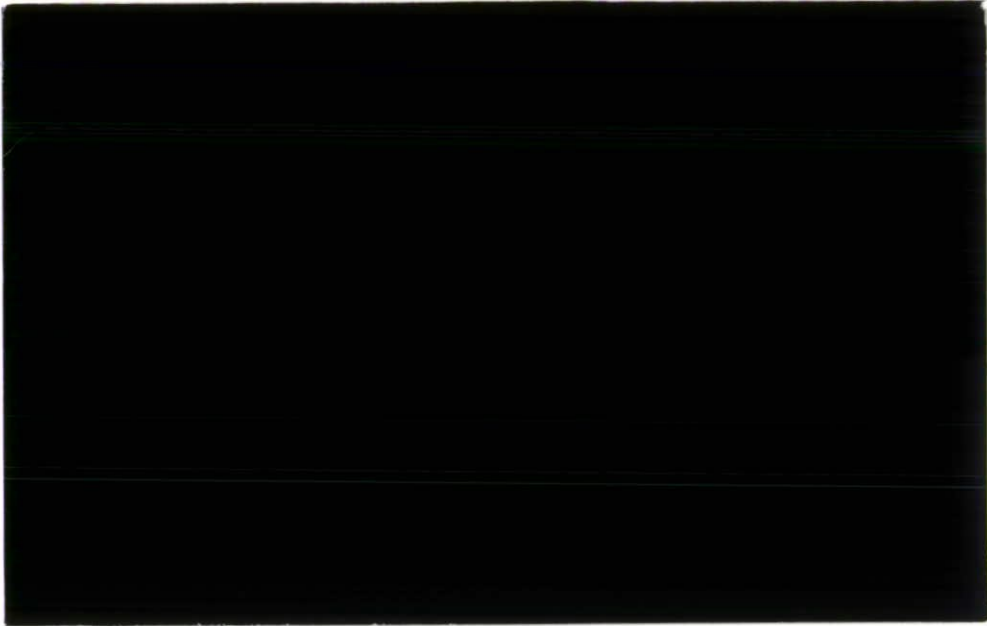




**Institute of
Hydrology**



1994 / 042

**THE INFLUENCE OF SELECTED SOIL MINERALS ON THE
TRASE SYSTEM I TIME-DOMAIN REFLECTOMETER (TDR)**

D.A. ROBINSON & C.H. BATCHELOR

INSTITUTE OF HYDROLOGY (INTERNAL REPORT)

SUSTAINABLE AGROHYDROLOGY SECTION

1994.

CONTENTS

- 1 EXECUTIVE SUMMARY
- 1.1 INTRODUCTION
- 1.2 PRINCIPLES OF OPERATION
- 1.3 TDR WAVEFORM ANALYSIS
- 2 REVIEW OF TDR AND SOIL MINERALOGY.
- 3 MATERIALS AND METHODS
- 4 IRON MINERALS
- 5 TITANIUM MINERALS
- 6 ZIRCON
- 7 CONCLUSIONS
- 8 FUTURE WORK
- 9 REFERENCES

1.

EXECUTIVE SUMMARY

Measurement of soil moisture content based on changes in dielectric constant has become a standard approach in recent years. Time domain reflectometry (TDR), in particular, has gained rapid acceptance as a technique for measuring soil moisture that can be used in a wide range of applications. One factor that dielectric soil moisture determining techniques have in common is the need for calibration. Early work in this field suggested soil type independent calibration, this has since been shown to be an over simplification with more recent work recommending in situ calibration.

Results presented from a series of experiments show the influence of selected iron, titanium and zircon minerals on the calibration of the time domain reflectometer. Magnetite showing the greatest effect by causing an uncertainty of up to 60% in the estimation of volumetric water content in the presence of 15% magnetite.

This work provides a preliminary look at the influence of some oxide minerals on TDR. This has greatest relevance to soils in the Tropics which may be highly weathered and contain a lot of oxides of iron, aluminium or titanium. These may also be the areas where accurate and precise measurements of water content are required as water may well be scarce. The precision of TDR is very high but the relationship between dielectric constant and water content has not been found to be straightforward. It is thus recommended that any work in the field has its own calibration in situ for the best results.

1.1

INTRODUCTION

Techniques such as Time Domain Reflectometry (TDR) and the capacitance probe are rapidly becoming recognised techniques for determining soil volumetric water content. The commercial availability of such techniques as the TDR means relatively easy access to rapid data acquisition, which is none radioactive and none destructive. The methods utilise the large comparative difference in dielectric constant between water(80), air (1) and most soil minerals(2-4). Information on the calibration of the techniques is becoming available in the literature, (Topp *et al.*1980; Roth *et al.* 1992; Jacobsen and Schjonning 1994 and Bell *et al.*1987) but still work is required to improve understanding of the effects of a variety of soil components on these calibrations. It is becoming clear that a single calibration is not satisfactory at present. Topp *et al.* (1980) and Roth *et al.* (1992) have shown that different TDR calibrations are required for mineral and organic soils. Jacobsen & Schonning (1994) have indicated the effects of bulk density on TDR calibration.

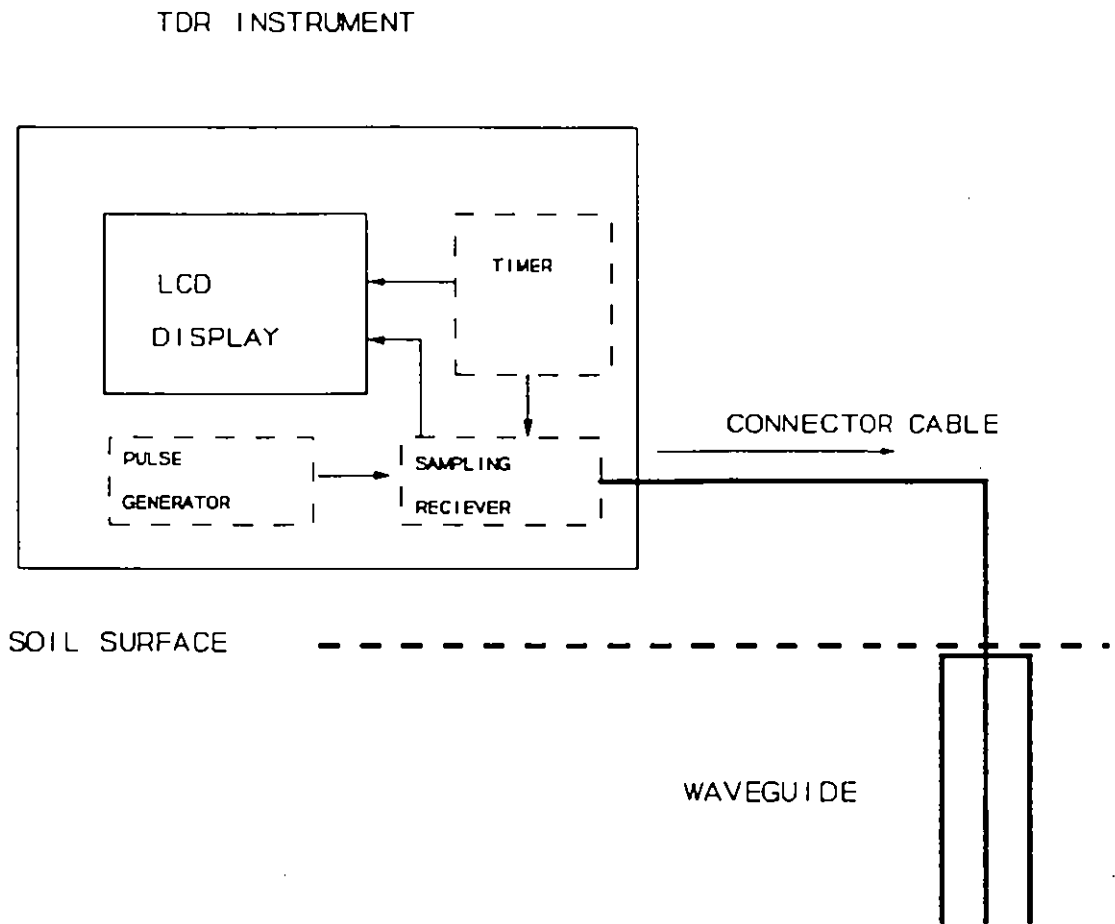
This report aims to examine a different aspect of the calibration, the influence of soil minerals. This is achieved through a series of experiments examining the influence of selected iron, titanium and zircon minerals. Soil minerals have thus far been considered to have very little influence on the calibrations of these techniques as the assumption is made that they all have low dielectric constants. This report examines whether this is a justified assumption by an examination of the literature followed with some exploratory laboratory experiments.

1.2

PRINCIPALS OF OPERATION

The TDR system is shown as a block diagram in Fig.1. It shows that the instrument consists of four major components as described by Topp & Davies (1985). The TDR pulse generator creates a fast rise time step voltage pulse in the order of 10^{-10} sec. This pulse travels past the receiver into the transmission line, it propagates down the line and through the balun which is there to achieve the maximum transmission of the pulse into the soil. The balun is an impedance matching transformer used to balance the pulse for use with the two wire waveguide design. At the soil-waveguide interphase there is an impedance mismatch which means part of the pulse is reflected back toward the instrument the rest of the signal propagates down the waveguide situated in the soil to the end where it is reflected back through the system and picked up by the sampling receiver.

