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RESEARCH MEMORANDUM

STUDY OF SOME DIELECTRIC PROPERTIES OF SUSPENSIONS OF
MAGNESIUM PARTICLES IN MINERAL OIL

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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

RESEARCH MEMORANDUMSTUDY OF SOME DIELECTRIC PROPERTIES OF SUSPENSIONS OF
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SUMMARY

The variation of dielectric constant has been measured as a function of the concentration of magnesium particles; the shape, size, and degree of oxidation of the particles; the temperature; and the frequency of oscillation. The variation of dielectric constant and settling level was investigated as a function of time. Also investigated were the effects of particle concentration, particle shape, and time on dielectric losses.

The two variables of primary importance affecting the dielectric constant were the concentration of the magnesium particles and the shape of the particles. Corrections for variations in temperature and degree of oxidation were made possible by the information gained. The dielectric constant was shown to be independent of particle size. The effects of oxide upon the variation of dielectric constant with frequency and the dielectric losses are discussed. Two processes, one consisting of dilution, the other of compression of particles, were advanced to explain the variation of dielectric constant with time as settling proceeds.

INTRODUCTION

Subject and Purpose of Investigation

Jet fuels consisting of suspensions of magnesium and boron particles in hydrocarbons are being studied at the NACA Lewis laboratory. Because such fuels have decided advantages over hydrocarbons alone (refs. 1 and 2), it is of considerable importance that careful investigations of their properties be made in order to obtain information which will be of use in the preparation of stable, freely flowing, and highly combustible fuels of this type. The present work is a continuation of the studies (ref. 3) of physical properties of such suspensions now being carried out at this laboratory.

Only a limited number of physical methods are available for the investigation of concentrated suspensions of metals (or nonmetals) in hydrocarbons. The best known of these methods are viscometry and settling rate measurements. While both of these methods are of considerable usefulness, they also have serious limitations.

Viscometry by its very nature is limited to systems under the influence of external shearing forces. These forces are of considerable importance in determining the motion of the suspensions through fuel systems or in the settling of particles. At the same time, however, little knowledge may be gained as to the rest behavior of suspensions.

Knowledge of the settling levels of particles in a suspension is also useful (ref. 3). No information is available as to the state of the system when such measurements are carried out before visible settling occurs. Furthermore, concentration gradients within unsettled and partly settled suspensions are not obtainable from simple settling-level measurements.

Another source of information lies in microscopic observations. Again, however, the study of dry powders or of particles in liquid films may fail to supply information upon the rest behavior of suspensions in bulk.

A method or methods which might give further information regarding the rest behavior of suspensions as well as the behavior under external shearing forces would be highly desirable. The study of the fundamental dielectric behavior of suspensions gives information upon the rest behavior of suspensions and, furthermore, offers possibilities of yielding information about the concentration gradients of the particles and the degree of flocculation in suspensions.

Previous Investigations

The dielectric constants of particles of such materials as potassium chloride crystals, lead chloride particles, particles of Wood's metal, and so forth, in such mediums as air, carbon tetrachloride, castor oil, and lubricating oil have been studied (ref. 4). From these studies it was concluded that the dielectric constant at a given volume fraction of suspended material depended upon the particle shape. The greater the deviation from spherical shape, the higher the dielectric constant is found to be for a random distribution of particles. The experimental results were compared with the various theoretical formulas which were available at the time, and it was concluded that the theoretical equation given in reference 5 was the most satisfactory. This equation has the form

$$1 - V = \frac{\epsilon_1 - \epsilon}{\epsilon_1 - \epsilon_2} \sqrt[3]{\frac{\epsilon_2}{\epsilon}} \quad (1)$$

where

- ϵ dielectric constant of dispersion
 ϵ_1 dielectric constant of dispersed material
 ϵ_2 dielectric constant of dispersing medium
 V volume fraction of solids

The equation holds only for spherical particles. For metallic spheres dispersed in nonpolar liquids, where $\epsilon_1/\epsilon_2 \rightarrow \infty$,

$$\epsilon = \epsilon_2(1 - V)^{-3} \quad (2)$$

which expanded in series form is

$$\epsilon = \epsilon_2(1 + 3V + 6V^2 + 10V^3 + \dots) \quad (3)$$

For low concentrations of the dispersed material,

$$\epsilon = \epsilon_2(1 + 3V) \quad (4)$$

Investigations have been carried out on the dielectric behavior at rest and under shear of suspensions of titanium dioxide, zinc, copper, iron, and carbon black particles in mineral oil and castor oil (ref. 6); and some measurements have been made upon paint pigments in similar vehicles (ref. 7). The behavior of the suspensions at rest and under shear was discussed in terms of modifications of equation (4). In reference 7 more recent theoretical work (ref. 8) which results in the limiting equation (4) also is mentioned. The modifications made result in a semiempirical equation:

$$\epsilon = \epsilon_2(1 + 3a_v fV) \quad (5)$$

where

- a_v agglomeration factor
 f shape factor

Equation (5) attempts to represent the behavior of nonspherical particles with a shape factor f which may form agglomerates of particles.

The theoretical equation given in reference 8 for small concentrations of spherical nonmetallic particles has the form

$$\epsilon = \epsilon_2 \left(1 + 3V \frac{\epsilon_1 - \epsilon_2}{\epsilon_1 + 2\epsilon_2} \right) \quad (6)$$

For higher concentrations of metallic spheres, the theoretical equation in reference 8 has the form

$$\epsilon = \epsilon_2 (1 - 3V)^{-1} \quad (7)$$

or expanded in series form is

$$\epsilon = \epsilon_2 (1 + 3V + 9V^2 + 27V^3 + \dots) \quad (8)$$

The preceding equation is based on different and probably more reasonable theoretical assumptions than equations (1) to (3). It may be seen that equation (7) would predict higher values of ϵ than equation (3) at concentrations where quadratic terms in V become important. Theoretical equations have also been derived for ellipsoidal particles (ref. 9).

Measurements have recently been made (ref. 10) on the dielectric constants and especially on the dielectric losses and conductivity of dilute suspensions of silica, iron, and carbon black particles as a function of shear, temperature, and frequency.

For spherical particles in suspension, the dielectric constant at a frequency f is related to the frequency at $f = \infty$ (infinite frequency) by the expression

$$\epsilon = \epsilon_\infty \left(1 + \frac{K}{1 + \omega^2 T^2} \right) \quad (9)$$

where

ϵ dielectric constant at a frequency f

ϵ_∞ dielectric constant at infinite frequency

ω $2\pi f$

$$K = \frac{9V\epsilon_2}{2\epsilon_2 + \epsilon_1}$$

$$T = \frac{2\epsilon_2 + \epsilon_1}{4\pi\sigma_1}$$

σ_1 conductivity of dispersed material

The loss angle or dielectric loss is given by

$$\tan \delta = \frac{K\omega T}{1 + K + \omega^2 T^2} \quad (10)$$

For nonspherical particles the analysis results in more complicated expressions (ref. 11) involving the elliptic integrals. Qualitatively, the dielectric constant and the dielectric loss should increase considerably in magnitude with increasing deviation from spherical shape.

The preceding equations are formally in agreement with those derived for polar liquids (refs. 12 and 13). Furthermore, since a dielectric sphere behaves like a dipole when acted upon by an external field, the resemblance is very close.

Scope of Present Investigation

In this investigation a considerable number of factors which were thought to be of importance in gaining fundamental information concerning the behavior of magnesium suspensions in mineral oil were studied. The variation of the dielectric constant and the dielectric loss was measured as a function of the concentration of magnesium particles, the temperature of the suspension, and the frequency of the alternating field. Since magnesium, especially in the finely divided state, is readily oxidized, the variation of the dielectric constant was measured when the oxide content was changed. The influence of particle size and shape upon the dielectric constant of magnesium - mineral oil suspensions was also determined. Finally, the dielectric constant was measured as settling proceeded in the cell. Settling rate measurements were made simultaneously in glass cylinders by reading the level to which the suspension had settled.

EXPERIMENTAL PROCEDURE

Sources and Description of Materials

The benzene, hexane, heptane, cyclohexane, methylcyclohexane, and toluene employed in the calibration and determination of accuracy and reproducibility of the dielectric-constant cells were purified by distillation through the Podbielniak columns at the NACA Lewis laboratory. The densities and refractive indices agreed within a few units in the fourth decimal place with National Bureau of Standards values (ref. 14).

Two samples of purified white mineral oil were used. The first sample had a density of 0.8714 gram per milliliter and a refractive index of 1.4762 at 20° C. The dielectric constant of this sample was 2.170 at 20° C and 2.137 at 50° C, and its Brookfield viscosity was about 36 centipoises at 30° C. The second sample had a density of 0.8805 gram per milliliter and a dielectric constant of 2.184 at 20° C. Its viscosity was about 100 centipoises at 30° C.

All the magnesium used was obtained from Golwynne Chemical Corporation and ranged in average particle diameter from 23 to 1.5 microns. The diameters were determined by an air permeability method (Fisher Sub-Sieve Sizer). A Roller analysis of the 14-micron powder is given in table I. The oxide contents were determined for each can of magnesium employed for the dielectric-constant determinations. The purest magnesium used was 98 to 99 percent, but in most cases the purity was in the range from 90 to 95 percent because of air oxidation. For the study of the effect of oxide content upon dielectric constant, 92 percent pure magnesium powder was obtained from 98 percent pure magnesium powder by allowing the latter to stand in a gallon can for about three months with the lid on but not tightly sealed.

Preparation of Suspensions

The suspensions were prepared by weighing first the magnesium and then the mineral oil into a beaker on a balance sensitive to 0.1 gram. The total weight was usually made up to 300 grams. A portion of this was used to fill the dielectric-constant cell and the remainder to fill the settling rate tube. The materials were vigorously stirred in the beaker with careful scraping of the sides of the beaker to remove any magnesium clinging to the glass. The material was stirred until it appeared smooth and homogeneous, allowed to stand for a few minutes, and stirred again. The material was poured into the measuring cell 15 to 20 minutes after mixing was begun. Some difficulty in mixing was encountered with the finest materials, less than 2 microns in diameter. Agglomerates of magnesium particles of this size range were sometimes difficult to disperse.

DIELECTRIC-CONSTANT APPARATUS

Description of Apparatus

3059 A bridge-type apparatus was employed in making the dielectric-constant and dielectric-loss measurements. The bridge used was a Schering capacitance bridge. The substitution method was used whereby the capacity C_X introduced by the measuring cell was removed by reducing the capacity of the precision condenser C_N in parallel with the measuring cell. The resistive component R_X was balanced out by C_B (fig. 1). This bridge can be balanced accurately up to 1 megacycle and can be used with resistances between 3×10^3 and 8×10^{11} ohms, the limits depending upon the frequency.

The generators used were R-C oscillators. One of the oscillators has a frequency range of 7 to 70,000 cycles per second, the other oscillator used has a range from 20 to 200,000 cycles. Since the variable precision condenser C_N within the bridge is corrected for worm-gear eccentricity at 1000 cycles per second, this frequency was used except when the effects of frequency upon dielectric constant were being determined.

The output from the oscillator was fed into the Schering bridge. The bridge output was then amplified by a decade amplifier and filtered either by a 1000-cycle-per-second band-pass filter or a bridged-T filter with 60-cycle-per-second reject. The detector was a vacuum-tube voltmeter. A stainless-steel sheet was placed under the entire apparatus to serve as a ground.

Dielectric-Constant Cells

The cells usually used in dielectric-constant investigations consist of rather thin-walled nickel, silver, or gold cylinders or plates held apart in the case of cylinders by quartz or Teflon spacers and attached to a glass apparatus. Usually, the space between electrodes is of the order of several millimeters. For the study of concentrated suspensions, a rather rugged cell is necessary because of the nature of the material used. Furthermore, the space between the electrodes must be considerably larger than usual to ensure the flow of the materials into the interelectrode space. Lastly, the cell must be readily disassembled, cleaned, and reassembled.

Since the ordinary dielectric-constant cells were not suitable, special cells were constructed. These cells (fig. 2) consisted of two concentric brass cylinders fitted into a Bakelite top. Both cylinders and the top were threaded. The cylinders were closed at the bottom, and the outer cylinder also served as a container for the material being

investigated. Two holes were tapped so that screws could make connection with the top edges of the cylinders and thus could serve as terminals. The cylinders were of sufficient difference in diameter, about 1/2 inch, to ensure a ready flow of material between them. These calls were rugged and were easily disassembled for cleaning.

In order to avoid long leads to the constant-temperature bath, the cell was placed in a stainless-steel water jacket, and water was pumped from the bath to the jacket and back. The thermoregulator was adjusted to compensate for the temperature gradient between the jacket and the bath. The temperature was maintained constant to at least $\pm 0.2^\circ \text{C}$. The small temperature coefficient of the dielectric constants of the materials made this degree of temperature control quite sufficient.

The cells were calibrated by using air and purified benzene for reference substances.

