NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM
REPORT 138

INSTRUMENTATION FOR MEASUREMENT OF MOISTURE

LITERATURE REVIEW AND RECOMMENDED RESEARCH

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NATIONAL RESEARCH COUNCIL
NATIONAL ACADEMY OF SCIENCES—NATIONAL ACADEMY OF ENGINEERING

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AREAS OF INTEREST:
MATERIALS AND CONSTRUCTION
SOILS, GEOLOGY, AND FOUNDATIONS

HIGHWAY RESEARCH BOARD
DIVISION OF ENGINEERING NATIONAL RESEARCH COUNCIL
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NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

Systematic, well-designed research provides the most effective approach to the solution of many problems facing highway administrators and engineers. Often, highway problems are of local interest and can best be studied by highway departments individually or in cooperation with their state universities and others. However, the accelerating growth of highway transportation develops increasingly complex problems of wide interest to highway authorities. These problems are best studied through a coordinated program of cooperative research.

In recognition of these needs, the highway administrators of the American Association of State Highway Officials initiated in 1962 an objective national highway research program employing modern scientific techniques. This program is supported on a continuing basis by funds from participating member states of the Association and it receives the full cooperation and support of the Federal Highway Administration, United States Department of Transportation.

The Highway Research Board of the National Academy of Sciences-National Research Council was requested by the Association to administer the research program because of the Board's recognized objectivity and understanding of modern research practices. The Board is uniquely suited for this purpose as: it maintains an extensive committee structure from which authorities on any highway transportation subject may be drawn; it possesses avenues of communications and cooperation with federal, state, and local governmental agencies, universities, and industry; its relationship to its parent organization, the National Academy of Sciences, a private, nonprofit institution, is an insurance of objectivity; it maintains a full-time research correlation staff of specialists in highway transportation matters to bring the findings of research directly to those who are in a position to use them.

The program is developed on the basis of research needs identified by chief administrators of the highway departments and by committees of AASHO. Each year, specific areas of research needs to be included in the program are proposed to the Academy and the Board by the American Association of State Highway Officials. Research projects to fulfill these needs are defined by the Board, and qualified research agencies are selected from those that have submitted proposals. Administration and surveillance of research contracts are responsibilities of the Academy and its Highway Research Board.

The needs for highway research are many, and the National Cooperative Highway Research Program can make significant contributions to the solution of highway transportation problems of mutual concern to many responsible groups. The program, however, is intended to complement rather than to substitute for or duplicate other highway research programs.

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The study reported herein was undertaken under the aegis of the National Academy of Sciences—National Research Council. The National Cooperative Highway Research Program, under which this study was made, is conducted by the Highway Research Board with the express approval of the Governing Board of the NRC. Such approval indicated that the Board considered that the problems studied in this program are of national significance; that solution of the problems requires scientific or technical competence, and that the resources of NRC are particularly suitable to the conduct of these studies. The institutional responsibilities of the NRC are discharged in the following manner: each specific problem, before it is accepted for study in the Program, is approved as appropriate for the NRC by the Program advisory committee and the Chairman of the Division of Engineering of the National Research Council.

The specific work to be performed in each problem area is defined by an advisory panel that then selects a research agency to do the work, monitors the work, and reviews the final reports. Members of the advisory panels are appointed by the Chairman of the Division of Engineering of the National Research Council. They are selected for their individual scholarly competence and judgment, with due consideration for the balance and breadth of disciplines.

Responsibility for the definition of this research project and for the publication of this report rests with the advisory panel. However, the opinions and conclusion expressed or implied are those of the research agency that performed the research, and are not necessarily those of the Highway Research Board, the National Research Council, the Federal Highway Administration, the American Association of State Highway Officials, nor the individual states participating in the Program.

Although reports in this category are not submitted for approval to the Academy membership nor to the Council, each report is reviewed and processed according to procedures established and monitored by the Academy's Report Review Committee. Such reviews are intended to determine, *inter alia*, whether the major questions and relevant points of view have been addressed, and whether the reported findings, conclusions and recommendations arose from the available data and information. Distribution of the report is permitted only after satisfactory completion of this review process.

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FOREWORD

By Staff
Highway Research Board

This report is the result of a comprehensive review of literature on all known methods of moisture measurement likely to be applicable to highway problems. It contains (1) a listing of highway moisture measurement problems, (2) a description of a number of applicable measurement principles, and (3) an evaluation model for estimating the best moisture measuring instrument for specific operating conditions. Soils and materials engineers will find information in the report useful during selection of instruments for particular moisture measurement problems. In addition, the study findings provide a sound basis for development of research programs aimed at more satisfactory resolution of moisture measurement needs in the highway field.

The majority of acceptance procedures for construction of highway embankments, subgrades, and base courses require determination of moisture content of the material being compacted. Moisture measurements also are associated with the storage and use of plant-processed materials such as aggregates used in pavements. Both the design and the performance of pavements are influenced by the moisture content of the substructure components. Other problems for which information on moisture content is of value to highway engineers are migration in embankments, soils susceptible to volume change, buildup immediately beneath pavements, groundwater fluctuations, research on performance of cemented pavement components, frost action, and long-term performance studies. The objectives of this study were to (1) review the state of knowledge with regard to suitability of existing techniques for the measurement of moisture in highway components, (2) identify techniques that may be applicable to highway moisture measurement problems but need further evaluation, and (3) identify problems for which the development of new moisture measuring instrumentation is needed and make recommendations concerning techniques that could lead to the resolving of this need.

The Research Triangle Institute staff conducted an extensive literature review within the highway field and in other disciplines to identify all known moisture measurement techniques that might be applicable to highway problems. The techniques were categorized by the physical, chemical, and electrical phenomena on which they are based. Altogether, 35 performance characteristics were selected and described for moisture measurement instruments. Where available from the literature reviewed, information on the various performance characteristics for each measurement technique is included in the report to provide a basis for the relative evaluation of several techniques for the accomplishment of a particular moisture measurement problem.

It appears that the ultimate need is for a very small, inexpensive device for measuring moisture that may be remotely monitored and that will rapidly and accurately indicate the amount and state of the water in the surrounding material. Although no such instrument was identified during this study, several instruments are available that are suitable for application to some of the current moisture measurement problems and a procedure has been developed for their relative evaluation.

It was substantiated that data are inadequate for evaluating the performance of many types of available instruments with regard to such characteristics as accuracy. As a result, studies to collect appropriate data on performance characteristics of instruments for measuring moisture in highway components are recommended. In addition, research programs are recommended to modify existing and to develop new sensors for some of the highway moisture measurement problems. Some 12 research programs are recommended in the report.

To provide a basis for further research in this problem area, the NCHRP advisory panel for the project reviewed the recommended research program summarized in Table 15 and prepared the following analysis:

Recommended Programs 4, 5, 6, and 8 are concerned with the measurement of moisture in pavement bases, subgrades, and earth materials. This is considered the top priority problem in the highway moisture measurement field. On the basis of the panel analysis, funds have been appropriated and NCHRP Projects 21-2 and 21-2(2). "Instrumentation for Moisture Measurement—Bases, Subgrades, and Earth Materials (Sensor Development)," have been initiated for the design and experimental verification of three sensors using the nuclear magnetic resonance, microwave, and fringe capacitance principles.

Recommended Programs 7, 10, 11, and 12 are concerned with moisture measurements in cemented materials such as portland cement and asphaltic concrete pavements. This problem is of major technical and economic importance. Research should be initiated as soon as funds can be made available.

Recommended Programs 1, 2, 3, and 9 are concerned with instrumentation to measure moisture in plant-processed materials. It is recommended that equipment manufacturers and materials suppliers be advised of the need for greater accuracy in the measurement of moisture in materials in storage bins and batch plants.

The advisory panel also recognized the importance to pavement design and performance of the interrelationship between moisture content, moisture state, and the stress-strain characteristics of the sub-pavement system under traffic. Pore pressure is a moisture state in need of further study because of its effect on stress-strain relationships of granular and earth materials in a pavement structure. Consequently, there is a need for exploratory research on instrumentation for in-situ measurement of pore pressure within the sub-pavement system and this problem should be considered within the scope of research on instrumentation for measurement of moisture.

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INSTRUMENTATION FOR MEASUREMENT OF MOISTURE

LITERATURE REVIEW AND RECOMMENDED RESEARCH

SUMMARY

The objective of this study was to evaluate the suitability of existing instrumentation and techniques and to stimulate development of modified or new procedures to measure the amount and state of water in highway components, such as embankments, subgrades, base courses, pavements, and structures. In accomplishing this objective, the researchers conducted an extensive literature review. Those techniques that appear to be immediately applicable to highway problems were evaluated using a linear decision model for comparing instrument performance characteristics with specific moisture measurement needs. A comprehensive list of performance characteristics with which to make this comparison was compiled.

The description of moisture measurement instrumentation is divided into categories according to the chemical and physical processes involved in the principle of measurement. Commercial instrumentation is available in most of these categories; however, as a rule, the instruments are not designed for use in highway engineering applications. The most suitable techniques currently available for use in highway problems are the gravimetric methods and nuclear scattering methods. For nondestructive surface or near-surface measurements, the nuclear method appears to be unexcelled. Where samples can be removed for analysis, gravimetric methods are most suitable. Oven drying is most commonly used, and normally is the standard method for correlating various highway component characteristics with moisture content. For field use, chemical extraction techniques appear to be most effective. Problems occur with all of these techniques; however, their performance is such that they can be of significant value in certain applications.

Uncontrolled moisture in highway construction and maintenance is a serious economic problem worthy of a sustained research effort to develop adequate instrumentation. Instrumentation needs to be developed and evaluated for such broad applications as unconsolidated materials in stockpiles, bins, and conveyors; compacted materials in depth or in layers; and cemented materials in layers and other shapes. Candidate methods for evaluation in these areas are nuclear, radiowave, microwave resistance, capacitance, hygrometric, tensiometric, thermal conductivity, and rapid chemical and thermal extraction techniques.

Certain modifications may improve the over-all performance of nuclear methods, with concurrent additional costs. One of these is the additional measurement of neutron-absorbing materials by means of the neutron gamma technique. Ignition tests also may be helpful in interpreting some measurements in clay materials.

Hygrometric techniques using aluminum oxide films might benefit from im-

proved construction methods. Extreme care in fabrication and the use of thick sputtered films should add to the quality of performance of these units.

Many of the measurement problems lacking sufficient instrumentation could be handled through the development of remote-sensing techniques. One particularly promising technique is that of implant telemetry. Moisture measurement principles that are compatible with this type of remote sensing should receive future research effort.

CHAPTER ONE

INTRODUCTION AND RESEARCH APPROACH

Water in its various states, when insufficient or in excess in the components of a highway system, adversely affects the service behavior. Despite recognition of the importance of the relationship between the presence of water and service behavior, the engineer has been hampered in his effort to provide predictable performance by the lack of instrumentation and techniques for adequate water or moisture measurement. The economic significance of the problem in highway construction and maintenance is evidenced by the large financial investment aimed at removal of excess water that causes loss of supporting capacity of subgrade soils and aggregate bases, embankment instability, and deterioration of pavements.

Techniques currently in use in the highway field to measure moisture content in situ generally are insufficient to meet the researchers' needs because of their high cost, time requirements, disturbance of the site, long-term instability, or a combination of these factors. Instrumentation is specifically needed for remote readout of local sensing, for remote sensing of subsurface conditions, for high-precision measurements, and for long-term continuous monitoring. Devices that can be installed permanently and portable devices for sampling at random locations are needed to provide data to allow for the development of new designs and the use of new materials.

The ultimate objective of research on this problem is to evaluate the suitability of existing instrumentation and techniques and to stimulate development of modified or new procedures to measure the amount and state of water in highway components such as embankments, subgrades, base courses, and structures.

During the course of this study, a literature search was conducted to determine the state of knowledge of moisture measurement instrumentation as it relates to the needs of the highway engineer. The results of this survey were used to assess the various specific needs for this instrumentation and to provide the basic data to evaluate the degree that the instrumentation satisfies these needs.

Definitions of performance were compiled to form the basis of comparing a measurement need with instrument capability. A linear decision model was used to apply individual weights to each performance requirement and sum the results to estimate the most desirable method for a particular need. This model was used to evaluate instrumentation for measuring moisture in base-course compaction control, the study of long-term soil-culvert interaction, and the study of moisture in concrete during drying. These areas were chosen as being representative of a larger group of moisture measurement problems that are being studied by using one or more available techniques.

The results of the literature search and the evaluation procedure indicated several additional areas of research that should receive attention. Seven programs were briefly defined in the area of evaluation of instrumentation for measurement of moisture in soil and mineral materials and mixtures; two were defined for development of new instrumentation for significant problems lacking sufficient instrumentation; and three were defined in the modification and application of nuclear methods.

CHAPTER TWO

FINDINGS

MOISTURE MEASUREMENT PROBLEMS

Moisture measurement problems are related to the storage and use of construction materials, particularly aggregates for use in concrete, and stockpiled, blended, or graded aggregate base materials. Moisture monitoring may be required for knowledge of moisture content at time of use (Walker et al., 1970), or for maintenance of a specified level of moisture as a condition of use. The latter condition is particularly true for aggregate and soil materials used directly in layer construction in highway embankments and other controlled fills. In most cases, knowledge of moisture content at time of use is sufficient for concrete aggregates.

Moisture measurement problems also are related to post-construction needs of short and long duration. Moisture problems for which information may be valuable include: moisture migration in embankments; loss of moisture in volume-change-susceptible soils, particularly those enveloped in moisture-control membranes; buildup of moisture immediately below pavements in locations with remote water tables; seasonal fluctuations in ground water tables, including intermittent springs; and change in moisture conditions due to adjacent construction activity or to inadvertent changes from unanticipated maintenance practices. Specific instrument requirements are discussed in the following.

Moisture in Fine Aggregates

Chutes and Bins

Mixing materials for highway construction, particularly concrete, would benefit from the use of a reliable, accurate instrument as a control element. The instrument will be exposed to hundreds of tons per hour. It must be placed in the chute, and will be exposed to a highly abrasive environment. It should be small enough so that it does not interfere with aggregate flow such as to form a dead pocket or aggregate buildup that would shield the probe or element from the active flow. It should have a short-time response on the order of seconds. The instrument size or the power requirements are not critical so long as the technique meets the noninterference criterion in the flow or bin.

Field Stockpiles and Storage Bins

This application requires a portable instrument that can be used by an engineer or a technician in the field. Accuracy may be sacrificed for portability in this case. This instrument need not make readings in situ, although it would be desirable.

Moisture in Coarse Aggregates in Chutes and Bins

The requirements for coarse aggregate moisture measurement are essentially the same as those for fine aggregate, except that the amount of moisture to be measured by weight of aggregate held or processed is decreased as the size of the aggregate is increased. For extremely large aggregates, the measurement of moisture may be unnecessary; for blended coarse aggregates and aggregates approaching fine aggregate in size, surface moisture held by aggregates may be appreciable. Generally, absorbed or nonsurface water held by aggregates is a constant for any aggregate supply, and does not require constant monitoring because most field aggregates are found in a saturated-plus moisture condition. One area where this condition of saturation is questionable is that of highly vesicular or otherwise porous aggregates recently processed or stored under cover. In this less-than-fully-saturated condition, knowledge of the degree of saturation would be important in concrete control, because a moisture adjustment would be required to allow for full saturation of accessible pores and vesicles.

Moisture in Concrete

Moisture measurement in concrete depends on whether concrete is fresh (plastic) or hardened. In fresh concrete, water content affects such properties as workability, blending, segregation, and potential strength gain. In most instances these properties are determined and controlled by means other than moisture measurement in fresh concrete. These means include moisture measurement in aggregates before weighing, appropriate adjustments of batch weights, including mixing water, consistency tests such as the slump test, and visual inspection.. For most concreting operations, measurement of moisture content in fresh concrete does not seem very important. However, with newer techniques (such as continuous feed mixing, and other types of volumetric batching and "tube" mixing), immediate sensing of mixture moisture for feedbacks to automatic adjustment of input quantities could become extremely useful. Moisture sensing also could be useful for no-slump mixes where normal consistency measurements will not indicate the quantity of lubricating water. In this sense, water control would be substituted for consistency control.

Moisture measurements of fresh concrete, then, probably would be made on unconsolidated mixtures, and perhaps techniques similar to those for aggregates in motion or process could be applicable.

Moisture measurement in hardened concrete is another matter entirely; here sensing would be desirable for a variety of reasons, including degree of hydration, degree of saturation, periodic moisture fluctuations, moisture gra-

dients, and relation of moisture changes to deterioration or durability.

Water exists in hardened concrete as (1) chemically fixed, as water of hydration in clinker constituents and aggregates, (2) absorptively fixed in cement gel and aggregates, (3) capillary fixed in cement gel and aggregates, and (4) free water. A single instrument is not likely to provide information on all forms of water present.

For instance, changes in density, thermal conductivity, heat capacity, and dielectric constant depend primarily on total amount of water present.

Electrical conductivity may depend on total amount of water, but it is influenced primarily by free water.

Resistance to frost action depends primarily on freezable water content, or free water plus loosely fixed capillary water. Other water present is important in that it affects the resistance to the stresses induced by freezing.

Resistance to high temperatures depends on the amount of water that will evaporate at elevated temperatures generating internal stress and strain.

Generally desirable characteristics of the instrumentation for making the foregoing measurements are (1) nondestructive, (2) telemetering, (3) continuous measuring for weeks or months, and (4) a large number of measuring points (Mertin, 1965).

Moisture in Base Materials

There is a general tendency in subgrade, subbase, and base materials to increase moisture. This increase may result from surface water infiltration or transport from adjacent areas. The result may be unsatisfactory pavement performance from swelling or decreased shear strength of supporting structures (LeFevre, Manke, 1968). Satisfactory measurement of moisture could be correlated to overall pavement performance, resulting in possible revisions in design methods and construction procedures.

This instrument needs to be portable for field use. It should be free from interference that may result from a variety of soil and aggregate types. This instrument needs to be of the remote or in-situ type.

Moisture In Situ in Concrete Aggregates, Embankments, Subgrades, and Cut Slopes

This instrument also requires portability for field use. Accuracy could be sacrificed for a remote sensor. The minimum requirement is for an instrument for making in-situ measurements using a very small probe.

Special Research-Related Needs

Much research is being conducted relating the physical characteristics of paving, grading, and structures in their variation and degradation with time. There is a distinct limitation to properly relating these physical characteristics to trafficability without concurrent measurements of moisture. These measurements, covering long periods of time, need to differentiate between the form of the water—whether it is bound or unbound, nonevaporable or evaporable. The physical state, whether liquid or frozen, and the nonhomogeneous distribution over relatively large volumes also need to be determined. Thus, there is a significant category of requirements, referred to as the research needs of moisture measurement in highway engineering. Two areas that currently are receiving attention are D-line cracking in concrete and ice formation in base and subgrade materials. A brief discussion of these and other research needs follows.

D-Line Cracking

D-line cracking is a moisture-related defect that occurs at corners and along the joints between concrete sections; it is evidenced by more or less parallel surface crack lines that advance from boundaries inward as deterioration progresses. The research needs here are to be able to measure the total amount of water in the locality of the crack formation. This water needs to be broken down into that (1) absorbed in the concrete, and (2) retained in hairline cracks and surface layers. These measurements need to be made simultaneously with soundness measurements to determine at what state in the history of the water variation the most serious cracking occurs. These measurements are difficult to make without disturbing the natural conditions of the pavement.

Lens Formation

The random freezing cycle experienced during cold weather results in extremely nonuniform distribution of the frozen water. This is related primarily to the difference in soil moisture tension and low vapor pressure near the ice surface. This low vapor pressure causes subterranean water to migrate upward. This migrated moisture freezes in layers or lenses because of the random variation of temperature. On thawing, these layers and lenses provide an excess local concentration of liquid water, resulting in saturated soil and base layers that provide inadequate support to the paving surface; severe failure is a probable consequence.

Frost Heaving

Frost heaving occurs when there is sufficient lens formation to actually raise or heave the pavement surface. Heaving can occur whether the freezing cycle is consistent or random; heaving of several feet has been observed in certain soils. Research is needed here to determine what moisture content can be tolerated in each type of soil without significant degradation of the structure.

Culvert Leakage or Infiltration

Culvert leakage or infiltration can be the beginning of major road failure. Discovery of such leakage at an early stage would allow preventive maintenance, with considerable savings in time and expense.

Subgrade Seepage

Surrounding geology may contribute a significant amount of water to the subgrade material, necessitating continual foundation repair. One problem with subgrade seepage in cut areas is that water flow may be seasonal, and not recognized as a potential defect during warm-weather con-

struction operations. Repair also may be made during warm weather when evidence of seepage has disappeared. Continuous monitoring of moisture variations would show presence of water seasonably and point to the possible need for remedial drainage, in addition to pavement repairs. Sufficient distribution of moisture or water measurement devices could localize such problems and, in many cases, result in repairs with a minimum of effort, and possibly even before damage occurs.

Large Soil Masses

The concurrent measurement of moisture, temperature, stress, and strain in large soil masses would be a significant research achievement. These measurements would characterize different grading formations, soil compositions, and environmental conditions to which the soil mass is exposed, and provide information on the stability of slopes and embankments.

Prediction and Characterization of Earth Movements

Water content in a large fill or embankment can be the key to its stability. Audio recording of localized soil activities can document the mechanics of earth movement. Moisture information added to this documentation could significantly improve the understanding of slides and facilitate the preventive action that is needed.

Long-Term Moisture Changes in Highway Structural Sections

The use of corings or access tubes to make long-term measurements in base material and other components of structural sections is expensive, unreliable, and detrimental. Remotely monitored sensors would be desirable in this application.

Soil Strength and Stability

The useful life of structures depends on the strength of their foundations, which, in the case of soil, may depend on moisture content. A reliable method of measuring moisture would be helpful in analysis of in-situ soil strength. Stability of drilled shafts is of special interest in Texas for dams and bridges (Ehlers et al., 1969).

Effects of Water on Properties of Concrete

Strength, modulus of elasticity, shrinkage, creep, etc., are directly related to water of hydration in cement gel. Rheological properties also depend on absorptively held water and, to a lesser extent, capillary water.

Evaluation of Surface Coating

The surface of bridge decks may gradually erode as a function of freeze-thaw cycles. Certain mixtures, such as linseed oil and kerosene, appear to improve surface performance. It would be desirable to relate this apparent improvement to specific moisture conditions.

Moisture Measurement Related to Highway Safety

Surface Moisture on Pavement

The hazards of highway travel are amplified by moisture on the wearing surfaces. This is particularly significant in the early stages of water film formation, before many drivers have altered their driving attitudes from those of dry conditions. Moisture sensors need to be developed to signal this condition and control visual warning systems.

Moisture on Reflective Signs

Moisture on the surface of reflective sign material may render it ineffective. If more suitable material cannot be found, this problem could be eliminated by monitoring the collection of moisture and providing a means for removing it as it occurs.

Moisture Bookkeeping Procedures

Moisture bookkeeping and estimation is an essential part of construction requiring heavy earth-moving equipment. More about the concept of moisture bookkeeping and modeling related to strategic and tactical military planning can be found in Thornthwaite (1957). Primary interest is in gain and loss of moisture in the upper 2 ft of soil that determine tractionability (Winner, 1964).

CURRENTLY APPLICABLE MOISTURE MEASUREMENT TECHNIQUES

A wide variety of techniques have been developed for measuring and controlling moisture in solids. In general, the closer the control required and the lower the moisture level, the more difficult is the measurement problem.

The rapidly responding instruments for continuous operation generally depend on electrical properties, such as resistance or capacitance, or on the absorption of neutrons, electromagnetic radiation, or other wave-like interactions. A calibration curve normally is required for relating the instrument reading to moisture content. Most of this instrumentation reduces the operation time by factors of 10 or more, compared with conventional oven drying. Continuous-type instrumentation also is compatible with automatic control procedures and telemetering requirements.

The following description of moisture measurement methods is summarized from a more comprehensive analysis in Appendix A. The methods are categorized by the physical, chemical, and electrical phenomena on which they are based. This classification results in a minimum of instances where a method falls into more than one category. From the standpoint of applications, there is a need for secondary classifications of laboratory, field, research, destructive, and nondestructive techniques.

Hygrometric Techniques

The general nature of the relationship between moisture content in porous materials and the relative humidity (RH) of the immediate atmosphere under equilibrium conditions is reasonably well known. Consequently, a

relatively large number of simple devices for indicating and recording RH have been conceived. There are basic disadvantages, however, in using these methods to measure RH in a small cavity inside a porous material and deriving the moisture content of the solid. These include deteriorating effects of soil components on the sensing element and the need for special calibration for each material to be sampled. Hygrometric methods have application, however, where RH in the material is related directly to other properties (e.g., the drying shrinkage of concrete).

Electrical Resistance Hygrometers

Chemical Salts and Acids.—The electrical resistance of sensors is modulated by changes in the water content of electrolytic processes. Most resistive humidity transducers consist essentially of a hygroscopic material that absorbs water from the surrounding atmosphere. Generally, the material contains a salt that disassociates in the presence of water. The concentration of these disassociated ions is determined by the measurement of the electrical resistance between the electrodes applied to the transducers. The hygroscopic material is deposited on a substrate of ion-impervious insulating surfaces, such as fibers, fabrics, or ceramics. Typical examples of hygroscopic materials that have been suggested are sulfuric acid, phosphoric acid, lithium chloride, calcium chloride, zinc chloride, and solutions of the tetrachlorides of tin, zirconium, hafnium, or lead. (Dunmore, 1938; Bouyoucos, Cook, 1965; Klein, Trescony, 1965; Wexler, Ruskin, 1965).

Aluminum Oxide.—Both resistance and capacitance of aluminum oxide films have been correlated with relative humidity.

The typical transducer is essentially a capacitor with a porous dielectric film of aluminum oxide. One capacitor plate should be permeable to water vapor to permit penetration into the aluminum oxide.

The porous oxide layer is formed by various means such as (1) sputtering, (2) vapor deposition, and (3) anodization. Anodization is more popular, and generally is an acid electrolyte (17.5 volume percent H_2SO_4, current density 100 ma/cm^2, 30 min).

Cutting, Jason, and Wood (1955) list advantages and disadvantages of aluminum oxide sensors (see Appendix A).

Electrolysis.—Electrolysis systems consist of a gas mixture flowing continuously over a thin layer of partially hydrated phosphorous pentoxide (P_2O_5) with the humidity in the gas absorbed by the P_2O_5 layer. A typical sensor is constructed with two platinum wires wound helically inside an insulating tube and then coated with a layer of P_2O_5.

A DC voltage is applied to the two noble wires, decomposing the water into gaseous H_2 and O_2. At equilibrium conditions the current flowing is proportional to moisture absorbed.

Disadvantages of the electrolysis system include the requirement for constant mass flow, constant temperature (induces an error of 0.3 percent/degree), and interference from gases that interact with P_2O_5.

Thermal System.—Although similar in appearance to the previous method, the thermal system operates on a different principle and permits an absolute determination of humidity. The element consists of a tubular wick made from glass fibers and impregnated with a hygroscopic salt (lithium chloride) insulated from a thin metal tube. Two silver wires are bifilar wound around the wick and connected to an AC voltage source with some means for current limiting. The salt absorbs humidity from the surrounding air and becomes electrically conductive. The current passing through the lithium chloride generates heat and tends to evaporate humidity from it. An equilibrium is soon reached when the layer neither gains nor loses water to the surrounding air. Equilibrium is reached at that temperature of the salt solution at which the partial pressure of water over a saturated solution just equals the ambient water vapor pressure. The temperature is measured by a resistance thermometer, thermistor, or thermocouple, and should be in thermal contact with the lithium chloride layer.

