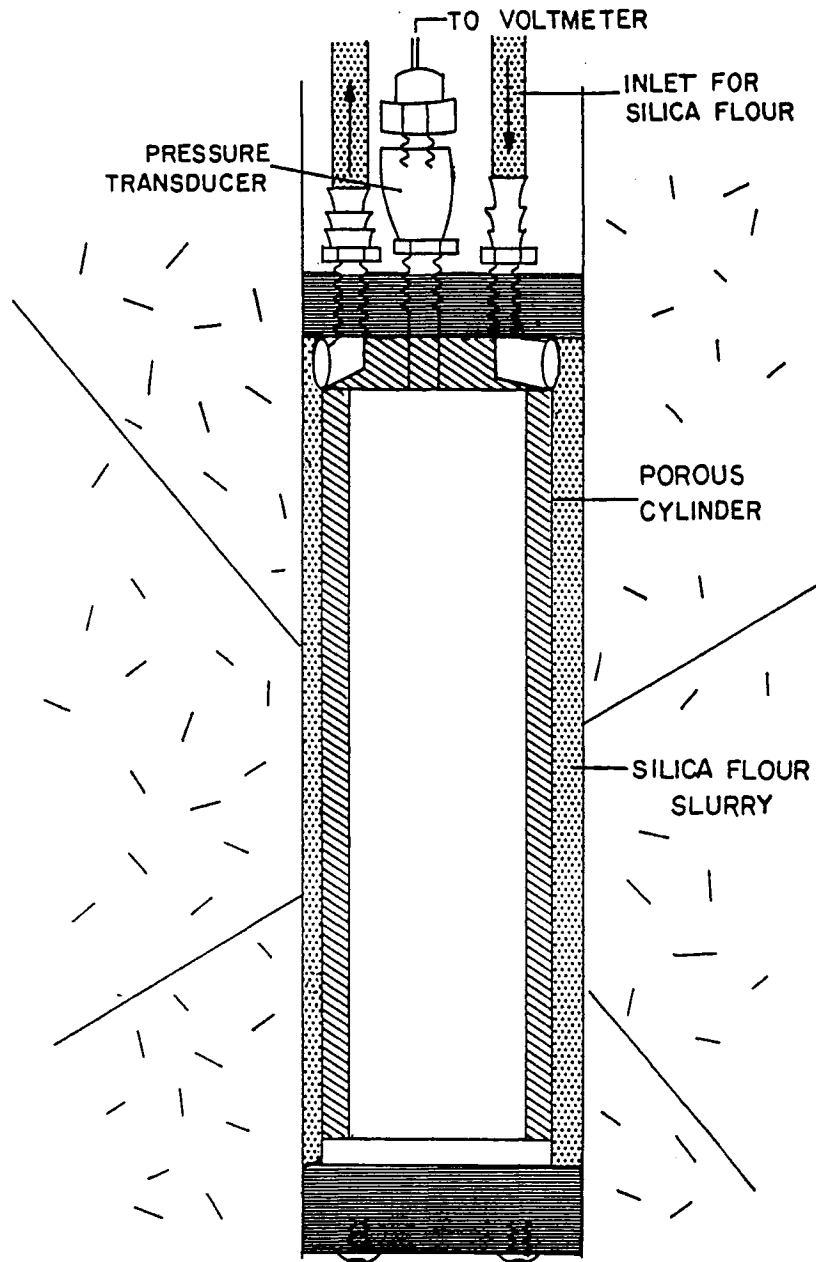


Figure 3.1. Diagram of the downhole tensiometer placed in a borehole in fractured rock.

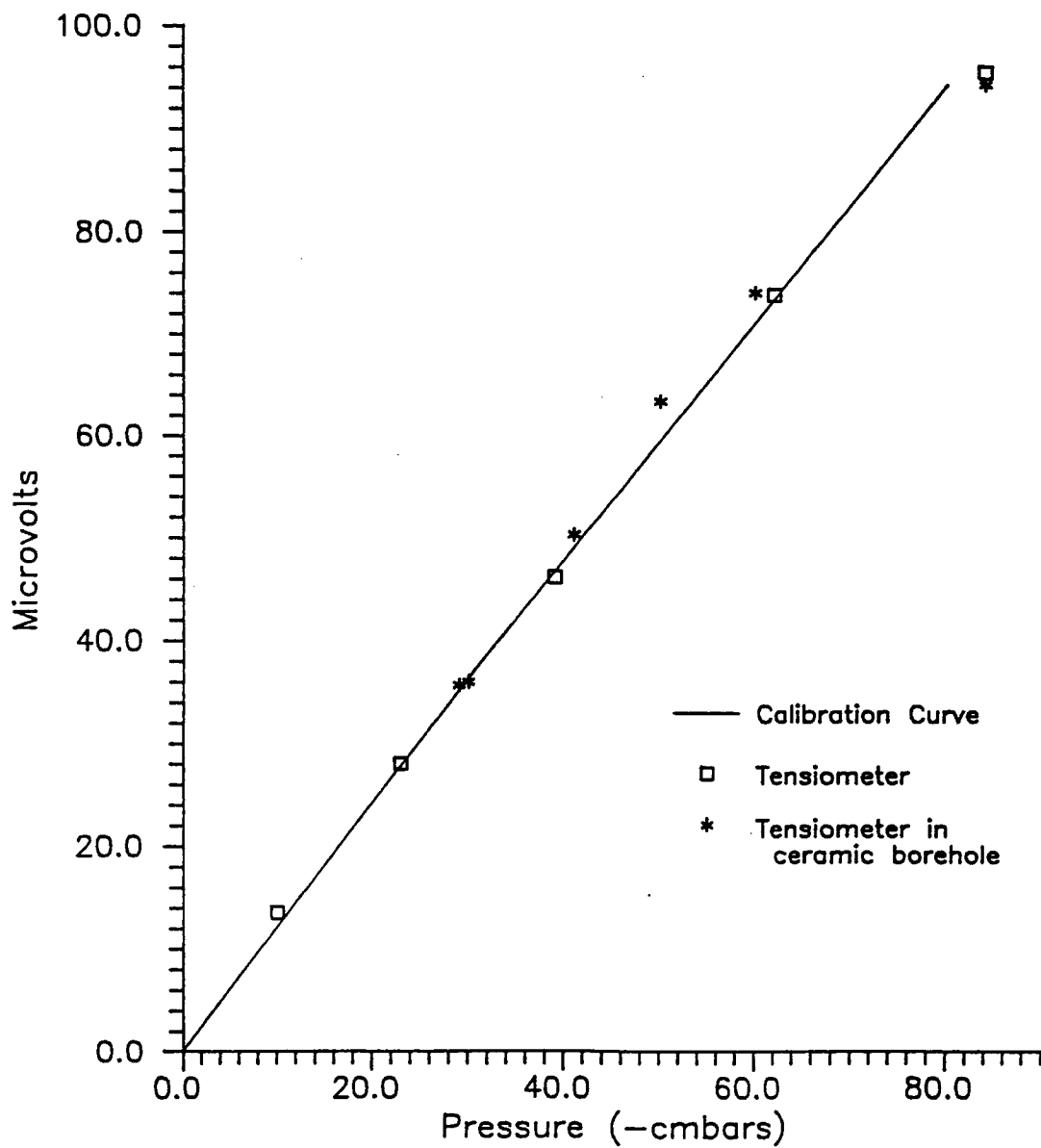


Figure 3.2. Downhole tensiometer response (microvolts) to various negative pressures induced with a vacuum pump. Results are compared with pressure transducer calibration curve.

The results give a near perfect fit with the pressure transducer calibration curve (Figure 3.2). The pressure transducer, with output in microvolts, gave stable readings after approximately five minutes at a constant negative pressure.

In the second laboratory experiment, a ceramic cylinder, with an inner diameter (4.7 cm) slightly larger than the tensiometer, was saturated and used as a "borehole" (see Figure 3.3). The annular space between the tensiometer and the "borehole" was filled with saturated silica sand to create the hydraulic connection with the ceramic borehole. The entire apparatus was placed in the plexiglass cylinder and various negative pressures were applied with the vacuum pump. The results, presented in Figure 3.2, give a near perfect fit with the calibration curve as well as the results of the previous experiment. Even though the second experiment involved the water potential equilibration of the saturated sand and the ceramic borehole, the response time of the tensiometer was nearly the same for both experiments (5 to 6 minutes).

The rapid and accurate response of the downhole tensiometer during laboratory experiments supports this method as viable for determining in situ rock water potentials when the water potentials are believed to be between 0 and -0.85 bars. Future field experimental procedures would involve placement of the tensiometer in a borehole. A slurry of silica flour or crushed rock would be forced in to the annulus between the borehole and the tensiometer creating the hydraulic connection. The pressure transducer measures the changes in water potential of the slurry as it equilibrates with the rock mass.

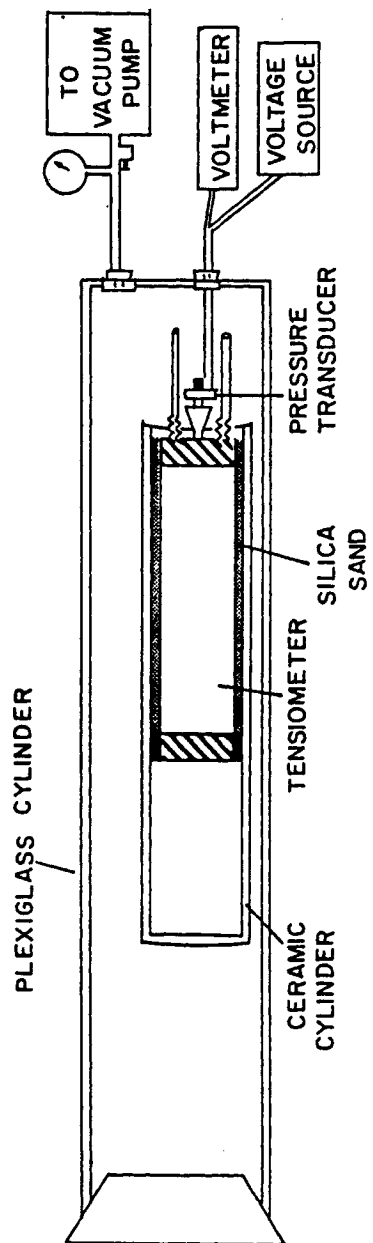


Figure 3.3 Diagram of the apparatus used to assess response of the downhole tensiometer at various negative pressures.

Continuous measurements could be made with a battery operated chart recorder, or measurements could be made periodically while the tensiometer is left in place for an extended period of time. The small amount of available water and the expected slow movement of water in rock would probably cause a lag time in the equilibration of the tensiometer.

3.2 Osmotic Tensiometer

The ability of the osmotic tensiometer to measure in situ matric potentials of unsaturated fractured rock was assessed. Figure 3.4 illustrates how the prototype osmotic tensiometer appears following insertion in a borehole of fractured rock. The core of the prototype (3.8 cm diameter x 91.4 cm stainless steel pipe) contains internal access tubes (0.32 cm diameter) which open to the annular space at each end of the device. A tubular semipermeable membrane provides the contact between moisture in the rock surrounding the borehole and the osmotic solution within the tensiometer. After the ends of the membranes are sealed by compressing them between a flat rubber gasket and an o-ring, a circular end plate is bolted to the stainless steel core.

Following insertion into a borehole, the annular space between the core and the membrane is filled with the osmotic solution. Filling is accomplished by applying positive pressure to the solution reservoir which is attached to the inlet access tube. After the device is filled, the valves on the vacuum (outlet) and inlet lines are closed. A pressure gauge or pressure transducer attached to the inlet line measures the hydrostatic pressure within the tensiometer. Subsequently,

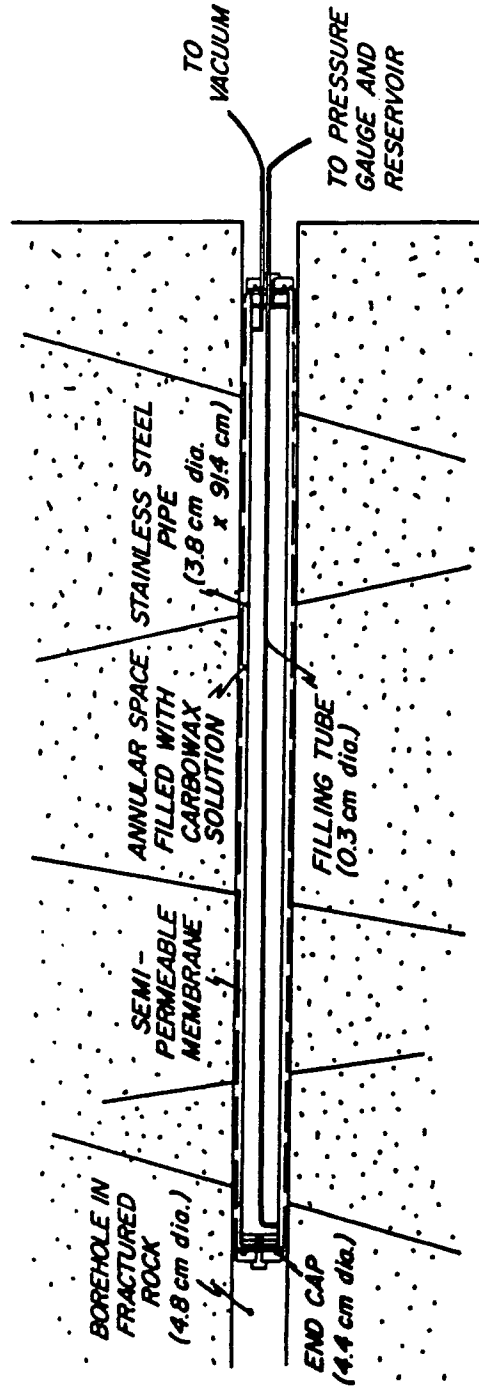


Figure 3.4 Diagram of the prototype osmotic tensiometer in a borehole in fractured rock.

water flows into or out of the device in response to relative changes in total potential on either side of the membrane. The average matric potential of the fractured rock mass in contact with the membrane is equal to the sum of the measured positive equilibrium hydrostatic pressure and the known osmotic potential of the osmotic solution.

The following subsections present laboratory and field studies conducted to assess the optimum combination of semipermeable membrane and osmotic solute for use in the prototype.

3.2.1. Membranes

The following criteria were used to select a semipermeable membrane:

1. maintains sufficient strength and flexibility to permit insertion into a borehole without damage and withstand several bars of hydrostatic pressure from within;
2. readily permeable to water at low pressure gradients;
3. permeable to inorganic solutes commonly found in water within the fractured rock;
4. impermeable to large organic molecules used to create an osmotic potential;
5. available in tubular form with diameters approximately equal to the boreholes;
6. resistant to microbial decomposition.

Of the various membranes tested, a regenerated natural cellulose membrane, Spectra/por 4, distributed by Spectrum Medical Industries, Inc., Los Angeles, CA, was the most acceptable. Spectra/por 4 has a molecular weight cutoff of 12,000 to 14,000 and a diameter similar to

that of the boreholes. The molecular weight cutoff for the membranes is defined on the basis of 90% retention of a globular protein over a 24 hour period. Since the membrane is a gel when wet, some enlargement of pore size may be expected when mechanical strain is applied thereby changing the solute retention characteristics of the membrane. Retention efficiency will also vary depending on the structure and chemical characteristics of a particular solute. For example, linear long chain molecules will pass through the membrane much more readily than a coiled spherical molecule of similar molecular weight. Additionally, the manufacturer's definition of molecular weight cutoff is not rigorously restrictive so that complete retention of a solute can only be achieved if the solute molecule is significantly larger than the specified molecular weight cutoff.

3.2.2. Solutes

The solute molecules utilized to create a known osmotic potential should be large enough to be retained by the semipermeable membrane, inert with respect to the membrane and metal used in the core of the tensiometer, chemically stable, and resist microbial decomposition. The organic solutes evaluated include; polyethylene glycol, polyvinyl pyrrolidone, and polyethylene oxide.

Polyethylene glycol, commonly referenced by its trade name, Carbowax (manufactured by Union Carbide), was utilized by previous investigators of the osmotic tensiometer. Carbowax polymers are available in numerous molecular weights (m.w.) between 200 and 20,000. Calibration curves relating osmotic potential to carbowax concentration in aqueous solutions are available in Zur, 1966 (6,000 m.w.) and Peck

and Rabbidge, 1969 (20,000 m.w.). The practical limit to the maximum osmotic potential obtainable with these solutions is about -15 bars.

Polyvinyl pyrrolidone (PVP) has a less linear and more rigid structure than Carbowax and is available in a wider range of molecular weights. PVP was initially developed for use as a blood plasma expander so most published research relate to this application. Experiments conducted during this study utilized 40,000 and 360,000 m.w. polymers and has a practical limit of obtainable osmotic potentials of about -2 bars. PVP is currently classified as a "cancer suspect agent". Due caution should be exercised in its use.

Polyethylene oxide (PEO) does not pose the potentials health risk associated with PVP use. Osmotic potentials of 0 to about -3 bars can be generated with 200,000 m.w. PEO. As the osmotic potential decreases, the viscosity of the solute increases thus limiting the range of osmotic potentials obtainable.

3.2.3. Experimental Methods

Various laboratory experiments were performed to evaluate the ability of the prototype osmotic tensiometer to measure water potentials within the borehole of a fractured rock. The prototype was tested in a concrete block, PVC pipe, a cotton cloth tube, and a porous ceramic pipe. Small plexiglass cells, which served as osmometers, were used to evaluate the relationship of concentration and osmotic potential.

Osmometer. The osmometer consists of cylindrical plexiglass cells separated by a semipermeable membrane supported by a porous glass disk (see Figure 3.5). The side of the device in contact with the membrane was filled with the osmotic solution such that no air was

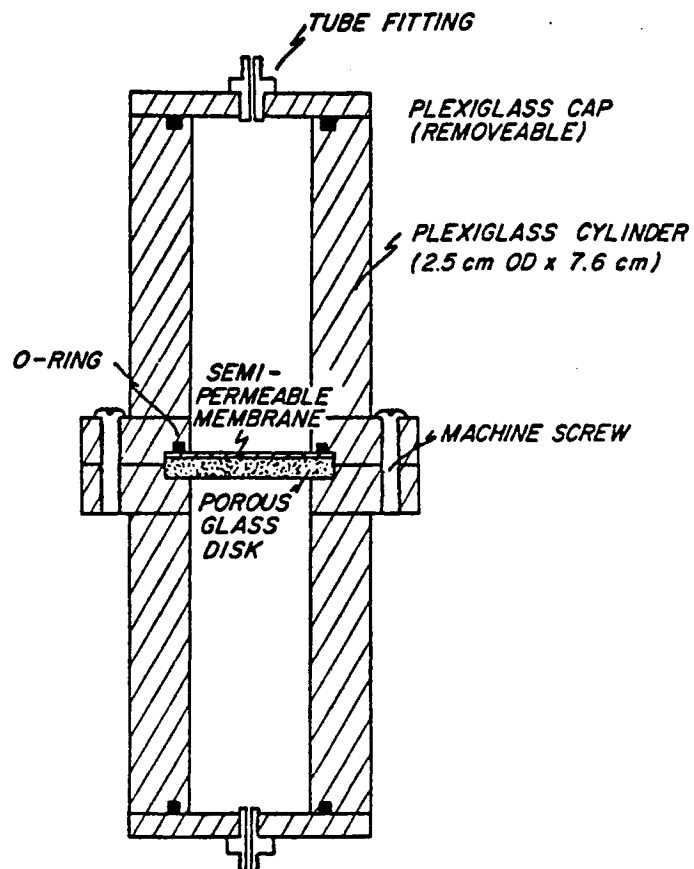


Figure 3.5. Diagram of plexiglass cells used to determine osmotic potentials of solutes.

trapped in the cell. It was then sealed and connected to a pressure transducer. The other side was partially filled with distilled water. Water flowed through the membrane into the osmotic solution until equilibrium was achieved. At equilibrium, the positive hydrostatic pressure in the cell with the osmotic solution was assumed to be equal in magnitude to the negative osmotic potential of the solution. Experiments of this kind were repeated using various concentrations of solutions containing different organic solutes in order to define a concentration vs. osmotic potential calibration curve for each solute. Figures 3.6 and 3.7 demonstrate examples of calibration curves for 200,000 m.w. PEO and 360,000 m.w. PVP.

Prototype Laboratory Experiments. A concrete block with a 4.75 cm diameter borehole was used to simulate a borehole in fractured rock. The prototype was inserted into the borehole and the concrete block was submerged in water to simulate saturated conditions. The block was allowed to dry, making it possible to observe the response of the tensiometer under changing moisture conditions. However, the use of the concrete block was abandoned because the osmotic potential of the water in the concrete matrix was unknown and uncontrollable.

A PVC pipe, about 10 cm longer than the prototype with an internal diameter about the same as that of the boreholes at the field sites, was also used to evaluate the prototype. The advantage of this setup was that the osmotic and matric potentials were known to be zero. The disadvantage was that water only flowed into or out of the tensiometer through a small ring of membrane at either end of the device. The major portion of the membrane surface area was nonconductive when the

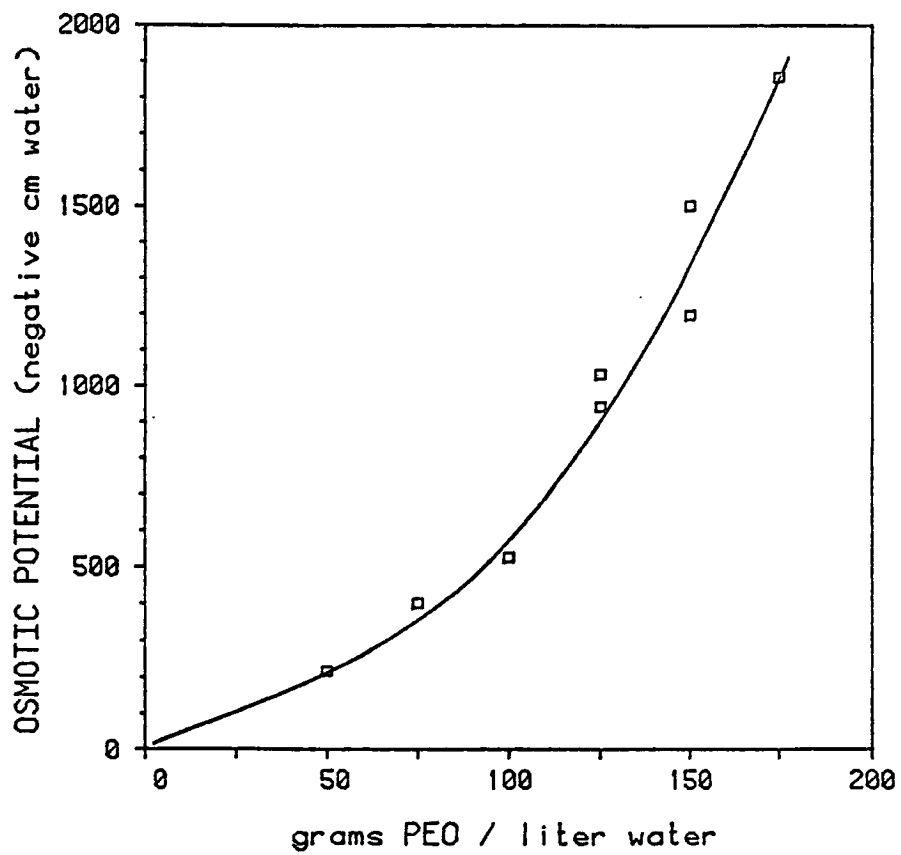


Figure 3.6. Concentration vs osmotic potential relationship determined for 200,000 m.w. Polyethylene oxide.

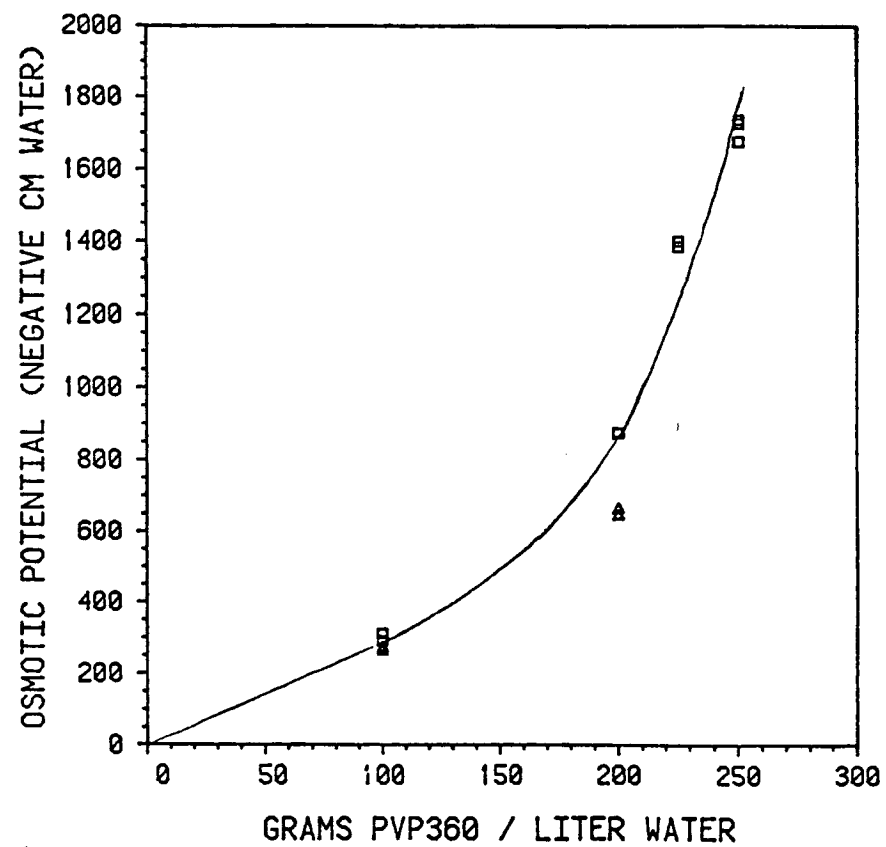


Figure 3.7 Calibration curve for 360,000 m.w. Polyvinyl pyrrolidone. Squares represent values determined using the plexiglass cells and triangles represent values determined using the prototype osmotic tensiometer in saturated ceramic pipe.

membrane was inflated against the inner surface of the impermeable PVC.

Insertion of the osmotic tensiometer in a cotton cloth tube having a diameter equal to that of the borehole followed by submergence in water permitted maximum flow of water through the entire membrane. This setup used to evaluate the prototype had the advantage of being able to see air bubbles trapped under the membrane. Additionally, leaks could easily be located in the fittings, welds, and O-ring seals by filling the tensiometer with compressed air. Instrument response over a 12.5 day period is shown in Figure 3.8. The membrane and cloth tube expanded to a diameter of 6.5 cm in the first 20 hours as water moved into the membrane. The hydrostatic pressure plateau reached at about 48 hours (310 to 315 cm of water) agrees with the osmotic pressure value (-331 cm of water) determined for the same 20,000 m.w. Carbowax solution (78 g/l) using the osmometer. The gradual pressure decay during the last 10 days of the experiment may be attributed to leakage of Carbowax through the membrane as it stretched thus widening the membrane pores. A disadvantage of this experimental setup was that the cloth tube and membrane stretched significantly as water flowed into the tensiometer. Substantial dilution of the osmotic solution resulted in reduction of the effective osmotic potential gradient across the membrane.