Instrumental Accuracy, Reproducibility, and Sensitivity

The reproducibility and accuracy of the apparatus were partly tested by the use of purified hydrocarbons. An example of the reproducibility of results is given by the following values for cyclohexane: 2.007, 2.008, 2.008; and methylcyclohexane: 2.009, 2.009, 2.010. The accuracy may be judged by a comparison of some values measured at 20°C with this apparatus with those given in the literature (also measured at 20°C) that are considered reliable:

Substance	This investigation	Literature	Reference
Hexane	1.890	1.890	15
Heptane	1.925	1.926	15
Cyclohexane	2.024	2.024	16
Toluene	2.386	2.387	17

According to the manufacturer, the Schering bridge has an accuracy of ± 0.2 micromicrofarad or ± 0.1 percent when capacity differences are measured. The results listed in the preceding table substantiate this estimate and seem to indicate an even better accuracy. The reproducibility with concentrated suspensions may be judged by the values of 7.10, 7.08, and 7.09 measured at different times for a 50 percent magnesium suspension of 14-micron particles of 98-percent purity in mineral oil. In general, the probable error in dielectric-constant determinations with suspensions varied from 0.1 to 1 percent, depending on the concentration size and shape of the particles; but it was often less than ± 0.25 percent. The larger errors in these determinations result from noninstrumental sources of error. These are, with their estimated magnitudes, as follows:

- (1) Nonhomogeneities due to imperfect mixing (error in extreme cases several hundredths of a dielectric-constant unit)
- (2) Rapid settling of particles in more dilute suspensions and at higher temperatures (may lead to errors of about ± 0.01 dielectric-constant unit)
- (3) Temperature fluctuations (less than instrumental error, that is, about ± 0.02 percent for an uncertainty in temperature of $\pm 0.2^{\circ}$ C)

The sensitivity of the apparatus could be adjusted by varying the oscillator output or the amplification. Usually the sensitivity of adjustment exceeded the readability of the capacity dial, which was about 0.05 micromicrofarad. The sensitivity was decreased when measurements were made at 100 cycles per second because of the inefficiency of the 60-cycle-per-second reject filter at 100 cycles per second. The sensitivity at 100 cycles per second was about ± 0.2 to ± 0.3 micromicrofarad.

The dielectric-loss readings usually could be reproduced to ± 5 percent.

Settling-Rate Measurements

Measurements of the settling rates of the magnesium suspensions were made at the same time the dielectric-constant measurements were made. The suspensions were placed in 100-milliliter graduated cylinders, which were kept in the constant-temperature bath between readings. The level between the supernatant liquid and the suspension was determined.

EXPERIMENTAL RESULTS AND DISCUSSION

Initial Values of Dielectric Constant

The initial dielectric-constant values used in the following sections are values obtained immediately after the cell was filled with the suspension.

Effect of concentration of particles on dielectric constant. - The values of the dielectric constants of the suspensions immediately after preparation are plotted against the weight percent of solids in figure 3(a) and against the volume percent in figure 3(b). The values plotted represent averages of two to four experimental determinations. These individual values, their mean values, and the estimated errors are given in table II.

The variation of dielectric constant with volume percent is of more theoretical significance than the variation with weight percent of solids. To calculate the volume percent, the following formula was used:

$$V = \frac{100d_L\omega_S}{\omega_S d_L + \omega_L d_S} \quad (11)$$

where

d_S, d_L densities of solid and liquid, respectively

ω_S, ω_L weight fraction of solid and liquid, respectively

The error in V is of the order of 0.25 to 0.5 percent. This error is introduced into the transformation because the density of the solid is the average of the densities of the pure magnesium powder and of a small amount of magnesium oxide. The average density was found by first analyzing the solid for the percentage of magnesium and then reading the density from a large graph of d_S against percent oxide content.

The curves in figure 3 present a number of interesting features. Compared with the experimental error of ± 0.01 to ± 0.02 in the dielectric constant, the dielectric-constant change is quite large, especially in the concentration region from 40 to 60 weight percent. Thus, in going from 50 to 60 weight percent, the dielectric constant changes by about 2.7 units. This means that an unknown suspension of the same material may be determined to ± 0.2 to ± 0.3 weight percent in this region. Determinations of differences from 0.5 to 1 weight percent and larger could be made with ease. From mineral oil alone to 60 percent magnesium (14μ) in mineral oil, the dielectric constant increased from 2.171 to 9.815.

The curves are both convex towards the concentration axes. In figure 3(b), the region below 10 to 15 percent by volume appears to be fairly linear. A least-squares treatment indicates that actually the experimental data at 10 percent by volume and below must be represented by a cubic equation. The experimental data plotted in figure 3(a) for 1.8-micron magnesium for 5 percent by volume and below are best represented by a quadratic equation. For a linear equation to be valid to better than 0.01 dielectric-constant unit, only data in the region of 2 percent by volume and below should be used. This may be verified by a series expansion of equations such as (3) and (8) by using various values of V , that is, 0.02, 0.05, 0.1 volume percent. The series representing the relation between ϵ and V are slowly converging series and attempts at simplification which result in cutting off the series expansion should be carried out with considerable caution.

In table III, the theoretical values for the dielectric constants of metallic spheres in mineral oil are calculated by means of equation (2), column 2, and equation (7), column 3, while the experimental results are given in column 6. The series expansion of equation (2) for metallic spheres has the form

$$\epsilon = \epsilon_2(1 + 3V + 6V^2 + 10V^3 + 15V^4 + 21V^5 + \dots) \quad (3)$$

while equation (7) may be written as

$$\epsilon = \epsilon_2(1 + 3V + 9V^2 + 27V^3 + 54V^4 + 162V^5 + \dots) \quad (8)$$

The differences in equations (2) and (7) result from the differing theoretical approaches. All the theoretical equations are derived on the assumption that the suspensions are fairly dilute and that no agglomeration of particles occurs. The method used to derive equation (7) is probably more satisfactory. The series expansions, equations (3) and (8), make it evident that when the third and fourth terms in the series become important the results of the two equations will disagree increasingly with each other.

In table III, columns 4 and 5, values are calculated with the inclusion of the shape factor f in the theoretical equations. This factor has been evaluated from the experimental dielectric-constant data for the 14-micron particles in mineral oil. (The shape factor will be discussed in more detail in the general discussion of the influence of shape on the dielectric constant.) A value of $3f = 3.2$ was employed. Equations (3) and (7) with the shape factor f included have the form

$$\frac{\epsilon_2}{(1 - fV)^3} = \epsilon_2(1 + 3fV + 6f^2V^2 + 10f^3V^3 + \dots) \quad (11)$$

$$\frac{\epsilon_2}{1 - 3fV} = \epsilon_2(1 + 3fV + 9f^2V^2 + 27f^3V^3 + \dots) \quad 9f^2V^2 < 1 \quad (12)$$

It may be seen that up to about 11 volume percent the equations including the shape factor give better agreement with experiment and that the modified equation (12) gives the best results. At the two highest concentrations, the modified equation (11) gives the best results. It is doubtful that any of the equations have applicability above about 10 volume percent because of the complex effect of neighboring particles on the local electric field of a given particle. (The coefficients of the terms in V^4 and higher in the theoretical equations which are becoming important in this concentration region may be grossly incorrect.) Furthermore, some particle agglomeration would be expected

in the region above 10 percent by volume. No attempt has been made to include the consequences of this effect in the theoretical equations.

In table IV data similar to those given in table III are listed for 1.8-micron magnesium particles. The shape factor employed was $f = 1.4$. For the three lowest concentrations, the modified equation (12) is slightly more satisfactory than the modified equation (11), while at 8 and 11 percent by volume, the modified equation (11) appears to be more satisfactory. Above 11 percent by volume both of the modified equations give values which are too high.

These two sets of results indicate the importance of including the appropriate shape factor in the theoretical equations. These results do not clearly indicate which of the modified equations is more satisfactory. Both give good agreement with experiment at low concentrations (< 10 volume percent).

Effect of shape of magnesium particles on dielectric constant. - The values of dielectric constants against concentration are plotted in figure 4 for four different shapes of magnesium particles. The 14-micron particles are nearly spherical while the three other powders with average particle diameters below 2 microns are irregular. The material made up of particles with an average particle diameter of 1.5 microns appears to be the most nonspherical and the most platelike from its large slope. All the material less than 2 microns in diameter appear as irregular plates under the microscope. The dielectric-constant results make it possible to distinguish between them in a quantitative manner. The shape factors will be discussed in more detail for two of the materials, the 14-micron average particle diameter and the 1.8-micron average particle diameter.

The shape factor may be evaluated from B in the series expansion $A + BV + CV^2 + \dots$ used to represent the experimental data. The shape factor $f = B/3\epsilon_2$ for metallic particles. For the 14-micron particles, the values of f calculated from the experimental data below 10 percent by volume for powders containing 98 percent magnesium and 100 percent magnesium (extrapolated value) combined with the slope of the linear equation with only the first two experimental points at 0 and 0.025 percent volume used give $3f = 3.2$ or $f = 1.07$. These particles are slightly ellipsoidal. Similar calculations for the 1.8-micron particles give $3f = 4.2$ and $f = 1.4$. The 1.8-micron particles should deviate from the spherical considerably more than the 14-micron particles. Microscopically, the 14-micron particles appear quite spherical, but small deviations would not be readily detectable. The value of $a/b \sim 0.3$ (where a/b is the axial ratio of a spheroid) calculated from $f = 1.4$ by using table V, reference 10, and unpublished data would indicate that the particles are somewhat flattened spheroids. Under the electron microscope, the 1.8-micron particles appear as somewhat irregular

spheroids or plates (fig. 5). If the particles were quite flat, say $a/b \sim 0.1$, then f would be greater than 3.

Unfortunately, for a random distribution of orientations of ellipsoidal particles with respect to the electric field, it does not seem possible to distinguish between two values of a/b without other information for a given value of f (see table V). If qualitative knowledge as to the shape is available, then a quantitative estimate can be uniquely given also. If shear is applied so that one of the axes of the ellipsoid is oriented parallel to the field, then a/b is a single-valued function of f , a unique choice may be made as to the shape, and a quantitative estimation may be given.

Effect of size of magnesium particles on dielectric constant. - It is assumed in the various theories on the dielectric constants of mixtures that, for the case of solid particles in a nonconductive dispersing medium, the dielectric constant is independent of particle size. Not much in the way of conclusive experimental evidence has been offered to support this assumption. In the present investigation, magnesium particles of three different particle diameters, 23, 14, and 4.6 microns, and of the same shape were employed to test this assumption. The results are listed in table VI. These results indicate that within the experimental errors involved the dielectric constant is independent of particle size for this system. The slightly lower dielectric-constant values for the 4.6-micron powder in mineral oil may be due to a small degree of incomplete dispersibility.

Effect of oxide content on dielectric constant. - The effect of the oxide content on the dielectric constant of magnesium suspensions in mineral oil was determined with the 14-micron powder. The difference between the dielectric constant of suspensions with various weight-percent total solids but containing about 2 percent oxide in the solid content in one case and about 9 percent oxide in the other is plotted in figure 6. As can be seen from figure 6, increasing the amount of oxide by 7 percent also increases the dielectric constant. This increase in dielectric constant with the 7-percent increase in oxide varies linearly with increasing total-solids concentration. The percentage of magnesium was determined by a gasometric analysis wherein the hydrogen evolved by adding acid to magnesium powder displaced a measured volume of water. These gasometric analyses are probably never more reproducible than ± 1 percent. The values of oxide content for the two samples of magnesium powder used to determine the effect of oxide content were analyzed quite carefully and are probably correct to ± 1 percent. The values given in figure 6 have been used to make possible extrapolations to 100 percent magnesium content and to correct the values obtained in the temperature-coefficient measurements to the same oxide content.

The increase in the dielectric constant with increasing oxide content is probably due to the flaking off of irregularly shaped plates of magnesium oxide from the magnesium particles. This effect amounted to about 0.1 dielectric-constant unit for a 50 percent suspension. If a value of $\epsilon = 9.8 \pm 0.5$ is used for the dielectric constant of magnesium oxide (ref. 18), the coefficient of the linear term of the equation derived for spheroidal particles dispersed in mineral oil (ref. 10 and unpublished data) would be 1.72 for the special case of spheres compared with 3.00 for metallic spheres. Thus the mere substitution of magnesium oxide spheres for magnesium spheres would lower the dielectric constant. If, however, the magnesium oxide particles are nonspherical and are in the form of very flat plates, then the linear coefficient would increase beyond 3.00 and the dielectric constant of the suspension would be larger than that of a suspension containing magnesium spheres only.

Effect of temperature on dielectric constant. - The temperature coefficients $d\epsilon/dt$ were determined for mineral oil and for 50- and 60-percent-by-weight suspensions of 14-micron magnesium particles in mineral oil in the temperature range from 20° to 50° C. The results are plotted in figure 7. The values of $d\epsilon/dt$ corrected for oxide content are given in the following table:

Material	$(d\epsilon/dt)/^{\circ}\text{C}$
Mineral oil (first sample)	-0.001
50 Percent magnesium (14 μ) in mineral oil	-0.008 \pm 0.002
60 Percent magnesium (14 μ) in mineral oil	-0.012 \pm 0.002

The temperature coefficients for concentrated magnesium suspensions are quite large compared with those for mineral oil only, and it would appear that the temperature coefficient decreases quite rapidly with decreasing concentration of magnesium particles in a suspension. Since corrections had to be made for the oxide content of the magnesium particles, the values for the temperature coefficients are not highly accurate.