White Hydrocal.—Bouyoucos and Cook (1965) describe what they consider to be the best hygrometer available. Stainless steel electrodes are cast in white hydrocal—a form of plaster of paris cement that sets hard, is pure, has low solubility, and has no added salts.

Capacitance Hygrometers

Capacitive Transducers.—Thc high dielectric constant of water (approx. 80) suggests the use of capacitive methods for determining water content of gases and solids. The low concentration of water in air, even at saturation, causes a very small variation in capacitance. The dielectric constant of gases under normal conditions changes from 1.000247 for dry air at 45°C; for saturation it is 1.000593. For such small capacitance changes very complex equipment for readout is required.

One way to increase this capacitance change is to displace the air with a material that will pick up an increased amount of water. A typical capacitive transducer is constructed with a thin plastic film of acetal resin, which is a crystalline form of highly polymerized formaldehyde. The capacitor plates are formed with evaporated gold electrodes thin enough to be pervious to water vapor yet electrically conductive.

Microwave Refractometer.—The microwave system is an arrangement to determine the resonant frequency of a cavity. The resonant frequency varies with the dielectric constant of the material in the cavity.

At an operating frequency of 10^{10} Hz, the difference in resonance frequency may be 0.27 MHz for one cavity filled with dry air and another filled with air plus a water vapor pressure of 100 mb.

Piezoelectric Sorption Hygrometer

In this method the sensing element is a quartz crystal coated with a hygroscopic material. The resonant frequency of such a crystal depends on the crystal mass (King, 1964). The mass, and, hence, resonant frequency, change with the adsorption of moisture. A typical sensitivity factor is about 1 Hz per Å thickness of added material.

Infrared Absorption and Transmission Hygrometers

The existence of optical absorption bands and transmission windows in the IR region provides a specific and accurate means of measuring humidity. Wood (1958) designed an optical instrument with a 12-in. absorption path and germanium narrow-band filters to compare the transmission of 2.54 μ and 2.60 μ. A rotating disk that alternately passes each wavelength is installed in front of a lead sulfide detector. The ratio of the amplitudes at the two frequencies is a function of water vapor concentration. Wavelengths of 2.3, 1.87, 1.37, and 1.12 also have been used.

Dimensionally Varying Element Hygrometers

Engineers have long been aware of the variation in size of certain materials as the humidity changes. The practicality of using this phenomenon in humidity measurement is evidenced by its wide use in industry (Fraade, 1963). Some of its desirable features are easy calibration, little manual know-how required, and the easy recording of results. It is characterized by poor accuracy and a host of other difficulties, but it performs well where these deficiencies can be tolerated.

Dew Point Hygrometers

Dew or frost point hygrometers depend on the measurement of temperature and are relatively simple and inexpensive devices. The temperature at which dew or frost appears on the surface of a cooled object denotes the point at which the RH at the surface is 100 percent. This temperature thus may be related to the partial pressure of water in the sample gas via the known saturation vapor pressure of water or ice.

Psychrometric Hygrometers

Psychrometric instruments form another major grouping of hygrometers. All such instruments depend on the cooling of a wetted thermometer and the subsequent temperature difference between the "wet bulb" and the "dry bulb" of the temperature sensor. Data usually are reduced by the use of the psychrometric formula or a set of tables based on this formula.

Electrical Resistance Techniques

The resistance of a material, its specific conductive capacity, and its dielectric losses vary with the amount of moisture it contains. An instrument for measuring one of these quantities thus can be calibrated in terms of moisture content.

Resistivity measurements can be made by direct contact of the electrodes with the material of concern. With this principle, a calibration first must be obtained for the specific material. The electrical resistance method developed by Bouyocous and Mick (1948) relies on the change of resistivity of the soil with changes in moisture content. Two electrodes covered with nylon, fiberglass fabric, or plaster of paris are buried in the soil and allowed to reach equilibrium before the measurement is made. This type of measurement is most accurate at very low moisture content. It has the disadvantage of a long response time to reach equilibrium.

Capacitance Techniques

The measurement of capacitance is one of the simplest, most rapid, and least expensive methods for determining soil moisture. This is because the dielectric constant for most soils is about 2.6, whereas the dielectric constant for free water between 15° and 35°C, within the frequency range of 10^5 to 10^8 Hz, is about 80. Fundamentally the method involves the measurement of the dielectric constant of the material between two electrodes. Thus, variation in the dielectric constant of nonwater components is an inherent source of error. Other inherent errors are variations in particle size, packing density, and ionized salts.

Nuclear Methods

Neutron Scattering

Since the pioneering work of Belcher, Cuykendall, and Sack (1950), much work has been devoted to a better understanding of the interaction between neutrons and the soil-water-detector system. The technique depends on the ability of hydrogen in water to slow down fast neutrons. The form of the hydrogen cannot be distinguished by this method. The measurement interferences existing in presently used commercial gauges consist of sensitivity to: (1) the total sample density, (2) sample composition (particularly the presence of thermal neutron absorbers, such as chlorine and boron, and of other moderators, such as hydrogenous material), (3) surface roughness, and (4) sample homogeneity. These measurement interferences can be minimized by several methods. One such method consists of the use of the new He^3-filled proportional counters to detect epithermal neutrons that are relatively insensitive to the presence of thermal neutron absorbers. Another method consists of the use of a gamma-ray gauge for the measurement of density and the subsequent use of calibration curves for various densities. Where each detector has a different sensitivity to composition, use of a dual gauge is another possible way to eliminate the effect of variable amounts of thermal neutron absorbers.

Comparative evaluation studies have led to the conclusion that interferences lead to errors in moisture measurement in soils (sometimes prohibitive errors), but the errors are commonly less than those for any other method (Stewart, Taylor, 1957; Johnson, 1962; Wofford, 1964; Ballard, Gardner, 1965; Waters, 1965; Gardner, Roberts, 1967; Hughes, Anday, 1967, 1970). Over-all, the neutron method appears to be the best method for nondestructive measurement of moisture in a 3- to 4-in. surface layer.

Gamma-Ray Interaction

Gamma-ray interaction is applicable where it is desired to measure moisture within a ½-in. layer of soil (Smith, Taylor, Smith, 1967). It is assumed that the specific

gravity of soil remains constant as moisture gain or loss changes the wet density. The wet density is determined by gamma transmission techniques. The relative change in moisture content can then be calculated from the change in wet density.

Neutron-Gamma Technique

Babinets and Zvol'skii (1966) proposed a logarithmic relation between moisture and the gamma radiation induced by neutron-activated atoms near the source of fast neutrons.

$$R = A + B \ln (w_H) \quad (1)$$

A and B are experimentally determined constants; w_H is the total hydrogen content. The mean deviation of their experimental results from this model was 0.02 g cm^{-3}.

Gravimetric Techniques

The gravimetric method of determining moisture content involves five steps: (1) collecting a soil sample, (2) weighing it, (3) removing the moisture, (4) weighing the dry sample or the removed water, and (5) calculating the moisture content. The gravimetric method is the most direct way of measuring soil moisture; therefore, it is required for calibrating equipment used in other moisture measuring techniques.

Oven Drying

A practical and easily controlled technique for water removal is oven drying. Heating raises the vapor pressure of the free water within the porous material. A reduced vapor pressure in the environment serves as a force to move this vapor out of the solid. This reduced pressure is obtained either by evacuation, or by dessication, or by the flowing of dry air over the samples. As the difference between the vapor pressure in the porous material and that of the environment decreases, the drying rate also decreases. At this time, a greater portion of the remaining moisture is tightly bound.

Freeze Drying

Freeze drying sometimes is referred to as lyophilization. This method offers no risk to decomposition of heat-sensitive materials. Freezing also has the effect of loosening the structure of organic materials so that additional moisture that remains after freeze drying is removed by oven drying. A combination of the two methods would result in greater removal of moisture.

Distillation

The sample is immersed in a liquid that is immiscible with water. Liquids are typically benzene, toluene, and xylene. The mixture is then raised to the boiling point of the liquid, which is normally below that of water. The refluxing column then feeds a calibrated bulb, where the liquid and water separate.

Dessicant Weight Gain

Dessicant weight gain can be used with other drying techniques (Geary, 1956). Inert gas or dry air is drawn over the samples and passed through a dessicant. The weight gain of the dessicant is assumed to be water. A dessicant may be chosen that passes volatiles other than water where normal drying techniques result in ambiguity.

Alcohol Burning

One of the more rapid gravimetric techniques is alcohol burning. This consists of mixing alcohol with the sample and igniting the mixture. Most of the water is removed during the process (Geary, 1956; Bouyoucos, 1937). The same is reweighed after the water-removal process. After three successive burnings the method correlates very closely to oven-drying results, even at high moisture content. (Antrim, 1970.)

Alcohol Extraction

Alcohol extraction is one of the simplest of the liquid-extraction methods (Bouyoucos, 1931). The water content is determined by the density of the alcohol-water mixture after extraction from the sample. A hydrometer can be used to measure the liquid density.

Calcium Carbide (Hydride)

The calcium carbide method is used widely. The moisture content usually is related to two measurements: (1) the decrease in the weight of the sample carbide mixture after evolution of acetylene, and (2) the rise in pressure inside an enclosure containing the mixture that determines the volume of gas produced. The latter is commonly known as the speedy moisture method. Extensive comparative evaluations of this method with oven-drying, nuclear, and alcohol-burning methods have been made (Antrim et al., 1970; L.B. Blystone, 1961). It is generally considered to be relatively accurate, inexpensive, and rapid.

Karl Fischer Reagent

The Karl Fischer reagent is used widely with the titration method for measuring moisture in solids. It is prepared by reacting SO_2 with iodine dissolved in pyridine and methyl alcohol. A more stable reagent is obtained by substituting ethylene glycol for methyl alcohol. The titration is conducted on the sample in methanol or other suitable solvents.

Immersion (Pycnometer)

The immersion method is based on classical procedures for determining specific gravity. It is in fairly common use in the measurement of loose materials such as sand and aggregates (Geary, 1956; Kirkham, Higgins, 1955).

Electromagnetic Radiation

Radiowaves

RF energy is highly absorbed by moisture. The method is not very specific, because many polar compounds also absorb this energy. Thus, the primary disadvantage of this technique is that it requires a calibration curve for each material on which it is to be used. The calibration slope is very steep, however, and with adequate control of other variables a fraction of a percent precision and accuracy can be achieved.

Microwaves

The moisture content of nonmetallic porous solids is related to the absorption of microwave energy. For many materials a linear relationship exists between moisture density and the logarithm of microwave attenuation (sometimes expressed in decibels). Microwaves between 3- and 30-cm wavelengths are most suitable from an economic standpoint. However, there are certain improvements in specificity below 3 cm, particularly with respect to soluble salts.

Nuclear Magnetic Resonance

When a sample is placed in a fixed magnetic field and a varying magnetic field, nuclear magnetic resonance results in an increased absorption of energy at specified frequencies of the varying magnetic field. The varying nuclear magnetization is converted into a voltage by using either the single-coil absorption technique or the quadrature-coil induction technique. The resonant frequency of the absorption or induction is characteristic of a particular nucleus. The highest signal-to-noise ratio for measuring water molecules is obtained by looking at the hydrogen nuclei spectra. This technique also responds to materials containing nonwater hydrogen. It is likewise responsive in varying degrees as the water is bound or free. The large size and the critical geometry of instrumentation dictates against its use in the field.

Infrared Reflection

A relatively new technique that is widely accepted in some industrial processes is based on the IR reflection that is proportional to the moisture content of the exposed solid. A limitation is that the interaction is primarily a surface interaction. If moisture is not homogeneous, significant errors can occur: Reflection signals of 1.94 μ and 1.80 μ are compared with the aid of a filter disk. A signal at 1.9 μ and a reference at 1.7 μ also may be used.

Tensiometry

The name "tensiometer" was used by Richards and Gardner (1936) as an unambiguous reference to the porous cup and vaccum gauge combination for measuring capillary tension or the security with which water is held in soil. More recently, permeable membranes have been used.

The term "pF," introduced by Schofield (1935), is commonly used to describe tensiometer response. It is defined as the common logarithm of the height of a water column in centimeters equivalent to the soil moisture tension.

Thermal Conductivity

The thermal conductivity method relies on the increase in thermal conductivity of porous materials with increasing moisture content. This principle can be applied through transmission in which a rise in temperature of the material is measured at a distance from the heat source. It also may be applied through dissipation, by measuring the temperature rise of the heating element. Although thermal contact may present problems, salt or variable composition of the sample material offer less interference than in any other technique.

Penetrometer

The basis for the penetrometer method is resistance to penetration or deformation. Some problems related to its use are that penetration equipment must be calibrated for each type of soil, and gravelly or stony soils are unsuitable. However, the method is very fast (Antrim et al., 1970).

Summary

Commercial suppliers of instrumentation for measurement of moisture by one or more of the foregoing techniques are given in Table 1. The inclusion of suppliers in the table occurred as a result of their being mentioned in research reports or as a result of their response to a written request for information. Other manufacturers of similar equipment may not have been identified during the literature survey or may have initiated production subsequent to report preparation.

COMPARATIVE EVALUATION

Following the identification of a moisture problem a systematic approach is recommended for choosing the appropriate measurement method. This effort should include the following steps:

1. Define the application in terms of the relative importance of basic instrument performance characteristics.
2. Review the available data on potential methods.
3. Conduct a preliminary evaluation to reduce candidate methods.
4. Conduct a laboratory evaluation of remaining methods.
5. Conduct a field evaluation of methods that perform satisfactorily in the laboratory.

To be as inclusive as possible in accomplishing these five steps, one should consider the following checklist of items: (1) material (or range of materials) whose moisture content is to be determined; (2) material properties that are under investigation; (3) state of water related to material properties; (3) measurement techniques that are responsive to a given state of water; and (4) cost minimization.

Terms for Describing Instrument Performance

The following terms are useful in describing the degree to which an instrument satisfies the requirement of its intended monitoring function. These may fall under the categories of: (1) physical characteristics of the instrument that may be measured, (2) measured responses of the instrument to well-defined test procedures, (3) data quality from field monitoring programs, or (4) functional capability. Definitions and discussion of these characteristics follow.

TABLE 1

COMMERCIAL INSTRUMENTATION

MEASUREMENT PRINCIPLE	MANUFACTURER
Hygrometric	American Standard Aminco Beckman Instruments Bell and Howell Consolidated Electrodynamics Corp. Foxboro Honeywell Hygrodynamics Manufacturers Engineering Phys-Chemical Research Corp. Reiss Engineering Co. Yellows Springs Instrument Co.
Electrical Resistance:	
Sample resistivity	Electronic Automation Systems Hartley Controls Corp. Mount Hope Machine Co. Sever-Hall
Sensor resistivity	Delmhorst Instrument Co. Soiltest
Capacitance	Forte Engineering Corp. Foxboro Co. James Hunter Machines Co. Moisture Register Co. Seedburo Equipment Co.
Nuclear	Campbell Pacific Nuclear Control Service Co. Pitman Limited Millworks Seaman Nuclear Corp. Texas Nuclear Corp. Troxler Electronic Laboratories
Gravimetric:	
Thermal extraction	Bell and Howell Brabender Central Scientific Co. Ohaus Scale Corp.
Chemical extraction	Ashworth & Co. Beckman Instruments E. H. Sargent & Co. Soiltest
Radiation:	
Mechanical Electromagnetic	Soiltest Anacon Boonton Polytechnic Co. General Electric Co. Moisture Register Co.
Tensiometry	NIC Instrument Co.
Gravimetric with Colorimetric	Technicon Control

Physical Characteristics

Portability: Measure of the capability to be moved. Small size and low weight are desirable. Durability and shape also may enter into the assessment of portability.

Size: The dimensions of the complete instrument. Height, width, and depth usually are adequate to describe size. The basic instrument is assumed to include the sampling, analysis, and detection systems, in one or more packages. Data display and recording systems are considered accessories unless they are an integral part of the instrument in a single cabinet.

Weight: Low weight is desirable, although its importance may vary from one user to another. It is a significant measurable characteristic related to portability.

Space Requirement: The dimensions of height, width, and depth required for installation, operation, and maintenance of an instrument. It includes space needed by auxiliary items and equipment, and swinging space for cabinet access doors and panels.

Auxiliary Equipment: Additional equipment required by the instrument to perform its measurement and data display function.

Power Requirement: The type of power and the amount required for operation.

Versatility: The capability of being used for more than one purpose; a laboratory instrument would be more desirable if it also could be used for field measurements.

Hazards: Sources of irritation or harm to which the instrument operator or other personnel may be exposed while using or servicing the instrument. Typical hazards are chemical, electrical, nuclear, and acoustical in nature.

Measured Responses to Standard Test Procedures

Temperature Effect: The change in instrument output per unit temperature change.

Interference: The positive or negative output caused by a substance other than the one being measured (see Interference Equivalent).

Interference Equivalent: The indicated moisture content that is attributable to a given concentration of an interferent. A low interference equivalent is desired for those interferences that are likely to be found in the instrument environment.

Specificity: The ratio of instrument response related to moisture and the response related to interfering substances.

Range: The minimum and maximum measurement limits. The effective range may be limited by the points where no readable response can be obtained. A more useful, effective range in analytical instrumentation is the range over which a single calibration curve gives sufficient calibration precision. This specification is of primary importance in matching an instrument and a specific measurement problem.

Set-up Time: The time required to prepare the instrument for operation after it has been transported from one location to another.

Warm-up Time: The time required for the instrument drift rate to be within the maximum limit specified for the instrument after having been shut down for at least 24 hours.

Response Time: The time required for the instrument to reach a new equilibrium. The important components of response time are defined in the following and are further clarified by Figure 1.

Initial Response Time (Lag Time), t_i: The time interval from a step change in the moisture content of the sample to the first corresponding change in the instrument output. This can be most reproducibly determined by extrapolating the slope at the 5 percent point, or using the 5 percent point as the point of reference.

Rise Time, t_r: The interval between the initial response time (t_i) and the time to 95 percent response (t_{95}), after a step increase in moisture (reference points other than 5 percent and 95 percent are often used).

Time to 95 percent Response, t_{95}: The time interval from the step change in moisture to a reading of 95 percent of the equilibrium response; $t_{95} = t_i + t_r$. There is no additional information in this term, but it is more convenient to determine than t_i and has more practical significance.

Fall Time, t_f: The interval between the initial response time, t_i, and the time to 95 percent response after a step decrease in moisture. This time is not necessarily equal to the rise time, but may be approximately the same.

Speed of Measurement: The time required to set up the instrument, calibrate, make the measurement, and perform any analysis necessary to present the instrument output in standard units.

Time Monitoring Capability: Time delay between successive measurement. For times less than 5 min, instruments commonly are termed "continuous"; for times greater than 5 min, "intermittent."

Sensitivity: Instrument output per unit input. The term is important in system design, but other terms describe system performance better (minimum detectable change, minimum detectable sensitivity, decision limit, detection limit, precision).

Sample Weight: The effective weight of sample that contributes to the instrument response.

Sample Volume: The effective volume of sample that contributes to the instrument response.

Physical State Sensitivity: Ability to distinguish between vapor, liquid, or solid form of water.

Moisture Type Sensitivity: Ability to distinguish the proportion of free water to bound water in a sample.

Radius of Influence: The distance at which additional sample contributes a negligible part of the total instrument response (closely related to sample volume).

Remote Monitoring Capability: The ability of the instrument to make remote or in-situ measurements.

Data Quality Determined by Field Monitoring Experience

Stability: A measure of the instrument drift. This term generally is used for long-term performance.

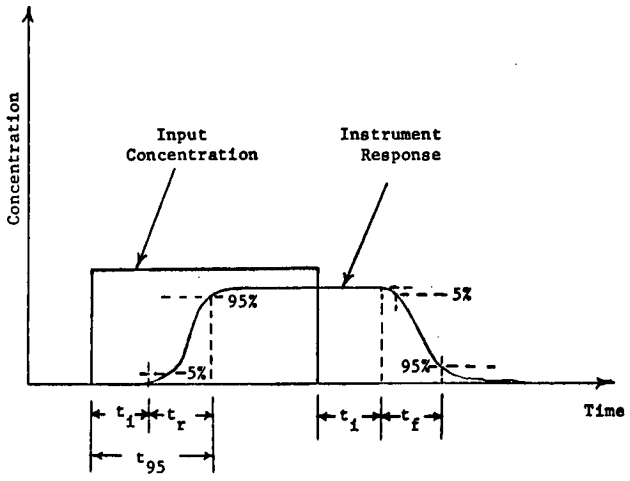


Figure 1. Instrument response time.

Accuracy: The degree of conformity of the measurement to a primary standard reference. An estimate of the accuracy normally is made by summing the known sources of error, such as those determined by interference, drift, precision, and reproducibility measurements.

Error: Difference between instrument reading and true reading when calibrating against a standard.

Tolerance: Permissible error or level of accuracy that is acceptable. Tolerance normally is specified as percent of scale reading or as percent of scale range. For a particular need, smaller tolerance may require more expensive instrumentation.

Precision: The degree of exactness of the instrument; the reproducibility that can be demonstrated by repeated measurement of the same sample; the degree of agreement between repeated measurement of the same sample expressed as the average deviation of the single results from the mean.

Calibration Precision: The variance of calibration data about the best fit calibration curve.

Zero Drift: The change in instrument output over a stated time period. Data with which to estimate this drift can be obtained through unadjusted, continuous operation, or they may be obtained by comparison of successive calibration data. Minimum zero drift is desirable, especially when one is measuring very low moisture content.

Span Drift: The change in instrument sensitivity over a stated period of time. This normally is determined together with zero drift.

Linearity: The maximum deviation between an actual instrument reading and the reading predicted by straight-line calibration drawn over the extent of its range. A linear response results in easier calibration.

Reproducibility: The attainability of the same output for a fixed input measured at intervals over a period of time. Estimates of this reproducibility may be influenced by the reproducibility of the calibration system.

Calibration Reproducibility: The variance between calibration data obtained at different times during the instrument operational period.

Minimum Detectable Sensitivity: The smallest amount of input concentration that can be detected with a specified degree of confidence. The statistical calculation of decision limit or detection limit from calibration data provides an unambiguous measure of this sensitivity. Much of the same information is contained in the zero drift and precision data.

Minimum Detectable Change: The smallest change in moisture content that can be detected at a given operating level. The equivalent information for this term is given by zero and span drift, and are best represented by confidence limits on the calibation curve. The minimum detectable change is estimated by the difference $y_{max} - y_{min}$ in Figure 2.

Decision Limit: The lowest signal, e_d in Figure 2, that can be distinguished from background with a specified degree of statistical confidence $(1 - \alpha)$.

Detection Limit: The moisture content, y_d in Figure 2, below which there is a specified probability, β, that the sample may erroneously be taken as zero.

Functional Capability

Availability: Obtainability from a supplier.

Practicability: Capability of being manufactured and applied to a specific problem.

Fragility: The delicacy of the instrument and the need for careful handling. Rugged instruments are preferred.

Durability: Ability to withstand normal use. This term is related to fragility under normal operating conditions.

Serviceability: Ease with which an instrument can be serviced and repaired. Long periods of repair indicate poor performance. Additional subjective judgment by the person performing the maintenance and repair is necessary in an evaluation.

Reliability (Failure): The mean time between failures. The record of field operation can provide the necessary data. Conclusions must be statistically qualified by the number of instruments being observed and the length of the observation period.

Maintenance Requirements: The frequency and severity of problems preventing proper operation of the instrument. The best estimate of this performance is the percentage of available time spent in maintenance. Parts and operator time are considered under cost.

Operational Period: The mean time over which the instrument can be expected to operate without maintenance recalibration or adjustment.

Lifetime: The expected total useful life of the instrument.

Instrument Cost: The cost of buying the instrument (capital cost), operating cost (operator time per sample, consumable supplies), and maintenance cost (spare parts, repair time). These three costs could be evaluated independently, depending on the economic needs of the user. Generally, however, lowest total cost would be the basis for selection.

Simplicity of Operation: Ease with which the instrument can be operated properly.

Operator Experience: Operator training time required.

Effect on Sample: The destructive or disturbing influence of the measurement method on the sample.

Applicable Materials: The composition, size, or shape of material most suited to an instrument's over-all performance.

Calibration: The development of a graphical or numerical relationship between instrument response and the moisture content of a set of standards.

Transfer Function: The inverse of the calibration equation.

Calibration Requirements: The time between calibrations, the time required to perform individual calibrations, the difficulty in using available standards, hysteresis effects, and nonlinear response.

Hysteresis: Apparent hysteresis is synonymous with lag, and disappears if measurements are conducted over a long enough time. Permanent hysteresis is a characteristic of the sensor. It is the difference in the response curve when going from zero to maximum and from maximum to zero.

Remote Sensing: The capability of sensing the sample condition at one position and relaying the signal response to another.

Type 1—Phenomena that are remote by nature.
Type 2—Wireless transmission of information.
Type 3—Physical contact with the sensor element.

For more information on this subject see Appendix C.

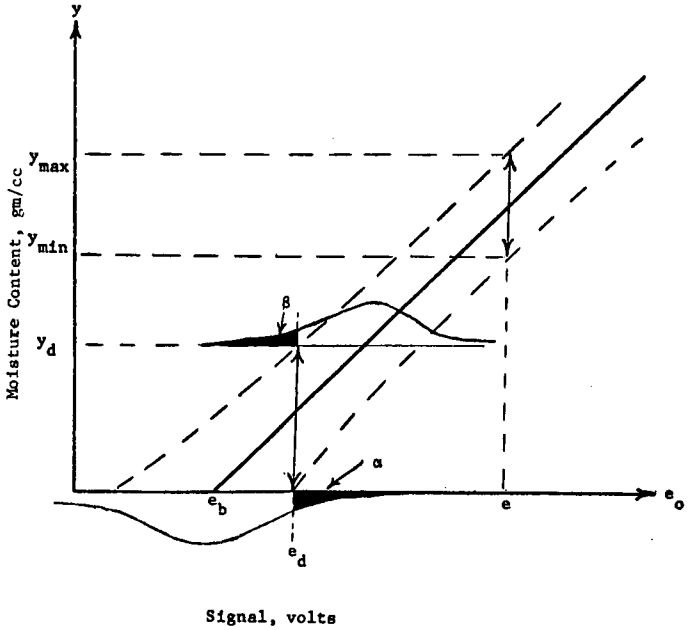


Figure 2. Transfer function with confidence limits. e_d *and* y_d *are the decision limit and the detection limit used by Hubaux and Vos (1970).*

Instrument Evaluation Model

The following instrument evaluation model is developed to provide an estimate of the best available instrument for specified operating conditions. This estimate is based on the relative importance of several performance characteris-

TABLE 2

COMPARISON MATRIX (EXAMPLE)

PERFORMANCE REQUIREMENT	METHOD 1	METHOD 2
Accuracy:		
Baseline drift (% span)	±5%/day	±10%/day
Span drift	±2%/day	±3%/day
Interference (% span)	Typically ±10%	Typically±10%
Portability:		
Weight (lb)	76	30
Size (cu ft)	15	10
	Has carrying handles	No handles required

tics and the probability as determined by experience that one instrument is better than another for a particular characteristic.

The value of a method for a measurement problem is given by

$$V_j = W_i P_{ij} \tag{2}$$

in which P_{ij} is the probability that method j is the best method, as determined by the characteristic i. W_i is a weighting factor or the probability that the characteristic i is the most important performance function. As an example to aid in understanding larger matrices, consider the smaller problem of two methods—say, Method 1 and Method 2—that are to be evaluated on the basis of two performance requirements; namely, accuracy and portability.

A *comparison matrix* is first set up that contains as much available data and subjective judgment as is needed to begin the evaluation. This is shown in Table 2. For a large matrix, a large worksheet is required, unless each method or characteristic is presented separately.

