DIELECTRIC PROPERTY MEASUREMENTS OF LIQUID FOOD SYSTEMS AT MICROWAVE FREQUENCIES

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ABSTRACT

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By

Kenneth Otto Weil

It has been recognized in the field of microwave food processing that there is a definite need for information concerning the dielectric properties of food materials. Such information will aid the development and design of sophisticated equipment and process systems.

This study deals with the resonant cavity perturbation technique for the measurement and calculation of the dielectric properties (dielectric constant, ε', loss factor, ε'', and loss tangent, $\tan \delta$) of liquid materials. The design and construction of the TM_{010} mode circular cylindrical cavities is discussed, along with the sample tubing used to support the liquid within the cavities and the preparation of the samples.

The dielectric properties were determined for distilled deionized water, 1.0, 1.5, and 2.0 M sodium chloride solutions, 10, 20, and 30% (by weight) aqueous solutions of dextrose, maltose, and dextrin, respectively,

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2.5, 5.0, and 10.0% (by weight) aqueous solutions of sodium caseinate, and a commercial sample of corn oil. Measurements were made at frequencies of 970, 1123, 1978, 2431, and 2973 MHz and at 28°C. The dielectric properties are reported as a function of concentration and/or frequency.

Favorable agreement is found between results using this method and literature values of dielectric properties in cases where materials have been investigated previously, usually by other methods.

There is a tendency for dielectric constants of the aqueous solutions to decrease as solute concentration is increased at given frequencies. Loss tangents tended to increase as a function of concentration and frequency.

With the sodium chloride solutions, the effect of conductivity on the loss tangent is noted. Based on the findings of the work with corn oil, it was concluded that the method would have to be altered slightly when measuring dielectric materials of low loss.

DIELECTRIC PROPERTY MEASUREMENTS OF LIQUID FOOD SYSTEMS AT MICROWAVE FREQUENCIES

By

Kenneth Otto Weil

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Figure Page

INTRODUCTION

The use of microwaves as a means of cooking or otherwise processing foods is a relatively new concept, and while it has yet to take the industry "by storm," it has many aspects that make it promising and desirable for both home and industrial applications. But, as is true of all ideas in their first stages of infancy, much information and methodology remains to be explored and developed before its full potential can be realized.

The very first attempts to adapt microwave energy to food processing were conducted on a trial and error basis (Morse and Revercomb, 1949), and proved both unsatisfactory and unscientific in that no systematic information could be gathered in this way. A better approach is to study the electrical properties of foods, particularly the dielectric properties described by loss tangents and dielectric constants. Pace (1967) followed this procedure in studies on fats, oils and potato chips, as did Van Dyke (1968) in working with ground beef. Such research may not be complete in itself, but it may indicate how a specific food will behave in the microwave equipment being used.

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Such information may then lead to the "tailoring" of a food product to the equipment available--again through the use of known dielectric properties of the basic constituents of the food. Both of these latter procedures, along with the ensuing data, could result in still another possible approach. Should present equipment and frequencies not be optimal for a food product, even with manipulation of its composition, it might be worthwhile to develop microwave equipment that would operate at frequencies other than those now used, or at least to design equipment which would optimize conditions for those frequencies that are available.

Whatever approach is taken (beyond the ineptness of "trial and error"), it is apparent that dielectric data for the product in question are needed. And since engineers make valuable use of dielectric data in the design and operation of microwave equipment and components, it is only good sense that the food scientist who is looking to the possibilities of microwave processing should also make use of such data to best design the food to the system, or possibly even the system to the food. It was with the thought of these possibilities in mind, that the data herein were gathered and examined.

LITERATURE REVIEW

A History of the Development of Microwaves

The discovery of microwaves is not recent, nor is their application limited to the food industry. Indeed, quite the opposite is true. Aref (1968) reports that during the past few decades the development of tools to generate, detect, and measure microwaves has allowed their use to expand into many areas of our daily life. The guiding of aircraft through fog and snow, the transmission of telephone conversations, the telemetering of information from data-gathering satellites, the carrying of trans-oceanic television signals--these are all applications of microwave technology. Badger (1970) writes that it was over one hundred years ago that James Clerk Maxwell, whose equations are used daily by design engineers, first predicted the existence of the electromagnetic waves that include microwaves. "Twenty years later, microwaves were generated for the first time by Hertz, who carried out some very ingenious experiments resulting in electromagnetic propagation over a distance of several feet." Both Pace (1967) and Olsen (1968) refer to Professor d'Arsonval's ensuing studies, at the College de France, which centered around the biological effects of high frequency electromagnetic fields.

According to Badger (1970), however, it was Hertz's experiments that

> caught the imagination of Marconi, who envisioned these electromagnetic waves as a means for communicating with ships at sea. At the turn of the century, he discovered that the longer radio waves would travel for great distances; this discovery marked the beginning of the valuable technology of radio. Microwaves (the very short waves) were forgotten in the rush to make practical, worldwide communications systems.

It was not until the 1940's with the Second World War and the consequent growth of the radar industry, that any real headway was made in the use of microwaves. And still another decade was to elapse before the first crude, but feasible, microwave oven was to be developed. Proctor and Goldblith (1953) reported that the radio frequency generators available prior to World War II and shortly after produced energy at frequencies of 20 to 150 MHz (1 MHz = 10^6 Hz, where 1 Hz = 1 cycle/second). In discussing the use of these frequencies, they cited problems due to electrical arcing which could be overcome at such frequencies only by lowering the electric field strength that was applied. Any lowering of electric field, however, requires a corresponding increase in frequency if the initial power level is to be maintained. This results from the fact that there is a direct relation of the electric field and frequency to power obtained, and it is the power that is desired to bring about heating in a reasonable length of time. But equipment and techniques capable of increasing power tube frequencies were not available at that time.

With the advent of radar came the use of the word "microwaves" (referring to frequencies above 300 MHz) as microwaves are the basis for radar. During the war, military installations used radar which was generated by tubes called magnetrons which had been developed by Boot and Randall (Badger, 1970). The magnetron utilizes the process of interaction between an electron beam and an electromagnetic field (in the presence of a strong static magnetic field) to generate microwave energy. Kinetic energy is given up by the beam electrons to the electromagnetic field. They were the first sources capable of producing high power microwave energy, generating frequencies of about 0.9 to 3 GHz (1 GHz = 10^9 Hz), as contrasted to the common electric power frequency of 60 Hz. The klystron, developed by the Varians during the same period, accomplished a similar task (Badger, 1970); again beam-EM wave interactions are exploited but without the need for strong static magnetic fields. These higher frequencies eliminated the earlier problems of arcing. With this problem overcome, and with peace finally becoming a reality, the interest in microwaves gained greater impetus in domestic industry.

Recognition of Microwave Processing by the Food Industry

The past two decades, in particular, have seen a fascination and excitement among food processors over the use of microwaves for the heating of food. The very characteristics of microwave energy have made it unlike any previous form of heating, be it a wood fire, gas flame, or electricity, as all these generate heat which must penetrate the product from the outside to the interior of the mass and each surrounds the food with solids, liquids, or gases which are at a much higher temperature than the cooking temperature of the food material. Microwaves, however, as Aref (1968) explains, are like light waves in that they

> pass through some materials and are reflected or absorbed by others. When they pass through material such as food, the molecules within the material attempt to align themselves with the direction of the electric field and oscillate around their axes. This oscillation creates intramolecular friction which produces heat. It is therefore often said that in microwave heating of foods, the food itself is the source of heating.
>
> Different materials have different tendencies for the intramolecular motion, and those exhibiting the motion more are termed "lossy." The degree of lossiness varies with frequency, temperature, and the nature of the material. Heat production, nevertheless, is almost instantaneous and takes place uniformly throughout the food particle or item. The limitations of conventional heating by conduction or convection, and the possibility of surface crust formation, are thus eliminated.

The basic advantages of microwave heating are best summarized as: (1) rapid heating; (2) a generally uniform heating throughout the mass; (3) selective heating of water and other lossy components; and (4) the ability to quickly control the microwave power incident upon (or applied to) the material being heated, that is, it can be turned on and off at the flick of a switch. Thus, microwave processing has presented new concepts in heating, such as the ability to heat a food while it is surrounded by cold air.

It should also be mentioned that as for the effect of microwaves on the nutritive value of foods, studies thus far have been few and the results relatively inconclusive (Anon., 1969b), though Goldblith, Tannenbaum and Wang (1968), and Harvey (1963) indicate that some work has shown microwave cooking to give better retention of vitamins.

The commercial market, particularly the vending and restaurant businesses, was the first to make use of microwave power in relation to food. In the late 40's a few venturesome restauranteurs were making use of the first cumbersome and complex microwave ovens. High price and low reliability held back growth through the 50's but the development of a compact one-kilowatt unit, operating on 110-volt power, and with a list price under $1000 caused the vending market to expand dramatically in mid-1965 (Moore, 1966). It has also been noted that the United States Army Natick Laboratories are developing a SPEED Field Feeding System utilizing microwave ovens (Dungan and Fox, 1969).

The market for microwave ovens for home use is still in its infancy, though the introduction of a browning unit by Tappan and a stronger emphasis on marketing may alter this picture considerably. It has been predicted that the growth of this market will reach one hundred million dollars by 1975 (Schiffman, 1970).

Present and Potential Industrial Applications

It is with the industrial market that some of the greatest potentials are yet to be explored. Among those applications that are presently in either experimental or developmental stages are cooking, baking, heating, blanching, thawing, pasteurization, sterilization, puffing, and drying, along with various combinations of these operations. One of the largest industrial systems to date is involved with the microwave pre-cooking of chicken. It consists of a cutting line, two conveyorized microwave cookers, chillers, bread and batter equipment, and a continuous freezer. The cooking is done while the chicken is bathed in a steam atmosphere, this procedure serving the two-fold purpose of preventing loss of both natural juices and moisture through evaporation as well as supplying a source of heat for heating the surface of the product while the microwave energy cooks the interior of the meat. The resultant product is preferable to those cooked by conventional methods in that it is more flavorful, juicier, and more tender. Yields also tend to be about 5% greater and, since microwaves penetrate into the product and the product heats very rapidly, bloody bone problems are non-existent (Olsen, 1968). Schiffman (1970) reports that "present installations utilizing this process are capable of producing as much as 3,000 pounds of precooked chicken per hour," and "the manufacturers of this equipment claim much reduced production costs in spite of the initial high capital investments, thereby allowing for very rapid payback for the system." Hartman (1969), Thamer, Town, and Robe (1971), and Anon. (1969a) have also reported most favorably on microwaves for the pre-cooking of chicken.

Another industry that has previously made extensive use of microwaves is the potato chip industry. The practice of finish drying by microwave heating evolved during the mid-1960's (Schiffman, 1970) and solved a substantial number of the potato chipper's problems, ranging from color control of the final product to raw potato purchase and storage costs. Olsen (1968) has asserted that

> more total kilowatts of microwave energy are devoted to drying potato chips than any other product. Their use allows the production of chips of saleable color, when the producer is forced to use storage or other potatoes of high sugar content. During the latter stage of the oil fry process, the temperature of the potato slice will climb to a point where a browning action takes place if high sugar is present. If the fry process is interrupted when color is optimum, the chips are still too high in moisture for the desired crispness. By passing these wet chips through a microwave drier, they can be dried to a point of

> crispness with little or no additional rise in temperature and therefore little or no darkening in color.

Smith (1966) and Anon. (1969a) concur on these benefits of the microwave processing of potato chips and, in addition, Smith stated in his earliest articles that taste acceptability and shelf life were as good as, or better, than those that were finished fried, and then prophesied that "microwave processing of potato chips is here to stay."

Decareau (1970) has noted that "studies at microwave frequency have (also) shown promise for the baking of items such as bread and pastries," and Russo (1971) cites the fact that there are already six microwave proofers presently operating in the production of yeast-raised doughnuts, and that by the end of 1971 fifteen different bakeries will have them. "Proof-baking" is generally used to refer to the microwave baking of doughs, as much proofing occurs concurrently with the baking process. The conventional form of dough proofing is dependent upon "the slow conduction of heat in a high-temperature proof box, whereas microwave proofing heats the dough very rapidly and with great uniformity (Schiffman, 1970). The new process accomplishes in four minutes what it took the conventional process 20 to 40 minutes to accomplish. The resulting advantages include not only significant savings in floor space, manpower, and capital investment, but greater ease of sanitation, better product uniformity, and a lower operating cost as well. Olsen

(1968) has also noted improved texture and flavor, the retention of odiferous volatiles, and the ability to bake a product directly in its plastic or paper container.

Blanching to inactivate the enzyme systems in fresh vegetables has also been explored as a microwave process.

> Since, in order to inactivate the enzymes, one would like to raise the temperature of the products as quickly as possible, and also because many of the enzymes lie deep within the tissues of the product the deep rapid penetrating action of microwaves make it ideal for this purpose. Experiments on microwave enzyme deactivation of corn-on-the-cob, for example, have been very successful, whereas the conventional steam or hot water blanching prior to freezing yields a product of low quality. (Schiffman, 1970)

Huxsoll, Dietrich and Morgan (1970) agree there are advantages to using microwaves for the blanching of corn-on-the-cob, but have difficulty justifying the cost factor.

Studies concerned with the blanching of Brussels sprouts (Dietrich, Huxsoll, and Guadagni, 1970) noted that sequences combining microwave and water blanching resulted in better chlorophyll and color retention than conventional methods. Further, Collins and McCarty's (1968) work with the blanching of whole potatoes led them to conclude that "results from this investigation indicate that microwave energy could be utilized to shorten the blanching period now employed." Successful studies have also been conducted on the blanching of various vegetables after they have been packaged in plastic pouches (Anon., 1963).

The first commercial installations for the microwave pre-cooking of bacon have passed the stages of experimentation and are now underway. Microwave heating in this field "gives good products with no curling or burning while maintaining high yields" (Schiffman, 1970). Studies of the microwave processing of wieners (Watanabe and Tape, 1969) and pasteurization of cured hams (Bengtsson, del Valle, and Green, 1969) also indicate promise for future development.

Microwave energy appreciably shortens pasteurization and sterilization times as well, and both Schiffmann (1970) and Olsen (1968) consider it especially effective for destroying fungi or molds, as might develop in baked goods. Work is also being done in the microwave pasteurization of seafood, potato-waste water (Hamid, Boerner, and Tong, 1970), raw milk (Hamid, Boulanger, Tong, Gallop, and Periera, 1969) and the sterilization of jams (Garrick, 1967).

Until recently, thawing and defrosting were much less feasible applications of microwave energy (Decareau, 1970), because of the quite different dielectric properties of ice and water. While liquid water heats well in a microwave field, ice being much less absorptive, does not. A new technique developed by Varian Associates, however, allows the microwaves to pass through the surface ice layer and heat the interior (Schiffman, 1970), and successful applications to meat, fish, fruits, and eggs have resulted. The microwave thawing process has also been used successfully with frozen cherries (Weil, et al., 1970).

Microwaves have also been studied for various other uses such as the finish-drying of rice and partial drying of potatoes (Aref, Brach, and Tape, 1969), the dehydration of applies (Nury and Salunkhe, 1968), and the opening of oysters (Learson and Stone, 1969). Schiffman (1970) also lists freeze-drying, the pre-heating of almonds, and the puffing of such vegetables and fruits as apples, carrots, potatoes, and red beets as applications where microwave energy can be applied to food processing.

Microwave energy, however, is not without its shortcomings, the most imposing of which as Huxsoll and Morgan (1968) note, in several instances, is cost. There are exceptions where overall cost may actually be reduced, as seen with poultry and potato chips; and where the advantages substantially increase quality and saleability, any additional investment is, of course, well justified. Nor is cost the only deterrent for the expansion of microwave usage. A lack of data concerning frequencies and dielectric constants has been observed by several researchers. In the study on cured hams

(Bengtsson, del Valle, and Green, 1969), it was noted that the "highest power efficiency that could be obtained with salted ham in the generator-electrode combination used was 25%, while 50-60% would be required in an industrial application" (for economic reasons), and that "with knowledge of the dielectric properties of the material to be heated, this could be obtained by proper design of the generator." Allan (1968) makes a similar request for such information as does Decareau (1965). Aref, Brach, and Tape (1967) urge the need "for information on dielectric properties of foods at the industrial microwave frequencies used at present." DeLoor and Meijboom (1966) further extol the virtues of using model systems for gathering such information. But it is Goldblith and Pace (1967) who state the problem especially well when they conclude that "studies are needed on the importance of frequency of microwaves, the effect of temperature and composition of the foodstuff and finally on the interrelation of these variables." They further suggest that "if the microwave process industry is to grow, meaningful research must be done on the responses of foods and their components to microwaves."

Microwave and Material Interaction

The need for knowledge of microwave-material interaction having been demonstrated, Tinga (1970) cites several instances where such knowledge can be applied for effective usage of microwave power. One instance involves the fact that the high heating rate which has been advantageous for the industrial use of microwaves has limitations that are often related to the physical properties (specific heat, conductivity, etc.) of the material being heated. A knowledge of these properties may lead to the determination, or at least estimation, of heating rates.

