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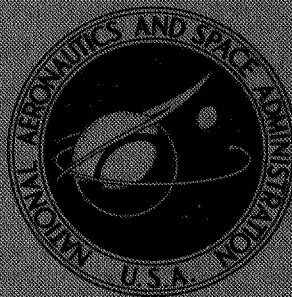
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SCREENING OF INORGANIC COMPOUNDS AS SOLVENTS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

The development of high-energy-density battery systems is hampered for want of electrolytes that are invariant in contact with electrodes fabricated from the materials found at either end of the electromotive force table. In an attempt to find a solvent that would be useful in making such an electrolyte, an investigation of inorganic compounds was conducted. The intent of this investigation was to screen those inorganic compounds which are liquid at room temperature and atmospheric pressure. The dielectric constant was chosen as the criterion for the screening. Measurements of the dielectric constant were made on those solvents with satisfactory physical properties.

A total of 45 compounds were selected on the basis of their physical state, 11 of which had dielectric constants high enough to warrant further study. The 11 selected compounds and their dielectric constants are bromine pentafluoride, 40.2; bromine trifluoride, 42.6; hydrazine, 52.9; iodine pentafluoride, 97.6; phosphorus oxychloride, 13.8; selenium monochloride, 55.0; selenium oxychloride, 46.5; thionyl chloride, 11.5; sulfuryl chloride, 10.8; arsenic trichloride, 12.5; and arsenic trifluoride, 17.5.

INTRODUCTION

Battery and fuel-cell systems presently in use are restricted in energy density by the medium in which the reaction occurs. Water is presently used as the electrolyte solvent. It prevents the use of high-energy-density electrodes, such as lithium and fluorine, since these electrodes react with water to evolve hydrogen and oxygen. In addition to being nonreactive with the electrodes, a solvent must effect the dissolution of ionic salts so that charge transport requirements can be met in an operating cell.

Much effort has been expended in search of a solvent that will provide the degree of solubility for ionic salts found in water, without undergoing decomposition. The search has centered largely on the organic compounds while the inorganic compounds (other than fused salts) as electrolytic solvents have not received much attention. It was, therefore, the intent of this investigation to evaluate certain inorganic compounds as solvents for

use in preparing electrolytes for high-energy-density cells.

Initially, compounds were chosen on the basis of being liquid at room temperature and atmospheric pressure. The dielectric constant was then taken as the criterion of selection since, in general, liquids with high dielectric constants may be expected to be better solvents for ionic substances than those with low dielectric constants. Experimental measurements were made of the dielectric constants of promising solvent materials.

METHOD OF ANALYSIS

An important requirement for a solvent is that it be capable of dissolving ionic material to such an extent that the specific resistance of the solution will be a few ohms-centimeter. The best indication of the ability of a solvent to dissolve a solute ionically is its dielectric constant. A material with a high dielectric constant is capable of separating charged ions and thus preventing significant ion pair formation.

The dielectric constant is defined by the equation

$$\epsilon = \frac{C_u}{C_{vac}} \tag{1}$$

where

C_u parallel capacitance of two parallel plates when space between them is filled with material being tested, F

C_{vac} parallel capacitance of two parallel plates when space between them is a vacuum, F

The dielectric constant ϵ of dry air at 23° C and 760 millimeters of mercury pressure is 1.00053 and for air saturated with water vapor is 1.00078. Thus, the error introduced by using the ratio of C_u/C_{air} rather than C_u/C_{vac} in equation (1) is less than 0.1 percent.

Capacitance was measured by an alternating-current bridge that allows independent variation of the frequency, voltage, and temperature. The changes in dielectric constant with frequency are produced by the dielectric polarizations that exist in the sample. At the frequencies between 10^0 and 10^6 cps (10^0 and 10^6 Hz), the dielectric constant does not vary (ref. 1). Below 10^0 cps (10^0 Hz), interfacial polarization is prominent. This polarization is caused by the interaction of the polar molecules with the electrode.