The best method found for simulating a borehole in rock was accomplished with a ceramic pipe, 0.3 cm thick wall and having an internal diameter of 4.75 cm. The ceramic pipe provided an excellent rigid, porous support for testing the prototype under a variety of conditions. By inserting the ceramic pipe containing the tensiometer in a larger diameter (15 cm) plexiglass pipe partially filled with water, a

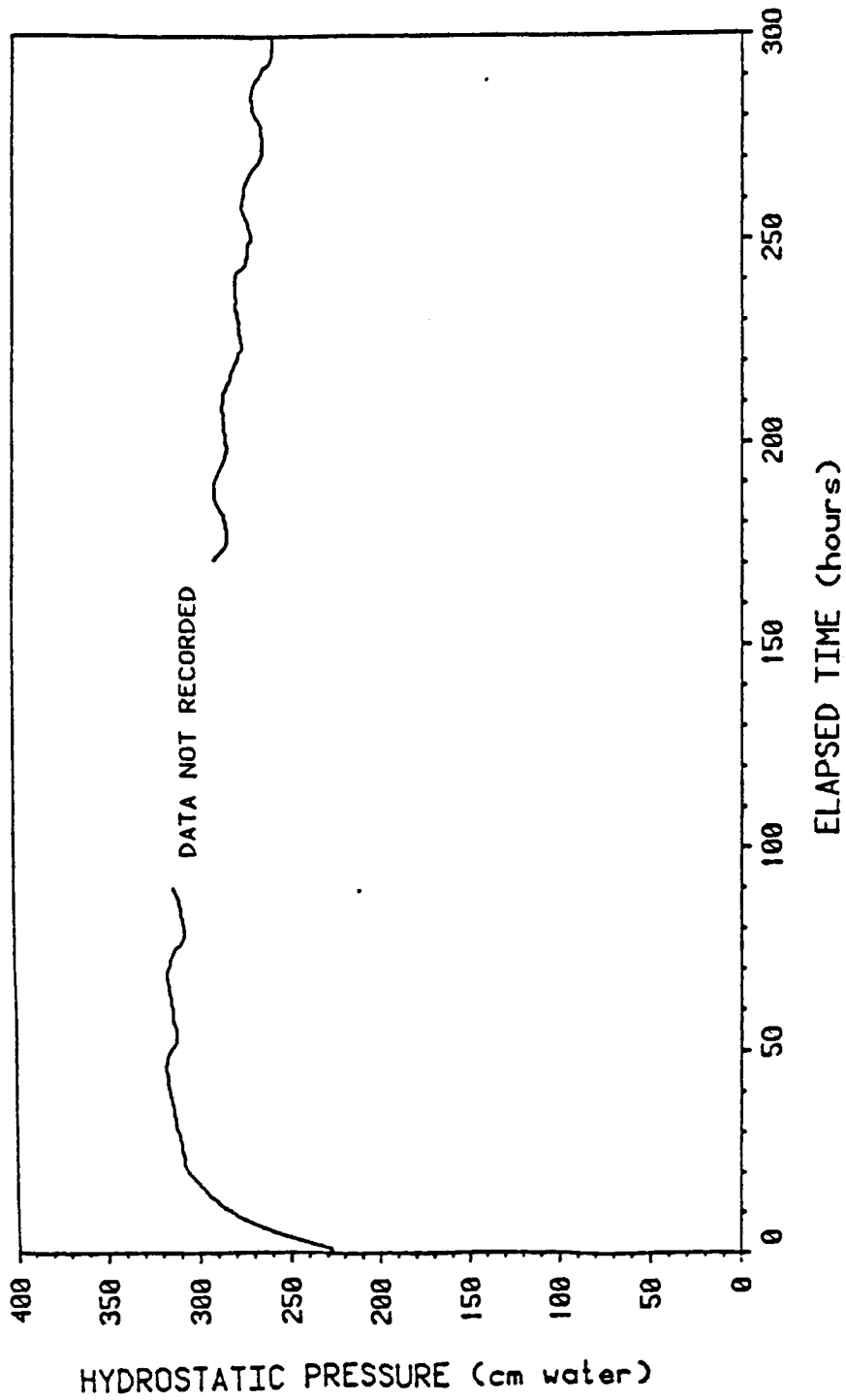


Figure 3.8 Tensiometer response curve for an experiment in which the portotype with Carbowax inserted into a cloth "borehole" and submerged in water.

series of external water potentials between 0 and -0.8 bars could be created (see Figure 3.9). The water potentials were created by applying a negative pressure (with a vacuum pump) to the airspace overlying the water. A comparison of the known negative air pressures applied and the hydrostatic pressure changes observed within the device was used to evaluate the response characteristics of the tensiometer. Ideally, the observed changes in hydrostatic pressure should be identical to the air pressure changes and the rate of reequilibration should be rapid enough to verify the usefulness of the tensiometer for measuring water potentials under changing field conditions. The major disadvantage to the experimental setup is that the limited range of external potentials created with a vacuum pump are much less than the potentials likely to be encountered in the field.

Experiments of this type described for the ceramic pipe were performed using Spectra/por 4 membranes and 360,000 m.w. PVP solute. Response curves for tests involving 200 and 250 g PVP 360K/l solutions are presented in Figures 3.10 through 3.12. Tensiometer response to a change in external potential began within a few minutes and reequilibration generally occurred in less than one hour. These results verify that the prototype osmotic tensiometer can be used to measure rapidly changing moisture conditions in a real situation. Data for these experiments are summarized in Table 3.1 in a form which permits assessment of the accuracy of the potential measurements made with the tensiometer. The first column tabulates the regulated changes of external air pressure. The second column is the observed changes in hydrostatic pressure within the tensiometer. Plus and minus signs

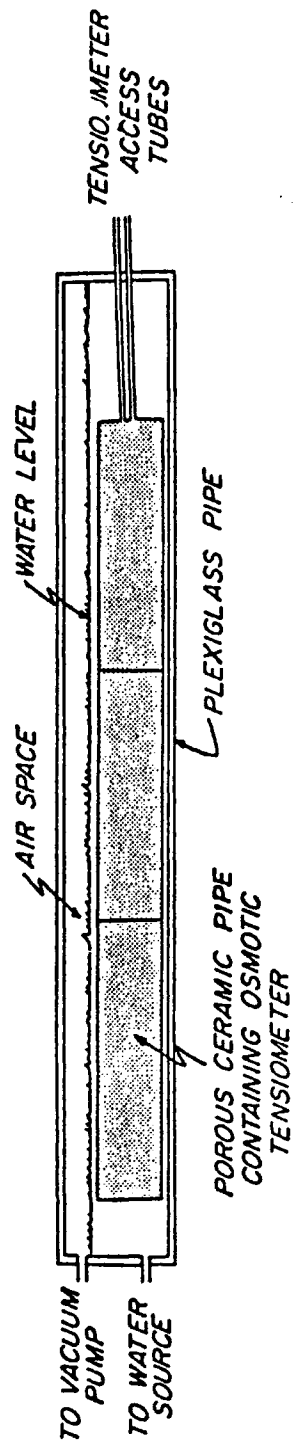


Figure 3.9 Diagram of apparatus used to assess osmotic tensiometer response under variable external potentials.

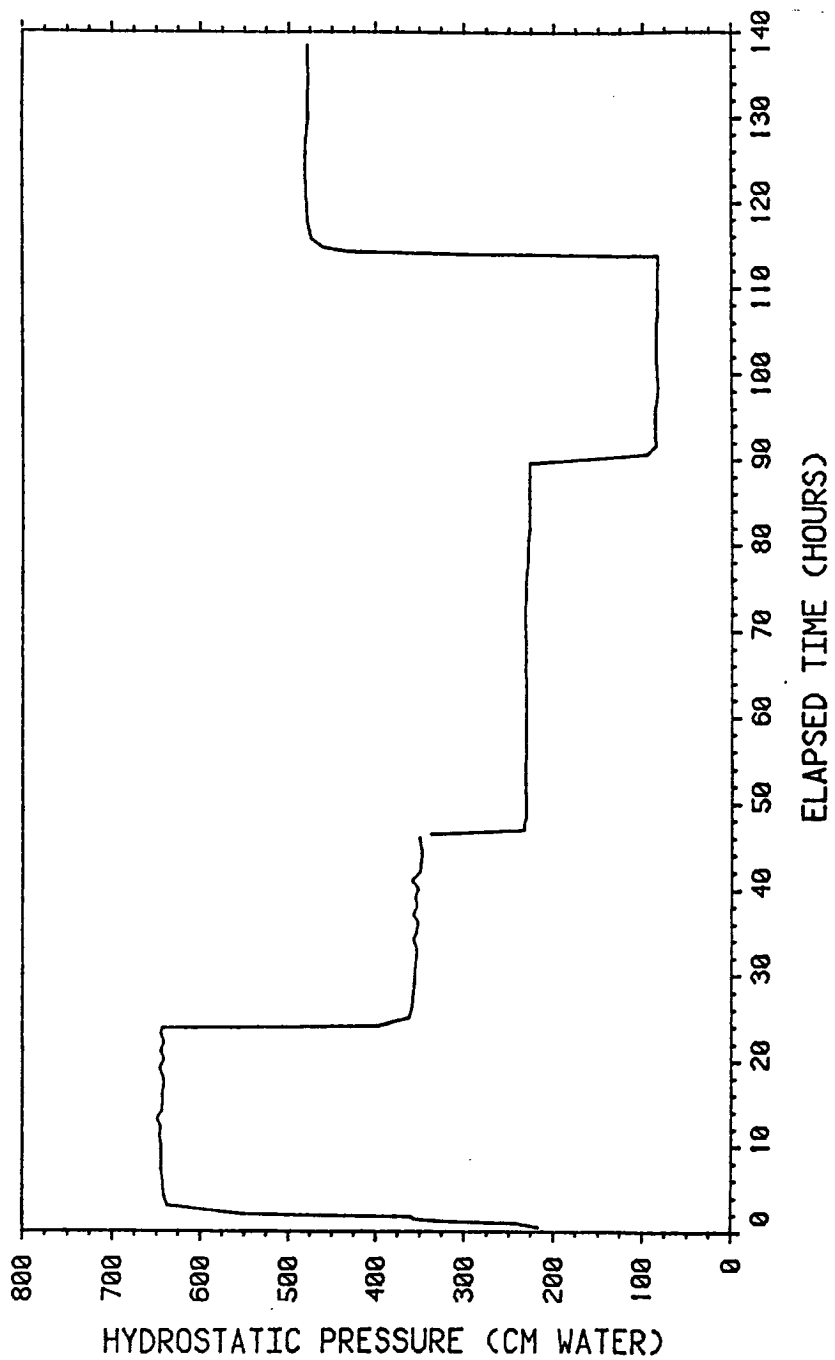


Figure 3.10 Osmotic tensiometer response to controlled changes in external potential. Tensiometer contains 200g PVP 360K/liter water with an osmotic potential of -850 cm of water.

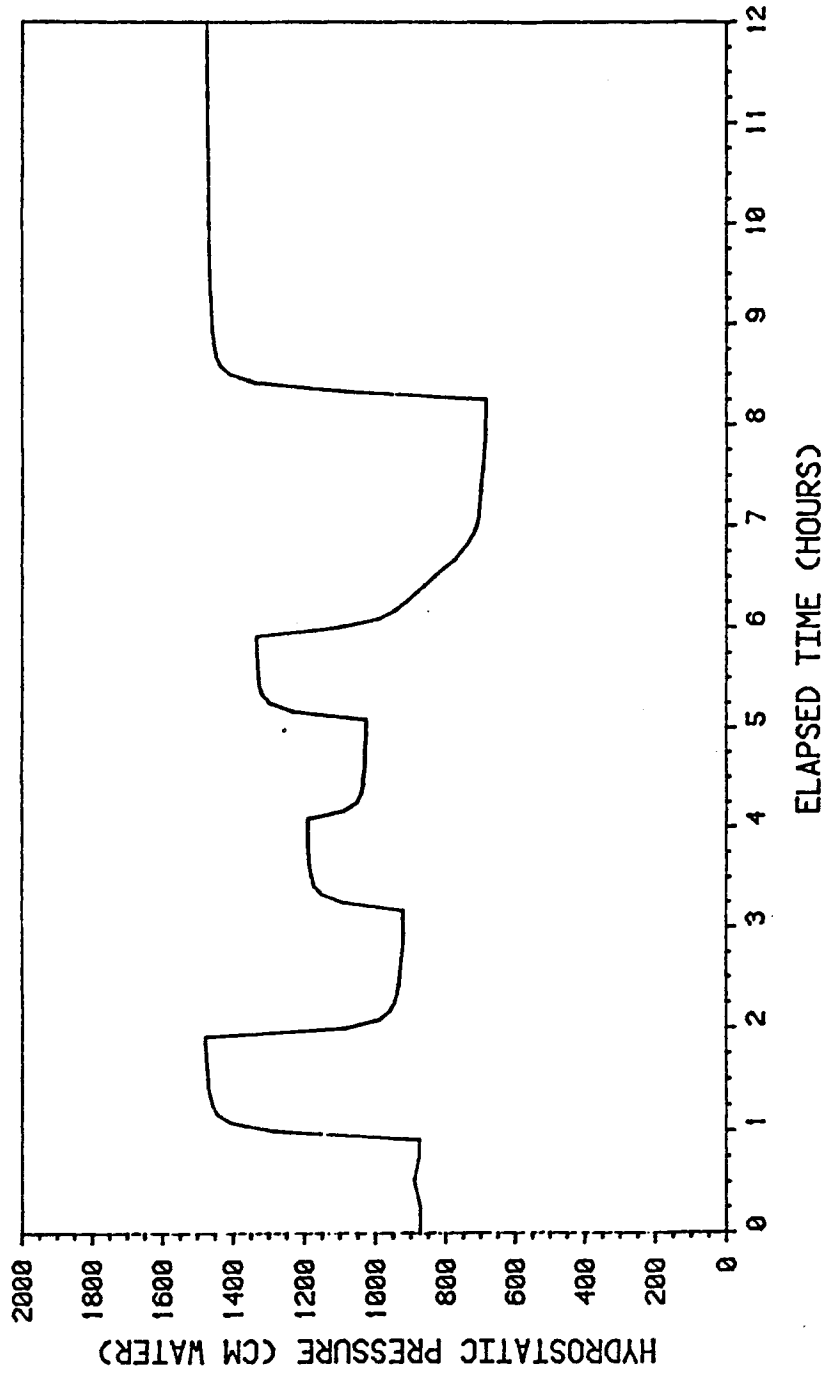


Figure 3.11 Osmotic tensiometer response to controlled changes in external potential. Tensiometer contains 250g PVP 360/liter water with an osmotic potentials of -1750 cm of water.

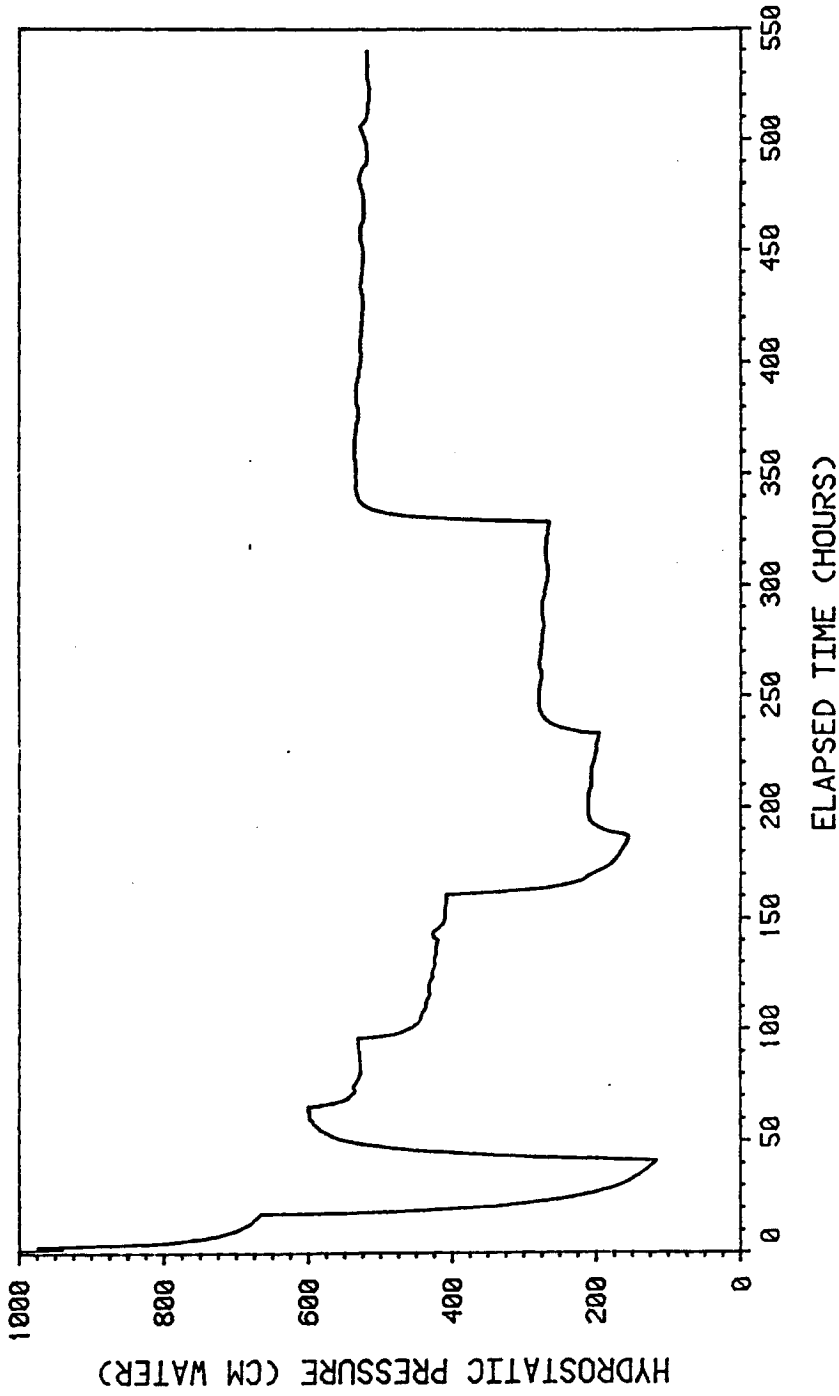


Figure 3.12 Osmotic tensiometer response to controlled changes in external potential. Tensiometer contains 200g PVP 360/liter water with an osmotic potential of -850 cm of water.

Table 3.1. Summary of induced air pressure changes and resulting hydrostatic pressure changes within the prototype osmotic tensiometer.

1. Air Pressure Change (cm H ₂ O)	2. Hydrostatic Pressure Change (cm H ₂ O)	3. Difference (cm H ₂ O)	4. Error (2 - 1)/1
FIRST EXPERIMENT (200g PVP360/1 H₂O)			
-307	-298	- 9	-.029
+307	+283	-24	-.078
-320	-310	-10	-0.31
-180	-183	+ 3	+.017
-131	-149	+18	+.137
+406	+396	-10	-.025
+131	+108	-23	-.176
SECOND EXPERIMENT (250g PVP360/1 H₂O)			
-755	-785	+30	+.040
+120	+115	- 5	-.042
+625	+603	-22	-.035
-584	-558	-26	-.045
+283	+271	-12	-.042
-171	-164	- 7	-.041
+321	+309	-12	-.037
-673	-653	-20	-.030
+824	+799	-25	-.030
THIRD EXPERIMENT (200g PVP360/1 H₂O)			
-611	-550	-61	-.100
+611	+484	-127	-.208
- 82	- 69	-13	-.159
-114	-125	+11	+.096
-285	-252	-33	-.116
+ 80	+ 41	-39	-.488
+104	+ 80	-24	-.231
+297	+260	-37	-.125

indicate pressure changes in positive or negative directions. Columns 3 and 4 show the measurement errors (differences between measured changes in air pressure and hydrostatic pressure) expressed in cm of water and decimal fraction of the induced air pressure. Errors are generally less than 25 cm in the first two experiments with the hydrostatic pressure change usually being less than the corresponding air pressure change. The larger errors observed during the third experiment may be due to errors in air pressure measurements rather than to errors in the tensiometer. Difficulties were encountered throughout this experiment in attempts to control and stabilize the negative air pressures within the apparatus.

As in previous experiments, gradual decay was noted during these experiments with regard to the sum of the measured hydrostatic pressure and absolute value of air pressure. The rate of decay varied from 0.1 to 0.5 cm/hour for the three experiments and did not pose problems as long as potential measurements are made over a period of a few days.

3.2.4 Field Experiments

This subsection presents field results of the osmotic tensiometer using three different solutes for measuring water potentials in fractured rock.

Carbowax. Water potential measurements were made in two boreholes in unsaturated, fractured granodiorite at the Santo Nino Mine field site (Rasmussen and Evans, 1986) using Spectra/Por 4 membranes filled with a solution of 20,000 m.w. Carbowax with an osmotic potential of -1020 cm of water. Figure 3.13 illustrates the resulting response curve. The initial pressure increase in Borehole 5.8 may be due to

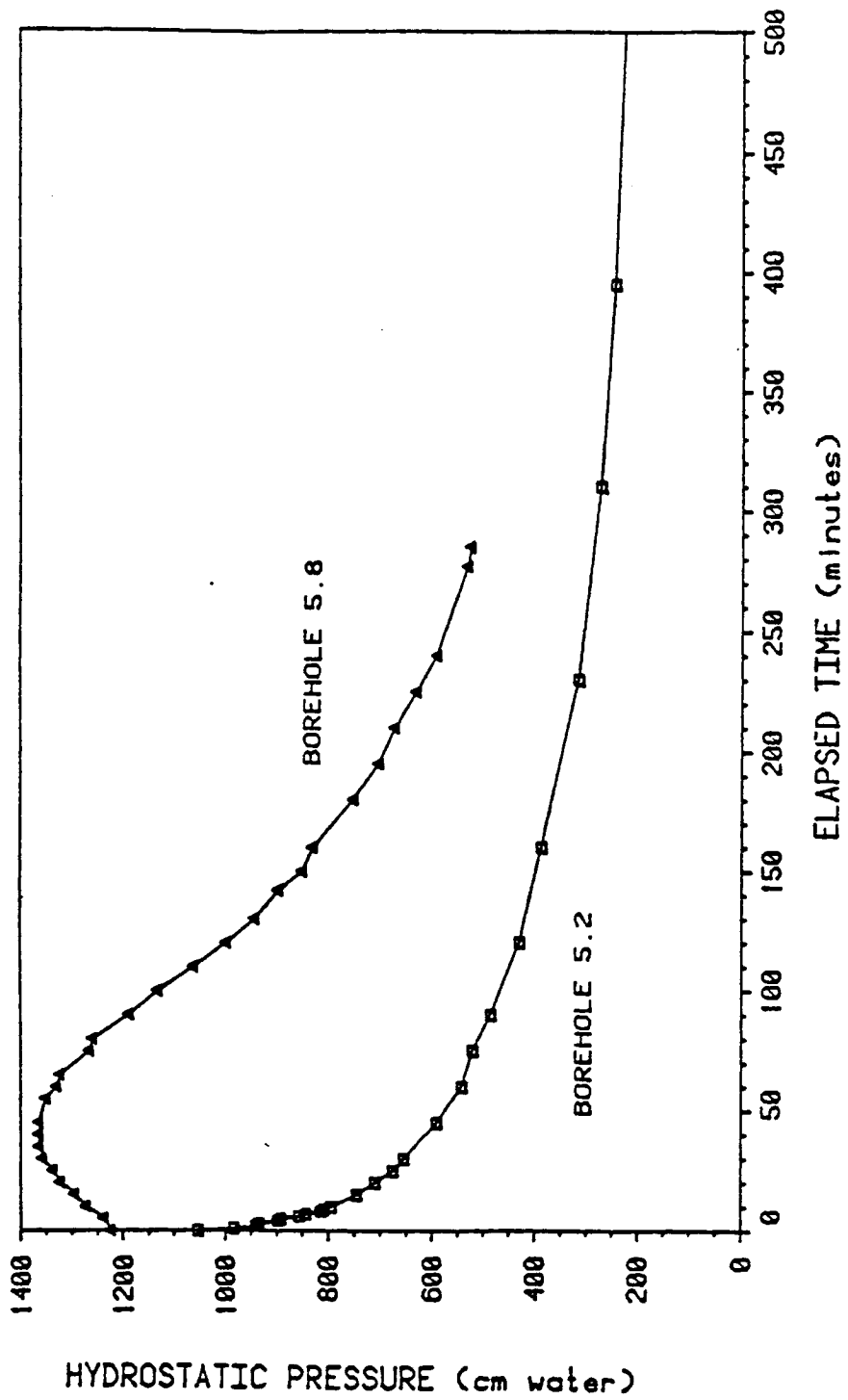


Figure 3.13 Tensiometer response curve in fractured granodiorite at the Santo Nino mine; tensiometer contains Carbowax solution.

temperature equilibration (the borehole temperature was higher than that of the Carbowax). Both response curves indicate a gradual pressure decay with time. An equilibrium value of about 200 cm of water was eventually reached in Borehole 5.2 which implies a total water potential of about -820 cm of water. Although this value seems reasonable, subsequent analysis proved the results to be questionable due to Carbowax leakage through the membrane.

PVP. Water potential measurements were made in Borehole 28.3 in an area of the Santo Nino mine where the granodiorite matrix appeared to be wet. The tensiometer was filled with 40,000 m.w. PVP solution having an osmotic potential of -1150 cm of water. The response curve is presented in Figure 3.14. The sharp drop at 7 hours was manually induced to verify that equilibrium had been reached. The curve suggests that the equilibrium hydrostatic pressure for the system was between 350 and 380 cm of water. Thus, the water potential in the fractured granodiorite was approximately -785 cm of water, which appears to be reasonable.