The *comparison matrix* is converted to the *decision matrix* of Table 3 on a statistical basis. It often may be necessary to rely heavily on engineering judgment.

Because P_{11} is twice as large as P_{12}, this is interpreted to mean that Method 1 is twice as likely to be first choice, if only accuracy is considered. Because only two choices are assumed to be available:

$$P_{11} + P_{12} = 1.0 \tag{3}$$

TABLE 3

DECISION MATRIX (EXAMPLE)

PERFORMANCE REQUIREMENT	METHOD 1	METHOD 2	SUM HORIZONTAL
Accuracy	$P_{11}=0.67$	$P_{12}=0.33$	1.0
Portability	$P_{21}=0.25$	$P_{22}=0.75$	1.0

Table 4 is the resultant *value matrix* determined by the product of the weight factor (w_i) and the decision matrix. Assume that portability is considered more important than accuracy, with $w_i = 0.60$ for portability and $w_i = 0.40$ for accuracy.

Because $V_2 > V_1$, Method 2 is the first choice. Here again, because only two choices are available, the probability of choosing Method 1 or Method 2 is 100 percent, so that

$$V_1 + V_2 = 1.0 \tag{4}$$

A reasonable question is, "How sensitive is the final choice to weight factor variation?" It may be expected to be sensitive to large variations of the weight factors, because these factors describe the application, and one method generally is not expected the best in all applications. It is not desirable, however, to reach a decision that may be reversed by small changes in the estimate of the

TABLE 4

VALUE MATRIX (EXAMPLE)

PERFORMANCE REQUIREMENT	WEIGHT FACTOR, W_i (%)	METHOD 1		METHOD 2	
		P_{ij}	W_iP_{ij}	P_{ij}	W_iP_{ij}
Accuracy	0.40	0.67	0.268	0.33	0.132
Portability	0.60	0.25	0.150	0.75	0.450
Total performance, V			0.418		0.582

importance of one performance requirement. A procedure known as *sensitivity analysis* adds to the confidence in the decision reached. In this analysis, shifts in relative ranking are observed as each weight factor is varied over a reasonable range. All other weight factors remain the same relative to each other, but normalization is maintained. This is especially simple to do for the sample problem. This result is given in Tables 5 and 6. Table 6 is the ranking that results from the value analysis of Table 5. It shows that Method 2 is the best choice if accuracy is rated at 60 percent or less and portability is rated at 40 percent or more.

This agrees with the comparison matrix that shows that Method 2 is best with respect to portability and Method 1

TABLE 7

COMPARISON MATRIX OF METHODS FOR MEASUREMENT OF MOISTURE

METHOD	AVAILABILITY	PORTABILITY	SAMPLE SIZE	EFFECT ON SAMPLE	ACCURACY (PRECISION, INTERFERENCE)	SPEED OF MEASUREMENT
GRAVIMETRIC, OVEN DRY	General use	Could be made portable, but normally is not	As required	Destructive	As precise as the technique for extracting the sample; volatile materials interfere	Many hours
GRAVIMETRIC, ALCOHOL BURNING	General use	Portable design	0.5–2 lb	Destructive	Similar to oven drying	Typically 5 min
GRAVIMETRIC, CALCIUM CARBIDE	General use	Portable design	13–26 grams	Destructive	Similar to oven drying; operator care and cleanliness also affect precision	Typically 1–5 min
NUCLEAR NEUTRON SCATTER	Units designed for highway use	Portable units in use	2–12 in., depending on moisture content	Nondestructive	Precision $\geq 0.1\%$ (dry), depending on counting time; interferences are density, neutron absorbers, and hydrogenous materials	Typically 1–5 min; 20 measurements in time of 1 gravimetric measurement
TENSIOMETRIC	Primarily for agricultural use, occasionally in highway use	Small, but not necessarily portable	Determined by region of moisture equilibrium	Requires installation in sample	Inadequate for many applications	A few minutes with small cavity and solid-state pressure transducer; others are much longer
CONDUCTIMETRIC, ELECTRICAL RESISTANCE	In limited use in R/D	Small, but not necessarily portable	Broadly defined by electrode position, but exact size is unpredictable	Nondestructive	Accuracy is poor for a variety of materials; salts interfere; 2 measurements give precision of one gravimetric	Real-time response except where electrodes are covered with fiberglass or other absorbent material
THERMAL CONDUCTIVITY	In limited use in R/D	Small but not necessarily portable	Small	Requires installation in sample	Thermal contact, density, and pore size are influential	Limited by time to install; non-equilibrium conditions can be used
HYGROMETRIC RESISTANCE	R/D units for concrete; primarily used in areas unrelated to highway engineering	Small but not necessarily portable	Region of vapor equilibrium	Normally requires installation in sample	Poor for ionic or hygromaterials or those containing volatile substances; hysteresis occurs	Limited by time to install and time to reach equilibrium with sample
MICROWAVE	Laboratory prototype	None available, but size not a major limitation	Depends on moisture content and frequency	Nondestructive	Less accurate than oven drying	Real-time measurement

[a] Depending on sensor type.

is best with respect to accuracy. Other ways to use sensitivity analysis have been described (Thompson, 1970).

The first step in applying the evaluation model is to construct the *comparison matrix* giving concise information about candidate methods that appear to be applicable to one or more highway problems. Performance characteristics are chosen that constitute important measurement requirements and for which reasonable estimates of the decision probabilities (P_{ij}) can be determined either from the data compiled in Appendix A or by engineering judgment.

The comparison matrix of Table 7 lists the candidate methods as column headings and the performance requirements as row headings. This table is used to consider four

MOISTURE TYPE	DURABILITY	RELIABILITY	STABILITY	HAZARDS	TEMPERATURE EFFECTS	LIFE	REMOTE MONITORING
Free or partially bound	Rugged	Little chance of failure	No unstable components	Thermal; electrical	Depends on oven temperature (~105° C)	>10 years	No
Free or partially bound	Rugged	Little chance of failure	No unstable components	Thermal; chemical	Very small	>10 years	No
Surface water	Rugged	Little chance of failure	No unstable components	Thermal; chemical	Corrections applied to calculations	>10 years	No
Primarily responds to hydrogen	Adequate except for extreme conditions	Proven electronic system	Electronic system requires occasional recalibration	Nuclear; electrical	Slight temperature dependence between 0–32° C determined by electronic system and detector	>5 years	Type 1
Free	Rugged	Air entry causes failure	Air entry changes calibration	Electrical	Strong temperature dependence	>6 months	Type 3
Free or partially bound	Generally rugged; electrical contact must be maintained	Good except in severe thermal or chemical environment	Contact is difficult to keep constant	Electrical	~3% resistance/ °C	1 mo to 1 yr [a]	Type 2 or 3
Free or partially bound	Generally rugged; electrical contact must be maintained	Electrical system is very simple	Thermal contact must be maintained	Electrical	Very small	>1 year	Type 3
Evaporable water at ambient temperature	Generally rugged; electrical contact must be maintained	Sensor failure depends on environment	Sensor aging occurs; this is improved by polystyrene insulation or moisture-permeable membranes	Electrical	Most have a strong negative temperature coefficient	1 mo to 2 yr [a]	Type 2 or 3
Free or partially bound	Adequate except for extreme conditions	Proven electronic system	Electronic stability required	Electrical	Sensitive to temperature	>5 years	Type 1

TABLE 5

VALUE MATRIX SENSITIVITY FOR A RANGE OF WEIGHT FACTORS (EXAMPLE)

WEIGHT FACTOR	METHOD 1	METHOD 2
Accuracy:		
20%	$V_1=0.333$	$V_2=0.667$
40%	0.418	0.582
60%	0.500	0.500
Portability:		
20%	0.583	0.417
40%	0.500	0.500
60%	0.418	0.582

TABLE 6

RANKING SENSITIVITY FOR A RANGE OF WEIGHT FACTORS (EXAMPLE)

PERFORMANCE REQUIREMENT	WEIGHT FACTOR		
	20%	40%	60%
Accuracy:			
1st choice	2	2	1 or 2
2nd choice	1	1	1 or 2
Portability:			
1st choice	1	1 or 2	2
2nd choice	2	1 or 2	1

basic measurement problems: (1) base-course compaction control, (2) long-term study of soil-culvert interaction, (3) inspection testing during and after construction, and (4) study of moisture in concrete during drying. Inspection of Table 7 reveals a heavy reliance on engineering judgment. This is attributed to lack of reliable data or to conflicting data in the literature. In an evaluation of the results of a field-test program comparing several instruments, the performance requirements included in the comparison matrix would coincide with the test objectives for each instrument.

Moisture measurement methods for base-course compaction control are evaluated in Tables 8 to 10. The decision probabilities of Table 8 are converted from the comparison matrix on a best-judgment basis. For this measurement problem, availability and accuracy were given the greatest weight in Table 9. Portability, sample size, and effect on samples were given medium weight. The results given in Table 9 indicate almost equal value for the gravimetric and nuclear methods, with a slight edge given to the latter. In Table 10 the individual weight factors are varied to determine the sensitivity of the evaluation. Performance requirements that have the greatest effect on the rankings are sample size, reliability, stability, hazards, lifetime, and remote sensing. Except for extreme variations, however, the results were consistent with the original weighting.

Moisture measurement related to the long-term study of soil culvert interaction is evaluated in Tables 11 and 12. Note that the decision probabilities for certain performance requirements such as effect on sample and remote sensing change with the type of problem being approached. These two terms are given the highest weight for this problem, with the result that the highest ranking is obtained for the electrical conductivity and the thermal conductivity methods.

TABLE 8

DECISION MATRIX—MOISTURE IN BASE COURSE COMPACTION CONTROL

PERFORMANCE CHARACTERISTIC	MEASUREMENT METHOD								
	GRAV. OVEN DRY	GRAV. ALCOHOL BURN	GRAV. CALCIUM CARB.	NUCLEAR	TENSI-OMETRY	ELECTRIC COND.	THERMAL COND.	HYGROM-ETRY	MICRO-WAVE
Availability	0.189	0.189	0.189	0.189	0.019	0.094	0.019	0.094	0.019
Portability	0.069	0.103	0.103	0.103	0.103	0.138	0.138	0.138	0.103
Sample size	0.160	0.160	0.160	0.120	0.040	0.080	0.040	0.120	0.120
Effect on sample	0.018	0.018	0.018	0.164	0.145	0.164	0.145	0.145	0.182
Accuracy	0.176	0.147	0.147	0.147	0.059	0.088	0.059	0.059	0.118
Speed of measure	0.065	0.097	0.129	0.161	0.032	0.161	0.161	0.032	0.161
Moisture type	0.140	0.140	0.140	0.105	0.088	0.088	0.088	0.088	0.123
Durability	0.125	0.139	0.139	0.111	0.083	0.111	0.111	0.083	0.097
Reliability	0.167	0.167	0.167	0.100	0.100	0.067	0.083	0.067	0.083
Time stability	0.152	0.152	0.152	0.091	0.106	0.076	0.106	0.076	0.091
Hazards	0.082	0.082	0.082	0.033	0.164	0.148	0.148	0.148	0.115
Temp. stability	0.152	0.152	0.152	0.121	0.076	0.076	0.106	0.076	0.091
Lifetime	0.222	0.222	0.222	0.111	0.022	0.022	0.044	0.022	0.111
Remote sensing	0.000	0.000	0.000	0.222	0.111	0.148	0.148	0.148	0.222

TABLE 9

VALUE MATRIX—MOISTURE IN BASE COURSE COMPACTION CONTROL

PERFORMANCE CHARACTERISTIC	WEIGHT FACTOR, w_i	MEASUREMENT METHOD: GRAV. OVEN DRY	GRAV. ALCOHOL BURN	GRAV. CALCIUM CARB.	NUCLEAR	TENSIOMETRY	ELECTRIC COND.	THERMAL COND.	HYGROMETRY	MICROWAVE
Availability	0.200	0.038	0.038	0.038	0.038	0.004	0.019	0.004	0.019	0.004
Portability	0.100	0.007	0.010	0.010	0.010	0.010	0.014	0.014	0.014	0.010
Sample size	0.100	0.016	0.016	0.016	0.012	0.004	0.008	0.004	0.012	0.012
Effect on sample	0.100	0.002	0.002	0.002	0.016	0.015	0.016	0.015	0.015	0.018
Accuracy	0.150	0.026	0.022	0.022	0.022	0.009	0.013	0.009	0.009	0.018
Speed of measure	0.050	0.003	0.005	0.006	0.008	0.002	0.008	0.008	0.002	0.008
Moisture type	0.050	0.007	0.007	0.007	0.005	0.004	0.004	0.004	0.004	0.006
Durability	0.030	0.004	0.004	0.004	0.003	0.002	0.003	0.003	0.002	0.003
Reliability	0.050	0.008	0.008	0.008	0.005	0.005	0.003	0.004	0.003	0.004
Time stability	0.030	0.005	0.005	0.005	0.003	0.003	0.002	0.003	0.002	0.003
Hazards	0.020	0.002	0.002	0.002	0.001	0.003	0.003	0.003	0.003	0.002
Temp. stability	0.050	0.008	0.008	0.008	0.006	0.004	0.004	0.005	0.004	0.005
Lifetime	0.050	0.011	0.011	0.011	0.006	0.001	0.001	0.002	0.001	0.006
Remote sensing	0.020	0.000	0.000	0.000	0.004	0.002	0.003	0.003	0.003	0.004
Total performance value		0.136	0.137	0.139	0.140	0.069	0.102	0.082	0.093	0.103

TABLE 11

DECISION MATRIX—MOISTURE IN LONG-TERM STUDY OF SOIL-CULVERT INTERACTION

PERFORMANCE CHARACTERISTIC	MEASUREMENT METHOD: GRAV. OVEN DRY	GRAV. ALCOHOL BURN	GRAV. CALCIUM CARB.	NUCLEAR	TENSIOMETRY	ELECTRIC COND.	THERMAL COND.	HYGROMETRY	MICROWAVE
Availability	0.189	0.189	0.189	0.189	0.019	0.094	0.019	0.094	0.019
Portability	0.069	0.103	0.103	0.103	0.103	0.138	0.138	0.138	0.103
Sample size	0.093	0.093	0.093	0.070	0.070	0.209	0.233	0.070	0.070
Effect on sample	0.025	0.025	0.025	0.100	0.100	0.225	0.200	0.200	0.100
Accuracy	0.176	0.147	0.147	0.147	0.059	0.088	0.059	0.059	0.118
Speed of measure	0.065	0.097	0.129	0.161	0.032	0.161	0.161	0.032	0.161
Moisture type	0.140	0.140	0.140	0.105	0.088	0.088	0.088	0.088	0.123
Durability	0.125	0.139	0.139	0.111	0.083	0.111	0.111	0.083	0.097
Reliability	0.167	0.167	0.167	0.100	0.100	0.067	0.083	0.067	0.083
Time stability	0.152	0.152	0.152	0.091	0.106	0.076	0.106	0.076	0.091
Hazards	0.082	0.082	0.082	0.033	0.164	0.148	0.148	0.148	0.115
Temp. stability	0.152	0.152	0.152	0.121	0.076	0.076	0.106	0.076	0.091
Lifetime	0.222	0.222	0.222	0.111	0.022	0.022	0.044	0.022	0.111
Remote sensing	0.000	0.000	0.000	0.160	0.040	0.240	0.240	0.240	0.080

TABLE 12

VALUE MATRIX—MOISTURE IN LONG-TERM STUDY OF SOIL-CULVERT INTERACTION

PERFORMANCE CHARACTERISTIC	WEIGHT FACTOR, w_i	MEASUREMENT METHOD: GRAV. OVEN DRY	GRAV. ALCOHOL BURN	GRAV. CALCIUM CARB.	NUCLEAR	TENSIOMETRY	ELECTRIC COND.	THERMAL COND.	HYGROMETRY	MICROWAVE
Availability	0.050	0.009	0.009	0.009	0.009	0.001	0.005	0.001	0.005	0.001
Portability	0.010	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
Sample size	0.100	0.009	0.009	0.009	0.007	0.007	0.021	0.023	0.007	0.007
Effect on sample	0.200	0.005	0.005	0.005	0.020	0.020	0.045	0.040	0.040	0.020
Accuracy	0.100	0.018	0.015	0.015	0.015	0.006	0.009	0.006	0.006	0.012
Speed of measure	0.010	0.001	0.001	0.001	0.002	0.000	0.002	0.002	0.000	0.002
Moisture type	0.030	0.004	0.004	0.004	0.003	0.003	0.003	0.003	0.003	0.004
Durability	0.040	0.005	0.006	0.006	0.004	0.003	0.004	0.004	0.003	0.004
Reliability	0.050	0.008	0.008	0.008	0.005	0.005	0.003	0.004	0.003	0.004
Time stability	0.050	0.008	0.008	0.008	0.005	0.005	0.004	0.005	0.004	0.005
Hazards	0.010	0.001	0.001	0.001	0.000	0.002	0.001	0.001	0.001	0.001
Temp. stability	0.050	0.008	0.008	0.008	0.006	0.004	0.004	0.005	0.004	0.005
Lifetime	0.100	0.022	0.022	0.022	0.011	0.002	0.002	0.004	0.002	0.011
Remote sensing	0.200	0.000	0.000	0.000	0.032	0.008	0.048	0.048	0.048	0.016
Total performance value		0.098	0.097	0.097	0.120	0.067	0.152	0.149	0.128	0.091

TABLE 10

SENSITIVITY OF THE VALUE MATRIX TO A CHANGE IN WEIGHT FACTORS—MOISTURE IN BASE COURSE COMPACTION CONTROL

PERFORMANCE CHARACTERISTIC	WEIGHT FACTOR, w_i	MEASUREMENTMETHOD								
		GRAV. OVEN DRY	GRAV. ALCOHOL BURN	GRAV. CALCIUM CARB.	NUCLEAR	TENSI-OMETRY	ELECTRIC COND.	THERMAL COND.	HYGROM-ETRY	MICRO-WAVE
Availability	(0.200)									
	0.000	0.123	0.124	0.126	0.127	0.081	0.104	0.097	0.093	0.124
	0.100	0.130	0.131	0.133	0.133	0.075	0.103	0.089	0.093	0.113
	0.300	0.143	0.144	0.145	0.146	0.062	0.101	0.074	0.093	0.092
Portability	(0.100)									
	0.000	0.144	0.141	0.143	0.144	0.065	0.099	0.075	0.088	0.103
	0.050	0.140	0.139	0.141	0.142	0.067	0.100	0.078	0.090	0.103
	0.200	0.129	0.133	0.135	0.136	0.072	0.106	0.088	0.098	0.103
	0.300	0.121	0.130	0.131	0.132	0.076	0.110	0.094	0.103	0.103
Sample size	(0.100)									
	0.000	0.133	0.135	0.136	0.142	0.072	0.105	0.086	0.090	0.101
	0.050	0.135	0.136	0.138	0.141	0.070	0.104	0.084	0.091	0.102
	0.200	0.139	0.140	0.141	0.137	0.065	0.100	0.077	0.096	0.105
	0.300	0.141	0.142	0.144	0.135	0.062	0.097	0.072	0.099	0.107
Effect on sample	(0.100)									
	0.000	0.149	0.150	0.152	0.137	0.060	0.096	0.074	0.087	0.094
	0.050	0.143	0.144	0.145	0.138	0.064	0.099	0.078	0.090	0.098
	0.200	0.123	0.124	0.125	0.142	0.077	0.109	0.089	0.099	0.112
	0.300	0.110	0.111	0.112	0.145	0.086	0.116	0.096	0.105	0.120
Accuracy	(0.150)									
	0.000	0.129	0.135	0.137	0.138	0.070	0.105	0.086	0.099	0.100
	0.050	0.131	0.136	0.138	0.139	0.070	0.104	0.084	0.097	0.101
	0.200	0.138	0.138	0.139	0.140	0.068	0.102	0.080	0.091	0.104
	0.300	0.143	0.139	0.140	0.141	0.067	0.100	0.078	0.087	0.105
Speed of measure	(0.050)									
	0.000	0.140	0.139	0.139	0.138	0.070	0.099	0.077	0.096	0.100
	0.100	0.132	0.135	0.138	0.141	0.067	0.106	0.086	0.090	0.106
	0.200	0.125	0.131	0.137	0.143	0.063	0.112	0.094	0.083	0.112
	0.300	0.117	0.127	0.136	0.145	0.059	0.118	0.103	0.077	0.118
Moisture type	(0.050)									
	0.000	0.136	0.137	0.139	0.141	0.068	0.103	0.081	0.093	0.102
	0.100	0.136	0.137	0.139	0.138	0.070	0.102	0.082	0.093	0.104
	0.200	0.137	0.138	0.139	0.134	0.072	0.100	0.082	0.092	0.106
	0.300	0.137	0.138	0.139	0.131	0.074	0.099	0.083	0.092	0.108
Durability	(0.030)									
	0.000	0.136	0.137	0.139	0.140	0.068	0.102	0.081	0.093	0.103
	0.100	0.135	0.137	0.139	0.138	0.070	0.103	0.084	0.092	0.102
	0.200	0.134	0.137	0.139	0.135	0.071	0.104	0.087	0.091	0.102
	0.300	0.133	0.138	0.139	0.132	0.073	0.105	0.090	0.090	0.101
Reliability	(0.050)									
	0.000	0.135	0.136	0.137	0.142	0.067	0.104	0.081	0.094	0.104
	0.100	0.138	0.139	0.140	0.138	0.070	0.101	0.082	0.092	0.102
	0.200	0.141	0.142	0.143	0.133	0.074	0.097	0.082	0.089	0.100
	0.300	0.144	0.145	0.146	0.129	0.077	0.093	0.082	0.086	0.098
Time stability	(0.030)									
	0.000	0.136	0.137	0.138	0.141	0.067	0.103	0.081	0.093	0.103
	0.100	0.137	0.138	0.140	0.136	0.071	0.101	0.083	0.092	0.102
	0.200	0.139	0.140	0.141	0.131	0.075	0.098	0.086	0.090	0.101
	0.300	0.140	0.141	0.142	0.126	0.079	0.095	0.088	0.088	0.099
Hazards	(0.020)									
	0.000	0.137	0.138	0.140	0.142	0.067	0.102	0.080	0.092	0.103
	0.100	0.132	0.133	0.134	0.131	0.076	0.106	0.087	0.097	0.104
	0.200	0.126	0.127	0.128	0.120	0.086	0.111	0.094	0.103	0.105
	0.300	0.121	0.121	0.123	0.109	0.096	0.115	0.100	0.109	0.106
Temp. stability	(0.050)									
	0.000	0.135	0.136	0.138	0.141	0.068	0.104	0.080	0.094	0.103
	0.100	0.137	0.138	0.139	0.139	0.069	0.101	0.083	0.092	0.102
	0.200	0.139	0.139	0.141	0.137	0.070	0.098	0.085	0.090	0.101
	0.300	0.140	0.141	0.142	0.135	0.070	0.095	0.088	0.088	0.100
Lifetime	(0.050)									
	0.000	0.132	0.133	0.134	0.141	0.071	0.107	0.083	0.097	0.102
	0.100	0.141	0.142	0.143	0.138	0.066	0.098	0.080	0.089	0.103
	0.200	0.150	0.151	0.152	0.135	0.061	0.090	0.076	0.082	0.104
	0.300	0.159	0.160	0.161	0.132	0.056	0.081	0.072	0.074	0.105
Remote sensing	(0.020)									
	0.000	0.139	0.140	0.142	0.138	0.068	0.102	0.080	0.092	0.100
	0.100	0.125	0.126	0.127	0.146	0.072	0.106	0.087	0.097	0.113
	0.200	0.111	0.112	0.113	0.155	0.076	0.111	0.094	0.103	0.125
	0.300	0.097	0.098	0.099	0.163	0.081	0.116	0.101	0.109	0.137

The problem of inspection testing during and after construction is weighted in about the same manner as base-course compaction control; therefore, no additional tables were prepared for this problem.

Instrumentation for the study of moisture in concrete during drying was evaluated in Tables 13 and 14. Highest weight was given to moisture type. Effect on sample and remote sensing were weighted heavily also. The highest ranking for hygrometric methods results because the bound or unbound condition of the moisture to be measured in this case is considered to relate directly to the equilibrium relative humidity. Thermal conductivity and electrical conductivity also ranked well. The high ranking of microwave methods is related primarily to methods for observing laboratory-controlled samples.

The evaluation model is seen to provide insight into the relationship between instrument methods and measurement problems—a relationship based on performance requirements. It suffers noticeably from lack of more specific data. Much more satisfactory results should be achieved in the evaluation of field tests that employ all the instruments under identical conditions.

TABLE 13

DECISION MATRIX—STUDY OF MOISTURE IN CONCRETE DURING DRYING

	MEASUREMENT METHOD								
PERFORMANCE CHARACTERISTIC	GRAV. OVEN DRY	GRAV. ALCOHOL BURN	GRAV. CALCIUM CARB.	NUCLEAR	TENSI-OMETRY	ELECTRIC COND.	THERMAL COND.	HYGROM-ETRY	MICRO-WAVE
Availability	0.189	0.189	0.189	0.189	0.019	0.094	0.019	0.094	0.019
Portability	0.069	0.103	0.103	0.103	0.103	0.138	0.138	0.138	0.103
Sample size	0.093	0.093	0.093	0.070	0.070	0.209	0.233	0.070	0.070
Effect on sample	0.022	0.022	0.022	0.087	0.087	0.196	0.174	0.174	0.217
Accuracy	0.176	0.147	0.147	0.147	0.059	0.088	0.059	0.059	0.118
Speed of measure	0.065	0.097	0.129	0.161	0.032	0.161	0.161	0.032	0.161
Moisture type	0.098	0.098	0.098	0.073	0.061	0.061	0.061	0.366	0.085
Durability	0.125	0.139	0.139	0.111	0.083	0.111	0.111	0.083	0.097
Reliability	0.167	0.167	0.167	0.100	0.100	0.067	0.083	0.067	0.083
Time stability	0.152	0.152	0.152	0.091	0.106	0.076	0.106	0.076	0.091
Hazards	0.082	0.082	0.082	0.033	0.164	0.148	0.148	0.148	0.115
Temp. stability	0.152	0.152	0.152	0.121	0.076	0.076	0.106	0.076	0.091
Lifetime	0.222	0.222	0.222	0.111	0.022	0.022	0.044	0.022	0.111
Remote sensing	0.000	0.000	0.000	0.148	0.037	0.222	0.222	0.148	0.222

TABLE 14

VALUE MATRIX—STUDY OF MOISTURE IN CONCRETE DURING DRYING

		MEASUREMENT METHOD								
PERFORMANCE CHARACTERISTIC	WEIGHT FACTOR, w_i	GRAV. OVEN DRY	GRAV. ALCOHOL BURN	GRAV. CALCIUM CARB.	NUCLEAR	TENSI-OMETRY	ELECTRIC COND.	THERMAL COND.	HYGROM-ETRY	MICRO-WAVE
Availability	0.050	0.009	0.009	0.009	0.009	0.001	0.005	0.001	0.005	0.001
Portability	0.010	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
Sample size	0.100	0.009	0.009	0.009	0.007	0.007	0.021	0.023	0.007	0.007
Effect on sample	0.150	0.003	0.003	0.003	0.013	0.013	0.029	0.026	0.026	0.033
Accuracy	0.100	0.018	0.015	0.015	0.015	0.006	0.009	0.006	0.006	0.012
Speed of measure	0.010	0.001	0.001	0.001	0.002	0.000	0.002	0.002	0.000	0.002
Moisture type	0.200	0.020	0.020	0.020	0.015	0.012	0.012	0.012	0.073	0.017
Durability	0.050	0.006	0.007	0.007	0.006	0.004	0.006	0.006	0.004	0.005
Reliability	0.050	0.008	0.008	0.008	0.005	0.005	0.003	0.004	0.003	0.004
Time stability	0.050	0.008	0.008	0.008	0.005	0.005	0.004	0.005	0.004	0.005
Hazards	0.010	0.001	0.001	0.001	0.000	0.002	0.001	0.001	0.001	0.001
Temp. stability	0.020	0.003	0.003	0.003	0.002	0.002	0.002	0.002	0.002	0.002
Lifetime	0.050	0.011	0.011	0.011	0.006	0.001	0.001	0.002	0.001	0.006
Remote sensing	0.150	0.000	0.000	0.000	0.022	0.006	0.033	0.033	0.022	0.033
Total performance value		0.098	0.096	0.096	0.107	0.065	0.129	0.126	0.156	0.127

CHAPTER THREE

RECOMMENDED RESEARCH

EVALUATION OF PROMISING TECHNIQUES

Most moisture measurement problems related to highway engineering would benefit significantly from a comparative evaluation of available or potentially available instrumentation. The following research problems are identified as having a relatively high priority, as having candidate measurement methods to be compared, and as being representative of characteristic measurement problems. In addition to candidate methods, the comparative evaluation also should include reference and supplementary methods; i.e., methods that are unsatisfactory for routine application but that may be used to compare and clarify the performance of more promising techniques.