At frequencies higher than 10^6 cps (10^6 Hz), dipole polarization occurs. This polarization is related to the microscopic nature of the material and depends on the relative

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orientation of the electrons and nuclei of the permanent dipoles in the applied field. These displacements are associated with changes in rotational and vibrational motions of the electrons and nuclei upon application of an electric field. The frequency dependence thus results from the inertial characteristics of the motions and the initial state of excitation of the system.

The dielectric constant - temperature relation of nonpolar liquids is a simple function of the thermal expansion of the liquid and is approximately 0.05 percent per $^{\circ}$C.

For polar liquids, an increase of temperature causes an increase in the relaxation frequencies of a molecule, which is a measure of the time required for randomizing of the orientation of the molecule after the electric field in which it has been alined is removed. These relaxation effects arise because the permanent dipole moment of the molecules experiences a torque in an electrical field, which tends to aline the dipole axis in the direction of the field. A sudden removal of the electrical field will cause a decay of their ordered state because of the randomizing agitation of the Brownian movement. This randomizing tendency is enhanced by an increase in temperature.

The relaxation frequency increases exponentially with temperature such that a tenfold increase in relaxation frequency may be produced by a temperature increment of 6° C. A temperature control of 0.2° C is therefore required to keep the change in relaxation frequency within a 0.1 percent. The effect of increasing the temperature of a dielectric is also reflected in the conductivity. This temperature effect appears in the capacitance calculation when a series circuit is used to obtain the capacitance, and this value is then converted to the equivalent parallel circuit, as shown in the appendix.

For practical application, all dielectric polarization, except interfacial, is independent of the existing potential gradient until a voltage is reached where either ionization or breakdown occurs. Dielectric breakdown is a catastrophic failure and will be obvious. Ionization can be detected by varying the voltage and measuring the capacitance.

To obtain dielectric values with an accuracy of 0.1 percent, it is necessary, from the preceding considerations of experimental variables, to operate at a frequency where the dielectric constant does not vary (10^{0} to 10^{6} cps or 10^{0} to 10^{6} Hz); at a temperature that is controlled to within 0.2° C; and at a voltage below that required for ionization of the solvent.

APPARATUS AND PROCEDURE

The equipment for the experimental determination of dielectric constants is composed of the test cell that contains the solvent and the electrodes, and the instrumentation.

Test Cells

The cells used for capacitance measurements were of two types. The first, a Wayne Kerr permittivity cell D121 (fig. 1) using four electrodes with a neutral exterior electrode, was used for nonfluoride samples with a dielectric constant up to 5 and a loss factor up to 0.1. Electrodes 1 and 3 are neutral electrodes that serve to guide the field into the tube and reduce surface leakage effects on the tube exterior. Electrodes 2 and 4 are the measurement electrodes. Calculations for the use of this cell are given in the appendix.

The second cell was fabricated from a polytetrafluoroethylene resin and platinum (fig. 2) for use with fluoride solvents and high dielectric solvents. The electrode diagram for this cell is shown in figure 3.

The outer neutral electrode was connected to all metal parts on the cell and thus prevented the capacitance to ground from interfering with the measurements. Since no provision had been made for edge effects, the change in capacitance due to sample material had to be calculated as shown in the appendix.

Instrumentation

The capacitance of the solvents was measured with a transformer ratio-arm bridge with an accuracy of 0.1 percent (ref. 2). The measurement is dependent on the product of the current and voltage ratio for each fixed standard used. Both of these ratios are easily obtained with an accuracy of 1 part per thousand. The need for fewer fixed standards allows the range of measurements to be increased above those found in substitution-arm bridges. The increased range of conductance at a fixed capacitance makes it possible to determine the capacitance of a material with a large loss factor. It is possible, by using the third lead, to measure a capacitance of 0.0002 picofarad at the end of long leads having thousands of times the capacitance of the sample and thus to be able to make measurements in remote areas, such as in a constant-temperature bath or a dry box.

By proper design of the test cell and the use of guard electrodes 1 and 3 (fig. 1) and the neutral electrode (fig. 3), stray capacitance associated with the unknown can be completely counteracted or calculated, as shown in the appendix. The use of the third electrode does not impair the sensitivity of the measurement.