PEO. Another field test conducted in Borehole 28.3 at the Santo Nino mine utilized a Spectra/Por 4 membrane and 180g PEO 200,000 m.w./liter water solution with an osmotic potential of -2000 cm of water. A cotton tube was slipped over the membrane prior to insertion into the borehole. After filling the tensiometer with the PEO solution, hydrostatic pressure within the device was monitored over a 6 day period. Data for the first 24 hours are presented in Figure 3.15. Hydrostatic pressure initially increased to a plateau value of 2358 cm of water and remained constant for 2 1/2 hours. This value is similar to

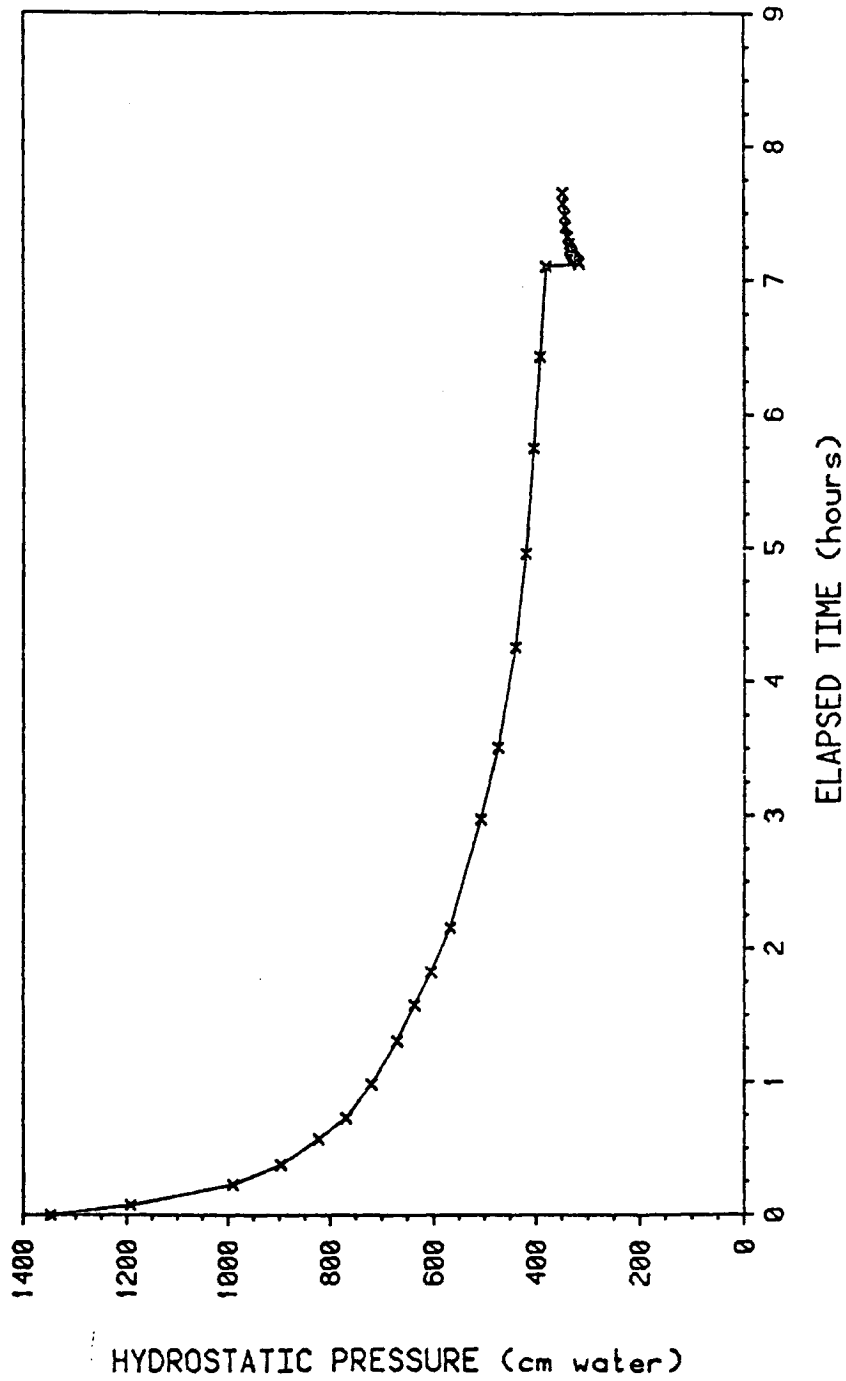


Figure 3.14 Response curve for the osmotic tensiometer in borehole 28.3 at Santo Nino mine. Tensiometer contained a 40,000 m.w. PVP solution.

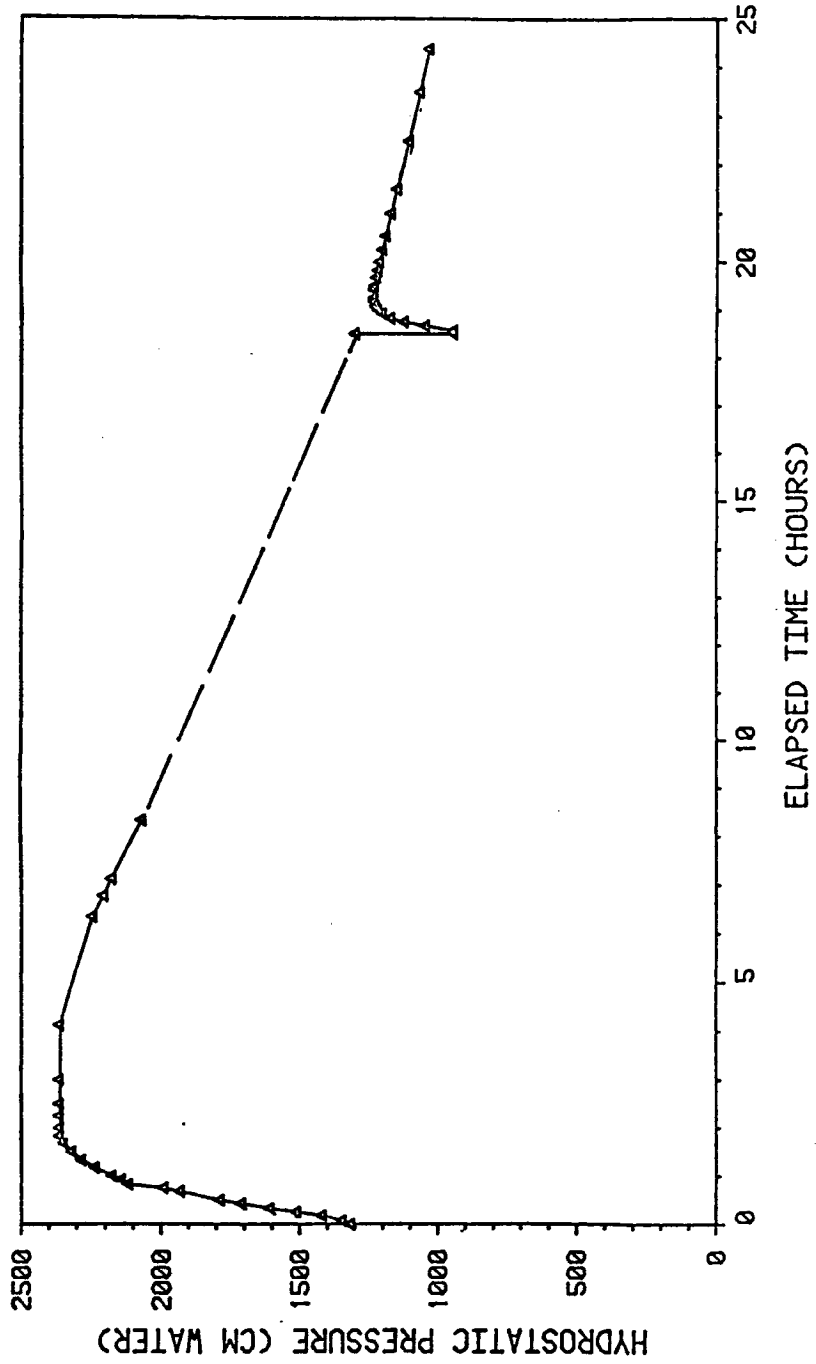


Figure 3.15 Response curve for the osmotic tensiometer in fractured granodiorite at the Santo Nino mine site.

the osmotic potential of the PEO solution within the tensiometer. This plateau was caused by the flow of water from the initially wet protective cloth into the tensiometer (i.e., the tensiometer was initially subjected to artificially saturated conditions). The decay of hydrostatic pressure was initially interpreted as reequilibration of moisture conditions, but the rate and extent of pressure decay during the next 20 hours cast doubt on the validity of the measurements. Data for days 2 to 6 are not plotted, but showed a continued pressure decay to a value less than 200 cm, thus suggesting an unreasonably low potential in the host rock of about -1800 cm of water. When the tensiometer was removed from the borehole, a random array of small stains on the protective cloth verified that PEO leakage had occurred through small pinholes in the membrane.

3.3 Field Psychrometers

For in situ measurements of water potential, the thermocouple psychrometer is protected by a cup-shaped shield made of wire screen or porous ceramic. The shield should permit rapid vapor equilibration between the soil, or rock, and the chamber. Essential components of thermocouple psychrometers include a reference thermocouple (copper-constantan), a measuring thermocouple (chromel-constantan), a protective shield, a Teflon base, and a length of four-strand wire (Figure 3.16). The reference thermocouple is embedded in epoxy within the base of the instrument, isolated from changes in moisture conditions, and is utilized to measure ambient temperature. The measuring thermocouple, supported by a Teflon base and protected by a shield, is exposed to ambient moisture conditions of the rock or soil being measured.

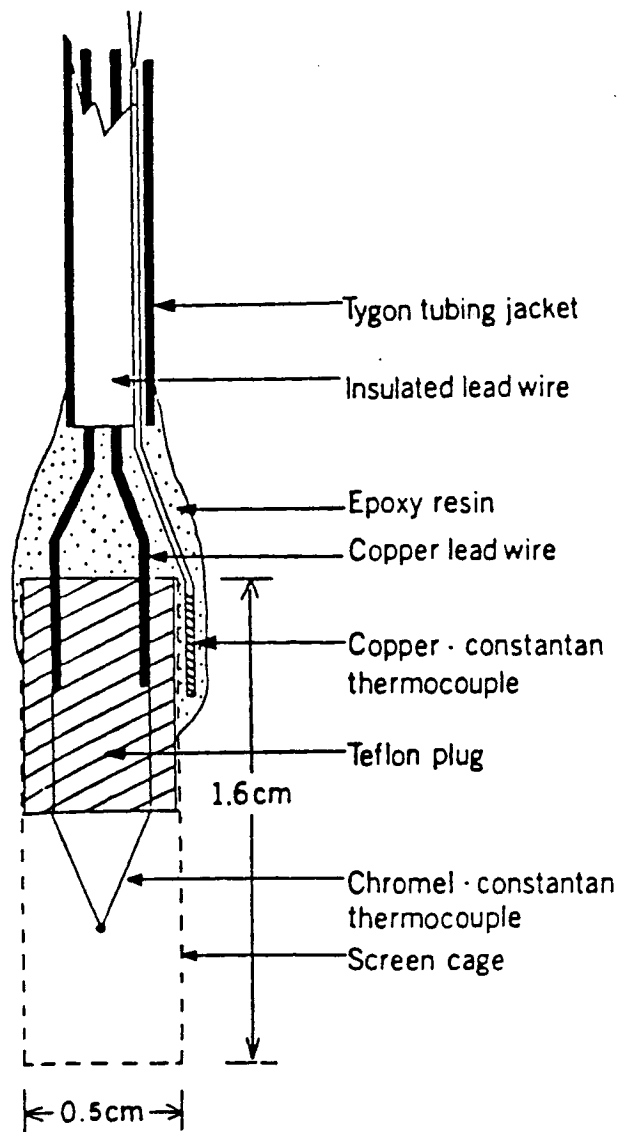


Figure 3.16. Median longitudinal section of a screen-enclosed thermocouple psychrometer.

Psychrometer measurements are made with control units/meters which provide the required switching, cooling circuitry, and signal amplification. Psychrometer response is read in microvolts using a built-in meter or chart recorder.

Because psychrometers are limited to measurements from -2 to -50 bars, a method was needed to define which extreme was prohibiting measurement. At the wet extreme (0 to -2 bars), failure was attributable to moisture evaporation from the wet-measuring thermocouple at a rate insufficient to produce a measurable temperature depression after the cooling current is terminated.

At dry extremes (less than -50 bars), the failure resulted from the fact that condensation cannot be induced on the measuring thermocouple by Peltier cooling. Utilizing the rate at which the measuring thermocouple cools when a fixed current is applied, a method was developed to differentiate between very wet and very dry conditions. A wet thermocouple should cool significantly slower than a dry thermocouple due to the large heat capacity of water films which condense on the thermocouple surface due to Peltier cooling under wet conditions.

Laboratory experiments were conducted with 8 Merrill (J.R.D. Merrill Specialty Equipment, Logan, Utah) screen-cage psychrometers equilibrated over distilled water (0 bars) and 1.6 molal NaCl solution (-76 bars). The results, presented in Figure 3.17 suggest that a distinction can be made by comparing the microvolt readings taken at a fixed time after Peltier cooling is initiated with corresponding values from calibration data. Ideally, one should compare field results for an

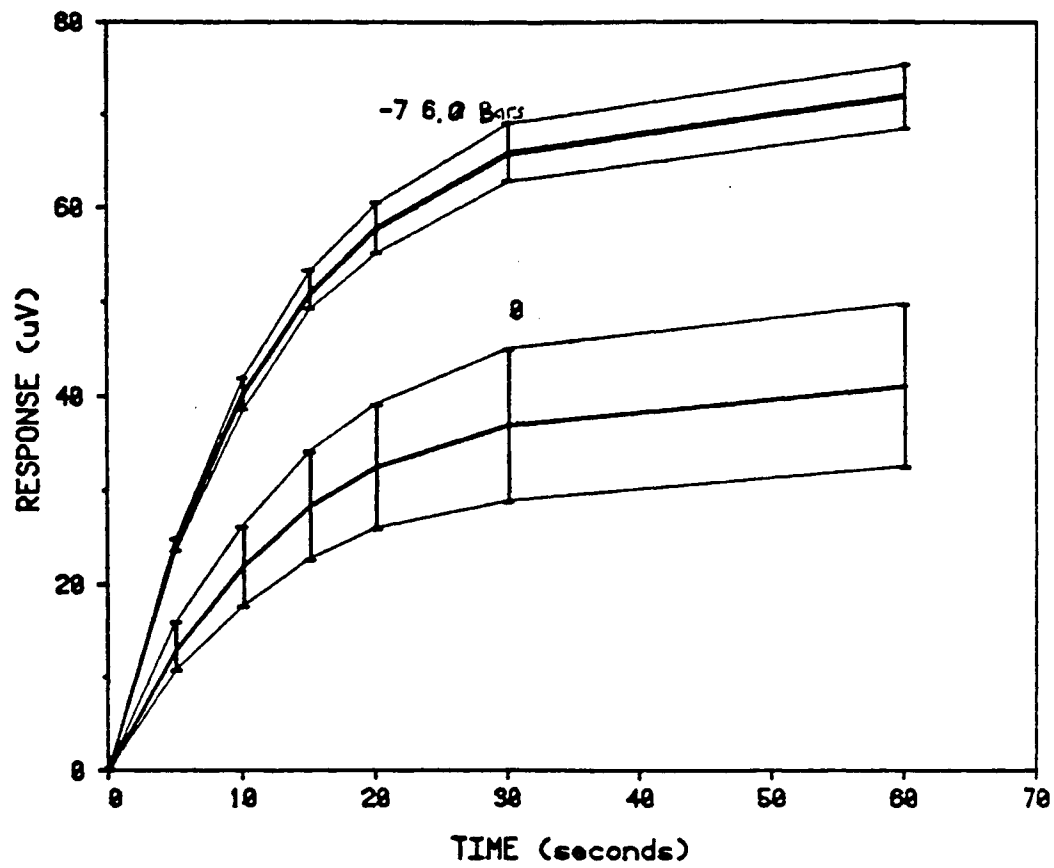


Figure 3.17. Average cooling rate curves for eight psychrometers under very wet and very dry conditions.

individual psychrometer with cooling curves developed specifically for that psychrometer.

3.3.1 Field Experiments

Thermocouple psychrometers have been emplaced for variable periods of time in six boreholes in the Pride-of-the-West mine, six boreholes in the Santo Nino mine, three boreholes in the Queen Creek road tunnel and one in the haulage tunnel in the Magma mine (Rasumussen and Evans, 1986). Typically, two psychrometers have been emplaced and sealed in each borehole using inflatable packers. After an initial equilibrium period, these psychrometers should measure an "average" moisture potential of the rock matrix and of all open fractures which intersect the borehole. Periodic measurements have been made at one to four week intervals in an effort to quantify temporal changes in moisture conditions within each borehole.

Results obtained from the Pride-of-the-West mine and Santo nino mine indicate that moisture potentials for all but Borehole 3 of Pride-of-the-West are out of the psychrometers' measurement range. Results for Borehole 3, presented graphically in Figure 3.18 indicate a wetting trend during the first several weeks followed by essentially constant moisture conditions during the next four months. The initial wetting period may have been the result of a gradual re-equilibration of moisture conditions within the borehole after it was sealed with the packer.

To determine if the response failure in boreholes 1, 4, 5, 6, and 8 was due to excessive moisture or dryness, cooling rate data were obtained. Previous laboratory calibration data suggest that microvolt

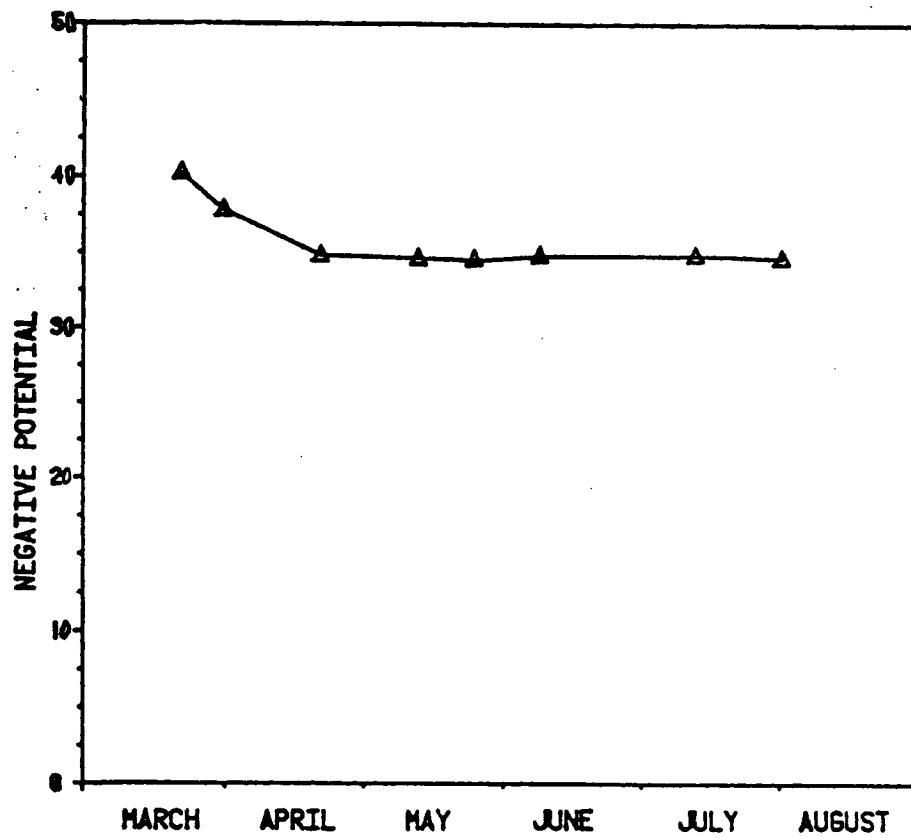


Figure 3.18. Moisture potentials measured in borehole #3 (Pride-of-the-West Mine) using psychrometer #150.

readings recorded after a 30 second cooling period which are less than 45 uv indicate that conditions are too wet; and if the readings are greater than 60 uv, then conditions are too dry. On this basis, Boreholes 5 and 6 appear to be too wet, and Boreholes 4 and 8 are too dry (Table 3.2). Substantiation of this interpretation of conditions in Borehole 5 was obtained when the packer was deflated and a significant volume of water poured out of the borehole. Some water staining on the mine wall beneath Borehole 6 suggests that this borehole was also too wet. Boreholes 4 and 8 show neither staining or excess moisture during the duration of the study.

Moisture conditions in the Santo Nino mine appeared to range from dry to very wet (Table 3.3). All six of the boreholes measured gave readings outside the effective range of the psychrometric and dew-point measurement methods. Using the cooling rate method and the same criteria as before, the results show that Boreholes W3, C1 and C2 were extremely dry, Borehole D1 was too wet and Boreholes D2 and W4 were inconclusive.

Psychrometer measurements taken in the haulage tunnel at the Magma mine and Queen Creek road tunnel east of Superior, Arizona were made in welded tuff. The welded tuff in the haulage tunnel remained very wet through the period of this study while tuff in the road tunnel was much dryer and has shown greater variation in moisture potential. Measurements made at the road tunnel are summarized in Table 3.4.

Road tunnel data for boreholes 1 and 2 indicate significant wetting (i.e. lower potentials) between May and June sampling dates. Borehole 3, which is located about 30 meters away from boreholes 1 and 2

Table 3.2 Summary of thermocouple psychrometer measurements in the Pride-of-the-West mine.

Borehole Number	Borehole Length (m)	Date of Measurement	Psychrometer Number	Borehole Temperature (°C)	Psychrometric Response (µV)	Moisture Potential (Mpa)		
1	4.4	3/22/82	M73 ^a	16.2	NR ^f			
			M152 ^a	16.3	NR			
			M73	16.3	NR			
			M152	16.1	NR			
			M73 ^b	16.3	NR			
		4/20/82	M152 ^b	16.5	NR			
3	>6.1	3/22/82	M150 ^a	16.7	13.7 ± 0.15	-4.01		
			M150	16.5	13.1 ± 0.1	-3.78		
			M150	16.5	12.1 ± 0.1	-3.49		
			M150	16.6	12.0 ± 0	-3.46		
			M150	16.6	12.0 ± 0	-3.45		
			M150	16.4	12.0 ± 0	-3.49		
			M150	16.6	12.1 ± 0.2	-3.48		
			M150	16.6	12.0 ± 0.05	-3.45		
					8/01/82			
					9/24/82 ^e			
4	4.0	5/11/82	M73 ^c	16.8	NR			
			M152 ^c	16.8	NR			
			M73	16.7	NR			
			M152	16.7	NR			
			M73	16.6	NR			
			M152	16.6	NR	64.0		
			M73 ^d	16.6	NR	67.2		
			M152 ^d	16.6	NR	64.0		
					7/12/82			

µV at
30 sec

Table 3.2 Summary of thermocouple psychrometer measurements in the Pride-of-the-West mine--continued.

Borehole Number	Borehole Length	Date of Measurement	Psychrometer Number	Borehole Temperature (°C)	Psychrometric Response (µV)	µV at 30 sec
5	>6.1m	4/20/82	M1519	16.4	NR	-
		5/11/82	M151	16.9	NR	-
		5/25/82	M151	16.6	NR	-
		6/08/82	M151	16.7	NR	86.4
		7/12/82 ^d	M151	16.9	NR	32.9
6	>6.1m	8/01/82	M73 ^h	-	NR	39.5
			M152	16.5	NR	-
		11/10/82 ^f	M73	16.5	NR	30.9
			M152	16.7	NR	28.4
			M153 ^c	16.5	NR	-
8	>6.1m	5/11/82	M153	16.6	NR	-
		5/25/82	M153	16.8	NR	66.8
		6/08/82	M153	16.7	NR	68.3
		7/12/82 ^d	M153	16.7	NR	-

- a. Psychrometers installed 3/18/82.
b. Borehole abandoned 4/20/82.
c. Psychrometers installed 4/20/82.
d. Borehole abandoned 7/12/82.
e. Packer found to be completely deflated; borehole abandoned.
f. NR indicates "no response".
g. Psychrometer installed 3/30/82.
h. Psychrometers installed 7/12/82.
i. Borehole abandoned 11/10/82.

Table 3.3 Summary of thermocouple psychrometer measurements in Santo Nino mine.

Borehole Number	Date of Measurement	Psychrometer Number	Borehole Temperature	Psychrometric Response	μV at 30 sec
D1	3/22/82	M76 ^a	10.2	NR	-
	3/30/82	M76	10.2	NR	-
	4/20/82	M76	10.1	NR	-
	5/11/82	M76	10.6	NR	-
	5/25/82	M76	10.6	NR	-
	6/08/82	M76	11.0	NR	49.0
	7/12/82	M76	11.0	NR	51.4
	7/27/82	M76	10.9	NR	40.4
	8/03/82	M5 ^b	11.2	NR	51.0
		M76	11.1	NR	37.6
	8/13/82	M5	11.4	NR	46.0
		M76	11.2	NR	35.9
	9/17/82	M5	11.4	NR	38.7
		M76	11.4	NR	34.8
	10/20/82	M5	11.3	NR	32.5
		M76	11.1	NR	33.6
D2	4/20/82	M210 ^c	11.0	NR	-
	5/11/82	M210	11.0	NR	-
	5/25/82	M210	11.2	NR	-
	6/08/82	M210	11.3	NR	46.8
	7/12/82	M210	11.3	NR	-
	7/27/82	M210	10.9	NR	56.5
	8/03/82	M20	11.5	NR	54.3
	8/13/82	M20	11.1	NR	56.8
		M210	11.4	NR	55.6
	9/17/82	M20	11.2	NR	55.8
		M210	11.4	NR	48.2
	10/20/82	M20	11.3	NR	56.3
		M210	11.6	NR	43.9

Table 3.3 Summary of thermocouple psychrometer measurements in Santo Nino mine--continued

Borehole Number	Date of Measurement	Psychrometer Number	Borehole Temperature °C	Psychrometric Response μV	μV at 30 sec
W1	3/22/82	M209e	9.5	NR	-
	3/30/82	M209f	9.6	NR	-
W3	5/25/82	M208g	9.9	NR	-
	6/8/82	M208	-	NR	49.5
	7/12/82	M208	10.8	NR	64.9
	7/27/82	M208	10.8	NR	63.2
		M6h	-	NR	59.5
	8/15/82	M208	11.1	NR	66.0
		M6	10.9	NR	55.0
	9/17/82	M208	11.3	NR	63.2
		M6	10.8	NR	59.2
	10/20/82	M208	10.9	NR	51.7
	M6	11.1	NR	55.6	
W4	4/20/82	M210 ⁱ	10.1	NR	-
	5/11/82	M210	10.5	NR	-
	5/25/82	M210	10.4	NR	-
	6/8/82	M210	10.4	NR	52.8
	7/12/82	M210	10.6	NR	46.0
	7/27/82	M210	10.9	NR	63.5
		M2J	11.4	NR	44.7
	8/15/82	M210	11.0	NR	57.6
		M2	11.0	NR	45.0
	9/17/82 ^k				

Table 3.3 Summary of thermocouple psychrometer measurements in Santo Nino mine--continued

Borehole Number	Date of Measurement	Psychrometer Number	Borehole Temperature °C	Psychrometric Response μV	μV at 30 sec
C1	7/27/82	M8 ¹	10.6	NR	73.6
		W6 ¹	10.6	NR	41.7
	8/13/82	M8	11.5	NR	71.0
		W6	12.0	NR	-
	9/17/82	M8	11.1	NR	76.8
		W6	10.9	NR	41.2
	10/20/82	M8	10.5	NR	67.6
		W6	10.3	NR	38.1
C2	7/27/82	M2 ¹	10.7	NR	61.9
		M3 ¹	10.7	NR	69.2
	8/13/82	M2	11.0	NR	-
		M3	11.1	NR	-
	9/17/82	M2	10.1	NR	58.6
		M3	10.5	NR	61.6
	10/20/82	M2	10.5	NR	48.0
		M3	10.9	NR	58.1

note:

- a. Psychrometer installed 3/18/82.
- b. Psychrometer installed 7/27/82.
- c. Psychrometer installed 3/30/82.
- d. Psychrometer installed 7/27/82.
- e. Psychrometer installed 3/18/82.
- f. Psychrometer removed 3/30/82.
- g. Psychrometer installed 5/11/82.
- h. Psychrometer installed 7/12/82.
- i. Psychrometer installed 3/18/82.
- j. Psychrometer installed 7/12/82.
- k. Packer had deflated; apparatus removed.
- l. Psychrometers installed 7/12/82.

