ON-LINE PROCESS ANALYSIS INNOVATION:
DICOMP™ SHUNTING DIELECTRIC SENSOR TECHNOLOGY

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ABSTRACT

The DiComp Shunting Dielectric Sensor (SDS) is a new patent-pending technology developed under the Small Business Innovation Research Program (SBIR) for NASA's Kennedy Space Center. The incorporation of a shunt electrode into a conventional fringing field dielectric sensor makes the SDS uniquely sensitive to changes in material dielectric properties in the kHz to MHz range which were previously detectable only at GHz measurement frequencies. The initial NASA application of the SDS for Nutrient Delivery Control has demonstrated SDS capabilities for thickness and concentration measurement of Hoagland nutrient solutions. The commercial introduction of DiComp SDS technology for concentration and percent solids measurements in dispersions, emulsions and solutions represents a new technology for process measurements for liquids in a variety of industries.

DIELECTRIC PROPERTIES OF MATERIALS

Material dielectrics are physical properties resulting from interaction between an alternating electric field and the ions and dipoles of a material. In dielectric materials, some of the electric field energy is stored while some energy is expended. Energy storage and consumption in the material are interrelated as a complex function of the electric field frequency (f), and are collectively known as the complex permittivity[1]. Complex permittivity is expressed as:

$$\varepsilon^*(f) = \varepsilon'(f) - j\varepsilon''(f) \qquad [1]$$

The real term of the permittivity, ε', is commonly known as the dielectric constant. The dielectric constant value depends strongly on the degree of polarity within the molecular structure of the material. A polar material, such as water, has high relative permittivity while non-polar materials, such as hydrocarbons, have low permittivity values.

The imaginary term of the permittivity, ε'', is commonly known as the loss factor. The loss factor value depends on the energy expended to align dipoles and move ions within the material, so it can be expressed as:

$$\varepsilon''(f) = \varepsilon_R''(f) + \varepsilon_C''(f) \qquad [2]$$

In this expression, ε_R'' is a measure of dipolar relaxation and ε_C'' is a measure of the ionic conductivity of the material. Both terms are a function of the electric field frequency. In general, at lower (kHz) frequencies the conductivity term dominates while at higher frequencies (GHz, or microwave) the dipolar term dominates.

DIELECTRIC MEASUREMENTS IN MATERIALS

Dielectrometry is the measurement and interpretation of the dielectric properties of materials. There are numerous sensors and instruments available whose measurements rely on one of more term of the material dielectrics. Commercially, these include conductivity meters, RF capacitance probes and microwave permittivity analyzers.

In general, each device has a sensor designed to pass an electric field through the material to be tested. Typically, a transmitting electrode and receiving electrode are used for this purpose. The coupling of the electric field between the two electrodes is dependent on the dielectric properties of the material between the electrodes. This coupling results in changes in the amplitude and phase of the original signal, as shown in Figure 1.
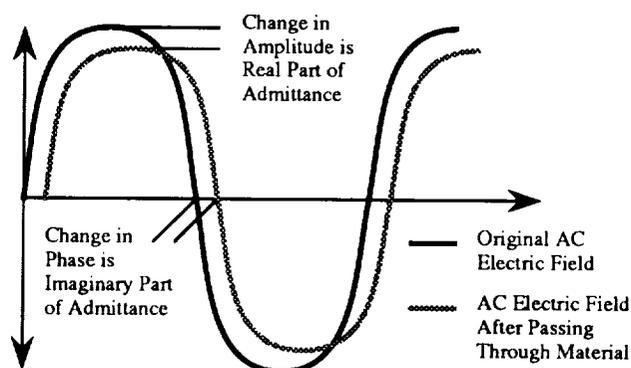
Figure 1: Changes in Measurement Signal Due to Material Under Test

The relative changes in the measurement signal are a unique function of the sensor's electrode configuration and the measurement circuitry. If the electric field can be modeled exactly, it is possible to calculate the material dielectrics by transforming the changes in amplitude and phase.