The depth of penetration of microwaves is another such area which affects the microwave process. Tinga (1970) explains this as the phenomenon that occurs because

> as microwave energy enters a material the energy density at the surface is usually greatest and this energy density falls off exponentially because as the microwaves penetrate deeper into the material they lose more and more of their energy to the material. Penetration depth is defined as the depth from the material surface where the energy density is 1/e or 36.8% of the energy density at the surface. This is an arbitrary figure but it is a measure of the ability of microwave energy at a specified frequency to penetrate a material and to provide useful energy at some point below the surface. Penetration depth (as well as heating rate), varies with frequency and again depends on certain physical properties of the material.

As these properties are discussed in turn, penetration depth and heating rate will again be dealt with.

Still another area in which knowledge of the microwave properties of materials would be useful involves the stability of the heating process. By stability is meant the change of heating rate with temperature and moisture content of the material.

Classes of Materials

In any discussion of a microwave process or system, there are three basic classes of materials to be considered, conductors which reflect (or are impenetrable to) microwaves, insulators which reflect and transmit microwaves but which absorb only negligible amounts of power, and dielectrics which have properties lying between those of conductors and insulators. As Tinga (1970) states, the materials to be treated are of the latter class and absorb microwave energy in varying degrees. "Such materials," he goes on,

> are often referred to as lossy dielectrics. Since most process materials can broadly be classified as dielectrics, it now becomes clear why one often hears the term dielectric properties, dielectric loss factors, dielectric loss tangent, in connection with microwave power. The dielectric properties of the process material (whether they are known or not), in the final analysis determine whether or not the material can suitably be treated with high frequency energy or microwave energy.

Mechanism of Interaction with Dielectrics

Several researchers, including Badger (1970), Proctor and Goldblith (1953), Harper and Tappel (1957), Pace (1967), and Van Dyke (1968), have described the mechanism of the microwave heating of dielectric materials. As Badger explains it, metallic substances are characterized by free electrons and when an electric current is applied a net drift of electrons results, thus creating an electric current. Insulators, on the other hand, have few free electrons, and little current flows when the electric field is applied. In the majority of dielectrics, the molecules may or may not have a net charge, but their charges are often distributed asymmetrically, as are those of insulators. Most molecules are therefore polar.

Badger cites water, with its asymmetric configuration of hydrogens and oxygen, as an example of a polar molecule. Proctor and Goldblith (1953) assert that each of the O-H bonds in the water molecule is polar because oxygen is more electronegative than hydrogen, and the two O-H bond polarities add up to give a net polarity to the molecule as a whole. The amount of polarity of the molecule depends not only on the difference in the electronegativity of the atoms, but also on the angle between the bonds. The bond angle for water is 105°; if it were larger, the polarity of the water would be smaller. That is, if the bond angle were 180°, as is true in CO_2, then the water molecule would be non-polar since the electronegativity of the two O-H bonds would cancel each other.

If a volume of water is placed between two metallic plates which are connected to a switch and battery in a circuit, and the switch is open, the water molecules are randomly oriented. When the switch is closed, however, and a strong electric field is applied, the polar molecules tend to align themselves with the electric field causing net polarization of the mass. "This degree of order within the volume of matter is a form of potential energy. When the switch is opened, the field is relaxed. The molecules revert to random orientation, and the stored potential energy is converted to heat" (Badger, 1970). The return to random orientation, according to Proctor and Goldblith (1953), is caused by such factors as brownian movement, thermal agitation, and convection currents in the water. The quantity of heat obtained in a single open-close operation is small, but with extended repetition it is additive and finally large enough to be useful.

This phenomenon can be described by the relationship $P \alpha E^2 fK$, where P is the absorbed power, E is the electric field, f is the frequency of relaxation (defined as $f = 1/\lambda$ and λ is relaxation time, the time required for random orientation to reoccur), and K is the loss factor of the material being heated. Power is expressed in kilowatts, the electric field in volts/meter and frequency in MHz.

> The quantity of heat which may be generated in the process material may be increased by increasing the electric field strength or the frequency. The loss factor can be changed only by modifying the process material. Assuming that we cannot modify the material, only the electric field and frequency are left as variables. The (strength of the) electric field is limited by sparking or arcing along the surface or in the volume of the material. Therefore, the only way to get significant power into most materials by this technique is by using microwave frequencies. (Badger, 1970)

Material Parameters Involved in Microwave Processing

The dielectric constant plays an extremely important role as far as the properties of a material and its behavior at microwave frequencies are concerned. As Tinga (1970) tells us, however, a dielectric constant, despite its name, is not constant. It depends on such varied parameters of a material as temperature, microwave frequency, and density, all of which will be discussed later.

Harper and Tappel (1957) attempt to define a dielectric constant much as Tinga (1970) does when he refers to it in physical terms as relating "the value of the electric field which is measured in volts per meter within the material to the value of the electric field externally applied to the material." As has been demonstrated with the water example, virtually all dielectrics are polarized by the application of an electric field. When this happens, the average internal electric field between molecules is reduced, the amount of reduction being proportional to the dielectric constant of the material. Thus, a large value of dielectric constant indicates a large reduction of the internal field (Tinga, 1970).

According to Redheffer, et al. (1952), Harper, Chichester, and Roberts (1962), and Tinga (1970), calculations involving dielectric constants use the symbol ε

to refer to the total dielectric constant of a material or, as referred to in electrical engineering terms, complex permittivity. But, as Tinga (1970) notes, "there are two things taking place within a dielectric when an electric field is applied to it. There is a reduction of the average internal field within the dielectric which gives rise to a real part of the dielectric constant, normally just called dielectric constant, ε', and the associated relaxation phenomenon which is described by the imaginery part of the dielectric constant, denoted by ε'', often called loss factor. Then the total complex dielectric constant is written as $\varepsilon = \varepsilon' - j\varepsilon''$. The $-j$ indicates a 90° phaseshift between the real and imaginary part of the dielectric constant." The loss tangent, $\tan \delta$, is defined as the ratio of the dielectric loss to the dielectric constant, or $\tan \delta = \varepsilon''/\varepsilon'$. The values of the dielectric constant and loss tangent determine the amount of energy reflected, absorbed, and transmitted by the material. Dielectric loss determines the amount of power that can be absorbed within a material. Though White (1968) gives a rigorous and detailed development of the appropriate power question, an approximate formula for calculating absorbed power is:

$$\text{Absorbed power, } P = (55.61 \times 10^{-4}) f E^2 \varepsilon'' \text{ watts/cm}^3$$

where f is the frequency in cycles per second, E is the electric field strength in volts/cm., and ε'' is the dielectric loss factor.

The increase in the material's temperature when microwaves are applied to the material can be stated as:

$$\text{Increase in temperature} = \frac{(8 \times 10^{-12}) f E^2 \varepsilon''}{\rho c}$$

where ρ is the density in grams/cm^3 and c is the specific heat in calories/gram °C.

> Both these formulas are subject to the assumptions that a uniform electric field exists within a volume of the material. The electric field strength, E, is the only parameter which is difficult to measure in actual practice. The formulas clearly show, however, the effect of the variables on the absorbed power and heating rate. (Tinga, 1970)

The dielectric constant and dielectric loss factor also determine the depth of effective energy penetration into a material, this penetration depth being defined as:

$$d = \frac{\lambda_o \sqrt{\varepsilon'}}{2\pi\varepsilon''} \text{ cm}$$

where d is penetration depth in cm, λ_o is the free space wavelength in cm defined by $\lambda_o = c/f$ (c being the velocity of light in cm/sec and f being the frequency) and ε' and ε'' are the dielectric constant and dielectric loss factor, respectively. Thus, while energy entering a material is dissipated in a shorter distance if the loss factor increases, this is offset somewhat by the fact that the dielectric constant also increases when ε'' increases. "At a depth d cm into a material the available power in the material has dropped to about 37% of its value at the surface" (Tinga, 1970).

A material's dielectric properties also affect the controllability of a process. Positive temperature coefficients lead to poor controllability of temperature and moisture content, whereas a decreasing dielectric loss with increasing temperature creates a leveling effect as does the increase of loss with increasing moisture content. When the latter conditions exist, it is possible to establish extremely good control over a material's temperature and moisture content profile.

Parameters Affecting Dielectric Properties

Harper and Tappel (1957) have asserted that although the dielectric constant and loss tangent (or power factor) are both properties of the material in question, there is no consistent relationship between them. This statement was made in reference to the application of dielectric heating as a heat supply in freeze-drying, but it applies equally well to other uses of microwave heating. Furthermore, it is pointed out that a high dielectric constant does not necessarily mean the power factor will be high. These two properties show a wide, but unpredictable variation with frequency and are also dependent on temperature.

Harper, Chichester and Roberts (1962) reinforce this statement in their assertion that both components of the dielectric constant usually depend on frequency and temperature in a manner that can be determined only by experimental measurement for any specific material. They found this to be a general situation in their measurements of the dielectric properties of peaches, pears, beef, and fat at various temperatures and frequencies in the microwave range.

Tinga (1970) also notes these two parameters as affecting the dielectric constant of a material (hence, the paradoxical "inconstancy" of the term) and, in addition, refers to moisture content as a third important factor. Water has a high dielectric constant, approximately 80, whereas the base material's dielectric constant is usually about 2; thus, as water is added, a material's average dielectric constant gradually increases. And while water's relaxation frequency (the frequency where its dielectric loss is maximum) is about 20 GHz, when mixed with other dielectrics with lower loss, it often causes a "maximum loss" much lower than 20 GHz. The mixing of different dielectrics, in general, causes very complex phenomena to occur.

> Fortunately a few rules of thumb can be used which indicate in which direction the dielectric constant will change when two simple substances are combined into a mixture: (1) the higher the moisture content the higher the dielectric constant; (2) dielectric loss usually increases with increasing moisture content but levels off at a value in the range of 20-30% and may decrease at still higher moisture contents; (3) the dielectric constant of a mixture will always lie between the values of that of its components. (Tinga, 1970)

As moisture content is a variable in microwave applications, so is temperature. Tinga (1970) states that this temperature dependence is quite complex in nature in that dielectric loss may either increase or decrease with temperature, depending upon moisture content or the content of whatever lossy component is present in the mixture. "Therefore, it is extremely important to know in more detail what functional relationships exist between these parameters in any one material."

Frequency, as noted, affects the dielectric constant of a material as does its temperature and moisture content dependence. However, because the designer is limited as to his choice of frequencies by Federal Communications Commission regulations, knowledge of the frequency dependence of the dielectric's characteristics is useful for instrumentation design primarily where different FCC regulations apply. The FCC approved microwave frequencies for Industrial-Scientific-Medical (ISM) use include: 915, 2450, 5800, and 22,125 MHz.

Still other parameters affecting dielectric constant are physical geometry, density, conductivity, thermal conductivity and specific heat. The latter three have a relatively indirect effect and are discussed more fully by Tinga (1970). Density can greatly affect the value of the complex dielectric constant in that the dielectric constant of air is 1.0 and the inclusion of air voids in samples will always reduce a material's effective dielectric constant. Physical geometry affects interaction phenomena, and especially equipment design, as density and size variations in the material may make dielectric property determination difficult. For these reasons, Pace (1967) took size into special account in his investigation of the dielectric properties of potatoes and potato chips, as did Van Dyke (1968) in his ground beef studies and Stuchly (1970) in his work with granualr solids containing water.

Methods of Measurement

In their efforts to measure the dielectric properties of a wide variety of organic and inorganic materials at microwave frequencies, researchers have devised and modified various methods for doing so. Indeed, it has been observed that there are over thirty methods presently in existence (Tinga and Edwards, 1962). As Harvey (1963) points out, however, these methods generally fall into three basic categories which he terms free-space, transmission line, and resonant-cavity techniques.

Free-space methods were used in the earliest measurements on liquids and are based on observation of the wavelength and absorption in a material using optical-type instruments.

Transmission-line methods depend upon the changes in phase and/or amplitude of a standing or traveling wave due to the presence of the dielectric (Harvey, 1963). These methods have been reviewed extensively by such authors as Montgomery (1947), von Hippel (1966), and Harvey (1963). The most notable of the transmission-line methods is the short circuit method developed and described by Roberts and von Hippel (1946). Dakin and Works (1947) later modified the computations involved for the case of low loss dielectrics. Pace (1967) made use of, and fully describes, such a method in his investigations with a precision slotted transmission line. It can be seen by referring to these methods, however, that there is a big disadvantage in the complexity of the equations used. Even though some charts are available which make the procedure somewhat easier, the accuracy is often questionable because of unfortunate choices of sample length and poor resolution of the charts. Also, in the case of solids, the samples must be accurately fitted to the dimensions of the transmission line sample holder.

The third category, that of resonant cavity techniques, is reviewed in detail, again by Montgomery (1947), von Hippel (1966), and Harvey (1963).

Unfortunately, the evaluation of dielectric constants is not always a simple and easy task, and all these methods suffer from one or more of the following disadvantages: (1) the calculations are difficult and tedious; (2) they are useful only for very limited ranges of loss tangent values; (3) elaborate measuring setups are needed; (4) sample preparation is often difficult; (5) information on both phase and amplitude is necessary; (6) poor samples often give rise to uninterpretable results; and (7) the experimental conditions required are difficult to achieve in practice.

A resonant cavity technique was chosen for the study reported herein because of the simplicity of the measurement and of the equations involved for arriving at the dielectric properties, the availability of the equipment, the relatively high accuracy of the method and the somewhat simple handling of samples, particularly liquids as in this case. The method involves measurements made by perturbation techniques as described by Horner, et al. (1946), Dunsmuir and Powles (1946), Birnbaum and Franeau (1949) and, later, Anon. (1970).

The method is described in detail in the section on methods and materials.

Measurement Studies of Food and Food-Related Materials

Recognizing the important role the dielectric properties of a food plays in high frequency power applications, a number of authors have made investigations of these properties using a variety of measurement methods. Pace (1967) provides a fairly comprehensive review of many dielectric property studies on food and food-related materials along with results. Most of the investigations reported recognize the temperature and frequency dependence of the dielectric properties. Unfortunately, much of the work that has been reported, particularly with regard to food systems, has been conducted at frequencies below the microwave range.

In a study reported by deLoor and Meijboom (1966) using transmission-line techniques, foods and other materials with high water contents (greater than 75% by volume) were measured for dielectric properties between 1.2 and 18 GHz. All results were found to lie on semicircular Cole-Cole arcs (ε' versus ε''), indicating a single relaxation time which they ascribed to the free water available. Stuchly (1970) investigated the dielectric properties of granular solids containing water at a frequency of 9.4 GHz. Buchanan, *et al*. (1952)

measured the dielectric properties of six aqueous protein solutions at microwave frequencies in an attempt to estimate protein hydration.

The dielectric properties of aqueous and alcoholic electrolytic solutions have been investigated by Hasted and Roderick (1958), the properties of aqueous ionic solutions by Hasted, Ritson and Collie (1947), the dielectric properties of water by Hasted (1961) and of water and heavy water by Collie, Hasted and Ritson (1948).

The research with food materials, however, has been scarce. Pace (1967) studied the dielectric properties of eleven commercial fats and oils, raw potatoes and potato chips using the slotted coaxial transmission line technique of von Hippel. Dielectric properties were reported as a function of temperature and frequency for the fats and oils. Dielectric properties were reported as a function of frequency, temperature, and moisture for potato chips. These frequencies ranged from 300 to 3000 MHz.

Van Dyke (1968) reported on the dielectric properties of ground beef in which he had varied the proximate analysis, also using von Hippel's technique. The dielectric properties were reported as a function of temperature, frequency and proximate analysis. As with Pace's study, the frequencies ranged from 300 to 3000 MHz.

In spite of the increased activity in the area of microwave power applications since about the mid-1960's, Goldblith and Pace (1967) have pointed out that there still remains "a paucity of data on the effects of these radiations on the food stuffs and their components." Aside from the investigations of Pace (1967) and Van Dyke (1968), apparently very little work in this area has been conducted.

METHODS AND MATERIALS

Introduction to Cavity Perturbation Theory

As mentioned previously, the method used throughout this research involves the resonant cavity perturbation technique. Various authors, including Montgomery (1947), von Hippel (1966) and Harvey (1963) have developed the theory which applies to resonant cavity techniques. These theoretical developments are all based on Maxwell's electromagnetic-field equations which, because of length and complexity, will not be cited here.

This study employs the method as described by Dunsmuir and Powles (1946), Birnbaum and Franeau (1949) and Anon. (1970) because of its relative ease of measurement and simple calculations. It is a resonant cavity technique based on cavity perturbation theory.

