76888

3649

AGRONOMY

A Series of Monographs

ıe American Society of Agronomy and Academic Press published the first six books in this series. ɜneral Editor of Monographs 1 to 6 was A. G. Norman. They are available through Academic Press, 11 Fifth Avenue, New York, NY 10003.

EDMUND MARSHALL: The Colloid Chemical of the Silicate Minerals. 1949

ſRON T. SHAW, *Editor*: Soil Physical Conditions and Plant Growth. 1952

D. JACOB, *Editor*: Fertilizer Technology and Resources in the United States. 1953

. H. PIERRE and A. G. NORMAN, *Editors*: Soil and Fertilizer Phosphate in Crop Nutrition. 1953

EORGE F. SPRAGUE, *Editor*: Corn and Corn Improvement. 1955

LEVITT: The Hardiness of Plants. 1956

he Monographs published since 1957 are available from the American Society of Agronomy, 677 S. Road, Madison, WI 53711.

AMES N. LUTHIN, *Editor*: Drainage of Agricultural Lands. 1957 *General Editor*, D.E. Gregg

RANKLIN A. COFFMAN, *Editor*: Oats and Oat Improvement. 1961 *Managing Editor*, H. L. Hamilton

. KLUTE, *Editor*: Methods of Soil Analysis. 1986
art 1—Physical and Mineralogical Methods. Second Edition. *Managing Editor*, R. C. Dinauer

. L. PAGE, *Editor*: Methods of Soil Analysis. 1982
art 2—Chemical and Microbiological Properties. Second Edition. *Managing Editor*, R. C. Dinauer

ſ. V. BARTHOLOMEW and F. E. CLARK, *Editors*: Soil Nitrogen. 1965
)ut of print; replaced by no. 22) *Managing Editor*, H. L. Hamilton

. M. HAGAN, H. R. HAISE, and T. W. EDMINSTER, *Editors*: Irrigation of gricultural Lands. 1967 *Managing Editor*, R. C. Dinauer

RED ADAMS, *Editor*: Soil Acidity and Liming. Second Edition. 1984 *Managing Editor*, R. C. Dinauer

. G. HEYNE, *Editor*: Wheat and Wheat Improvement. Second Edition. 1987 *Managing Editor*, S. H. Mickelson

.. A. HANSON and F. V. JUSKA, *Editors*: Turfgrass Science. 1969 *Managing Editor*, H. L. Hamilton

:LARENCE H. HANSON, *Editor*: Alfalfa Science and Technology. 1972 *Managing Editor*, H. L. Hamilton

. R. WILCOX, *Editor*: Soybeans: Improvement, Production, and Uses. Second Edition. 1987 *Managing Editor*, D. A. Fuccillo

AN VAN SCHILFGAARDE, *Editor*: Drainage for Agriculture. 1974 *Managing Editor*, R. C. Dinauer

)EORGE F. SPRAGUE, *Editor*: Corn and Corn Improvement. 1977 *Managing Editor*, D. A. Fuccillo

ACK F. CARTER, *Editor*: Sunflower Science and Technology. 1978 *Managing Editor*, D. A. Fuccillo

ıOBERT C. BUCKNER and L. P. BUSH, *Editors*: Tall Fescue. 1979 *Managing Editor*, D. A. Fuccillo

ſ. T. BEATTY, G. W. PETERSEN, and L. D. SWINDALE, *Editors*: Planning the Uses and Management of Land. 1979 *Managing Editor*, R. C. Dinauer

ſ. J. STEVENSON, *Editor*: Nitrogen in Agricultural Soils. 1982 *Managing Editor*, R. C. Dinauer

H. E. DREGNE and W. O. WILLIS, *Editors*: Dryland Agriculture. 1983 *Managing Editor*, D. A. Fuccillo

R. J. KOHEL and C. F. LEWIS, *Editors*: Cotton. 1984 *Managing Editor*, D. A. Fuccillo

N. L. TAYLOR, *Editor*: Clover Science and Technology. 1985 *Managing Editor*, D. A. Fuccillo

D. C. RASMUSSON, *Editor*: Barley. 1985 *Managing Editor*, D. A. Fuccillo

M. A. TABATABAI, *Editor*: Sulfur in Agriculture. 1986 *Managing Editor*, R. C. Dinauer

R. A. OLSON and K. J. FREY, *Editors*: Nutritional Quality of Cereal Grains: Genetic and Agronomic Improvement. 1987 *Managing Editor*, S. H. Mickelson

A. A. HANSON, *Editor*: Alfalfa and Alfalfa Improvement. 1988 *Managing Editor*, S. H. Mickelson

F-704 P.002 T-453 +5056653866 From-EES-10 14:21 08-28-03

METHODS OF SOIL ANALYSIS

76888

Part 1

Physical and Mineralogical Methods

Second Edition

Arnold Klute, *Editor*

Editorial Committee

G. S. Campbell
D. R. Nielsen
R. D. Jackson
A. Klute, chair
M. M. Mortland

1186 pp © 1986

Senior Managing Editor: RICHARD C. DINAUER
Editor-in-Chief ASA Publications: DWAYNE R. BUXTON
Editor-in-Chief SSSA Publications: JOHN J. MORTVEDT

Number 9 (Part 1) in the series

AGRONOMY

American Society of Agronomy, Inc.
Soil Science Society of America, Inc.
Publisher
Madison, Wisconsin USA

1986

LOS ALAMOS NATIONAL LABORATORY
AUG 2 2 1989
LIBRARIES PROPERTY

09-29-03 14:21 From-EES-10 +5056653866 T-453 P.003 F-704

21 Water Content

WALTER H. GARDNER
Washington State University
Pullman, Washington

21-1 GENERAL INFORMATION

Direct or indirect measures of soil water content are needed in practically every type of soil study. In the field, knowledge of the water available for plant growth requires a direct measure of water content or a measure of some index of water content. In the laboratory, determining and reporting many physical and chemical properties of soil necessitates knowledge of water content. In soils work, water content traditionally has been expressed as the ratio of the mass of water present in a sample to the mass of the sample after it has beeen dried to constant weight, or as the volume of water present in a unit volume of the sample. In either case the amount of water in the sample is needed. To determine this the water must be removed and measured, or the mass of the sample must be determined before and after removal of water. And criteria must exist for deciding at what point the sample is "dry." For a great many purposes where high precision and reproducibility are not required, a precise definition of the term "dry" is not required. Where high precision does become important it should be recognized that "dry" is a subjective term and must be defined. Where high precision is implied in water content figures, such figures must be accompanied by the definition and a description of the procedure used to determine the water content. Traditionally, the most frequently used definition for a dry soil is the mass of a soil sample after it has come to constant weight in an oven at a temperature between 100 and 110°C. The choice of this particular temperature range appears not to have been based upon scientific consideration of the drying characteristics of soil; and in practice, samples are not always dried to constant weight.

Water content as usually used in soils work is either a dimensionless ratio of two masses or two volumes or is given as a mass per unit volume. When either of the dimensionless ratios is multiplied by 100, such values become percentages, and the basis (mass or volume) should be stated. Where no indication is given, the figure generally may be assumed to be on a mass basis because the determination usually involves getting mass-basis figures first and then converting them to volume-basis figures.

Copyright 1986 © American Society of Agronomy—Soil Science Society of America, 677 South Segoe Road, Madison, WI 53711, USA. *Methods of Soil Analysis, Part I. Physical and Mineralogical Methods*—Agronomy Monograph no. 9 (2nd Edition)

08-29-03 14:21 From-EES-10 +5056653866 T-453 P.004 F-704

Occasionally the ratio of mass of water to that of the wet soil is used. Conversions from one basis to the other easily are made by use of the formulas,

$$\theta_{ww} = \theta_{dw}/(1 + \theta_{dw}), \quad \theta_{dw} = \theta_{ww}/(1 - \theta_{ww}) \qquad [1]$$

where θ is the water content, and the subscripts *dw* and *ww* refer to dry-weight and wet-weight basis.

If water content is desired on a volume basis, the volume from which the sample was taken must be known. A sampling device which takes a known volume of soil (Richards & Stumpf, 1960) may be used; or the bulk density of the soil (mass of soil per unit volume), as obtained from independent measurements in the same area, may be used. Where a bulk density is known or assumed, volume-basis water contents may be obtained from the mass-basis figures by use of the formula

$$\theta_{vb} = (\rho_b/\rho_w)\theta_{dw} \qquad [2]$$

where ρ_b is the bulk density of the soil, ρ_w is the density of water (this usually may be taken as unity in g/cm^3 or Mg/m^3 units, but must be present for dimensional consistency), and the subscripts *vb* and *dw* refer to volume basis and dry-weight basis. The accuracy of the inferred volume-basis water content depends upon the accuracy of the bulk density figure used as well as upon the accuracy of the dry-weight water-content figure. Both depend upon the accuracy of the figure for the "dry" mass of the soil. In some types of work water content also is expressed as the ratio of the volumetric water content to that which would exist at saturation, or the *saturation ratio*. On this basis the water content of a saturated soil is unity. Further information on measurement of bulk density may be found in chapter 13.

Computations of water content on a volume basis require a correct measure of bulk density. Considering the variability of soil, even in an area as small as 1 m^2, some error nearly always is involved. However, for many purposes where volume-basis water contents are needed, the error probably is no more important than the error involved in representing the water content at a particular depth in the field by a single number. Taylor (1955), working with Millville loam (which is a relatively uniform soil insofar as observable physical and chemical properties are concerned), reported coefficients of variation of 17 and 20% for gravimetric measurements of the water content of samples of soil from field plots under irrigation by furrow and sprinkler methods, respectively. Similar variation has been observed on apparently uniform areas of Palouse silt loam in the dryland areas of southeastern Washington. For precise work, statistically sound methods must be used for sampling and for reporting such measurements (see chapters 1, 2 and 3; Cline, 1944).

When water is applied to soil by irrigation or rainfall, the quantity

layer. To obtain the volume of water applied to a given area would require multiplication of the depth by the area, measured in the same length units. In like manner the quantity of water in a soil profile may be reported as the depth of the water present if it were to be accumulated in a layer. In this case, however, the number is not quantitatively meaningful without an association being made with some particular profile depth defined by a number or by a description of the part of the profile under consideration, such as the rooting zone, plow depth, or depth to bedrock. In the case of uniform rainfall the concept of "depth of water" matches the usual way of visualizing the amount of water involved, as well as the way in which it is measured in an ordinary catchment device. However, when the amount of water in the soil is to be measured or computed the number used is volume of water in unit volume of soil (measured on a volume basis or inferred using Eq. [2]) multiplied by the incremental volume used (unit area × length of depth increment) and summed for the entire profile under consideration. To reduce this to the same basis as for rainfall, the area (unit, in this case) is divided out:

$$(\text{vol/vol})(\text{unit area} \times \text{length})/(\text{unit area}) = \text{length}. \qquad [3]$$

Specification of a "depth" of accumulated water in such fashion, unless the context makes its meaning unequivocal, should be accompanied by a modifier such as "in the rooting zone." The length or "depth" may be specified in millimeters, centimeters or meters. If specified as a depth per some depth increment, as has been common in the past, this dimensionless number would be multiplied by the number of increments of concern to give the total depth of water in a defined soil depth, providing that the water content over the total depth is uniform and that units are properly taken into account.

Common expressions for water in soil in the USA have been "inches per foot," "acre-inches," "acre-feet," and "acre-feet per acre." The term "acre-feet" (and similarly, "acre-inch") often has been improperly used to mean "acre-feet per acre" or "feet" of water rather than a volume of water, which it really represents. Similar problems have existed with other units, such as the hectare and metric units of length. Eq. [3] may be seen to be independent of units except for the length of the depth increment. This can be in any convenient system of units. To obtain the volume of water contained in soil on a given size of farm unit, as may be needed in farm water accounting, the depth or length computed by Eq. [3] would be multiplied by the area of the field in question, measured in appropriate units.

21-2 DIRECT METHODS

21-2.1 General Principles

Determination of water content of soils may be accomplished by direct and indirect methods. Direct methods may be regarded as those

09-29-03 14:21 From-EES-10 +5056653866 T-453 P.005 F-704

methods wherein water is removed from a sample by evaporation, leaching, or chemical reaction, with the amount removed being determined. Determination of the amount removed may be by one or more of the following methods: (i) measurement of loss of weight of the sample, (ii) collection by distillation or absorption in a desiccant and measurement of the amount of water removed, (iii) extraction of the water with substances which will replace it in the sample and measurement of some physical or chemical property of the extracting material that is quantitatively affected by water content, or (iv) quantitative measurement of reaction products displaced from a sample. In each of these methods the water and soil are somehow separated, and the amount of water removed is measured or inferred.

The key problem in water content determination in porous materials has to do with the definition of the dry state. Soil is made up of colloidal and noncolloidal mineral particles, organic materials, volatile liquids, water, and chemical substances dissolved in water. Of the mineral fraction of the soil, the noncolloidal particles probably are the easiest with which to deal. At ordinary room temperatures and room humidities, such materials will have a small quantity of adsorbed water that is removed easily from their surfaces by heating. Only at elevated temperatures does water of crystallization associated with these minerals come off. The status of water in the colloidal particles of the mineral fraction is more complicated. Water present in the colloidal fraction may be considered in two categories. *Structural water*, is water derived from components of the mineral lattice itself, whereas *adsorbed water* is water that is attached to the mineral lattice but is not a structural component of the lattice. It often is difficult to distinguish between the two categories. Some adsorbed water may be located with respect to the lattice in such a way that the difficulty of removal is comparable to that for structural water. (Difficulty here refers generally to the temperature required for removal and possibly to the fineness of the particles.) The exact situation depends upon the nature of the particular mineral.

Some representative thermal dehydration curves (after Nutting, 1943) are presented in Fig. 21-1. (It should be recognized that the character of the thermal dehydration curve depends upon the purity of the sample, so that it is unlikely that curves for the same type of minerals constructed by various workers would be identical.) The curves in Fig. 21-1 show the weight loss of samples that were initially at room temperature and in equilibrium with room humidity of approximately 60%, as the samples were heated to constant weight at various temperatures. The weight at a temperature of 800°C or slightly higher is taken to be unity. It is not clear from the curves what part of the water is adsorbed water and what part is structural water. But it should be obvious from these curves that for colloidal minerals it is important to specify the exact temperature of dry[illegible] [illegible]f precision is required. Considering that the temperature control of o[illegible]ary laboratory ovens is not very good, precision water content

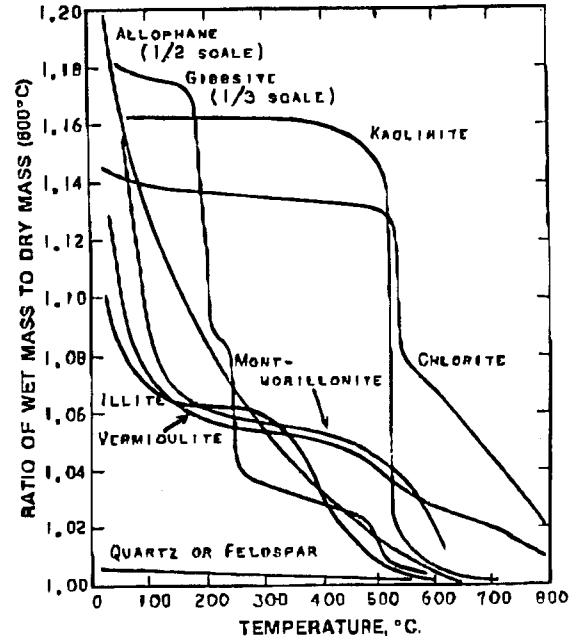

Fig. 21-1. Wet mass of a unit dry mass of a clay mineral as a function of drying temperature. The sample is presumed to be dry at a temperature of 800°C or greater. Water content percentages on a mass basis for this drying temperature may be obtained by subtracting 1 from the ordinate figure and multiplying by 100. (The numbers to the right of the decimal thus may be regarded as percentages.) (Nutting, 1943)

measurements should not be expected except where extraordinary attention is given to temperature measurement and control.

Although it is not possible to generalize on the precision of oven temperature control, it can be noted that unless the temperature control of an oven has been checked, it is not safe to assume that control in the specified 100 to 110°C range is achieved in ordinary laboratory ovens. Several ovens in Washington State University soils laboratories showed variations as large as +15°C over a period of a few weeks (without a sample load) and temperature differences within the ovens of as much as 40°C. This was true of forced-draft ovens as well as convection ovens. Temperature variation with elevation in the oven was found to be as great at 40 to 100°C for several convection-type ovens at the Massachusetts Institute of Technology (Lambe, 1949). Since radiation heating could be a factor in some ovens, precision work requires that temperature measurements be made within the soil sample rather than in the oven atmosphere, as is done with conventional oven thermometers.

Insofar as most of the colloidal minerals are concerned, the temperature sensitivity of water content measurements would be reduced materially by selection of a temperature range where water [illegible]ss changes with increasing temperature are smaller than they are fr[illegible]00 to 110°C.

09-29-03 14:21 From-EES-10 +5056653866 T-453 P.006 F-704

Judging from the curves of Fig. 21–1, the range between 165 and 175°C would be better than the traditional range of 100 to 110°C. However, excessive oxidation and decomposition of soil organic matter in this high temperature range would prevent its general adoption, although in special cases it might be appropriate.