In the simple case of parallel plate electrodes, a cell constant can be used to characterize the amplitude changes at low frequency and predict the ohmic conductivity of the material. More complex geometries, such as fringing field-type sensors, require the use of more elaborate Fourier transforms to predict material properties from impedance or admittance measurements.

Since the objective of most dielectric measurements is to detect material composition changes, it is not necessary to obtain the absolute value of the material's dielectric properties. It is necessary only that changes in the measurement signals are a function of relative changes in the dielectric properties due to changes in material composition.

LIMITATIONS OF CURRENT DIELECTRIC MEASUREMENTS

Most practical measurement applications involve mixtures of two or more components. The mixture may be relatively stable, as in solutions, emulsions, slurries and dispersions, or may change with time as in a chemical reaction. In each case, the mixture is comprised of components with different contributions to the dielectric properties of the whole.

Solutions, for example, will typically exhibit changes in the ionic conductivity term of the loss factor as the solute concentration changes. Slurries and emulsions will typically exhibit changes in dielectric constant, as their components have lower dielectric constants than the water carrier. Often, a mixture will have both suspended and dissolved components, so both dielectric properties will be affected by changed in composition of the mixture.

If one component has a distinct contribution to the dielectric properties of the mixture at some measurement frequency, then it can be quantified if the instrument system is sufficiently sensitive to the changes in ε' and ε'' at the optimal measurement frequency for a particular component. However, current commercial dielectric measurement systems have limitations in sensitivity.

Conductivity meters are sensitive only to ionic conductivity (ε_C'') and operate a fixed low frequency. RF capacitance probes are limited to measurement in materials with low loss factors. Microwave permittivity systems operate a very high (GHz) frequencies, and therefore cannot detect dielectric relaxations which occur in the low RF (1kHz - 1MHz) region[2]. The ideal dielectric measurement system should have the capability to make measurements sensitive to either ε' or ε'', even at very high loss factors. Such as system should also make measurements at multiple frequencies across the RF spectrum to predict the composition of a wide range of mixtures of materials.

SHUNTING DIELECTRIC MEASUREMENT INNOVATION

Axiomatics has discovered a new dielectric sensor configuration which provides unique sensitivity to changes in both ε' and ε'' across a broad RF spectrum. This innovation is the patent-pending Shunting Dielectric Sensor (SDS).

A conventional fringing field sensor (right) is shown in Figure 2, along with a representation of the coupling of the electric field between the two electrodes.
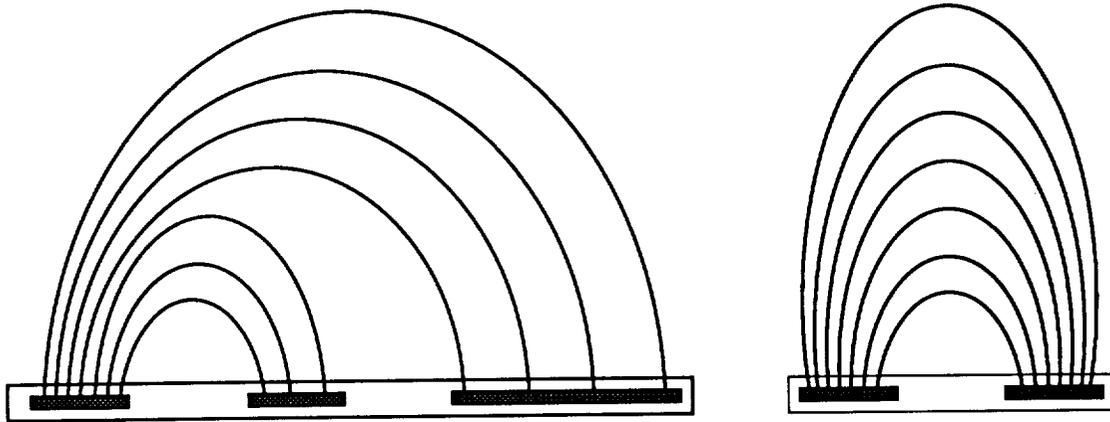
Figure 2: Field Behavior in Shunting (Left) and Conventional Dielectric Sensors