Horner, *et al*. (1946) and Anon. (1970) describe a method for measuring the dielectric properties of solid dielectrics in the microwave range using a cylindrical cavity resonator. In such methods a variable frequency oscillator is used along with the cavity to measure the permittivity and loss tangent (tan δ) of cylindrical rod samples of low loss solid dielectric materials. The cavity resonator is excited in the dominant (lowest resonant frequency) TM_{010} mode of oscillation and the permittivity of the dielectric is determined from the resonant frequencies of the cavity when completely air-filled, and when containing the dielectric sample. The TM_{010} mode designation refers to a transverse magnetic EM propagation in which the electric field is purely longitudinal and the magnetic field is purely circumferential. The dielectric power factor is determined from the change in the corresponding quality (Q) factors of the same system. The Q factors are determined from a transmission measurement of the cavity's resonance or Q curve, which relates the square of the field strength (energy density) within the cavity to excitation frequency. According to Dunsmuir and Powles (1946) it is possible to obtain accuracies of 1 per cent in permittivity and 2 per cent in power factor measurements.

If the method is to be applied to the measurement of the dielectric properties of liquid samples, these must be retained in the necessary rod form by use of a thin-walled container. The method was originally based on a development by Anon. (1970) for material perturbation of the TM_{010} circular cylindrical cavity mode. The material, even though in a thin-walled container, was assumed to be a thin dielectric rod. As it turned out, the Dunsmuir and Powles development paralleled this and confirmed the assumption that a thin wall of containing material would have negligible effect upon measurements of the dielectric properties of the liquid material.

Dunsmuir and Powles (1946) provide the mathematical analysis for such a three layer dielectric system which leads to expressions for the permittivity and tan δ values of the liquid sample as functions of the resonant frequency and Q factor of the resonant cavity with and without the sample. As is pointed out later in their analysis, if the wall thickness of the container approaches zero, the expressions reduce to those derived for the two layer, solid dielectric case by Horner, et al. (1946) and Anon. (1970).

The following is the theory of the TM_{010} resonant cavity containing three dielectric layers as developed by Dunsmuir and Powles (1946).

The Resonant Condition and the Permittivity Relations

The system being considered is a circular cylindrical resonator (Figure 1) consisting of three regions designated by the subscripts (a), (b) and (c) respectively, containing media of different dielectric properties. The bountry surfaces between adjacent regions are concentric cylinders of radii "b," "c" meters and the highly conducting outer wall is of radius "a" meters.

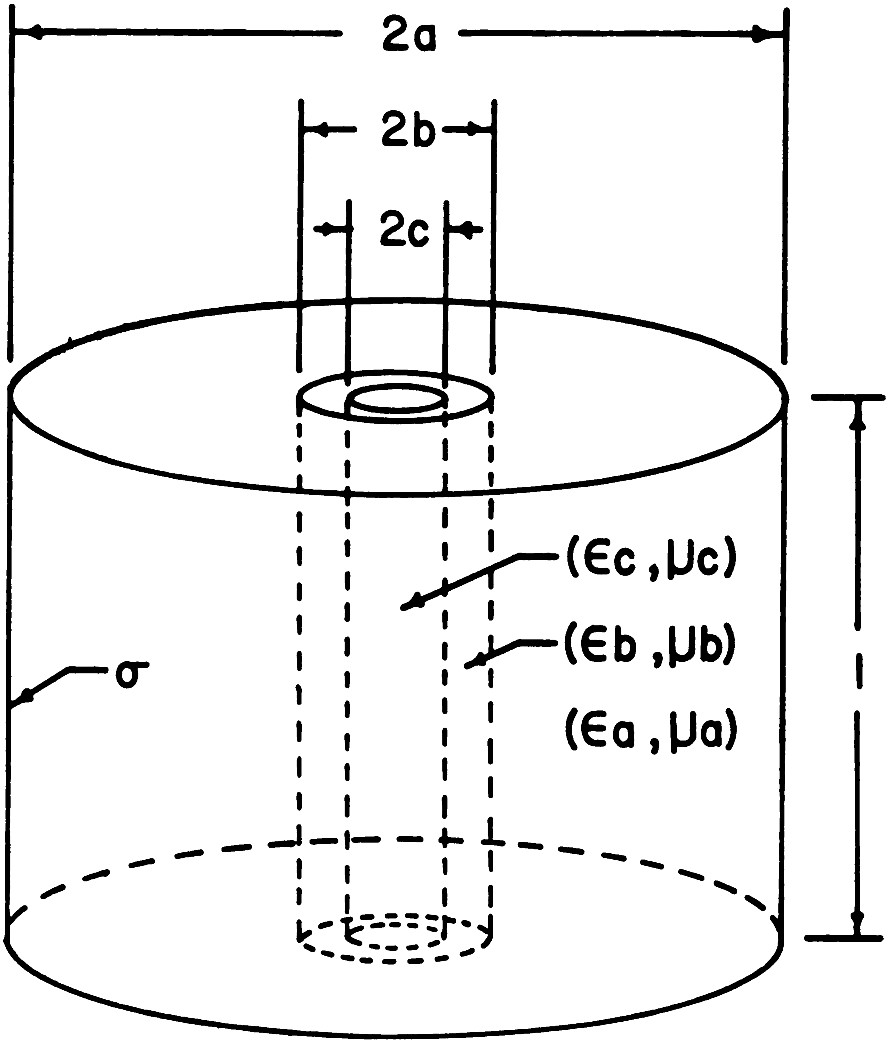

Figure 1. Circular cylindrical resonator consisting of three regions.

The field components for the TM_{010} mode are then given in M.K.S. units and in terms of cylindrical polar coordinates as

$$H_\theta = \{AJ_1(\beta r) + BY_1(\beta r)\}\, e^{j\omega t} \text{ amp/meter}$$

(1)

$$E_z = -j\sqrt{\frac{\mu}{\varepsilon}}\{AJ_o(\beta r) + BY_o(\beta r)\}e^{j\omega t} \text{ volts/meter}$$

where H_θ and E_z are the radial magnetic field intensity and the longitudinal electric field intensity, respectively, and J and Y are Bessel functions of the first and second kinds, respectively. The amplitude constants A, B and propagation constant $\beta = \omega\sqrt{\varepsilon\mu}$ are chosen appropriately for each of the regions (a), (b), and (c).

Relations between the values taken by A, B within these regions are obtained from consideration of the physical (or boundary) conditions at the boundary surfaces, that is:

(1) that the tangential component of electric field intensity is zero at the outer wall r = a, and

(2) that the tangential components of electric and magnetic intensity are continuous at the boundary surfaces r = b and r = c.

From these, Dunsmuir and Powles obtained equations (2), in which it is assumed that the permeability has the value μ_a ($\mu_a = \mu_o = 4\pi \times 10^{-7}$ for virtually all dielectrics) everywhere within the system.

$$(2)\quad \left\{ \begin{aligned} & A_a J_o(\beta_a a) + B_a Y_o(\beta_a a) = 0 \\ & A_a J_1(\beta_a b) + B_a Y_1(\beta_a b) - A_b J_1(\beta_b b) - B_b Y_1(\beta_b b) = 0 \\ & \frac{A_a}{\sqrt{\varepsilon_a}} J_o(\beta_a b) + \frac{B_a}{\sqrt{\varepsilon_a}} Y_o(\beta_a b) - \frac{A_b}{\sqrt{\varepsilon_b}} J_o(\beta_b b) - \frac{B_b}{\sqrt{\varepsilon_b}} Y_o(\beta_b b) = 0 \\ & A_b J_1(\beta_b c) + B_b Y_1(\beta_b c) - A_c J_1(\beta_c c) = 0 \\ & \frac{A_b}{\sqrt{\varepsilon_b}} J_o(\beta_b c) + \frac{B_b}{\sqrt{\varepsilon_b}} Y_o(\beta_b c) - \frac{A_c}{\sqrt{\varepsilon_c}} J_o(\beta_c c) = 0 \end{aligned} \right.$$

where $\beta_a = \omega\sqrt{\mu_a \varepsilon_a}$, $\beta_b = \sqrt{\dfrac{\varepsilon_b}{\varepsilon_a}}\,\beta_a$ and $\beta_c = \sqrt{\dfrac{\varepsilon_c}{\varepsilon_a}}\,\beta_a$

The quantities M_1, M_2, M_3, M_4, and M are then defined as:

$$(3)\quad \left\{ \begin{aligned} & M_1 = J_o(\beta_b b) Y_1(\beta_b c) - J_1(\beta_b c) Y_o(\beta_b b), \\ & M_2 = J_1(\beta_b b) Y_1(\beta_b c) - J_1(\beta_b c) Y_1(\beta_b b), \\ & M_3 = J_o(\beta_b b) Y_o(\beta_b c) - J_o(\beta_b c) Y_o(\beta_b b), \\ & M_4 = J_1(\beta_b b) Y_o(\beta_b c) - J_o(\beta_b c) Y_1(\beta_b b), \end{aligned} \right.$$

$$(4)\qquad \begin{aligned} M &= \frac{Y_1(\beta_a b)/J_1(\beta_a b) - Y_o(\beta_a a)/J_o(\beta_a a)}{Y_o(\beta_a b)/J_o(\beta_a b) - Y_o(\beta_a b)/J_o(\beta_a a)} \\ &= 1 + \frac{1}{F}\,\frac{a}{b}\,\frac{J_o(\beta_a a)}{J_1(\beta_a a)} \end{aligned}$$

(5) where $$F = \left[Y_o(\beta_a a) J_o(\beta_a b) - Y_o(\beta_a b) J_o(\beta_a a) \right] \frac{\pi \beta_a a}{2}$$

Eliminating the amplitude constants A_a, A_b, A_c, B_a, and B_b from equations (2) result in

$$\sqrt{\frac{\varepsilon_c}{\varepsilon_b}} \frac{J_1(\beta_a c)}{J_o(\beta_a c)} = \frac{M_2 - \sqrt{\frac{\varepsilon_a}{\varepsilon_b}} \frac{J_1(\beta_a b)}{J_o(\beta_a b)} M_1 M}{M_4 - \sqrt{\frac{\varepsilon_a}{\varepsilon_b}} \frac{J_1(\beta_a b)}{J_o(\beta_a b)} M_3 M} \tag{6}$$

The transcendental equation (6) relates the permittivities and the dimensions of the three regions to the resonant frequency, but is not suitable for computation. Dunsmuir expresses this in more convenient terms in equation (10) by use of the approximation

$$\frac{J_1(x)}{J_o(x)} \doteq \frac{x}{2} \left[1 + \frac{x^2}{8} \right] \tag{7}$$

and the following approximate expressions for M_1, M_2, M_3, and M_4

$$\begin{cases} M_1 = - \frac{2}{\pi \beta_b c} \\ M_2 = M_1 \beta_b (b - c), \\ M_3 = M_2, \\ M_4 = - \frac{c}{b} \cdot M_1 \end{cases} \tag{8}$$

Expressions (8) are obtained by expansion in Taylor series assuming that (b-c) is small compared with b and c and by use of the identity

$$(9) \qquad J_1(x)Y_o(x) - Y_1(x)J_o(x) = \frac{2}{\pi x}$$

This leads to the equation

$$(10) \qquad \frac{\varepsilon_c}{\varepsilon_a} = \frac{\frac{b^2}{c^2}\left[1 + \frac{(\beta_a b)^2}{8} M - M_b\left(1 - \frac{(\beta_a b)^2}{8}\right)\right]}{1 + \frac{(\beta_a b)^2}{8}\left[M - M_b\right]}$$

where

$$M_b = 2\,\frac{\varepsilon_b}{\varepsilon_a}\,\frac{(b-c)}{b}$$

The expression (10) is suitable for direct calculation of the relative permittivity, $\frac{\varepsilon_c}{\varepsilon_a}$ of the sample. Computation of the function M in equation (4) is made easier by using the graphical plot of numerical values for the function F, equation (5), given by Horner, et al. (1946).

The relative permittivity, $\frac{\varepsilon_b}{\varepsilon_a}$ of the containing material is assumed known but a highly accurate value is not necessary. The stated degree of inaccuracy introduced by the approximation is less than 1 per cent for values of $\frac{\varepsilon_b}{\varepsilon_a}$ less than 4 and of $\frac{(b-c)}{a}$ less than 1/40.

In the case of a very thin walled containing material where ($b/a < 1/50$), further approximations lead to:

$$(11) \qquad \frac{\varepsilon_c}{\varepsilon_a} = 1 + 0.539 \, \frac{a^2}{c^2} \, \frac{(df)}{f_o}$$

where (df) is the change in resonant frequency due to the presence of liquid sample and f_o is the unperturbed resonant frequency. Expression (11) is stated to be useful for liquids of high loss ($\tan \delta > 0.006$), in which case a sample of small diameter must be used.

Power Factor Relations

Referring to the field equations (1), which may be written for brevity's sake as

$$A_a J_o(\beta_a r) + B_a Y_o(\beta_a r) = {}_aZ_o(\beta_a r)$$

etc., where $Z_o(x)$ is known as a cylinder function (a Bessel function of the first or second kind). The field equations for the EM field in region (a) then become

$$(1') \qquad \begin{aligned} H_\theta &= {}_aZ_1(\beta_a r) e^{j\omega t} \\ E_z &= \sqrt{\frac{\mu_a}{\varepsilon_a}} \, {}_aZ_o(\beta_a r) e^{j\omega t} \end{aligned}$$

and similar relations hold for the (b) and (c) regions.

The procedure for calculating the Q value of the system makes use of the relation

$$(12) \qquad Q = \omega\frac{\text{stored energy}}{\text{mean power loss}}$$

described by Horner, et al. (1946). Thus the energy stored is given by

$$W = 2\pi l \int_{o}^{a} \frac{r\mu H^2}{2}\, dr = \pi l \int_{o}^{a} r \cdot {}_{\nu}Z_1^2(\beta_{\nu} r)dr$$

where ν encompasses the regions (a), (b) and (c), such that

$$(13) \qquad W = \frac{1}{2}\, l\pi\mu\{a^2\, {}_{a}Z_1^2(\beta_a a) + b^2(1 - \frac{\varepsilon_a}{\varepsilon_b})\, {}_{b}Z_o^2(\beta_b b)$$

$$+ c^2(1 - \frac{\varepsilon_b}{\varepsilon_c}\, {}_{c}Z_o^2(\beta_c c)$$

The power loss in the liquid dielectric region (c) is

$$(14) \qquad \left\{ \begin{aligned} P &= 2\pi l \int_{o}^{c} \frac{r}{2}\, q_c\, E^2\, dr \\ &= \frac{1}{2}\, \pi l \omega\mu \tan \delta_c \cdot c^2\{{}_{c}Z_1^2(\beta_c c) + {}_{c}Z_o^2(\beta_c c)\} \end{aligned} \right.$$

where q_c is the effective conductivity of the liquid and $\tan \delta_c = q_c/\omega\varepsilon_c$.

Q_2 is defined as the Q factor of the cavity system with the hollow liquid sample container filled with a sample dielectric, and Q_1 as the corresponding Q value if the container were filled with loss-free liquid of the same permittivity. It then follows from (12) and (13) and the definition of Q that

$$\text{(15)}\quad \tan\delta_c = \frac{a^2\,{}_aZ_1^2(\beta_a a) + b^2\left(1 - \frac{\varepsilon_a}{\varepsilon_b}\right){}_bZ_o^2(\beta_a b) + c^2\left(1 - \frac{\varepsilon_b}{\varepsilon_c}\right){}_cZ_o^2(\beta_c c)}{c^2\{{}_cZ_1^2(\beta_c c) + {}_cZ_o^2(\beta_c c)\}} \times \left[\frac{1}{Q_2} - \frac{1}{Q_1}\right]$$

which reduces to the approximate form (16) when the container wall is thin.

$$\text{(16)}\quad \tan\delta_c = \frac{\left\{\frac{a^2}{c^2 F_b{}^2} + \frac{b^2}{c^2}\left(\frac{\varepsilon_b}{\varepsilon_a} - 1\right)\right\}\left\{1 - 2\beta_c(b-c)\,\frac{J_1(\beta_c c)}{J_o(\beta_c c)}\right\} + \frac{\varepsilon_c - \varepsilon_b}{\varepsilon_a}}{\frac{\varepsilon_c}{\varepsilon_a}\left\{1 + \frac{J_1^2(\beta_c c)}{J_o^2(\beta_c c)}\right\}} \times \left[\frac{1}{Q_2} - \frac{1}{Q_1}\right]$$

F_b is the function F, defined in (5), expressed in terms of the ratio b/a.

When the relative permittivity $\frac{\varepsilon_b}{\varepsilon_a}$ and the power factor $\tan\delta_b$ of the container are known, Q_1 may be derived from the Q factor Q_o of the completely air-filled resonator by means of the relation $Q_1 = Q_o\ \times$

$$(17)\left\{\frac{\left\{\frac{a^2}{c^2F_b{}^2}+\frac{b^2}{c^2}\left(\frac{\varepsilon_b}{\varepsilon_a}-1\right)\right\}\left\{1-2\beta_c(b-c)\frac{J_1(\beta_c c)}{J_o(\beta_c c)}\right\}+\frac{\varepsilon_c-\varepsilon_b}{\varepsilon_a}}{\frac{a}{a+1}\sqrt{\frac{f_o}{f_1}}\left[\left\{\frac{a(a+1)}{1^2F_b{}^2}+\frac{b^2}{c^2}\left(\frac{\varepsilon_b}{\varepsilon_a}-1\right)\right\}\left\{1-2\beta_b(b-c)\frac{J_1(\beta_c c)}{J_o(\beta_c c)}\right\}+\frac{\varepsilon_c-\varepsilon_b}{\varepsilon_a}\right]}\right)$$

$$+\ 2\ \frac{b-c}{c}\ \frac{\varepsilon_b}{\varepsilon_a}\ Q_o\ \tan\ \delta_b$$

where the frequencies f_o and f_1 correspond to Q_o and Q_1 respectively.

