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## Alternating Current Method Of Measuring The Electrical Conductivity Of Dielectric Liquids

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## Alternating Current Method of Measuring the Electrical Conductivity of Dielectric Liquids

Henry E. Fischer; Ralph S. Carson



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### Protection Circuit Insulated for High Voltage

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(Received 14 December 1966)

**P**ROTECTION of expensive millimeter wave klystron tubes from heater over- and under-voltage was needed. The circuit giving this protection is described here. The basic circuit is adaptable to a variety of other protective functions merely by changing one component.

Figure 1 shows the protection circuit. The sensor is an optical meter relay with adjustable "set points." The optical meter relay uses a light-variable resistor for operating external circuits and is substituted in place of the normal heater voltmeter. This provides a stable sensor with an on-off output. The required high voltage insulation (the klystron heater floats above ground at the beam potential, which is approximately 2.5 kV for our millimeter wave tubes) is provided by an insulated transformer T1 on the input to the circuit and by the use of a light beam which triggers Q<sub>3</sub>, the 3P30 light-sensitive switch, on the output. The 3P30 and its associated circuit can thus be operated at ground potential.

The operation of the circuit is as follows: The light variable resistor R<sub>2</sub> is part of the optical meter relay circuit. The resistance of R<sub>2</sub> is small when the meter pointer lies between the set points. When the operating

voltage on the klystron heater lies between the pointers, transistors Q<sub>1</sub> and Q<sub>2</sub> conduct, lighting lamp I1 and triggering Q<sub>3</sub>. This energizes relay K1 and allows power to be applied to the protected equipment. If the voltage varies so that the meter pointer passes either set point, then Q<sub>1</sub>, Q<sub>2</sub>, Q<sub>3</sub>, I1, and the relay all turn off, removing power to the tube. Two of these circuits are needed, one for each set point. The power supply provides electrical energy for several circuits in parallel. The circuit is fail-safe in that any failure which turns off the lamp or the phototransistor turns off the system or prevents it from being turned on.

The circuit makes setting of the desired protection voltages easy, since the set points are clearly visible on the meter face and no instruments are required.

This circuit can be used in many types of on-off applications by changing the optical meter relay. These relays come in ranges of microamperes to amperes and millivolts to kilovolts without change of the output light variable resistance. Therefore, the circuit outside the meter relay remains the same.

### Alternating Current Method of Measuring the Electrical Conductivity of Dielectric Liquids

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**W**HEN direct voltages are employed in determining the conductivity of dielectric liquids, the much-discussed polarization effects are present. This polarization phenomenon, which has not been completely explained, causes difficulty in obtaining a very accurate measurement of electrical conductivity. In spite of this problem, it is generally agreed in the literature that direct voltages continue to be used for this purpose. This general agreement stems mainly from the fact that in order to obtain meaningful measurements using an alternating voltage, the period of the applied voltage must be long compared to the charge relaxation time of the liquid. This requires quite low frequency sources for some dielectric liquids with low conductivity.

This article demonstrates an ac method of measuring the conductivity of dielectric liquids in which the liquid itself becomes an integral part of a Wien-bridge oscillator. This is done by replacing the normal parallel resistance-capacitance tank circuit with a parallel-plate capacitor immersed in the liquid. This gives an equivalent resistance and capacitance in parallel. The equivalent resistance can be approximated by

$$R_2 = L/\sigma A \quad (1)$$

PROTECTIVE CIRCUIT SCHEMATIC

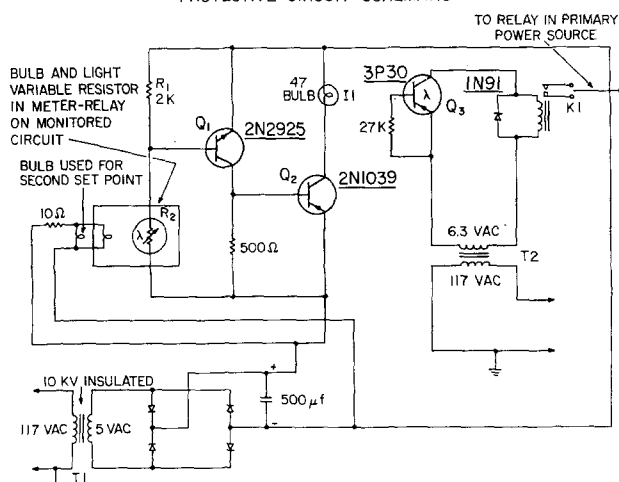


FIG. 1. Protective circuit schematic.

and the equivalent capacitance by

$$C_2 = \epsilon A/L, \quad (2)$$

where  $L$  is the plate separation in centimeters,  $A$  is the total plate area in  $\text{cm}^2$ ,  $\sigma$  is the liquid conductivity in reciprocal  $\Omega\text{-cm}$ , and  $\epsilon$  is the liquid permittivity equal to the product of the relative dielectric constant  $\epsilon_r$  of the liquid times the permittivity of free space,  $8.85 \times 10^{-14}$  F/cm.