The sign of a voltage or current can be reversed to allow measurements in all four quadrants of the complex plane and thus to allow materials with semiconductor or electrochemical-pseudoinductance (ref. 3) to be measured.

The measurement of capacitance and resistance is accomplished with a parallel

circuit, which makes it possible to determine the dielectric constant of a material with a high conductivity or loss factor. By definition the dielectric constant is the ratio of parallel capacitances. Measurements obtained by the use of a series circuit have to be converted to a parallel value, as shown in the appendix. It is necessary to limit the loss factor to 0.1 in series measurements to keep the error in converting series capacitance to less than 1 percent.

Auxiliary equipment allows the bridge to be used at frequencies from 10 to 20 kcps (10 to 20 kHz). The bridge and associated equipment needed for capacitance measurements are shown in figure 4.

The capacitance of the assembled cell was measured with ambient air between the electrodes and then transferred to a dry box that contained a dry-nitrogen atmosphere. A sample of solvent was then added to the cell through the filling port and the cell was sealed. It was removed from the dry box, placed in a constant temperature air chamber, and the capacitance of the cell was again obtained. The conditions of measurement for the cell with the air and sample were (1) temperature, $25^{\circ} \pm 0.1^{\circ}$ C; (2) potential applied to the electrodes, 640 millivolts; and (3) frequency, 19 kcps (19 kHz). The dielectric constant was calculated, as shown in the appendix.

RESULTS AND DISCUSSION

The compounds considered for evaluation and their dielectric constants are listed in table I. With the exception of antimony pentachloride and thionylchloride, the available dielectric values reported in the literature (also listed in table I) agree with the values obtained experimentally. No explanation can be extended for discrepancies in the two exceptions. The experiment was repeated after purifying the material by distillation, and the dielectric value did not differ by more than the experimental error from the original measurement.

The correlation between the dielectric constants of bromine pentafluoride, bromine trifluoride, and iodine pentafluoride is again ascribed to differences in purity. Bromine pentafluoride (ref. 4) and iodine pentafluoride (ref. 5) were studied spectroscopically and are both tetragonal pyramids with four fluorine atoms in a square planar configuration about the iodine or bromine atom and the fifth fluorine atom on an axis normal to the plane. It would be expected that these two compounds would then possess similar polarizations. The higher dielectric constant of bromine trifluoride relative to that of bromine pentafluoride would indicate a possible covalent compound of $BrF_3 \cdot F_2$. This would be substantiated by the higher vapor pressure found for the sample of BrF_5 investigated (1 atm at 0° C).

A correlation appears to exist between the dielectric constant and the type of halide

atom attached to the molecule. The dielectric constants measured followed the order $F > Cl > Br > I$. This order is also shown in the literature for organic compounds (ref. 6) as given in the following table:

Compound	Dielectric constant, ϵ
Fluorobenzene	6.373
Chlorobenzene	5.641
Bromobenzene	5.391
Iodobenzene	4.486

The number of dielectric constants available for oxysalts is not sufficient to allow an analysis of this classification. Four such compounds were studied: selenium oxychloride, phosphorus oxychloride, vanadium oxytrichloride, and chromium oxychloride. Of these, the transition elements, chromium and vanadium showed low dielectric constants, 2.6 and 2.9, respectively. The dielectric constants for selenium and phosphorus oxychloride are 46.5 and 13.8, respectively.

All the halogen halides showed high dielectric constants, with the exception of bromine pentafluoride, the abnormality of which was discussed previously.

The effect of temperature on the dielectric constant was less than the experimental error of the equipment used. The temperature coefficient of the materials listed in reference 7 shows a negative change in the dielectric constant of less than 1×10^{-2} for a 1° C change in temperature, that is, $-dE/dT < 1 \times 10^{-2}$.

Compounds for which the dielectric constants were not determined included iodine monochloride, selenium tetrafluoride, and selenium oxyfluoride.

