

FINAL REPORT

MEASUREMENT OF TRANSIENT
MOISTURE GRADIENTS IN
CONCRETE SLABS

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Joint
Highway
Research
Project

by

J.R. BELL

PURDUE UNIVERSITY
LAFAYETTE INDIANA

Final Report

MEASUREMENT OF TRANSIENT MOISTURE GRADIENTS IN CONCRETE SLABS

TO: K. B. Woods, Director January 30, 1963
Joint Highway Research Project

FROM: H. L. Michael, Associate Director File: 9-7-3
Joint Highway Research Project Project: C-36-63C

Attached is a report entitled, "Measurement of Transient Moisture Gradients in Concrete Slabs" by J. R. Bell, research engineer on our staff. This is the final report on this research project and is a summary report of the research which has been conducted on the project during the past five years.

The first seven pages of the report are a summary of the findings and results. Although no moisture meter suitable for measuring transient moisture gradients in concrete pavement by non-destructive means was found for all conditions, a wealth of knowledge on the subject of this research was obtained. The dielectric properties of concrete were found to be potentially accurate indicators of moisture content in concrete over a sufficient range of ambient conditions to be of considerable value in studying the behavior of concrete pavements.

It is anticipated that this final report will also be a paper which will be submitted for publication, subject of course to review and approval of the participating groups. The report is submitted for review and approval for publication.

The submission of this report and its acceptance by all the parties to the research will terminate this research project.

Respectfully submitted,

Harold L. Michael
Harold L. Michael, Secretary

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Final Report

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GRADIENTS IN CONCRETE SLABS

by

J. R. Bell
Research Engineer

Joint Highway Research Project
Project No: C-36-63C
File No: 9-7-3

Purdue University
Lafayette, Indiana

January 30, 1963

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Special acknowledgment is due Dr. G. A. Leonards, Professor of Soil Mechanics, Purdue University, who guided this investigation and reviewed the manuscript.

Many individuals contributed to this project and to each of those the author expresses his deepest appreciation.

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SUMMARY

The investigations summarized in this report are the results of a study sponsored jointly by the United States Bureau of Public Roads, the Indiana State Highway Department, and the Joint Highway Research Project of Purdue University, to develop a moisture meter suitable for measuring transient moisture gradients in concrete pavements by non-destructive means. Specifically, it was desired that a meter suitable for use in connection with investigations of the moisture-volume relationship and the resulting warping of concrete pavements be developed.

The study was initiated in March 1958 with the first phase having the following objectives:

- (1) A literature survey to compare the available methods of measuring the moisture content of porous media by non-destructive means,
- (2) limited laboratory investigations of the more promising of these methods, and
- (3) selection of the most promising methods for intensive investigation in the second phase of the study.

On the basis of the literature review it was concluded that, of the methods surveyed, capacitance measurements were inherently the most promising and it was decided to investigate this procedure in the laboratory.

The initial laboratory tests consisted, for the most part, of measuring the capacitance of thin mortar disks at various water contents in a parallel plate capacitor by means of a low frequency (60 cps) AC capacitance bridge. The results showed these measurements to be sensitive

to changes in water content only at water contents below about 2 percent. From data available in the literature, and from the companion volume-water content tests made for this study, it was concluded that most of the volume change due to shrinkage in concrete mixes used for highway pavements occurred at water contents below about 4 percent, and that the desirable moisture content range to be measured should be up to 6 percent. Therefore, from the standpoint of obtaining either an absolute measure of water content, or of developing an indirect procedure for measuring volume changes by capacitance measurements, the results showed an apparent lack of sensitivity at higher water contents. The data and conclusions from this initial phase of the study were reported in Progress Report No. 1 (1)*.

A study of the theory of capacitance measurements did not indicate any serious inherent limitations to the capacitance measurement method. It was concluded, therefore, that the difficulties encountered were the result of instrumentation problems. For the most part, the instrumentation problems stemmed from the fact that the apparent dielectric constant as indicated by capacitance measurements is not a true constant. It is a function not only of the material being tested, but also of the test conditions. The most important factors influencing the test results are boundary conditions and the electrical frequency at which the test instruments are operated. A troublesome boundary condition is that of obtaining good contact between the capacitor plates and the dielectric material being tested. Poor contact lowers the sensitivity of the measurements. The problems associated with frequency of operation are more complex. The test frequency controls the effects of such factors as conductance, polarization, and force fields acting on the water molecules.

*Numbers in parentheses refer to the Bibliography at the end of this report.

The influences of conductance are reduced as frequency is increased, tending to increase the effective range, but the other factors tend to reduce the sensitivity. The problem, therefore, is to balance the physical size and shape of the capacitor and the frequency and sensitivity of the measuring instrument to the electrical properties of the dielectric - in this case, moist concrete.

To test the feasibility of designing apparatus of suitable range and sensitivity, a second series of capacitance tests was performed at a higher electrical frequency (600,000 cps). At this frequency the range in moisture content that could be measured increased without seriously reducing the sensitivity. The capacitance measurements were subject to changes in the structure of the concrete with time and reflected the sorption hystereses in the wetting-drying cycle; however, the test results showed that the inaccuracies resulting from these causes were not prohibitive (these factors also limit the accuracy of other indirect moisture measuring methods). The results of the higher frequency tests showed that for a well-cured mortar the capacitance method could indicate moisture contents with an accuracy of ± 0.5 percent (moisture content) for water contents between 1 percent and 6 percent. This range brackets the moisture contents that are of interest from the standpoint of moisture-volume relationships in concrete highway slabs.

From these results, which were reported in Progress Report No. 2 (2), it was concluded that the dielectric constant is a satisfactory indicator of the water content of hardened concrete and that the capacitance method was worthy of an intensive study to develop/instrumentation necessary to permit routine insitu moisture measurements. Accordingly, the second phase of the study was undertaken with the purpose of

developing and testing the necessary measuring instruments, sensing elements, and procedures to allow insitu field testing of the moisture content of concrete pavement slabs.

A 10^6 cps General Radio Capacitance Assembly and accessory equipment was acquired, and a test capacitor was designed, built, and calibrated. With this equipment it was possible, for the first time, to determine the actual dielectric constant-water content relationship for concrete. All previous work had been with the apparent capacitance-water content relationship, which is a function not only of the material but also of the test. Work was also started on the design and testing of an appropriate sensing element for embedment in the concrete test section.

Preliminary tests with this equipment indicated several important problems. First, the sensing element and field instrumentation design required a prior knowledge of the dielectric properties of concrete, which was not available either in the literature or from the initial testing program. Secondly, it was discovered that the dielectric constant of concrete is a function not only of the water content but also of the conductivity of the pore water. This latter discovery was very disturbing, because one of the reasons for selecting the capacitance method for detailed study was a supposed freedom from large influences due to variations in the salt content of the pore fluid. This development raised serious doubts concerning the validity of the conclusions drawn from the initial phase of the study. It was now evident that in spite of the results presented by previous investigators, the conclusions drawn from the simplified theory used to interpret the test data, and the test results obtained in the initial phase of the study, that the capacitance method had inherent limitations which would permit reliable measurements

of water content only within a restricted range of ambient conditions. The data that provide the basis for this conclusion were reported in Progress Report No. 3 (3).

At this juncture the entire investigation was re-evaluated. While there was no longer any doubt concerning the restricted range of applicability of the capacitance method, it was by no means established whether or not this range fell within that which is of interest in the pavement design problem. Considering the investment of time and material that had already been made, it was concluded that the practical range of applicability of the capacitance method should be delineated. For this purpose it was necessary to:

- (1) develop a more rigorous theory for interpreting the test data,
- (2) isolate the factors which influence the dielectric properties of hardened concrete, and
- (3) establish the range over which these factors could vary so that the precision of the capacitance method would still yield results of practical interest.

The final phase of the study was divided into two parts. The first part consisted of an extension of the theory of high loss dielectrics. The second part was a laboratory study of the dielectric constant-water content and dielectric conductivity-water content relationships for one mortar and one concrete mix through ten cycles of drying and wetting. In those tests, one half of the samples was oven dried to 105°C and the other half was dried to 60°C. All dielectric constant determinations were from permittivity tests made at a frequency of 10⁶ cps. Dielectric conductivity was determined both from direct

resistance measurements at 10 cps and from the 10^6 cps permittivity tests. Special tests were conducted to indicate the influence of the salt concentration of the pore fluid on these relationships. Procedures and equipment used in these tests were selected on the basis of their suitability for adaptation to field studies if the laboratory tests indicated that field applications were justified. The results of these investigations were presented in Progress Report No. 4 (4). It was shown that the relationships between water content and the dielectric properties of Portland cement concrete and mortar are not constant relationships. They are strongly influenced by the electrical frequency at which they are measured, the salt content of the pore water, and the conditions under which drying and rewetting take place.

The extremes of the variations of these relationships are sufficiently large to make electrical moisture meters unsuitable for all possible environmental conditions. In this study, large variations occurred only when thin specimens were dried to 105°C or where the NaCl concentration of the pore water was increased to 1.0 Normal. When thicker samples (3-inch disks approximately $\frac{1}{8}$ inch thick) were dried to only 60°C and salts were not added, the variations were minor. This is true for at least ten cycles of drying and rewetting with distilled water. For these conditions, permittivity measurements at 10^6 cps are potentially capable of indicating moisture contents in concrete within ± 0.25 percent up to water contents of 6 to 8 percent. Such accuracies would be adequate for many purposes, and conditions comparable to drying at 60°C and no large increases in salt concentration of the pore water are frequently encountered in practice. However, corrections would have to be made for increases in the dry weight of concrete with aging.

Dielectric conductivity-water content relationships determined by low frequency resistance measurements indicated variations of ± 0.5 percent up to water contents of 6 to 8 percent for the conditions of 60°C maximum temperature and ten cycles of wetting and drying. There are indications that this greater variation is due, at least partially, to the test conditions and would not occur within a larger test section. Because of the simplicity of this method it should not be discarded until all possibilities for its use have been explored.

The dielectric properties of concrete are potentially accurate indicators of moisture content in concrete over a sufficient range of ambient conditions to be of considerable value in studying the behavior of concrete pavements. For these methods to be successful, the instrumentation must be carefully designed to suit the specific dielectric properties of concrete, due consideration being given to dielectric constant, dielectric conductivity, frequency, and the electrical characteristics of the contact between sensing element and concrete.

The following sections of this report present a summary of the results of the several investigations conducted as a part of this study. To avoid obscuring the main trends of the report, brief descriptions of equipment and test procedures and only typical test data are presented. For complete results and detailed descriptions the reader is referred to the Progress Report where this information is contained.

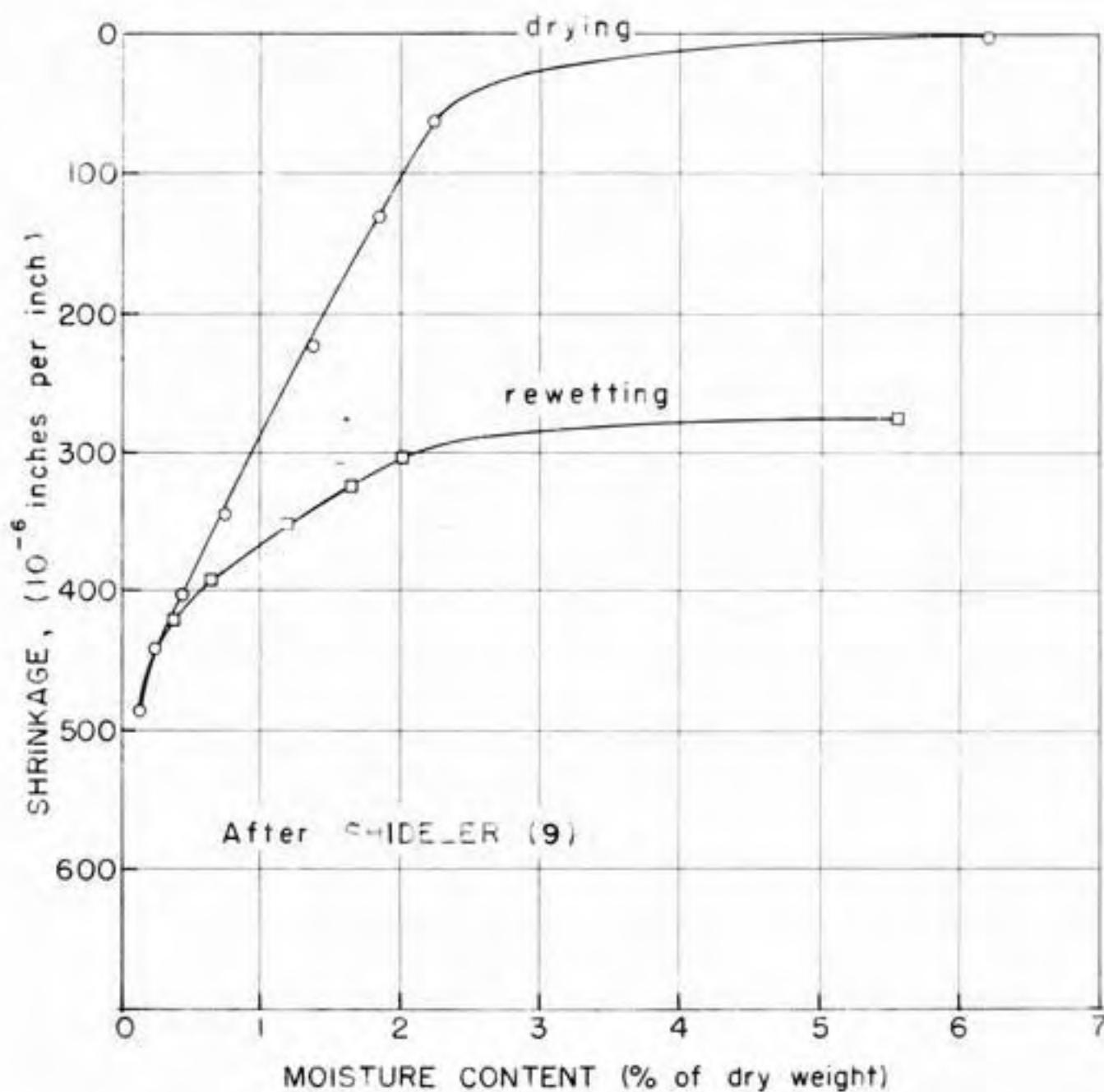
REVIEW OF LITERATURE

Moisture-Volume Relationships of Concrete

There is an extensive literature on water in concrete and its effect on the properties of concrete; however, much of the published information is qualitative in nature because reliable methods of measuring water content in non-destructive tests have not been available (5, 6). The published data are difficult to interpret because in many cases the descriptions of test conditions are incomplete. This has resulted in many apparent discrepancies; however, there are a few general trends, which are discussed below.

Menzel (7) states: "With all of the different types of concrete, whether rich or lean, dense or porous, etc., significant shrinkage begins at a moisture content corresponding roughly to the equilibrium value that would ultimately be attained with exposure to a 99 to 100 percent relative humidity". Therefore, much of the water may be removed before appreciable shrinkage occurs, which limits the moisture content range of interest in this study to relatively low water contents. This statement is borne out by tests on masonry blocks by Kalousek (8) and Shideler (9). A plot of shrinkage vs. water content prepared from Shideler's data is shown on Figure 1. The data show that nearly all of the volume change occurred at water contents below 2 to 3 percent (of the dry weight). This same general trend is shown by Yoshida (10) for cement paste; however, in the absence of aggregates, the shrinkage begins at somewhat higher water contents. The shrinkage data obtained in this study (Figures 6-10) further confirm these facts.

FIG.1.- SHRINKAGE vs. WATER CONTENT
FOR CONCRETE MASONRY UNITS.



The above data are not for concrete mixes used in highway pavements; however, they consistently show that nearly all shrinkage occurs at moisture contents lower than that corresponding to about 40 percent of total absorption as determined by ASTM Designation C140-52. The results of tests by Blackman (11) show that total absorption for highway pavements would almost certainly correspond to water contents of less than 10 percent by dry weight. This indicates that the most critical moisture content range is below 4 percent by dry weight. Teller and Sutherland (6) found by gravimetric tests the average moisture content of a slab at the Arlington Test Road to be 3.5 percent in the summer and 3.8 percent in the winter. Thus, the range in water content of interest in this study was taken as 0-6 percent.

Theoretical considerations by Harr and Leoniards (12) indicated that it is desirable, with respect to slab warping studies, to measure the water content with sufficient accuracy to indicate length changes resulting from moisture variations equivalent to a temperature change of about 5°F. Hveem (13) has shown that changes in the moisture content of 30 oven-dried thin concrete disks, sawed from cores from pavements, which were soaked for seven days resulted (for 2/3 of the specimens) in length changes greater than those resulting from a 90°F. temperature increase. Also, a study of Shideler's data (Figure 1) shows that in the critical range a 2 percent moisture increase results in a volume increase equivalent to a temperature increase of 300°F or more. Thus, a sensitivity of about 0.25 percent (moisture content) can be considered a desirable objective.

Many factors other than water content influence the volume-water content relationship of concrete. Some of these factors are

mix design, type and size of aggregate, type of curing, age, cycles of wetting and drying, carbonation, and others. (7, 8, 9, 10, 14). For these reasons it was decided to discard the possibility of obtaining direct correlations between volume change and any indirect measurement of water content. Rather, it was decided to attempt the development of a general procedure for measuring the transient variations in water content; in any given application, the corresponding volume changes would be obtained by calibration for the specific conditions of interest.

Moisture Measuring Methods - General

Literature on the subject of moisture measuring techniques is extensive and there are several general reviews and bibliographies available (15, 16, 17). Many of the methods have been applied to concrete, soils, and a variety of other materials with widely varying degrees of success. The results are confusing, as one author will report a method to be reliable while another reports it as less than worthless. Therefore, in this study an attempt was made to evaluate the various methods on the basis of their relative inherent advantages and disadvantages and not solely on their reported reliabilities.

The definition of water content, as used throughout this paper, is the ratio of the weight of water lost upon drying to constant weight at 105°C to the weight of solids at this temperature. Direct gravimetric determinations of transient moisture gradients is unsatisfactory for the purposes of pavement warping studies because it requires destruction of the concrete slab during testing. A possible modification would be to construct the slab with removable segments which would periodically be weighed, oven dried, weighed and returned to the slab. This would cause discontinuities in the slab, could provide avenues for the entrance of water, would not give a continuous record because a

long time delay would be required before equilibrium was re-established after the segment was replaced, and the oven drying would alter the structure of the segment so that it would not remain the same as the rest of the slab.

Indirect methods measure some property of the concrete which is a function of water content and can be used for in-place, non-destructive measurements. These methods all present similar problems of range and sensitivity, the necessity of calibration, the speed of response to moisture changes, the effective zone of influence, determination of what the device actually measures, the effect of changes other than moisture (i.e., changes in density, temperature, structures, and salt concentration) degree of contact between the sensing element and the material being measured, the requirement that some object or cavity be placed in the concrete and, in some cases, the necessity for a hygroscopic material surrounding the sensing element which is supposedly in (moisture) equilibrium with the material being tested.

A variety of indirect methods are discussed and compared in Progress Report No. 1(1). Their relative advantages and disadvantages are summarized in Table I. It was concluded that the capacitance method was inherently the most promising, and it was decided to investigate this method in detail.