The response of the conventional sensor can be characterized by its RC time constant, which is a function of the geometric configuration of the sensor and the dielectric behavior of any material present in the electric field:

$$RC_C = A * \tau \qquad [3]$$

In this expression, A is a sensor geometry constant and τ is the relaxation time constant of the material in the field. τ is an indication of the time rate at which stationary equilibrium will be reached after the initiation of the electric field. Typically τ is expressed as the ratio of the static permittivity to the steady conductivity. As the conductivity increases, τ and RC_C decrease — this relationship limits capacitance systems to materials with low conductivity.

Axiomatics discovered that the placement of a third electrode in proximity to the conventional electrode pair provides a dominant coupling of the electric field. This shunting of the electric field (left), illustrated in Figure 2, dramatically changes the coupling of the field to the sensing electrode. In effect, the RC time constant for the Shunting Dielectric Sensor (SDS) is multiplied by a term related to the geometry of the shunt:

$$RC_{SHUNT} = B_{SHUNT} * RC_C \qquad [4]$$

As a result, the RC time constant of the SDS sensor is several orders of magnitude higher than the conventional two-electrode sensor. The SDS sensor is therefore significantly more sensitive to changes in τ, regardless of which component of the complex dielectrics is responsible for the change. This sensitivity permits the SDS sensor to be used in measurements of both low and very high conductivity.

More importantly, the SDS sensor has a frequency response which makes it sensitive to non-dispersive dielectric phenomena at much lower frequencies than conventional sensors. Many mixtures, particularly emulsions, have interfacial polarizations which exhibit dielectric relaxations in the MHz spectrum. Complex admittance measurements using the SDS sensor in this frequency range can yield valuable information about the quality and composition of the emulsion.

DICOMP SDS APPLICATIONS

Establishing the response of the DiComp SDS sensor to particular industrial applications has only recently begun. The unique behavior of the SDS makes even the limited published data on conventional dielectric measurements of little value. As with the introduction of any new technology, each application must be approached empirically to establish the measurement response of the SDS sensor for a given material.

In the spirit of the SBIR program, Axiomatics began sales and initial tests with commercial customers in May 1991. The success of this early commercial testing lead to a formal product introduction of our DiComp Analyzer for

liquids at the Instrument Society of America ISA/92[3] exhibition in October 1992. The DiComp SDS system has been successful employed in a variety of applications measuring concentration or percent solids in dispersions, emulsions and solutions. The materials detected include acids, alcohols, caustics, halides, hydrocarbons, monomers and water.

In general, the approach to each new application follows a procedure designed to identify the optimal measurement parameters. The first step in this approach is to use the SDS sensor for a frequency scan of a sample with known composition, over the operating range of the Analyzer of 1kHz to 8MHz. By selectively varying constituents in the known sample, a "fingerprint" is generated which establishes the relative contribution of each constituent as a function of frequency. A combination of frequencies may then be used to predict the constituent parts of the sample using a straightforward function of the complex admittance measurements.

