RESEARCH PAPER RP585

Part of Bureau of Standards Journal of Research, Vol. 11, August 1933

EFFECT OF TEMPERATURE AND FREQUENCY ON THE DIELECTRIC CONSTANT, POWER FACTOR, AND CON-DUCTIVITY OF COMPOUNDS OF PURIFIED RUBBER AND SULPHUR

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ABSTRACT

The dielectric constant, power factor, and conductivity of purified rubber and of a series of its compounds with sulphur were determined at temperatures from -75° to 235° C. The dielectric constant and power factor were measured at five frequencies from 60 to $300,000 \sim$ and the apparent conductivity at about 0.002 second and at one minute after the application of potential. The results of the measurements are expressed in both tabular and graphic form. At 25° C. and $1,000 \sim$ the dielectric constant of purified rubber containing no sulphur was 2.37. With increasing sulphur content the dielectric constant increased to a maximum of about 3.75 at 11.5 percent sulphur, then decreased to a minimum of 2.70 at 22 percent sulphur and again increased to 2.82 at 32 percent sulphur. Under similar circumstances, the power factor increased from 1.6×10^{-3} for the rubber alone to a maximum of 93.8×10^{-3} for the compound containing 13.5percent sulphur; it then decreased to about 4.0×10^{-3} at 20 percent sulphur, and again slowly increased to 5.1×10^{-3} at 32 percent sulphur. The 1-minute conductivity was 2.3×10^{-17} mol/cm for the rubber alone. With 12 percent sulphur, it was only 0.5×10^{-17} , while at 18 percent sulphur. The 1-minute conductivity was 2.3×10^{-17} mol/cm for the rubber alone. With 12 percent sulphur, it compositions between 22 and 32 percent sulphur. Changes of the temperature or the frequency at which the measurements were made shifted the maxima and minima in these curves and modified their heights. For example, at -25° C, the maximum dielectric constant at $1,000 \sim$ was 2.8, and was obtained with a compound containing 4 percent sulphur, while at 145° C. the maximum was 4.5 and was obtained for a compound containing about 26 percent sulphur. Comparison of the results of this investigation on purified rubber with previous work done with crude rubber indicates that purification alters the values obtained for the electrical pr

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I. INTRODUCTION

The authors have previously reported results ¹ on the electrical properties of a series of rubber-sulphur compounds which were made from commercial rubber and were measured at 25° C. with $1,000 \sim$. The present paper reports results on the electrical properties of a similar series of compounds made from purified rubber and measured over a wide range of temperature and frequency. Sufficient data have been obtained to enable one to determine the dielectric constant and power factor for any composition of rubber and sulphur at any useful temperature and at any normal frequency.

The results obtained in this paper do not entirely confirm the conclusions of the previous paper. When the electrical measurements reported in that paper were plotted as functions of the percent of combined sulphur, curves were obtained which showed changes in direction at compositions corresponding to simple, empirical formulas. From this evidence as well as from a consideration of changes in density with composition, it was inferred that

The existence of a definite compound of empirical formula $(C_5H_8)_2S$ is indicated by all the properties which have been studied. Evidence for the other three compounds $(C_5H_8)_4S$, $(C_5H_8)_4S_3$, and $(C_5H_8)_4S_3$ is less complete.

Soon after that paper was published it was pointed out by Kimura, Aizawa, and Takeuchi² that the electrical properties are functions of the temperature, and by Kitchin³ that they are functions of the fre-quency at which measurements are made. Kimura and Namikawa⁴

 ¹ H. L. Curtis, A. T. McPherson, and A. H. Scott, Density and Electrical Properties of the System, Rubber-Sulphur, Part II. Electrical Properties of Rubber-Sulphur Compounds, B.S.,Sci. Papers, vol. 22 (S560), p. 398, 1927.
 ² S. Kimura, T. Aizawa, and T. Takeuchi, Dielectric Properties of the Rubber-Sulphur System, Inst. Elec.Engrs.J. (Japan) pp. 1274-1277, December 1928.
 ³ Donald W. Kitchin, Power Factor and Dielectric Constant in Viscous Dielectrics, Am.Inst.Elec. Engrs.J., vol. 48, p. 495, 1929.
 ⁴ S. Kimura and N. Namikawa, The Specific Gravity and Thermal Expansion of the Rubber-Sulphur System, Soc.Chem.Indust.J. (Japan), Supplemental Binding, vol. 32, p. 196B, 1929.

pointed out that the density and coefficient of expansion of the rubbersulphur compounds likewise varies with the temperature. Hence, the positions of the maxima and minima which were observed in the earlier paper and were attributed to the formation of definite compounds of rubber and sulphur bear no relation to the chemical structure of the vulcanized rubber.

The present paper is primarily concerned with the description of methods of experimental measurement of the electrical properties and the presentation of the results. A consideration of the results in the light of modern dielectric theory will form the subject of a separate paper.

II. PREPARATION OF SPECIMENS

The specimens were prepared by molding and vulcanizing thin sheets of different rubber-sulphur compounds between aluminum plates which later served as electrodes. The process of preparation consisted in purifying the rubber, mixing it with sulphur, molding it between prepared electrodes, and vulcanizing the specimen thus formed in an atmosphere of inert gas.

1. PURIFICATION OF RUBBER

The purification of rubber consisted in the digestion of crude rubber or latex with water at about 180° C., followed by washing with water, extraction with alcohol, and drying in an atmosphere of inert gas. The digestion hydrolyzed the proteins, while the washing and extraction served to remove the protein-hydrolysis products, the resins, the sugars, and part of the inorganic salts. The product, thus obtained, contained about 99.5 percent of rubber hydrocarbon. The principal impurities were inorganic salts, as indicated by ash, and residual quantities of resins and protein-hydrolysis products, as indicated by a few hundredths percent of oxygen and nitrogen. This method of purification afforded a simple and convenient method for making rubber hydrocarbon of reasonably high purity in lots of several kilograms. A detailed description of the method of purification and a discussion of the properties of the purified rubber is given elsewhere.⁵

2. MIXING OF RUBBER AND SULPHUR

The purified rubber was usually mixed with sulphur in an internal mixer in an atmosphere of nitrogen or carbon dioxide, but some batches were mixed carefully on a roll mill in air. The rubber and the sulphur were weighed separately and the batch was weighed after mixing. The weight of the batch seldom differed from the sum of the weights of the constituents by more than 1 part in 1,000. Any greater difference was due to a loss of sulphur by dusting and the requisite quantity of sulphur was added to correct this loss. The batches, after mixing, were stored in an atmosphere of inert gas until used for making specimens.

The sulphur employed was a commercial grade of ground, rhombic sulphur, specially prepared for use in rubber manufacture. Analysis indicated no impurities except a trace of ash.

⁵ A. T. McPherson, A Method for the Purification of Rubber and Properties of Purified Rubber, B.S. Jour. Research (RP449), vol. 8, p. 751, 1932.

3. ELECTRODES

The electrodes were circular disks cut from aluminum sheet and were approximately 0.025 cm in thickness. They were used in pairs 24 and 26 cm in diameter, respectively, as indicated in figure 1. The electrodes were of two types—one type was made from ordinary polished aluminum sheet, and the other from aluminum sheet perforated with fine holes. The perforated aluminum sheet used for this purpose had 97 perforations per square centimeter; each perforation was about 0.05 cm in diameter and was separated from adjacent perforations by about the same distance.

The electrodes of polished sheets were employed in measurements at 25° C. These ordinary electrodes were not satisfactory, however, for specimens that were to be measured over a range of temperature, because differences in thermal expansion frequently caused separation of the rubber and metal. For this reason, specimens that were to be employed in determining the effect of temperature were made with electrodes of perforated aluminum. Since the rubber was molded



FIGURE 1.—Cross section of molded specimen.

into the perforations, excellent attachment to the electrodes was secured at all times.

4. MOLDING OF SPECIMENS

The electrical test specimens were made by placing the requisite quantity of the unvulcanized rubber-sulphur stock in the form of a sheet about 1 cm thick between a pair of electrodes and pressing it out in a hydraulic press to form a sheet about 0.075 cm thick between the electrodes. The exact thickness was controlled by the use of a large spacing ring between the platens of the press. Enough of the rubber-sulphur stock was used to permit considerable overflow beyond the edge of the electrodes. The overflow carried most of the entrapped air with it, and thus minimized the quantity of air enclosed in the specimen. A large sheet of aluminum foil was placed on each side of the specimen in order to prevent the overflow from sticking to the platens of the press. The foil was left on the specimen during vulcanization and up to the time the electrical measurements were made, in order to protect the rubber from oxidation and contamination.

Care was exercised to secure a uniform thickness that could be measured with the accuracy requisite for the determination of the dielectric constant. The platens of the press were plane to within 0.01 cm over the entire bearing surfaces. The molding was done at about 100° C., and the specimen was left between the platens of the press for 10 minutes in order to permit the rubber to flow as much as it would. The best specimens that were obtained were uniform in thickness to within 2 percent of the thickness of the rubber, or about 15 μ , over their entire area, but in some cases the thickness of the specimens varied by as much as 5 or 10 percent. These large variations were traced to inequalities or irregularities in the platens and could be eliminated by regrinding which was done whenever they were discovered.

5. VULCANIZATION OF SPECIMENS

After molding, the specimens were vulcanized in an autoclave. Those made during the early part of the investigation were vulcanized in open steam, but the majority of the specimens were vulcanized under pressure in an autoclave filled with carbon dioxide, hydrogen, or nitrogen. An autoclave for this purpose was improvised from a standard 12-inch iron pipe cap on which a rigid iron plate was bolted as a cover. A gasket made from a ring of wire solder effectively prevented the leakage of gas.

The time of vulcanization in the gas-filled autoclave was between 25 and 40 hours at 140° C., with the gas pressure at 50 or 60 lbs./in.² in excess of the pressure of the atmosphere. Vulcanization in steam was conducted for about 15 hours at 149° C. This relatively long time of vulcanization was intended to bring practically all the sulphur into combination with the rubber. Analysis indicated that this purpose was accomplished in the case of all specimens except those containing the maximum amount of sulphur. Specimens compounded with 32 percent of sulphur were found to contain 0.5 to 0.8 percent of sulphur in the uncombined state. Those compounded with 28 percent of sulphur contained 0.1 to 0.2 percent of free sulphur, while the specimens of lower sulphur content contained not more than 0.1 percent of the element in the uncombined state.

III. MEASUREMENT OF THE DIMENSIONS OF SPECIMENS

The dielectric constant and the conductivity of the material of a flat specimen with ordinary electrodes can be computed from the results of electrical measurements, the thickness between the electrodes, and the area of the smaller electrode.

That, however, cannot be done for specimens having perforated electrodes. Here the electrical properties may be arrived at indirectly by determining experimentally a shape factor for each specimen—a procedure which amounts to finding the dimensions of a parallel plate capacitor which would be equivalent to the specimen under consideration.

1. MEASUREMENT OF THICKNESS

The majority of the measurements of thickness were made with the dial gage which was used by the authors in previous investigations.⁶ This gage, however, was found to be unsuitable because the pressure exerted by the plunger, which had a very small foot, reduced the thickness of the rubber of the softer specimens even through the thin aluminum electrodes. The error from this source was as much as 2 percent for some soft rubber specimens but was negligible in the case

⁶ See footnote 1 (p. 174) and also H. L. Curtis and A. T. McPherson, Dielectric Constant, Power Factor, and Resistivity of Rubber and Gutta-Percha, B.S.Tech. Papers, vol. 19, p. 669, 1925.

of hard rubber. A correction for each composition was determined and was applied to the measurements which had been made with the dial gage.