Effect of frequency of oscillation on dielectric constant. - The variation of the dielectric constant with the frequency of oscillation was studied in the frequency range from 10^2 to 2×10^5 cycles per second. The values at 100 cycles per second are somewhat less accurate than those obtained at higher frequencies because of 60-cycle-per-second interference which could not be completely filtered out. The results are given in figure 8.

In figure 8(a), there is evidence of a small but experimentally significant decrease in the dielectric constant with increasing frequency. The larger decreases in the dielectric constants for concentrations of the highly nonspherical particles (1.5- and 1.9- μ magnesium powders),

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which are much lower (20 volume percent) than the concentration employed for the nearly spherical 14-micron powder (60 volume percent), clearly indicates the importance of the shape of the particles upon this phenomenon. It is true that the change with frequency in the case of the 14-micron material is of the same magnitude as the experimental error. These particular results possibly may not be experimentally significant.

In figure 8(b), the effects of concentration upon the frequency effect are illustrated. The decrease $\Delta\epsilon = \epsilon_{\nu,2} - \epsilon_{\nu,1}$ ($\nu_2 = 100$ cps) with increasing frequency increases in magnitude quite regularly with the increasing concentration of particles.

Equation (9) may be written in the form

$$\epsilon = \epsilon_{\infty} \left[1 + \frac{3V}{\frac{2\epsilon_2 + \epsilon_1}{3\epsilon_2} + \frac{(2\epsilon_2 + \epsilon_1)^3 \omega^2}{48\pi^2 \epsilon_2 \sigma_1^2}} \right] \quad (9)$$

For metallic particles dispersed in mineral oil, $\epsilon_1/\epsilon_2 \rightarrow \infty$; hence $\epsilon = \epsilon_{\infty}$. Since the second term tends toward zero, this result which predicts no frequency effect is not in accord with the experimental results.

Actually, of course, the magnesium particles are coated with magnesium oxide. This layer of oxide is not continuous but tends to crack and blister because of the high density of the oxide and the curvature of the particles. The oxide coating on the magnesium particles will tend to flake off readily. This small amount of irregular flakes of magnesium oxide in the suspension would result in a finite effect of frequency upon the dielectric constant being observed.

The shells of magnesium oxide on the magnesium particles would probably also contribute to the frequency effect. Since these shells would have the same shape as the particles, they could well be responsible for the observed effect of shape on the frequency dependence of the dielectric constant.

Variation of Dielectric Constant with Time

Dielectric-constant curve. - The variation of dielectric constant with time as settling proceeds in the measuring cell is shown for various magnesium concentrations at 20°, 30°, and 50° C in figure 9. When the suspension is dilute enough, or when the particles are fairly large, a representative curve may be obtained in a few hours. The curve may or

may not show a sharp maximum in the first few minutes, but it is generally found thereafter that the dielectric constant decreases to a broad minimum and then begins to increase again (figs. 9(a) to (f)).

The initial maximum may be due to the filling of air spaces in the more viscous suspension or the escape of small air bubbles. It might be explained by a process of agglomeration during the first few minutes the material is being measured. However, since the effect is less prominent in the more concentrated suspensions where agglomeration would be most likely, this explanation is not too satisfactory.

The remainder of the curve may be explained in terms of two processes of settling. In the first process, particles that are large compared with the average particle size are settling out through the suspension. The decrease in concentration through the suspension is not compensated for by the small amount of concentrated material packed at the bottom. The net result is a decrease in dielectric constant. This process would be expected to be important early in the settling process. As time continues, the entire mass of suspension settles down, leaving an increasing amount of supernatant liquid above it. This second process may be thought of as a compressional phase of the settling process. The effect of this process on the dielectric constant may be calculated by considering the following model: The dielectric-constant cell may be considered as two parallel cylindrical condensers. The upper condenser is in the region where only supernatant liquid is found and the dielectric constant is that of the mineral oil. The lower condenser has compressed suspension between its cylinders. If the approximation is made that the compression is uniform, the concentration of the compressed suspension may be calculated and, hence, from previous results its dielectric constant. Since the two condensers are considered in parallel, the capacities are additive and so are the dielectric constants.

As an example, a 20-percent-by-volume suspension in a hypothetical dielectric-constant cell, which is 100 milliliters in volume, will be considered. Initially the dielectric constant was 4.5 units. When 20 milliliters of supernatant liquid are present, the volume concentration of solid in the suspension is 25 percent. The dielectric constant would be $\frac{1 \times 2.2 + 4 \times 5.4}{5} = 4.8$ units. The total average dielectric constant has increased by 0.3 unit. If settling proceeds further until there are 40 milliliters of supernatant liquid, the volume concentration of solids has increased to 33 percent. The dielectric constant would then be $\frac{2 \times 2.2 + 3 \times 7.1}{5} = 5.1$ units. Thus, compression leads to a continuously increasing total average dielectric constant.

Thus, two processes, dilution and compression, are acting on the suspension. The former is decreasing the dielectric constant and the latter is increasing the dielectric constant. At the observed minimum, the two processes are cancelling each other and then the compressional process becomes predominant and the dielectric constant increases again.

This explanation is admittedly a qualitative one which ignores important factors such as concentration gradients and cannot be used to predict quantitatively the shapes of the dielectric-constant against time curves which are observed. It does explain the effects observed in terms of factors which should be important in the settling process.

Settling-level curves. - The settling-level ratio $\frac{h}{h_0} \times 100$ is also plotted in figure 9. The value of the settling level h is the level between supernatant liquid and settled suspension, while h_0 is the level of suspension before settling begins. In a number of the dilute suspensions (e.g., fig. 9(a)) settling-level curves are incomplete. This results from the difficulty in making a reading of the level. In these suspensions the level appears to be quite high for a period of time, although nonhomogeneities are visible in the suspension itself. After a time, a considerable portion of the suspension becomes partly transparent because of the settling of a sufficient amount of larger material. Finer particles in considerable amounts are left suspended in the supernatant liquid in these cases.

Dielectric Losses of Magnesium Suspensions

At the same time that the capacity measurements were made, the dissipation factor, or dielectric loss, was measured with the bridge. The accuracy of these measurements was considerably lower than those giving the dielectric constant.

It was found that the dielectric losses of magnesium suspensions in mineral oil increased with increasing concentration of magnesium particles, as may be seen in table VII. Increased deviation from spherical shape also increased the dielectric losses somewhat. The concentration effect has been investigated previously (ref. 10) for silica suspensions. Both the absolute values of the dielectric losses and the changes in the dielectric losses are much greater than those given here. The increases in dielectric loss with increased deviations from spherical shape have been discussed theoretically in reference 11.

If equation (9) is rewritten as

$$\tan \delta = \frac{\omega}{\frac{4\pi\sigma_1}{9\epsilon_2 V} + \frac{4\pi\sigma_2}{2\epsilon_2 + \epsilon_1} + \frac{(2\epsilon_2 + \epsilon_1)^2}{36\pi\epsilon_2\sigma_1 V}} \omega^2$$

then as $\frac{\epsilon_1}{\epsilon_2} \rightarrow \infty$, that is, for metallic particles in suspensions,

$$\tan \delta \rightarrow 0$$

The explanation herein is probably quite similar to that used to explain why there is an effect of frequency upon dielectric constant. The presence of a dielectric shell around the metal particle and the flakes of magnesium oxide which have fallen off the oxide shell are probably partly responsible for the losses observed. The losses of the mineral oil and benzene may be due to space charges (ref. 19).

The dielectric losses were found to increase with increasing temperature from 20° to 50° C. A change in dielectric loss with time was also observed. At 20° C, the losses usually decreased by 5 to 10 percent during the course of the determination when the 14-micron magnesium particles were dispersed in mineral oil. The less than 2-micron material showed a somewhat greater decrease in dielectric loss with time. Some representative values are given in table VIII. In contrast with these results, at 50° C the dielectric losses were found to increase by 5 to 10 percent with time.

These time effects upon the dielectric loss may be associated in part with concentration changes or with increased wetting of the particles. The increases in dielectric loss may be associated in some instances with high humidities which resulted in water absorption.

SUMMARY OF RESULTS

The initial dielectric constants (time = 0) of suspensions of magnesium particles in mineral oil were determined as a function of concentration, temperature, particle size and shape, degree of oxidation, and frequency of oscillation. The increases of dielectric constant with increasing particle concentration and increased deviation from spherical shape were the most pronounced variations found. The dielectric constant increased from 2.171 to 9.815 in going from the mineral oil alone to 60 percent magnesium in mineral oil, even for almost spherical particles. The variation of dielectric constant in dilute suspensions allowed a shape factor to be determined for a given type of particle which was related quantitatively to the shape of the particle. The dielectric constant decreased with increasing temperature. The temperature coefficient

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increased with increasing concentration. An increase in the oxide content increased the dielectric constant. This effect amounted to about 0.1 dielectric-constant unit for a 50 percent suspension. The effect may be due to the flaking off of irregularly shaped oxide particles. The dielectric constants of magnesium suspensions were shown to be independent of particle size. The difference between the dielectric constant at 100 cycles per second and at a higher frequency $\Delta\epsilon$ was shown to increase with increasing frequency of oscillation. The magnitude of the increase of $\Delta\epsilon$ increased with increasing concentration and with increasing deviation from spherical shape. Since the dielectric constants of suspensions of metal particles in mineral oil should be independent of frequency, the effects observed were ascribed to the oxide shells around the metal particles and to flakes of magnesium oxide in suspension.

The variation of dielectric constant with time as settling proceeded was also investigated. The curves found experimentally were explained qualitatively on the basis of two processes. One of these was a process of dilution resulting from the settling out of large particles during the initial stages of the over-all settling process. The second process was a compression of the suspension which resulted in a net increase in the dielectric constant which began to predominate later. This interpretation serves to explain in a qualitative manner the experimental curves which were convex to the time axis with a wide minimum.

Settling-level curves were determined along with the dielectric-constant data.

The dielectric losses were shown to increase with increasing concentration and increasing deviation from spherical shape. Since the losses for metallic particles in mineral oil should be zero, the losses observed were probably partially caused by the presence of the magnesium oxide. The small losses observed even in the mineral oil may be due to space charges.

Time variations in the dielectric losses as settling proceeds were also investigated.

CONCLUDING REMARKS

A considerable mass of data upon various dielectric phenomena has been obtained in a relatively unexplored field. Some of the effects studied can be explained quantitatively, a larger portion only semi-quantitatively or qualitatively. Much still remains to be done upon the theoretical aspects of the subject before a precise quantitative treatment may be given to the experimental material.

Practically, the results indicate several possible valuable procedures: The dielectric constant may be employed to determine concentrations of suspensions or slurries once a calibration is made. Studies in the dilute concentration range (less than 2 percent by volume) will allow the evaluation of experimental shape factors which may be used to evaluate the equivalent shapes of particles in terms of spheres, spheroids, and the limiting cases of disks and needles. The concentration gradients in suspensions are obtainable by means of dielectric-constant measurements if a dipping dielectric-constant cell or a "multicapacitor" settling-rate tube were employed. Furthermore, the deflocculation of suspensions may be investigated by dielectric-constant measurements by using shear or surface-active agents to deflocculate.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 3, 1953

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TABLE I. - ROLLER ANALYSIS OF 14-MICRON POWDER

Average particle diameter, μ	Weight percent
0-8.5	13.5
8.5-17	21.9
17-26	23.5
26-40	40.0
>40	1.1

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TABLE II. - DIELECTRIC-CONSTANT VALUES FOR 14-MICRON MAGNESIUM
PARTICLES OF AVERAGE PARTICLE SIZE AND 98-PERCENT
PURITY IN MINERAL OIL (FIRST SAMPLE) AT 20° C

Solids, weight percent	Values	Average	Average deviation
0	2.170, 2.170, 2.170, 2.167, 2.177	2.171	± 0.003
10	2.63, 2.62	2.625	± 0.01
15	2.94, 2.94	2.94	± 0.00
20	3.29, 3.29, 3.29	3.29	± 0.00
30	4.16, 4.11, 4.15, 4.16	4.145	± 0.02
40	5.43, 5.35, 5.41, 5.40	5.40	± 0.02
50	7.08, 7.10, 7.09	7.09	± 0.01
60	9.83, 9.80	9.815	± 0.02

TABLE III. - COMPARISON OF THEORETICAL AND EXPERIMENTAL VALUES OF
DIELECTRIC CONSTANT FOR 14-MICRON MAGNESIUM PARTICLES IN
MINERAL OIL (FIRST SAMPLE) AT 20° C

1	2	3	4	5	6
V (fraction)	$\frac{2.170}{(1 - V)^3}$	$\frac{2.170}{1 - 3V}$	$\frac{2.170}{(1 - 1.07V)^3}$	$\frac{2.170}{1 - 3.2V}$	Experiment
0.026	2.34	2.35	2.36	2.37	2.37
.053	2.56	2.57	2.58	2.61	2.62
.081	2.80	2.87	2.85	2.93	2.94
.111	3.09	3.25	3.17	3.36	3.29
.177	3.90	4.63	4.07	----	4.15
.250	5.14	8.68	5.53	----	5.40

