

W.M. Cornelis (ed.) Proceedings of the International Workshop on (i) Technical aspects and use of wind tunnels for wind-erosion control; (ii) Combined effect of wind and water on erosion processes, Ghent, Belgium. 17–18 Nov. 1997. I.C.E. Special Report No. 1998/1. International Centre for Eremology, Univ. of Ghent, Belgium.

Sterk, G., A.F.G. Jacobs, and J.H. van Boxel. 1998. The effect of turbulent flow structures on saltation sand transport in the atmospheric boundary layer. *Earth Surf. Proc. Landforms* 23:877–887.

Sterk, G., and W.P. Spaan. 1997. Wind erosion control with crop residues in the Sahel. *Soil Sci. Soc. Am. J.* 61:911–917.

Wallace, J.M.H., H. Eckelmann, and R.S. Brodkey. 1972. The wall region in turbulent shear flow. *J. Fluid Mech.* 54:39–48.

Wolfe, S.A., and W.G. Nickling. 1993. The protective role of sparse vegetation in wind erosion. *Progr. Phys. Geograph.* 17:50–68.

## Soil Particle Concentrations and Size Analysis Using a Dielectric Method

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### ABSTRACT

Limitations of traditional methods for particle-size analysis warrant the investigation of new techniques. An alternative method based on the difference between the dielectric constant of soil solids ( $\approx 4$ ) and dispersing solution ( $\approx 81$ ) was developed. We determined changes in suspended sediment concentrations ( $C$ ) using a coaxial probe placed on the surface of a dispersed soil suspension by monitoring changes in the apparent dielectric constant with time following complete mixing. A single-point calibration for each sample was obtained using the known initial concentration. A refractive index ( $n$ ) model of the suspension dielectric properties gave the slope of a  $C$  vs.  $n$  curve for changes in silt-size (0.002–0.05 mm) particles. A magnetic stirring rod was used to homogenize the dispersion, and temperature changes were minimized given the rapid measurement time. Using the dielectric method, particle-size distributions were measured on a 1- to 2-g sample with 400-s settling time because the effective depth of measurement was only 1.5 mm. Wet sieving was used to remove the sand fraction. Comparisons between the silt and clay fractions obtained using the dielectric and pipette methods were in agreement. The combination of speed, automation, small sample size, and nearly continuous data should be balanced against the higher cost of the equipment necessary for the dielectric method.

FOR MANY YEARS, sedimentation methods have been used for measuring soil particle-size distributions. In the hydrometer method, first described by Bouyoucos (1925), the floatation depth of a hydrometer is measured as a function of time, providing an indication of the solution density. Soil particles, being denser than the aqueous solution in which they are dispersed, tend to settle with time and the fraction of particles remaining in suspension at a given measurement depth is estimated from the fluid density. An analysis involving Stokes' law (Casagrande, 1934), which states that the terminal velocity of a particle is proportional to the square of particle radius, is used to estimate the particle-size distribution. A more accurate approach using the same theory is the pipette method, which apparently was developed more or less simultaneously by three different research groups (Jennings et al., 1922; Krauss, 1923; Robinson,

1922). In this approach, solution, containing sediment particles, is directly sampled at a given depth and time, and particle concentrations are measured by gravimetric analysis. Because of its greater accuracy, the pipette method is often used as a standard to which other particle-size methods are compared (Gee and Bauder, 1986).

Standard instruments for the pipette and hydrometer methods of soil particle-size analysis are not usually automated to provide continuous readings of particle sizes. Continuous, or nearly continuous, measurements of particle-size analysis include scattering techniques (light, neutron, and x-ray), size exclusion and hydrodynamic chromatography, field flow fractionation, electrozone sensing, sedimentation and centrifugation, sieving and filtration, ultrasonic measurements, aerosol time-of-flight measurements, and electric birefringence transient measurements (Barth and Flippen, 1995). Correlative studies between the pipette technique and more automated means of particle-size analysis (e.g., Pennington and Lewis, 1979; Konert and Vandenberghe, 1997) show that the clay fraction ( $< 2 \mu\text{m}$ ) is either occasionally or systematically underreported by as much as 30 to 66%, most likely related to assumptions regarding shape factors for nonspherical particles inherent in each method, including sedimentation. It has been argued (Syvitski et al., 1991) that particle-size analyzers should no longer have their results compared with classical techniques such as sieving and pipetting. However, since soil size fractions are classically defined in terms of equivalent spheres with regard to Stokes' law, acceptance of techniques other than sedimentation may represent a break with methods in use in soil science for more than 60 yr. Interestingly, recent developments in soil particle-size analysis include the use of a sedimentation and gamma attenuation approach (Oliveira et al., 1997) to yield nearly continuous measurements, although these involve expensive and hazardous equipment.

We will describe here a rapid radio-wave electronic system for continuously monitoring particle concentration in a settling soil dispersion, which we believe to be a novel application of well-established principles. Differences in apparent dielectric constant of soil particles ( $\approx 4$ ), soil air ( $\approx 1$ ), and water ( $\approx 81$ ) have been used for years as the basis of in situ determination of the amount of soil water in a matrix of soil particles by time

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domain reflectometry (TDR; Topp et al., 1980). We propose using differences in the dielectric constant to measure the concentration of dispersed soil particles settling in a solution of sodium polymetaphosphate by measuring the refractive index (the square root of apparent dielectric constant) with a coaxial probe dielectric water content sensor and interpreting the combined effects of soil solids, bound water, and dispersing solution on the refractive index measured in solution using an extension of the refractive index model presented by Whalley, (1993) that was applied to the soil matrix. Our objective is to describe the new method and the theory on which it relies.

## THEORY

The time ( $t$ ) required for a particle of diameter ( $d$ ) to settle a distance ( $h$ ) traveling at its terminal velocity (given by Stokes' law) depends on the density of the particle ( $\rho_p$ ), the density of the liquid ( $\rho_l$ ), and the viscosity of the liquid ( $\eta$ ). It is typically assumed (Gee and Bauder, 1986) that all particles with diameters  $>d$  will be absent (settled out) and those with diameters  $<d$  will be present in their initial concentration after a time given by:

$$t = 18\eta h / [g(\rho_p - \rho_l)d^2] \quad [1]$$

where  $g$  is the gravitational constant ( $9.8 \text{ m s}^{-2}$ ). Thus, by measuring the soil concentration at some calculated time and given depth, a measure is made of the concentration of particles with diameters less than a given diameter. Such a measure of the clay fraction ( $d < 2 \mu\text{m}$ ) for a typical depth of 10 cm has an associated settling period on the order of 8 h, but if the depth of sampling is reduced to 1.5 mm, this time is reduced to  $\approx 400$  s. Such a shallow depth of measurement is not feasible with any method of measurement previously described in the literature, but is possible with the method presented here.

