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A STUDY OF THE DIELECTRIC CONSTANT OF CHARGE MATERIAL

Summary

Charge material made in either a vapor or liquid phase reactor was found to have a dielectric constant of 3.2 which was quite independent of frequency and temperature. However if the TCl_4 was sublimed or melted and then cooled, the dielectric constant became variable, increasing with decreasing frequency and rising temperature. The temperature dependence was in the opposite direction from that given in the Debye equation. Several theories are postulated to explain the dielectric behavior of charge material but none of them is satisfactory.

Preliminary Discussion

If we were to measure the capacity (C_1) of an evacuated condenser and then were to fill it with some material substance and remeasure the capacitance (C_2) we would find that the latter capacitance is larger. If this experiment were repeated with various sizes and shapes of condensers we would find that the ratio C_2/C_1 is constant for any one material. This constant is called the dielectric constant. It can be shown that this same constant gives the ratio of the electric fields, E_1/E_2, at any point in the condenser and that it also is equal to the square of the ratio of the velocity of propagation of the electromagnetic waves in the condenser under the two conditions of experiment. Any of these relationships can also be used as a definition of dielectric constant.

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Approved for public release
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Now it happens that the material media on which dielectric measurements are made also have a measurable resistance, R. Thus, although the impedance of an evacuated condenser is $\frac{-j}{\omega C}$ (where ω is 2π times the frequency of the impressed A.C. voltage, C the capacitance of the condenser, and $j=\sqrt{-1}$), that of a condenser filled with a material is not merely $-j/\omega C\epsilon$ (where ϵ is the dielectric constant), but $-j/\omega C\epsilon + R$. This expression can be simplified by writing the impedance as

$$\frac{-j}{\omega C(\epsilon' - j\epsilon'')} \qquad (1)$$

where $(\epsilon' - j\epsilon'')$ can be called the complex dielectric constant. ϵ' is intimately connected with the old ϵ, and ϵ'' with R. In experimental papers ϵ' is called the dielectric constant.

Before we describe the actual mechanics of the measurements we shall indicate how we came to investigate this problem. This laboratory was asked to determine the dielectric constant of charge material for use in production. It was found (CD-462) that at 1000 cycles a sample of sublimed charge material had a dielectric constant of about 10 whereas the dielectric constant of samples of unsublimed materials (made by either the liquid or vapor phase process) was about 3. We were immediately interested in the possible reason for the different behavior of the sublimed and unsublimed samples and wondered whether an extended study of the effect might not clarify some production problems. For example, do these two dielectric constants indicate that there are two different crystalline forms of charge material with different vapor pressures?

In order to answer the above question and to clarify the situation a study was made of the frequency and temperature dependence of the complex dielectric constant of samples of charge material prepared or treated by different methods. A block

diagram (Fig. 1) of the experimental set up appears at the right. We can consider as the independent variables in our experiments the frequency, $\frac{\omega}{2\pi}$, and the absolute temperature T; the dependent variables were the readings on the C and the $R\omega C$ dials of the bridge. An oscillograph was used to indicate balance of the bridge. The C and R refer respectively to the equivalent series capacitance and the equivalent series resistance of any impedance measured with the bridge. Expressing the impedance in terms of equation (1) it can readily be shown that:

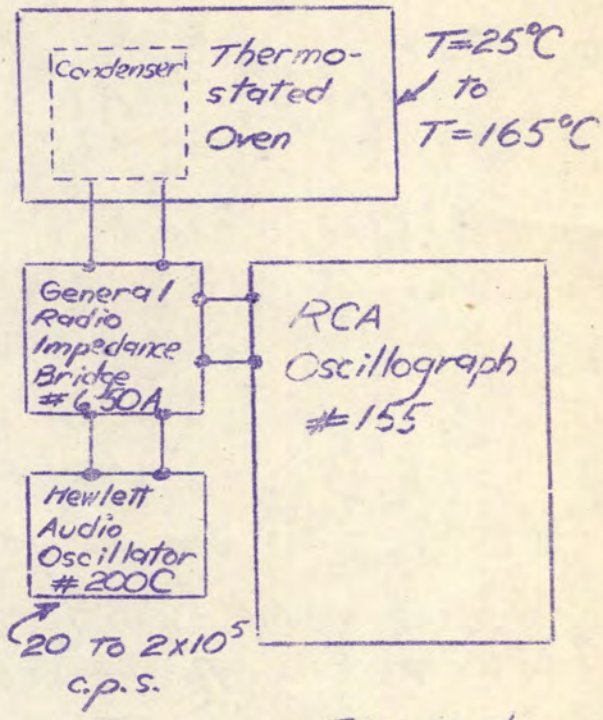


Figure 1

$$\epsilon' = \frac{1}{C_E}\frac{C}{1+(R\omega C)^2}; \quad \epsilon'' = \frac{1}{C_E}\frac{C(R\omega C)}{1+(R\omega C)^2} \qquad (2)$$