Capacitance Moisture Meters

In principle the capacitance moisture meter is a simple device. It is based on the fact that if electrodes having a geometric capacitance C_0 are embedded in a material with a relative dielectric constant k^* there capacitance will be^{*}

$$C = C_0 k^* \quad (1)$$

*The notation is given on Page 82

TABLE I - COMPARISON OF MOISTURE MEASURING METHODS

Method	Disadvantages	Advantages
Resistance Method	<ol style="list-style-type: none"> Influenced by small variations in composition of pore water Some methods require hygroscopic materials which increases response time and limits range 	<ol style="list-style-type: none"> Very simple and inexpensive
Ionic Barrier Resistance Method	<ol style="list-style-type: none"> Hygroscopic material required (increased response time and limited range) Life of Ionic membranes unknown 	<ol style="list-style-type: none"> Free of effects of pore water composition Range of sensitivity may be controlled
Relative Humidity and Suction Methods	<ol style="list-style-type: none"> Range limited to lower water contents Cavities required in concrete slab Some methods require hygroscopic materials 	<ol style="list-style-type: none"> Relatively free of effects of changes in pore water composition Simple calibration procedure
Neutron Radiation Method	<ol style="list-style-type: none"> Detects all water present-combined and evaporable water Zone of influence relatively large Cavities required in concrete Complex expensive equipment Special instrumentation problems of designing small probe and of maintaining critical probe spacing 	<ol style="list-style-type: none"> Free of effects of changing pore water composition Wide range Relatively free of problems of contact between probes and materials being tested

TABLE I (cont) - COMPARISON OF MOISTURE MEASURING METHODS

Method	Disadvantages	Advantages
H. F. Radiation Method	1. Combines disadvantages of both the resistance and the capacitance methods	1. No special advantages
Sonic Radiation Method	1. Extreme precision of measurement required 2. Very sensitive to changes in structure (deterioration of the concrete)	1. Free of effects of changing salt concentration of pore fluid
Heat Transfer Method	1. Serious problems of contact between sensing element and porous material 2. Sensitive to changes in structure and density 3. Measurements cause moisture migration 4. Large zone of influence	1. Free of effects of changing pore water composition
Dielectric Constant (capacitance) Method	1. Dielectric constant of water a function of the force field acting on the water 2. Sensitive to degree of contact between the sensing elements and the material being tested 3. Special instrumentation problem - the measured dielectric constant is a function of the electrical frequency of the measuring instruments	1. Relatively free from effects of small changes in composition of pore fluid 2. Rapid response to changes in water content 3. Small zone of influence - well adapted to measurement of moisture gradients

The capacitance moisture meter consists of a sensing element which is a suitably calibrated capacitor employing the material whose water content is desired as the dielectric and a device for measuring the capacitance of this capacitor. Equation 1 permits the determination of the relative dielectric constant of the material from these measurements. The relative dielectric constant of a material such as concrete is a function of its water content, and if the dielectric constant-water content relationship of the material is known, the water content can be determined from the capacitance measurement.

Such a moisture meter apparently has several advantages. The instrumentation principle is simple; the sensing element is inert and does not depend on a moisture equilibrium between some hydrophilic material and the material being measured. The moisture is sensed by an electric field and therefore gives immediate response to transient moisture changes. Also, the relative dielectric constants of most solid minerals are in the range from 5 to 10 (18) while the value for water may be on the order of 80 (19), indicating that the method should have high sensitivity to small moisture changes.

Two important assumptions are implied in the capacitance method of measuring moisture contents. First, it is assumed that the capacitance of the sensing capacitor can conveniently be measured in such a way as to reflect the true dielectric constant of the material being studied and second, it is assumed that the dielectric constant-water content relationship is constant for the conditions of the investigations and can readily be determined by some appropriate calibration procedure. To evaluate the potential of this method in a given set of circumstances, the validity of these assumptions must be established for the conditions of the particular situation.

Reference could not be found in the literature to the study of the capacitance method relative to concrete, but the apparent advantages of this method have led numerous investigators to study its application to moisture determination in a wide variety of other materials including soils, soybeans, wheat, cotton, gelatin, paper pulp, carrots, and beef steak.

One of the most striking characteristics of these researches is the lack of consistency in the results. For example, Balls (20) reports excellent results with cotton and found an almost linear relationship between water content and measured capacitance, while Lill (21), working with soils, was unsuccessful primarily due to an inability to balance out or otherwise compensate for stray capacitances and the effects of the conductivity of the soil. Lill used a tuned oscillator circuit instead of a capacitance bridge which most other investigators have used to measure the capacitance. He attributes most of his instrumentation problems to this fact, and suggests that a "Schering" type bridge would have been superior. Lill also used insulated electrodes and noted that the cell was very sensitive to the degree of contact with the soil. He further states that the cell was more sensitive to changes of the conductivity of the soil than to capacitance changes. Most of the other investigators claim limited success, depending upon their purposes. The best results were generally obtained with the simpler materials such as gelatin and cotton, and the least success was achieved with the more complex materials, notably soils. The following discussion is restricted to studies on soils and rocks, unless otherwise noted, as these materials have more in common with concrete than do many of the others.

Balls (20), using a tuned oscillator and insulated electrodes, obtained relatively consistent results at a frequency of 2×10^6 cps with

soils. His data showed that the capacitance increased by a factor of 5 as the water content was increased from 0 to 60 percent.

DePlater (22), utilizing a bridge circuit at 1000 cps and bare electrodes, reports consistent results on a "clay loam" and indicates that the capacitance of his setup depended only on the water content of the soil. However, Cashen (23) measured the equivalent parallel capacitance and conductance of a series of soil blocks, and obtained results which depended on the electrodes used. With mercury electrodes, all soils gave capacitance-water content curves of the same general type, which he attributes to a large capacitance of the soil-electrode interface. Results using carbon electrodes were less consistent but showed the same general effects.

Wallihan (24), using a modification of the capacitance method incorporating a plaster of Paris block between the electrodes, obtained consistent results with insulated plates, but the use of non-insulated plates were unsuccessful where the electrical resistance between the plates was as low as 10 ohms. He also reports that the frequency of the alternating voltage used in the bridge apparently did not affect the results obtained with insulated plates. With non-insulated plates, however, low frequencies gave the highest readings. Baherjee and Joshi (25) who studied the dielectric constant of soil at radio frequencies also found that the dielectric constant decreases with an increase of frequency and that the relative dielectric constant increased from about 4 or 5 at a water content of 6% to a value of about 17 at a water content of 14% when measured at a frequency of 70×10^6 cps.

Cownie and Palmer (26), investigating the effect of moisture on the electrical properties of soil at a frequency of 430×10^6 cps using a coaxial transmission line terminating in clay samples as their test

cell, found that the relative dielectric constant varied from 4 to 31 as the water content was increased from 4 to 47%. These results compared favorably with data at very high frequencies reported by other investigators. They also found that the dielectric constant increased first at an increasing rate with water content and then at a decreasing rate at the higher water contents. This double inflection could not be predicted by simple mixing theories. They explain the phenomenon on the basis that the first water added to the soil (below about 15 to 20%) is strongly held by the force fields of the clay and, acting as bound water, has a dielectric constant of the same order as ice, while water above 20% acts as free water with a relative dielectric constant of about 80. Edlefsen (27), in discussing the dielectric method and summarizing the findings of several investigators, points out that a given quantity of water distributed evenly through a soil produces a greater change in the dielectric constant of comparatively dry soil than it does in the same soil when relatively wet, and contrary to the results of Cownie and Palmer, states that the relation is practically linear up to the moisture equivalent of the particular soil. Edlefsen also points out that the dielectric constant decreases, other factors being constant, as the temperature increases. This is supported by the fact that the dielectric constant of water decreases with increasing temperature (19). He also states that the dielectric constant of the soil water system is a function of the salt concentration of the pore water. Fletcher (28) also reports an influence of the salt concentration of the pore fluid at 3.9×10^6 cps but only for concentrations of less than 0.1 gm per 100 cc in the case of NaCl, which was the only salt he investigated. Addition of NaCl in excess of this amount had no additional effect. This point is confused rather than

clarified when free water is considered because Hasted (29), Smith-Rose (30), and Von Hippel (31) all indicate that the salt concentration of bulk water has only a small effect on its dielectric constant at frequencies in the low megacycle range. However, Hasted also presents data showing the salt concentrations greater than about 1 Normal significantly decrease the dielectric constant of water for frequencies greater than $1,000 \times 10^6$ cps.

Anderson (32) measured the capacitance of several soils as a function of water content using bare electrodes at a frequency of about 1,000 cps. He found that the curves for all soils were very similar with the capacitance increasing very rapidly with water content up to approximately the moisture equivalent of the soil and remaining nearly constant for higher water contents. Anderson also found that the increase in capacitance with water content was about $450 \mu\text{uf}$ as opposed to a predicted value of about $16 \mu\text{uf}$ assuming a relative dielectric constant of 5 for the solids and 80 for the water. He suggested that the anomalously high dielectric constants could be the result of very high polarizability of the adsorbed water layer or as a result of polarization of the non-insulated electrodes. He also stated that contact between the electrodes and the soil is not important as long as it is reproducible, which was the case for the soils he tested. On the other hand, Childs (33), in a discussion of electrical methods of measuring soil moisture, shows that for certain conditions of low frequency and high conductivity of the dielectric it would be possible for contact capacitances to produce high capacitance readings such as those reported by Anderson.

Anomalous dielectric constant values have also been measured for a wide range of materials other than soils. Most significant to this study are those for rocks. Evjen (34) estimated a value for the relative

dielectric constant of a sedimentary rock section to be 10^8 . Recently Keller and Licastro (35) have measured values greater than 10^6 for sedimentary cores in the natural state. Keller and Licastro made their measurements at frequencies ranging from 50 cps to 30×10^6 cps. They found the very high values at the lower frequencies. At frequencies above 10×10^6 cps all values were less than 80. This dispersion effect was only significant for wet cores and is obviously associated with the contained water. They also measured the resistivity of the cores. The wetter cores show an almost constant resistivity for the lower frequencies decreasing with frequency only above about 10^6 cps. The dry cores on the other hand show an almost linear decrease with frequency on a log-log plot for the full range of frequencies studied. A portion of their data is reproduced in Figure 2.

Keller and Licastro show, based on a dispersion model proposed by Jolliffe (36), that the measured dielectric constant values are much too high to be attributed entirely to electrode polarization. They further explain the anomalously high dielectric constants on the basis of an interfacial or space-charge polarization phenomena.

Dielectric Properties of Concrete

Very little actual data is available pertaining to the dielectric properties of concrete. A paper by Hammond and Robson (37) contains the only data on the dielectric constant which could be found in the literature. Steniour (38) has referred to the results in this paper as the standard for the cement industry. These workers found anomalously high dielectric constants and dispersion for frequencies in the range of 50 to 25,000 cps. They also obtained somewhat different values for different types of cement, and the dielectric constant values for pastes were significantly higher than the corresponding values for concrete. All that can be learned about

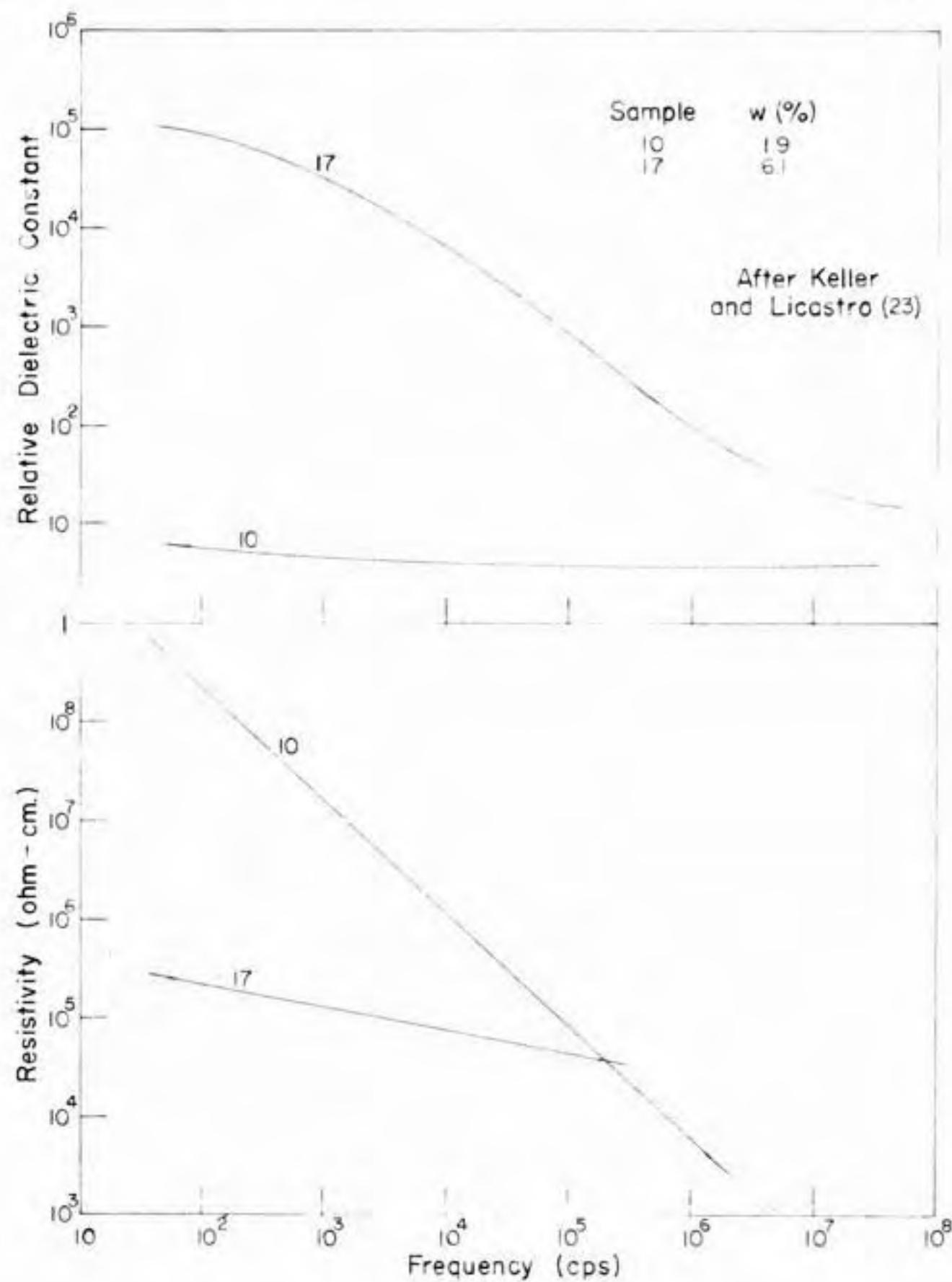


FIG. 2 RELATIVE DIELECTRIC CONSTANT AND RESISTIVITY OF NATURAL-STATE MORRISON CORES

the dielectric constant-water content relationship from these data is that, within the range of the relatively low water contents tested, the dielectric constant generally increases with increases in water content. A portion of Hammond and Robson's results are presented as Figure 3. The trends of these results are, in general, consistent for those of Keller and Licastro (35) for rocks (Figure 2).

The conductivity of concretes and pastes have been studied by several investigators, and it is generally accepted that this property is strongly influenced by several factors other than water content. Spencer (39) studied this property in 1938 and states that at the same water content resistivity of a concrete may vary by 40% with changes in salt content of the pore fluid. Decoux, Roland, and Barnes (40) find that the resistivity at a given water content of a fresh concrete may vary from that of an aged concrete by a factor of 100 because of salts in the pore fluid. They also show that certain admixtures such as blast furnace slag can have a significant effect on the conductivity.

The conductivity of concrete must also be strongly affected by the mix design, curing, and other factors. Powers, Copeland, and Mann (41) present results which show that water-cement ratio, type of cement, curing and fineness of cement can control the degree to which a hardened cement paste will contain continuous capillary pores. Whether or not the cement paste of a concrete contained continuous capillaries would have a pronounced effect on the electrical conductivity of the concrete.

Theory of High Loss Dielectrics

This section reviews only those aspects of the general theory of dielectrics which are necessary to explain some of the apparently

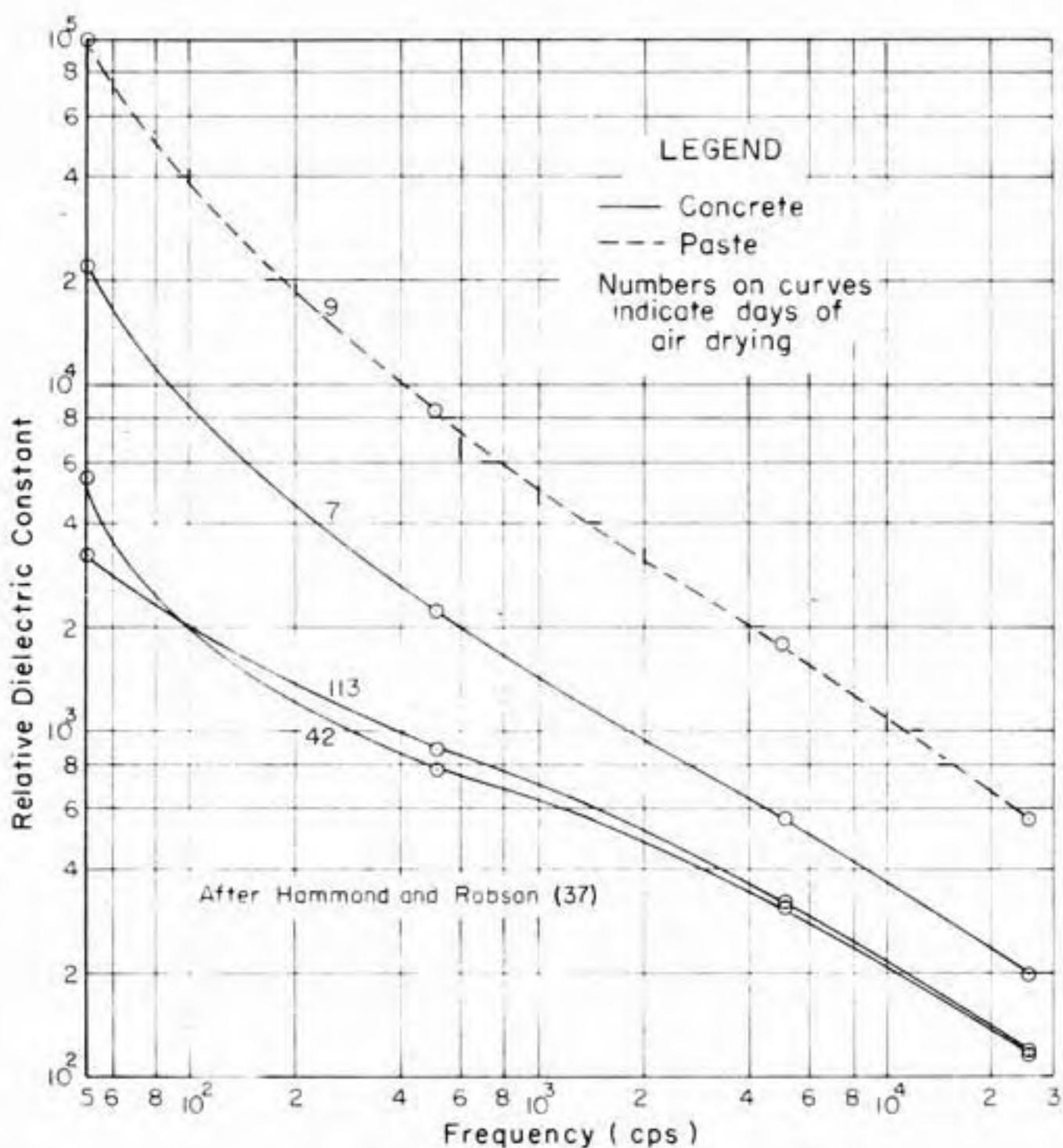


FIG. 3 RELATIVE DIELECTRIC CONSTANT OF CONCRETE

contradictory results recorded in the literature on electrical moisture meters. A more complete review is available in Progress Report No. 4 (4).