TABLE IV. - COMPARISON OF THEORETICAL AND EXPERIMENTAL VALUES OF
DIELECTRIC CONSTANT FOR 1.8-MICRON MAGNESIUM PARTICLES IN
MINERAL OIL (SECOND SAMPLE) AT 20° C

V (fraction)	$\frac{2.184}{(1 - V)^3}$	$\frac{2.184}{1 - 3V}$	$\frac{2.184}{(1 - 1.4V)^3}$	$\frac{2.184}{1 - 4.2V}$	Experiment
0.010	2.25	2.26	2.28	2.28	2.29
.026	2.36	2.36	2.44	2.45	2.46
.053	2.57	2.59	2.75	2.81	2.80
.081	2.82	2.89	3.14	3.31	3.19
.111	3.11	3.28	3.62	----	3.65
.177	3.92	4.66	5.13	----	4.91
.250	5.17	8.73	7.95	----	6.60

TABLE V. - VALUES OF SHAPE FACTOR f FOR
METALLIC PARTICLES AT VARIOUS ECCENTRICI-
TIES a/b FOR RANDOM DISTRIBUTION

a/b	f	a/b	f
0.1	3.320	1.2	1.01
.2	1.930	1.4	1.04
.3	1.480	1.6	1.08
.4	1.270	1.8	1.12
.5	1.150	2.0	1.18
.6	1.080	2.5	1.34
.7	1.040	3.0	1.52
.8	1.020	3.5	1.73
.9	1.004	4.0	1.95
1.0	1.000	5.0	2.46
		10.0	5.93

TABLE VI. - DIELECTRIC CONSTANTS OF MAGNESIUM
PARTICLES OF VARIOUS DIAMETERS AND APPROXI-
MATELY 91-PERCENT PURITY IN MINERAL
OIL (FIRST SAMPLE) AT 20° C

Solids, weight percent	Average particle diameter, μ		
	4.6	14	23
10	2.62	2.63	-----
15	2.94	2.94	-----
20	3.29	3.32	-----
30	4.13	4.19	-----
40	5.40	5.43	5.42
50	-----	7.18	7.18
60	-----	9.89	10.00

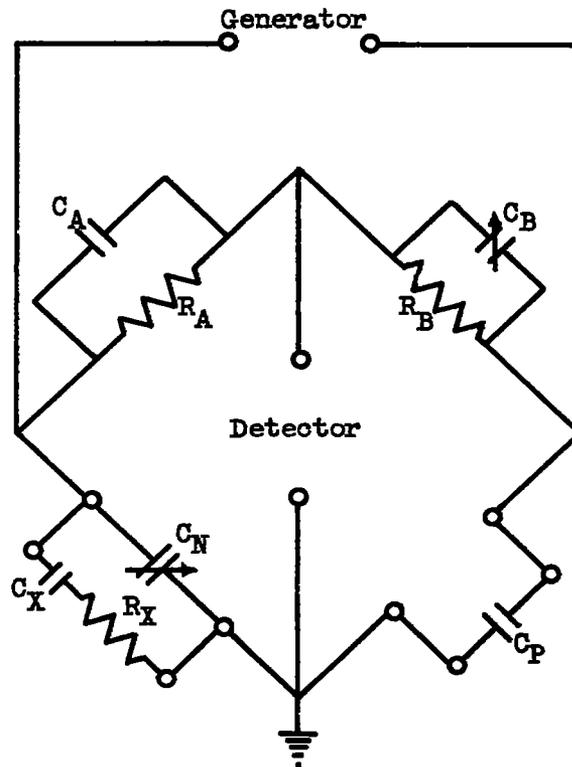
TABLE VII. - DIELECTRIC LOSSES OF MAGNESIUM SUSPENSIONS

Material	Dielectric loss		
	14- μ powder, °C		2- μ powder, °C
	20	50	20
Benzene ^a	0.0006	0.0014	0.0007
Mineral oil (first sample) ^a	.0006	.0014	.0007
10 Percent magnesium in oil	.0006	.0018	.0010
20 Percent magnesium in oil	.0007	.0022	.0018
30 Percent magnesium in oil	.0008	-----	-----
40 Percent magnesium in oil	.0009	-----	-----
50 Percent magnesium in oil	.0011	-----	-----
60 Percent magnesium in oil	.0013	-----	-----

^aMost of the dielectric loss given here is in the Bakelite top of the cell, although there is a small amount of residue loss in the liquid. The other values would be reduced by the same amount so that the conclusions remain unaffected.

TABLE VIII. - TIME BEHAVIOR OF DIELECTRIC LOSSES OF MAGNESIUM SUSPENSIONS IN MINERAL OIL (FIRST SAMPLE) AT 20° C

Material	Time, min	Dielectric loss
60 Percent magnesium (14 μ)	0	0.00130
	10	.00128
	30	.00127
	60	.00124
	90	.00123
20 Percent magnesium (1.9 μ)	0	0.0019
	30	.0019
	60	.0018
	150	.0018
	300	.0016



- C_B set of capacitors used to balance out resistive component R_X (the dial reading of C_B is the dissipation-factor reading)
- C_N internal precision condenser with worm-gear correction calibration
- C_P external condenser fixed at greater than $100\mu\mu\text{f}$
- C_X, R_X capacitive and resistive components of unknown material in dielectric-constant cell

Figure 1. - Schering bridge apparatus.

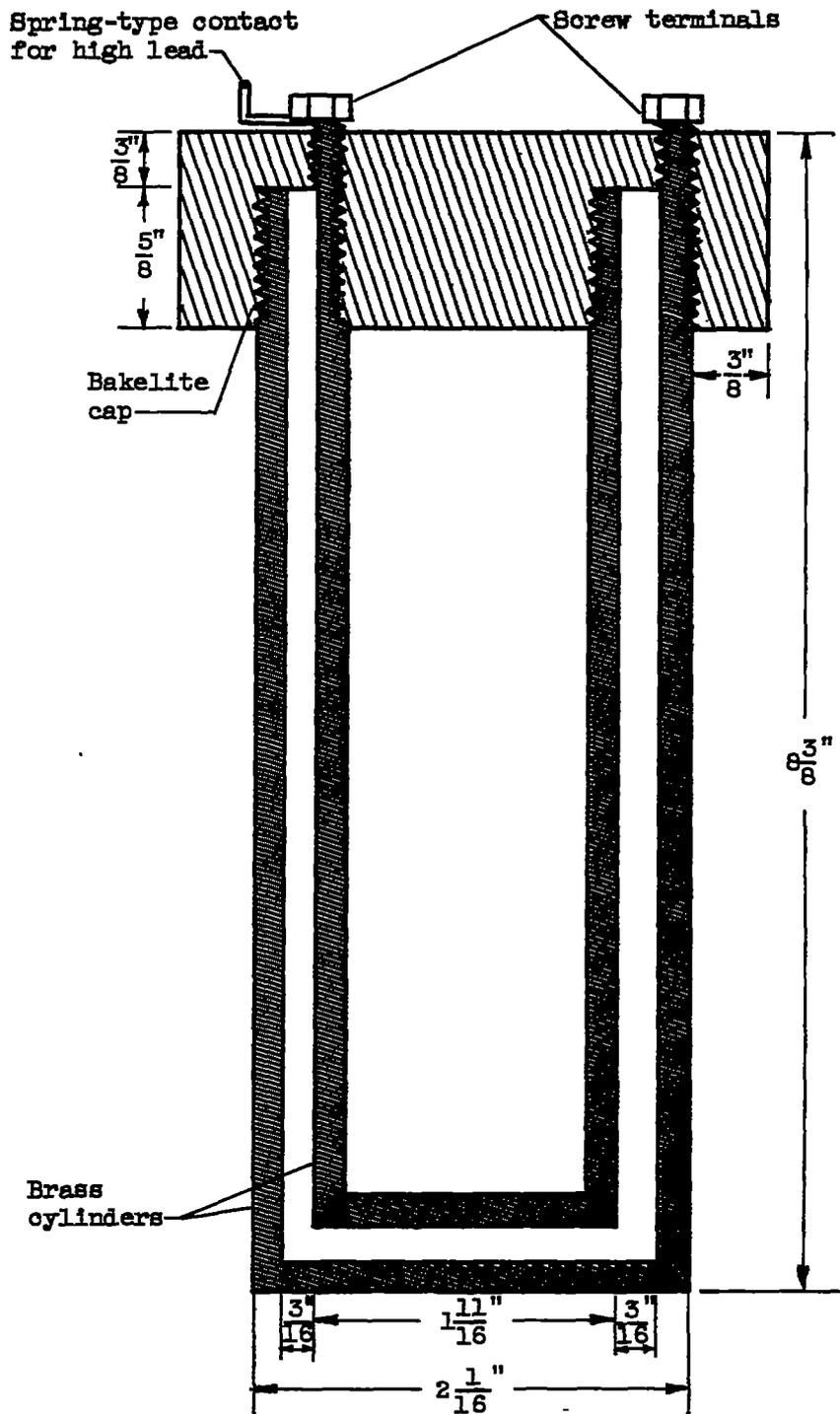
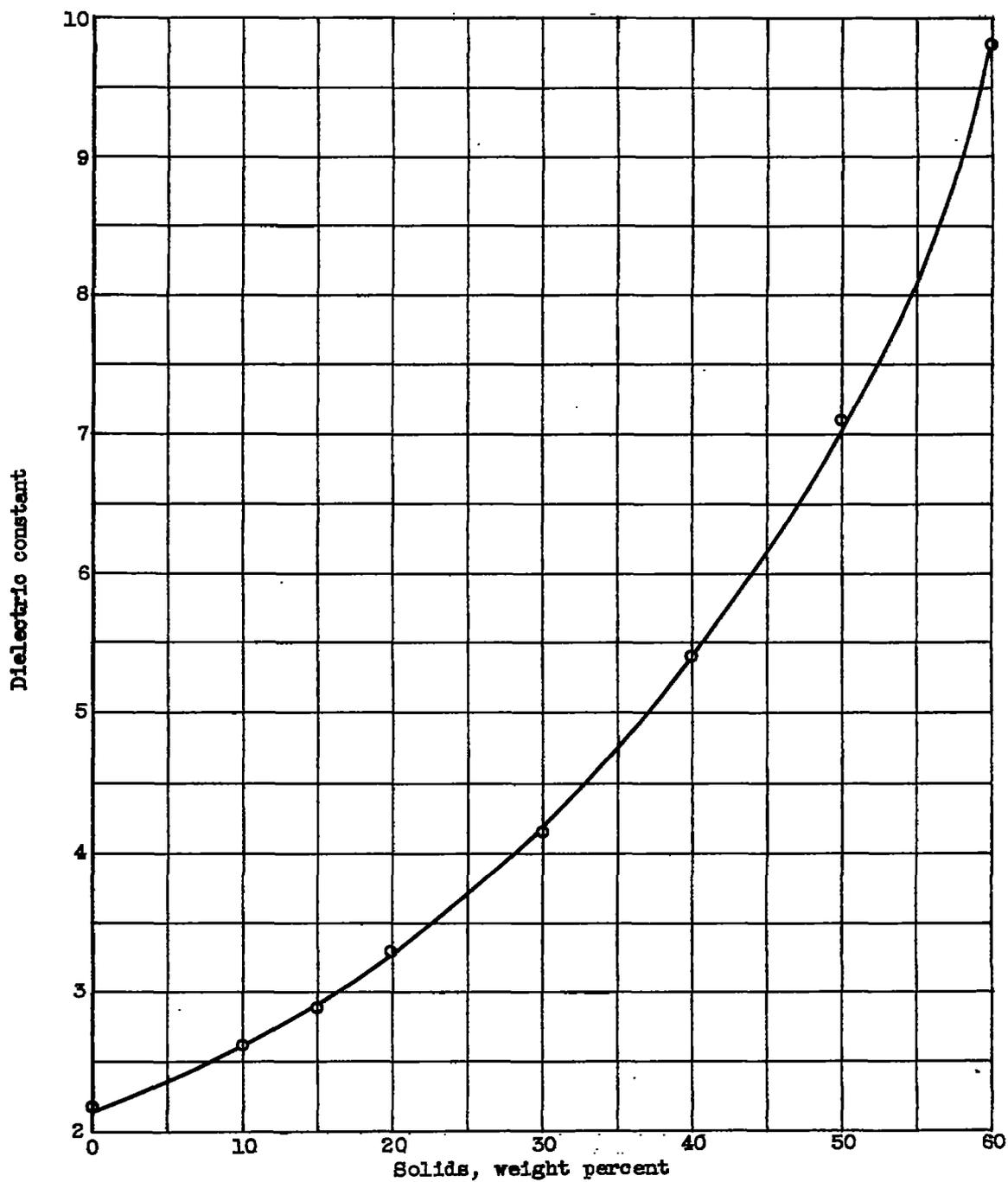


Figure 2. - Dielectric-constant measuring cell.