This relation is not as complicated as it seems since some of the terms have already been calculated in (16). The two expressions (16) and (17) are accurate to better than 1 per cent for values of $\frac{\varepsilon_b}{\varepsilon_a}$ less than 4, and of $(\frac{b-c}{a})$ less than 1/40, as in the case of the permittivity relation. For containers of small radius, again, with b/a less than 1/50, expression (16) reduces to

$$(18)\qquad \tan\ \delta = \frac{0.269}{\varepsilon_c/\varepsilon_a}\ \frac{a^2}{c^2}\left[\frac{1}{Q_2}-\frac{1}{Q_1}\right]$$

Expressions (11) and (18) are essentially the same as those developed by Anon. (1970) or

$$(19)\qquad \frac{(df)}{f_o} = -\ \frac{(\varepsilon'-1)}{2}\ \left(\frac{b}{a}\right)^2\left[\frac{J_o^2(p_{01}b/a + J_1^2(p_{01}b/a)}{J_1^2(p_{01})}\right]$$

$$(20)\qquad \left[\frac{1}{Q}-\frac{1}{Q_o}\right] = \varepsilon''\left(\frac{b}{a}\right)^2\left[\frac{J_o^2(p_{01}b/a) + J_1^2(p_{01}b/a)}{J_1^2(p_{01})}\right]$$

where ε' is the real part of the complex permittivity and ε'' is the imaginary part, remembering that tan δ can be defined as $\varepsilon''/\varepsilon'$. $J_0(x)$ and $J_1(x)$ are Bessel functions of zero and first order, and $P_{01} = 2.405$ is a constant for the TM_{010} resonant cavity. These latter two expressions were considered valid according to the Dunsmuir and Powles development and were thus used throughout this investigation.

Equipment Used for the Cavity Perturbation Technique

Measurements were carried out using five cavities with design resonant frequencies of 970, 1131, 2000, 2450, and 3000 MHz, respectively. Details of their design and construction are given in a later section.

The two lower frequency cavities were used in a microwave circuit with the following auxiliary equipment:

1. A modulating power supply, operated with 1kHz square wave amplitude modulation and minimum modulated amplitude, General Radio Co., Type 1264-A, Serial #875.

2. A 900-2000 MHz unit oscillator, operated at an output level to give full scale reading on the standing wave meter, General Radio Co., Type 1218 B, Serial #435.

3. A frequency meter; Hewlett Packard, Model 536A, Serial #3130.

4. A standing wave indicator; Hewlett Packard, Model 415B.
5. A coaxial switch; Transco Products, Inc., 1 pole 6 throw, Type M-1560-6, Serial #115.

Also included in the circuit were three 10 db attenuators (General Radio Co., Type 874 - G 10), a square law crystal detector and tee (General Radio Co., Type 874 - VQL), a 50 ohm termination (General Radio, Type 874 - W50BL, Serial #5242) and General Radio Co. coaxial transmission cables.

The complete circuit as used for both the 970 and 1131 MHz cavities is shown diagramatically in Figure 2.

The three higher frequency cavities were energized by a sweep oscillator and their resonance or Q curves, obtained by transmission measurements, displayed on an oscilloscope screen. The auxilliary equipment used in this circuit consisted of:

1. A sweep oscillator with sweep range of 2000-4000 MHz, operated as an internal sweep modulation source with recurrent sweep. Settings: cathode current, 8.5 ma., anode selector, off, recurrent sweep at a sweep rate of 16,000 MHz/sec., and Δ frequency, 210 MHz.

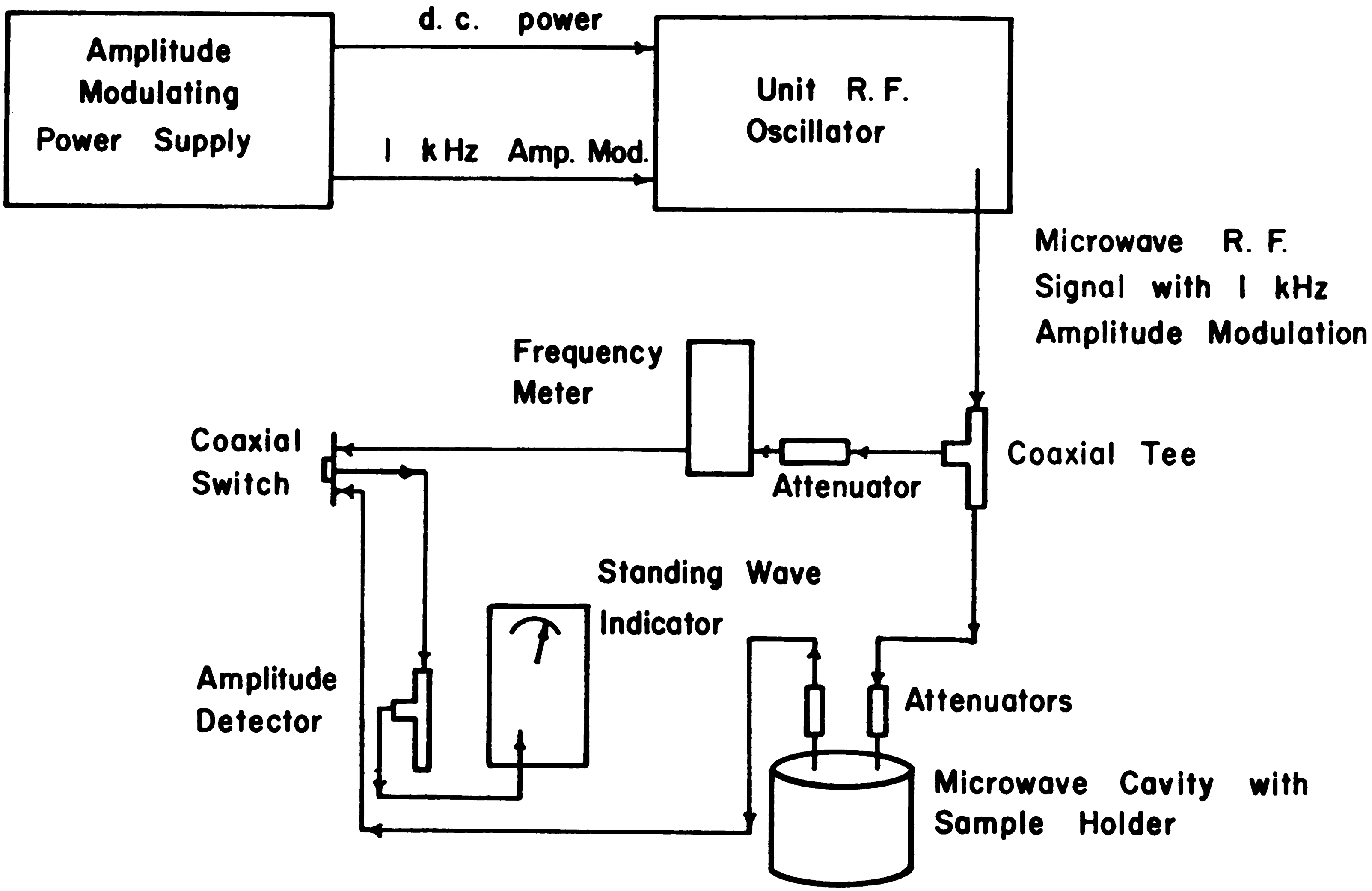

Figure 2. Microwave circuit used with the 970 and 1123 MHz resonant cavities.

2. An oscilloscope (Tektronix Type 581A) with a dual trace plug-in unit (Type 82), operated to give a full screen trace of the cavity resonance curves.
3. A frequency meter; Hewlett Packard, Model 536A, Serial #3130.

Also included in the circuit were two 10 db altenators (General Radio Co., Type 874 G 10), a square law crystal detector and tee (General Radio Co., Type 874-VQL) a 50 ohm termination (General Radio, Type 874-W50BL, Serial #5242 and General Radio coaxial transmission cables.

The complete microwave circuit used for the 2000, 2450, and 3000 MHz cavities is shown diagramatically in Figure 3.

Design and Construction of Resonant Cavities

It was desired to study the dielectric properties of the samples at frequencies encompassing those frequencies presently being used commercially, namely 915 and 2450 MHz. Because it was impossible to obtain standard brass tubing of large enough diameter, a cavity at 915 MHz could not be constructed. Thus, the more convenient frequency, 970 MHz, was decided upon.

The resonant frequency of a TM_{010} mode circular cylindrical cavity is given as

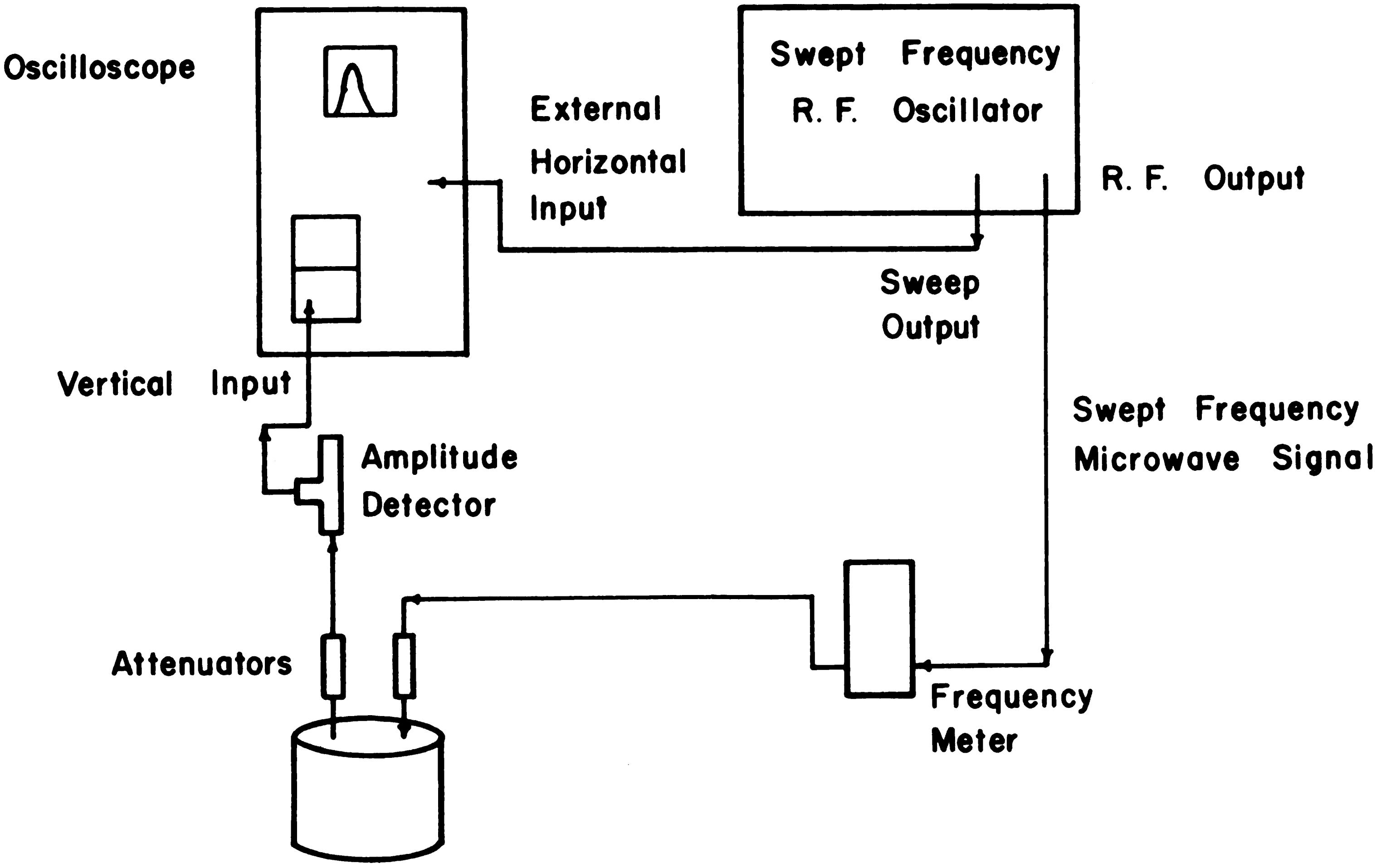

Figure 3. Microwave circuit used with the 1978, 2431, and 2973 MHz resonant cavities.

$$f_o = \frac{2.405}{2\pi a\sqrt{\mu\varepsilon}}$$

where a is the inside radius of the cavity, and $v = \frac{1}{\sqrt{\mu\varepsilon}}$ is the propagation velocity of EM waves in the medium filling the cavity or 3×10^8 m./sec. for air. Thus, for example, in designing the 3000 MHz cavity the inside radius is given as

$$a = \frac{(2.405)(3 \times 10^8 \text{ m./sec.})}{2\pi(3 \times 10^9\text{/sec.})}$$

and a = 0.03830 or 3.830 cm. The actual measured resonant frequency will be just slightly lower than the design frequency due to the skin-effect and holes in the walls for coupling of the excitation and detection probes.

The height, d, of the cavities was chosen such that $(2a/d)^2$ is greater than 1 according to the mode chart for circular cylindrical air-filled cavities (Anon., 1969). This allows excitation of only the TM_{010} mode at the frequencies employed. Thus, all cavities were designed with d = a. This also allows the designed Q factor for each cavity to be large, which is desirable.

The Q factor for a TM_{010} mode circular cylindrical cavity is given as

$$Q_o = \frac{\lambda}{\delta}\,\frac{2.405}{2\pi(a/d + 1)}$$

where δ = skin depth = $1/\sqrt{\pi f_o \mu \sigma}$

$\mu = \mu_o$ = $4\pi \times 10^{-7}$ for air

σ = 5.8×10^7, the conductivity for brass, the material of construction for all five cavities

f_o = the design resonant frequency

λ = wavelength at the resonant frequency = $3 \times 10^8/f_o$

a = inside radius of cavity

d = inside height of cavity

In the case of the 3000 MHz cavity, again where a = d = 3.830 cm., then $Q_0 = 7.653 \times 10^3$.

According to such design calculations as summarized in Table 1, the five resonant cavities were machined and constructed using standard brass tubing for the cylindrical walls and 1/4" brass plate for two end plates.

TABLE 1.--Design values for the resonant cavities.

Cavity	f_o X 10^8Hz	Q_o, X 10^4	a, cm	d, cm.	$(2a/d)^2$
1	9.70	2.791	11.747	11.747	4
2	11.31	1.2470	10.160	10.160	4
3	20.00	0.9375	5.744	5.744	4
4	24.50	0.8467	4.689	4.689	4
5	30.00	0.7653	3.830	3.830	4

A 1/16" hole was drilled through the end plates along the axis of each cavity to allow a thin walled tube, used as a sample holder, to be inserted coaxially through the cavities. In addition, two holes were drilled and threaded at mid-radius along a diameter of one end plate on each cavity. Each of these two holes was fitted with straight metallic probes coupled to coaxial transmission systems, one probe for providing excitation (introducing energy as a transmitting antenna) to a cavity, the other to detect the amplitude of the TM_{010} mode EM field excited in the cavity (extracting energy as a receiving antenna). A probe length of 24 mm. was determined to give near critical coupling and a satisfactory curve response for the 1131 MHz cavity. The probe lengths for each of the other four cavities were then based on this value, being scaled according to resonant frequency. These lengths were 27.8, 13.5, 11.0, and 9.0 mm. for the 970, 2000, 2450, and 3000 MHz cavities respectively. A diagram of a typical cavity appears in Figure 4.

