Cavity Perturbation Technique for Measurement of Dielectric Properties of Some Agri-food Materials

by

Meda S. Venkatesh

Department of Agricultural & Biosystems Engineering
McGill University, Montreal

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Department of Agricultural and Biosystems Engineering
Macdonald Campus of McGill University
Ste-Anne-de-Bellevue, (QC), H9X3V9, CANADA

ABSTRACT

Meda Venkatesh **M.Sc. (Agr & Biosystems Eng.)**

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Optimal utilization of microwave energy requires more basic knowledge of the dielectric properties of the material under investigation.

The dielectric properties of materials subjected to microwaves are known to depend on moisture content, temperature and density of the material as well as the frequency of the applied microwave field.

This thesis deals with the study of cavity perturbation technique and its feasibility to determine the dielectric properties of various agri-food materials.

In the study, linear relationships between roots of the dielectric properties and density found in the literature were confirmed to be valid for pulverized grains tested with the cavity perturbation method. The effect of particle size on dielectric properties of chopped grain is also reported as a quadratic relationship.

The dielectric properties (ε' & ε'') of materials such as tylose (methyl hydroxy ethyl cellulose); organic solvents such as ethanol, hexane and their mixtures; edible oils such as canola, soya and sunflower oils were determined at various temperatures, frequencies, salt content and other relevant parameters, using cavity perturbation technique.

RÉSUMÉ

L'utilisation optimale de l'énergie micro-onde requiert une connaissance approfondie des propriétés diélectriques des matériaux traités.

Les propriétés diélectriques des matériaux soumis aux micro-ondes dépendent du taux d'humidité, de la température et de la densité de la matière de même que de la fréquence du champ micro-onde.

Cette etude a également examiné la méthode de perturbation de cavités et son utilisation potentielle dans le secteur agro-alimentaire.

Dans cette étude, une modélisation linéaire entre la racine des propriétés diélectriques de grains hachés et la densité a été validé avec la méthode de perturbation des cavités. L'effet de la grosseur des particules sur les propriétés diélectriques a été identifié comme étant quadratique.

Les propriétés diélectriques de matériaux tel le tylose$^{MC.}$; de solvants comme l'éthanol et l'hexane ainsi que leur combinaison; d'huiles comestibles comme l'huile de canola, l'huile de soya ou l'huile de tournesol ont aussi été determinées à différents niveaux de température, fréquence, pourcentage de sels, etc. en utilisant la méthode de perturbation des cavités.

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Symbols

ε_o	=	absolute permittivity of vacuum (8.854 X 10^{-12} F/m)
ε^*	=	complex permittivity
ε_r'	=	relative dielectric constant (= ε' or eps')
ε_r''	=	loss factor (= ε" or eps")
tanδ	=	loss tangent or dissipation factor
ω	=	angular frequency (rad/sec)
σ	=	conductivity
λ	=	wavelength (m)
TM_{mnk}	=	transverse magnetic; directions : x,y,z
m_1, m_2, m_3	=	constants in eqns. 2.3, 2.4, 2.5
e	=	constant dependent on the material in eqn. 2.4
ρ	=	density (t/m^3)
a, b, c	=	constants in eqns 2.6, 2.7
A_o, A_1	=	regression coefficients
r^2	=	coefficient of determination
s.e.	=	standard error of estimate
MHz	=	mega hertz (10^6 Hz or cps)
GHz	=	giga hertz (10^9 Hz or cps)
Q	=	quality factor of the cavity
P	=	power loss
ω_r	=	resonant frequency (f), (rad/sec)
W_{max}	=	energy received by the resonator
C_p	=	bidirectional coupler
M	=	phase shifter
C	=	cavity
A	=	amplifier

A_t	=	attenuator
T	=	delay
dB	=	decibels
v_s	=	sample volume (mm^3)
µl	=	microlitre (10^{-6})
$_o$	=	subcript meaning "unloaded" (empty)

I INTRODUCTION

Microwaves have been of interest in drying/heating of agri-food materials primarily because the radiant transfer of energy to the moisture within the material by-passes the slow conduction stage that limits the efficiency of conventional processing technology. Also, in regions where hydro power is available, fossil fuel combustion can be eliminated from such applications (Raghavan and Alvo, 1993). In recent years interest has grown rapidly in extending the application of microwave energy to the processing of a wide variety of new and engineered materials including ceramics, polymers, composites, pharmaceutical, foods and biological materials.

Microwave energy provides a clean, rapid and efficient heating over a wide range of temperatures (up to 2000°C or more) as well as new degrees of freedom and flexibility over conventional processing methods. Microwave processing is fundamentally different from conventional heating because electromagnetic energy is directly transferred to and absorbed by the material being processed. Consequently, this energy is converted into heat within the material and thus provides energy savings by eliminating the large thermal mass of conventional furnaces. The unique property of microwave energy is to penetrate and produce heat within food materials. This characteristic of the microwaves has better advantages when compared to other conventional heat processing techniques.

Processes generally considered suitable for microwave applications include dehydration, freeze-drying, blanching, baking, thawing, pasteurization, sterilization, curing, and pre-cooking. Progress of microwave processing of food has been reviewed by Decareau (1985), Rosenberg and Bogl (1987a, b) and Giese (1992). Also, studies on use of microwaves for microbial control, enzyme inactivation, pest control, etc have been reported (Raghavan et al, 1995). Systematic studies of the microwave drying kinetics and resulting quality of various grains (wheat: Kudra et al. 1994; corn: Shivhare et al. 1991a, b;

Shivhare et al.1993) have been recently reported. Tulasidas et al. (1995) showed that the specific energy requirements for drying grapes in a microwave field are 1/4 or less of those for drying in a convective regime for the same product quality, and one might expect that they also be substantially lower for drying grain.

As the use of microwave heating increases in the processing of foods, better understanding of the interaction of microwaves with food materials is necessary for successful application. The "permittivities" (dielectric properties) of agri-food materials and their constituents are the key factors in understanding these interactions. Dielectric properties of agricultural products and food materials are dependent on the frequency of applied electromagnetic fields and the moisture content and temperature of the materials (Nelson, 1973a), however the quantitative nature of that dependence is not well established for many complex agri-food materials, such as tylose and reheated edible oils.

1.1 Aspects of microwave interactions with biological material

Dielectric properties refer to the intrinsic electrical properties that affect how materials like foods interact with electromagnetic fields such as microwaves. Foods respond only to the electric portion of the microwave field. Dielectric properties are defined in terms of dielectric constant (ε') and dielectric loss factor (ε''). ε' is a measure of the ability of a material to heat by absorbing microwaves. ε'' actually refers to effective loss factor which also includes direct current (d.c.) conductivity effect. The power dissipated inside a material is proportional to ε'', the effective loss factor. The ratio, ($\varepsilon''/\varepsilon'$), is called the loss tangent or dissipation factor and is a measure of the material's ability to generate heat (Mudget, 1990). Dielectric properties of water and aqueous ionic solutions have been studied extensively by Collie et al. (1947), Grant et al. (1957), Hasted (1973) and Hans-Juergen Blume (1980). Because microwave radiation is part of the electromagnetic spectrum, it behaves very

much like the more familiar visible light radiation we experience daily. Similar to the laws of optics, materials interact with microwaves in three ways. Firstly, they reflect microwave radiation impinging on them. Secondly, they transmit microwaves that have entered into them. Finally, they absorb some of the microwave energy being transmitted through them.

There are different measuring techniques to determine the dielectric properties of a material under test and "cavity perturbation" is one such techniques. In the cavity perturbation technique, when a small sample of dielectric material (eg., ceramics, agri-food) is introduced into a resonant cavity, the frequency of resonance (Δf) is changed by a small quantity as well as the quality factor (Q-factor) of the cavity. These effects on the circuit parameters are usually quantified which results in the measurement of the dielectric properties of the sample. An advantage of the cavity perturbation technique is the simplicity of the measurement set up. It does not require complicated calibrations and tuning since a Network Analyzer can accomplish most of the sensing functions.

Various materials, including alcohols and some organic solvents, also exhibit dielectric properties that make them suitable for heating with microwave and dielectric energy and so, behave similarly to water.

In preliminary studies accomplished, it was observed that particle size might be having an effect on the dielectric properties. The effects of moisture content, temperature, and frequency have been well documented (Nelson et al, 1994), it is feasible and necessary to concentrate on the other unknown data such as effect of particle size and bulk density on dielectric properties of cereal grains (corn, wheat) and the dielectric behaviour of selected agri-food materials, using "Cavity Perturbation Technique".

1.2 Hypothesis

Knowledge of the dielectric properties of materials to be processed by microwave is essential for proper design of microwave applicators used to

predict temperatures during microwave heating. In the design of any microwave heating/drying/processing operation, it is important to know the functional relationships between the dielectric properties of the material and its moisture and temperature since they change during the process and will affect the process efficiency substantially if not taken into account. Most of the work on microwave drying has been concentrated on whole kernels for storage drying, and the relationships between dielectric properties have been established for many of the species studies. One of the questions is : what factors would be influencing the microwave drying kinetics and dielectric properties of kernels that are crushed or powdered prior to heat processing applications such as microwave food processing, cereal drying, etc.

Data on dielectric properties for a number of agricultural and food products are available in a recent bibliography (Kent 1987). However, the data on many of the complex agri-foods and aromatic compounds are not found in the literature. There is a growing need to study & investigate different measuring techniques for determination of dielectric properties of complex, selected agri-food materials as MW energy and application is increasing and energy efficient. Therefore, a study was carried out to determine the dielectric properties of selected agri-food materials, using the cavity perturbation technique.

1.3 Objectives

The objectives of this study were :

- to determine the effects of particle size and bulk density on the dielectric properties of corn and wheat grains.
- to study the dielectric behaviour of selected agri-food materials like tylose and edible oils at different processing frequencies and temperatures.
- to determine the dielectric properties of organic solvents such as ethanol, hexane and their mixtures.

II REVIEW OF LITERATURE

2.1 Introduction to Microwaves

2.1.1 Description

Microwaves are electromagnetic waves of very short wavelength and are composed of electrical and magnetic energy. They extend over the wavelength range of 0.1 mm to 1 m (300 MHz to 3000 GHz frequency). Microwaves lying in the extremely short wavelength region are called submillimeter waves. Microwaves include UHF (ultra high frequency), SHF (super high frequency), and EHF (extreme high frequency) waves. Microwaves (MW) are coherent and polarized electromagnetic (EM) waves. In the electromagnetic spectrum, the microwave frequency range lies between the radio and infrared regions. Figures 2.1 and 2.2 describe the EM spectrum and it's propagation along z direction.

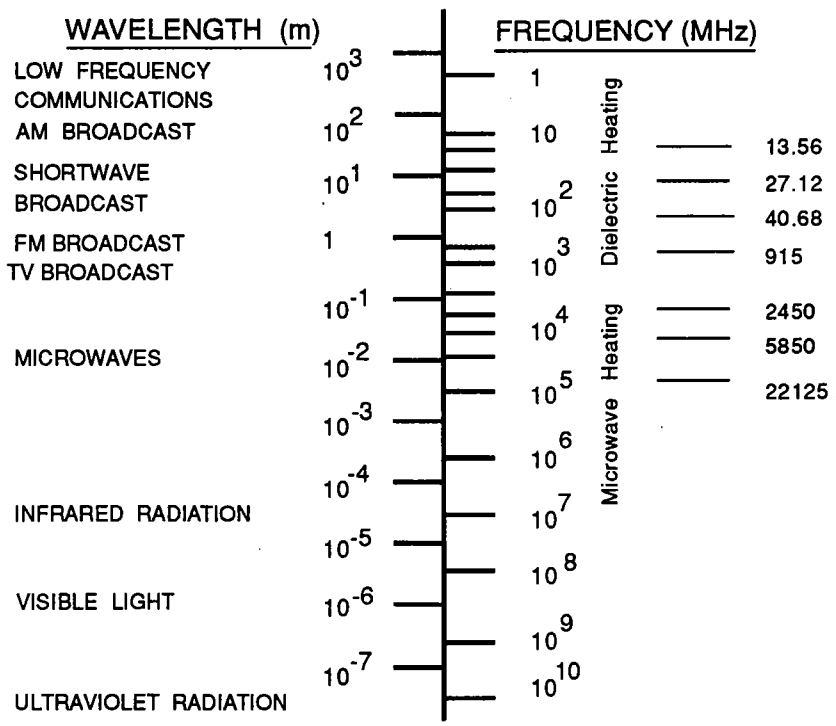

Figure 2.1 The Electromagnetic spectrum

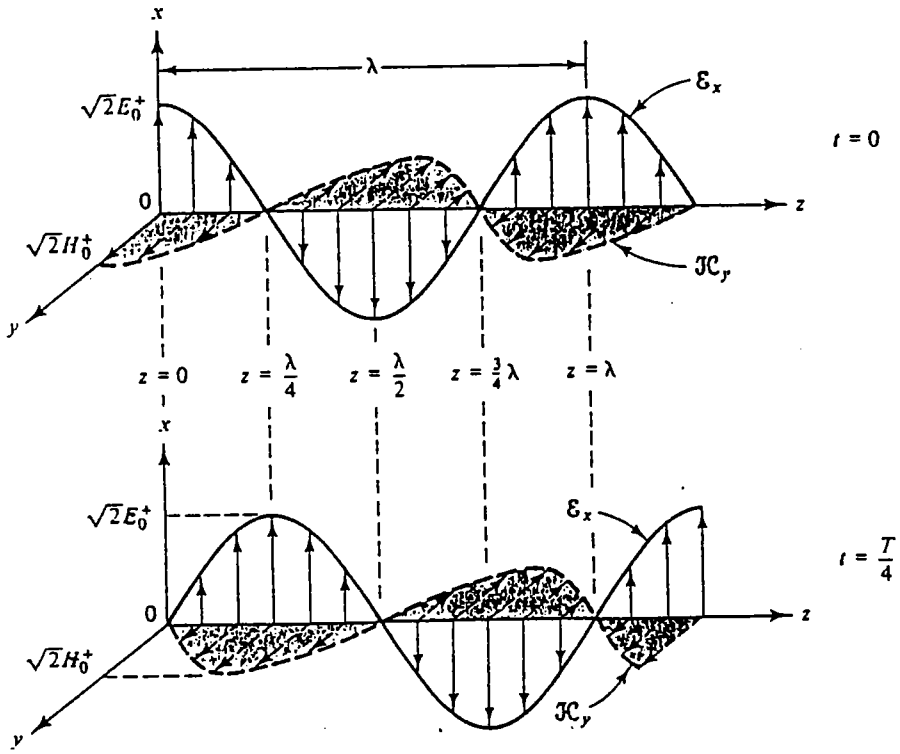

Figure 2.2 Description of an electromagnetic wave travelling in the positive z direction (shown at t=0 and t=T/4).

2.1.2 Permittivity

The permittivity of a material is an important parameter to consider in microwave drying/heating/processing applications because of its relation with process variables such as material moisture content, bulk density and temperature as well as frequency of the alternating electromagnetic field.

It is represented by the equation :

$$\varepsilon^* = \varepsilon_r' - j\,\varepsilon_r'' \qquad (2.1)$$

where ε_r' is the dielectric constant and ε_r'' the dielectric loss factor. The real and the imaginary parts of the complex relative permittivity are also called complex dielectric constants. ε' and ε'' are referred to as the dielectric

properties (suffix "r" refers to the relative parameter).

2.1.3 ε', ε" and tanδ

The dielectric constant (ε') is associated with the ability of a dielectric material to store or couple electromagnetic energy whereas the dielectric loss factor (ε") represents its ability to dissipate energy. The three mathematically linked dielectric properties one has to understand are: ε'- the dielectric constant (relative to air); ε"- the loss factor and tanδ - the dissipation factor or loss tangent. ε' is usually referred to as the "dielectric constant". It indicates how well a material will sustain an electric (microwave) field; in other words, how much of the microwave energy generated in microwave oven is concentrated in it. It also determines how much energy is reflected from the surface of the food and how much is transmitted into it.

2.2 Microwave (MW) - material interaction mechanism

The high frequency microwave field, oscillating at 2450 MHz in microwave ovens, influences the vibrational energy in the water molecule and other dipoles to cause frictional heating. While materials other than water may be dipolar or may behave as dipoles due to the stress of the electric field; water usually dominates probably because it is pervasive at high concentrations in most foods. In fact, one can speculate that if foods did not contain so much water there would not have been microwave ovens, today. When microwaves penetrate and propagate through a material, the internal electric fields generated within the volume of the material induce translational motion to ionic or molecular dipoles. The resistance of these induced motions causes losses, attenuating the electric field and volumetrically heating the material. One of the heating mechanisms is ionic conduction, which is a type of resistance heating that depends on the acceleration of ions through solutions and the resulting multiple billiard-ball-like collisions. Two factors that affect food materials' dielectric properties are its charge due to ionic concentrations

and water. The major interaction mechanism that causes heating is the dipolar rotation, which depends on the influence of the microwave field on dipoles. In foods, the ions of salt-sodium and chlorine have a major affect upon the penetration depth as explained below.

Microwaves interact with foods in three ways: reflected, transmitted or absorbed (Figure 2.3). The dissipation factor, tanδ, indicates how well the microwave field inside the food is converted to heat. The higher the value of tanδ the more microwave energy will be converted to heat. The loss factor describes the lossiness or how lossy a product is ie., how well a material absorbs the microwave fields passing through it and converts it to heat. A lossy food with a high ε", such as ham, will heat very quickly; however, the heating will be near the surface (Schiffmann, 1993).

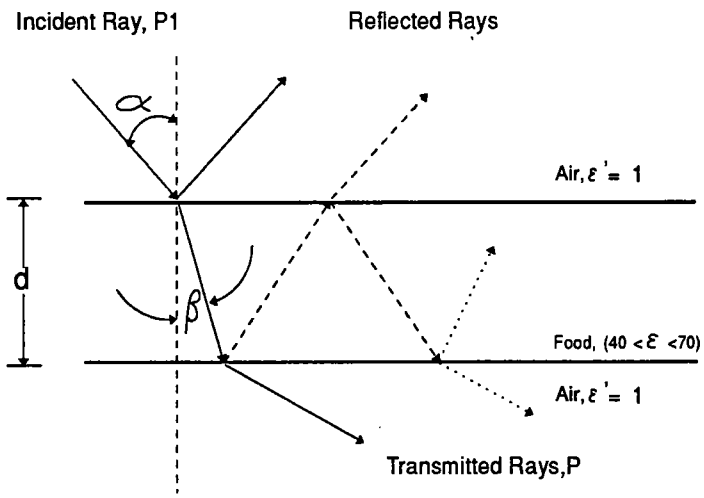

Figure 2.3 Shows MW interaction through a slab of food material.

The materials that interact with electromagnetic fields are divided into four categories : conductors, insulators, dielectrics and magnetic compounds. Those

properties that govern whether a material may be successfully heated by a dielectric or microwave field are the dielectric properties : dielectric constant ε', loss tangent or dissipation factor (tanδ), and the loss factor ε". The dielectric constant of a mixture is usually a type of weighted average of those of its components.

2.3 Importance of dielectric properties in MW food processing

The knowledge of dielectric properties and how these depend on composition, temperature, frequency is a must for a better understanding and analysis of microwave heating of foods and biological materials. During microwave and high frequency heating/processing many variables in the food affect the heating performance. Among the most significant is the permittivity of the food since it describes how a material interacts with microwaves. The characterization of dielectric properties is vital to quantifying microwave/materials interactions. Dielectric materials such as ceramics, polymers, teflon and composites have low thermal conductivities and thus only absorb microwaves to varying degrees depending on the material.

Comprehensive overviews of dielectric properties at microwave frequencies for a wide variety of foods and agricultural (biological) products have also been reported by Bengtsson and Risman (1971), Nelson (1973), Ohlsson and Bengtsson (1975), and Kent (1987). However, most dielectric properties for food materials have been reported for temperatures below 65°C. Ohlsson and Bengtsson (1975) measured dielectric properties of several foods at 450, 900 and 2800 MHz from 40 to 140°C. As microwave pasteurization and sterilization are increasingly applied (Giese, 1992), dielectric properties of food and biological materials at wider temperature ranges are needed as a basis for fundamental studies of such processes. Fundamental studies of microwave food sterilization require dielectric data over the temperature and frequency range of practical interest.

Microwave energy for drying agricultural commodities has been

extensively studied (Otten and St. John, 1988; Shivhare et al. 1992; Raghavan et al, 1992). A concerted effort was made to obtain reliable data on material properties (von Hippel, 1954). The most important factors affecting the dielectric properties of grain as stated by Nelson (1982) are; the moisture content, the temperature and the frequency of the alternating field. Density had also been identified as a major source of variation for ε' and ε'' (Kent, 1977; Nelson, 1983a, b; Nelson, 1984a, b; Kent and Kress-Rogers, 1986 and Nelson and You, 1989). The density dependence of the dielectric properties of materials must be accounted for in elaborating functions determining grain moisture content (Powell et al.; 1988; Meyer and Schilz, 1980). This relation could also be used in the control of continuous on-line processing of the grain. In the case of moisture determination by microwave methods, the frequency used should be above 5 GHz to avoid the influence of ionic conductivity and bound water relaxation (Kraszewski, 1988). For this reason, studies of dielectric properties vs density relationships studies have been conducted at high frequencies. However, the size of microwave components is usually proportional to the wavelength and therefore, inversely proportional to frequency.

Dielectric properties can be conveniently used to measure density as well as the water content of any biological material. The knowledge of these properties were considered very useful in constructing ovens, in selection of packaging materials, cooking utensils, design of proper heating equipment and in developing/formulating foods (Ryyanen, 1985; Ohlsson et al. 1987).

2.3.1 Dielectric properties and significance of moisture (water) content

Water is the major constituent of most moist foods and thus its dielectric properties also determine to a great extent, the dielectric properties of the food. The knowledge of the dielectric properties of water is essential in estimating the dielectric properties of the majority of the foods. Microwave heating is

greatly affected by presence of water in foods (von Hippel, 1954; Mudget, 1990; Nelson, 1990). Water is the major absorber of microwave energy and consequently, higher the moisture content the better the heating of food. The organic constituents of food which are dielectrically inert ($\varepsilon' < 3$ and $\varepsilon'' < 0.1$), when compared to aqueous ionic fluids or water, may be considered transparent to MW (Mudget, 1990). The components of low specific heat will be the major factors in heating only when the residual traces of water are bound and unaffected by the rapidly changing MW field at low moisture levels in foods. In the case of high carbohydrate foods and syrups, the dissolved sugars (in water) are the main MW susceptors (Mudget, 1990). Dielectric properties of aqueous sugar solutions of different concentrations have been measured and compared with those of grapes of similar moisture concentrations (Tulasidas and Raghavan, 1992). The amount of free moisture in a substance greatly affects its dielectric constant since water has a high dielectric constant (~ 78) at room temperature; that of base materials is of the order of 2. Thus with a larger percentage of water the dielectric constant generally increases with increasing moisture content but levels off at values in the range of 20% - 30% and will decrease at still higher moistures. The values of ε' and ε'' of a food material play a critical role in determining the interaction of the microwave electric field with the material. A "map" of foods plotted against their dielectric parameters was introduced by Bengtsson and Risman (1971). The range of dielectric responses of materials and their ability to absorb microwaves is perhaps one of the most widely used features of microwave processing. For example, water is a strong, broad-band absorber of microwaves and therefore is widely used in selective heating for processing food (Craig et al. 1995). The effect of microwave and conventional heating on water mobility (self-diffusion coefficient "D") and dielectric properties was investigated (Umbach et al. 1992). They conclude that, although the self-diffusion coefficient (D) and dielectric properties together measure water behaviour, they examine different aspects of the microstructure of a starch-

gluten-water system.

While alcohols and dissolved carbohydrates are active ingredients in some foods and beverages, their effects are negligible in most food products, except for high carbohydrate foods, such as bakery products or syrups, and alcoholic beverages. The effects are related to stabilization of hydrogen bonding patterns through hydroxyl-water interactions (Buck, 1965; Roebuck et al., 1972).

2.3.2 Effect of temperature, moisture content and applied frequency on dielectric properties.

The temperature dependence of a dielectric constant is quite complex and may increase or decrease with temperature depending upon the material. The dielectric properties of some hydrocolloids such as powdered potato starch, locust bean gum and carrageenin were found to increase regularly with moisture content in the range of 0 to 20 % wet basis (w.b) at 2.45 GHz (Nelson et al. 1991). The dielectric properties of these hydrocolloids were also found to increase with temperature over the range from 20°C to 100°C, and the temperature difference increased markedly as moisture content increased (Prakash et al. 1991). The dielectric properties of the whole (solid) material of the above hydrocolloids were reported to be greater than the corresponding properties of their powdered materials and they exhibited similar relationships with moisture content and temperature (Nelson et al. 1991).