If a sinusoidal voltage is applied to a capacitor incorporating a leaky dielectric, the capacitor will draw a charging current leading the voltage by a phase angle of 90° and a loss current in phase with the voltage. The loss of a dielectric is often designated in terms of a dissipation factor D or loss tangent, $\tan \delta$. Thus,

$$D = \tan \delta = \frac{\text{loss current}}{\text{charging current}} \quad (2)$$

If the dielectric is characterized by a complex permittivity ϵ^*

$$\epsilon^* = \epsilon' - j\epsilon'' \quad (3)$$

where, ϵ' = dielectric constant

ϵ'' = loss factor

$$j = \sqrt{-1}$$

The total current flowing through the capacitor is

$$I = (\omega \epsilon' j + \omega \epsilon'') V \frac{C}{\epsilon_0} = j\omega V C_0 k^* \quad (4)$$

where $\omega = 2\pi \times$ frequency of applied voltage

V = applied voltage

C_0 = capacitance of the capacitor with a vacuum
as the dielectric

ϵ_0 = dielectric constant of a vacuum

$k^* = k' - jk'' = \frac{\epsilon^*}{\epsilon_0} =$ relative complex permittivity ($k' =$
relative dielectric constant and $k'' =$ relative loss
factor)

and the loss tangent may be written

$$\tan \delta = \frac{\epsilon''}{\epsilon'} = \frac{k''}{k'} \quad (5)$$

For this notation, the current density J traversing a parallel plate capacitor under an applied field strength E is

$$J = (j\omega \epsilon' + \omega \epsilon'') E \quad (6)$$

Representing this current density graphically (Figure 4), it is apparent that the quantity

$$\sigma = \omega \epsilon' \quad (7)$$

has the properties of conductivity and therefore is called dielectric conductivity.

A dielectric material inserted between the plates of a capacitor increases the storage capacity of the capacitor by neutralizing charges at the electrode surfaces. If a dielectric composed of dipolar molecules is assumed, this neutralization may be visualized as the result of the orientation of the dipoles to oppose the applied field. The process of orientation to oppose the applied field is known as dielectric polarization and was first recognized by Faraday prior to 1837. The polarization is proportional to the ease with which the dielectric may be polarized or its polarizability. Polarizability may be defined as the average polarization per unit field strength per contributing elementary particle. The greater the polarizability of a material the greater will be its dielectric constant.

Four mechanisms of polarization are recognized: 1) electronic, 2) atomic, 3) orientation, and 4) space charge or interfacial polarization. The first three polarization mechanisms are all forms of the dipole polarization previously discussed and are due to charges locally bound in atoms, in molecules, or in the structures of solids and liquids. They result from permanent molecular dipoles (orientation polarization) or dipoles induced by external fields in atoms (electronic polarization) and in molecules (atomic polarization). The fourth type -- space charge (or interfacial) polarization -- results from charge carriers that are usually present and more or less free to move through the dielectric

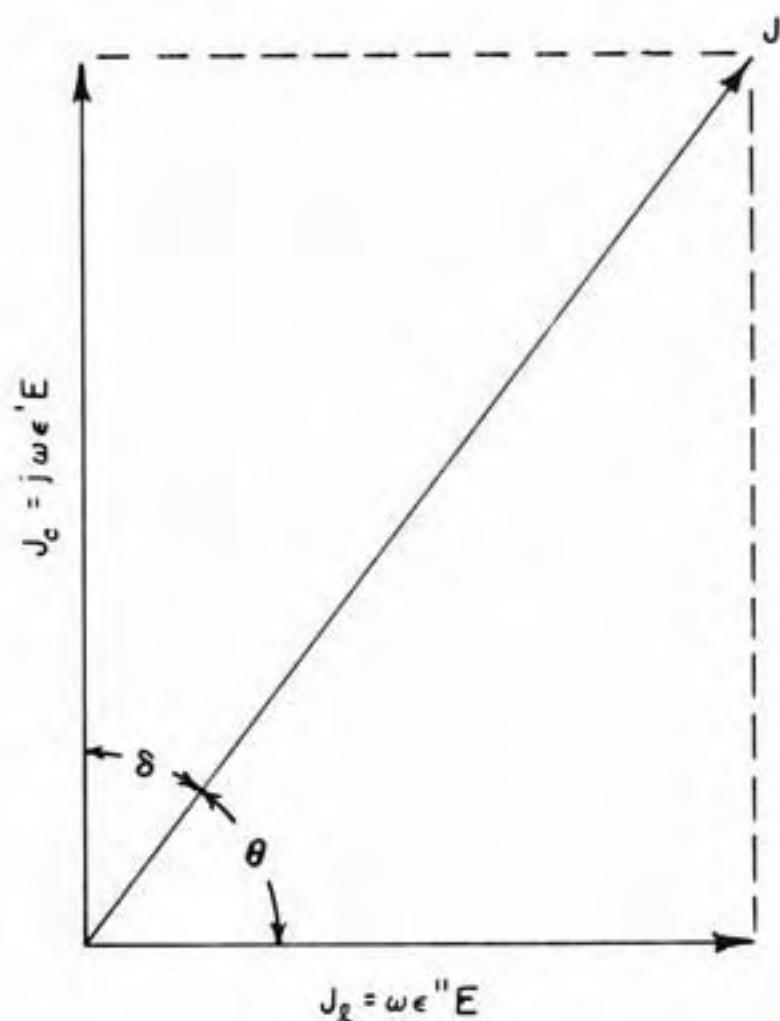


FIG. 4 - CURRENT DENSITIES OF A CAPACITOR WITH LOSS

under the influence of the applied field. When such carriers are impeded in their movement and become trapped in the material or at interfaces, space charge concentrations result in a macroscopic field distortion which increases the capacitance.

The total polarizability α is the sum of the electronic polarizability α_e , the atomic polarizability α_a , the orientation polarizability α_o , and the space-charge polarizability α_s . There are theories relating microscopic structure to the dielectric constant for gases at low pressures and some pure liquids and crystals, but at the present time this is completely out of the question for complex systems such as concrete. Microscopic considerations, however, do aid in understanding qualitatively some of the observed phenomena.

These considerations show that the dielectric constant of a material cannot be a constant because it is a function of the polarizability which is in turn a function of frequency, temperature, local fields, applied field strength, availability and freedom of charge carriers within the dielectric, and local field distortions.

The dielectric conductivity will also show the influence of these factors. This is true because dielectric conductivity is a function not only of ohmic conductivity but also of the power consumed in polarizing the material; therefore, it must reflect the control these factors have over the polarizability of the material.

Considering the work of Keller and Licastro (35) and the nature of concrete, space-charge polarization may play an important role in concrete. This mechanism unlike the other polarization mechanisms which are produced by displacement of bound charges is produced by traveling charges. Space-charge or interfacial polarization was recognized prior

to 1892 by Maxwell (42), whose classical two-layer capacitor serves as a model for this polarization mechanism. In this model the dielectric consists of two sheets characterized by their dielectric properties k'_1 , σ_1 and k'_2 , σ_2 respectively, and $\sigma_1 > \sigma_2$. This compound dielectric possesses a dielectric constant which is macroscopically indistinguishable from that of simple orientation polarization; however, the loss factor contains in addition to an orientation loss term an ohmic-conductivity term caused by the σ_1 and σ_2 factors. From the molecular point of view, because of the unequal conductivities, charge carriers pile up at the interface between sheet 1 and sheet 2 and cause field distortions resulting in a higher dielectric constant than would be predicted from a simple series combination of the capacitances resulting from k'_1 and k'_2 .

Von Hippel (43) has shown that dividing the layers 1 and 2 into thinner layers and arranging them in any manner does not affect the complex permittivity of the resulting dielectric. However, a change of the relative geometry of the two media, for example from sheets to cylinders or spheres of material 1 in a matrix of material 2, results in a very different permittivity (44). It is apparent that the simple mixing rules frequently used can hold true only for very specific special distributions which eliminate space-charge polarization.

If space-charge polarization is to be avoided, the charge carriers must be able to move freely through the dielectric, be capable of discharging freely at one electrode, and be replaced simultaneously at the other electrode. This combination of conditions is very unlikely in non-metals (45). Space-charge polarization at the interface between the dielectric and the electrodes has received much notoriety under the name of electrode polarization; however, until very recently it has been almost completely overlooked as a possibility within the body of the dielectric.

It is, of course, impossible to predict quantitatively the dielectric constant-water content relationship of a concrete, but these considerations assist materially with the interpretation of permittivity measurements.

Permittivity Measurements

The relationship between charging and loss current densities (Figure 4) suggests the representation of capacitors with loss by circuits composed of lumped ideal capacitances and resistances. The most obvious such representation is the simple parallel RC circuit. When this circuit is connected to an AC voltage source, the current becomes

$$I = \left(j\omega C + \frac{1}{R}\right)V \quad (6)$$

For electrical equivalency of this RC circuit to a real capacitor with a leaky dielectric, its current must be identical in magnitude and phase with that of the real capacitor, and it follows that

$$k^2 = \frac{C}{C_0} \quad (9)$$

$$= \frac{\epsilon_0}{RC_0} \quad (10)$$

and

$$D = \tan \delta = \frac{1}{\omega RC} \quad (11)$$

where C and R are the equivalent parallel ideal capacitance and resistance respectively, C_0 is the geometric capacitance of the real capacitance, k^2 and σ are the relative dielectric constant and dielectric conductivity of the real dielectric at the angular frequency ω , and δ is the loss angle for this dielectric. It must be remembered that R is not the ohmic resistance of the dielectric but is a purely conductive resistance which dissipates

the same power as that lost in the dielectric by conductance plus all other power-consuming phenomena. Any two of these equations completely describe the real dielectric in terms of an equivalent parallel circuit composed of R and C.

The analogy between a real capacitor and a RC circuit of ideal lumped circuit components is a very useful concept in many applications and provides the basis for the measurement of capacitance.

A variety of methods has been developed to determine the dielectric properties of materials. The primary factors controlling the method best suited to a given application are the frequency at which the measurements are to be made and the loss of the dielectric. For frequencies up to about 10^8 cps, lumped circuits are used, and above 10^8 cps, distributed circuits are used to determine permittivity. Neither complex permittivity nor its components, dielectric constant and loss factor, are measured directly but are calculated from measurements of other parameters. The selection of parameters measured depends on the frequency range used.

Very low frequencies are not suitable for moist concrete because of the high concentration of mobile ions which lead to electrode polarization problems. Very high frequencies result in increased instrumentation problems. In the frequency range from about 10 to 10^7 cps the most generally useful method for dielectrics with loss is the AC bridge method (46). In this method the parameters measured are usually relative capacitance and dissipation factor. The dielectric constant and loss factor or dielectric conductivity are obtained by suitable calculations using these parameters.

Figure 5 is a schematic diagram of the basic Schering bridge circuit which experience has shown to be especially well suited to

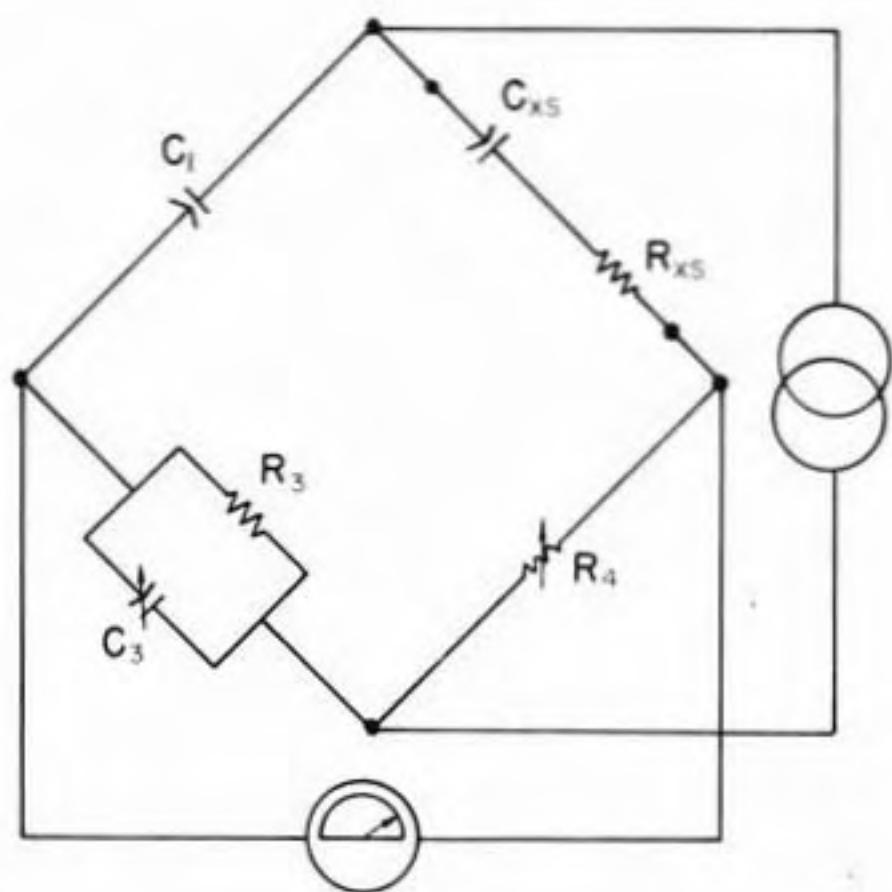


FIG. 5 - BASIC SCHERING BRIDGE CIRCUIT

measurements on high loss dielectrics. The balance equations for this bridge are

$$\frac{R_2 R_3}{R_1} = R_{xs} \quad (12)$$

and

$$C_3 \frac{R_1}{R_2} = C_{xs} \quad (13)$$

In this circuit C_3 and R_4 are the variable components to achieve balance with the unknown capacitor represented by the series equivalent components C_{xs} and R_{xs} . A dial on R_4 calibrated in capacitance units may be used to indicate the value of C_{xs} . For this bridge the dissipation factor can be expressed

$$D_x = \frac{1}{\omega C_3 R_3} \quad (14)$$

therefore, a dial on C_3 is usually calibrated to read the dissipation factor directly. With C_{xs} and D_x known it is a simple matter to calculate dielectric constant and loss factor.

As the frequency is increased, stray capacitances and inductances within the bridge become important, and appropriate compensating components, as well as shielding, must be added to the bridge, greatly complicating its design, construction and calibration.

The values for C_{xs} and D_x indicated by the bridge are not equal to the respective values for the sample being measured. C_{xs} and D_x contain other capacitance and loss terms. The leads connecting the sample and bridge have capacitance and resistance which increases both C_{xs} and D_x . Also there will be fringe capacitance between the electrodes around the sample and stray capacitances between the high and ground. These extraneous effects must be eliminated either by appropriate equipment design or by calculations. Detailed procedures for handling these factors are given by Field (47).

Another important consideration in capacitance measurements is the quality of the contact between the electrodes and the sample being tested. If this contact is less than perfect, there will be a finite contact capacitance C_c in series with the specimen capacitance C_s . Considering perfect capacitors without loss, the total capacitance C_x would be

$$C_x = \frac{C_c C_s}{C_c + C_s} \quad (15)$$

From this expression, $C_x \rightarrow C_s$ as $C_c \rightarrow \infty$ and $C_x = \frac{C_s}{2}$ for $C_c = C_s$. Therefore, unless the contact capacitance is much greater than the specimen capacitance, C_c must be determined and its influence taken into account.

Summary

In considering the various moisture measuring methods the resistance method was eliminated from serious consideration because of its susceptibility to changes in pore water composition which may cause larger resistance variations than even relatively large changes in water content.

The relative humidity and suction methods, radiation methods, and heat transfer methods were rejected because while they offer no special advantages over the ionic barrier resistance method or the dielectric constant method they suffer from a wide variety of additional disadvantages. At first the ionic barrier method appears to be the most promising method; however, while this method is considered to be deserving of additional study, the capacitance method was judged to be inherently superior for two reasons: 1) the ionic barrier requires a hygroscopic material surrounding the electrodes which increases its response time, and 2) the useful life of the ionic barrier is unknown.

It is well established that the dielectric constant-water content relationship of a complex material such as soil or concrete is not constant; it is certainly a function of frequency, and there are strong indications that it is also a function of the local electrical fields acting on the pore water and its associated ionic concentration. The degree of contact between the electrodes of the sensing capacitor and the dielectric material is also an important factor: for some conditions the effect of incomplete contact can completely override the effect of changes in moisture content. Furthermore, conventional formulae for predicting the dielectric constant of a mixture of dielectrics do not apply to complex, high-loss dielectrics such as concrete. Studies have shown that it is possible for the dielectric constant of these materials, for a given set of conditions, to be considerably greater than the dielectric constant of any of their constituents.

The use of dielectric properties as moisture indicators is predicated on the assumption that appropriate measurements can be made on sensing elements which will actually reflect these properties, that the relationship between these properties and water content will remain sufficiently constant during the period over which moisture determinations are desired, and that this relationship can be established by some appropriate calibration procedure. From the review of previous work it is by no means clear whether or not these considerations can be met with sufficient precision for any given application.

The most important question is whether or not the relationships between water content and the dielectric properties of concrete remain constant to a sufficient degree of accuracy. The laboratory tests conducted in this study were to provide a basis for answering this question.

LABORATORY INVESTIGATIONS

General

This section briefly outlines the procedures and summarizes the results of the various laboratory test series conducted in the course of this study. These test series were as follows:

Shrinkage vs. Water Content [2]

Capacitance vs. Water Content ~ 60 cps [1]

Capacitance vs. Water Content ~ 600,000 cps [2]

Permittivity vs. Water Content ~ 10^6 cps [4]

Resistance vs. Water Content ~ 10 cps [4]

The numbers in the brackets indicate the Progress Reports which present detailed discussions and complete results of the test series.

All tests with the exception of the conductivity tests were performed on samples prepared in the form of thin disks 1/8 to 1/4 inches thick and 3 inches in diameter. The conductivity measurements were made on prisms approximately 1/4 x 1 x 2 inches long. Portland cement mortar specimens were used except for the permittivity and conductivity tests where both a mortar and a concrete mix were tested.

All specimens were prepared from 3 x 6 inch cylinders cured one day in the mold and several days in a 100°F water bath. Upon removal from the bath the cylinders were sliced on a concrete saw and the specimens ground to the desired size on conventional petrographic grinding wheels. The faces of the capacitance and permittivity specimens were polished to give good contact with the test capacitor. During preparation the specimens were not allowed to dry; therefore, the first drying phase occurred during the actual testing.

of

The samples were tested through a number/cycles of drying and wetting. Moisture control was accomplished by allowing evaporation to (or absorption from) the test chamber atmosphere. Because the purpose of the proposed moisture meter was to measure transient moisture contents, no effort was made to insure equilibrium with the moisture environment. It was observed that the 105°C dry weights of the specimens increased during the testing period. A correction was applied for this factor, and all water contents quoted are relative to 105°C dry weight at the time the corresponding electrical measurement was made.

The permittivity and conductivity tests were performed at $30 \pm 1^\circ\text{C}$. All other tests were performed at laboratory temperature.

The shrinkage measurements were made by means of an Ames strain gage arrangement which measured the spacing of two brass plugs set at opposite ends of a diameter of the disk.

The conductivity relationships were calculated from direct resistance measurements made between the ends of the test prisms at 10 cps with an A.C. resistance bridge constructed especially for these tests.

All capacitance and permittivity measurements were made on parallel plate capacitors containing the test disk as the dielectric. The plates of the test capacitors were insulated with a thin Teflon sheet. The 60 cps measurements were made with a General Radio Co. Type 740 A.C. bridge. The 600,000 cps measurements were made with a special bridge fabricated in our laboratory. Neither of these tests permitted calculating the true dielectric constant from the test results. Apparent capacitance was the only parameter which could be determined for the conditions of these tests.