The method employs a measurement of refractive index (the square root of apparent dielectric constant) in solution as a function of time during settling. As the soil particles with a low refractive index ( $n = 2$ ) settle out of the measurement volume and are replaced with water ( $n = 9$ ), the refractive index increases. Changes in refractive index can be used to determine concentration changes based on the refractive index of particles settling out of solution. Further, if the initial concentration is known and initial refractive index is measured, then a known point (calibration) is obtained and only relative measurements of refractive index are needed. In an ideal Stokes' model of particle settling, throughout the period when silt settles out of the measurement volume, the clay concentration does not change. Thus, the poorly understood dielectric properties of clay and associated bound water (Hilhorst and Dirksen, 1994) are approximately a constant throughout the duration of the experiment.

The approach taken by Whalley (1993) modeling TDR measurements of soil water content was based on the refractive indices and volume fractions of soil

constituents. This model was extended and applied to solutions composed of solvent mixtures in Starr et al. (2000), where agreement between model and measured refractive index was about  $\pm 0.2$ . Predictive equations of the model were determined by equating the refractive index ( $n$ ) measured in composite dispersion with the sum of the refractive indices of each component weighted by the respective component volume fraction. Although this model was introduced (Whalley, 1993) with little more than an intuitive theoretical underpinning and to our knowledge has not been derived from first principles of electromagnetic physics, it does lead to linear and closed-form equations, as will be shown. In our model of soil dispersion, the volume fractions were converted to a mass per unit volume, or concentration ( $C$ ), using the density ( $\rho$ ) of the respective constituent ( $2.65 \text{ kg L}^{-1}$  for soil solids and  $1 \text{ kg L}^{-1}$  for water) resulting in Eq. [2]:

$$n = \sum_i n_i C_i / \rho_i \quad [2]$$

The sum across all constituents ( $i$ ) could include free water, soil separates, organic matter, and bound water. Thus, a more specific equation to represent soil dispersion can be written where the refractive index of the composite dispersion equals a sum of terms specific to each constituent:

$$n = n_{sa} C_{sa} / \rho_{sa} + n_{si} C_{si} / \rho_{si} + n_c C_c / \rho_c + n_{om} C_{om} / \rho_{om} + n_b C_b / \rho_b + n_w C_w / \rho_w \quad [3]$$

In Eq. [3]  $n$ ,  $\rho$ , and  $C$  have the following subscripts: sa, si, c, om, b, and w, defining the sand, silt, clay, organic matter, bound water, and free water constituents, respectively. The  $n$  of soil separates can be assumed to be 2 (apparent dielectric constant of 4) and their density can be assigned a value of  $2.65 \text{ kg L}^{-1}$ , whereas free water has an  $n$  of 9 and a density of  $1 \text{ kg L}^{-1}$ . To obtain complete dispersion (Gee and Bauder, 1986) of soils with appreciable organic matter, destruction or removal of the organic matter is necessary and the organic matter term will, therefore, be negligible. In a settling experiment, the concentration of soil separates,  $C_s$ , is a parameter that must be determined and it is described by the following equation:

$$C_s = C_{sa} + C_{si} + C_c \quad [4]$$

It is desirable to consider changes in concentration as they relate to refractive index changes in a settling dispersion because changes in refractive index may be much more accurately measured and because this allows for simplification of the model, as will be shown. Prior to the time when clay begins to settle out of the measurement volume,  $C_c$  and  $C_b$  are invariant. If, in addition, sand has been removed from the dispersion by sieving and organic matter has been destroyed (as is typical with the pipette method) then the rate of change of refractive index with concentration when silt is settling can be derived by differentiating Eq. [3]:

$$\partial n / \partial C_{si} = (n_{si} / \rho_{si}) + (n_w / \rho_w) \partial C_w / \partial C_{si} \quad [5]$$

It is also straightforward to show that for silt settling:

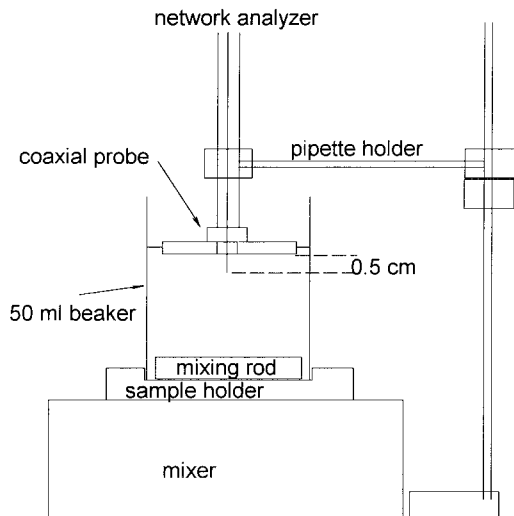


Fig. 1. Schematic of the dielectric particle-size analysis system.

$$\partial C_w / \partial C_{si} = -\rho_w / \rho_{si} \quad [6]$$

So by substitution with Eq. [6], Eq. [5] can be rewritten as:

$$\partial n / \partial C_{si} = (n_{si} - n_w) / \rho_{si} \quad [7]$$

yielding the result that the derivative of  $n$  with respect to concentration during silt settling is a constant given by the right-hand side of Eq. [7]. Integration of Eq. [7] from the initial time (when there is a complete homogeneous dispersion) to some later time (prior to clay settling) yields the following:

$$n(t) - n(0) = (n_{si} - n_w)[C_{si}(t) - C_{si}(0)] / \rho_{si} \quad [8]$$

Notice that the difference in silt concentration equals the difference in total soil concentration ( $C_s$ ) during this time period. Substituting  $[C_s(t) - C_s(0)]$  for  $[C_{si}(t) - C_{si}(0)]$  and rearranging terms, a governing equation is derived that has a closed mathematical form independent of the comparatively unknown clay and bound water quantities:

$$C_s(t) - C_s(0) = \rho_{si}[n(t) - n(0)] / (n_{si} - n_w) \quad [9]$$

This is a useful equation for determining concentration vs. time, because the change in refractive index  $[n(t) -$

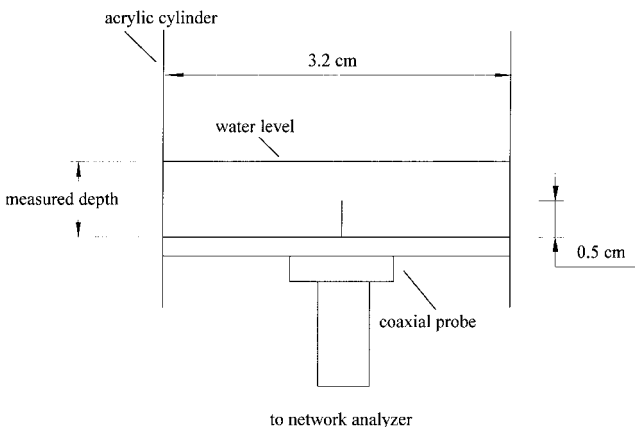


Fig. 2. Probe arrangement for determining effective probe measurement depth.

