THE MEASUREMENT OF DISSIPATION FACTOR, DIELECTRIC CONSTANT AND RESISTIVITY OF LIQUIDS

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INTRODUCTION

Manufacturers and users of dielectric fluids must have adequate methods for measuring the quality of these liquids. Dissipation factor or power factor and DC resistivity of the fluids are measures of the electrical losses which a given fluid will permit when used as insulation. For this reason, it is common practice to measure these properties to determine whether or not an insulating liquid is suitable for use in a capacitor, transformer, cable or other electrical apparatus. Dielectric constant,which is a measure of the ability of a material to store electrical energy when used as the dielectric of a capacitor, is also an important electrical property of an insulating fluid.

Measurements of these properties are useful not only as sales and acceptance specifications, they are also valuable measures of the deterioration of a fluid during use. In addition, they serve to determine the effect of other materials of construction used in the equipment on the quality of the fluid insulation and to determine the effect of additives used to prevent degradation in use. They can be used to control the quality of in-process materials and to guide process development work.

It is therefore important that reliable methods for the measurement of these properties be available. The ASTM has recognized the importance of

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such methods and covers them in methods D924 and D1169. In spite of the existence of these methods, measurements of dissipation factor and resistivity often give highly erratic results, particularly with highly purified fluids and fluids of higher dielectric constant such as askarels. A given worker sometimes finds that successive dissipation factor and resistivity measurements on the same sample differ by a factor of five or more. Different laboratories often disagree by even larger factors. In an attempt to improve this situation, we have tried to learn the causes of some of the discrepancies and to eliminate them so that more reliable measurements of dissipation factor or power factor and resistivity could be made.

REQUIREMENTS FOR ACCURATE MEASUREMENTS

The requirements for making accurate measurements of the dissipation factor, dielectric constant and resistivity of dielectric fluids are the following:

First, one must have an adequate A.C. bridge for measuring dissipation factor and capacitance as well as a D.C. bridge for measuring resistance. Fortunately, there are a number of excellent ones on the market. For A.C. measurements, the modern transformer ratio arm capacitance bridge has many advantages. It permits making three terminal measurements without balancing a guard circuit. This means that one can make three terminal measurements more easily than two terminal measurements can be made with a standard resistance arm bridge. This is because one direct measurement without a separate measurement of lead capacitance sufficies to measure the capacitance of the sample volume of the cell. Moreover, the effects of stray capacitance variations

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are eliminated. We have found that the Electro Scientific Industries Type 701 Capacitance Measuring Assembly is extremely convenient for our work. It is a 0.01% accuracy bridge capable of measuring dissipation factors in the neighborhood of 0.0001.

The General Radio Type 1644A Megohm Bridge gives a 1% resistance accuracy up to 10^{12} ohms after self-calibration, decreasing to 10% at 10^{14} ohms. Figure 1 is a photograph of the two bridges.

The precision and accuracy of these bridges can b e demonstrated against capacitance, dissipation factor and resistance standards. The poor accuracy of measurements on insulating fluids is not caused by the electrical measuring equipment itself.

Next, one must have suitable measuring cells. They must be mechanically stable, chemically resistant, have essentially zero power factor, and be easily cleaned. We have found that Balsbaugh Type 350G cells modified to our specifications are quite satisfactory. We have them made of 316 SS instead of monel, have the jack on the outer electrode mounted vertically, and have the screwed-on name plate omitted. Figure 2 is a photograph of two of these cells and the cell vessels in which they are used. One is shown in the vessel with leads attached and lid and support clamp for the vessel in place.

In addition, the sample must be maintained at a known constant temperature while protected from the effects of light, temporary overheating and other influences which may cause degradation. For this purpose, we use a covered glycerine thermostat controlled at $100.0 \pm 0.01^{\circ}C$. Glycerine is used as the bath fluid because it can be readily washed off the cell vessels with water.

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A liquid filled thermostat permits bringing the sample to measuring temperature quickly and reproducibly. The thermostat is shown in Figure 3.

Another important requirement is that the composition of the sample be representative. Also, it must not change sufficiently during the measurement to affect the result of the measurement. In the case of a dielectric constant measurement this is not too difficult because relatively large amounts of impurities produce a comparatively small change in dielectric constant. One one-hundredth of a percent (1 part in 10^4) of water which has a dielectric constant of 80 added to a sample of dielectric fluid having a dielectric constant of five would produce a change of only a little over one-tenth percent in the dielectric constant. Contaminants with lower dielectric constants than that of water would produce proportionately less effect.