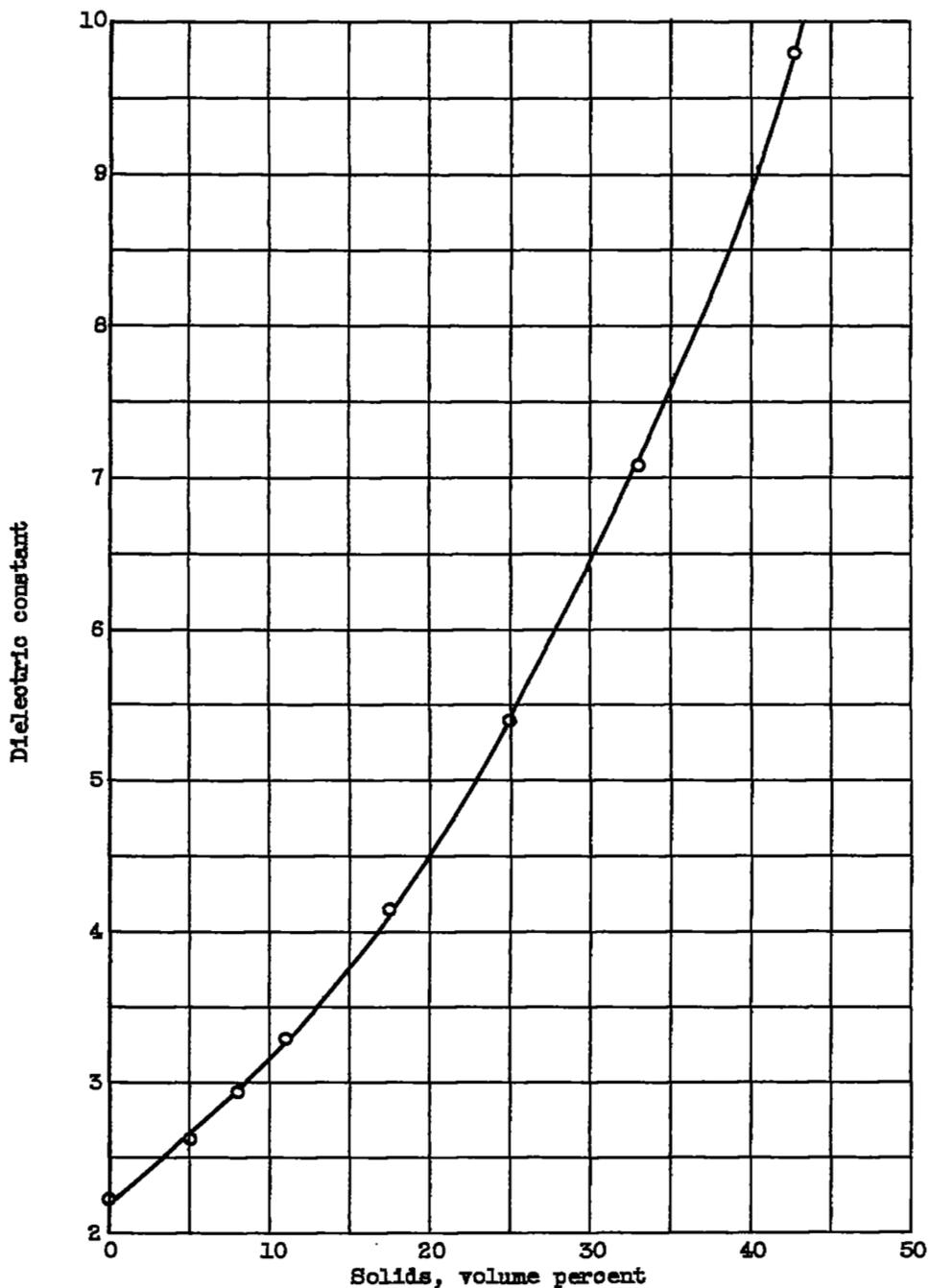
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(a) Weight percent.

Figure 3. - Variation of dielectric constant with concentration of solids in a suspension of 14-micron magnesium powder in mineral oil (first sample) at 20°C.



(b) Volume percent.

Figure 3. - Concluded. Variation of dielectric constant with concentration of solids in a suspension of 14-micron magnesium powder in mineral oil (first sample) at 20° C.

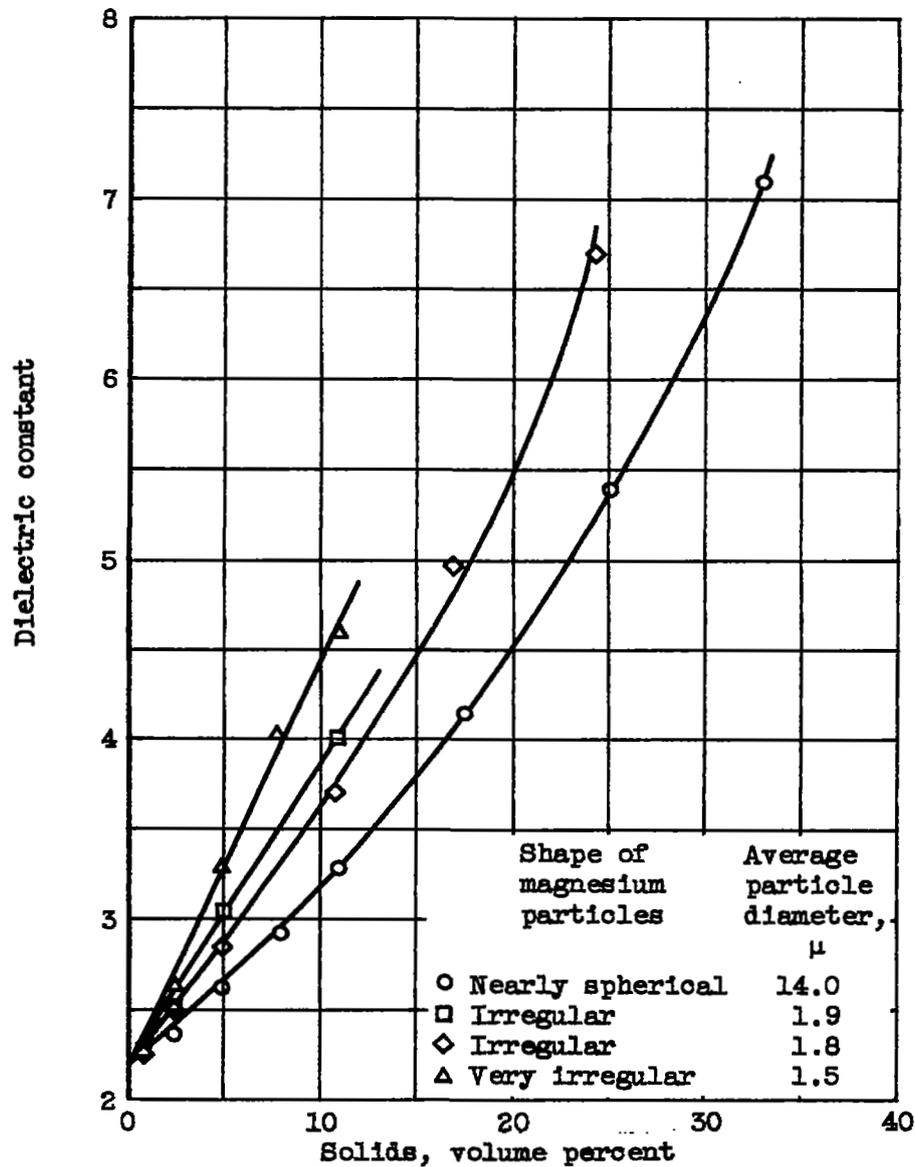
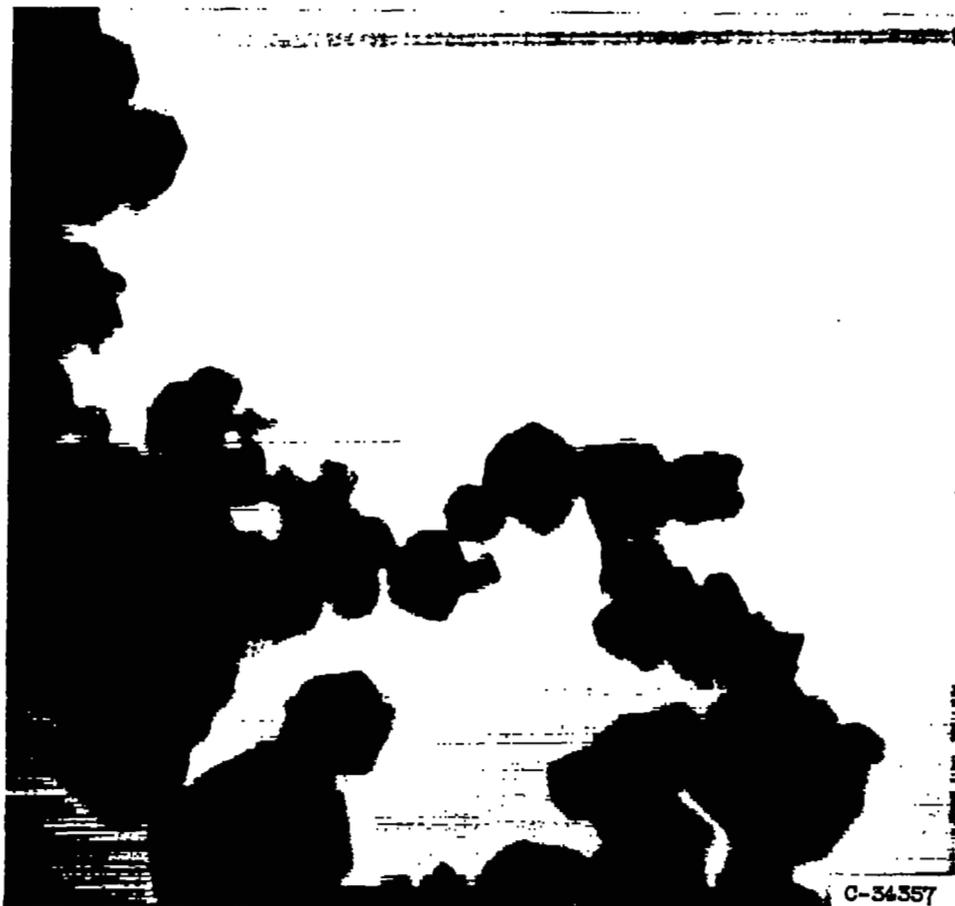


Figure 4. - Variation of dielectric constant with concentration and particle shape of magnesium powder in mineral oil (both samples) at 20° C.



1 μ

Figure 5. - Electron-microscope photograph of atomized magnesium powder. Average particle diameter, less than 2 microns.

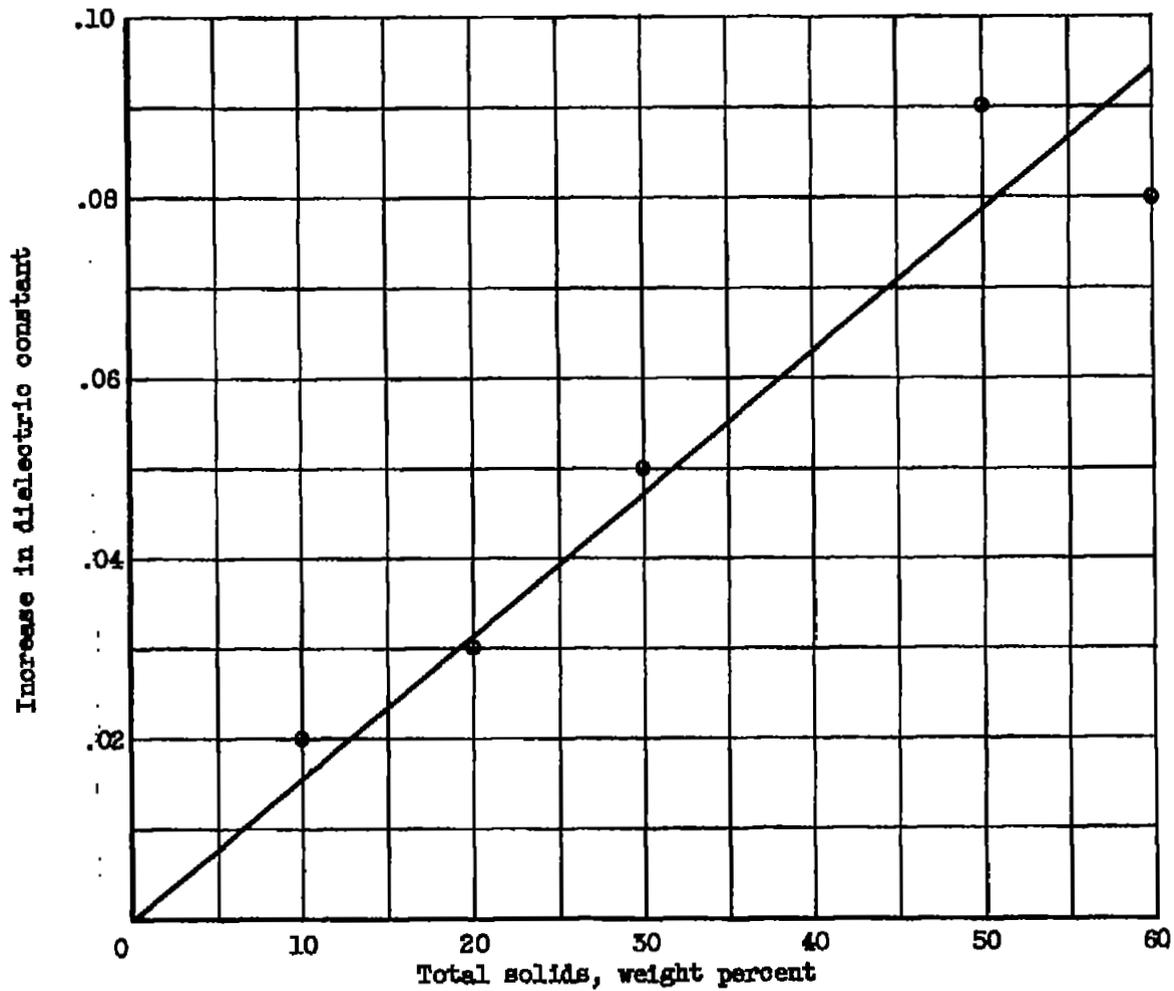


Figure 6. - Effect of 7 percent increase of oxide content on dielectric constant at various total-solids concentration of 14-micron magnesium powder in mineral oil at 20° C.