Special emphasis should be given to the evaluation of nuclear methods, radio frequency and microwave techniques, and fringe capacitance techniques.

Nuclear methods are well established in the area of compaction control. They are potentially applicable in many other areas, especially when nuclear density or mass measurements can be performed concurrently.

Radio frequency and microwave methods are potentially applicable to measurement problems characterized by low moisture content, freedom from dielectric interferences, and availability of concurrent density or mass data. Instruments with multiple-frequency capability and temperature compensation should be most useful.

The fringe capacitance method should be considered for measurement of moisture in soil or other compactible materials and for research problems requiring remote sensing. The impedance-matching techniques described by Thomas (1966) are considered to be a necessity for optimum performance of this method.

The specific areas for evaluation may be considered to be the components of a larger program to evaluate instrumentation for measurement of moisture in soil and mineral materials and mixtures.

Research Program 1

Title: Evaluation of Instrumentation for Moisture Measurement in Unconsolidated Materials in Stockpiles.

Candidate Methods: Nuclear, fringe capacitance, resistivity, electromagnetic radiation.

Reference Methods: Oven dry, alcohol burning, calcium carbide.

Research Program 2

Title: Evaluation of Instrumentation for Moisture Measurement in Unconsolidated Materials in Storage and Weigh Bins.

Candidate Methods: Nuclear, radiowave, microwave, capacitance, resistivity, hygrometric.

Reference Methods: Same as Program 1.

Research Program 3

Title: Evaluation of Instrumentation for Moisture Measurement in Unconsolidated Materials in Motion.

Candidate Methods: Nuclear, radiowave, microwave, capacitance, infrared.

Reference Methods: Nuclear (density), oven dry, alcohol burning, calcium carbide.

Research Program 4

Title: Evaluation of Instrumentation for Moisture Measurement of Compacted Materials in Depth.

Candidate Methods: Nuclear, fringe capacitance, hygrometric, resistivity, thermal conductivity, tensiometric.

Reference Methods: Oven dry, calcium carbide, alcohol burning.

Research Program 5

Title: Evaluation of Instrumentation for Moisture Measurement in Compacted Materials in Discrete Layers.

Candidate Methods: Nuclear, fringe capacitance, radiowave, resistivity.

Reference Methods: Oven dry, alcohol burning, calcium carbide, penetrometer.

Research Program 6

Title: Evaluation of Instrumentation for Moisture Measurement in In-Situ Materials in Depth.

Candidate Methods: Nuclear, resistance, thermal conductivity, tensiometric, hygrometric.

Reference Methods: Oven dry, alcohol burning, calcium carbide, penetrometer.

Research Program 7

Title: Evaluation of Instrumentation for Moisture Measurement in Cemented Materials in Layers and Other Shapes.

Candidate Methods: Nuclear, resistivity, thermal conductivity, hygrometric, radiowave, microwave.

Reference Methods: Oven drying.

Program:

Each of the foregoing research problems has essentially the same basic research plan:

1. Literature search and review.

2. Choose candidate methods.
3. Optimize instrument design to match field-test conditions (except for commercially adapted systems).
4. Develop operating procedures under laboratory conditions or through the use of a simulation model.
5. Install instruments in field-test environment.
6. Data analysis—apply the evaluation model to test results to determine the most suitable method(s); relate the tests results to problem solutions.
7. Reports.

Costs and priorities are summarized in Table 15.

MOISTURE MEASUREMENT PROBLEMS LACKING SUFFICIENT INSTRUMENTATION

Insufficient instrumentation for a given measurement problem may be attributed to lack of prior awareness of the problem, lack of effort to solve the problem, or technological difficulty of the problem. The following problems (as noted in Chapter Two) are hampered by the extreme difficulty of performing the measurements necessary to study the problem. A significant contribution to the solution of these problems could result from the development of a small, rugged, remote sensor for measuring moisture in highway materials.

Instrument for Measuring Moisture in Concrete

A primary problem encountered in measuring moisture is the many ways in which water exists in concrete. This problem may require the simultaneous measurement by many different methods. There has been much interest recently concerning the nature of moisture movement and rate of moisture loss in the presence of temperature gradients (McDonald, 1970; Thompson, Dempsey, 1970). Through the verification of theoretical models (including the interrelation of moisture and temperature) projections may be made into the more difficult problems that defy accurate measurement.

D-Line Cracking

The problem with D-line cracking is determining the amount, type, and state of the water present, and resolving minute inhomogeneities, without interfering with the normal freeze-thaw process.

Lens Formation and Frost Heaving

Spatial resolution is important here but not as much as in the foregoing problems. It would be helpful to determine the state of water in a nondisturbing manner.

Subgrade Seepage

Sufficient distribution of remote moisture measurement devices could localize subgrade seepage and in many cases result in repairs with a minimum of effort, and possibly even before damage has actually occurred.

Culvert Infiltration and Leakage

One solution to culvert infiltration and leakage might be the installation of many small sensors characterized by low maintenance and low cost.

TABLE 15

RESEARCH PROGRAM SUMMARY

PROGRAM NO. AND TITLE	TIME (MO.)	COST ($)	PRIORITY INDEX
1 Evaluation of Instrumentation for Moisture Measurement in Unconsolidated Materials in Stockpiles	18	82,000	22
2 Evaluation of Instrumentation for Moisture Measurement in Unconsolidated Materials in Storage and Weigh Bins	24	123,000	25
3 Evaluation of Instrumentation for Moisture Measurement in Unconsolidated Materials in Motion	24	164,000	26
4 Evaluation of Instrumentation for Moisture Measurement of Compacted Materials in Depth	18	95,000	22
5 Evaluation of Instrumentation for Moisture Measurement in Compacted Materials in Discrete Layers	18	91,000	27
6 Evaluation of Instrumentation for Moisture Measurement in In Situ Materials in Depth	18	95,000	21
7 Evaluation of Instrumentation for Moisture Measurement in Cemented Materials in Layers and Other Shapes	24	75,000	25
8 Development of Miniature Remote Sensors for Measuring Moisture in Highway Materials	24	118,000	26
9 Improved Field Method for Continuously Measuring Moisture Content of Bulk Aggregates	12	73,000	26
10 Improved Nuclear Method for Moisture Measurement in Highway Materials	12	62,000	27
11 Evaluation and Development of Radioactive Techniques for Evaluating Moisture, Density, and Strength of Pavement Components	24	122,000	24
12 Evaluation and Adaptation of Radioactive Techniques for Measurement of Moisture in Highway Materials (other than base courses under construction)	18	91,000	24
Total cost		1,191,000	

Gradation and Characterization of Earth Movements

Remote sensing is a necessary requirement, together with a large number of inexpensive sensors. The effective sample size must be extremely large.

Long-Term Moisture Changes in Highway Structural Section

Small sensors that can be remotely monitored and that do not interfere with normal component behavior are required.

Research Program 8

Title: Development of Miniature Remote Sensors for Measuring Moisture in Highway Materials.

Program:

1. Literature search and review.
2. Design and develop miniature moisture sensors that operate on the basis of resistance, capacitance, and thermal conductivity, and can be monitored remotely by physical contact with the sensor or by electromagnetic signal transmission.
3. Conduct laboratory and field evaluation.
4. Data analysis and reports.

The problem of monitoring moisture content of bulk aggregates was identified by Walker et al. (1970) as having a substantial priority. Water in aggregates must be included in determining total water of concrete paste. Because the water/cement ratio is of primary importance to the performance characteristics of concrete, the moisture content of the aggregate needs to be measured continuously to determine the amount of water to be added to the mix. Although there are suitable batch methods in use, there is no method that can continuously and accurately measure the moisture content of aggregate as it exists in stockpiles and bins.

Research Program 9

Title: Improved Field Method for Continuously Measuring Moisture Content of Bulk Aggregates.

Program (From Walker et al., 1970):

1. Literature search and review.
2. Develop a field method for continuously and accurately measuring moisture content of an aggregate mass.
3. Test and evaluate method.
4. Data analysis, synthesis, and reports.

SUGGESTED MODIFICATIONS OF AVAILABLE TECHNIQUES

Although the nuclear method is probably the most suitable method for in-situ measurement of moisture in compactible materials, there are potential modifications to this measurement that would reduce its susceptibility to interferences. It is assumed that bulk density corrections normally are applied. Interferences occur from slow neutron-absorbing materials as well as organic or hydrogenous materials. Although it is not widely considered, the total absorbing power of some of the rare earths such as gadolinium in the earth's crust is much higher than that of the more abundant or commonly considered elements, such as iron, boron, and chlorine. This may account for the occasional anomalies in nuclear measurements and also the frequent coincidence of the compensating effects of hydrogenous material and absorbing elements in some clays. A modification that would indicate the presence of abnormally high neutron absorbers would be to use the neutron-gamma technique in conjunction with the neutron scattering method. This should reduce some of the interference errors due to absorbers. Ignition tests also should be made occasionally in troublesome soils. These two added measurements should contribute to understanding and confidence in the nuclear method.

Research Program 10

Title: Improved Nuclear Method for Moisture Measurement in Highway Materials.

Program:

1. Literature search and review.
2. Develop and evaluate a nuclear method for measuring moisture, using the combined principles of neutron scattering and the neutron-gamma reaction.
3. Data analysis and reports.

Research Program 11 (Walker et al., 1970)

Title: Evaluation and Development of Radioactive Techniques for Evaluating Moisture, Density, and Strength of Pavement Components.

Research Program 12

Title: Evaluation and Adaptation of Radioactive Techniques for Measurement of Moisture in Highway Materials (other than base courses under construction).

The 12 research programs are summarized in Table 15. The priority index and costs were obtained in the manner used by Walker et al. (1970). The ease of solution was given a weight of 1; value of solution, 3; ease of implementation, 1; and geographical significance, 2. The costs were based on an average rate of $3,000 per man-month. Estimates of equipment costs varied between $5,000 and $20,000.

The total cost of the 12 programs is $1,191,000. The total cost of the two programs having the top priority index is $153,000. The top five programs have a total cost of $508,000. These programs represent needed research in the area of moisture measurement instrumentation. The cost is small in comparison with the costs associated with the problems they are intended to solve. Although all of these problems are not likely to be solved in one or two years, a significant contribution can be made through a continuous research effort guided by an awareness of present technological capabilities and the proper perspective of moisture measurement requirements in the many facets of highway engineering activities.

APPENDIX A

MOISTURE MEASUREMENT TECHNIQUES

In searching for all possible methods of measuring moisture, it was found convenient to divide potential methods by the chemical and physical processes on which the measurement principle is based. In this way the methods were broken down into sufficiently small categories in which the title is a partial description of the method used. It is not the purpose of this study to completely describe and characterize each of these methods and document the extensive research in this area. Consequently, an effort was made to be brief, and yet present the information needed by the highway engineer. Some background information is provided on the principle behind each moisture measurement, and as much information as possible is presented concerning the instrument performance characteristics. In many cases little specific information on these characteristics was available. In other cases extensive information was found in research reports, textbooks, and journals. In those sections of this appendix where it is desirable to present the results of many publications, an abbreviated format of presentation is used: the material is grouped by the author or agency conducting the work, and date (month/year) of publication. Pertinent material is described by a brief review, followed by a summary listing of the performance characteristics for which the work contributed additional information. The performance characteristics are indicated on the left by numbers from 1 to 35, which represent the numbered definitions of performance listed in Table A-1. Where only limited information on a particular method was available, it is presented in textual form.

1.0 HYGROMETRIC TECHNIQUES

Among the earliest references in hygroscopy are the works of Hooke (1665) and Boyle (1725). Leonardo da Vinci also is credited with the application of the relationship between relative humidity (RH) and the physical properties of hair. Considering the technological advances made in modern times, it is surprising that the hair hygrometer introduced by de Saussure in 1783 is widely used in industry today (Fraade, 1963).

The general nature of the relationship between moisture content in porous materials and the RH of the immediate atmosphere under equilibrium conditions is reasonably well known. Consequently, a relatively large number of simple devices for indicating and recording RH have been conceived. There are basic disadvantages, however, in using these methods to measure RH in a small cavity inside a porous material and deriving the moisture content of the solid. These include deteriorating effects of soil components on the sensing element, and the need for a special calibration for each material to be sampled. The latter is the result of the funadmental dependence on the moisture-retention characteristics of the material and the RH of the cavity. These methods have application, however, where RH in the material is related directly to other properties; e.g., the drying shrinkage of concrete. In evaluating hygrometric methods, the following characteristics are especially significant: (1) range of water contents of interest, (2) hysteresis effects in both the material and the sensitive element, (3) the size of the sensitive element, (4) the type of water to be measured, and (5) the durability of the sensor in the particular medium.

For a detailed study of humidity sensors, see Wexler and Ruskin (1965).

1.1 Electrical Resistance Hygrometer

1.1.1 Chemical Salts and Acids

The electrical resistance of sensors is modulated by changes in the water content of volumetric and electrolytic processes. Most resistive humidity transducers consist essentially of a hygroscopic material that absorbs water from the surrounding atmosphere. Generally, the material contains a salt that disassociates in the presence of water. The concentration of these disassociated ions is determined by the measurement of the electrical resistance between the electrodes applied to the transducers. The hygroscopic material is deposited on a substrate—either ion-impervious insulating surfaces, or fibers, or fabrics, or ceramics. Typical examples of hygroscopic materials that have been sug-

TABLE A-1

INDEX OF PERFORMANCE CHARACTERISTICS USED FOR THE LITERATURE REVIEW [a]

1. Availability	20. Temperature effects
2. Practicability	21. Interference
3. Portability	22. Radius of influence
4. Size	23. Sensitivity
5. Versatility	24. Range
6. Fragility	25. Accuracy
7. Durability	26. Stability
8. Serviceability	27. Precision
9. Simplicity of operation	28. Response time
10. Operator experience	29. Speed of measurement
11. Reliability	30. Time monitoring capability
12. Lifetime	31. Remote monitoring capability
13. Cost	32. Power requirement
14. Hazards	33. Calibration requirements
15. Sample weight	34. Maintenance requirements
16. Simple volume	35. Application materials
17. Effect on sample	
18. Physical state	
19. Moisture type	

[a] Terms are defined in Chapter Two.

gested are sulfuric acid, phosphoric acid, lithium chloride, calcium chloride, zinc chloride, and solutions of the tetrachlorides of tin, zirconium, hafnium, or lead (Wexler, Ruskin, 1965).

Reliability.—For accuracy, speed of response, and stability, some authors consider the most successful humidity transducers to be those fabricated on an insulating polystyrene form supported with two wires of a noble metal that are wound in the form of two parallel helixes (Dunmore, 1938). Others (Bouyoucos, Cook, 1965) consider lithium chloride (LiCl) and similar elements to be unreliable and short-lived, even with plastic sealers. Nielsen (1967) found them inadequate for long periods of time, or high RH.

Gause and Tucker (1940) used a Dunmore-type electric hygrometer coated with polyvinyl acetate. Bakelite washers were replaced by polystyrene washers as binding posts to prevent leakage at high humidities. They concluded that electrical resistance hygrometers are well suited to measure RH in concrete.

Temperature Effects.—The resistance at constant humidity depends strongly on the temperature; therefore, either operation at a constant temperature or effective compensation is required. A negative temperature coefficient normally is encountered (White, 1954).

Interference.—Ionic or hygroscopic materials cannot be tested. Certain volatile materials will interfere, such as acetone, acetylene, acid vapors, alkaline vapors, amines, ammonia, ethylene oxide, glycol vapors, salt air, sulfur dioxide, and saturated air subject to condensation (Roth, 1966).

Klein and Trescony (1965) suggest two potential solutions to problems with resistance hygrometers:

1. Hygroscopic sensory element that is unaffected by solutions of salts in the meter environment.
2. Protective covering, such as a membrane that presents a barrier to salt ions and is permeable to moisture.

For applications in fresh concrete or deep fills, they chose an ionic barrier moisture meter that was developed at the University of California (Blythe, 1957).

The hygroscopic sensory element is formed by dipping in a concentrated solution of polyvinyl alcohol in alcohol and water. This coating is dried at 60°C. Klein and Trescony (1965, Figs. 2 and 3) show the moisture meter and the monitoring circuit.

Leaching tests indicated that if the sensing element is encased successively in shells of an anionic and a cationic permselective membrane the moisture meter operates without salt interference.

Sensitivity.—A commercial unit produced by Honeywell (Roth, 1966, p. 84) has a lower limit of detection (LLD) of 0.5 percent (dry). This system encloses the sample in a small volume and allows about 10 min for the vapor pressure to reach equilibrium. The sensor is a thin film of LiCl on interlacing gold grids on a plastic base.

The resistance of salt solution is given by Schaffer (1946):

$$R = L^2W/mA \qquad \text{(A-1)}$$

in which

L = length of film;
W = equivalent weight of solute in film of electrolyte;
m = mass of solute; and
A = equivalent conductivity.

The sensitivity is obtained by differentiation with respect to relative humidity:

$$\frac{\partial R}{\partial(\mathrm{RH})} = \frac{L^2W}{m}(-1/A^2)\frac{\partial A}{\partial(\mathrm{RH})} \qquad \text{(A-2)}$$

Notice the inverse dependence on mass of solute. Experiments with LiCl solutions show a decreasing sensitivity as the amount of LiCl is increased.

As indicators of the moisture content of the solid enclosing the cavity, the sensitivity falls off for a cavity RH greater than 90 percent. Near saturation it is especially poor, with some materials doubling the moisture content as the RH of the cavity goes from 99 to 100 percent (Monfore, 1970).

Range.—The resistance of such transducers ranges from about 10,000 ohms to 10 megohms. The resistance variation corresponds to a limited range of RH of approximately 25 to 35 percent. Several transducer elements are required to cover the total range from approximately 2 to 99 percent RH. The different ranges can be made by using different concentrations of LiCl in the hygroscopic layer deposited on the insulated form. The individual sensors in the unit described by Roth covered a range of 4 percent (dry).

White (1954) indicated that a sodium dichromate solution was better than LiCl at higher humidity and temperature. The resistance of an alumina core saturated with sodium dichromate was found to vary linearly with RH between 30 and 99 percent. It was found that with a heater current a variety of responses could be achieved with possible control applications.

Ionic crystals have been considered as fractional dewpoint indicators (Wylie, 1955, 1962). The fractional dewpoint is indicated by the sensor temperature for which the surface resistance reaches some constant value. The RH at that temperature is then near the equilibrium RH of the crystal, such as: K_2SO_4 (99 percent), NaCl (76 percent), $CaCl_2$ (30 percent), and LiCl (15 percent). The method is limited and would require temperature measurement and control for implementation.

Response Time.—If the hygroscopic material is very thick, a long time is required for it to come into equilibrium with the surrounding atmosphere. Therefore, the dynamic response is poor and the indication of the system will show a time lag, perhaps of several hours. If the hygroscopic body is very thin, the physical process is limited to the surface, or to extremely near the surface layer, and the system is easily influenced by the surface contamination and mechanical injury, resulting in the measurements becoming nonreproducible. The sensing elements should be protected from long exposure to environments of 100 percent RH, because condensation is likely to damage the vulnerable surface.

The speed of response is high for LiCl sensors with respect to the hair hygrometer. If exposed to an abrupt

large change of RH at room temperature, the element will assume 63 percent of its equilibrium resistance within several seconds. However, the speed of the response depends on the air turbulence in the vicinity of the sensor. In a cavity, diffusion is the limiting mechanism. The speed of response also depends on the magnitude of the humidity change and on the temperature. The resistance changes faster when the humidity varies from a low to a higher value than it does in the opposite direction. The response time generally increases with increasing temperature. Faster response transducers are described by Wexler et al. (1962) using potassium metaphosphate in the humidity range of 83 to 100 percent. These transducers will reach 63 percent of the final resistance in approximately ½ sec at room temperature and in about 10 sec at —20°C. The response time of this transducer is decreased by factor of 20 to 30, compared to the previously described LiCl sensor.

The response time of sodium dichromate elements is a few minutes at room temperature and higher humidity values. However, at 0°C and humidity of less than 30 percent this time increases to about 1 hr. Barium fluoride films have been designed with a 1-sec response time at —20°C and 3 sec at —30°C. Response is essentially instantaneous at higher temperatures.

Accuracy.—Transducers of the described type are accurate and reproduce to within ±2.5 percent. However, special sensing elements with an error that does not exceed ±1.5 percent are commercially available (Wexler, Ruskin, 1965). The calibration curves generally are nonlinear, and an empirical calibration is required. Hysteresis is almost always a problem in the material being monitored, and frequently occurs with the sensor as well. The interpretation of results for the worst cases is thus very difficult.

Applicable Materials.—Shiba and Ueda (1965) suggest the use of resistance hygrometry for estimating the drying rate of a concrete wall. The difference in vapor pressure of ambient air and the surface of the wall was found to have an exponential relation with time.

$$\Delta p = ae^{-nt} \qquad \text{(A-3)}$$

The constants were determined for mortar blocks and were found to be: $a = 7.3$ Torr, and $n = 0.2$ day^{-1}.

The stability of these sensors in most materials for long periods of time probably is inadequate. Some improvement may be gained by the use of RH wells (Monfore, 1963).

1.1.2 Aluminum Oxide

Both resistance and capacitance of aluminum oxide films have been correlated with RH. This would allow its classification together with techniques 1.2 as well as techniques 1.1. The two techniques, however, are treated as one for this sensor.

Ansbacher and Jason (1953) and Cutting, Jason, and Wood (1955) have investigated and described an electrical hygrometer based on variations of the electrical properties of aluminum oxide with changes in humidity. Jason (1965) also has considered some of the basic limitations of this sensor.

The typical transducer is essentially a capacitor with a porous dielectric film of aluminum oxide. One capacitor plate should be permeable to water vapor to permit penetration into the aluminum oxide.

The porous oxide layer is formed by various means such as (1) sputtering, (2) vapor deposition, and (3) anodization. Anodization is more popular and is generally an acid electrolyte (17.5 volume percent H_2SO_4, current density 100 ma cm^{-2}, 30 min).

The apparent dielectric constant of such an oxide in the vicinity of saturation ranges from 1,000 to 8,000. A typical calibration curve is shown in Figure A-1.

The sensitivity decreases near the saturation values of RH. Typical accuracy is estimated to be about ±3 percent (Cutting, Jason, Wood, 1955). The dynamic response to a sudden variation of ambient humidity depends on the magnitude and direction of such a change. Response times range on the order of 10 to 100 sec. Response is slower and slight hysteresis effects are noticeable at high degrees of humidity. The resistance increases and the capacitance decreases, causing aging effects during the first few months after oxide formation. The transducer output is practically independent of temperature between 0° and 80°C. Changes in resistance and capacitance with variations in relative humidity may be irreversible in the temperature range of 100° to 400°C.

Cutting, Jason, and Wood (1955) list the following advantages and disadvantages of aluminum oxide sensors:

1. *Advantages:*
   (a) Insensitive to temperature variation from 0° to 80°C.
   (b) With care some calibration curve can be used for many units.
   (c) Change fabrication for different characteristics.
   (d) Displays humidity as one electrical parameter.
   (e) Physical size can be varied.
   (f) Air velocity insensitive.
   (g) Remote monitoring possible.
   (h) Stable after aging.
   (i) Rapid response time.
   (j) Rugged construction.
2. *Disadvantages:*
   (a) Problems above 90% RH are slow response, drift, hysteresis, low sensitivity, and possible destruction of element.
   (b) Applied voltage above 80% of formation voltage may cause temporary breakdown.
   (c) Oil or grease ruins response.

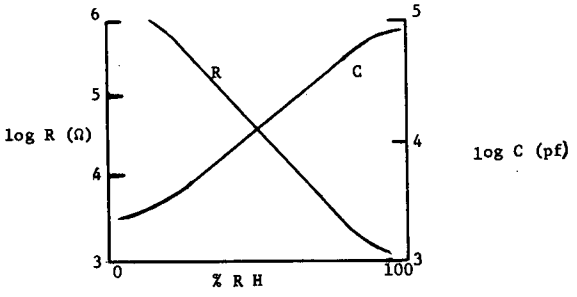


Figure A-1. Typical calibration curve.

Results of other research with aluminum oxide are given in the following, in the format mentioned at the beginning of this appendix. The numbers on the left refer to performance characteristics listed in Table A-1. The order is chronological.

Stover (6/63)*

Anodized aluminum (99.99%) 0.075 mil (~1.9 μ) thick is used. Fabrication formula is 50% sulfuric acid, 90°F, 25 min, and 12 a (AC) per ft².

4 Foil is 3 mils × 3/16 in. × 5/8 in. and weighs about 12 mg.
20 Linearity of 2% between 10 and 90% RH (good response curve on p. 634) between −80° and 120°F.
21 Condensation on surface has no permanent effect. (May not be true in presence of salts.)
23 Too-thin coating of gold causes high resistance; too-thick coating causes tight layer that does not allow moisture to migrate freely.
24 Range is 0-100% (0.000614-145 mm Hg, −106° to 130°F dewpoint, 1 ppm up to pure water).
26 Recalibration may be required if the unit is subject to severe conditions, or stored for long periods.
28 75°F, 100% → 50% RH . . . . . $\tau < 300$ msec.
75°F, 0% → 50% RH . . . . . $\tau < 100$ msec.

Parametrics (12/64)

Units were similar to those used by Stover, who consulted on the program.
Behaved as a pure capacitor ($Z \sim 1/f$). Any convenient frequency can be used.
4 Element dimensions: 3/16 × 5/8 × 0.003 in.
23 Elements were calibrated to frost points of −100°C. A linear response was obtained of impedance vs frost point.
33 Shift of 5°C in frost point resulted from high-vacuum storage for 4 mo.

Chleck (12/66)

Stover consulted in fabrication of sensor but does not give detailed description. Results are inconsistent with Brousaides (1968). Fabrication is an art.
25 1. Measured absolute humidity.
2. ±1.5° to 3°C error in dew point.
3. Good stability and aging.
4. Negligible hysteresis.
28 Response time is 2 sec at ambient conditions.

Locke (8/67)

Describes construction and operation of a small instrument for Martian water vapor detection.
4 Instrument weighs 0.8 lb with a volume of 36 in³.
25 Error is ±2°C dew point.
32 Power required is 0.4 w.

* Refers to date; (6/63) = June 1963.