Accurate measurements of thickness were made by means of a gage which operates with a relatively slight contact pressure. This gage is a modification of one devised by Dye and described by Rolt.⁷ As is shown diagramatically in figure 2, it consists of a steel bar supported



FIG. 2.—Bar gage.

The bar rotates on the spherical surfaces at eachfend of the bar. The scale is at right angles to the axis of the bar and the tilting of the bar moves the spot of light along the scale. The micrometer head is provided so that the approximate thickness of the specimen can be set on it and only the variations in thickness need be measured by the bar. The optical lever system is so adjusted that the gage reads in microns.

present investigation. This was established by comparing the volume of rubber in a specimen as found by dimensional measurements with the volume indicated by the method of hydrostatic weighings. The two methods gave the same volume, within experimental error, whereas if there had been any appreciable compression the volume computed from the dimensional measurements would have been the smaller.

on three hemispherical surfaces, the ones at the ends resting hardened steel on supports which are lapped optically flat, and the one at the center resting on the end of a micrometer head. The hemispherical surface at the center is 1 mm off the line of centers of the other two; thus any vertical displacement of this spherical surface tilts the bar. The optical lever system from the top of the bar is so adjusted that the vertical displacement can be read directly in microns. The micrometer head is provided so that the approximate thickness of the specimen can be set on it and the optical lever system need only take care of the variations in thickness of the specimen. The bar is so balanced that the pressure of the specimen is small and the compression is negligible for purposes of the

⁷ F. H. Rolt, Gages and Fine Measurements, vol. 1, p. 339. The Macmillan Co., New York, N.Y., 1929.

The thickness of each specimen was measured at 19 points, which were so distributed as to represent equal areas. A pattern was employed as a convenient means for locating the points. To test the reproducibility of the average so obtained, several specimens were measured repeatedly with the pattern shifted each time to a new position. The largest variation found in the average thickness was about 1 percent.

The thickness of the rubber in a specimen was found by subtracting the average thickness of the aluminum electrodes from the over-all thickness of the specimen. This was done mechanically by offsetting the zero of the measuring instrument by an amount equal to the thickness of the aluminum plates of the particular specimen under observation.

The thickness of the aluminum plates was ascertained either by the use of the optical gage desecribed above or by weighing them, measuring their diameters and computing the thickness from these data and the known specific gravity of the aluminum. Measurements on aluminum plates at different points indicated that they were sufficiently uniform to justify the use of the simple average thickness.

2. MEASUREMENT OF AREA

The area of the specimen used in the computation of the dielectric constant and conductivity was obtained by correcting the area of the smaller electrode for the fringe or edge effect. This correction was included in the equations used to compute these electrical properties and is discussed in a subsequent section of this paper.

The area of the smaller electrode was computed from the average diameter, as determined by measurements with a steel scale in different directions. The diameters of a given electrode seldom varied by more than 0.5 mm, and the accuracy with which the area was determined was about 0.5 percent.

3. CHANGE IN DIMENSIONS WITH TEMPERATURE

The change in area of the electrical test specimen due to thermal expansion was small and therefore no correction was applied. Taking the coefficient of expansion of aluminum as 23×10^{-6} per °C., the change in area would be about 1 percent for a temperature change of 210° C, which is the largest difference between the temperature of dimensional measurement and the temperature of electrical measurement reported in this investigation. In the majority of measurements the change was considerably less than 1 percent.

The change in thickness with temperature was, however, of significant magnitude, not only because the coefficient of expansion of rubber is much greater than that of aluminum, but also because the rubber held in a thin sheet between the aluminum plates is free to move in only one direction, so that any contraction or expansion is manifest almost wholly as a change in thickness, the lateral dimensions changing only as much as the aluminum.

To investigate experimentally the change in thickness with temperature, the thickness gage described above was mounted in an electric oven and measurements were made on representative samples at several temperatures from 25° to 150° C. The results were found to be in accord with those computed or extrapolated from the volume coefficients of expansion reported by Kimura and Namikawa,⁸so these latter coefficients were used as a basis for correcting all determinations for change in thickness with temperature. These corrections amounted in extreme cases to as much as 13 percent.

IV. MOUNTING OF SPECIMENS FOR ELECTRICAL MEASUREMENT

The specimens were assembled for the electrical measurements in a measuring chamber which was provided with a means for connecting to the specimens. Two different measuring chambers were employed, one being used solely at 25° C. and the other at temperatures below or above 25° C. Several different methods were used for securing and controlling the temperature, but the same method of assembling the specimens and making electrical connections was employed throughout the work.

1. ELECTRICAL CONNECTIONS TO TEST SPECIMENS

The test specimens were assembled by pairs into test units, two specimens of the same composition being placed with their smaller electrodes together. Electrical connection was made to the smaller electrodes by placing between them an aluminum disk which was provided with a slender projection that extended from between the specimens. Connection was made to the larger electrodes by pressing against them aluminum disks having projections which extended beyond the edges of the specimens. When the larger electrodes were maintained at earth potential and the smaller, inner electrodes at an elevated potential, the test unit constituted a shielded condenser. In practice as many as 10 of these test units were placed one on top of another in the constant temperature chamber at one time. The smaller electrodes were connected to well-insulated, individual measuring leads. The larger electrodes were connected to a common lead. In Figure 3 a stack of test units assembled for electrical measurements is shown outside the measuring chamber.

In order to insure good electrical contact between the electrodes and the aluminum disks that were used to establish connection to the leads, brass filings were sprinkled on the surfaces and a weight of about 7 kg was placed on the assembly of specimens. Measurements indicated that this reduced the contact resistance to less than 0.005 ohm. Contact resistances at other connections were reduced by sandpapering or scraping the metal surfaces. The weight placed on the top of the specimens was assumed to have no appreciable effect on the thickness of the specimens, since application of the weight did not change the capacitance as measured with $1,000 \sim$.

2. CHAMBER FOR MEASUREMENTS AT 25° C

The chamber in which measurements were made at 25° C. was a large earthenware jar mounted in a thermostatically controlled oil bath. The test units were placed on a hard rubber disk which was suspended from a hard rubber cover, the cover being seated on a ledge inside the jar. Electrical connections to the specimens were established by means of brass rods passing through the cover. These were insulated by paraffin bushings. The humidity was kept low in the

See footnote 4.

chamber by means of a large tray of anhydrous, granular calcium chloride at the bottom. Circulation of the air in the chamber by means of a fan insured uniformly low humidity as well as constant tem-

perature throughout. A further description of this constant temperature chamber, together with an illustration showing the mounting of the specimens in it, and the method of obtaining the lead capacitance, h as been given in a previous publication.⁹

The chamber in which measurements were made over a wide range of temperature was a Pyrex glass jar about 30 cm in diameter mounted in a constant temper-The ature bath. test units were mounted on an aluminum disk suspended from a bakelite cover which rested on the top of the jar. The cover with some test units mounted on the suspended aluminum disk is shown in figure 3. As is shown, a portion of the center of the cover was cut out and a smaller Pyrex jar was sus-



FIGURE 3.—Specimens mounted for electrical measurement at temperatures from -75° to 235° C.

pended in this opening so that the bottom of this jar was about 5 cm above the specimens and the top extended above the cover. This

The specimens are mounted on the aluminum disk suspended from the bakelite cover of the constant temperature chamber (not shown). Measurements are made after the temperature and humidity become constant. The specimens are connected to the bridge by the vertical brass rods. For temperatures below 25° C. part of the cooling liquid is put in the jar mounted in the cover; this produces circulation of the gas in the chamber. When temperatures above 25° C. are used, this jar is filled with a thermal insulating material.

⁹ See reference in footnote 1.

provided a means for stirring the air in the chamber at temperatures below room temperature since a part of the cooling liquid used in the bath could be put in the smaller jar thereby producing convection currents. Pyrex glass tubes were inserted in the cover for the lead rods to pass through in order to furnish better electrical insulation than was afforded by the bakelite. A ring of bakelite attached to the smaller jar about 7 cm below the cover afforded additional support to the tubes which insulated the leads, and also provided some thermal insulation for the chamber. No fan was provided for stirring the air in the chamber since the heating and the cooling arrangements were such that convection could be relied upon to maintain a reasonably uniform temperature. The temperature at the center of the stack of specimens was determined by means of a thermocouple. This thermocouple was imbedded in an aluminum plate which was placed between the test units. Electrical measurements were made when the temperature at the center of the assembly was within 0.5° C. of the temperature of the bath.

Low relative humidity in the measuring chamber was insured by the use of anhydrous calcium chloride as a drying agent, and by the passage of a slow stream of carbon dioxide from a cylinder into the chamber. The carbon dioxide escaped through the annular spaces between the Pyrex glass insulating tubes and the brass rods which served as leads and prevented the entrance of air or moisture into the measuring chamber.

Small electric heaters were placed around the Pyrex glass insulating tubes as an additional precaution against the condensation of frost on the leads when measurements were being made at low temperatures.

4. MEANS FOR MAINTAINING CONSTANT TEMPERATURE

The chamber which was operated at 25° C. was mounted in a wellstirred oil bath that was provided with thermostatic control. The thermostat operated with a double contact so that the bath was either heated or cooled according to the temperature of the room. The heating was obtained by means of an electrical heating unit, and the cooling by the use of a small pump which circulated cold water through a coil in the bath.

The chamber which was used over the wide range of temperatures could be mounted in either a liquid bath or an air bath. Liquid baths were used below 25° C., water cooled by ice being employed in the range 25° to 0° C., and alcohol cooled by carbon dioxide snow in the range 0° to -75° C. The liquid bath had a volume of about 10 gallons and was well insulated so that no difficulty was encountered in regulating the temperature to within 1° C. of that desired by adding ice or carbon dioxide snow as it was needed. A liquid bath was also maintained in the smaller cylinder of the measuring chamber and was kept at the same temperature as the main bath. With a cooling surface immediately above the specimens, it was possible to maintain them at a uniform temperature without a forced circulation of air in the chamber.

The air bath which was employed for heating the measuring chamber above 25° C. was provided with an electric heater, a circulating fan, and a bimetallic thermostat. It was well insulated and the temperature of the air in it could be maintained to within 1° C., of the desired temperature. When the measuring chamber was placed in this air bath, the smaller jar was simply filled with a thermal insulating material. In this case, the air in the chamber was sufficiently stirred by convection to maintain the specimens at a uniform temperature, since the heat was applied to the bottom and sides.

V. ELECTRICAL MEASUREMENTS

Two types of electrical measurements were made—one type was the primary measurements on specimens having ordinary electrodes; the other type was the relative measurements on specimens having perforated electrodes. The primary measurements were made at 25° C. and 1,000 ~ for the purpose of computing the dielectric constant and conductivity from the observed dimensions of the specimens and the measured capacitance and conductance, respectively. The dielectric constant thus obtained was used with the capacitance of a test unit of identical composition, but having perforated electrodes, to compute a shape factor for this test unit. This shape factor and the thermal expansion of the specimens were used to determine the dielectric constant and conductivity from the capacitance and conductance, respectively, as measured between temperatures of -75° and 235° C. Although for making measurements over this extended temperature range, two different measuring chambers were used and different means were employed to secure the desired temperatures, the same method of assembling the specimens and making the electrical connections to them was employed throughout the investigation.

The electrical measurements which were made on the specimens were the capacitance, power factor, and conductance. The capacitance and the power factor were measured at five frequencies and the conductance at two elapsed times after electrification. The methods used in part of the measurements have been described elsewhere and will be only briefly sketched here. The dielectric constant and conductivity were computed from these measurements and the dimensions of the specimens.