The trouble comes when we make dissipation factor and resistivity measurements. As little as 0.1 part per billion (1 part in 10^{10}) of some impurities can increase the dissipation factor of a sample of askarel by 0.02. This is intolerable if the sample has a dissipation factor which is of the same order of magnitude or smaller. Any soluble ionizable impurity has an effect on dissipation factor and resistivity far beyond what most people would think possible. Colloidal impurities can also affect these properties but to a much smaller degree. Their effect is less because the mobility of colloidal particals is less than that of ions. It is therefore necessary to use techniques of sample handling which will prevent contamination of the sample by minute traces of impurity during storage, transfer and measurement. Sample containers must be carefully cleaned and dried. They should be of

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amber glass with Teflon or aluminum foil cap liners and kept in a dark place. Table 1 shows the effect of storage of samples exposed to different light intensities. The data demonstrate that protection from light during storage is essential and that properly stored samples do not change with time. Samples for measurement should be withdrawn from the bottles by means of a carefully cleaned pipette rather than by pouring. The measuring electrodes and the glass vessel for the cell must be cleaned with special care. An outline of the cleaning procedure used in our laboratories is as follows:

1. Xylene vapor clean 15 minutes.
2. Clean in boiling 29% NH_4OH 10 minutes.
3. Steam clean for 30 minutes.
4. Acetone vapor clean 15 minutes.
5. Dry in 100°C. oven 30 minutes.

Figure 4 shows the vapor cleaning baths for the cells and Figure 5 those for the cell vessels. Neither the electrodes nor the electrode vessel are touched by the operator's hands during the cleaning process. Details of the equipment required, the cleaning method and the measurement are given in an operating procedure available on request.

The measurement is carried out as follows: Pipette a sufficient amount of fluid to come above the cell guard electrode into the cell vessel and place the cell in it. Attach the electrical leads and supporting clamp and place the cell vessel in the 100.0°C. thermostat. Connect the leads to the capacitance bridge. After 15 minutes balance the bridge and record the dissipation factor and capacitance. Make a second measurement 30 minutes after placing the

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cell in the bath. The 30 minutes values are reported. The ratio of the sample filled cell capacitance to the cell air capacitance gives the dielectric constant. Comparison of the 15 and 30 minute capacitance readings serves to make certain that the sample has reached temperature equilibrium. Comparison of the 15 and 30 minute dissipation factor values gives additional information. If the dissipation factor increases, it is likely that the cell or vessel have not been properly cleaned. An increase might possibly indicate that enough light is reaching the cell to cause photo-dissociation of askarels or that the sample is being oxidized to form ionizable impurities.

Decreasing dissipation factors indicate loss of some material from the sample. Dissipation factor of a dry askarel sample decreases much less than that of a wet one. The reason is that water is lost during measurement at 100°C. The water loss can be confirmed by Karl Fisher titration. The loss is probably due to vaporization of the water since samples measured at 40°C. show very little decrease in dissipation factor or loss of water with time.

The D.C. resistivity measurement is made after the A.C. measurements. Five hundred volts are applied to the cell and the resistance is measured one minute after application. The cell constant is obtained by a direct measurement of the air capacitance of the cell and multiplying by the theoretical factor, 11.3. In making resistivity measurements, strict adherence to the voltage and time specification is necessary since the measured value always increases very rapidly with increasing time and voltage and it is not practical, nor would it be meaningful, to wait to measure an equilibrium value. The increase can be attributed to electrochemical and electrophoretic processes

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which cause a decrease in the concentration of charge carrying ionic or colloidal impurities. This process can be used to prepare materials of very high resistivity.

DISCUSSION

The above methods are an extension of the ASTM methods. They point out the most modern and convenient to use equipment. Most important of all, they emphasize the need to avoid changes in sample composition and the techniques for sample handling and cell cleaning which make this possible.

It should be obvious from what has been said above that there are many causes for poor agreement among dissipation factor and resistivity measurements. However, most or perhaps all of them can be said to be caused by variation in composition of the sample, either prior to or during the measurement. It is an unusually difficult task to make a meaningful measurement when what we are measuring is influenced by variation of the amount of components present at the 0.1 part per billion level. However, the techniques described in the detailed procedure referred to previously permit an order of magnitude improvement in such measurements. Table 2 shows the reproducibility of the data obtained.