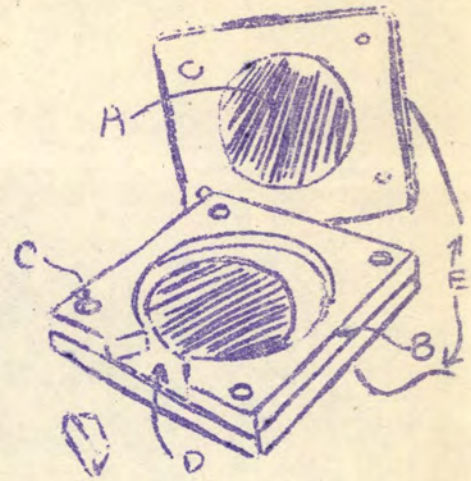
where C_E is the capacitance of the empty condenser. Thus ϵ' and ϵ'' can be considered as dependent variables.

Several condensers were used in the course of these experiments. Figure 2 illustrates their general construction. Since TCl_4 is very hygroscopic, the condensers were always filled in a dry box and sealed shut.

Before the data are considered, a few of the classical ideas on the subject of dielectric constant will be reviewed. The simplest dielectric medium is a gas consisting of non-polar molecules far enough apart to have negligible interactions. If an electric field were applied to the gas, the molecules would have their center of negative charge displaced relative to the center of positive charge. The dipole momement, m, thus induced, is found to be proportional to the electric field, E, acting on each molecule, i.e. $m = \alpha E$, where α is called the polarizablity. From

this equation and the concept that the dielectric constant is equal to the ratio of electric fields E_1/E_2 at any point in the condenser, the well known Clausius-Mosotti equation is derived, (N being Avogadro's number, and M/ρ the ratio of the molecular weight to the density):

$$\frac{(\epsilon-1)M}{(\epsilon+2)\rho} = \frac{4\pi N\alpha}{3} \qquad (3)$$



A. Metal Condenser Plate on insulator; Area 300 cm^2.
B. Porcelain or lucite spacer width .5 cm.
C. Bolt Holes.
D. Opening for filling Condenser.
E. Lead Wires.

Fig. 2

If, however, the gas molecules possess their own permanent dipole moments, the above expression must be extended to include the contribution of these dipoles to the dielectric constant; for the dipoles will tend to rotate in the direction of the field. The dipole moment per unit volume so produced must be considered as well as the one that is induced. It was Debye who first carried out this extension to the theory. He showed that if we assume Boltzmann's law for the distribution of energy among the molecules of such a gas we find that:

$$\frac{\epsilon-1}{\epsilon+2}\frac{M}{\rho} = (4/3)N\pi\alpha + (4/3)N\pi\frac{\mu^2}{3KT}\left(\frac{1}{1+j\omega\tau}\right) \qquad (4)$$

where μ is the permanent dipole moment, k is Boltzamann's constant, τ is the relaxation time (i.e. the time for 1/e of the lined-up molecules to return to random distribution upon removal of the field).

If we assume that the total ohmic resistance of the dielectric is due to the permanent dipoles, we can set $\epsilon = \epsilon' - j\epsilon''$. Then equation (4) can be rewritten to show

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the theoretical dependence of ϵ' and ϵ'' on the frequency and temperature for polar gases. Instead of retaining α, N, k, μ, etc. it is customary to use the more general parameters:

ϵ_0, the dielectric constant at optical frequencies, (i.e. ϵ' for $\omega = \infty$)

ϵ_s, the static dielectric constant, (i.e. ϵ' for $\omega = 0$)

and $\beta = \left(\frac{\epsilon_s + 2}{\epsilon_0 + 2}\right)\tau$.

In terms of these we get

$$\epsilon' = \epsilon_0 + \frac{\epsilon_s - \epsilon_0}{1 + \beta^2\omega^2}$$

$$\epsilon'' = \frac{(\epsilon_s - \epsilon_0)\beta\omega}{1 + \beta^2\omega^2} \qquad (5)$$

The general shape of these curves is given in (Fig. 3). At low frequencies there are long periods of rest between dipole rotations and thus ϵ' is large and fairly constant. At high frequencies the amplitude of dipole rotation approaches zero because of the relative sluggishness of the permanent dipoles; in fact ϵ_0 is entirely due to induction. The permanent dipoles therefore do the most moving at intermediate frequencies (the region of dispersion) and one would expect that the power expenditure (and hence ϵ'') would have a maximum here.