Fig. 1



1.3

TDR WAVEFORM ANALYSIS

The TDR relies on the interpretation of a waveform to calculate the dielectric constant. The Trase TDR contains software which does this automatically. The manual does not describe how this is done but does however state that the trase waveguides contain a marker point so the TDR knows where to place its time domain capture window to sample the waveform. This is an excellent advantage ensuring that the TDR always measures from the same start point. It also means that long cables can be used up to 30 m. The drawback from an experimental point of view is that only the commercial waveguides can be used successfully with the system.

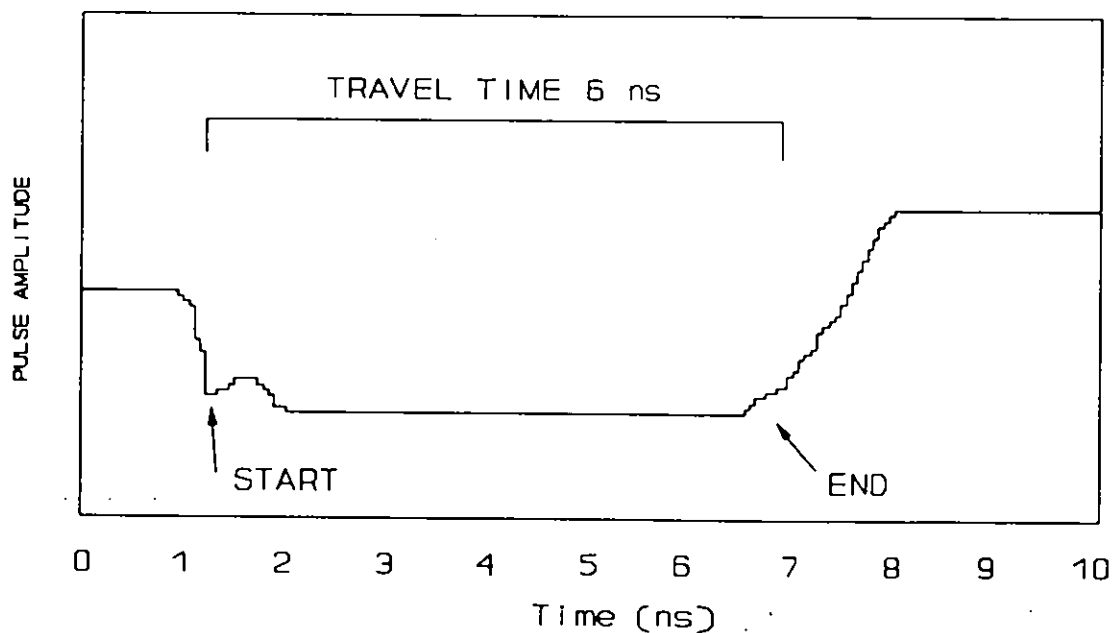
WAVEFORM INTERPRETATION

The importance of waveform analysis to the determination of dielectric constant cannot be stressed enough. It is critical to the calculation of K_a and the subsequent estimation of water content. The calculation of the dielectric constant comes from the travel time of the pulse along the waveguide. The apparent dielectric constant, K_a , is determined from the formula below:

$$K_a = \left(\frac{tc}{L} \right)^2$$

Where t is the travel time of the pulse in nanoseconds, c is the speed of light (29.979 cm/ns) and L is the length of the waveguides. A worked example of this is illustrated in Fig.2 .

TDR WAVEFORM IN BULK WATER

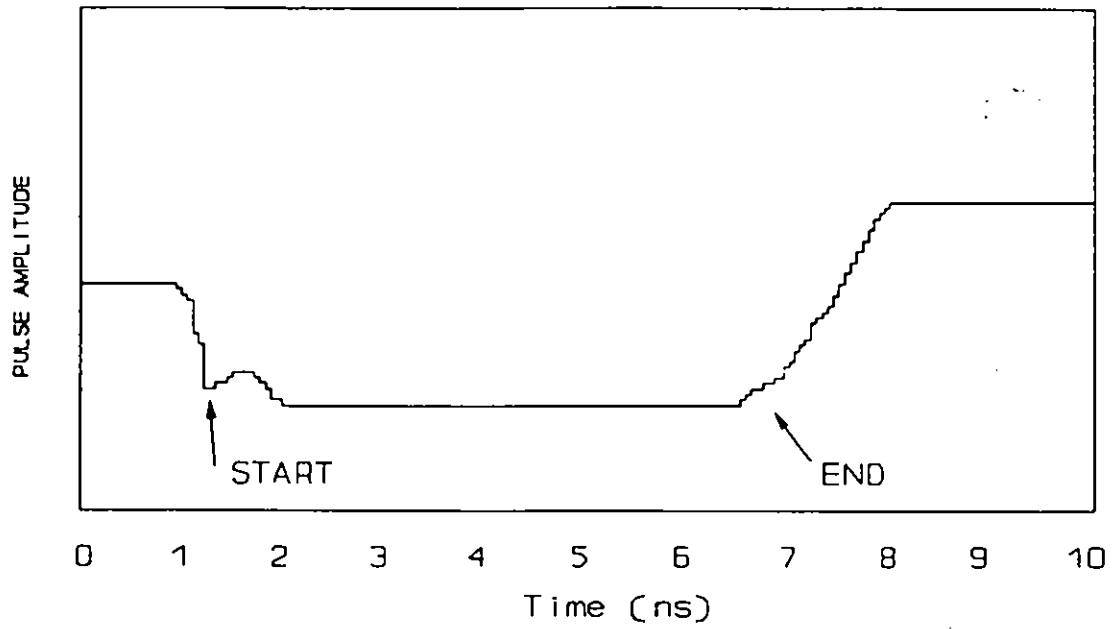


$$K_a = \left[\frac{6 * 29.979}{20} \right]^2 = 80.9$$

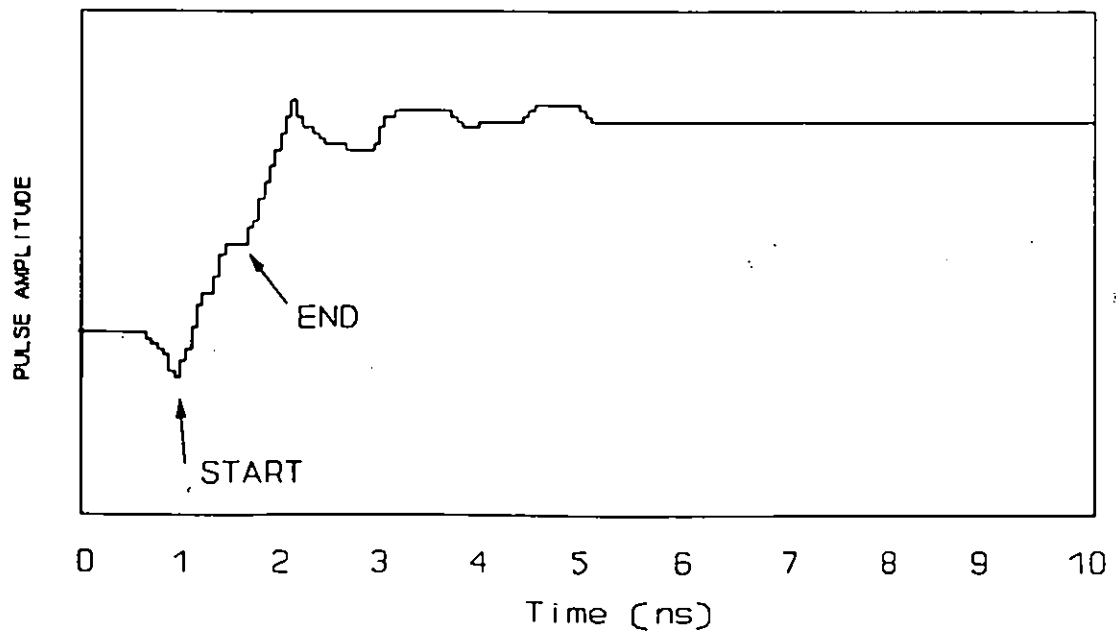
Fig.2

Fig.3 shows how idealised waveforms appear for water (A) and air (B), the diagrams show the start and reflection of the pulse off the end of the waveguide.

TDR WAVEFORM IN BULK WATER



TDR WAVEFORM IN AIR



2.

TDR AND SOIL MINERALOGY

This section reviews the dielectric behaviour of soil minerals. It begins with a brief discussion of dielectric constant as applied to water and soil minerals. The assumption that soil minerals have low dielectric constants is then critically examined on the basis of published literature. The implications of this conclude the chapter with examples taken from laboratory calibrations for clay minerals and iron rich soils.