$n(0)]$  can be measured as a function of time during settling, the initial concentration  $C_s(0)$  is known or can be determined by oven drying, and the density and refractive indices of the silt and water are constants that can be accurately estimated.

Water is bound to the clay fraction and both the density of this bound water and its refractive index may be variable, depending among other things, on how tightly the water is bound. For simplicity, the bound water may be assumed to have a refractive index of 2 and a density of  $1 \text{ kg L}^{-1}$ . The concentration of bound water can be assumed proportional to the concentration of clay because the bound water is integrally associated with clay in a lattice type structure. The constant of proportionality, here termed the bound water fraction,  $\eta$ , can be expressed by the equation:

$$\eta = C_b / C_c \quad [10]$$

This model or simple treatment of clay bound water is something an oversimplification, but provided  $\eta$  is measurable (as will be shown), it gives insight into bound water-clay interactions. To show this, a determination of  $\eta$  was obtained by considering dispersion with only the clay fraction. This type of dispersion can be isolated by sedimentation fractionation (Gee and Bauder, 1986). As before, changes in refractive index with changes in concentration are to be considered, but the organic matter, sand, and silt terms of Eq. [3] are all eliminated. In a manner similar to the derivation of Eq. [7], the following can be shown by differentiating Eq. [3] with respect to concentration of clay:

$$\partial n / \partial C_c = (n_c / \rho_c) + (\eta n_b / \rho_b) - (n_w / \rho_c) \quad [11]$$

Solving Eq. [11] for  $\eta$  yields an equation that can be evaluated provided the change in refractive index with clay concentration is measured and the refractive indices and density of clay and bound water are estimated:

$$\eta = \rho_b(\partial n / \partial C_c - n_c / \rho_c + n_w / \rho_c) / n_b \quad [12]$$

The slope of an  $n$  vs.  $C_c$  curve ( $\partial n / \partial C_c$ ) can be measured

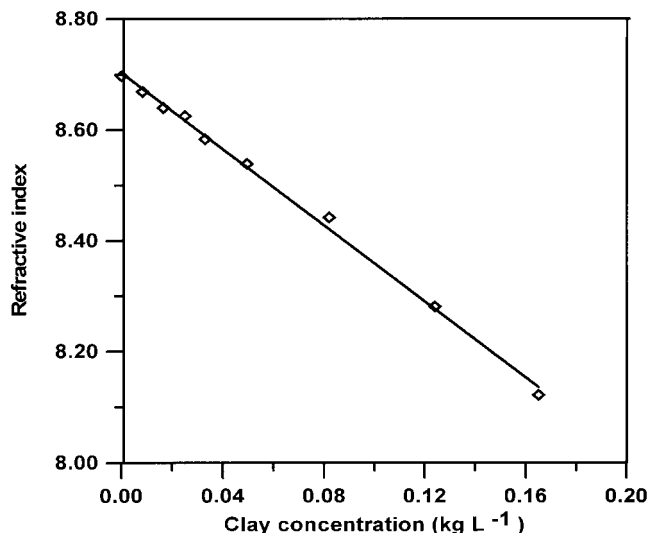


Fig. 3. Relationship between refractive index and concentration of the clay fraction (<2 μm) of Plano soil.

by repeatedly diluting a clay dispersion and measuring  $n$  at each dilution step, then plotting the data and fitting a linear regression line. From this plot,  $\eta$  can be determined using Eq. [12]. One of the most significant aspects of this model is the prediction that the measured refractive index will be linearly related to the particle concentration during settling.

Variation in soil bulk density, organic matter, and clay content (with associated bound water) can cause slight variations in soil dielectric properties thus requiring calibration of dielectric water content sensors such as time domain reflectometers for highly accurate readings in fine-textured and organic soils (Topp et al., 1994). In our experiments, the organic matter was destroyed (with  $H_2O_2$ ) prior to dispersion, the clay and bound water are invariant throughout the experiment, air is not present, and the particle concentration (equivalent to bulk density in soils) is the only soil variable causing changes in the refractive index. If the range of refractive index of silt were from 1.75 to 2.25 (soil particle apparent dielectric constant may range from 3 to 5), the slope of the  $C$  vs.  $n$  curve [ $\rho_{si}/(n_{si} - n_w)$  as in Eq. [9)] varies by only  $\pm 3.5\%$  of its value for  $n_{si} = 2$ , leading to a small but acceptable error in concentration. Similarly, in the soil matrix, Hook and Livingston (1996) found that the slope of the water content vs.  $n$  curve was nearly invariant (using TDR) and approximately equaled the slope predicted by Whalley (1993). In theory, Eq. [9] should be applicable to the silt fraction of all soils without modification.

## MATERIALS AND METHODS

The refractive index ( $n$ ), was measured at a frequency of 795 MHz using a HP8712B vector network analyzer (Hewlett

Packard, Santa Rosa, CA)<sup>1</sup> and a custom-made coaxial probe dielectric water content sensor (Starr et al., 2000). This probe was constructed from a coaxial cable with the ground connected to a metal flange (large stainless steel washer) and the center conductor connected to a spiked extension (male connector pin) extending 0.5 cm beyond the flange. The phase shift of 795 MHz sine waves was measured repeatedly (201 measurements per trace). These data were displayed on the analyzer and mean phase shift was recorded for further analysis. Refractive index is linear in relation to phase shift (Starr, 1997) when changes in  $n$  are small. A relationship between  $n$  and phase shift was derived (Starr et al., 2000) across a broader  $n$  range ( $n = 1-9$ ) for this probe and frequency and the Starr et al. (2000) method has been used for reducing the measured mean phase shift into refractive index in this study. The trace phase shift was observed to change with soil particle concentration during settling experiments. The trace observations were also used for detecting trapped air bubbles in the dispersion. A previous study (Starr et al., 2000) showed agreement between refractive index model and  $n$  measurements of about  $\pm 0.2$  in the absolute sense. When measuring small  $n$  changes the accuracy will be considerably greater than for absolute  $n$  determination.

The coaxial probe was placed at the top (surface) (Fig. 1) of a 50-mL glass cylinder (beaker) containing 40 mL of a mixture containing  $5 \text{ g L}^{-1}$  sodium polymetaphosphate, water, and soil along with a magnetic stirring rod. A pipette holder was used to raise and lower the probe into contact with the sample surface. A wooden sample holder secured the sample during mixing and provided insulation from the warming effects of the magnetic mixer. Parafilm was used to seal the top of the beaker to prevent evaporation.

The prediction of the mixing model that the refractive index was linearly related to the particle concentration was tested

<sup>1</sup> Mention of a trade name or company does not constitute endorsement by the USDA-ARS.