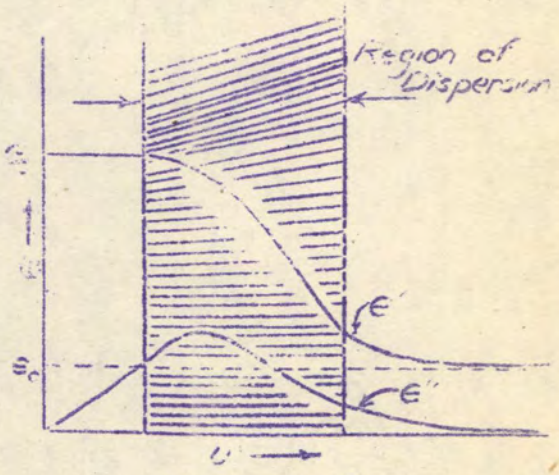

Figure 3

Although equation (4) was derived on the assumption that we were dealing with a polar gas, equations (5) are somewhat more general. They can in fact be derived without reference to dipoles at all. The qualitative discussion in the preceding

paragraph would hold for any polarization phenomenon of slow response, whether it be for dipoles in the more complicated fields in a liquid or solid, or for films of ions moving on crystal surfaces, etc. It is not too surprising therefore that equations (5) have been found to describe fairly well the behavior of the dielectric constant of solid dielectrics. We have fitted our experimental results to these equations and sought values for the parameters ϵ_0, ϵ_s, and β for each sample of material that exhibited variation of dielectric constant with temperature and frequency.

In many cases where equations (5) were fitted to data on various solid dielectrics it was found necessary to assume either several values of β, a continuous band of β values, or both in order to approximate the experimental curves. This is physically reasonable inasmuch as a single relaxation time implies the existence of a fixed resistance of the surrounding medium and a fixed elastic constant. But in a solid the coupling between molecules is complicated, and the resistant and elastic constants vary with orientation. Hence a series of discrete values or some continuous distribution of β would seem logical.

Numerical Results

There is a distinct difference in the dependence of dielectric constant on frequency between TCl_4 samples that had been sublimed and those that had not.

The unsublimed TCl_4 samples all showed very little frequency dependence of ϵ'. The general trend was a 2-15% decrease in ϵ' from the value at 100 cycles (the lowest frequency obtainable with the bridge) to that at 3000 cycles. From this point to 200 kilocycles (the high frequency limit of our apparatus) ϵ' remained fairly constant. Graph I on the next page gives the curve for a typical sample. Since even at low frequencies the rate of change of ϵ' with frequency was slow and since the bridge is unreliable at these frequencies, we may consider ϵ' for unsublimed materials to be independent of frequency.

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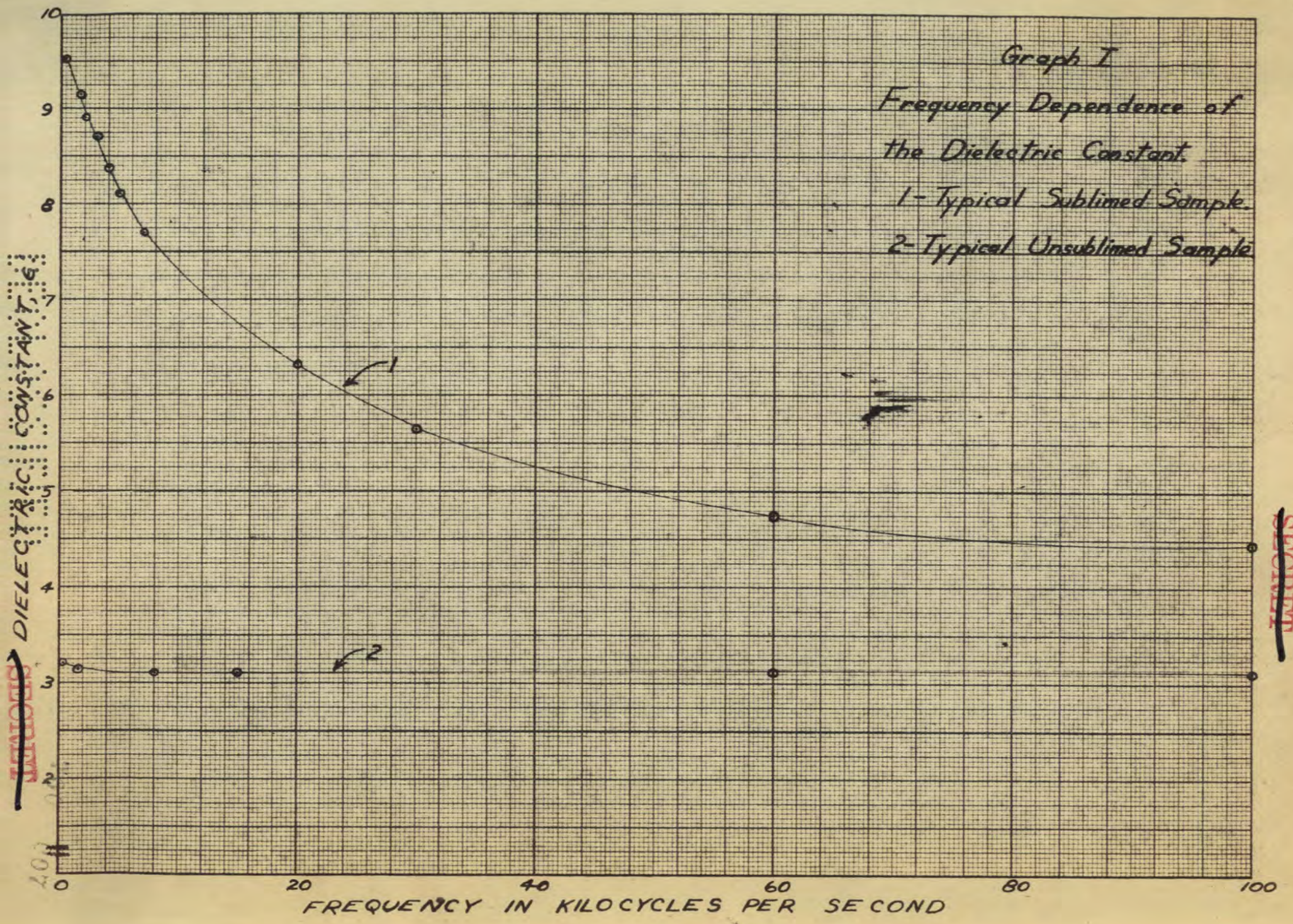