Table 3.4 Moisture potential as measured using thermocouple psychrometers in the dew point and psychrometric modes in welded tuff at Superior Road tunnel.

BOREHOLE	PSYCHROMETER	METHOD	1/23/84	3/1/84	3/2/84	3/14/84	4/5/84	5/10/84	6/6/84	6/14/84
#1	M26	DP	-	-	21.5	20.3	19.8	27.5	15.3	15.8
		PS	33.5	29.3	30.0	28.6	27.3	28.0	16.5	17.4
	M27	DP	-	-	21.8	20.9	20.0	24.0	15.5	17.0
		PS	37.5	29.8	29.8	28.5	27.1	29.1	16.8	20.8
#2	M28	DP	-	-	22.9	21.3	24.2	* 21.0	13.6	-
		PS	-	35.5	34.6	32.7	32.4	24.4	12.7	-
	M29	DP	-	-	16.4	18.7	-	* 21.9	20.0	-
		PS	-	25.0	24.5	25.1	-	26.3	22.8	-
#3	M23	DP	-	-	-	<A	<A	4.5	5.7	4.5
		PS	-	<A	-	<A	<A	6.0	8.2	6.8
	M24	DP	-	-	-	2.8	1.5	2.8	5.1	3.8
		PS	-	<A	-	3.5	<A	3.7	5.4	3.5

*Psychrometers M28 and M29 were replaced with psychrometers M22 and M30 following the 4/8/84 readings.

has consistently been much wetter than the other holes and shows a slight drying trend throughout the measurement period, with the possible exception of the last date.

Each borehole in the Queen Creek road tunnel contains two psychrometers located adjacent to one another. Comparison of results obtained from corresponding pairs of psychrometers within the same borehole indicates different measured potentials. These discrepancies show the extreme variability of the psychrometer measurements and cast doubt on the precise magnitude of water potentials capable of being measured using thermocouple psychrometers. However, the results presented in Table 3.4 do suggest that temporal trends in water potential can be followed using this method even if the absolute values are subject to uncertainty.

3.4 Absorber Method

The absorber method is based on the principle that a moist soil or rock in contact with a filter paper will exchange water until potential equilibrium is achieved. At equilibrium, the water potential of the rock and the filter papers will be the same. Previous authors (Gardner, 1937; Fawcett and Collis-George, 1967; McQueen and Miller, 1968; Hamblin, 1981) have discussed the water potential relationship of filter papers equilibrated with different soil types of variable moisture contents as well as defining a MRC for various brands of filter papers. A significant portion of this study was devoted to the evaluation of the water potential relationship of filter papers equilibrated with unsaturated rock samples in the laboratory and in situ.

3.4.1 Moisture Release Curve

The initial procedure used to evaluate the water potential relationship of filter paper was to describe the MRC of a particular brand of filter paper. Two methods were used to evaluate the water potential of the filter papers; Method I used the thermocouple psychrometer SC-10A (described in section 2.3) to measure the water potential of filter papers, and Method II involved the equilibration of filter papers on ceramic pressure plate extractors.

Method I. This method involved placing two Whatman No. 42 filter papers around the inner circumference of each sample cup of the SC-10A. The filter papers were wetted such that no excess water remained at the bottom of the sample cup. The sample cups were then sealed with rubber stoppers to limit evaporation losses and weighed on a scale accurate to 10^{-4} grams. The stoppers were removed and the sample cups placed in the psychrometer SC-10A for vapor equilibration and water potential measurement. After all the water potential measurements were made, the sample cups were removed and placed in an oven to slightly dry the filter papers (2 to 5 minutes at 105°C). The samples were then allowed to cool and the process repeated until the filter papers were completely dry. In this manner, the water potential of the filter paper measured with the thermocouple psychrometer SC-10A, and water content (percent of dry weight) determined gravimetrically, defined the MRC for the filter papers for Method I.

Method II. This method utilized two types of pressure plate extractors typically used in soil moisture studies; the 15-bar ceramic plate extractor and the Tempe pressure cell, both manufactured by

Soilmoisture Corporation, Santa Barbara, California. Pressure plate extractors are limited to a range of 0 to -15 bars while the Tempe pressure cells, although similar to the 15-bar extractor, are smaller and limited to 0 to 1 bar of pressure. The Tempe cell is used to accurately equilibrate samples in the high water potential range (near saturation).

A saturated ceramic plate was placed inside the 15-bar extractor vessel with saturated samples of soil, rock or filter papers set on the plate. The vessel was then sealed and a pressure applied using a nitrogen tank. When water no longer passed through the pressure plate and out of the pressure vessel, the samples were assumed to be at water potential equilibrium with the applied pressure. At this point, it was assumed that the samples had the same value for matric potential as the applied pressure but opposite in sign. A similar procedure was used for the Tempe pressure cell.

Method II involved the equilibration of Whatman No. 42 filter papers on either the 15-bar pressure plate or the Tempe cell. After the filter papers had equilibrated, the pressure was released and the filter papers were quickly transferred to previously weighed bottles, which were then sealed and weighed. After weighing, the filter papers were placed in an oven at 105°C for approximately 24 hours and the dry weight was determined. To define the MRC with Method II, the water potential measurements were taken from the amount of pressure applied within the extractor vessel and the water content (percent of dry weight) was measured gravimetrically.

The resulting MRC for filter papers using Methods I and II are

presented and compared with MRC's developed by previous authors in Figure 3.19. The curve produced by Method I diverges from Method II at a water potential measurement of approximately -2.0 bars. This divergence is due to the inability of the thermocouple psychrometer to accurately measure high water potentials (near saturation). The pressure extractor, particularly the Tempe cell, is a more reliable method of determining water potentials near saturation (Rahi, 1986).

The combination MRC described by Method I and II is similar to the MRC developed by Hamblin (1981) and Fawcett and Collis-George (1967) but is shifted slightly to the right. The shift may be due to differences in individual filter papers although, experiments done by Hamblin in 1981 showed that Whatman No. 42 filter papers had the same characteristics over a two year study. Variations in experimental procedures could also create this shift.

The calibration methods used by Hamblin (1981) are very similar to those used in Method II. However, the procedure he used to determine the extreme high and low water potentials differ from the methods used during this research. Hamblin calibrated Whatman No. 42 filter papers against a suction plate up to -0.07 bars, pressure plate to -0.7 bars, pressure membrane plate to -15 bars, and exposed the filter papers to a saturated salt solution at 20°C equivalent to -55.0 bars.

McQueen and Miller calibrated Schleicher and Schuell No. 589 filter papers by exposing the filter papers to saturated salt solutions in closed, isothermal containers for water potentials less than -15 bars. For water potentials from -0.1 to -15 bars the filter papers were placed in contact with soils previously equilibrated on a pressure

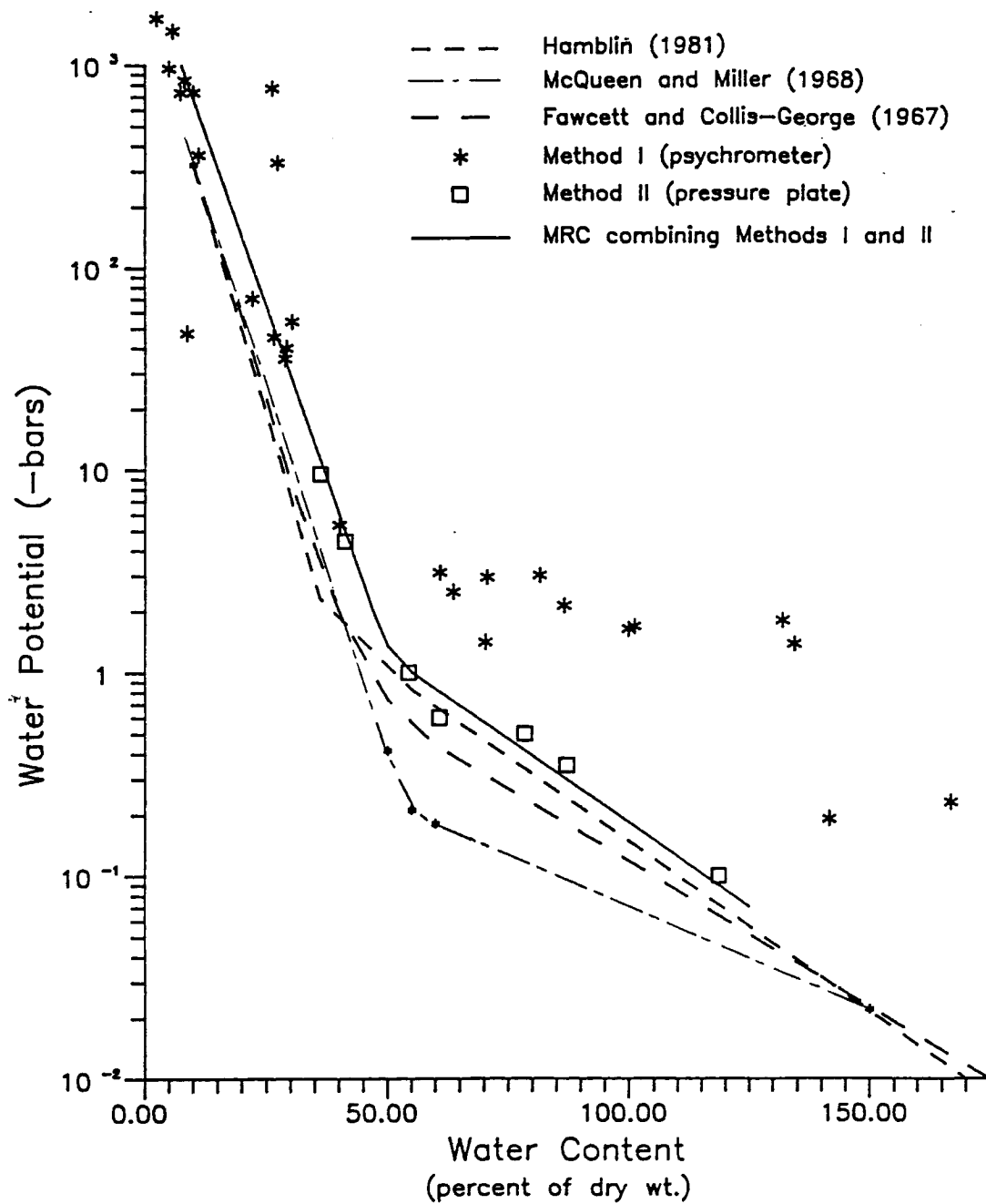


Figure 3.19. Filter paper moisture release curves described by three authors and a curve developed during this study.

membrane extractor. Calibration for water potentials greater than -0.2 bars were determined from field samples obtained at known heights above the water table. The McQueen and Miller MRC is very similar to the other MRC's in Figure 3.19 for water potentials below -2.0 bars. The divergence of the McQueen and Miller MRC above -2.0 bars may be attributed to the different water retention characteristics of Schleicher and Schuell filter papers compared with Whatman No. 42 filter papers. Filter papers were calibrated at high water potentials by placing the filter papers in contact with equilibrated soil. This method is much different than the methods used in this study where the filter papers were equilibrated directly on the pressure plate.

The methods used for determining a MRC during this study were most similar to the methods used by Hamblin; water potentials of filter papers were determined directly (on pressure plates or with psychrometers) with no soil contact. By comparing the MRC defined by this study and the MRC described by Hamblin, it appears that greatest errors occur along the steepest slope of the MRC (water potentials less than -0.6 bars). For example, if the filter paper water content is 20 percent of dry weight the water potential ranges from -50 to -150 bars depending on which MRC one refers. The errors are much less at higher water contents where a filter paper at 50 percent of dry weight gives a range of water potentials from -1.0 to -1.5 bars.

The MRC defined by Hamblin and the MRC developed using Methods I and II (shown in Figure 3.19) are referred to in the remaining sections of this chapter. These sections evaluate the relationships of the MRC of filter papers and the water potential of the filter papers when

equilibrated with soil and rock water potentials. The filter paper MRC is also used to verify measurements of laboratory and field water potential and water content.

3.4.2. Comparison of Water Potential Measurements

The water potential of filter papers and soils measured with the thermocouple psychrometer SC-10A was compared with water potentials of filter papers and soils created by the pressure plate extractors.

The pressure plate extractor method is widely used to define the MRC for soils (Richards, 1965). At equilibrium, the pressure applied within the extractor vessel is equivalent, but opposite in sign, to the matric potential of the equilibrated sample. As an initial experiment, some of the Schleicher and Schuell filter papers were covered by a buffer of filter paper wrapped around the filter paper strips with no soil contact. Other Schleicher and Schuell filter papers were placed below, in the middle and on top of columns of silty loam soil. All of the samples were saturated and equilibrated on the porous plate at 2.9 bars of pressure for approximately 24 hours. The pressure was released and the filter papers were quickly placed in the sample cups of the psychrometer SC-10A to measure the water potential of the filter papers. The values of water potential determined by the SC-10A are presented in Table 3.5.

The water potential of the filter papers that most closely match the applied pressure were filter papers enclosed within larger filter papers which act as a buffer. The filter papers in contact with the silty loam have a lower potential (drier) than the potential predicted by the amount of applied pressure.

Table 3.5. Water potential of Schleicher and Schuell filter papers determined with the thermocouple psychrometer SC-10A after equilibration on a pressure extractor at 2.9 bars for 24 hours.

	<u>Description</u>	<u>Water Potential</u> (applied 2.9 bars for 24 hours)
1	Single filter papers on top of 1" column of silty loam.	-4.77
2	Single filter paper on top of 1/2" column of silty loam.	-5.26

3	Single filter paper in middle of column of silty loam soil.	-5.60
4	(same)	-6.65

5	Single filter paper below the soil column.	-4.92
6	(same)	-4.03

7	Filter paper enclosed with in larger filter paper (no contact with soil).	-2.39
8	(same)	-2.57
9	(same)	-1.25

Because of the variance of filter paper water potentials in contact with soils, the following experiment involved the use solely of filter paper buffers. In some cases, filter paper strips were doubled or tripled together within the sample cups of the SC-10A to increase the available water within the cup and expediate equilibration time. Figure 3.20 shows ten separate experiments involving single and double Schleicher and Schuell filter paper strips. The filter papers were covered by a filter paper buffer, saturated, and placed directly on a pressure plate for equilibration at pressures varying from 1 to 5 bars for durations from 24 to 90 hours. Following equilibration, the filter papers were quickly placed in the psychrometer sample cups and the water potential determined with the thermocouple psychrometer SC-10A. Experiments 1-5 involved only single filter paper strips in the sample cups and showed great variation from the applied pressure. Experiments 2, 3 and 5 gave higher water potential readings (wetter) than predicted from the applied pressure, whereas in experiments 1 and 4, the filter papers were predominantly of lower water potential. The variations of experiments 1 and 4 may be due to evaporation losses when transferring filter papers from the pressure extractor to the SC-10A. Other sources of error in experiments 1 to 5 may be caused by inadequate equilibration of filter papers either on the pressure plate or within the SC-10A.

When the filter papers were doubled together, as in experiments 6 to 10, the scatter of the data decreased, but continued to vary from the applied pressure. To test the ability of the SC-10A to measure very high water potentials (near saturation), experiments 6 to 10 involved very small amounts of applied pressure. A silty loam soil was equilibrated

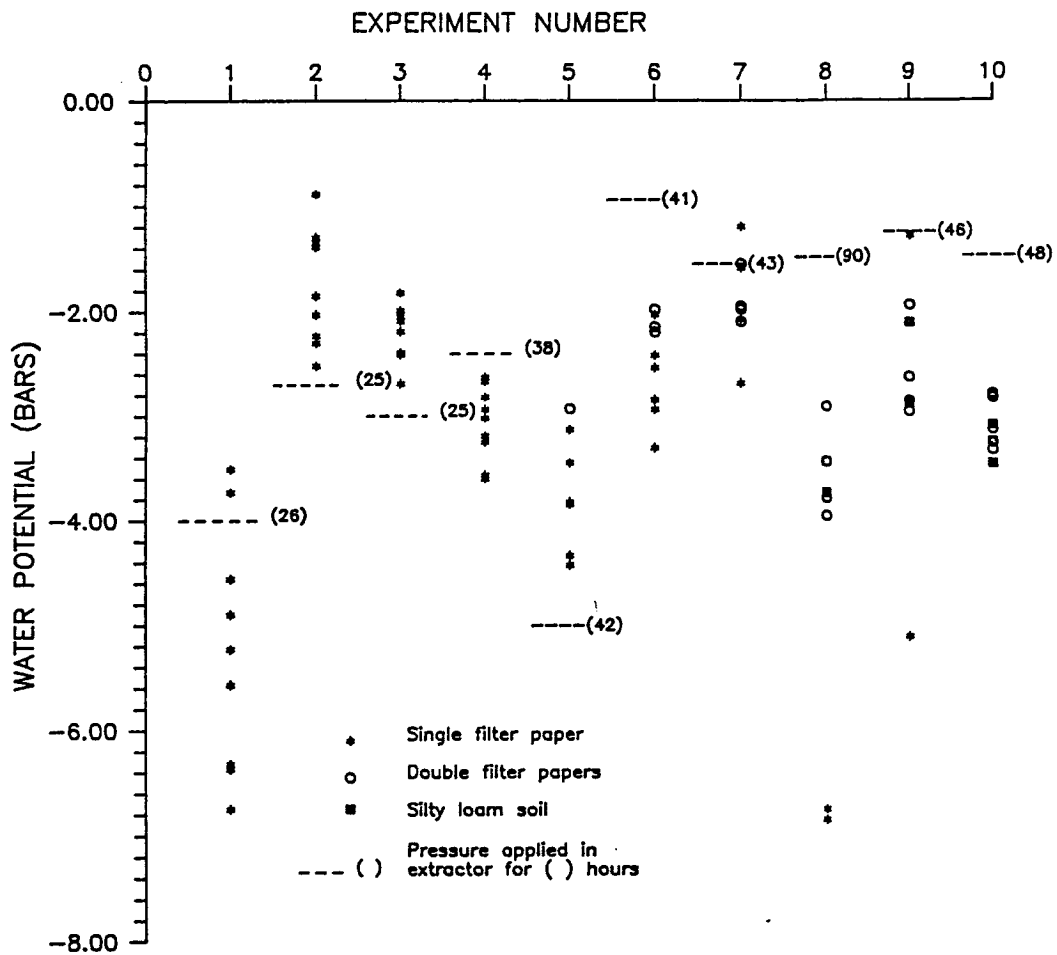


Figure 3.20. Ten experiments involving the equilibration of filter papers on a pressure plate followed by filter paper water potential measurements made with the thermocouple psychrometer SC-10A.

separately but on the same pressure plate as single and double filter papers in experiments 8 to 10. A low pressure (1.3 to 1.5 bars) was applied for various lengths of time. Soil samples and filter papers were placed in separate sample chambers of the SC-10A and their water potentials determined independently. Although the results of experiments 8 to 10 gave lower water potential values than the applied pressure, the water potential of the doubled filter papers and the silty loam were very similar. Again, the divergence from the applied pressure may be due to the inability of the SC-10A to measure water potentials near saturation. Additionally, if the relative humidity in the extractor vessel was not near 100 percent, then the filter papers and soil may have lost water to vapor as they equilibrated causing the water potential values to be lower than predicted by the applied pressure. In the remaining experiments, wet paper towels were placed along the inner walls of the pressure vessel to increase the relative humidity within the vessel. The applied pressure differed significantly from the filter paper and soil water potential measurements. However, the filter paper and soil water potential measurements were similar and suggest that the the water potentials of the two mediums had equilibrated.

Experiments 11, 12 and 13, of Figure 3.21, involved the same procedure as experiments 1 to 10, however silica sand and distilled water were used to see if there is a significant osmotic potential causing errors in previous experiments where a silty loam was used. The water potentials of the triple filter papers covered with a filter paper buffer and silica sand in experiment 12 (-1.5 to -2.7 bars) were very similar to the applied pressure (2.31 bars). However, in experiment 13 the filter

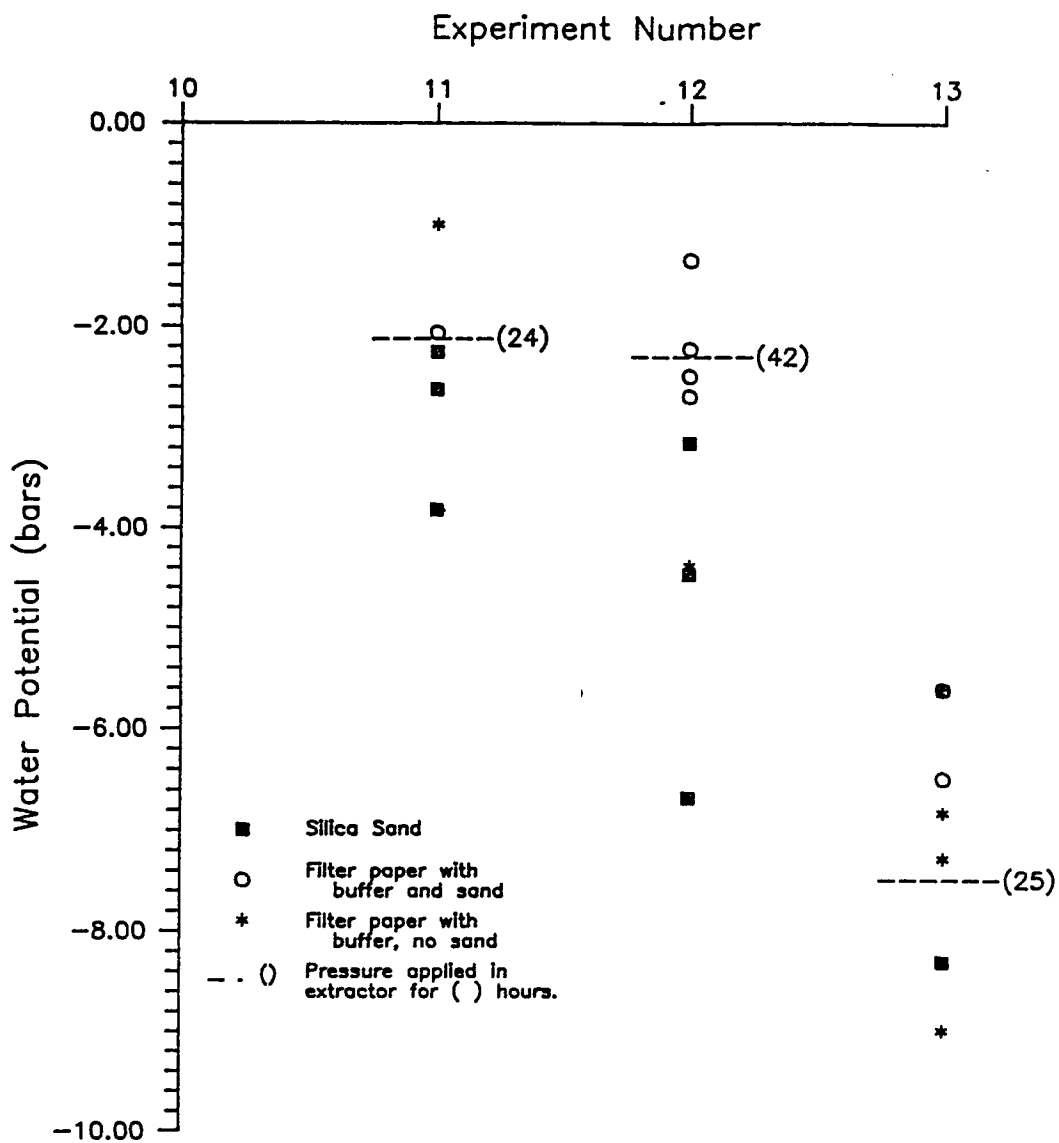


Figure 3.21. Three experiments involving the equilibration of filter papers and soil on a pressure plate. Water potentials of the filter papers were determined with the SC-10A.

papers without contact with the silica sand are also similar to the applied pressure. Whether or not filter papers came in contact with silica sand did not appear to affect the water potential of the filter papers.

A large scatter of the water potential of silica sand was observed in all three experiments. The variance of silica sand water potentials makes it difficult to make any conclusions concerning errors created by osmotic potentials. However, the water potential of the silica sand was generally less than the applied pressure. The low water potentials of the silica sand may be due to the disturbance of the sand while transferring from the pressure plate to the sample chambers of the SC-10A. Water held loosely between the sand particles evaporated, thereby lowering the soil water potential. Thus, the transfer and disturbance of the sample had a much more significant effect on the water potential of the sand than it did on the silty loam.

3.4.3. Comparison of Brands of Filter Paper

Two brands of filter papers were used by previous authors: Schleicher and Schuell No. 589 white ribbon used by McQueen and Miller (1968), and Al-Khafaf and Hanks (1974); and Whatman No.42 used by Fawcett and Collis-George (1967), and Hamblin (1981). Experiments were performed with these two filter papers as well as Millipore filter papers to see if there was a significant difference between brands of filter papers in their ability to achieve water potential equilibrium with a pressure plate, soil and rock.

Double and triple strips of filter papers, set on a ceramic plate and covered by a filter paper buffer, were saturated, placed in a

pressure vessel and a pressure applied. After equilibration, the filter papers were quickly placed in the chambers of the SC-10A. The results are presented in Figure 3.22.

The Millipore papers in the four experiments have higher water potentials than the other filter papers and higher than the applied pressure. Filter papers are classified by the size of the particles retained by the filter paper. The Millipore papers have a retention capacity of 0.45 microns. Whatman No. 42 papers retain particles larger than 5 microns and Schleicher and Schuell No. 589 filter papers have a retention capacity of 8 microns.