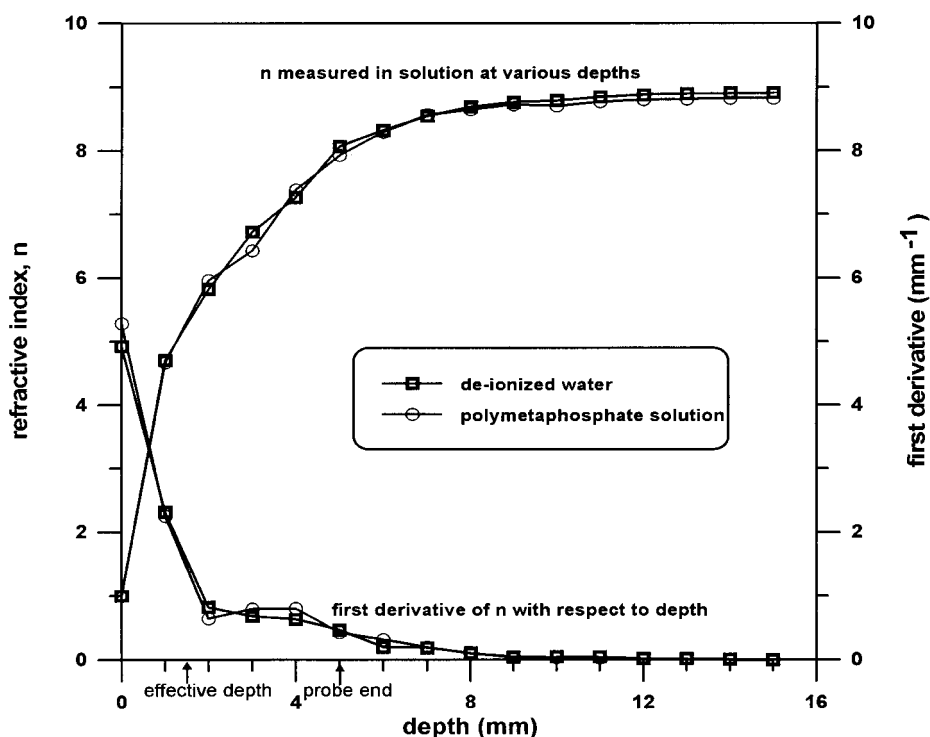


Fig. 4. Measurement depth of the coaxial probe. Derivatives calculated by the modified Savitzky-Golay method of Barak (1995).



using particles that would not appreciably settle during measurement (i.e., clay-size particles). For this purpose, a Plano silt loam soil (mixed, mesic, Typic Argiudoll) from Arlington, WI was dispersed with sodium polymetaphosphate solution and the  $<2\text{-}\mu\text{m}$  fraction was decanted. The clay fraction of this soil is dominated by interstratified smectite-illite with lesser amounts of illite, kaolinite, and quartz (Liu et al., 1997). A series of dilutions with additional dispersant solution were prepared and the refractive indices were measured in the apparatus described above.

The measurement depth of the coaxial probe was investigated by inverting the probe with the spiked extension pointed upward and affixing an acrylic cylinder around the outside ground plane (3.2-cm diameter) with paraffin wax (Fig. 2). A few simple tests were conducted to ensure that the chamber contained the entire measurement volume of the probe. With the chamber filled with water, first a glass stirring rod and a then a razor blade were inserted into the chamber to see, roughly, where the instrument began to respond to these aberrations in the chamber. These objects caused appreciable  $n$  changes only when they were within  $\approx 1$  cm of the spiked extension either diametrically or vertically. Placing ones hands around the chamber and observing the signal showed that there was a small shift in measured  $n$  presumably caused by a small amount of transmitted and reflected radiation from the probe; however, this was deemed negligible.

Solution, both deionized water and  $5\text{ g L}^{-1}$  sodium polymetaphosphate, was added incrementally and both height of solution and the refractive index,  $n$ , were measured. The effective measurement depth was further measured using the silt fraction of a pulverized silica sand powder (obtained from Timco

Inc., Sauk City, WI), which was wet sieved to remove sand and decanted repeatedly to remove clay-sized particles (after Gee and Bauder, 1986). This silica silt was further fractionated by mixing in a 1-L cylinder and drawing 40-mL samples at a depth of 10 cm at selected times to obtain samples with known particle-size ranges (calculated from Eq. [1]) for analysis with the dielectric method. Settling time of particles with a known minimum diameter of silt ( $2\ \mu\text{m}$ ) was used to calculate the effective depth of measurement from Eq. [1].

Soil samples taken from different locations in the soil profile of Plano silt were also analyzed with both the pipette method and the dielectric method. The standard pipette method protocol (Gee and Bauder, 1986) was used for preparing soil dispersions with organic matter removed with a  $\text{H}_2\text{O}_2$  treatment and wet sieving to remove sand. One-liter samples were prepared and pipette analysis conducted to determine the silt and clay concentrations. These samples were then dispersed again and 40-mL subsamples were extracted for analysis by the dielectric method.

Since achieving a uniform dispersion at the initiation of the experiment and measuring  $n(0)$  are critical to the success of the measurement method, the mixer was run at sufficiently high speed to stir particles, but slow enough that air bubbles did not enter under the ground plane of the probe. Spinning the soil solution in this manner causes particles to accumulate at the edge of the beaker as in a centrifuge. When the mixer is turned off, the magnetic mixing rod stops immediately, applying a braking effect on the swirling mixture. This causes the fluid at the edges to circulate downward and flow upward at the center, homogenizing the mixture. By first rapidly swirling the mixture, then stopping and starting the mixer three