Del Pico (10/67)

Used aluminum with 1.5% manganese. Anodized in 17.5% (V/V) H_2SO_4 at 25°C, 50 ma cm^{-2} for 30 min. Aquadog electrodes were applied. Table 1 (p. 15) gives solutions for calibrating RH.
Films were about 30 μ thick.
21 Polar gases such as ammonia, CO_2, CO, NO_2, and SO_2 interfere, but low concentrations should give negligible effect.

Morrissey, Brousaides (10/67)

Takes issue with Chleck (1966). Time constant may be longer and element may have temperature as well as RH coefficient. Glueckauf (1947) predicts an inverse dependence of response time on pressure.

Lai, Hidy (8/68)

Used 20,000- to 40,000-Å-thick sputtered films on a thin quartz fiber. Electrodes were gold on platinum with a gap between. Al_2O_3 was sputtered on gap and topped with porous graphite.
23 C_p and R curves shown on p. 1200.
24 Range is from 30 to 90%.
28 Response time is about 0.1 sec.

Disadvantages:

1. Low sensitivity at less than 20% RH.
2. Loss of sensitivity in storage for a few days.
3. Hysteresis-like effect given longer response time for changes from high humidity to low, then vice versa.
4. Limited range.

Barton, Maffei (10/68)

In nuclear reactor gases.
12 Unit performed satisfactorily for a year or more.
24 Range is from −110°C (<0.01 ppm) to 10°C (>10,000 ppm).
28 Response time was less than 10 sec.

Brousaides (10/68)

This technique is based on the fact that thin, porous films of Al_2O_3 are hygroscopic. Common practice is to prepare films by anodization in a porous-oxide-producing electrolyte. Behavior has not been satisfactorily explained in terms of diffusion and sorption theory. Large variations in reported results may be related to different construction techniques. Stover (1963) used AC anodization and Jason (1963) used DC anodization.
Figure 2 (p. 3) shows the sensor configuration.
20 The temperature dependence is approx. 10% RH per °C.
23 Response curves are shallow.
Factor of 2 change occurs between 20 and 95% RH. (The ML-476 carbon element changes by factor of 100.)

25 Accuracy is limited by temperature control and hysteresis. Hysteresis gives error of 10% RH or greater. Some error may be apparent hysteresis due to an excessively long time constant. Permanent hysteresis also is an unavoidable aspect of most porous materials. The "ink-bottle" theory advanced by Kraemer (1931) and McBain (1936) is based upon meniscus advance and decline in pores of varied geometry. Meniscus formation in the capillary is determined by the Kelvin equation:

$$\ln(P/p_0) = \frac{-2\gamma V \cos\theta}{R\,T\,r_m}$$

in which p_0 is the saturation vapor pressure; γ is the surface tension; θ is the contact angle; V is the molecular volume of the liquid; r_m is the mean radius of the meniscus; R is the gas constant; and T is the temperature. The partial pressure required to fill the neck ($r_m = r_n$) is less than that required to fill the bottle ($r_m = r_b$). Thus, during sorption when the meniscus reaches the neck, it fills spontaneously. However, during desorption, the pressure must fall below that required to empty the neck, resulting in significant hysteresis. (Soaking procedures reduce this effect, possibly by permanently filling the smaller pores.)

28 Response times of different units varied from 5 to 80 sec for 63% response for increasing RH. For decreasing RH, the response is about ¼ these values, 90% response times are as long as 15 min.

1.1.3 Electrolysis

The electrolysis system consists of a gas mixture flowing continuously over a thin layer of partially hydrated phosphorous pentoxide (P_2O_5), with the humidity in the gas absorbed by the P_2O_5 layer. A typical sensor is constructed with two platinum wires wound helically inside an insulating tube and then coated with a layer of P_2O_5.

A DC voltage is applied to the two noble wires, decomposing the water into gaseous H_2 and O_2. The DC voltage must be much larger than the polarization voltage (~ 2 V).

The current, I, during the time, t, will decompose a mass of water of

$$M_{\text{water}} = AIt \qquad \text{(A-4)}$$

For a humid gas flowing continuously with a constant-mass flow rate,

$$V_m = \frac{M_{\text{mix}}}{t} \cong \frac{M_{\text{gas}}}{t} \qquad \text{(A-5)}$$

the system will reach equilibrium when the rate of water absorbed by the P_2O_5 layer is equal to that decomposed by electrolysis. The current, I, is

$$I = K\,\frac{V_W}{V_g}\,V_m \qquad \text{(A-6)}$$

in which V_W and V_g are the partial volume of water vapor and gas. The constant, K, is

$$K = \frac{2 \times 96500}{18.016} \times \frac{W_{\text{water}}}{W_{\text{gas}}} \qquad \text{(A-7)}$$

in which W_{water} and W_{gas} are gram-molecular weights of the water and of the gas.

For air, Eq. A-7 becomes

$$I = 6.6 \times 10^{-3}\,V_m \frac{V_W}{V_{\text{air}}} \qquad \text{(A-8)}$$

in which V_W/V_{air} is the concentration by volume of water in air in parts per million (ppm). For example, for a volume flow of 100 cc/min, the current will be 13.2 μa/ppm by volume at a temperature of 25°C and a pressure of 1 atm.

The instrument is particularly useful for very small concentrations of water in a gas, usually between 1 to 1,000 ppm, with the lower limit of 1 ppm. Accuracy of the electrolysis system is between ±3 and ±5 percent (Beckman Instruments, Inc., *Bull. 4101*). For lower ranges of humidity, the water in the gas will not reach equilibrium with the hydrated P_2O_5 layer. Where the "efficiency" is low, empirical calibration frequently is required (Taylor, 1956).

The time response for a sudden increase in water vapor concentration is approximately 1 min to reach 63 percent of the final indication. An equivalent decrease in humidity corresponds to 2 min.

Disadvantages of the electrolysis system include the requirement for constant mass flow, constant temperature (induces an error of 0.3 percent/degree) and the interference from gases that would interact with P_2O_5.

Some specific findings of other work follow.

Fraade (4/63)

Device reported by Keidel in February 1956 at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. It is based on Faraday's laws of coulometry.

1 The technique is licensed by E. I. du Pont and manufactured by Consolidated Electrodynamics Corp., Beckman Instruments, and Manufacturers Engineering and Equipment Corp.

2 Rapid acceptance of this technique has led to industrial models with explosion-proof housing, rugged and simple components.

31 Modular design permits signal transmission to a remote central location.

Honnell, Hibbits (8/68)

An evaluation of accuracy is conducted.

The sensor is constructed of a bifilar helical winding of rhodium wire potted in plastic. Moisture is absorbed by a P_2O_5 dessicant deposited between the windings. The hydrated P_2O_5 is electrolyzed with a 70-v DC potential releasing H_2 and O_2.

1 The unit is the Electrolytic Hygrometer, Model 17901, manufactured by Beckman Instruments.

16 100 ml (gas) is sampled in 1 min.

19 Sensor responds to unbound water.

23 The sensitivity is 1 ppm.

24 The range is 20-260 ppm.
25 Accuracy is ±5% or 2 ppm, whichever is greater.
30 System provides continuous operation.
31 It is suitable for remote monitoring.

Roth (8/66)

Description: This is an electrolysis system, but is used in a gravimetric fashion in that the sample is weighed, heated to volatize the moisture, and the resultant vapor is carried into an electrolytic cell by a stream of dry nitrogen.
1 Consolidated Electrodynamics; Manufacturers Engineering. The former is a single-cell instrument; the latter, a dual cell.
21 Ammonia, amines, and alcohol are known to interfere.
23 Lower limit of detection is 1 μg of water.
24 1 μg to 0.1 g of water. Materials with moisture of less than 10% are most suitable.
25 Calibration can be obtained with such standards as sodium tartrate dihydrate. Calibration curves are not necessary.
28 Response is practically instantaneous, but time is required to volatize the moisture.
29 10 to 30 min are required for measuring most materials.
30 Intermittent operation.
35 Can be used with any solid material, preferably granular or powdered.

Thacker (8/67)

1 Solids Moisture Analyzer, Type 26-321A, made by Consolidated Electrodynamics Corp.
23 Lower limit of detection is 10^{-7} g.

Barton, Maffei (10/68)

Nuclear reactor gases are studied.
20 Residual conductivity (zero moisture) varies with temperature. This is less significant at higher moisture levels.
23 Performance is poor at less than 1 ppm.

Kreider (9/68)

Moisture in solid radioactive material is measured.
1 Consolidated Electrodynamics Corp.
24 Range is 1 μg to 10 mg per sample.
25 Accuracy is 2% or ±20 μg, whichever is greater (manufacturer's specifications).

MacCready (9/62)

A miniature breadboard model P_2O_5 system was built and studied for use in Martian atmosphere.
2 Permits only zero moisture (i.e., dessicant effect).
4 Weight = 11 oz. Volume = 450 cc.
21 Appears to be somewhat sensitive to hydrogen, as follows: $P_2O_5 + XH_2 \rightarrow H_2O +$ some compound of hydrogen.
25 Minimum detection limit is 0.5 ppm.
32 Power required is 0.5 w.

Meteorology Research, Inc. (9/65)

An extension and conclusion of earlier work (MacCready, 1962). Functional problems were significant.

1.1.4 Thermal System

Although similar in appearance to a previously described method, the thermal system operates on a different principle and permits an absolute determination of humidity. The element consists of a tubular wick made from glass fibers and impregnated with a hygroscopic salt (LiCl) insulated from a thin metal tube. Two silver wires are bifilar wound around the wick and connected to an AC voltage source with some means for current limiting. The salt absorbs humidity from the surrounding air and becomes electrically conductive. The current passing through the LiCl generates heat and tends to evaporate humidity from it. An equilibrium is soon reached when the layer neither gains water nor loses water to the surrounding air. Equilibrium is reached at that temperature of the salt solution at which the partial pressure of water over a saturated solution just equals the ambient water vapor pressure. The temperature is measured by means of a resistance thermometer, thermistor, or thermocouple, and should be in thermal contact with the LiCl layer.

The vapor pressure of saturated LiCl solution at different temperatures is accurately known from tables (Wexler, Ruskin, 1965); hence, the instrument needs no empirical calibration. The output can be calibrated in dew-point temperature directly. The instrument can be used over a range of relative humidities from about 15 to 100 percent at temperatures of −30° to about 70°C. The regions of highest precision are between −12° and +34°C and above 41°C dew point.

The error of the instrument is on the order of ±2° to 3°F dew-point temperature. To reach 98 percent of the equilibrium temperature requires about 2 to 4 min. The instrument is susceptible to wind speed and water droplets. Cell reconditioning is recommended every 90 to 100 days (Hickes, 1947; Conover, 1950).

A commercial instrument of this design is made by Foxboro and Minneapolis-Honeywell Regulator Co. for operation between 40° and 120°F ambient temperature. Fraade (1963) listed the following interferences: sulfides, ammonia, suspended ionic solids or vapors, acid vapors, vapors of hygroscopic liquids such as glycerine or glycols, reactive organic vapors such as ethylene oxide, alcohols, or ketones.

A different heating approach is described by Byrne and Rose (1968). A saturated solution of LiCl in an absorbent tissue between capacitor plates is heated with a 2-w 70-MHz oscillator. The equilibrium temperature is a measure of RH. The operating range is from 1.3 to 30 mb vapor pressure. Byrne and Rose found no significant hysteresis.

1.1.5 White Hydrocal

Bouyoucos and Cook (1965) describe what they consider to be the best hygrometer available. Stainless steel electrodes are cast in white hydrocal. This is a form of plaster

of paris cement that sets hard, is pure, has low solubility, and has no added salts. This unit operates over a range of 12 to 100 percent RH and is independent of temperature between 2° and 32°C. When calibrated with saturated salt solutions the resistance as a function of RH exhibits a very small hysteresis. Recalibration should not be necessary for at least a year. The over-all accuracy of the unit is between 1 and 2 percent up to 75 percent RH, and between 2 and 3 percent from 75 to 100 percent RH. The design reflects the authors' previous experience in using resistance probes for soil moisture measurements.

1.2 Dielectric Systems

1.2.1 Capacitive Transducers

The high dielectric constant of water (approximately 80) suggests the use of capacitive methods for determining water content of gases and solids. (Discussion of water content in solids appears in the capacitance section.) The low concentration of water in air, even at saturation, causes a very small variation in capacitance. The dielectric constant of gases under normal conditions changes from 1.000247 for dry air at 45°C; for saturation it is 1.000593. For such small capacitance changes, very complex equipment for readout is required.

Capacitive systems have been developed using two concentric cylinders for the capacitor plates (Webb, Neugebauer, 1954). The capacitance change in response to water vapor variation causes a frequency variation of an oscillator at about 2 MHz, indicated by the beat frequency method. Dynamic time response is limited only by the time required for flushing the cell. The system is capable of detecting changes of 1 mg of water vapor per liter of air. Typical flow rates permit 50 percent of the final indication in 0.1 sec.

Sensors of this type are usable in the range of 10 to 100 percent RH over a temperature range of −35° to 80°C (Nelson, 1965). A sensor with a typical capacitance of 500 picofarads at 25°C will result in a capacitance variation of 11 percent for a RH change from 10 to 90 percent. The dynamic response is 15 sec at 25°C, but increases to 1 hr at −35°C.

These sensors show a substantial variation of capacitance with temperature as well as RH; however, it appears feasible to compensate with additional circuitry.

Improvements in the capacitive method have resulted from the work of Nelson and Amdur (1965). The main disadvantage of this technique is the relatively small change in capacitance of the sensor with a change in humidity. One way to increase this capacitance change is to displace the air with a material that will pick up an increased amount of water. Some desirable qualities of the dielectric material are: (1) low water absorption (<½ percent at 100 percent RH), (2) high permeability to water vapor, (3) low permeability to contaminants, (4) good mechanical strength, (5) high bulk resistivity, and (6) stability with wide ranges of temperature and humidity.

Such a capacitive transducer is constructed with a thin plastic film of acetal resin, which is a crystalline form of highly polymerized formaldehyde. The capacitor plates are formed with evaporated gold electrodes thin enough to be pervious to water vapor yet electrically conductive.

Charlson and Buettner (1963, 1964) used a hygroscopic liquid to improve the performance of this technique. The sensor consists of a porous plate capacitor through which air passes. Fluoropak granules covered with a thin layer of a hygroscopic liquid form the dielectric. The humidity is described by the relative capacitive response:

$$\mathrm{RH} = \frac{A\Delta C}{A\Delta C + 1} \tag{A-9}$$

in which A is the experimentally derived sensitivity coefficient approximately equal to 0.1 pF^{-1}. Liquids used are carbowax, polyethylene glycol, glycerol, ethylene glycol, and triethlene glycol dimenthyl ether. The sensor is free of hysteresis, and experimentally exhibits a 63 percent response in 10 sec. This response time is theoretically given by

$$t_{0.63} = \frac{n_s}{k_g P_T} \tag{A-10}$$

in which $t_{0.63} = 63$ percent response time;

n_s = number of moles of hygroscopic solvent;
k_g = gas phase mass transfer coefficient (mole sec^{-1} mb^{-1}); and
P_T = saturation vapor pressure of water at sensor temperature.

Many problems are evident with this system. The temperature coefficient is about 6 percent per degree Centigrade. Interference occurs with ethanol, carbon monoxide, and other polar molecules. Irreversible damage may result from O_3, NO_2, NO, SO_2, or a RH of more than 50 percent.

The accuracy of the measurement is 3 to 5 percent or about 0.1 mb in field tests with a precision of about 1 percent. The minimum detectable level is 0.1 mb (−40°C frost point); the developers believe it is possible to reduce this to 10^{-3} mb, or better.

1.2.2 Microwave Refractometer

The microwave system is an arrangement to determine the resonant frequency of a cavity. The resonant frequency varies with the dielectric constant of the material in the cavity. If f_0 is the resonance frequency of a cavity containing a reference gas (dielectric constant t_0) and f_1 is the resonance frequency of the cavity with the gas to be measured, then

$$\frac{\epsilon_1}{\epsilon_0} = \left(\frac{f_1}{f_0}\right)^2 \tag{A-11}$$

The quantity ϵ is the complex dielectric constant of the form $\epsilon = A + jB$; however, experiments have shown the loss factor for gases and water vapor may be neglected in the region 10^{10} Hz (Sargent, 1955).

A frequency modulator shifts the Klystron frequency in such a fashion that one cavity will resonate before the other cavity if the gases in the two cavities have different dielectric constants. The resonance maximum at the detector will be displaced in time by an amount that is pro-

portional to the difference between the resonant frequencies of the cavities. The gas temperature and pressure in the cavity remain constant.

At an operating frequency of 10^{10} Hz the difference in resonance frequency is 0.27 MHz for one cavity filled with dry air and another filled with air plus a water vapor pressure of 100 mb.

Response time is determined by the flushing time of the cavities.

The wide range, accuracy, and sensitivity of this technique makes it superior to all other systems. Electronic and material specifications, however, are stringent (Birnbaum, Kryder, Lyons, 1951; Birnbaum, Chatterje, 1952; Bussey, Birnbaum, 1953).

A microwave refractometer of different construction is described elsewhere (Crain, 1948, 1950; Crain, Deame, 1952).

Several types of double tuned cavities are discussed by McGavin and Vetter (1965). Response is a function of pressure, temperature, and humidity. Over-all accuracy of a laboratory model is 2 percent absolute humidity.

Brown and Billeter (1967) estimated the feasibility for three methods of monitoring trace moisture in reactor coolant gases such as helium: (1) frequency shift of a resonant cavity (dielectric constant change), (2) phase shift in a waveguide (dielectric), (3) attenuation in a waveguide. The third method requires a long path (0.1 dB km^{-1} at 22 GHz). The first two exhibit decreasing sensitivity with temperature.

The microwave refractometer is a very accurate technique for measuring RH if the temperature is controlled accurately. However, it does not appear to be easily adaptable to the measurement of moisture in most highway components.

1.3 Piezoelectric Sorption

In this method the sensing element is a quartz crystal coated with a hygroscopic material. The resonant frequency of such a crystal depends on the crystal mass (King, 1964). The mass (and, hence, resonant frequency) changes with the adsorption of moisture. A typical sensitivity factor is about 1 Hz per Å thickness of added material.

To date, the technique has not been applied to highway engineering problems. It has the disadvantages characteristic of other hygrometric methods. The Model 510 Moisture Analyzer (E. I. du Pont de Nemours & Co.) operates on this principle to measure moisture in gases. Two crystals are alternately exposed to sample air and dry air. The frequency difference between the two is indicated on the analyzer scale in parts per million (ppm) water vapor by volume.

The range of the instrument described is from 1 to 25,000 ppm (V/V).

The accuracy is ±5% of full scale. The lowest range is 0 to 5 ppm.

Precision and accuracy remain stable up to 18 mo.

Although applicable to flowing gases, the method would require extensive modification before it could be used for moisture in solids.

1.4 Optics

1.4.1 Emission

Potential methods for analyzing water content of moon materials were studied by van Tassel and Salisbury (1964). Remote sensing methods considered were radio waves, infrared (IR), visible, ultraviolet, and X-radiation. IR emission was considered to be the least ambiguous. If particles are micron sized, the identifying spectral structure is lost. Particles greater than 15 μ (or the wavelength of the IR source used) produce a diagnostic spectrum.

No advantage for this technique is seen at this time.

1.4.2 Absorption and Transmission

The existence of optical absorption bands and transmission windows in the IR region provides a specific and accurate means of measuring humidity. Wood (1958) designed an optical instrument with a 12-in. absorption path and germanium narrow-band filters to compare the transmission at 2.54 μ and 2.60 μ. The ratio of the amplitudes at the two frequencies is a function of water vapor concentration. A rotating disk that alternately passes each wavelength is installed in front of a lead sulfide detector. This technique is similar to a U.S. Weather Bureau rotating section filter.

Fractional absorption (A) is given by

$$A = CW^{0.5}(P + p)^{0.23} \qquad \text{(A-12)}$$

in which

W = water vapor concentration;
P = total gas pressure; and
p = water vapor pressure.

At concentrations below 0.2 g m^{-3}, variations of 1 mg m^{-3} (1 ppm) can be detected. The 95 percent response time is less than 5 sec.

Accuracy of the system is ±0.5°F (dew point at 32°F), ±1°F (dew point at −20°F), and ±2°F (dew point at −50°F).

Brenden, Coleman, and Moore (1967) used a similar technique but compared absorption at 2.55 μ and 2.3 μ. This matches the best lead sulfide response, but over-all accuracy was not improved.

Salkowski (1966) used IR absorption at 2.8 μ to measure moisture in jet fuel and residual fuel.

The optical-acoustic gas detection method has been designed for water vapor (Mikhaylenko, 1966). In this method the radiant energy of an IR source is absorbed in a sample cell and a reference cell. Expansion caused by the absorption of this energy provides the signal for the instrument output. The response time is about 20 sec.

Interference by CO_2 and other gases normally is about ±0.1 percent of range. The maximum range is 15 to 100 percent. Over-all accuracy is ±2.5 percent of range, with hysteresis accounting for ±1 percent.

Other than those between 2.5 μ and 3.0 μ, absorption bands of water that have been used are near 1.87 μ, 1.37 μ, and 1.12 μ. These shorter wavelengths have weaker absorption bands and sometimes are used to analyze moisture in solids (see technique 6.2.4).

1.5 Dimensionally Varying Elements

Engineers have long been aware of the variation in size of certain materials as the humidity changes. The practicality of using this phenomenon in humidity measurement is evidenced by its wide use in industry (Fraade, 1963). Some of its desirable features are easy calibration, little manual know-how required, and easy recording of results. It is characterized by poor accuracy and a host of other difficulties, but it performs well where these deficiencies can be tolerated.

Monfore (1963) developed a system for determining moisture in concrete based on the expansion of a dacron thread monitored by a wire strain gauge. The accuracy was found to be ±2 percent of the calibration value. The response time was less than 5 min.

A special need exists for a small probe for measuring moisture condition in concrete specimens that are to be fire tested, because moisture content and distribution affect the test results for fire resistance. The Monfore type was found to be better than the Dunmore type (LiCl) for this application (Abrams, Monfore, 1965). Narrow-range Dunmore types require 10 elements to cover the range between 2 and 99 percent RH. A copper wire in series with the wire gives sufficient thermal compensation between 70° and 80°F. A two-point calibration (0 and 100 percent) requires about 20 min. Humidity wells are required for concrete. Little difference is noted between 5/32- and 1-in.-diameter wells. The units have a long life and are reusable in many samples.

Dimensional variation adapts readily to vibrating wire strain gauges. A vibrating wire "telehygrometer" is described by de Castro (1962). Using 16-cm white oak and scotch pine strips as sensing elements, an accuracy of ±2 percent RH is obtained over the range of 5 to 27 percent. The maximum operating range is a function of soil type. Hysteresis of up to 10 percent RH is noted.

This method should be adaptable to remote monitoring of RH in porous cavities. The apparent need is for sensing materials that are compatible with the highway component environment as well as strain sensing devices. Ceramic materials are potentially useful, but their response to moisture variation normally is very long (Cole, Birtwistle, 1969).

The use of very thin materials and miniature strain gauges could minimize this deficiency.

1.6 Dew Point

Dew or frost point hygrometers depend on the measurement of temperature and are relatively simple and inexpensive devices. The temperature at which dew or frost appears on the surface of a cooled object denotes the point at which the RH at the surface is 100 percent. This temperature thus may be related to the partial pressure of water in the sample gas via the known saturation vapor pressure of water or ice. Many different methods of cooling a surface and of detecting the formation of condensation have been used. Modern improvements in these methods, including the use of Peltier devices for cooling and photometric detection of condensation, have both added to the cost of the device and afforded an accurate and useful instrument (Wexler, Ruskin, 1965). Commercially produced instruments of this type may be the most accurate hygrometers currently available. However, the slow response necessitated by the thermal characteristics of Peltier coolers limits their application to relatively static situations. Such instruments may be useful for calibration of faster-responding sensors.

Commercial instruments are produced by Illinois Testing Laboratories, Compu Dyne Corp., and Technology/Versatronics. Interference may be caused by condensable vapor such as hydrocarbons (Fraade, 1963).

In the Technology/Versatronics (1968) instrument a thermoelectric cooler is used and a quartz crystal serves as an internal reflector that is affected by condensation on its surface. The accuracy of this instrument is about 0.5°F dew point.

Units designed for smallness and portability include one using alpha radiation attenuation by a condensing film at the dew point (Brousaides, Morrissey, 1967), and another that uses a small jet of freon from a 1-lb tank to cool the condensation mirror. The latter unit weighs 1,800 g and operates for only 1 hr.

The most likely candidate for measuring RH in a cavity is a small unit designed for space application by Cambridge Systems (1966). It weighs 2 lb 4 oz. The sensor is 1 in. in diameter and 3 in. long; the control unit is 6 × 2 × 2.5 in., including telemetry circuitry. Other specifications are:

Linearity	1.2°F.
Range	40° to 100°F dew point or greater.
Accuracy	±1°F dew point.
Response time	<10 sec.
Power	<3 w (28 v DC).

1.7 Psychrometry

Psychrometric instruments form another major grouping of hygrometers. All such instruments depend on the cooling of a wetted thermometer and the subsequent temperature difference between the "wet bulb" and the "dry bulb" of the temperature sensor. Data usually are reduced by the use of the psychrometric formula or a set of tables based on this formula. Although this principle and its theories have been known more than 250 years, definitive papers on the sensitivity and time response of the psychrometer were still being presented in the 1930's. The chief advantage of the psychrometer is its simplicity. If the ambient air has a velocity of more than 3 m sec^{-1}, two thermometers, one covered with a wet wick, are all that is needed. If wind is nonexistent, aspiration must be provided either by a fan or by moving the wet bulb through the air, as with the sling psychrometer (Assman, 1892). Details of the theory for psychrometric methods are found in Arnold (1933), Spilhaus (1937), and Wexler, Ruskin (1965).

Difficulties leading to erroneous readings arise when the RH is low and the wet bulb depression is large. Further problems arise with the response time with wet bulb temperatures of less than 0°C.

Fraade (1963) describes an instrument manufactured by Mine Safety Appliances Company. The technique is based

on the wet-dry principle. Materials such as cotton and wool absorb; their temperature rises. Upon desorption, their temperature falls. Alternate exposure to sample gas followed by dry gas results in a fluctuation in temperature that is related to the RH of the sample gas. Each cycle requires about 4 min.

The requirement of a flowing gas prevents the use of this technique in solids.

2.0 ELECTRICAL RESISTIVITY

The resistance of a material, its specific conductive capacity, and its dielectric losses vary with the amount of moisture it contains. An instrument for measuring one of these quantities thus can be calibrated in terms of moisture content.

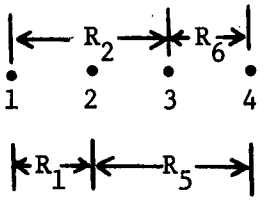
Some of the earliest work of importance in this area was done by Whitney et al. (1897) in soil and Spencer (1938) in concrete. The use of gypsum absorbent blocks has resulted from many difficulties in making direct contact, salt interference, and similar problems (Bouyoucos, 1940). Other types of absorbents used are nylon and gypsum, fiberglass and gypsum (Bourget, 1958), aluminous cement (Croney et al., 1952), and portland cement mortar (Mertin, 1965). Four-point electrical probes have been used (McCorkle, 1931, and Neto, 1962) to overcome electrode contact problems.

Instrument characteristics that have received most of the attention in application and evaluation studies are response time, size, interference, sensitivity, and accuracy. The advantages of the method generally are considered to be the small size, simplicity of operation, and rapid response. The disadvantages are primarily those characteristics that are reflected in the relatively poor accuracy of the method.