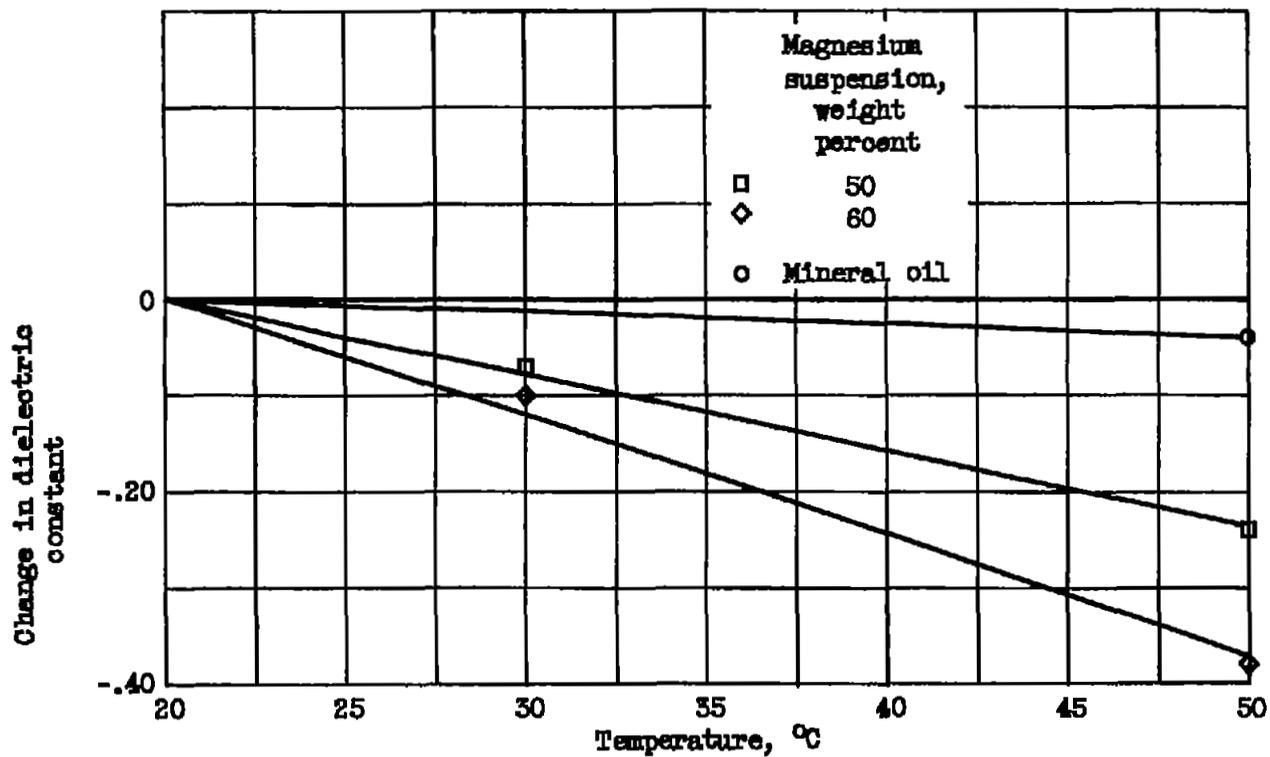
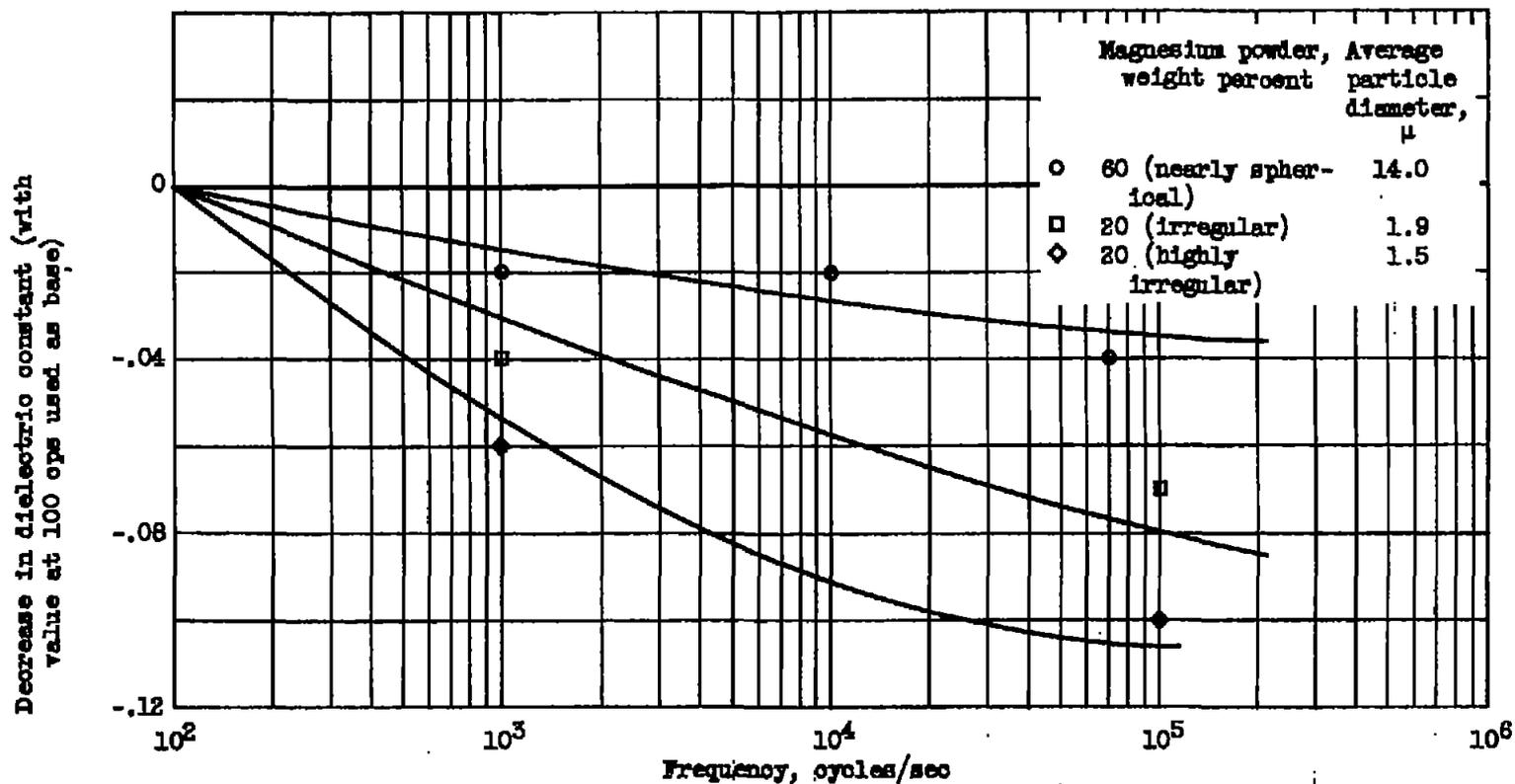
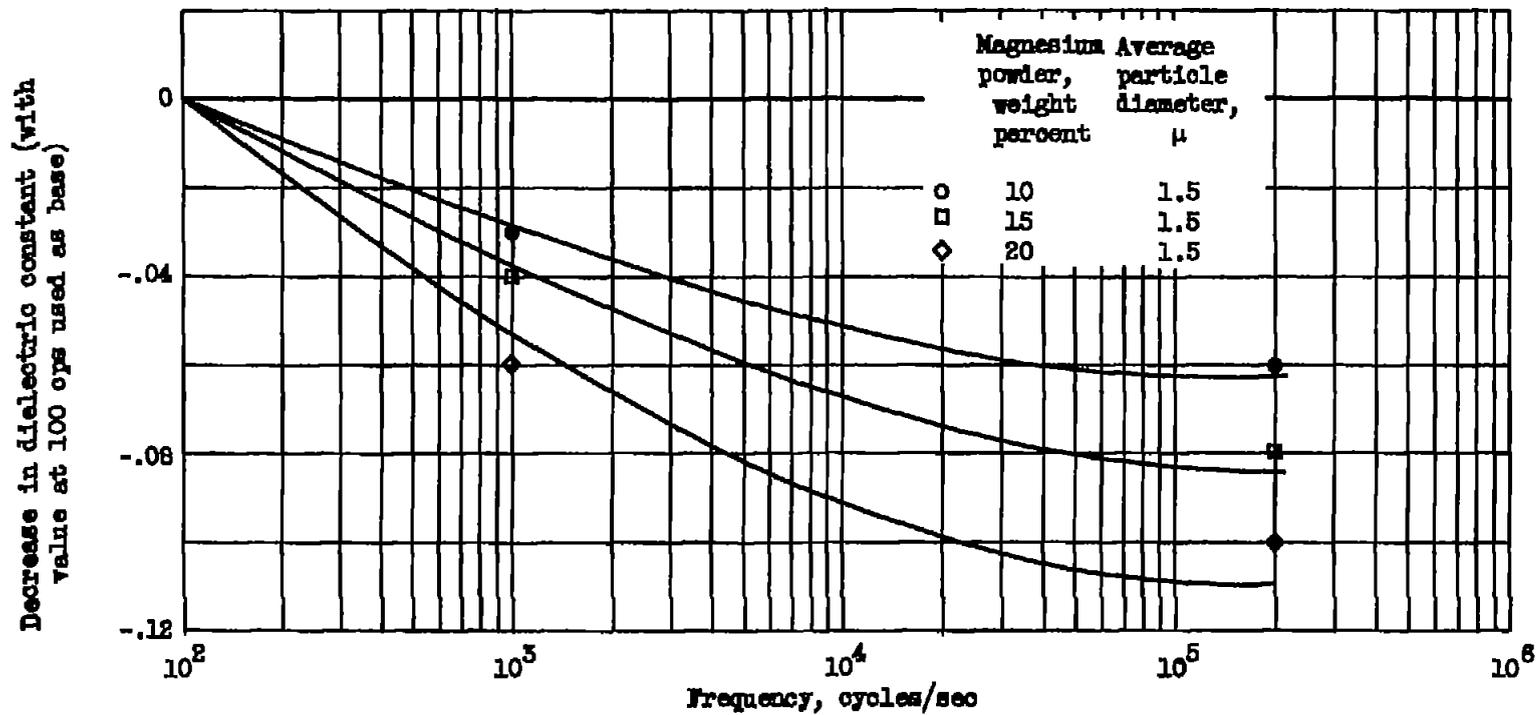


Figure 7. - Variation of dielectric constants of mineral oil and concentrated suspensions of 14-micron magnesium powder in mineral oil (first sample) with temperature.