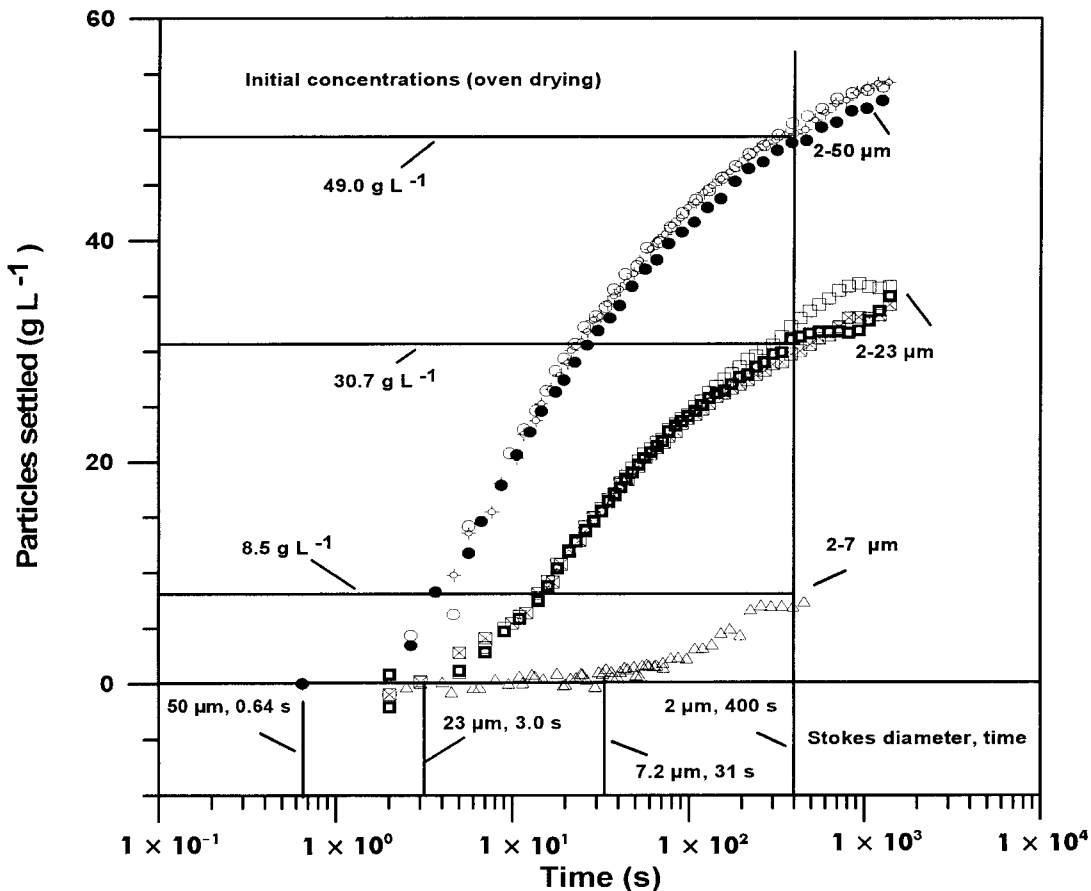


Fig. 5. Testing and calibration of the dielectric system in silica silt.

or more times at  $\approx 2$ -s intervals, a homogeneous dispersion is obtained. The measurement of initial concentration must be made within the first 2 s after the mixer is turned off, during the period when the central upward flow and turbulence are sufficient to overcome the rapid settling of the larger silt particles.

Temperature has a significant effect on the magnitude of the measured refractive index because of the well-known temperature dependence of  $n_w$  (Weast et al., 1986). The success of this experiment relies on measurements of the changes in refractive index, not on its absolute value. Provided that temperature does not change during the 400-s interval of settling, temperature corrections are theoretically unnecessary. However, the typical particle concentration and refractive index changes during particle settling are so small that a temperature change of  $1^\circ\text{C}$  causes unacceptable errors. This potential pitfall was discovered when samples were exposed to open air and evaporation from the sample caused significant ( $\approx 3^\circ\text{C}$ ) reduction in the sample temperature. When a cooler-than-ambient sample was covered with the coaxial probe, the temperature of the solution increased for almost 30 min and caused erroneous results. This problem was solved by either covering the sample with cellophane prior to and during measurement or by waiting until temperature had equilibrated. Another temperature problem occurs when a wet probe is exposed to air and the temperature of the metal probe rapidly drops because of evaporative cooling. Care should be exercised to quickly wipe the probe dry after each sample run to control

this potential pitfall, as well as to avoid temperatures caused by heating upon contact with bare hands. Although temperature correction could have been applied, in these experiments, with careful handling and operation, it was judged unnecessary.

## RESULTS AND DISCUSSION

The component mixing model to interpret the combined effects of soil solids, bound water, and dispersing solution on the refractive index predicts a linear relationship between particle concentration and refractive index. Such a linear relationship was measured for the  $<2\text{-}\mu\text{m}$  fraction of Plano silt loam (Fig. 3). The use of clay, particularly an expansible clay such as the smectitic-illitic clay used here, adds a level of complexity to the problem due to the presence of bound water associated with the clay itself. The mixing model can be used to calculate the bound water fraction,  $\eta$ , from these data using the formula in Eq. [12], yielding a value of  $0.14\text{ kg bound water kg}^{-1}\text{ clay}$ . By contrast, the bound water measured in a bentonite suspension by the same technique (data not shown) was  $1.75\text{ kg kg}^{-1}$ , an order of magnitude higher, indicating that much more water is tightly bound by bentonite than the naturally occurring clay minerals in the Plano surface soil. Because of differences in the dielectric properties of clay miner-

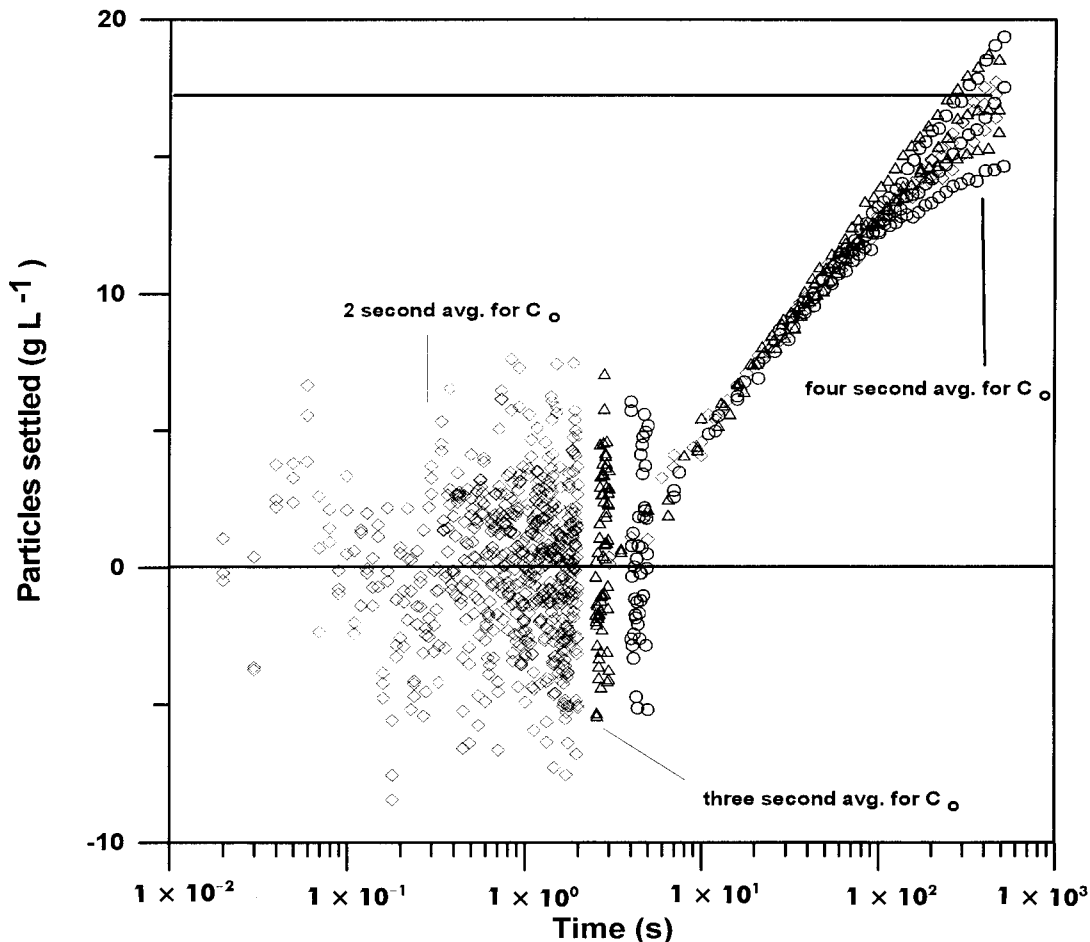


Fig. 6. Time interval of initial signal averaging and its impact on concentration measurements.