Sample Tubing

The selection of the containing material (tubing) for the samples had to meet the following requirements. The tubing had to be uniform in cross-section, to be unaffected either physically or chemically by the liquid samples, and to be of low permittivity and power factor

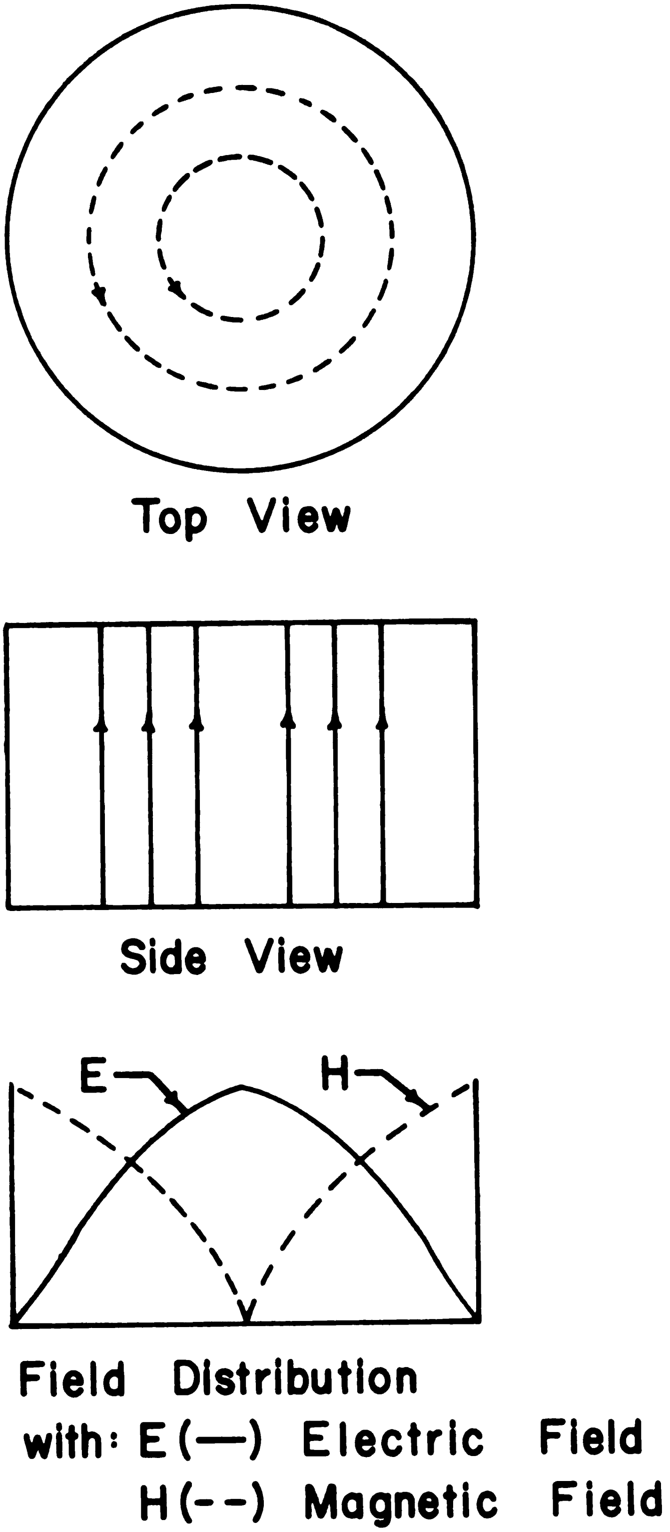

Figure 4. TM_{010} mode circular cylindrical cavity.

in order to minimize the effect of the tubing on the resonance frequency and Q factor of the system.

A number of materials were tested including glass capillary, Teflon tubing and Tygon tubing. It was found, however, that polyethylene tubing (Clay Adams, intramedic polyethylene tubing, medical formulation PHF) met the tubing requirements best. The sizes of tubing chosen were designated PE 20 (nominal dimensions: 0.015" I.D. x 0.043 O.D.) and PE 50 (nominal dimensions: 0.023" I.D. x 0.038" O.D.) by the manufacturer. Both sizes of tubing were used throughout the investigation since, theoretically, as long as the tube size is small enough and meets the above requirements, the results from measurements on the sample for each tubing should be the same. According to von Hippel (1966), polyethylene has dielectric properties of $\varepsilon' = 2.25$ and $\tan \delta = 3 \times 10^{-4}$. The two sizes of tubing used in each measurement also afforded a check on the consistency of the method and the validity of the theoretical analysis. Liquid samples were gravity fed through the tubing.

Samples and Preparation

The method as discussed is developed to handle liquid samples of high loss. It was applied to liquid samples including:

1 - distilled deionized water

2 - aqueous solutions of sodium chloride

3 - aqueous solutions of dextrose

4 - aqueous solutions of maltose

5 - aqueous solutions of dextrin

6 - aqueous solutions of sodium caseinate

7 - samples of a commercial corn oil.

The water and solutions of sodium chloride were used as standards for comparison, since a few literature values are available for these at microwave frequencies. The sodium chloride (analyzed reagent grade, Mallinckradt) was prepared into 1.0, 1.5, and 2.0 M solutions using distilled, deionized water.

The dextrose (anhydrous granular, analyzed reagent grade, Baker) was prepared into 10, 20, and 30% (by weight) solutions using distilled deionized water.

The maltose (Bacto Maltose "Difco" certified reagent grade), was prepared into 10, 20, and 30% (by weight) solutions using distilled, deionized water.

The dextrin (special, bacteriological grade, Nutritional Biochemicals Corporation) was prepared into 10, 20 and 30% (by weight) slurries using distilled, deionized water. The proximate analysis of the dextrin as given by the supplier was:

Starch: none

Ash: 0.5%

Moisture: 0.10%

Reducing sugar: 5.0%

Sodium caseinate (Nutritional Biochemicals Corporation) was prepared into 2.5, 5.0 and 10.0% (by weight) solutions. The proximate analysis as given by the supplier was:

Protein (N x 6.38)(dry basis): 92.5-94.5%

Moisture: 5.0%

Ash: 4.0%

Fat: 1.5%

Sodium: 1.3%

In order to solubilize the protein, each solution was prepared using deionized, distilled water made up to pH 11 using sodium hydroxide. It was necessary to readjust these protein solutions, then, all to the same pH of 8.0 with 0.1 N sodium hydroxide, which did not appreciably change the composition of the solutions.

The commercial corn oil used (Mazola, Best Foods Division of CPC International, Inc.) contained additives of isopropyl citrate and methyl silicone for freshness as declared on the label. The corn oil was used as is.

Each sample was measured immediately upon preparation in both the PE 20 and PE 50 sample tubing, at the five specified frequencies and at room temperature.

Calculation of ε', ε'', and tan δ from Experimental Data

Before making any measurements on the samples it was necessary to obtain an accurate measure of the inside diameters of both the PE 20 and PE 50 sample tubes used throughout this investigation. These tube diameters, as far as the calculation of dielectric properties are concerned, become the diameters of the columns of liquid sample filling the tubes. An accurate knowledge of these diameters is necessary since, as will be seen later, the sample diameter is a limiting factor with respect to the accuracy of the ε' value. The diameters of representative cross-sections of each of the tubes was measured using a microscope with a calibrated eyepiece. The PE 20 tubing had an average diameter of 0.392 mm while the average diameter of the PE 50 tubing was 0.538 mm.

The measured diameter for the tube used for a particular measurement (with reference to equations (19) and (20)) is used to determine the radius of the sample in inches, designated as "b" in each equation. Next, the resonant frequency of the cavity, f_o, containing the empty sample tube is determined. This is done, in the case of the circuit used for the two lowest frequencies (Figure 2), by first adjusting the frequency dial of the unit oscillator to give a maximum deflection on the standing wave meter. The frequency is determined by rotating the calibrated frequency meter to the frequency resulting in a minimum deflection of the standing wave meter. This frequency corresponds to f_o for the particular cavity and sample tubing used in a measurement. With the circuit used for the three higher frequencies (Figure 3), the resonant frequency is determined using the frequency meter. The meter causes a "pip" at the maximum point (maximum power) of the full screen trace of the cavity resonance curve on the oscilloscope. This frequency is taken as f_o.

In either case the resonant frequency, f_o, is used directly in equation (19) and also in the determination of the radius of the cavity, "a," under the test conditions. The cavity radius is obtained from the equation for resonant frequency of a TM_{010} mode circular cylindrical cavity

$$F_o = \frac{2.405}{2\pi a\sqrt{\mu\varepsilon}}$$

where $\sqrt{\mu\varepsilon}$ is given as 3×10^8m/sec. and "a" is expressed in inches. The cavity radius, "a," so determined, also appears in equations (19) and (20).

The quality factor, Q_o, for the cavity containing the empty sample tubing is determined next. At the two low frequencies this is done by adjusting the frequency upward with the unit oscillator until the needle of the standing wave meter reads 3 db. (the upper half power point). This frequency is determined as before using the frequency meter. The frequency is then adjusted downward with the unit oscillator until the needle of the standing wave meter again reads 3 db. (the lower half power point) and the frequency is read.

The quality factor, Q_o, for the cavity containing the empty sample tubing is given as

$$Q_o = \frac{f_o}{B}$$

where f_o is the resonant frequency and B is the difference in frequency between the upper and lower half power points.

For the three higher frequencies, B was determined simply by measuring the distance across the full screen trace of the resonance curve on the oscilloscope at one-half the maximum power. The horizontal scale of the oscilloscope screen (frequency) was calibrated to allow such a measurement. The quality factor, Q_0, so determined appears in equation (20).

To complete all the measurements necessary to calculate the dielectric properties, ε' and ε'', from equations (19) and (20), respectively, the resonant frequency, f, and quality factor, Q, are re-determined with sample filling the sample tubing. In equation (19) df is the difference $(f - f_0)$. Thus, the ε' and ε'' values were calculated directly from equations (19) and (20) for each sample, sample tubing, and resonant cavity.

The loss tangent, tan δ, is calculated from the values of ε' and ε'' or $\tan \delta = \varepsilon''/\varepsilon'$.

RESULTS AND DISCUSSION

The microwave dielectric properties of a variety of aqueous solutions of food materials and one commercial corn oil sample were determined according to the methods described previously. The dielectric constant, ε', loss factor, ε", and loss tangent, tan δ, were measured at five frequencies including 970, 1123, 1978, 2431, and 2973 MHz, all at room temperature (28°C.±3°C.).

Except for the cavity with a resonant frequency of 970 MHz, all the cavities had slightly lower actual measured frequencies than the calculated frequencies given in Table 1, due as mentioned to the skin effect and holes in the walls for the probes. The actual Q values were also lower than the calculated values given in Table 1, due most probably to the lack of critical coupling of the probes and to coupling of losses from the external circuit to the cavity due to attenuation of the excitation and output signals.

The dielectric properties as calculated from the measurements on all the samples are presented in Table 2. The dielectric properties of ε' and tan δ as a function of

TABLE 2.--Dielectric properties of the materials studied at 28°C.

Frequency, MHz:	970		1123		1978		2431		2973	
Sample Tubing diameter, mm.:	0.392	0.538	0.392	0.538	0.392	0.538	0.392	0.538	0.392	0.538
Sample:										
Distilled, deionized water										
ε'	73.8[a]	69.4	76.9	79.9	68.8	81.0	76.1	74.3	75.3	75.1
ε"	3.4	—	9.8	7.1	3.6	5.2	7.6	7.2	9.4	10.5
tan δ	0.046	--[b]	0.128	0.089	0.053	0.064	0.100	0.096	0.124	0.140
	(71. , 3.4, 0.046)		(78.4, 8.5, 0.109)		(74.9, 4.4, 0.058)		(75.2, 7.4, 0.098)		(75.2, 9.9, 0.132)	
Sodium chloride										
ε'	62.3	60.7	148.9	43.5	45.4	66.2	59.9	61.7	61.0	55.4
1.0M ε"	138.4	160.6	292.9	85.8	79.2	81.8	67.5	66.1	64.9	64.8
tan δ	2.222	2.646	1.966	1.971	1.745	1.236	1.127	1.071	1.064	1.168
	(61.5, 149.5, 2.434)				(55.8, 80.5, 1.491)		(60.8, 66.8, 1.099)		(58.2, 64.8, 1.116)	
ε'	72.5	58.0	148.9	40.5	43.1	56.4	51.1	48.2	53.8	50.9
1.5M ε"	215.3	216.9	408.6	110.2	114.9	122.6	91.0	78.8	81.0	75.6
tan δ	2.972	3.741	2.744	2.723	2.663	2.173	1.781	1.632	1.506	1.485
	(65.2, 216.1, 3.356)				(49.8, 118.8, 2.418)		(49.7, 84.9), 1.707)		(52.4, 78.3, 1.495)	
ε'	72.5	52.6	138.5	43.5	32.8	49.9	41.7	50.1	48.4	48.3
2.0M ε"	262.4	279.9	510.6	133.0	130.8	155.3	106.7	104.3	98.6	89.7
tan δ	3.62	5.325	3.687	3.055	3.982	3.112	2.557	2.082	2.038	1.856
	(62.5, 271.1, 4.473)				(41.4, 143.0, 3.547)		(45.9, 105.5, 2.320)		(48.4, 94.2, 1.947)	
Dextrose										
ε'	72.8	74.3	75.5	76.9	89.3	78.6	71.7	73.1	64.1	71.0
10% ε"	--	0.26	3.2	9.3	9.0	8.0	9.2	10.8	11.2	11.9
tan δ	--	0.003	0.043	0.121	0.101	0.101	0.129	0.148	0.174	0.167
	(73.5, 0.26, 0.003)		(76.2, 6.3, 0.082)		(84.0, 8.5, 0.101)		(72.4, 10.0, 0.138)		(67.6, 11.5, 0.171)	
ε'	72.8	74.3	75.5	73.8	71.7	66.8	64.8	70.8	64.1	66.7
20% ε"	--	1.83	8.9	6.3	11.9	12.9	12.3	12.5	13.6	14.2
tan δ	--	0.025	0.118	0.085	0.166	0.194	0.189	0.177	0.213	0.212
	(73.5, 1.83, 0.025)		(74.7, 7.6, 0.101)		(69.2, 12.4, 0.180)		(67.8, 12.4, 0.183)		(65.4, 13.9, 0.212)	
ε'	62.6	68.8	69.7	67.8	54.0	56.6	56.6	63.4	56.1	60.9
30% ε"	6.3	2.5	6.1	9.3	14.8	16.2	15.4	13.3	17.2	16.4
tan δ	0.101	0.037	0.087	0.137	0.275	0.285	0.272	0.209	0.306	0.269
	(65.7, 4.4, 0.069)		(68.7, 7.7, 0.112)		(55.3, 15.5, 0.280)		(60.0, 14.3, 0.241)		(58.5, 16.8, 0.287)	
Maltose										
ε'	62.6	58.0	81.1	79.9	63.5	82.3	76.7	70.1	73.8	76.3
10% ε"	0.69	1.59	5.82	7.7	8.8	7.9	10.6	10.6	12.9	13.3
tan δ	0.011	0.027	0.072	0.096	0.138	0.096	0.138	0.152	0.175	0.174
	(60.3, 1.1, 0.019)		(80.5, 6.7, 0.084)		(72.9, 8.3, 0.117)		(73.4, 10.6, 0.145)		(75.0, 13.1, 0.174)	
ε'	57.4	58.0	69.7	73.8	53.2	81.8	73.7	67.9	67.4	72.1
20% ε"	3.2	0.23	11.5	7.7	8.7	9.4	13.3	12.8	15.3	5.0
tan δ	0.057	0.004	0.166	0.104	0.163	0.115	0.180	0.188	0.227	0.208
	(57.7, 1.7, 0.030)		(71.8, 9.6, 0.135)		(67.5, 9.0, 0.139)		(70.8, 13.0, 0.184)		(69.7, 15.1, 0.217)	

Maltose											
	ε'	62.6	49.8	64.0	67.8	55.4	70.0	73.1	63.4	63.9	66.3
30%	ε"	0.69	5.2	8.7	9.1	11.7	11.0	14.9	16.0	16.9	17.1
	tan δ	0.011	0.105	0.136	0.135	0.210	0.157	0.204	0.253	0.265	0.258
		(56.2, 3.0, 0.058)		(65.9, 8.9, 0.135)		(62.7, 11.3, 0.184)		(68.2, 15.5, 0.229)		(65.1, 17.0, 0.261)	
Dextrin											
	ε'	83.1	66.1	69.7	79.9	53.5	75.7	59.0	72.2	71.8	67.9
10%	ε"	2.9	3.0	11.6	4.7	5.9	4.8	7.5	8.0	8.6	10.0
	tan δ	0.034	0.045	0.167	0.059	0.111	0.063	0.127	0.111	0.120	0.147
		(74.6, 2.9, 0.040)		(74.8, 8.1, 0.113)		(64.6, 5.4, 0.087)		(65.6, 7.8, 0.119)		(69.8, 9.3, 0.134)	
	ε'	72.8	58.0	69.7	79.9	58.6	80.8	55.9	64.3	67.5	63.4
20%	ε"	3.3	0.72	17.3	7.7	8.9	6.3	9.0	8.8	11.1	11.3
	tan δ	0.045	0.012	0.249	0.097	0.151	0.078	0.161	0.137	0.164	0.178
		(65.4, 2.0, 0.029)		(74.8, 12.5, 0.173)		(69.7, 7.6, 0.114)		(60.1, 8.9, 0.149)		(65.5, 11.2, 0.171)	
	ε'	67.7	55.3	69.7	61.7	52.1	63.0	52.0	58.9	62.7	59.1
30%	ε"	11.9	3.9	17.3	--	8.8	7.6	8.9	10.4	12.7	12.6
	tan δ	0.176	0.071	0.249	--	0.170	0.121	0.171	0.177	0.202	0.214
		(61.5, 7.9, 0.123)		(65.7)		(57.5, 8.2, 0.145)		(55.4, 9.6, 0.174)		(60.9, 12.7, 0.20)	
Sodium caseinate											
	ε'	67.7	66.1	69.8	76.9	71.1	69.9	71.4	68.7	69.0	70.2
2.5%	ε"	--	4.1	7.3	11.5	8.5	7.6	5.9	9.1	10.6	12.2
	tan δ	--	0.063	0.104	0.150	0.119	0.108	0.082	0.133	0.153	0.174
		(66.9)		(73.3, 9.4, 0.127)		(70.5, 8.0, 0.114)		(70.0, 7.5, 0.108)		(69.6, 11.4, 0.163)	
	ε'	57.4	60.7	64.0	73.8	66.7	70.3	74.5	67.7	69.6	68.9
5.0%	ε"	2.8	1.4	4.3	13.0	8.5	9.0	6.0	9.2	13.1	12.6
	tan δ	0.049	0.023	0.068	0.176	0.127	0.128	0.081	0.135	0.188	0.182
		(59.1, 2.1, 0.036)		(68.9, 8.7, 0.122)		(68.5, 8.8, 0.128)		(71.1, 7.6, 0.108)		(69.2, 12.8, 0.185)	
	ε'	57.4	60.7	64.0	70.8	66.7	62.0	71.3	64.4	66.1	65.6
10.0%	ε"	11.4	5.5	15.5	14.5	11.0	10.5	7.4	11.0	13.8	13.5
	tan δ	0.199	0.090	0.242	0.205	0.165	0.169	0.103	0.171	0.209	0.205
		(59.1, 8.5, 0.145)		(67.4, 15.0, 0.223)		(64.3, 10.7, 0.167)		(67.9, 9.2, 0.137)		(65.9, 13.6, 0.207)	
Corn Oil											
	ε'	--	--	--	--	8.4	5.5	1.6	2.2	3.3	2.5
	ε"	--	--	--	--	6.0	4.2	1.6	0.7	1.4	1.1
	tan δ	--	--	--	--	0.720	0.778	0.994	0.341	0.421	0.443
						(6.9, 5.1, 0.749)		(1.9, 1.2, 0.667)		(2.9, 1.3, 0.432)	

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[a] The last decimal place may or may not be significant in all figures.

