

DSW 330602

STLCOPCB4077246

PROCEDURE FOR MEASURING DISSIPATION FACTOR OR
POWER FACTOR, DIELECTRIC CONSTANT AND RESISTIVITY OF ASKARELS

INTRODUCTION

This procedure covers the measurement of dissipation factor, dielectric constant or permittivity and resistivity of electrical insulating fluids. The dissipation factor and the power factor of a fluid differ by less than one percent of the numerical value when the dissipation factor is less than 0.14. Since good insulating fluids have dissipation factors much below this, their dissipation factors and power factors can be considered equal for ordinary purposes. In some cases, power factor is given in percent. Care must be used in comparing values to be sure that all are given as fractions or in percent. Calculations given in this procedure give the fractional value of the dissipation factor. To convert these values to percent, multiply by 100.

Precautions: In the early development of this procedure Aroclor 1242 was found to be light sensitive. Light from sources such as fluorescent lamps, mercury vapor lamps, and incandescent light bulbs caused a marked increase in dissipation factor and a decrease in resistivity. For this reason all dissipation factor and resistivity measurements should be made in subdued light.

When measuring fluids having low dissipation factor values such as those found in Askarel fluids, it is extremely important that the utmost care be taken to prevent contamination of the sample. Minute quantities of contamination introduced into the sample either from the test cell, glassware, or in other ways will cause extreme differences in dissipation factor values. As little as 0.1 part per billion of some impurities can be detected. Therefore, the test

DSW 330603

cells must be handled only with the cell holders or tongs. No part of the test cell or glassware which might contact the sample should be touched with hands or any support. Strict adherence to the cleaning and sample preparation sections of the procedure is a must. Dissipation factor measurements have been made using test cells that were cleaned and stored for 16 hours in the drying oven with satisfactory results; however, longer storage periods give increased dissipation factor values.

The cells are ordinarily cleaned without disassembling. However, if coatings or impurities remain after the standard cleaning procedure the cells can be taken apart and cleaned with soap and water on a soft cloth or bristle brush. This should not be done unless several passes through the standard cleaning procedure fail to get the cell clean enough.

*To take the cell apart for cleaning, first unscrew the 874 connector shell and pin from the top. Then unscrew the outer electrode from the body of the cell. Finally, unscrew the knurled nut from the center electrode and push it gently out of the body. No attempt to further disassemble the cell should be made. To reassemble, reverse the above procedure.

After cleaning the cell parts with soap and water, it should be reassembled, rinsed with distilled water and reagent grade acetone, then put through the standard cleaning procedure until it gives the correct value when used to measure a known good sample.

Caution should be exercised when making resistance measurements on the Type 1644A Megohm Bridge. Never touch the cell or leads from the test cell to the bridge when in the charge zero or measure position. 500 volts

is applied to the cell in either position. Always return the switch to the discharge position after completing the resistance measurement before attempting to remove the leads from the bridge to the test cell. The red light on the upper right corner of the panel is on when the switch is in either position. However, it is important to look at the switch itself; the pilot light could be burned out.

APPARATUS

1. Electro Scientific Industries Capacitance Measuring System Model 701.
2. General Radio Type 1644A Megohm Bridge.
3. 110-24 Volt Step Down Transformer, Selenium Rectifier type RT201, Chicago Standard Transformer Corp., Chicago, Ill., with plug in adaptor unit for 60 cycle measurements.
4. Special Plug in Connector for measurements above 60 cycles
5. Two General Radio Type 874-Q9 or 777-Q3 adaptors.
6. General Radio Decade Resistor Type 1433-L or box made using one General Radio Type 510D and one 510E decade resistor in series in a suitable metal box.
7. General Radio Type 274 NQS Patch Cord.
8. Coaxial Cable lead (General Radio Type 874 Patch Cord Cat. No. R22A).
9. Patch cord with banana plug terminals such as H. H. Smith Type 1863-36-102, Allied Radio Stock No. 47D1904.
10. Balsaugh Test Cells (Stainless Steel) Type 350G modified according to Monsanto specifications.