The dielectric constant for iodine monochloride was not obtained because the conductivity was so high that no reliable capacitance data could be obtained. The dissociation constant for $2ICl = I_2 + Cl_2$ is 1.6×10^{-10} (ref. 8). This value does not agree with the high conductivity obtained with this compound and reported (ref. 9) as 4.4×10^{-3} ohm^{-1} centimeter^{-1} since pure IBr has a conductivity of 5×10^{-6} ohm^{-1} centimeter^{-1} at 55° C (ref. 9). The literature shows that the dissociation constant for the reaction $ICl + Cl^- = ICl_2^-$ is 1.7×10^2 (ref. 8). Therefore, the presence of the chloride ion as a difficultly removed impurity in the ICl would lead to the production of the ICl_2^- ion. This is a possible explanation for the conductivity observed. This compound has been studied as a solvent by Gutman (ref. 9).

Selenium tetrafluoride and selenium oxyfluoride were not investigated because of the prohibitive price of the materials.

CONCLUSIONS

The results of dielectric determinations show the possibility of using some of the investigated materials as solvents for electrolytes. The following are the best potential solvents of those evaluated and have dielectric constants sufficiently high to expect that soluble ionic salts would be ionized: Bromine pentafluoride, bromine trifluoride, hydrazine, iodine pentafluoride, phosphorus oxychloride, selenium monochloride, selenium oxychloride, thionyl chloride, sulfuryl chloride, arsenic trichloride, and arsenic trifluoride.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, April 20, 1967,
120-34-01-09-22.

APPENDIX - CALCULATIONS AND MATHEMATICAL

CONSIDERATIONS OF DIELECTRIC CONSTANTS

Cell Type 1 - Wayne Kerr Cell

The cell constant for cell type 1, the Wayne Kerr cell, was first obtained through the use of a standard fluid with a known dielectric constant. The dielectric constant of the standard liquid is given by the formula

$$\epsilon_m = \frac{\dfrac{C_{Hg}}{C_a} - 1}{\dfrac{C_{Hg}}{C_s} - 1} \tag{A1}$$

where

ϵ_m measured dielectric constant

C_{Hg} measured capacitance of tube filled with mercury, F

C_a measured capacitance of tube filled with air, F

C_s measured capacitance of tube filled with standard liquid, F

The difference between ϵ_m obtained from equation (A1) and the true value, expressed as a percentage, is the cell constant.

When a material that has an appreciable loss factor (0.1) is measured the measured capacitance for the substance must be converted into the series form and the capacitance of the glass wall subtracted. The corrected value must then be converted back to the parallel form (as shown in eq. (A9)) before calculating the dielectric constant.

Cell Type 2 - Polytetrafluoroethylene-Platinum Cell

The dielectric determination of cell type 2, the polytetrafluoroethylene-platinum cell, is obtainable by two methods, direct calculation and the use of standard liquids.

Direct calculation. - The cell has provisions for eliminating capacitance to ground but not edge capacitance. The edge effect can be calculated from cell geometry by the equation

$$C_e = (0.019\ K_s' - 0.058\ \log T + 0.010\ \text{cm}) \times 2(d_1 + T) \tag{A2}$$

where

C_e edge effect

K_s' approximate value of sample dielectric constant

T   space between electrodes, cm

d_1 diameter of center electrode, cm

When the values for the cell used are substituted, equation (A2) becomes

$$C_e = 0.259\ K_s' + 0.587 \tag{A3}$$

These equations assume an idealized cell with smooth concentric cylinders of equal height and with coplanar ends. The capacitance of the cell can be calculated for the case in which the space between the electrodes contains a vacuum:

$$C_{vac} = \frac{0.24160 \times L}{\log \dfrac{d_2}{d_1}} \tag{A4}$$

where

L   length of electrodes, cm

d_2 inner diameter of outer electrode, cm

d_1 outer diameter of inner electrode, cm

When the dimensions of the cell are substituted, a capacitance of 6.162 picofarads is obtained. The capacitance of the cell and the edge capacitance are in parallel. The edge capacitance of the cell containing a vacuum between the electrodes with a K_s' of 1 is 0.846. The theoretical capacitance of this cell containing a vacuum between the electrodes is therefore 7.01 picofarads. In practice, it was found that the capacitance of air is 6.74 picofarads, which constitutes an error of 3.85 percent, which is in good agreement with the calculation of an idealized system. Correcting equation (A3) for nonideal conditions produced the equation

$$C_e \cong 0.249\ K_s + 0.565 \tag{A5}$$

Figure 6 is a plot of the edge effect capacitance as a function of the dielectric constant.