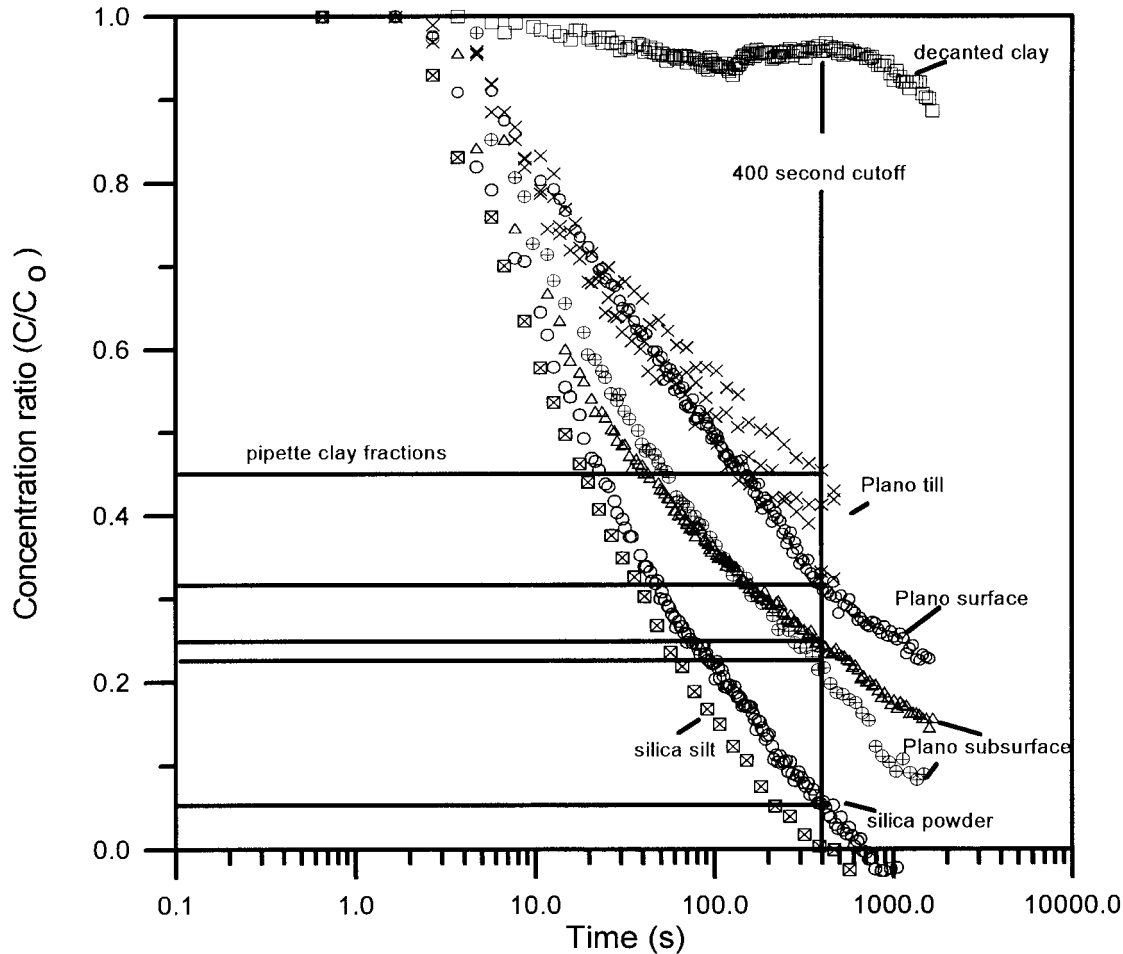


Fig. 7. Concentration ratio ( $C/C_0$ ) vs. time for several soils.

als and associated bound water, the line in Fig. 3 should not be used for a calibration describing silt settling, but does provide confirmation of the mixing model.

The probe response vs. measurement depth (Fig. 4) was virtually identical for both deionized water and sodium polymetaphosphate solution. The greatest incremental response was measured for the first 1 mm, which was  $\approx 2.5$  times more sensitive than the 1- to 2-mm increment; at slightly greater than 1-mm depth, the probe reached 50% of its value at infinite depth. Measurements at subsequent probe depths show a rapid decline in sensitivity toward the end of the spiked extension (5 mm), and the probe was essentially insensitive beyond 8 mm. The similarity in measurement for both deionized water and for the much more conductive ( $3.3 \text{ dS m}^{-1}$  at  $25^\circ\text{C}$ ) sodium polymetaphosphate solution (Fig. 4) allay concern that these reflectivity measurements might be affected by the electrical conductivity of the chemical dispersant.

To obtain a more appropriate measurement to determine the apparent depth for applying Stokes' law on the basis of particle settling, a set of measurements for calibration and testing (Fig. 5) using silica powder of known size ranges and initial concentrations was used. Shown in Fig. 5 are data on particles settled ( $C - C_0$ ) calculated from Eq. [9] vs. time for three size ranges

( $2-50$ ,  $2-23$ , and  $2-7 \mu\text{m}$ ). Three repetitions of the settling experiment (for the  $2-50$  and  $2-23 \mu\text{m}$  size ranges) show a close grouping of concentrations, indicating good repeatability, and particles settled equals the initial concentration after  $\approx 400$  s (there was only one replication for the  $2-7 \mu\text{m}$  size range). Essentially, after 400 s, all the particles in the dispersion have settled below the apparent depth of measurement. Because the smallest particles (here  $2 \mu\text{m}$ ) settle last, a value of  $2 \mu\text{m}$  and 400 s can be used to calculate an effective probe measurement depth from Eq. [1], yielding a result of 1.5 mm. This depth is in general agreement with the approximation for median depth (1–2 mm) and is slightly less than the weighted average depth (2.2 mm) from Fig. 4.