The 10^6 cps permittivity measurements were obtained with a General Radio Co. Type 1610-AH Capacitance Measuring Assembly. For these

tests not only the apparent capacitance but also the true dielectric constant and dielectric conductivity could be calculated from the results. Details of these calculations are presented in Progress Report No. 4 (4).

Test Results

Shrinkage

Shrinkage tests were performed on five specimens. Two of these were mortars prepared with quartz sand. One disk was further steam cured at 150 psi in an autoclave. The remaining three disks were composed of mortars incorporating a limestone sand. One of these limestone mortar specimens was further cured in water at room temperature for 96 days and during testing was only dried to 60°C. These samples were tested through a minimum of two cycles of drying and rewetting. Results representative of these shrinkage tests are presented as Figures 6-9. The results for Disk C-S (Figure 8) are also presented on Figure 10 as a semi-logarithmic plot with shrinkage plotted against log of water content.

Capacitance - 60 cps

Capacitance measurements at 60 cps were made on three test disks. All of these disks were mortars prepared with a quartz sand. One of the specimens was cured in the autoclave at 150 psi. Results typical of this series are given on Figures 11 and 12. The capacitance values plotted are apparent capacitance values and include the influence of the contact capacitance as well as the capacitance of the mortar disk.

FIG. 6 - SHRINKAGE vs. WATER CONTENT - DISK # 9

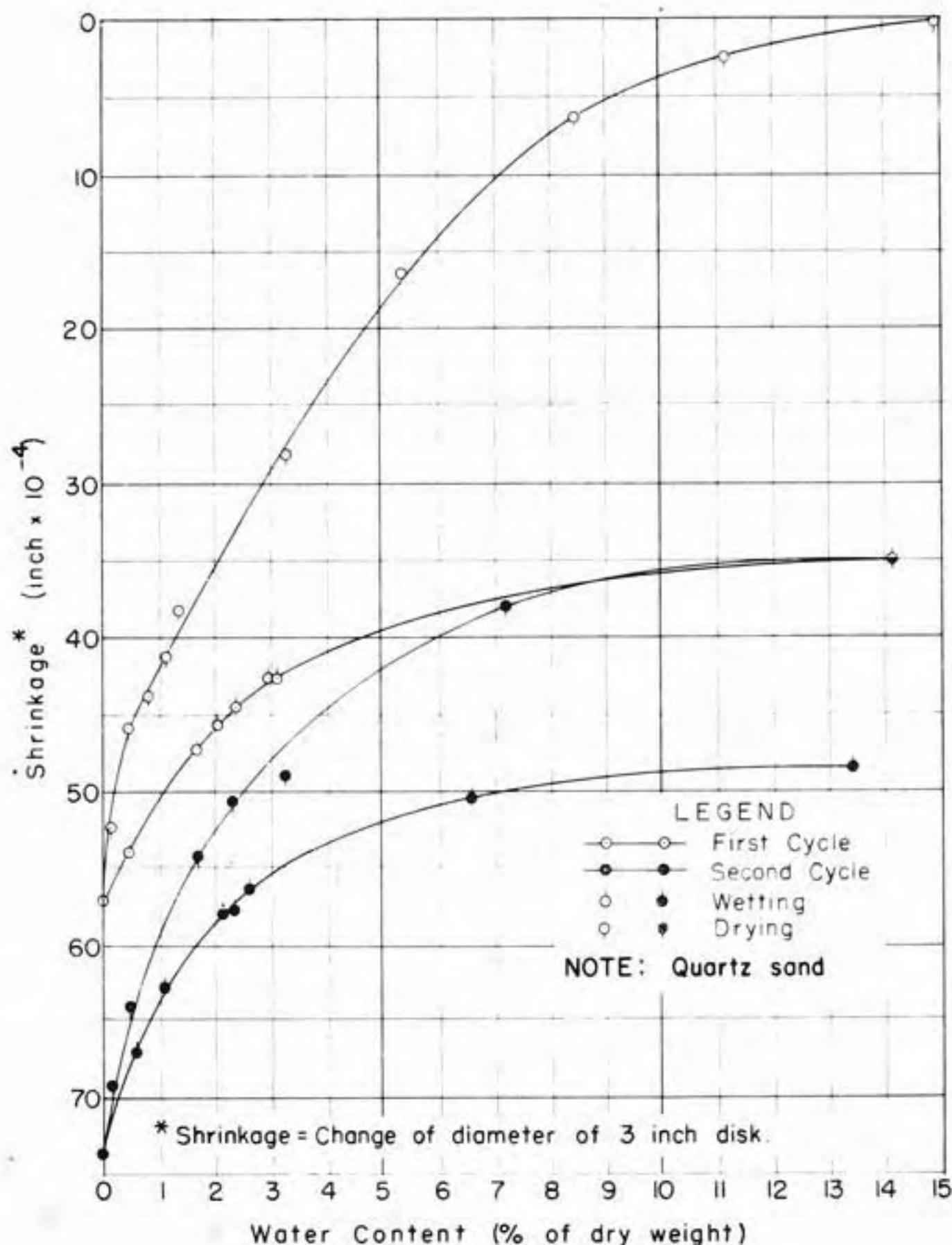


FIG. 7 - SHRINKAGE vs. WATER CONTENT- DISK # 2

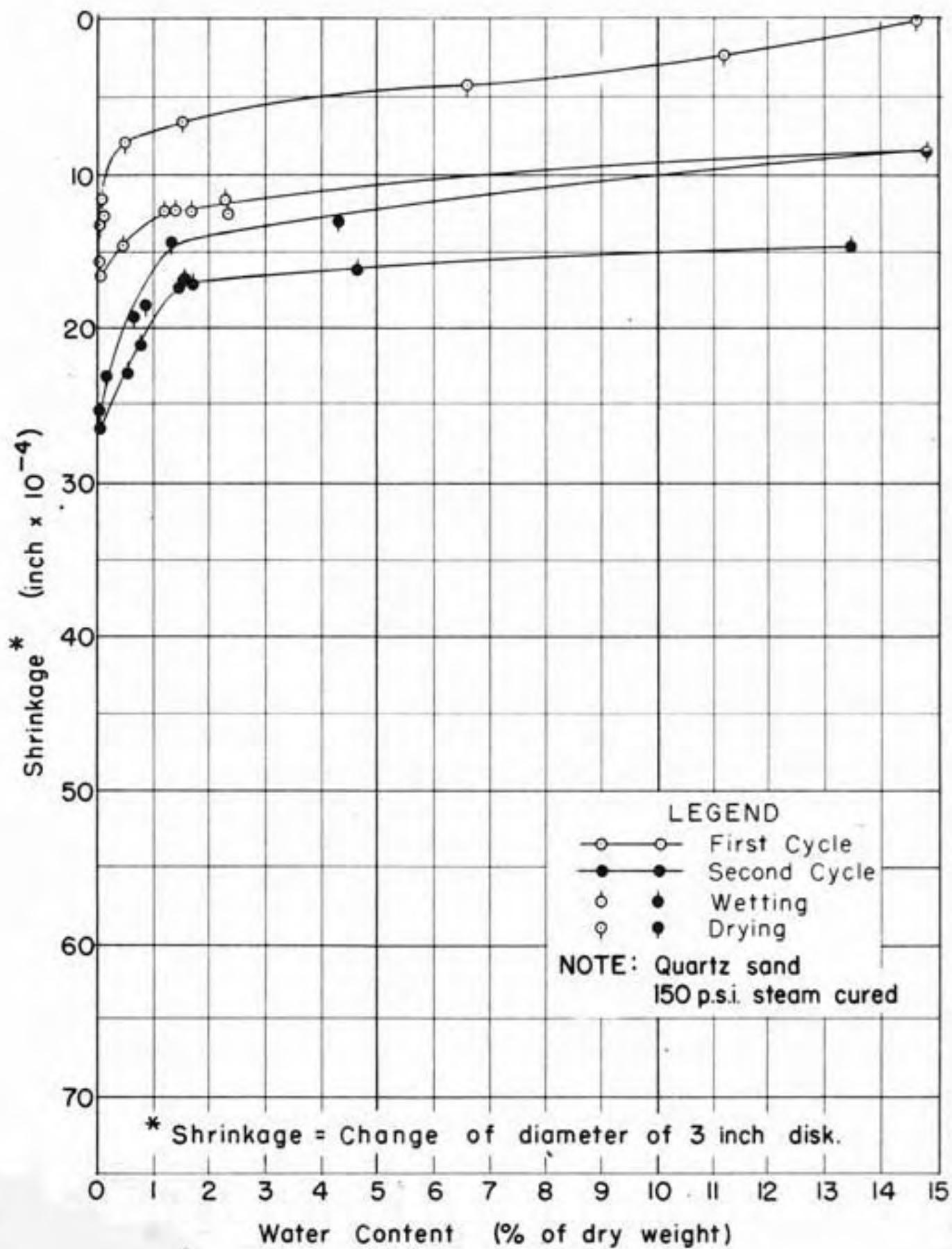


FIG. 8 - SHRINKAGE Vs WATER CONTENT- DISK C-S

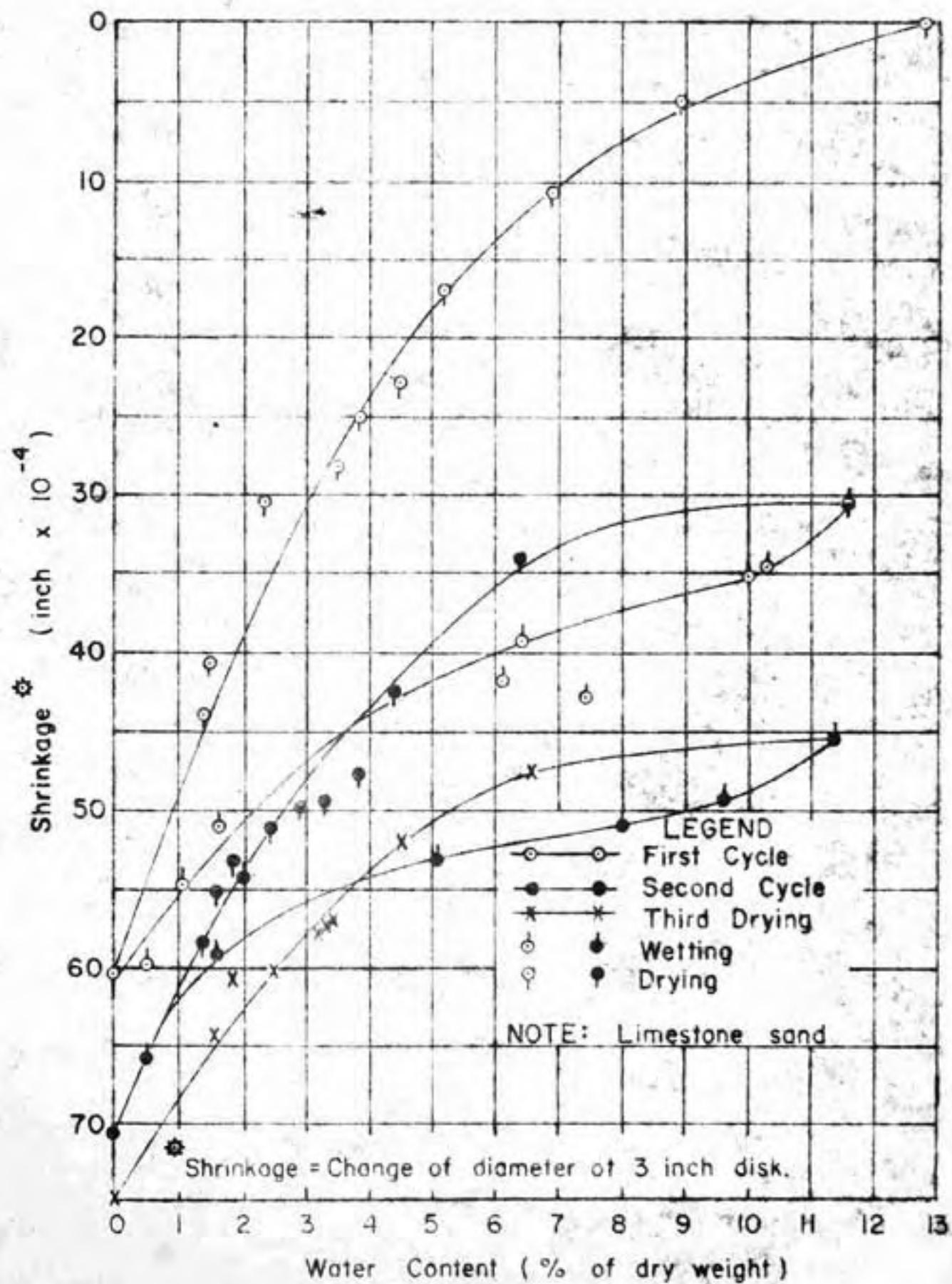


FIG. 9 - SHRINKAGE Vs WATER CONTENT- DISK B-S

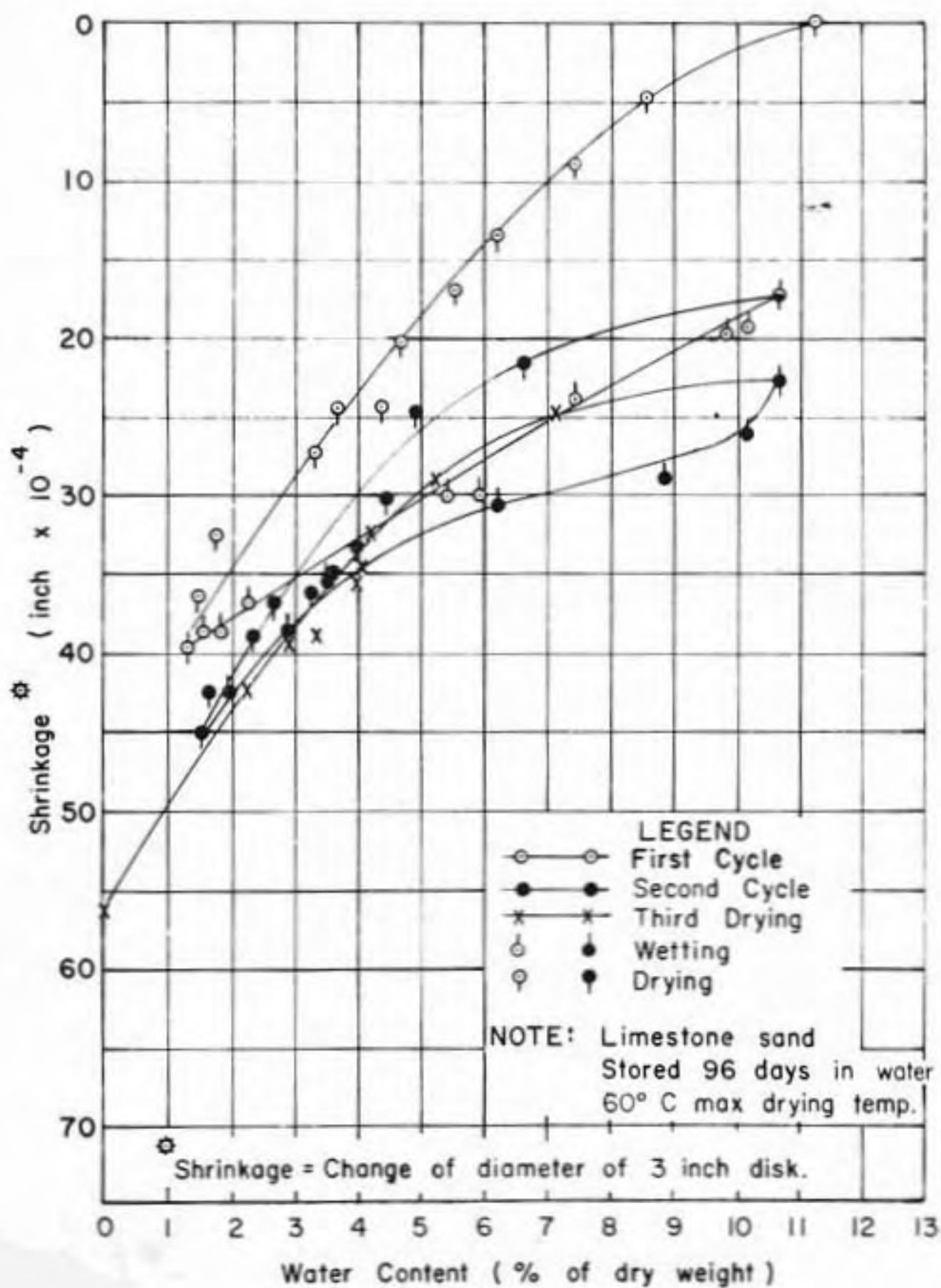


FIG. 10 - SHRINKAGE VS LOG OF WATER CONTENT - DISK C-S

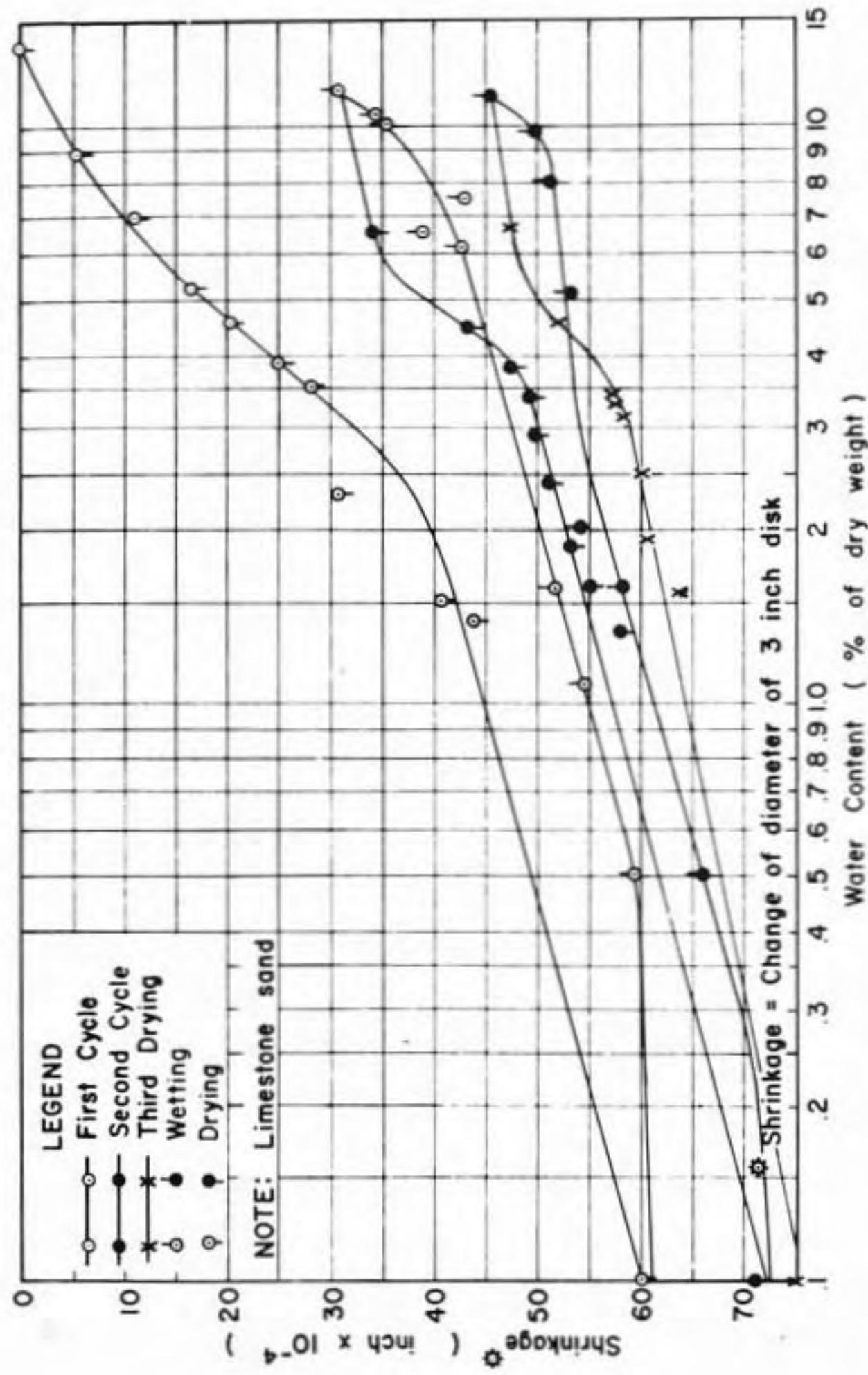


FIG. II - CAPACITANCE vs WATER CONTENT - DISK # 8
(60 c.p.s.)