The time constant of the parallel combination is

$$R_2 C_2 = (L/\sigma A)(\epsilon A/L) = \epsilon/\sigma. \quad (3)$$

It is known that the charge relaxation time of an electrically conducting material is equal to the time constant  $\epsilon/\sigma$  associated with the exponential decay of a charge-density disturbance within the material. The charge relaxation time used here is not the dielectric relaxation time which is determined by molecular activity and polarization decay or build-up. Charge relaxation time is determined by the mobilities of ions and impurities within the liquid. Equation (3) relates charge relaxation time to the time constant  $R_2 C_2$  of a parallel resistance-capacitance combination. Therefore if a circuit can be found whose operation is sensitive to such a parallel combination, and if  $R_2 C_2$  can be calculated accurately from measurements made on the circuit, then the charge relaxation time can be determined. A Wien-bridge oscillator is one such circuit.

The frequency-determining equation of a Wien-bridge oscillator is

$$f = 1/2\pi(R_1 C_1 R_2 C_2)^{1/2}, \quad (4)$$

where  $R_1$ ,  $C_1$ ,  $R_2$ , and  $C_2$  are as shown in Fig. 1. Solving for  $R_2 C_2$  from Eq. (4) yields

$$R_2 C_2 = 1/(2\pi)^2 f^2 R_1 C_1. \quad (5)$$

Since the oscillator can be constructed such that  $R_1$  and  $C_1$  are known, and the frequency of oscillation can be measured, then Eq. (5) gives the charge relaxation time  $\epsilon/\sigma$ .

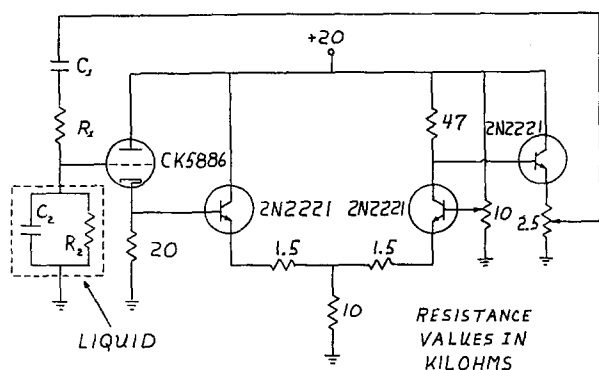


FIG. 1. Schematic diagram of device.

From Eqs. (3) and (5) the conductivity is obtained as

$$\sigma = \epsilon(2\pi)^2 f^2 R_1 C_1. \quad (6)$$

Values of the dimensionless relative dielectric constant  $\epsilon_r$  are commonly tabulated for liquids. Thus Eq. (6) becomes

$$\sigma = 3.5 \times 10^{-12} \epsilon_r f^2 R_1 C_1. \quad (7)$$

Equation (7) gives  $\sigma$  in reciprocal  $\Omega\text{-cm}$  units with  $f$  in hertz,  $R_1$  in ohms, and  $C_1$  in farads.

The oscillator used must have an input impedance considerably higher than that across the dielectric liquid in order that the input impedance does not influence the frequency of oscillation. This high input impedance was obtained by using a cathode-coupled electrometer tube for the input device. The oscillator was made direct coupled in order to eliminate phase shift at low frequencies due to coupling capacitors. Finally, the output of the differential amplifier was emitter-follower coupled back to the bridge input. The emitter-follower stage was employed to give a low output impedance which is an implicit assumption in Eq. (4). The entire circuit was then mounted in a shielded box to minimize stray field effects.

The capacitor which was immersed in the liquid to form the  $R_2 C_2$  combination was a small 10 pF radio-type variable. It has four stator plates and three rotor plates made of nickel plated brass. The rotor plates were semi-circular with a diameter of 28.6 mm, and the stator plates were about 34.9 by 12.7 mm. The plate spacing was about 2.38 mm. The actual physical dimensions are not particularly critical but too close spacing makes it difficult to eliminate air bubbles.

Several carbon and wire-wound resistors ranging from 1000  $\Omega$  to 20 M $\Omega$ , and several mica and ceramic capacitors ranging from 20 pF to 1000 pF, were mounted on plugs. These components were measured to an accuracy of 1% and simply plugged in as  $R_1$  and  $C_1$  to set the frequency of oscillation.

The frequency was measured with a frequency counter. Good sinusoidal waveshapes were obtained readily over a range from about 20 sec per cycle to over 50 kHz. In all cases it was desirable to adjust the setting of the 2.5 k output potentiometer to the lowest value for which oscillation occurred. The voltage across the plates of the liquid cell was only a few millivolts.

The device was used in the laboratory to measure the conductivities of three dielectric liquids—distilled water, glycerin, and chloroform. These initial measurements are not intended to be the most accurate since the capacitor plates were not platinized nor were there any means of closely controlling the purity of the liquids. However, with these limitations the readings obtained were in good agreement with published values. The average values of the conductivities obtained were: distilled water— $1.28 \times 10^{-6}$

$\Omega^{-1} \text{ cm}^{-1}$ , glycerin— $3.03 \times 10^{-8} \Omega^{-1} \text{ cm}^{-1}$ , and chloroform— $8.88 \times 10^{-10} \Omega^{-1} \text{ cm}^{-1}$ . The foregoing are called average values because the conductivities of the liquids varied slightly with frequency. The frequency of oscillation was always chosen to be small compared to the reciprocal of the charge relaxation time to make sure that the charge relaxation time requirement, mentioned earlier, was satisfied.