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In Table I are listed the stable (high frequency) values of ϵ' for several samples. It should be noted that the value of ϵ' does not depend on the method of preparation of the unsublimed charge material. The dielectric constant of TCl_4 made in a vapor phase (V.P.) reactor approximately equals that of the tetrachloride from a liquid phase (L.P.) reactor.

TABLE I

Dielectric Constant of Unsublimed Charge Material

Sample	Preparation	
A	V.P.	3.2
B	V.P.	3.2
C	L.P.	3.1
D	L.P.	3.5
E	L.P.	3.7
F	L.P.	4.1

Samples of sublimed charge material from production all showed a marked dependence of dielectric constant on frequency. (See Graph I for a typical experimental curve.) The values of the parameters ϵ_s and ϵ_o obtained from the data for several samples appear in Table II.

The data for each sample consists of a series of values of C and $R\omega C$ for various frequencies. In addition we have the capacity C_o of the empty condenser and the capacity of the lead wires to the condenser which is subtracted from C and C_o before the ratio C/C_o is taken. ϵ' and ϵ'' were calculated at each frequency according to equations (2). It was found that ϵ'' did not go through a maximum as in Fig. 3., but was a monotonic decreasing function of frequency. But the fact that the probable error of the $R\omega C$ reading at low frequencies was 20% and the existence of ionic conduction through the charge material would imply that the ϵ'' results should not be expected to fit equation (5) at low frequencies. The case with ϵ' is different. Although the $R\omega C$ readings do influence its value somewhat at low frequencies, it is mostly determined by C.

The values of ϵ_s in Table II were obtained by graphical extrapolation of the curve to zero frequency, neglecting those points for which $R\omega C$ exceeded three tenths. It was impossible to make a direct measurement of ϵ_s; for although it is quite easy

to measure the capacitance of an air condenser by discharging it through a ballistic galvanometer, a condenser full of charge material does not discharge in a short enough time for the measurement to be ballistic.

The values of ϵ_0 in Table II were also obtained graphically. Since the slope of the ϵ' curve is

$$\frac{-8\pi^2\beta^2 f(\epsilon'-\epsilon_0)^2}{\epsilon_s-\epsilon_0},$$

a value for ϵ_0 and for β can be obtained (assuming the extrapolated ϵ_s) from the consideration of any pair of points on the experimental curve. Many pairs of points were used and the various values of ϵ_0 for any one sample were found to be consistent. The average value of ϵ_0 for each sample appears in Table II. From the shape of the curves we might assume that there is a band of β values around $\beta = 10^{-5}$. The theoretical curves based on $\beta = n \times 10^{-5}$ for several values of n are compared with an experimental curve in Figure 4.