DSW 330605

11. Constant Temperature Bath, a 12" x 12" Pyrex jar, insulated.
 - a. Constant Temperature of 100°C. is maintained with a Fisher Proportional Controller using a thermister probe as a temperature sensing device.
 - b. A 125 watt Cenco knife blade heater is used for intermittent heating controlled by the Fisher Proportional Controller.
 - c. A 450 watt Cenco knife blade heater is used as a continuous heater controlled by a variac to supply enough heat so that the intermittent heater is on about half the time.
 - d. A Bodine stirring motor with attached stainless steel propeller is used to maintain good agitation of bath liquid. The propeller is surrounded by a draft tube. It should draw the bath fluid up through the draft tube.
 - e. Glycerine is used for the bath medium with the liquid level maintained to within 3/4 inch from the top edge of the bath cover.
 - f. Standardized Centigrade thermometer for temperature read out (Princo Q-7, range 75°-110°C.)
 - g. Bath cover constructed from Extren 500 manufactured by J. T. Ryerson & Son, Inc., 2558 West 16th St., Chicago, Ill. 60608.
12. Drying Oven (Fisher Isotemp Junior Model 202)
13. Xylene Vapor Bath containing calcium oxide lumps (Test Cell Cleaning).

DSW 330606

14. Boiling 29% NH_4OH Bath (Test Cell Cleaning). A 1.5 liter beaker.
15. Distilled H_2O steam bath (Test Cell Cleaning).
16. Acetone Vapor Bath (Test Cell Cleaning).
17. Xylene Vapor Bath (Glassware Cleaning).
18. Distilled H_2O Steamer (Glassware Cleaning).
19. Sample Vessel (Glass).
20. Teflon Lid for Sample Vessel.
21. Supporting Clamp (Sample Vessel).
22. Special Wrench for removing center pin connection from test cell.
23. 50 ml. Pyrex Pipette (Taper Tip cut off to allow faster sample delivery).
24. Fisher Thermix Hot Plate Cat. No. 11-493 (Acetone Vapor Bath).
25. Teflon Covered Stirring Bar (For Acetone Vapor Bath).
26. Laboratory Tongs (Fisher Cat. No. 15-202).

Drawings of the items of apparatus which cannot be obtained from laboratory supply companies are attached. Numbers on drawings correspond to numbers on apparatus list.

ELECTRICAL CONNECTIONS

1. Electro Scientific Industries Capacitance Measuring System, Model 701.
 - a. With the power switch off, plug the power cord of the Model 701 into a standard 110 volt outlet. This will supply power for the bridge null detector and internal power supply.

- b. Remove the plug-in shield covering the generator output and bridge input terminals and the links which connect them. To make measurements at 60 Hz, connect the 12 volt terminals of the 110-24 volt transformer to the terminals marked GEN 1 and 2 on the bridge through a switch. A GE Type 47 pilot lamp connected in series with a 100 ohm resistance across the 24 volt leads serves to indicate when the bridge power is on. The switch and pilot lamp with its resistor are mounted in a small metal shield box with banana plug connectors in its back so that the 60 Hz 24 volt source may be plugged into or disconnected from the generator terminals of the bridge quickly and easily. The 110 volt terminals of the transformer are connected to a 110 volt outlet with a suitable cord and plug.
- c. To make measurements at other frequencies than 60 Hz, connect the generator output terminals marked BEN OUT to the bridge GEN 1 and 2 terminals with the links provided or more conveniently with a connector made of a $1/4'' \times 1-1/4'' \times 1-3/4''$ Lucite block with 4 properly spaced banana plugs mounted in it and wired to connect the proper terminals.
- d. To make dissipation factor measurements at higher values than provided by the Dissipation Factor Dial of the bridge, make the following connections: Remove the link connecting the EXT D ADJ terminals. Plug one end of a General Radio

Type 274 NQS double plug patch cord into these terminals and the other into the terminals of the decade resistor.

- e. The Balsbaugh cells are connected to the unknown terminals of the bridge as follows: Plug the center connector of a General Radio 874-Q9 or 777-Q3 Adaptor into the Unknown No. 1 terminal so that its shield terminal connects to the GRD terminal. Use the General Radio Type 874 Patch Cord to connect between the adaptor and the Type 874 connector on top of the Balsbaugh cell. Plug one end of the banana plug patch cord into the Unknown No. 2 terminal of the bridge and the other end into the banana plug jack at the top of the cell.