Microwave heating has recently been applied to aid the extraction of natural products from plant materials. The microwave extraction of crude fat, antinutritives and organophosphate pesticides for chromatographic analysis of an essential oil from *Lippia sidoides* Cham and selection of vegetable extracts have been reported (Chen and Spiro, 1994). Studies on factors that determine the rate of microwave heating of a mixture of leaves and solvent and the rates of extraction of such systems carried out isothermally and under microwave conditions have been also been reported (Chen and Spiro, 1994). The

microwave heating characteristics of extraction mixtures consisting of rosemary or peppermint leaves suspended in hexane, ethanol and hexane plus ethanol mixtures were studied. It was found that there was a temperature rise in the mixture, which depends on the dielectric properties of the solvents and the leaves in question (Chen and Spiro, 1994). Both the dielectric constant and loss factor of grapes at 2.45 GHz were found to decrease with decreasing moisture content in the range of 80% -15% (w.b) (Tulasidas et al. 1995). In the case of high moisture in grapes the dielectric constant and loss factor decreased with increasing temperature and a reverse trend was observed in the lower moisture ranges (Tulasidas and Raghavan, 1995).

Temperatures for small samples (5 ml) in a microwave oven cavity was well controlled by a feedback temperature control system which uses shielded thermocouple (Ramaswamy and van de Voort, 1991).

The dielectric measurements of a number of moist food stuffs at 2800 MHz in the temperature range of -20°C to +60°C have been extended to the frequencies of 450 and 900 MHz using the cavity perturbation technique. The results shown are in good agreement with literature data. Also, a good agreement has been established between the data and those using a different measuring technique (Bengtsson et al. 1974). Food dielectric properties are primarily determined by moisture and salt contents (Mudget, 1974; Swami and Mudget, 1981).

ε' and ε" increased significantly with falling frequency for most foods and in most of the cases the dielectric data increased sharply with temperature during thawing operation (Ohlsson et al. 1974). The influence of different water and salt contents were significantly large, especially at 450 and 900 MHz and it was also found that at temperature above 60°C, ε' decreased gradually whereas ε" increased particulary at lower frequencies (Bengtsson et al., 1975).

The cavity perturbation technique has been found reliable and useful for measuring dielectric data of foods over a wide range of frequency, temperature

and composition and the data generated were in good agreement with available literature data (Ohlsson et al. 1974).

2.3.3 Penetration Depth

The penetration depth (d_p) is commonly defined as the depth into a sample where the microwave power has dropped to 36.8% of its initial value. This value is defined as the distance into a sample where the power has dropped to one half or 50 % of its original value. The commonly used equation for determining the value of the penetration depth is given by :

$$d_p = \frac{\lambda_o \sqrt{\varepsilon'}}{2\pi\varepsilon''} \qquad (2.2)$$

where, λ_o = free space microwave wavelength
(for 2450 MHz, λ_o = 12.2 cm)

2.3.4 Effect of penetration depth & frequency on dielectric properties

Ohlsson, et al (1975) compared the values of ε' and ε'' at 2800, 900 and 450 MHz and found that there was a significant increase in dielectric loss and increase in temperature dependence with decreasing frequency, resulting in considerably smaller differences in penetration depth and in calculated temperature profiles. Generally, the greater the moisture and salt contents, the shallower the microwave penetration depth and consequently, the less uniform the heating rate throughout the product.

It is important to note that food electrical and physical properties which affect microwave heating change dramatically at temperatures below freezing point. While microwaves heat foods internally, the depth of penetration of the energy varies in different foods. The depth is largely controlled by the dielectric properties.

In general, the designers follow three rules :

- ✡ The higher ε", the greater the lossiness of the food and the better it will absorb microwaves and heat.
- ✡ The smaller the d_p, the more surface and uneven heating.
- ✡ High salt concentrations lead to small d_p and possible runaway heating as ε" is likely to increase with temperature.

It has been reported that 900 MHz microwaves lose more energy than 2450 MHz microwaves in certain materials, whereas the reverse is true in other materials; in some materials, loss is the same at both frequencies. Where depth of penetration is desired in a given material, one can choose the microwave frequency with the lower loss factor. It is also shown that under similar conditions, by the time half of their incident energy is lost, 900 MHz microwaves will penetrate water to a depth of 76 mm, whereas 2450 MHz microwaves will penetrate to a depth of only about 10 mm (Potter et al. 1996).

2.3.5 Grain bulk density and its effect on dielectric properties

The bulk density of grain is the second most important factor, after moisture content, affecting the dielectric properties of grain at a given frequency and temperature (Nelson, 1982). It has been shown that certain functions of the dielectric properties of particulate materials vary linearly with the bulk density of the air-particle mixtures (Nelson, 1983a, b,; 1984a).

2.3.5.1 Review of mathematical relationships

Certain functions of the dielectric properties of particulate materials vary linearly with the bulk density of the air-particle mixtures.
These relationships were :

$$\sqrt{(\varepsilon_r')} = m_1 \rho + 1 \qquad (2.3)$$

$$\sqrt{(\varepsilon_r'' + e)} = m_2 \rho + \sqrt{(e)} \qquad (2.4)$$

$$\sqrt[3]{(\varepsilon_r')} = m_3 \rho + 1 \qquad (2.5)$$

where m_1, m_2, m_3 are constants for the slope of the single linear regressions and "e" is a constant dependent upon the material. Equations 2.3 and 2.4 are consistent with the quadratic relationship between dielectric properties and density reported by Kent (1977) for data on fish meal:

$$\varepsilon_r' = a\ \rho^2 + b\ \rho + 1 \qquad (2.6)$$

$$\varepsilon_r'' = c\ \rho^2 + d\ \rho \qquad (2.7)$$

where a, b, c and d are constants. The similarity between Equations 2.3 and 2.6 and Equations 2.4 and 2.7 is evident if one considers that: $a = m_1^2$; $b = 2\ m_1$; $c = m_2^2$; and $d = 2m_2\ (e)^{1/2}$.

Comparison of Equations 2.4 and 2.7 leads to the following condition:

$$e = \frac{d^2}{4c} \qquad (2.8)$$

Equation 2.5 was reported to give a more reliable determination of the dielectric constant (Nelson, 1983a, b; 1984a; Nelson and You, 1989). It is also consistent with proven mixture formulas which specify the additivity of the cube roots of the dielectric constants of the constituents of a mixture when taken in proportion to their volume fractions (Nelson, 1984a).

All the equations from (2.3) to (2.8) are found to concord with the theory that when $\rho = 0$, the values of the dielectric properties are those of free space (or air); $\varepsilon_r' = 1$ and $\varepsilon_r'' = 0$. The advantage of Equations 2.3 to 2.5 is that they can be easily extrapolated to values of the density exceeding the range of measurements. This was found to be quite helpful in the case of grains (corn, wheat). An estimation of the dielectric properties of the kernel might be attempted by compromising a sample of grain up to kernel density (around 1.3

g/cm^3), which is very difficult with fragile microwave components. The same result was obtained by measuring the dielectric properties of the material over a certain range of easily attainable density (usually upto 1.1 t/m^3) and then extrapolating the straight lines to desired kernel density.

2.3.6 Dielectric properties and MW sterilization.

Ohlsson and Bengtsson (1975), concluded that the dielectric properties of foods and their dependence on temperature, frequency and composition greatly influence the temperature distribution developed during microwave sterilization. Studies on computer simulation in microwave sterilization indicate that the resulting dielectric properties depend on the food composition, temperature as well as on the processing frequency (Ohlsson et al. 1975).

2.3.7 Dielectric properties and Quality of agri-food material

Dielectric properties and the quality of a substance can be correlated, for example: either high or lower frequencies can be used to measure the quality of fish and meat (Kent & Kress Rogers, 1987).

Dielectric properties (ε' and ε") were compared to conventional methods of analysis (viscosity, refractive index, iodine value, peroxide value & the fatty acids) for evaluating the frying quality of a blend of cotton seed and sunflower oils and also for predicting deterioration occurring during heating of oil as a large portion of edible oil is consumed in frying of foods (El-Shami et al., 1992).

Dielectric constant was found to be the most significant indicator for quality control in commercial deep fat frying operations and also concluded that polymer content and changes in dielectric constant are useful for monitoring frying oil quality (Smith et al, 1986).

2.3.7.1 Water quality detection

The dielectric behaviour of artificially polluted water, and polluted water collected from various industrial locations were carefully analyzed and the

results suggested the use of a novel and effective technique for detecting the pollutants in water at microwave frequencies based on the measurement of complex permittivity of polluted water (Raveendranath et al. 1995).

2.4 MW heating and effects on thermo-physical properties of a material

Positive and negative charged ions of dissolved salts in foods, such as common table salt, sodium chloride, also interact with an electrical field by migrating toward oppositely charged regions of the electrical field and disrupt hydrogen bonds with water to generate additional heat. Magnetic field interactions are negligible, since foods contain only trace amounts of magnetic minerals such as nickel, cobalt, and iron. The heat generated within a product by the electrical interactions is then transferred throughout the product by conventional thermal conduction. Engineering properties of biological materials are very useful in microwave applications. In particular; thermal, electrical and physical properties play a significant role. Microwave penetration depths within the product are determined by its electrical and physical properties and can vary significantly with chemical composition and temperature of the product and the processing frequency.

The electrical and physical properties of foods/biological materials which determine microwave penetration depth, conventional heat transfer, and overall heating rate are the dielectric constant and loss factor, heat capacity (specific heat) and density. The dielectric properties and some related electrical properties which affect the transmission and absorption of microwave energy are rigorously defined in classic references on the nature of electromagnetic waves and applications of dielectric materials by von Hippel (1954a, b).

Food solids do not absorb microwave energy in high or intermediate - moisture product, although dried food solids do absorb energy. On the other hand, low-moisture products heat more uniformly because of their low heat

capacities (Schiffman, 1986). Moisture content and temperature of product also affect rates of conventional heat transfer (internal conduction and surface convection) which are determined by thermal diffusivity (ratio of thermal capacity to specific heat and density). Heat is also lost from the product to the microwave oven as a result of surface cooling by moisture evaporation.

2.5 Microwave development

Microwave processing offers many potential benefits to industry including time and energy savings, floor space reductions, and better use of assets. Major areas of ongoing research in microwave processing include microwave/material interactions, dielectric measurements, microwave equipment design, process scale-up and evaluation, and new materials development. The establishment of multidisciplinary teams to research, develop, and market innovative approaches to employing microwaves will determine the rate and extent of the growth of a technology that offers much more than a convenient method for heating our food (Craig et al. 1995).

2.6 ISM frequency

In North America, only frequencies of 915 MHz and 2450 MHz are being used for Industrial, Scientific and Medical (ISM) microwave applications to avoid interference with telecommunications, defence and maritime applications. At these frequencies, the penetration depth in the material will allow a reasonable processing rate/cost ratio. Therefore, it is interesting to investigate on the applicability of the existing models to lower microwave frequencies. The lower frequency systems refers to either the HF (3-30 MHz) and VHF (30-300 MHz). HF includes dielectric and radio frequency (RF) heating. It is generally accepted that dielectric heating is done at frequencies between 1 and 100 MHz, whereas microwave heating occurs between 300 MHz and 300 GHz.

2.7 Measurement of dielectric properties

A number of measuring techniques are available to measure the dielectric properties of agri-food materials (Nelson, 1994; von Hippel, 1954). Measurements of the dielectric properties are performed by numerous methods employing various sizes and shapes (Westphal et al. 1980) of material. Most measurements in the past have been performed in the frequency domain (FD), but more recently are used in the time domain (TD) (Stuchly et al. 1978). Recent reviews have included methods for both frequency-domain and time-domain techniques (Kaattze and Giese, 1980; Afsar et al., 1986). The dielectric properties of the sample material is calculated from the measured reflection for non resonant methods, and from the measured resonant frequency and Q-factor for resonant methods. Techniques for the measurement of dielectric properties of materials are numerous. At frequencies of interest for dielectric heating below 200 MHz, impedance bridges and resonant circuits have traditionally been used to determine the characteristics of capacitive sample holders with and without a dielectric sample from which the dielectric properties are calculated. At frequencies above 200 MHz and into the microwave region, transmission-line and resonant techniques have been useful. Principles and techniques of RF and microwave dielectric property measurements have been discussed in several classic reviews of such methods (von Hippel, 1954; Altschuler, 1963; Bussey, 1967; Franceschetti, 1967). The development of measuring dielectric properties of materials over a wide range of frequencies (van Gemert, 1973; Kaatzs and Giese, 1980) have been studied. As modern impedance and network analyzers have become available the task of measuring dielectric properties over a wide frequency ranges has become even more efficient. Several techniques are used to measure the dielectric properties of food materials (Sucher and Fox, 1963; de Loor and Meijboom, 1966; Bengtsson and Risman, 1971; Metaxas and Meredith, 1983). Among these, cavity Perturbation is frequently used for measuring dielectric properties of homogeneous food materials because of its simplicity, easy data reduction,

accuracy, and high temperature capability. The measurement errors intrinsic to the open-ended probe for low-loss materials make these measurements difficult. A very sensitive and accurate technique for determining low-loss sample properties is the perturbation technique. This measurement utilizes the change in frequency and the change in absorption characteristics of a tuned resonant cavity.

2.7.1 Waveguide and Coaxial Transmission Line Method

Early efforts in characterizing dielectric properties of materials was carried out at the Massachusetts Institute of Technology (Roberts and von Hippel, 1946; von Hippel, 1954b). The values of ε' and ε'' were derived from transmission line theory, which indicated that these parameters could be determined by measuring the phase and amplitude of a reflected microwave signal from a sample of material placed against the end of a short-circuited transmission line such as a waveguide or a coaxial line. For a waveguide structure, rectangular samples that fit into the dimensions of the waveguide at the frequency being measured are required. For coaxial lines, an annular sample is needed. The thickness of the sample should be approximately one-quarter wavelength within the sample. Preparing an optimal sample, therefore requires guessing the dielectric constant of the material being measured so that the wavelength can be determined. Typical thickness at 2450 MHz range from 0.5 cm for woods to 1.9 cm for fats and oils.

The dielectric parameters can be easily and inexpensively obtained by the transmission line technique, particularly if one utilizes a slotted line and standing-wave indicator (Nelson et al. 1974). A more sophisticated implementation of the technique utilizes a swept-frequency network analyzer, where the impedance is measured automatically as a function of frequency (Hewlett Packard, 1985).

Dielectric sample holder design for the particular materials of interest is an important aspect of the measurement technique. Coaxial-line

sample holders have worked well for measurements on grain and seed samples with a Q-meter at frequencies from 50 MHz (Nelson et al., 1953; Nelson, 1979a). Coaxial line sample holders with open-circuit terminations and with the sample holders modeled as transmission-line sections were used for similar measurements with bridges and admittance meters at frequencies between 50 and 500 MHz (Jorgenesen et al. 1970; Stetson and Nelson, 1970). Coaxial-line and rectangular wave-guide sample holders were used with various microwave measurement systems assembled for dielectric properties determination on grain, seed, and fruit and vegetable tissue samples at frequencies from 1 to 22 GHz (Nelson, 1972, 1973b, 1980, 1983a). The same sample holders were also useful for measurements on pulverised coal and mineral samples (Nelson et al. 1980; Nelson et al. 1989).

2.7.2 Transmission line technique

This technique is cumbersome because the sample must be made into a slab or annular geometry. At 2450 MHz the sample size is somewhat large particularly for fats and oils. Commonly available waveguide test equipment for 2450 MHz is designated WR-284. For measurements at 915 MHz only the coaxial line technique is practical due to the large size of waveguide required. Liquids and viscous-fluid type foods can be measured with this method by using a sample holder at the end of a vertical transmission line.

2.7.3 Open ended probe technique

A method that circumvents many disadvantages of the transmission line measurement technique was pioneered by Stuchly and Stuchly (1980). The technique calculates the dielectric parameters from the phase and amplitude of the reflected signal at the end of an open-ended coaxial line inserted into a sample to be measured. Care must be exercised with this technique because errors are introduced at very low and very high frequencies as well as for low values of dielectric constant and loss factor. This technique is valid for 915

and 2450 MHz for materials with loss factors > 1. Interpretation for lower-loss materials such as fats and oils must be treated with caution. Typical open-ended probes utilize 3.5 mm diameter coaxial line. For measurement of solid samples probes with flat flanges may be utilized (Hewlett Packard, 1991). The open-ended probe technique has been successfully commercialized and the required software and hardware are available. Figure 2.4 shows the coaxial line transmission technique with an open ended probe used to determine the dielectric properties of the material under test.

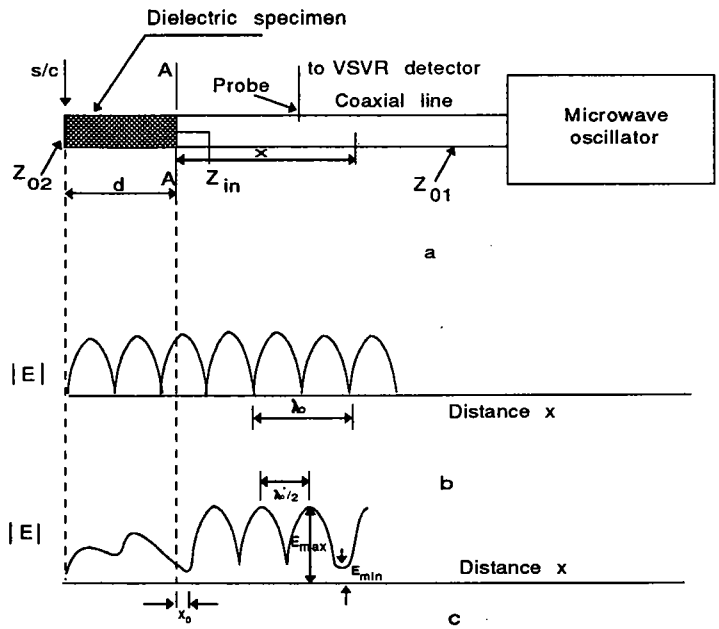

Figure 2.4 Method for dielectric material measurements. General form of apparatus and standing wave pattern.

2.8 Theoretical aspects - cavity perturbation

When a small sample of dielectric material is introduced into a resonant cavity, the frequency of resonance (Δf) is changed by a small amount as well as the quality factor Q of the cavity. These effects on the parameters are usually quantified which results in the measurement of the dielectric properties of the sample. The shift in resonance frequency is considered to be mainly correlated to the dielectric constant while the change in the Q factor is associated to the dielectric loss.

2.8.1 Q-factor and cavity resonator

The Q-factor is referred to as the quantification of the sharpness of the peak in the resonance curve. Thus, when an object is introduced in the cavity, the resonance frequency will decrease and the Q factor will be lowered, causing a broader, flatter resonance curve (Kraszewski and Nelson, 1994). Figure 2.5 shows an example of the resonant curve with and without a perturbing dielectric object in the cavity. The Q-factor of the resonator is generally defined as :

$$Q = \frac{\omega_r W_{max}}{p} = \frac{2\pi W_{max}}{PT} \qquad (2.9)$$

with

$$\omega_r = 2\pi f_r = \frac{2\pi}{T} = \text{resonant frequency} \qquad (2.10)$$

W_{max} = energy received by the resonator

P = Power loss converted by the resonator

With cavity resonators, one can achieve a very high unloaded Q, with well conducting and smooth inner surfaces up to many times 10^4. The Q-factor of the cavity resonator decreases proportionally with $1/(f)^{1/2}$. The introduction

of the dielectric (agri-food material) in the resonator increases its resonant wavelength. The measurement is made by placing a sample completely through the centre of a waveguide that has been made into a cavity. The cavity is made by placing two plates with central holes on either side of a section of waveguide (1.5 times waveguide length). These plates are called irises and give the waveguide a very narrow frequency range for the transmission of microwave energy. Changes in the centre frequency and width of this transmission characteristic, when a sample is inserted, provide information to calculate the dielectric constant and loss factor of the sample.

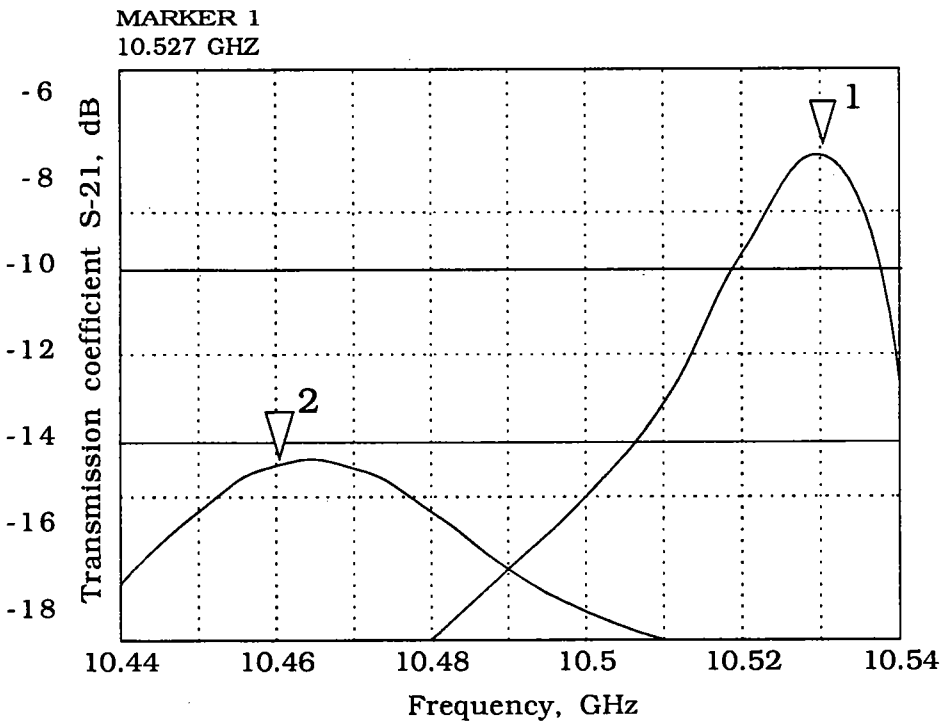

Figure 2.5 Example of a resonant curve with the perturbing dielectric object (2) and without the object (1)

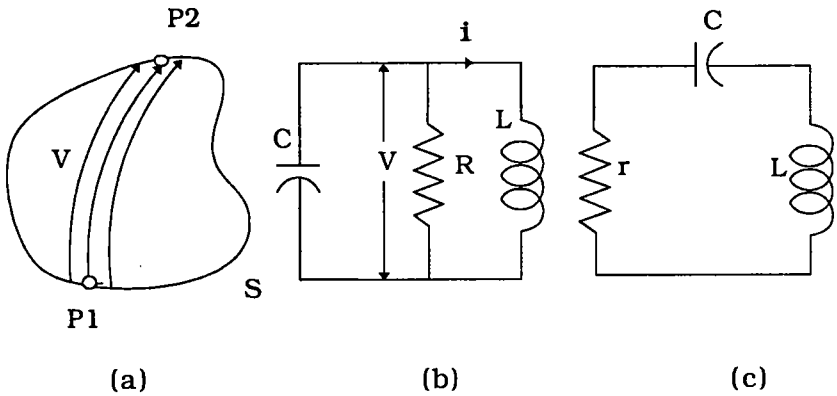

Figure 2.6 Cavity resonator of the equivalent circuit (a) cavity(b) parallel circuit (c) series circuit

The circuit configuration built in a microwave circuit is called a microwave resonator. The wall material is made of a good conducting substance such as brass, copper, etc. In the cavity resonator, the electrical energy is stored in the electrical field and the magnetic energy is stored in the magnetic fields. The energy of the waves oscillate back and forth between the electrical and the magnetic fields. In this system the stored energy reaches a maximum when the microwave frequency in the circuit corresponds to the resonant frequency of the cavity resonator. In the case of resonance, the sum of electrical and magnetic energy is constant over time. A continuous transformation between the electrical and magnetic field energy takes place.

2.8.2 Field Distribution and Configuration

It is known that a time-varying magnetic field produces a time-varying electric field (Faraday's law) which, in turn produces a time-varying magnetic

field (Ampere's law)... and so forth. Since one type of field produces the other in a plane normal to it, the electric and magnetic fields are always perpendicular to each other and travel at the same speed. The simplest method of describing this phenomenon is to orient each field as function of only one coordinate in a 3-D. Figure 2.7 shows an electromagnetic wave at two different times in the 3-D coordinate system.

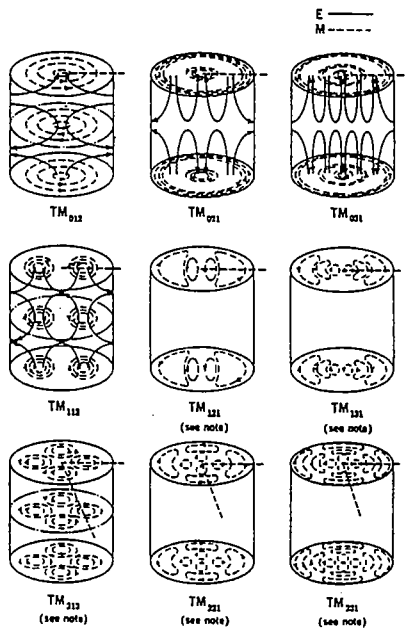

Figure 2.7 Sketch of an electromagnetic-field configuration of TM_{mnk} cylindrical cavity.