Dielectric theory and the water molecule

"The dielectric properties of a substance are interpreted in terms of the electric moments, both permanent and induced, of the molecules which compose it." (Hastead, 1973) The water molecule has both a permanent dipole moment and a polarizability which is induced by an electric field and is proportional to its magnitude. Water is the only common rock forming substance to have a marked molecular polarizability, (Carmichael, 1982). In the presence of an electric field the dipoles of the molecule orientate within the field. If the field alternates the water molecules realign with each cycle. Reorientation of the dipoles occurs until the field reverses so rapidly for the molecule to keep up. At this point 'dielectric loss' is said to occur and the dielectric constant reduces.

Dielectric constant (ϵ) can be a misleading term. It is only constant over limited frequency, temperature and pressure. The term permittivity is used in some of the literature and is a less misleading term. The frequency dependence of the dielectric constant of water is stable in the frequency range of the TDR 10 MHz - 1 GHz. The temperature dependence is an important consideration, ice has a dielectric constant of 4. Free water is 80.37 at 20 °C and falls as the temperature rises so that at 25 °C it is 78.54.

The static dielectric constant (ϵ_s)

This is the dielectric constant where the dipole alignment with the electric field is time-independent. The values are often high due to the influence of conduction processes.

Dielectric loss and dispersion ($j\epsilon''$)

The presence of an alternating electric field may give the dipoles insufficient time to realign with the field such that dielectric loss is said to occur. Dielectric loss is dependant on frequency, the higher it becomes the more likely the dipoles are to be incapable of keeping up with the reorientation. At these higher frequencies the orientation of the permanent dipoles no longer contributes to the dielectric constant. In the case of water this does not occur until 17 GHz.

Dispersion in conductivity

Some dielectrics are found to display conduction which arises from actual charge transport. An example of this is ionic conductivity in electrolyte solutions. This conduction would usually be described by a volume conductivity (S/m). The influence this has is to contribute to the dielectric dispersion. The complex dielectric constant is thus as follows:

$$\epsilon^* = \epsilon' - j\epsilon''$$

ϵ^* is the complex representation of the dielectric constant.

ϵ' is the real part of the dielectric constant (Static value)

$j\epsilon''$ is the imaginary part of the dielectric constant (Dispersion)

The dielectric behaviour of the soil solution.

The dielectric constant of bulk water at 20 °C is 80.37. The soil solution however is not bulk water it is a weak electrolyte subject to forces of attraction by mineral surfaces. The presence of ions in water has a tendency to reduce the rotational mobility of the water molecules and thus reduce the dielectric constant. As the concentration increases so the ionic conductivity also increases adding to dielectric dispersion. Reduction of rotational ability appears to be true of molecules close to mineral surfaces, this is discussed more fully under the next section on clay minerals.

Clay minerals

Clay minerals are an important constituent of many mineral soils. The influence they may have on the dielectric behaviour of a moist soil is therefore of great importance. The low frequency (1kHz - 3 MHz) dielectric constant of clay minerals was examined by Fical (1959).

Table. 1

The influence of frequency on the dielectric constant of clay minerals

| Clay mineral | Frèquency (kHz) | Temperature (°C) | Dielectric constant |
|-----------------|--------------------|-----------------------|---------------------|
| Kaolinite | 45.58 | 75 | 6.45 |
| | 2,879 | 75 | 5.24 |
| Halloysite | 35.01 | 75 | 13.91 |
| | 2,361.5 | 75 | 9.42 |
| Montmorillonite | 469.8 | 75 | 15.3 |
| | 2,246.3 | 75 | 10.41 |
| Illite | 63.22 | 75 | 13.91 |
| | 2,413.5 | 75 | 8.44 |

(Altered from Ficai, 1959)

The clay minerals were observed to have dielectric constants related to the presence of bound water. Dielectric constants were observed to reduce both on drying and with increasing frequency.

The behaviour of water in contact with clay surfaces has been the focus of a study by Hoekstra & Doyle, (1970). The general conclusions of this work were that surface bound water behaves in a very structured manner almost like ice and hence has a low dielectric constant (4) at the higher radio frequencies.

Ionic conduction is known to be an important contributor to dielectric dispersion. Ionic conduction is also known to occur in clay minerals. The amount of conduction is related to the surface density of charge and the surface area. These parameters are listed in Table. 2 for a variety of clay minerals.

Table. 2

The surface area, CEC and surface charge density of clay minerals

| | CEC (mol _c kg ⁻¹) | Surface charge density (μmol _c kg ⁻¹) | Surface area (m ² kg ⁻¹) |
|----------------------------------|---|--|--|
| 1:1 Kaolinite | 0.02-0.06 | 1-6 | (1-2)×10 ⁴ |
| 2:1 Smectite | 1 | 1 | 8×10 ⁵ |
| Illite | 0.3 | 3 | 1×10 ⁵ |
| Vermiculite | 1.4 | 2 | 8×10 ⁵ |
| Fe and Al oxides | 0.005 | 0.2 | 3×10 ⁴ |
| Amorphous to X-rays Allophane | 0.8 | 1.5 | (5-7)×10 ⁵ |

The kaolinite has a low surface area and low exchange capacity and would be expected to be associated with a low ionic conductivity. The interlayers of the 2:1 minerals provide space into which cations can be adsorbed. In illite clays the interlayer contains potassium which fixes the structure rigidly. The vermiculite has a partially expanding lattice, whereas the smectite has a fully expanding lattice. The high surface area of the smectite, its capacity to swell and its ability to adsorb ions means it should create a reasonably electrically conductive material. The consequences of this are likely to be that in heavy textured soils ionic conduction is likely to be high adding significantly to dielectric dispersion.

Other minerals

The dielectric constants of a variety of minerals are given in Table. 3. These were compiled from Carmichael, (1982).

Table. 3

| MINERAL | SOURCE & ORIENTATION | DIELECTRIC CONSTANT RADIO FREQUENCIES |
|---------------------------|----------------------|--|
| <i>Silicate Minerals</i> | | |
| Quartz | Across optic axis | 4.96 |
| | Normal to optic axis | 4.11 |
| Plagioclase feldspar | Along a axis | 5.58 - 7.15 |
| Orthoclase feldspar | | 5.5 |
| Biotite mica | | 6.19 - 9.3 |
| Muscovite mica | | 6.19 - 8 |
| Zircon | | 8.5 - 12 |
| <i>Phosphate Minerals</i> | | |
| Apatite | | 7.4 - 10.47 |
| <i>Carbonate Minerals</i> | | |
| Calcite | | 7.8 - 8.5 |
| Dolomite | | 6.8 - 8 |
| <i>Halide Minerals</i> | | |
| Halite | | 5.7 - 6.2 |
| <i>Oxide Minerals</i> | | |
| Hematite | | 25 |
| Rutile | | 31 - 170 |
| Anatase | | 425 |
| <i>Sulphate Minerals</i> | | |
| Anhydrite | | 5.7 - 6.3 |
| Gypsum | | 5 - 11.5 |

(Altered from Carmichael, 1982)

The table indicates that many of these minerals which may appear in soils do not have dielectric constants below 4 as assumed. The exact frequencies at which these measurements were taken is not given such that they may be high or low radio frequencies. If they were

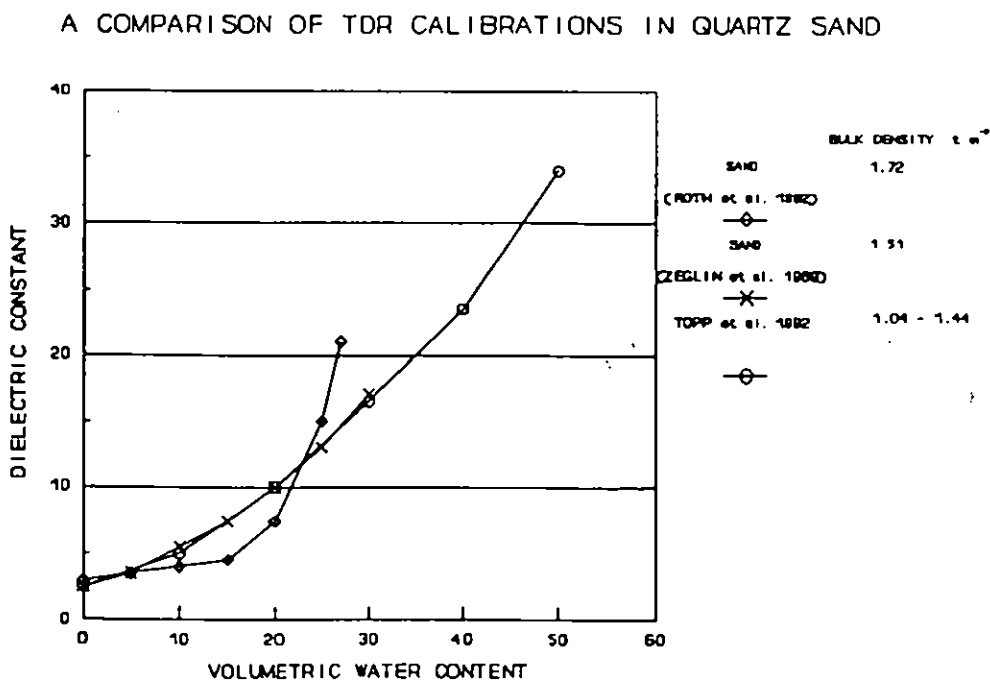
taken at low frequencies there is scope for further dielectric loss which would reduce the dielectric constant.