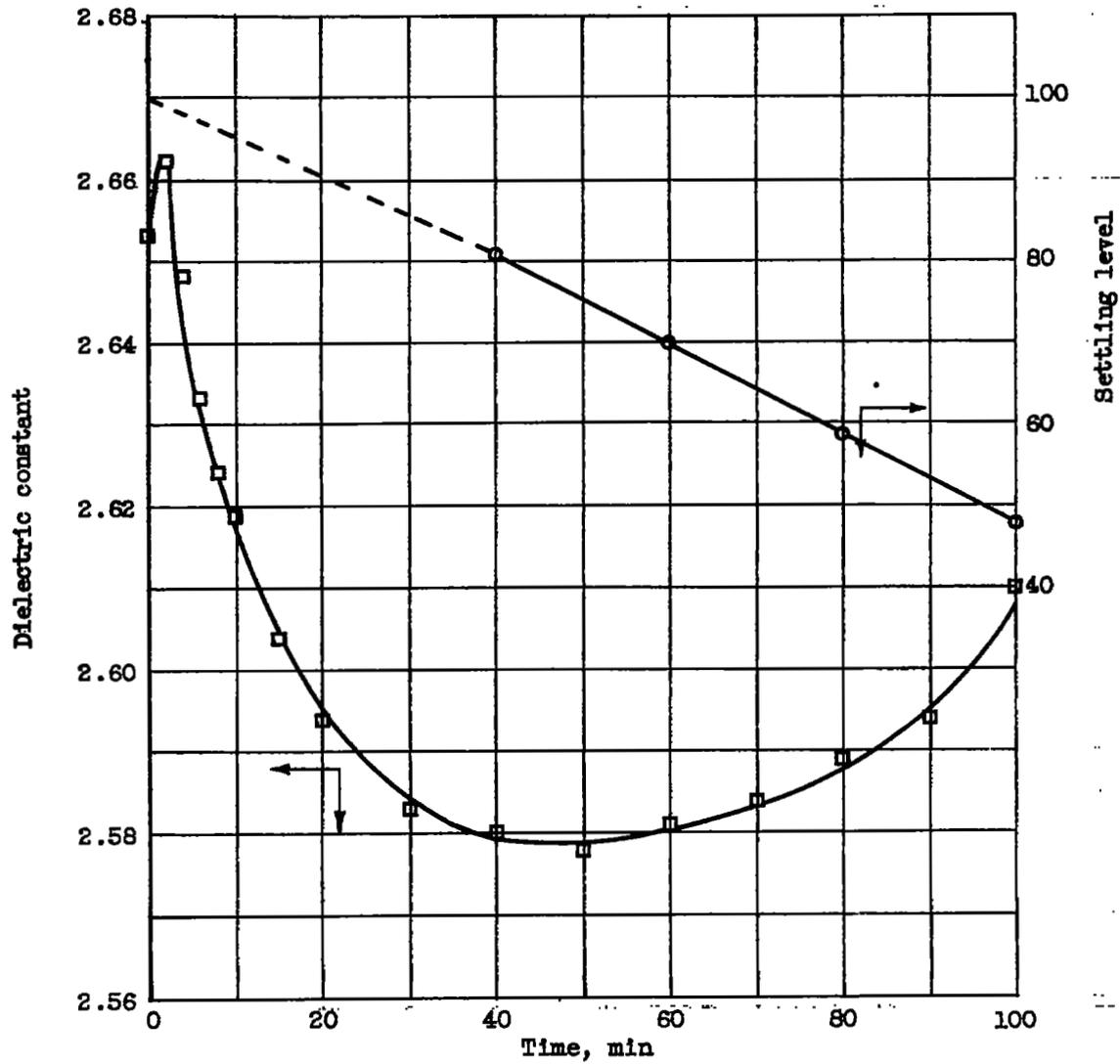
(a) Effect of particle shape.

Figure 8. - Variation of dielectric constant with frequency of oscillation for some suspensions of magnesium powders in mineral oil (first sample) at 20° C.



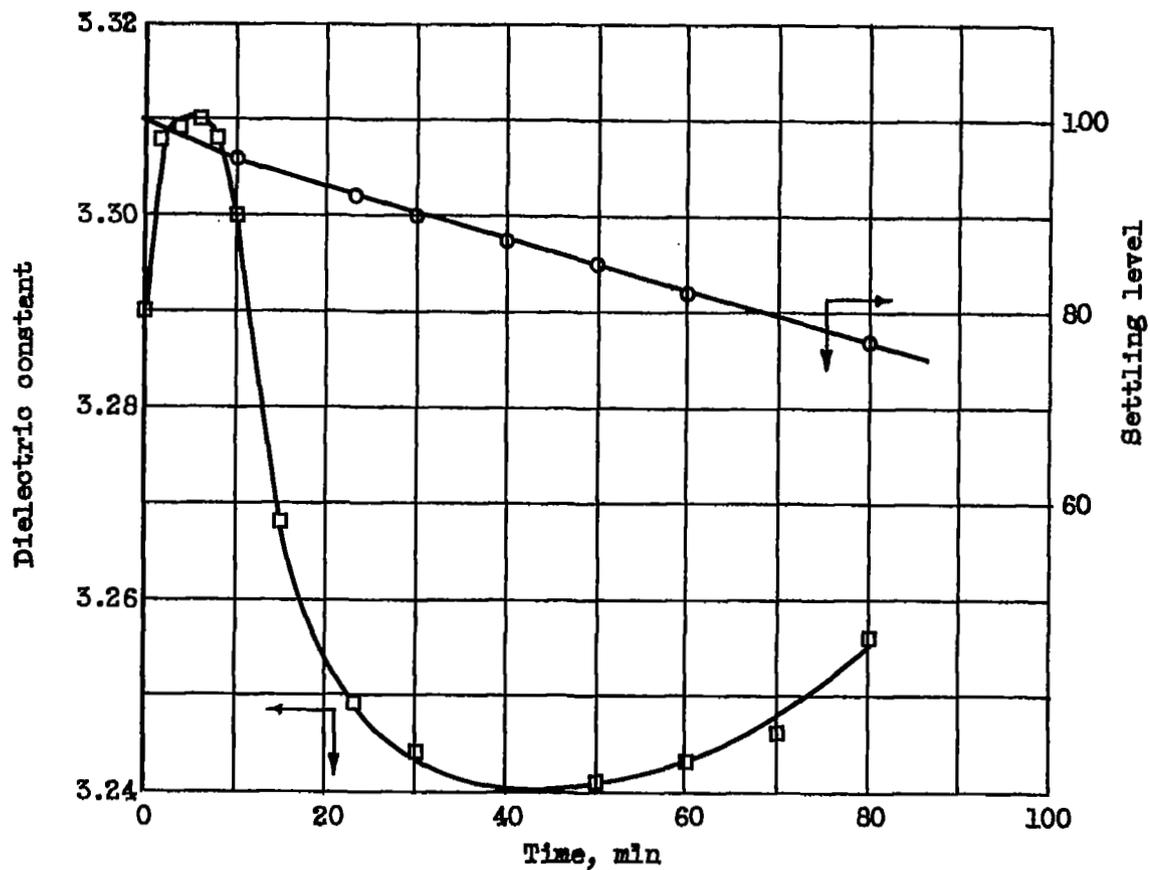
(b) Effect of concentration.

Figure 8. - Concluded. Variation of dielectric constant with frequency of oscillation for some suspensions of magnesium powders in mineral oil (first sample) at 20° C.



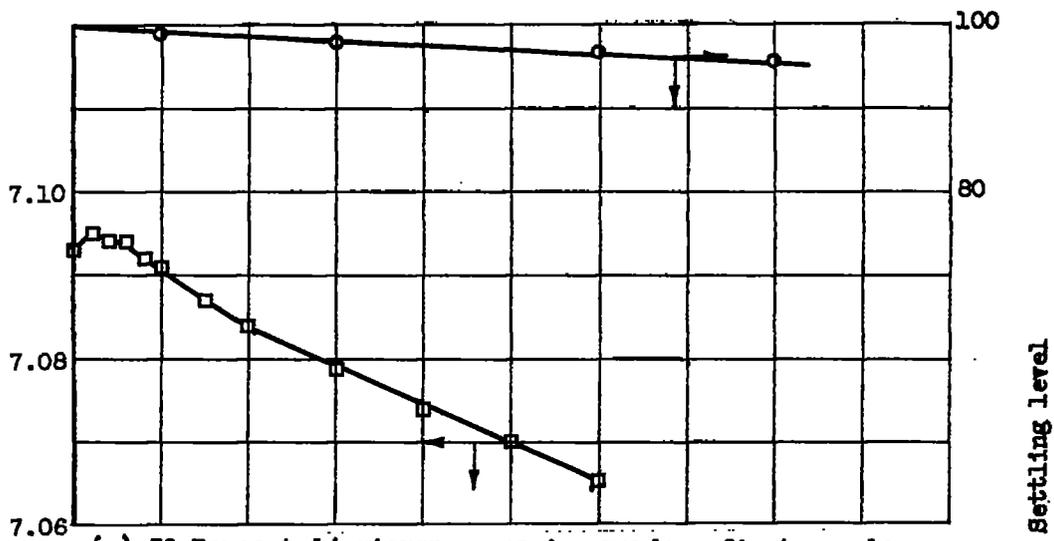
(a) 10 Percent 14-micron magnesium powder; first sample at 20° C.

Figure 9. - Variation of dielectric constant and settling level with time of magnesium powder in mineral oil.

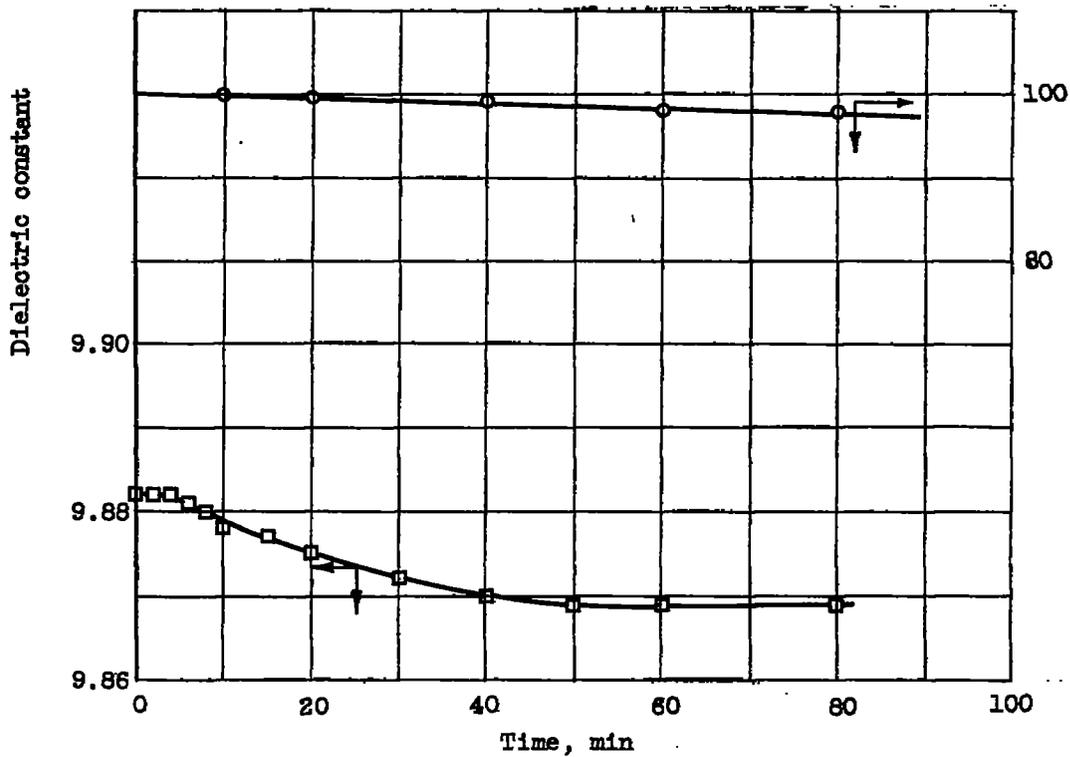


(b) 20 Percent 14-micron magnesium powder; first sample at 20° C.

Figure 9. - Continued. Variation of dielectric constant and settling level with time of magnesium powder in mineral oil.

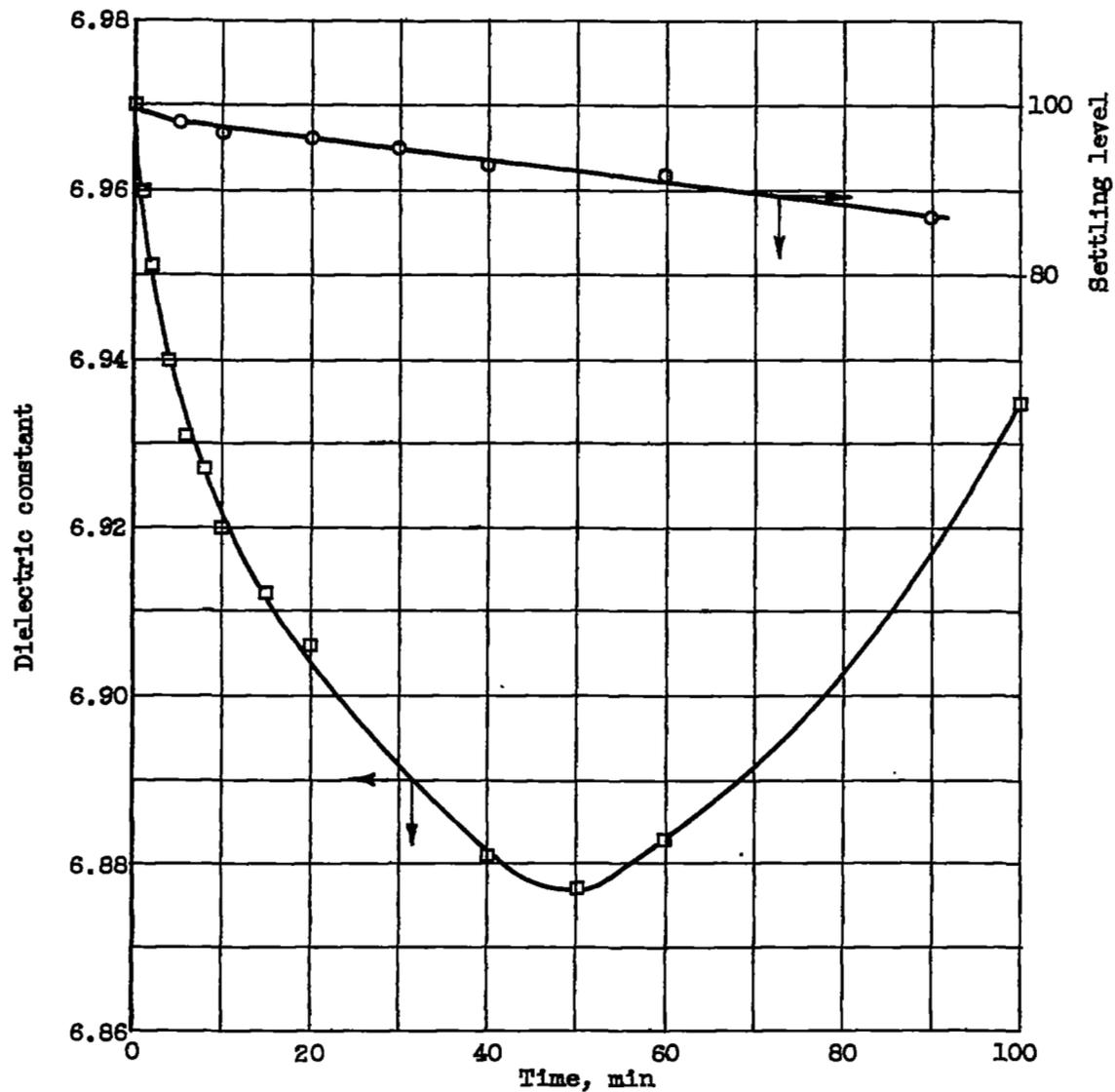


(c) 50 Percent 14-micron magnesium powder; first sample at 20° C.