1. CAPACITANCE AND POWER FACTOR

The capitance and power factor measurements at 60, 1,000, and $3,000 \sim$ were made with the modified Rosa series inductance bridge, a diagram of which is shown in figure 4. A telephone receiver was used as the detector for 1,000 and 3,000 \sim , and a vibration galvanometer for 60 \sim . The manipulation of this bridge is described in the legend.

A modification of the resonance method described by Dellinger and Preston ¹⁰ was used to measure the capacitance and power factor at 100,000 and 300,000 \sim . A diagram of this set-up is shown in figure 5. The method of making the measurements is described in the legend.

The source of 60 \sim frequency was the local power circuit. The frequency of the power supply was so well controlled that it was sufficiently accurate for these measurements.

¹⁰ J. H. Dellinger and J. L. Preston, Methods of Measurement of Properties of Electrical Insulating Materials, B.S. Sci. Papers, vol. 19 (S471), p. 39; 1923. The method here referred to is described on p. 43.

The frequencies of 1,000 and 3,000 \sim were obtained from a motor generator driven from a direct-current supply. At full speed this machine supplied a current at $3,000 \sim$. It was run at one third full speed to obtain a current at $1,000 \sim$. The use of a remote-control system and a frequency meter made it possible to control the 1,000 \sim



FIGURE 4.—Modified Rosa bridge.

To make a measurement of capacitance and power factor several cycles of adjustment must be made in which the detector is connected first to the bridge and then to the Wagner earthing system. First connect the specimen in the bridge not only by its regular lead X but also by the dummy lead Y. The cycle con-sists in first connecting K to H and balancing by adjusting C and L, then connecting K to J and balancing by adjusting P' and L'. This cycle is repeated until the throwing of K from J to X does not affect the balance of the bridge or of the Wagner earthing system. Let the reading of the left-hand inductor be L. Connect a calibrated capacitor S into the bridge in place of the specimen and make another cycle of adjust-ments varying S instead of C. Let the readings of the calibrated capacitor and left-hand inductor be S₁ and L₂. Connect the specimen into the bridge as before with the exception of the dummy lead Y which is disconnected not only at Z, but also at the specimen. Adjust the bridge in the same way as when both leads were connected. Let the reading of the left-hand inductor be L₂. Again substitute the calibrated capacitor for the specimen and make the cycle of adjustments. Let the reading of the calibrated capacitor for the specimen and make the cycle of adjustments. The the reading of the calibrated capacitor be S₂. The reading of the inductor will be L₂ as before. The capacitance of the dummy lead and hence of the measuring lead is S₁-S₂. The capacitance of the specimen is S₂-(S₁-S₂)=2 S₂-S₁. If L₁=L₄ as is usually the case, and R=P, the power factor F is given with sufficient approximation by the equation $-u e(L_2-L_1)$

$$F = \frac{\omega(L_2 - L_1)}{R}$$

where ω is 2π times the frequency of the alternating current used in the measurement, and R and P are the total resistances of the arms A H and B H. If the lead capacitance is not to be determined, the first part of this procedure is omitted.

frequency with high accuracy. No frequency meter was available for use with the 3,000 \sim frequency, but the generator was steady at full speed and after being set with a tachometer and stop watch gave a frequency which was sufficiently accurate for purposes of the present investigation.

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The radio frequencies of 100,000 and 300,000 \sim were obtained from a radio-frequency generator using a 250-watt power tube in a tuned plate circuit. This generator was adjusted for the desired frequency and the output was checked at frequent intervals by means of a calibrated frequency meter.



FIGURE 5.—Set-up for radio-frequency measurements.

The set-up consists of an inductance coil L, a noninductive decade resistance R, and a vacuum thermo-couple T connected in series to the midpoints K of a double pole, double throw switch by means of which the calibrated variable air capacitor C, or the test unit C_2 and parallel variable air capacitor C_4 may be connected into the circuit. The measurement of capacitance and power factor consists in adjusting the circuit containing first the specimen and then the calibrated air capacitor to resonance with the source. The test unit is first introduced into the circuit by connecting K to H, R being set at zero resistance, and resonance is then obtained by adjusting C_4 . The coil is so chosen that C_6 will be small. The coupling between the source and the resonance circuit is so adjusted by moving the source that a large deflection of the galvanometer is obtained. The noninductive resistance R is then increased until the angular deflec-tion of the galvanometer is decreased to one fourth. Let this resistance be R_1 . The capacitor C_4 is intro-duced into the circuit by connecting K to J, R is set at zero resistance, and C is adjusted until resonance is obtained. The coupling is again adjusted to give a large angular deflection of the galvanometer, and then this deflection is decreased to one fourth by increasing R. Call this resistance R_2 . The capacitance of the specimen C_4 is then C_2 , where C_4 is the capacitor and the leads. The power factor is given with sufficient accuracy by the equation $R_{10} = R_{10} C_1^2 (R_1 - R_2)$

$$F = \frac{\omega C_s(R_1 - R_2)}{C_x}$$

2. DIELECTRIC CONSTANT

The dielectric constant K of the medium of a capacitor is given by the equation

$$K = \frac{C}{C_o} = AC \tag{1}$$

where C is the measured capacitance, C_o is the capacitance which the capacitor would have if vacuum were its dielectric, and $A = \frac{1}{C_0}$ is the shape factor of the capacitor. The specimens having ordinary electrodes were so designed and prepared that C_o and hence A could be computed from the measurements of the dimensions. For a test unit the value of C_o in micromicrofarads is given to a sufficient approximation by the equation

$$C_o = \frac{A_1 d_2 + A_2 d_1 + 8r d^2 [ln \ 2 + (1+x) ln \ (1+x) - x \ ln \ x]}{11.31 \ d_1 d_2}$$
(2)

where A_1 and A_2 are the areas and d_1 and d_2 are the thicknesses of the individual specimens making up the test unit, d is the average of d_1 and d_2 , r is the average of r_1 and r_2 , which are the radii of the



FIGURE 6.—Set-up for measuring the "1-minute" conductance.

To make a measurement, the specimen is shorted, the shunt set at the lowest ratio, the reversing switch closed, and the deflection of the galvanometer noted when K_2 is closed. The deflection of the galvanometer is also noted when the reversing switch is thrown in the opposite direction. Call the average deflection d_m , the shunt ratio X_m , and the potential applied V_m . Next open the shorting key and obtain an average deflection as before, changing the shunt ratio until a readable deflection is obtained and using a higher potential if desired. The specimen must be shorted for two minutes between readings. Call the deflection at the end of one minute after the potential is applied d_x , the potential V_x , and the shunt ratio X_z . The conductance G is then given by the equation.

$$G = \frac{d_x}{d_m} \cdot \frac{X_m}{X_x} \cdot \frac{V_m}{V_x} \cdot \frac{1}{10^6} \,\mathrm{mho}$$

small electrodes of the two specimens, $x ext{ is } \frac{t}{4d}$ where t is the total thickness of the two middle electrodes, and ln signifies the natural logarithm.¹¹ The dimensions are to be expressed in centimeters. This

equation takes into account the edge effect, which is small. As the computation of C_o and hence of A from measured dimensions is impracticable for test units having perforated electrodes, these test units were not used for primary determinations of the dielectric constant, but values of the dielectric constant were derived indirectly from measurements with them, a shape factor being deter-

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¹¹ A. Gunthershulse, Measurement of the Dielectric Constant and Dipole Moment, Handbuch der Physik., vol. 16, chap. 20.

mined for each test unit at 25° C. This shape factor was obtained from measurements of the capacitance of two test units of the same composition, one having ordinary electrodes and the other perforated electrodes. The dielectric constant of the rubber was calculated from the measurements on the test unit having ordinary electrodes. This value was used in conjunction with the capacitance of the test unit having perforated electrodes to obtain its shape factor. The test units having perforated electrodes were used for measurements over a wide range of temperature and frequency, and the shape factors, corrected for the thermal expansion of the rubber, were used for the calculation of the dielectric constant

3. CONDUCTANCE

The conductance G of a test unit was determined by measuring, with a suitable apparatus, the current which flowed through the specimen under an applied electromotive force of known value. Since the value obtained for the conductance depended on the elapsed time between the application of the electromotive force and the measurement of the current, two different time intervals were used in this investigation—1 minute, which is the usual time in measurements of this sort, and a much shorter interval (about 0.002 second). The set-up for measuring the conductance at the end of 1 minute, which may be designated as the "1-minute" conductance, was the same as that described previously,¹² a self-explanatory diagram of which is shown in figure 6.

The "short-time" conductance; that is, the conductance obtained at the end of about 0.002 second, was measured with the aid of a Helmholtz pendulum, as shown diagrammatically in figure 7.

Although the time interval between the actions of S_2 and S_3 was usually 0.001 second, in some instances the conductance was so high that the galvanometer would deflect off the scale when this time interval was used, and in other instances the conductance was so low that the galvanometer deflection was too small to read. The deflection was made readable by shortening or lengthening the time interval between the actions of S_2 and S_3 , although this affected the results, since the current was changing with time. For the highest conductance, this time interval was reduced to 0.0005 second, and the values obtained were higher than would have been obtained if the usual time interval had been employed. For the lowest conductances, the time interval was increased to 0.005 second which made the results lower than they would have been had the usual time been employed. However, the results are usable since high accuracy cannot be obtained in such measurements. All the "short-time" conductivity measurements are tabulated as though they were measured at 0.002 second after the potential was applied.

4. CONDUCTIVITY

The conductivity γ of the material of a test unit was obtained by means of the equation

$$\gamma = BG \tag{3}$$

¹² H. L. Curtis and A. T. McPherson, Dielectric Constant, Power Factor, and Resistivity of Rubber and Gutta-Percha, B.S.Tech. Papers, vol. 19, p. 669. The set-up here referred to is described on p. 678.

where G is the conductance of the test unit and B the shape factor for



FIGURE 7.—Set-up for measuring "short-time" conductance.

The "short time" conductance is measured with the aid of a Helmholtz pendulum. The specimen is connected into the circuit and the reversing switch closed. When the pendulum is released, it first strikes the prop from under S_1 , allowing this switch to close. The pendulum then strikes S_2 0.0015 second later and opens it, which allows the current to flow through the galvanometer. The pendulum opens S_2 a predetermined time later (usually 0.001 second). The current which flows through the galvanometer between the openings of S_2 and S_3 gives a ballistic throw to the galvanometer. The conductance G is given by

$$G = \frac{Kd}{ET}$$

where K is the galvanometer constant, d is the ballistic deflection, E is the potential applied, and T is the time between the actions of S_2 and S_3 .

while the reproducibility will be touched on briefly in subsequent divisions of the paper.

the shape factor for conductivity. The shape factor for conductivity is connected with the shape factor for dielectric constant by the equation

$$B = \frac{A}{11.31} \qquad (4)$$

where A is the shape factor for dielectric constant when the capacitance is measured in micromicrofarads. The shape factor for conductivity for each test unit at each temperature was obtained from the corresponding shape factor for the dielectric constant.

5. ACCURACY OF DETERMINATIONS

The accuracy which can be assigned to the electrical constants of rubber of a definite composition depends not only on the accuracy with which the electrical and mechanimeasurements cal can be made on a particular specimen, but also on the accuracy with which specimens of a given composition can be reproduced at different times and from different lots of crude The accurubber. racy of the measurements will be discussed in this section,

Scott, McPherson] Curtis

(a) EFFECT OF ERRORS IN DIMENSIONAL MEASUREMENTS

As already stated, the probable errors involved in the dimensional measurements are of the order of 1 or 2 percent. These impose the chief limitation on the accuracy of the dielectric constant measurement. Conductance measurements, on the other hand, are subject to errors, common to this type of measurement, of such magnitude that the accuracy of dimensional measurements is of relatively small importance. Power factor determinations are, of course, independent of the dimensions of the specimens.