2. General Radio Type 1644A Megohm Bridge

- a. With the bridge power switch off, plug the power cord into a 110 volt outlet.
- b. Connect the Balsbaugh cell to the bridge as follows. Plug the center connector of a General Radio 874-Q9 or 777-Q3 Adaptor into the bridge terminal marked + Unknown. Connect the shield terminal of the adaptor to the bridge terminal marked Guard. Connect the Guard and Gnd terminals of the bridge with one of the links supplied with the bridge. Then use the Type 874 patch cord to connect the 874 adaptor on the bridge and the Type 874 connector at the top of the cell. Use the banana plug patch cord to connect the Unknown - terminal of the bridge and the banana plug jack at the top of the cell.

DSW 330609

MATERIALS

1. Glycerin for constant temperature bath.
2. Xylene, Reagent Grade.
3. 29% Ammonium Hydroxide, Reagent Grade.
4. Distilled H_2O .
5. Acetone, Reagent Grade.
6. Benzene (Non-Reagent Grade).
7. Acetone (Non-Reagent Grade).
8. Calcium Oxide Lump Form (Fisher Cat. No. C-116).
9. Boiling Chips (Used in cleaning baths).

PROCEDURE

A. Clean Test Cell

1. Using tongs place test cell into cell holder.
2. Clean in xylene vapor bath for 15 mins.
3. Clean in boiling 29% NH_4OH for 10 mins.
4. Clean in distilled H_2O steam bath for 30 mins.
5. Clean in acetone vapor bath for 15 mins.
6. Dry in $100^{\circ}C$ - $110^{\circ}C$ oven for 30 mins.

Note: Cells must be handled only with cell holders or

tongs. The cleaning baths require cleaning once per week.

B. Cleaning Glassware

1. Clean Pipette.
 - a. Use tongs to hold pipette.
 - b. Benzene wash.
 - c. Acetone wash.

DSW 330610

- d. Distilled H_2O wash.
- e. Reagent grade acetone wash.
- f. Draw clean air through pipette.
- g. Dry by flaming with bunsen burner.
- h. Draw clean air through pipette.

Note: No part of the pipette which might contact sample should be touched with hands or any support.

2. Clean Sample Vessel

- a. Pour sample into solvent can.
- b. Benzene wash.
- c. Acetone wash.
- d. Rinse off glycerin with water.
- e. Acetone wash.
- f. Clean in xylene vapor bath for 15 mins.
- g. Fill $3/4$ full with 29% NH_4OH boil for 10 mins.
- h. Steam for 30 mins.
- i. Dry at 100° - $110^{\circ}C$ for 30 mins.
- j. Draw clean air through vessel before using.

C. Sample Preparation

- 1. Remove sample vessel from drying oven. Use tongs.
- 2. Draw clean air through sample vessel.
- 3. Pipette sample into sample vessel by means of a suction bulb.
- 4. Remove test cell from drying oven and insert coaxial cable.
connector shell and pin.

DSW 330611

5. Place test cell in sample (check to make sure that the sample level is just above the bottom of the holes on the unguarded electrode).
6. Fasten sample vessel supporting clamp in such a position to allow 1/8 inch of the vessel to extend above the top edge of the clamp. This insures proper immersion depth in the bath.
7. Connect leads to the cell and put on Teflon lid.
8. Place sample vessel in the 100°C constant temperature bath and record entrance time.

Note: The required amount of sample is just enough to insure sample flowing through the holes on the unguarded electrode (outside). This amount is approx. 50 ml.

D. General Operating Procedure for Electro Scientific Industries
Model 861A AC Generator Detector

1. 60 cycle Measurements
 - a. Plug in the step-down transformer adaptor into Gen. position 1 & 2 on capacitance bridge.
 - b. Turn the AC line switch on, allow 15 min. warm up.
 - c. Rotate the output Power control fully counterclockwise.
 - d. Set Frequency Control for 60 cycles.
 - e. Set Range Control at the 1X position.
 - f. Set the Selectivity control to the Sharp position.
 - g. Turn the Log-Linear Switch to the Log position.