Assuming the retention capacity of a filter paper represents the papers approximate pore size, the capillary rise equation can be used to determine the pressure required to drain the pores. The capillary rise equation is as follows:

$$h = \frac{2 \gamma \cos \theta}{\rho g r} \quad (2)$$

where h is the height of rise; γ is the surface tension; θ is the angle of contact; ρ is the density of water; g is the acceleration due to gravity; and r is the radius of the pore. The pressure required to drain the pore is determined by:

$$P = h g \rho \quad (3)$$

For the three brands of filter papers, the following pressures are required to drain the pores of the filter papers: 6.4 bars for Millipore papers; 0.58 bars for Whatman No. 42 papers; 0.36 bars for Schleicher and Schuell papers. The 3-bar ceramic plate, with a pore size

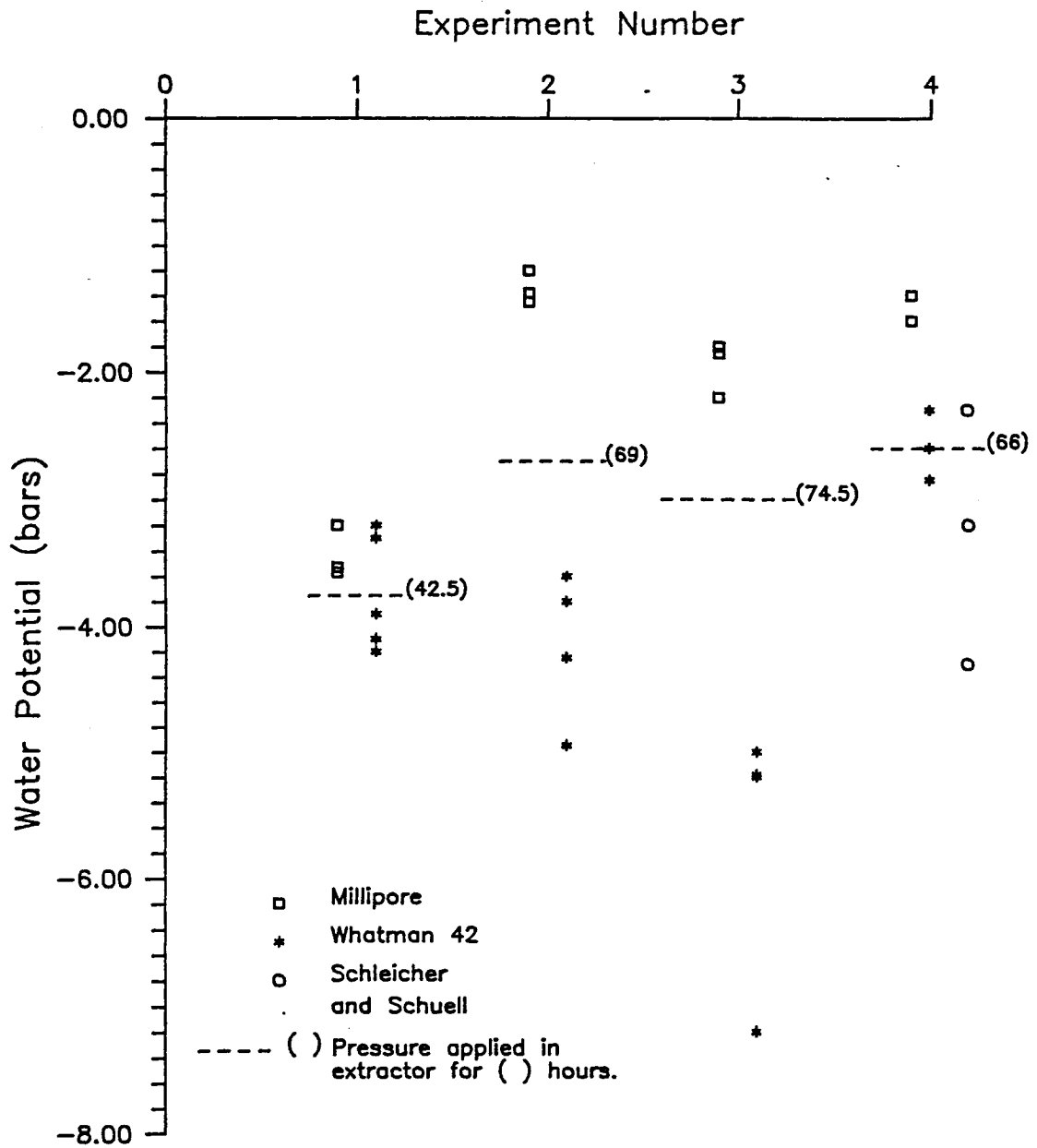


Figure 3.22. Four experiments comparing the water potential of 3 types of filter papers equilibrated on a pressure plate and measured with the SC-10A.

of 0.8 microns, remains saturated when pressures less than 3.6 bars are applied. Thus, it is reasonable that Millipore filter papers exhibit the highest water potentials because pressures greater than those applied during this experiment were necessary to drain water from the small pores of the filter paper.

The Whatman filter papers generally have lower water potential values than the applied pressure. The variance of water potential values could be attributed to handling and evaporation; evaporation may be more significant with the Whatman and Schleicher and Schuell filter papers, with their larger pores, than the Millipore filter papers. The large variance in water potentials of the Schleicher and Schuell filter papers is exhibited in the numerous experiments involving this brand of filter paper (see Figure 3.20). Due to the variance of all the filter paper brands, it is difficult to conclude which brand of filter paper is superior for this research. The following experiments involve the use of various filter papers for different types of experiments. The results of each experiment are then evaluated separately and compared.

3.4.4. Filter Papers Equilibrated with Soil

Al-Khafaf and Hanks (1974) described a method where both matric and osmotic potentials could be determined for a soil sample using filter papers. A filter paper in intimate contact with the soil measures only the matric potential. A filter paper in a closed chamber with the soil but not in contact with the soil measures the sum of the matric and osmotic potential because the vapor pressure in the soil air is a function of matric and osmotic potentials. The following experiments are very similar to the experiments described by Al-Khafaf and Hanks.

After a saturated porous plate was set in the pressure vessel, about a 100 grams of a sandy loam was placed on the plate and saturated. A constant pressure was applied until equilibrium was achieved, and the soil was then carefully removed and placed in soil sample cans. Schleicher and Schuell No. 589 white ribbon filter papers previously treated with actidione (an antibacterial reagent) and air dried, were placed below the soil with a paper towel above the filter paper to keep it free of soil. Two more filter papers were placed on a small rubber cylinder above the soil (Figure 3.23). The sample cans were sealed and placed in a constant temperature room for equilibration. After an equilibration time greater than 8 days, the filter papers, as well as some soil, were removed and placed separately in the sample cups of the SC-10A for water potential measurement. The results are presented in Figure 3.24.

Al-Khafaf and Hanks (1974) developed a MRC which combined the water content of the equilibrated filter papers, determined gravimetrically (percent of dry weight), and the water potential of the corresponding soil, measured with a sample chamber thermocouple psychrometer or pressure extractor. The experiments conducted in this research differed from Al-Khafaf and Hanks in that the filter papers, after equilibrating with soil, were placed directly in the thermocouple psychrometer SC-10A for water potential measurement. A small amount of the equilibrated soil was also placed in separate sample cups of the SC-10A and a comparison was made of water potentials of the soils and filter papers of various types of contact.

The results of experiments 1 to 3 (Figure 3.24) show water

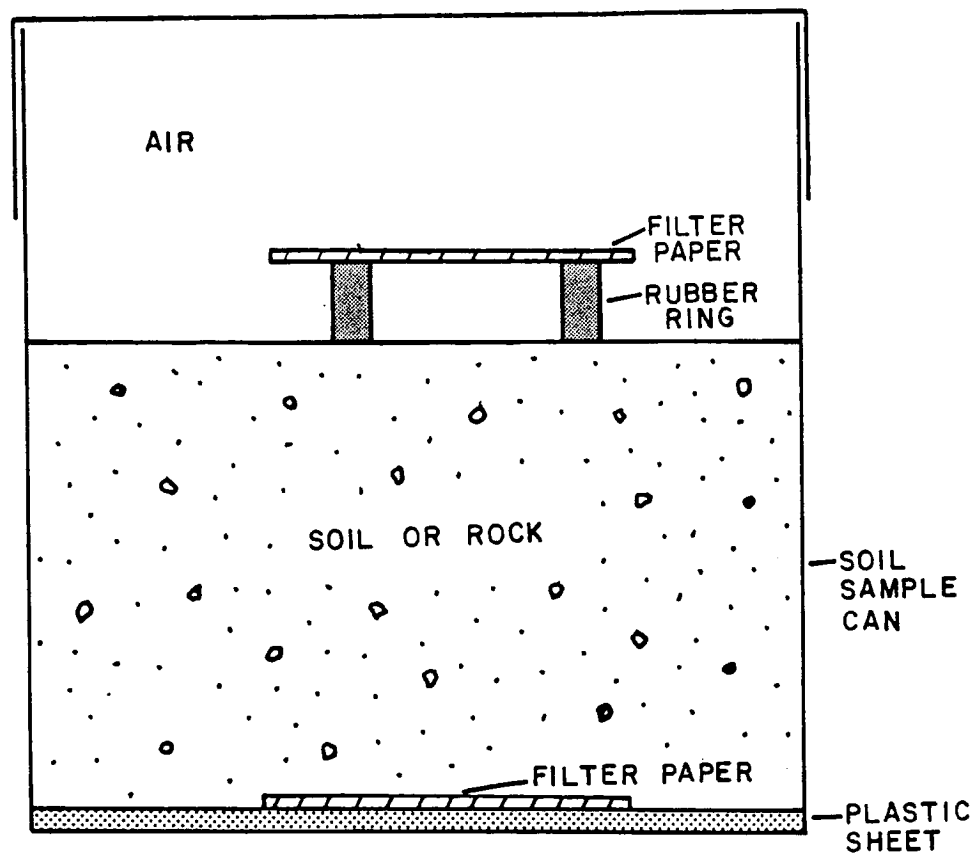


Figure 3.23. Cross sectional diagram of experimental set up used to measure matric and osmotic potentials of soil or rock depending on the type of contact.

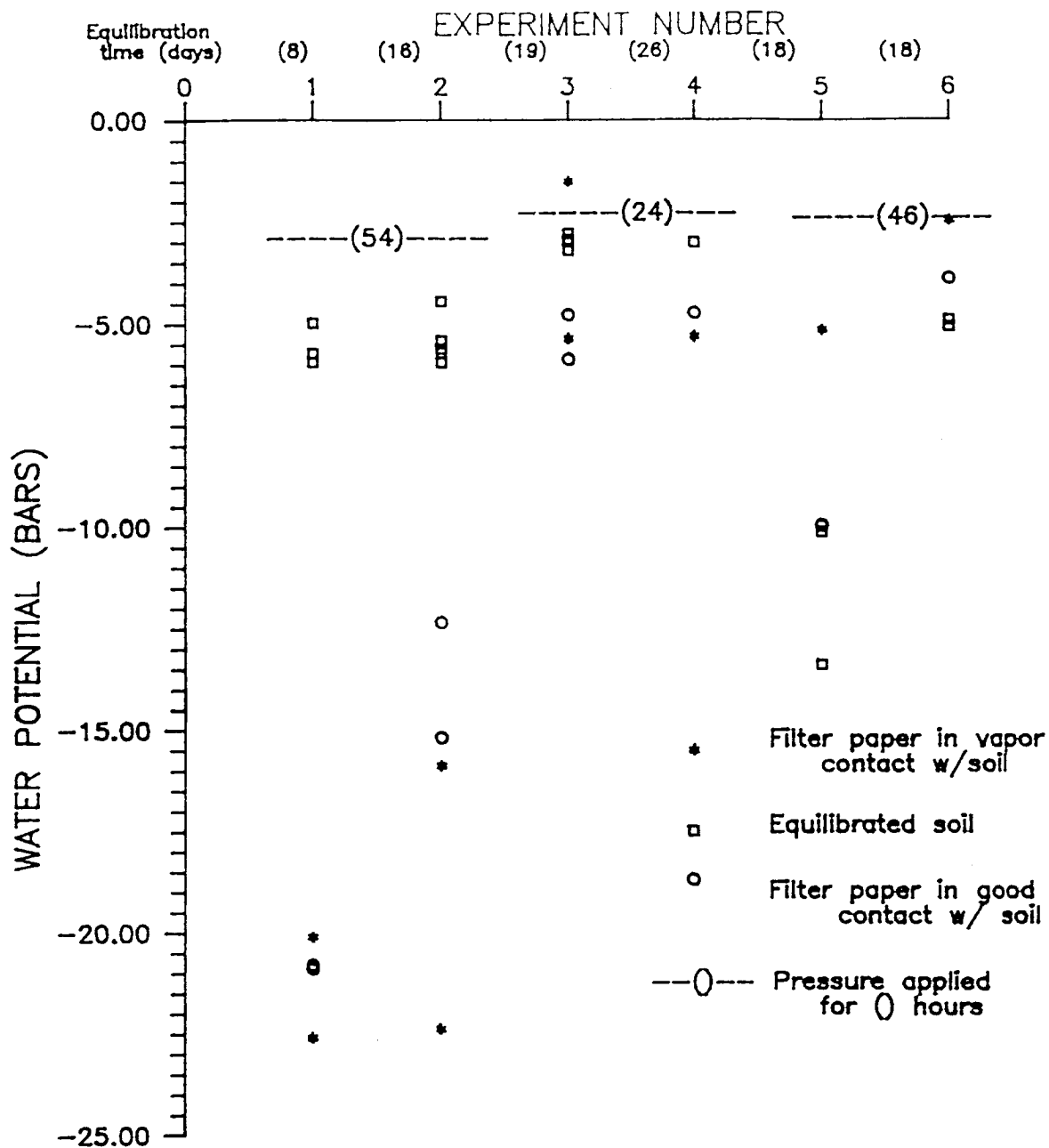


Figure 3.24. Results from experiment involving the equilibration of soil on a pressure plate followed by equilibration of filter papers placed in direct and vapor contact with soil.

potentials of soil and filter papers to be consistently lower than the applied pressure. The water potential of the soil samples are generally closer to the water potential predicted by the applied pressure. Experiments 2 and 3 involved a silty loam subjected to the same amount of pressure, however experiment 2 equilibrated with the filter papers for 8 days and experiment 3 equilibrated with filter papers for 16 days. The water potential of the soils after 8 and 16 days of equilibration remained relatively constant (-4.45 to -5.9 bars). However, the water potential of the filter papers equilibrated for 16 days was higher (closest to the water potential of the soil) than the filter papers equilibrated for 8 days. Al-Khafaf and Hanks (1974) equilibrated the filter papers with the soil for only 2 days, however these experiments show that even after 16 days of equilibration the filter papers do not have the same water potential as the soil. Al-Khafaf and Hanks did not take filter paper water potential measurements with a thermocouple psychrometer but assumed that the filter paper water potential had equilibrated with the soil water potential. Hence, the findings of this experiment do not agree with the procedural assumptions used by Al-Khafaf and Hanks to define their MRC.

The filter papers used in experiment 3 were not treated with actidione to inhibit bacterial degradation and all 4 filter papers showed evidence of mold. It is unclear how the mold affects the water potential readings. Bacterial degradation and mold would presumably affect the weight of the filter paper causing an error in the calculation of water content. Mold and decay of a filter paper in good contact with soil as well as mold on the soil were also evident in experiment 4.

The filter paper water potentials of experiments 4 and 5 resembled the soil water potentials very closely; the water potential of the clay loam soil ranged from -2.8 to -3.4 bars and the water potential of the filter papers ranged from -4.73 to -5.88 bars. The water potential of the soil was also similar to the water potential predicted by the applied pressure (-2.05). The filter paper in vapor contact which exhibited a high water potential of -1.5 bars, may be in error due to water condensing on and falling from the roof of the sample can onto the filter paper. For the clay loam used in experiments 2 to 5, the time required for water potential equilibration of the filter papers with soil appears to be approximately 20 days. The concentration of actidione was ineffective in preventing bacterial growth and mold on the filter papers when the equilibration time was at least 19 days.

Experiments 6 and 7 were performed with clean silica sand and distilled water in order to decrease mold and bacterial growth as well as osmotic effects. The soil sample can in experiment 6 was transported from one lab to another lab 10 miles away thereby disturbing the sand and lowering the soil water potential. McQueen and Miller (1968) noted that a transported sample with a low stress (high water potential) may change its stress value without a change in moisture content by simply rearranging its pore sizes and shapes. Experiments 6 and 7 were the only experiments where the water potential of the filter papers were higher than the water potential of the soil. This result may be attributed to the disturbance of the sample in experiment 6 or due to the matric potential gradient between the coarse silica sand and the fine pores of the filter paper causing the water to migrate from the sand to the filter paper.

The results of filter papers in vapor contact, measuring matric and osmotic potentials, were inconclusive. The water potentials of these filter papers were in error due to condensation on sample can lids which dropped onto the filter papers.

3.4.5 Filter Papers Equilibrated with Rock

Cores of Coconino Sandstone and nonwelded white unit of the Apache Leap tuff with diameters of 5 cm and 6.3 cm were cut into slabs of 0.6 to 2.3 cm in thickness. The slabs were initially placed in a vacuum dessicator to remove as much air as possible from the pores of the rock cores. Water was slowly introduced into the evacuated dessicator until the slabs were immersed in water. After a vacuum was maintained for at least two hours it was assumed that the sample was essentially saturated. The slabs were prepared for equilibration on the pressure plate by putting tissue paper and a thin layer of wet bentonite clay on the contact surface of the core and ceramic plate. The clay provided the hydraulic contact with the ceramic plate and the tissue paper was used to keep the rock slabs clean of bentonite clay. The rock samples were placed on the saturated pressure plate and sealed within the pressure vessel and a pressure applied. The interior circumference of the vessel was lined with wet paper towels to create a humid environment. After an equilibrium time of 66 to 93 hours, the pressure was released and the slabs were placed in sample cans with filter papers set in between the slabs. The sample cans were closed, sealed and placed in a constant temperature room for equilibration (22 to 36 days). The filter papers were removed and placed in the thermocouple psychrometer SC-10A for water potential measurement.

Sandstone. The results of the experiments involving the equilibration of sandstone slabs with various brands of filter papers are presented in Figure 3.25. The equilibrated Whatman No. 42 filter papers of experiment 1 had water potential values of -0.9 to -1.5 bars and the rocks surrounding the filter papers were equilibrated at 2.7 bars of pressure. The assumption was made that the equilibrated rock has a water potential of -2.7 bars; the actual water potential of the sandstone was not measured. Therefore, the error may be the result of the rock not reaching equilibrium with the applied pressure. In addition, the reliability of the thermocouple psychrometer measurements at high water potentials is questionable.

Experiment 2 utilized sandstone slabs equilibrated for 74 hours at 3 bars of pressure. The filter papers equilibrated between the rock slabs for 27 days in a constant temperature room. The water potential readings were very scattered, with experiment 2b being farthest from the applied pressure (-420 and -650 bars). This experiment used the thicker sandstone slabs (1.6 to 1.8 cm) which may not have been initially fully saturated. To verify that the large variance was due to the rocks having a low water potential and not due to the inability of the filter papers to equilibrate with the rock water potential, the water content (percent of dry weight) of the rocks surrounding the filter papers was calculated. The weights of all the rock slabs within a sample can were measured together, thus the rock water content is an average of all the rocks in contact with the filter papers of each experiment. The weight of the rocks was measured after equilibration with the filter papers and the dry

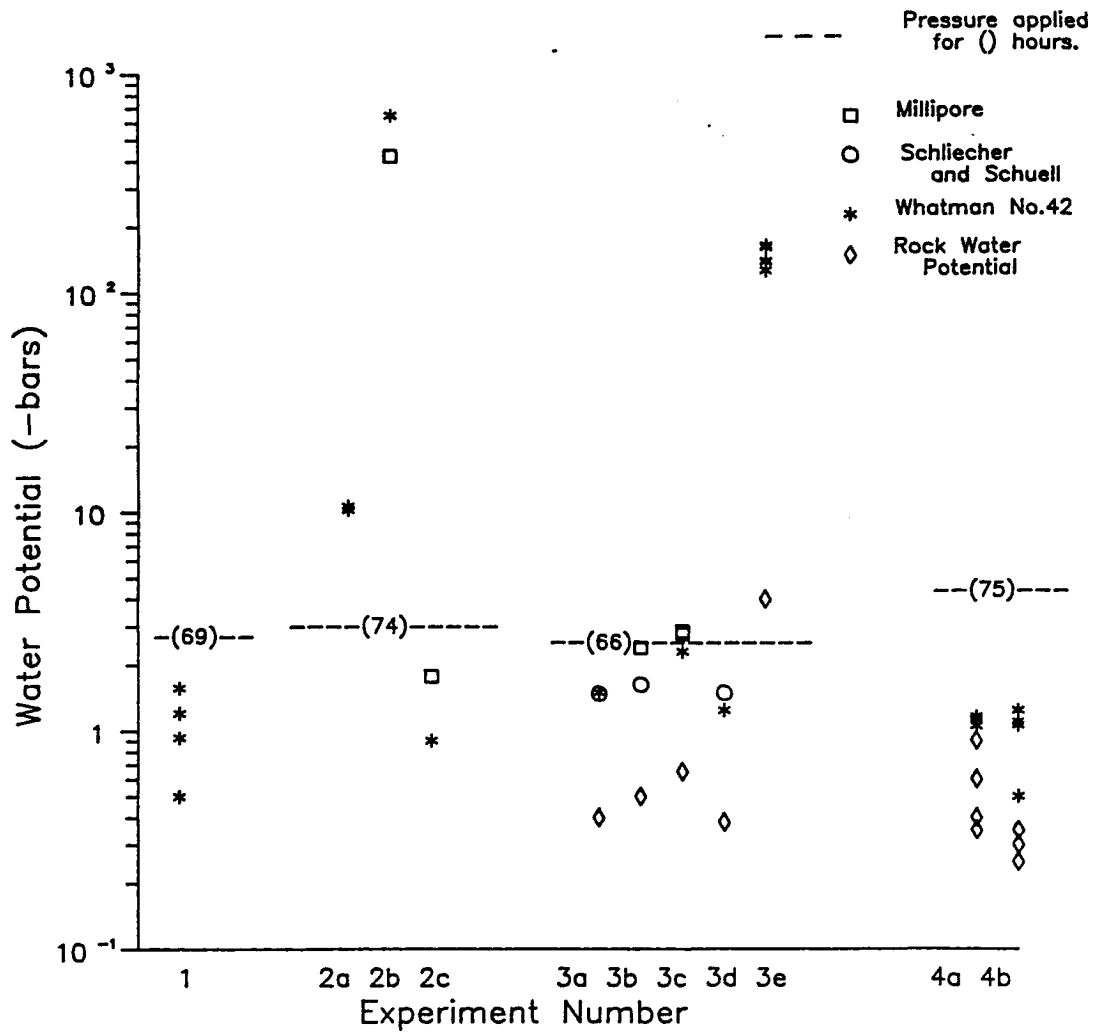


Figure 3.25. Results from experiments involving the equilibration of sandstone slabs with various brands of filter papers.

weight was measured after drying the rocks at 105°C for at least 2 days.

The average rock water content of experiment 2b was 0.12 percent of dry weight. In experiment 2c, the water potential of the filter papers was at the opposite extreme of 2b, closer to the applied pressure of 3 bars (-0.9 and -1.8 bars), and the average rock water content was 1.85 percent of dry weight. Thus, the extreme variance of the water potentials of the filter papers was not due to the inability of the filter papers to equilibrate with sandstone. The variance of the filter paper water potential may be attributed to the difficulty in saturating the thicker sandstone slabs and equilibrating the slabs by application of constant pressure on a ceramic plate in the pressure vessel.

For experiment 3, the sandstone slabs were equilibrated on the pressure plate at 2.6 bars for 66 hours and the filter papers equilibrated in between the sandstone slabs for 36 days. The results for 3a to 3d were encouraging; the water potentials of the various brands of filter papers are very similar to the applied pressure, ranging from -1.2 to -2.9 bars. The Millipore filter papers are closer to the applied pressure ranging from -2.4 to -2.9 bars, while the Schleicher and Schuell filter papers have water potentials consistently near -1.5 bars for experiments 3a, 3b and 3d. Whatman No. 42 filter papers gave the greatest variation of water potential values ranging from -1.2 to -2.4 bars. The average rock water content (percent of dry weight) was calculated for the three or four sandstone slabs surrounding the filter papers. The average rock water contents ranged from 0.24 to 1.84 percent and varied correspondingly with the variation of filter paper water potentials:

<u>Exp.</u>	<u>Rock</u>	<u>Filter Paper</u>
	<u>Water Content</u> (percent of dry weight)	<u>Water potential</u> (bars)
3e	0.24	-127 to -166
3c	0.86	-2.4, -2.8
3b	1.45	-1.6, -2.4
3a	1.75	-1.5, -1.7
3d	1.84	-1.2, -1.4

As the water content of the sandstone increased, the water potential of the filter papers increased.

For experiments 3 and 4, the water content of the filter papers was also determined. The results were plotted and compared with the MRC previously described in section 3.4.1, and are shown in Figure 3.26. The data at the lower water potentials have the best fit while the data at higher water potentials are more scattered due to the uncertain ability of the SC-10A in measuring water potentials greater than -2 bars (high water potentials). The fact that the data fit the MRC shows that the filter papers continue to have the same relationship between water potential and water content even when the filter papers are equilibrated with rocks, hence confirming the validity of the results.

In experiment 4, the rock water content (by volume) was determined for each sandstone slab within a sample can. The MRC for Coconino sandstone (see Figure 3.27), developed by Rahi (1986), was used to estimate the water potentials of the sandstone slabs. The sandstone slabs were left on the pressure plate at 4.5 bars of pressure for 75 hours.

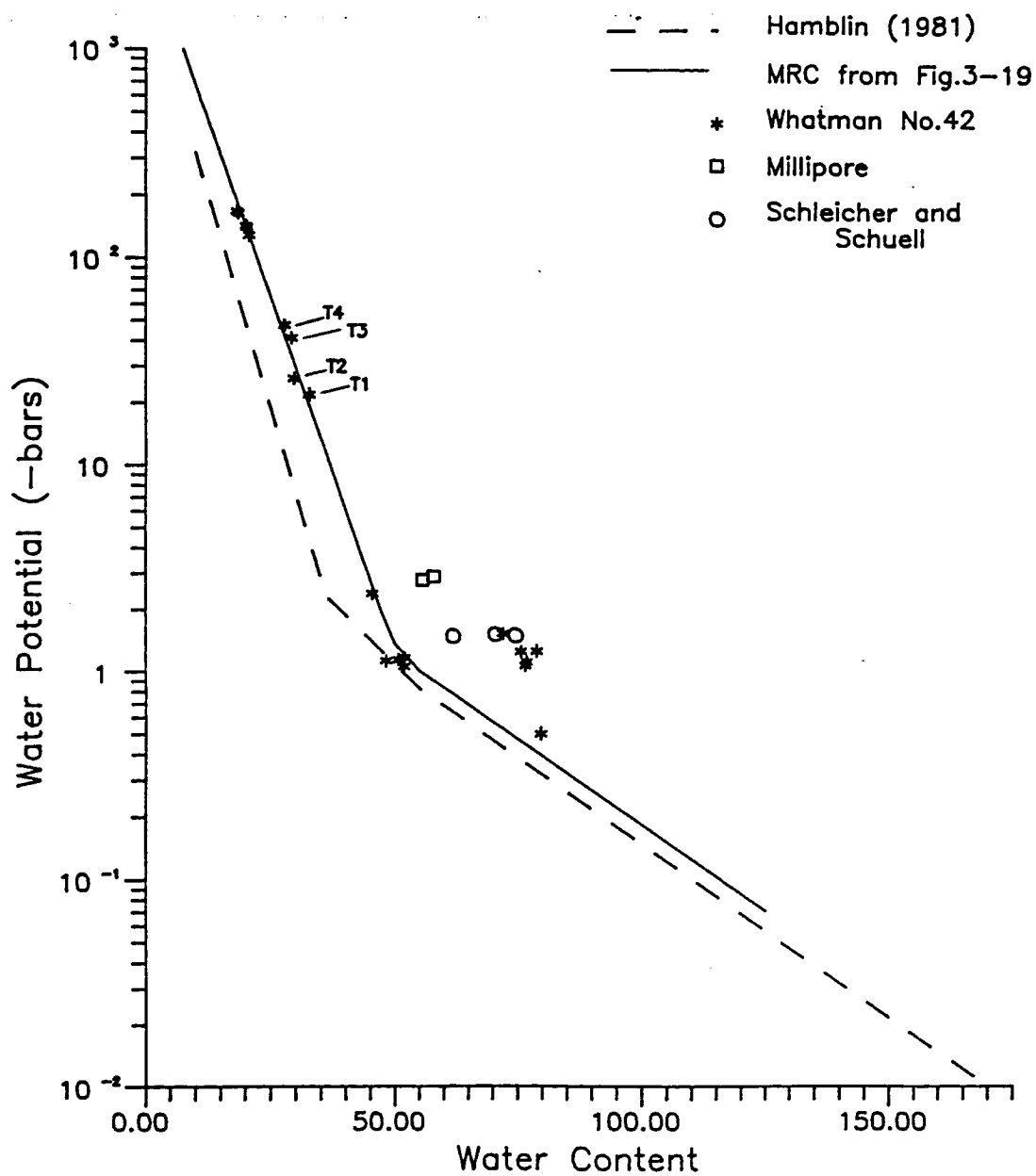


Figure 3.26. Results from filter papers equilibrated with sandstone slabs compared with the MRC developed previously.