For example, Figures 3 and 4 show the real and imaginary components of the complex admittance measurements for two materials as a function of frequency. Material A, which is representative of a material such as deionized water, has a high τ and therefore exhibits a low frequency peak in its imaginary admittance component with a relatively flat response in the real admittance component. Material B, which has the characteristic response of a material with low τ, exhibits peak response at high frequency in both the real and imaginary admittance components.
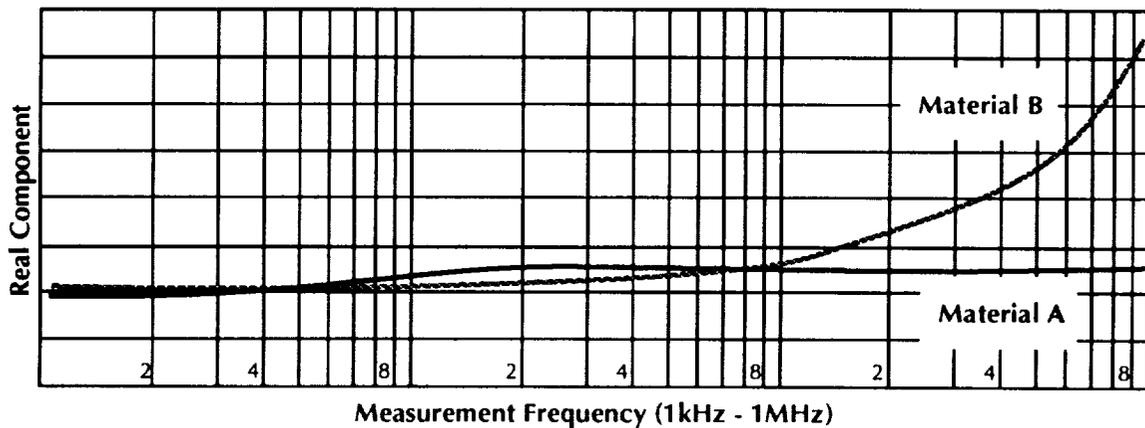

Figure 3: Real Admittance Response for Two Materials as a Function of Frequency
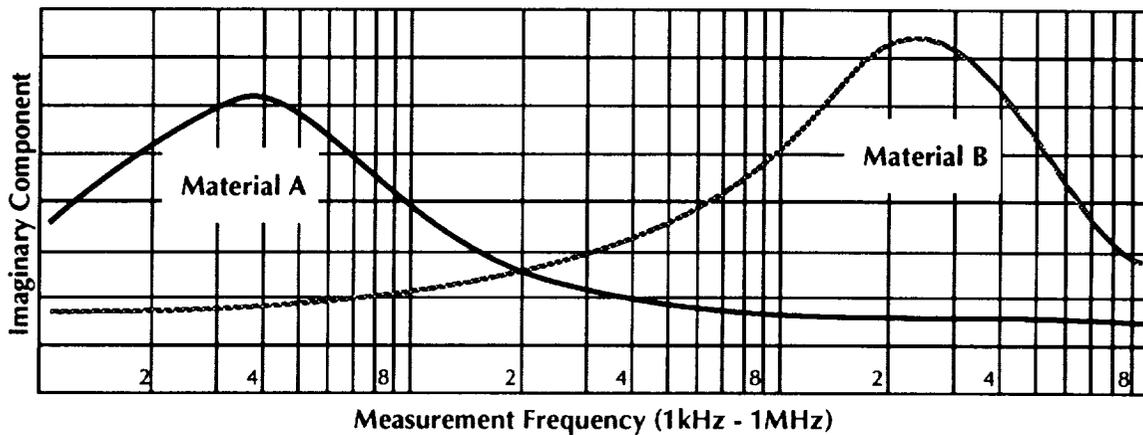

Figure 4: Imaginary Admittance Response for Two Materials as a Function of Frequency

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Mixtures of the two constituent materials will result in a shift of the peaks towards the middle of the range, with corresponding drops in the magnitude of the values. A simple polynomial function of the admittance at one frequency can then be used to predict the relative concentrations — low frequency if Material A is the solvent or high frequency if Material B is the solvent.

The addition of a third constituent material, such as a salt or acid, will change the admittance response further as a result of lowering the τ of the mixture. Although this effect may be evident across the frequency range, it will be more pronounced at higher frequencies. A high frequency measurement can therefore be used to determine the salt content, then compensate the low frequency measurement to predict the concentration of the other variable. This technique can be applied to determining hydrocarbon contamination levels in seawater.