(b) POSSIBLE SOURCES OF ERROR IN ELECTRICAL CONNECTIONS AND MEASUREMENTS

In the measurement of capacitance care was taken to make the bridge errors as small as possible. The substitution method was used to eliminate from the measurements the capacitance between pieces of apparatus and between apparatus and ground. The errors in the bridge itself were considered to be negligible in comparison with other errors in the determination of dielectric constant.

It was necessary to make a correction for the capacitance of the leads which extended from the test unit to the outside of the constant temperature bath. The lead capacitance was not determined each time, but the average of a large number of determinations was taken as the capacitance of each lead. This average value did not differ from the measured value in any case by more than $5\mu\mu$ f which corresponds to a maximum error of 0.4 percent in the determination of the capacitance of a test unit. The method of determining the lead capacitance is described in the legend under figure 4.

A possible source of error in the determination of the capacitance of a test unit resided in the fact that the individual members of the test unit did not necessarily have their small electrodes precisely in juxtaposition. But from the procedure used in assembling a test unit, it is unlikely that the centers of the small electrodes were out of line by as much as 1 mm. An eccentricity of this amount would give a maximum error of 0.3 percent. As a matter of fact when several test units were repeatedly taken apart, reassembled, and remeasured, the maximum variation in the capacitance which was observed was never greater than 0.2 percent.

Contact resistances in the circuit were a potential source of error in the capacitance and power factor measurements. At the lower frequencies the errors due to any such resistances as may have been present were negligible, but at radio frequencies it was necessary to take special precautions to see that these resistances were so small that they did not cause any appreciable error. For these frequencies, all the metal parts were scraped before connections were made, and metal filings were put between each electrode and its connecting plate.

No guard rings were used in making any of the measurements, but it was unlikely that any significant errors arose from their omission because the specimens were kept in an atmosphere of low relative humidity while being measured.

(c) INDEPENDENT DETERMINATION OF DIELECTRIC CONSTANT

A check was made on the determination of the dielectric constant of a few compounds at 25° C. by using an entirely different method for ascertaining it. The method consisted in measuring the dielectric constant by means of a parallel plate capacitor with a guard ring. Care was taken to have the guard ring at all times as near the potential of the guarded plate as possible. Measurements were made on a sheet of rubber prepared from the same batch as a pair of specimens which constituted a test unit. The sheet of rubber was placed between the parallel plates of the capacitor and an air gap was left between the sheet and the upper plate. The bridge containing this capacitor was then balanced. The rubber sheet was removed from between the plates and the bridge was again brought to balance by changing the distance between the plates. The change in distance between the plates was determined by making readings on an engraved scale with a telescope and micrometer eyepiece. The dielectric constant was then obtained from the equation

$$K = \frac{t}{t - (d' - d'')} \tag{5}$$

where t is the thickness of the sheet of rubber and (d'-d'') is the change in distance between the plates. Values of the dielectric constant obtained by this method agreed to within 2 percent with those obtained by the other method.

(d) EFFECT OF THE CONDITION OF ELECTRODE SURFACES

• Some experiments were made to determine whether or not the condition of the electrode surfaces had any appreciable effect on the measured values of the capacitance and power factor. Such an effect has been reported for aluminum electrodes.¹³ Several specimens were made up with aluminum electrodes which had been anodized (oxy-dized) like those used in that work. No appreciable difference was found between the values of the dielectric constant and power factor obtained with such electrodes and those obtained with ordinary electrodes.

VI. VALUES OF ELECTRICAL PROPERTIES

The measurements on rubber-sulphur compounds which are here reported show the effect of composition, temperature, and frequency on the electrical properties. From an experimental standpoint the results fall into two general groups. In the first group are the measurements at 25° C. on specimens having ordinary electrodes. In the second group are the determinations which were made over a range of temperatures and frequencies on specimens having perforated electrodes. The second group of measurements are dependent on the first since a shape factor was obtained from capacitance determinations on specimens of identical composition having the two types of electrodes.

There is a further difference between the two groups of measurements in that the values reported for the electrical properties in the first group represent, in most cases, averages of several determinations, while those reported in the second group represent single determinations.

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¹³ H. J. Curtis, The Dielectric Constant of Liquid Sulphur, J. Chem. Phys., vol. 1, p. 160; February 1933. Curtis obtained temperature curves of dielectric constant and power factor similar to those which were obtained for rubber, but the values were not the same on different runs. When the aluminum electrodes were anodized, quite different curves were obtained which could be readily reproduced.

The primary measurements, which constitute the first group of results, were made at a single temperature, 25° C., and at a single frequency, $1,000 \sim$, and consequently these data can be summed up in one table or figure. The comparative measurements, however, were made at a number of temperatures and at several frequencies. To express these results concisely, tables and figures are given showing the relation of the electrical properties to two simultaneous variables—composition and temperature. The effect of the third variable, frequency, is shown by giving separate tables and figures for each frequency.

1. ELECTRICAL PROPERTIES AT 25° C

Electrical measurements at 25° C. were made on compounds of purified rubber and sulphur covering the range from unvulcanized rubber to completely vulcanized hard rubber. Twenty-two different



FIGURE 8.—Relation of the dielectric constant, power factor, and "1-minute" conductivity of rubber-sulphur compounds to the percent of sulphur.

The power factor is given in units of 1×10^{-3} and the "1-minute" conductivity in units of 1×10^{-17} mho/cm.

compositions were employed, these being spaced at intervals of 0.5 to 2.0 percent sulphur so as to precisely define the changes in electrical properties with composition. From 1 to 16 different test units or pairs of specimens of each composition were prepared and measured, the average number being 7. Dielectric constant and power factor measurements were made at $1,000 \sim$ and the conductance measurements were made 1 minute after the application of potential to the specimen.

The results are summarized in table 1 and are shown graphically in figure 8, in which the three electrical properties are plotted as functions of the composition expressed as percent of sulphur. Each of the properties has characteristic features, but it should be noted that each has a maximum at some composition.

(a) DIELECTRIC CONSTANT

The dielectric constant of unvulcanized rubber was found to be 2.37. With increasing sulphur content the dielectric constant increased almost linearly to a maximum of about 3.75 in the neighborhood of 11.5 percent sulphur. With larger sulphur content, it decreased rapidly and reached a minimum of 2.70 at about 22 percent sulphur. Above 22 percent sulphur, there was a slow increase in dielectric constant and a value of 2.82 was observed for rubber containing 32 percent sulphur.

The average deviation of individual dielectric constant determinations from the mean of those of the same composition, made under identical conditions, was less than 1 percent in the majority of cases.

 TABLE 1.—Dielectric constant, power factor, and conductivity of compounds of purified rubber and sulphur at 25° C.

	Dielectri cycl	c constar es per se	nt at 1,000 cond	Power fac I	ctor at 1, Der secon	000 cycles d	"1-minute" conductivity				
Sulphur (in percent)	Number of deter- mina- tions	Aver- age value	Average devia- tion from mean	Number of deter- mina- tions	Aver- age value	Average devia- tion from mean	Number of deter- mina- tions	Aver- age value	A verage devia- tion from mean		
0 2 4 68	17 2 6 4 1 3	2, 37 2, 68 2, 89 3, 22 3, 49	Percent 0.7 .9 .8 .8 1.2	19 3 7 5 7	$\begin{array}{c} \times 10^{-3} \\ 1.6 \\ 1.8 \\ 2.1 \\ 4.6 \\ 13.5 \end{array}$	Percent 25 11 10 6 10	16 3 6 5 7	×10-17 2.3 1.3 1.1 .9 .9	Percent 266 69 64 49 33		
10 10.5 12 13.5 14	$\begin{array}{c} 4\\6\\4\\4\\3\end{array}$	3. 69 3. 70 3. 74 3. 47 3. 39	.7 .5 .4 .6 1.7	5 7 5 5 4	34. 1 42. 5 73. 9 93. 8 90. 0	5 4 7 3 3	5 6 5 5 4	.6 .6 .5 .8 1.1	43 28 36 15 29		
16 18 19 20	4 3 6 2	$\begin{array}{c} 2.90\\ 2.76\\ 2.74\\ 2.71\end{array}$	$1.1 \\ .4 \\ .6 \\ 1.3$	4 4 8 3	42.7 10.3 8.0 3.2	7 16 30 18	5 4 9 3	$ \begin{array}{c} 6.0 \\ 38. \\ 29. \\ 8.1 \end{array} $	30 14 7 32		
22 23 24 26	$\begin{array}{c} 2\\ 1\\ 1\\ 6\end{array}$	2.70 2.77 2.74 2.75	.7	2 2 1 7	$\begin{array}{c} 4.3\\ 2.6\\ 3.3\\ 4.7\end{array}$	0 4 28	2 2 1 7	$1.0 \\ 1.2 \\ 1.0 \\ 1.0$	10 8 20		
28 29 30 32	1 1 8	2.80 2.83 2.82	.8	1 1 1 10	4.0 4.2 4.8 5.1	21	1 1 1 9	1.0 .8 1.3 1.5			

¹2 other values were discarded because 1 was unreasonably high and the other unreasonably low.

(b) POWER FACTOR

The power factor of unvulcanized rubber was 1.6×10^{-3} . There was a slow decrease in power factor up to about 4 percent sulphur, and above this composition the increase became relatively rapid until a maximum of 93.8×10^{-3} was reached at about 13.5 percent sulphur. With further increase in sulphur content, the power factor decreased rapidly and reached a minimum value of 2.6×10^{-3} at about 23 percent sulphur. Throughout the range of hard-rubber compounds the power factor increased slowly and attained a value of 5.1×10^{-3} for 32 percent sulphur.

The average deviation of power factor determinations from the mean of each composition amounts to as much as 30 percent in some instances and is 11 percent for the group as a whole. Since probable errors in measurement amount to less than 5 percent for the lower power factors and to less than 1 percent for the higher power factors, this deviation from the mean may be attributed to the possible effect of incidental impurities which, in small proportion, may materially alter the power factor of a dielectric.

(c) CONDUCTIVITY

The curve relating "1-minute" conductivity to composition is similar to the curve for the power factor in that both show similar sharp and high maxima. The conductivity of unvulcanized rubber is 2.3×10^{-17} mho/cm. This decreases with increasing sulphur content and reaches a minimum value of 0.5×10^{-17} at about 12 percent sulphur. Above this minimum a sharp increase takes place and at 18 percent sulphur there is a maximum conductivity of 38×10^{-17} . Beyond the maximum there is an equally sharp decrease so that at 22 percent sulphur the conductivity is only 1×10^{-17} . Above this point, throughout the hard-rubber range, the conductivity increases very slightly.

The variation in conductivity between individual samples of the same composition is considerable, as would be expected from the nature of the results that are obtained in conductivity measurements on dielectrics in general. This variation is as much as 70 percent in one instance, and is, on the average, 31 percent. A negligible part of this variation is doubtless attendant on circumstances having to do with the dimensional or electrical determinations, but most of it is probably due to slight accidental variations in the composition of the rubber.

2. MEASUREMENTS AT -75° TO 235° C

The measurements which were carried out to determine the effect of temperature and frequency on the electrical properties covered the range from -75° to 235° C. at intervals of about 10° and were made at five frequencies, namely, 60, 1,000, 3,000, 100,000, and $300,000 \sim$. The conductivity was measured at two time intervals after the application of potential, namely, 0.002 second and 1 minute. The compositions on which the measurements were made covered the range of possible rubber-sulphur compounds at slightly wider intervals than the previous series of measurements at 25° C., there being 14 instead of 22 different percentages of sulphur. As has been indicated previously, these specimens were all made with perforated aluminum sheets as electrodes, and, instead of measuring the thickness and area, a shape factor was determined and was used in computing the dielectric constant and conductivity values.