2.8.3 Field Modes

As the electric and magnetic fields are perpendicular to each other and both lie in a plane transverse to the direction of propagation, the uniform plane wave is called a transverse electromagnetic wave (TEM) wave. Usually, a resonant cavity is made of a portion of a waveguide or a circular waveguide. Plane wave propagation in the cavity can follow the transverse magnetic field (TM) or the transverse electric (TE) mode. The second alphabet designates which field has its direction always and everywhere transverse to the direction

of wave propagation. The mode notation also includes three subscripts in the form of TM_{mnk} or TE_{mnk}. The alphabet "m" represents the number of full cycles of transverse field variation in one revolution through 2 radians of diameter; "n" represents the number of zeros of the transverse field along the radial of a guide; "k" represents the number of repetition of the TE_{mnk} - TM_{mnk} mode with the opposite phase of the field configurations in the axial direction. Some frequent mode of propagation are displayed in Figure 2.8.

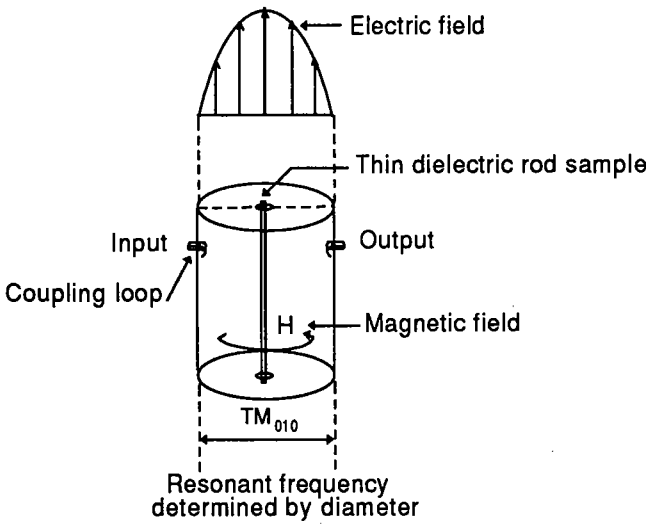

Figure 2.8 The TM_{010} cavity commonly used for perturbation measurement upto 10 GHz

The TM_{010} mode cavity (Figure 2.8), has been referred (Akyel, 1991) to as the simplest, convenient, and relatively less complex (mathematical calculations); geometrically simple and stable system. A typical mode chart of a right circular cylinder of different cavity modes is attached (Appendix E). This particular set up was therefore effectively used for all the measurements conducted in this study.

2.8.4 Active Cavity Perturbation (ACP) Technique

Conventionally, the displacement of the resonance curve as a function of the frequency and deformation (Q-factor) are measured with and without a sample. Those perturbations of the cavity parameters are in general measured by a frequency sweeper, a power sensor or an oscilloscope to record the resonance frequency and its variation in time (Akyel, 1991). These instruments are disposed in an open circuit arrangement starting with the source and ending with the signal detection. Network analyzers are used to observe any change in the cavity's characteristics.

However, a complete circuit requires several other components as shown in Figure 2.9. Bidirectional couplers (C_p) are required to control the incident and the reflected waves. A "coupler" links between the source of the electromagnetic signal and the field inside the cavity. A phase shifter (M) will bring back all the signals to the same phase at every cycle around the loop. One or more attenuators (A_t) can be disposed along the circuit to lower the power before fragile components. However, phase and power are not measured in the cycle since only frequencies are necessary to compile the dielectric properties. In an active cavity perturbation (ACP), the same principles apply for the determination of the dielectric properties from the shift in the resonant frequency and the change in Q. However, the active system simply represents a new approach to obtain the resonance frequency and the Q factor without considering much about their origin. In ACP, the operation of the system is mainly based on the information concerning the phase of the cavity rather than the frequency. The circuit is closed in a loop composed of amplifiers, phase shifters, attenuators, modulators and the actual resonant cavity. The closed loop circuit very much resembles microwave oscillator circuit using an amplifying element and positive feedback.

An oscillation condition is produced whenever proper conditions of phase and amplitude are satisfied in the feedback loop (Akyel and Bosisio, 1990). The principal characteristic of the ACP apparatus is that it oscillates at the

resonance frequency. In the simplest case, an active system consist of only an amplifier (A), a cavity (C) and a certain length of wire causing a delay (T).

For ease of measurement, a network analyzer can be used to

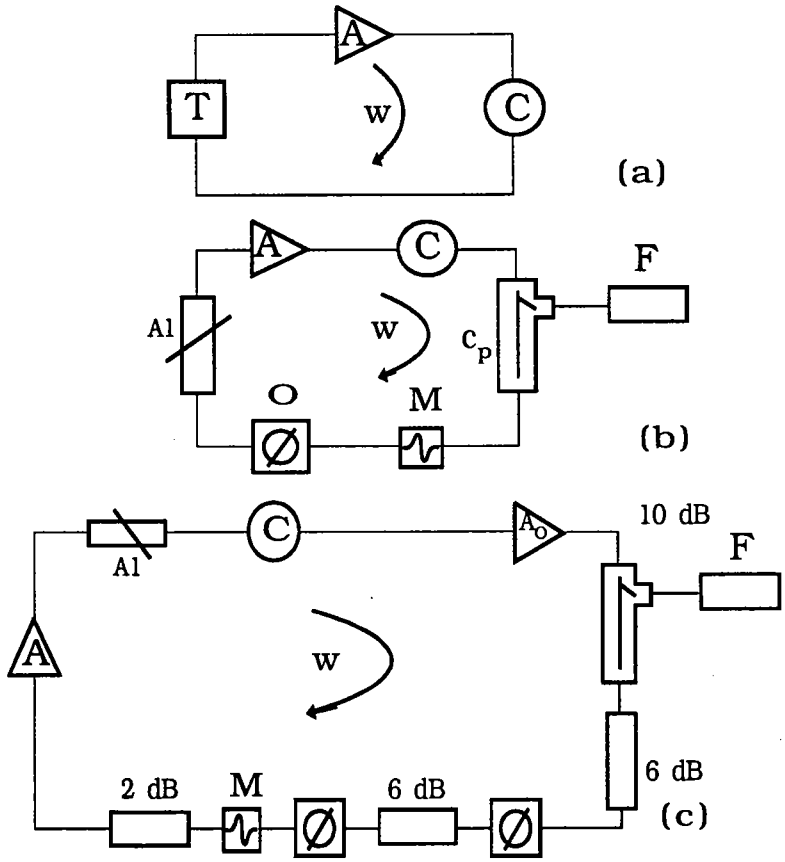

Figure 2.9 Active system under different configurations. (a) = minimum system (b) one amplifier system (c) two amplifier system.

automatically display changes in frequency and width (Engelder and Buffler, 1991). A recommended waveguide cavity design with full theory and design details is available as a standard procedure published by the American Society for Testing and Materials (ASTM, 1986). Use of cavity perturbation technique is extensively used for the study of dielectric properties of various agri-food materials such as grain, milk, raisins (Akyel, 1995).

Figure 2.10 Shows a block diagram of an arrangement of a single mode active cavity measurements (ACP) used in permittivity measurements.

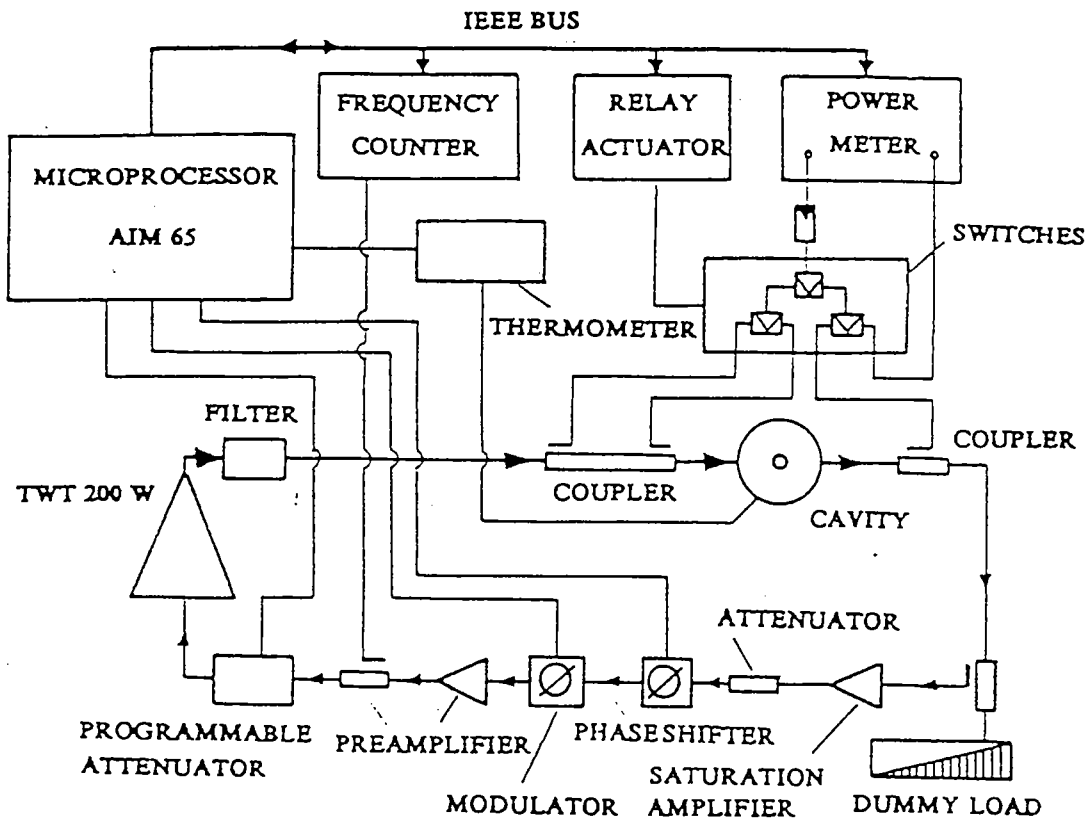

Figure 2.10 Shows a block diagram of an arrangement of a single mode active cavity measurement (ACP) used in permittivity measurements.

2.8.5 Coaxial line reflection method

In the reflection methods the test sample permittivity is obtained from the measurements of the reflection coefficient (the scattering parameter S_{11}) at a defined reference plane usually at the interface of the test dielectric. The reflection coefficient may be measured by a slotted line or a network analyzer,

or by forming a resonator terminated by the sample in the frequency domain (FD) or by a time domain (TD) spectrometer (Stuchly and Stuchly, 1978).

2.9 Recommended sample preparation

Sample geometries can be of circular, rectangular, or square cross section with a recommended maximum dimension of approximately 0.318 cm for a 2450 MHz system. Circular cross section is recommended because the measurement is independent of rotational orientation of the sample. This geometry however may be more difficult than a square cross section for fabrication. For a WR-284 cavity a cavity length of 11.56 cm is appropriate to give an approximate resonance frequency of 2450 MHz. A sample length of 6.35 cm will adequately extend through the waveguide.

2.9.1 Preparation of solid samples

For solid materials, samples in the form of rods can be formed, molded, or machined directly from their material into microwave transparent test tubes or tubing. The material of the sample holder is an important factor to consider prior to subjecting the sample into electromagnetic fields. Although quartz is preferable, borosilicate glass is acceptable, but not the ordinary glass. Wall thickness should be as thin as practical, commensurate with required mechanical rigidity. Paper or plastic straws may also be used if glass is not available.

2.9.2 Preparation of liquid samples

Liquids are filled into test-tube sample holders with a pipette. Small diameter pipettes themselves also make excellent sample holders; 100 microlitre (μL) pipettes are used for a low-loss materials and 10 μL pipettes for high-loss materials. Materials that can be melted are poured into sample holders and allowed to solidify. This technique is appropriate if the material does not change its properties following melting and resolidification.

III MATERIALS AND METHODS

In this chapter, the materials tested and their preparation are described. Because semi-solid materials can be difficult to insert into the micropipettes used in the cavity perturbation technique of measurement of dielectric properties, a special device was designed to facilitate their insertion. The equipment is described in section 3.3. Standard procedures were used for the other materials (grain, liquids).

Data acquisition and mathematical aspects of determination of dielectric properties are also described.

3.1 Grain Samples: Selection and Preparation

Samples of clean yellow-dent field corn (*Zea Mays L.*,) and hard red winter wheat (*Triticum Aestivum L.*,) were used for the experiment. The corn moisture content was evaluated (13.65%, w.b) as per the method prescribed by ASAE (American Society of Agricultural Engineers) Standard S352.2 DEC 92. The moisture content of wheat sample was 9.22% (w.b). These values of moisture content are typical of the good storage conditions in which the grain was stored after the drying operation. The moisture content was kept at these equilibrium values to assure a stable moisture content of the powder since the grain was to be ground.

For each type of grain, 3 samples with different particle-size distributions were prepared (see sections 3.1.1 and 3.1.2 below). In addition, 1 whole kernel sample was prepared for each. In the case of corn, a ground sample was prepared and sieved to 7 particle-size ranges. Each of these ranges provided a sample to test the relationship between dielectric properties and bulk properties (bulk density and particle size). For wheat, only 6 such samples were prepared. Thus 21 grain samples were used to measure the dielectric properties at different densities. All measurements were taken at 915 MHz and 24°C temperature. The moisture content was constant for all

samples : 13.65% (w.b) for corn and 9.22% (w.b) for wheat.

3.1.1 Grain particle size

Samples of both corn and wheat were ground in three mixtures of different particle sizes. The operation consisted more in chopping than in grinding since a simple Osterizer™ home blender was used. The resulting mixture was a combination of particles of different sizes ranging from fine powder to half-grain particles. Different residence times in the blender were used to obtain different particle size distributions. The three different size of corn were labelled C1, C2 and C3 whereas the wheat was labelled W1, W2 and W3. A sample of 200 g was chopped for each labelled sample out of which 50 g was quickly sealed in sample bottles and kept at 4°C until 12 hrs before the dielectric properties measurement. The remaining 150 g of each sample were used in a sieving test to determine the particle size distribution.

3.1.2 Sieving

The sieves used were U.S. Std.# 4, 8, 12, 20, 35, 60 and 140 for corn and U.S. Std.# 12, 20, 35, 60 and 140 for wheat. A suitable sieve shaker (Ro-tap™) was used. The time of shaking and method of data analysis were in confirmation with the ASAE Standard S 319.2 for feed materials. After completion of the appropriate shaking time the material left in each sieve was precisely weighed and transferred into sample bottles and kept at 4°C until 12 hrs before the dielectric properties measurements were done. These bottles were labelled according to the type of grain and sieve number. For example, CS 12 stands for Corn sample taken from sieve # 12. Only one set of such bottles was filled for each grain. Because the grain was originally coming from an original source, care was taken to avoid any occurance of differences between sieved material of different batches. The shaking method was standardised and was used for all the runs.

3.1.3 Sample conditioning

As a reference point, bottles were filled with samples of whole kernels of both grains for further dielectric properties measurement. They were labelled as "WGC" for whole grain corn and "WGW" for whole grain wheat. All the bottles were stored at 4°C until the experiments started. 12 hrs before the start of the experiments the bottles were taken out and kept at room temperature to stabilize slowly in order to avoid condensation in the bottles. Kernel density was calculated by measuring the volume of water displaced by a known mass of grains. An average of over 10 readings resulted in a value of 1.193 t/m^3 for corn and 1.187 t/m^3 for wheat.

3.1.4 Sample holder for grain particles

The sample holder consisted of a piece of teflon (low ε" material) carefully machined to fit the cavity and to withstand sufficient pressure to bring bulk density to above 1.0 t/m^3. At such a density, a straight line of sufficient reliability would allow extrapolation of the dielectric properties to kernel density.

Initially the sample holder was filled with the grain sample, loosely and a sample height gage made of a calibrated steel rod was inserted to form a plane sample surface at the top of the material. The scale scribed on the rod corresponded directly to the volume of the sample which was calculated as the inside cross-sectional area by the length. Weighing of the sample allowed for density calculation on site.

3.2 Preparation of liquid samples: organic solvents and edible Oils

Organic solvents such as ethanol, ethanol and hexane mixtures were mixed in different compositions (100% ethanol, 90% ethanol+10% hexane, 70% ethanol+30% hexane, etc.) and carefully sealed in corked laboratory glass bottles. Commercially available cooking oils such as canola, sunflower and soya were stored in the refrigerator for 2 days and small quantities were

drawn and kept in ambient conditions for 1 - 2 hours before the start of measurements. The sample bottles were kept in ambient conditions until the dielectric measurements were performed. All the measurements on organic solvents were done at 2450 MHz and at temperatures from 22-60°C. Tests on the oils were performed at 3 frequencies (2450, 915 and 400 MHz) and temperatures ranging from -30°C to 75°C. The various combinations of temperature and frequency used on the liquids are shown in Table 3.1.

Table 3.1 Selected frequencies and temperature range used for liquid samples such as edible oils (canola, soya and sunflower) and organic solvents such as ethanol and hexane mixtures.

Sample	Frequency (MHz)	Temperature range (°C)
Edible oils	2450 & 915 400	-30 to 75 Room temperature
Organic solvents	2450	22 to 60

The liquid samples were pipetted into standard cornings (10, 50,100 µl) pipettes which are made of pyrex/borosilicate glass. The size of the micro-sampling pipettes depend on the lossiness of the sample to be tested. High loss materials (biological) requires lower range micropipettes since the cavity demands the small sample size for the signal to interact with such samples. One end of the pipet was sealed using a standard tube sealant (wax, silicone rubber) which is a very low loss material by itself. Volume of the sample (mm^3) was calculated by knowing the height of the sample or the cavity and the rated capacity of the micropipette. The volume of the sample (v_s) was calculated by the ratio of the heights of the cavity and the sample respectively.

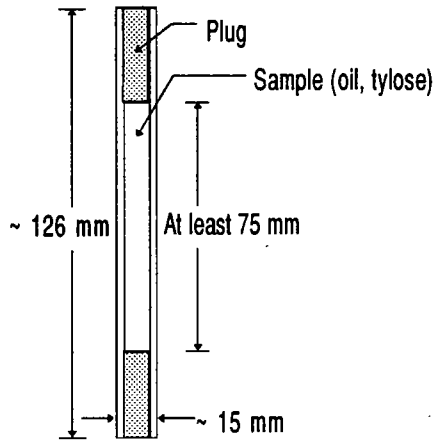

Figure 3.1 Schematic of a micropipette pyrex (100µl).

3.3 Preparation of Semi-Solid Material: Tylose ™

Tylose MH 1000 (methylhydroxyethyl cellulose) was used as the sample in the "semi-solid" category of this experiment. It was available commercially in the form of a thick "paste" with 77% moisture content. The number "1000" represents its viscosity grade. Since it could not be filled into a delicate 10µl micro-sampling pipette manually, a new equipment (Figure 3.2) was designed and built to accomplish this task.

Basically, the equipment gripped the micropipette at both ends. One end was connected to a vacuum pump and the other to a manually operated screw press. The sample was carefully filled inside the hollow acrylic section through which the sample was forced. The acrylic tube was internally machined in such a way that the sample coming out at the other end would flow easily into the pipet. The handle of the screw press was rotated manually before the vacuum pump was switched on to give an initial push for the material inside the micro-sampling pipette. Presence of high water content in tylose suggested the use of 10 µl pipette.

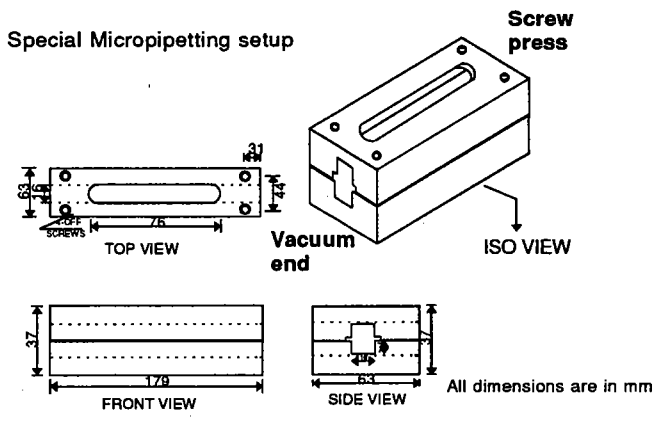

Figure 3.2 Special micropipeting set up.

The whole unit was housed inside a precisely machined aluminum compartment which has a window to observe the pipetting action. The upper half of the aluminum compartment was unscrewed to remove the pipet for each run. Suitable rubber padding were provided for the housing of the pipet assembly to avoid breaking the fragile pipet. Care was taken to seal off the pipet ends (silicone rubber, wax) immediately to avoid air infiltration that could affect the dielectric properties at below freezing temperatures. The dielectric properties of water and ice are reported to be different (Nelson, 1976). Using the newly built equipment, it took less than a minute to successfully fill the sample in the micropipettes. The samples were labelled and stored in the cold room until their dielectric properties were measured. Before the final measurements were done the pipettes filled with the sample were kept in ambient condition for 1-2 hrs.

3.4 Instrumentation and Software

The operation of the apparatus was made user friendly with the development of an appropriate Quick Basic Program. When using the software, it first prompts the user to enter the parameters which will stay constant regardless of the samples. It first asks for the resonance frequency of the cavity, in order to know what frequency to sweep during the sensing operation. The next thing the operator must enter is the volume of the empty cavity, for further use as v_c. The last input is the shape of the cavity (rectangular or circular) which will help to use the proper value of Bessel constants in the equations. The Bessel constant is dependent on the geometry of the resonant cavity.

When proceeding with sample testing, the next step is to put the empty sample holder in the cavity and press enter on the PC Keyboard. The program then activates the network analyzer to read ΔF and Q_o for empty cavity but with the sample holder in place so that the tested material is the only addition in the real test. The program then asks to put the sample in the sample holder and press enter. It then takes only a few seconds for the program to find ΔF and Q. The next information the operator is asked to enter is the volume of the sample v_s. The program then has all the variable to calculate ε' and ε'' and display them on the screen, ready for another measurement. A sketch of the setup is shown in Figure 3.3.

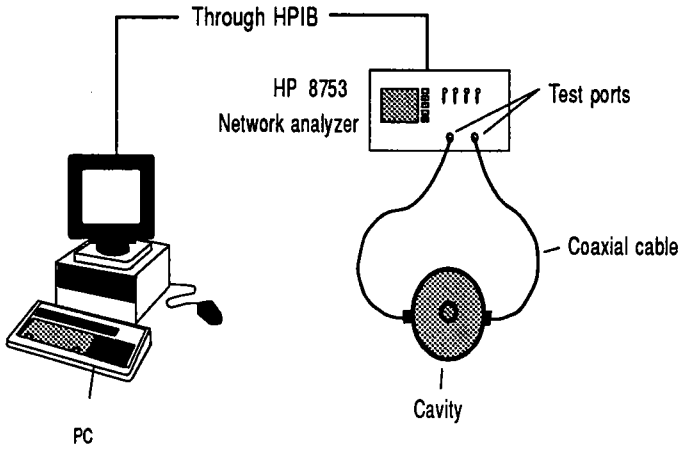

Figure 3.3 Schematic of the experimental set up.

3.5 Measurement System and Methods

As mentioned earlier, one of system's main characteristics is the use of the information contained in the phase of the signal. This method constitutes a short cut to improve the measuring speed. However, in order to correlate the frequency to the phase, the system must find a reference value of Q using the longer approach and then proper calibration can lead to great time savings. This reference value is called Q_o and the detailed description is in Akyel (1991). This method (Figure 3.4) requires a high resolution programmable attenuator (0.1 dB by change). The method is based on the complete sweeping (at least 360°) by an electronic programmable phase shifter. At start, the attenuator is initialized at a value which assures a gain inferior to 1 for the whole system. Also, no oscillation is observed. The value of the attenuator is then decreased by a regular step until an oscillation is obtained. For each step the phase shifter sweeps a range of 360°. A confining algorithm was developed to "touch" the peak of the resonance curve. The system goes forward by a regular step of reasonable value (1 or 2 dB) until a first "cut" of the curve is

obtained for a certain range of phase values. Then, the step size is decreased by one and the increment is divided in half and second "cut" of the curve is obtained. The same procedure is repeated (diminishing step by one and dividing increment by two) until the attenuator's resolution is at a limit. This is the "touching point"; of the resonance curve. In fact, it consists of a small region limited by two values close to each other. The point in the middle of this zone is then considered as the resonance frequency at the peak of the amplitude curve. Figure 3.4 shows the relation between amplitude (top curve) and phase (bottom curve) on a frequency graph.