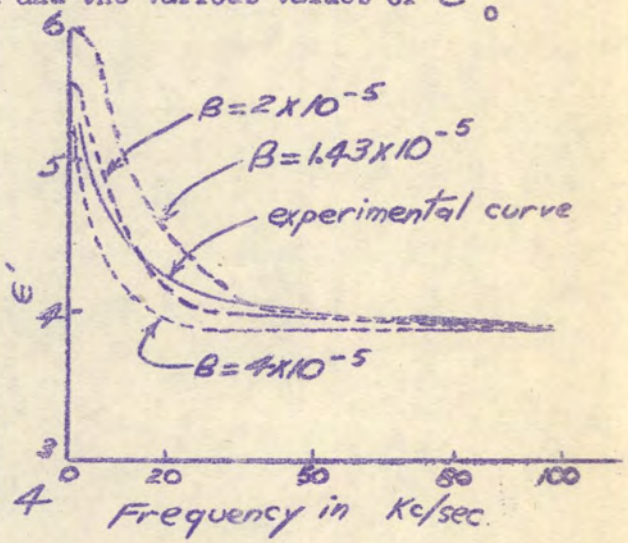

Figure 4

From an inspection of Table I and Table II, the reader will note that the values of ϵ_0 for the sublimed material are nearly equal to the values of ϵ' for the unsublimed material. Since it is usually assumed that the dielectric constant at high frequencies, or ϵ_0, is entirely due to induction, the values of ϵ' for the unsublimed material can be visualized as being due to molecular polarization alone. Subsituting $\epsilon' = 3.2$ in equation (3) and using the packing density (2.5 g./ml) instead of the real density (4.7), the polarizability of TCl_4 is found to be 2.5×10^{-23}.

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On the assumption of the molecule being a perfectly conducting sphere, this corresponds to a molecular radius of 2.9 Å, which is a reasonable value.

TABLE II

Dielectric Constant of Sublimed and Specially Treated Charge Material

Sample	Preparation	Special Treatment	Static Dielectric Constant, ϵ_s	High Frequency Dielectric Constant, ϵ_o
G	Sublimed	None	9.7	4.2
H	Sublimed	None	10.6	4.7
I	Sublimed	None	12.2	4.5
J	Sublimed	None	10.4	4.3
K	Sublimed	None	10.4	4.2
L	Sublimed	None	13.6	6.0
D	Unsublimed	After Melting in a Bomb (p.10)	7.0	2.8
M	Unsublimed	After Melting in a Bomb (p.10)	10.0	3.9
N	Unsublimed	After Melting in a Bomb (p.10)	12.2	5.4
I	Sublimed	After Melting in a Bomb (p.10)	11.5	5.5
G	Sublimed	Heated to 400°C (p.10)	5.7	3.9
C	Sublimed	Heated to 400°C (p.10)	3.22	2.9
L	Sublimed	Resublimation in Glass App.	4.7	3.2

In Graph II the effect of temperature on the variation of dielectric constant with frequency is given for a particular sample of sublimed material. We feel that the curves represent values very near equilibrium since they were approached both from higher and lower temperatures (see Figure 5). The rate of achievement of temperature equilibrium is slow, for ϵ' did not become constant until the condenser was held at a given temperature for about 24 hours. Furthermore it was necessary to preheat the charge to 400° C and seal the laden condenser to achieve reproducibility.

The unsublimed material showed very small temperature dependence of dielectric

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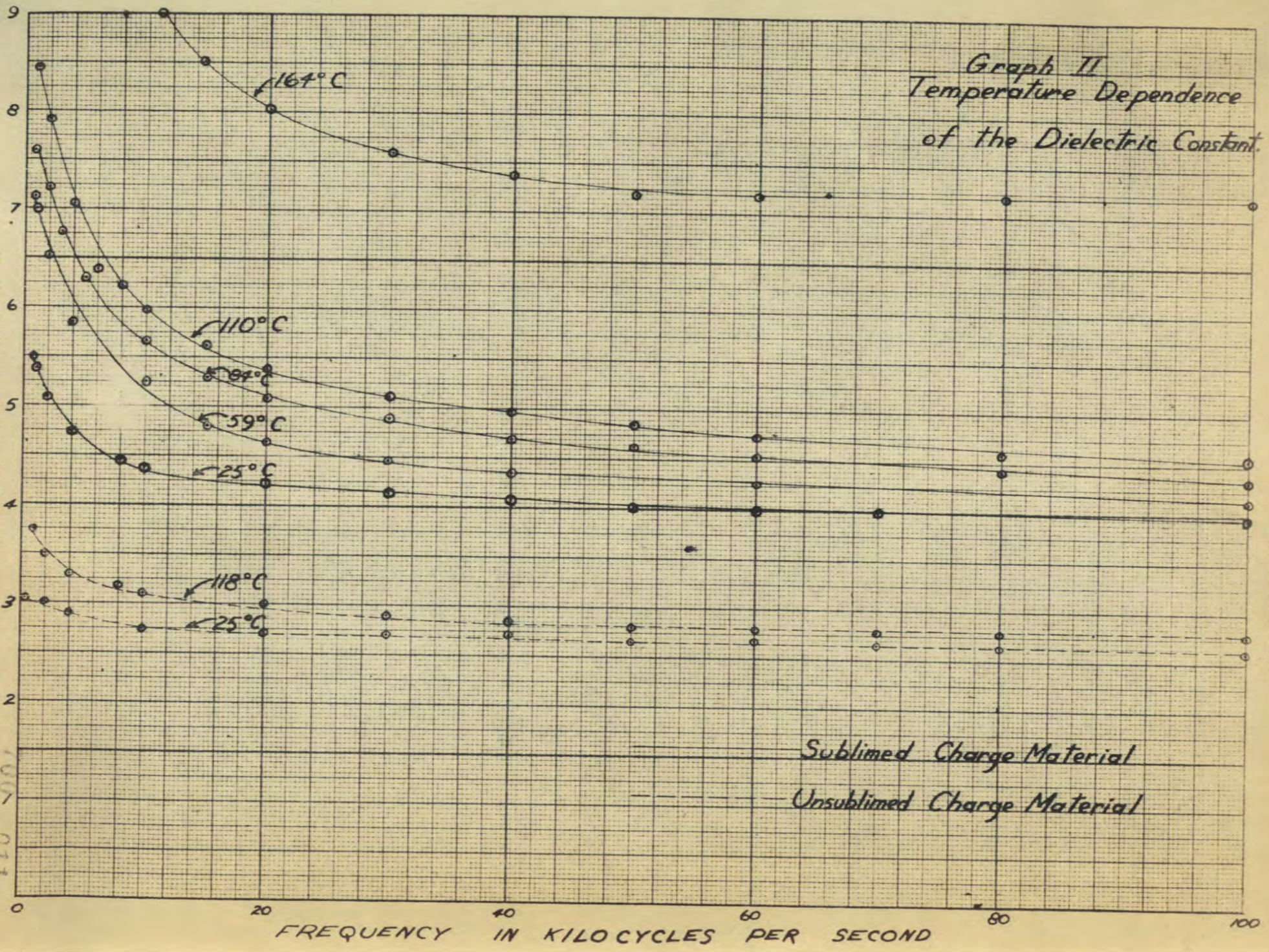