Independent corroboration of the results, using procedures employing conductivity cells of known cell constant, was not possible because the equipment was not readily available. However, the general accuracy of the method was tested. When known resistors and capacitors were used as  $R_2$  and  $C_2$  to simulate the range in which chloroform and glycerin fall, the agreement between measured frequency and the calculated frequency was within the 1% accuracy of component values.

### Ultrathin $\text{Al}_2\text{O}_3$ Support Films for Electron Microscopy\*

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(Received 7 November 1966; and in final form, 8 December 1966)

THE following technique has been used very successfully in the preparation of  $\text{Al}_2\text{O}_3$  films in the range of 75–100 Å and below. It was developed during a feasibility study on biological field ion microscopy (sponsored by the National Institutes of Health). A need arose for dielectric support films of 100 Å and thinner, but there was no method available for readily and easily producing them. Hence, this technique was developed.

The oxide films were formed by anodizing aluminum foil. Only selected areas of the aluminum were chemically removed by means of photo-lithography, leaving the remainder as a support grid.

The starting material used was 0.05 mm thick aluminum foil obtained from our own materials division. Sections of wrinkle-free foil 6.5 cm square were carefully cut and then degreased in baths of acetone, trichlorethylene, trichloroethylene, and alcohol. Any residual oxide formed during the manufacture of the foil was removed from the mirror-finished side by swabbing with a normal solution of sodium hydroxide and rinsing in distilled water. If this residual oxide was not completely removed, the thickness-voltage relationship of 15 Å/V could not have been used to control thickness.

The anodizing solution<sup>1</sup> used was sodium hydrogen phosphate (48 g), sulphuric acid (2 ml) and water (400 ml). With the voltage set to a predetermined level to obtain a desired thickness, the foil was anodized for thirty

seconds. Any abrupt change in current, other than the desired smooth decay, indicated a faulty specimen, which was discarded. The specimen was washed and dried upon

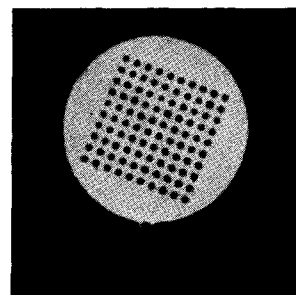


FIG. 1. The finished specimen is a grid pattern of 0.076 mm circular holes, over which lies the freely-suspended aluminum oxide film. The grid array is  $10 \times 10$  for a total of 100 openings.

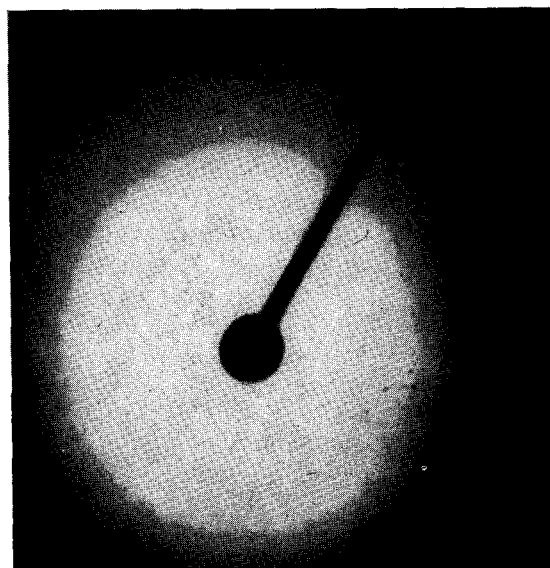


FIG. 2. Studies by electron diffraction show the oxide films to have no detectable structure of their own.

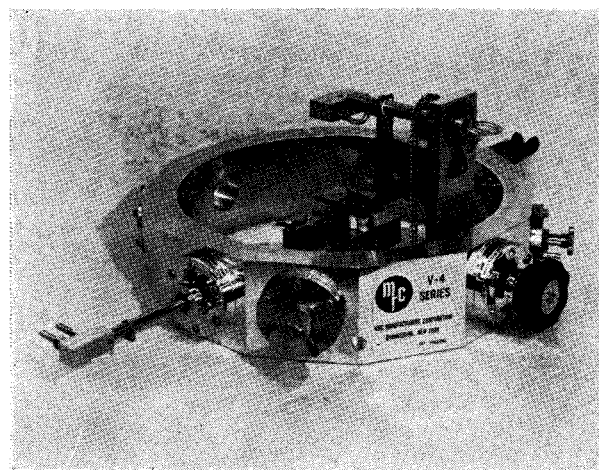


FIG. 3. MRC V4-7000 ultrashadowing module. It is important that only the shielded Ta filament and the evaporating material were heated, thereby keeping the amount of radiated heat reaching the specimen to a minimum. While evaporating tungsten at 3600°C, the substrate temperature was recorded at 38°C.