Once the resonance frequency " f " is found, the value of Q is obtained by computing the ratio of f over the frequency band 3 dB below the amplitude of " f ".

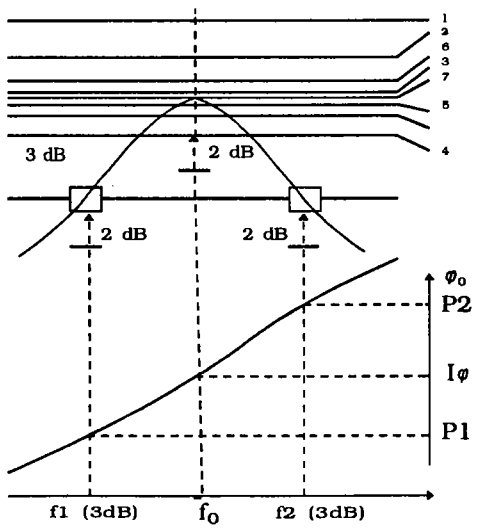

Figure 3.4 Absolute measurement of quality Q_o factor.

3.6 Mathematical representation

$$Q = \frac{f}{f_2(3dB) - f_1(3dB)} \tag{3.1}$$

where :

f = resonance frequency

f_2 (3dB) = f(+3dB)

f_1 (3dB) = f(-3dB)

Accomplishing this procedure with the empty cavity results in the determination of Q_o, the Q factor of the empty cavity. With this reference Q factor, it is then possible to obtain the Q factor with samples without having to go through the whole procedure again using a simple relation of frequencies:

$$Q_s = Q_o * \frac{\Delta\omega_O}{\Delta\omega_S} * \frac{\omega_S}{\omega_O} \tag{3.2}$$

where :

Q_s = Q of cavity loaded with a sample

Q_o = Q of empty cavity (without sample)

ω_o = resonance frequency of empty cavity

ω_s = resonance frequency of loaded cavity

$\Delta\omega_o$ = resonance frequency band (±3dB) of empty cavity

$\Delta\omega_s$ = resonance frequency band (±3dB) of loaded cavity

3.7 Calculation of Dielectric Properties

The following equations apply to all methods using cavity perturbation technique and therefore are not restricted to active cavity perturbation (ACP) only. The perturbation theory has been discussed by many authors (Waldron,

1967). It establishes a relationship between the properties of the cavity (Δf and Q) and the dielectric properties of the material (ε' and ε"). The transmission factor (ΔT) must first be described as :

$$\frac{\Delta T}{Q_o} = \left(\frac{1}{Q_s} - \frac{1}{Q_o}\right) \tag{3.3}$$

$$\Delta F = 2(\varepsilon' - 1)\, K\, f_o \left(\frac{v_s}{v_o}\right) \tag{3.4}$$

$$\varepsilon' = 1 + \frac{0.5}{K} \left(\frac{v_s}{v_o}\right) \left(\frac{\Delta f}{f_o}\right) \tag{3.5}$$

$$\frac{\Delta T}{Q_o} = \left(\frac{1}{Q_s} - \frac{1}{Q_o}\right) = 4\varepsilon'' K^2 \left(\frac{v_s}{v_o}\right) \tag{3.6}$$

$$\varepsilon'' = 0.25 K^{-2} \left(\frac{v_o}{v_s}\right) \left(\frac{1}{Q_s} - \frac{1}{Q_o}\right) \tag{3.7}$$

For the cavity perturbation set up used in this study, the constant K was computed and the final equations for ε' and ε" were in this form :

$$\varepsilon' = 1 + 0.539 \left(\frac{v_s}{v_o}\right) \left(\frac{\Delta f}{f_o}\right) \tag{3.8}$$

$$\varepsilon'' = 0.269 \left(\frac{v_o}{v_s}\right) \left(\frac{\Delta f}{f_o}\right) \tag{3.9}$$

where:

ε' = dielectric constant

ε'' = dielectric loss factor

v_s = volume of the sample (mm^3)

v_o = volume of the cavity (mm^3)

k = factor dependent upon shape of the object, orientation and permittivity

The following equations used in the Quick-Basic program for data acquisition were in the form :

$$\varepsilon' = 1 + \frac{(Bessel * v_o * (\Delta f)}{f_o\ (v_s + 1.0\ e^{-06})} \tag{3.10}$$

$$\varepsilon'' = \frac{(Bessel\ /\ 2) * \Delta\ Q * v_o}{(v_s + 1.0\ e^{-06})} \tag{3.11}$$

where Bessel = Bessel constant which is dependent on the geometry of the resonant cavity (similar to k). For a rectangular cavity, Bessel = 0.5 and for a circular cavity, Bessel = 0.539.

3.8 System Utilization

3.8.1 Active cavity set up

As mentioned in section 3.4, the operation of the system was made user friendly with the development of an appropriate software using Quick Basic (Appendix D). The software is mouse driven. To obtain the dielectric properties of a sample, the steps followed are :

* Launching the software from the Windows environment.
* Asking for initial measurement to compute Q_o.
* Asking to save Q_o as a reference value.
* Inputting the volume of the sample v_s.
* Then, the dielectric properties are displayed as well as other information about the network.

3.8.2 Instrumentation (comments/discussion)

The ACP method has been developed to accelerate the process of measurement of the resonant frequency in the early 1980's. It should not be forgotten that the need for this particular aspect has been decreased since the event of high speed computers. However, the system described here is very versatile since it can work with any resonator with a single or multiple modes. Some adjustments are needed for the latter. The active cavity system also allows to heat the sample in the cavity which cannot be accomplished by using a network analyzer.

Because of the recent availability of faster and improved network analyzers, such pieces of equipment can be used to accomplish the same task as the active circuit. In the present study of determining the dielectric properties of various agri-food materials, the resonant cavity was hooked directly to a HP 8753 network analyzer. Because of built-in programs, the analyzer could do most of the job for finding Δf and Q. This has an effect of simplifying the Basic Program, and is only required to communicate with the analyzer and transform the changes to Δf and Q into ε' and ε''.

3.8.3 Data Acquisition / Network analyzer set up

The data acquisition system consists in a Data Translation™ DT2817 card for which the specifications are included (Appendix B). This card has 32 lines of input/output that are divided into four 8-bit ports. It has a built-in multiplexer which can allow sequential operation of channels. Three main components are connected to the card; the attenuator is controlled through D/A conversion as well as the phase shifter. Both instruments require 12 bits of resolution. The power sensor is hooked as an input and uses 12 bits also. It uses the A/D conversion capabilities of the card. Multiplexer is effectively used to perform many functions simultaneously.

Because of this, the frequency meter is connected to the General Purpose Interface Bus of the PC because it is an instrument which comes with its own

data conversion system and would not be compatible to the card. Figure 3.5 shows the general layout of the data acquisition equipment.

The set up using a Hewlett-Packard HP 8752 network analyzer is quite simple. The analyzer is connected to the General Purpose Interface Bus (GPIB) of a PC and works on remote mode i.e. waiting for the computer to send a signal initiating a measurement routine. The cavity is connected to the test ports of the network analyzer through appropriate coaxial cables.

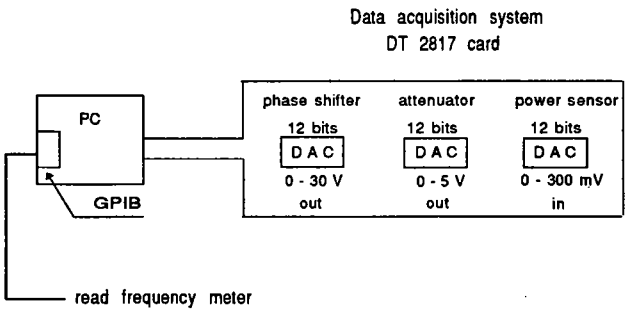

Figure 3.5 General layout of the data acquisition system

3.8.4 Critical Cavity Parameter

The dimensions of the cavities are to be determined using very specific rules. Cylindrical cavities have been used more often when compared to rectangular ones (Akyel, 1991), and their dimensions are determined more easily. For these reasons, only the equation for the size of the cylindrical cavities is shown here;

Cylindrical cavity in the TM_{010} mode :

where : f= resonance frequency in GHz

$$f = (\frac{22.966}{D}) \quad (3.12)$$

D= inside diameter in cm

The only restriction to the height of the cavity is that it must be less than 0.5 D. Also, it is recommended that the best material to be used for the cavity should be brass, copper or aluminum. Samples of high moisture contents are not recommended for high frequency cavities (5 GHz and above). The rule of thumb is that the sample size should be equal or smaller to (1/1000 th) of the volume of the empty cavity in order to prevent too many perturbations. Figure 3.6 shows a simple rectangular cavity arrangement with the test sample.

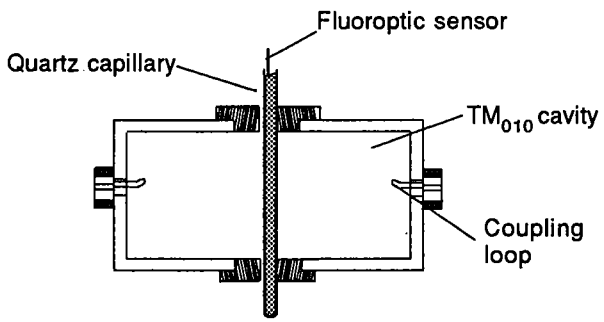

Figure 3.6 Test sample geometry and cavity arrangements

IV RESULTS AND DISCUSSION

4.1 Bulk density effect (grain) and regression model

The first step in this work was to determine the relationship between the dielectric properties and bulk densities of grains. The approach taken was to grind the grain and then pack the material to different densities. In this way, we could evaluate the relationships between the dielectric properties and packing properties of the same material. Results for corn (C1) are presented in Table 4.1 below.

Table 4.1 Dielectric constants ε_r' and dielectric loss factors ε_r'' of chopped corn C1 sample with similar densities.

Density (t/m^3)	ε'	ε''
0.585	2.51	0.2721
0.618	2.51	0.2772
0.619	2.79	0.3425
0.758	3.04	0.4112
0.904	3.04	0.4112
0.951	4.15	0.7263
1.058	4.88	0.9507
1.102	5.24	1.0552

Linear regressions were obtained for the following models:

$$\sqrt{(\varepsilon_r')} = A_o + A_1 \rho \tag{4.1}$$

$$\sqrt[3]{(\varepsilon_r')} = A_o + A_1 \rho \tag{4.2}$$

where A_o and A_1 are regression coefficients. The point (0,1) was included in the data for all regressions to account for the dielectric constant of air when the sample holder is empty. The results of the regressions are listed in Tables 4.2 and 4.3 where the regression coefficients A_o and A_1 appear, as well as the values of the coefficient of determination r^2, and the standard error of estimate s.e. Most of the values obtained for A_o, the axis intercept, are close to the theoretical value of 1. Inclusion of the (0,1) intercept emphasizes this similarity between the regression models and the theoretical models, as mentioned earlier in Equations 2.3 and 2.4. The cube root model seems to better represent the phenomenon as earlier reported. The r^2 values were also higher for the cube root model. The reliability of this simple linear regression made it possible to extrapolate for the dielectric constant at densities outside the range of measurements.

Figures 4.1 to 4.2 show the relationship between the dielectric constant and bulk density for corn (C2) and wheat (WS 60). The square and cubic roots of the dielectric constant (ε') are also displayed as data points with the corresponding regression models shown as solid lines. The point (0,1) is inserted as the ε'- axis intercept. These results confirm the validity of the existing linear regression models to the data obtained by the cavity perturbation method.

Table 4.2 Coefficients of regression model relating the dielectric constant ε_r' to the density ρ for corn @ 24°C and 915 MHz.

		$(\varepsilon_r')^{1/2} = A_o + A_1\rho$				$(\varepsilon_r')^{1/3} = A_o + A_1\rho$			
Sample	Density range (t/m^3)	A_o	A_1	r^2	s.e	A_o	A_1	r^2	s.e
C1	0.585-1.102	0.9236	1.1612	0.977	0.0635	0.9716	0.6636	0.989	0.0254
C2	0.622-1.122	0.9413	1.1764	0.988	0.0479	0.9804	0.6709	0.996	0.0164
C3	0.650-1.111	0.9488	1.1919	0.988	0.0459	0.9839	0.6810	0.996	0.0152
WGC	0.534-0.807	0.9988	1.1937	1.000	0.0064	1.0037	0.7076	0.999	0.0062
CS4	0.507-0.747	0.9927	1.1400	0.997	0.0185	0.9994	0.6818	0.998	0.0082
CS8	0.611-0.898	0.9879	1.1273	0.997	0.0184	0.9983	0.6643	0.999	0.0061
CS12	0.582-0.885	0.9775	1.1088	0.993	0.0288	0.9925	0.6547	0.997	0.0108
CS20	0.660-0.964	0.9604	1.0260	0.977	0.0583	0.9818	0.6052	0.985	0.0273
CS35	0.528-1.003	0.9492	1.0552	0.986	0.0415	0.9800	0.6171	0.993	0.0171
CS60	0.523-1.026	0.9694	1.0450	0.996	0.0218	0.9939	0.6076	0.999	0.0066
CS140	0.378-0.992	0.9576	1.0383	0.994	0.0267	0.9884	0.6046	0.998	0.0089

Table 4.3 Coefficients of regression model relating the dielectric constant ε_r' to the density ρ for wheat @ 24°C and 915 MHz.

		$(\varepsilon_r')^{1/2} = A_o + A_1\rho$				$(\varepsilon_r')^{1/3} = A_o + A_1\rho$			
Sample	Density range (t/m^3)	A_o	A_1	r^2	s.e	A_o	A_1	r^2	s.e
W1	0.726-1.090	0.9744	0.9326	0.990	0.0320	0.9917	0.5477	0.994	0.0142
W2	0.680-1.056	0.9809	0.9152	0.995	0.0217	0.9949	0.5412	0.998	0.0078
W3	0.670-1.052	0.9738	0.9489	0.992	0.0270	0.9917	0.5586	0.997	0.0101
WWG	0.707-1.004	0.9873	0.8962	0.995	0.0266	0.9957	0.5345	0.998	0.0103
WS12	0.668-0.981	0.9907	0.9205	0.998	0.0166	0.9980	0.5495	1.000	0.0042
WS20	0.670-0.996	0.9888	0.9179	0.996	0.0232	0.9968	0.5486	0.998	0.0090
WS35	0.596-0.988	0.9705	0.9172	0.990	0.0336	0.9886	0.5452	0.996	0.0103
WS60	0.475-1.088	0.9631	0.9152	0.992	0.0255	0.9875	0.5416	0.997	0.0042
WS140	0.618-1.024	0.9786	0.8856	0.994	0.0206	0.9936	0.5274	0.998	0.0090
WP	0.459-0.812	0.9834	0.8448	0.993	0.0204	0.9938	0.5146	0.997	0.0132

A similar approach was taken to examine the relationship between ε_r'' and e. In this case, however, only the square root model was used. In order to find e in Equation 2.4, the following regression was obtained:

$$\varepsilon_r'' = A_o + A_1 \rho + A_2 \rho^2 \tag{4.3}$$

Coefficients for Equation 4.3 are listed in Tables 4.4 and 4.5. The coordinate (0,0) was included in all regressions. The values obtained for A_o are nearly zero and therefore negligibly small for all samples. Thus Equation 2.3 was found similar to Equation 2.7 and the value of e for use in Equation 2.4 was determined by Equation 2.8. The regression of $(\varepsilon''+e)^{1/2}$ on density ρ, then could be calculated by:

$$\sqrt{(\varepsilon_r'' + e)} = A_o + A_1 \rho \tag{4.4}$$

The regression coefficients for this linear regression are listed in Tables 4.4 and 4.5. As for the ε_r' vs ρ relationship, the values of A_o are close to the expected figures. The r^2 values also indicate a good reliability of the simple linear regressions for all samples.

Figures 4.3 and 4.4 show the relationship between the dielectric loss factor (ε'') and the bulk density for corn (C2) and wheat (WS 60). These samples were arbitrarily chosen. In this case, only the square root model was used. The experimental and modified values of ε'' are shown as points where as the models described in Equations 4.3 and 4.4 are shown as solid lines. The coordinate (0,0) is included and the models explain well the variation of ε_r'' with respect to density.

The main reason for developing these models was its simplicity in extrapolating the linear relationships to the whole kernel density (1.3 t/m^3) of both grains.

Table 4.4 Coefficients of regression model relating the dielectric loss factor ε_r'' to the density ρ for corn @ 24°C and 915 MHz.

		$(\varepsilon_r'') = A_o+A_1\rho+A_2\rho^2$				$(\varepsilon_r''+e)^{1/2} = A_o + A_1\rho$			
Sample	Density range (t/m^3)	A_o	A_1	A_2	r^2	A_o	A_1	r^2	s.e
C1	0.585-1.102	0.0073	-0.2391	1.0587	0.994	-0.0207	0.9259	0.994	0.0255
C2	0.622-1.122	0.0018	-0.1672	0.9973	0.998	-0.0165	0.9265	0.998	0.0158
C3	0.650-1.111	0.0022	-0.1946	1.0856	0.998	-0.0156	0.9568	0.998	0.0166
WGC	0.534-0.807	-0.0009	0.1825	0.7431	0.996	0.0102	1.0016	0.997	0.0170
CS4	0.507-0.747	0.0007	0.0596	0.8627	0.997	0.0045	0.9723	0.999	0.0104
CS8	0.611-0.898	0.0000	-0.0167	0.9336	0.998	-0.0036	0.9291	0.999	0.0075
CS12	0.582-0.885	0.0000	-0.1239	0.9747	0.982	-0.0078	0.9144	0.998	0.0116
CS20	0.660-0.964	-0.0011	-0.1117	0.9384	0.995	-0.0074	0.9083	0.997	0.0169
CS35	0.528-1.003	0.0001	-0.0252	0.7865	0.999	-0.0032	0.8734	1.000	0.0057
CS60	0.523-1.026	0.0006	0.0068	0.7030	0.999	-0.0021	0.8415	1.000	0.0051
CS140	0.378-0.992	0.0053	0.0422	0.6387	0.999	-0.0223	0.8114	0.996	0.0157

Table 4.5 Coefficients of regression model relating the dielectric loss factor ε_r'' to the density ρ for wheat @ 24°C and 915 MHz.

		$(\varepsilon_r'') = A_o+A_1\rho+A_2\rho^2$				$(\varepsilon_r''+e)^{1/2}=A_o+A_1\rho$			
Sample	Density range (t/m^3)	A_o	A_1	A_2	r^2	A_o	A_1	r^2	s.e
W1	0.726-1.090	-0.0024	-0.0143	0.4971	0.963	-0.0055	0.6973	0.987	0.0269
W2	0.680-1.056	-0.0006	-0.0117	0.4850	0.997	-0.0023	0.6888	0.999	0.0076
W3	0.670-1.052	0.0009	-0.0391	0.5603	0.997	-0.0027	0.7228	0.998	0.0090
WGW	0.707-1.004	0.0003	-0.0541	0.5450	0.999	-0.0123	0.6256	0.990	0.0123
WS12	0.668-0.981	-0.0002	0.0581	0.4666	1.000	0.0033	0.7301	1.000	0.0063
WS20	0.670-0.966	0.0000	-0.0244	0.5546	0.996	-0.0013	0.7263	0.999	0.0104
WS35	0.596-0.988	0.0011	-0.0142	0.5178	0.995	0.0004	0.7083	0.997	0.0143
WS60	0.475-1.088	0.0012	0.0235	0.4628	0.998	0.0081	0.6938	0.998	0.0092
WS140	0.618-1.024	0.0002	-0.0297	0.5029	0.987	-0.0032	0.6878	0.999	0.0058
WP	0.459-0.812	0.0010	-0.0710	0.3700	0.996	-0.0122	0.6805	0.994	0.0149

4.2 Particle size effect: quadratic regressions & nominal sieve diameter

In order to analyze the effect of particle size, the linear relationships found previously were used to find the dielectric properties at a given density. It was decided to evaluate them at both kernel density and typical bulk density of the grain sample. This exercise was conducted only for the samples which were the result of a separation by sieving operation; CS4 to CS140 and WS12 to WS140. The cubic root model was the only one used to extract values of ε_r'. This is justified by the method previously discussed (1.193 t/m^3) for corn and (1.187 t/m^3) for wheat. Because typical bulk densities were in the range of the measurements, the values found in ASAE standard D241, 4 Feb 93 (0.721 t/m^3 for corn and 0.772 t/m^3 for wheat) were used.

These empirical values of the dielectric constants were then fitted with quadratic regressions:

$$\varepsilon_{re}' = A_o + A_1 d + A_2 d^2 \tag{4.5}$$

where the e subscript refers to empirical values and d is the nominal sieve diameter of the sieve from which the sample was collected.

Figure 4.5 shows the quadratic regression of empirical dielectric loss factor (ε_{re}'') on nominal sieve diameter (d) for corn.

$$\varepsilon_{re}'' = B_o + B_1 d + B_2 d^2 \tag{4.6}$$

Coefficients for the regressions are listed in Table 4.6. The quadratic relationship seems appropriate. The regression line, however fits well the rest of the points on this Figure 4.5. It would seem appropriate especially for the regression of ε_{re}'' on d.

Table 4.6 shows the results of the quadratic regressions of dielectric properties to nominal sieve diameter. The r^2 values show good representation of the model.

Table 4.6 Coefficients for regression equations relating the dielectric constant ε_r' and the dielectric loss factor ε_r'' to nominal sieve diameter d. Density is expressed in t/m^3.

	ε'				ε''			
Regression parameters	Corn Bulk density	Corn kernel density	Wheat bulk density	Wheat kernel density	Corn bulk density	Corn kernel density	Wheat bulk density	Wheat kernel density
A_0	2.8327	4.8774	2.7075	4.1664	0.5857	0.9763	0.5189	0.8002
A_1	0.1912	0.4398	0.2583	0.5745	0.0528	0.0850	0.0726	0.1072
A_2	-0.0186	-0.0438	-0.0925	-0.2270	-0.0059	-0.0097	-0.0253	-0.0039
r^2	0.8730	0.9030	0.9620	0.9630	0.8610	0.8770	0.9420	0.9760

Thus the fact that quadratic or cubic models could explain most of the variation of the dielectric properties on diameter suggests that further relationships can be examined. These polynomials could be modified to look like Equations 2.3 to 2.5 with ρ replaced by d. Further work on this topic is recommended. It would then be possible to express the functions of the dielectric properties (square root and cube root) with respect to a plane having axes ρ and d.

The nominal sieve diameter is believed to be a good representation of the particle size. If segregation of the material occurs due to sieving, the same amount of segregation is likely to be obtained in particles crushed or powdered to the same size.

A tentative study of the effect of geometric mean diameter (gmd) of the samples of chopped grains on dielectric properties was initiated. However, the three different sizes of mixture did not constitute enough points to show any regular pattern in the behaviour of these relations. The mean geometric diameter might not be the best figure to represent the particle size in this research.

4.3 Dielectric properties and behaviour of tylose

Tylose is a biological material with moisture content (77% w.b.), which freezes between 0°C and (-25)°C. The dielectric properties of tylose were determined at a MW frequency of 2450 MHz and for different temperature ranges (freezing and thawing). The temperature of the jacketed resonant cavity was controlled by a glycol recirculation temperature controlled bath. At frozen state, tylose behaved as a very low loss material (low ε'' and $\tan\delta > 0.5$); however it reversed this trend at higher temperatures (above 20°C). Tylose is not a homogeneous sample however it is a water soluble complex compound.

Tylose MH 1000™ (Hoechst celanese corporation) was available in the form of thick paste with viscosity grade "1000", ready to knead into different shapes. However, filling these samples in the micropipette was performed using a special micropipeting equipment (as discussed in section 3.3).

Tylose is used as a thickening, stabilising and a plasticizing agent; also, tylose (with salt) finds useful application in the food processing area i.e. to simulate the salty food or high loss material like "ham", proper phantom food samples are assessed to achieve simplified numerical models.

Figures 4.6 and 4.7 indicate the dielectric properties (ε', ε'') and behaviour of tylose, with and without salt (2%, 3% and 4%), at MW frequency of 2450 MHz as determined by the cavity perturbation technique. ε'' shows an increasing trend for tylose (> 2 % salt) where as ε' values were found to be consistent.

Figures A1 to A4 in Appendix A indicate the frequency response (1 GHz to 6 GHz) of tylose at room temperature, as measured by 1-port reflectometer technique, with an open-ended coaxial line hooked on to a network analyzer. This method uses "air" and "pure water" as reference media for calibration. The probe was held in direct contact with the material under test (tylose) for few seconds and the indicated results (Table 4.7) were found to be consistent with the results obtained by the cavity perturbation technique. However, it was found that there was some variation in the values of the $\varepsilon'$and ε'' as

compared to different measuring techniques.