Similar strategies have been applied to mixtures such as slurries and emulsions, where the solvent or carrier is typically water with dissolved solids and the other constituent is typically suspended solids or oils with relatively low permittivity. In each case, predictions for each constituent can be made through a calibration procedure in which representative samples are created in the laboratory. The constituents of the samples are selectively varied, and the admittance measurements at various frequencies are recorded. By comparing the sample admittance measurements against actual constituent values determined by laboratory analytical measurement, a predictive function can be established.

In industrial applications involving continuous production, it may not be possible to create representative process samples in the laboratory. However, a pilot or experimental facility often exists where the SDS sensor can be installed in a pilot process, and the calibration procedure is adapted to rely on simple additions or dilutions performed in a flow loop. By comparing process admittance measurements against known addition and dilution values, a predictive function can be established.

Where no pilot facility exists to permit controlled experiments, it is still possible to perform the calibration procedure using a sensor installed in an actual production process. Although the process is under positive control, there will be variations over time in the constituents and these variations are normally recorded as part of routine laboratory monitoring.

For these cases, Axiomatics has developed software to digitally record admittance data in a form compatible with popular spreadsheet packages. The SDS admittance data is then merged with laboratory measurements of process variation, and a calibration is developed using on-line data. Extrapolation of the calibration function outside of the range of actual measured values can generally be used to predict extremely out-of-specification product.

Axiomatics has developed a number of different sensor geometries to accommodate various pilot and production processes as part of an on-going commercialization program.

NASA NUTRIENT DELIVERY SYSTEM APPLICATION

The initial application of the SDS technology in the Controlled Ecological Life Support System (CELSS) Nutrient Delivery System (NDS) at NASA's Kennedy Space Center required the unique capability to predict both the concentration of a solution and the thickness of the solution layer over the sensor.

The CELSS experiment is designed to test new technologies to reliably provide optimized nutrient solution[4] quality and flow rates to sustain crop growth in a micro- or variable-gravity environment. The NDS control system must therefore include sensors for measuring both thickness and concentration of the nutrient solution delivered to one of several alternative NDS plant growth surfaces being evaluated by NASA, including the continuous-flow NDS in the Biomass Production Chamber[5] (BPC) at the Kennedy Space Center Breadboard facility, the porous tube NDS[6] in the Plant Growth Unit (PGU), and the porous stainless steel NDS[7].

Conventional capacitive sensors cannot be used in this application because the nutrient solution has a very high loss factor. Conductivity sensors can measure bulk concentration, but are unsuitable for measurement of nutrient solution thickness, due to electrode interference with plant growth and variances in conductivity of the solution. Infrared detectors are also inappropriate, due to interference from plant roots and biomass.

The SDS provided an innovative and unique solution to the CELSS problem. The SDS sensor could be embedded in the NDS plant growth surface without interference, and its non-invasive measurements do not affect plant growth.

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Axiomatics designed an SDS sensor for the NDS[8] with a geometry B_{Shunt} designed to provide the necessary sensitivity for both thickness and concentration independent of the choice of plant growth surface. The NDS sensor design is shown in Figure 5.
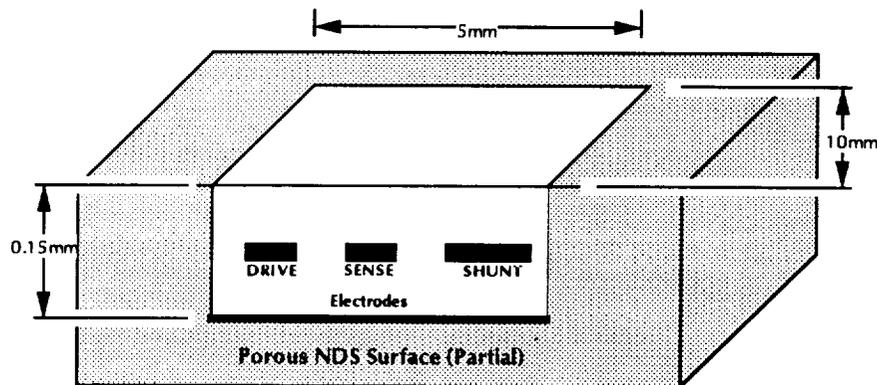