Some of the limitations of using an apparent depth with this type of probe are seen when the particles continue to settle after 400 s. This is because some of the smaller particles are still settling from the lower regions of the measurement volume where the probe has some sensitivity. Effective depth, calculated in this way, is therefore a calibration to determine size distribution for the silt-clay boundary at  $2 \mu\text{m}$ ; however, it can also be used to calculate the times at which particles begin to settle. These times, shown in Fig. 5 for the 50, 23, and  $7 \mu\text{m}$  limits, agree quite well with the times when the particles are observed to start settling. In the

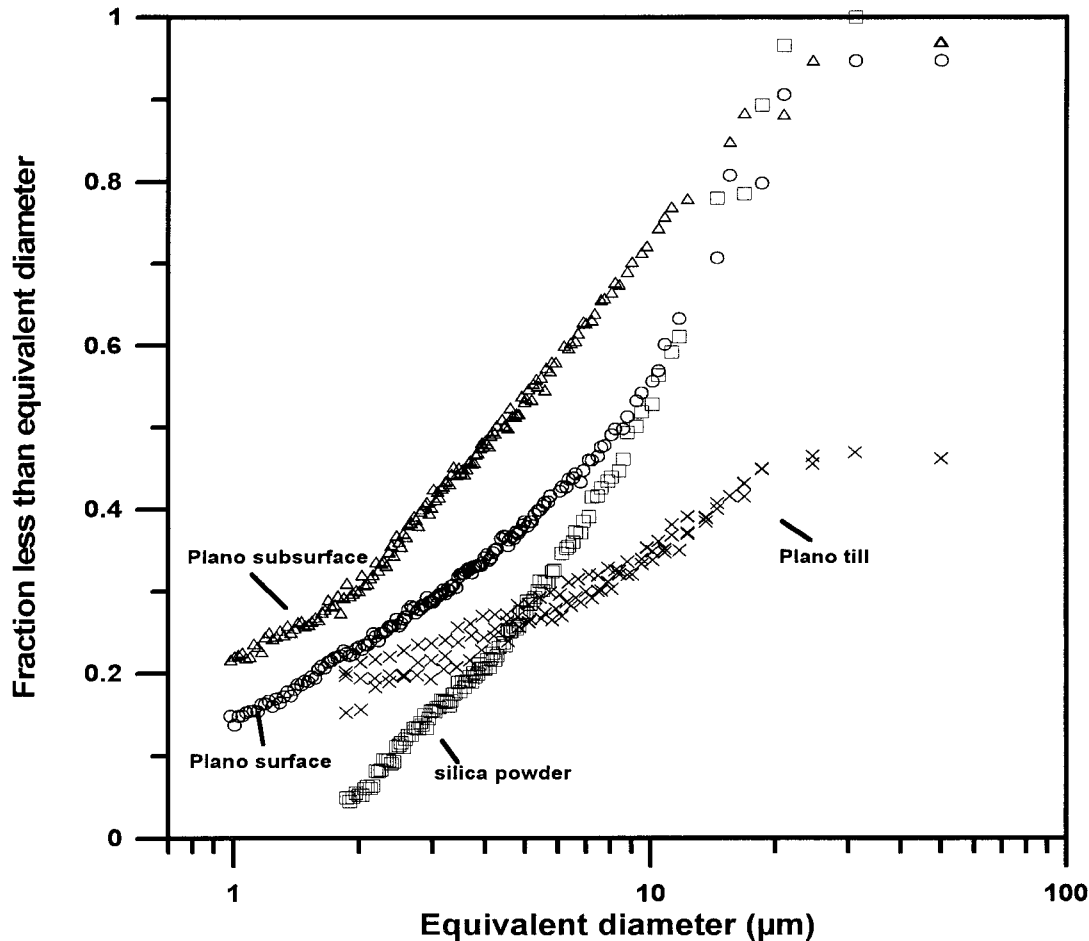


Fig. 8. Summation curves for several soils.

case of  $7 \mu\text{m}$ , the particles begin settling slightly before the calculated time of 31 s and this small aberration can also be explained by error in using an effective depth. These small deviations notwithstanding, the application of Stokes' law and the effective depth calculation do appear to work as expected for describing the settling process.

The initiation of settling was 0.64 s for the  $50\text{-}\mu\text{m}$  limit, and this led us to believe that there might be less than a second to measure initial concentration before silt began to settle. However, turbulence from mixing the chamber keeps silt in suspension for  $\approx 2$  s. Measurements of the type shown in Fig. 6 show that waiting beyond 2 s to measure  $n(0)$  leads to unacceptable errors. Silica silt settling data are shown in Fig. 6 for 2-, 3-, and 4-s signal averaging to determine  $n(0)$ , and the repeatability of the experiment is seen to deteriorate with time of initial measurement beyond 2 s. However, there was no obvious trend in the data that would suggest settling before 2 s. Signal averaging of 200 raw data points across a time period of 1.4 s was used in the particle-size analysis measurements that are described below.

The concentration ratio  $[C(t)/C(0)]$  can also be calculated from Eq. [9] provided the initial concentration is known [we determined  $C(0)$  by oven drying the 40-mL sample]. A plot of concentration ratio vs. time (Fig. 7) shows the settling behavior for decanted clay, silica

powder ( $<50 \mu\text{m}$ ), silica silt ( $2\text{--}50 \mu\text{m}$ ), and several samples from a Plano soil: Plano surface (A horizon), two samples from the Plano subsurface ( $B_1$  horizon), and one from the till (glacial parent material). The dielectric method was used on subsamples from a 1-L dispersion that was also analyzed using the pipette method and the fractional concentration of silt/(silt + clay) is shown graphically to agree very well between the two methods at 400 s. There was considerably more spread in the till soil data than the other samples. This till dispersion had a low initial concentration ( $\approx 8 \text{ g L}^{-1}$ ) because most of the sample was sieved out as sand and coarse fragments. This concentration ( $8 \text{ g L}^{-1}$ ) is about the minimum required for the dielectric method of particle-size analysis. With lower concentrations, the results are increasingly marginal.

The experimental apparatus does not have an ideal measurement volume (i.e., a point measurement at a precisely defined depth) for the application of Stokes' law. This may result in observations that deviate from the predictions of the Stokes' law model. For example, the decanted clay ( $<2 \mu\text{m}$ ) begins to settle somewhat before the predicted 400 s and the silica silt (which would have entirely settled out of an ideal measurement volume) continues settling after the predicted 400 s, observed by the slightly negative concentration ratio occurring after 400 s. However, neither of these devia-



tions from the ideal behavior poses serious problems for determining the fraction of clay in a mixture of silt and clay, as evidenced by the pipette method calculations of the fraction of clay/(silt + clay) for the Plano soils. These values intersected the  $C(t)/C(0)$  vs. time curve (Fig. 7) near the predicted 400-s cutoff time, when  $C(t)/C(0) = \text{clay}/(\text{silt} + \text{clay})$ , indicating satisfactory agreement between the dielectric and pipette methods.