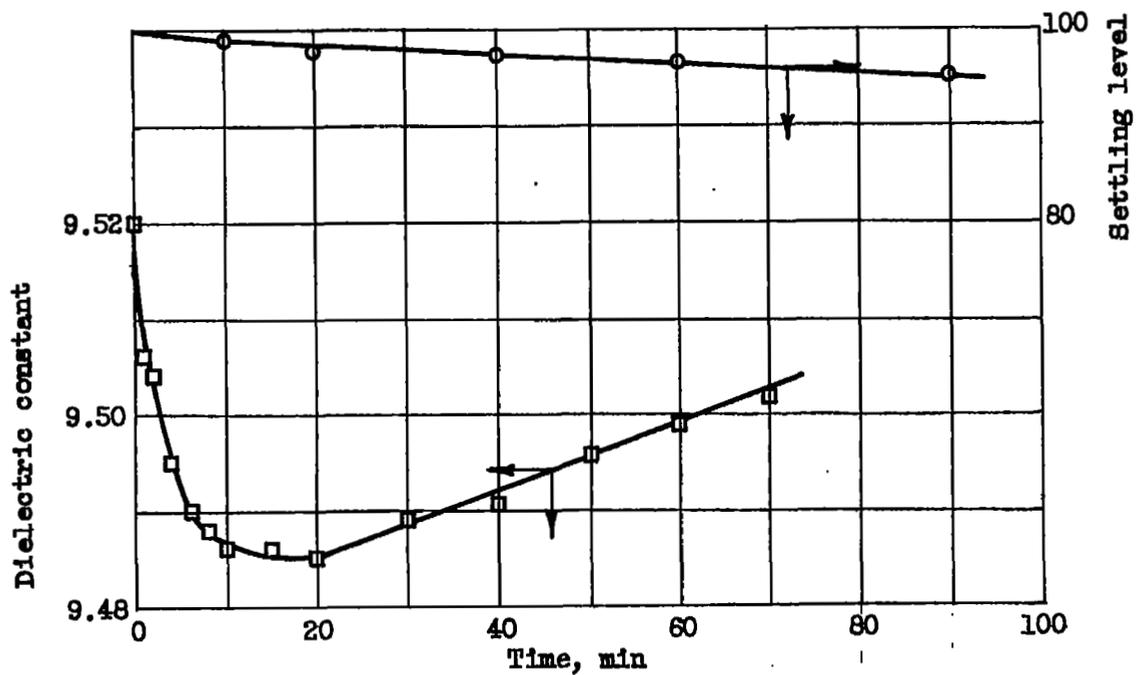
(d) 60 Percent 14-micron magnesium powder; first sample at 20° C.

Figure 9... Continued. Variation of dielectric constant and settling level with time of magnesium powder in mineral oil.



(e) 50 Percent 14-micron magnesium powder; first sample at 50° C.

Figure 9. - Continued. Variation of dielectric constant and settling level with time of magnesium powder in mineral oil.



(f) 60 Percent 14-micron magnesium powder; first sample at 50° C.

Figure 9. - Continued.. Variation of dielectric constant and settling level with time of magnesium powder in mineral oil.

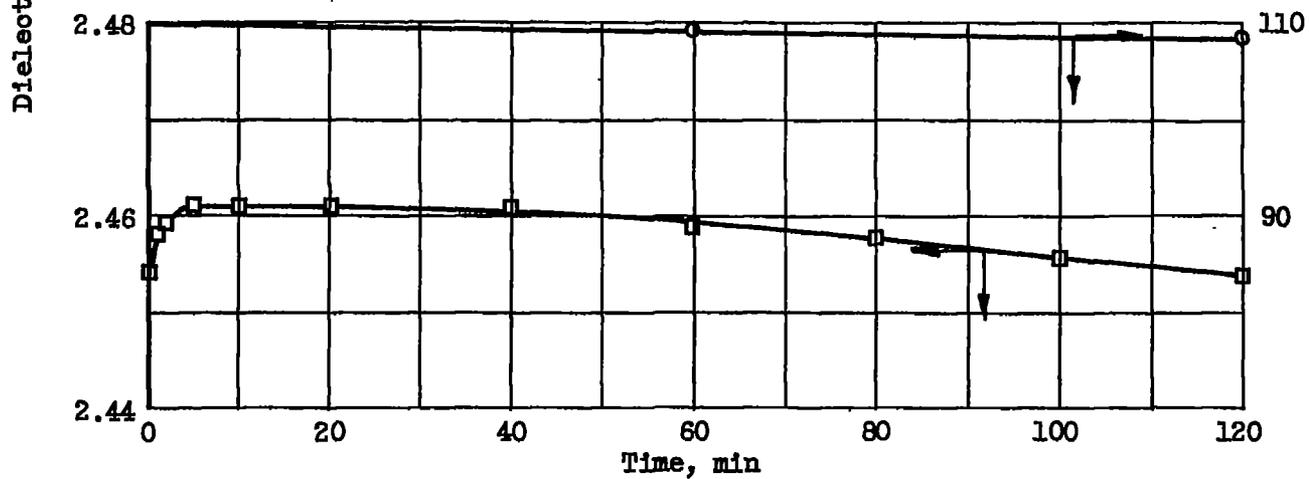
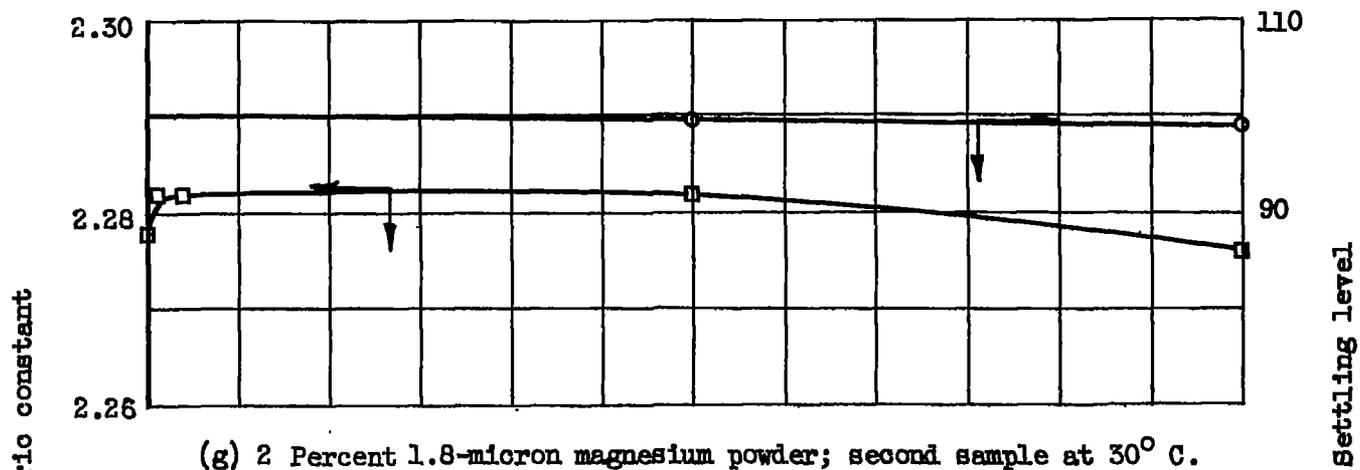


Figure 9. - Continued. Variation of dielectric constant and settling level with time of magnesium powder in mineral oil.

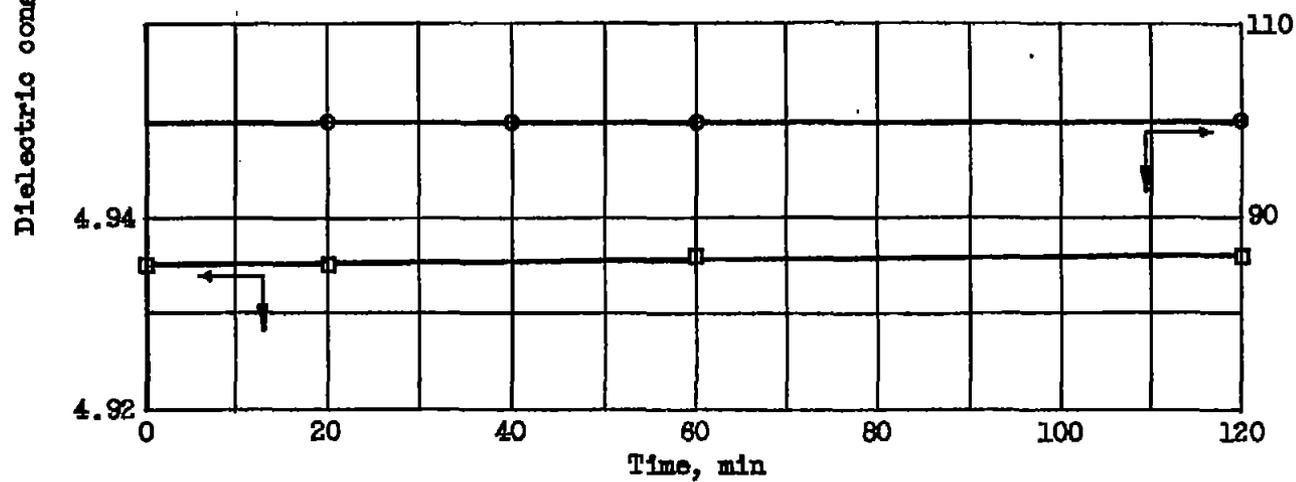
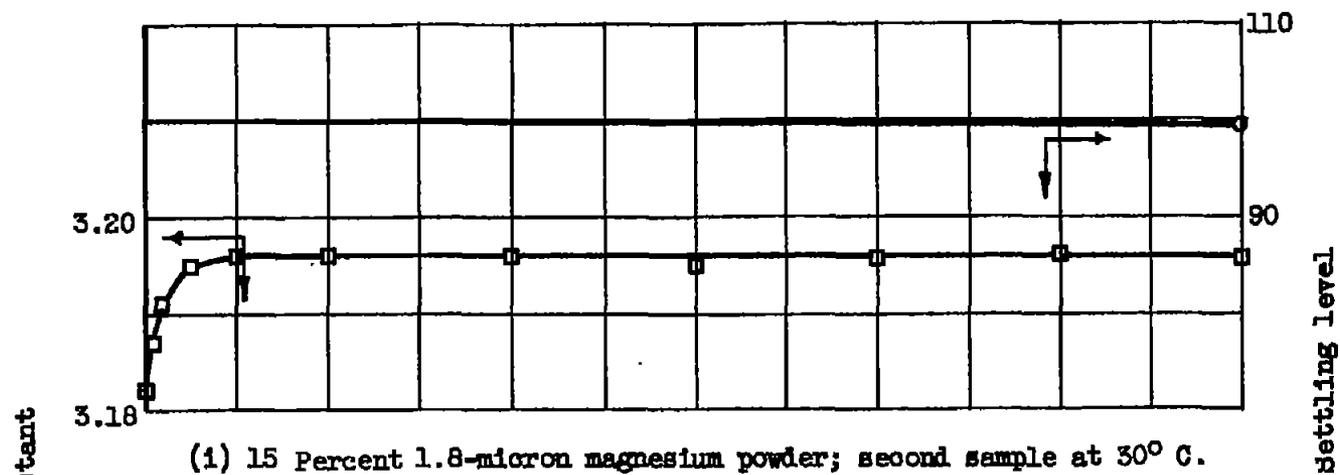


Figure 9. - Concluded. Variation of dielectric constant and settling level with time of magnesium powder in mineral oil.

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