The problem of defining "dry conditions" in the organic fraction of the soil is even more difficult than defining such conditions for the colloidal mineral fraction. The organic fraction of soils consists of undecomposed plant fragments, such as roots, and resistant decomposition products such as polysaccharides or polyuronides as well as intermediate products. Volatile liquids other than water also may be present. If subjected to too high temperatures, organic materials are oxidized or decomposed and lost from the sample. The temperature at which excessive oxidation occurs would be difficult to specify, but 50 °C seems to be a common temperature used to dry organic materials and is thought by many investigators to be sufficiently low to avoid loss of organic matter by oxidation.

Little is said in the soils literature about weight changes due to oxidation and decomposition of the organic fraction in soil samples undergoing drying. However, many investigators readily admit that they often have observed weight changes in soils being dried over periods of many days to many weeks. In our own laboratory we have observed weight changes in samples during drying over periods as long as 15 days. A silt loam soil, containing about 3% organic matter and with illite predominating in the clay fraction, lost weight which corresponded to 0.3% water content from the 2nd to the 10th day in a forced-draft oven at 100 to 110°C. Whether this represents water loss or oxidation and decomposition is difficult to tell.

Extensive studies have been made on the drying of food products by food technologists interested in dehydration. Here efforts seem to be more in the direction of finding a drying technique that will yield reproducible results rather than in finding a method wherein the dry status is unequivocal. A reference method used by food technologists (Makower, 1950) involves drying ground vegetable material at room temperature in a vacuum over a desiccant that permits practically no water vapor pressure (magnesium perchlorate). Drying time is long (6 to 9 months). The assumption is made that with no air present decomposition and oxidation are negligible. Even as a reference standard, this method is painfully slow and other methods have been sought.

One modification that has reduced the drying time from months to days (Makower and Nielsen, 1948) involves lyophilization or freeze-drying. The fresh weight of the organic matter is obtained first and then water is added to the sample to cause it to swell. In the swollen state, the sample then is frozen, and most of the water is removed by sublimation. In this state the mechanical structure of the sample essentially is fixed, and evaporation can proceed much more rapidly than under conditions where the

porosity of the sample is reduced drastically through shrinking. After most of the water has been removed, drying can be continued at higher temperatures. A redrying procedure involving drying at temperatures between 50 and 70°C also has been developed by Makower et al. (1946) as a reference standard. They reported that considerable decomposition of vegetables takes place at temperatures above 70°C.

Since many soils contain only small amounts of organic material, much of which is fairly stable, inaccuracies introduced by uncertainties in drying of organic materials often may be negligible. However, since drying temperatures normally used for soils are well above those considered safe for organic materials, decomposition and oxidation should be expected and considered in the development of drying techniques and taken into account in reporting data. Where precise water content values are required, precautions must be taken to assure that minimum losses due to oxidation or decomposition occur and that all samples to be compared are dried to constant weight or to some arbitrary state of dryness which, by careful timing, can be reproduced. The measured water content of a soil which contains more than negligible quantities of organic material must be regarded as an arbitrary value, depending upon a state of dryness defined by the method used rather than upon a dry state which could be regarded as unequivocal.

In many salt-affected soils, considerable material exists in the soil solution. If water is leached out of the sample by some liquid such as alcohol, most of the dissolved materials would be removed along with the water. On the other hand, if the soil is dried in an oven only the volatile materials are lost. Depending upon the quantities of dissolved substances present, the method of water removal could make considerable difference. It is possible to have water content differences as great as several tenths of a percent, depending upon whether water was removed by leaching or by evaporation. It is a moot question whether or not the dissolved substances should be considered to be part of the "dry" soil. Most procedures in common use involve oven-drying or calibration against oven-drying data, so that dissolved salts are counted as part of the "dry" soil.

It appears from the literature that oven-drying methods often may have been accepted too readily as reference standards for water content measurement. The situation is complicated by lack of detail in the literature concerning oven-drying procedures used for the water content measurements that are reported. Drying time rarely is mentioned, and the specification of oven temperature appears to be perfunctory if mentioned at all. There usually is no evidence to suggest that an investigator has questioned oven-drying procedures. By implication, scatter in many kinds of data based upon or related to water content measurements fails to be associated with the water content measurement. In some cases, other methods for obtaining water content are compared to oven-drying methods, and any lack of reproducibility observed is ascribed automat-

09-29-03 14:21 From-EES-10 +5056653866 T-453 P.007 F-704

ically to the other method rather than to the oven-drying procedure. Accuracy itself is measured against oven-drying methods. As a consequence of this misplaced trust in oven-drying procedures, there may be good reason to re-evaluate some of the other methods for water content determination and possibly a few other types of analytical procedures when water content determinations are critical.

Gravimetric water content measurements usually involve three independent measurements, the wet (x) and dry (y) weights of the sample and the tare weight (t). These may be combined in this way

$$[(x - t)/(y - t)] - 1 = \theta \qquad [4]$$

to provide water content θ. With each weighing, there is associated an error e, which is the balance error. The balance error is made up of two parts, a random error having to do with the reproducibility of a particular balance reading, and a bias which is the difference between an average reading and the true value. The bias is likely to be different at different weight values, but since a balance may be calibrated such bias may be eliminated to any desired degree of accuracy. A constant bias, such as might result from an improper adjustment of the zero on an automatic balance, introduces no error in the water content ratio provided it is the same for each weighing and the tare weight is not balanced out. This may be seen when such an error e is applied uniformly to each measurement appearing in Eq. [4]

$$[(x + e) - (t + e)]/[(y + e) - (t + e)] - 1 = \theta . \qquad [5]$$

The error term may be seen to cancel out.

The effect of random error may be evaluated by determining the variance σ^2 of the ratio, $(x - t)/(y - t)$, in Eq. [4]. The variance of this ratio with reasonable assumptions may be approximated by

$$\sigma^2_{x-t/y-t} = [1/(y - t)^2]\{\sigma^2_{x-t} + [(x - t)^2/(y - t)^2]\sigma^2_{y-t}\} . \qquad [6]$$

The variances of the numerator and denominator are given as

$$\sigma^2_{x-t} = \sigma^2_x + \sigma^2_t \qquad [7]$$

$$\sigma^2_{y-t} = \sigma^2_y + \sigma^2_t . \qquad [8]$$

Substituting Eq. [7] and [8] into [6] gives

$$\sigma^2 x - t/y - t = \frac{1}{(y - t)^2}\left[\sigma^2_x + \sigma^2_t + \frac{(x - t)^2}{(y - t)^2}(\sigma^2_y + \sigma^2_t)\right]. \qquad [9]$$

Since the variances due to the random error of all three weighings are likely to be nearly the same, Eq. [9] is approximated by

$$\sigma^2_{x-t/y-t} = 2\sigma^2_{x,y,t}\left[1 + \frac{(x - t)^2}{(y - t)^2}\right] \Big/ (y - t)^2 \qquad [10]$$

Combining Eq. [4] with Eq. [10] and replacing $(y - t)$ by the dry mass of the soil z gives

$$\sigma^2_\theta = \sigma^2_{x-t/y-t} = (2\sigma^2_{x,y,t}/z^2)[1 + (\theta + 1)^2]$$
$$= (2\sigma^2_{x,y,t}/z^2)(\theta^2 + 2\theta + 2) . \qquad [11]$$

For water contents and sample sizes ordinarily encountered in soils work, θ^2 is negligible so that Eq. [11] further reduces to

$$\sigma^2_\theta = (4\sigma^2_{x,y,t}/z^2)(\theta + 1) \qquad [12]$$

where the subscript, $(x - t)/(y - t)$, is replaced by θ for water content. Written in terms of standard deviations ($V = \sigma^2$), this becomes

$$\sigma_\theta = (2\sigma_{x,y,t}/z)\,(\theta + 1)^{1/2} . \qquad [13]$$

If a tare compensation device is used so that each water content determination involves only two weighings, Eq. [6] becomes

$$\sigma^2_{x/y} = (1/y^2)[\sigma^2_x + (x/y)^2\,\sigma^2_y] , \qquad [14]$$

and Eq. [9] is not needed, since the tare weight is not obtained independently. However, if the tare compensation involves a constant bias, this does affect the accuracy of the resulting water content figure. The effect on water content values of a constant bias due to a tare error or other causes, which may be either positive or negative, may be obtained by solving explicity for E in the equation

$$[(x + e)/(y + e)] - 1 = \theta + E \qquad [15]$$

which, when combined with $\theta = (x - y)/y$, reduces to

$$E = (-e\theta)/(e + y) \qquad [16]$$

or when e is small compared to y, it reduces to

$$E = -\theta(e/y) . \qquad [17]$$

If the ratio e/y does not exceed 0.001 (e.g., 100 m[illegible]0 g, 10 mg/10 g

09-29-03 14:22 From-EES-10 +5056653866 T-453 P.008/029 F-704

or 1 mg/1 g), the error will not exceed 0.1% water content up to water contents of 100%. If the ratio e/y does not exceed 0.0001, the error will not exceed 0.01% water content over a similar range. This applies to any constant bias of a balance as well. The random error when tare compensation is used may be obtained from Eq. [14] using a similar development and with the same assumptions as used for Eq. [13]. The standard deviation is

$$\sigma_\theta = \sigma_{x,y}\,[1 + (\theta + 1)^2]^{1/2}/y \qquad [18]$$

which, when θ^2 is regarded as negligible, reduces to

$$\sigma_\theta = (2)^{1/2}\,\sigma_{x,y}\,(\theta + 1)^{1/2}/y \qquad [19]$$

when tare compensation is used.

Balance precision and accuracy are reported in various ways by manufacturers, and the meaning of the terminology used is not always clear. Hence, it would be difficult to provide general instructions for using balance ratings as provided by the manufacturer to compute the variance or standard deviation in a water content measurement. However, the standard deviation for a particular balance at a specified load may be obtained experimentally with small effort. Hence, water content measurement errors are given here in terms of standard deviations. (For statistical methods for computing standard deviations, consult an elementary book on statistical analyses or books on chemical analyses such as *Chemical Computations and Errors* by Crumpler and Yoe (1946).)

If the limit of precision in water content measurement E due only to random weighing errors is taken to be 3σ, then from Eq. [13],

$$E = 3\sigma_\theta = 6\sigma_{x,y,l}\,(\theta + 1)^{1/2}/z \qquad [20]$$

where a tare weighing is involved. At 3σ, 99.7% of the measurements would be within $\pm E$; at 2σ, 95%; and at σ, 68%. If the dry weight z in Eq. [11] is replaced by the wet weight, $z' = z/(\theta + 1)$, then Eq. [20] (again neglecting θ^2 and higher powers) becomes:

$$E = 6\sigma_{x,y,l}\,(\theta + 1)^{3/2}/z' = 6\sigma_{x,y,l}\,(3\theta + 1)^{1/2}/z'\,. \qquad [21]$$

Neglect of θ^2 and higher powers leads to E values that are slightly too small for large values of θ in Eq. [20] and [21]. However, the magnitude is correct, and the practical value of the expressions is enhanced by their simplicity. In cases where tare compensation is used, the equation corresponding to Eq. [20] is

$$E = 3\sigma_\theta = 3(2)^{1/2}\sigma_{x,y}(\theta + 1)^{1/2}/y = 4.24\sigma_{x,y}(\theta + 1)^{1/2}/y\,. \qquad [22]$$

A balance should be read to $\pm\,\sigma$. (The precision of some balances is

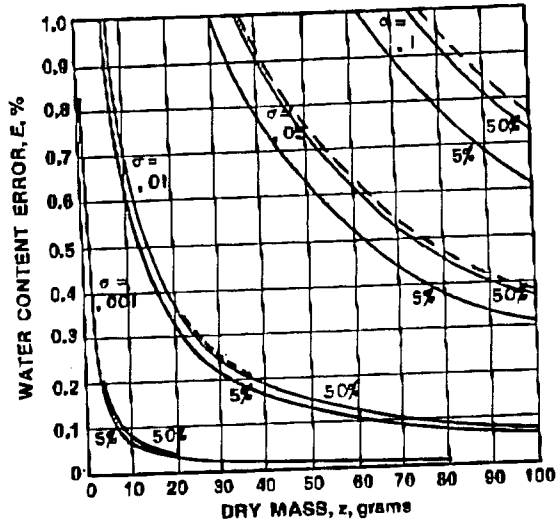

Fig. 21-2. Error in water content measurements as a function of the dry mass of the sample at various weighing precisions and for 5 and 50% water contents on a dry-mass basis (Eq. [20]). The value of E is $3\,\sigma_\theta$, and the weighing precision is the standard deviation σ for the balance in grams. When tare compensation is used, E should be multiplied by $2^{1/2}/2$, and where n replicate determinations are made, by a factor of $1/n^{1/2}$. If z is taken as the wet mass of the sample, the error E from the graph should be multiplied by $(\theta + 1)$, as can be seen from Eq. [21]. The dashed curves, for 50% water content, result when the θ^2 term in Eqs. [11] and [18] is not ignored.

reported as $\pm e$, where e is taken to be 3σ. The Metler K-7 is of this type, the precision being given as $\pm$ 0.03 g. The standard deviation at loads of approximately 100 g was found experimentally to be about 0.01 g on four different balances of this type. The bias of each balance was $< \pm 0.01$ g at the same loading.)

The precision measurement may be improved by a factor of $1/n^{1/2}$ by making n replicate determinations. Values of σ for various balance standard deviations $\sigma_{x,y,z}$, sample dry masses E, and water contents are shown in Fig. 21-2. When tare compensation is used the value of E should be multiplied by $2^{1/2}/2$, and where n replicate determinations are to be made, by a factor of $1/n^{1/2}$.

21-2.2 Gravimetry With Oven Drying

21-2.2.1 INTRODUCTION

Water content measurements by gravimetric methods involve weighing the wet sample, removing the water, and reweighing the sample to determine the amount of water removed. Water content then is obtained by dividing the difference between wet and dry masses by the mass of the dry sample to obtain the ratio of the mass of water to the mass of the dry soil. When multiplied by 100, this becomes the percentage of water in the sample on a dry-mass (or, as often expressed, on a dry-

09-29-03 14:22 From-EES-10 +5056653866 T-453 P.009/029 F-704

weight) basis. Water content may be described in other ways as indicated in section 21-1. Water may be removed from the sample in any of a number of ways, the principal method in common use being the oven-drying method described here. Accuracy and reproducibility of water content measurements, assuming that weighing precision is consistent with desired precision of water content measurement, depend upon the drying technique and the care with which it is used. (See discussion in section 21-2.1).

21-2.2.2 METHOD

21-2.2.2.1 Special apparatus. Apparatus required for gravimetric determination of water content may be used in many different forms, and so exact specifications are not needed. Requirements include an auger or sampling tube or some other suitable device to take a soil sample, soil containers with tight-fitting lids, an oven with means for controlling the temperature to 100 to 110°C, a desiccator with active desiccant, and a balance for weighing the samples. In the field, if soil samples are taken under conditions where evaporation losses may be of sufficient magnitude to affect the desired accuracy of measurement, special equipment for weighing the samples immediately or reducing evaporative loss must be used. Both convective and forced-draft ovens are used, and for precise work a vacuum oven is of particular value. Balances used range all the way from analytical balances to rough platform scales, depending upon the size of the sample to be taken and the precision of measurement desired.

21-2.2.2.2 Procedure The procedure to be used must vary with the circumstances of measurement and the equipment. Since these vary widely it is impossible to specify a detailed standard procedure that will fit all of the many uses made of water content measurements. The procedure given here is intended for use in routine work where moderate precision (say, measurements having a precision of ± 0.5% water content) is desired. Replication must depend upon the nature of the sample and soil system for which water content is desired, but it is suggested that samples be run in duplicate as a minimum.

Place samples of 1 to 100 g of soil in weighing bottles or metal cans with tight-fitting lids. Weigh the samples immediately, or store them in such a way that evaporation is negligible. Refer to Fig. 21-2 to find the required weighing precision. (The balance need not be read to a precision greatly exceeding the standard deviation for the balance.) Place the sample in a drying oven with the lid off, and dry it to constant weight. Remove the sample from the oven, replace the cover, and place it in a desiccator containing active desiccant (e.g., magnesium perchlorate or calcium sulfate) until cool. Weigh it again, and also determine the tare weight of the sa[illegible] container. Compute the water content by one of the following for[illegible]las:

$$\theta_{dw} = \frac{(\text{weight of wet soil + tare}) - (\text{weight of dry soil + tare})}{(\text{weight of dry soil + tare}) - (\text{tare})} \quad [23]$$

$$= \frac{(\text{weight of wet soil + tare}) - (\text{tare})}{(\text{weight of dry soil + tare}) - (\text{tare})} - 1 \quad [24]$$

$$= \frac{\text{weight of wet soil}}{\text{weight of dry soil}} - 1. \quad [25]$$

The third of these equations is useful where standardized cans are usec and the tare weight is balanced out in the weighing process so that th sample weight is obtained directly. Multiplication by 100 gives the per centage of water in the sample on a dry-mass basis.