In spite of the problems cited, Mertin (1965) concluded that electrical conductivity methods most nearly satisfied four important requirements for measuring moisture in concrete: (1) nondestructive, (2) telemetering, (3) continuous measuring for weeks or months, and (4) a large number of measuring points.

2.1 Sample Resistivity

Resistivity measurements can be made by direct contact of the electrodes with the material of concern. With this principle, a calibration must first be obtained for the specific material. The electrical resistance method developed by Bouyoucos and Mick (1948) relies on the change of resistivity of the soil with changes in moisture content. Two electrodes covered with nylon, fiberglass fabric, or plaster of paris are buried in the soil and allowed to reach equilibrium before the measurement is made. This type of measurement is most accurate at very low moisture content. It has the disadvantage of a long response time to reach equilibrium.



$$R_s = \frac{R_5 + R_2 - R_6 - R_1}{K}$$

Figure A-2. Measurement of resistances.

Some typical examples of electrode configurations are (1) sample cells for loose material, (2) clamps for boards, (3) rollers or parallel plates for moving sheet material, (4) probes for plunging into loose material, (5) sword electrodes for insertion between stacked sheets, (6) needle electrodes for penetrating wood or textile fiber, and (6) surface electrodes for pressing against flat surfaces (Geary, 1956).

The resistivity measurements are related to the free water content (Monfore, 1968), although some authors believe partially bound water also plays a role (Roth, 1966).

2.1.1 Interference

Even in the early work of Whitney et al. (1897) the soil resistivity was found to depend on soil type and the presence of ionized salts. This is an inherent problem with the method, because the conductivity of the sample or sensor material results from impurities and soluble components in the material that are dissolved in the water. The interpretation of results obtained using these methods must recognize the limitations imposed by the basic source of the changes in resistivity of moist materials.

The variable contact problem can be partially overcome by using four electrodes. McCorkle (1931) placed his electrodes in a line about 2 ft apart. The resistances were then measured as shown in Figure A-2, in which K depends on probe length and distance between probes.

Smaller electrodes have been used. The portable hand unit of Szuk (1965) attempted to achieve reproducibility in the contact pressure applied for each measurement. This unit and others require calibration for each type of soil or material.

2.1.2 Sensitivity

The sensitivity of the method is relatively high, except near saturation. Neto (1962) found this to be true also with the four-point probe.

2.1.3 Accuracy

With a rather limited variation in type of material the accuracy is usually adequate. Kondo and Norose (1962) made surface measurements on mortar and plaster with an estimated standard deviation of less than 1 percent, using conducting rubber electrodes with a constant contact pressure. The errors in McCorkle's data appear to be about 2 percent (dry).

Szuk (1962) made moisture measurements on sand and gravel samples by adding a potassium chloride solution. This masked the effect of electrolytes dissolved out of the sample, but the effect of particle size remained significant. His accuracy was about 0.5 percent.

2.1.4 *Other Applications*

Szuk observed a significant feature with concrete in the form of an inflection point that occurs in the resistance profile as the w/c ratio gradually changes with the addition of water. This corresponds to the point at which all particle surfaces are wet, and coincides with the maximum crushing strength of the concrete. This inflection point is also useful in indicating the wetting point of various aggregates.

Roth (1966) describes an instrument for measuring moisture in sheet materials manufactured by several companies (Electronic Automation Systems, Mt. Hope Machine Co., Hartley Controls Corp.). The method is a nondestructive surface measurement in which the instrument continually balances the resistance and capacitance of the test material, using two well-balanced servo-loops. Percentage of moisture is proportional to the position of the resistance balancing loop. Compensation is provided for other materials that contribute to the conductivity of the sample. The transfer function is a direct exponential relationship with percentage of moisture in the low moisture range. The over-all range is 3 to 50 percent (dry). An accuracy of ±0.5 percent is claimed. The response time of the system is limited only by electrical components so that it is suitable for rapid measurement and control processes.

2.2 Sensor Resistivity

The fundamental difference between sensor resistivity methods and sample resistivity methods lies in the requirement that the sensor reach moisture equilibrium with the sample. Thus, the sensor resistance depends on (1) the relative attraction of the soil and the matrix material (sensor) for moisture, (2) the amount of moisture present, (3) the rate that water transfer can be made from one material to the other, and (4) the electrical conductivity of the components (moisture, solution, soil) within the electrical influence of the electrodes.

The most common sensor materials that are used for soil and concrete are gypsum (plaster of paris), nylon-gypsum, fiberglass-gypsum, and resin-treated gypsum. A relationship exists between this technique and the tensiometric and hygrometric techniques. The difference lies in the equilibrium mechanism. Closs (1954) developed a calibration technique for the resistance block and the soil moisture tension based on the relation between the freezing point depression ($\Delta\tau$) due to pressure on the water. The resistance is measured at the freezing point and the moisture tension is given by

$$pF = 4.097 + \log \Delta\tau \qquad \text{(A-13)}$$

Nielsen (1967) used a gypsum cell to relate shrinkage and creep to moisture content in concrete. Capacitance and phase difference at 1 KHz were calibrated with RH. Care must be taken in the comparison of results obtained by the different methods, however, because vapor transport and equilibrium may be entirely different from total moisture transport between two materials. Moisture retention relationships vary between materials and between soils.

2.2.1 *Portability*

Salaruddin and Khasbardar (1967) describe a portable unit. Most applications take the form of inserted probes or semi-permanent sensor installation.

2.2.2 *Reliability*

Clean procedures in preparing gypsum improve reliability (Nielsen, 1967). Soil moisture blocks are more dependable at low moisture content below field capacity than at higher moisture content (Johnson, 1962).

2.2.3 *Temperature Effects*

These units are temperature-sensitive, but it is not their major cause of error (Taylor, 1955; Bourget et al., 1958). A 3.5 percent change in resistance per degree Centigrade occurs in the portable unit mentioned previously.

2.2.4 *Interference*

All units are sensitive to salts (Bourget et al., 1958). NaCl content greater than 0.01 N (1,800 ppm) causes significant deviation (Hancox, Walker, 1966). Fiberglass units are more sensitive to salts than are gypsum units (Taylor, 1955).

2.2.5 *Sensitivity*

An S-shaped response curve is common with the sensitivity being low at the higher moisture content (Building Research Station, Watford, 1962; Johnson, 1962). The sensors of Hancox and Walker (1966) exhibited a log-log relation between resistance and moisture.

$$R = K(c\phi)^{-n} \qquad \text{(A-14)}$$

in which

c = ratio of moisture content to the value at saturation; and
ϕ = porosity.

Typical values for the constants are

$K = (3.2 \pm 0.2) \times 10^3\ \Omega$
$n = 1.3 \pm 0.05$

These sensors were formed as plaster cylinders 5 cm long and 0.5 to 1 cm in diameter, with conducting silver point at each end as the electrodes.

2.2.6 *Range*

Fiberglass blocks normally have a greater range than gypsum. Salaruddin and Khasbardar (1967) used two sensing elements: (1) a combination of fiberglass and gypsum for the high moisture range, and (2) gypsum for the low moisture range. The resistance changes from about 500 ohms to 10^5 ohms for the normal range of gypsum blocks.

2.2.7 *Accuracy*

Bouyoucos (1961) found an increase in accuracy and sensitivity by using 80 parts of water to 100 parts of

gypsum. This ratio gives increased pore space, with some sacrifice of stability. The variation between measurements by different units made by Hancox and Walker was $< \pm 1.5$ percent. The best accuracy claimed by the developers of soil moisture blocks is 1 percent.

The data taken at the Building Research Station, Watford (1962), with different types of limestone and brick indicated that 90 percent of the data were within ± 2 percent (dry).

Penner (1962) contends that their accuracy is good enough for irrigation purposes, but not good enough for most other applications.

Ashcroft and Taylor (1953) used plaster resistance blocks to measure moisture tension in 32 plots, with 8 different locations in each plot at depths of 6, 12, 18 and 24 in. The variability for locations and plots was much greater than the variability for the resistance blocks. This justified their use, even though the coefficients of variability were 6 to 12 percent. Because block data are much faster than sampling data, the number of measurements can be increased to gain a reduction in the location variability coefficient.

Relating accuracy and range, Bourget et al. (1958) concluded that gypsum is best above 0.3 atm tension. Nylon-gypsum is best below 0.3 atm tension. Fiberglass-gypsum with big electrodes is the best compromise from 0 to 15 atm tension.

2.2.8 Stability

Nylon, fiberglass, and resin treatment are added to gypsum to stabilize its use for long periods of time. This is especially true for the more sensitive blocks of Bouyoucos (1961). Several years' stability of calibration has been claimed in some cases.

2.2.9 Precision

Taylor (1955) has compared the precision of several methods. He found that two resistance measurements or two neutron measurements are equivalent to one gravimetric determination. To detect a mean difference between groups of less than 10 percent requires 8 resistance, 5 neutron, and 4 gravimetric measurements.

2.2.10 Speed of Measurement

From the foregoing work, 80 to 100 resistance measurements equal 20 neutron measurements and 1 gravimetric measurement.

3.0 CAPACITANCE TECHNIQUES

The measurement of capacitance is one of the simplest, most rapid, and least expensive methods for determining soil moisture. This is because the dielectric constant for most soils is about 2.6, whereas the dielectric constant for free water between 15° and 35°C, within the frequency range of 10^5 to 10^8 Hz, is about 80. Fundamentally the method involves the measurement of the dielectric constant of the material between two electrodes. Thus, variation in the dielectric constant of nonwater components is an inherent source of error. Other inherent errors are variations in particle size, packing density, and ionized salts.

A comprehensive treatment of the method appears in Wexler (1965). Roth (1966) compared the method with other methods suitable for application in industry. Monfore (1970) and Geary (1956) reviewed its application in highway engineering.

3.1 Sample Capacitance

Many relatively inexpensive instruments have been designed and used to measure the moisture of solids, based on the sample capacitance method. They are primarily low-frequency designs, and have been successful only when the variability of nonwater components was small, or when high accuracy was less important than speed and low cost.

An instrument operating in the vhf range offers much superior performance but is inherently larger, more complicated, and more expensive. One such instrument has been designed by Thomas (1966), with the sensing element in the form of a long, slender probe to be inserted in the sample. The sensitivity of the probe was improved by designing electrodes with a large fringing field. Other problems normally associated with capacitance methods (such as temperature dependence and ionized salt interference) contribute their effect through the imaginary component of the complex dielectric constant. By operating above 30 MHz these effects were significantly reduced. A Wayne-Kerr (B 801) vhf admittance bridge was used to measure the capacitance in the prototype instrument. Low or moderately priced meter-type instruments were not commercially available at that time, but Watson indicated that development work to meet this need was being conducted in his laboratory. Test results with this instrument were excellent using a wide variety of soils. A functional relation between the volume fraction (θ) of water in soils and the charge of capacitance (ΔC) when the probe was inserted was determined together with the standard error, $S(\theta_k)$, in a given measurement (θ_k). These functions are:

$$\theta = 0.245(\Delta C - 2.6) \qquad 0 < \theta < 0.1 \qquad \text{(A-15)}$$

$$S(\theta_k) = 0.07\,\theta_k \qquad \text{(A-16)}$$

$$\theta = 0.38 \log(\Delta C - 0.21) \qquad 0.045 < \theta < 0.45 \qquad \text{(A-17)}$$

$$S(\theta_k) < 0.005 \qquad \text{(A-18)}$$

Lower-frequency methods are less accurate. Leach and Neilson (1960) designed a one-terminal capacitance probe to measure moisture in bales of jute.

Gagne and Outwater (1961, 1965) designed a small portable surface prototype for measuring moisture in laminated plastic rocket cases. Permittivity and dissipation factors were measured with a balanced bridge powered by a stable crystal-controlled oscillator at 460 Hz. The dimensions were $5\frac{1}{4} \times 7 \times 3\frac{1}{2}$ in. A 9-v dry cell operated the instrument intermittently for more than 100 hr.

A surface unit consisting of two stainless steel plates 1 in. wide and 1 in. apart, operating at 1 KHz, was used by de Plater (1955). Penetration was determined to be about ½ in. Response time was about ½ min. A separate calibration was required for significantly different soils.

Hancox (1966) and Bell et al. (1963) also noted the need for different calibration curves and dissolved salt interference. Except for these effects, Bell et al. determined an accuracy of 0.25 percent below 6 percent total moisture (dry) in concrete.

Roth (1966) evaluated the method for measuring moisture in granular and sheet materials in industry. Instruments manufactured by Foxboro Co., James Hunter Machines Co., Forte Engineering Corp., and Moisture Register Company were identified. The minimum detectable limit of this group of instruments was 2 percent (dry). The range of measurement was 2 to 25 percent (dry). The precision was estimated to be ±2 percent. Calibration for each material was required. One of their desirable attributes was their almost instantaneous response.

3.2 Sensor Capacitance

It is somewhat difficult to distinguish sensor capacitance from the capacitance hygrometer methods or the electrical tensiometer methods. It is easy to see how the problems encountered in the sample capacitance method are compounded with the sensor technique. In addition to the problems of relating the capacitance of the sensor material to the moisture in the sensor, the interface between the sensor and the sample introduces variability into the measurement problem. This is related to quality of contact, physical characteristics of sensor and sample surfaces, temperature, and other moisture transport parameters. Some of the early work was done with plaster of paris blocks, similar to the resistance-type measurements. The accuracy appears to be sufficient in agriculture for determining moisture content of soil between the permanent wilting percentage and the moisture equivalent points (Anderson and Edlefsen, 1942). Sufficient accuracy, however, has not been demonstrated for highway engineering measurements of moisture content. As indicated in the discussion of hygrometric techniques and tensiometric methods, there may be other uses where quantities other than the free water content are important.

4.0 NUCLEAR METHODS

4.1 Neutron Scattering

Since the pioneering work of Belcher, Cuykendall, and Sack (1950) much work has been devoted to a better understanding of the interaction between neutrons and the soil-water-detector system. The technique depends on the ability of hydrogen in water to slow down fast neutrons. The form of the hydrogen cannot be distinguished by this method. The measurement interferences existing in presently used commercial gauges consist of sensitivity to: (1) the total sample density, (2) sample composition (particularly the presence of thermal neutron absorbers, such as chlorine and boron, and of other moderators, such as hydrogenous material), (3) surface roughness, and (4) sample homogeneity. These measurement interferences can be minimized by several methods. One such method consists of the use of the new He^3-filled proportional counters to detect epithermal neutrons that are relatively insensitive to the presence of thermal neutron absorbers. Another method consists of the use of a gamma-ray gauge for the measurement of density and the subsequent use of calibration curves for various densities. Use of a dual gauge where each detector has a different sensitivity to composition is another possible way to eliminate the effect of variable amounts of thermal neutron absorbers.

Recent theoretical studies on neutron gauge design have been conducted by Gemmell, McGregor, and Moss (1966), Ølgaard and Haahr (1967), Gardner and Roberts (1967), Nagy and Vertes (1968), McDougall, Dunn, and Gardner (1969), Lippold, Carnesale, and Gardner (1969), and Ølgaard (1969). These studies used multigroup theory and Monte Carlo methods to estimate the effects of the interferences discussed previously. The results in some cases were used to optimize the gauge design with respect to minimizing interference effects.

The results of a survey by the American Society of Civil Engineers (Roberts, Waananen, 1964) indicate that about 80 percent of the nuclear gauges used by highway departments are used to obtain data for construction. The survey also indicated that surface gauges rather than depth gauges were used (by a ratio of 11 to 4). Research studies in recent years have shown a similar distribution of emphasis.

Comparative evaluation studies have led to the conclusion that the previously mentioned interferences lead to errors in moisture measurement in soils (sometimes prohibitive errors), but the errors are commonly less than those for any other method (Stewart, Taylor, 1957; Johnson, 1962; Wofford, 1964; Ballard, Gardner, 1965; Waters, 1965; Gardner, Roberts, 1967; Hughes, Anday, 1967, 1970). Over-all, the nuclear method appears to be the best method for measurement of moisture in a 3- to 4-in. surface layer. The relatively high cost has been a deterrent to its use in some cases; but, unlike with some commercial instruments, the cost of nuclear gauges has decreased for several years to around $3,000 to $4,000. This is not a prohibitive cost for an instrument that is to be used on a routine daily basis.

Extensive material was reviewed in the literature. To present as much of it as possible, an abbreviated format is used that is similar to the abstracting notes that were taken during the literature review. The first entry is the author and the date (month/year) of publication. The numbers on the left represent the 35 performance criteria that formed the basis of the review and evaluation (Table A-1). The order is chronological.

Gardner, Kirkham (5/52)

Gives theory of methods. Tabulates scattering coefficients of a few elements.

16 Shows effective range for water of ~12 in.

Sharpe (3/53)

23

D(cm)	−8	3	5	10
S/N	3.5	5	8	6

Variation of source to base of counter dimensions indicates best discrimination at 5 cm. $B^{10}F_3$ counter length

is 15 cm. (No indication of effect of difference in CR.)

$$S/N = \frac{\text{Count rate (8\% water)}}{\text{Count rate (dry sand)}}$$

Stone, Kirkham, Read (10/55)

Operation of depth units limited to depths of less than 9 in.

13 Parts cost about $750.

15 45 lb.

25 Accurate within the standard deviation of gravimetric determinations.

Holmes (3/56)

Reviews theory. The mean distance between source and detector must be determined.

21 Absorber depression shown with NaCl solution.

25 Gets a linear calibration curve up to 30% (Vol) for loam, clay, and sand.

van Bavel, Underwood, Swanson (7/56)

Experimental surface and depth probes are evaluated.

22 Resolution is not better than 25 cm. The radius of influence is a function

$$R = 15\sqrt[3]{100/W_v} \quad \text{cm}$$

23 For linear response, detector should not extend more than 17 cm from source (can turn up or down).

25 0.6% (Vol) below 30% (Vol) moisture content for a 5-cm-diameter hole in a 60-cm layer.

Geary (56)

21 Hydrogenous material will give an effective moisture reading. Materials in the sample having a high absorption cross-section will reduce the moisture reading. The slow neutron count is related primarily to the weight of water per unit volume. To convert to percentage moisture by weight it is necessary to know the density of the material. Fifteen references are given.

Stewart, Taylor (2/57)

25 A 2-year field study is reported. Correlation coefficient at 15-cm depth was 0.76. Correlation coefficient at depths from 30 to 122 cm was 0.95 to 0.98. For depths between 30 and 125 cm, variation is 7 to 9% of predicted regression value. Results are better for measurements at same depth.
Plaster resistance blocks were studied at the same time and gave a coefficient of variability of 15% of mean value. This method is more accurate for measuring tension at more than 1 atm. Below 1 atm the tensiometer is most reliable, but errors are introduced in converting moisture.
Over-all, the nuclear method is most accurate.

Pawlin, Spinks (3/57)

Used neutron gauge on concrete. Experimented with steel reflector for surface gauge.

25 Concrete and sand-sugar calibration curve indicated error of less than 0.02 gm cm^{-3}.

Marias, de V. Smit (2/58)

On five different soils, three different calibration curves were determined to be significantly different.

25 The resulting error ranged as high as 2 pcf around 15 pcf moisture content.

Holmes, Turner (3/58)

A linear response curve is obtained up to 100% moisture. A polyethylene access tube is used. Resolving time correction was made.

16 $R = 20$ cm at 0.2 g/cc
$= 11$ cm at 1.0 g/cc } vertical

Davidson, Nielsen, Perrier (5/59)

1 Nuclear Chicago

20 5% decrease in count going from −20° to 30°C. Count went to zero between 32° and 46°C.

Holmes, Jenkinson (59)

21 100 ppm boron reduces slope by 10%.
Bound water of clays and chemical absorbers tend to cancel.

Stone, Shaw, Kirkham (6/60)

25 Coefficient of variation equals standard deviation divided by the mean ($C = s/m$).
For moisture content which yields about 17,000 counts, $C < 1\%$.

27 Where 7 gravimetric measuring sites are required, only 3 nuclear sites would be required.

Templeman (5/61)

13 Economical as measured by costs per determination. 1 system is equivalent to a 5-man crew.

16 0.5 ft^3.

25 Equal to or better than other techniques.
Minimizes human error.

26 0.4 pcf with 95% confidence in 1 min.

29 2-3 min.
5-to-1 time saving in fine-grain materials.
15-to-1 time saving in base-course materials.

van Bavel, Nielsen, Davidson (10/61)

End-source and center-source designs were compared and absorber solutions were studied as standards.

1 Nuclear Chicago, Troxler.

16 Effects noted from soil within cylindrical volume up to 3 ft. There is a noticeable difference between 3 and 4 ft.

23 Response or calibration curve turns up for short, and turns down for long counters. Troxler unit is approximately linear and intermediate in length. End-source system has two- to threefold greater efficiency than center source.

van Bavel (12/61)

16 Down to 15 cm.
23 400 cpm per 1% (Vol).
25 Large errors result from stratification or irregular surface.
27 1% (Vol).
29 5 min.

Burn (6/62)

Discusses in much detail the procedures and requirements for artificial standards for calibration purposes.

Smith, Weber, Campbell (10/62)

1 Nuclear Chicago, Hidrodensimeter.
20 Scaler inoperative below 32°F; operable up to 140°F.
25 Standard error = 0.8 pcf.
26 Spread of 2 to 4 pcf over 3 months, 1 pcf day to day.
27 Thin layer of sand less than 1/16 in. improves performance.
34 Much time spent in maintenance.

Johnson (62)

Energy loss is large when neutrons collide with low-atomic-weight atoms. The hydrogen in water is the most effective of all elements.

Summary: Only methods in common use are gravimetric, electrical resistance, tensiometric, radioactive. The ideal method has yet to be developed. The gravimetric method requires less experience than indirect methods and also requires more effort under some conditions. Indirect methods are most desirable if continuous or frequent measurements are necessary. Radioactive methods probably are the best in this case, but are more expensive.

16 The soil volume measured in this manner is bulb-shaped and has a radius of 6-15 in., depending on moisture content and soil density.
25 With calibration with type of soil to be tested, the accuracy is within 1-2% volume. Readings close to the surface are affected by the position of the probe. Near the air-surface interface the count is lower than at greater depth.

Time errors may be minimized by a standard count cycle of 2 min.

Access tubes must be kept free of excess moisture.
33 Calibration is required for the type and size of casing used for each installation.

The radiation hazard is minimized by proper handling. The method is time-consuming if calibration time is included. Equipment is heavy and delicate; equipment failures are likely. Re-calibration is required after repairs. Repairs may require services of an electronic specialist.

Merriam, Copeland (1/63)

27 Random instrument error in moisture percent by volume is given by

$$E = \sqrt{K(P_v/ST)}$$

in which

K = number of standard deviations;
P_v = slope of the calibration curve in cpm/P_v; and
T = sampling time.

He contends that earlier comparisons of end-vs-center locations did not take into consideration the much larger counting tubes in the end-source geometry.

B. G. Richards (8/63)

25 Australian soils with high clay content can result in errors of ±7% gravimetric moisture. An Australian-made instrument was judged inadequate for subgrade moisture investigations.

Lawless, Macgillivray, Nixon (9/63)

Determined gauge response at interfaces (air-soil; wet soil-dry soil).
1 Troxler, Nuclear Chicago.
23 End source probe is 1½ to 2 times as efficient in terms of cpm/mC. This advantage decreases at higher moisture contents.
25 1. Near soil surface, corrections must be applied to depth probes; this effect extends as deep as 18 in. for dry soil.
2. Gauges underestimate moisture in stratified profiles (or where moisture gradients occur).

Burn (3/64)

Leda clay in the valleys of Ottawa and St. Lawrence Rivers gives a depression of slow-neutron counting rate of 11%; scintillation nuclear detector was used. A calibration curve is included using different calibration materials.
21 4% reduction in count for 2% dry weight of potassium at 50% moisture content.
25 ±0.5 lb/cu ft.
26 Standard deviation of 3 sets of profiles for one day are 0.18 lb/cu ft.

Wofford (3/64)

16 30 times sand-cone test volume.
24 Normal operation between 2-30 pcf.
25 Fewer inherent errors than sand cone.
25 3 pcf at 4 in. in embankment material.
4 pcf at 6 in. in stone base courses.
33 A minimum of 10 tests required.

Preiss, Grant (8/64)

Paper studies optimum design for soil or concrete. Detector: (1) insensitive to fast neutrons, gamma rays, and slow neutrons below 0.8 EV.; (2) high sensitivity to epithermal neutrons. One solution is cadmium covering over normal detector to give 0.4 EV cut-off. 0.015 in. Cd reduces CR by almost factor of 10.

21 With 0.015-in. Cd shield, 8% ±4% reduction in count is seen per gm cm^{-3} NaCl. Absorbers are given in Table 2 in terms of NaCl equivalent. A systematic correction can be applied by using the slope of the calibration curve.

$$\Delta w = \frac{\Delta CR}{R^1} = \frac{\Delta CR}{dCR/dw} \text{(absorption)}$$

25 Time to achieve a certain accuracy due to counting statistics is

$$T = \frac{CR}{(R^1 \sigma_w)^2}$$

Roberts, Wyndham, Waananen (11/64)

This paper reports the results of a survey by the ASCE Task Force on use of neutron meters. Observational procedures are reviewed.

1 Nuclear Chicago, Test Lab. Corp., Troxler.
Hydrodensimeter made by Viatec Division of Tellurometer.
Kaiser-Tempe made by Kaiser Aircraft and Electronics.
Mobile logging unit by Dresser Research.
Most sources are 3-5 mc.
Use by highway departments (15 responses):

Data for construction	12
Data for agriculture	1
Data for other use	1
Equipment for depth moisture	4
Equipment for surface moisture	11

10 Effective application requires trained personnel.
11 Most reliable of available methods.
13 High initial cost.
17 Nondestructive nature is primary advantage of this method for studying moisture movement and profiles.
20 Ratemeters made require shading in field use.
21 Close fit of access tubes required in depth measurements, organic soils with organic salts.
25 High accuracy.
Interpretation of data is important in such effects as air-soil interface, response to gradients, effect of stones and stratification. Smooth surfaces and uniform meter contact are required.
27 Good reproducibility.
29 Observation time is reduced from weeks to days; gives on-the-spot guide to compaction.
30 Ideal for repeated measurements.
33 Of total users, 44 used manufacturer's calibration; 74 indicated field calibration.
Calibration required for special soils and different access tubes or surface conditions.
34 Radiation safety requirements.
Equipment and cable breakdown are problems resulting in significant down-time.

Ølgaard (1/65)

A 3-group diffusion-theory model is applied to neutron scattering in soil and results are compared with experiment. Concluded that theory was sufficient for present measurement capability.

16 Sphere of importance is consistent with theory used and model fits data better than van Bavel model. For typical soil:

$$R = \frac{100}{1.4 + 0.1\, M_v} \text{ cm}$$

M_v = moisture percent (Vol)

Composition and cross-section data that were used are included.

21 Interferences were indicated to be density, absorbers, and hydrogen content of dry soil.

Benz, Willis, Nielsen, Sandoval (6/65)

Advantages:

1. Greater accuracy if calibrated.
2. Response is convertible directly into volume moisture percent.
3. Successive samples can be taken from the same location.

Disadvantages: Dependence on soil composition, especially neutron absorbers.

1 Nuclear Chicago instrument is used in this study.
21 During field calibration as much as 20% reduction in rate was observed for high-salinity soils. In the laboratory NaCl in a water bath resulted in approximately 5% depression of counting rate for 5,000 ppm. Results were nonlinear.