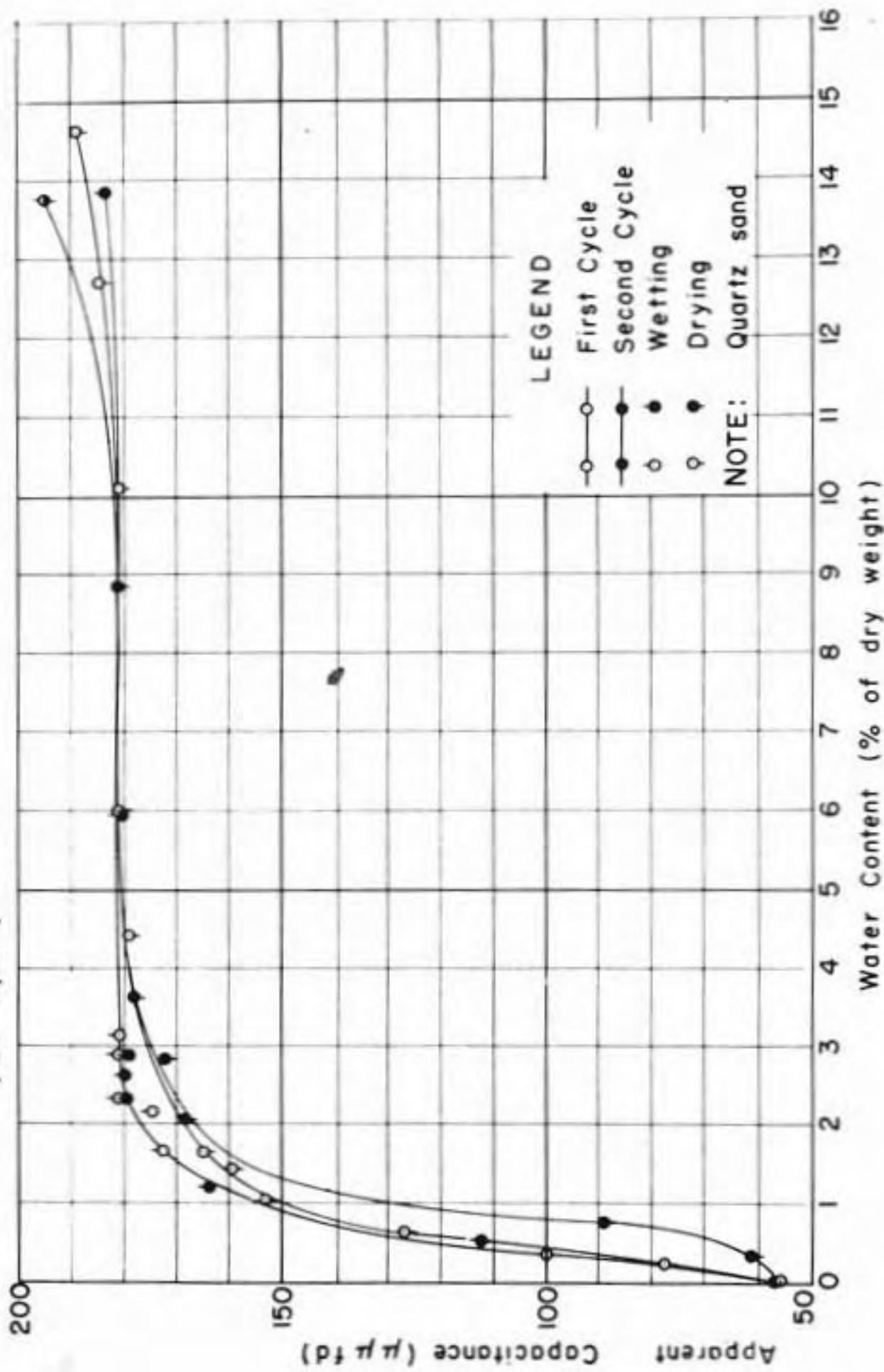
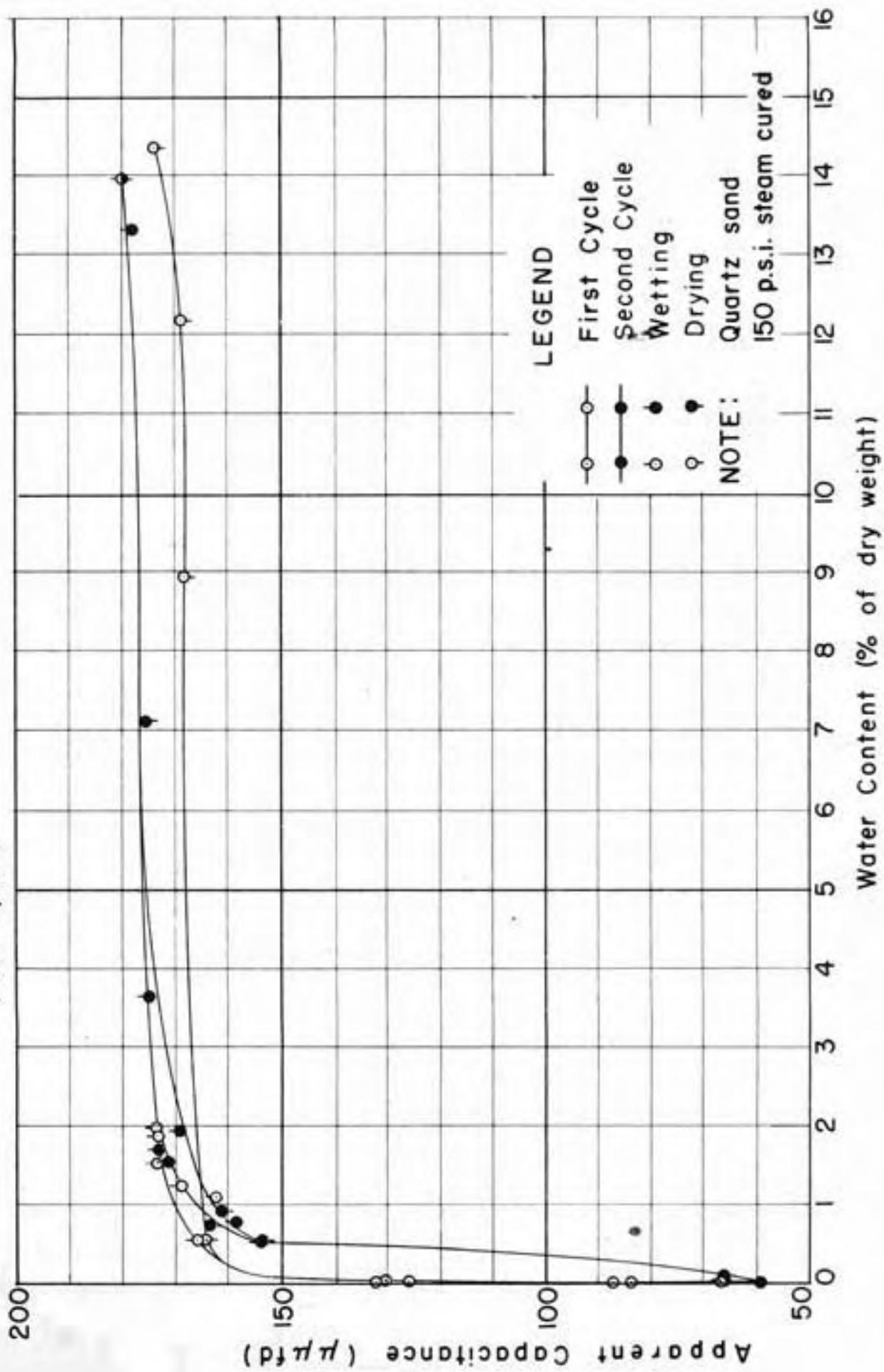


FIG.12- CAPACITANCE vs. WATER CONTENT- DISK # 1
(60 c.p.s.)



Capacitance - 600,000 cps

Capacitance tests were performed on five disks at a frequency of 600,000 cps. Four of these were mortar disks prepared with a limestone sand and the fifth was one of the quartz sand mortar disks used in the 60 cps tests re-tested at this (higher) frequency. Typical results of these tests are shown on Figures 13 and 14. One of the disks was tested using a maximum drying temperature of 60°C rather than the standard temperature of 105°C used for the other samples (Figure 14).

The impedance bridge used for this test series was not calibrated to read capacitance directly. Therefore, the capacitance dial reading values plotted are not actual capacitance but simply dial readings which indicate relative capacitance. These readings are a function of the contact capacitance of the test capacitor and the capacitance of the mortar disk, but the actual capacitance is not known; therefore, these values cannot be compared directly with the values from other test series. Only relative comparisons can be made.

Permittivity - 10^6 cps

Permittivity tests were conducted on eight specimens. Four of these specimens were mortar and four were concrete. Both mixes incorporated a limestone aggregate. Half of the specimens were dried at 105°C and half were dried at only 60°C at the end of each drying phase. Disks of two thicknesses (approximately 4 mm. and 7 mm.) were tested.

The results of these tests for two disks - C-1 and CT-2 - are presented on Figures 15 through 20. These results are typical of the results from this test series. Both of these disks were concrete specimens. The results for the mortar specimens had different absolute values, but the trends were the same. Disk C-1 was a thin disk dried to 105°C

FIG. 13 - CAPACITANCE DIAL READING Vs WATER CONTENT -
DISK C-C (600,000 c.p.s.)

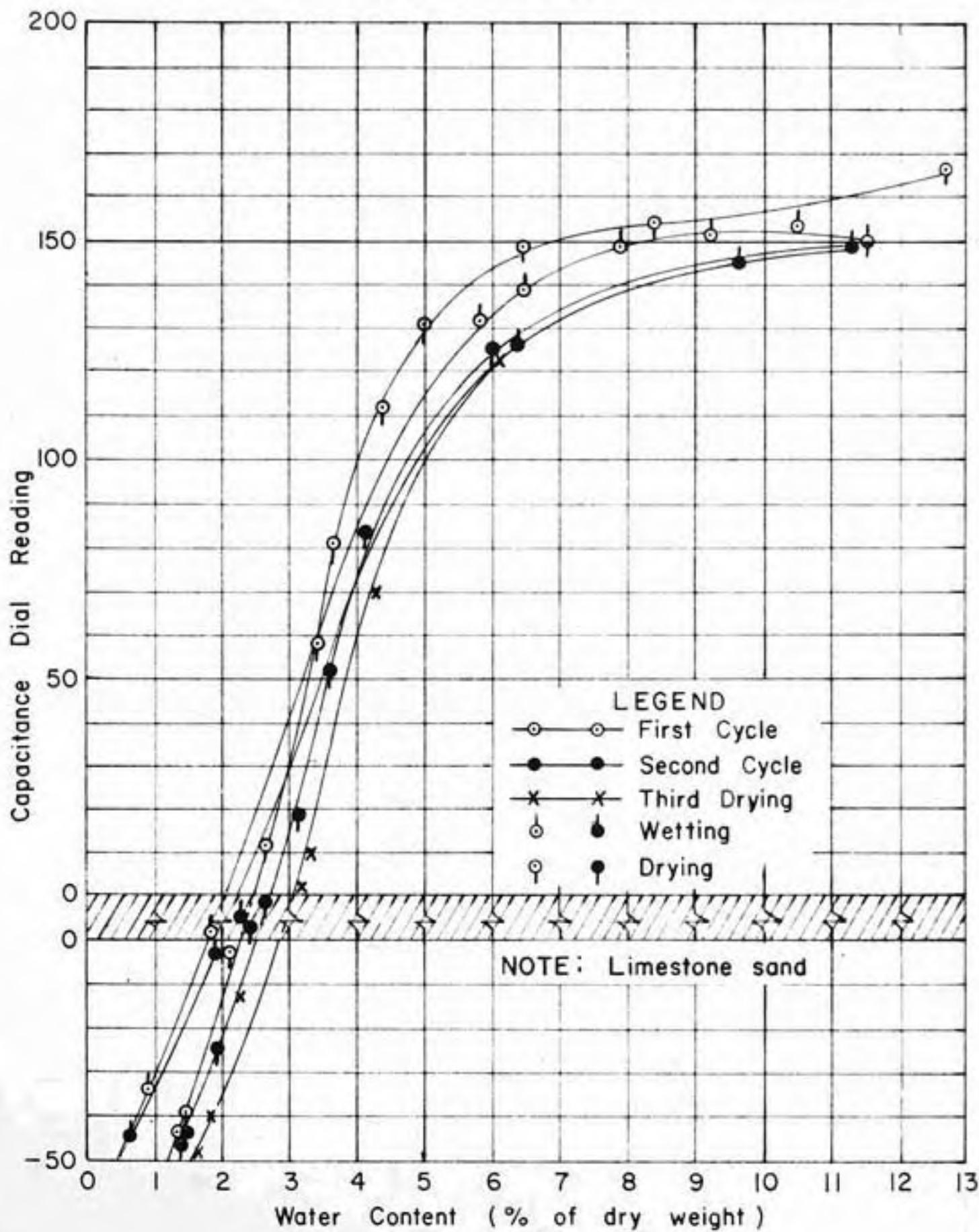
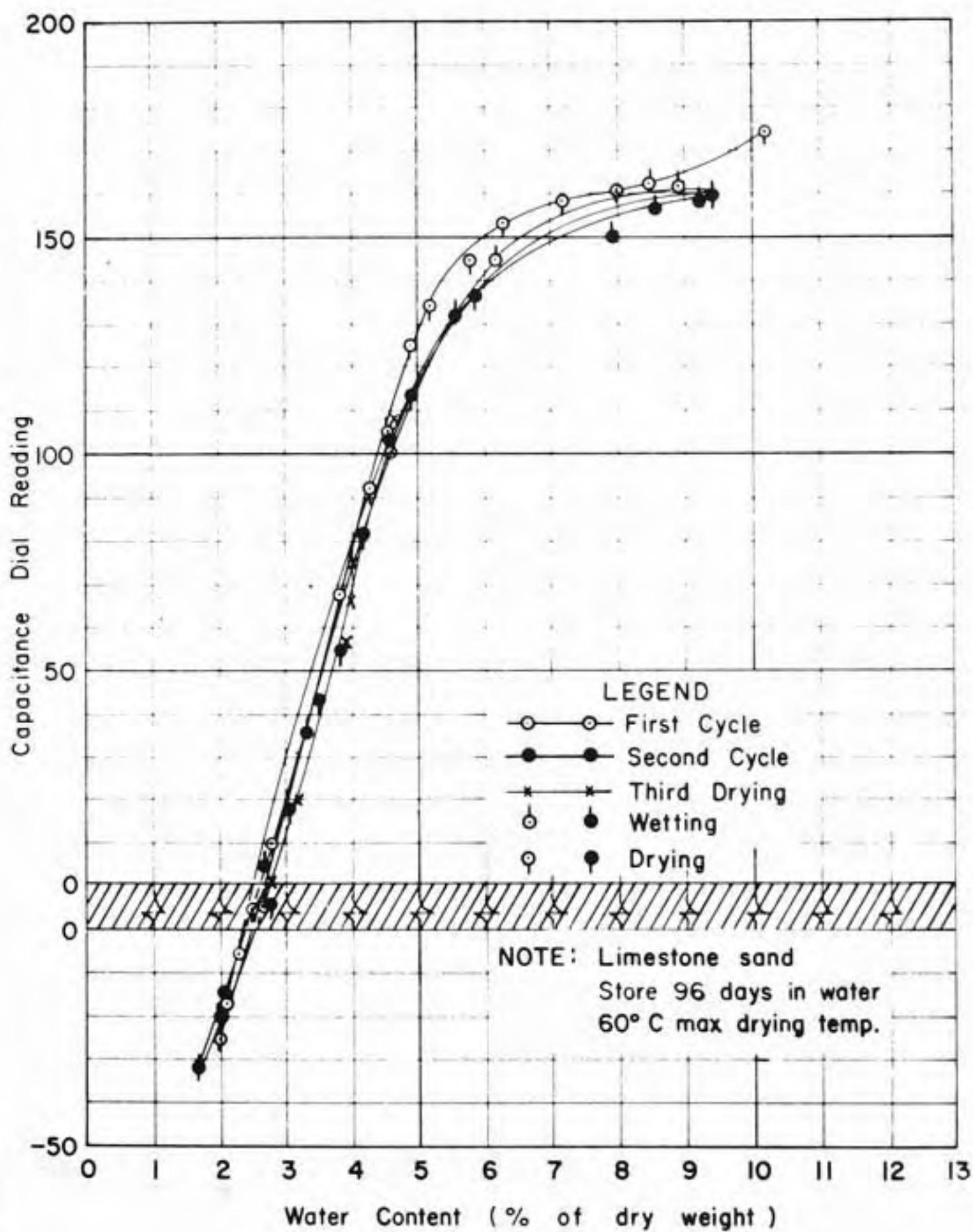


FIG. 14 -CAPACITANCE DIAL READING Vs WATER CONTENT-
DISK B-C (600,000 c.p.s.)