21-2.2.2.3 Comments. The time necessary to reach constant weigh (the term being loosely used here, since constant weight rarely is obtainec except for very sandy soils containing little or no organic matter) wil depend upon the type of oven used, the size or depth of the sample, anc the nature of soil. If a forced-draft oven is used, 10 h usually is considerec sufficient. If a convection oven is used, samples should be dried for a least 24 h, and precautions should be taken to avoid adding wet sample: during the last half of the drying period. Also, additional time should b added if the oven is loaded heavily. Water contents for samples that ar to be compared should be determined using precisely the same methoc for each measurement. For more precise work, other considerations ar involved, which are discussed below.

An alternative method may be used for drying soil. Radiation-dryin using an infrared or ordinary heat lamp, often in association with a built in balance, can be used for soil water content measurements where lov precision is adequate. Several such instruments containing a built-in in frared heat lamp, a torsion or analytic balance, and a scale for direc reading of wet-mass basis water contents are available from scientifi supply houses under the name of "moisture determination balance."

The uncertainty of the drying temperature makes radiation-dryin methods less accurate than those using closely controlled constant-tem perature drying ovens. However, the method is rapid, requiring only few minutes to dry the soil; and when the built-in balance is used, wet mass basis water content values ordinarily may be read directly from scale. These may be converted to dry-mass figures by Eq. [1]. When usin radiation-drying, care should be taken to avoid excessive heating of th sample.

Water content values for stony or gravelly soils, both on a mass anc volume basis, can be grossly misleading. The problem arises from th fact that a large rock can occupy appreciable volume in a soil sample anc contribute appreciably to the mass without making a commensurate con tribution to the porosity or water capacity of the soil. Mass-basis wate content figures are lower than corresponding values for a soil on a rock free basis because of the excessive contribution to t[illegible]ry mass made b

08-29-03 14:22 From-EES-10 +5056653866 T-453 P.010/029 F-704

a rock which may have a bulk density of about 2.6 g cm^{-3} compared to that of the finer soil material, which will usually range from 1.0 to 1.6 g cm^{-3}. A mass-basis water content figure of 10% based upon the dry mass of a gravelly or stony soil having a bulk density of 2.0 g cm^{-3} would represent a water content of 20% if based upon the dry mass of the finer fraction with a bulk density of 1.6 g cm^{-3}.

It is important when presenting water content data on gravelly or stony soils to specify the basis of measurement—particularly the size fraction on which it is made. Water retention in stony soils also is discussed in chapter 26.

The two types of water content figures of greatest interest are water content per unit bulk volume and water content per unit mass of the fine fraction. The volume-basis figure makes it possible to compute the volume of water per unit area in, say, a root zone. The mass-basis figure for the fine fraction is usually the figure obtained from gravimetric analyses or from a wilting-point water-content matric potential determination, and is the figure which would ordinarily be used to compare water conditions from place to place in a soil. The relationship between these two types of water content values is

$$\theta_{vb} = (\theta_{dmf}\rho_b/\rho_w)/(1 + M_{stone}/M_{fines}) \qquad [26]$$

where θ_{vb} is the volume of water in a unit volume of the whole soil, θ_{dmf} is the water content on a dry-mass basis for the fine fraction, ρ_b and ρ_w are the bulk densities of the whole soil (including stones) and of water, and M_{stone} and M_{fines} are the dry masses of the stone and fine fractions.

Water content on a volume basis or bulk density may be determined by taking a sample of known volume, oven-drying it according to procedures already described, and dividing the difference between wet and oven-dry mass by the volume to give water content, or dividing the oven-dry mass by the volume to give bulk density. The heterogeneity of gravelly and stony soils and the variability that usually exists from point to point in the soil make for low precision. Because of this low precision, it is possible to discard large stones prior to oven-drying without greatly affecting the precision. Rocks and stones to be discarded are carefully and quickly brushed to avoid soil loss and to reduce evaporation loss, and then are weighed.

Conventional cylindrical tube samplers may be used in some gravelly and stony soils. However, as the number or size of stones increases, the utility of such sampling devices diminishes. In these kinds of soils it is important to determine the volume sampled each time a water content determination is to be made. Where core-type samplers can be used this is not difficult. However, where large rocks and stones interfere seriously, other methods must be used. One useful method involves sampling with a spade or shovel and determining the volume of the hole which is dug.

The volume may be measured by placing a rubber or plastic membran in the hole and filling it with water from a container filled to a know volume. The quantity of water used in filling the hole is determined easily Grain millet or dry sand, which flow easily and pack easily to constan bulk density, also may be used to fill the hole; and if the material is t be discarded, the rubber or plastic membrane is not needed. A descriptio of the method is given in chapter 13. No simple, inexpensive method have as yet been developed for sampling beyond shallow depths withou digging an access hole.

Certain general requirements must be met in the development of procedure for obtaining accurate and reproducible water content mea surements. Foremost of these is the requirement that the sample be drie at a specified temperature to constant weight with nothing being lost bu water. This rarely is possible with a colloidal material like soil, partic ularly if it contains any appreciable organic matter, as is discussed earlie in the section on general principles (21–2.1). Weight losses during dryin at 100 to 110°C for periods as long as 15 days have been observed i soils ranging from fine sands to silty clay loams. Because of this it i important to specify the details of the drying procedure used in reportin water contents where precise values are needed.

Since accuracy in water content determinations hinges upon existenc of a definable dry condition which, with soil, can only be based upo subjective judgment, it is more appropriate to refer to reproducibilit than to accuracy. Reproducibility in water content measurements can b achieved in two ways: (i) treating every sample of a set to be compare exactly the same way in terms of such things as sample size and dept in the container, drying temperature, and drying time; or (ii) followin techniques that lead to equilibria which are as nearly independent of suc variables as is possible. From the latter point of view, the nature of th thermal dehydration curves (Fig. 21–1) suggests the desirability of choos ing a drying temperature in a region where the weight change of th colloidal constituents with temperature is at a minimum, as is discusse in section 21–2.1. Also, as is discussed in the same section, vacuum dryin at relatively low temperature, with the temperature being controlled care fully and specified when reporting, probably provides the most repr ducible drying data.

Reproducibility in water content measurements also may depen upon the technique used to avoid absorption of water from the air durin cooling and prior to weighing. While absorption may be negligible in dr ambient conditions or for low-precision work, in certain mineralogic studies or studies of other types, where small weight changes may b confounding, an unacceptable error may be introduced. A method i volving flame-sealing of small vials containing the soil prior to coolin has been described by Kittrick and Hope (1970).

09-29-03 14:22 From-EES-10 +5056653866 T-453 P.011/029 F-704

21-2.3 Gravimetry With Microwave Oven Drying

21-2.3.1 INTRODUCTION

An alternative to conventional oven-drying of soil (and of plant materials, as well) involves heating by means of microwave energy, which will penetrate into and vaporize water throughout a soil sample. Although not designed specifically for highest efficiency in drying soil samples, commercial microwave ovens, using 0.9- to 300-GHz frequencies in the electromagnetic spectrum, may be used. Polished metals reflect microwave energy, but heat is generated to some degree in most materials by absorption of microwave energy, and to a considerably greater degree in polar water than in most soil materials. Thus, soil water temperature quickly is raised to the boiling point where, because of consumption of heat in vaporizing the water, it remains until absorption of microwave energy by the soil and water exceeds the rate of energy consumption by vaporization. At this time the temperature rapidly rises. If heating is discontinued after the absorbed water is gone and before soil temperature rises to a point where organic matter is appreciably oxidized or structural water is driven off, the dryness achieved should match that achieved using conventional oven-drying. However, this is not easily accomplished and it constitutes the most serious obstacle to achieving accuracy and reproducibility in microwave drying.

The amount of microwave energy absorbed, which determines drying time needed, depends upon the quantity and absorptive characteristics of the material being dried, and hence upon the nature and quantity of energy-absorbing materials present and their water content. And since a flat plateau does not exist in the water loss vs. time curve for microwave drying (Fig. 21-3) (as is assumed and is approximately true for most practical measurements using conventional ovens where temperature is held constant), strictly speaking only a single sample—or duplicate samples of the same soil and same size and assumed to have nearly the same

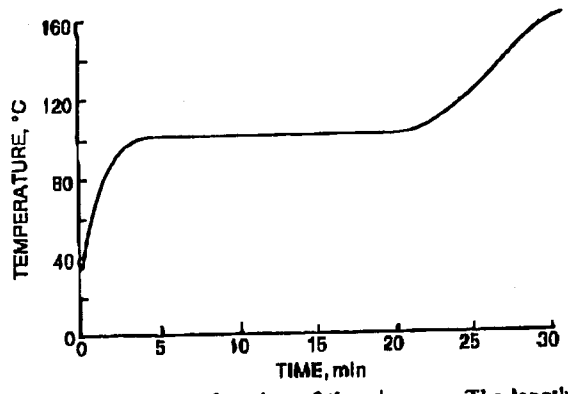

Fig. [illegible] Sample temperature as a function of time in oven. The length of the plateau v[illegible]dth sample size, water content, and soil type.

water content—may be dried during a single oven run. However, for many purposes errors associated with temperature rise above the standard drying temperature may not always reduce the accuracy and reproducibility of a moisture content determination beyond acceptable limits (Gee and Dodson, 1981; Gilbert, 1974; Hankin and Sawhney, 1978; Miller et al., 1974). Curves shown in Fig. 21-4 illustrate something of the nature of the problem. Here, samples of soils at several different initial water contents have been removed from the microwave oven at the end of different drying periods and the water content computed under the assumption that the sample is dry. After drying periods ranging from 6 to 20 min the computed water content tends to approach a constant value, which may be taken as the correct initial water content. It is evident that selection of an unequivocal value is not always possible.

21-2.3.2 METHOD

21-2.3.2.1 Special Apparatus. A household microwave oven may be used. Preferably the oven will have a uniform microwave field over the load surface, which is achieved by means of a rotating microwave reflector or by a rotating loading platform, or possibly by both. It also will have an automatic shut-off switch, which operates when the oven load, as a consequence of drying, is reduced and excessive microwave energy is reflected back to an absorbing heat sensor.

Glass or paper cups holding about 25 g of soil are required. Some plastic materials can be used, but many plastics melt at temperatures reached by the container or its sample. Where saturated or near-saturated samples are to be dried, a lid with a small vent may be used to prevent loss of sample from boiling.

21-2.3.2.2 Procedure. The procedure to be used must vary with type

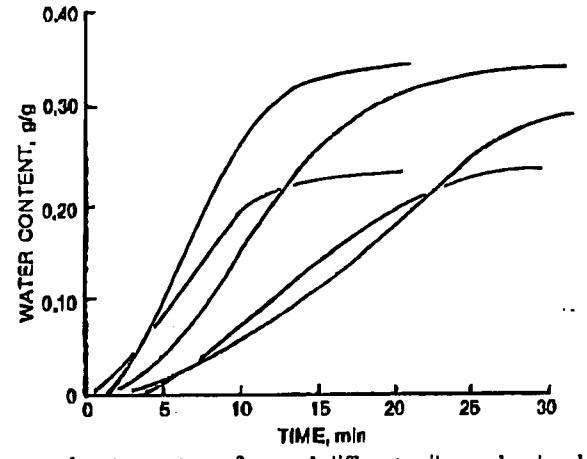

Fig. 21-4. Computed water content of several different soil samples, based upon sample weight reached at various times in the oven. These curves vary widely with sample size, water content, and soil type.

08-29-03 14:23 From-EES-10 +5056653866 T-453 P.012/029 F-704

of soil, accuracy desired, and the performance of the microwave oven to be used. Where accuracy of ± 1% is satisfactory (under some conditions this may be ± 0.5%), if the oven has previously been used for soil water content determination and found to have a fairly uniform distribution of microwave energy over the area where samples are placed, and if temperatures reached by samples are not more than about 300°C, the technique to be used is approximately the same as that for conventional oven-drying. Drying times of the order of 20 min have been used on samples of approximately 20 g, but some experience with a particular oven, sample size, and water content may be needed to determine this. Where greater accuracy is required, additional precautions must be taken. These precautions depend upon expected sources of errors, but could involve making drying tests on uniform samples to evaluate uniformity of the microwave energy field, making temperature measurements on samples of differing size and water content at different drying times, and determination of water loss vs. temperature relationships for the particular soils involved. In situ temperature measurements are difficult to make because of the absorption of microwave energy by the temperature sensing device and its possible destruction. However, satisfactory measurements may be made by inserting a low thermal mass thermocouple into the soil sample immediately upon opening the oven door.

21–2.3.2.3 Comments. Since drying time and temperature reached by the sample depend upon its size, water content, and composition (including organic matter), standardization of a drying technique for all materials and water contents is not possible. Hence, where high accuracy is important the standard of reference must be a reliable oven-drying technique, preferably with a vacuum oven having accurate and precise temperature control (see section 21–2.2.2.3). Under many conditions where drying times are chosen so that temperature always rises above the boiling point of water, the computed water contents always will be equal to or higher than the standard value for 105 to 110°C drying. Experience with a given oven, soil, sample size, and range of water content sometimes may permit subtraction of a small corrective term to reduce such bias.

Because the microwave field in a microwave oven is not completely uniform, temperatures reached by samples distributed over the oven floor also may not be uniform. As indicated earlier, small errors associated with nonuniformity in heating often may be neglected where high accuracy is not required. However, where accuracy is important some guidance is required as to the size of possible errors which may result from unequal heating. Two major sources of such errors are organic matter oxidation and removal of structural water. Both of these would result in bias so as to yield water content values that are too large. The organic matter problem exists in conventional oven-drying and is not easily resolved, except that an upper limit is defined by the proportion of the sample which is organic and subject to loss through oxidation.

As discussed in section 21–2.1, General Principles, and shown in Fig. 21–1, loss of structural water upon drying of clay minerals depends upon drying temperature, and for accurate work, drying temperature must be controlled closely. The size of errors, $\Delta\theta$, to be expected from excessive heating—which always results in a value too high—may be obtained as follows. The mass basis water content, θ_T of a wet sample, M_{wT}, dried to constant weight, M_T at a temperature, T, is $(M_{wT}/M_T - 1)$, so that

$$\Delta\theta = \theta_T - \theta_{105} = M_{ws}/M_T - 1 - (M_{ws}/M_{105} - 1)$$
$$= [(M_{ws}/M_T) - M_{ws})/M_{105})] . \qquad [27]$$

An illustration of the magnitude of possible errors which might occur with different clay minerals may be obtained using data from the temperature–water loss curves shown in Fig. 21–1. The ordinate in this figure gives the mass of a sample which would be reduced to a constant mass of unity after drying at a temperature of 800°C; thus the ordinate number minus unity and divided by unity would give the water content on a g/g/ basis, e.g., $(1.04 - 1)/1 = 0.04$, or the ordinate value is $(\theta_{800} + 1)$. Multiplication of Eq. [27] by M_{800}/M_{800} permits rewriting this equation as

$$\Delta\theta = [(M_{800}/M_T) - (M_{800}/M_{105})]M_{ws}/M_{800}$$
$$= [1/(\theta_{T,800} + 1) - 1/(\theta_{105,800} + 1)](\theta_{s,800} + 1) \qquad [28]$$

where $\theta_{T,800} = M_T/M_{800} - 1$; $\theta_{105,800} = M_{105}/M_{800} - 1$ and $\theta_{s,800} = M_{ws}/M_{800} - 1$. Quantities $\theta_T + 1$ and $\theta_{105} + 1$ may be obtained from the ordinate in Fig. 21–1 for different drying temperatures and for several different clay minerals. The quantity $\theta_{s,800}$ is the sample water content based upon drying to constant weight at 800°C. The range of water contents of interest on this basis will not differ appreciably from what the range would be for 105°C drying. Assuming this range to be from about 0.05 to 0.60 g/g for illite clay the approximate error $\Delta\theta$ for drying temperatures different from the standard 105°C drying temperature may be obtained. The bracketed portion of Eq. [28] for 200 and 105°C becomes $(1/1.062) - 1/1.067) = 0.0044$. Multiplying this by $0.05 + 1$ and by $0.60 + 1$ gives 0.0046 and 0.0070 or an error range of approximately 0.46 to 0.70% water content. Similarly, computations for 300 and 105°C over the same range of water contents result in an error range of approximately 0.75 to 1.14%, and for 400 and 105°C the range is approximately 3.5 to 5.4%. For illite the water loss curve is nearly flat in the 150 to 300° range and steepens appreciably in the 300 to 500°C range.

Most common soils contain appreciable soil particles larger than clay size and which are less affected by drying at higher than usual drying temperatures. Assuming that only 1/3 of a sample is clay and 2/3 is unaffected by over-heating the error need be associated only with 1/3 of the sample so that the error range for 200°C becomes 0.15 to 0.23%; for

08-29-03 14:23 From-EES-10 +5056653866 T-453 P.013/029 F-704

300°, 0.25 to 0.38%; and for 400°, 1.2 to 1.8%. Larger errors would be found for some clay soils as may be inferred from the curves given for other clay types, particularly if higher temperatures than those considered here are reached in a part of the sample.

Further error may occur due to nonuniform microwave heating in the various mineral components of a nonuniform sample. Sizes of errors anticipated by the above computations have been found by the author and by Gee and Dodson (1981) with soils of several different types.

Special microwave equipment for automatic drying, weighing, and computation of water content is commercially available. However, most of the same limitations as discussed above apply. Furthermore, the automatic weighing feature limits measurements to single samples and a measurement time of 10 to 20 min each. Thus, these have limited usefulness in much soils work where multiple samples are required.