Ballard, Gardner (65)

A review of simple models is given, together with the definition of important neutron transport parameters. A literature review of experimentally determined accuracies and interferences is given in Table 9. Type of system and operating range are included.
A simple regression model is proposed that is based on the absorption and scattering probabilities of the material to be measured.

$$P_s = \rho \sum_{i=1}^{n} \omega_i \sigma_{si} \xi_i / A_i$$

in which

P_s = probability of a thermalizing collision;
ρ = density of medium;
ω_i = weight fraction of the ith element in barns per atom;
σ_{si} = scattering cross-section of the ith element;
A_i = mass of the ith element; and
ξ_i = logarithm of the average energy decrement per collision of the ith element.

The probability of thermal neutron absorption is

$$P_a = \rho \sum_{i=1}^{n} \omega_1 \sigma_{ai} / A_i$$

in which

σ_{ai} = thermal neutron cross-section of the ith element.

The model proposed is

$$R - B = a(P_s)^b \exp(cP_s + dP_a)$$

in which

R = counting rate of detector;
B = background counting rate; and
a, b, c, d = constants determined by a regression analysis using calibration standards.

25 Best accuracy in the review was 1% (Vol); the worst results were obtained by Mintzer (6/60) with 35% error using a single calibration for 4 New York soils.

Cermak (65)

25 Water in sand, 0.8%.

Kasi, Koskinen (65)

Combined Monte Carlo and diffusion theory calculations are used.

21 Large effect of density is shown in Figure 4 (p. 6) (theory).

23 Increased sensitivity with iron reflector on back is shown in Figure 7 (p. 8).

Waters (65)

Neutron method appears to be best method available for surface measurements of concrete and masonry structures.

1 Hydrodensimeter made by Viatec Division of Tellurometer.

25 A wide variation (as much as 0.1 g cm^{-3}) for different materials was observed. Differences correlated with absorbers present in significant quantities such as 0.02% boron in plywood and 3 to 5 ppm gadolinium and other rare earths in some cements.

Roth (8/66)

Description: Hydrogen, because of its close mass relationship to neutrons, is a very efficient moderator. The neutron technique generally operates with a fast-neutron source and a slow- or thermal-neutron detector for those neutrons that the sample has slowed. It is fundamentally a volumetric measure of moisture content. A simultaneous measure of density by means of gamma-ray scattering is sometimes used to convert volumetric moisture to a weight basis. A ratio signal from the gamma-ray detector and the thermo-neutron detector is functionally related to percentage of water on a weight basis.

1 Nuclear Chicago, Kay-Ray Division of Kay Electric Co., Numec Instrument and Controls Corp.

13 $5,000-$50,000 (1966).

17 Nondisturbing.

18 Incapable.

19 Total moisture.

21 All materials interact with neutrons, but normally in a minor way compared to hydrogen or moisture. Variation of hydrogenous material other than water would cause serious error.

24 2-80%.

25 Depends on the material being measured. It is about 0.2% if the material has zero or a fixed hydrogen content.

27 Can be 0.2%.

29 Instantaneous.

30 Continuous.

35 Most solids. For a conveyor-belt application, the sample must be 16 in. wide and at least 2 in. thick. See Figure 10 (p. 88).

Beskin (66)

23 Boron detector with frontal placement of source has the highest sensitivity up to 30% moisture (Vol). Lateral placement of source is the only one of several configurations that had a linear correlation curve.

Slow neutron density (at r) is given by:

$$N(r) = \frac{Q\tau}{8L_f^3}\exp\left(-\frac{r}{L_f}\right)$$

in which

Q = fast-neutron source length;
r = distance from source;
τ = lifetime; and
L_f = moderation path length.

$$L_f = \frac{1}{n\sigma_s}\sqrt{\frac{j\alpha_s}{3}}$$

in which

n = atomic density;
α_s = anisotropic scattering coefficient;
σ_s = neutron-scattering cross-section per atom; and
j = amount of collisions resulting in thermalization ($E = 0.025$).

Gemmell, McGregor, Moss (66)

Presents a multigroup theory.

21 Density appears to have a positive correlation with response.

Boron decreased response by 25%/100 ppm at 0.2 gm cm^{-3}, ($\rho = 0.99$ g cm^{-3})

van Bavel, Stirk (3/67)

Soil water inventories were made at one site to a depth of 170 cm. A gamma-free Am^{241}-Be neutron source was used in a Troxler N104 probe.

25 0.6% of total moisture.

27 0.2% of total moisture.

33 Separate calibration within 15 cm of surface.

Ølgaard, Haahr (5/67)

Applies 3-group diffusion to gauge design (sensitivity) and influence of density and composition.

33 Some normalization constants are determined for using a theoretical calibration model.

Stone, Barefoot, Garton (8/67)

21 Interference may result from ignition noise of reciprocating engines. Coupling is electromagnetic, apparently through cable connection. Count rate almost doubled near a fast-idling engine. Effect decreases with distance. Coiling cable around distributor bead gave 16,000 spurious counts per minute.

Gardner, Roberts (67)

A calibration model based on 2-group diffusion theory for a slightly absorbing spherical cavity in a homogeneous medium is

$$R = \frac{K_1}{\Sigma_a(L_1 + K_2)(L_2 + K_4)(L_1 + L_2)} + K_3$$

If the cavity is nonabsorbing, $K_2 = K_4$. R is gauge response; Σ_a is the microscopic thermal absorption cross-section; L_1 is the fast diffusion length; L_2 is the thermal diffusion length; and K_1, K_2, and K_3 are gauge constants. Details of calculating soil moisture parameters and application of the model are given [Hughes, Anday, 1967].

16 3-5-in. depth for $W < 10$ pcf; 1-in. depth for $W > 40$ pcf.

25 Using 4 calibration standards, one calibration curve was obtained by the model and another was obtained by fitting a straight line. The fit gave a standard error of 1.144 pcf for the straight line and 0.352 pcf for the model (3 constants vs 2). Results on the five test-site soils at the Virginia Conference, however, showed little difference between the two.

Hughes, Anday (67)

21 High iron content causes wider variability between gauges.

25 For 30 gauges, standard deviation for calibration curves ranged from 0.1 to 4.4 pcf. The average standard deviation was 1.2 pcf. Tests on five sections resulted in standard deviations between gauges of 1.5, 1.8, 2.9 (19.8% Fe), 2.2, 2.0 pcf.

33 Different manufacturers using different standards for calibration have a much wider variation than when they use the same standards.

Nagy, Vertes (11/68)

Standard deviation may be written

$$\sigma_M/M = 1/(S\sqrt{Pq})$$

in which

S = relative sensitivity of probe;
P = efficiency of probe; and
q = source strength.

21 Using 3-group theory, corrections for variation in dry bulk density are calculated. Significant corrections can be made with curves and equations that are presented.

LeFevre, Manke (68)

1 Troxler attempted to develop a calibration procedure using dual-purpose standards for both moisture and density. He used 55-gal drums cut to a height of 24 in. and an average volume of 5 cu ft.

23 Linear between 5 and 30 pcf.

25 Significantly different slope from manufacturer's calibration curve developed from cadmium chloride standards.

33 Calibrate for type of soil being tested.

McDougall, Dunn, Gardner (4/69)

25 Surface effect error (E):

ρ = normal indicated density;
ρ' = indicated density in a position 1/16 in. above sample; and
$E = \rho - \rho'$.

Statistical counting rate error:

$$\sigma_s(\rho) = \frac{\partial \rho}{\partial R}\,\sigma(R)$$

Using more than one count for composition correction gives

$$\sigma_s(\rho) = \sqrt{\left(\frac{\partial \rho}{\partial R_1}\right)^2 \sigma^2(R_1) + \left(\frac{\partial \rho}{\partial R_2}\right)^2 \sigma^2(R_2)}$$

Composition and other errors:
Grouping all remaining errors that are determined experimentally results in

$$\sigma_c(\rho) = \sqrt{\frac{\Sigma(\rho_1 - \hat{\rho}_1)^2}{n}}$$

33 A lengthy calibration model is given based on Monte Carlo calculations. A series of curves are drawn with density and equivalent iron content. Average standard deviation for 3 gauges and 15 test sites was 1.72 pcf. Results indicate a large effect of both sample density and composition. Commercial gauges have good reproducibility. A multiple-curve laboratory calibration shows promise of increasing accuracy.

Ehlers, Reese, Anagnos (6/69)

1 Troxler Electronic Labs, Inc.

9 Drill holes are used for depth probe (procedures are outlined by Heiliger and Haliburton).

13 Relatively high cost.

16 0.5 cu ft average.

19 Total water (Belcher et al., 1952) indicate response to bound water which is not removed at 110°C.

20 No temperature effects between 40° and 110°F.

21 Influence of rocks: fluctuations were observed, but they remained within 1.25 pcf (1% dry weight).

23 Most sensitive with source located near midpoint of detector.

25 2% (dry weight) or less variation with gravimetric.

26 Coefficient of variation of 1%.

27 Lower at high moisture.
Air gap variation results in decrease of 0.4% per 1/16 in. of gap for 13-15% moisture range.
33 Uncertainty in generality of calibration when applied to diverse materials.
Requires understanding of error that may occur and that may be tolerated.

Cameron (69)

A good review is given. Need is for simple, accurate model besides group theory.
Advantages: rapid, precise, nondestructive, large-volume average, lightweight, portable, reliable, simple, inexperienced operators.
Detector using ^{6}Li-loaded glass scintillators may be used for moisture and density measurements.
It is common to have an accidental correlation between bound water content and absorbing elements, so that the two cancel each other (as was observed in work of Anday and Hughes).
Precision: Electronic ±0.1%.
Accuracy: 1% (Vol) or 0.01 g cm^{-3}.

Hönig, Pospišilova, Klablena, Papež (69) (unpublished)

Probably the most comprehensive collaborative testing program of international commercial gauges. Important characteristics that were considered are:

1. Calibration relationships.
2. Temperature stability.
3. Chemical influences of measured medium.
4. Optimum measuring time.
5. Resolution of subsurface probes.
6. Depth effect of surface probes.
7. Short- and long-time stability.
8. Simplicity of operation.
9. Measurement of time.
10. Design of scaler.
11. Design of probes.
12. Weight of complete instruments.
13. Suitability of instruments for field work (including auxiliaries for driving in access tubes and for surface leveling).
14. Charging of batteries.
15. Servicing arrangements.
16. Safety of work.

Results of this program will be published soon.

Lippold, Carnesale, Gardner (69)

A Monte Carlo simulation was used for gauge calibration and design optimization.
Predicted effects of density and thermal neutron absorbers (in terms of iron equivalent) are shown in Figure 3.

Ølgaard (69)

Presents 3-group diffusion theory and experimental results for depth probe. Preliminary studies using Monte Carlo technique for surface probes indicate the need for simpler model. Theoretical models can be used in gauge design (sensitivity, etc.).
21 Density effects are shown in Figure 4 (p. 70).
33 Discrepancies between calibration curves for low moisture can result from too-small calibration samples.

Monfore (1/70)

16 Radius from 4-18 in.
Thickness from 4-24 in.
19 Measures total water content.
21 Scattering and absorption of other atomic nuclei are not negligible. Cadmium shield reduces composition dependence.
25 Calibration of meters has received considerable attention.

Anday, Hughes (6/70)

25 1 calibration curve: 3 pcf standard error.
2 calibration curves:

	CLASS A	CLASS B
Optimum moisture content (%)	>18	<18
Percent passing No. 200 screen	>48	<48
Percent sand	<30	>30
Standard error (pcf)	1.5	0.84
Correlation coefficient	0.907	0.971

No trend for addition of Fe_2O_3 was noted.

In Class B ignition loss at 1,000°C was relatively low; content of neutron absorbers also was low (i.e. Fe_2O_3, TiO_2). (Density was not indicated.)

4.2 Gamma-Ray Interaction

Two gamma-ray-interaction methods have been proposed for highly specialized applications. The first is applicable where it is desired to measure moisture within a ½-in. layer of soil (Smith, Taylor, Smith, 1967). This technique assumes that the specific gravity of soil remains constant as moisture gain or loss changes the wet density. The wet density is determined by gamma transmission technique.

$$W_v = \rho(\text{def}) - \rho(\text{dry})\frac{\rho(\text{soil})}{\rho(\text{dry})}(100) \qquad \text{(A-19)}$$

Field capacity moisture deficit, ρ(def), was defined as a relative measurement because both ρ(soil) and ρ(dry) are undetermined. The density measurement at or near field capacity is designated as ρ_1. Subsequent measurements of ρ_2 give the moisture deficit with respect to field capacity moisture.

$$W_{v1} - W_{v2} = (\rho_1 - \rho_2)\frac{(100)}{\rho_w} \qquad \text{(A-20)}$$

in which

W_{v1} = volume moisture percent at field capacity;
W_{v2} = volume moisture percent at subsequent time; and
ρ_w = density of water.

The standard error was determined to be 2.35 percent (Vol).

A second method was studied by Babinets and Zvol'skii (1966). They proposed a logarithmic relation as the first approximation of the gamma radiation induced by neutron-activated atoms and detected near the source of fast neutrons.

$$R = A + B \ln(w_H) \qquad \text{(A-21)}$$

A and B are experimentally determined constants; w_H is the total hydrogen content. The mean deviation of their experimental results from this model was 0.02 g cm^{-3}.

Neither of the foregoing methods appears to be a substitute for more conventional methods, but they may be useful for special applications. The second method is a good candidate for incorporation into the operation of conventional moisture gauges to provide a dual-gauge capability for discrimination of neutron absorbers.

5.0 GRAVIMETRIC TECHNIQUES

The gravimetric technique is the most commonly used technique for measuring moisture in soil. Some general applications of this technique appear in Wexler (1965), Monfore (1970), and Geary (1956).

The gravimetric method of determining moisture content involves five steps: (1) collecting a soil sample, (2) weighing it, (3) removing the moisture, (4) weighing the dry sample or the removed water, and (5) calculating the moisture content. The gravimetric method is the most direct way of measuring soil moisture; therefore, it is required for calibrating equipment used in other moisture measuring techniques. To collect the best soil samples, the soil should be homogenous; just moist enough to permit easy cutting by the sampling equipment; and free from roots, stones, organic matter, and other unwanted particulates. In practice, these conditions are seldom met. The technique and equipment used for sample collection should be such that the samples do not lose or gain moisture or otherwise become altered or contaminated during sampling in the first weighing of the sample material. In collecting samples, one must proceed with caution when sampling through a wet layer into a drier layer. The sampling equipment should be kept as dry as possible to prevent water from running down the hole into the drier material. In sampling some very wet soils, some of the water may be squeezed out during the compaction and probably will indicate less than the correct value of moisture content. In sampling hard, dry, fine-textured sediments, it is difficult to drive the core barrels or sampling tubes into the sample region. Dry, coarse-textured sediments may slide out of the sampling tube or the core barrel as it is withdrawn from the sample hole. The gravimetric method requires considerable time and effort to collect samples, especially from depths greater than a few feet, and to oven dry and weigh the many samples required for most projects. For many projects the sampling procedure (making numerous holes) alters the area of the experiment. Under some conditions, the holes may have to be refilled and packed. It is desirable to discuss some background information on the kinds of water usually found in materials. Water in materials may be fixed to the solid phase in various degrees ranging from chemically bound to essentially unbound or free. The removal of chemically bound water generally drastically changes the properties of the solid material. The chemically bound water often is described as the nonevaporable water. It should be noted that aggregates, concrete, and soils may contain an appreciable amount of nonevaporable water.

The evaporable water customarily is defined as that which is removed under a vacuum of 0.5 microns of mercury at 23°C. This pressure and temperature correspond approximately to the water lost during oven drying at 105°C. The evaporable water in soils frequently is classified as (1) hydroscopic, and (2) gravitational. The hydroscopic water is under the influence of surface forces; the gravitational water is that part that will drain from the soil under the force of gravity.

5.1 Thermal Extraction

5.1.1 Oven Drying

A practical and easily controlled technique for water removal is oven drying. Heating raises the vapor pressure of the free water within the porous material. A reduced vapor pressure in the environment serves as a force to move this vapor out of the solid. This reduced pressure is obtained either by evacuation, or dessication, or the flowing of dry air over the samples. As the difference between the vapor pressure in the porous material and that of the environment gets small, so does the drying rate. At this time, a greater portion of the remaining moisture is tightly bound.

Although oven drying is the widely accepted standard for measurement of moisture, there are sources of error that must be considered. Interferences are present in the form of absorbed volatiles, sample decomposition, water of crystallization, and adsorbed gas into or out of the sample. An impervious crust may trap moisture within the solid. Moisture or gases may be adsorbed between the time the sample is dried and the time it is weighed.

Dryness of the sample when drying stops depends on ambient vapor pressure. Turbulent drying air also may remove solids. At the same time water may not be removed that is partially bound. Under these conditions absolute equilibrium is never reached. Thus, a standard drying time—say, 1 hr—has to be specified.

Several multisample units are available to speed the measurement process and provide economy where large numbers of samples are to be measured. Two of these are described by Roth (1966). The units accepted a sample of approximately 10 g. The range of measurement was between 1 and 100 percent. The accuracy and precision are quoted at 0.2 percent. Between 5 min and 1 hr is required for each drying cycle. The units are applicable to any oven-driable material, preferably with a maximum of surface area.

The long times required for oven drying have led to searches for faster methods. Microwave ovens have been applied to this problem; these were found to be very fast and agreed well with standard oven drying procedures at

105 to 110°C (Ryley, 1969). GHz waves are absorbed by polar molecules. After water removal, some soils were found to cool; others were found to heat. In the foregoing work, a laboratory prototype oven was used. Its cost was about five times that of the conventional oven. Ryley found that primarily unbound water is removed. At higher operating levels the removal of some bound water is evident by the greater loss in weight of high-clay-content soils when compared to conventional oven drying. An over-all agreement of 0.4 percent was found when this method was compared with standard oven drying methods. Sample time is between 10 and 15 min.

Microwave ovens should be especially good for cement-stabilized soils. One precaution is noted: fuel ash materials may ignite. A unique control feature was added by Algee, Callaghan, Creelman (1969). The load voltage-standing wave ratio was found to be a good indicator of the time if the sample is "just dry." A change in the reflected signal occurs as moisture is removed. Samples of 200 to 300 g were used and an accuracy of 0.5 percent was obtained, as compared to a conventional oven drying method at 110 ±5°C. Some samples were dried for 3 min at 2 kW (2.45 GHz). Others were dried for 30 min at 200 w (2.987 GHz).

It is often desired to measure the water of hydration in hardened concrete. Wesche and Schlotmann (1962) found that loss on ignition was a more accurate method than determination of specific volume or specific density. Care was taken to deduct the CO_2 content.

5.1.2 Freeze Drying

Freeze drying sometimes is referred to as lyophilization. This method offers no risk to decomposition of heat-sensitive materials. Freezing also has the effect of loosening the structure of organic materials so that additional moisture that remains after freeze drying is removed by oven drying. A combination of the two methods would result in greater removal of moisture.

A vacuum with a dry-ice cold trap has been compared with a vacuum dessicator drying method to measure non-evaporable water in hardened portland cement paste (Copeland, Hayes, 1953). The dry-ice method was found to be simpler and resulted in a precision of 0.0008 g/g of cement. The magnesium perchlorate-dihydrate procedure resulted in a precision of 0.0012 g/g of cement. The vacuum dessicator gives a value of 1.084 times the moisture value obtained by the dry-ice method.

5.1.3 Distillation

The Brown-Duvel moisture tester uses this technique (Geary, 1956). The sample is immersed in a liquid that is immiscible with water. Liquids are typically benzene, toluene, and xylene. The mixture is then raised to the boiling point of the liquid, which is normally below that of water. The refluxing column then feeds a calibrated bulb, where the liquid and water separate. The volume of the bulb is calibrated accordingly as the liquid is lighter or heavier than water. Many of the references that Geary uses apply primarily to organic solids.

5.1.4 Heating in Oil

Geary (1956) also discusses the method of heating the sample in nonvolatile oil. This is an empirical technique that may be used for specialty applications. The few references given by Geary are not especially important in the measurement of highway components.

5.1.5 Dessicant Weight Gain

Dessicant weight gain can be used with other drying techniques (Geary, 1956). Inert gas or dry air is drawn over the samples and passed through a dessicant. The weight gain of the dessicant is assumed to be water. This technique often is applied to coal. A dessicant may be chosen that passes volatiles other than water where normal drying techniques result in ambiguity. Thus, this is a good supporting technique.

5.1.6 Alcohol Burning

One of the more rapid gravimetric techniques is alcohol burning. This consists of mixing alcohol with the sample and igniting the mixture. Most of the water is removed during the process (Geary, 1956; Bouyoucos, 1937). The sample is reweighed after the water-removal process. Antrim et al. (1970) found that three successive burnings were required for high moisture content. A correlation coefficient of 0.999 with oven-dry results was obtained with a standard error of 0.42 percent (dry).

5.2 Chemical Extraction

The three principal categories of chemical extraction are alcohol, calcium carbide (hydride), and the Karl Fischer reagent. In addition to these methods, the refractive index of dioxan has been measured and related to water in solution (Geary, 1956). Other measurements on extraction liquids related to water content are depression of freezing point, elevation of boiling point, separation temperature of ethyl alcohol and petroleum, electrical conductivity, and capacitance.

5.2.1 Alcohol

Alcohol extraction is one of the simplest of the liquid-extraction methods (Bouyoucos, 1931). The water content is determined by the density of the alcohol-water mixture after extraction from the sample. A hydrometer can be used to measure the liquid density.

A slightly different approach was used by Hancock and Hudgins (1954). A soil sample was mixed with a 70 percent ethyl alcohol-30 percent acetone solution and sodium chloride. The conductivity of the solution was related to the moisture extracted from the soil. The accuracy was determined to be ½ percent dry-weight basis.

5.2.2 Calcium Carbide (Hydride)

The calcium carbide method is used widely. The moisture content usually is related to two measurements: (1) the decrease in the weight of the sample carbide mixture after evolution of acetylene, and (2) the rise in pressure inside

an enclosure containing the mixture that determines the volume of gas produced. The latter is commonly known as the speedy moisture method and is produced by Ashworth & Co. (Geary, 1956) and Soiltest, Inc. Extensive comparative evaluations of this method with oven-drying, nuclear, and alcohol-burning methods have been made (Antrim et al., 1970; Blystone, 1961). It is generally considered to be relatively accurate, inexpensive, and rapid.

The calcium carbide (CaC_2) reaction with moisture proceeds as follows: $CaC_2 + 2H_2O \rightarrow Ca(OH)_2 + C_2H_2$. The calcium carbide gas pressure method measures the moisture content indirectly by gauging the pressure of C_2H_2 gas generated by the reaction in a closed vessel. The tester used in the work of Blystone is a hollow aluminum vessel, with a pressure gauge on one end and a cap with a clamping arrangement on the other. A 26-g sample size is used. Two steel balls, 1¼ in. in diameter, were effective in pulverizing soil samples. Attempts to correlate the temperature rise of the sample and holder with moisture were unsuccessful. This study led to a recommendation of a six-step procedure for this method of measurement:

1. Place three measures (approx. 22 g) of calcium carbide and two 1¼-in. steel balls in the large chamber of the moisture tester.
2. Using the tared scale, weigh a 26-g sample of soil. A 13-g sample should be used if the moisture content is much greater than 20 percent. Moisture percentage is given on a dry-weight basis.
3. Place the soil sample in the cap. With the pressure vessel in a horizontal position, insert the cap in it, and tighten the clamp to seal the cap to the unit.
4. Raise the moisture tester to a vertical position so that the soil in the cap falls into the pressure vessel.
5. Holding the moisture tester horizontally, manually rotate the device for 10 sec so that the steel balls are put into orbit around the inside circumference. Rest for 20 sec. Repeat the shake/rest cycle for a total of 3 min. Do not allow the steel balls to fall against either the cap or the orifice leading to the dial because this might cause damage.
6. Read the pressure gauge of the moisture tester and determine the moisture content of the soil on a dry-weight basis from the calibration curve.

As compared with the oven-dried methods, with this method the average moisture content difference is approximately 0.5 percent dry weight (Blystone, 1961). The measurement range is from 0 to 45 percent dry weight.

Applications are considered to be in-place density tests, auger and split-spoon samples, sands used in concrete mixtures, and proper moisture content for earth work. Field laboratory applications may include hygroscopic moisture, development of compaction curves, and low-value liquid and plastic limits (Blystone, 1961).

The hydride method is similar to the carbide method. The reaction is: $CaH_2 + 2H_2O \rightarrow Ca(OH)_2 + 2H_2$.

The reaction is exothermic, with a standard heat of reaction ($\Delta H°$) equal to -122.38 Kcal mole^{-1}. The standard free energy of the reaction ($\Delta F°$) is a highly negative -121.24 Kcal mole^{-1}. When the reaction is complete the cell is cooled to 20°C. The pressure in the cell is a measure of the H_2, and, thus, the moisture of the sample. The average error for this method was found to be ±0.1 percent (wet) for a 0.5-g sample, and ±0.05 percent (wet) for a 1.0-g sample (Güven, Kerr, 1965). This method measures free water that is held by physical adsorption similar to condensation forces. The heat for each is 5 to 10 Kcal mole^{-1}. The rate of adsorption is very rapid. Chemisorption holds water tightly, similar to chemical bonds. Hydration involves a discontinuous adsorption of water. The energy may be more or less than that of physical adsorption.

Because heating to 105 to 110°C removes some chemisorbed water and water of hydration, the results of this method may vary significantly from the weight loss during oven drying. This difference is typically 5 to 15 percent (wet).

5.2.3 Karl Fischer Reagent

The Karl Fischer reagent is used widely with the titration method for measuring moisture in solids. It is prepared by reacting SO_2 with iodine dissolved in pyridine and methyl alcohol. A more stable reagent is obtained by substituting ethylene glycol for methyl alcohol. The titration is conducted on the sample in methanol or other suitable solvents. Both direct and back titration are effective. The endpoint is commonly determined electronically. When free iodine in excess reagent is present, the sample solution will conduct electricity. A sharp change in conductivity is observed at the equivalence point. Two commercial instruments using this principle are described by Roth (1966). These are the Auto-Aquatrator (manufactured by E. H. Sargent & Co.) and the Aquameter (manufactured by Beckman Instruments, Inc.).

This method measures the total bound and free water that will react with the reagent. It is very specific relative to other techniques, because many common substances are inert to the Fischer reagent. Interference may come from (1) oxidants such as chromates, dichromates, cupric and ferric salts, higher oxides, and peroxides; (2) reductants such as sulfates, sulfides, and stannous salts; and (3) borates, basic oxides, and salts of weak oxyacids that might react with the reagent to form water.

The technique is extremely sensitive; the lower limit of detection is 0.01 percent. Samples with water content up to pure water can be measured. The precision of measurement is 0.025 ml of titrant.

The response time is normally a few minutes. However, the total measurement time may be long if interfering materials are present, or if moisture is difficult to extract. Thus, finely divided solids require a shorter measurement time than coarser materials.

5.3 Mechanical Extraction

Samples containing high water content can be mechanically pressed to remove water (Geary, 1956). The weight before and after compression can be used to calculate the water content, or the weight of the water removed can be measured. This technique has not been applied to highway components, and there are no obvious applications to which it is suited.

5.4 Immersion (Pycnometer)

The immersion method is based on classical procedures for determining specific gravity. It is in fairly common use in the measurement of loose materials such as sand and aggregates (Geary, 1956). Some of the early work in this area is attributed to Tortensson and Eriksson (1936). Garton and Crow (1954) used a 150-cc laboratory bottle equipped with an aluminum stopper with a hole in the center. Soil was added to the bottle and it was weighed. Water was added to completely fill the bottle and it was weighed again. Moisture percentage was determined by:

$$P_w = \frac{W_p - W_m}{W_p(W_m - 1)} \times 100 \qquad \text{(A-22)}$$

in which

P_w = moisture percent of dry weight;
W_p = specific weight of soil particles; and
W_m = specific weight of moist soil.