The most significant group in this table are the oxides which would appear to have very high dielectric constants. These kinds of minerals are most likely to be found in tropical soils and would suggest scientific investigation is required into the feasibility of applying TDR principals to tropical soils.

TDR LABORATORY CALIBRATIONS

Quartz sand

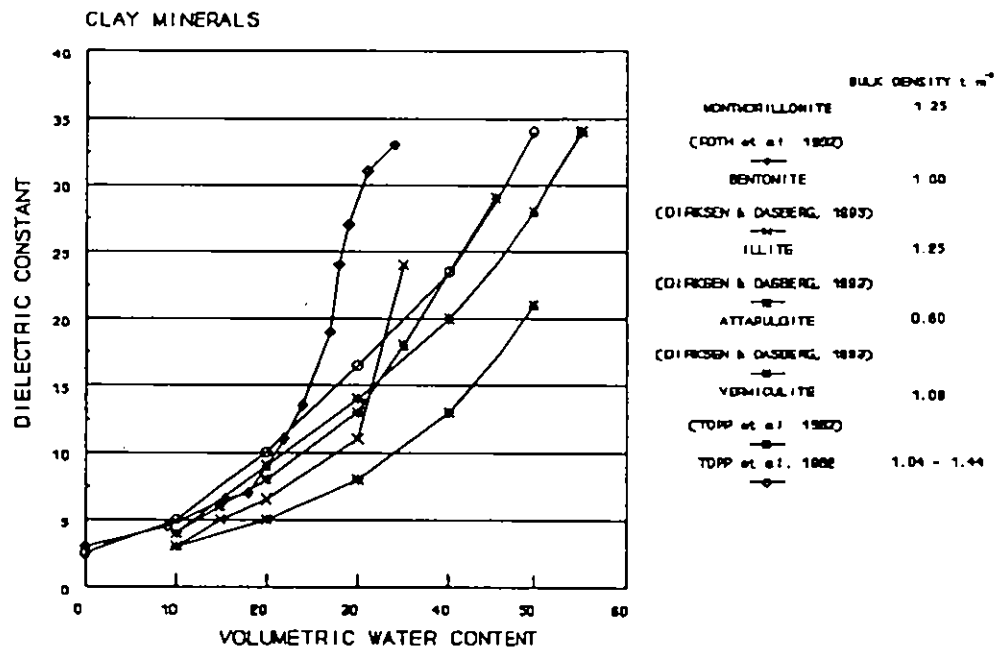
Two TDR calibrations using quartz sand are compared to the Topp *et al.* (1980) calibration in Fig.4. The Topp *et al.* (1980) calibration corresponds well with the calibration of Zeglin *et al.* (1989) in quartz sand. The calibration of Roth *et al.* (1992) shows a distinct difference from the other two calibrations. The difference in bulk density may be a contributory factor, but more likely is the difference in instrument used. Both the Topp *et al.* (1980) and the Zeglin *et al.* (1989) calibrations were carried out with the same TDR device. The work of Roth *et al.* (1992) was carried out using a TDR miniprobe which may account for the differences in calibration.



Clay minerals

A selection of published calibrations in clay minerals are shown in Fig.5. It is instantly apparent that there is a significant difference between the calibrations in the different clay minerals. The differences may be due to the use of different equipment. The difference in bulk density appears to be a significant factor with a drop in measured dielectric constant as the bulk density decreases for the same water content.

Fig.5.



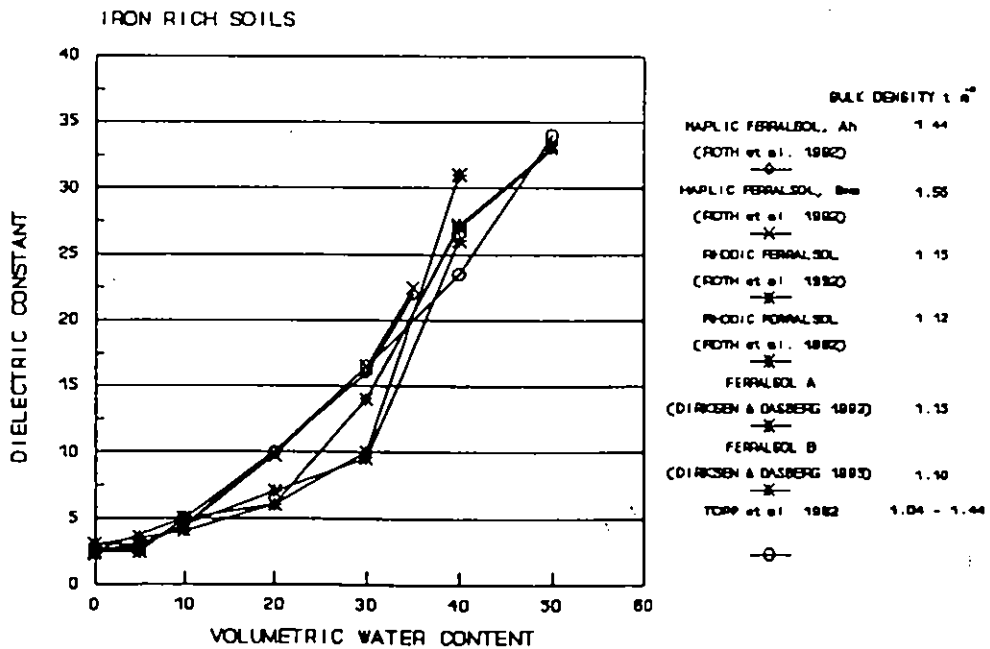
The montmorillonite/bentonite appear to have a very different calibration, this is due to the way in which water is held within the crystal lattice. This certainly requires further investigation.

Iron rich soils

Calibrations for iron rich soils are given in Fig.6. The calibrations are again quite different in comparison with Topp *et al.* (1980). The literature suggests that oxide minerals

of iron and aluminium which may be present in these soils may have high dielectric constants. This does not appear to be the case for these soils as the measured dielectric constant when dry was approximately 3 to 4 similar to both the sand, clay and Topp *et al.* (1980) calibrations. The calibration differences observed tend to occur with increasing water content. They could be due to a hysteresis effect caused by the experimental design which uses a suction device to empty pores to give each volumetric water content. The problem with using such devices is that the spatial distribution of water within the column is unknown. It also means that a potential gradient exists down the column again altering the spatial distribution of water throughout the column. The presence of minerals with a high dielectric constant does not appear to be an influential factor in the case of the calibrations shown.

Fig.6



Time Domain Reflectometer

The TDR system used for the experimentation was the Trase system I manufactured by Soilmoisture Equipment Corps, USA. and described by anon, (1990).

Instrument performance

The performance of the instrument was monitored over an experimental period of 8 weeks. This was achieved by checking the stability of the readings in air and deionised water. Readings were also sampled before work was undertaken each day. This was carried out at the constant temperature of 24°C. The performance achieved maintained a high level of precision throughout the experimental period.

Trials in fine silica sand

The experimentation was carried out using oven dry fine silica sand. The sand was oven dried at 105°C for 24 hours. The principal of the experimentation was to use the sand as a matrix adding to it the ground minerals by mass. Two litres of sand were used with a bulk density of 1.7g cm³. The sand used was settled by agitation of the container this was then weighed so that the exact mass of sand was known. The mineral was added and mixed by hand until a uniform colour was achieved. 2.65 g cm³ was used for the mass of sand, hence in 2 litres 53 g was taken to represent 1%.

The container used for the experimentation was constructed from PVC pipe. The dimensions were, diameter 10.2 cm and height 30 cm. The column was filled to 24.5 cm to create a volume of 2 litres. The column was used like a weighing lysimeter. It had a perforated base so that the column could be wetted from the bottom by placing it in a bucket of deionised water avoiding the effects of air entrapment.

TDR measurements

The sand was transferred into the TDR measuring container. The 20 cm wave guide was then inserted into the sand and readings were taken. After five readings the column was emptied, remixed and repacked with the same mixture and then five more readings were taken. Waveforms for all the mixtures tested were stored and saved. The column had a water content of 37.8% by volume of water added, the TDR readings reflected this with measurements of 37.7%. The dry column gave a volumetric water content of 1.1 by the TDR even though the sand had been oven dried this is because of the calibration used for Ka against water content.

Mineral samples tested

The mineral samples used were a mixture of mineral ore samples. The rutile and zircon minerals were prepared ground samples <0.05 mm and 95% assay and were provided as samples by mineral companies. The ilmenite, magnetite, hematite and goethite were all rock mineral samples which were crushed and ground to the same size as the rutile and zircon and then analyzed semi quantitatively by X-ray diffraction analysis. The results are given in Table 4.