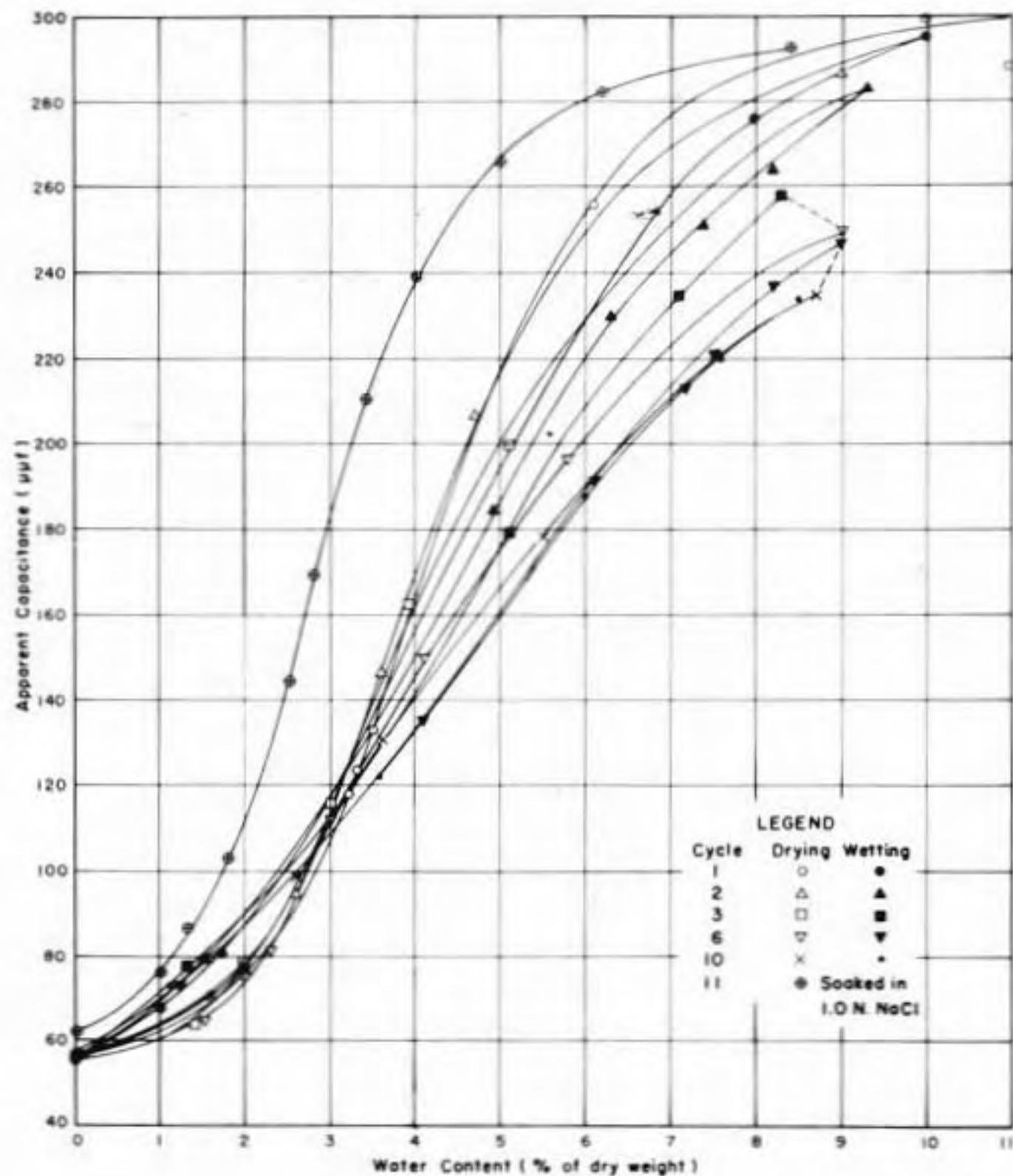


FIG. 15 APPARENT CAPACITANCE AT 10^6 cps VS. WATER CONTENT FOR DISK C-1

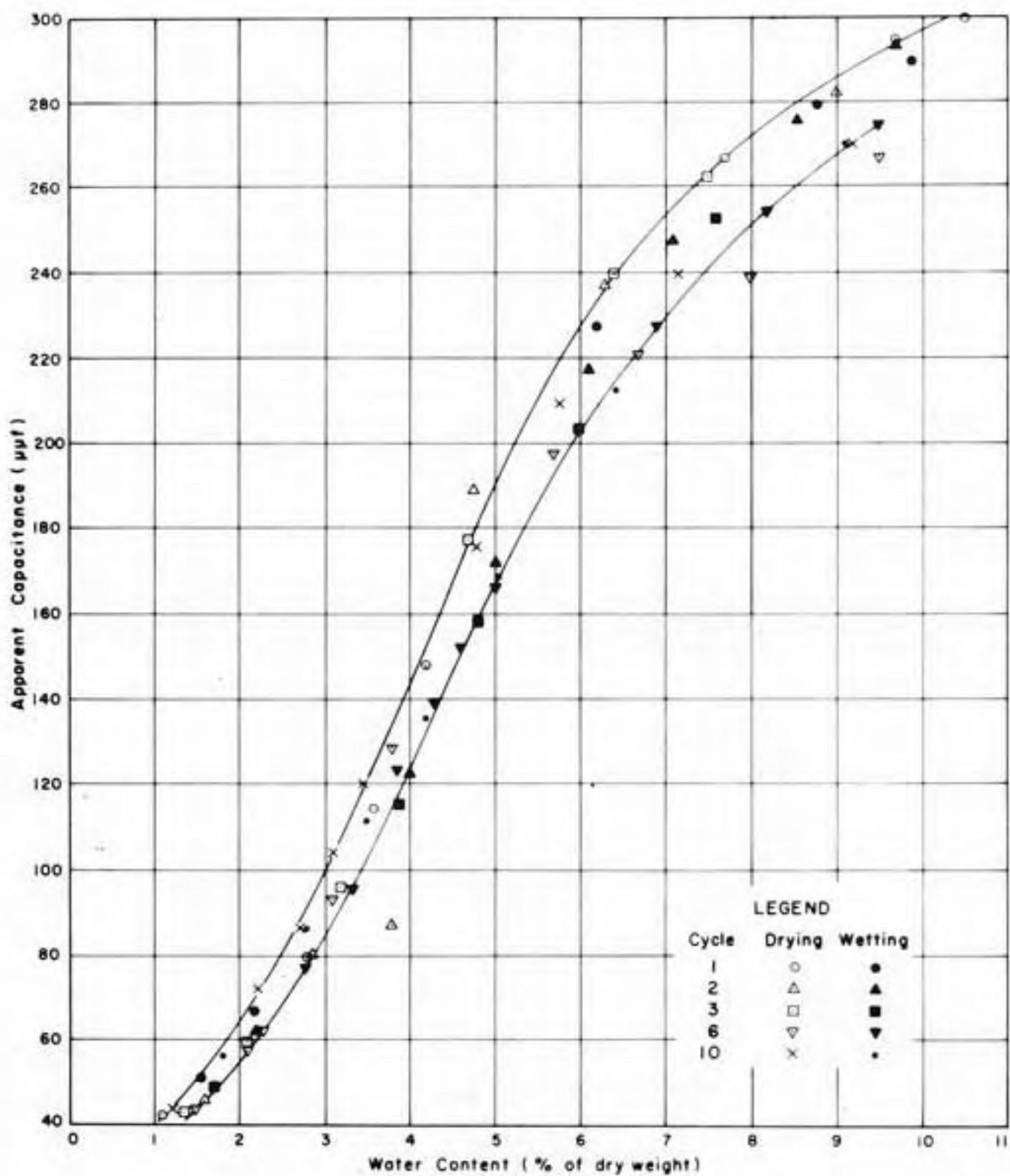


FIG. 16 APPARENT CAPACITANCE AT 10^6 cps VS. WATER CONTENT FOR DISK CT-2

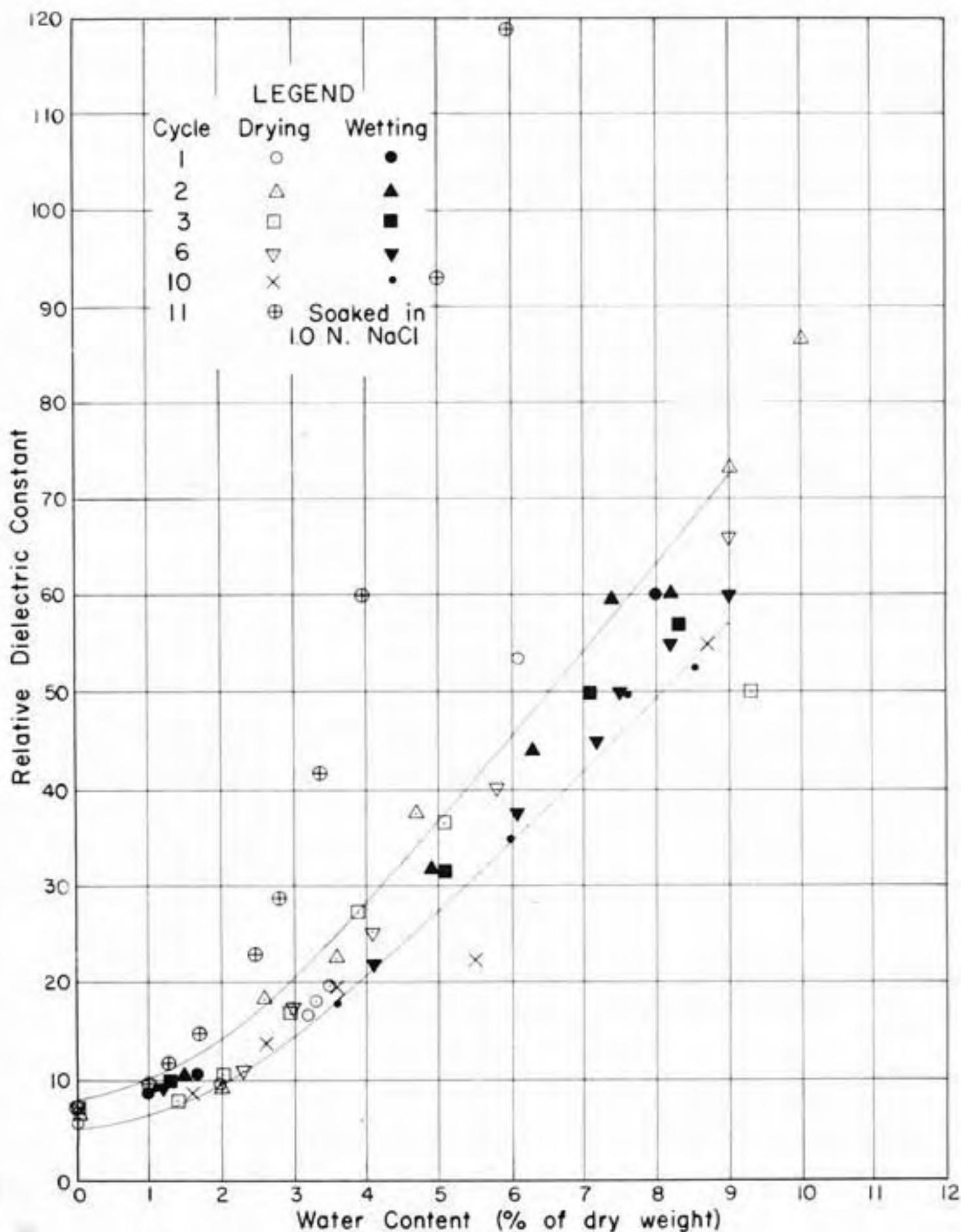


FIG. 17 RELATIVE DIELECTRIC CONSTANT AT 10^6 cps VS.
WATER CONTENT FOR DISK C-1

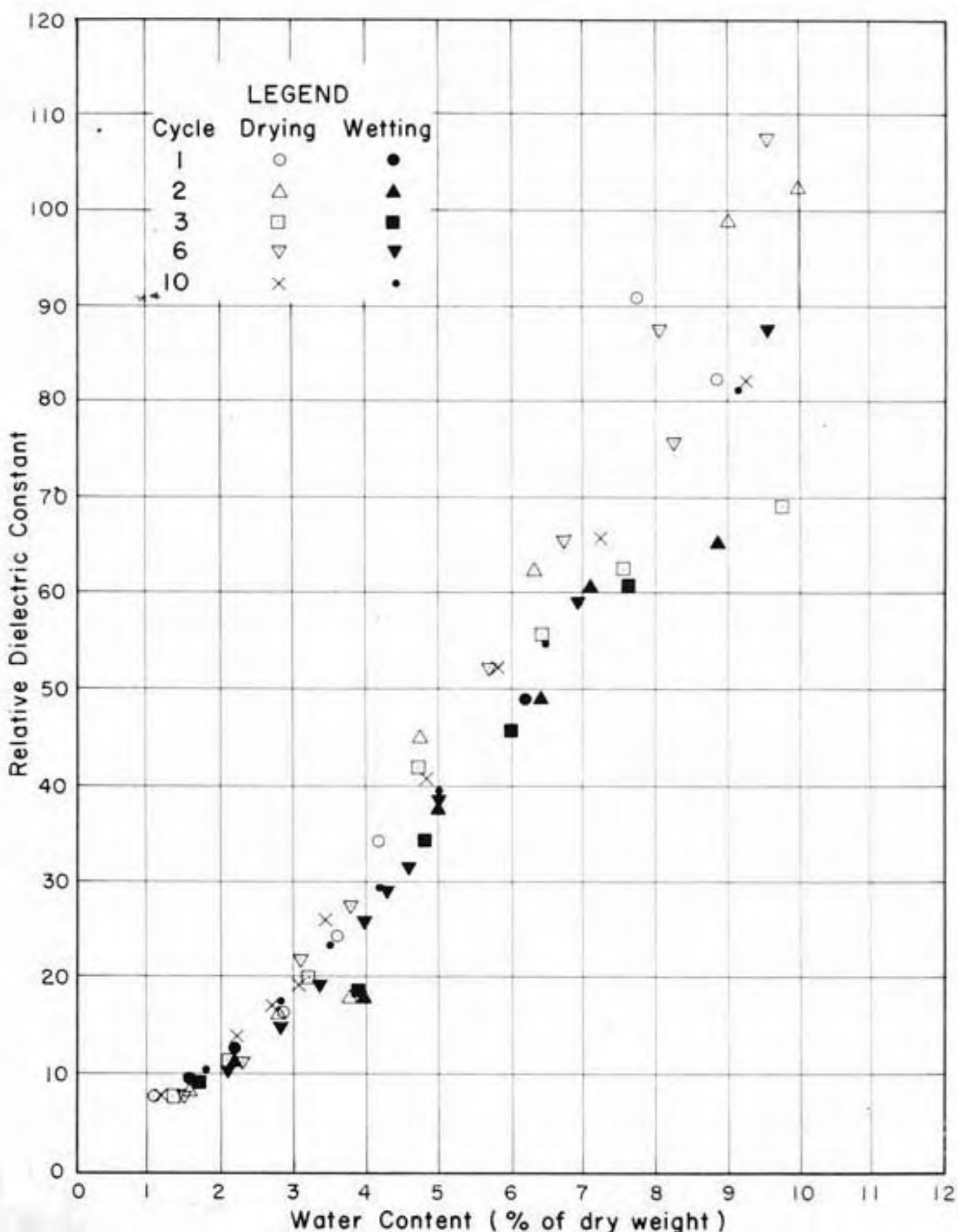


FIG. 18 RELATIVE DIELECTRIC CONSTANT AT 10^6 cps VS. WATER CONTENT FOR DISK CT-2

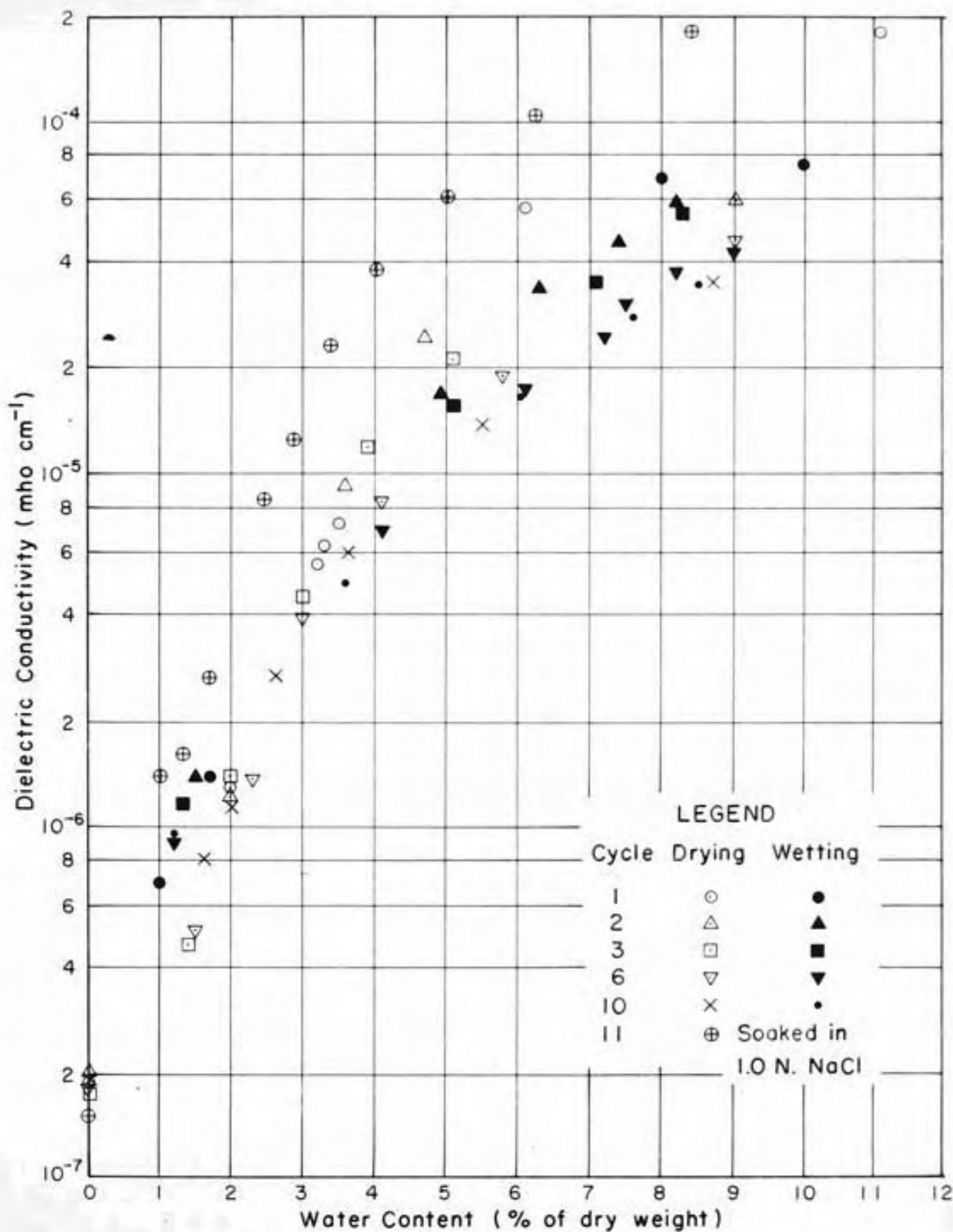


FIG. 19 DIELECTRIC CONDUCTIVITY AT 10^6 cps VS.
WATER CONTENT FOR DISK C-1

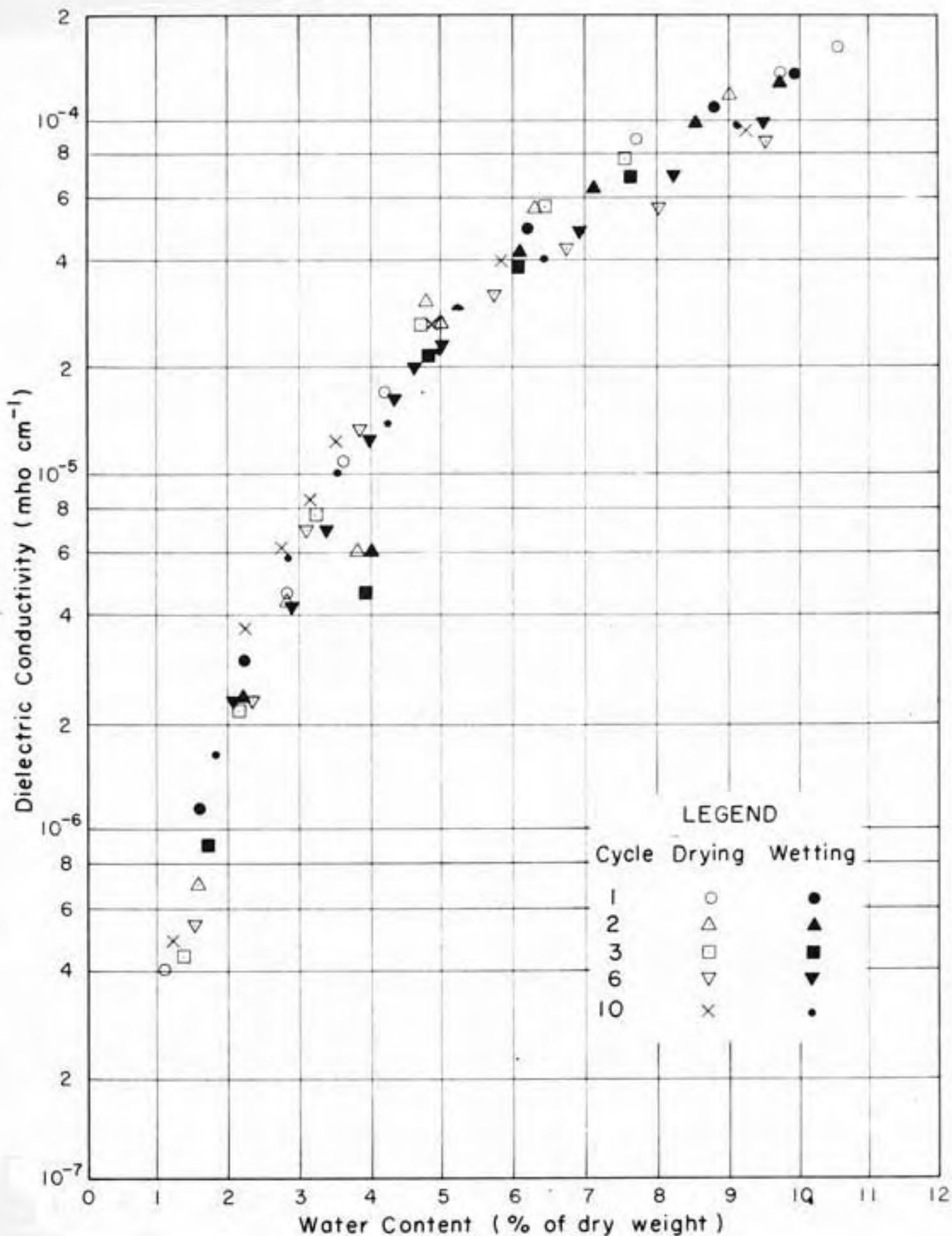


FIG. 20 DIELECTRIC CONDUCTIVITY AT 10^6 cps VS. WATER CONTENT FOR DISK CT-2

and disk CT-2 was a thicker disk and was dried to only 60°C. These two conditions represented the extremes of the test variables. Both disks were tested through ten cycles of drying and rewetting with distilled water. In addition disk C-1 was soaked in a 1.0 Normal NaCl solution after the tenth drying and tested through an additional drying phase.