The variation in the values of the dielectric properties using different measuring techniques were attributed to the following reasons :

* Different measuring techniques lead to some variation which is reported by many authors.
* Tylose being rich in moisture, air locked in the sample holder (micropipette) has contributed to the variation particularly at below freezing temperatures where there is a phase change of water into ice.

Table 4.7 Comparative results of the measurements of the dielectric properties and its variation of tylose sample at MW frequency (2450 MHz) and 22°C.

	Cavity Method		Reflectometer Method		% variation (x100)	
Sample	ε'	ε"	ε'	ε"	ε'	ε"
Tylose (0% salt)	83.4	14.90	54.5	11.5	0.346	0.228
Tylose (2% salt)	82.54	15.96	54.50	25.50	0.350	0.374
Tylose (3% salt)	80.36	29.327	54.0	31.0	0.328	0.053
Tylose (4% salt)	80.58	34.20	54.25	31.0	0.326	0.093

4.4 Dielectric properties and behaviour of organic solvents

Organic solvents such as ethanol and hexane were mixed in various standard proportions and the dielectric properties were determined.

Figures 4.8 and 4.9 indicate the behaviour of ε' and ε" of ethanol and the mixture of ethanol and hexane, at temperature ranging from 22°C to 62°C. The values of ε' decreased as the % of ethanol decreased. The same trend can be observed with respect to ε". Samples of pure ethanol exhibited higher values of the dielectric properties. The trends of ε' with temperature were identical for ethanol concentrations of 10 to 30% and were flat from 37 to 62°C. At higher concentrations of ethanol, the trend with temperature was to

increase throughout the range. In the case of the loss factor, ε", the temperature trends of the mixtures were quite flat. Basically, ethanol is a better absorber of microwave energy than is hexane.

4.5 Dielectric properties and behaviour of edible oils

Figures 4.10 to 4.15 show the variation of dielectric properties (ε' and ε") of different edible oils (canola, soya and sunflower oils) at different temperatures and frequencies. All the three oils have shown similar responses at MW frequency of 2450 M Hz whereas at 915 MHz, the values of ε' and ε" for all the three oils were found to be inconsistent, possibly due to contribution of the constituents of the sample. Fats that are liquid at room temperature are called oils. Oils and fats are made up of fatty acid esters of glycerol. Edible fats and oils come from both plant and vegetable sources and have important functional and nutritional properties in agri-foods. Monoglycerides and diglycerides decompose at temperatures ranging from 160°C to 190°C (Potter et al. 1996). The ingredients of the above said oils and their behaviour at different processing temperatures and frequencies will have an effect on the dielectric properties. However, the processing frequency is an important parameter which needs to be carefully monitored by the food processing industries and the home MW oven manufacturers.

Dielectric properties of canola oil and milk (2% homogenised) were measured at 915 MHz and 400 MHz @ 24°C and compared. Results (Tables 4.8 and 4.9) indicate the average values of 7 replicates.

Table 4.8 Comparative dielectric properties of oil and milk @ $f = 915$ *MHz*.

Sample	ε'	ε"	Q_o	Q_s
Canola oil	6.12	0.188	3592.684	511.6357
Milk	70.74	14.41	3592.712	3326.6368

Table 4.9 Comparative dielectric properties of oil and milk @ $f = 400$ *MHz.*

Sample	ε'	ε"	Q_o	Q_s
Canola oil	5.8	0.1902	7281.615	7035.698
Milk	58.34	16.4625	6981.659	1530.443

The values of ε' of both samples decreased with decreasing frequency (from 915 MHz to 400 MHz) and ε" increased with decreasing frequency. However, the values of the quality Q-factor reached higher and desirable values at 400 MHz.

The majority of the parameters involved in the MW equipment design are product oriented. Dielectric data and quality of various edible oils (reheated), need to be upgraded due to the fact that the oils are increasingly becoming popular and used in the frying and cooking processes in the food and hotel industries.

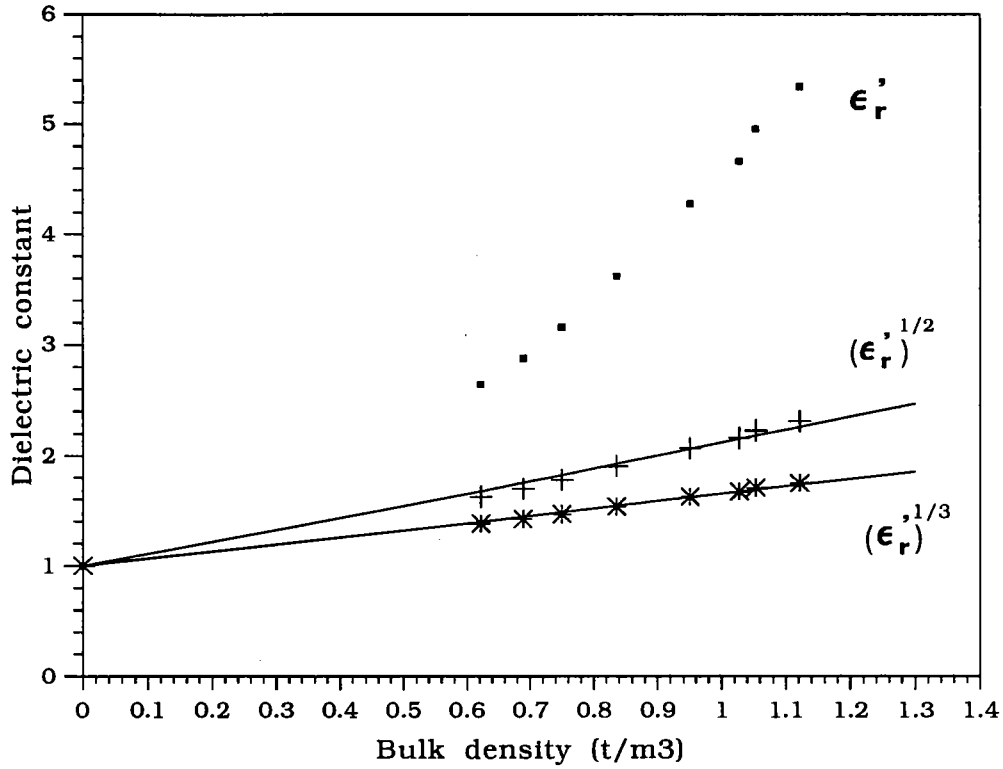

Figure 4.1 Functions of ε_r' vs density for C2 chopped corn sample.

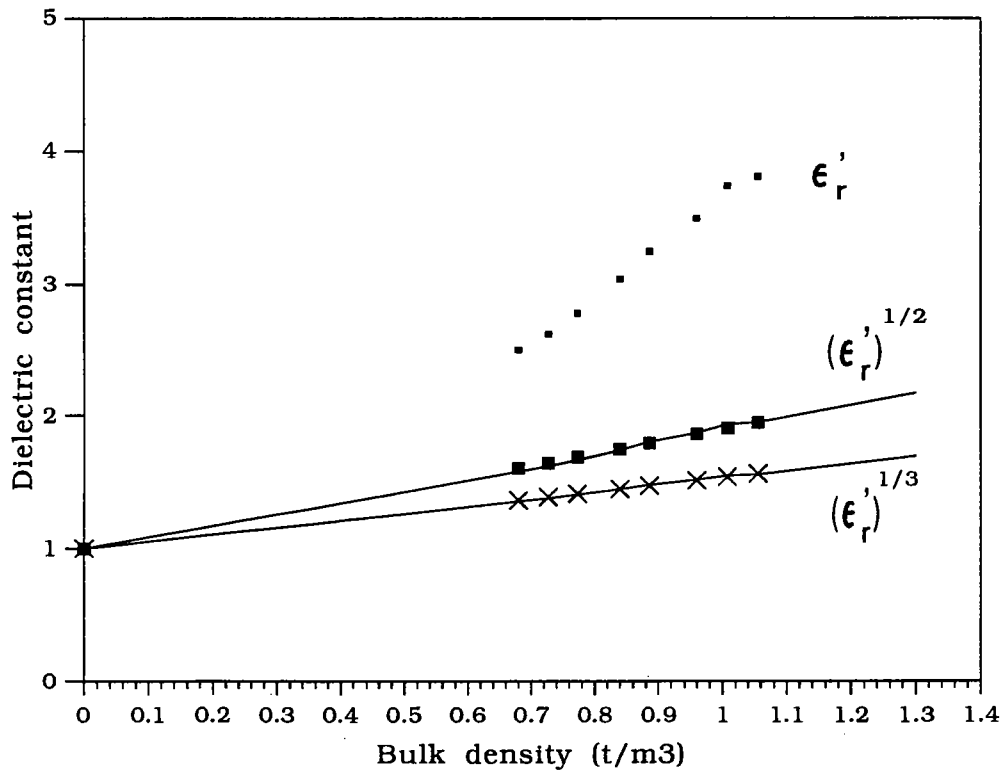

Figure 4.2 Functions of ε_r' vs bulk density for WS 60 sieved wheat sample.

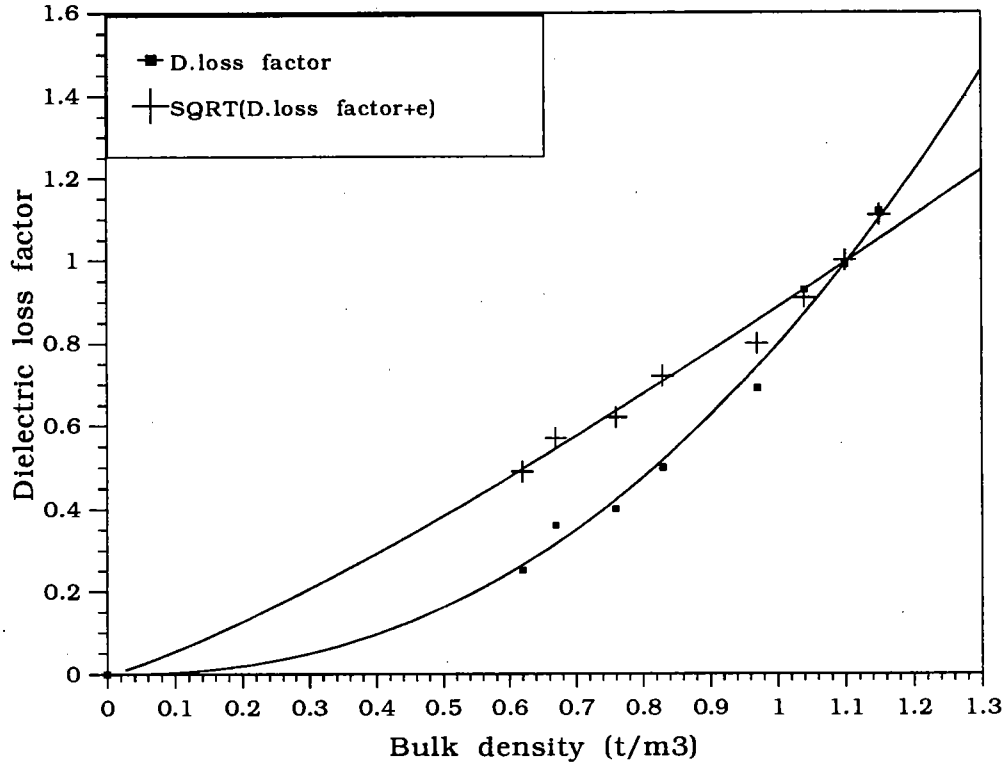

Figure 4.3 Functions of ε_r'' vs bulk density for C2 chopped corn sample.

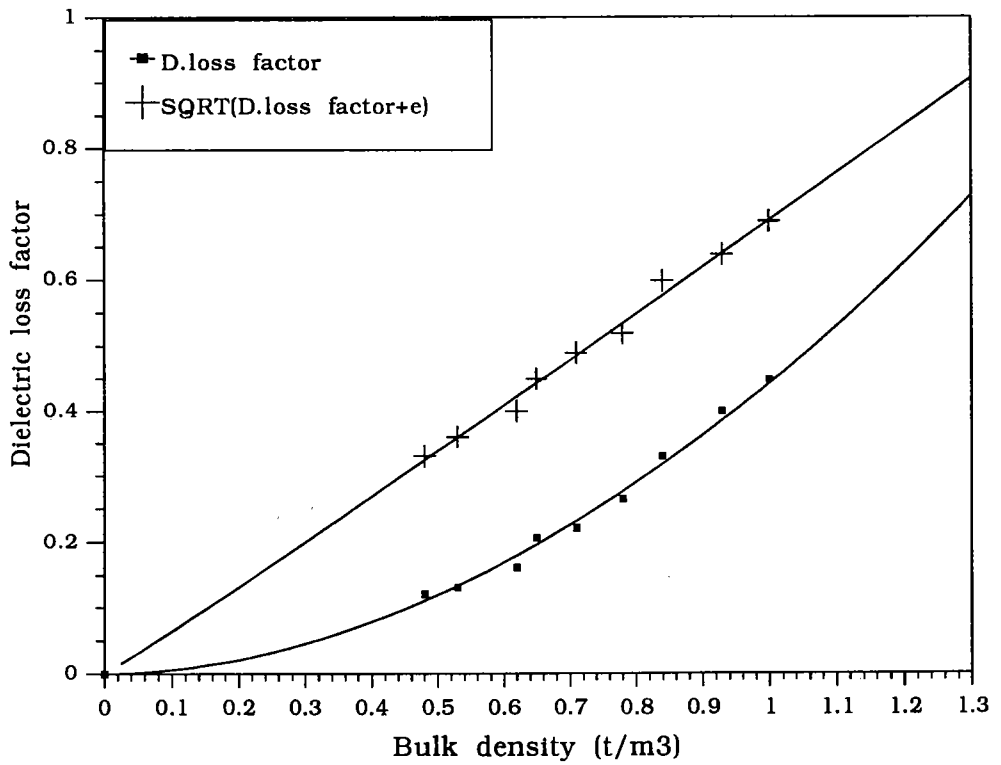

Figure 4.4 Functions of ε_r'' vs bulk density for WS 60 sieved wheat sample.

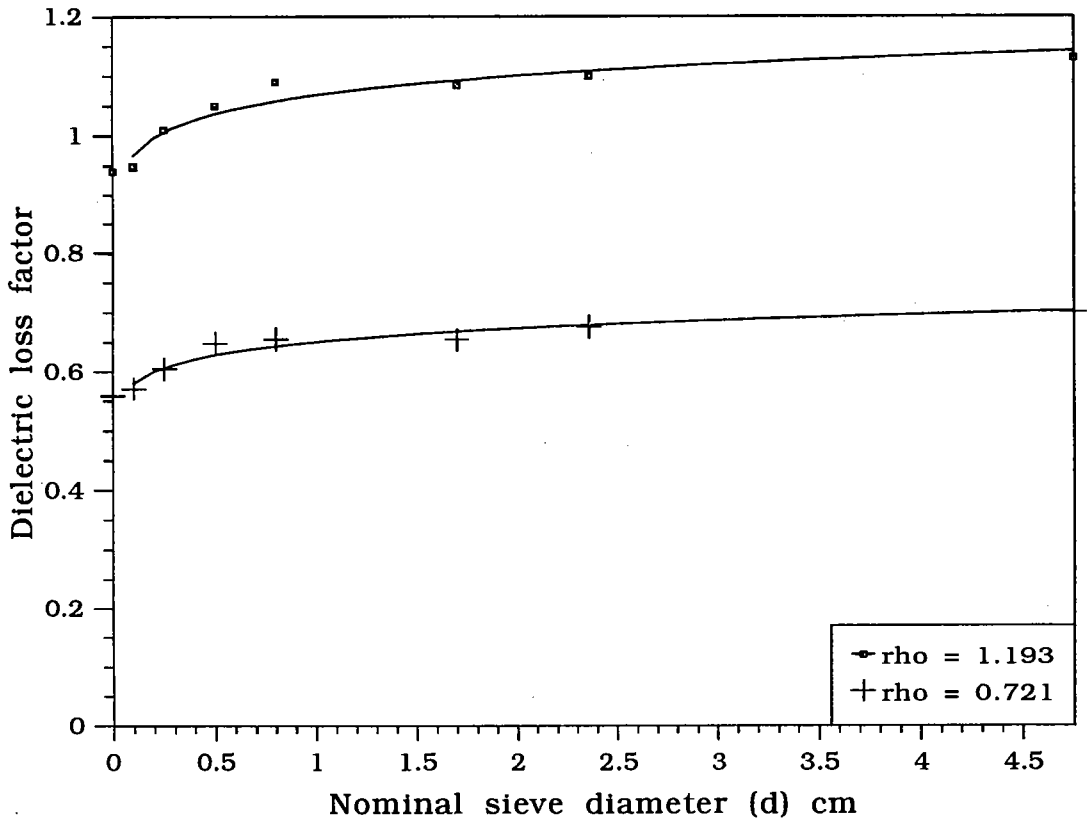

Figure 4.5 Quadratic regression of empirical dielectric loss factor ε_{re}" on nominal sieve diameter "d" for corn sample.

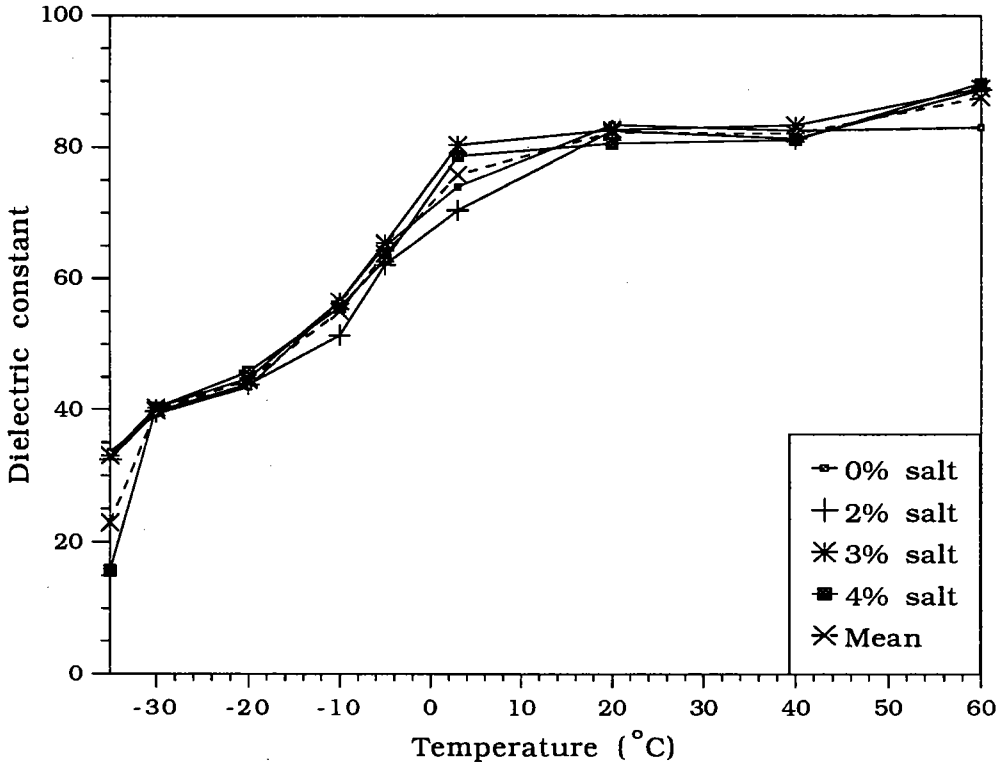

Figure 4.6 Dielectric constant vs temperature of tylose and it's different salt compositions at 2450 MHz frequency.

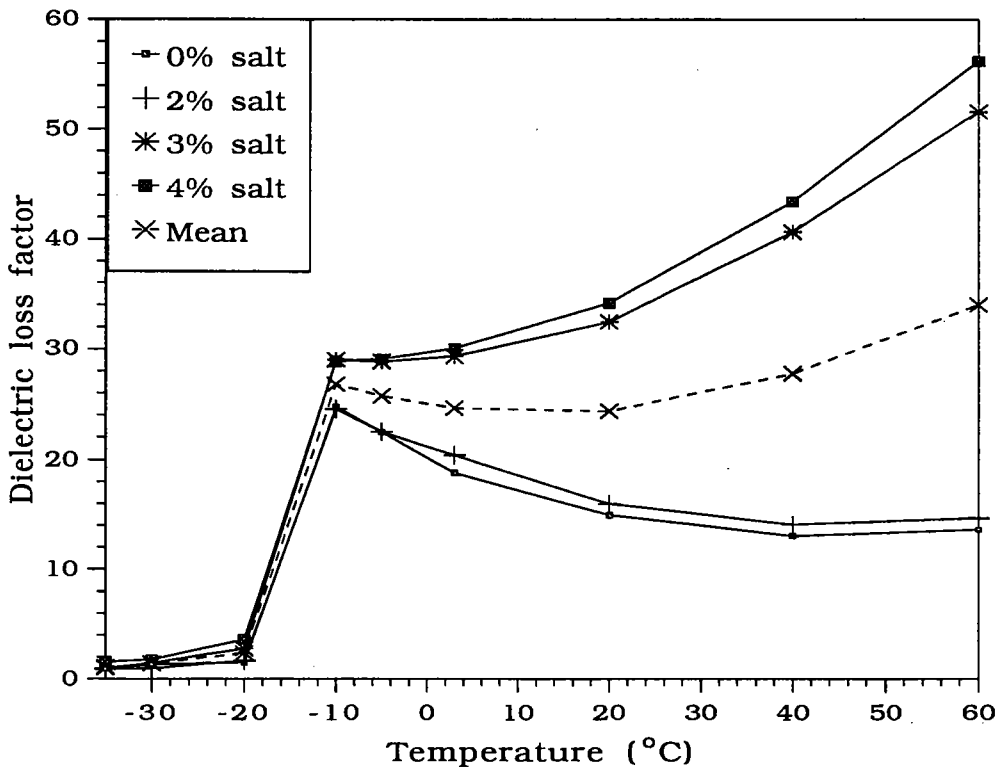

Figure 4.7 Dielectric loss factor vs temperature of tylose and it's different salt compositions.

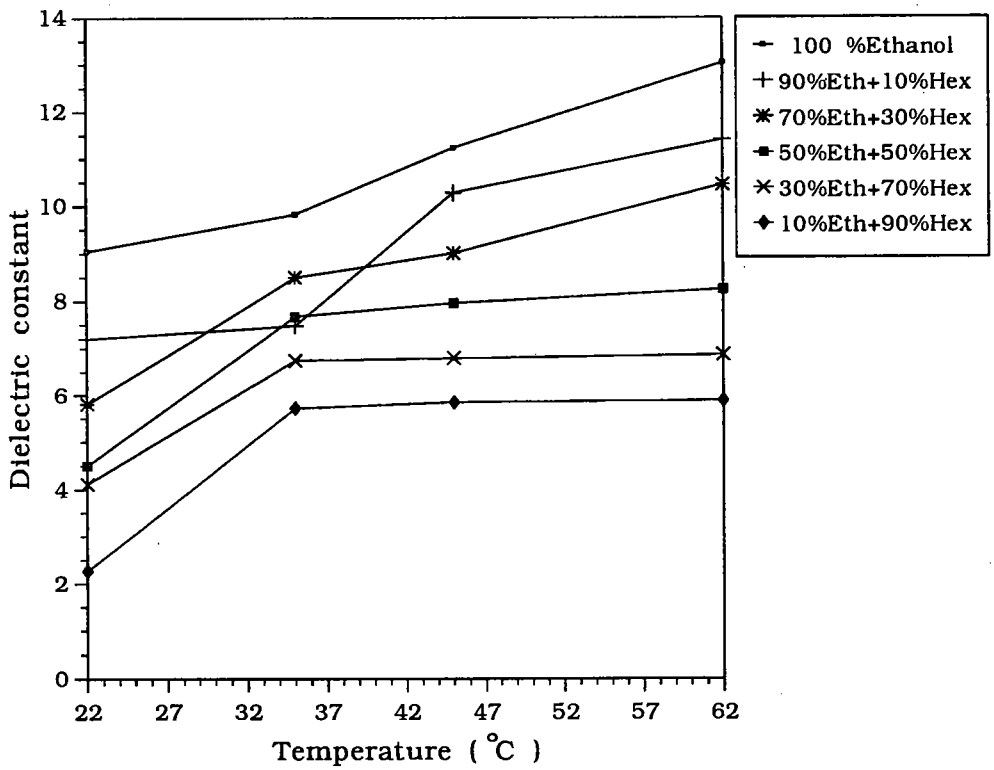

Figure 4.8 Dielectric constant vs temperature of ethanol and hexane mixtures at f = 2450 MHz.