For specimens containing not over 12 percent of sulphur, the test units used above 25° C. were not the same as those used below 25° C., but for higher percentages of sulphur each test unit was used throughout the entire range of temperature.

Only a single pair of specimens constituting a single test unit of each composition was used, but the consistency of the change in electrical properties with temperature and frequency affords a reasonable indication that there were no large experimental errors.

Measurements are here reported for temperatures between -75° C. and the highest temperature at which accurate measurements could be made. The lower limit was fixed by the lowest temperature which could be readily maintained with carbon dioxide snow. The upper limit was different for different samples, depending either on the decomposition of the sample during the time the observations were in progress, on its change in shape because the sample began to flow, or on its increase in conductivity. The samples were generally meas-

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ured at temperatures higher than herein reported, the upper limit for reporting results being fixed after the measurements were completed.

In determining the temperature at which a sample had deteriorated to a point where the results should not be reported, the decision was





Each line connects points having equal values of dielectric constant. A is the temperature limit set by the flow of the specimen. B is the temperature limit set by the decomposition of the specimens. C is the temperature limit set by the high conductance.



FIGURE 10.—Isograms of the dielectric constant of rubber measured at 1,000 \sim . For meaning of symbols see figure 9.

made that, when a sample had lost an amount of sulphur equal to 0.3 percent of the original weight of the sample, it was no longer representative. Using the results of Cummings,¹⁴ the temperatures were

¹⁴ A. D. Cummings, Effect of Temperature on the Evolution of Hydrogen Sulphide from Vulcanized Rubber, B.S.Jour. Research, vol. 9 (RP464), p. 163; 1932. determined at which the samples of each composition would lose this amount of sulphur under the conditions at which the electrical measurements were made; namely, increasing the temperature in steps of 10° or 20° and maintaining the specimens at each tempera-



FIGURE 11.—Isograms of the dielectric constant of rubber measured at 100,000 \sim . For meaning of symbols see figure 9.

ture for 8 hours. A change in shape caused by the flowing of the material of the sample affected only the one having 0 percent



FIGURE 12.—Isograms of dielectric constant of rubber measured at $300,000 \sim$. For meaning of symbols see figure 9.

sulphur. It is known that such material flows readily at temperatures above 105° C. A temperature limit was imposed by the increase in the conductivity of certain samples because neither it nor its accompanying power factor could be measured without a redesign of the

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apparatus, which did not seem to be necessary. The figures show which one of these causes decided the upper limit.

The effect of the temperature on the relation between electrical properties and the composition is shown by the use of contour charts in which temperature and composition are abscissa and ordinate, respectively, while the electrical properties are represented by isograms which may be visualized as the contour lines of a solid model. The subsequent discussion will refer to the charts as representations of such models. While the model for each property has distinctive features, they have one feature in common, namely, a prominent diagonal ridge in which each property goes through a maximum.

(a) DIELECTRIC CONSTANT

The variation of the dielectric constant with composition and temperature is shown in five tables (2 to 6), each table giving the results at a different frequency. The corresponding isograms are shown in figures 9 to 12. The results at 3,000 \sim are not shown graphically because of close similarity to results at 1,000 \sim .

TABLE	2.—Dielectric	constant	of	compounds	of	purified	rubber	and	sulphur	at
			60	cycles per se	con	d				

Temperature (in ° C.)					P	ercent	of com	bined	sulphu	ır				
(in ° C.)	0	2	4	6	8	10	12	14	18	20	23	26	29	32
-75 -65	2, 45 2, 43	2.45 2.44	2.53 2.52	2.58 2.57	2, 58 2, 56	2.56 2.55	2.62 2.61	2.62	2.63	2.66	2.65	2.69	2. 71	2.75
-55 -45 -35	2.43 2.45 2.50	2.45 2.53 2.73	2.52 2.56 2.80	2.57 2.58 2.68	2.57 2.57 2.57	2.56 2.58 2.55	2. 61 2. 61 2. 60	2.63	2.65	2.69	2.68	2.72	2.73	2.75
-27	2.52	2.79	3.03	3.02	2. 71	2. 59	2.62	2.01	2.01	2. 70	2.70	2. 11	2.10	2.10
-10 0	2.52 2.50 2.40 2.37	2.75 2.77 2.74 2.74	3.09 3.07 2.08	3.37 3.36 3.28	2. 50 3. 44 3. 59 3. 57	3. 11 3. 57 3. 80	2.80	2. 67 2. 79 3. 37	2.68 2.68 2.70	2.70 2.71 2.71 2.71	2.70	2.76	2. 78	2. 75
25	2.36	2.67	2.93 2.90	3. 21 3. 16	3. 51 3. 47	3. 78 3. 73	4.04	3.92 4.15	2. 10 2. 84 3. 21	2.74 2.80	2.74 2.73	2.77	2. 81	2.84
45 56	2.36	2.62	2.87	3. 13	3. 41	3.67	3.95	4.19 4.13	3.86 4.37	3.06 3.67 4.17	2.74	2.77 2.79	2.79	2.83
75	2.00		2.02	0.00	0.01		0.01	4.01	4.45	4. 50	3. 61	2. 92	2.83	2.80
85 95 105	2.35	2.58	2.79	3.00 2.92	3. 22	3. 44 3. 33	3. 68 3. 56	3.95 3.89 3.81	4. 39 4. 31 4. 22	4. 57 4. 53 4. 45	4.22 4.56 4.64	3. 22 3. 80 4. 41	2.87 2.96 3.09	2.89 2.93 3.08
115		2. 52	2. 68	2.85	3. 05	 3. 23	3. 45	3. 68	4.06	4. 28	4. 59 4. 51	4.63 4.73	3.75 4.43	3.42 4.13
135 145 168		2.48 2.45	2.63	2.79	2.98 2.91	3.15 3.06	3.35 3.24	3.59 3.48	3.94 3.82	4.16	$\frac{4.37}{4.20}$	4.63 4.56	4.63 4.66	4.58
195		2.40	2.52	2.66	2.83	2.98	3. 16							

Temperature (in ° C.)					P	ercent	of com	bined	sulphu	ır				
(in ° C.)	0	2	4	6	8	10	12	14	18	20	23	26	29	32
	$2.42 \\ 2.42$	2. 43 2. 44	$2.51 \\ 2.51$	2.56 2.56	2.55 2.56	$2.54 \\ 2.55$	2.59 2.59	2. 61	2. 62	2.64	2. 63	2.66	2. 68	2. 71
-55 -45	2.42	2.44	2.51	2.57 2.56	2.56 2.56 2.56	2.55 2.55 2.56	2.60	2.62	2.64	2.66	2.65	2.68	2.70	2.76
	2. 50	2. 55	2. 35	2. 68	2. 50	2.56	2. 61	2.02	2.00	2.00	2.01	2.11	2.10	2.10
-20 -10	2.52 2.51 2.40	2.75 2.75 2.73	2.93 3.03 3.04	$\begin{array}{c} 2.90 \\ 3.17 \\ 3.28 \end{array}$	2.69 2.96 3.32	2.60 2.73 3.06	2.62 2.66 2.83	2.63 2.64 2.69	2.66 2.66 2.67	2.69 2.70 2.71	2.68	2.73	2.74	2.77
14	2.37	2.69	2.97	3. 27	3. 48	3. 52	3. 30	2.93	2.68	2.70	2.70			
25 35 45	2.37 2.35 2.34	2.66 2.64 2.61	2.92 2.88 2.85	3. 20 3. 15 3. 11	3. 48 3. 44 3. 39	3.67 3.68 3.64	3.74 3.88 3.88 3.88	3. 37 3. 77 3. 98 4. 05	2.76 2.91 3.25 3.75	2.73 2.76 2.86 3.14	2.73 2.72 2.72 2.72 2.72	2.76 2.76 2.76 2.76 2.76	2.79	2.82
35	2.33	2. 57	2.80	3. 04	3. 30	3. 53	3.79	4.04	4. 10 4. 26	3. 55	2.87	2.78	2.80	2.83
35 95	2.33	2. 57	2.78	2.99	3. 21	3. 43	3.67	3. 93 3. 86	4. 30 4. 26	4. 29 4. 38	3. 55 4. 05	3.00 3.31	2.84 2.90	2.86 2.90
l05 l15	2. 33	2. 55	2.73	2.91	3. 12	3. 32	3. 55	3. 81	4. 20	4.38 4.34	4.32 4.43	$3.69 \\ 4.15$	$3.03 \\ 3.31$	2.96 3.15
125 135		2. 51	2.67	2.85	3. 05 	3. 23 	3.44	3. 67 	4. 05	4. 27	4.45 4.40	4. 43 4. 49	3.80 4.18	3. 55 3. 97
145 156		2.47	2.62	2.79	2.98	3.15	3.35	3. 58	3.94	4.16	4.35	4.50 4.45	4. 44	
168		2.43 2.39	2.57 2.51	2.72 2.65	2.90 2.82	3.06 2.95	3.25 3.11	3. 47 3. 36	$3.81 \\ 3.74$	4.02	4, 19			

 TABLE 3.—Dielectric constant of compounds of purified rubber and sulphur at 1,000 cycles per second

 TABLE 4.—Dielectric constant of compounds of purified rubber and sulphur at 3,000 cycles per second

Temperature (in ° C.)					P	ercent	of com	bined	sulphu	ır				
(in ° C.)	0	2	4	6	8	10	12	14	18	20	23	26	29	32
	2.42 2.42	2. 43 2. 43	2.50 2.51	2.55	2.54	2.53	2, 58 2, 60	2.61	2.61	2. 63	2.61	2.65	2.67	2.70
	2. 42 2. 42 2. 47	2. 43 2. 44 2. 48	2. 51 2. 51 2. 54	2.50 2.55 2.57	2.56 2.56 2.56	2.55 2.55 2.55	2.60 2.60 2.61	2.61 2.62	2. 63	2.60	2. 64	2. 67	2. 69	2.72
-27 -20 -10	2.52 2.51 2.50	2.60 2.69 2.73	2.66 2.81 2.98	2.62 2.76 3.04	2.57 2.62 2.83	2.55 2.57 2.65	2.61 2.61 2.64	2.62	2.65	2.68	2.68	2.72	2.73	2.76
0 14	2.40 2.37	2.73 2.69	3. 02 2. 96	3. 20 3. 24	3, 11 3, 40	2, 90 3, 35	2, 74 3, 12	2.66 2.82	2.66 2.67	2.69 2.70	2.79 2.70	2 73	2.75	2.78
25 35 45	2.36 2.34 2,34	2.66 2.62 2.61	2, 92 2, 87 2, 85	3.20 3.13 3.11	3.44 3.42 3.39	3. 57 3. 63 3. 62	3.54 3.79 3.83	3.18 3.53 3.83	2.74 2.83 3.09	2.72 2.74 2.82	$\begin{array}{c} 2.72\\ 2.71\\ 2.71\\ 2.71\end{array}$	2.75 2.75 2.76	2.78 2.78	2.81
55 65	2.32	2. 57	2.80	3.04	3.30	3. 58 3. 53	3.82 3.78	3.98 3.98	3.54 3.88	3.05 3.35	2.76 2.84	2.76 2.78	2, 80	2.82
75 85 95	2.33	2, 56	2.78	2.99	3.21	3.42	3.67	3.96 3.92 3.86	4.15 4.24 4.22	3.81 4.10 4.28	$3.06 \\ 3.36 \\ 3.77$	$2.84 \\ 2.95 \\ 3.17 $	2.81 2.83 2.88	2.84 2.85 2.89
115	2. 32	2. 54	2.73	2.91	3.12	3.33	3. 56	3. 81	4, 19	4.33	4, 13 4, 32	3. 51 3. 91	2.99 3.20	2.96
125 135 145		2. 51	2. 67	2.85	3. 04 2. 97	3. 23	3. 44	3. 57	4.04	4.25 4.20 4.15	4.30 4.36 4.33	4.20 4.38 4.44	3. 56 3. 94 4. 28	3. 35 3. 73
156 168 195		2.42	2.56	2.72	2.89	3.06	3. 24 3. 12	3.47	3.81 3.68	4,01	4. 18	4. 41		
235		2. 33	2.44	2. 56	2.70	2.84	2.98							