21-3 INDIRECT METHODS

21-3.1 Introduction

Certain physical and physical-chemical properties of soil vary with water content. However, the relationship between such properties and water content usually is complicated. Both the pore structure and constituents of the soil solution are involved in these relationships. Also, some of these properties, even under conditions where all other factors are held constant, are not determined uniquely by water content. The wetting history of the soil is a factor in many instances, particularly where water must flow into and out of a sensing device such as a porous block. Despite many limitations, some of these properties, with appropriate calibration, can be useful in characterizing soil with respect to its water content.

Wetting history often must be considered because water content of soil for a given energy status (temperature being constant) will be greater if the soil has reached a given water content by drying than by wetting. This phenomenon, known as hysteresis, is discussed in more detail in Chapter 26. For present purposes however, it is sufficient to point out that whether or not a pore becomes water-filled during a wetting process depends upon the size of the pore itself, whereas in a drying process the emptying of the pore depends upon the size of the channels connecting it with other pores in the system. It is possible, therefore, to have two soil samples with identical porosities but different water contents at equilibrium with each other if their wetting histories have been different.

Indirect methods involve measurement of some property of the soil that is affected by soil water content or measurement of a property of some object placed in the soil, usually a porous absorber, which comes to water equilibrium with the soil. The water content of a porous absorber at equilibrium depends upon the energy status of the water rather than

upon the water content of the soil with which it is in contact. For example, a soil with fine pores will contain more water than a soil with coarse pores at equal matric potential. Hence, if the properties of the porous absorber are to provide an indication of water content, calibration against the water content of a sample of the soil in which the absorber is to be used is required, together with some indication of the wetting history of the soil. Electrical or thermal properties of the absorber or weight changes in the absorber are indications of its water content. Methods involving weighing the absorber (Richards and Weaver, 1943) have not become widely used, probably because of technical complications associated with the weighing process.

Neutron scattering and neutron and gamma ray absorption are affected by water content of a porous material and may be adapted to water content measurement. Although generally requiring calibration and therefore considered to be indirect methods, under some conditions radiation measurements can be converted directly to water content on the basis of theoretical considerations. Hence, such methods might be considered to be direct methods. There are conditions where radiation water content measurements are subject to less error than gravimetric measurements. For example, where spatial variation is high and is a sizeable confounding factor in repeated gravimetric sampling, radiation measurements, which can be made repeatedly in the same soil volume, have a definite advantage.

The need for indirect methods for obtaining water content or indices of water content is evident when the time and labor involved in gravimetric sampling are considered. In addition to requiring a waiting time for oven-drying, such determinations are destructive, and therefore each sample must be taken at a different place in the soil system under study. Destructive sampling may disturb an experiment and may increase the possibility that a change in water content with position in a sampling area may be interpreted falsely as a change in water content with time at a particular location. Many of the indirect methods permit frequent or continuous measurements in the same place and, after equipment is installed, with only small expenditure of time. Thus, if a suitable calibration curve is available, changes in water content with time can be approximated.

Although this chapter is concerned primarily with measurement of water content or inferences of water content from other measurements, it should be pointed out that for many purposes water content is less useful than certain other properties of the soil-water system which depend upon water content. For example, in studies involving plant growth, matric potential in the soil has greater meaning provided that, in soils containing more than small quantities of soluble salts, a term taking into account osmotic potential is added. It is common practice to calibrate some of the indirect methods for evaluating water conditions in soil in terms of matric potential rather than water content. The subject of water

09-29-03 14:23 From-EES-10 +5056653866 T-453 P.014/029 F-704

potential is discussed in greater detail and methods for its measurement are given in chapters 23 through 26.

21-3.2 Electrical Conductivity and Capacitance

21-3.2.1 PRINCIPLES

Electrical and thermal conductivity and electrical capacitance of porous materials vary with water content. Such properties of materials can ordinarily be measured with great precision; and if a reliable correlation with water content existed, methods based upon measurement of these properties would have considerable utility. Unfortunately, such measurements made directly in soil rather than in a porous body inserted in soil have not resulted in unique correlations with water content and have not come into general use. The most thoroughly tested of the methods has involved measurement of electrical resistance (e.g., Edlefsen & Anderson, 1941; Kirkham & Taylor, 1950). Soil heterogeneity, which prevents uniform flow of current in the soil mass, and uncertain electrical contact between electrodes and soil seem to be the major obstacles to successful use of direct electrical resistance methods. Electromagnetic properties of soil measured by means of buried coaxial cables have been used for water content measurement (Topp, 1980).

Many of the problems involved in measurement of electrical and thermal conductivity and electrical capacitance in soil are avoided by use of porous blocks containing suitable electrodes and imbedded in the soil. When these blocks reach equilibrium, i.e., when water ceases to flow into or out of the blocks, their electrical or thermal properties often are regarded as an index of soil water content. However, the associated soil water content must be obtained from a calibration curve made using soil from the site where the block is used, because the equilibrium between a block and soil is a matric potential equilibrium and not a water-content equilibrium. Different soils have different water content vs. matric potential curves, variations among soils often being as much as several hundred percent. For example, a fine sandy loam may have a water content of 5% at −1.5 MPa matric potential, whereas a clay loam may have a water content of, say, 13% at the same potential. As a consequence, calibration of a porous block against matric potential often may be considered more reasonable and more useful than calibration against water content (see chapter 25).

Hysteresis enters into the problem of inferring water content from measurements made on porous blocks even though a calibration curve for a particular soil is available. The water content of both soil and block depends in part upon wetting history. Ideally, two calibration curves are needed: one for drying, extending from very wet to very dry, and one for wetting, where the starting point is in the very dry range. These two curves are considered to close at the endpoints and to provide an envelope which would contain all possible intermediate curves. However, because it is

difficult to wet a soil only part way, the wetting curve is usually not made. And in many practical situations, the starting point is unknown. Hence, the curve traced out as the soil wets or dries is unknown, and water content can be known only to lie at some point between the limiting wetting and drying curves. Resulting errors in water content inferences depend upon the nature of the soil in question and its wetting or drying history, but easily can be 20% or more (Taylor et al., 1961). Nevertheless, blocks often are used to indicate water content of soil even though the precision in such use is rather low. However, the popular use of porous blocks likely stems from their utility as indicators of water conditions favorable or unfavorable to plant growth (matric potential as opposed to water content), rather than from their ability to indicate soil water content.

Thermal conductivity and electrical capacitance measurements in porous blocks, although favorably reported on from time to time in the literature, have not come into general use (see Fletcher, 1939; Shaw & Baver, 1940; Anderson & Edlefsen, 1942; de Plater, 1955; Bloodworth & Page, 1957; Phene, et al., 1971a, 1971b). This probably results from the fact that electrical conductivity is measured easily and porous blocks for such use are easy to construct. Therefore, the most common porous-block technique involves measurement of electrical conductivity. The porous-block method which follows is for such measurements.

21-3.2.2 METHOD

21-3.2.2.1 Special Apparatus.

1. Wheatstone Bridge for measuring resistance: Bridges in common use are of the alternating current type (usually 1000 cycle) to avoid polarization at the electrodes in the porous block. Both null-point and deflection-type instruments are used. Digital read-out instruments which can be used in data acquisition systems are useful. Resistances to be measured range from a few hundred ohms to 200 000 or more ohms, a single calibration curve often covering as much as 100 000 ohms.
2. Porous blocks: Blocks now available are made of a variety of porous materials ranging from nylon cloth (Bouyoucos, 1949) and fiberglass (Coleman & Hendrix, 1949; Cummings & Chandler, 1949; England, 1965) to casting plasters (Bouyoucos & Mick, 1940; Perrier & Marsh, 1958; Cannell & Asbell, 1964; and numerous others), the most common being some form of gypsum. Various grades and kinds of casting plaster are mixed with different amounts of water and in a variety of ways, including in some instances pouring the mix into the mold in a partial vacuum. In some instances resin is added to the mix, which changes the electrical characteristics and decomposition rate in the soil (Bouyoucos, 1953). The method of preparation as well as the mix itself governs the porosity of the block and the resulting response

09-29-03 14:23 From-EES-10 +5056653866 T-453 P.015/029 F-704

curve. Some types of gypsum last longer in soil than others.

Several different electrode systems are in common use. The simplest consists of two tinned wires about 35 mm long (made using ordinary twin-conductor, rubber-insulated lamp cord). These are imbedded 1 or 2 cm apart in a rectangular porous block roughly 1 by 3 by 5 cm in size. Cylindrical blocks also are in use. These consist of a cylindrical screen (usually made of stainless steel) surrounding a central post or in some instances a second, smaller cylindrical screen. Such blocks are 2 to 3 cm in diameter and about 3 cm long. A parallel-screen system, using rectangular blocks, has been used by several investigators and is reported to have less lag in coming to equilibrium than other types of blocks.

3. Calibration container: Prepare a small screen box (window screen or hardware cloth soldered together), open at one end and of suitable dimensions to contain the block and a layer of soil at least 2 cm thick around the block (Kelley, 1944).
4. Equipment for determining a reference water content of the soil used in the calibration (section 21-2.2.2.1 or 21-2.4.2.1).

21-3.2.2.2 Procedure. Calibrate each block in soil typical of the site in which it is to be used, and with packing to about the same bulk density. To carry out the calibration, saturate the block with water, preferably with vacuum soaking, and weigh the wet block, its attached leads, and the screen box together to obtain a tare weight. Moisten the soil to be used to a state where it can be packed around the block to approximately its field density, mix the soil thoroughly, and take a sample for water content determination. Then pack the soil around the block in the screen box and weigh the entire apparatus. Using the water content determined independently by gravimetric methods (see section 21-2.2 or 21-2.3), compute the dry mass of the soil in the box. This will be

$$\text{soil dry mass} = \frac{(\text{wet mass} + \text{tare}) - (\text{tare})}{\%\ \text{water content}/100 + 1} \qquad [29]$$

where the water content figure is on a dry-mass basis. At all subsequent calibration points the water content percentage will be

$$\underset{\text{dry mass basis}}{\text{water content, \%,}} = \frac{100[(\text{tare} + \text{wet soil}) - (\text{tare} + \text{dry soil})]}{\text{dry soil}}. \qquad [30]$$

Wet the soil in the screen box to near saturation, weigh the entire assembly, and then measure the block resistance to determine the first calibration point. Allow water to evaporate from the apparatus in the air until the desired weight for the next calibration point is reached. After the desired weight is reached, place the entire apparatus in a closed container (such as a desiccator without desiccant) in the dark at uniform temperature. Leave it overnight or longer, to permit the water to equilibrate in the block and soil. Alternatively, embed the block in soil on the porous plate used in obtaining the water content vs. matric potential curve (see chapter 26). Water content then may be varied by changing the gas pressure used. After the soil comes to equilibrium water content, measure the resistance. Plot the resistance as a function of water content. (Three-cycle semilogarithmic paper is convenient.)

To install a block in the field, form a hole vertically from the surface or horizontally in the side of a trench. Wet the block thoroughly, place it in the hole, and then pack soil around it to assure good contact with the surrounding soil. Bring the leads to the surface, running them horizontally for a short distance beneath the surface to assure that no continuous channel exists along the leads for passage of free water. After equilibrium is reached, usually overnight, make resistance measurements as desired and convert them to water content values with a calibration curve.

21-3.2.2.3 Comments. In calibration as well as under field conditions, true water content equilibria rarely are reached, particularly in the dry range. However, the uncertainty of the water content inference, at best, does not justify an elaborate and time-consuming calibration. Under practical conditions, when the resistance reading approaches a constant value, equilibrium may be assumed to be close. In the wet range, for most porous blocks, the resistance change with changing water content is small and the precision is low. Precision also is affected by changes in the calibration curve over successive wetting and drying cycles (Cannell, 1958).

Restricted water flow at the interface between the smooth face of a porous block and coarse soil materials creates some problems in the use of blocks in sandy soil. However, coating a block with a porous material such as diatomaceous earth can reduce this problem appreciably. An envelope of fine-grained material placed around tensiometer cups (A. Cass and G.S. Campbell. Tensiometer response in coarse grained soils, Western Society of Soil Science, Eugene, OR, June 1981 unpublished), has produced a significant improvement in interface conductivity. Nonetheless, some problems do exist in use of blocks in coarse soils where flow of water is restricted by low unsaturated conductivities.

It is common practice to place blocks into uniform groups according to their resistance at saturation and to calibrate only selected blocks from each group (Tanner et al., 1949). This practice does not completely ensure obtaining groups of blocks with like calibration curves; but considering the low precision of the method when it is used for water content determination, it probably is an adequate procedure.

The calibration procedure described is for desorption. The procedure cannot be reversed easily to provide a sorption curve because of difficulties associated with partially wetting a soil mass. However, two tedious processes have been used in the author's laboratory to provide sorption curves in the dryer part of the water content range where wetting is

09-29-03 14:24 From-EES-10 +5056653866 T-453 P.016/029 F-704

difficult. One involves condensing water into soil from the air under carefully controlled temperature and humidity at the water potential required to provide the desired water content. The second involves thorough mixing of finely pulverized ice in appropriate quantities into dry soil at low temperatures and then slowly warming to melt the ice. Control of pore size distribution affecting wet range water content is difficult in the latter method.

Calibration often is carried out in the field by obtaining water content values gravimetrically and plotting them against resistance readings. Or, blocks are placed in pots containing growing plants and the water content determined by weighing the pot, with an estimate of plant weight being subtracted (Cannell, 1958).

Electrical conductivity of a porous block depends upon the electrolyte concentration of the conducting fluid as well as upon the cross section of this fluid or water content. In a porous block made from an inert material, the electrolytes that carry the current come from the soil solution. Even a small change in electrolyte concentration will influence the resistance. In blocks made from gypsum the electrolyte concentration corresponds primarily to that of a saturated solution of calcium sulfate. Variations in the soil solution due to fertilization have relatively little influence upon the electrolyte concentration in such blocks and therefore relatively little influence on resistance. Such blocks also may be used without serious difficulty in slightly saline soils (where soil extract conductivities are less than approximately 2 mmho/cm^2 (Taylor, 1955).

Blocks made from gypsum compounds gradually deteriorate in soil, particularly in sodic soils and in soil where the water table frequently is at high levels. However, those made from hydrocal have lasted for upwards of 6 years in some soils. Blocks made from ordinary plaster of paris have been known to deteriorate in a single season beyond the point where they can be used.

It is difficult to specify an expected precision for water content measurements using electrical conductivity blocks because of the many sources of error involved. The precision depends not only upon the care used in manufacture, selection, and calibration of blocks but also upon factors of hysteresis which are out of the control of the operator. However, it appears that precision better than +2% water content should not be expected and that errors as great as 100% easily are possible. On-the-site checking as the blocks are used appears necessary if confidence is to be developed in water content inferences to be made. On the other hand, where porous blocks are used as a measure of matric potential rather than water content, considerably better performance is possible. Calibration against matric potential may be carried out using porous-plate and pressure-membrance equipment (chapter 25) with special pass-through electrical contacts.

21-3.3 Neutron Thermalization

21-3.3.1 PRINCIPLES

Hydrogen nuclei have a marked property for scattering and slowing neutrons. This property is exploited in the neutron method for measuring water content. High-energy neutrons (5.05 MeV) emitted from a radioactive substance such as radium–beryllium or americium–beryllium [^{9}Be(α,n)^{12}C] are slowed and changed in direction by elastic collisions with atomic nuclei. This process is called *thermalization*, the neutrons being reduced in energy to about the thermal energy of atoms in a substance at room temperature.

Neutrons interact with matter in two general ways: by elastic and inelastic scattering, and by interactions leading to capture with a consequent emission of energy or of other nuclear particles. The probability of any particular interaction depends upon neutron energy and characteristics of the nuclei encountered. These characteristics are described generally by the nuclear cross section, measured using a unit of area called the "barn," which is 10^{-24} cm^2. The larger the probability of a particular interaction, the larger is the nuclear cross section.

The two major factors involved in scattering and slowing of neutrons are the transfer of energy at each collision and statistical probability of collision.

The average energy transfer at collision of a neutron with other nuclei depends largely upon the mass number of the nuclei encountered. The average number of collisions required to slow a neutron from 2 MeV to thermal energies is 18 for hydrogen, 67 for lithium, 86 for beryllium, 114 for carbon, 150 for oxygen, and $9A + 6$ for nuclei with large mass numbers A (Weinberg & Wigner, 1958, Table 10.1).

The statistical probability of collision is dealt with using the concept of "scattering cross section," which is a statistically derived, cross-sectional area measured in barns which is proportional to the probability of collision—in this case, between neutrons and other nuclei. This scattering cross section depends upon the nature of the nuclei encountered and the energy of the neutron. The scattering cross section for hydrogen varies from about 1 barn at 10 MeV to about 13 barns at 0.1 MeV. The cross section varies considerably as the neutron continues to lose energy, but is somewhat higher in the thermal energy range. Other elements found in soil with appreciable scattering cross sections (2–5 barns) are beryllium, carbon, nitrogen, oxygen, and fluorine.

Considering both energy transfer and scattering cross-section, it is evident that hydrogen, having a nucleus of about the same size and mass as the neutron, has a much greater thermalizing effect on fast neutrons than any other element. In addition, when both hydrogen and oxygen are considered, water has a marked effect on slowing or thermalizing neutrons. This is particularly true in the thermal range.