On the basis of what is now known, it would seem logical to abandon the measurement of D.C. resistivity of dielectric fluids to indicate their quality. There are many reasons for doing so. The main one is that the resistivity of a sample is constantly changing during the measurement due to electrical clean up of charge carrying impurities. Therefore, no "correct" value can

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be measured. The value obtained is a purely empirical one. It is not desirable to use such a value unless no better one is available. Another disadvantage is that as the fluid becomes purer, the resistivity tends toward infinity. This misleads many who use resistivity data into thinking that there are very large differences between samples when in fact only minute traces of perhaps 0.01 part per billion of impurities cause the difference.

Now that dissipation factor measurements can be made as conveniently or more conveniently than D.C. resistivity measurements, it would seem more desirable to use dissipation factor measurements to determine the quality of dielectric fluids. One reason for this is that such measurements are not sensitive to the voltage applied to the cell during the measurement or to the length of time voltage is applied. We have made measurements with a bridge which applies about 20 volts to the cell and with another which imposes 1000 volts on the cell with no significant difference in the values obtained. Please note that this statement applies only to the measurement of liquid samples. The values obtained, at least with a low voltage bridge, are independent of whether the voltage is applied to the cell continuously or only while an actual measurement is being made. At frequencies of the order of 60 Hz, dissipation factor is essentially a measure of the conductivity of the fluid. Conductivity in very dilute solutions is directly portional to the concentration of charge carrying impurities so that it tends towards zero as the impurity concentration approaches zero. The changes in dissipation factor as purity is increased, therefore, do not tend to over emphasize the difference among samples as resistivity values do. Therefore, dissipation

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factor would appear to be a more desirable measurement which should supplant D.C. resistivity as a measure of dielectric fluid quality.

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DATA SUMMARY

SHOWING THE EFFECT OF LIGHT ON THE DISSIPATION FACTOR

Aroclor 1242, Anniston Spl. #5 (8-25-67)

	Spl. Stored in the Dark After Splitting (10-30-67) (Standard Method)		Spl. Stored in an Amber Bottle in the Light (10-30-67)		Spl. Stored in a Clear Bottle in the Light (11-1-67)	
	11-1-67	2-8-68	11-1-67	2-8-68	11-2-67	2-8-68
Dissipation Factor at 15 Mins.	.00144	.00186	.00150	.00336	.00420	.02658
30 Mins.	.00150	.00156	.00156	.00318	.00318	.02514
60 Mins.	.00150	.00120	.00132	.00288	.00204	.02286
90 Mins.	.00138	.00114	.00126	.00204	.00162	.02190
Resistivity (500 Volts) after 90 Min.	$55,392 \times 10^9$	$15,409 \times 10^{10}$	$73,856 \times 10^9$	$4,431 \times 10^{10}$	$57,783 \times 10^9$	$1,610 \times 10^9$

TABLE 1

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REPRODUCIBILITY DATA

Aroclor 1242, Anniston Spl. #4 (8-19-68)
(Page OR 117182) (10-1-68)

Test No.	15 Mins.	Deviation From Mean	30 Mins.	Deviation From Mean	(30 Mins.) Resistivity	Deviation from Mean
974	.00030	.000015	.00030	.00003	213×10^{12}	5×10^{12}
975	.00024	.000075	.00018	.00009	192×10^{12}	4×10^{12}
979	.00036	.000045	.00030	.00003	194×10^{12}	6×10^{12}
980	.00036	.000045	.00030	.00003	153×10^{12}	35×10^{12}
Mean	.000315		.00027		188×10^{12}	
Mean Deviation		.000045		.000045		17.5×10^{12}

Aroclor 1242, Lot KH-456 (3-15-68)
(Page OR 117186) (10-2-68)

Test No.	15 Mins.	Deviation From Mean	30 Mins.	Deviation From Mean	(30 Mins.) Resistivity	Deviation From Mean
984	.01014	.00054	.00852	.00008	15×10^{12}	4×10^{12}
986	.01212	.00146	.00870	.00026	11×10^{12}	0
990	.00942	.00126	.00828	.00016	12×10^{12}	1×10^{12}
991	.01104	.00036	.00828	.00016	6×10^{12}	5×10^{12}
Mean	.01068		.00844		11×10^{12}	
Mean Deviation		.00090		.00016		2.5×10^{12}

TABLE 2

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