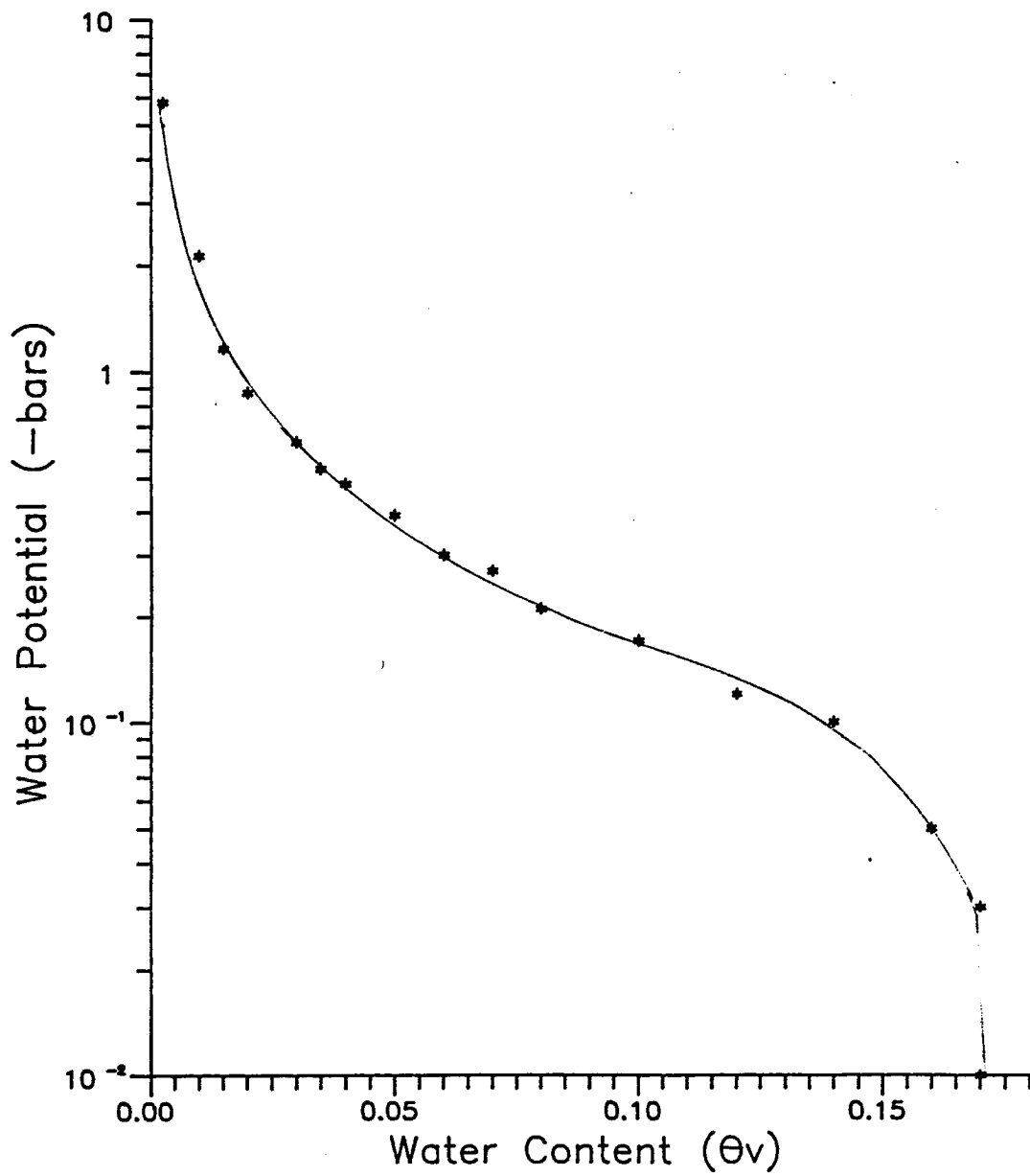


Figure 3.27. A Coconino sandstone MRC developed by Rahi (1986).

Whatman No. 42 filter papers were equilibrated between the sandstone slabs for 20 days. The results are as follows:

<u>Experiment 4a</u>			<u>Experiment 4b</u>		
Water potential (bars)			Water potential (bars)		
filter paper	rock	thickness	filter paper	rock	thickness
	-1.0	.8		-0.25	.6
-1.13	-0.4	.6	-0.50	-0.18	.6
-1.05	-0.5	.85	-1.10	-0.32	.65
-1.16	-0.32	.8	-1.06	-1.2	.7
-1.12	-0.9	.7	-1.24	-0.25	.7

The water potentials estimated from the MRC for the sandstone slabs are higher than the pressure induced within the pressure vessel (4.5 bars). The 15-bar ceramic plate is required for experiments where the pressure exceeds 3 bars. Experiments involving pressures of 0 to 3 bars were accomplished on a 3-bar ceramic plate. The 15-bar ceramic plate is manufactured such that air will not pass through the pores of the plate until an excess of 15 bars of pressure exists within the pressure vessel. The pore size of the 15-bar ceramic plate is approximately 0.16 microns.

The results of this experiment show that 75 hours at 4.5 bars of pressure is not enough time for the water potential of the sandstone slabs to equilibrate with the applied pressure. The water potential of the filter papers measured with the SC-10A were very consistent (-0.5 to -1.24 bars) and near the high water potential extreme capable of being measured by the SC-10A. More time was required for the filter papers to

equilibrate with the rock. Some errors may also be associated with the MRC curve described by Rahi (1986) and the procedure used to measure rock water content, or the limited ability of the SC-10A to accurately measure high water potentials. However, the comparison between the equilibrated filter papers and sandstone was reasonable. It is also noteworthy that the filter paper with the highest water potential (-0.5 bars) was surrounded by the two sandstone slabs with the highest water potentials (-0.25 and -0.18 bars). The rock water potential did not appear to be influenced by the thickness (or volume) of the sandstone slabs.

Tuff. The same experimental procedures used with the sandstone slabs were used with cores of Apache Leap Tuff. Rock cores, 6.3 cm in diameter and 1.0 cm thick, were saturated and equilibrated on a ceramic plate. The results of these experiments are shown in Table 3.6.

The water potential of the filter papers is much lower than the applied pressure. The water content and water potential of the tuff samples were not calculated making it impossible to determine if the errors result from the filter papers or the rock. The results of experiment 3 were added to Figure 3.23 and found that the filter papers (points T1 to T4) fit the filter paper MRC.

3.4.6 Effects of Pressure on Equilibration of Rock and Filter Papers

In these experiments, the effects of pressure or a compressive force on the equilibration of rocks with filter papers was examined. The purpose of applying pressure was to simulate field experiments involving an inflated packer which forces filter papers against a borehole wall. Accurate weight measurements of the rock samples were made in order to

Table 3.6. Experiments involving equilibration of Apache Leap tuff with various types of filter papers for different lengths of time.

Experiment 1. Tuff equilibrated in pressure extractor at 2.7 bars for 69 hours, then equilibrated with filter papers for 22 days.

	Filter Paper Water potential (bars)
3 Whatman No.42	-22.7
	-21.87

Experiment 2. Tuff equilibrated in pressure extractor at 2.7 bars for 69 hours, then equilibrated with filter papers for 26 days.

3 Whatman No.42	-30.5
	-25.6

Experiment 3. Tuff equilibrated in pressure extractor at 2.7 bars for 93 hours, then equilibrated with filter papers for 29 days.

		Filter Paper Water Content	
3 Whatman No.42	-21.7	32.6	T1
3 Schleicher and Schuell	-26.0	29.5	T2
2 Whatman No.42	-40.8	29.0	T3
3 Whatman No.42	-47.0	27.7	T4

calculate rock water potentials and compare them with filter paper water potentials.

Sandstone. Saturated sandstone slabs were placed on the 3-bar ceramic plate within the pressure vessel and equilibrated for 93 hours at 2.6 bars of pressure. Following equilibration, Whatman No. 42 filter papers were placed between the rock slabs. A column of seven sandstone slabs separated by filter papers were wrapped tightly in polyurethane and surrounded by foam insulation to prevent evaporation and large temperature fluxuations. A large rock weighing approximately 2 kilograms was then placed on top of this column and left to equilibrate for 18 days. Two sample cans containing sandstone slabs separated by filter papers were also sealed and equilibrated in a constant temperature room for 18 days. The results are presented in Table 3.7.

The water potential of the filter papers under applied pressure exhibited a significant increase towards the bottom of the column. The gradient is much less significant when no additional force is applied. The water potential of the sandstone, estimated from the MRC described by Rahi (1986), is presented in Figure 3.27. The filter paper on the bottom of the column with the applied pressure was the only one that had a water potential value near the range of the rock water potentials. For the column of sandstone slabs with the additional pressure of a 2 kg rock, it appeared that water moved from the top of the column towards the bottom. Not only was there a gradient of filter paper water potentials, the rock water potentials generally exhibited an increase in values toward the bottom of the column as well. The results also show that equilibration of filter paper and rock water potentials takes

Table 3.7 A comparison of two experiments involving filter papers equilibrated with sandstone slabs. Experiment I involved the addition of a 2 kg rock placed on top of the column of sandstone slabs. Experiment II involved columns of sandstone slabs with no applied pressure.

I. Experiment with 2 kg weight:

		<u>Filter paper</u>	<u>Rock*</u>
	Water Content	Water potential	Water potential
	(percent dry weight)	(bars)	(bars)
Top			-0.58
1	18.2	-68.5	-1.50
2	20.7	-40.5	-0.87
3	19.5	-36.5	-0.72
4	19.5	-32.6	-0.77
5	23.9	-12.0	-0.72
6	31.4	-2.55	-0.58

II. Experiment without additional weight:

			-0.74
1	18.9	-52.6	-2.40
2	23.1	-38.0	-0.63
3	21.5	-36.6	-1.74
			-0.58
1	15.8	-107.1	-0.74
2	16.0	-110	-0.73
3	16.6	-96	-1.74
4	14.2	-117.6	-0.92

* Rock-water potentials are estimated from the MRC for sandstone defined by Rahi (1986) presented in figure 3.27.

longer than 18 days when pressure is not applied.

To prevent the creation of a water potential gradient, the following experiments were set up with a single rock slab placed on a circular rubber pad covered with aluminum foil. Two filter papers were set in between the rock and the aluminum covered rubber pad. The rock, filter paper and pad were wrapped tightly in polyurethane and taped. Three large rocks , 9.7, 14.1, and 15.2kg were used to simulate pressures used in field experiments. The filter papers have an average surface area of 5.4 square centimeters, therefore the approximate pressures supplied by the rocks were 1.68, 2.44, and 2.64 bars. For the two sets of experiments presented in Table 3.8 and 3.9, the filter papers equilibrated with the rock slabs with the additional pressure for 90.5 to 281 hours.

The first set of experiments (I, II, and III) presented in Table 3.8 involved the equilibration of rocks in the pressure vessel at 2.7 bars for 166 hours. The rock water content was determined before equilibration with the Whatman filter papers and after equilibration to see if a significant amount of water was lost during equilibration. The rock water potentials were estimated from the MRC for sandstone defined by Rahi (1986) (Figure 3.27). The water potential of the rocks before equilibration with the filter papers ranged from -0.26 to -1.16 bars and after equilibration ranged from -0.3 to -2.8 bars. The change of rock water potentials from before and after equilibration range from 0.02 to 1.83 bars. The results of this experiment show that all the rocks have a much higher water potential than predicted by the applied pressure. Thus, even after 7 days at 2.7 bars, the sandstone slabs did not fully

Table 3.8. Summary of experimental results involving the equilibration of sandstone slabs, previously equilibrated on a pressure plate at 2.7 bars for 166 hours, with Whatman No. 42 filter papers and the addition of pressure.

I. Equilibrated at 2.64 bars for 90.5 hours.

<u>Rock No.</u>	<u>Filter Paper</u>		<u>Rock</u>		ψ Average (bars)
	θ % dry wt	ψ (bars)	ψ BE (bars)	ψ AE (bars)	
9	27.1	-19.1	-0.53	-0.73	-0.63
10	35.4	-2.6	-0.39	-0.41	-0.39
11	27.6	-11.5	-0.65	-0.76	-0.72
12	36.5	-2.1	-0.49	-0.58	-0.55
13	56.6	-1.0	-0.37	-0.40	-0.39

II. Equilibrated at 2.44 bars for 190 hours.

1	30.6	-9.2	-0.53	-0.76	-0.65
2	25.9	-25.2	-0.51	-0.77	-0.65
3	28.8	-13.4	-0.74	-1.35	-0.92
4	24.5	-36.3	-0.74	-1.50	-0.92
5	22.3	-50.4	-0.92	-2.12	-1.35
6	18.1	-102.9	-0.74	-1.74	-0.92
7	24.5	-30.3	-0.92	-1.93	-1.16
8	20.2	-79.6	-0.92	-2.40	-1.50

III. Equilibrated at 1.68 bars for 281 hours.

16	21.3	-82.4	-0.73	-1.93	-0.97
17	19.8	-82.0	-0.97	-2.80	-1.74
18	28.5	-16.1	-0.73	-1.50	-0.92
21	27.1	-29.8	-1.16	-2.12	-1.74

ψ BE is the rock-water potential (bars) before equilibration with filter papers.

ψ AE is the rock-water potential (bars) after equilibration with filter papers.

ψ Average is the average rock-water potential determined from the average water content of the data taken before and after equilibration with filter papers.

equilibrate with the applied pressure within the pressure vessel.

In experiment I, the rocks with the highest water potential correspond with the Whatman filter papers with the highest water potential. Although the filter papers in contact with rocks 10, 12 and 13 have water potentials one to two bars lower than the rock water potential of all the data collected, these filter papers most nearly approximate the rock water potential. In experiment II, the filter paper with a water potential most similar to the rock water potentials is rock 1 with a corresponding filter paper water potential of -9.2 bars. Although this is nearly nine bars lower than the corresponding rock water potential, the rock in contact with the filter paper has the highest water potential of the rocks in that experiment. This relationship between the highest filter paper water potential corresponding with the highest rock water potential is also evident in experiment III. None of the experiments appear to have a strict one to one relationship between the rock and filter paper water potentials.

Experiments V, VI and VII were performed with the saturated sandstone slabs equilibrated in a pressure vessel at 2.18 bars for 14 days, and the filter papers equilibrated with the rocks for 188 to 270 hours with additional pressure. The results of this set of experiments are presented in Table 3.9. The rock water potentials before equilibration with the filter papers ranged from -0.58 to -1.16 bars. Thus, none of the sandstone slabs fully equilibrated with the applied pressure even after 14 days at 2.18 bars of pressure. After equilibration with the filter papers, the rock water potentials ranged from -0.87 to -2.8. As with the first set of experiments, the filter papers with the highest

Table 3.9. Summary of experimental results involving the equilibration of sandstone slabs, previously equilibrated at 2.18 bars in a pressure extractor for 14 days, with Whatman No. 42 filter papers and additional pressure.

V. Equilibrated with 2.64 bars for 188 hours.

Rock no.	Filter Paper		Rock		
	θ % dry wt.	ψ (bars)	ψ BE (bars)	ψ AE (bars)	ψ Average (bars)
16	22.6	-57.6	-0.73	-1.35	-0.92
12	28.9	-15.6	-0.68	-0.76	-0.72
13	28.5	-18.8	-0.72	-0.87	-0.74
23	25.9	-25.3	-0.87	-1.50	-1.06
11	28.3	-20.7	-0.73	-0.87	-0.76

VI. Equilibrated with 2.44 bars for 258 hours.

5	17.3	-145.5	-0.96	-2.80	-1.50
6	20.1	-9.3	-0.74	-1.50	-0.92
7	21.6	-62.8	-0.97	-2.12	-1.50
8	20.1	-87.2	-0.76	-2.12	-1.16
9	24.3	-48.8	-0.58	-0.97	-0.72
10	27.5	-27.5	-0.68	-0.92	-0.74

VII. Equilibrated with 1.68 bars for 270 hours.

1	22.2	-57.0	-0.73	-1.74	-0.92
2	26.1	-28.7	-0.58	-0.77	-0.70
3	23.2	-47.0	-0.92	-2.12	-1.35
4	19.5	-98.5	-0.72	-2.03	-0.92
17	22.1	-58.0	-0.92	-2.40	-1.50
21	25.8	-31.0	-1.16	-2.40	-1.74
23	25.8	-29.0	-0.72	-0.97	-0.77

ψ_{BE} is the water potential (bars) before equilibration with filter papers.

ψ_{AE} is the water potential (bars) after equilibration with filter papers.

ψ Average is the average water potential (bars) determined from the average water content of the before and after equilibration data.

water potentials were generally equilibrated with sandstone slabs with the highest water potential.

The water potential of the filter papers and corresponding average rock water potentials for all six experiments were plotted on a semi-log graph (Figure 3.28). The average water content before and after equilibration with the filter papers was used to determine the average rock water potential.

Tuff. Experiments similar to those described previously with sandstone slabs were set up using rock cores from the slightly welded unit of the Apache Leap tuff, near Superior, Arizona. The results are presented in Table 3.10. The tuff cores were saturated in the vacuum dessicator and equilibrated in the pressure extractor; experiment IV equilibrated at 2.3 bars for 11 days, and experiment X equilibrated at 5 bars for 20 days. The tuff cores were removed, two Whatman No. 42 filter papers were set in between the rock and the aluminum covered rubber pad and wrapped tightly in polyurethane and tape. An additional pressure of 2.64 bars was applied to the rock cores for 21 days in experiment IV and 25 days in experiment X. The water potential of the tuff slabs was estimated by the MRC developed by Rahi (1986) shown in Figure 3.29. The average rock water potential was calculated from the average rock water content data taken before and after equilibration with the filter papers. The filter paper water content and water potential measurements were plotted on the filter paper MRC and compare very well with the MRC developed in this research (see Figure 3.19) and the MRC by Hamblin (1981). The results are presented in Figure 3.30.

For both experiments, all the rock water potential values are

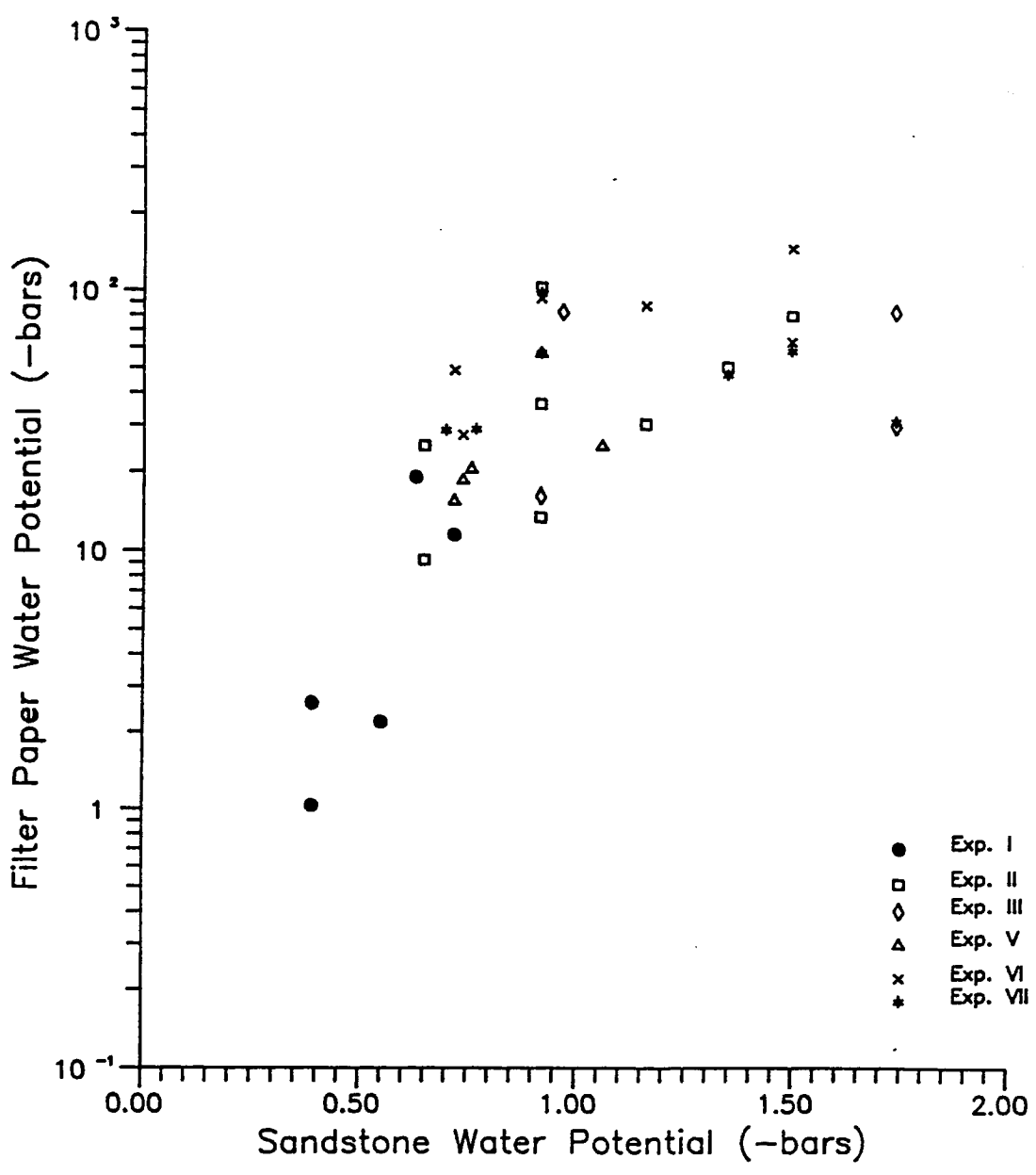


Figure 3.28 Filter paper water potentials plotted with corresponding sandstone water potentials.

Table 3.10. Summary of experimental results involving the equilibration of Apache Leap tuff with Whatman No. 42 filter papers and additional pressure.

IV. Equilibrated tuff in pressure extractor at 2.3 bars for 11 days.
Equilibrated filter papers with tuff for 21 days with 2.64 bars.

<u>Rock no.</u>	<u>Filter Paper</u>		<u>Rock</u>		
	θ % dry wt.	ψ (bars)	ψ_{BE} (bars)	ψ_{AE} (bars)	ψ Average (bars)
1	27.9	-8.7	-3.8	-5.6	-5.4
2	27.3	-14.4	-6.1	-9.4	-7.6
3	25.5	-19.6	-5.4	-7.7	-6.4
4	25.9	-19.2	-4.6	-6.8	-6.1
5	29.3	-7.6	-3.2	-5.8	-3.7

X. Equilibrated tuff in pressure extractor at 5 bars for 20 days.
Equilibrated filter papers with tuff for 25 days with 2.64 bars.

1	23.9	-44.1	-8.2	-11.3	-9.6
2	21.4	-63.9	-12.1	-24.2	-16.4
3	22.9	-49.2	-9.6	-14.6	-12.1
4	23.9	-50.6	-8.0	-15.3	-12.1
5	23.9	-38.9	-10.8	-16.4	-14.6

ψ_{BE} is the water potential (bars) before equilibration with filter papers.

ψ_{AE} is the water potential (bars) after equilibration with filter papers.

ψ Average is the average water potential (bars) determined from the average water content from the before and after equilibration data.

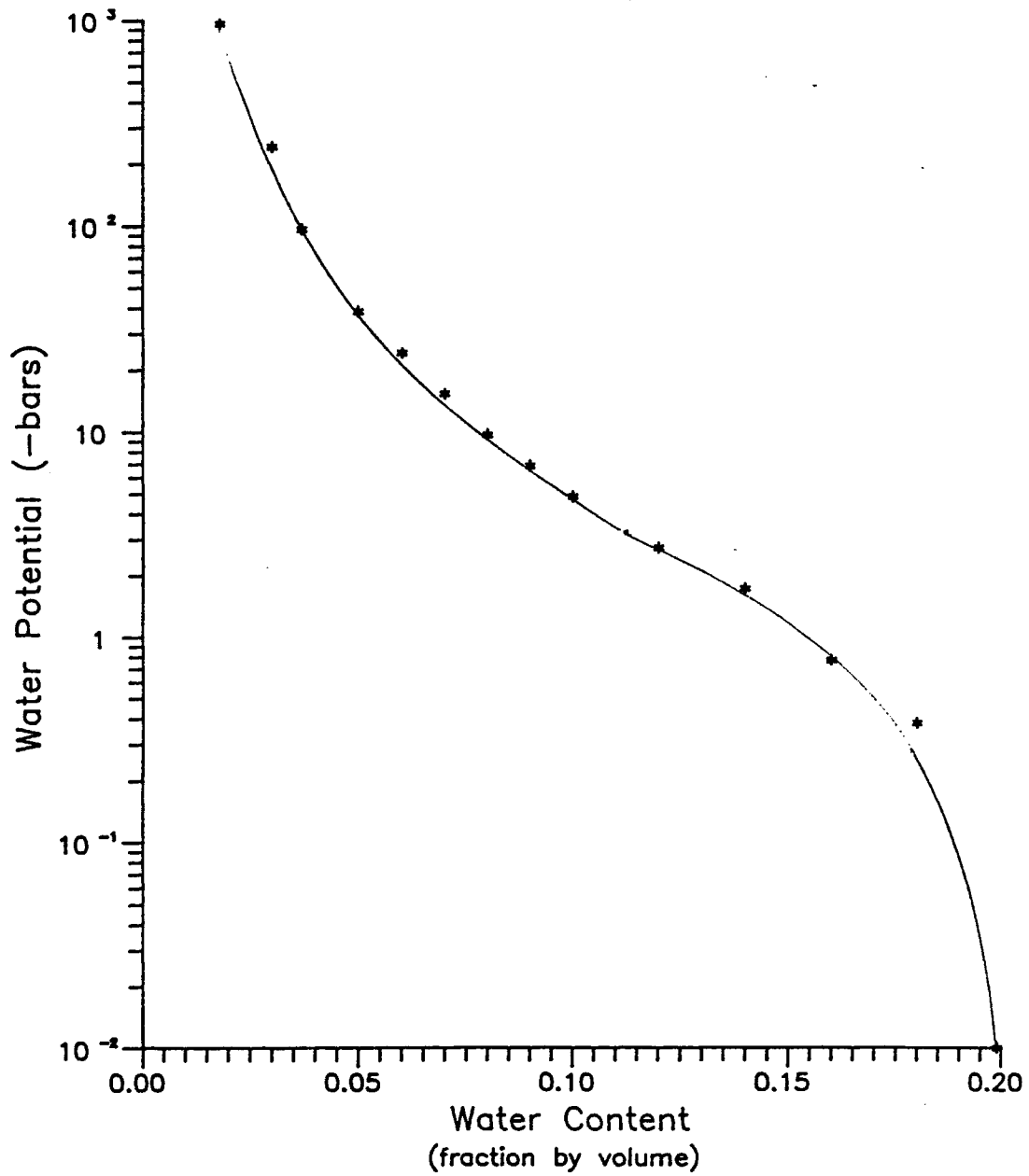


Figure 3.29. Moisture release curve for Apache Leap tuff (slightly welded) developed by Rahi (1986).