Figure 5: Cutaway View of NDS Sensor

The sensor is constructed of flexible materials to conform to the curved surface of the porous tube growth surface. A wettable surface insures that the thickness of nutrient solution over the sensor is the same as the adjacent area. An architecture which supports multiple NDS sensors is shown in Figure 6. The design employs digital impedance analyzer technology specifically developed to meet the performance and space requirements of the NDS application.
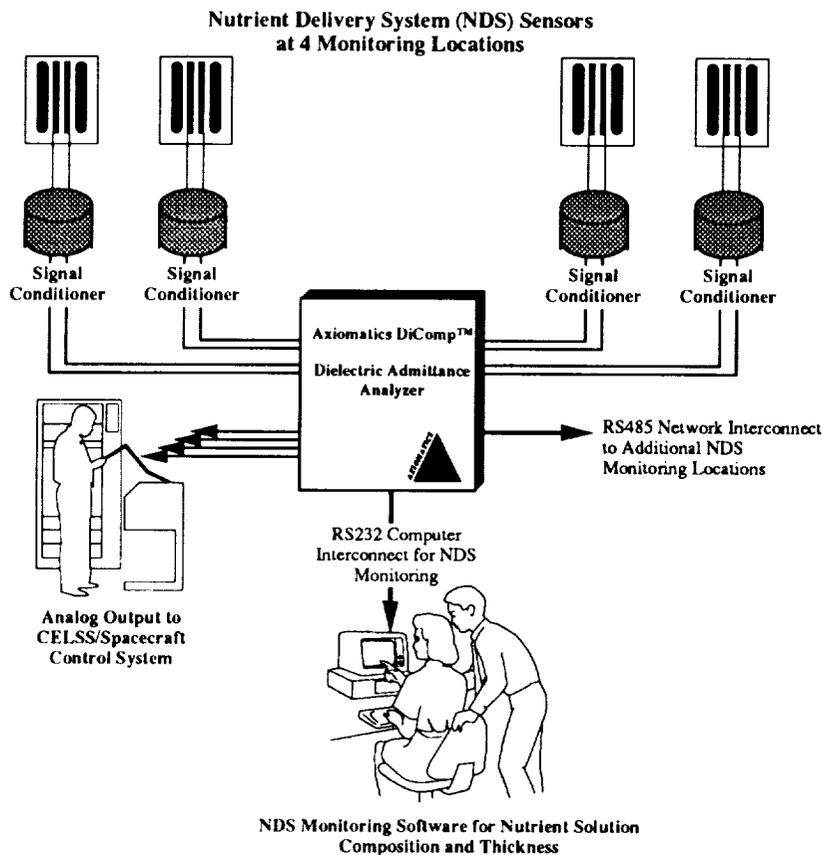