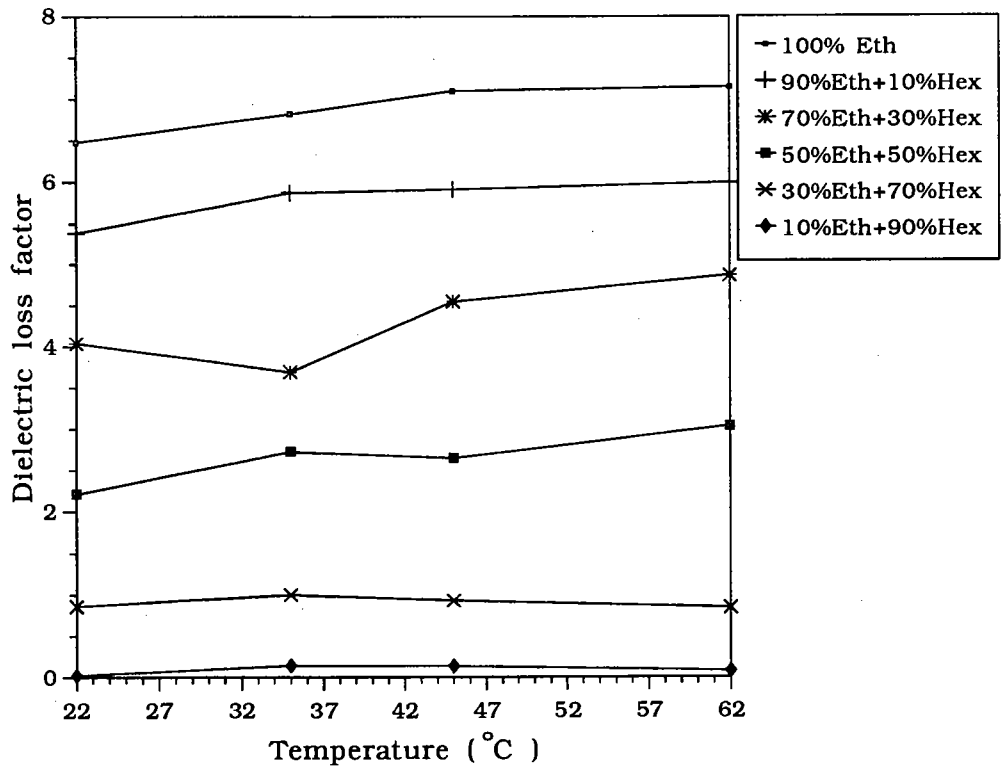

Figure 4.9 Dielectric loss factor vs temperature of ethanol and hexane mixtures at f = 2450 MHz.

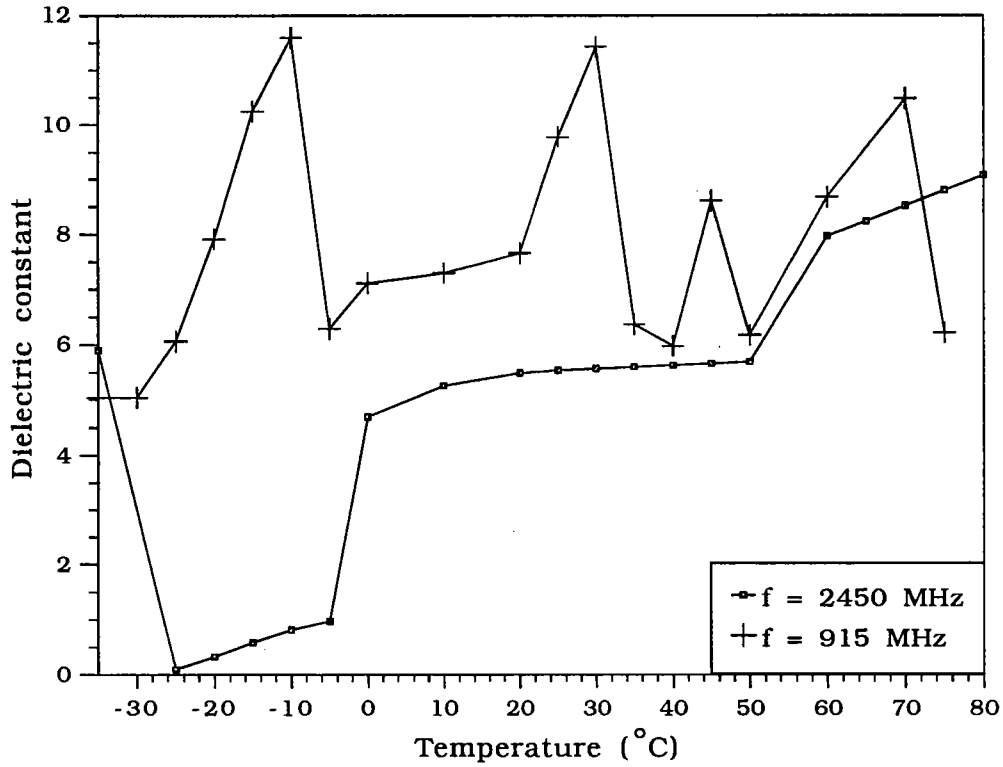

Figure 4.10 Dielectric constant vs temperature of canola oil. f = 2450 MHz and 915 MHz.

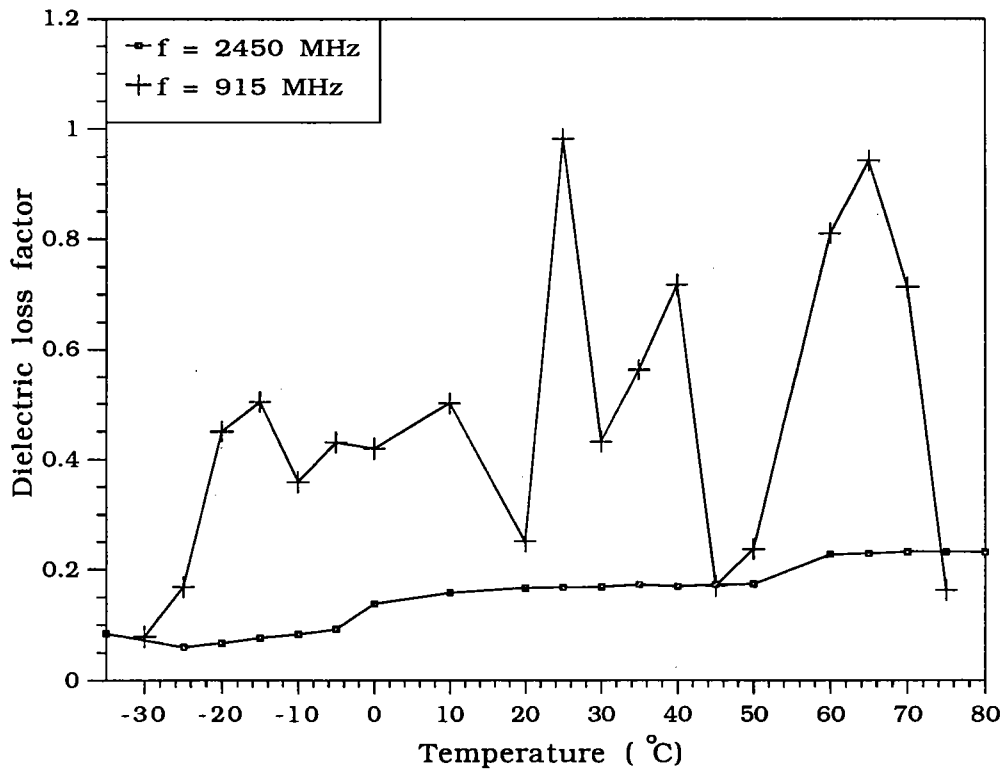

Figure 4.11 Dielectric loss factor vs temperature of canola oil. f = 2450 MHz and 915 MHz.

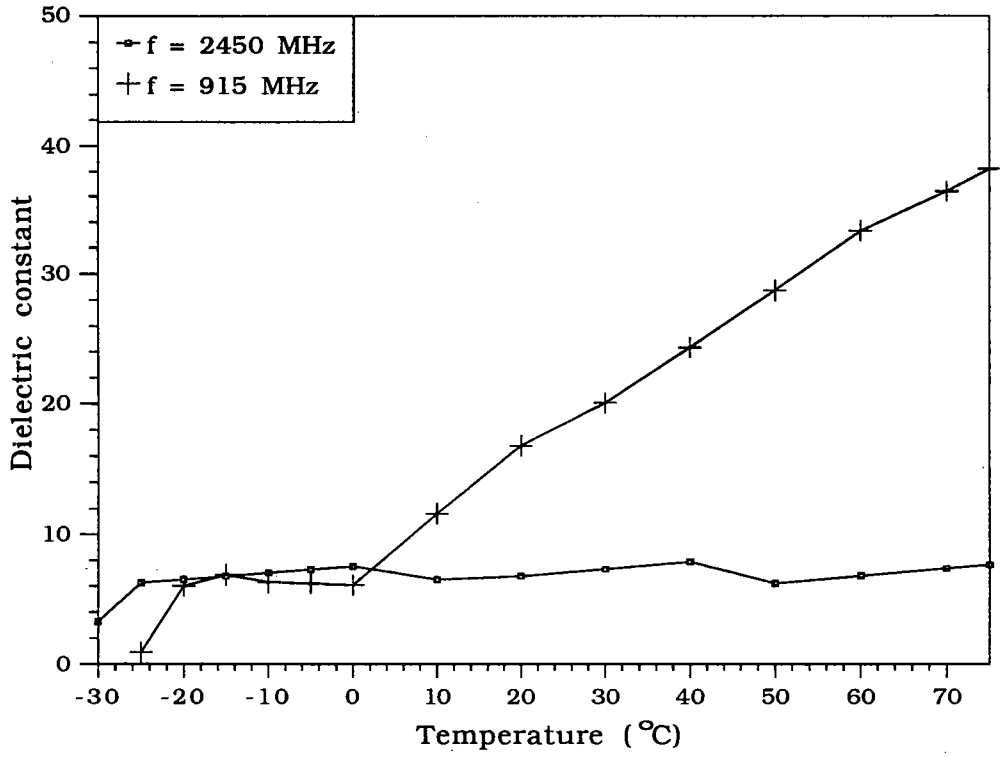

Figure 4.12 Dielectric constant vs temperature of soya oil. f = 2450 MHz and 915 MHz.

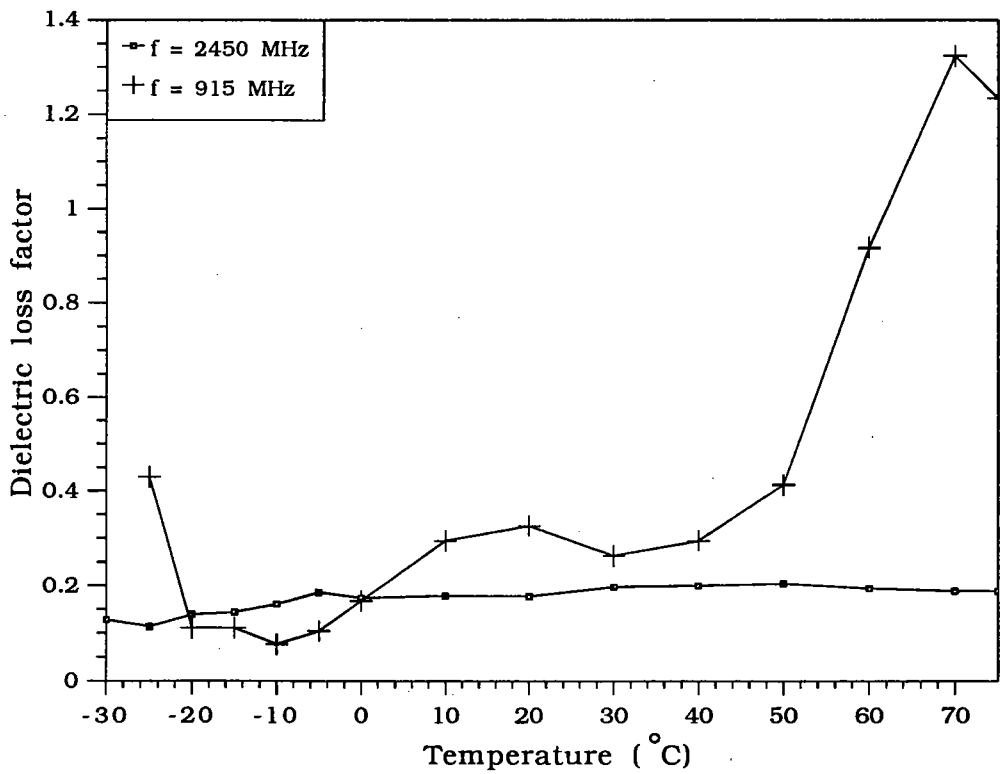

Figure 4.13 Dielectric loss factor vs temperature of soya oil. f = 2450 MHz and 915 MHz.

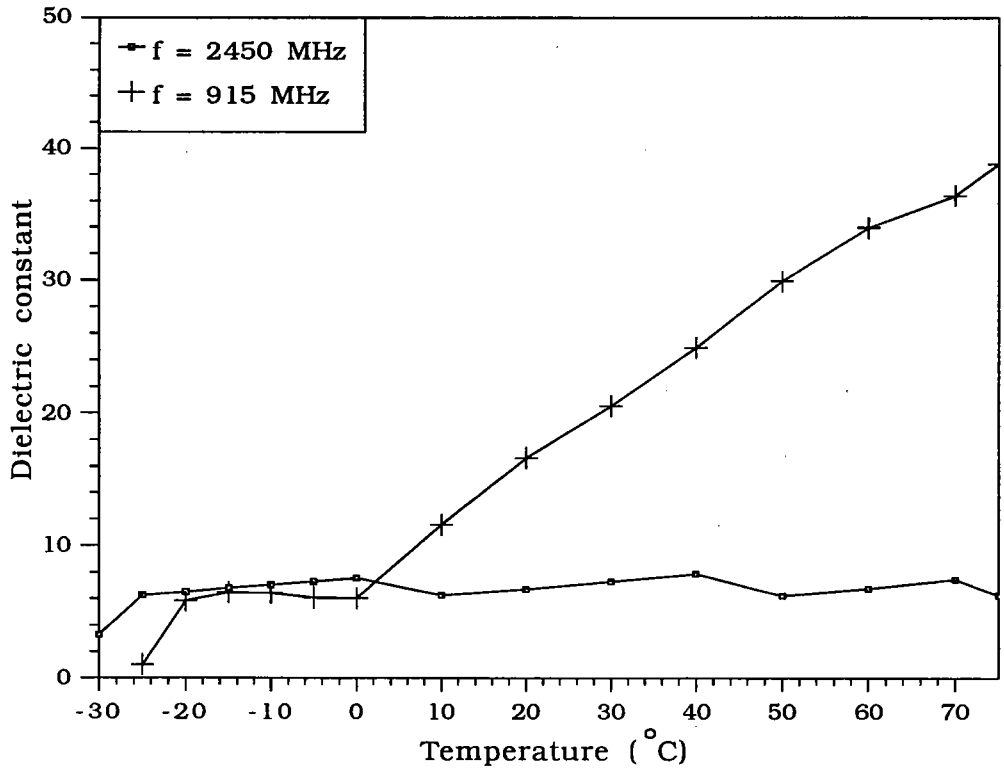

Figure 4.14 Dielectric constant vs temperature of sunflower oil at f = 2450 MHz and 915 MHz.

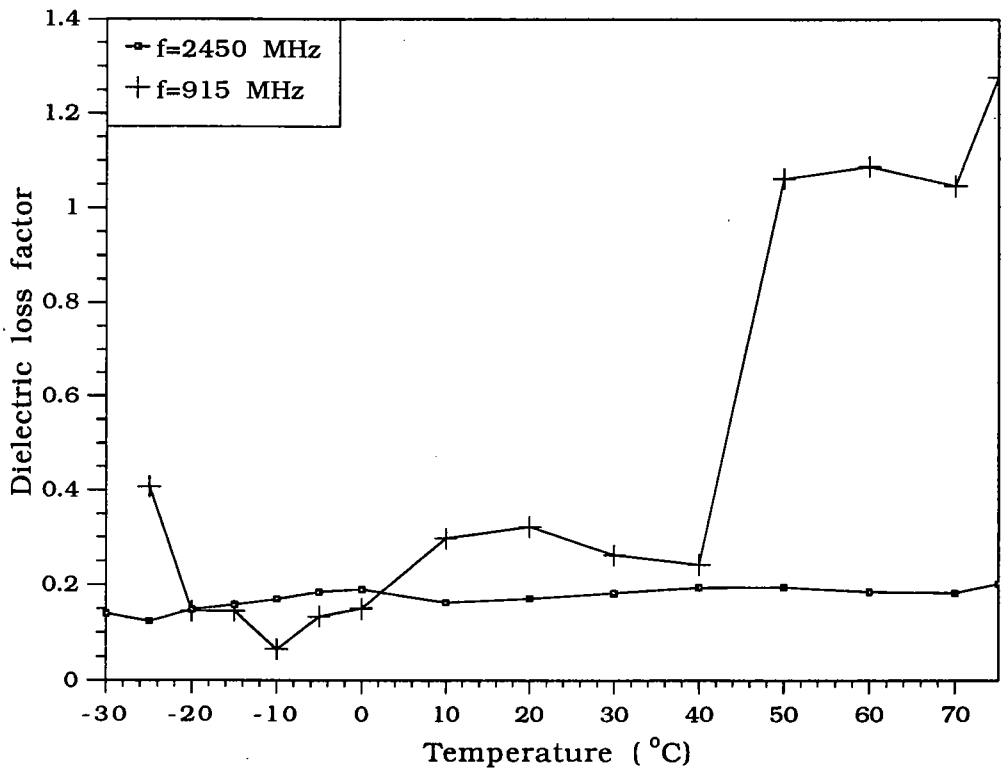

Figure 4.15 Dielectric loss factor vs temperature of sunflower oil at f = 2450 MHz and 915 MHz.

VI SUMMARY & CONCLUSIONS

A study of the dielectric properties of selected agri-food materials was carried out. The cavity perturbation technique was used to make the measurements. This technique appeared to work very well, given that the measured values gave rise to empirical regression models whose predictions closely resembled those of the theoretical models of the relationships between grain bulk density and their dielectric properties. Particle size distribution of milled grain and its connection with the dielectric properties were also studied.

The dielectric properties of a semi-solid material, tylose, were studied at various temperatures and frequencies. In order to load samples of such materials into micropipettes of 10µl, it was necessary to develop a specialized apparatus. The apparatus "micro-sampling unit" is termed and described. The dielectric properties of tylose were interesting in that they tended to be lower than those of water at temperatures below 0°C but were substantially higher at temperatures above 30°C. This is an example of the odd behaviour one can expect from materials that may be in food products, which indicates the importance of knowing the composition of foods in the design of microwave applications for the industry.

The dielectric properties of ethanol and various ethanol-hexane mixtures were determined. The mixtures behaved fairly regularly. It was clear that the ability of a mixture to absorb and react to microwaves increased with increasing ethanol concentration regardless of temperature in the range of 22°C to 62°C. These data indicate the potential of using microwaves for solvent extractions.

The behaviour of ε' and ε'' as a function of frequency and temperature was examined for the edible oils such as canola, soya and sunflower. At 915 MHz, the behaviour for canola oil was quite irregular. At this frequency, the other oils showed a trend to much higher loss factor and dielectric constant at higher temperatures. At 2450 MHz, neither soya nor

sunflower oils showed much response of dielectric properties to temperature. The dielectric properties of canola oil both exhibited a tendency to increase with temperature at 2450 MHz. Without an analysis of their chemical compositions and more detailed study of the dielectric properties of their constituents, it is difficult to further comment on the behaviour.

Conclusions :

1. Existing linear models were found to describe adequately the relationship between functions of the dielectric properties and density of chopped, powdered and whole kernel corn and wheat.
2. Using these models to eliminate the effect of density, models relating the dielectric properties to a size correlated parameter, the nominal sieve opening, were developed.
3. Further studies could be aimed at elaborating a model that would describe the dielectric properties as a function of both density and particle size. This work suggests that such model could be represented by a surface in 3-dimensions (dielectric properties, density, particle size).
4. The cavity perturbation technique is an effective tool for measuring the dielectric properties of various agri-food and biological materials with varying process parameters (frequency, temperature, moisture content, etc).
5. More detailed work on the dielectric properties of edible oils should be done. In particular, the relationships of the dielectric properties to chemical composition (eg. fatty acid distribution) should be elaborated.

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APPENDICES

APPENDIX A

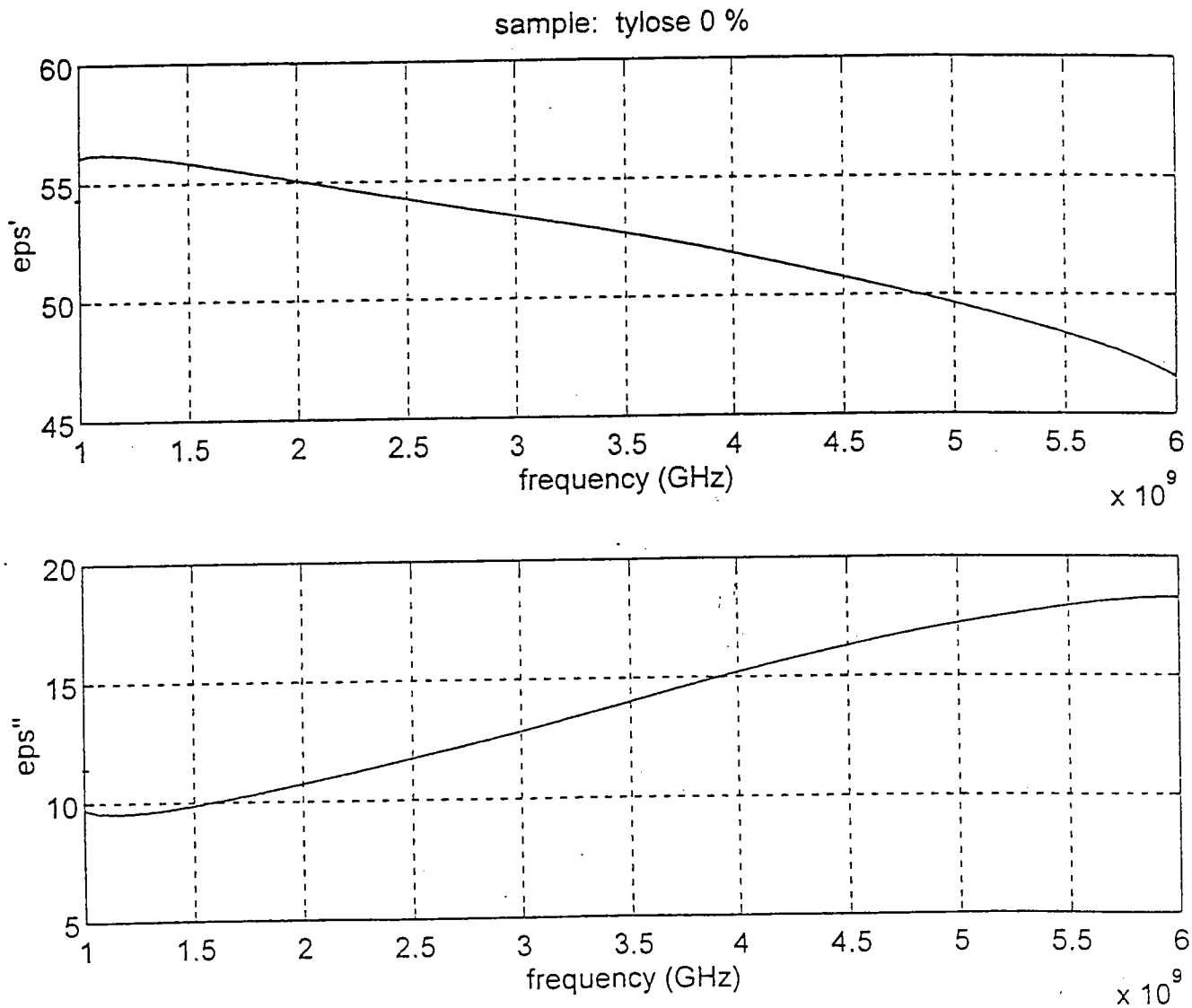


Fig A1. Dielectric properties (ε',ε") of tylose (0% salt) as a function of frequency (1 to 6 GHz), @ 22°C.