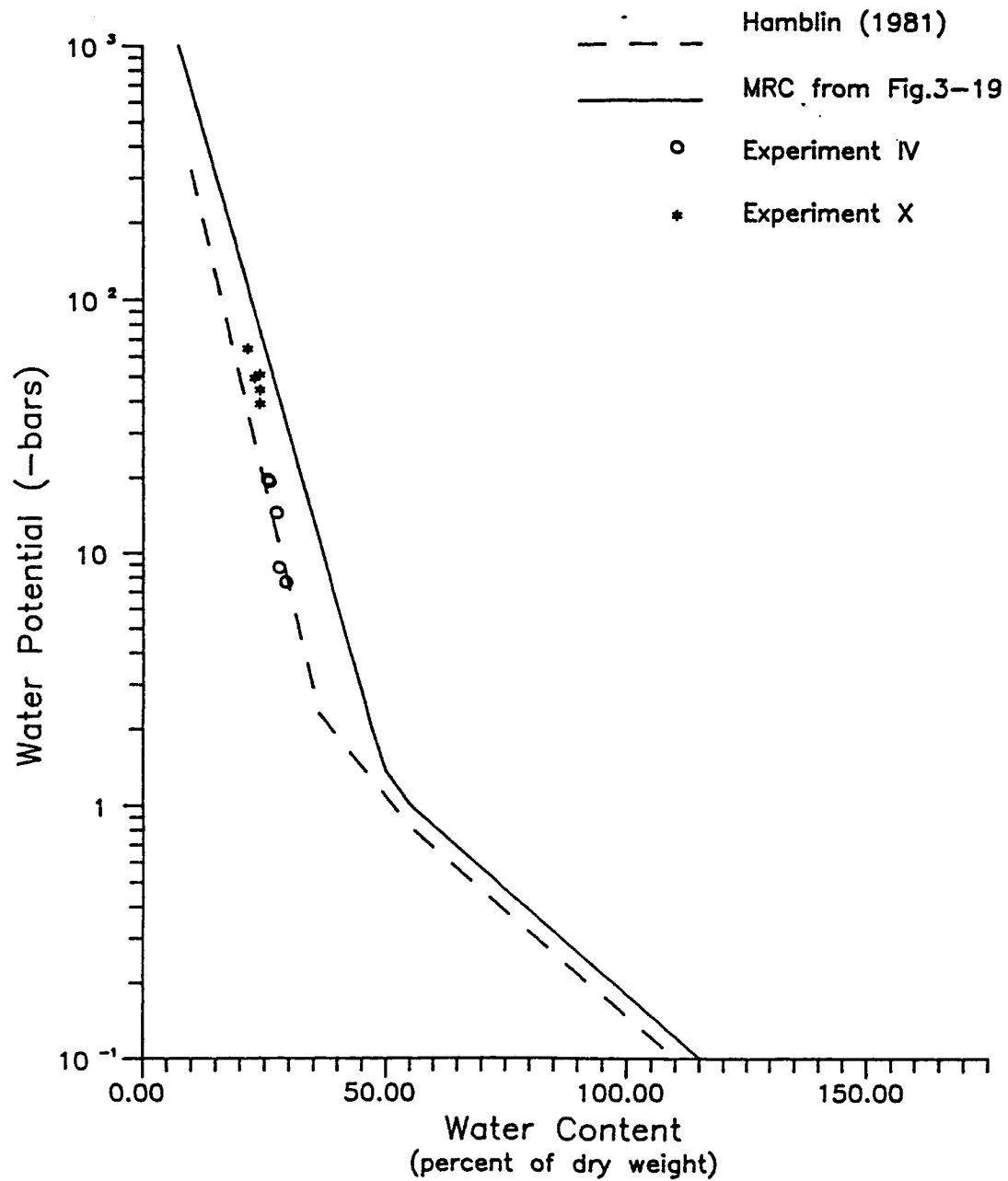


Figure 3.30. Filter paper water content and potential for papers equilibrated with Apache Leap tuff plotted with MRC's described previously.

lower than the applied pressure. In experiment IV the filter paper water potentials are 3 to 13 bars less than the corresponding average rock water potentials. Equilibration time of 21 days with additional pressure of 2.64 bars was apparently not enough time for filter papers to attain the same water potential value as the tuff cores.

Experiment X involved the equilibration of the tuff cores with 5 bars of pressure for 20 days. The rock water potentials estimated by Rahi's MRC give values ranging from -9.6 to -14.6 bars. The filter paper water potentials were lower than the corresponding average rock water potential by 24.3 to 47.5 bars. In this experiment, the filter papers varied more dramatically from the rock water potential than Experiment IV, even after 25 days of equilibration with the addition of 2.64 bars. The rock water potential and corresponding filter paper water potential data are plotted in Figure 3.31. A relationship, other than the predicted one to one relationship, appears to be significant and can be described by the following linear equation:

$$Y = 0.194 X - 3.27 \quad (4)$$

where Y is the tuff water potentials; X is the filter paper water potential; and the correlation coefficient is 0.917.

3.4.7. Field Experiments

A series of doubled Millipore and Whatman No. 42 filter papers covered by a filter paper buffer were carefully taped to aluminum foil which covered a section of small inflatable rubber packers (0.3 meters long). The packers were carefully placed in horizontal boreholes in the

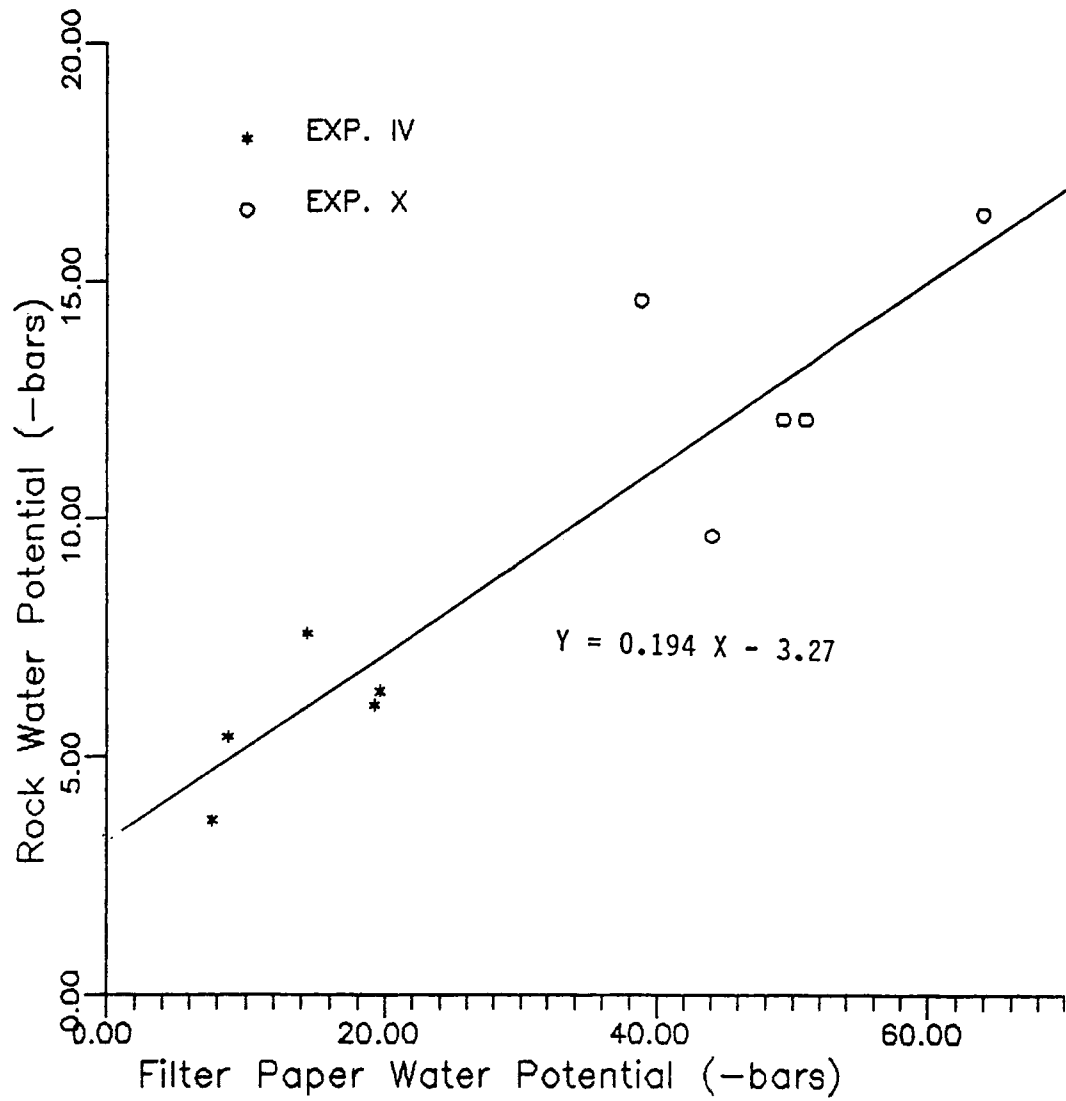


Figure 3.31. Tuff water potential vs corresponding Whatman No. 42 filter paper water potential.

Queen Creek road tunnel near Superior, Arizona. When a packer was in place, a bicycle pump was used to inflate the packer to about 2 bars. The borehole was sealed and the filter paper-packer assembly left to equilibrate with the water potential of the welded tuff. After a period of equilibration, the packer was deflated and taken out of the borehole. The filter papers were removed and placed in the chambers of the thermocouple psychrometer SC-10A for water potential measurement. After all the measurements had been taken, the filter papers were removed and quickly placed in small (2 dram) glass bottles, transported to the laboratory and weighed. The filter papers were dried and the water content (percent of dry weight) was calculated. The glass bottles had a plastic lid and paper septum with a certain initial water content. A correction factor was determined for the amount of water lost from the septum when the lids and glass bottles containing the filter papers were dried.

For the first set of field experiments involving boreholes 1, 3 and 4, the packers were placed approximately 108 cm into the borehole, inflated to 2 bars and left to equilibrate for 38 days. The results are presented in Table 3.11. The water potential of the filter papers in Borehole 1 were very low, with the Millipore papers being the lowest at -655 bars. According to standard psychrometer measurements made two years earlier, Borehole 1 had water potential values of -15 to -20 bars for the month of June (see Table 3.4).

Measurements made with the standard psychrometer in Borehole 3 has consistently given higher water potential readings than Boreholes 1 and 2 (see Table 3.4). This trend is also evident in the water

Table 3.11. Results from field experiments involving 39 days (6/16/86 to 7/24/86) of filter paper equilibration with welded tuff of the boreholes in the road tunnel near Superior, Arizona.

Filter paper	Water content (% of dry wt.)	Water potential (bars)	Psychrometer readings (6/84) (bars)
<u>Borehole 1</u>			
Whatman 42	13.0	-125.1	-15 to -20
Whatman 42	17.4	-181.2	
Whatman 42	12.0	-211.9	
Millipore	3.8	-655.8	
<u>Borehole 3</u>			
Whatman 42	19.8	-57.9	-3.5 to -8.2
Whatman 42	15.6	-68.4	
Whatman 42	24.6	-50.8	
Millipore	24.2	-3.62	
Millipore	22.2	-5.04	
<u>Borehole 4</u>			
Whatman 42	27.2	-4.5	
Whatman 42	28.6	-6.2	
Whatman 42	23.0	-22.4	
Millipore	41.8	-3.46	
Millipore	21.4	-4.2	
Millipore	44.8	-3.55	

potential measurements made with the SC-10A of the equilibrated filter papers. The Millipore filter papers used in Borehole 3 have water potentials approximately 50 bars higher than the Whatman filter papers. The water potentials estimated with the Millipore papers are very similar to the measurements made two years earlier with the standard psychrometers (-3.5 to -8.2 bars). Boreholes 3 and 4 are parallel and about 1 meter apart. Field psychrometer data was not recorded for Borehole 4, but it was assumed that the water potential data of Borehole 3 would be nearly the same as Borehole 4. In Borehole 4, the water potential of the two types of filter papers were generally very similar. The Whatman No. 42 filter papers had water potentials values of -4.5, -6.2, and -22.4 bars. The water potential of the Millipore filter papers were -3.46, -4.2, and -3.55 bars. The rock water potentials determined with the filter papers agree with the rock water potential assumed to exist in Borehole 4, ranging from -3.5 to -8.2 bars.

The filter paper water content and water potential values are plotted on the filter paper MRC in Figure 3.32. Generally, the results fit the MRC very well.

The second set of field data involved equilibrating Whatman No. 42 and Millipore filter papers for 23 days in Boreholes 2 and 3. An extension was attached to the packer in Borehole 2 enabling the packer to be placed 200 cm into the borehole. A packer was placed 77 cm into Borehole 3. The results of this set of experiments are presented in Table 3.12 and were added to the MRC of Figure 3.32.

The calculated water content of the filter papers in Borehole 2 may be in error because the amount of water lost from the septum of the

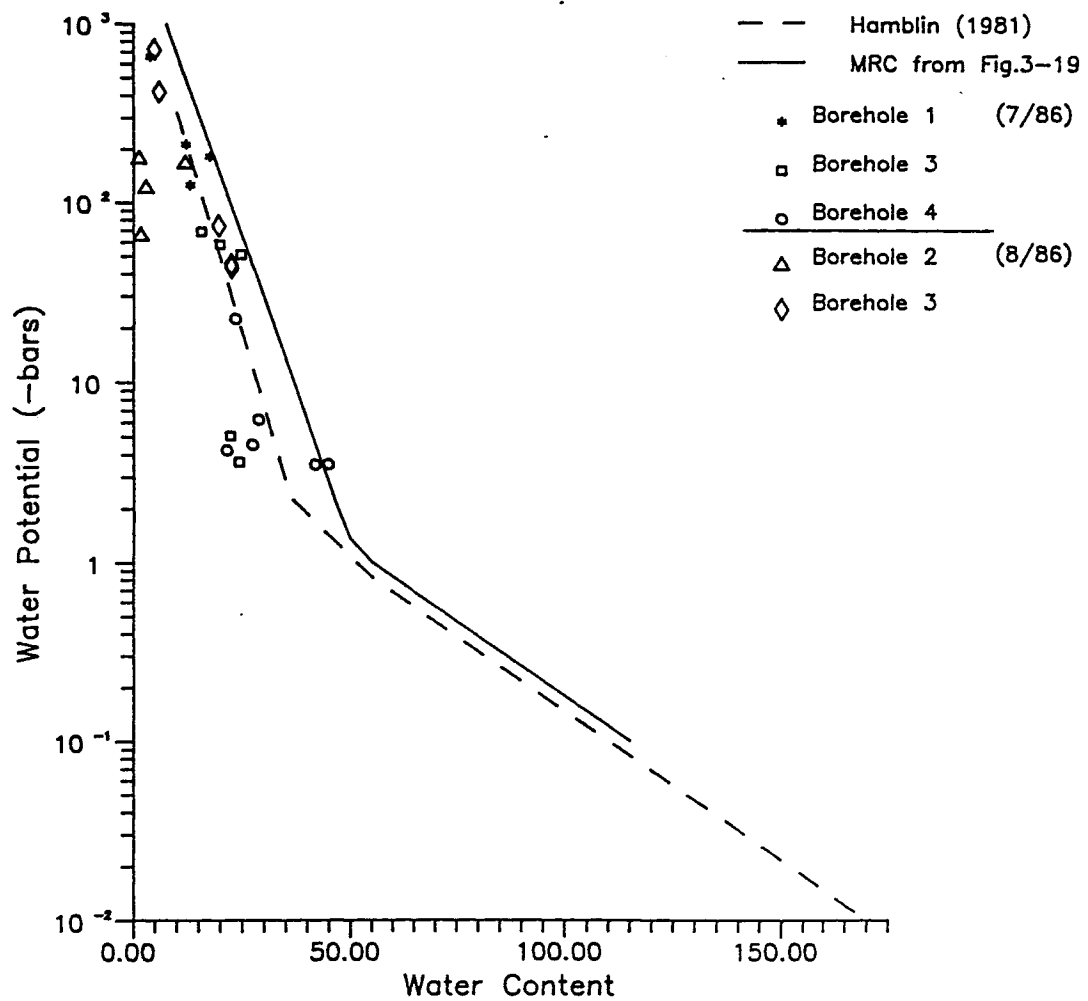


Figure 3.32. Filter paper water content and potential values taken after equilibration in four boreholes in the Queen Creek road tunnel plotted with MRC's described previously.

Table 3.12. Results from field experiments involving 23 days (8/23/86 to 9/15/86) of equilibration of filter papers and welded tuff in boreholes at the road tunnel site near Superior, Arizona.

Filter paper	Water content (% of dry wt.)	Water potential (bars)	Psychrometer readings (6/84) (bars)
<u>Borehole 2</u>			
Whatman 42	1.81	-66.4	-12.7 to -22.8
Whatman 42	2.86	-123.0	
Whatman 42	11.9	-160.0	
Millipore	--	-142.7	
Millipore	--	-168.0	
Millipore	1.27	-180.0	
<u>Borehole 3</u>			
Millipore	5.9	-418	-3.5 to -8.2
Millipore	4.8	-719	
Millipore	--	-716	
Whatman 42	22.5	-43.0	
Whatman 42	22.4	-45.0	
Whatman 42	19.6	-73.5	

weighing bottle was estimated after the bottles were dried. The water potentials of both types of filter papers were very low, ranging from -66.4 to -180 bars. The water potential readings made with the field psychrometers two years earlier ranged from -12.7 to -27.8 bars.

Borehole 2 is parallel and about 1 meter away from Borehole 1 where the rock water potentials determined with the absorber method for the first set of field experiments (7/86) ranged from -125 to -211 bars for the Whatman filter papers.

The packer-filter paper assembly was placed 77 cm into Borehole 3. The water potential results of the Millipore papers taken during the first set of field experiments differed dramatically from the data taken during the second set while the Whatman No. 42 papers remained very consistent. The Millipore filter paper water potentials were very dry, ranging from -418 to -719 bars. The Millipore papers are very brittle, particularly after being dried, making them sensitive to handling and making it difficult to accurately determine their water content. Improper handling of the brittle filter papers may have caused these data to give unreasonably low water potentials.

The water potential of the Whatman No.42 filter papers varied from -43 to -73.5 bars. The estimated rock water potentials are lower than the values made two years previous with the field psychrometers, ranging from -3.5 to -8.2 bars, but are very similar to values determined during the first set of field experiments. The particular area of measurement, 70 to 100 cm into the borehole, may have a lower water potential than the average "atmospheric" water potential measured with the field psychrometers. The psychrometers are not in direct

contact with the borehole and are influenced by the water vapor changes throughout the borehole.

The psychrometer data gathered two years ago (6/84) is assumed to represent the typical range of water potentials for that month. The rock water potentials within the borehole are affected by changes in precipitation cycles and influences of other unrelated experiments performed in the borehole. Thus, the field psychrometer data may be used as a reference, but not as a precise measure, of current rock water potentials.

With the exception of Borehole 2, the field experimental data fit the filter paper MRC. The duplicated experiments in Borehole 3 gave similar results with the Whatman No. 42 filter papers while the Millipore papers differed by an order of magnitude. Although the water potential of Millipore papers were consistent within each experiment, when experiments were duplicated, as in Borehole 3, the water potential results varied significantly. From these conclusions, the Whatman No. 42 filter papers would be preferred over the Millipore filter papers for future field experiments.

CHAPTER FOUR

SUMMARY AND CONCLUSIONS

This study tested and evaluated field methods for measuring water potentials of unsaturated fractured rock. These methods were the downhole tensiometer, osmotic tensiometer, thermocouple psychrometer, and the absorber method. All were originally designed for soil water potential measurements and were adapted in this study for field measurements of rock water potential.

The downhole tensiometer was an adaptation of the standard soil tensiometer and, like the soil tensiometer, was limited to water potential measurements from 0 to -0.8 bars. For accurate rock water potential measurements, the most important criterion was the assurance of good hydraulic contact between the ceramic cylinder of the tensiometer, the injected silica flour slurry, and the borehole wall. A pressure transducer attached to the tensiometer measured the changes in internal pressure as the silica flour equilibrated with the rock water potential. The rapid and accurate response achieved during laboratory experiments supported this method as a viable way to determine in situ rock water potentials estimated to be greater than -0.8 bars. The possible utility of the downhole tensiometer is that it can be placed in particular areas of a borehole to measure water potentials in fractured and non-fractured rock. Future field and laboratory experiments are

necessary to assess the accuracy of water potential measurements with extended use.

The osmotic tensiometer consisted of a stainless steel core covered by the tubular membrane. The annular space between the core and the membrane was filled with osmotic solute until the membrane was in contact with the borehole wall. Internal hydrostatic pressure changes caused by water flowing into or out of the device in response to relative changes in total potential were measured with a pressure transducer. The average matric potential of the fractured rock mass in contact with the membrane was determined by summing the measured positive hydrostatic pressure and the known osmotic potential of the osmotic solution.

Optimal functioning of the osmotic tensiometer depended on the combination of a durable membrane that was permeable to water, and a solute with a molecular weight large enough to be retained by the membrane. Spectra/por 4 membranes and PEO of 200,000 m.w. were found to be the most suitable combination. The maximum range of osmotic potentials obtainable with PEO of 200,000 m.w. was about 0 to -3 bars.

Laboratory experiments involving a ceramic "borehole" where water potentials were induced with a vacuum pump, verified that the tensiometer can be used to measure rapidly changing moisture conditions. The field experiments made in boreholes in the Santo Nino mine were less definitive. A continual decay of hydrostatic pressure throughout a 6 day experiment and visible stains on the protective cloth, were evidence that solute leakage had occurred through small pinholes in the membrane. Changes in the design of the tensiometer or an alternative combination

of membranes and solutes is needed to make the osmotic tensiometer a viable method for measuring rock water potentials in a borehole.

Thermocouple psychrometers were sealed in boreholes at four different field sites. The psychrometers measured an average water potential of the rock matrix and of all open fractures intersecting the borehole. Because psychrometers are limited to water potential measurements of -2 to -50 bars, a method was devised to determine which extreme was prohibiting measurement. This method was used for eleven of the sixteen boreholes measured.

Comparison of results obtained from corresponding pairs of psychrometers within the same boreholes of the Queen Creek road tunnel site indicated different measured potentials. These discrepancies indicate the possible impreciseness of water potential measurements made using thermocouple psychrometers. In addition, the psychrometers measure the average matrix and osmotic potentials that exist in the vapor of a borehole rather than measuring the potential of the rock directly. Still, the field studies did show that thermocouple psychrometers can be used to determine temporal trends in water potentials even if absolute values are subject to uncertainty.

The absorber method involved the equilibration of rock water potentials with filter papers. After equilibration, the filter papers were placed in the thermocouple psychrometer sample changer to determine the filter paper's water potential. In laboratory experiments using cores of welded tuff, a relationship was described between the water potential of tuff and filter paper (see equation 4).

Continued laboratory experiments equilibrating water potentials of tuff and filter papers would be necessary to determine the required equilibration time and to substantiate the relationship described by equation 4. Emphasis on modeling the necessary equilibration time may result in a more reliable estimation of the time required for equilibrating the water potential of tuff and filter papers.

The absorber method, unlike the field psychrometers, is able to measure water potentials of a particular area of a borehole. Thus, by allowing filter papers to simultaneously equilibrate throughout a borehole, water potential gradients can be determined.

Recent investigations for high level radioactive waste repositories has stimulated interest in the characterization of unsaturated flow in fractured rocks. Unsaturated flow is dictated by the water potential gradients within the geologic medium. The four methods evaluated in this study to determine water potentials in fractured rock covered different ranges of measurement and uncertainty. Initial use of field psychrometers sealed in a borehole can reveal the range of water potentials. If the potentials range from 0 to -0.8 bars, the downhole tensiometer can be effective. The osmotic tensiometer can be used for potentials from 0 to -3 bars, however, improvements to the design are necessary before this method can be recommended for use in the field. For water potentials ranging from -2 to -100 bars, the absorber method can be used.

The four methods discussed in this thesis need modifications to improve the accuracy of measurement of rock water potentials, especially for determining the hydraulic head gradient for flow calculations.

APPENDIX A

Thermocouple Psychrometers

This appendix contains detailed descriptions for laboratory calibration methods of thermocouple psychrometers. The discussion is primarily based on the information presented in the instruction manual for the Wescor HR-33T Dew Point Microvoltmeter (Wescor, 1979), a Utah State University Bulletin (Wiebe et al., 1971) and an instruction manual for the Thermocouple Psychrometer SC-10A (Decagon devices, 1981).

Thermocouple psychrometers are used to determine moisture potential indirectly by measuring temperature phenomena (dewpoint temperature and temperature depression associated with evaporation) which are functions of the relative humidity of the environment in which the psychrometers are placed. Each psychrometer will produce slightly different responses to identical moisture potentials due to variability in the thermocouple characteristics. Thus, although it is possible to approximate the relationship between moisture potential and psychrometer response using theoretical and empirical equations (Wiebe et al., 1971; Wescor, 1979), it is necessary to define response characteristics of individual psychrometers through calibration routine if precise field measurements of moisture potential are to be determined.

Psychrometer response (as read using a microvoltmeter) is a function of temperature as well as moisture potential. Consequently, the calibration procedure described in this section involves a series of

measurements made over a range of temperatures and potentials likely to be encountered in field situations.

Equipment used during the calibration procedure includes a constant temperature water bath, water-tight calibration chambers, a microvoltmeter-control unit, and a series of salt solutions with predetermined osmotic potentials. The refrigerated water bath should be the type in which water is circulated with a pump (as opposed to a magnetic mixing device which can interfere microvolt readings) and which is capable of accurately maintaining subambient temperatures to within 0.1°C over a period of several hours. The bath should be large enough to hold the required number of calibration chambers, as well as a minimum of the first 7 to 10 centimeters of psychrometer lead wire protruding from the calibration chamber.

Calibration chambers can be purchased commercially (J.R.D. Merrill Specialty Equipment, Logan, Utah) or constructed from a variety of materials. The primary requirement of the chamber is that no vapor sources or sinks be present other than those provided by the calibration solution. It is desirable to maximize the free liquid surface area within the chamber and to minimize the volume of air surrounding the psychrometer in order to facilitate rapid vapor equilibration.

Microvoltmeter-control units can be purchased from several sources including Wescor, Inc., Logan, Utah, and J.R.D. Merrill Specialty Equipment, Logan, Utah. Some units are suitable for measurements using both psychrometric and dewpoint modes, while others lack the internal circuitry required for dewpoint measurements. Output from the microvoltmeter-control unit can be fed into an optional strip chart

recorder to provide a visual record of psychrometer response curves. These curves can then be diagnosed to identify problems as well as providing a record of the shape, slope, and width of response plateaus corresponding to psychrometric and dewpoint values.