The apparent capacitance-water content relationships for these two disks are shown on Figures 15 and 16. The apparent capacitances contain both the effects of the contact and the disk capacitances and is comparable to the capacitance results obtained at 60 and 600,000 cps. For the conditions of these tests it was possible to calculate the relative dielectric constant and dielectric conductivity from the test data. These relationships are presented on Figures 17 and 18 and Figures 19 and 20, respectively.

Resistance - 10 cps

Resistance measurements were made on four prisms (approximately 1/2" x 1" x 2") with an AC bridge at 10 cps. Two mortar and two concrete specimens of the same mixes as those used in the permittivity tests were studied. One specimen of each mix was dried to 105°C during the test period, and one was dried to 60°C. Dielectric conductivity was calculated from the resistance data, and this relationship for the two concrete disks tested is presented on Figures 21 and 22. Prism RC-1 was dried to 105°C, and prism RC-2 was dried to 60°C. Both specimens were tested through ten cycles of drying and rewetting with distilled water. RC-1 was soaked in a 1.0 Normal NaCl solution after the tenth drying and tested for one additional drying phase.

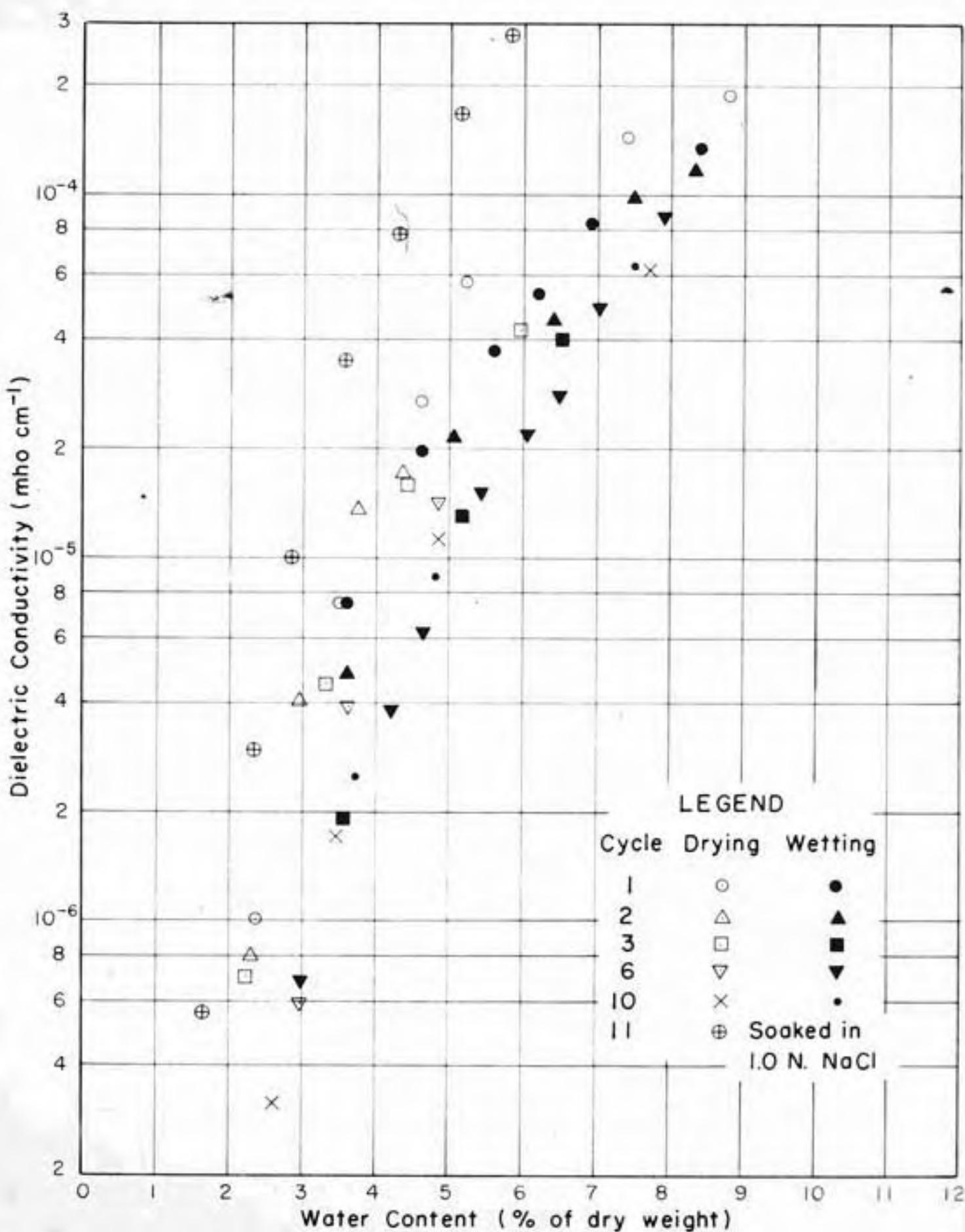


FIG. 21 DIELECTRIC CONDUCTIVITY AT 10 cps VS. WATER CONTENT FOR SAMPLE RC-1

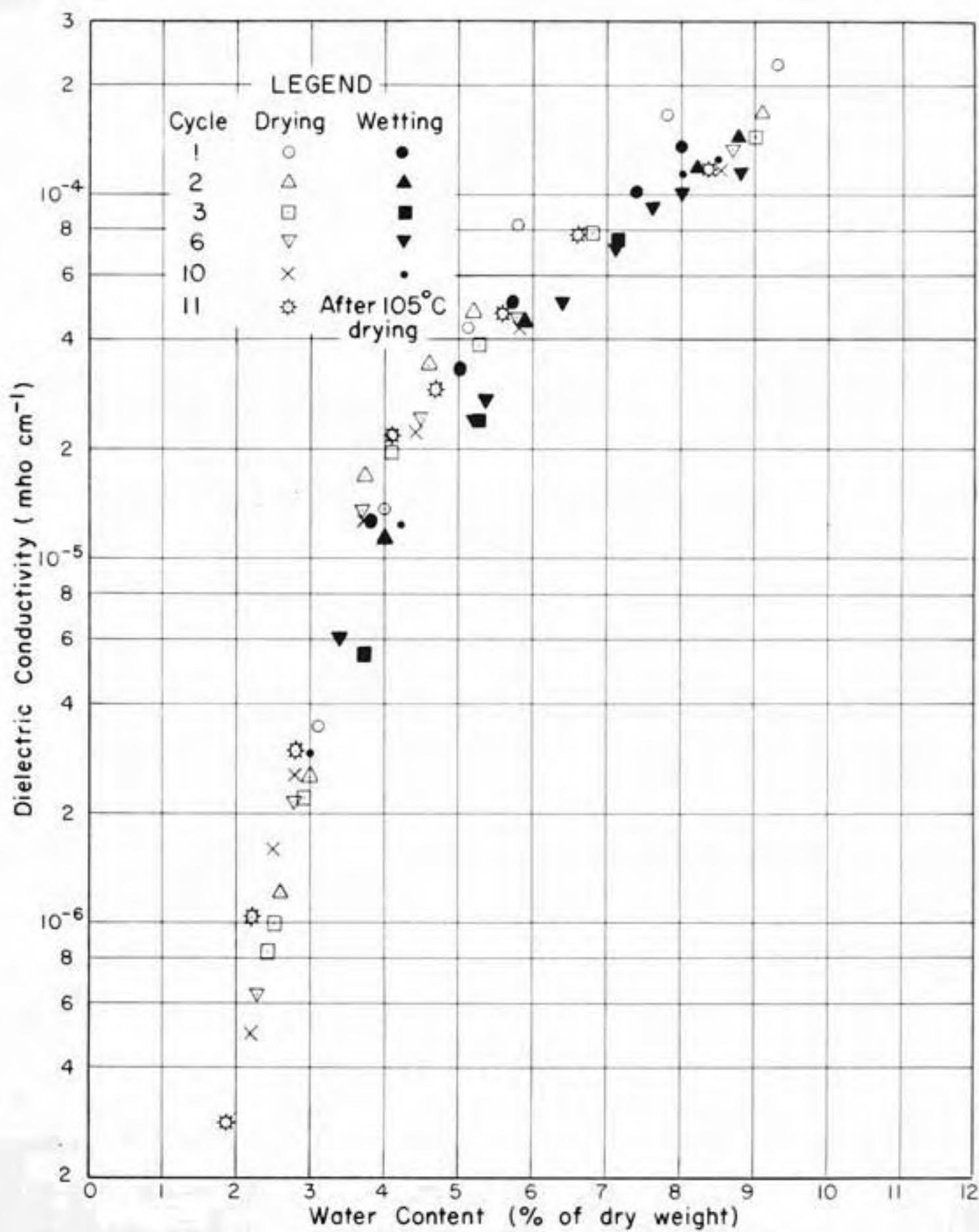


FIG. 22 DIELECTRIC CONDUCTIVITY AT 10 cps VS. WATER CONTENT FOR SAMPLE RC-2

DISCUSSION OF RESULTS

Test Results

The shrinkage of Portland cement concrete is influenced by many variables such as curing, cement, aggregate, and moisture history, which were not thoroughly investigated in this study. However, the requirements for a satisfactory meter to appraise the effects of moisture gradients on warping can be estimated from the data available.

Examination of Figures 6 through 10 shows that by far the greatest amount of volume change occurs at water contents less than about 7 percent with the possible exception of the first drying. In concrete, the presence of the relatively inert coarse aggregate moves the volume change-water content curve to the left.

Neglecting the first drying, the maximum value for the moisture coefficient of expansion of mortar indicated in this study is approximately 5×10^{-4} inches per inch per percent moisture content, which occurs at water contents below 1 percent. This corresponds approximately to the effect of a temperature variation of 50°F. Shideler's data (9) indicate the coefficient of expansion for concrete to be half that for mortar. Thus, if moisture contents are to be used to predict volume changes with an accuracy equivalent to the volume change resulting from a temperature change of 5°F, the moisture determination must be accurate to within ± 0.2 percent.

For practical problems such precise measurements would not be required. In a highway pavement slab there would be two factors acting to reduce the accuracy required. First, the minimum water content would generally be appreciably above that corresponding to the range of maximum

rate of shrinkage, and the moisture coefficient of expansion would therefore be considerably smaller than the maximum given above (the maximum coefficient of expansion at a water content of 2 percent is only about $\frac{1}{3}$ that at 1 percent). Second, with each drying of the concrete the rate of shrinkage decreases. Accordingly, an accuracy in moisture measurement of $\pm \frac{1}{2}$ percent would probably be satisfactory for many cases, while an accuracy of ± 0.25 percent would be sufficient to comprise practically all field conditions of interest in highway pavement design.

The shrinkage data show that, in using moisture content as an indicator of volume change, measuring the moisture content is only part of the problem. The other part is to predict the shrinkage after the water content has been determined. The problem arises from the fact that the volume-water content relationship of a concrete is constantly changing. Therefore, it must be possible to predict how this relationship will change with time.

The measurements made in this study were not sufficiently precise or comprehensive to provide a basis for such predictions, but they do show certain trends. The semi-log plots of the shrinkage data (e.g. Figure 10) are particularly useful in this respect. The absolute values of shrinkage showed considerable variation between disks, but the curves are all similar in shape and undergo the same evolution. The regularity of the changes in the shrinkage-moisture relationship upon successive wetting-drying cycles suggest that these changes may be predictable; however, this problem will require additional study before the matter can be resolved.

Certain trends in the apparent capacitance relationships are shown very clearly by the data from this investigation. First, the sensitivity of apparent capacitance to moisture changes is markedly

affected by frequency. At low frequencies it is restricted to very low water contents. Other factors remaining constant, the range of sensitivity increases with frequency at least within the range of frequencies tested in these studies. For the conditions of these tests the sensitive range at 10^6 cps extended to at least 6 percent for the first cycle and in most instances up to 8 or 9 percent for subsequent cycles. When the disks were soaked in the salt solution, the sensitive range was restricted to water contents below about 6 percent. At 60 cps the sensitive range was restricted to water contents less than 2 percent in all cases.

Second, there is a shifting of the curves with subsequent cycles of drying and wetting. Third, there is a distinct hysteresis in the drying-wetting cycle. Both the hysteresis and shifting of the curves appear to become less pronounced upon subsequent cycles.

The hysteresis occurred to a lesser or greater degree in all cases, but the marked shifting of the curves on subsequent cycles only occurred with thin discs dried to 105°C during the test period. For thin disks dried to only 60°C and for the thicker disks regardless of their maximum drying temperature this tendency was greatly reduced if apparent at all.

Considering the 10^6 cps data, it becomes apparent that at the higher water contents these curves are controlled almost completely by the conductance of the disks and the contact capacitance between the disks and the test capacitor. The dielectric constant of the disks has a negligible effect at the very high water contents. For the thicknesses of disks C-1 and CT-2, the capacitance of Disk CT-2 should be about $2/3$ that of C-1 for the same water content, assuming dielectric constants approximately equal for equal water contents. However, the curves show that the apparent capacitance for disk CT-2 is the larger one at the high water content. This can be the case only if the contact capacitance for CT-2 is the greater of the two and if the resistances of the samples are

low enough to make the apparent capacitance approach this contact capacitance. This situation is represented near the left-hand edge of Figure 23. Extending the curves on Figures 15 and 16 to estimate their asymptotes, the contact capacitances of disks C-1 and CT-2 were estimated to be $300 \mu\text{uf}$ and $320 \mu\text{uf}$, respectively. The values of C_b for disk C-1 approach an asymptote at high water contents, especially during the first two cycles and subsequent salt concentration test, and permit a good estimate of the contact capacitance. The thicker disk (CT-2) has a higher resistance and therefore does not approach its asymptote as closely, and its contact capacitance cannot be estimated with the same precision.

The curve for C-1 after salt saturation is much higher than even the first drying cycle and gives additional evidence of the dependency of these values on the conductance of the sample.

For the extreme conditions of drying a thin disk to 105°C and soaking in a 1.0 Normal NaCl solution, the range of water contents at which the same apparent capacitance can be obtained is very wide, being about 5 percent or more at high water contents and exceeding 1 percent even at the low water contents for disk C-1 at 10^6 cps. However, for the less severe conditions of 60°C drying and for a thick disk at the same frequency, as represented by disk CT-2, the range of water contents at which the same apparent capacitance can be obtained is only about .5 percent up to water contents of about 6 percent and from 1.0 to 1.5 percent at the highest water contents.

Both the 10 cps dielectric conductivity (Figures 21 and 22) and the 10^6 cps dielectric conductivity (Figures 19 and 20) show the same general trends as the apparent capacitance curves. The conductivity decreases with successive cycles and exhibits a hysteresis in the cycle

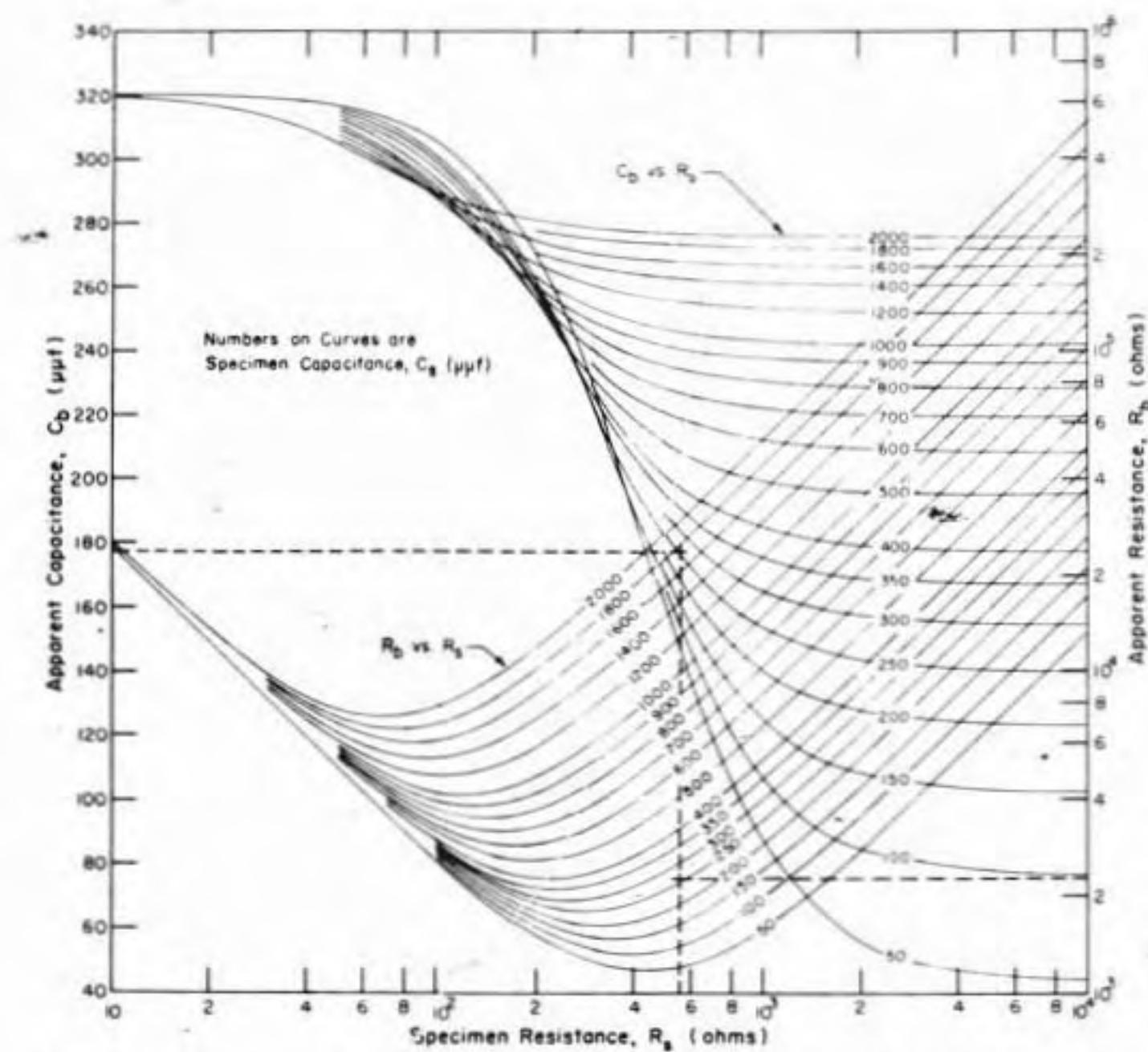


FIG. 23 GRAPH FOR DETERMINATION OF SPECIMEN CAPACITANCE AND RESISTANCE FROM APPARENT CAPACITANCE AND RESISTANCE FOR A CONTACT CAPACITANCE OF 320 μuf AND A FREQUENCY OF 10⁶ cps

for the samples dried to 105°C. The samples dried to 60°C show less variation between cycles than those dried to 105°C; however, the hysteresis remains essentially unchanged. For the 10⁶ cps data the thick samples show less variation than the thin disks. This last comparison cannot be made for the low frequency data as all of these test specimens are approximately the same thickness as the thick disks. Comparing only the results for thick disks with the 10 cps dielectric conductivity data, the 10⁶ cps results show less variations than do the low frequency tests.