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Temperature (in ° C.)	1				Р	ercent	of com	bined	sulphu	ir			-	
(in ° C.)	0	2	4	6	8	10	12	14	18	20	23	26	29	32
-75 -55	2. 43 2. 42	2.44 2.44	2.52 2.52	2.56 2.56	2.55 2.55	2.54 2.54	2.58 2.59	2.60	2, 59	2.62	2.60	2.63	2.65	2.69
-45 -40 -35	2.41	2. 43	2. 51	2. 55	2. 55	2. 54	2.59	2.61	2.62	2.65	2.63	2.66	2.68	2.70
-27 -20	2.52 2.51	2.45 2.47	2.53 2.55	2.55 2.57	2.54 2.55	2 . 54 2. 54	2.60 2.59							
-10 0 14	2.48 2.39 2.37	$ \begin{array}{c c} 2.55 \\ 2.64 \\ 2.68 \end{array} $	2.62 2.76 2.92	$ \begin{array}{c c} 2.62 \\ 2.74 \\ 2.96 \end{array} $	2.58 2.65 2.84	2.56 2.60 2.72	2. 60 2. 62 2. 70	2.62 2.67	2.64 2.66	2.68 2.69	2.67 2.69	$2.71 \\ 2.72$	2.73 2.74	2.76
25 35 45 55 65	2. 36 2. 36 2. 34 2. 34	2, 69 2, 63 2, 61 2, 58	2.98 2.87 2.85 2.83 2.83 2.81	3.08 3.07 3.07 3.06 3.04	3. 05 3. 22 3. 29 3. 29 3. 29 3. 28	2.91 3.18 3.35 3.43 3.45	2.83 3.05 3.29 3.47 3.58	2.75 2.89 3.04 3.29 3.49	2.68 2.72 2.79 2.91 3.07	2.70 2.71 2.74 2.81 2.90	2.70 2.69 2.71 2.73 2.78	2.74 2.73 2.74 2.75 2.75 2.76	2.75 2.75 2.76 2.76	2.782.782.79-2.81
75 85 95	2, 23	2.57	2.79	2,99	3.25 3.20	3.44 3.40 3.37	3.62 3.61 3.58	3.64 3.73 3.75 2.74	3. 28 3. 56 3. 75	3.04 3.27 3.48	2.84 2.95 3.09	2.79 2.85 2.93	2.79 2.81 2.84	2.82 2.84 2.86
105		2. 55	2. 73	2.92	0.10 	3. 23 3. 27	3. 49	3.74	3.93	3.87	3. 28 3. 50	3.18	2.90	2.90
125 135 145 156		2. 51	2. 67	2.85	2.98	3. 15	3.35	3. 73 3. 63 3. 59	3.95 3.93 3.90	3.97 4.02 4.03 4.02	3. 89 4. 00 4. 07	3. 56 3. 77 3. 97	3. 23 3. 44	3. 04
168 195		2. 43 2. 38	2.57 2.51	2.72 2.65	2, 91 2, 82	3. 06 2. 96	3. 25 3. 14	3. 49 3. 34	3. 81 3. 63	3. 99	4.09			
211 225 235		2, 33	2. 44	2.56	2. 70	2.80	2, 92	3. 27 3. 19 	3. 54					

TABLE 5.—Dielectric constant of	compounds.	of purified rubber	and sulphur	at 100,000
	cycles per	second		-

 TABLE 6.—Dielectric constant of compounds of purified rubber and sulphur at 300,000
 cycles per second

Temperature					Р	ercent	of con	nbined	sulphu	ır				
(ш 0.)	· 0	2	4	6	8	10	12	14	18	20	23	26	29	32
-75 -55	2.45 2.43	2.45 2.46	2.54 2.54	2.58 2.58	2.57 2.57	2.55 2.56	2.59 2.60	2.63	2.61	2.64	2.62	2.64	2.67	2.70
-45 -40	2.42	2.45	2.53	2.56	2.57	2.56	2.61	2.64	2.64	2.67	2.65	2.67	2.69	2.71
-27 -20	2. 53	2.45	2.53	2.56 2.57	2.55	2.55	2.61							
-10 0	2.50 2.41 2.40	2.50 2.58	2.58	2.60	2.57 2 61 2 72	2.56	2.60	2.64	2.66	2.70	2.68	2.71	2.73	2.76
25	2.38 2.37	2. 01	2. 85 2. 92 2. 87	2. 03 2. 97 3. 01	2.90	2.07	2. 75	2. 75	2.69	2.73	2.71	2.73	2.74	2.78
45	2.36	2.63	2.86	3.06	3.20 3.25	3.18	3. 10 3. 27	2.92	2.77	2.75	2.72	2.74	2.76	2.78
75	2.30	2.00	2.80	3.00	3. 27 3. 26	3. 41	3. 44	3. 27 3. 47 3. 63	2.90	2.87	2.78	2.70	2.79	2.80
95 105	2.34	2. 55	2. 82	2. 95	3. 20 3. 17	3. 39 3. 36	3.58 3.56 3.56	3.68 3.72	3. 53 3. 71	3. 29 3. 50	3. 02 3. 16	2.90 2.98	2.81	2.85 2.85 2.89
115		2, 52	2.69	2.88	3. 09	3. 31 3. 27	3. 51 3. 48	3.70 3.69	3.82 3.87	3.67 3.82	3 32 3.51	3.08 3.22	2.95	2.93 3.01
135 145 156		2.48	2.64	2. 81	3. 01	3. 18	3.40	3.66 3.61	3.89 3.88 3.87	3.91 3.98 3.99	3.67 3.83 3.94	3.36 3.56 3.74	3. 12 3. 28	3.08
168 181		2.44	2. 59	2.75	2.94	3. 10	3. 29	3. 52	3. 83	3. 99 3. 95	4.04			
195 211 225		2.39	2. 53	2.68	2.85	3.00	3. 16	3.38 3.31 3.22	3.67 3.58					
235		2.34	2.46	2, 58	2.72	2.83	2.96							

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All of the charts have as a prominent feature the diagonal ridge which was mentioned previously. This ridge increases in height from low to high percentages of sulphur. On the low temperature side of the ridge, there is a steep slope which falls away to an almost level area. This level area represents a region in which the dielectric constant changes little with composition and temperature. On the high temperature side of the ridge, the slope is gradual and fairly uniform. In this region the dielectric constant decreases with rising temperature and increases as the percentage of sulphur becomes larger.

A change of frequency changes the height and position of the maximum or ridge, and changes the steepness of the slope on the low sulphur side of the ridge. Increasing the frequency has the effect of shifting the maximum to lower percentages of sulphur and to higher temperatures. Or, in other words, with increasing frequency, the diagonal ridge is displaced toward the right of the chart. The height of the maximum becomes less as the frequency is increased—that is,



FIGURE 13.—Effect of frequency and temperature on the dielectric constant of rubbersulphur compounds containing 4, 12, and 23 percent sulphur.

the rate of change of the dielectric constant with composition and temperature is less rapid the higher the frequency. The regions on either side of the ridge are affected relatively little by changes in frequency.

The composition-temperature relation shown in the isograms in figures 9 to 12 may also be represented for particular rubber-sulphur compounds, as in figure 13, where the dielectric constants at 60, 1,000, and 100,000~are plotted against the temperature for compounds containing 4, 12, and 23 percent of sulphur. The relation of the curves for a single frequency indicates not only the manner in which, with increasing sulphur, the value of the maximum of the dielectric constant increases, but also how its position shifts to higher temperatures. It is on account of this shift in position that the curves for the compounds containing the highest percentages of sulphur do not reach maxima below temperatures at which decomposition becomes appreciable. These curves also show clearly how the maxima move to higher temperatures with increasing frequency.

(b) POWER FACTOR

The results which show the relation of power factor to composition and temperature are given in tables 7 to 11, there being 1 table for each of the 5 frequencies. Corresponding isograms are given in fig-







FIGURE 15.—Isograms of the power factor of rubber measured at 1,000 \sim . For meaning of symbols see figure 14.

ures 14 to 17 for four of the frequencies, the chart for $3,000 \sim \text{being}$ omitted because of its similarity to the chart for $1,000 \sim$. The charts are characterized by a prominent diagonal ridge, simi-

lar in position to that for the dielectric constant. There are, how-

ever, two differences between the charts for the power factor and for the dielectric constant. Approximately level areas in which the power factor is little affected by changes in either temperature or



FIGURE 16.—Isograms of the power factor of rubber measured at $100,000 \sim$. For meaning of symbols see figure 14.



FIGURE 17.—Isograms of the power factor of rubber measured at $300,000 \sim$. For meaning of symbols see figure 14.

composition occur not merely on one but on both sides of the ridge, and on the high temperature side of the chart there is the beginning of a steep upward slope which in some cases is not shown, as thermal decomposition sets the limit at a lower temperature.

Tomporatura					Р	ercent	of com	bined	sulphu	ır				
(in ° C.)	0	2	4	6	8	10	12	14	18	20	23	26	29	32
	3.0	2.8	2.8	3. 2 1. 1	3.0	3.1 1.0	3.4 1.5	1.9	2.6	3. 9	4.7	5.5	4.3	4.2
-55	4.5	3.6 30.4	2.3	1.6	1.0	1.1	1.5	2.1	2.6	3.6	4.3	5.2	5.1	3. 9
-35	0	28.9	53.9	39.1	8.1	.6	0.2	8.7	7.7	6.0	5.9	4.3	4.5	4.1
-25 -20	1.1	11.9 5.3	38.8 20.2	69.2 52.0	40.3 74.3	9.8 33.2	2.3	2.3	2.1	2.3	2.7	3.4	3.8	4.1
0 14	2.1 1.6	2. 4 2. 3 1. 6	4.0 1.8	19.7 8.4 2.7	27.5 7.7	69.4 24.4	40.8 82.4 62.0	32.3 84.4	2. 2 2. 3 4. 6	2.2 2.3 1.5	2.6 1.6	3.3	4.2	4. 8
25	2.3	1.9	1.6 3.0	1.7 2.8	3.3 2.8	9.2 4.3	28.4 10.9	69.8 38.7	25.6 71.5	3.0 12.4	1.7	2.2 2.1	3.2	3. (
45	4.5	2.8	1.9	1.7	1.4	2.2	4.5	18.6	90. 4 54. 0	49.6	3.4 13.2	2.8 3.4	3.2	3. 4
65	5.4	2.7	1.7	1.4	1.0	1.1	1.5	4.1	33.6	78.9	39.4	4.6	3.2	3.7
75 85 95	5.5	2.8	1.8	1.9	1.1	1.0	.9	2.3 1.9	$13.6 \\ 6.6 \\ 4.0$	43.8 22.7 10.1	88.4 82.2 47.3	18.9 55.0 89.0	3.2 3.0 14.0	3.4 4.0
105 115	5.3	3.4	2.4	2.5	1.1	1.2	1.0	1.0	3.6	6.7	23. 1 12. 1	74.0 42.9	48.1 86.0	22. 7 51. 1
125		3. 5	2.7	2.7	1.6	1.7	1.5	.9	4.8	6. 6	9.1	18.2 9.5	69.2 37.1	88.4
145 168 105		4.0 6.2 10.7	4.0 6.4	4.2 8.0	2.6 6.4 26.9	3.1 8.2	3.4 10.4 70.3	$\begin{array}{c} 1.9\\ 8.2 \end{array}$	$4.3 \\ 12.5$	$5.9 \\ 13.1$	$5.1 \\ 6.7$	4.9	14.7	

TABLE 7.—Power factor of compounds of purified rubber and sulphur at 60 cycles per second $^{\rm 1}$

¹ Multiply the values in the table by 10^{-3} to obtain the power factor.