[b] Parenthetical figures are average values of ε', ε", and tan δ, respectively.

frequency and contration from these data are presented graphically in Figures 5 through 14. The corn oil data are not presented graphically, since there appears to be inconsistency in the results.

A few general statements can be made from the data and graphs. In most cases, it can be seen that the higher the solute concentration at a given frequency, the lower is the resulting ε'. This is evident by referring to Figures 5 to 9. This is consistent with the statements by Tinga (1970) that the higher the moisture content the higher the dielectric constant and that the dielectric constant of a mixture will be between the values of those of its components, in this case water and solute.

As seen in Figures 10 to 14, there is a tendency for the loss tangents to increase for the majority of samples at a given frequency as solute concentration increases. There is also a tendency for these to increase as a function of frequency. This again, is in agreement with the latter part of a statement by Tinga (1970) which implies that loss may increase with lower moisture contents.

Pace (1970) in his investigations at 300, 1000 and 3000 MHz concluded that at constant temperatures, the dielectric constants decreased as frequency increased. This is generally the situation which existed in this study if the values at only 1123 and 2973 MHz are considered. At

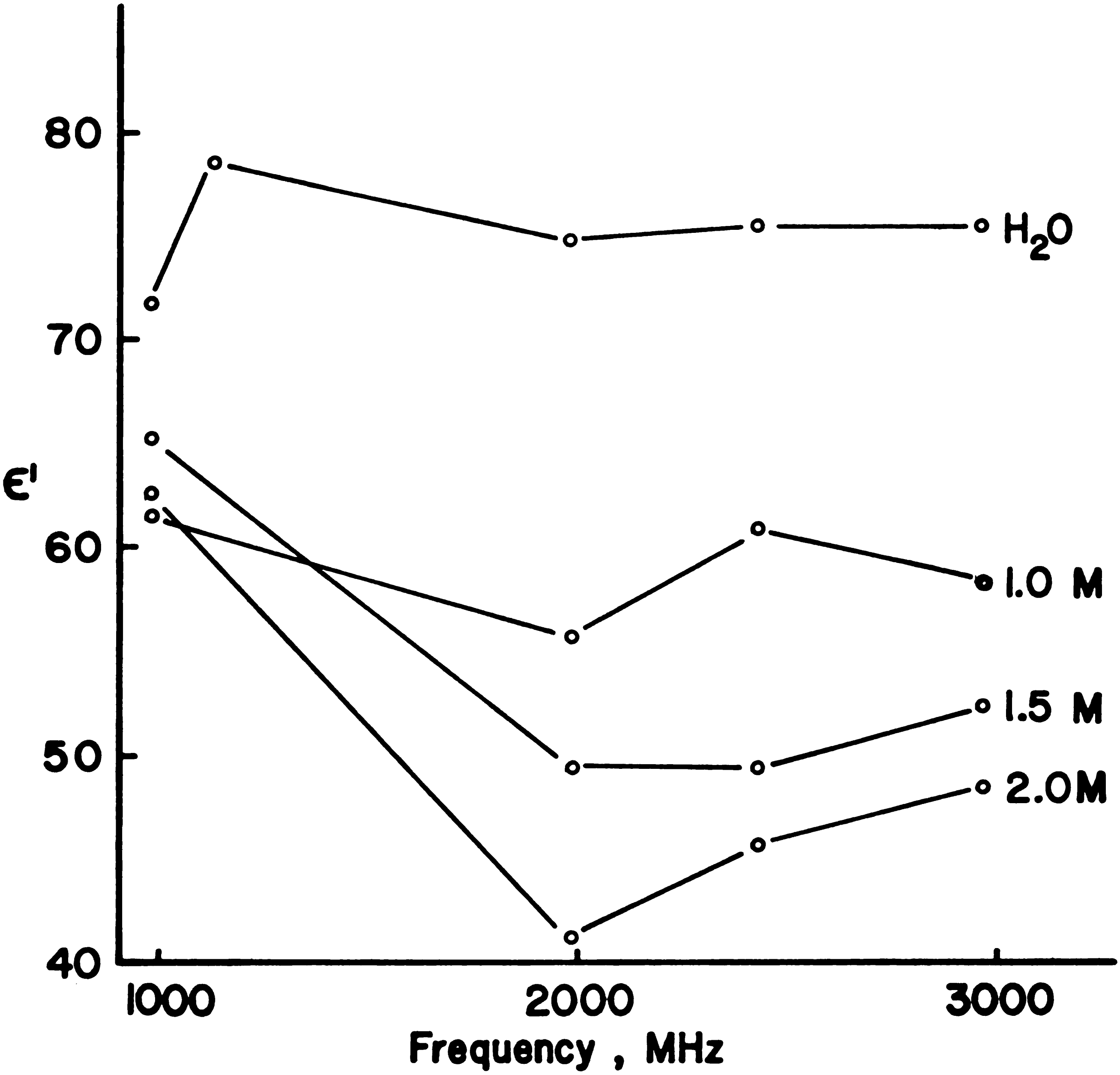

Figure 5. Dielectric constant (ε') as a function of frequency at 28°C. for aqueous sodium chloride solutions.

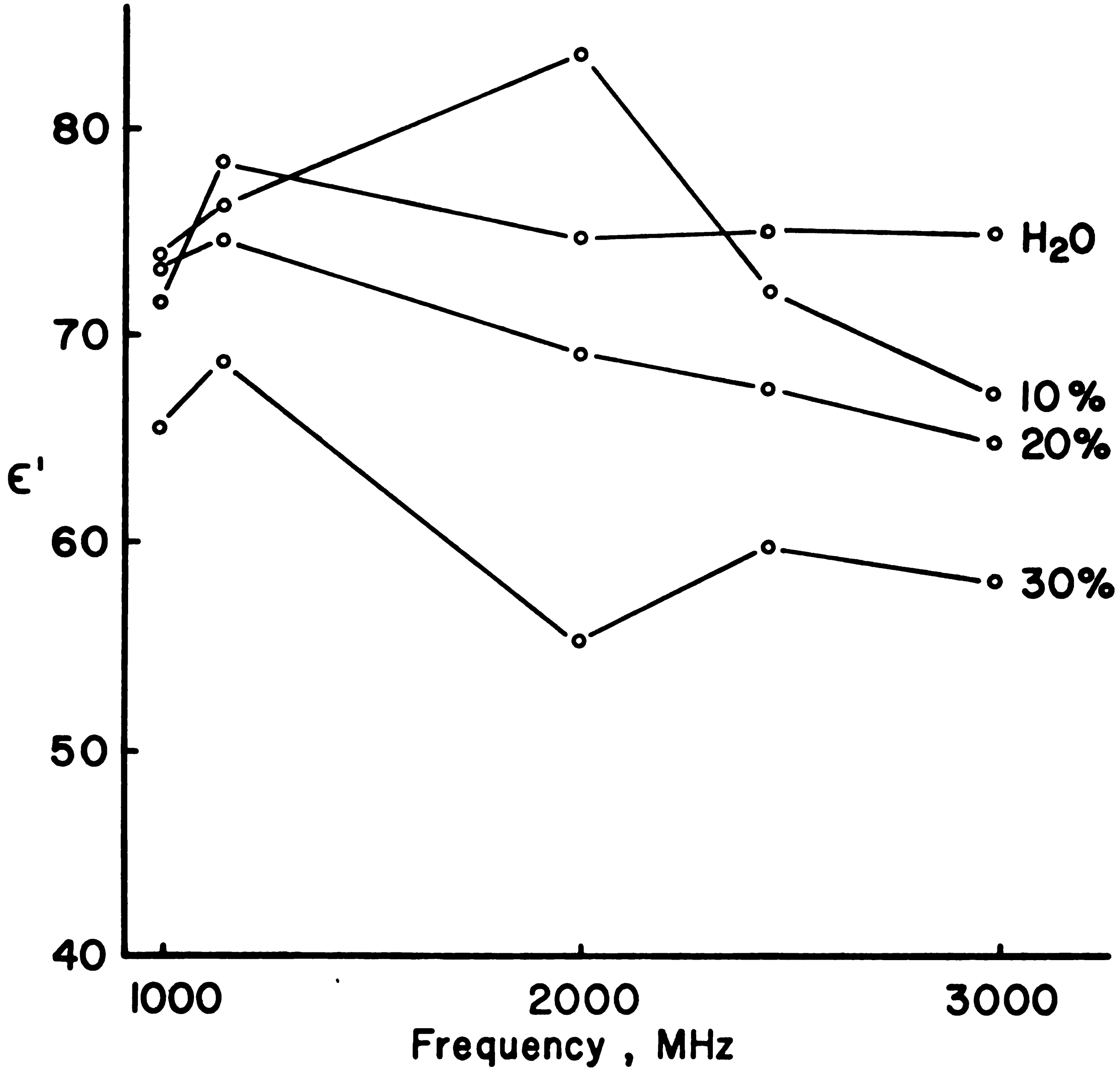

Figure 6. Dielectric constant (ε') as a function of frequency at 28°C. for aqueous dextrose solutions.

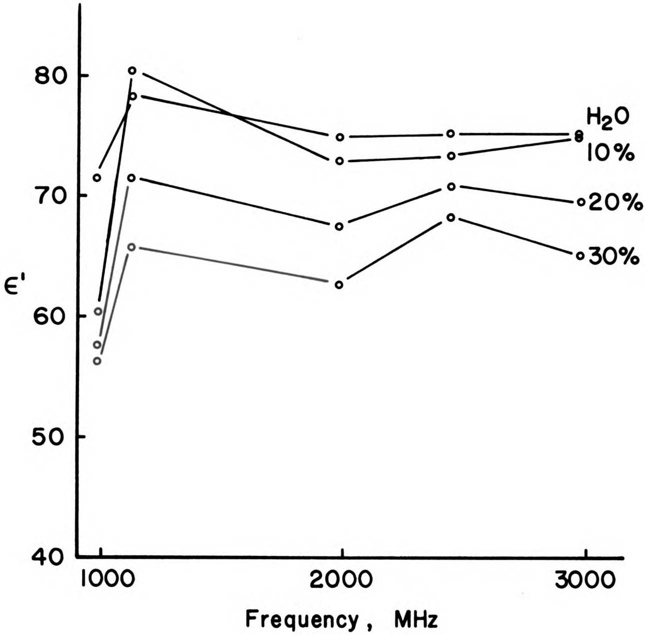

Figure 7. Dielectric constant (ε') as a function of frequency at 28°C. for aqueous maltose solutions.

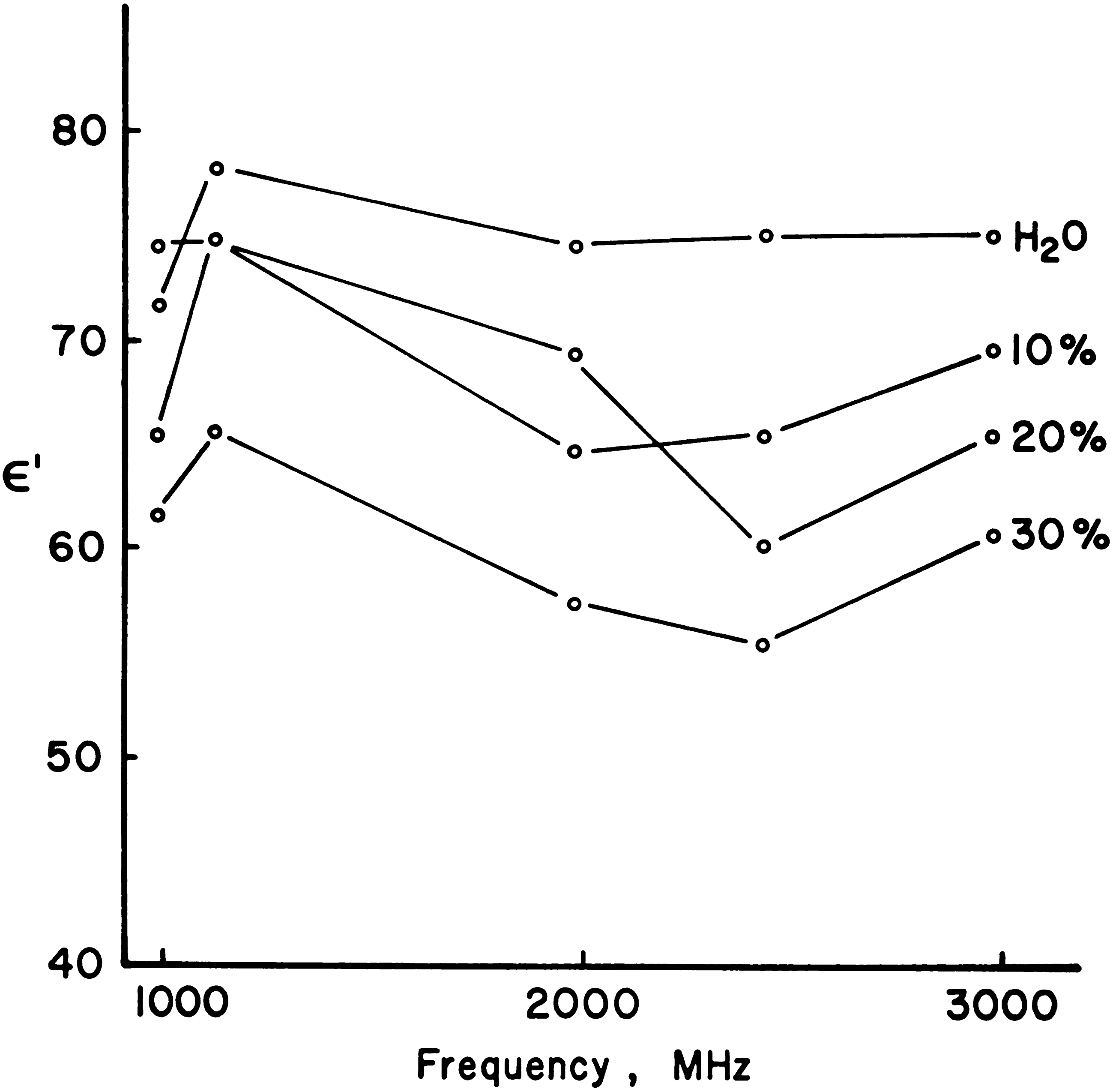

Figure 8. Dielectric constant (ε') as a function of frequency at 28°C. for aqueous dextrin slurries.