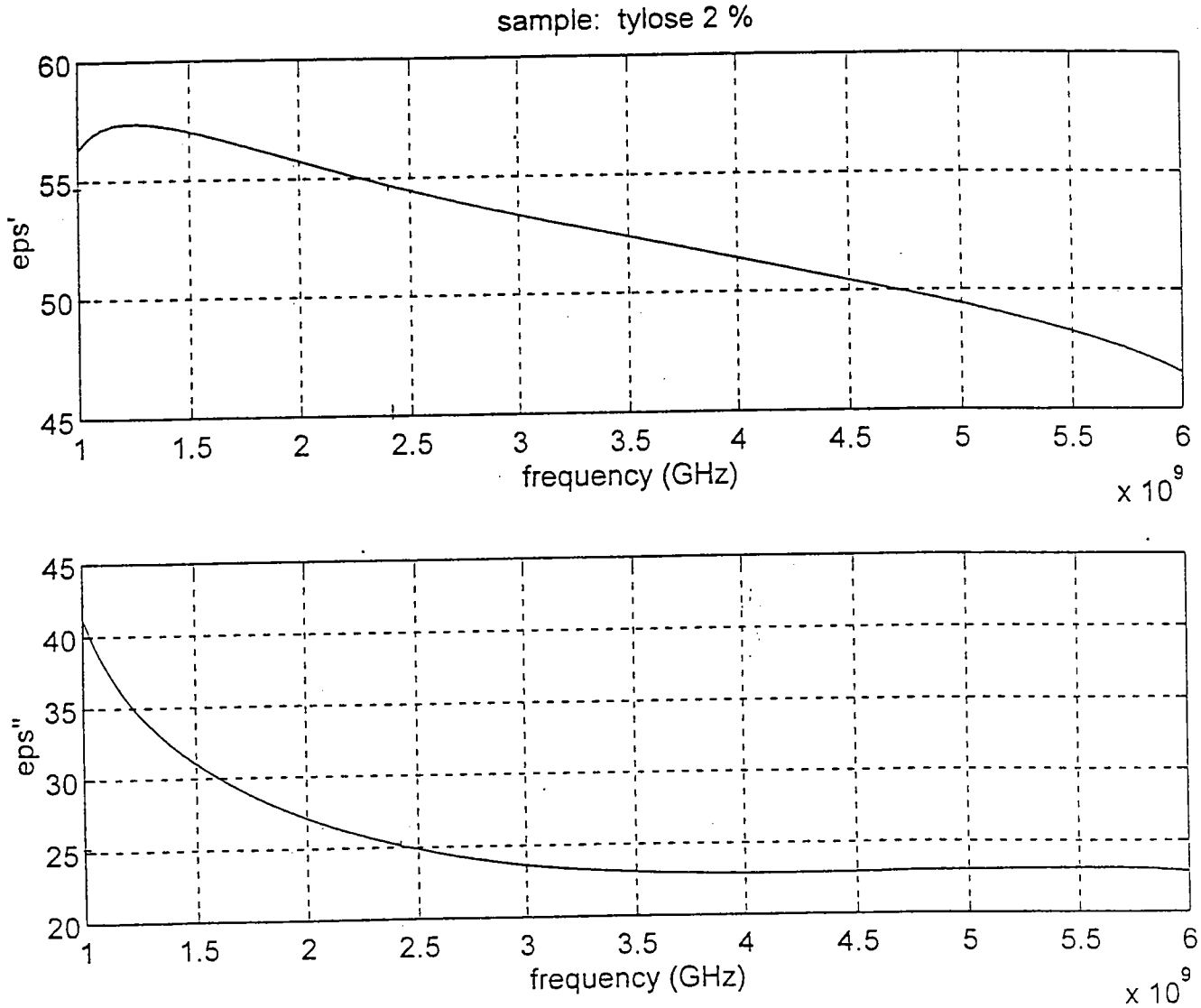


Fig A2. Dielectric properties (ε',ε") of tylose (2% salt)
as a function of frequency (1 to 6 GHz), @ 22°C.

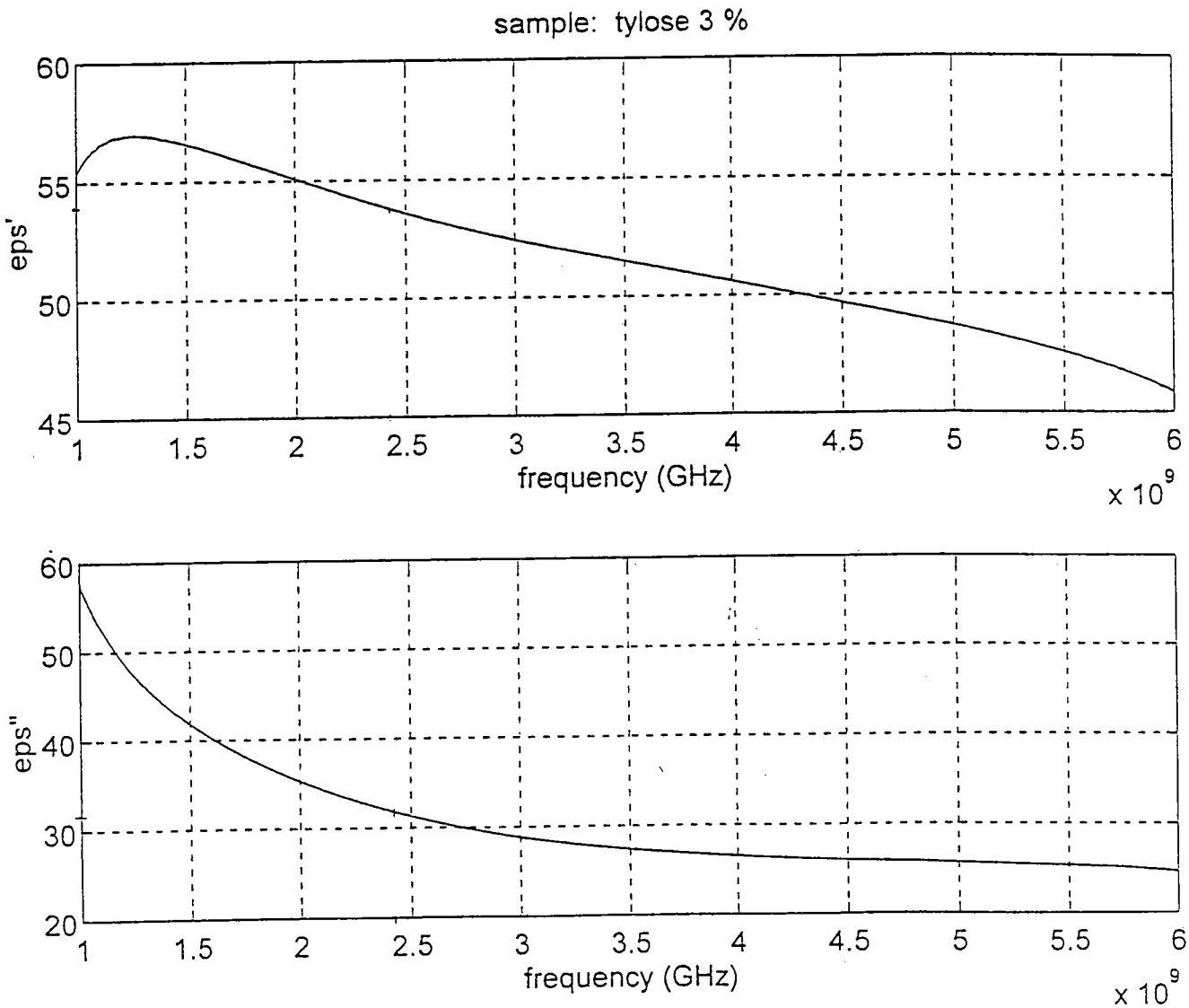


Fig A3. Dielectric properties (ε',ε") of tylose (3% salt) as a function of frequency (1 to 6 GHz), @ 22°C.

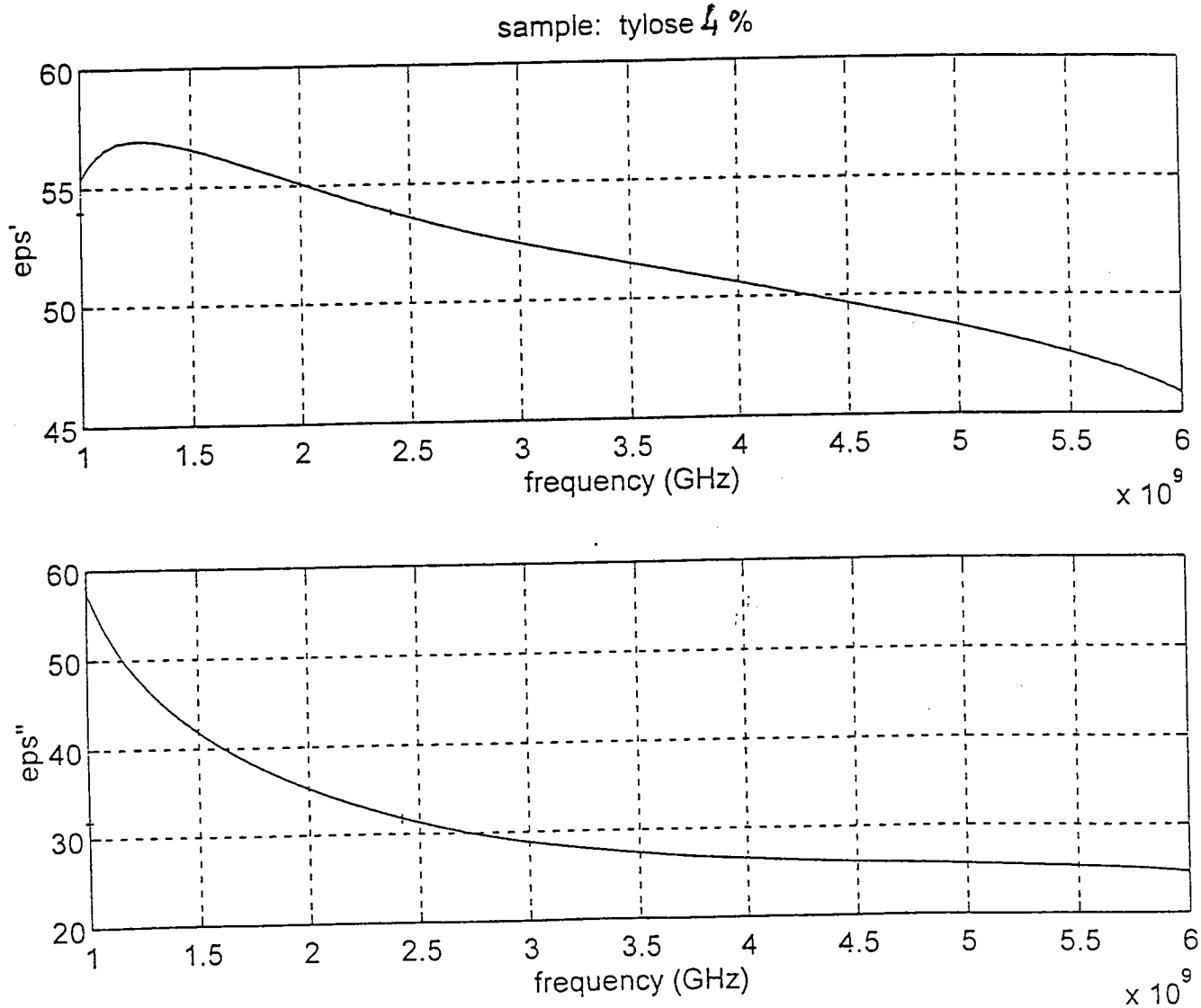


Fig A4. Dielectric properties (ε',ε") of tylose (4% salt) as a function of frequency (1 to 6 GHz), @ 22°C.

DATA TRANSLATION®

IBM Personal Computer
DIGITAL INPUT/OUTPUT BOARD
DT2817

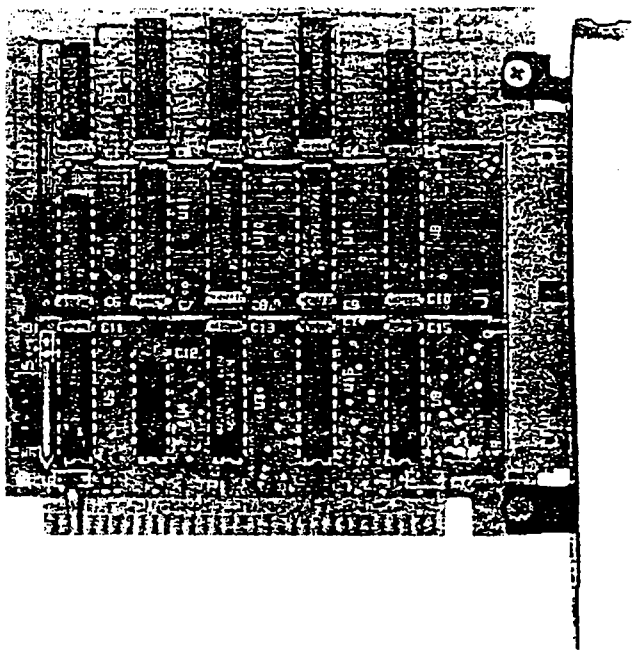
FEATURES

- Low cost, half-size IBM PC/XT/AT compatible digital I/O board
- 32 lines of input/output
- 4 8-bit ports programmable as input or output
- Capable of driving OPTO 22™ or Potter & Brumfield™ panels directly
- Shipped with comprehensive user manual
- Manual has sample BASIC programs that can be used as is or modified for user applications

DESCRIPTION

The DT2817 is a half-size, low cost digital I/O interface that gives users of the IBM PC/XT/AT access to a large number of digital signals. The DT2817 features 32 lines of digital I/O that are divided into four 8-bit ports. Each port can be software programmed for either digital inputs or outputs.

The board plugs into any I/O expansion slot for easy installation. Shipped with the DT2817 is a set of sample BASIC programs. These programs test the board to ensure proper operation and may be used as is or modified for user applications. The DT2817, along with the companion half-size DT2814 16-channel analog-to-digital and the DT2815 8-channel digital-to-analog boards make up a series of low cost, medium performance analog and digital I/O system boards for the IBM PC.



M—4310

Figure 1. The DT2817 provides low cost digital I/O for the IBM PC/XT/AT and compatibles.

OPTO 22 is a trademak of OPTO 22, Huntington Beach; Potter & Brumfield is a trademark of Potter and Brumfield Division AMF Incorporated.

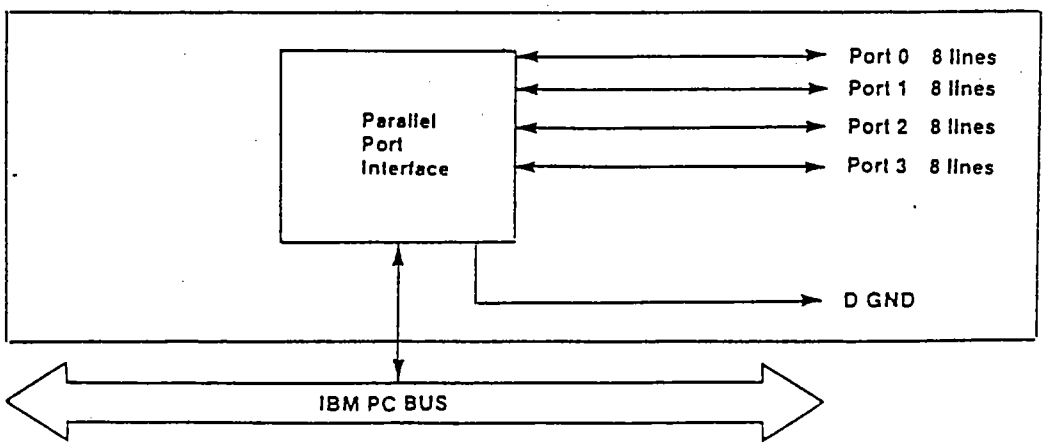
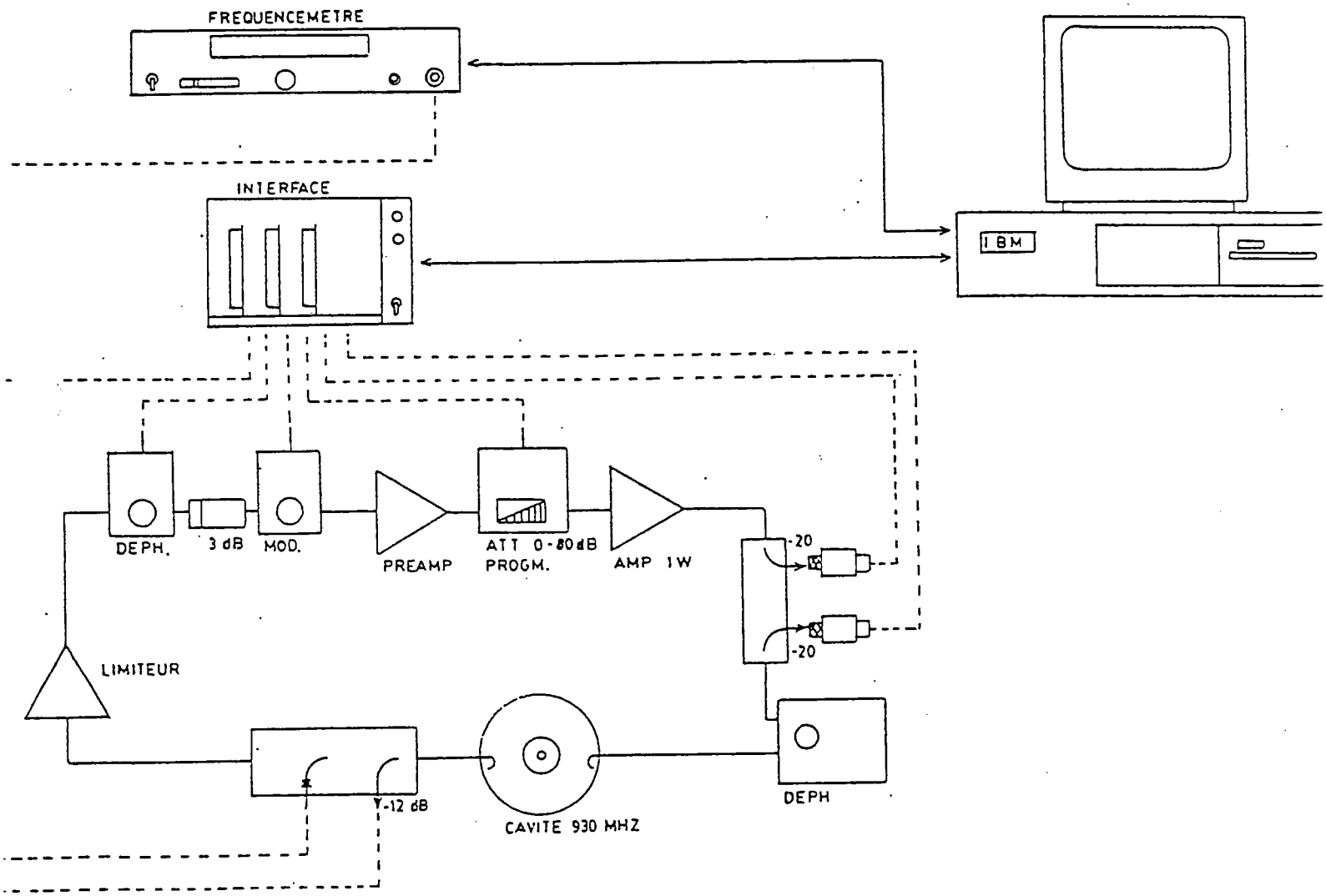


Figure 2. Functional block diagram of the DT2817.

Data Translation, Inc. 100 Locke Drive, Marlboro, MA 01752-1192 USA (617) 481-3700
Data Translation Ltd., The Mulberry Business Park, Wokingham, Berkshire RG11 2QJ, U.K. (0734) 793838

APPENDIX C

SYSTEME DE MESURE DE PERMITTIVITE A 915 Mhz



APPENDIX D

```
DECLARE SUB epsilon ()
DECLARE SUB vide ()
DECLARE SUB ech ()
DECLARE SUB stopcom ()
DECLARE SUB config ()
DECLARE SUB stimul ()
DECLARE SUB mesure ()

    ' Programme de control pour cavite avec le 8510


 COMMON SHARED /blk1/ fst, fsp, pt, rd, cav, wrt, CMD, BDNAME, FILE AS STRING
 COMMON SHARED /blk2/ FQ AS INTEGER
 COMMON SHARED /BLK3/ F0, fg, fd, bessel, fv, fs, Qv, Qs AS SINGLE
 COMMON SHARED /blk4/ fstart, fstop, fc, Vc, Vs, ate, Q AS SINGLE

 COMMON IBSTA%, IBERR%, IBCNT%          'declaration pour GPIB

     CMD$ = SPACE$(20)                  ' command buffer
   '  rd$ = SPACE$(50)                   ' read buffer
   '  wrt$ = SPACE$(100)                   ' write buffer
     BDNAME$ = SPACE$(7)                ' board/device name
     FILE$ = SPACE$(50)                 ' file name


CLS
        LOCATE 3, 3
           PRINT " Mettre  L'adresse de l'analyseur a 2 "
           PRINT
           INPUT " Mettre l'appareil en mode 'LOCAL'  "; a

           CLS
        LOCATE 3, 3

        INPUT " Quel est la frequence de resonnance de la cavite 'Ghz' "; fc
        PRINT
        INPUT " Quel est le volume de la cavite 'mm cube' "; Vc
        PRINT
11 :    INPUT " Quel est le type de cavite  'C' ou 'R' "; cav$
          cav$ = UCASE$(cav$)


          IF cav$ = "C" THEN
          bessel = .539
             GOTO 12
          END IF
          IF cav$ = "R" THEN
          bessel = .5
             GOTO 12
          ELSE
             GOTO 11
          END IF
12 :

        fstart = fc - (.2 * fc)
        fstop = fc + (.2 * fc)

    ' Initialisation de l'analyseur
```

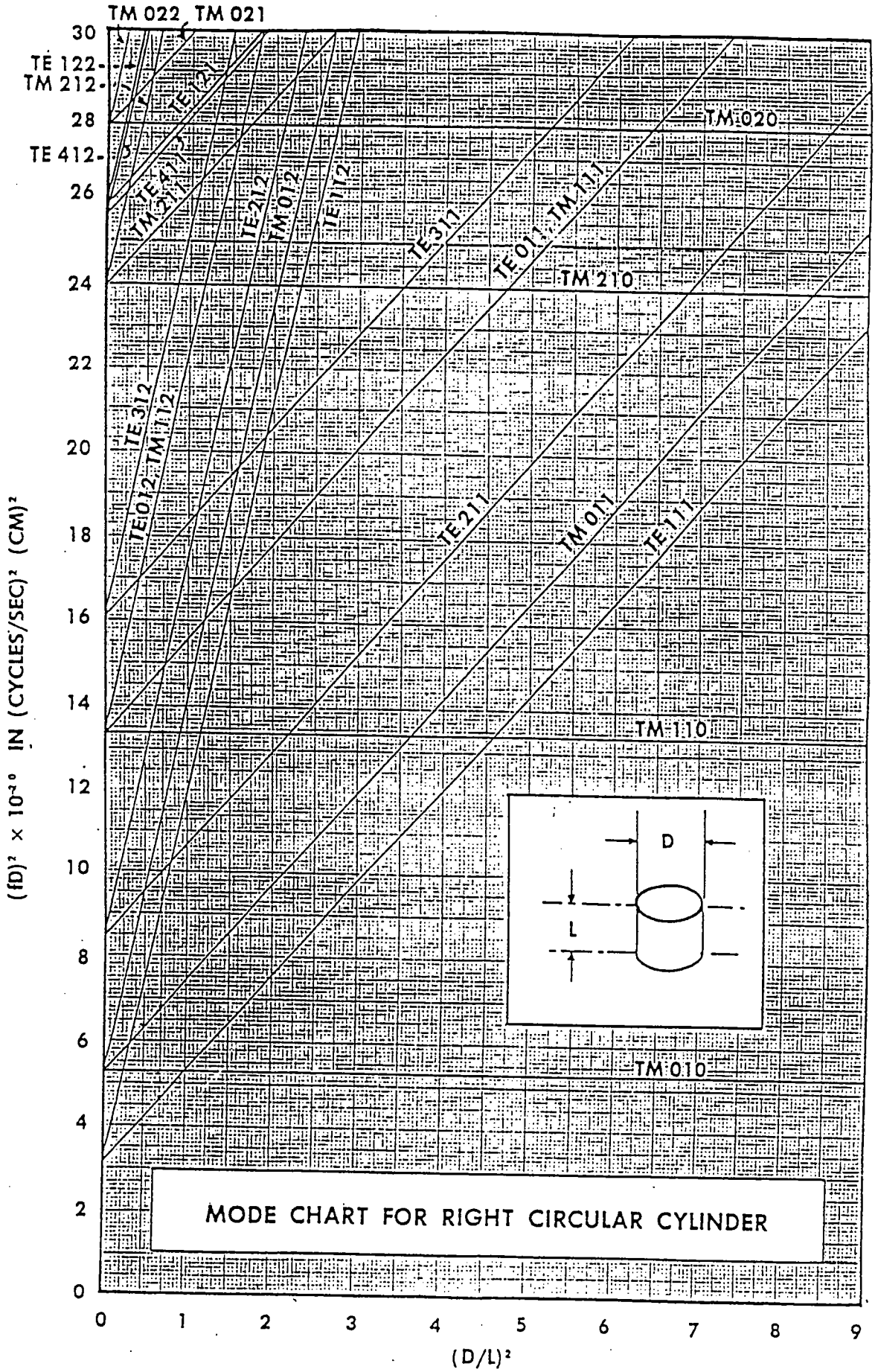
```
        CALL config             'configuration de gpig


       'Calcul de epsilon
10 :

      CALL epsilon
      BEEP
      LOCATE 20, 2
      INPUT " Voulez-vous refaire une autre mesure 'O/N' "; rep$
      IF UCASE$(rep$) = "O" THEN
          GOTO 10
          END IF

 CALL stopcom          'reset la communication
   CALL stopcom



END


SUB config STATIC

DEV$ = "GPIB0"
         CALL IBFIND(DEV$, GPIB0%)
DEV$ = "HP8753"
         CALL IBFIND(DEV$, FQ%)
         V% = &H2
         CALL IBPAD(FQ%, V%)       'address du 8510
         CALL IBCLR(FQ%)
         CALL IBSIC(GPIB0%)
         V% = 1
         CALL IBCAC(GPIB0%, V%)
         CALL IBTMO(FQ%, 13)
    '   wrt$ = "PRES;"
    '   CALL IBWRT(FQ%, wrt$)


END SUB

SUB ech STATIC


       ' cette routine fait la mesure de l'echantillon

33 :
         CALL stimul
       ate = 6
       CALL mesure
       fs = F0
       Qs = Q
        IF Qs > 10000 THEN
        GOTO 33
        END IF
```

```
END SUB

SUB epsilon STATIC


  CLS
  LOCATE 5, 5
      PRINT " CALCUL DE EPSILON "
      PRINT
      PRINT " Parametre de cavite vide "
      PRINT
      PRINT " Faire la mesure de la cavite vide "
      PRINT
      INPUT " Prepare la cavite puis 'ENTER' "; a
      CALL vide
      CLS
          BEEP
          PRINT " Cavite vide termine"

44 :
LOCATE 1, 5
     PRINT " Mesure de l'echantillon"
     PRINT
     INPUT " Mettre l'echantillon dans la cavite puis 'ENTER' "; a
     CALL ech
     BEEP
     CLS
     PRINT "Mesure de l'echantillon  termine"

        'calcul de epsilon

         deltaf = (fv - fs) / fs
          deltaQ = (1 / Qs) - (1 / Qv)
          LOCATE 6, 1
          PRINT "Qvide "; Qv
          PRINT "Qech  "; Qs


         PRINT
         INPUT " Quel est le volume du sample 'mm cube'  "; Vs


          '  PRINT bessel, deltaf, Vc, Vs
               EP1 = 1 + (bessel * Vc * deltaf / (Vs + .000001))
                   IF EP1 < 0 THEN
                          EP1 = 0
                     END IF
               EP1 = INT(EP1 * 100) / 100

               EP2 = (bessel / 2) * deltaQ * Vc / (Vs + .000001)

                    IF EP2 < 0 THEN
                          EP2 = 0
                    END IF
                    EP2 = INT(EP2 * 10000) / 10000
```