TABLE	8.—Power	factor of	of compound	s of pu	rified ru	ubber and	sulphur d	at 1,000	cycles
			p_{i}	er secon	nd 1				

Memperature	Percent of combined sulphur													
(in ° C.)	0	2	4	6	8	10	12	14	18	20	23	26	29	32
-75	1.4	1.1	1.3	1.6	2.0 1.7	1.9	2.2 2.1	4.2	3.8	4.6	5.3	6. 0	5.2	5.0
	2,4	1.8 9.2	1.4	1.4	1.4	1.4	1.9	2.6	4.0	4.7	5.8	6.4	5.9	5.8
-35	3.3	35. 9	28, 5	13.6	4.7	2.1	1.9	3.3	4.1	4.8	5.7	5.7	5.7	5.2
-27 -20	2.1	36.4 20.7	53.5 49.3	39.4 68.0	14.1 39.8	4.6 13.7	2.3	2.6	3.0	3.4	4.3	4.7	5.2	5.1
0 14	1.3 1.2 1.1		$ \begin{array}{r} 27.2 \\ 12.6 \\ 4.1 \end{array} $	59.3 29.0 10.3	78.1 69.9 31.9	45.5 84.9 70.6	10. 2 50. 8 89. 8	4.3 15.6 71.7	2.8 3.1 3.9	3.3 3.3 2.3	$3.9 \\ 2.7$	4.8	5.2	5.2
25	1.4	1.6	2.2	4.1	13.1	34.3	70.6	89.5	13.5	3.0	2.5	3.2	4.2	4.3
45	2.8	1.0 2.2	2.1 2.0	3. 2 2. 6	0.9 3.6	8.2	42.0 19.3	79.0 54.2 29.9	80.0	24.5	2.0 3.3 7 1	3.3 3.3	4.2	4.7
65	3.3	2.5	1.9	2.0	1.8	2.9	5.0	17.5	74.9	91.0	20.3	4.5	4.1	4.3
75	3.7	2. 9	2.0	1.7	 1. 1	1.5	2.1	9.7 6.8	45.2 26.8	85.6 61.7	46.4 88.1	11.4 28.3	4.2	4.4
105	3.7	3.2	2.0	1.6	. 9	1.0	1.1	3.2 2.2	13.9 8.5	37. 3 22. 4	89.2 66.2	66. 1 90. 3	10. 3 23. 9	6.4 13.1
115		3.0	1.8	1.4	8	7		1.1	3.0	12.8 7:2	40.8	85.7 57.7	58.2 88.8	34.4
136 145		3.0	1.9			.9			1.6	3.0	13.5 8.0	39.0 22.8	82.0 54.5	91.0
156												11.0		
168 195		3.0 3.7	2.0 3.0	1.6 3.5	1.2 3.1	1.2	1.1	$1.0 \\ 69.1$	1.6 145.6	2.1	2. 5			
230		21. 5	28.1	47.0	68.5	142.0	162.8							

¹ Multiply the values in the table by 10^{-3} to obtain the power factor.

TABLE	9.—Power	factor of	of compound	ls oj	f purified	l rubber	and	sulphur	at 3,000	cycles
			p	er s	econd 1					

							1. Sec. 1. Sec								
Temperature		Percent of combined sulphur													
(in ° C.)	0	2	4	6	8	10	12	14	18	20	23	26	29	32	
-75	1.4	0.9	1.2 1.4	1.9	2.1	1.9	2.1	3. 2	3. 3	3.2	5.3	5.8	5.2	4.7	
-55 -45	1.7	1.4	1.4	1.3	1.6	1.6	2.1	2.6	4.1	4.7	5.8	6.1	5.8	2.5	
-35	3.5	20.2	14.1	6.8	3.4	2, 1	1.7	2.4	3.7	4.6	5.5	5.9	6.0	5.8	
-27 -20	2.5 1.8	38.4 32.1	42.8 56.1	25. 2 53. 1	9.9 23.9	4.3 8.7	2.7 3.8	2.3	3.2	4.1	4.8	5.4	5.5	5.3	
—10 0	1.2 1.3	15.4 6.7	41.8 20.2	69.7 43.7	63.8 79.8	30.0 71.6	11.5 35.2	4.2 11.6	3.0 3.0	3.8 3.6	4.2	5.0	5.5	5. 3	
14	.8	2.5	6.5	17.0	48.0	84.9	83.2	44.0	3.6	2.6	3.2				
25 35		$1.8 \\ 1.5 \\ 0.0 $	3.4	7.5	23.5 12.6	53.9 30.2	87.9 63.0	83.2 91.2	11.4 28.4	3.5	3.3	3.9	4.8	4.7	
45 55	2.5	2.0	2.1	3.8	6.2	14.9 8.4	33.1 18.9	73.4	66.4 95.4	19.3 52.0	3.3	3.9	4.9	5.3	
69	2.7	2. 2	1.8	2.3	2.6	4.8	10. 2	28.5	91.4	82.9	16.2	4.3	4.2	4.2	
/0 85	2.8	2.5	1.7	1.9	1.3	1.9	3.6	16.5	04. 7 42. 5	94. 0 79. 9	42.5	9.5	4.3	4.4	
95								6.0	24.9	55.6	93.1	49.4	9. 0	6.6	
105	3.0	2.8	1.9	1.5	.9	1.1	1.7	3.8	15.9	37.0	82.9	83. 2	21.1	12.5	
115									8.9	21.7	58.3	91.5	46.4	27.1	
136		2.8	2. 5	1.4	1.0	.8	. 1	1.9	5.3	12.8	30.0 23.8	58.0	80.0	98, 9	
145		4.2	2.0	1.3	.7	. 6	.6	.9	2.3	5.3	15.1	39.3	73.4		
156			1.0						1 2		4 1	20.8			
195		2.9	2.1	$\frac{1.4}{2.1}$	1.6	2.0	2.3	31.4	53.0	2.0	4.1				
235		9.4	11.2	18.4	25.9	47.4	52.5								

¹ Multiply the values in this table by 10^{-3} to obtain the power factors.

TABLE 10.—Power factor of compounds of purified rubber and sulphur at 100,000 cycles per second $^{\rm 1}$

Temperature	Percent of combined sulphur													
(in ° C.)	0	2	4	6	8	10	12	14	18	20	23	26	29	32
-75 -55	0.8 1.0	0.7 1.3	$1.1 \\ 1.5$	$1.2 \\ 1.5$	1.4 1.8	$1.6 \\ 2.0$	1.7 2.1	2.6	3.4	3. 7	4.0	4.1	3.9	3. 5
-45 -40	2.4	2.6	2.6	2.4	2.0	2.1	2.2	3.2	4.6	5.1	5.8	6.1	5.9	5.2
	3.5	13.5	11.4	3. 8 7. 3	4.5	2. 2 3. 2	2.8							
-20 -10	3.2 2.2	$24.6 \\ 37.7$	$\begin{array}{c} 21.\ 2\\ 42.\ 3\end{array}$	$\begin{array}{c}14.4\\33.3\end{array}$	7.6 18.5	4.8 10.2	3.6 6.0							
0 14	1.4 .6	33.5 17.5	57.5 45.7	$61.4 \\ 64.2$	42, 1 76, 5	23.7 59.5	12.8 35.1	6, 9 19, 1	5.5 6.0	5.8 5.5	7.1 6.7	7.9 7.6	7.9	7.0
25 35	.8 1.3	$11.6 \\ 4.2$	$31.2 \\ 13.0$	45, 8 30, 5	74. 8 57. 1	$\begin{array}{c} 85.1\\ 86.6\end{array}$	$\begin{array}{c} 64.2\\ 93.1 \end{array}$	38. 1 67. 9	9.4 17.4	5.8 8.2	6.3 5.7	7.4 6.3	7.5 6.8	7.2 6.6
45 55	1.3	2.9	5.3	18.3 12.1	37.2 23.8	65.8 46.2	91.0 75.0	85.5 96.0	29. 2 53. 3	12.6 23.7	6.1 7.9	$\begin{array}{c} 6.2\\ 6.3\\ 6.7\end{array}$	6.8	6.3
75	1, 0	1.9	4.1	9.4	9.9	30. 2 19. 3	36. 1	67. 0	97.5	65.0	21.5	9.0	6.6	6.4
85 95	1.3	2.3	3.4	4.3	6.8	12.8 8.7	25.8 16.0	47.8 34.9	95.8 81.9	88.1 97.3	39.0 61.1	15.0 25.3	7.1 9.5	6.8 8.1
105 115	1.6	2.4	2.5	3.0	3.5	6.3 4.2	10.5 5.3	$ \begin{array}{c} 24.4 \\ 17.7 \end{array} $	64. 2 46. 6	91.4 72.7	83. 1 94. 3	40.6 63.2	13.5 23.0	16.8
125 135		2.3	2. 2	2.4	2. 2	3.6	4.1	11.4 8.4	$34.7 \\ 24.9$	57.7 44.0	91. 1 77. 8	81. 2 92. 4	39.6 61.1	28.6 47.6
145 156 168		2.3	1.9	2.1	1.2	2.1	2.9	6.2	17.9	33.2 24.3	66.8 48.1 31.2	89.8 75.3	83. 2	
195		2.3	2.0	1. 8	1. 5	1.6	1.5	3. 7	6.7					
211225								5.0 7.6	7.2				-	
235		2.8	2.1	2.1	2.1	3.8	2.8							

¹ Multiply the values in this table by 10^{-3} to obtain the power factors.