The sample calculation of the dielectric constant of 1, 2-dichloroethane is made as follows:

$$\frac{C_u}{C_{air}} = \frac{63.92 \text{ pF}}{6.74 \text{ pF}} = 9.48$$

$$C_{air} = 6.74$$

$$C_{1, 2\text{-dichloroethane}} = 63.92$$

$$C_e \text{ due to } K_s' \text{ of } 9.48 = 2.92$$

$$C_e \text{ due to } K_s' \text{ of air} = 0.81$$

$$\frac{C_u - C_{u,e}}{C_{air} - C_{air,e}} = \frac{63.92 - 2.92}{6.74 - 0.81} = 10.31$$

The dielectric constant of 1, 2-dichloroethane is reported as 10.36; therefore, the experimental value is in error by 0.5 percent, which is within experimental error.

Cell constant using standard solvent. - It is also possible to use a cell constant obtained by running a sample with a known dielectric constant. The equation is

$$C_c = \frac{E_K C_{vac} - C_K}{E_K - 1} \tag{A6}$$

where

C_c cell constant

E_K dielectric constant of standard solvent

C_{vac} measured vacuum capacitance

C_K measured capacitance when cell is filled with liquid of dielectric constant E_K

The dielectric constant E_X of an unknown material is obtained by the use of the equation

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$$E_X = \frac{C_X - C_c}{C_{air} - C_K} \tag{A7}$$

The sample calculation of the dielectric constant of selenium monobromide is made by substituting the following values into equation (A6):

Capacitance of filled cell, pF

Selenium monobromide 39.8

1,2-dichloroethane 62.4

Air 6.74

Dielectric constant of 1,2-dichloroethane 10.36

Substitution of these values in equation (A6) gives the cell constant:

$$C_e = \frac{(10.36)(6.74) - (62.4)}{10.36 - 1}$$

$$= 0.79 \text{ picofarad}$$

The capacitance of the unknown of air and the cell constant is substituted into equation (A7) to yield the dielectric constant of the unknown:

$$E_X = \frac{39.8 - 0.79}{6.74 - 0.79} = \frac{39.0}{5.95} = 6.56$$

The edge capacitance is dependent on the dielectric constant of the sample, and, therefore a standard material close to the unknown should be used in calculating a cell constant. To illustrate this, the dielectric constant of isopropyl alcohol was determined from the direct calculation and standard solvent methods with the use of 1,2-dichloroethane as the standard.

Mathematic Comparison of Direct and Standard Solvent Methods

Direct calculation. - The calculation of the dielectric constant of isopropyl alcohol with the use of the direct method is as follows:

Capacitance of unknown = 113.1 pF

Capacitance of air = 6.7 pF

C_e due to K_s' of 16.9 = 4.8

$$E_X = \frac{113.1 - 4.8}{6.7 - 0.8} = 18.4$$

Standard solvent method. - The calculation of the dielectric constant of isopropyl alcohol with the standard solvent method is as follows:

Cell constant = 0.8 (from direct calculation)

$$E_X = \frac{113.1 - 0.8}{6.7 - 0.8} = 19.0$$

The dielectric constant of isopropyl alcohol is 18.3, an error of 0.5 percent, which is in agreement with our system (for the direct calculation), and an error of 3.8 percent for the standard solvent method using 1,2-dichloroethane as the standard.

Dissipation Factor

The dissipation factor of an insulating material is the ratio of the conductance A of a capacitor in which the material is the dielectric to its susceptance B or is the ratio of its parallel reactance X_p to its parallel resistance R_p and is represented by the symbol D:

$$D = \frac{1}{R_p w C_p} = w R_s C_s \tag{A8}$$

where w is 2π times the frequency in cycles per second (Hz).