A 1.5-mm apparent depth was used in Stokes' law to calculate the appropriate particle diameters for the classical summation curves (Fig. 8). The data are graphed as the fraction less than particle diameter vs. particle diameter, showing several of the expected features for these soils. The higher clay content of the subsurface as compared with the surface horizon and the greater coarse fraction of the glacial till were all expected, as was the generally high silt concentration of this Plano silt loam soil. The data for diameters above  $\approx 10 \mu\text{m}$  are affected by the early turbulence in the settling chamber causing the summation curves to appear flatter in that region than they actually are. For example, the summation curve in the 20- to 50- $\mu\text{m}$  range of Plano till shows a surprisingly flat summation curve, suggesting that this soil has almost no coarse silt. The summation curve for Plano surface drops much faster than the subsurface and till in the range of 10 to 50  $\mu\text{m}$ , indicating that the surface soil has a larger fraction of coarse silt particles than does the subsurface.

Theoretically, Stokes' law applies when the turbulent period of time ( $\approx 3$  s) is small compared with the settling time for a given diameter, which is true for  $\approx 10 \mu\text{m}$  and lower. It is not currently known how reliable the data below 2  $\mu\text{m}$  are. Once clay begins to settle, it will carry bound water and may have a bigger impact on the refractive index changes than silt. Based on these considerations, the measured summation curves are more reliable in an absolute sense between 2 and 10  $\mu\text{m}$  than outside of this range. It requires only  $\approx 27$  min of settling to reach an equivalent Stokes' diameter of 1  $\mu\text{m}$  using the dielectric method, compared with 1 d of settling with the traditional pipette method.

## SUMMARY AND CONCLUSIONS

A dielectric method of soil particle concentration measurement and size analysis has been described, with emphasis placed on the underlying theory and one possible approach to methodology. The coaxial probe-network analyzer system has distinct advantages over the pipette method of particle-size analysis, primarily with regard to speed (400-s settling time to the silt-clay boundary), automation, small sample size (1–2 g soil sample), and the amount of data obtained (nearly continuous). Although capable of making measurements below 2  $\mu\text{m}$ , additional research is needed to determine the dielectric properties of clay. Calibration is obtained from the known initial concentration and measured initial refractive index, and the theoretical slope is also used for the  $C$  vs.  $n$  curve. The system relies on relative measurements of changes in refractive index as they

relate to changes in concentration. Good agreement with the pipette method for the silt-clay boundary indicates that this is a promising method that warrants further study. If, as suggested by both the theory and the data presented here, the same slope can be applied for the calibration of all silt, the method may find widespread applicability in soil science.

## REFERENCES

- Barak, P. 1995. Smoothing and differentiation by adaptive-degree polynomial filter (modified Savitzky-Golay method). *Anal. Chem.* 67:2758–2762.
- Barth, H.G., and R.B. Flippen. 1995. Particle size analysis. *Anal. Chem.* 67:257R–272R.
- Bouyoucos, G.J. 1925. The hydrometer as a new method for the mechanical analysis of soils. *Soil Sci.* 23:343–353.
- Casagrande, A. 1934. Die aräometer-methode zur bestimmung der kornverteilung von boden und anderen materialien. Julius Springer, Berlin.
- Gee, G.W., and J.W. Bauder. 1986. Particle size analysis. p. 383–411. In A. Klute (ed.) *Methods of soil analysis*. Part 1. 2nd ed. Agron. Monogr. 9. ASA and SSSA, Madison, WI.
- Hilhorst, M.A., and C. Dirksen. 1994. Water content sensors: Time domain versus frequency domain. p. 23–33. In *Symp. and workshop on time domain reflectometry in environmental, infrastructure, and mining applications*. 7–9 Sept. 1994. Northwestern Univ., Evanston, IL. Spec. publ. SP19–94. U.S. Dep. of Interior, Bureau of Mines, Washington, DC.
- Hook, W.R., and N.J. Livingston. 1996. Errors in converting time domain reflectometry measurements of propagation velocity to estimates of soil water content. *Soil Sci. Soc. Am. J.* 60:35–41.
- Jennings, D.S., M.D. Thomas, and W. Gardner. 1922. A new method of mechanical analysis of soils. *Soil Sci.* 14:485–499.
- Konert, M., and J. Vandenberghe. 1997. Comparison of laser grain size analysis with pipette and sieve analysis: A solution for the underestimation of the clay fraction. *Sedimentology* 44:523–535.
- Kraus, G. 1923. Über eine neue methode der mechanischen bodenanalyse. *Int. Mitt. Bodenk.* 13:147–160.
- Liu, Y.J., D.A. Laird, and P. Barak. 1997. Release and fixation of ammonium and potassium under long-term fertility management. *Soil Sci. Soc. Am. J.* 61:310–314.
- Oliveira, J.C.M., C.M.P. Vaz, K. Reichardt, and D. Swartzendruber. 1997. Improved soil particle-size analysis by gamma-ray attenuation. *Soil Sci. Soc. Am. J.* 61:23–26.
- Pennington, K.L., and G.C. Lewis. 1979. A comparison of electronic and pipet methods for mechanical analysis of soils. *Soil Sci.* 128: 280–284.
- Robinson, G.W. 1922. A new method for the mechanical analysis of soils and other dispersions. *J. Agric. Sci.* 12:306–321.
- Starr, G.C. 1997. New approaches to soil water content measurement using electromagnetic methods. Ph.D. diss. Univ. of Wisconsin, Madison. Diss. Abstr. DAI-B58/09.
- Starr, G.C., B. Lowery, and E.T. Cooley. 2000. Soil water content determination using a network analyzer and coaxial probe. *Soil Sci. Soc. Am. J.* 64:867–872 (this issue).
- Syvitski, J.P.M., K.W.G. Leblanc, and K.W. Asprey. 1991. Interlaboratory instrument calibration experiment. p. 174–193. In J.P.M. Syvitski (ed.) *Principles, methods, and applications of particle size analysis*. Cambridge Univ. Press, New York.
- Topp, G.C., J.L. Davis, and A.P. Annan. 1980. Electromagnetic determination of soil water content: Measurement in coaxial transmission lines. *Water Resour. Res.* 16:574–582.
- Topp, G.C., S.J. Zegelin, and I. White. 1994. Monitoring soil water content using TDR: An overview of progress. p. 67–79. In *Symp. and workshop on time domain reflectometry in environmental, infrastructure, and mining applications*. 7–9 Sept. 1994. Northwestern Univ., Evanston, IL. Spec. publ. SP19–94. U.S. Dep. of Interior, Bureau of Mines, Washington, DC.
- Weast, R.C., M.J. Astle, and W.H. Beyer (ed.) 1986. *CRC handbook of chemistry and physics*. CRC Press, Boca Raton, FL.
- Whalley, W.R. 1993. Considerations on the use of time domain reflectometry (TDR) for measuring soil water content. *J. Soil Sci.* 44:1–9.