The maximum variation in conductivity at the same water content for these tests is on the order of a factor of 10, which is smaller than that reported by other investigators. The dielectric conductivities at 10⁶ cps are higher at low water contents and lower at high water contents than the corresponding 10 cps dielectric conductivities, which is consistent with the results of Keller and Licastro (35) shown on Figure 2. These differences are least at the high water contents, which is also indicated by their data.

For the dielectric conductivities calculated from the 10⁶ cps permittivity tests the most severe conditions indicated a maximum range of water contents which could yield the same conductivity value of about 5 percent or more, which is about the same as the spread for the apparent capacitance data. Also, for the less severe conditions of 60°C drying and no salts added, the spread was only about 0.5 percent up to water contents of 6 percent. The spread in dielectric conductivity as measured by the resistance tests at 10 cps was about twice as great for the conditions of 60°C drying compared with the 10⁶ cps data.

The relatively larger variation in the low frequency dielectric conductivity results are not so much the result of shifting of the data with successive cycles of drying and wetting as the result of a larger

hysteresis in the cycle. Holmes (48), working with porous sandstone cores, has investigated the influence of allowing evaporation at the surface of the samples during resistance measurements. He found that the distribution of moisture over the cross-section of the core is important and that whether or not evaporation is permitted has a pronounced effect on the resistance measurements. This factor may contribute to the greater hysteresis effects found in the low frequency measurements.

Evaporation could also be a contributing factor in the hysteresis effect in the permittivity measurements. The effect in the permittivity test should be smaller because of the larger ratio of area to perimeter of these samples and because during testing evaporation is essentially prevented from the faces of the disks by the plates of the capacitance test cell. Holmes' tests indicate that holding the disks in the test cell for only two minutes would eliminate much of the evaporation effect for all but the lowest water contents.

The dielectric conductivity data also show the interesting result that, after ten cycles of drying to 60°C , drying the sample to 105°C does not cause any significant shifting of the curves on the subsequent drying after re-wetting with distilled water (see Figure 22).

The conductivity test results support the contention that the curve shifting and hysteresis effects shown by the apparent capacitance data are largely the results of changes in dielectric conductivity; however, the relative dielectric constant-water content relationships, (Figures 17 and 18) as calculated from the 10^6 cps permittivity data, show that other factors must also be acting as these curves also show the same trends. The dielectric constant-water content data exhibit the same shifts with successive cycles, the same hysteresis trends, and the same relationships between the magnitude of these variations and the sample size and

drying temperature as do the apparent capacitance and conductivity data. The same factors which affect the conductivity are evidently also acting to influence the dielectric constant values. Also, anomalously high dielectric constants are indicated. In nearly all instances values greater than the dielectric constant of bulk water are indicated.

The anomalously high dielectric constant and the variations in the dielectric constant-water content relationship can be rationalized if it is assumed that space-charge or interfacial polarization has an important role in controlling the dielectric constant of concrete. This type of polarization requires the presence of charge carriers free to move under the influence of the applied electric field. In moist concrete these charge carriers could be provided by the ions contained in the pore water. To produce interfacial polarization the charge carriers must be impeded in their motion to cause concentrations of charges which would increase the storage capacity of the capacitor. The solids in the concrete would obstruct the motion of the charge carriers and fulfill this second condition for space-charge polarization. At a given frequency, field strength, and temperature the magnitude of the space charge polarization produced would depend on the ion concentration and the pore structure (pore size, continuity, and tortuosity) and probably the types of ions, — the same factors which are dominant in controlling conductivity. The test results do not permit any conclusions regarding the relative influences of these various factors. All that can be said is that within the range of conditions employed in the tests those factors which result in net decreases in conductivity also result in decreases in the dielectric constant and vice versa. The large dielectric constants obtained for disk C-1 after soaking in a NaCl solution add further support to the validity of these statements.

At low water contents the relative dielectric constant data are consistent. It is also shown by the flat slope of these curves at the very low water contents that the dielectric constant is relatively insensitive to changes in water content in this range. This may reflect the influence of the surface forces of the cement gel in orienting the water in the gel pores, thereby reducing the dielectric constant of the water to the same order of magnitude as that of the solid matter.

As the water content increases, the random scatter of the relative dielectric constant also increases. This scattering of points makes it difficult to evaluate the true variations in this relationship, but they are at least as great, and possibly greater, than the apparent capacitance and 10^6 cps dielectric conductivity relationships. Soaking in the 1.0 Normal NaCl solution causes relatively greater changes in the dielectric constant than in the other properties, and there are some indications that the change during the first drying is also greater for this property.

The results of the tests in this study give numerous illustrations of the problem encountered in measuring the dielectric properties of concrete in the laboratory and suggest some additional problems which would be encountered in field measurements.

An important feature of the data obtained is the occasional erratic scatter of some of the data points. This is especially true at the higher water contents and for the dielectric constant results and is due largely to the sensitivity of the calculation procedures to small inaccuracies in the test data. Errors in the test readings can be magnified many times when R_s is very small. At the highest water contents it is sometimes impossible even to approximate the dielectric constant. The dielectric conductivity determinations are also subject to errors for this same reason; however, the resulting error in R_s is not as large as the error in C_s .

As the specimen resistance increases, the sensitivity of the calculations to changes in the contact capacitance is reduced; therefore, it is desirable to proportion the test capacitor to permit working as far to the right on Figure 23 as possible. Unfortunately the control over this factor is limited. From Equation 9

$$C_s = k' C_0 \quad (16)$$

and from Equation 10

$$R_s = \frac{\epsilon_0}{\sigma C_0} \quad (17)$$

The only factor in these expressions which can be controlled is the geometric capacitance C_0 . To increase the specimen resistance the geometric capacitance must be reduced. This also decreases the value of the specimen capacitance reducing the accuracy with which it can be measured. Thus a compromise in the selection of C_0 will be necessary in the design of moisture-measuring equipment.

As R_s is increased, a conflict also develops between the necessity to know the contact capacitance to calculate the dielectric constant and dielectric conductivity and the desirability of working on the more accurate portions of the interpretative graphs. If the specimen resistance is increased sufficiently that the apparent capacitance curve no longer approaches its asymptote, the contact capacitance cannot be accurately estimated. For these reasons, it does not appear that any approach adaptable to field use would yield dielectric constant or dielectric conductivity results significantly better than those obtained for the thick disks in this study.

The problems involved with the determination of dielectric constant and dielectric conductivity combined with the smooth curves and relative freedom from erratic scattering of points in the apparent capacitance-water content relationship suggests the possibility of using

this relationship as a moisture indicator. This would lead to calibration problems because the apparent capacitance is not a property of the material. It is a function of the complex permittivity of the material, but it is also a function of the contact capacitance and the geometry of the sensing electrodes. Any empirical calibration for the apparent capacitance-water content relationship would have to be accomplished under exactly the same conditions with respect to the contact capacitance and geometric capacitance of the test capacitor as would be experienced in the prototype tests. This would require that calibration be accomplished by embedding sensing elements as nearly identical to those used in the field test as possible in relatively large concrete specimens and calibrating them by varying the water content of these specimens, or the elements would have to be calibrated during the field tests by relating their readings to gravimetric moisture determinations on companion dummy test sections. Both of these approaches would be slow, laborious, and inaccurate.

Indirect calibration techniques could be used to establish the apparent capacitance-water content relationship. The conductivity and relative dielectric constant-water content relationships could be established by methods similar to those used in this study and the apparent capacitance-water content relationship calculated for the sensing capacitors to be used in the field tests. This would require that the contact and geometric capacitance of the sensing capacitor be known. The geometric capacitance could be determined by measurements made with the sensing capacitor immersed in liquids of known dielectric constant. The contact capacitance could be determined similarly using salt water solutions if it is assumed or could be established by test that the electrode to liquid contact capacitance would essentially duplicate the electrode to concrete contact capacitance for elements cast in concrete. If this could not be established, the contact capacitance could be estimated by

monitoring the first drying after the element was cast in the concrete and the contact capacitance taken as the asymptote of the C_0 -water content curve.

This study has shown that all the dielectric properties of concrete are sensitive to large increases in the NaCl concentration of the pore water, drying to temperatures significantly above 60°C during the first few cycles, and the thickness of the sample tested. These factors dictate that if a valid correlation is to be established the calibration tests must be conducted under conditions similar to those for which the correlation is to be used.

The dry weight of concrete increases with age. The magnitude and rate of this increase depends on the environment in which the aging takes place. If corrections are not made for variations in dry weight during testing, the test results will shift successively to the right and indicate much greater variations in the relationships between the dielectric properties and water content than actually occur. The water contents used in all of the relationships studied in this investigation represent the water evaporable at 105°C relative to the 105°C dry weight at the time of the measurement. Some of the random scatter of data points for the samples dried to 60°C may be attributed to difficulties in relating the water contents of these samples to a 105°C dry weight basis.

Evaluation of Various Electrical Methods

Considering the basic trends and variations under all conditions tested, there is little to choose between the various properties investigated as moisture indicators. The apparent capacitance, dielectric conductivity (low and high frequency), and relative dielectric constant all exhibit significant variations under certain conditions and none can be

used as indicators of water content in concrete under all possible conditions. It is also true, however, that the most serious variations occur only under extreme conditions. Hence, a realistic evaluation can be made only with respect to the conditions under which actual moisture determinations are likely to be made.

In many applications—for example, a concrete pavement slab—the section tested would be relatively massive, would be dried under atmospheric conditions, and, at least in many localities, would not have salts added to the pore fluid. These conditions would correspond fairly closely to those of the thick samples dried to only 60°C and subject only to leaching. The results for these samples show a minimum of variation and shifting of their relationship with water content on successive cycles of wetting and drying. Also, for many applications the maximum moisture content of interest would not exceed 6 or 8 percent. For example, it is unlikely that the moisture content of a concrete pavement would normally exceed 8 percent (6), and in the study of certain properties related to moisture content (such as shrinkage), the moisture range of the greatest importance is less than this value (7). Under such conditions, the dielectric constant, dielectric conductivity, or apparent capacitance as determined from 10⁶ cps permittivity measurements could be used successfully as moisture indicators. The indicated accuracy of the determinations would be better than $\pm .25$ percent for water contents not exceeding 6 percent.

The indicated accuracy for the dielectric conductivity as determined from low frequency resistance tests is about $\pm .5$ percent for these same conditions. This is as good as that claimed for supposedly superior methods such as the plaster of Paris or nylon resistance cells (49), but it is only about one-half as good as the 10⁶ cps permittivity data; however, as previously discussed, there are indications that the variation

in the low frequency conductivity relationships found in these tests may be greater than would be encountered under actual field conditions.

When practical considerations of instrumentation, calibration, and interpretation are considered, the relative advantages and disadvantages of the various relationships as moisture indicators become more apparent. The results of this study permit making comparisons between five electric moisture-meter schemes as follows:

1. The Capacitance Method

This method would use the apparent capacitance (C_b)-water content relationship as determined by an empirical calibration. The apparent capacitance in the field would be determined from permittivity measurements on a suitable sensing capacitor embedded in the concrete test section, but the dielectric properties of the concrete would not actually be determined.

2. The Dielectric Constant Method

This method would utilize the relative dielectric constant-water content relationship established by laboratory tests similar to the permittivity tests used in this study. The relative dielectric constant of the concrete in the field would be calculated from appropriate permittivity tests.

3. The Dielectric Conductivity Method

This method is identical to the dielectric constant method except that the dielectric conductivity would be calculated from the field permittivity tests.

4. The Complex Permittivity Method

The apparent capacitance-water content curve would be calculated for the field sensing capacitor from the dielectric constant and dielectric conductivity-water content relationships of

the concrete as determined by laboratory calibration tests and a knowledge of the contact and geometric capacitances of the field capacitor. The apparent capacitance of this sensing capacitor embedded in the concrete test section would then be determined from field permittivity tests.

5. The Resistance Method

This method would use the dielectric conductivity as determined from resistance tests at relatively low frequencies. The calibration could be established by laboratory tests similar to the resistance tests in this study. Field measurements would determine the resistance of a sensing element embedded in the concrete test specimen, and the dielectric conductivity would be calculated from this resistance.

The first four methods are all based on permittivity measurements and require the same instrumentation, namely, relatively high frequencies and insulated electrodes, and share all of the associated problems with shielding and the contact capacitance. In addition, the contact and geometric capacitances of the sensing element must be predetermined for all but the capacitance method.

The capacitance method is the same method that has been studied by numerous investigators and reported in the literature. It appears to offer an advantage in the simplicity of calibration and the fact that it does not require extensive data reduction. In actuality, however, calibration is the major disadvantage to this method. Because this is a purely empirical method that depends upon unknown instrumentation factors as well as the properties of the concrete, the calibration must be performed with test capacitors and for conditions identical to those which will be involved in the field tests. This leads to cumbersome and inaccurate calibration procedures.

The difference between the other three permittivity methods (methods 2, 3 and 4) is in the methods used to interpret the results. The dielectric constant and dielectric conductivity are calculated from field permittivity measurements and compared with laboratory calibration curves to determine the corresponding water content. In the complex permittivity method the apparent capacitance is calculated from the field permittivity tests and correlated with an apparent capacitance-water content relationship calculated from laboratory permittivity tests and the characteristics of the field sensing capacitor. The results of this study show that apparent capacitance is less sensitive to small errors in the test data than either dielectric constant or dielectric conductivity. Furthermore, the dielectric constant and dielectric conductivity need only be calculated from laboratory test data which can be more closely controlled than the field tests; erratic scattering of occasional points can readily be identified and neglected in establishing average curves for the dielectric constant and dielectric conductivity water content relationships. For these reasons the complex permittivity method is potentially an excellent method of measuring moisture contents in concrete for many possible applications. However, before it can be considered a practical method, the instrumentation required for field measurements must be developed, and the problem of temperature effects must be studied and appropriate corrections developed if required.

The resistance method utilizes dielectric conductivity as the moisture indicator. In this respect, it is identical with the dielectric conductivity method. The difference between these methods is the way this property is determined. In the resistance method the dielectric conductivity is determined from resistance measurements instead of permittivity

measurements. This is accomplished by designing the instrumentation to make the charging current through the sample small with respect to the loss current. This permits making the measurements at relatively low frequency and with bare electrodes, thereby greatly simplifying both the instrumentation and the necessary calculations. Simplicity is the main advantage of this method. The results of this study show wider variations of the dielectric conductivity-water content relationship as determined by the resistance method than from the permittivity relationship, suggesting that the resistance method would be less accurate than the complex permittivity method. However, there are indications that this greater variability may not exist under field conditions. As long as this possibility exists, the resistance method should not be discarded without further detailed study of all the factors involved.

CONCLUSIONS

On the basis of the information obtained in this study, it is concluded:

1. To be satisfactory for use in connection with moisture-volume relationship investigations in concrete pavements, a moisture meter must be capable of measuring water contents in hardened concrete between about 1 and 6 percent. For mortars the range of interest extends to approximately 10 percent.
2. To indicate volume changes due to moisture variations with an accuracy which will limit the error to an amount equivalent to a temperature change of 5°F , the accuracy of the moisture measurements must be at least $\pm 0.5\%$. If measurements are to be made in the first drying cycle to water contents of 1% or less, an accuracy of about 0.25% would be required.
3. In studies where water content is to be used to predict the shrinkage of concrete, the problem of establishing the water content-volume relationship at any given time is equally as important as the actual moisture measurement. This relationship is constantly changing, and among other things, is a function of the moisture history of the concrete. There are indications that it may be possible to predict the moisture-volume relationship at any time if the moisture history is known. It should at least be possible to predict changes in volume with change in moisture content at any given time.
4. The relationships between the dielectric properties of concrete and water content are not constant. They are influenced by numerous factors other than water content -- notably electrical frequency,

salt content of the pore fluid, and environment under which the concrete is aged. However, the conditions which lead to large variations in the relationships between dielectric properties and water-content -- such as a drying temperature of 105°C or soaking in 1.0 Normal NaCl solutions -- are unrealistic with respect to the environments in which moisture determinations would be desired in many practical applications.

5. For moderate environmental conditions the dielectric properties of concrete as determined from permittivity measurements are potentially capable of indicating the water content of hardened concrete with an accuracy of $\pm 0.25\%$ for moisture contents up to 6 percent. However, for these accuracies to be attainable, the instrumentation must be carefully designed for the specific dielectric properties of concrete, due consideration being given to dielectric constant, dielectric conductivity, frequency, and electrical characteristics of the contact between sensing element and the concrete.
6. Low frequency resistance measurements indicate a potential accuracy only half that of the permittivity tests, but there are indications that this accuracy could be improved. Due to the relative simplicity of the instrumentation, and of the data reduction involved, this method should not be discarded until its possibilities have been re-evaluated in the light of the findings of this study.
7. The dielectric properties correlate with evaporable water. Corrections must be made for changes of dry weight of the concrete or mortar during the test period.

8. The relationships between the dielectric properties and water content and probably the shrinkage relationship as well are strongly influenced by the details of the test procedures and conditions. Great care must be exercised to insure that calibration tests realistically represent the conditions for which the actual moisture meter method or calibration procedure is to be used.

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NOTATION

C	capacitance
C_b	apparent capacitance (parallel equivalent capacitance of combined specimen and contact)
C_c	contact capacitance
C_o	geometric capacitance
C_s	specimen capacitance
C_x	unknown capacitance
C_{xs}	series equivalent capacitance of unknown capacitor
cps	cycles per second
D	dissipation factor = $\tan \delta$
D_x	dissipation factor of unknown capacitor
E	applied electric field strength
I	current
J	current density
J_c	charging current density
J_A	loss current density
j	$\sqrt{-1}$
R	resistance
R_b	apparent resistance (parallel equivalent resistance of combined specimen and contact)
R_s	parallel equivalent resistance of specimen
R_{xs}	series equivalent resistance of unknown capacitor
V	instantaneous voltage
α	polarisability
α_a	atomic polarisability
α_e	electronic polarisability
α_o	orientation (dipole) polarisability

α_s	space charge (interfacial) polarizability
δ	loss angle
$\tan \delta$	loss tangent = D
ϵ_0	dielectric constant of free space (vacuum)
ϵ'	dielectric constant
ϵ''	loss factor
ϵ^*	complex permittivity
θ	power factor angle
k'	relative dielectric constant
k''	relative loss factor
k^*	complex relative permittivity
μ_{uf}	unit of capacitance equal to 10^{-12} farads
ν	frequency
σ	dielectric conductivity = $\omega \epsilon''$
ω	angular frequency = $2\pi\nu$