A high value of w is preferable, but as shown earlier, a frequency above 10^6 cps (10^6 Hz) produces dipole polarization. For the best case, where $w = 2\pi 10^6$ and $C_p = 100$ picofarads, R_p has to have a minimum value of 15.9×10^3 ohms for the dissipation factor to be 0.1. For the worst case, where $w = 2\pi 10^6$ and $C_p = 1$ picofarad, R_p has to have a minimum value of 15.9×10^5 ohms for the dissipation factor to be 0.1.

The relation between series and parallel capacitance is

$$C_p = \frac{C_s}{1 + D^2} \qquad \text{(A9)}$$

The series capacity is larger than the parallel capacity by less than 1 percent for a dissipation factor of 0.1 and by less than 0.1 percent for a dissipation factor of 0.03 (ref. 1).

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TABLE I. - DIELECTRIC CONSTANTS OF INORGANIC LIQUIDS

Compound	Dielectric constant, ϵ		
	Experimental	Ref. 7	Ref. 10
Antimony pentafluoride	6.5	-----	----
Antimony pentachloride	5.0	3.22	----
Arsenic trifluoride	17.5	-----	----
Arsenic trichloride	12.5	-----	----
Arsenic tribromide	9.3	-----	----
Boron tribromide	2.6	2.58	----
Bromine pentafluoride	40.2	-----	----
Bromine trifluoride	42.6	-----	----
Chromium oxychloride	2.6	-----	----
Germanium tetrachloride	2.1	-----	----
Hydrazine	52.9	52.9	51.7
Iodine pentafluoride	97.6	-----	----
Iodine monochloride	----	-----	----
Iron pentacarbonyl	2.8	-----	----
Lead tetrachloride	2.8	-----	----
Manganese heptaoxide	3.3	-----	----
Molybdenum hexafluoride	2.9	-----	----
Phosphorus trichloride	3.4	3.43	----
Phosphorus tribromide	3.9	-----	----
Phosphorus oxychloride	13.8	13.3	13.9
Rhenium hexafluoride	2.9	-----	----
Selenium tetrafluoride	----	-----	----
Selenium monochloride	55.0	-----	----
Selenium monobromide	6.4	-----	----
Selenium oxyfluoride	----	-----	----
Selenium oxychloride	46.5	-----	----
Silicon tetrabromide	2.6	-----	----
Silicon tetrachloride	2.4	2.4	----
Sulfur monobromide	4.0	-----	----
Sulfur monochloride	7.5	-----	----
Sulfur dichloride	4.6	-----	----
Sulfuryl bromide	9.1	-----	----
Sulfuryl chloride	10.8	10.0	----
Stannic tetrachloride	3.2	2.8	----
Stannic dichloride dibromide	6.9	-----	----
Stannic dichloride diiodide	6.9	-----	----
Thallium tetrachloride	2.1	-----	----
Thionyl chloride	11.5	9.25	----
Thiophosphoryl dibromomonochloride	6.4	-----	----
Thiophosphoryl chloride	5.8	-----	----
Titanium tetrachloride	2.7	2.80	----
Vanadium tetrachloride	3.1	-----	----
Vanadium oxytrichloride	2.9	-----	----
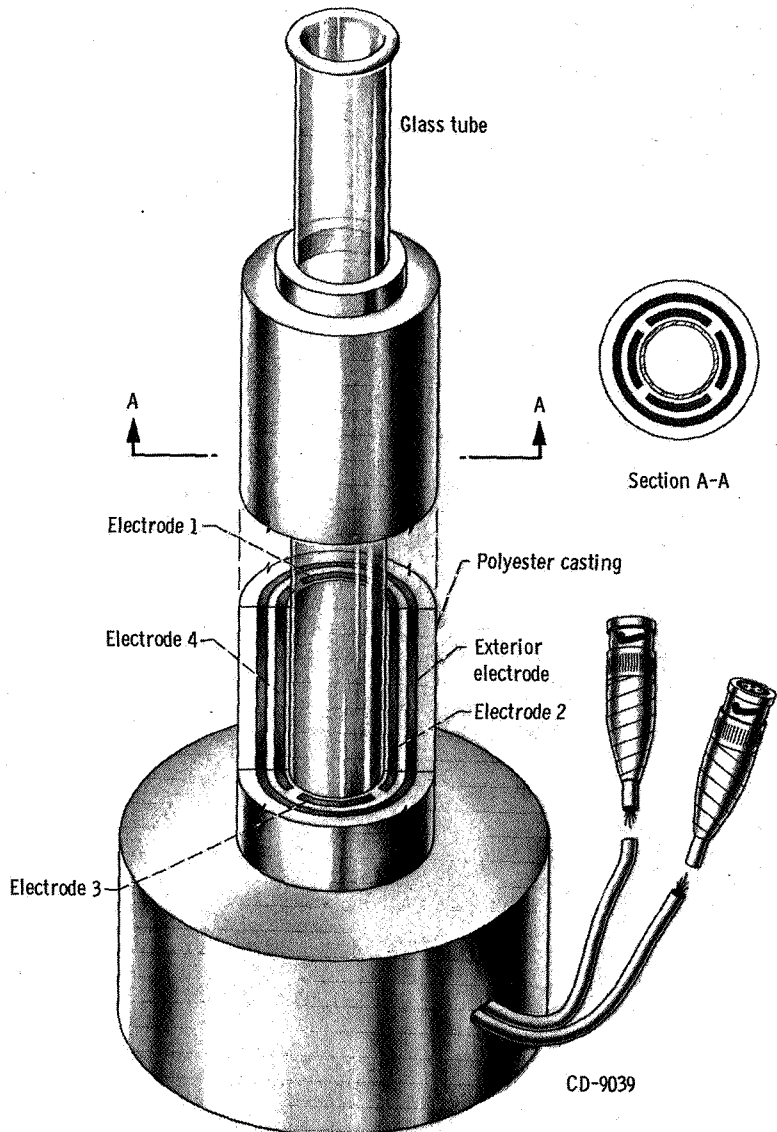
Figure 1. - Wayne Kerr permittivity cell.