A convenient curve can be plotted for 0 to 40 percent moisture for W_m of 1.80 to 2.50 and 2.52 to 2.68.

The mean difference between this technique and the oven-drying method was determined to be 0.152 percent. Obviously, the moisture determined in this manner is free water in the sand. This is especially important when one is using wet sand in proportioning concrete.

Results obtained by Wilde and Spyridakis (1962) during field tests were not nearly as accurate as the foregoing. They attributed an error of 2 percent to the normal variations of specific gravity of soil particles.

6.0 RADIATION TECHNIQUES

With modern refinements in electronic techniques there has been a concurrent effort to apply mechanical and electromagnetic waves to the measurement of moisture because of their compatibility with electronic systems. Nuclear methods could be grouped under this category as nuclear radiation.

6.1 Mechanical

6.1.1 Ultrasonics

Ultrasonic energy propagation was studied experimentally at frequencies of 15.9, 19.8, 114, and 142 MHz by Mack and Brach (1966). Samples were sealed in 130-cm^3 containers and placed between matched transmitter and receiver transducers. The 114-MHz signal provided the best discrimination between water and the solid matrix. A laboratory-type oscillator was used in the studies, manufactured by Marconi Instruments Ltd. The Model TF 1246 was used in the range of 40 KHz to 50 MHz; the Model TF 1247 was used in the range of 20 to 300 MHz.

A correlation coefficient of 0.95 was obtained for a wide variety of materials at 114 MHz. This was improved to 0.97 by including an effect for density variation. The moisture content ranged from 0 to 56 g water/130 cm^3. At lower frequencies the range is limited from 0 to 12 g water/130 cm^3. An instrument sensitivity was observed of 1.20 μamp/g water.

Studies of the effect of temperature indicated little variance from 0° to 30°C. However, a marked difference occurred on freezing.

Interference from salt ions and organic material was found at 15.9, 19.8, and 59.0 MHz. Very little interference was found at 114 and 142 MHz.

6.2 Electromagnetic

6.2.1 Radiowaves

By definition, radiowaves include microwaves; however, an attempt is made here to separate the two in terms of frequency. Microwaves are considered to start between 300 and 1,000 MHz and to extend up to 300 GHz. The radiowave section considers frequencies below microwave frequencies. Although there is some overlap, the purpose of making this distinction is the difference in equipment used at the different frequencies. Similarities also may be seen in the capacitance and resistance parameters.

RF energy is highly absorbed by moisture. The method is not very specific, because many polar compounds also absorb this energy. Thus, the primary disadvantage of this technique is that it requires a calibration curve for each material on which it is to be used. The calibration slope is very steep, however, and with adequate control of other variables the precision and accuracy can be achieved in the low fractions of a percent. Roth (1966) has noted the availability of instruments by Boonton Polytechnic Co. and Moisture Register Co. The operating range is between 0.01 and 60 percent (dry). Specific materials may have a much more restricted range (e.g., concrete blocks have a range of 10 to 50 percent; molding sand has a range of 2 to 15 percent). Probe-type electrodes normally are designed for the particular material to be measured.

Using an airplane overflight technique, Geleynse and Barringer (1965) accomplished remote sensing of conductive bodies located in the ground by transmitting a train of high-powered unipolar electromagnetic pulses of half-sine-wave shape and receiving the reflected signal. The pulses have a base width of 1.5×10^{-3} sec and are spaced 2×10^{-3} sec apart (3.5×10^{-3} sec between peaks). Pulse decay in the receiver depends on conductivity of the reflecting material. Close inspection of the return pulse is required for interpretation, but it was found that ore bodies could be detected at depths of 50 to 100 ft. Increased conductivity of wet soils also has a characteristic effect on the reflected pulse.

The transmitter is several hundred ampere-turns around a small airplane. The receiver is a coil towed in a bird behind the plane at a height of about 100 ft. The total weight of the equipment is 600 lb.

A very high frequency sweeping technique with a log spiral antenna also was used to study layered structures. An undetermined calibration shift was related to the sample homogeneity. Layered structures or nonhomogeneous water content were difficult to analyze. This could be resolved only by elevating the transmitter and receiver such as with an airplane. A spatial resolution was obtained (using delay line correlation techniques) of 2 in. in a total layer spacing of 20 in.

6.2.2 *Microwaves*

The moisture content of nonmetallic porous solids is related to the absorption of microwave energy. For many materials a linear relationship exists between moisture density and the logarithm of microwave attenuation (sometimes expressed in decibels). Microwaves between 3- and 30-cm wavelengths are most suitable from an economic standpoint. However, there are certain improvements in specificity below 3 cm, particularly with respect to soluble salts.

Busker (1968) gives a capsule summary of microwave absorption at different frequency ranges. He considers the 22-GHz absorption peak a good candidate for moisture measurement. It is noted that the attenuation of bound water is low, whereas the attenuation of free water is high. The temperature dependence goes from a negative temperature coefficient at high moisture content to a positive coefficient at low moisture content. Work at the Building Research Station (1962) considered moisture measurement in walls at 7.5- and 12.0-cm wavelengths. The mathematics to describe the attenuation of the sample is:

$$I = I_0 \exp\left(-2\pi\eta \frac{l}{\lambda}\right) \qquad \text{(A-23)}$$

in which

I = transmitted intensity;
I_0 = initial intensity;
λ = wavelength;
l = thickness of material,

$$\eta = \frac{1}{2n} \frac{(\epsilon_s - \epsilon_\infty)\,\lambda_s/\lambda}{1 + (\lambda_s/\lambda)^2} + \frac{2\sigma\lambda}{c} \qquad \text{(A-24)}$$

ϵ_s = dielectric constant at 0 frequency;
ϵ_∞ = dielectric constant at ∞ frequency;
n = refractive index;
σ = conductivity;
λ_s = characteristic relaxation wavelength; and
c = velocity of light.

Dissolved salts contribute to the conductivity term. This term becomes negligible in sea water below 3 cm. The need for calibration curves for different material is indicated by the following absorption coefficients:

Clay bricks	8 db/g water/sq cm
Sand/lime bricks	5 db/g water/sq cm
Concrete	9 db/g water/sq cm

Watson (1965) noted that the method is not nearly as accurate as oven drying, but it is much faster.

Roth (1966) gave the commercial manufacturers of this type of equipment as Charter Industrial Engineering Co. (2.45 and 10.68 KHz) and Microwave Instruments (2.8 and 9.4 KHz). The range of these instruments is from 1 to 90 percent (dry). The response is sensitive to other polar materials, to thickness and bulk density variation, and to small changes in temperature.

Lundien (1966) concluded that estimates of moisture content of deep homogeneous samples could be made. Pulsed radar sensors were tested in a hangar-like enclosure at frequencies from 0.297 to 31.543 GHz. The equipment used was very bulky.

Microwave radiometers also require bulky equipment. Johnson and Gravitte (1966) mounted a microwave radiometer on a pneumatic-tired cart. Penetration was 2 to 4 in. in soil with low moisture content. For high moisture content, the signal was reflected essentially at the surface.

Using a 16-ft mobile laboratory on a 1½-ton flatbed truck, Edgerton (1968) found that microwave radiometers were dependent on sensor frequency, polarization, soil moisture particle size, and surface roughness. The last two influence the general shape and slope of the curves of radiometric vs antenna viewing angle.

Moisture caused response curves to shift along the temperature axis. Microwave temperature difference between tidal mud and dry soil is about 120°K. A qualitative relationship was found between brightness temperature and soil bearing strength.

6.2.3 *Nuclear Magnetic Resonance*

When a sample is placed in a fixed magnetic field and a varying magnetic field, nuclear magnetic resonance results in an increased absorption of energy at specified frequencies of the varying magnetic field. The varying nuclear magnetization is converted into a voltage by using either the single-coil absorption technique or the quadrature-coil induction technique. The resonant frequency of the absorption or induction is characteristic of a particular nucleus. The highest signal-to-noise ratio for measuring water molecules is obtained by looking at the hydrogen nuclei spectra. Obviously, this technique is difficult to apply to materials containing nonwater hydrogen. It is likewise responsive in varying degrees as the water is bound or free. The large size and the critical geometry of instrumentation restricts its use to the laboratory. Geary (1956) notes that this measurement is directly related to the water content of the material. Unlike with nuclear-scattering methods, the signal obtained from bound hydrogen water is different from that of free hydrogen or hydrogen in solid material. There may be some overlap in signals from hydrogen-containing liquids, such as fat or oils. Because this equipment in its present state of development is large and highly complex, its use in measuring moisture in highway materials is restricted.

6.2.4 *Infrared Reflection*

A relatively new technique that is widely accepted in some industrial processes is based on the infrared (IR) reflection that is proportional to the moisture content of the exposed solid. A limitation is that the interaction is primarily a surface interaction. If moisture is not homogenous, significant errors can occur. Galluzzo (1968) indicated the cost of such equipment to be $5,000 to $15,000. The range of measurement is between 0.05 and 80 percent (dry). There is obvious interference by other materials that reflect IR. The method can be used for most solids, preferably sheet or granular, where rapid response is required.

An instrument to measure moisture in paper was evaluated by Hardacker (1968). Reflection signals of 1.94 μ and 1.80 μ were compared with the aid of a filter disk. This particular instrument was the Inframike made by

General Electric. The range is from 0 to 40 percent moisture (dry). The accuracy was determined to be 0.1 to 0.3 percent. A drift of 0.1 percent was observed in 7 days.

Various sources of error were found. A 0.75 percent change was found from 20° to 170°F. A 10 percent basis weight change gives a 0.3 percent change in moisture indication. Plasticizers also affected the response. Spacing between the gauge and the surface was critical, with a 0.1 percent error for a 0.05-in. deviation.

An instrument made by Anacon Inc. (1968) also uses the filter wheel technique. The reflection of 1.9-μ radiation indicates moisture. The 1.7-μ wavelength is used as a reference.

7.0 TENSIOMETRY

7.1 Porous Cup

The name "tensiometer" was used by Richards and Gardner (1936) as an unambiguous reference to the porous cup and vacuum gauge combination for measuring capillary tension or the security with which water is held in soil. This term replaced earlier designations such as "capillary potentiometer" (Gardner et al., 1922), "soil hygrometer" (Heck, 1934), and "soil moisture meter" (Rogers, 1935).

The term "pF," introduced by Schofield (1935), is commonly used to describe tensiometer response. It is defined as the common logarithm of the height of a water column in centimeters equivalent to the soil moisture tension.

Richards and Gardner (1936) found that air entry in dry soil was a serious problem with the early porous cups. They observed hysteresis errors of about 3 percent. Temperature corrections were based on the temperature dependence of surface tension.

Tensiometers were more fully developed by Richards (1942), following earlier work by Neal, Richards, and Russell (1937, 1939) and Stoeckeler and Aamodt (1940). Agriculture has been the most notable beneficiary where high accuracy is not required. Using inexpensive porous cups costing less than $1.00 each, Kenworthy (1945) obtained sufficient air entry values and determined laboratory and field calibrations to have an accuracy of 5 percent (dry). The cup conductance of about 0.02 cm^3 $atm.^{-1}$ sec^{-1} gave a response time of less than 1 min.

In a revue of tensiometers, Johnson (1962) was somewhat more generous in his estimate of the accuracy attainable. He indicated about 2 percent using a vacuum gauge, and a little better using a mercury manometer. Taylor (1955) attributed the largest source of error to air bubbles. His units were pretested at 20 psi or greater, but dissolved air in water passing through the cup was indicated as an additional source of bubbles.

Temperature also has been a source of error. Haise and Kelley (1950) and Taylor (1955) attributed large diurnal variations to temperature-related vapor-pressure gradients and distillation transfer between cup and soil.

Hysteresis is significant with this technique. A moisture tension curve generally indicates a higher soil moisture tension during drying than during wetting. Some of this is real, possibly from the ink-bottle effect, but part of it is apparent as a result of the long response times, on the order of ½ hr to many hours. Several workers have devised methods for reducing this response time.

Miller (1951) and Dumbleton and West (1968) used a manometer pressure balance system to measure tension without flow of moisture through the porous cup. An improvement in accuracy also results in that moisture is neither gained nor lost from the sample. The time response is about 30 sec for a reading accuracy of 0.1 cm water.

Although salt concentration in the soil may affect the tensiometric method, it is considered to interfere less than with electrical methods (Johnson, 1962). Organic content of soil also results in a shift in the calibration (Stoeckeler, Aamodt, 1940).

Jumikis (1969) has been studying the problem of moisture transfer under freeze-thaw cycling conditions. He includes 85 references in his report, many of which report his previous work. Porosity appeared to be the principal factor in determining film flow, vapor flow, or pure vapor diffusion. In common soils, where the porosity is less than 50 percent, the most effective mechanism for upward flow of moisture is the mechanism of film flow. His moisture-related measurements include a tensiometer plus gravimetric measurement at termination of his experiments. He used a conical-shaped tensiometer with effective surface area of 30.52 cm^2. The porosity was 67 percent with an average capillary radius of 0.3 microns. The bubbling gauge pressure (equal to air-entry value) is 2 atm.

The air entry value is the tensiometer pressure difference necessary to bring about an air leak through the porous wall when the wall is saturated with distilled water. Normally when one is measuring soil tensions up to about 1 atm., the tensiometer must test an air-entry value greater than 1 atm.

During freezing the tensiometers used by Jumikis were found to contain ice; however, no damage was found on thawing. His restriction of about 0.85-atm water pressure apparently was the result of the porosity of his sensing element and the use of flexible tubing to transmit pressure.

Combining pressure transducers with tensiometers in recent years has resulted in an improvement in over-all performance of this method. Klute and Peters (1962) used a strain gauge pressure transducer to obtain a short-time response of less than 1 sec, including the recorder. Based on cup conductance (κ) and gauge sensitivity, the time constant (τ) was figured to be 0.02 sec from the relationship

$$\tau = 1/\kappa S \qquad \text{(A-25)}$$

The gauge sensitivity was 3×10^3mb cm^{-3}. With the strain gauge bridge voltage at 6 v, a 4-mv-per-volt transducer output corresponds to 12 μv cm^{-1} of water. This resulted in a minimum detectable change of 3 mm of water. With the exception of the recorder drive, the unit was powered by a 6-v dry cell (or a regulated power supply).

Thiel, Fouss, and Leech (1963) developed a pressure transducer ceramic tip combination for laboratory and field use for measuring hydrostatic pressures in a porous medium. As a deflection sensor, linearly variable differential transformers (LVDT) were found to be superior to strain gauges and semiconductor force transducers. The system cost $50, assembled and calibrated.

For maximum sensitivity a stainless steel diaphragm should be designed for a specific operating range. The maximum height of water that the diaphragm can withstand is

$$h = \frac{S_r t^2}{0.027 a^2} \qquad \text{(A-26)}$$

in which

S_r = maximum radial stress in the diaphragm (44,000 psi is a typical proportional limit for stainless steel);
a = diaphragm radius;
t = diaphragm thickness; and
h = water pressure inches of water.

A typical gauge sensitivity is 0.3 v/50 cm water.

The unit was powered by a 6-v audio-oscillator at 10 kHz. The resulting accuracy was ±0.5 cm water.

Watson (1963, 1965, 1967) has contributed to the development and application of self-contained pressure transducer tensiometers. He lists their advantages for field use as: (1) rapid response of system due to small volume displacement of transducer, (2) convenience of obtaining a chart record of the pressure changes with time, (3) avoidance of diurnal temperature effects such as those that occur in tensiometer-manometric systems with above-ground components, (4) applicability of the method to the measurement of the capillary pressure at depth, (5) relative ease in which the system can be instrumented for automatic control, and (6) elimination of the response problems that occur in soils of low conductivity when one is using a selector valve to switch from one tensiometer to another.

Commercially available pressure transducers are small in size, highly accurate, and very stable. This should make it possible to achieve sensitivities and responses in the field equivalent to those achieved in the laboratory. A typical transducer is Type PM-131TC manufactured by Statham Instruments, Inc. Characteristics of this transducer are as follows:

CHARACTERISTIC	VALUE
Rated excitation	5 v DC or AC
Nominal output	4 mv/v
Nominal bridge resistance	350 ohms
Hysteresis and linearity	0.75% normal, 0.25% special
Compensated temperature range	—65° to 250°F
Thermal shift: Sensitivity	0.01%/°F
Thermal shift: Zero	0.01%/°F
Weight	¼ oz
Differential pressure ranges	±2.5, ±5, ±10, ±15, ±25
Differential overload	200% of range
Hydraulic sensitivity (S)	5.9×10^{-5} cc/mb

Pressure changes of 0.2 mb are detectable with this system. With a ceramic 600-mb bubbling pressure, the time constant is approximately 0.2 sec. This method is particularly applicable to the movement of water in unsaturated soil in field situations. This equipment appears to be able to achieve increased accuracy and response with a minimum of development effort and fabrication complexities.

7.2 Permeable Membrane

A permeable membrane tensiometer is similar to the porous cup tensiometer. Instead of capillary flow through a porous ceramic wall, the flow mechanism of a permeable membrane is more nearly described by osmosis. According to Coleman and Marsh (1961), seven components of a soil stress tensor are determined by the moisture content density, shear strength, and total free energy depression of soil water. The components are the principal total stresses, σ_1, σ_2, σ_3; pore water pressure, μ; pore air pressure, π; osmotic pressure, P; and temperature, θ. A model relating these parameters would be very useful in the design of a functional field instrument.

For laboratory use, Coleman and Marsh developed two pressure-membrane apparatus, one for low and one for high pressures. Another laboratory technique for high pressures has been designed by L. A. Richards (Patent No. 2353760—Pressure membrane extraction apparatus).

A field unit is known commercially as the Aquapot. It is manufactured by NIC Instrument Co., Victoria, Australia.

8.0 THERMAL CONDUCTIVITY

The thermal conductivity method relies on the increase in thermal conductivity of porous materials with increasing moisture content. This principle can be applied through transmission in which a rise in temperature of the material is measured at a distance from the heat source. It also may be applied through dissipation, by measuring the temperature rise of the heating element. In the former case, the response time is a function of the spacing between the thermocouples and the heating element. Although thermal contact may present problems, there are inherent advantages to the technique that make it suitable for incorporation into highway embankments, or pavements, or structures where moisture is to be measured. Thermocouples and heating wires normally have little disturbing effect on these materials, and salt or other compositions of the material offer less interference than in any other technique. The continuous monitoring of variations of moisture with time is the most notable need to which this technique may be applied. According to Johnson (1962), the transmission type or thermal conductivity cell has been the most satisfactory.

Shaw and Baver (1939) applied a laboratory apparatus of their own design to a variety of soils. No hysteresis was observed with their method, but a wide variation in calibration curves was obtained for different soils. The reproducibility with the same soil was relatively good, being less than 1 percent (dry). The sensitivity of the method was found to be increased by the fact that conductivity and heat capacity change with moisture content in the same direction. Their instrument was designed to keep the temperature difference between the heat source in the soil very small to prevent the movement of moisture from the point where heat is applied.

The temperature rise is given approximately by

$$dQ/dt = K\Delta T + A\left(1 - e\frac{-Kt}{C}\right) \qquad \text{(A-27)}$$

when heat is added at a constant rate. A and K are constants, and C is the specific heat.

A negligible effect from ambient temperature of soil between 0° to 54°C was found. Also, a negligible effect from 10,000 ppm KCl was observed.

Because heat transport in moist, porous materials occurs with some distillation of water vapor, de Vries (1952) proposed a nonstationary method and small temperature gradients for measuring thermal conductivity of soils.

The soil parameters of primary interest are thermal conductivity (λ) and thermal diffusivity (a). At a point, 4, from the center of a long heated wire, the rise of temperature (θ) is given by

$$\theta = (q/4\pi\lambda)\{-Ei(-r^2/4at)\} \qquad \text{(A-28)}$$

in which q is the amount of heat produced per unit time and unit length of the source, and the elliptic integral is given by

$$Ei(x) = \int_x^\infty (1/x)\exp(-x)dx$$

$$= 0.5772 + \ln x - \frac{x}{1\cdot 1!} + \frac{x^2}{2\cdot 2!}\cdots \qquad \text{(A-29)}$$

Experiments were carried out with a relatively small sensor buried in the soil. Measurements were taken after about 60 sec (when the temperature difference has a logarithmic dependence on time) and were found to be a good indicator of soil moisture changes.

Additional development work has been done by Winterkorn (1956), Bloodworth (1967), and Vos (1965). Using a thermistor as a combination heating element and temperature indicator, Bloodworth found that the technique was unaffected by salt concentration and had greatest sensitivity below field capacity. He also found that calibration was required for different soils and that soil temperature corrections must be applied, but the measurements could be easily made. Vos (1965) looked at the nonsteady state with twin probes. The technique was found to be a very good measure of thermal conductivity, but caution was expressed concerning the errors occurring, especially due to moisture migration under a temperature gradient.

Globus and Kaganov (1965) developed a thermistor probe-type instrument in which they attempted to get around the problem of sensitivity to ambient temperature. The unit weighed 2.5 kg, was powered by a pen-light battery, and provided temperature compensation by means of a variable resistance. They attributed the sensitivity to type of soil to the variation in pore size distribution, structural peculiarities, and density. These variations can be greater than 20 percent (dry). A porous envelope was found to improve performance through improved reproducibility of physical contact.

9.0 MISCELLANEOUS METHODS

9.1 Vapor Pressure

Upon evaporation, the volumetric expansion of water is very large. When the sample is enclosed in a small volume and the temperature is raised to evaporate the water that it contains, the pressure of the container will rise. This pressure can then be related directly to the moisture content. Minute amounts of moisture can be measured through the use of small volume, heating, or condensation and reevaporation techniques (Geary, 1956). Volatile materials in the sample would be expected to interfere.

9.2 Colorimetry

The reaction of cobalt chloride with moisture produces a measurable color change proportional to moisture content. An automatic system manufactured by Technicon Control, Inc., is described by Roth (1966). A bead chain feeder serves to measure the amount of solids taken in so that this technique might be considered a gravimetric method with the colorimetric technique for measuring the evolved moisture.

This technique is highly specific, although materials that form insoluable compounds with cobalt chloride ions will interfere. Samples must be removed for analysis, and the state of the water measured is somewhere between unbound and partially bound. The measurement range is between 0.01 and 20 percent. The measurement is continuous, with a response time of between 2 and 3 min. The precision is limited to the reproducibility of the bead sampler, which is about ± 10 mg. This limits high accuracy and precision to materials with a moisture content greater than 5 percent. This technique is most applicable to free-flowing uniform granular materials that can be brought to the instrument by conveyor.

9.3 Penetrometer

The basis for the penetrometer method is resistance to penetration or deformation. Geary (1959) reviewed its use in highway materials and referenced some of the earlier work. The penetration device has become known as the Proctor Needle (Proctor, 1933). Some problems related to its use are that penetration equipment must be calibrated for each type of soil, and gravelly or stony soils are unsuitable. However, the method is very fast and often is used in correlation studies with other test methods (Antrim et al., 1967). A commercial device is distributed by Soiltest, Inc.

APPENDIX B

BIBLIOGRAPHY

The literature survey portion of this research effort encountered several thousand references. This was expected for such a broad coverage of moisture measurement techniques. Included in this bibliography are those publications that were either referenced in this report, or reviewed and rated for usefulness of contents, or for which abstracts were reviewed. Brackets following each bibliographic item provide additional information concerning measurement methods reported. For instance, [4.1, R1] indicates the measurement category that refers to Appendix A, where section 4.1 is devoted to neutron-scattering techniques; R1 is the review rating code. The following abbreviations are used:

R1—Good experimental data on a specific measurement method.
R2—Good general data on moisture measurement.
R3—Reportable data with questionable interpretation.
R4—Vague applicability.
R5—Inapplicable.
Abs.—Only the abstract was available for review.
Gen.—General moisture-related work.
Sur.—Survey of many methods.
Bib.—Bibliography.
Tel.—Telemetering.

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APPENDIX C

REMOTE SENSING

The remote measurement field can be divided into three basic categories: (1) phenomena that are remote by nature, (2) wireless transmission of information, and (3) physical contact with the sensor element. Of the three categories, the first is the most attractive; however, it is the most difficult, and to a large extent has eluded the researcher, except for a few specific cases such as the nuclear techniques for near-surface measurements. This category includes the transmission and reflection of electromagnetic and acoustical energy as well as nuclear particles. A good understanding of these phenomena within the highway engineering environment should prove fruitful in the development of future measurement methods. The major limitation to date in this field has been the lack of understanding of interactions and interferences.

The second category involves the transmission of data from a point of interest to a remote location. This category includes the radio transmitter. The sensing mechanism is considered separately from the transmission mechanisms. A typical application of this category would involve the implanting of a transmitter with the sensor in the location of the measurement and then telemetering the data outside the system. The major problem here is the lack of a good, inexpensive energy source that will last long enough to meet present requirements and not significantly disturb the sample.

The third technique involves a physical link with the sensor element. An example here is a capacitance probe implanted into the medium of interest, with wires attached to the probe and extending outside the medium to the readout system. The range of materials, geometry, and sensing techniques that can be considered is very wide. This category of techniques has found some use in the research field, but in general is frustrating and difficult to use on any broad scale.

The second category appears to be the most fruitful area of development at present. The technology of implant telemetry has reached a stage of development where it deserves serious consideration for highway engineering applications, particularly in the area of moisture-related research (Caceres, 1965; see also NASA *Rept. SP-5023, Rept. SP-5054,* and *Tech. Brief 66-10057*). The incentive for this development has been the combined requirements of the aerospace industry and medical implant biotelemetry. Some of these requirements are small size and weight, high reliability, low power drain, and long life.

A small circuit developed by MacKay is shown in Figure C-1 (MacKay, 1965). This circuit may be used to demonstrate the basic compatibility of this technique with most of the moisture measurement techniques that have been identified. A notable exception is the nuclear method. Variation of the capacitance, C_2, with moisture could be transmitted externally. The resistance, R, could be soil resistance or a hygroscopic film resistance. By replacing R with a thermistor, soil temperature could be measured. Soil moisture tension could be measured by allowing pressure changes to move the transmitter core, M.

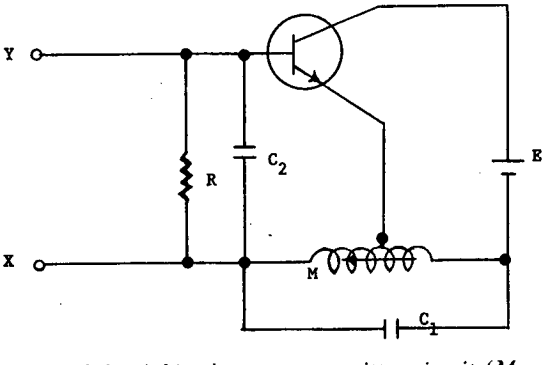

Figure C-1. A biotelemetry transmitter circuit (MacKay, 1965).

Although the simple circuit of Figure C-1 is inadequate in terms of long life and stability, the general technique can be greatly improved by using advanced microelectronic circuit techniques. For example, Fryer (1965) has reported an implantable microwatt transmitter that accurately measures and telemeters deep body temperature for up to two years.

There are commercial units available (American Electronic Laboratories, for instance), for special application, but effective use of this technique will require further work. Significant benefits should result from an applied research and development program for the purpose of adapting implant telemetry to specific moisture measurement methods to meet the needs of the highway engineer.

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—* A Critical Review of Literature Treating Methods of Identifying Aggregates Subject to Destructive Volume Change When Frozen in Concrete and a Proposed Program of Research—Intermediate Report (Proj. 4-3(2)), 81 p., $1.80

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