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constant, compared to that noted for the sublimed material (Graph II).

In order to determine the cause of the above experimental effects, several rather qualitative studies were made. When unsublimed charge material was heated up about 200° C above the melting point (Ca. 580° C) in a stainless steel bomb and then cooled to room temperature, the dielectric constant of the material that had been melted varied with frequency similarly to the sublimed material. Whether the cooling of the melt was done quickly (in several seconds by dry ice) or slowly (by lowering the furnace temperature stepwise so that more than 24 hours were needed to reach room temperature), the variation of dielectric constant with frequency after cooling was the same for any given melt. Heating sublimed charge material until it melted and then cooling it caused no appreciable change in the shape of the dielectric constant-frequency curves.

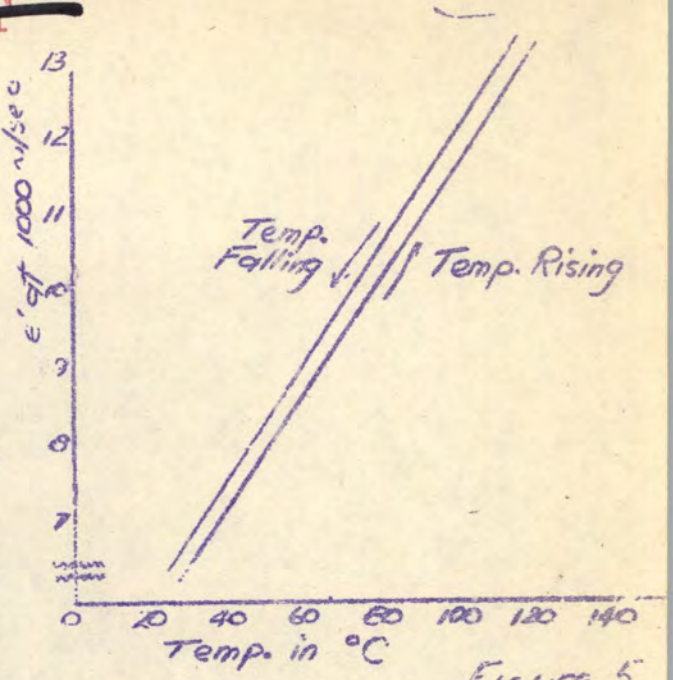

Figure 5

On the other hand when the same sublimed material was heated only to 400° C and then cooled, ϵ_s was somewhat reduced. (Incidentally this reduced value of ϵ_s was reproducible when the temperature was varied, whereas ϵ_s for the unheated samples was not. This is the reason that the samples used to study the temperature variation were preheated.) Heating unsublimed charge material had a comparatively small effect.

Resublimation of sublimed material in an evacuated glass apparatus caused ϵ_s to be reduced. This is an interesting effect and might be ascribed to either purification or a difference in the temperature of the condensing surfaces of the production and glass sublimation equipment.