TABLE.4

| | MAGNETITE | HEMATITE | GOETHITE |
|---------------|-----------|----------|----------|
| MAGNETITE | 62% | | |
| HEMATITE | | 71% | |
| GOETHITE | | | 99% |
| QUARTZ | 20% | 29% | 1% |
| STILPNOMELANE | 18% | | |
| ILMENITE | > 95% | | |
| RUTILE | > 95% | | |
| ZIRCON | > 95% | | |

Influence of Iron minerals on TDR

This section examines the influence of Iron minerals on TDR determination of dielectric constant. A brief review of iron minerals in soils is followed by the results obtained in experimental work. It examines the systems ability to assess dielectric constant K_a , and then examines the relationship between K_a and volumetric water content.

Occurrence of Iron in soils

Iron minerals in a variety of different forms occur in a large proportion of soils world wide. The more well known Iron minerals in soil are, goethite, hematite, ferrihydrite, lepidocrocite, magnetite and maghemite. The most abundant of which are goethite and hematite. Their presence is often strongly visible through the yellow through brown to red colours they impart on soils. They may occur in large quantities in Oxisols, Ultisols, Alfisols and Inceptisols. Their large presence in Oxisols and Ultisols has led to the creation of ferritic and oxidic families, (Soil Taxonomy, Soil Survey Staff, 1975). To enter the ferritic category there must be over 40% iron oxide in the soil. These soils are most abundant in the Tropics and Sub Tropics through Africa, Asia and the Americas.

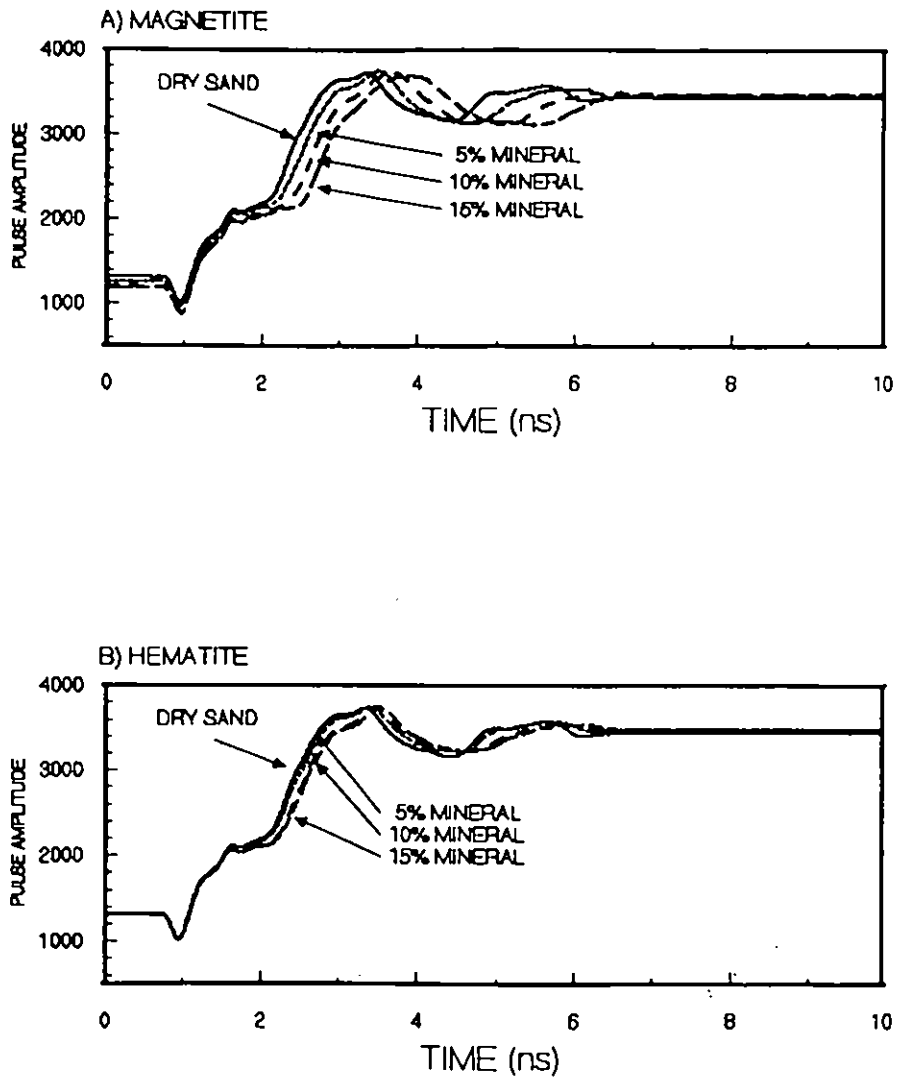
Magnetite and maghemite are both magnetic iron minerals. Magnetite is considered lithogenic derived from parent materials of an igneous or metamorphic nature, where as maghemite is a secondary mineral formed pedogenically in soils. Their presence in soils is as accessory minerals and they sometimes make up a very significant proportion of the heavy fraction especially on those soils derived on basic parent materials. The main occurrence is in the highly weathered Oxisols and Ultisols, and the immature Inceptisols formed on iron rich parent material.

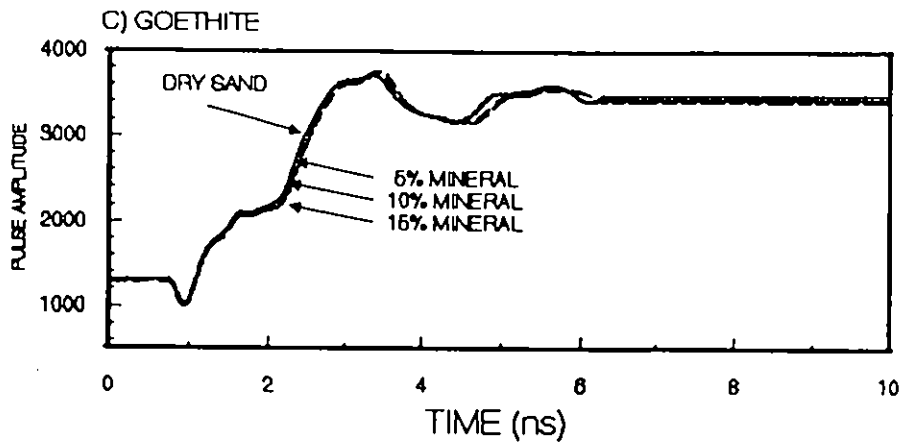
TDR dry sand and mineral mixtures

The waveforms produced by the TDR are shown in Fig.8, magnetite Fig.8a had the greatest influence as it increased in proportion. This is evidenced by the increased travel time of the reflection as the magnetite proportion increased. Manual interpretation the travel time increased from 1 ns in dry sand to 1.5 ns with a 15% proportion of magnetite mixed in. This corresponded to an increase in dielectric constant of 2.5. The hematite showed a similar

response in that the waveform moved to the right with the increased proportion of mineral, but to a far lesser extent than that for magnetite. The goethite Fig.8c had no effect on increasing the travel time of the pulse.

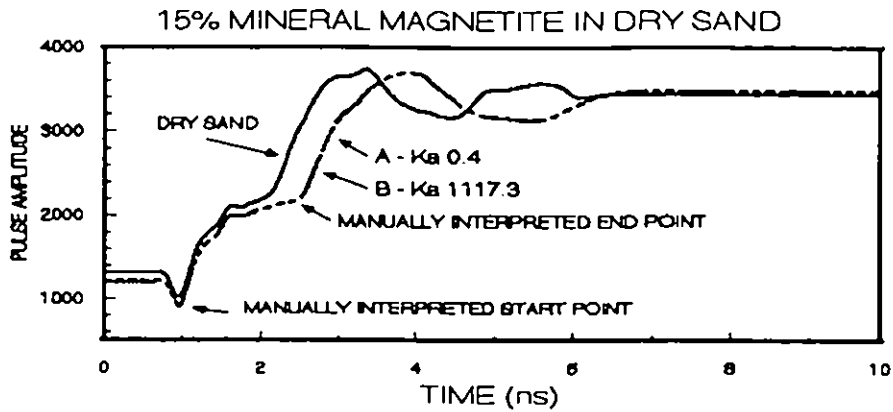
Fig.8





The readings of dielectric constant generated by the Trase sometimes differed from the manually interpreted waveforms. This was most apparent for the 15% magnetite mixture. A wide range of dielectric constants were given by the TDR ranging between 0 and 1117. The waveforms of these readings are shown in Fig.9. The reason for this diverse set of readings comes down to the way the Trase software interprets the waveform. As shown in Fig.9 the waveforms are nearly identical as they were for all the readings. This is good because it means that the precision of the system is good. The problem arises with the software's calculation of the K_a . The software finds it difficult to locate the point where the signal was reflected off the end of the waveguides. This means that each time it may choose a different end point. This fault lies in the software and not the TDR system, the precision of the software is low. It means that it is vital that one monitors the waveforms produced by the TDR to ensure that the correct interpretation is given by the software which in 95% of usage should be correct.