Figure 6: NDS Sensor System Architecture

In the NDS application, the target thickness for the nutrient solution at the plant growth surface is 0.5mm. The NDS sensor has been optimized to provide maximum sensitivity over the range of 0 to 0.5mm, although the sensor has been shown to be capable of accurate measurements to approximately 2.0mm. Within the target thickness range, repeatable accuracy of ±0.05mm (0.002 inches) has been demonstrated, as shown in Figure 7.
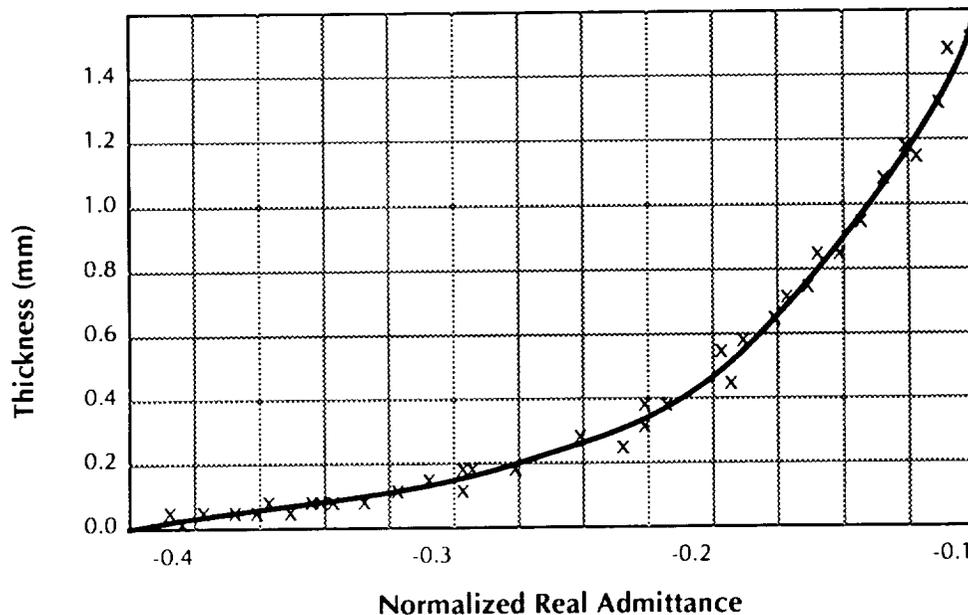

Figure 7: Plot of NDS Sensor Thickness Sensitivity

The development of the NDS sensor system is nearly complete and installation at the CELSS facility at NASA Kennedy Space Center is set for January 1993.

SUMMARY

DiComp SDS technology has numerous industrial applications in single or multi-component liquid streams over a broad range of concentrations from high percent to trace part-per-million. The unique SDS sensor can be flexibly configured to work with nearly any material or process, while the DiComp system retains the stability and low cost of RF frequency operation.

Axiomatics is building on its initial commercial success in measuring concentration or percent solids in dispersions, emulsions and solutions. The broad range of liquid streams which can be used make the DiComp applicable in agricultural productions, chemical processing, foods and beverages, petrochemicals, pharmaceuticals, polymers, pulp and paper and textiles.

REFERENCES

1 W.M. Siebert, Circuits, Signals and Systems, MIT Press, Section 2.5 1989.

2 M Clausse, Dielectric Properties of Emulsions and Related Systems, Encyclopedia of Emulsion Technology.

3 DiComp, ISA/Today, p.21, October 19, 1992.

4 D.R. Hoagland and D.I. Arnon, The Water Culture Method for Growing Plants Without Soil, Circular 347, University of California at Berkeley 1938.

5 R.P. Prince and W.M. Knott, CELSS Breadboard Project at the Kennedy Space Center, Lunar Base Agriculture: Soils for Plant Growth, ASA-CSAA-SSSA, Madison, WI pp. 155-163 (1989).

6   T.W. Dreschel and J.C. Sager, Control of Water and Nutrients Using a Porous Tube: A Method for Growing Plants in Space, *HortScience*, 24(6), pp. 944-947 (Dec 1989).

7   H.V. Koontz, R.P. Prince, and W.L. Berry, A Porous Stainless Steel Membrane System for Extraterrestrial Crop Production, *HortScience*, 25(6), p.770 (June 1990).

8   F.A. Waldman and C.R. Davis, Novel Sensor Technology for Monitoring and Control of Critical Plant Nutrient Parameters, 29th Plenary Meeting of the Committee of Space Research, *Advances in Space Research*, to be published.