The series of known potentials required for the psychrometer calibration procedure are generated using sodium chloride (NaCl) and potassium chloride (KCl) solutions having known osmotic potentials. Several millimeters of solution can be used to form a free liquid surface in the bottom of the calibration chamber or the interior walls of the calibration chamber can be lined with filter paper soaked with the salt solution. Osmotic potentials of the calibration solutions should cover the range of moisture potentials values anticipated in field situations. The range typically encompasses the effective measurement range (-2.0 bars to -50 bars) of the thermocouple psychrometer.

Osmotic potentials associated with various NaCl and KCl concentrations are determined by using the following equations (based on Raoult's law and Kelvin's law):

$$(A.1) \quad e / e_0 = 55.556 / (55.556 + O_s)$$

$$(A.2) \quad \psi = (RT / V) \ln(e/e_0) = 0.4617 T \ln(e/e_0)$$

where O_s is osmolality (Os/kg), e/e_0 is relative humidity (dimensionless), ψ is osmotic potential (MPa), and T is absolute

temperature (K). Osmolality values corresponding to various KCl and NaCl concentrations can be obtained from standard chemical references.

A.1. Procedures

Psychrometric measurements can be made using either the dewpoint method, the psychrometric method, or a combination of both. The psychrometric method is based on measurement of the temperature depression of a wet thermocouple caused by the evaporation of water from its surface. The dewpoint is based on the maintenance (by electronic means) of wet thermocouple temperature such that the amount of condensation is equal to the amount of evaporation from the thermocouple under ambient humidity and temperature. However, either method may at different times appear to be the better choice, depending on the experimental conditions and circumstances. General calibration and measurement procedures for the dewpoint and psychrometric methods are outlined below.

A.1.1. Dewpoint Method

Prior to calibrating psychrometers for the dewpoint method, it is first necessary to experimentally determine cooling coefficients (π_V) for individual psychrometers. The cooling coefficient is defined as the maximum junction temperature depression which results from the passage of a specified nominally optimum cooling current through the junction under dry conditions (Wescor, 1979). Once the coefficient has been determined for a given psychrometer, it is then possible to adjust the electronic gain of the microvoltmeter-control unit prior to making dewpoint measurements.

Cooling coefficients are determined after the psychrometers have been allowed to equilibrate under dry conditions (less than 90 percent relative humidity). An empty ice chest serves as an excellent constant temperature / constant humidity chamber. A cooling current is applied to a psychrometer for a short period of time (approximately 10 seconds) and the electronic gain of the control unit is adjusted until the microvolt readings holds at a constant value between 15 and 30 μV . The corresponding cooling coefficient is determined by pressing a button on the instrument and reading a value from the microvoltmeter. The cooling coefficients should be corrected to a common temperature (e.g., 25°C) using relationship recommended by the psychrometer manufacturer, and an average for each psychrometer should be calculated.

Psychrometer response is affected by cleanliness of the measuring thermocouple. Deposits of salt or organic matter on the thermocouple reduce measurement accuracy and prolong reequilibration time required between measurements. The measuring thermocouple should be cleaned with a zero-residue organic solvent and several rinses of distilled-deionized water. The psychrometer should be dried thoroughly by blowing clean air over its surface and/or allowing it to set overnight in a clean environment.

Psychrometers are then sealed within calibration chambers containing a salt solution with a known osmotic potential. The volume of solution used should be adequate to completely cover the bottom of the chamber to ensure that changes in salt concentrations resulting from subsequent evaporation of water during the equilibration process will not be significant. Calibration chambers containing psychrometers are

then submerged in a constant temperature water bath. Thermal and vapor conditions are then allowed to equilibrate within the chambers. The proper equilibration duration is determined by making a series of measurements over a period of several days until sequential measurements produce the same readings.

Figure A.1 illustrates results of psychrometric and dewpoint measurements over a 48 hour period following placement of four initially dry screen cage psychrometers in individual calibration chambers containing a 25 bar NaCl solution. Results suggest that a minimum of 24 to 48 hours may be required for initial equilibration using this type of psychrometer and calibration chamber. Less time is required following a subsequent adjustment of the water bath temperature or a change in the salt solution within the chamber. The smaller, commercially available chambers required much shorter equilibration periods (2 to 4 hours).

Dewpoint measurements can then be made following detailed procedures specified in the instruction manual for the particular microvoltmeter-control unit being used. The procedures typically include:

- 1) Measuring the calibration chamber temperature using the reference electrode located within the psychrometer base;
- 2) Calculating and setting the appropriate cooling coefficient;
- 3) Zeroing the microvoltmeter;
- 4) Applying a cooling current to the measuring thermocouple for a fixed interval of time; and
- 5) Recording the microvolt reading corresponding to the dewpoint plateau.

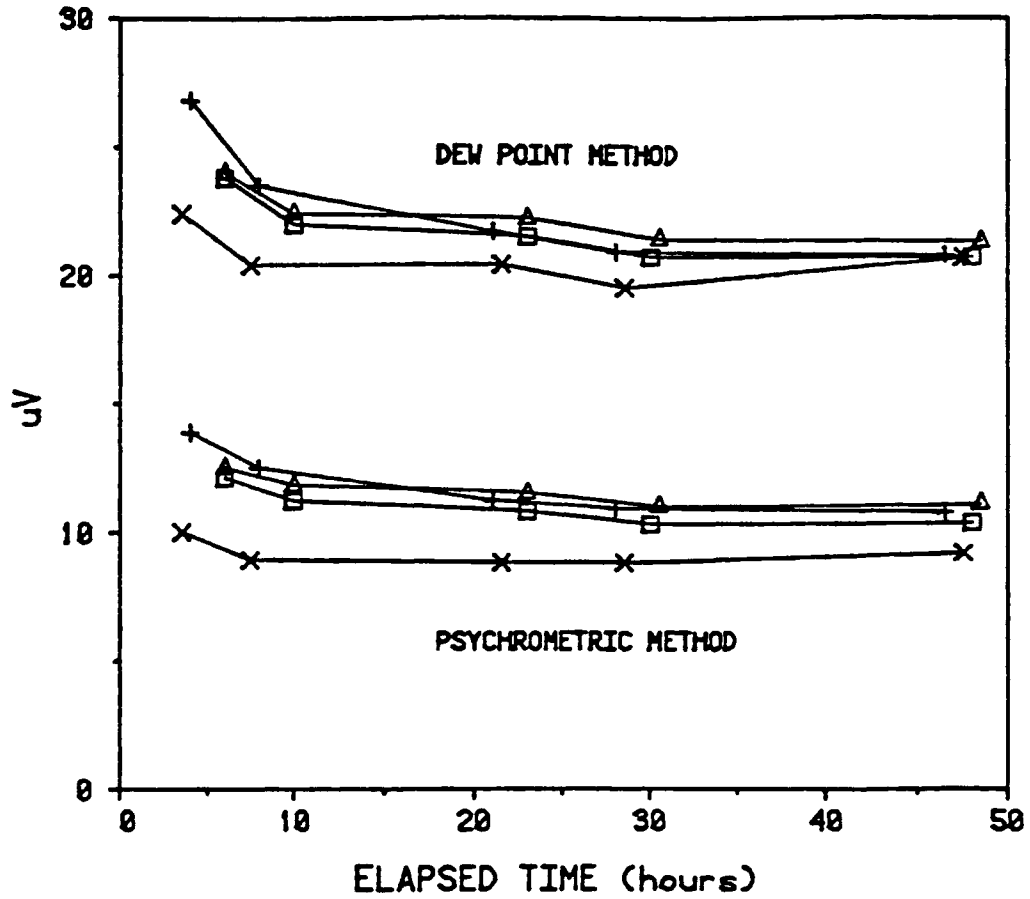


Figure A.1 Equilibration curves for four initially dry screen cage psychrometers placed in calibration chambers containing a 2.5 MPa NaCl solution.

Three of four readings should be obtained using each psychrometer for each solute-temperature combination. The cooling current is applied for 5 to 6 seconds for potentials in the range of -2 to -5 bars, 10 to 15 seconds for potentials near -25 bars, and 30 to 40 seconds for potentials near -50 bars. If the cooling current is too short, the measuring thermocouple surfaces will not be thoroughly wetted and reproducibility of measurements will be poor.

After all psychrometers have been calibrated for a given temperature-osmotic potential combination the water bath temperature should be reset at a new value. Measurements should be made at 3 or 4 different temperatures near the range of values likely to occur in the field situations.

When all the measurements are complete, remove the calibration chambers from the water bath, carefully open the chambers, discard the salt solution, thoroughly clean, rinse and dry the chambers, fill the base with the next solution, and reassemble and submerge the chambers. Repeat these procedures 3 or 4 times using salt solutions having osmotic potentials adequately covering the potential range from about -2 to -50 bars. If four solutions and four temperature values are used, then the resulting 16 data points will adequately define a calibration curve for each psychrometer.

A.1.2. Psychrometric Method

Measurements made using the psychrometric method do not require the determination or setting of cooling coefficient. Calibration steps outlined in the preceding section otherwise remain valid for the psychrometric method. The only difference is the measurement of

interest is the microvolt plateau corresponding to depression of the thermocouple temperature caused by evaporation of water from the thermocouple surface. Refer to the appropriate instruction manual for more detailed measurement procedures.

A.1.3. Combined Method

Dewpoint and psychrometric measurements can be made sequentially with some microvoltmeters simply by switching the meter controls to the psychrometric measurement position following completion of the dewpoint measurement. Use of the combined method is recommended for field measurements because it provides two estimates of moisture potential for a single procedure.

A.2. Data Analysis

Computer programs have been developed (Meyn and White, 1972), to analyze calibration data in a three-dimensional sense. It is simpler, however, to combine response and potential data as a single parameter and to plot results against temperature on a two-dimensional graph. The later method has been utilized in this study and is summarized below.

- 1) Calculate mean dewpoint and psychrometric response value for each solution-temperature combination evaluated during the calibration procedure.
- 2) Calculate osmotic potential values (in bars) for each solution-temperature combination using the equations presented previously (Raoult's Law and Kelvin's Law).
- 3) Divide each mean dewpoint and psychrometric response value by the appropriate osmotic potential value.

- 4) For each psychrometer, plot all $\mu\text{V}/\text{bar}$ values against corresponding temperature values. Psychrometric method results should plot as a straight line. Dewpoint method results should plot nearly horizontally. Individual data points lying a significant distance away from the apparent dewpoint and psychrometric calibration lines should be ignored because they probably indicate that the calibration system had not reached temperature or vapor equilibrium. This situation could be caused by a leak in the calibration chamber or by insufficient equilibration times.
- 5) Calculate linear regression equations using temperature and $\mu\text{V}/\text{bar}$ values judged to be reliable in step (4). Use these equations to accurately plot the slope and intercept of the calibration lines for each psychrometer for the dewpoint and psychrometric methods.
- 6) Subsequently, the calibration lines derived in the previous steps can be used to calculate moisture potential values (in bars) corresponding to temperature and microvolt data gathered in the field.

The entire data analysis outlined in steps 1) to 6) can be readily accomplished using a computer, although an appropriate program was not developed for this research.

APPENDIX B

THERMOCOUPLE PSYCHROMETER SAMPLE CHANGER

The psychrometer SC-10A can be calibrated for measurement of water potentials in the dry range (below -70 bars) and also for water potentials near saturation (0 bars). The procedure requires the determination of a unique psychrometer constant for a particular thermocouple.

B.1 Calibration Procedure at Low Water Potential.

Calibration for the low water potential range is accomplished with saturated salt solutions. The solutions are made by adding an excess of the salt to water such that salt is visible in the solution even after a few days of equilibration. Water potentials and water activities (or relative humidity) of several saturated salt solutions are given in Table B.1. Calibration can be accomplished with a single solution, but additional solutions provide a check and increase confidence in the measurement.

Select at least one salt which is near the range of water activity-potential of the samples and prepare the saturated salt solution. Fill one or more cups about half full (about 1 ml) with the salt solution and place them in the sample changer. Be sure that no solution remains on the top or outside surfaces of the cup. Carefully rotate the selector knob to avoid splashing the solutions. After the calibration solutions are loaded, put distilled water in the cup in position zero.

Once all of the samples are loaded, wait 15-20 minutes for temperature equilibrium and then rotate the selector to position zero and raise the water reservoir cup to wet the thermocouple. Lower the cup, rotate the selector to the calibration solution and seal the chamber. If the chamber humidity is lower than the humidity in the sample changer (generally the case for low activity solutions) the microvolt reading will increase to a maximum, hold that value for a time and then decrease to zero as water disappears from the thermocouple. A reading is taken at the highest steady value obtained. Repeat the wetting and reading procedure for each calibration solution.

B.2. Calibration Procedure at High Water Potential

The procedure already presented for measurement of low water potentials can generally be applied for measurement of high water potentials using calibration solutions from Table B.2. For higher sample humidities, the microvolt reading starts high and decreases exponentially toward a steady value, which it generally attains within 5-10 minutes. After reading the steady microvolt output, determine and record the chamber temperature. Repeat the wetting and reading procedure for each calibration solution and record the microvolt readings and chamber temperatures.

An alternative calibration procedure for high water potentials consists of obtaining a calibration curve such as the one shown in Figure B.1, using KCl or NaCl solutions given in Table B.2. The calibration curve is useful for converting microvolt readings to water potential. The scatter of the points from a line will give some idea of

Table B.1 Water potentials and water activities of selected saturated solutions at 20°C. To find water activities at other temperatures, see Greenspan (1975).

Salt Solution	Water Potential kJ/kg or MPa	Water Activity
LiCl	-294.35	0.113
MgCl ₂	-149.38	0.331
Mg(NO ₃) ₂	-82.24	0.544
NaCl	-37.99	0.755
KCl	-21.77	0.851
KNO ₃	-7.47	0.946

Table B.2 Water potentials and water activities of KCl and NaCl solutions at 20°C. To find water potentials at other temperatures use $WP_T = WP_{293} T/293$

Concentration Moles/kg	Bars or J/kg x 10 ⁻²		Water Activity	
	NaCl	KCl	NaCl	KCl
0.05	-2.32	-2.32	0.99828	0.99828
0.10	-4.54	-4.52	0.99664	0.99666
0.02	-9.01	-8.88	0.99335	0.99344
0.30	-13.49	-13.26	0.99006	0.99023
0.40	-17.93	-17.60	0.98681	0.98705
0.50	-22.42	-21.90	0.98353	0.98391
0.60	-26.99	-26.22	0.98021	0.98076
0.70	-31.59	-30.61	0.97687	0.97758
0.80	-36.18	-35.01	0.97356	0.97440
0.90	-40.87	-39.31	0.97018	0.97130
1.00	-45.58	-43.72	0.96680	0.96813

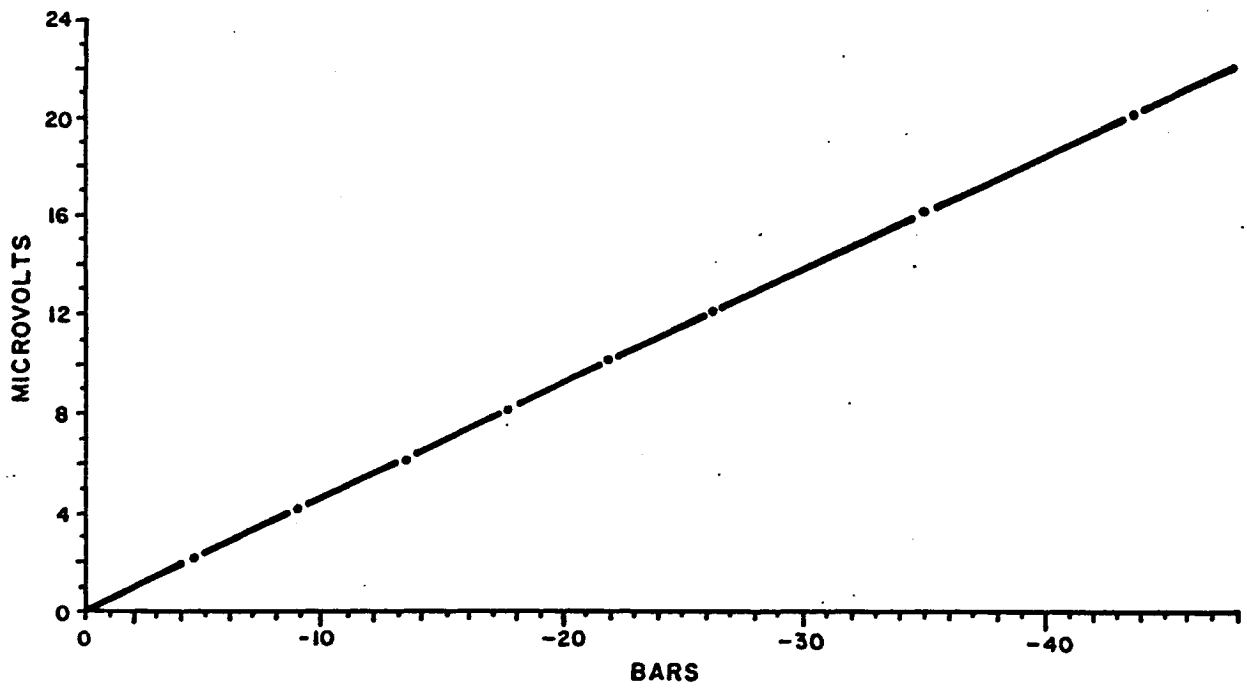


Figure B.1. Calibration curve for high water potential readings using various concentrations of KCl at 20°C.

how accurate the readings on unknown samples are likely to be. In addition to the calibration curve, it is often useful to run standards along with samples in order to verify the reliability of the readings. For a typical calibration, a minimum of 5 solutions are required at concentrations of 0.1, 0.3, 0.5, 0.7, and 1.0 molal.

Include a distilled water sample to give a zero reading. Table B.2 gives the water potentials of KCl and NaCl solutions of various molalities. For potentials not shown, linear interpolation will give values with sufficient accuracy.

Best accuracy is obtained at high water potential if the walls of the sample cups are covered with the solution. This is accomplished by cutting filter paper strips 12mm x 45mm and placing them around the walls of the cups. Use only enough calibration solution to wet the filter paper and cover the bottom of the cup.

Relatively small errors in solution concentration which result from evaporation of water from the solution in the psychrometer cup or transfer of water or salt from one solution to another in the dropper can cause measurable errors in the psychrometer calibration. Once all of the calibrating solutions have been loaded, fill the small well in the water source for the measuring junction, and allow 20 to 30 minutes for temperature equilibration. During equilibration, the selector should be set so that the thermocouple is over the driest sample to minimize the possibility of condensation on the thermocouple mount.

When temperature equilibrium has been achieved, turn the selector to position zero and lift the lever to place water on the measuring junction. Lower the lever and turn the selector to position

1, and seal the chamber by lifting the lever. The microvolt meter reading should decrease rapidly, and finally reach a constant value. At water potentials in the 0 to -4 bar range (water activities in the 1 to 0.99704 range), a constant reading may take ten minutes or longer. At lower water potentials, 2 to 3 minutes should be adequate.

The thermocouple should be rewet between readings, however, at high water potentials the thermocouple will generally hold enough water for several readings. Repeat this procedure for all of the calibration solutions.

Calibration data should be plotted as shown in Figure B.3. The results should lie on a smooth line over the entire range of water potentials, however, departures from a smooth line indicate problems either in calibration procedure or in the equipment.

For very careful work, calibration should be checked against one or two known potential activities each time a sample is run. The psychrometer is sensitive to temperature changes; the psychrometer output increases about 2.5 percent per degree as temperature increases.

B.3 Calibration Equations and Curves

A calibration curve, such as Figure B.1, for high activity solutions, can be produced from the microvolt readings and used to convert microvolt measurements to water potential for a particular temperature. However, it is more convenient to use the psychrometer equation. To use the equation, a psychrometer constant must be determined for the psychrometer.

The psychrometer constant (g) is calculated from:

$$g = e'_w - a_w e' / (T_a - T_w) \quad (\text{B.1})$$

where $T_a - T_w$ is calculated from the microvolt reading, assuming that the thermocouple output is $60 \mu\text{V}/\text{C}$; a_w is the water activity (relative humidity) of the calibration solution; and e' is the saturation vapor pressure, calculated from:

$$e' = \exp(52.57633 - 6790.4985 / T_k - 5.02808 \ln(T_k)) \quad (\text{B.2})$$

where T_k is the Kelvin temperature read during calibration.

A simple calibration program can be used to calculate the psychrometer constant (g) as well as the water activity and water potential of a sample. To calculate the water activity or relative humidity of a sample, use the equation:

$$a_w = e'_w - g(T_a - T_w) / e' \quad (\text{B.3})$$

The water potential is related to the water activity (i.e., relative humidity) by the equation:

$$\text{WP} = R T / M \ln(a_w) \quad (\text{B.4})$$

where WP is the water potential; R is the gas-constant; T is the Kelvin temperature; M is the molecular weight of water; and a_w is the water activity of the sealed chamber.

REFERENCES

- Al-Khafaf, S. and R.J. Hanks, 1974. Estimation of the filter paper method for estimating soil water potential. *Soil Sci.*, 117:194-199.
- Brown, R.W. and R.S. Johnston, 1976. Extended field use of screen covered thermocouple psychrometers. *Agron. J.*, 68:995-996.
- Campbell, E.C., G.S. Campbell and W.K. Barlow, 1973. A dewpoint hygrometer for water potential measurement. *Agr. Meteor.* 12:113-121.
- Campbell, G.S., W.D. Zollinger, and S.A. Taylor, 1966. Sample changer for the thermocouple psychrometer: construction and some applications. *Agron. J.*, 58:315-318.
- Daniel, D.E., J.M. Hamilton, and R.E. Olsen, 1981. Suitability of thermocouple psychrometers for studying moisture movement in unsaturated soils. Permeability and groundwater contaminant transport, ASTM STP 746, T.F. Zimmis and C.O. Riggs, Eds, Am. Soc. for Testing and Materials, p. 84-100.
- Fawcett R.G. and N. Collis-George, 1967. A filter paper method for determining the moisture characteristics of soil. *Aust. J. Exp. Agric. Anim. Husb.*, 5:201-205.
- Gardner, Robert, 1937. A method of measuring the capillary tension of soil moisture over a wide moisture range. *Soil Sci.*, 43:277-283.
- Gardner W. et. al., 1922. The capillary potential function and its relation to irrigation practice. *Phys. Rev.*, 20:196.
- Gradmann, H. 1934. Uber die Messung von Bodensaugwerten. Cited in McQueen I.S. and R.F. Miller, 1968. *Soil Sci.*, 106:225-231.
- Hamblin, A.P., 1981. Filter-paper method for routine measurement of field water potential. *J. Hydrol.*, 53:355-360.
- Hansen, H.C., 1926. The water-retaining power of the soil. *J. Ecol.*, 14:111-119.
- Hunter, A.S. and D.J. Keeley, 1946. Changes in the construction of soil moisture tensiometers for field use. *Soil Sci.* 61:215-218.
- Kemper, W.D. and N.A. Evans, 1963. Movement of water as effected by free energy and pressure gradients: III. Restriction of solutes by membranes. *Soil Sci. Soc. Am. Proc.*, 27:485-490.

- Klute, A. and D.B. Peters, 1962. A recording tensiometer with a short response time. *Soil Sci. Soc. Am. Proc.*, 26:87-88.
- Long, F. Leslie, 1984. A field system for automatically measuring soil water potential. *Soil Sci.*, 137:227-230.
- Marthaler, H.P., W. Vogelsanger, F. Richard, and P.J. Wierenga, 1983. A pressure transducer for field tensiometers. *Soil Sci. Soc. Am. J.*, 47:624-627.
- Marvil, Joshua D., June 1986. USGS Denver Colorado, personal communication.
- McQueen, I.S. and R.F. Miller, 1968. Calibration and evaluation of a wide range of gravimetric method for measuring moisture stress. *Soil Sci.*, 106:225-231.
- Merril, S.D. and S.L. Rawlins, 1972. Field measurement of soil water potential with thermocouple psychrometers. *Soil Sci.* 113:102-109.
- Meyn, R.L. and R.S. White, 1972. Calibration of thermocouple psychrometer: A suggested procedure for development of a reliable predictive model. pp. 56-63, in R.W. Brown and B.P. van Haveren (eds.), Psychrometer in Water Relations Research. Utah State University, p.342.
- Peck A.J. and R.M. Rabbidge, 1966. Soil-water potential: Direct measurement by a new technique. *Science*, 151:385-386.
- Peck A.J. and R.M. Rabbidge, 1969. Design and performance of an osmotic tensiometer for measuring capillary potential. *Soil Sci. Soc. Am. Proc.*, 33:196-202.
- Rahi, K.A., 1986. Hydraulic conductivity assessment for a variably-saturated rock matrix. University of Arizona, Masters thesis.
- Rasmussen, T.C. and D.D. Evans, 1986. Unsaturated flow and transport through fractured rock-related to high level waste repositories: Phase II, U.S. Nuclear Regulatory Commission Report, NUREG/CR-4655.
- Rice, R., 1969. A fast-response, field tensiometer system. *Trans. Am. Soc. Agric. Engr.*, 12:48-50.
- Richards, L.A., 1947. Pressure membrane apparatus, construction and use. *Agric. Engr.* 28:451.
- Richards, L.A., 1949. Methods of measuring soil moisture tension. *Soil Sci.*, 68:95-112.

- Richards, L.A. and W. Gardner, 1936. Tensiometer for measuring capillary tension of soil water. *Am. Soc. Agronomy J.*, 28:352-358.
- Richards, L.A., and G. Ogata, 1958. Thermocouple for vapor pressure measurement in biological and soil systems at high humidity. *Science*, 128:1089-1090.
- Richards, S.J., 1965. Soil suction measurements with tensiometers, pp. 153-163, in C.A. Black (ed.), *Methods of soil analysis*, Am. Soc. of Agronomy, Madison, WI.
- Spanner, D.C., 1951. The peltier effect and its use in the measurement of suction pressure. *J. Exp. Botany*, 2:145-168.
- Thomas, M.D., 1921. Aqueous vapor pressure of soils. *Soil Sci.* 11:409.
- Watson, K.K., 1965. Some operating characteristics of a rapid response tensiometer system. *Water Resour. Res.* 1:577-586.
- Wescor Inc., 1979. Instruction manual for Wescor HR-33T dew point microvoltmeter, Wescor Inc., Logan Utah.
- Wiebe, H.H., G.W. Campbell, W.H. Gardner, S.L. Rawlins, J.W. Cary, and R.W. Brown, 1971. Measurement of plant and soil water status, Bulletin 484, Utah Agri. Exp. Stn., Logan, Utah.
- Williams, J. and C.F. Shaykewich, 1969. An evaluation of polysthylene glycol (P.E.G.) 6,000 and P.E.G. 20,000 in the osmotic control of soil water potential. *Canadian J. Soi. Sci.*, 49:397-401.
- Williams, O.B. and R.H. Sedgley, 1965. A simplified filter paper method for determining the 15-atmosphere percentage in soils. *Aust. J. Exp. Agric. Anim. Husb.*, 5:201-205.
- Williams, T.H. Lee, 1978. An automatic scanning and recording tensio meter system. *J. Hydrol.*, 39:175-183.
- Zur, B., 1966. Osmotic control of the matric soil-water potential I. Soil-water system. *Soil Sci.*, 102:394-398.