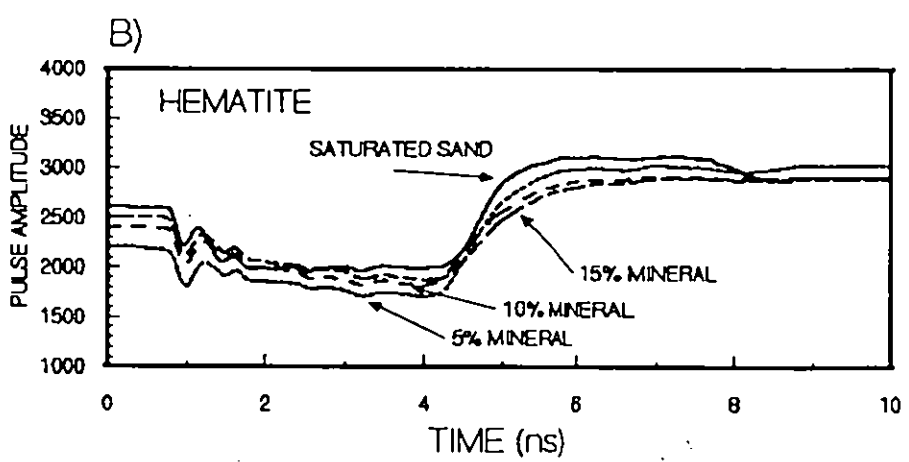
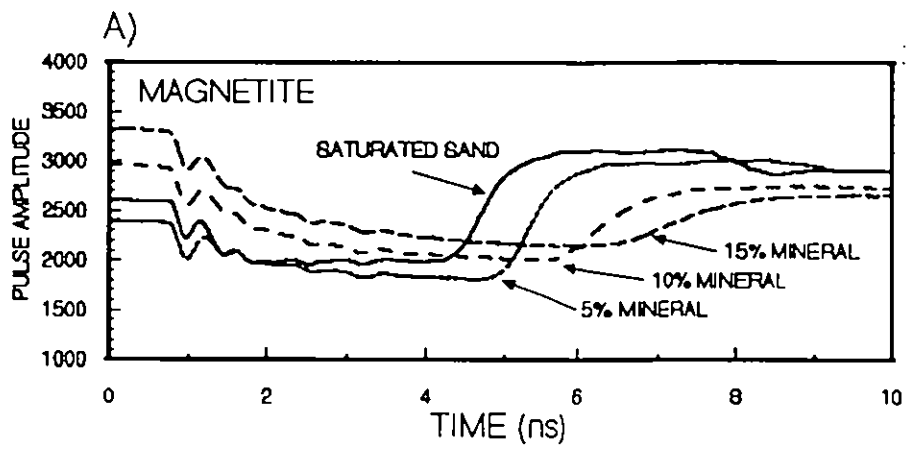
Fig.9

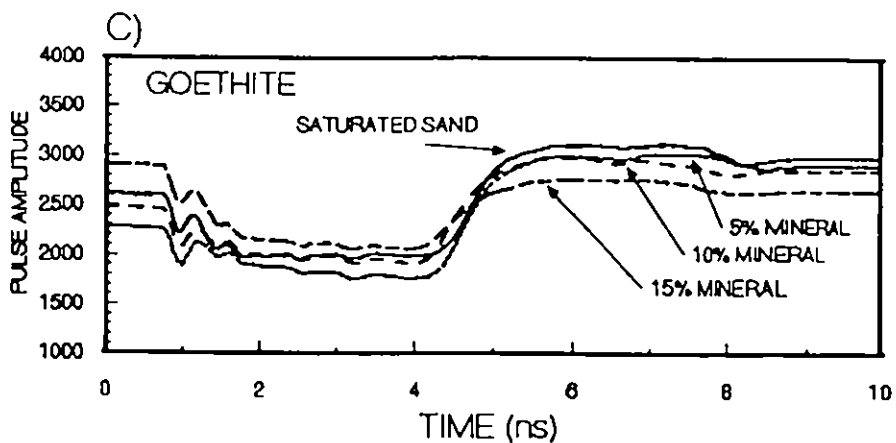


TDR saturated sand and mineral mixtures

The TDR waveforms produced from the saturated mineral mixtures are shown in Fig.10. Fig.10a shows the influence of magnetite. In theory the waveforms should have reduced in travel time and moved to the left indicating the reduced volume of water due to the reduced porosity. It is immediately apparent that this was not the case in the presence of magnetite. The waveforms very substantially increase in travel time and the magnitude of the reflected signal dramatically decreases. The influence of 15% magnetite increases the K_a to 70 from saturated sand which was about 23. In comparison the hematite and goethite did not have such a marked effect. The magnitude of the reflected signal is decreased, most substantially for the goethite. This does not however dramatically increase the travel time of the waves and hence the dielectric constant measured. The end point produced by the hematite mix stays in the same place, it would appear that the hematite compensates for the reduced water content in keeping the K_a about the same at 23 rather than allowing it to reduce as one would expect when the water content is reduced. The sand goethite mix follows the reduction in water content with reduced travel time of the wave as the proportion of the goethite increases.

Fig.10





Possible mechanisms influencing the techniques

The following provides discussion and speculation as to possible reasons as to why the instruments behave in a particular way. The greatest influence of the iron minerals on both of the techniques occurs when the mixtures are saturated. This would suggest that the presence of the water somehow exaggerates the influence of the minerals. The TDR is sensitive to changes in electrical conductivity and this has been a proposed use for simultaneous readings of water content and electrical conductivity Topp *et al.* (1988). The TDR waveform is attenuated by increasing electrical conductivity of the medium, i.e. the magnitude of the reflected signal is reduced. Attenuation loss is visible for the magnetite mixture waveforms and the goethite. Speculating it would appear that the addition of water allows the flow of electrons reducing the resistance and increasing the electrical conductivity of the system.

The electrical conductivity of the medium reduces the magnitude of the reflected signal from the TDR however it is considered opinion that the travel time of the pulse remains unaffected. The increase in the travel times of the waves in the magnetite mix and to a certain extent the hematite mix may be attributable to another mechanism. Roth *et al.* (1992) examined the relationship between magnetic soils from Brazil and their influence on TDR. They concluded that the magnetic permeability of a soil didn't influence the TDR

readings. The theory is that for soil water content measurements the TDR assumes dielectric loss of the soil to be equal to zero, and the magnetic permeability to be equal to unity, Davies and Chudobiak, (1975). According to Roth *et al.* (1992), " This implies that the soil approximates to a perfect dielectric and has no magnetic properties. The velocity of the pulse propagation is inversely proportional to both the square root of the medium's,(soil) dielectric constant and the square root of its magnetic permeability. Therefore the calibration function of a soil that shows susceptibility to an extraneous magnetic field may differ from the one that is valid for a non-magnetic soil, leading to an over estimation of readings of soil moisture." The workers concluded from their work that magnetic minerals had no influence. The chemical data for the work showed the presence of total Iron in the soil at 18% it didn't show the form it was in, which this work has shown to be most important. The influence of both a magnetic material and one with a higher dielectric constant should be independent of the presence of water and thus any exaggeration shown in the presence of water must be due to some other influence.

The exaggeration exhibited due to the presence of water was very significant. It is most easily explained by suggesting that electrical conductivity is increased with the presence of water and that this not only attenuates the wave but also increases the travel time. This appears the most plausible and simple explanation but disagrees with the literature

Influence on water content readings

The relationship between dielectric constant and water content has been demonstrated by a number of authors (Hoekstra and Delaney, 1975; Topp *et al.* 1980). The large relative difference between the K_a of air (1), soil materials(2-4) and water (80) means that an increase in dielectric constant corresponds to an increase in water content. On the evidence provided by the data one can no longer assume that the dielectric constant of a soil mineral will always lie in the 2-4 range and hence the calibration supplied with the Trase TDR in the presence of certain minerals will over estimate the water content of the soil. The presence of the goethite at 15% is unlikely to alter readings of V.W.C.. The presence of 15% hematite may alter readings by as much as 6%. The presence of 15% magnetite may raise water content readings by 60%. The percentages of mineral quoted are those added by mass so that in reality 15% magnetite is about 9% magnetite. This means that small quantities of magnetite ie 1 or 2% will still have a considerable influence on V.W.C. readings if present in soil.

Hematite, present in large quantities eg in ferric horizons may raise readings of water content by ten or fifteen percent. The presence of maghemite in soil may create a similar influence to magnetite. The influence of other iron minerals such as ferrihydrite or lepidocrocite is yet to be assessed but is probably minimal for the TDR. This means that for some iron rich soils the TDR will require its own in situ calibration, in a very few soils spacial variability of minerals may rule out its use altogether.

Influence of titanium minerals on TDR

This section briefly examines the occurrence of titanium minerals in soils. It then presents the results obtained from experimental work on rutile and ilmenite.