Glass tube

Section A-A

Electrode 1

Polyester casting

Electrode 4

Exterior electrode

Electrode 2

Electrode 3

CD-9039
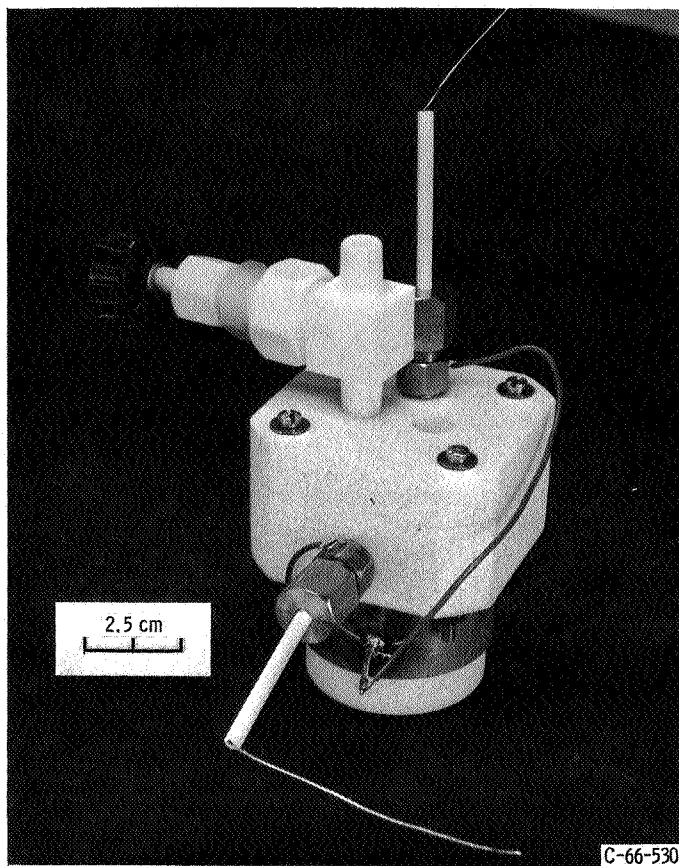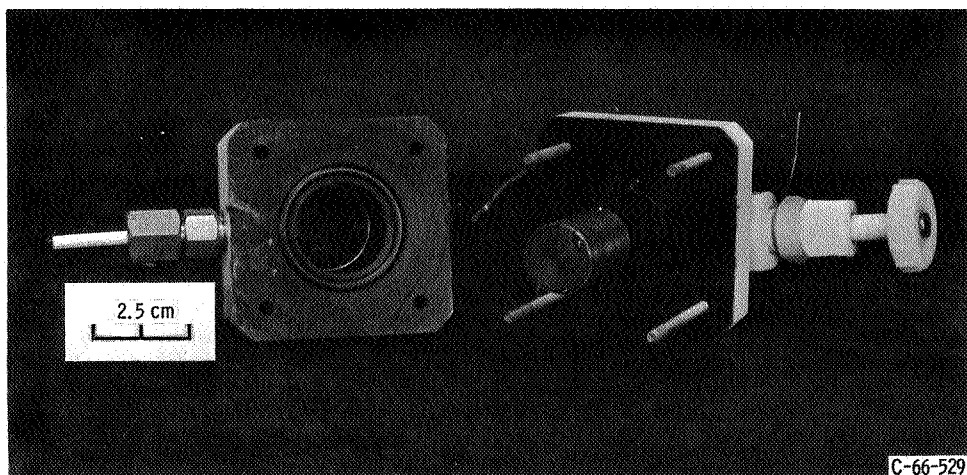
(a) Assembled.