09-29-03 14:24 From-EES-10 +5056653866 T-453 P.017/029 F-704

The quantity of hydrogen in the soil ranges from near zero for dry coarse sand to as much as 8% of the mass of a fine-textured soil with 50% water content when structural water (see section 21–2.1) is included in the computation. Most of the hydrogen in soil is associated with water, and lesser amounts with organic matter.

As fast neutrons lose energy and become thermalized, another nuclear-matter interaction becomes increasingly important—neutron capture with the release of other nuclear particles or energy. Of the elements usually present in soil in quantities of 1% or greater, the capture cross-section for thermal neutrons is greatest for iron (2.53 barns) and potassium (2.07 barns). The other most common elements in the soil, silicon, aluminum, hydrogen, carbon, and oxygen, have capture cross-sections of 0.16, 0.23, 0.33, 0.003, and 0.0002 barns. Several elements present in soil in small (or even minute) quantities, such as cadmium with an absorption cross section of 2450 barns, boron with 755 barns, lithium with 71 barns, or chlorine with 34 barns, can have an appreciable effect on neutron capture.

When a fast neutron source is placed in moist soil it immediately becomes surrounded by a cloud of thermal neutrons. The density of this cloud represents an equilibrium between the rate of emission of fast neutrons, their thermalization by nuclei such as those of hydrogen, and their capture by absorbing nuclei, as determined by their concentration and capture cross-section.

The scattering cross-section and the concentration of hydrogen nuclei determine the distance from the source a fast neutron must travel before making a sufficient number of collisions to become thermalized. The farther a neutron travels from the source the larger the volume which will be occupied by thermal neutrons and the lower their density. With the number of slow neutrons involved, the absorption capacity of the soil for neutron capture is essentially infinite, and the rate of capture depends only upon thermal neutron concentration and the combined capture cross-section of the elements in the soil. If the capture cross-section, except for that due to water, remains constant (i.e., chemical composition constant), then the thermal neutron density may be calibrated against water concentration on a volume basis. Thermal neutron density is easily measured with a detector, insensitive to fast neutrons, which is placed in the vicinity of the fast neutron source. Thermal neutrons interact with a boron trifluoride gas in the detector, releasing an alpha particle, which is attracted to a negative high-voltage electrode within the detector. This creates a short electrical pulse, which registers as a count in an associated electronic scaling unit. The source usually is placed at the bottom of the detector tube or against the side or as an annular ring about the detector. This probe can be lowered through an access hole into the soil and measurements obtained for conversion to water content (Belcher et al., 1950; Gardner & Kirkham, 1952).

The nature of the neutron-scattering and thermalization process imposes an important restriction on the resolution of water content measurements. The volume of soil involved in the measurement will depend upon the concentration of scattering nuclei and thus largely upon water content, and upon the energies of the emitted fast neutrons. The strength of the neutron source affects the thermal neutron density and is involved in the counting statistics, but does not affect the range of the fast neutrons. Experimental work with neutron sources indicates that the practical resolution ranges from about a 16-cm radius at saturation to 70 cm at near zero water content (Van Bavel et al., 1956). The radius of the sphere of influence accounting for 95% of the neutron flux which would be obtained in an infinite medium is given by Olgaard (1965) in an empirical equation as

$$R = 100 \text{ cm}/(1.4 + 10\, m) \qquad [31]$$

where m is the water content in g/cm^3. However, the parameters in the equation may be expected to change somewhat with differences in chemical composition of the soil. Lack of high resolution makes it impossible to detect accurately any discontinuity or sharp change in water content gradient in a soil profile (McHenry, 1963). In particular, measurements close to the soil surface are unreliable because of the discontinuity at the interface between soil and air; and measurements usually are not made with well-type equipment any closer than about 18 cm from the surface. Therefore, water-content distribution curves for soil profiles containing steep water-content gradients will be rounded and inaccurate in detail; however, they are likely to be of sufficient accuracy for many practical uses. In particular, water content *changes* within a profile can be obtained with considerably greater accuracy than is possible in the determination of the profile water content itself.

Surface probes, in which the slow-neutron detector is laid horizontally on the surface of the soil with the fast-neutron source beside it, make it possible to obtain water content measurements in surface soil where the well-type unit is inadequate, but with considerably less accuracy. Most surface probes involve use of a moderator rich in hydrogen (such as paraffin or polyethylene) over the top of source and detector to compensate partially for the discontinuity at the interface between soil and air. Experimental work with surface units indicates a sensitive depth of from 15 to 35 cm (Van Bavel et al., 1961; de Vries & King, 1961; Phillips et al., 1960). Where water content of the surface soil is not uniform, say under conditions of rapid surface evaporation or superficial wetting by low rainfall, or where the surface of the soil is rough, precision falls off materially (Van Bavel et al., 1961).

In theory it should be possible to determine the density of thermalized neutrons in the vicinity of a fast-neutron source from the chemical composition of the soil in the absence of water, and then to relate changing water content to increases in this thermalized neutron density as mea-

09-29-03 14:24 From-EES-10 +5056653866 T-453 P.018/029 F-704

sured with an appropriate detector. However, the several types of neutron-matter interactions, together with the many chemical constituents of soil and variations in bulk density (Holmes, 1966; Lal, 1974; Greacen & Hignett, 1979), make this impractical and some form of calibration becomes necessary. Some success has been achieved through laboratory calibration of soil materials (Vachaud et al., 1977). Although still difficult, calibration is somewhat simplified by the fact that for most commercial neutron source–detector units the calibration curve relating thermal neutron density to water content is essentially linear over the range of interest, so that the calibration equation or curve may be defined by as few as two points if they are available with sufficient accuracy. Further simplication, although the calibration process remains difficult, is associated with the fortuitous coincidence that some differences in composition and texture among soils are compensated partially by differences in the neutron-matter interactive processes, so that a single calibration curve may approximately fit a small group of soils of similar chemical composition or a soil which varies with depth or spatial distribution. However, for high accuracy or under conditions where a soil is suspected to deviate markedly from the normal, extensive calibration procedures may be required. See Van Bavel et al. (1961), Holmes and Jenkinson (1959), Stolzy and Cahoon (1957), Holmes (1956), Olgaard (1965), Sinclair and Williams (1979), Zuber and Cameron (1966), Vachaud et al., (1977), and Lal (1974) for these procedures.

The quantity of hydrogen in soil, apart from absorbed water, depends upon organic matter content and the nature of the mineralogical components. Certain clay minerals contain appreciable structural water (see section 21-2.1), which is not removed by oven-drying at 100 to 110°C. For several minerals (Fig. 22-1) as much as 20% additional water can be removed by heating to about 800°C. On the other hand, coarse sands composed of quartz or feldspars have almost no water associated with them at 100 to 110°C. It is significant that the construction of many calibration curves for neutron water-content equipment has involved use of sand to provide some water contents and loams and clays for other water contents, often without apparent difficulty. The explanation for this apparent anomaly possibly involves a nearly perfect balance between increased scattering due to hydrogen in organic matter or in the structural hydrogen of clays, which tends to increase thermal neutron density, and increased neutron capture associated with a different chemical composition, which tends to decrease thermal neutron density. Clay materials that retain large amounts of structural water also are known to contain higher concentrations of such elements as boron, lithium, chlorine, and iron, which are good neutron absorbers compared to the elements composing sands. Evidence that small quantities of good neutron absorbers can affect a calibration curve has been obtained by Holmes and Jenkinson (1959). They added boron to a soil at rates of 65, 156, and 245 mg/kg and noted that increasing boron concentration decreased the slope of the

counting-rate vs. water-content curve. It is evident that the right amounts of neutron absorbers could compensate for increased neutron-scattering due to organic or structural hydrogen in a soil. It also is evident that a soil containing uncommon excesses of such elements as boron, chlorine, or iron could have a different calibration curve than that for a more normal soil. An additional fact which must be acknowledged in the calibration problem is that tightly bound hydrogen nuclei interact differently with neutrons than do hydrogen nuclei of water.

21-3.3.2 METHOD

21-3.3.2.1. Special Apparatus.

1. Neutron moisture depth probe and meter consisting of a source of fast neutrons (usually americium-241/beryllium), a detector for thermalized neutrons, a protective shield composed of lead (for gamma ray absorption) and polyethylene or paraffin (for neutron absorption) which serves also as a reference standard, and a scaler for registering counts or, in conjunction with a built-in computer, a meter for direct display of water content. The neutron source usually is either a small capsule located on the side of the detector cylinder or an annular ring placed around the detector. Such units are commercially available with several different neutron-source strengths and arrangements. Separate gamma ray density units for back-scatter bulk density measurements or a density probe combined with a moisture probe also are available commercially. A separate neutron moisture meter for use on surface soil also is available.
2. Soil auger for installing access tubing, slightly smaller than the tubing to assure a tight fit.
3. Thin-wall aluminum, steel, or plastic access tubing. Several sizes of tubing are used, but the size should be consistent with the probe size to reduce errors associated with an air gap, which may be created between the probe and tubing wall.
4. Calibration curves, or calibration parameters for units involving an integral computer, and a moisture content read-out.
5. Film badges, leak test-kits.
6. State or federal license, as may be required.

21-3.3.2.2 Procedure.

1. Use the soil auger to form the hole for installation of the access tubing. In many soils, particularly where loose materials are present, it is advisable to drill the access hole through the access tubing, advancing the tubing little by little as the hole is drilled. This is important because of the influence that air space in the vicinity of the access hole has upon the thermalized neutron distribution and consequently the neutron count. The access tubing usually is left so as to protrude about 10 cm above the soil surface and is covered with an empty can or

09-29-03 14:24 From-EES-10 +5056653866 T-453 P.019/029 F-704

stopper between readings to keep water and debris out. In situations where water might enter, a tight cap or rubber stopper may be necessary at the lower end of the tube.

2. To make a measurement, place the probe unit over the access tube preparatory to lowering it into the hole. Select an appropriate counting time and make several standard counts while the probe is in the shield. The measurement used then will be the ratio of the count taken at a particular position of the probe to the average of counts taken in the standard. This will correct for any electronic changes in the counting circuits which otherwise might confound the measurement.
3. Make one or more counts at each selected depth in the profile. Since the zone of influence for 5-MeV neutrons is roughly spherical with a radius of about 15 cm in wet soil and 70 cm in dry soil, the depth increment should be no greater than 15 cm.
4. Use the calibration curve to reduce count ratios (count in soil/count in standard shield) to volumetric water content, or read water content directly if the equipment has the required built-in computer.
5. The procedure for use of the surface probe is comparable, except that precautions must be taken to assure a smooth surface so that air gaps are not present between the surface probe and the soil.

21-3.3.2.3. Comments. With reasonable attention to safety rules supplied by the manufacturer, the health hazard involved in using the equipment is small. The important precautions are the following: (i) Keep the probe in its shield at all times except when it is lowered into the soil for measurement. (ii) Reduce exposure to the small amount of radiation escaping the shield by keeping several feet away, except when changing the position of the probe, and by keeping the open end of the probe and shield pointed away from personnel. (iii) Carry the probe in the field on a cart or on a sling between two persons if more than a few minutes is involved in getting the equipment to a position for use. (iv) Transport the probe in the back of a truck, a car trunk, or for short periods in the unoccupied rear seat. (v) Have operators wear a film badge at waist level. (vi) When the probe is not in use, lock it in a storage room. Label the container plainly to indicate radioactive content. (vii) Have a leak test performed on the source by a competent safety officer semiannually, or as may be prescribed by the radiation license (the manufacturer can advise on this). (viii) See that probe maintenance is performed only by personnel trained in servicing radioactive equipment.

For many practical uses of the neutron equipment, the manufacturer's calibration curve is adequate. However, calibration may be required if a soil has unusual neutron-absorption characteristics or if the access tubing is of different size or material from that in which the probe was calibrated. For maximum reproducibility of water content measurements, the probe should fit the access tubing as closely as practical.

A field calibration check may be used for detecting large differences in calibration due to unusual soil conditions. However, field calibration

should not be relied upon for precision work because of the inaccuracies associated with the determination of the volume-basis water content values required. These inaccuracies arise from errors in measurement of both the mass-basis water content (see section 21-2.2) and the bulk density. Field variation in both water content and bulk density (which can occur over a relatively short distance, particularly in a heterogeneous soil) introduces an additional confounding factor. For these reasons, unless the calibration curve is considerably in error, water content inferences from the neutron measurements may well be better than measurements obtained by direct sampling.

Accurate calibration curves require the use of large homogeneous bodies of soil with carefully regulated water content. Some useful checking can be done with substitute neutron absorbers (such as $CdCl_2$–water solutions) in small containers. However, the techniques are sufficiently complicated that persons attempting calibration should consult original literature sources such as Van Bavel et al. (1961), Holmes (1956), Holmes and Jenkinson (1959), Stone et al. (1960), Hewlett et al. (1964), Sinclair and Williams (1979), and Zuber and Cameron (1966), or a review by Visvalingham and Tandy (1972).

An empirical equation for water content in terms of the count ratio, used over the range of water contents of usual interest, is

$$\theta = a + bf \qquad [32]$$

where f is the count rate ratio, I/I_{std}, and a and b are parameters which depend upon soil characteristics and the standard count when the neutron source and detector are in the shield. The count ratio usually has a range of from near zero to about 1.7 at saturation, depending upon the characteristics of the soil and composition of the standard absorber. At low water contents the equation departs from linearity because in real soil the count rate, I, never is zero. The parameter a depends in part upon the bulk density and is closest to zero in soils which have a low bulk density and, when dry, contain the smallest quantities of moderating substances. The parameter b is essentially independent of bulk density and depends upon the presence of substances in the soil, such as structural water (as opposed to absorbed water) and chemical materials, which are effective in the neutron thermalization and capture process. Such substances as iron, boron, molybdenum, and cadmium significantly affect the value of b. Inhomogeneities, such as stratification in mineral density and composition, or even sharp changes in water content at interfaces, where the porosity may change abruptly, can have a marked effect upon the calibration curve where it is constructed from field measurements. Sharp changes in water content distort the shape of the volume-of-influence boundary, which is given approximately by Eq. [31].

Laboratory calibration usually can be done with greater precision than field calibration, providing that the volume of soil exceeds that

09-29-03 14:25 From-EES-10 +5056653866 T-453 P.020/029 F-704

of the volume of influence as given by Eq. [31]. However, when the laboratory calibration curve is used on field measurements it must be recognized that it will deviate from a true calibration to the degree that a nonhomogeneous field soil deviates from the calibration soil both spatially and in its neutron thermalizing and capture constituents. Thus, the accuracy of a calibration curve, whether done in the field or in the laboratory, often is highly subjective.

Bearing in mind the many difficulties associated with calibration, high precision and accuracy require painstaking effort and (usually) separate curves covering the area where the method is to be used. However, for precision and accuracy comparable to that usually achieved with gravimetric methods (roughly + 0.005 g/cm^3), considering field variability, reasonable calibration curves easily may be produced. At this level of accuracy it often is adequate to determine, with considerable care, two points on the calibration curve, one very wet and one dry, and to draw a straight line between. Alternatively, calibration data may be obtained over a range of water contents and a calibration equation determined statistically.

Both water content and change in water content with time at a fixed position are of interest. Ignoring spatial variation in water content and assuming the validity of the empirical equation relating water content and neutron count ratio, f, given by Eq. [32], error equations may be written, one for water content and one for change in water content at a fixed position,

$$\theta = a + bf \qquad [32]$$

$$\Delta\theta = a + bf_1 - (a + bf_2) = b(f_1 - f_2) = b\Delta f. \qquad [33]$$

The corresponding variances are

$$\sigma_\theta^2 = \sigma_a^2 + b^2\sigma_f^2 + f^2\sigma_b^2 \qquad [34]$$

$$\sigma_{\Delta\theta}^2 = b^2\sigma_{\Delta f}^2 + \Delta f^2\sigma_b^2. \qquad [35]$$

Variances σ_a^2 and σ_b^2 for water content depend upon spatial variations in soil constituents and profile structure and the degree to which calibration conditions resemble particular field conditions. Such variations can involve bias as well as unidentified error associated with site variation. Detailed determination of these variances would involve site studies, but reasonable estimates of the magnitude of the standard deviations often may be made. Assuming that accurate placement of the source-detector probe is possible, σ_a^2 and σ_b^2 become essentially zero for water content change at a fixed location in the soil. An exception to this may occur if

sharp interfaces or wetting-fronts exist in a manner so as to seriously alter the shape or size of the volume of influence as the water content changes.

An additional factor of some importance where high accuracy in measurements is desired involves perturbations in water content caused by temperature changes in soil surrounding a neutron-probe access-tube induced by heat conduction along the tube. However, Hanks and Bowers (1960) measured changes in water content around access tubes and concluded that the influence usually would be minor compared with other sources of error.