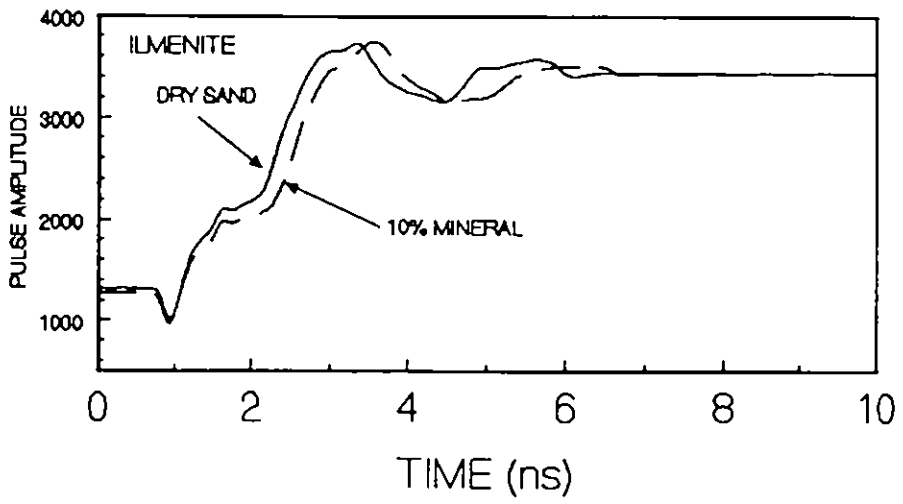
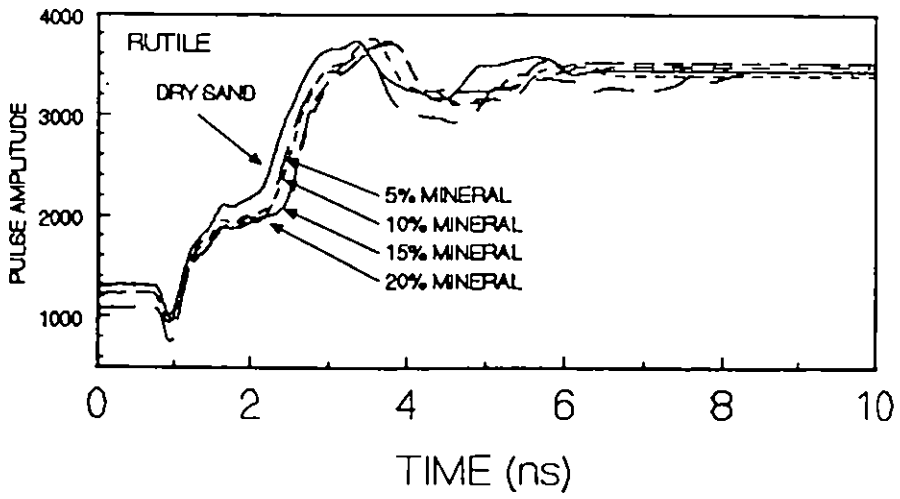
The occurrence of titanium minerals in soils

The occurrence of titanium minerals in soils is generally minor. Estimations of amounts vary the majority of soils worldwide contain < 1% titanium. This is usually in the form of anatase, brookite, rutile, ilmenite or sphene. Though quantities are usually small its significance grows in certain soils, usually those which are formed on titanium rich parent materials. Quantities of up to 25% (TiO₂) have been reported for certain fractions from Hawaiian soils (Sherman, 1952). Significant quantities have been reported in the A horizons of peaty Podzols from Ireland and Scotland (Bain, 1976). The resistance of these minerals to weathering means they will often be present in a variety of soils after the parent material has been through a number of weathering cycles. The study examines rutile and ilmenite as anatase is difficult to obtain in large quantities in its mineral form.

TDR response in dry sand and mineral mixtures

The influence of the titanium minerals on the TDR waveforms is shown in Fig.11a. In theory the pulses should remain in the same position as the dry sand, this is not the case and they move to the right. The response moves the waves to the right increasing the travel time of the pulse. Unlike the response to magnetite the increase in travel time on each occasion is not as clear cut. The waveform created depended to an extent on the orientation of the waveguide. This influence is very important as readings may differ according to the orientation of the mineral fraction with the waveguide. This makes random readings across an area containing rutile more difficult to have confidence in. Ilmenite appears to cause much the same response as the rutile increasing the travel time of the pulse, Fig.11b.

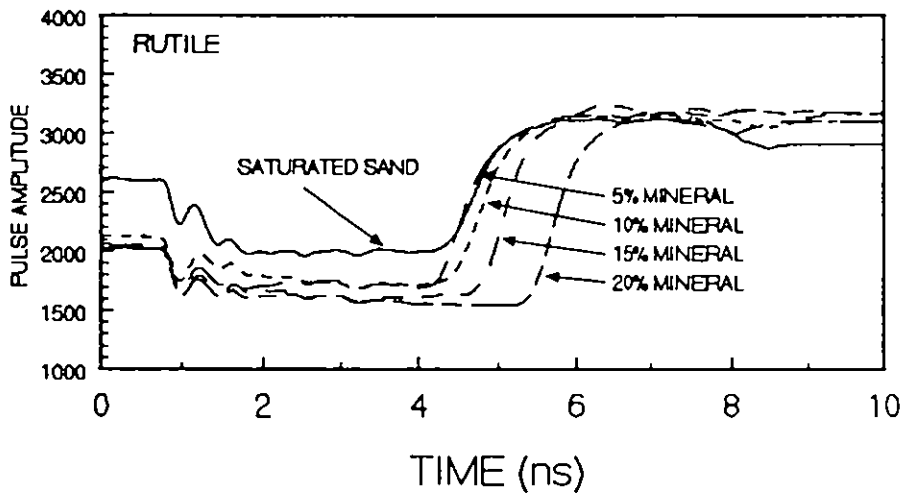
Fig.11



TDR response in saturated sand mineral mixtures

The two minerals examined exerted a substantial effect on the response of the TDR. An examination of the waveforms in Fig.12a and b clearly shows an increase in the travel times of the pulses in the presence of both of the minerals. It is interesting to note the apparent increased strength of reflection in the waves generated in the presence of rutile. The end points tend towards becoming more right angled ie the reflection off the end of the guides is much sharper, Fig.12a. The Ilmenite creates a different waveform with a poor reflection and much greater attenuation of the pulse, Fig.12b. The response due to increasing proportions of mineral was to increase the Ka readings in both cases.

Fig.12a



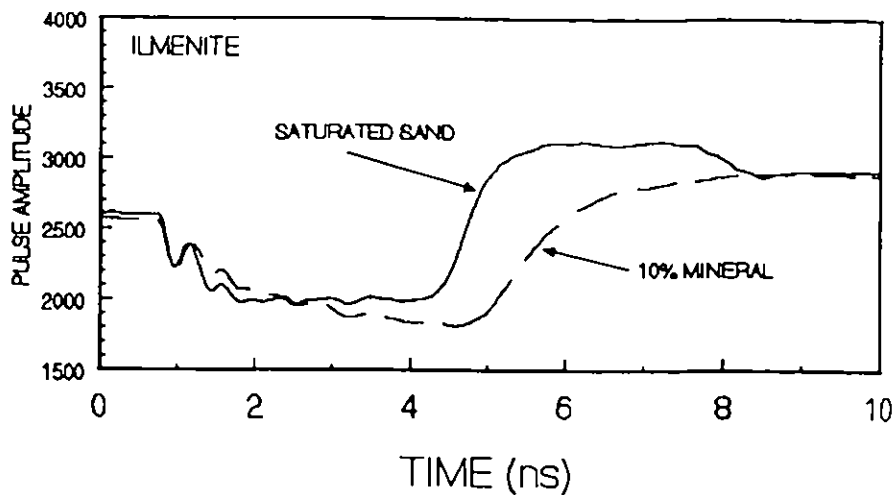


Fig.12b

Possible mechanisms influencing the techniques

The titanium minerals follow a similar response pattern to that seen in the presence of iron minerals. This is that the greatest influence occurs at saturation. The influence of the minerals under saturation is possibly due to an increase in electrical conductivity in the presence of water. The strength of the reflected waves in the presence of rutile would suggest that the electrical conductivity was not increasing. It would therefore appear that the influence of the rutile was something other than this. The influence of rutile and ilmenite creates more questions to be answered about their behaviour with TDR waveforms. An important step would be to examine changes in electrical conductivity with the addition of water.

Influence on water content readings

Increases in K_a due to the presence of titanium minerals will lead to errors in the estimation of water content using a standard calibration. 20 % rutile will mean that an over estimation of 20% water content is made, whereas for ilmenite 10% would result in an over estimation of 30%. The ilmenite can be clearly seen to have a much more significant influence for the minerals studied.

Influence of zircon on TDR

This section examines the influence of zircon on TDR. It gives a brief introduction on the occurrence of zircon in soils, then presents the results of the experimental work obtained from the TDR.