(b) Disassembled.

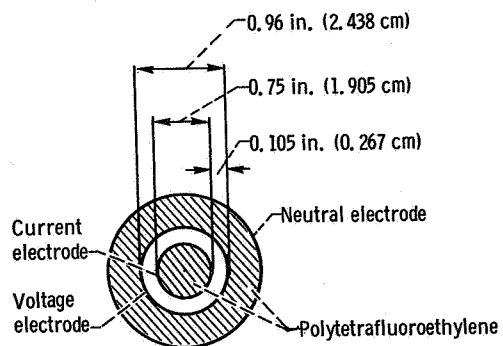Figure 2. – Test cell fabricated from polytetrafluoroethylene resin and platinum.

Figure 3. - Diagram of electrode for polytetrafluoroethylene-platinum cell.

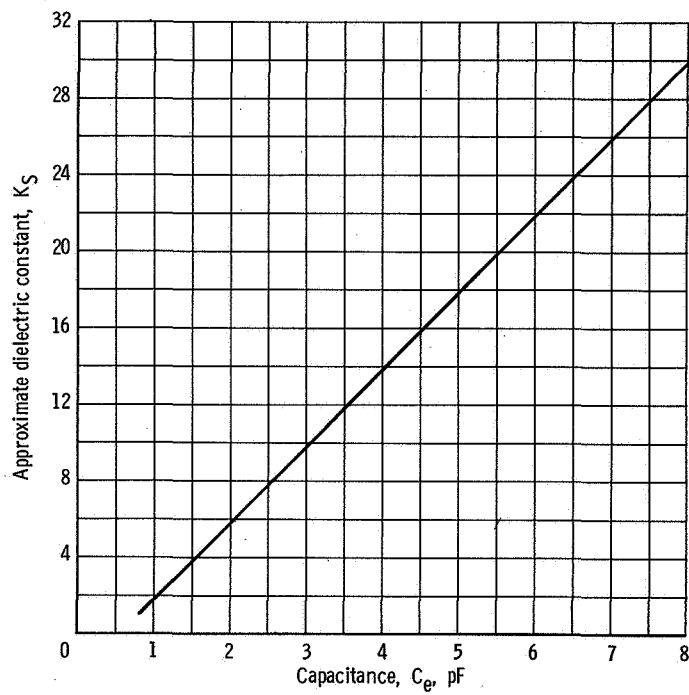Figure 4. - Bridge and associated equipment used for capacitance measurements.

Figure 5. – Edge effect as function of dielectric constant.