As can be seen in Figure 6, the dependence of ϵ_s, on temperature for the sub-

limed charge material (data from Graph I) is the opposite from that which is predicted by Debye. According to the Debye equation ϵ_s decreases with increasing temperature, in fact:

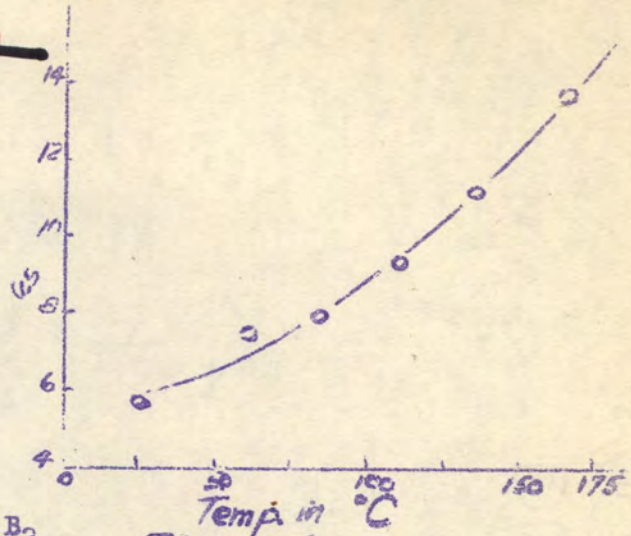

Figure 6
(see equation (4))

$$\frac{\epsilon_s - 1}{\epsilon_s + 2} = B_1 + \frac{B_2}{T} \qquad (6)$$

But in our experiments ϵ_s increased with rising temperature. However it is possible to make our data fit the Debye relationship if we assume that the amount of material in which free dipole rotation occurs increases with the temperature.

Accordingly, it is postulated that there exists an equilibrium between a fraction of the material with variable dielectric constant (see equation (6)) and the remainder. The dielectric constant of this remainder is constant and is reasonably taken to be ϵ_o. Mathematically expressed:

$$K = Ae^{-\frac{\Delta H}{RT}} \qquad (7)$$

where K is the equilibrium constant and ΔH, the heat of reaction for the transition, at any temperature, T. A is simply a constant.

If the additional assumption is made that the dielectric of the mixture is related to those of the components through*

$$\epsilon = \frac{\epsilon_1 \epsilon_2}{P_2 \epsilon_1 + P_1 \epsilon_2} \qquad (8)$$

(where P_1 is the mole fraction of the first material etc. i.e. $K = \frac{P_1}{P_2}$) then the measured ϵ can be expressed in terms of the temperature T. In fact substitution

*There is no general relationship for the dielectric constant of a mixture of solids. The one given here is based on the assumption that the solids are separated into 2 parallel layers. The error is fortunately small even for such an oversimplification.

from equations (6), (7) and (8) readily gives:

$$\epsilon = \frac{\epsilon_0\left[Ae^{-\frac{\Delta F}{RT}} + 1\right]\left[\frac{1 + 2B_1 + \frac{2B_2}{T}}{1 - B_1 - \frac{B_2}{T}}\right]}{\epsilon_0 + \left[\frac{1 + 2B_1 + \frac{2B_2}{T}}{1 - B_1 - \frac{B_2}{T}}\right]Ae^{-\frac{\Delta F}{RT}}} \qquad (9)$$

and if this ϵ vs. T curve is fitted to the experimental one, the parameters ΔF, A and μ can be evaluated. The result for our data is:

$\Delta F =$ 2.7 KCal/mole
$\mu =$ 3.7 Debyes
$A =$ 13.5

which makes K = .14 at room temperature and .44 at 100° C.

Implications

The main results that have been established are that the dielectric constant of sublimed charge materials varies with the temperature and frequency. The variation for unsublimed material is relatively negligible.

Our object in this section is to look into possible explanations of this difference in behavior of the two materials and to account as far as possible for the shape of the observed curves.

What then causes the dispersion in sublimed TCl_4 charge material? It is either the TCl_4 itself or some impurity. Let us deal with the latter possibility first. Unsublimed charge material as supplied to us contains the following impurities, listed in the order of their usual abundance: $TOCl_2$, HCl, TCl_5, CCl_4, phosgene, and usually some hexavalent tuballoy compounds as well as some salts of other metals. After production sublimation and the usual inadvertent exposures to the air, charge material contains $TOCl_2$, HCl, $T_2O_2Cl_2$, $CrCl_3$, $NiCl_2$, $FeCl_3$ and some other T-O-Cl

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compounds. The total amount of impurity in any sample is usually less than 2%. We measured the dielectric constant of $TOCl_2$ and TCl_5 and found $\epsilon = 2.4$ and 1.9 respectively with no appreciable variation with frequency. It may, however, be argued that these substances (and other of the impurities) have polar molecules which are free to rotate when they are impurities in the TCl_4 lattice, but are tightly bound in their own crystal lattice. But even this assumption leads to insurmountable difficulties, as will now be shown:

The Debye equation for a polar gas can be written:

$$\mu = \left(\frac{9MkT}{4\pi N\rho}\left[\frac{\epsilon_s - 1}{\epsilon_s + 2} - \frac{\epsilon_o - 1}{\epsilon_o + 2}\right]\right)^{1/2} . \qquad (10)$$

Assuming that the dispersion causing impurity is spread as a gas throughout the charge, this formula and our data would permit the calculation of an upper bound of $\left(\frac{M}{\rho}\right)_{gas}$. The largest values of μ found in the literature are about 3 Debyes (i.e. 3×10^{-18} e.s.u.-cm) so that assuming the impurity has so large a dipole moment

$$\left(\frac{M}{\rho}\right)_{gas} \leq \frac{4\pi N}{9kT}\left[\frac{9 \times 10^{-36}}{\frac{\epsilon_s - 1}{\epsilon_s + 2} - \frac{\epsilon_o - 1}{\epsilon_o + 2}}\right]. \qquad (11)$$

Taking a typical sublimed sample, we can substitute the values of ϵ_s and ϵ_o for a given temperature in the preceding inequality (although ϵ_o would actually be due to the polarization of TCl_4 molecules as well, the assignment of the full ϵ_o to the impurity merely strengthens the inequality). We find:

$$\left(\frac{M}{\rho}\right)_{gas} \leq 600$$

or the moles per cm^3 would exceed $\frac{1}{600}$. But the impurity is not really dispersed as a gas. And although the relation between μ and ϵ for solids is unknown, it is certainly true that in this case the dipoles will be less free to rotate than in a gas and to get the same observed ϵ for a given μ there would have to be more moles

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per c.c. than for the gas. Hence it is certainly true that if one assumes that a polar impurity causes the dispersion, its concentration must be least 1/600 moles/c.c. of TCl_4. The molar concentration of the impurity would have to be greater than 1/5 that of the TCl_4. If the impurity were one of large molecular weight, it would form an appreciable part of the charge material, and this is not in accord with chemical analysis. Any polar impurity would therefore have to be of low molecular weight.

Since great quantities of HCl are driven off upon heating the charge material and HCl is known to be polar ($\mu = 1.03$ Deb.), one might assume that HCl is the light impurity that is sought. But μ is low for HCl and one would have to assume that HCl is present in molar concentration twice that of the TCl_4 to account for the observed dielectric constant. This too is at variance with known facts. (Furthermore the unsublimed material seems to adsorb as much HCl as the sublimed, and aerating charge material in steam, $H_2O + TCl_4 \rightarrow TOCl_2 + 2HCl$, does not raise the dielectric constant.) Thus it appears that HCl is not the impurity we are after. In general, any polar impurity would have to be assumed in greater concentration than possible to explain the observed dispersion.

It seems likely too, that the assumption of an ionized impurity which would cause the dispersion by displacement of ions (located in crystal imperfections, or surface films, for example) would hardly account for the observed results unless very special conditions were assumed.

It appears therefore that the dispersion should be explained on the basis of the polarity of TCl_4 itself. An X-ray analysis (A-2210) by Money indicated that the TCl_4 molecule is non-polar in a crystalline sample supplied by Kraus. However we might make the rather improbable assumption that the gaseous TCl_4 molecule is polar and is merely distorted into a symmetrical non-polar shape in the crystal. Accordingly the unsublimed material would be crystalline. The sublimed material on the other hand

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would have a freer structure, (Perhaps it would be amorphous, since solids deposited from their vapors often are amorphous, especially if the receiving surface is cold.) and its molecules would be relatively undistorted and hence polar. To explain the observed dependence of ϵ_s on temperature, the freedom of rotation of these dipoles would be assumed to vary with temperature.

Although this hypothesis seems to conflict least with experiment, it strains credulity sufficiently to make it an unacceptable explanation of the data. One might ask, "If some molecules in the sublimed material are free to rotate in the electric field, why can they not rotate into such a position that they will go into the crystal state?" Moreover the dipole moment calculated on page 12 is extremely large. It is very unlikely that a molecule with such a moment can be strained sufficiently in a crystal lattice to reduce the moment to zero. Thus it appears that we are at present no more able to derive an adequate explanation of our observations on the assumption of polar TCl_4 than on the assumption that the charge material contains some polar impurity.

Conclusion

Since this data cannot be readily fitted to a variation of the standard Debye treatment it appears that it is in the class of experimental results on dielectric phenomena in solids which call for a basically new theory.

From the dielectric data alone no definite conclusion can be reached about the existence of two forms of charge material at room temperature. In order to clarify this matter X-ray diffraction studies are being made on our samples elsewhere (see M2120). We are also trying to determine if there is an appreciable heat of transition between sublimed and unsublimed charge material by measuring heats of solution. If there is a heat of transitition, it will naturally affect the vapor pressure and the

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rate of sublimation.

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