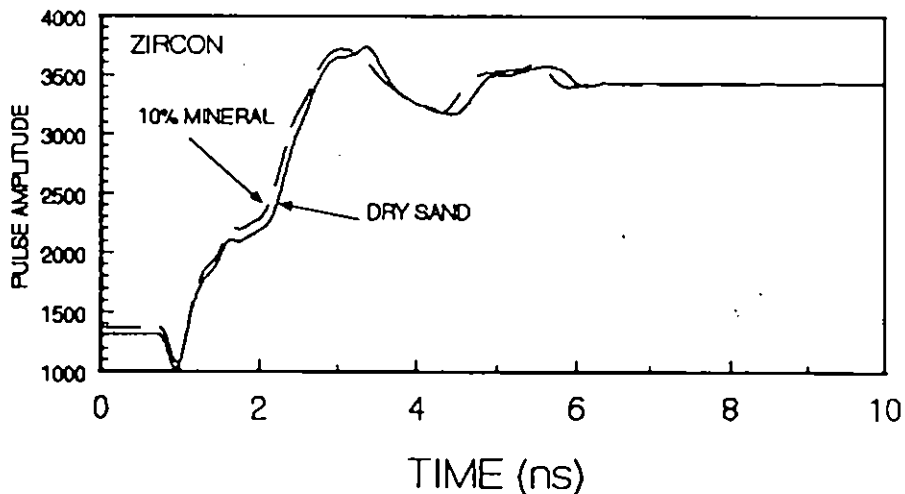
The occurrence of Zircon in soils

Zircon is the most common zirconium mineral in soils, (Allen & Hajek, 1989). It is a lithogenic mineral which is exceptionally tough often surviving many weathering cycles. The majority of soils are said to have at least traces of zircon, it tending to be most abundant in the surface horizons of highly weathered soils.

TDR dry sand and mineral mixtures

The waveforms of dry sand and 10% zircon are shown in Fig.13. It is immediately apparent that the two waves closely correspond giving the same value of K_a . In the dry mixture the zircon has no influence on the waveform.

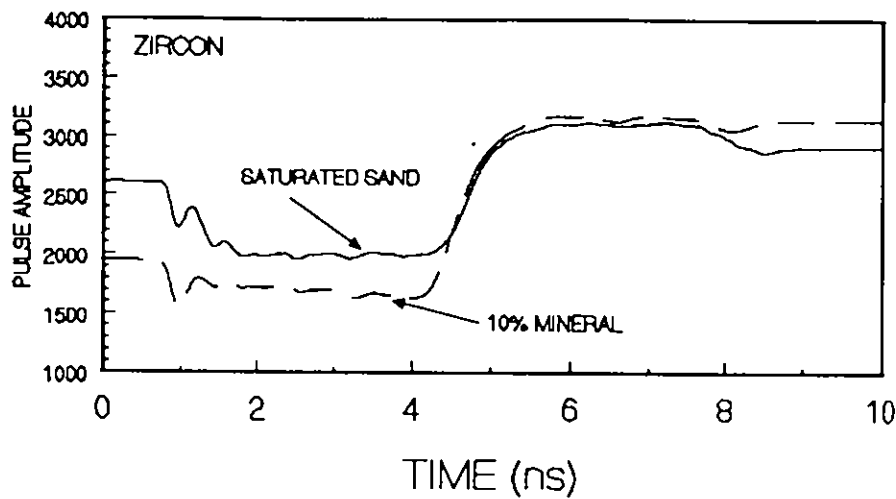
Fig.13



TDR saturated sand and mineral mixtures

The waveforms in Fig.14 behave as considered opinion would predict. The addition of 10% mineral reduced the water content and resulted in the wave moving to the left with the travel time being decreased. It is interesting to note that the reflected wave in the presence of the zircon appears to be much sharper than in the saturated sand suggesting a decrease in electrical conductivity according to the theory of the relationship between pulse attenuation and electrical conductivity.

Fig.14



The influence on water content readings

The zircon creates no apparent effect on the readings of the TDR and hence its presence in soils should not alter the K_a value determined and hence have no effect on water content readings.

7.

CONCLUSIONS

TDR is proving to be a very successful technique for soil moisture estimation. This work has illustrated that to fully realise its potential some further work is required to give understanding into the influence of certain mineral fractions. The major conclusions of the report are listed below with recommended solutions.

Magnetite, hematite, rutile and ilmenite have high dielectric constants thereby requiring differing calibrations to that of a standard such as Topp *et al.* (1980). This work would suggest that for soils where these minerals may be present in large quantities in situ calibration should be undertaken. The spacial distribution of these minerals may render the TDR technique unusable. Electrical conductivity (E_c) within soils appears to be a highly influential factor on TDR. This is already known and is used as a simultaneous measure of E_c of soils. The precise role of minerals such as those described has not been examined and may be important. TDR with subjective waveform analysis will mean that accurate water content determination will not easily be achieved in all soils due to the large variety of influences on the way in which it measures and calculates water content. It would appear that the minerals studied do not have a linear effect on the dielectric constant measured in the presence of water. This may make them potentially difficult to calibrate for.

8.

FUTURE WORK

To extend the range and capabilities of the TDR a few lines of further investigation are suggested.

1. The study of the influence of hematite on TDR. Hematite is a common mineral especially in tropical soils where 30 - 40% may occur.
2. Comparison of different TDR equipment and their calibration as some of the reported differences in the literature may be due to this.
3. Examination of the influence of minerals on electrical conductivity.
4. Investigation as to the influence of a magnetic field.
5. Investigation into the role of clay minerals, the influence of bound water and the effect on electrical conductivity.

- Anon, 1990. Trase System I manual. Soilmoisture Equip. Corp., P.O.Box 30025, CA 93105, USA.
- Allen, B.L. & Hajek B.F. 1989. Mineral occurrence in soil environments. In Minerals in soil environments., 2nd Ed. (Dixon J.B. & Weed S.B.), pp.258-259. SSSA, Inc. Madison, Wisconsin, USA.
- Bain, D.C. 1976. A titanium rich soil clay. *Journal of Soil Science*. 27,68-70
- Bell, J.P., Dean, T.J. and Hodnett, M.G. 1987. Soil moisture measurement by an improved capacitance technique, Part II. Field techniques, evaluation and calibration. *J. Hydrol.* 93: 79-90.
- Carmichael, R.S., 1982. Handbook of physical properties of rocks. CRC Press, Inc., 2000 N.W. 24th street, Boca Raton, Florida.
- Davies, J.L. & Chudobiak, W.J. 1975. *In situ* meter for measuring relative permeativity. Paper 75-1A. Geological Survey of Canada. Energy, Mines and Resources of Canada, Ottawa.
- Dirksen C. & S. Dasberg, 1993. Improved calibration of time domain reflectometry soil water content measurements. *Soil. Sci. Soc. Am. J.* 57, p.660-667.
- Ficai, C., 1959. Caracteristiques des matieres argileuses etablies en mesurant les constantes et les pertes dielectriques dans le domaine de frequence compris entre 30 kHz et 3 MHz, *Bull. Soc. Franc. Ceram.*, 42: 7-16.
- Hastead, J.B., 1973. Aqueous dielectrics, Chapman and Hall, London.
- Hoekstra, P. & Delaney, A. 1974 Dielectric properties of soils at UHF and microwave frequencies. *Journal of Geophysical Research* 79, 1699-1708.
- Hoekstra, P. & Doyle W.T., 1971. Dielectric relaxation of surface adsorbed water. *Journal of colloid interface science*. 36, 513 - 521.
- Jacobsen O.H. & Schjonning P., 1994. A laboratory calibration of time domain reflectometry for soil water measurement including effects of bulk density and texture. *Jour. of Hydrol.* (In print)
- Jacobsen O.H. & Schjonning P., 1994. Field measurements of soil water content during a drying period using time domain reflectometry and gravimetry. *Jour. of Hydrol.* (In print)
- Roth, C.H., M.A. Malicki & R. Plagge, 1992. Empirical evaluation of the relationship between soil dielectric constant and volumetric water content and the basis for calibrating soil moisture measurements by TDR. *Journal of Soil Science* 43, 1-13.

Sherman, G.D. 1952. The TiO_2 content of Hawaiian soils and its significance. *Soil Sci. Soc. Am. Proc.* 16, 15-18.

Soil Survey Staff 1975. *Soil Taxonomy*. Agricultural Handbook 436. Washington DC: Soil Conservation Service.

Topp, G.C., Davies, J.L. & Annan, A.P. 1980. Electromagnetic determination of soil water content: measurements in coaxial transmission lines. *Water Resources Research* 16, 574-582.

Topp, G.C. and Davies, J.L. 1985 Time-domain reflectometry (TDR) and its application to irrigation scheduling. In *Advances in Irrigation Vol.3* (Hillel D.), pp.107-127. Academic Press Inc. 24/28 Oval Road, London.

Topp, G.C., Yanuka, M., Zebchuk, W.D., and Zeglin, S.J. 1988. Determination of electrical conductivity using Time Domain Reflectometry: Soil and water experiments in coaxial lines. *Water Resour. Res.* 24:945-952

Zeglin, S.J., White, I. and Jenkins D.R., 1989. Improved field probes for soil water content and electrical conductivity measurement using Time Domain Reflectometry. *Water Resour. Res.* 25:2367-2376