Errors in water content measurements associated only with random neutron emission may be computed with Eq. [34], where variances involving the calibration parameters are taken as zero. The variance in water content, σ_θ^2, is $b^2\sigma_f^2$ or $b^2\sigma_{I_t/I_{std}t'}^2$, where t is the time counted at rate I and I_{std}. The error may be reduced by counting in the standard for a longer time, t', which usually is practical because of the infrequent need for making this count during a series of measurements. Inclusion of this in the error analysis requires replacing $I_{std}t$ with $I_{std}t'/n$, with $t'/t = n$. Thus,

$$\sigma_\theta^2 = \sigma_{nIt/(I_{std}t')}^2 = b^2[\sigma_{nIt}^2 + (nIt)^2/(I_{std}t')^2\,\sigma_{I_{std}t'}^2]/(I_{std}t')^2 \qquad [36]$$

which, with $\sigma_{nIt}^2 = n^2\sigma_{It}^2 + (It)^2\sigma_n^2$ and with $\sigma_n^2 = 0$, reduces to

$$\sigma_\theta^2 = n^2b^2[It/(I_{std}t') + (It)^2/(I_{std}t')^2]/(I_{std}t') \qquad [37]$$

where, for a Poisson distribution in neutron emission from the source, $\sigma_{It}^2 = It$ and $\sigma_{I_{std}t'}^2 = I_{std}t'$. Recalling that $I_{std}t' = nI_{std}t$ and that $f = It/I_{std}t$, Eq. [37] becomes

$$\sigma_\theta^2 = b^2(f + f^2/n)/(I_{std}t) = fb^2(1 + f/n)/I_{std}t;$$

$$\sigma_\theta = b[f(1 + f/n)/I_{std}t]^{1/2} \qquad [38]$$

From Eq. [38] it may be seen that increasing the value of n by making long counts in the standard will reduce the standard deviation in water content caused by random neutron emission. The variance is largest in wet soil, so that for a large value of f, say 1.6, $b = 0.5$ and for the counting time in the standard (e.g., $I_{std}t = 4 \times 10^4$) being the same as for other counts ($n = 1$) the standard deviation in water content is 0.005 g/cm^3. Increasing counting time in the standard by a factor of 5 reduces this to 0.0036 g/cm^3. For 95% certainty ($2\sigma_\theta$) water content may be known to about 0.007 g/cm^3 or to about 0.7%, neglecting errors in a and b of the calibrating equation.

Variance in the difference in two counts, $\Delta f = f_1 - f_2$, is the sum of the two variances, $\sigma_{f_1}^2$ and $\sigma_{f_2}^2$, so that the variance and standard deviation in water content due solely to variation in the count ratio, f, are

09-29-03 14:25 From-EES-10 +5056653866 T-453 P.021/029 F-704

$$\sigma_{\Delta\theta}^2 = 2\bar{f}b^2(1 + \bar{f}/2)/I_{std}t; \qquad \sigma_{\Delta\theta} = b[2\bar{f}(1 + \bar{f}/2)/(I_{std}t)]^{1/2} \quad [39]$$

where $(f_1 + f_2)/2 = \bar{f}$, the average of the counts made at two different times. In the wet range where the variance is greatest, say $\bar{f} = 1.6$, *and for* $t = 5$, $b = 0.5$ *and* $I_{std}t = 40\,000$, $\sigma_{\Delta\theta} = 0.005$ g/cm^3. Thus, at a certainty of 95%, water content change may be known to the nearest 1% water content ($2 \times 0.005 \times 100$). Little improvement is achieved by going beyond $t = 5$. The accuracy in measurements of water content *change* associated with random neutron emission is slightly less than that for water content itself, but errors due to variations in parameters a and b usually are not involved as in water content measurements, so that the overall accuracy generally is better. Neither variance in water content nor water content change given in Eq. [38] and [39] have involved variance due to spatial variation in water content, except as parameters a and b may be involved. Spatial variation can be large, exceeding 0.001 (standard deviation of $1 \times 10^{-3/2} = 0.03$ g/cm^3), thus somewhat reducing the importance of high accuracy in measurements of a and b and of f.

The parameters, a and b in Eq. [32] and subsequent equations, for a given soil sample of large enough volume to encompass the effective volume of influence, may be determined to almost any desired precision by carefully adjusting bulk density and water content to known values and making long neutron counts. At a single location in the field, errors in determination of these parameters depend upon the accuracy of the independent measure of water content in the soil volume that is seen by the source-detector unit. Such errors would impart a fixed bias to subsequent measurements. Larger errors, however, usually are associated with field variation of soil properties that determine the size of parameters a and b. The sizes of both a fixed bias and variation in spatial distribution of soil properties affecting a and b can only be determined by studies made in the field or upon samples taken from the field. However, experience often will permit estimates of the probable size of such errors. The effect of such errors upon water content inference may be determined by converting them to variances and applying Eq. [34].

21-3.4 Gamma Ray or Neutron Attenuation

21-3.4.1. PRINCIPLES

Principles of absorption by matter of both gamma rays and neutrons are well known. The degree to which a beam of monoenergetic gamma rays is attenuated or reduced in intensity in passing through a soil column depends upon its constituent elements and the overall density of the column. If the constituents and bulk density of the soil without its water remain constant, then changes in attenuation represent changes in water content. Or, if measurements are made at two different gamma ray energies the attenuation equations may be solved simultaneously to provide both water content and soil bulk density. Since bulk density often changes

somewhat with wetting and drying, use of the dual gamma technique (Gardner & Calissendorff, 1967; Soane, 1967; Corey et al., 1971; Gardner et al., 1972) improves the accuracy of water content measurements over what is possible where bulk density must be assumed to remain constant.

The attenuation equation, neglecting air, is

$$I_m/I_o = \exp[-S(\mu_s\rho_s + \mu_w\theta) - 2S'\mu_c\rho_c] \quad [40]$$

where I_m/I_o is the ratio of the transmitted to incident flux for the moist soil, μ_c, μ_s, and μ_w are the mass attenuation coefficients for the container material, soil, and water respectively, θ is mass of water per unit bulk volume of soil, ρ_c is the density of the container, S' is the thickness of its wall, ρ_s is the bulk density of the soil and S is the thickness of the soil column. The intensity of the incident monoenergetic gamma beam is proportional to the intensity at the source and inversely proportional to the square of the distance from the source. Since gamma ray sources are of various sizes and dimensions, some deviation from the inverse square of distance rule is possible. This rule holds strictly only for uniform radiation from an infinitely small source. However, for a ^{137}Cs source, believed to have an active projected area of at least several square millimeters, the falloff was closely proportional to the inverse square of the distance in the range of from 30 to 90 cm as measured using a 12-cm long by 0.33 cm by 1.2 cm collimator against the source and a 10-cm long by 0.1 cm collimator against the scintillation crystal. A correction for air adsorption was made.

The corresponding equation for a dry soil is

$$I_d/I_o = \exp(-S\mu_s\rho_s - 2S'\mu_c\rho_c)\,. \quad [41]$$

Division of Eq. [39] by Eq. [40] yields

$$I_m/I_d = \exp(-\mu_w\theta S) \quad [42]$$

or

$$\theta = \ln(I_m/I_d)/-\mu_w S \quad [43]$$

which is useful under conditions where bulk density may be presumed to be constant. If a perfectly collimated gamma beam of uniform energy were used, and if all scattered and secondary radiation were eliminated, the established value of μ_w, available from independent work, could be used. However, satisfactory calibration curves can be obtained experimentally, usually with greater practicality.

The single gamma ray attenuation method has been successfully used to follow water content change in laboratory columns [illegible] Gurr and Marshall (1960), Ferguson and Gardner (1962), Gurr (1[illegible]), Rawlins and

08-29-03 14:25 From-EES-10 +5056653866 T-453 P.022/029 F-704

Gardner (1963), Davidson et al., (1963), Reginato (1974) Reginato and Van Bavel (1964), and by more recent investigators. It was used to monitor water content change in the root zone in growing plants by Ashton (1956) and by Hsieh et al., (1972).

Two techniques are available for dual gamma scanning. The first involves independent measurements of gamma ray attenuation closely spaced in time, usually using ^{241}Am at 0.060 MeV and ^{137}Cs at 0.662 MeV, where either the soil column or the scanning gamma ray equipment is moved with precision from one set-up to another for the separate measurements. Use of the attenuation measurements and available parameters permits solution of the two simultaneous equations, for energy a

$$I_a = I_{ca}\exp[-S(\mu_{sa}\rho + \mu_{wa}\theta) - S'\mu_{ca}\rho_c] \quad [44]$$

and for energy b

$$I_b = I_{cb}\exp[-S(\mu_{sb}\rho + \mu_{wb}\theta) - S'\mu_{cb}\rho_c] \quad [45]$$

to yield

$$\theta = [\mu_{sb}\ln(I_a/I_{ca}) - \mu_{sa}\ln(I_b/I_{cb})]/Sk \quad [46]$$

$$\rho = [\mu_{wa}\ln(I_b/I_{cb}) - \mu_{wb}\ln(I_a/I_{ca})]/Sk \quad [47]$$

for water content, θ, and bulk density, ρ, where the attenuation coefficients for dry soil and water are μ_s and μ_w with additional subscripts, a and b to designate the gamma energy used, S is the column thickness, $k = \mu_{sa}\mu_{wb} - \mu_{sb}\mu_{wa}$, and the container counts, I_{ca} and I_{cb}, replace I_{oa} and I_{ob} to eliminate the container terms from Eq. [44] and [45].

The second dual gamma scanning technique involves simultaneous measurement, using two single channel analyzers or a multichannel analyzer to make simultaneous gamma counts at the two energy levels involved, and placing the ^{137}Cs gamma source behind the ^{241}Am source so that the higher-energy ^{137}Cs gamma rays pass through the ^{241}Am source. With appropriate geometry so that the soil traversed by both the collimated cesium and americium beams is close to the same length, the only additional problem, beyond those involved where two separate measurements are taken at close to the same time, is that of correcting the americium count for down-scattered gammas coming from the cesium source. Nofziger and Swartzendruber (1974) have shown that the probability for downscatter of 0.662 MeV gammas for cesium into the 0.060 MeV energy range of americium in the soil traversed by a narrowly collimated gamma beam is negligible and that the major downscatter occurs in the scintillation crystal. They have shown that it is possible to obtain

a curve relating the intensity of low-energy gammas in the 0.060 MeV range to the 0.662 MeV gamma intensity, thus producing a correction term to be subtracted from the measured low-energy americium intensity. The empirical correction, e, in counts/second to be subtracted from the low-energy count rate is of the form of a cubic polynomial

$$e = A + Bi_c + Ci_c^2 + Di_c^3 \quad [48]$$

where i_c is the measured cesium intensity in counts/sec. For the gamma ray system of Nofziger and Swartsendruber (1974), the coefficients are given by $A = 11.343$ counts/s, $B = 0.10056$, $C = 9.5979 \times 10^{-8}$ s/count and $D = -2.1916 \times 10^{-11}$ s^2/count^2 over the range of cesium intensities from 3000 to 14000 counts/s. This correction equation was obtained from measurements made with the americium removed and replaced with a brass plug. The correction was shown to be independent of the material in the gamma-ray beam.

Water-content measurement techniques using neutron attentuation rather than gamma radiation are similar, but more specific to water. Although neutrons are scattered and absorbed in some degree by all kinds of nuclei in the soil, hydrogen is by far the most effective. Hence, attentuation change is relatively sensitive to water-content change (see section 21-3.3). However, high neutron fluxes, not readily available outside of a reactor facility, are required and field application would be difficult. Because of the current impracticality of the neutron attentuation method for general use, the method will not be described here in detail, and the reader is referred to an AEC report (Stewart & Gardner, 1969) for further information. The method has been successfully used at the 100-kW nuclear reactor at Washington State University, where fast and thermal neutron fluxes available at beam ports are 4×10^{12} and 1.2×10^{12} neutrons/cm^2 per s.

21-3.4.2. METHOD

21-3.4.2.1. Special Apparatus.

21-3.4.2.1.1 Sources of Gamma Radiation

Cesium-137, which emits gamma rays at 0.662 MeV and has a half-life of 30 years, and ^{241}Am at 0.060 MeV and a half-life of 470 years, are well suited for water-content measurements. The size of sources required depends upon the use to be made of the equipment. Sources of 20 or 25 mCi have been satisfactorily used. However, where rapidly changing water content is to be followed, so that counting times of only a few seconds are required, or where resolution of the order of a millimeter or less is required, much larger sources are desirable. Sources from 100 to 500 mCi have proved satisfactory under these more stringent conditions. However,

09-28-03 14:25 From-EES-10 +5056653866 T-453 P.023/029 F-704

self-absorption limits the practical size of ^{241}Am. Lead shielding required for safe operation increases, of course, with increasing source size. Either of the sources, or both for concurrent measurements, may be used.

21-3.4.2.1.2 Lead Shields and Collimators

Sources are housed in lead shields with suitable collimating holes or slits. With protective plugs for collimating slits, shields serve also as storage containers. Ideally, collimating holes should be drilled appropriately to serve for gross measurements and to accept lead plugs with smaller collimating holes or slits for measurements where greater resolution is desired. Holes about 3 cm in diameter will accommodate a collimating plug containing a 1- by 20-mm slit or any number of other slits or cylindrical openings that might be desired. Collimating plugs should have the form of bolts with large heads to cover the space between bolt and lead block.

For good spatial resolution, the gamma rays, emitted from the source in a solid angle, should be passed through a collimator which is as long as possible, consistent with source size and desired count rate, so as to be close to parallel as they enter the detecting crystal. For errors associated with collimation of the order of 0.001 g/cm^3 in a soil column 10 cm thick, the collimator for ^{137}Cs should be about 8 cm long on both source and detector side. For a tapered collimator slit 0.45 × 45 mm at the detector crystal face, the spatial resolution for cesium has been found to be only slightly greater than slit size, or about 0.5 mm. For dual gamma measurements the ^{241}Am system should have similar collimators, so that the shape of the gamma ray beam as it passes through the soil is comparable to that for the cesium. However, the practical size of an americium source is limited by self-absorption to roughly 22 mCi/mm^2, so that path and collimator length suited to cesium may severely reduce the count rate possible with americium and make compromise necessary. The length of collimator material needed for the 0.060-MeV gamma photons from ^{241}Am is roughly 1/50 of that needed for 0.62 gamma photons from ^{137}Cs. Although lead is used ordinarily for collimators and protective shielding, tungston absorbs gamma energy from two times as well (at a thickness of 1.2 cm) as lead to many times as well, and may be machined to closer tolerances. Hence, it often is used in place of lead.

The dimensions of protective shielding depend upon source strength; but, for greatest convenience where weight is not an important factor, blocks should be thick to reduce radiation in the vicinity to values only slightly above natural background, so that special precautions required in the vicinity of such radiation hazards may be minimized. Normal background varies from 0.01 to 0.03 mR (milliroentgen)/hour. One milliroentgen in air is about 5% less than 1 mrad in tissue (the rad is the unit of physical radiation dose), so that for practical considerations they ma[illegible] regarded as equivalent.

[illegible]he design of shielding and collimators, it is necessary to determine

the dose rate in terms of source strength, shielding thickness, and distance from the source. This may be determined by the following formula. The dose rate in mrad per hour at a distance R in cm from a point-source of gamma radiation at strength A in mCi and energy E_o in MeV, and with shielding of thickness t in cm of a material with a linear attenuation coefficient in cm^{-1}, is (as inferred from Eq. [2], [3], and [4] of Blizard (1958))

$$D(R,t) = 2.134 \times 10^6\, B(\mu,t)\, (\mu_a/\rho)\, E_o[A \exp(-\mu t)/(4\pi R^2)] \quad [49]$$

where $B(\mu,t)$ is the build-up factor, μ_a/ρ in cm^2/g is the energy-absorption mass-attenuation coefficient for the source energy and the material in which the dose is to be calculated (ρ is its density) and where the numerical constant has units of g × rad/MeV × mCi × hr). For gamma sources with more than one peak in an energy range that would contribute significantly to the dose rate, Eq. [49] must be applied to each peak and the dose rates summed.

For 0.662-MeV gamma radiation from a ^{137}Cs source and for 0.060-MeV gamma radiation from a ^{241}Am source, μ_a/ρ, measured in tissue, is about 0.0323 cm^2/g and 0.033 cm^2/g (Evans, 1968). For the same two gamma sources, μ is 0.717/cm and 46.4/cm, and the build-up factor $B(\mu,t)$ for lead thicknesses from 2 to 15 cm is approximated by $1.4 + 0.21t$ (a dimensionless factor) for the ^{137}Cs. The build-up factor for ^{241}Am is close to unity. The dose rate equations for ^{137}Cs and ^{241}Am inferred from data in Tables 1 and 4 (Blizard, 1958) and Evans (1968), are:

$$^{137}\text{Cs: } D(R,t) = (5.1 + 0.76t)10^3\, A \exp(-0.717t)/R^2 \text{ mrad/hour} \quad [50]$$

$$^{241}\text{Am: } D(R,t) = 336.24\, A \exp(-46.4t)/R^2 \text{ mrad/hour} \quad [51]$$

where R is the sum of the shield thickness, t, and the distance from the shield surface.

For a 500-mCi ^{137}Cs source with 13.6 cm of lead shielding, the radiation at the surface of the lead is about 2.5 mrad/hour, and at 1 m from the surface it is about 0.03 mrad/hour, or the equivalent of background radiation. Shielding required for a ^{241}Am source of comparable strength is approximately 3 mm, with radiation at 1 m being negligible compared to natural background.