Temperature (in ° C.)	Percent of combined sulphur													
	0	2	4	6	8	10	12	14	18	20	23	26	29	32
-75 -55	0.7 1.2	0.9 1.2	1.0 1.5	$1.2 \\ 1.6$	1.8 1.8	$1.8 \\ 1.9$	1.9 1.9	2.4	3.0	3. 2	3.6	3.5	3.6	3. 5
-45 -40 -35	2.1 2.4	2.2 	2.4	2.3	2.2 2.6	2.3 2.3	2.4	3.3	4.3	4.6	5.4	5.5	5.3	4.7
-27	3.2	8.5	8.1	5.7	3.7	3.0	2.8							
-10	2.7	29.7 37.6 27.0	28.9 49.4	21.9 44.0 67.8	13.0 27.7 62.4	7.8	5.6 10.1	6.7	6.1 7 3	6.4	7.7	8.1	7.8	7.3
25	1.0	19.5	42.6	62.9	79.4	70.0	45.1	28.2	9.6	7.1	7.9	8.5	8.5	7.7
45 55	1.1	4.5	$ \begin{array}{c} 22.4 \\ 13.2 \\ 9.0 \end{array} $	48.8 30.7 21.7	75.5 58.1 40.1	89.2 87.3 65.9	90.9 90.5	50.0 74.8 93.5	13. 5 24. 9 41. 8	9.0 13.3 20.9	7.5 7.7 9.7	7.9 7.7 8.0	8. 3 7. 8	7.6
65 75	1.3	2.7	6.2	13.4	25.6 17.9	49. 1 32. 2	76.0 60.0	98.9	64.0 89.0	33. 2 55. 0	9.8 20.8	8.2 10.8	5.8 7.7	7.6
85 95 105	1.4	2.8	4.7	9.0	13.3 9.3 6.7	$ \begin{array}{c c} 24.8 \\ 17.2 \\ 12.3 \end{array} $	41.4 30.0 21.2	70.1 51.7 38.8	101. 0 98. 9 85. 5	75.7 93.4 98.2	32.5 51.7 69.7	15.6 23.2 34.6	8.4 10.6 14.3	7.4
115						8.3	14.2	29.5	66.1	92.9	83.9	52.5	22.4	17.1
125 135 145		2.4	2.0	2.7	2.7	4.3	6.0	16.3 12.0	40.1	63. 2 50. 6	94.0 86.2	86.6 94.2	48. 2 72. 2	36.8
168		2.3	2.1	2.4	2.2	3.5	4.1	7.4	23.8	40.1 27.8	68.1 46.3	92.4		
181 195 211		2.2	2.0	2.2	1.8	2.4	2.6	5.8 5.2	10. 8 9. 4	19.4				
225 235		2.8	2.4	2.1	2.1	4.9	2.2	5.6						

TABLE	11.—Power	factor	of	compounds of	purified	rubber	and	sulphur	at	300,000
				cycles per s	second 1					

¹ Multiply the values in this table by 10^{-3} to obtain the power factors.



FIGURE 18.—The effect of frequency and temperature on the power factor of rubbersulphur compounds containing 4, 12, and 23 percent of sulphur.

A comparison of the isograms in figures 14 to 17 shows that a change in frequency has the same type of effect on the power factor that it has on the dielectric constant, in that the characteristic diagonal ridge is shifted toward the high temperature side of the diagram

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with increasing frequency. The steepness of the slopes on both sides of the ridge becomes less with increasing frequency. Unlike the dielectric constant for the same compound, increase of frequency does not change the height of the maximum very much and apparently increases it rather than decreases it.

The relation of power factor to temperature and composition may also be illustrated for particular compositions as in figure 18, where the power factors at 60, 1,000, and 100,000 \sim are plotted against the temperature for compounds containing 4, 12, and 23 percent sulphur. The height of the maximum at a single frequency increases rapidly with sulphur content up to about 12 percent of sulphur. Further increase of the sulphur content does not materially change the height.



FIGURE 19.—Isograms of "1-minute" conductivity.

Each line connects points having equal values of the conductivity. The values are given in mho/cm. A is the limit set by the flow of the specimen. B is the limit set by the decomposition of the specimens. Measurements were not recorded below E because of difficulties with the apparatus.

The position of the maximum is shifted to higher temperatures as the sulphur content is increased. These curves also show the manner in which the maxima move to higher temperatures with increasing frequency.

(c) CONDUCTIVITY

The data pertaining to the effect of composition and temperature on the conductivity are given in the form of isograms, which are shown in figures 19 and 20, respectively.

The chart for the "1-minute" conductivity somewhat resembles similar charts for the power factor and dielectric constant in spite of the fact that it has been necessary to use a different kind of scale in spacing the isograms; that is, using a power of 10 between isograms instead of a fixed numerical difference. The diagonal ridge is fairly well defined. Along most of its length, it rises to an approximately uniform height of a little more than 10^{-16} mho/cm, but at its low sulphur end it goes to above 5×10^{-15} mho/cm at -50° C. There is a decided increase of conductivity with temperature above 25° C., and this increase makes difficult the location of the ridge at high percentages of sulphur. For many specimens, the conductivity at 225° C. is 10,000,000 times that at 25° C. Since measurements were made on a single set of specimens, and small amounts of impurities may cause considerable variation in the conductivity, precise significance should not be attached to minor features or irregularities in the isograms.

The "short-time" conductivity chart bears a closer resemblance to the power factor than to the "1-minute" conductivity chart. The diagonal ridge is high and well defined, and it has steep slopes. On either side is located an area of low conductivity where changes in temperature or composition produce little effect. At low temperatures and low percentages of sulphur, the ridge almost disappears, but with rising temperature and increasing sulphur content the ridge reaches a height of about 100 times that of the low conductivity area



FIGURE 20.—Isograms of "short-time" conductivity.

Each line connects points having equal values of conductivity. The values are given in units of 1×10^{-12} mho/cm. A is the limit set by flow of specimen. B is limit set by decomposition of specimen. D is limit set by range of apparatus. Measurements were not recorded below E because of difficulties with the apparatus.

on either side. As in the case of the power factor, the upward slope becomes evident only at the very edge of the chart on the high temperature side. Its similarity to the chart of 60-cycle power factor is particularly marked. The ridge falls at the same place on both, suggesting a possible connection between the "short-time" conductivity and the 60-cycle power factor. This will be studied further.

A comparison of the "short-time" and the "1-minute" conductivities for one compound containing 12 percent of sulphur is shown on a semilogarithmic scale in figure 21. This figure indicates that the "short-time" conductivity is considerably higher than the "1-minute" conductivity at ordinary temperatures, but that the two approach and become very nearly the same at the highest temperatures used in the measurements. Comparison of the two conductivities for other compositions led to similar conclusions.

VII. COMPARISON OF ELECTRICAL PROPERTIES OF COM-POUNDS OF PURIFIED RUBBER AND SULPHUR WITH THOSE OF CRUDE RUBBER AND SULPHUR

A comparison of the results reported in this paper with those previously reported for crude rubber indicates that purification of the rubber has only a relatively small effect on the dielectric constant and power factor, and a larger effect on the conductivity. The curves in figure 8 of this paper were made under the same conditions as the curves in figures 8, 9, and 10 of our previous paper giving results on crude rubber.¹⁵ The curve of dielectric constant for compounds of purified rubber is slightly lower than the corrresponding curve for crude rubber except that the maximum value at about 11 percent of sulphur is the same (3.75) for the two. The values of the dielectric



FIGURE 21.—"Short-time" and "1-minute" conductivities for a rubber containing 12 percent of sulphur.

constant for purified rubber specimens are, on the average, about 0.06 lower than those of the corresponding crude rubber specimens on the low sulphur side of the maximum and about 0.08 lower on the high sulphur side of the maximum. The maxima of the two curves occur at the same composition for both.

The power factors of the purified rubber specimens are lower than those of the corresponding crude rubber specimens except in the region of the maximum, where the power factor of the purified rubber appears to rise to a higher maximum than that of crude rubber. In the regions of low and of high percent of sulphur, the power factors of specimens of purified rubber are generally 30 to 50 percent less than those of crude rubber.

The conductivity of purified rubber is not so readily compared with that of the previous work on crude rubber, since in the previous paper the resistivity was plotted instead of the conductivity. A comparison of values shows that the conductivity of crude rubber is from 5 to

¹⁵ See reference in footnote 1, p. 411.

20 times as much as the corresponding specimens of purified rubber excepting in the region of the maximum where there seems to be little difference between the two.

The values for the dielectric constant and power factor of crude rubber as determined by Kitchin¹⁶ and by Kimura¹⁷ and his associates are appreciably larger than those herein reported. The curves which they give indicate that purification of the rubber does not change the temperature or the frequency at which either the dielectric constant or power factor is a maximum.

VIII. SELECTION OF RUBBER COMPOUNDS FOR SPECIFIC USES

The properties of rubber and its compounds with sulphur have a direct bearing on many practical problems because such sulphur compounds form the basis of virtually all commercial rubber products. While the commercial rubber products are usually compounded with fillers and other ingredients which affect the electrical properties of the product, the effects of these are superimposed on the basic properties of the rubber-sulphur compounds.

If rubber of low dielectric constant is required, as for the insulation of submarine telephone or telegraph cables, an inspection of isograms in figures 9 to 12 indicates clearly that the best results are to be obtained with the lowest practicable proportion of combined sulphur. For example, at 25° C. and $1,000 \sim$ the rubber alone has a dielectric constant of 2.4; with 1 percent of combined sulphur, the dielectric constant is about 2.5, and with 3 percent of sulphur it is about 2.8. If a dielectric constant less than 2.8 is necessary, percentages of combined sulphur less than 3 percent must be used. If it is desirable that the insulation have the mechanical properties of the high sulphur or hard-rubber range, a dielectric constant as low as 2.75 can be obtained. The use of the minimum percent of sulphur in such cases is in accordance with recent developments in cable engineering,¹⁸ some of which have resulted from this investigation.

When a low power factor is needed, the frequency used must be taken into account. At 60 \sim and 25° C., specimens containing up to 8 percent of combined sulphur have power factors of less than 4×10^{-3} , but the range of composition having this low power factor at 300,000 \sim lies below 1 percent of combined sulphur. About as low a power factor can be obtained with high sulphur content; that is, above 20 percent at 60 \sim . However, the power factor in this range of composition increases but little with frequency rising to about 8×10^{-3} at 25° C. and 300,000 ∼.

The results show that purification of the rubber makes it possible to obtain soft rubber having as low conductivity as hard rubber, whereas if the rubber is not purified the soft rubber has much higher conductivity than the hard rubber. It is of advantage, then, to purify the rubber when a low conductivity is desired. If the rubber is purified, it makes little difference, as far as conductivity at 25° C., is concerned, what composition is used with the exception of the range

¹⁶ Donald W. Kitchin, Studies in the Vulcanization of Rubber. Part V. Dielectric Constant and Power Factor of Vulcanized Rubber, Ind. & Eng. Chem., vol. 24, p. 549, May 1932.

 ¹³ See footnote 2.
 ¹⁴ H. E. Rowand, Part I, E. W. Davis and G. J. Crowdes; Part II: Submarine Telephone Cable. Electrician, 107, p. 721, November 1931.
 A. R. Kemp, Paragutta, A New Insulating Material for Submarine Cables, Bell System Techn. J., vol. 10, p. 132, 1931.

from 14 to 24 percent of sulphur where the conductivity is definitely higher.

The conductivity of rubber does not necessarily increase with temperature as is usually assumed in the preparation of tables and formulas which are used in specifications for correction of temperature. This is clearly shown by figure 19. It may either increase or decrease according to the temperature range used. For instance, the conductivity of a specimen containing 8 percent of sulphur decreases with temperature from 0° C. to about 20° C. and then increases as the temperature is increased above this. This indicates quite clearly the inadequacy of the usual formulas for correcting the conductivity or resistivity for differences in temperature.

IX. CONCLUSION

The electrical measurements given in this paper differ from those previously reported by the authors and by other investigators in that they were made on specimens prepared from purified rubber. The purification, which involved the removal of proteins; resins, and water-soluble salts, affected all the electrical properties to some extent, but did not alter the general character of the variation in electrical properties with composition, temperature, or frequency.

The results of the present investigation afford comprehensive data on the electrical properties of rubber-sulphur compounds, and may form a basis for designing rubber compounds for specific electrical uses. They also demonstrate the inadequacy of the simple numerical coefficients that are sometimes employed to evaluate the changes in the electrical properties with temperature and frequency. A discussion of the results from the standpoint of modern dielectric

A discussion of the results from the standpoint of modern dielectric theory is not included in the present paper, but is contemplated or a separate paper.

X. ACKNOWLEDGMENT

Acknowledgment is made of the important assistance which A. D. Cummings rendered in preparing many of the specimens used in this investigation and in helping with some of the electrical measurements.

WASHINGTON, April 17, 1933.

205-33-4