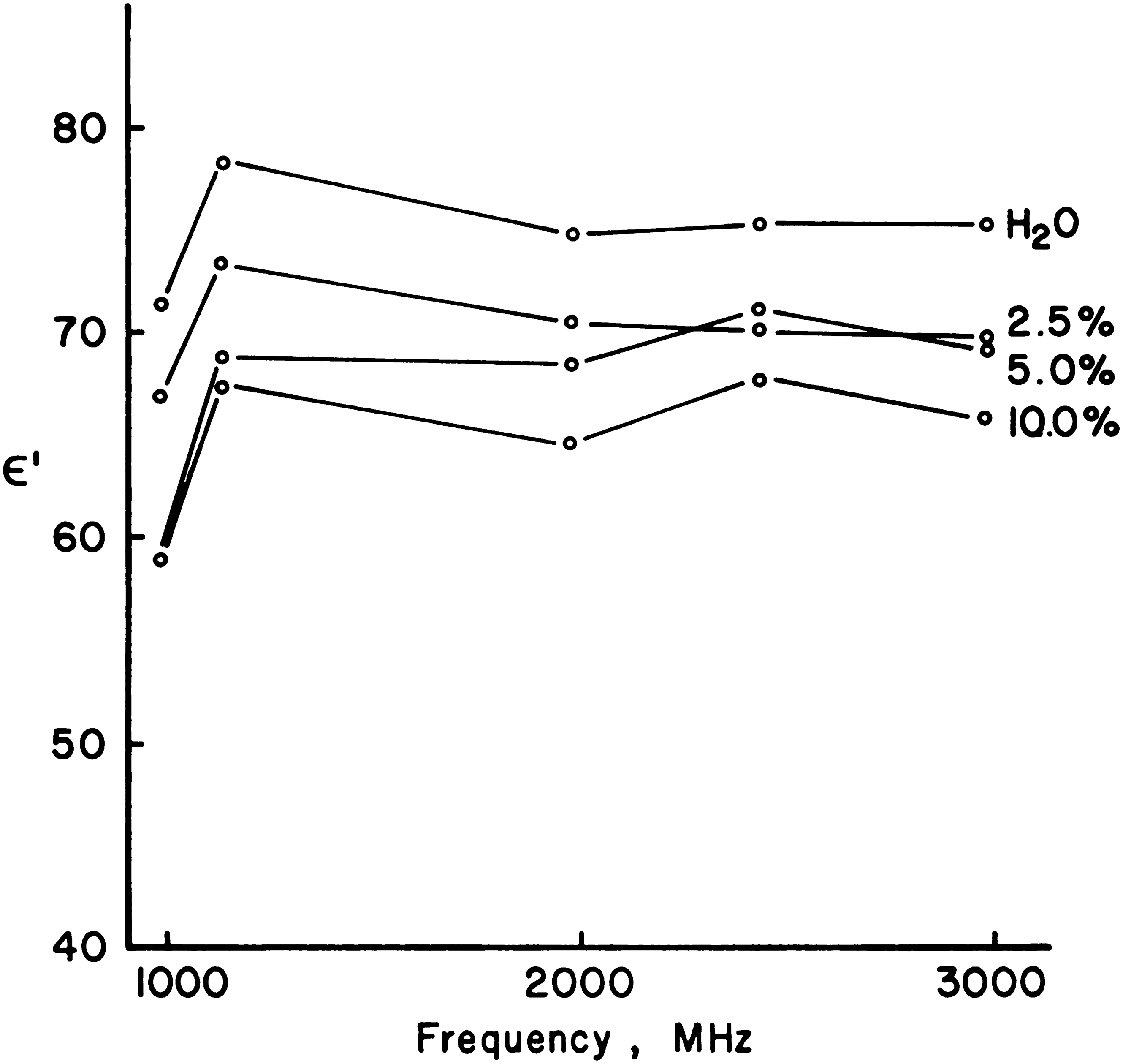

Figure 9. Dielectric constant (ε') as a function of frequency at 28°C. for aqueous sodium caseinate solutions.

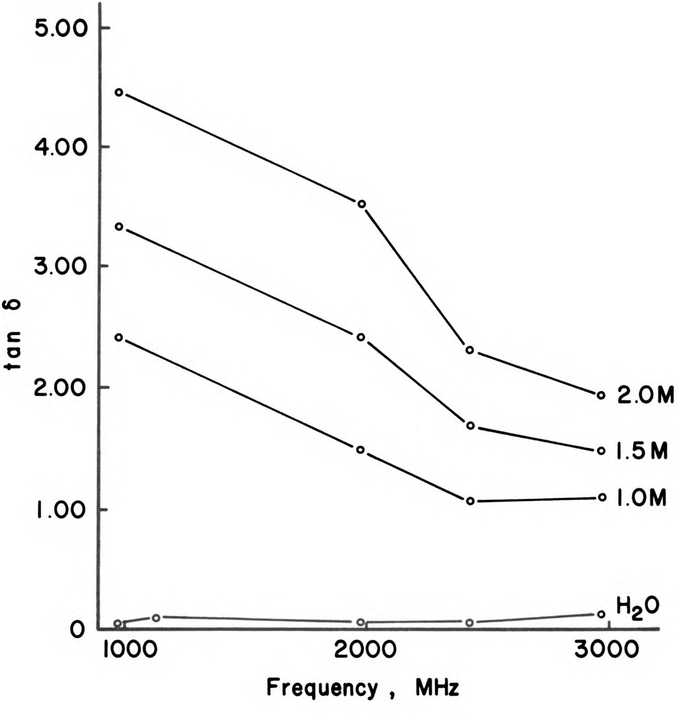

Figure 10. Loss tangent (tan δ) as a function of frequency at 28°C. for aqueous sodium chloride solutions.

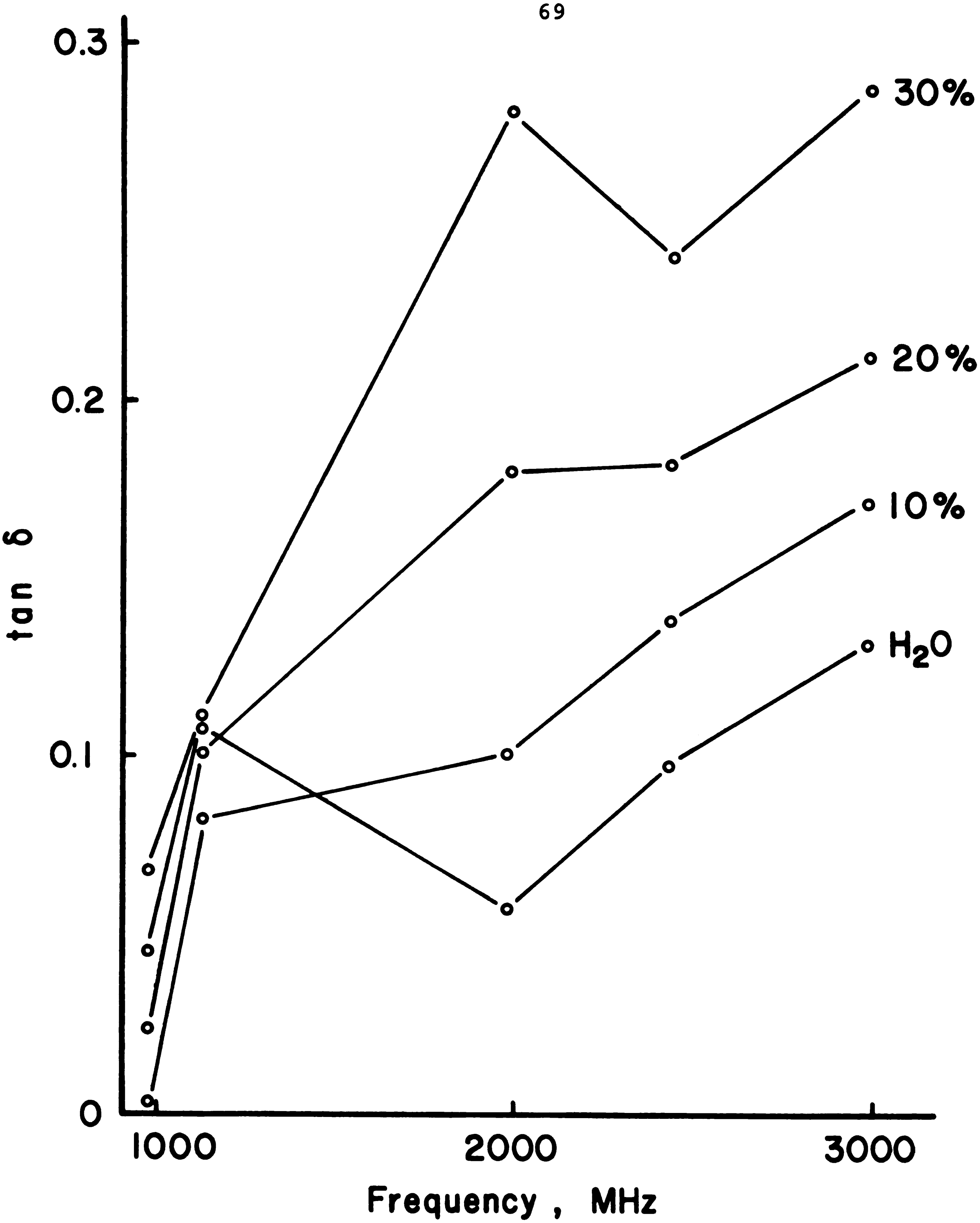

Figure 11. Loss tangent (tan δ) as a function of frequency at 28°C. for aqueous dextrose solutions.

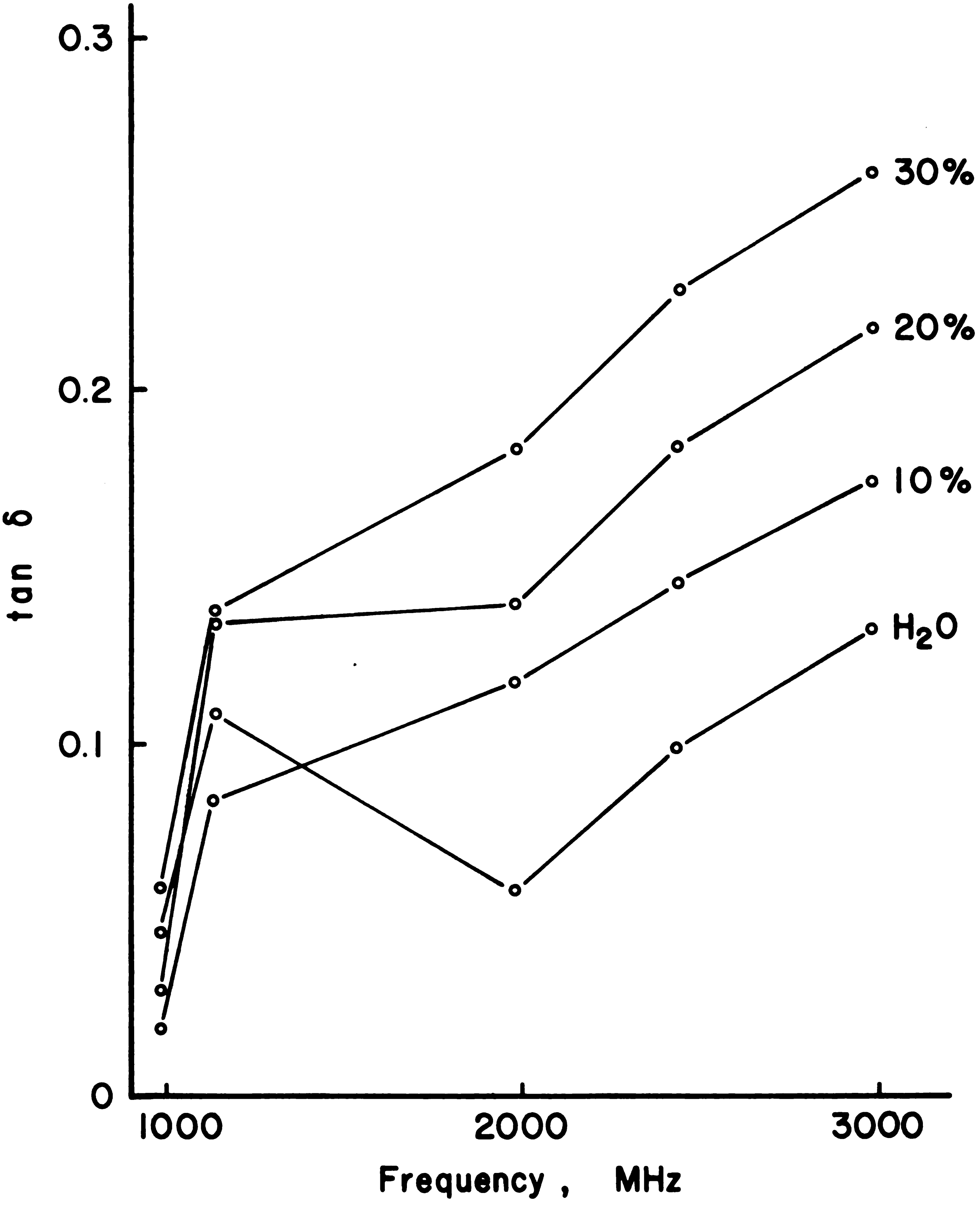

Figure 12. Loss tangent (tan δ) as a function of frequency at 28°C. for aqueous maltose solutions.

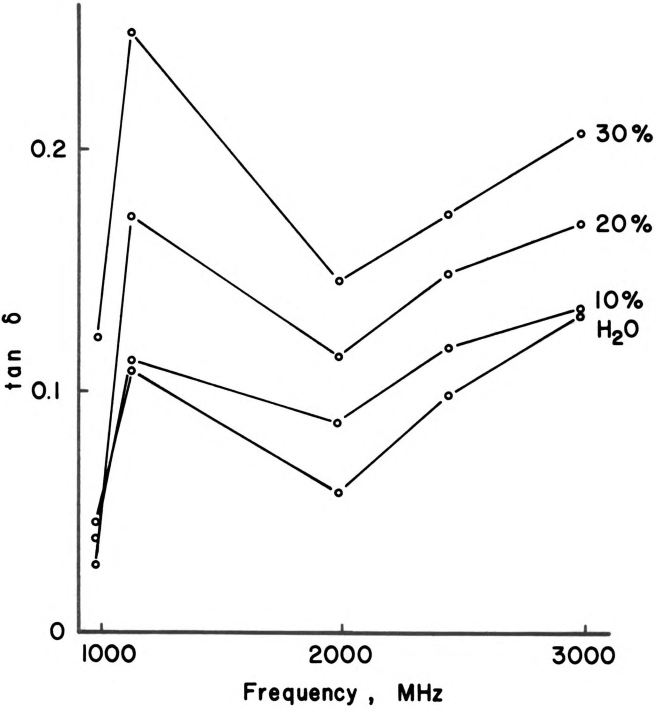

Figure 13. Loss tangent (tan δ) as a function of frequency at 28°C. for aqueous dextrin slurries.

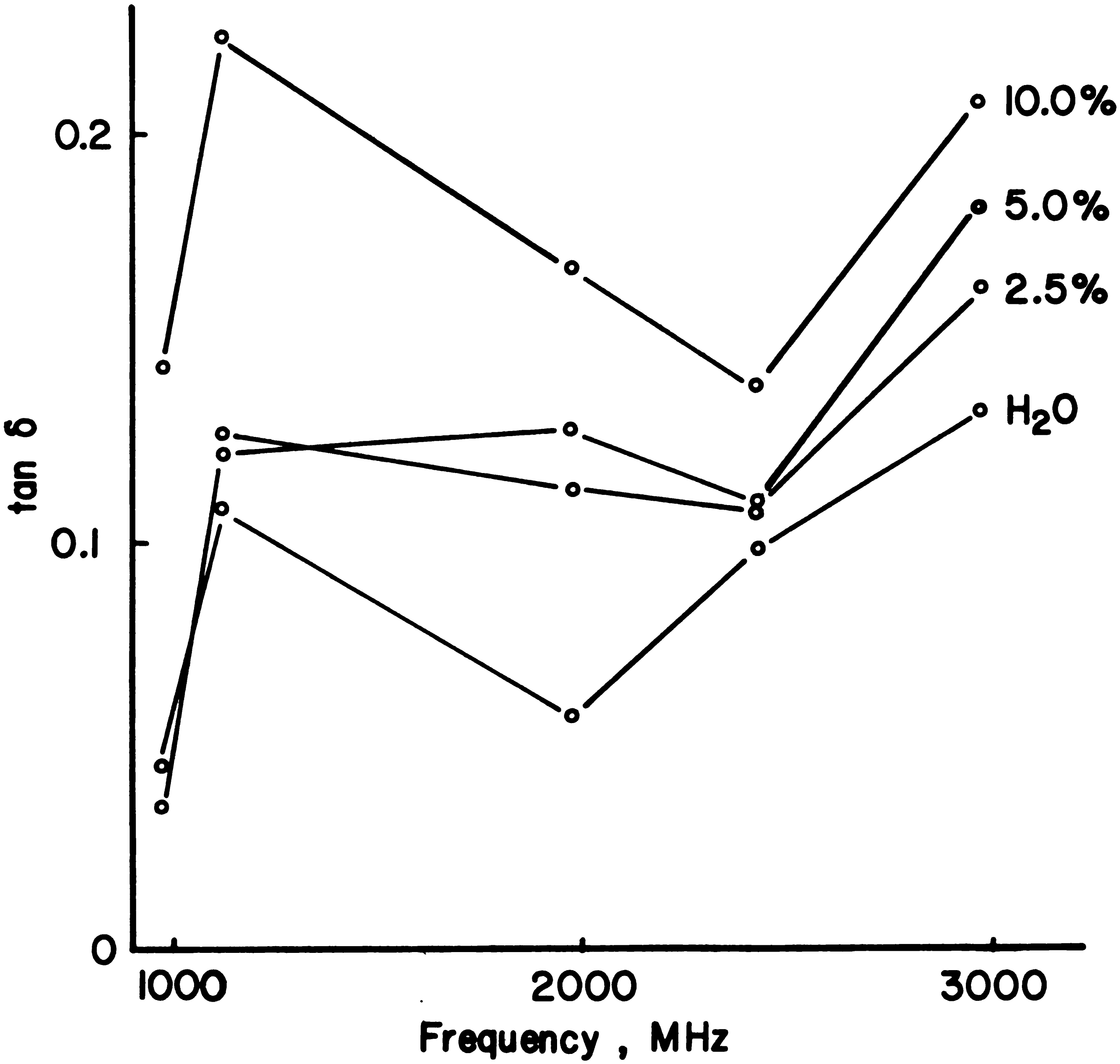

Figure 14. Loss tangent (tan δ) as a function of frequency at 28°C. for aqueous sodium caseinate solutions.

frequencies between these, however, there tends to be some variation. In all cases, it was found that there is an increase of ε' between 970 and 1123 MHz.