The maximum exposure limitation given by the U. S. Nuclear Regulatory Commission is 1.25 rem/calendar quarter (the rem is the "roentgen equivalent, man") for persons 18 years of age or older. For gamma radiation, 1 rem is the equivalent of 1 rad, so that for a 7-hour exposure/day for 65 days (ordinary working days/quarter year), 0.03 to 2.5 mrad/hour results in an accumulation of 0.014 to 1.14 rad/[illegible]ter, well within U. S. regulations. For a more complete description [illegible] applicable regu-

08-28-03 14:26 From-EES-10 +5056653866 T-453 P.024/029 F-704

lations in the USA see part 20 of the Federal Code (U.S. Nuclear Regulatory Commission, 1985).

21-3.4.2.1.3 Gamma-sensitive Probe

Scintillation-type gamma-sensitive probes are the most satisfactory. A 5-cm phototube probe containing a preamplifier and equipped with a 2.5-cm thick thallium-activated sodium iodide crystal is adequate for many measurements. However, the crystal size may be increased for greater sensitivity. A second lead collimator about 5 cm long for ^{137}Cs (or much less for ^{241}Am), containing a thin slit or hole, is aligned with the source collimator, with an intervening space large enough to accommodate the soil container. The scintillation crystal, with a surrounding lead shield of approximately the same thickness as used around the source, is placed against the collimator. Where sequential measurements at two gamma energies are to be made, two probes may be desired.

21-3.4.2.1.4. Soil Container and Mechanism for Orienting Soil in Beam

Specifications for the soil container depend upon the nature of an experiment. However, two factors should be considered for optimum water content measurement. First, the container walls through which the beam will pass should be as thin as practical and of low density, and should not absorb water. Where a small source is to be used so that counting rate is limiting, it often is desirable to arrange holes in the container at desired counting positions, covering them with Mylar film. Second, the thickness of the soil through which the beam is to pass should be about 10 to 35 cm for ordinary soil density when ^{137}Cs is used as a gamma source. For ^{241}Am, thickness should be 2 to 8 cm. For dual gamma measurements the curves in Figure 21-5 should be used. Equation [61] in section 21-3.4.2.3 can be used to compute optimum soil column thickness for single gamma. The mechanism for positioning the soil container in the beam can take many forms, depending upon the nature of an experiment. In some cases, because of the weight of the lead shielding, it is easiest to move the soil container in the beam. A rack-and-pinion-operated sliding table or elevator works well.

21-3.4.2.1.5 Scaler or Rate Meter

A number of different types of scalers or rate meters can be used. A single- or multiple-channel analyzer with a built-in adjustable amplifier-discriminator and with a resolution time of 1 μs or less and a preset timer is desirable. Data acquisition systems, incorporating computer programs to accept and refine data, may be used for direct output of water contents and bulk densities.

21-3.4.2.2 Procedure. Before it is possible to infer water content

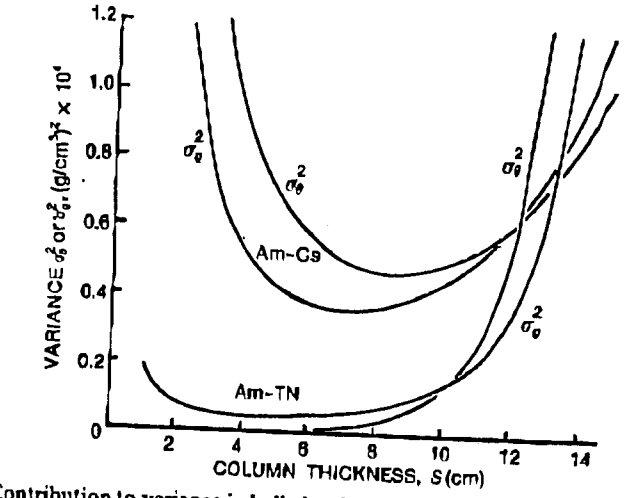

Fig. 21-5. Contribution to variance in bulk density or water content from random emission of radiation sources for double gamma measurements. Mass attenuation coefficients have the following values: for ^{241}Am $\mu_{m} = 0.305$ cm^2/g, $\mu_{w} = 0.2036$; for ^{137}Cs $\mu_{s} = 0.0769$, $\mu_{w} = 0.0858$; for thermal neutrons (TN) $\mu_{m} = 0.4130$ and $\mu_{w} = 2.632$.

from gamma ray measurements, it is necessary to evaluate the attenuation coefficient, μ_w, and the count through dry soil and the container, I_d, in Eq. [43], when a single gamma energy is to be used. Or, for concurrent measurement of water content and bulk density using two gamma energies with Eqs. [46] and [47], it is necessary to evaluate the attenuation coefficients for dry soil and for water, μ_s and μ_w, at the two different energies. Also, the gamma count for the container walls, I_c, must be obtained at the two energies and the column thickness, S, must be measured.

Measurements of I_d and I_c are similar, I_d being obtained by packing the soil column to be used with oven-dry soil and scanning the column over its length, using sufficiently long counting times to reduce the error due to random emission of gamma photons to any desired level (see section 21-3.4.2.3 for a discussion of errors). Air-dry soil may be used, providing that the count is corrected using Eq. [42], where θ is the water content of the soil determined gravimetrically, g/g, and converted to g/cm^3 through multiplication by the density of water, usually taken as unity. In single-energy systems values of I_d and I_c must be uniform throughout the length of a soil column under study, or their values at measuring positions along the column must be obtained and tabulated for use in water-content computations to be made from gamma ray counts at each position. Also, I_d may change with swelling or shrinking of the soil in wetting and drying situations, which introduces error in single-energy systems. Using container materials of uniform dimensions, I_c likely will be uniform over a column so that a single value may be obtained using the empty container. This value may then be used throughout a series of measurements. However, it is the

09-28-03 14:26 From-EES-10 +5056653866 T-453 P.025/029 F-704

to the count in the empty container, I_c, that is used in the attenuation equations for the dual-energy systems. This fact makes it possible to reduce the effect of any instrument drift on measurements by making periodic measurements of I_c and using them to obtain the ratio, I/I_c. This may be done using an absorber made from two pieces of the container wall material, so as to simulate the wall. Other absorbers may be used as well. Attenuation in air usually may be ignored, as it is negligible in most measurements involved with soil.

For dual gamma measurements, the values of the attenuation coefficients for soil and water must be obtained with considerable precision (see section 21–3.4.2.3). These measurements are made by placing soil in small containers having parallel walls at precisely known spacing, with the same geometry as that for the soil columns to be used. The bulk density is computed from mass and volume measurements. Careful counts are made on the containers without soil to determine I_c, and air-dry soil then is packed into the containers as uniformily as possible with a correction later made for the air-dry water content. (Oven-dry soil may be used to eliminate need for a water-content correction. However, absorption of moisture from the atmosphere during measurement may introduce some error.) Gamma counts then are made at several positions of the gamma beam in the soil, and the average value computed. Counting times may be set at values to produce any desired precision in the attenuation coefficients (the larger the count, the greater the precision). A similar measurement is made for water, with the values obtained usually being close to the theoretical value but usually not identical because of dependence upon the geometry of the attenuation system. (Mass attenuation coefficients for water generally are reported to be 0.197 and 0.088 cm^2/g for 0.060- and 0.662-MeV gamma rays. Experimental values for gamma beams used on soil columns often are from 1 to 6% less.) Eq. [43], with $\theta = 1$ g/cm^3 (usually) and I_d replaced by the container count, I_c, is used to compute the attenuation coefficients for water. The equation for computing the values for soil is

$$\mu_s = [-\mu_w\theta_{ad} - \ln(I_{ad}/I_c)/S]/\rho_s \qquad [52]$$

where ρ_s is the dry bulk density in g/cm^3 computed for the container soil, θ_{ad} is the air-dry water content in g/cm^3, S is the thickness of the soil column in centimeters, and I_{ad}/I_c is the ratio of count in air-dry soil to count through the empty container.

21–3.4.2.3. Comments. The precision of gamma ray methods for measuring water content and bulk density varies with the thickness and density of the soil column, the adsorption characteristics of the soil, and the size of the gamma count in a moist and dry soil column. The error analysis of the single-gamma technique which follows provides useful desi[illegible]iteria for systems, including optimum column thickness. Since the va[illegible]nce in gamma-ray emission is the gamma-ray count itself, count

intensity, I, must be replaced by the number of counts, It, where t is th[illegible] counting time in Eq. [43]; thus,

$$\theta = -\ln (I_m t/I_d t)/(\mu_w S) \qquad [53]$$

and the variance, with reasonable assumptions, becomes

$$\sigma_\theta^2 = \sigma^2_{\ln(I_m t/I_d t)}/(\mu_w^2 S^2) \qquad [54]$$

so that the standard deviation is

$$\sigma_\theta = \sigma_{\ln(I_m t/I_d t)}/(\mu_w S) \qquad [55]$$

To reduce the error, the counting time for $I_d t$, which is measured infrequently and usually under conditions where time is not pressing, is increased by a factor of 3 or 4 over that of $I_m t$. However, the computation requires that t be the same for both $I_m t$ and $I_d t$. Hence the longer count $I_d t'$ is reduced to comparable size through division by n, where $n = t'/t$. Thus $I_d t'/n$ is the time-equivalent count to $I_d t$ and may replace it in Eqs. [54] and [55].

The standard deviation of $\ln(I_m t/I_d t'/n)$ is

$$\sigma_{\ln}(I_m t/I_d t'/n) = \frac{I_d t'/n}{I_m t}\,\sigma(I_m t/I_d t'/n)$$

$$= \frac{I_d t'/n}{I_m t}\,\frac{1}{I_d t'/n}\left[\sigma^2_{I_m t} + \frac{(I_m t)^2}{(I_d t'/n)^2}\,\sigma^2_{(I_d t'/n)}\right]^{1/2}. \qquad [56]$$

Using Eq. [56] with

$$\sigma_{I_m t} = (I_m t)^{1/2} \text{ and } \sigma_{(I_d t'/n)} = (I_d t')^{1/2}/n$$

Eq. [55] becomes

$$\sigma_\theta = 1/[\mu_w S\,(I_m t)^{1/2}]\,[1 + I_m t/(I_d t')]^{1/2}$$

$$= 1/(\mu_w S\,(I_m t)^{1/2})\,[1 + I_m t/(n I_d t)]^{1/2}. \qquad [57]$$

Where n is 3 or 4, the second term under the radical may be neglected with only small effect on σ_θ (ca 10%), so that Eq. [57] becomes

$$\sigma_\theta = 1/[\mu_w S\,(I_m t)^{1/2}] \qquad [58]$$

or, multiplying Eq. [40] by t/t and substituting into Eq. [58] yields

$$\sigma_\theta = \exp[S(\mu_s\rho_s + \mu_w\theta)/2 + S'\mu_c\rho_c]/[\mu_w S(\text{[illegible]})^{1/2}]. \qquad [59]$$

08-29-03 14:26 From-EES-10 +5056653866 T-453 P.026/029 F-704

The optimum value of soil-column thickness, S, may be obtained by equating $d\sigma_\theta/dS$, obtained by differentiation of Eq. [59], to zero:

$$\frac{d\sigma_\theta}{ds} = \frac{\exp[S(\mu_s\rho_s + \mu_w\theta)/2]\exp(S'\mu_c\rho_c)}{\mu_w S(I_0 t)^{1/2}} \times [1/2(\mu_s\rho_s + \mu_w\theta) - (1/S)] = 0 \qquad [60]$$

and

$$S = 2/(\mu_s\rho_s + \mu_w\theta) \qquad [61]$$

For ^{241}Am with $\mu_s = 0.307$ and $\mu_w = 0.195$ cm^2/g and for a bulk density of 1.2 g/cm^3, the optimum soil-column thickness ranges between 5 and 4 cm for low to high water-contents. Similarly, for ^{137}Cs with $\mu_s = 0.076$ and $\mu_w = 0.082$ cm^2/g, the optimum soil-column thickness ranges from 20 to 15 cm.

A complete error analysis for use with Eq. [46] and [47] for dual gamma measurements is given in Gardner et al., (1972). However, a brief discussion of the two most important factors involved in the design of experimental soil columns—errors associated with random gamma ray emission which determine the optimum column thickness, and errors in measurement of column thickness—is given here. The error equation may be written

$$\sigma_\theta^2 = \left(\frac{\partial\theta}{\partial I_a t}\right)^2\sigma_{I_a}^2 + \left(\frac{\partial\theta}{\partial I_{ca} t}\right)^2\sigma_{I_{ca}}^2 + \left(\frac{\partial\theta}{\partial I_b t}\right)^2\sigma_{I_b}^2 + \left(\frac{\partial\theta}{\partial I_{cb} t}\right)^2\sigma_{I_{cb}}^2 + \left(\frac{\partial\theta}{\partial S}\right)^2\sigma_S^2 + \left(\frac{\partial\theta}{\partial\mu_{sa}}\right)^2\sigma_{\mu_{sa}}^2 + \left(\frac{\partial\theta}{\partial\mu_{sb}}\right)^2\sigma_{\mu_{sb}}^2 + \left(\frac{\partial\theta}{\partial\mu_{wa}}\right)^2\sigma_{\mu_{wa}}^2 + \left(\frac{\partial\theta}{\partial\mu_{wb}}\right)^2\sigma_{\mu_{wb}}^2 . \qquad [62]$$

For bulk density, θ is replaced by ρ. Carrying out the partial differentiation indicated in this equation yields appropriate equations giving the contribution to variance in water content or bulk density of each of the parameters in terms of a variance which may be assumed for measurement of that particular parameter. Gamma ray emission follows a Poisson distribution for which the variance, $\sigma_{I_i}^2$, is the number of counts, It. Hence, it is possible, for a particular count, to obtain relationships between variances in water content or bulk density and variances associated with (i) the container in the absence of soil (this count to be made over appropriate times so as to be the same for both gamma sources), (ii) the various absorption coefficients, (iii) the water content and bulk density, and (iv) the thickness of the soil column. For 10^6 counts through the empty container and mid-range values of water content and bulk density (0.15 and

1.2 g/cm^3), the curves for variance in water content and in bulk density are shown in Fig. 21-5 in terms of soil-column thickness. Curves also are given for the combination of ^{241}Am and thermal neutrons. From these curves it may be observed that optimum column thickness for bulk density is about 7 cm (a range of 5–10 cm) and for water content is about 8.5 cm (average of about 7–10 cm). A similar analysis involving variation in column thickness from a designated value (a bias rather than a variance for repeated measurements at the same position) shows that for an accuracy contribution in water content of 0.001 g/cm^3 the error in column thickness must be less than 0.67%, or for 0.01 g/cm^3 less than 6.7%. Similarly, for an accuracy contribution in bulk density of 0.001 g/cm^3, the error in column thickness must be less than 0.083%, or for 0.01 g/cm^3 less than 0.83%. For water and homogeneous soil the attenuation coefficients may be determined by repeated measurements to any desired accuracy, using a particular gamma-scanning set-up. Hence these factors do not enter into design, but the relevant accuracy evaluation may be carried out similarly using the appropriate term in Eq. [62].

The above analyses assume a rectangular soil column. If a cylindrical column is used it is obvious that the attenuation coefficients must be carefully made on cylindrical samples, with the column positioned precisely in the gamma beam to avoid error. (The difference in column thickness over a gamma-beam cross-section would be absorbed in the attenuation coefficients in this case.)

For all gamma counting, a correction for the resolving time of the gamma-ray counting system must be used. This is the time between successive counts during which an additional count cannot be recorded (see Fritten, 1969; Gardner et al., 1972). A corrected count rate, N, for the counting period used may be computed using the equation

$$N = R/(1 - \tau R) \qquad [63]$$

where R is the observed count rate over the prescribed counting time and τ is the dead time per count. The value of τ may be determined experimentally using particular equipment in the range of settings to be involved and by making counts through air, R_0, separately for each of two iron blocks of equal thickness (thickness such as to give a counting rate at least as high as that expected in measurements to be made) and averaging them, R_1; and making counts for both blocks together, R_2. The equation for τ (Gardner et al., 1972) is

$$\tau = (R_1^2 - R_0R_2)/[R_1^2(R_0 + R_2) - 2R_0R_1R_2] . \qquad [64]$$

The basic gamma count rate I_0 with only air between the source and scintillation crystal, and hence the precision and resolution possible in water-content measurement, depends upon the strength of the gamma source, the range of gamma energies counted, the geometry of the equip-

08-29-03 14:26 From-EES-10 +5056653866 T-453 P.027/029 F-704

ment, and the efficiency with which gamma radiation is measured. The source emits gamma rays in all directions, and the intensity of the gamma field decreases with the square of the distance from the source (neglecting the absorption of air in the path and assuming a point source) (section 21–3.4.1). Under these conditions, the fraction of the radiation reaching the face of the scintillation crystal depends upon the degree of collimation and the distance from the source. With an absorber in the beam, some scattered and secondary radiation reaches the crystal so that the inverse square law no longer holds. However, if a single-channel or multichannel analyzer or a discriminator is used so that only gamma rays of the maximum energy emitted from the source are counted, the effect of scattered and secondary radiation is eliminated.