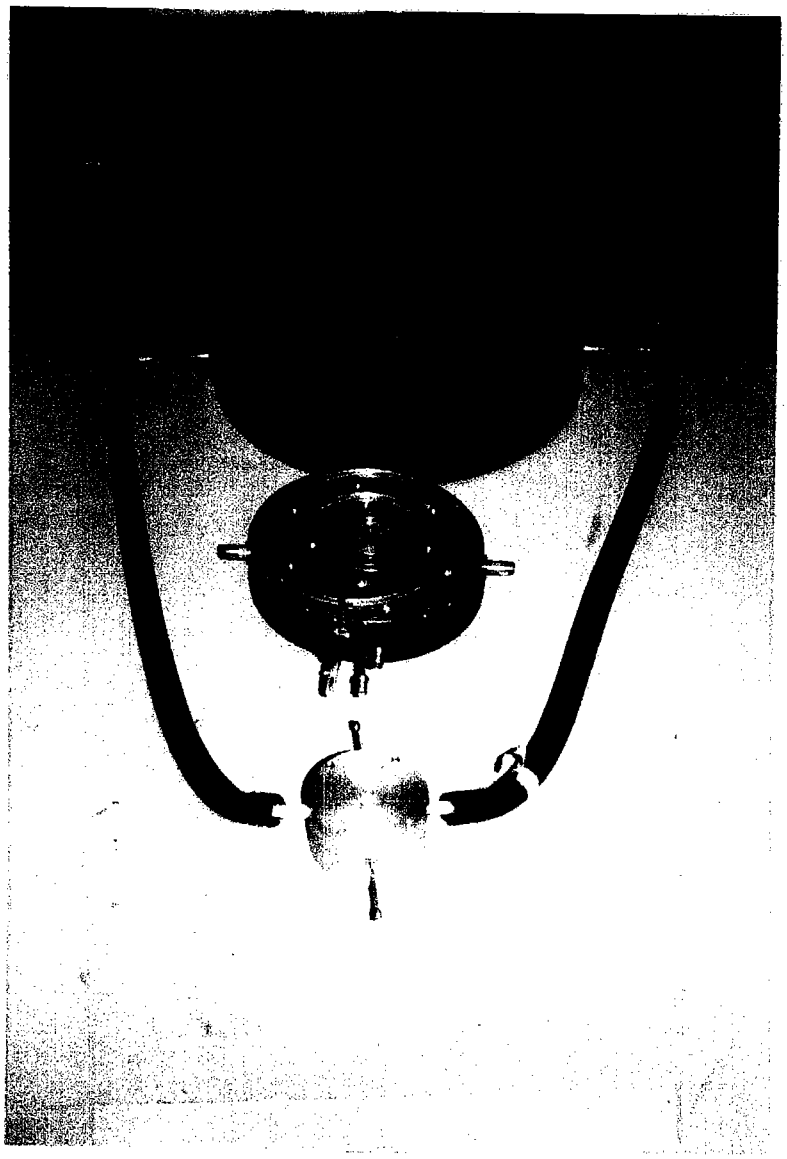
```
        LOCATE 12, 1
        PRINT "Epsilon prime  = "; EP1
        PRINT "Epsilon second = "; EP2
        PRINT "Frequence de res "; F0
        PRINT "Q vide           "; Qv
        PRINT "Q echantillon    "; Qs
     LOCATE 20, 1

      INPUT "voulez-vous refaire une mesure d'echantillon 'O/N' "; rep$
      IF UCASE$(rep$) = "O" THEN
      GOTO 44
      END IF


END SUB

SUB mesure STATIC

      rd$ = SPACE$(50)                    ' read buffer
      wrt$ = SPACE$(100)                   ' write buffer


    ' Choix du mode dans l'analyseur

            wrt$ = "CHAN1; S21; LOGM;SING;REFP 9 "
            CALL IBWRT(FQ%, wrt$)
            wrt$ = "MARK1; SEAMAX; OUTPMARK;"
    ' mesure de la frequence centrale

            CALL IBWRT(FQ%, wrt$)
            CALL IBRD(FQ%, rd$)
            F0 = VAL(RIGHT$(rd$, 19))

     ' attenuation  pour la mesure du Q
        ' PRINT rd$, F0
        ' INPUT a

            VZ = VAL(LEFT$(rd$, 14)) - ate
            ATT$ = "SEATARG" + STR$(VZ) + " DB;" + "SEAL;" + "OUTPMARK;"
            wrt$ = ATT$
            CALL IBWRT(FQ%, wrt$)
      ' Mesure de la frequence gauche

            CALL IBRD(FQ%, rd$)
            fg = VAL(RIGHT$(rd$, 19))
      ' Mesure de la frequence droite
        '  PRINT rd$, fg
            wrt$ = " SEAR; OUTPMARK;"
            CALL IBWRT(FQ%, wrt$)
            CALL IBRD(FQ%, rd$)
            fd = VAL(RIGHT$(rd$, 19))
         '   PRINT rd$, fd
        ' INPUT a
         alf = (10 ^ (ate / 10) - 1) ^ .5
         IF fg = 0 THEN
            fg = 1
            END IF
          IF fd = 0 THEN
          fd = 1
          END IF
```

```
          Q = (F0 * alf) / (fd - fg)

      '   PRINT fg, F0, fd, Q
      '   INPUT a



END SUB

SUB stimul STATIC


         ' stimulus de depart

              fst$ = STR$(fstart)
              fsp$ = STR$(fstop)
              pt$ = STR$(801)

        wrt$ = "STAR " + fst$ + " GHz;" + "STOP " + fsp$ + " GHz;"

         CALL IBWRT(FQ%, wrt$)
         wrt$ = "POIN" + pt$
         CALL IBWRT(FQ%, wrt$)


      ' trouver la bande a -10 DB

         ate = 15
           CALL mesure
           ghz = 1000000000

         fst$ = STR$(fg / ghz)
         fsp$ = STR$(fd / ghz)

            wrt$ = "STAR " + fst$ + " GHz;" + "STOP " + fsp$ + " GHz;"

            CALL IBWRT(FQ%, wrt$)
            wrt$ = "POIN" + pt$
            CALL IBWRT(FQ%, wrt$)


END SUB

SUB stopcom STATIC



V% = 1
CALL IBONL(FQ%, V%)
CALL IBONL(GPIB0%, V%)

V% = 0
CALL IBONL(FQ%, V%)
CALL IBONL(GPIB0%, V%)
```

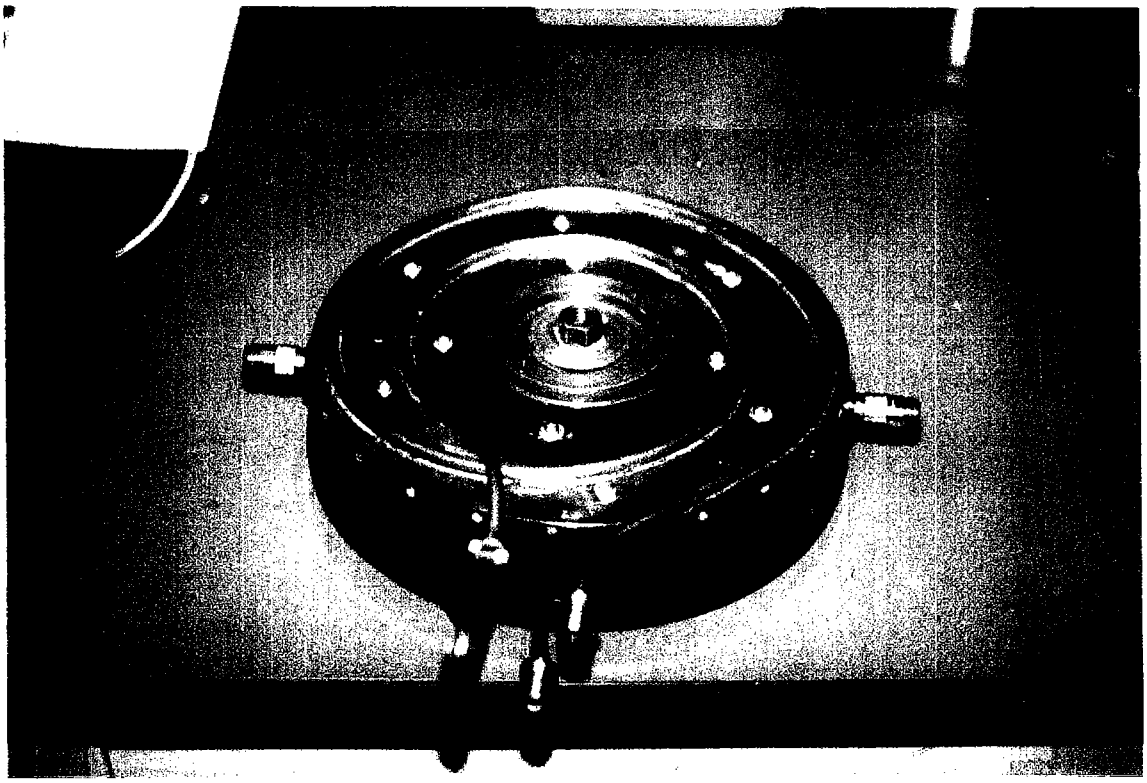
```
CALL IBLOC(FQ%)



END SUB

SUB vide STATIC

      ' cette routine fait la mesure de la cavite vide

34 :
      CALL stimul
      ate = 6
      CALL mesure
      fv = F0
      Qv = Q
      IF Qv > 10000 THEN
      GOTO 34
      END IF

END SUB
```


TM 022, TM 021
TE 122
TM 212
TE 412
TM 020
TM 210
TM 110
TM 010
TE 312
TE 212
TM 012
TE 112
TE 311
TE 011
TM 111
TE 211
TM 011
TE 111
TE 012, TM 112
TM 312, TE 412
D
L
MODE CHART FOR RIGHT CIRCULAR CYLINDER
(fD)² × 10⁻⁶ IN (CYCLES/SEC)² (CM)²
(D/L)²
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
1
3
5
7
9

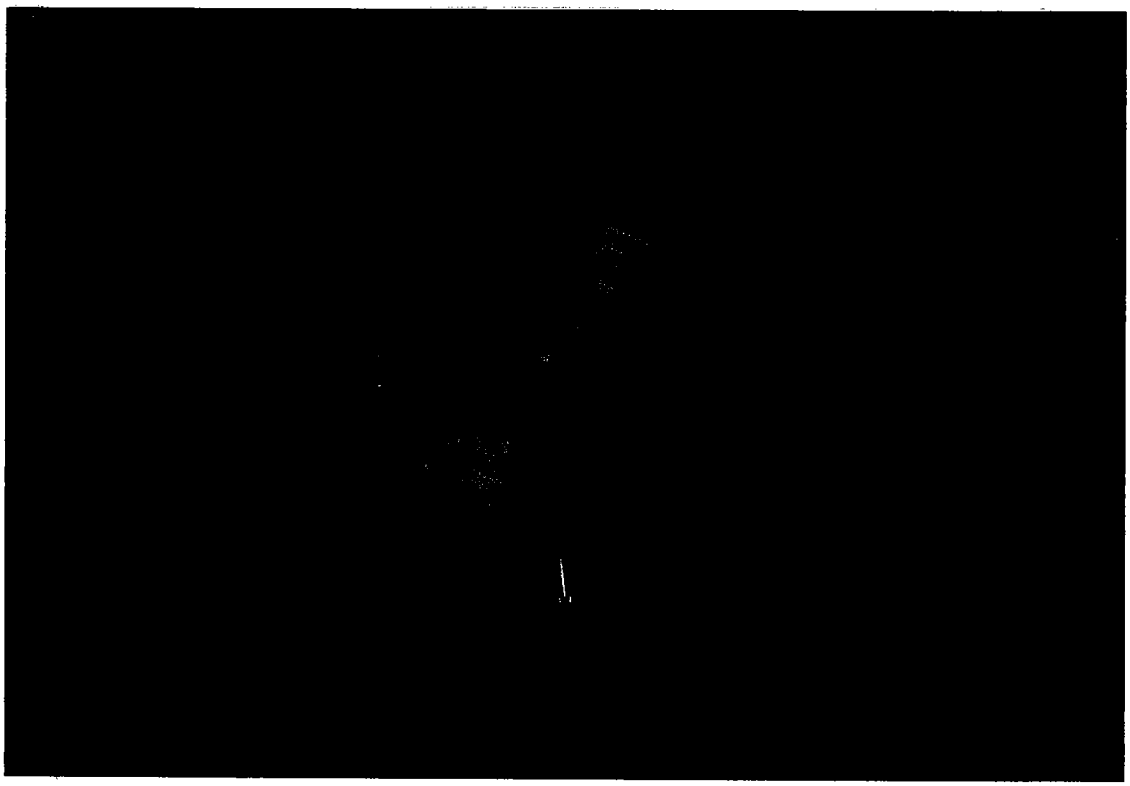
APPENDIX F



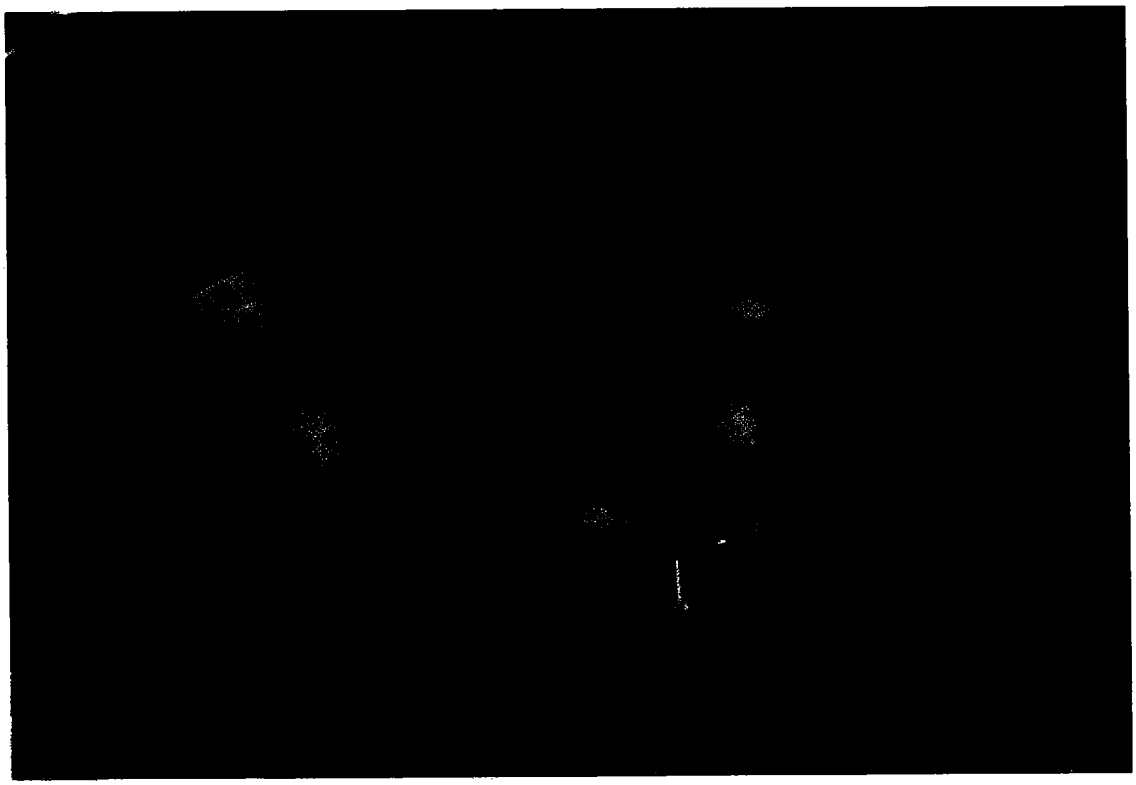
F1 Shows the 400, 915 & 2450 MHz cavities.



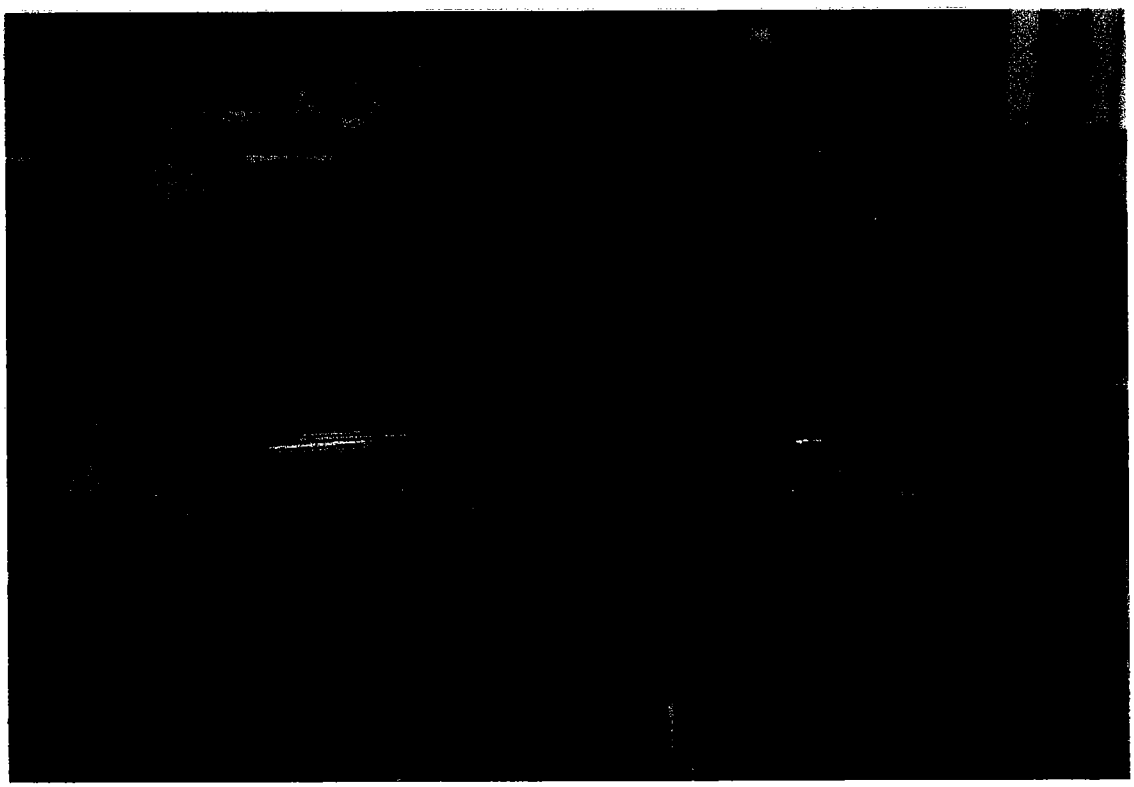
F2 A 915 MHz cavity with temperature control coils.



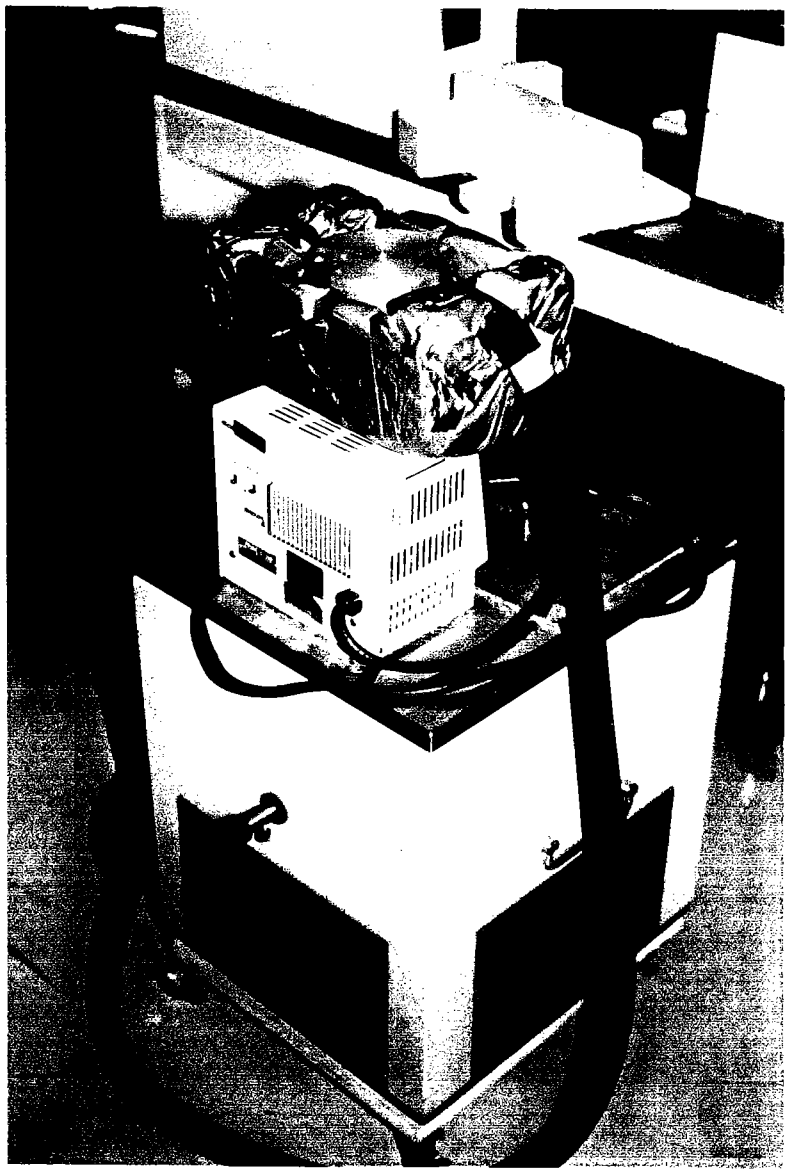
F3 Shows a 2450 MHz cavity with 2 ends for the coaxial probes.



F4 Details of a 2450 MHz cavity with a coupler.



F5 Shows an experimental set up using 915 MHz cavity.



F6 Shows an insulated 2450 MHz cavity hooked on to a temperature control bath.