The loss tangent decreased with increasing frequency for the solutions containing the electrolyte sodium chloride, but tended to increase for the other non-ionic materials investigated. These results for sodium chloride are in accord with the ground beef studies of Van Dyke (1968). Pace (1967) found that at about 25°C. the loss tangent decreased with increasing frequency from 1000 to 3000 MHz, but at higher temperatures increased within this frequency range.

Turning to more specific findings of this study with the distilled deionized water used as the solvent, there is a slight rise in the ε' value from 970 to 1123 MHz, followed by a slight decline and leveling off from 1123 to 2973 MHz. The tan δ value was also found to increase between 970 and 1123 MHz, followed by a decline and then a rise from 1123 to 2973 MHz.

Of the polar liquids, the dielectric properties of water have received considerable attention. These properties have been measured over a wide range of frequencies and temperatures. It is generally observed that the ε' value decreases with increases in frequency and the ε" and tan δ values increase with increases in frequency, much as is seen in the present study. Values for comparison at the specific frequencies used here are difficult to find in the literature. Also, water of different quality tends to exhibit somewhat different values. Collie, et al. (1948) reported ε' and ε'' values for water of unspecified quality at 20°C. as 77 and 12, respectively, at a frequency of 3000 MHz. Harvey (1963) also reported ε' and ε'' values of 77.2 and 13.1, respectively, for water, again of unspecified quality, at the same temperature and frequency. Von Hippel (1966) reported ε' and $\tan \delta$ values for conductivity water at 25°C. as 76.7 and 0.1570, respectively, at a frequency of 3000 MHz. These values compare favorably with the average values of ε', ε'' and $\tan \delta$ determined herein at 2973 MHz, these being 75.2, 9.9, and 0.132, respectively.

Hasted (1961), in discussing the dielectric properties of electrolyte solutions points out that for years there has been much disagreement, both theoretically and experimentally, in reporting these values. From studies of the literature, it can even be difficult to decide whether the dielectric constant of an ionic solution increases or decreases with increasing concentration. He goes on to state that the explanation of the experimental differences is to be sought in the masking effect of the very large conductivities. The imaginary part of the dielectric constant, ε'', is indistinguishable from the ohmic correction, $2\sigma/f$, where σ is the a.c.

conductivity. Thus at all frequencies below about 3000 MHz the solutions exhibit enormous loss tangents, which make the usual experimental methods inaccurate. The alternatives which remain are the use of methods such as those depending on the forces produced by electric fields, and the use of frequencies above 3000 MHz. The use of these alternatives has also resulted in inconsistency of data, apparently due to the fact that the frequency and concentration ranges studied have been widely different, so that whole regions of dispersion may lie between them.

For this study, it was decided to investigate the dielectric properties of sodium chloride solutions since, like water, electrolyte solutions have been investigated extensively in the past. It was believed that comparison of the values obtained by the method used here with values reported in the literature would give an indication of the validity of the method. Food materials have received little attention in the past, and in applying the present method, it appeared important to establish its validity.

Since it was found that very dilute solutions would give only slight deviations from the dielectric properties of the water, sodium chloride concentrations of 1.0, 1.5, and 2.0 M were used. On the other hand, Hasted, et al. (1948) reported that working with solutions with very high values of tan δ greatly limits the accuracy of the measurement.

The data for the sodium chloride solutions studied here indicate decreasing ε' values with increasing solute concentrations, and decreasing tan δ values with increasing solute concentration at a constant frequency. Hasted, et al. (1948) reported ε' and ε" values for 1.0, 1.5, and 2.0 N sodium chloride solutions at 21°C. and 3000 MHz. The ε' values were 67.4, 61.5, and 56.2, respectively, for each of these solutions. The average ε' values of 58.2, 52.4, and 48.4 determined by the present study compare favorably with these, considering that they were determined at a slightly higher temperature and that with increasing temperature, ε' values tend to decrease. The values for ε" do not agree with those of Hasted, although the tan δ values derived from ε" and ε' appear to fall in a similar range and increase with increasing concentration at 3000 MHz as reported by Okress (1968).

In order to be able to make some general conclusions about the relationship of monosaccharide units in a carbohydrate molecule (in this case, dextrose and its polymers) and the dielectric properties, dextrose, maltose, and dextrin solutions were investigated. Large differences and trends are not apparent from the data. In most cases, for a given frequency, the ε' values decreased as the solute concentration increased. Some deviation from this trend did occur with the 20% dextrin sample, which may have been due to some inhomogeneity of the slurry in the sample tubing during the measurement. The data point for 10% dextrose at 1978 MHz also showed some inconsistency. Repeating the measurements for this point, however, resulted in similar values for ε'. The most reasonable explanation that can be given is that this deviation probably represents an inaccuracy in the method. Because change is occasionally quite minimal, the small frequency shifts which result when the sample is introduced into the cavity are often difficult to pinpoint beyond approximation. As will be mentioned later, the use of a digital frequency meter, rather than the cavity type used, would tend to reduce such inaccuracy.

For a given frequency, the tan δ values generally increased as the solute concentration increased. This is opposite to what was seen with the sodium chloride solutions and may reflect the conductivity effects mentioned previously in connection with electrolyte solutions. For the most part, dextrose and dextrin had higher ε' values for the lower frequencies of 970 and 1123 MHz, whereas maltose showed little variation of ε' value with frequency, except at 970 MHz where the ε' values were lowest. Dextrose and maltose, on the other hand, had higher tan δ values at the three highest frequencies. Dextrin, also, tended to have higher tan δ values at the highest frequencies (except for the data at 1123 MHz), but not as high as dextrose and maltose.

As stated, it was hoped that greater differences might have been found among the three carbohydrates. As might be expected, the ε' values were essentially less than those of water at the various frequencies and the loss tangents greater than those of the water. Dextrose and dextrin solutions resulted in greater differences from water with respect to their ε' values, whereas the dextrose and maltose solutions resulted in greater differences from the water with respect to the tan δ values, especially at the higher frequencies.

Sodium caseinate was chosen for study, since it is a common protein supplement to many manufactured foods. Its dielectric properties in aqueous solutions might reflect the type of response a food containing it might have in a microwave field. The change of ε' of sodium caseinate solutions with frequency was similar to that of water; that is, a slight rise between 970 and 1123 MHz. followed by a slight decline and tendency to level off from 1123 to 2973 MHz. Again, at a given frequency, the ε' values tended to decrease as solute concentration increased. The tan δ values, also at a given frequency, showed variations similar to that of water, although they were higher for the protein solutions and showed an increase as protein concentration increased. The ε' and tan δ values both tended to be higher for all concentrations at the four highest frequencies. The ε' values appear to almost vary linearly with protein concentration at a given frequency.

Rosen (1966) indicates that few measurements of the dielectric properties of protein solutions have been made in the microwave range. He points out, however, that the electrical properties of molecules to some extent influence molecular interactions and are probably of particular significance in the interactions of macromolecules, such as protein. Full knowledge of these factors is important if the behavior of proteins and other macromolecules is to be fully understood. Buchanan, et al. (1952) studied the high frequency dielectric properties of six aqueous protein solutions (not including sodium caseinate) in terms of water "irrotationally bound" to the protein. In their study, attempts were made to estimate the maximum amount of bound water as a function of axial ratio of the protein assuming spheroid molecules. One of the frequencies employed had a wavelength of 9.22 cm., (about 3250 MHz), which closely corresponds to the frequency of 2973 MHz used in the present study (λ = 10.1 cm.). Their values of ε' and ε" for the six proteins at this frequency and 25°C. compare very closely with those found herein. For instance, horse methemoglobin at pH 7 and concentrations of 3.4, 6.7, and 10.1% resulted in ε' values of 72.5, 69.2, and 67.0, respectively, and ε" values of 12.1, 12.3, and 11.7, respectively. The results of the present study with sodium caseinate at pH 8.0 and concentrations of 2.5, 5.0, and 10.0% gave average ε' values of 69.6, 69.2, and 65.9, respectively, and average ε" values of 11.4, 12.8, and 13.6, respectively. Moreover, the results of Buchanan, et al. (1952) indicate a decline in ε' values with increases in frequency (for frequencies of 3000 MHz and higher) and a decline of ε' values with increasing protein concentration at constant frequency, much as noted with the present study. Their ε" values tended to increase with frequency, also, as observed in this present study. These same values for Buchanan, et al. (1952) decreased with increasing protein concentration at constant frequency which was the reverse of that noted here. This latter finding may have been due to the lower frequencies used in this study.

Measurements were made using the corn oil to determine whether the method, which had thus far been found to correlate with literature values for high loss materials, would be equally valid for what is reportedly (Pace, 1967) a low loss food material. Based on the observations of Dunsmuir and Powles (1946) using non-polar organic liquids of low ε' and tan δ values, the resonant cavity method should prove reliable. However, they made their measurements using sample tubes of much larger diameter, which were not readily available for the present study. The dielectric properties of the corn oil, characterized by small ε' and ε" values, combined with the small diameter of the sample tubing did not provide sufficient perturbation of the cavities to insure accurate measurements, especially with those cavities at 970 and 1123 MHz. The ε' values at 2431 and 2973 MHz appear more favorable compared with the findings of Pace (1967). However, the ε" and tan δ values seem much too high.

As discussed previously, the resonant cavity perturbation technique for measurement of dielectric properties proved advantageous with respect to the simplicity of the measurements and of the equations involved for arriving at the dielectric properties. Handling of the samples also proved to be an easy matter. This was in contrast to a transmission line method which had been employed initially. The transmission method, although adapted to handle the liquid samples, proved difficult with respect to measurement and calculation of the dielectric samples and was thus discontinued. The cavity perturbation technique, as was employed, does have its limitations as found with the measurements on corn oil of low ε' and tan δ values. To be able to have achieved a measureable shift in the resonant frequency from its unperturbed value for determination of ε' would have required a large diameter sample tubing. This same size tubing, however, would have resulted in too large a shift in the Q value (beyond the perturbation theory). Thus it would have required two sizes of sample tubing, larger than those used here, but different from each other to make an assessment of the dielectric properties of the oil. As it turned out, the lower actual Q factors of the cavities from the calculated Q factors allowed for greater ease of measurement of small Q factor differences with the other samples.

All in all, the method proved satisfactory when working with the other samples. Generally good agreement was found between the two sizes of sample tubing, especially at the higher frequencies, giving some indication of the validity of the theory when working with liquids of high loss. This leads to some suggestion of improvement of the method over what was actually used for this study. For one, the use of a digital frequency meter (not available for this study) would have allowed greater accuracy in reading frequency shifts due to the presence of the sample in the cavity. Secondly, the use of a sweep oscillator at the two lower frequencies (again, not available for this study) as was used at the three highest frequencies would lead to more accurate measurements. It appears from the data that greater consistency of results was achieved using the sweep oscillator. Some of the deviations of the data, especially for the PE 20 and 50 tubing at 1978 MHz, may have been due to the fact that this frequency was at the lower limit for the sweep oscillator. A third suggestion concerns the square law detector crystal which was used. The Q response for the cavities is dependent on the square law detector crystal, which squares the voltage signal from the cavity and thus displays this voltage as power (power is proportional to the square of voltage) on the standing wave meter or oscilloscope, depending on which circuit was used. One problem occurs if the voltage is too high and thus drives the crystal beyond square law detection (a case which would result if the sample size is too large). A second problem is that the detector crystal used had a detection limit range anywhere from a factor of about 1.5 to about 2.5 so that an actual squaring of the voltage may not have taken place. It is thus recommended to use a true square law detector or, better still, a power meter to indicate the power.

It is difficult to assess the method for error. There are errors due to the sample size, the measurements themselves, and the equipment, all of which have been alluded to. Horner, _et al_., (1946), attempted to assign some measure of accuracy to the resonant method using a TM_{010} mode cavity for solid rod shaped samples of low dielectric properties. Their estimates of accuracy were based on assigning standard deviations to each of the parameters affecting the final results for the dielectric constant and the loss tangent. These standard deviations were based on a series of measurements of the parameters and calculations of the corresponding standard deviations of the dielectric constant and loss tangent. They assumed a normal distribution of errors with 50% of the results having an error less than 0.675 times and 95% of the results having an error less than 1.96 times the standard deviation. Their analysis indicated that the limiting factor with respect to the dielectric constant, ε', is the accuracy with which the samples are prepared and their diameters measured. They arrived at a total standard deviation of about 0.15% for all ε' determinations.

Concerning the loss tangent, tan δ, the dominant source of error lies in the Q measurement on the cavity containing the sample. Again, the analysis by Horner, et al., (1946), indicates that the standard deviation of loss tangent measurements may be expected to be less than 2%.

As mentioned previously in the methods and materials section, the degree of inaccuracy introduced by the approximations used for ε' and tan δ is less than 1% for values of $\varepsilon_b/\varepsilon_a$ less than 4 and of b/a less than 1/50. These conditions are met with the method used for this investigation. Dunsmuir and Powles (1946) tested these approximations on a series of non-polar organic liquids at frequencies of 663, 1290, 3210, and 3490 MHz, respectively, and different sample sizes. Good agreement was found between the values obtained for ε' and those available from the literature. They estimated the actual accuracy of the tan δ values at the two higher frequencies to be ± 5% and ± 10% at the two lower frequencies. Greatest accuracy of the tan δ measurement was achieved when introduction of the liquid sample reduced the Q factor by about one-third. It was necessary for lossy liquids, therefore, to use very small sample containers, which decreases the importance of losses in these containers.

SUMMARY AND CONCLUSIONS

The dielectric properties of water, aqueous solutions of food or food related materials, and corn oil were determined at microwave frequencies of 970, 1123, 1978, 2431, and 2973 MHz and 28°C. The method employed for these determinations was a resonant cavity perturbation technique which, previously, had been applied only to a limited degree with such systems.

It was found through this investigation that the resonant cavity perturbation technique as employed is apparently valid. The results for dielectric properties of materials studied compare favorably with those reported for the same, or similar, materials in the literature.

It was generally found, for all solutions studied, that the dielectric constant, ε', decreased as solute concentration increased at given frequencies.

ε' values of the solutions tended to be lower than those of the water used throughout. Some deviations from this trend are discussed, notably with the 10% dextrose solution at 1978 MHz, and must be ascribed to an error inherent in the method under the conditions used. Such

conditions made it difficult to ascertain a frequency shift when sample was introduced into the test cavity.

The 20% dextrin slurry similarly resulted in a noticeable variation from the trends at this same frequency. This may have been due to inhomogeneity of the slurry in the sample tubing. It should also be pointed out that deviations that were found to occur at 1978 MHz may have been due, at least in part, to the fact that this frequency was at the lower limit of the sweep oscillator used for these measurements.

Loss tangents, tan δ, were found to increase for the majority of samples at given frequencies as solution concentration was increased and, also, to increase as a function of frequency.

Very little difference was found among the results of the solutions of the three carbohydrates under the conditions investigated. Thus, the conclusion which can be made from this study is that the effect the number of monosaccharide units in a carbohydrate might have on the dielectric properties as measured is negligible.

The results for the sodium caseinate solution agreed with the major trends found in this investigation as well as with published results of other protein studies at microwave frequencies.

That part of the study dealing with the dielectric properties of corn oil was unsuccessful. As pointed out, the dielectric properties of this type of material are too low to be reliably determined under the conditions used. The sample tubing was too small to allow a measureable perturbation of the cavities, particularly at 970 and 1123 MHz, and the method of measuring the frequency shift due to presence of the sample in the cavity was not critical enough when working with corn oil.

This research focused on only a few variables of some relatively simple systems, but it is representative of the type of research which must be conducted if the microwave food process industry is to explore, develop, and eventually achieve its potential.

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