With the gamma-ray detector set to count only a narrow range of gamma energies, the resolution possible for given precision, source strength, and experimental arrangement can be computed. If the area of the collimator at the face of the crystal is a (assuming this to be the minimum collimator restriction) and the distance from the crystal face is r, then the fraction of the total radiation reaching the crystal, neglecting the absorption of air in the path, is $a/(4\pi r^2)$. The count rate I_0 is proportional to the product of this fraction and the strength of the source Z:

$$I_0 = k(a/4\pi r^2)Z \qquad [65]$$

with

$$k = 3.70 \times 10^7 f[1 - \exp(-\mu_p s)] \qquad [66]$$

where the constant, 3.70×10^7, is the number of disintegrations per second associated with 1 mCi of any radioactive nuclide; f is the number of photoelectrons produced per disintegration (0.851 for ^{137}Cs and 0.359 for ^{241}Am), μ_p is the linear attenuation coefficient for the thallium-activated sodium iodide crystal, which is the fractional decrease in intensity per unit length traversed, s. The value of μ_p is 0.0352 cm^{-1} for 0.62-MeV photons of ^{137}Cs and 22.12 cm^{-1} for 0.060-MeV photons of ^{241}Am. For a scintillation crystal approximately 5 cm in diameter and 5 cm deep, k has the value of 5.08×10^6 photoelectrons s^{-1} mCi^{-1} for ^{137}Cs and 13.18×10^6 photoelectrons s^{-1} mCi^{-1} for ^{241}Am. Eq. [65] gives the maximum count rate obtainable in air for a particular equipment geometry, collimation, and source strength. Using narrow collimation, values considerably less than this may be expected where perfect alignment of collimators is difficult to achieve.

Replacing the count N_0 in Eq. [59] by its equivalent I_0t, substituting this into Eq. [65], and solving explicitly for the source strength leads to

$$Z = 4\pi r^2 \exp[S(\mu_s\rho_s + \mu_w\theta) + 2S\mu_c\rho_c]/(ka\mu_w^2S^2\sigma_\theta^2 t). \qquad [67]$$

For a typical situation at high water content (0.4 g/cm^3) and bulk density (1.3 g/cm^3) where the error is largest, a 10-cm wide soil column contained in a plastic box with 0.62-cm thick walls having a mass attenuation coefficient of 0.08747 cm^2/g and a density of 1.19 g/cm^3 would require a 200-mCi ^{137}Cs source for 60-s counting to yield a precision of $\pm$ 0.5% water content ($3\sigma_\theta = 0.005$ g/cm^3), with tapering collimation at the source and on the detector side having a slit cross-section of 1 by 40 mm at the face of a 5-cm deep scintillation crystal. With imperfect alignment of a narrow slit, the count rate would be significantly reduced, and a somewhat larger source strength would be required. The attenuation coefficients used in the above computation are 0.07785 and 0.08559 cm^2/g for soil and water, and the value of k is 5.081×10^6 photoelectrons/s.

If long counting-times are practicable in an experiment, high precision is possible with relatively small sources and at high resolution. However, if rapid measurement is important, then either the resolution must be low or the source strength high. In such cases, since k increases with increasing crystal size, large scintillation crystals are desirable. If resolution and counting time are not important factors, as in an experiment involving measurement of gross water content in a container of soil with growing plants, source strengths of the order of 20 mCi may be adequate. Also, small crystals are considerably less expensive than large ones, and crystals of the order of 2 to 3 cm may often be adequate under such conditions.

In the foregoing discussion it has been assumed that the soil traversed by the gamma beam had spatially uniform physical and chemical properties so that the density and attenuation coefficients could be represented by single values. In stratified soil this is not always the case, particularly in systems that change with time, and the interpretation of calculated bulk densities and water contents becomes complicated. Special problems to be encountered in measurements made in stratified soil and some methods for dealing with them are discussed by Goit et al. (1978) and by Nofziger (1978).

21–4 REFERENCES

Anderson, A. B. C., and N. E. Edlefsen. 1942. Electrical capacity of the 2-electrode plaster of Paris block as an indicator of soil moisture content. Soil Sci. 54:35–46.

Ashton, F. M. 1956. Effects of a series of cycles of alternating low and high soil water contents on the rate of apparent photosynthesis in sugar cane. Plant Physiol. 31:266–274.

Belcher, D. J., T. R. Cuykendall, and H. S. Sack. 1950. The measurement of soil moisture and density by neutron and gamma ray scattering. Civil Aeron. Admin. Tech. Develop. Rep. no. 127. Washington, DC.

Blizard, E. P. 1958. Nuclear radiation shielding. *In* H. Etherington (ed.) Nuclear engineering handbook. McGraw-Hill, New York.

Bloodworth, M. E., and J. B. Page. 1957. Use of thermistors for the measurement of soil moisture and temperature. Soil Sci. Soc. Am. Proc. 21:11–15.

Bouyoucos, G. J. 1949. Nylon electrical resistance unit for continuous m[illegible]ment of soil moisture in the field. Soil Sci. 67:319–330.

09-28-03 14:27 From-EES-10 T-453 P.028/029 F-704 +5056653866

Bouyoucos, G. J. 1953. More durable plaster of Paris moisture blocks. Soil Sci. 76:447–451.

Bouyoucos, G. J., and A. H. Mick. 1940. An electrical resistance method for the continuous measurement of soil moisture under field conditions. Michigan Agric. Exp. Stn. Tech. Bull. 172. East Lansing, MI.

Cannell, G. H. 1958. Effect of drying cycles on changes in resistance of soil moisture units. Soil Sci. Soc. Am. Proc. 22:379–382.

Cannell, G. H., and C. W. Asbell. 1964. Prefabrication of mold and construction of cylindrical electrode-type resistance units. Soil Sci. 97:108–112.

Cline, M. G. 1944. Principles of soil sampling. Soil Sci. 58:275–288.

Colman, E. A., and T. M. Hendrix. 1949. Fiberglass electrical soil-moisture instrument. Soil Sci. 67:425–438.

Corey, J. C., S. F. Peterson, and M. A. Wakat. 1971. Measurement of attenuation of ^{137}Cs and ^{241}Am gamma rays for soil density and water content determinations. Soil Sci. Soc. Am. Proc. 35:215–219.

Crumpler, T. B., and J. H. Yoe. 1946. Chemical computations and errors. John Wiley & Sons, Inc., New York.

Cummings, R. W., and R. F. Chandler, Jr. 1940. A field comparison of the electrothermal and gypsum block electrical resistance methods with the tensiometer method for estimating soil moisture in situ. Soil Sci. Soc. Am. Proc. 5:80–85.

Davidson, J. M., J. W. Biggar, and D. R. Nielsen. 1963. Gamma-radiation attenuation for measuring bulk density and transient water flow in porous materials. J. Geophys. Res. 68:4777–4783.

de Plater, C. V. 1955. A portable capacitance-type soil moisture meter. Soil Sci. 80:391–395.

de Vries, J., and K. M. King. 1961. Note on the volume of influence of a neutron surface moisture probe. Can. J. Soil Sci. 41:253–257.

Edlefsen, N. E., and A. B. C. Anderson. 1941. The four-electrode resistance method for measuring soil moisture content under field conditions. Soil Sci. 51:367–376.

England, C. B. 1965. Changes in fiber-glass soil moisture-electrical resistance elements in long-term installations. Soil Sci. Soc. Am. Proc. 29:229–231.

Evans, R. D. 1968. X-ray and γ-ray interactions. *In* F. H. Attix and W. C. Roesch (ed). Radiation dosimetry, Vol. 1, Fundamentals. 2nd ed. Academic Press, New York.

Ferguson, A. H., and W. H. Gardner. 1962. Water content measurement in soil columns by gamma ray absorption. Soil Sci. Am. Proc. 26:11–14.

Fletcher, J. E. 1939. A dielectric method for determining soil moisture. Soil Sci. Soc. Am. Proc. 4:84–88.

Fritton, D. D. 1969. Resolving time, mass absorption coefficient, and water content with gamma ray attenuation. Soil Sci. Soc. Amer. Proc. 33:651–655.

Gardner, W. H., and C. Calissendorff. 1967. Gamma ray and neutron attenuation measurement of soil bulk density and water content. p. 101–113. *In* Isotope and radiation techniques. Proc. of Symp. Techniques in Soil Physics and Irrigation Studies, Instanbul, IAEA Vienna.

Gardner, W. H., G. S. Campbell, and C. Calissendorff. 1972. Systematic and random errors in dual gamma energy soil bulk density and water content measurements. Soil Sci. Soc. Am. Proc. 36:393–398.

Gardner, Wilford, and D. Kirkham. 1952. Determination of soil moisture by neutron scattering. Soil Sci. 73:391–401.

Gee, G. W., and M. E. Dodson. 1981. Soil water content by microwave drying: a routine procedure. Soil Sci. Soc. Am. J. 45:1234–1237.

Gilbert, P. A. 1974. Microwave oven used for rapid determination of soil water contents. Rep. No. 13, Misc. paper No. 3-478, U. S. Army Engineer Waterways Exp. Stn., Vicksburg, MS.

Goit, J. B., P. H. Groenevelt, B. D. Kay, and J. G. P. Loch. 1978. The applicability of dual gamma scanning to freezing soils and the problem of stratification. Soil Sci. Soc. Am. J. 42:858–863.

Greacen, E. L., and C. T. Hignett. 1979. Sources of bias in the field calibration of a neutron meter. Aust. J. Soil Res. 17:405–415.

Gurr, C. G. 1962. Use of gamma rays in measuring water content and permeability in unsaturated columns of soil. Soil Sci. 94:224–229.

Gurr, C. G., and T. J. Marshall. 1960. Unsaturated permeability—its measurement and its

estimation from other properties of the material. Trans. Int. Congr. S[illegible]ci., 7th. 1:306–310.

Hankin, L., and B. L. Sawhney. 1978. Soil moisture determination using microwave radiation. Soil Sci. 126:313–315.

Hanks, R. J., and S. A. Bowers. 1960. Neutron meter access tube influences soil temperature. Soil Sci. Soc. Am. Proc. 24:62–63.

Hewlett, J. D., J. E. Douglass, and J. L. Clutter. 1964. Instrumental and soil moisture variance using the neutron-scattering method. Soil Sci. 97:19–24.

Holmes, J. W. 1956. Calibration and field use of the neutron scattering method of measuring soil water content. Aust. J. Appl. Sci. 7:45–58.

Holmes, J. W. 1966. Influence of bulk density of the soil on neutron moisture meter calibration. Soil Sci. 102:355–360.

Holmes, J. W., and A. F. Jenkinson. 1959. Techniques for using the neutron moisture meter. J. Agric. Eng. Res. 4:100–109.

Hsieh, J. J. C., W. H. Gardner, and G. S. Campbell. 1972. Experimental control of soil water content in the vicinity of root hairs. Soil Sci. Soc. Am. Proc. 36:418–421.

Kelley, O. J. 1944. A rapid method for calibration of various instruments for measuring soil moisture in situ. Soil Sci. 58:433–440.

Kirkham, D., and G. S. Taylor. 1950. Some tests of a four-electrode probe for soil moisture measurement. Soil Sci. Soc. Am. Proc. 14:42–46.

Kittrick, J. A., and E. W. Hope. 1970. Preventing water resorption in weight loss determinations. Soil Sci. Soc. Am. Proc. 34:536–537.

Lal, R. 1974. The effect of soil texture and density on the neutron and density probe calibration for some tropical soils. Soil Sci. 117:183–190.

Lambe, T. W. 1949. How dry is a dry soil? Proc. 29th Annual Meeting, Highway Research Board 29:491–496.

Makower, B. 1950. Determination of water in some dehydrated foods. Adv. Chem. 3:37–54.

Makower, B. and E. Nielsen. 1948. Use of lyophilization in determination of moisture content of dehydrated vegetables. Anal. Chem. 20:856–858.

Makower, B., S. M. Chastain, and E. Nielsen. 1946. Moisture determination in dehydrated vegetables. Vacuum-oven method. Ind. Eng. Chem. 38:725–731.

McHenry, J. R. 1963. Theory and application of neutron scattering in the measurement of soil moisture. Soil Sci. 95:294–307.

Miller, R. J., R. B. Smith, and J. W. Biggar. 1974. Soil water content: microwave oven method. Soil Sci. Soc. Am. Proc. 38:535–537.

Nofziger, D. L. 1978. Errors in gamma-ray measurement of water content and bulk density in nonuniform soils. Soil Sci. Soc. Am. J. 42:845–850.

Nofziger, D. L., and D. Swartzendruber. 1974. Material content of binary physical mixtures as measured with a dual-energy beam of gamma rays. J. Appl. Phys. 45:5443–5449.

Nutting, P. G. 1943. Some standard thermal dehydration curves of minerals. U. S. Geol. Survey, Prof. Paper 197-E.

Olgaard, P. L. 1965. On the theory of the neutronic method for measuring the water content of soil. Danish Atomic Energy Comm., Riso Rep. 97.

Perrier, E. R., and A. W. Marsh. 1958. Performance characteristics of various electrical resistance units and gypsum materials. Soil Sci. 86:140–147.

Phene, C. J., G. J. Hoffman, and S. L. Rawlins. 1971a. Measuring soil matric potential in situ by sensing heat dissipation within a porous body: I. Theory and sensor construction. Soil Sci. Soc. Am. Proc. 35:27–32.

Phene, C. J., G. J. Hoffman, and S. L. Rawlins. 1971b. Measuring soil matric potential in situ by sensing heat dissipation within a porous body: II. Experimental results. Soil Sci. Soc. Am. Proc. 35:225–229.

Phillips, R. E., C. R. Jensen, and D. Kirkham. 1960. Use of radiation equipment for plow-layer density and moisture. Soil Sci. 89:2–7.

Rawlins, S. L., and W. H. Gardner. 1963. A test of the validity of the diffusion equation for unsaturated flow of soil water. Soil Sci. Soc. Am. Proc. 27:507–511.

Reginato, R. J. 1974. Gamma radiation measurements of bulk density changes in a soil pedon following irrigation. Soil Sci. Soc. Am. Proc. 38:24–29.

Reginato, R. J., and C. H. M. van Bavel. 1964. Soil-water measurement with gamma attentuation. Soil Sci. Soc. Am. Proc. 28:721–724.

Richards, L. A., and H. T. Stumpf. 1960. Volumetric soil sampler. Soil Sci. 89:108–110.

Richards, L. A., and L. R. Weaver. 1943. The sorption-block soil moisture meter and hysteresis effects related to its operation. J. Am. Soc. Agron. 35:1002–1011.

Shaw, B. T., and L. D. Baver. 1940. An electrothermal method for following moisture changes in soil in situ. Soil Sci. Soc. Am. Proc. (1939) 4:78–83.

Sinclair, D. F., and J. Williams. 1979. Components of variance involved in estimating soil water content and water content change using a neutron moisture meter. Aust. J. Soil Res. 17:237–47.

Soane, B. D. 1967. Dual energy gamma-ray transmission for coincident measurement of water content and dry bulk density of soil. Nature (London) 214:1273–1274.

Stewart, G. L., and W. H. Gardner. 1969. Water content measurement by neutron attenuation for application to study of unsaturated flow of soil water. AEC Rep. No. RLO-1543-5. Washington, DC.

Stolzy, L. H., and G. A. Cahoon. 1957. A field-calibrated portable neutron rate meter for measuring soil moisture in citrus orchards. Soil Sci. Soc. Am. Proc. 21:571–575.

Stone, J. F., R. H. Shaw, and D. Kirkham. 1960. Statistical parameters and reproducibility of the neutron method of measuring soil moisture. Soil Sci. Soc. Am. Proc. 24:435–438.

Tanner, C. B., E. Abrams, and J. C. Zubriski. 1949. Gypsum moisture-block calibration based on electrical conductivity in distilled water. Soil Sci. Soc. Am. Proc. 13:62–65.

Taylor, S. A. 1955. Field determinations of soil moisture. Agric. Eng. 36:654–659.

Taylor, S. A., D. D. Evans, and W. D. Kemper. 1961. Evaluating soil water. Utah Agric. Exp. Stn. Bull. 426.

Topp, G. C. 1980. Electromagnetic determination of soil water content: measurements in coaxial transmission lines. Water Resources 16:574–582.

U. S. Nuclear Regulatory Commission. 1985. Rules and regulations. Title 10, Chap. I, Code of Federal Regulations, Part 20, Standards for protection against radiation. U.S. Supt. of Public. Doc. Washington, D.C.

Vachaud, G., J. M. Royer, and J. D. Cooper. 1977. Comparison of methods of calibration of a neutron probe by gravimetry or neutron-capture model. J. Hydrol. 34:343–356.

Van Bavel, C. H. M., D. R. Nielsen, and J. M. Davidson. 1961. Calibration and characteristics of two neutron moisture probes. Soil Sci. Soc. Am. Proc. 25:329–334.

Van Bavel, C. H. M., N. Underwood, and R. W. Swanson. 1956. Soil moisture measurement by neutron moderation. Soil Sci. 82:29–41.

Visvalingam, M., and J. D. Tandy. 1972. The neutron method for measuring soil moisture content—a review. J. Soil Sci., 23:499–511.

Weinberg, A. M., and E. P. Wigner. 1958. The physical theory of neutron chain reactors. Univ. of Chicago Press, Chicago.

Zuber, A. and J. F. Cameron. 1966. Neutron soil moisture gauges. Atomic Energy Rev. 4:143–167.

08-29-03 14:27 From-EES-10 +5056653866 T-453 P.029/029 F-704