DSW 330612

- h. Turn outer Sensitivity control knob to such a position that a deflection on the meter can be seen. (Not more than 50% of scale)
- i. Rotate the Fine Tuning control to the position giving the maximum meter deflection. Detector is now properly tuned for 60 cycle measurements.

2. For Frequencies Above 60 Cycles

- a. Plug in special adaptor in generator out terminals 1 & 2.
- b. Turn the AC line switch on.
- c. Set the output Impedance switch to 100-ohm position.
- d. Rotate the output Power control full clockwise.
- e. Set Frequency Control for desired frequency.
- f. Set Range Switch to match frequency.
- g. Set Selectivity control to Sharp position.
- h. Turn the Log-Linear Switch to the Log position.
- i. Turn outer Sensitivity Control knob to such a position that a deflection on the meter can be seen. (Not more than 50% of scale)
- j. Rotate Fine Tuning control to the position giving the maximum meter deflection. Detector is now properly tuned.

E. Make Dissipation Factor Measurements Using ESI Type 701

Capacitance Measuring System

- 1. Turn on bridge null detector (allow 15 mins. warm up).
- 2. Set Frequency control to desired frequency using proper plug in unit, tune detector for maximum sensitivity (See General Operating Procedure for Model 861A AC Generator Detector, Section E).

DSW 330613

3. Set Capacitance range switch in the Picofarads C range.
4. Set Dissipation factor range switch to the 10^{-3} position.
5. Connect leads from the test cell to the bridge.
6. Apply bridge power.
7. Adjust bridge sensitivity (inner knob) until needle reads 40 on the meter scale.
8. Balance the bridge by adjusting the capacitance and then the dissipation factor knobs alternately until the best null is achieved at 10^2 sensitivity range.
9. A 1K and 10K step external decade resistance box is used to extend the bridge dissipation factor scale readings. Each 1000 ohm step on the decade box is equal to 100 divisions on the dissipation factor scale. It then becomes necessary to divide the total reading of the decade box by 10. This value is added to the Dissipation Factor scale reading.
10. Record the capacitance and dissipation factor readings 30 mins. after placing sample in the constant temperature bath.

Formula for calculating dissipation factor:

$$(S + R) \times A \times F = \text{Dissipation Factor}$$

Where S = Dissipation factor scale reading

R = External Decade box reading in ohms divided by 10

A = Dissipation factor range

F = Frequency in KHz (kilocycles)

Example for 60 cycle measurements:

$$S = 41, A = 10^{-3}, F = 6 \times 10^{-2}, R = \text{not needed to balance bridge}$$

From the formula $S \times A \times F = \text{Dissipation Factor}$

$$(41 \times 10^{-3}) 6 \times 10^{-2} =$$

$$41 \times 6 \times 10^{-5} = .00246$$

Example when the External Decade Box is needed to extend the

Dissipation Factor Scale readings

$$S = 41, A = 10^{-3}, F = 6 \times 10^{-2}, R = 250$$

From the formula $(S + R) \times A \times F = \text{Dissipation Factor}$

$$(41 + 250) \times 10^{-3} \times 6 \times 10^{-2} =$$

$$291 \times 6 \times 10^{-5} = .01746$$

Example For 100 cycle measurements

$$S = 41, A = 10^{-3}, F = 1 \times 10^{-1}, R = \text{not needed}$$

From the formula $S \times A \times F = \text{Dissipation Factor}$

$$41 \times 10^{-3} \times 1 \times 10^{-1} =$$

$$41 \times 10^{-4} = 0.0041$$

Example for 1000 cycle measurements

$S = 41$, $A = 10^{-3}$, $F = 1$, $R =$ not needed to balance bridge

From the formula $S \times A \times F =$ Dissipation Factor

$$41 \times 10^{-3} \times 1 = .041$$

Dielectric constant calculations can be made by using the following formula.

Formula for calculating Dielectric Constants .

$$\frac{C_s}{C_a} = \text{Dielectric Constant}$$

C_s = Measured capacitance of sample

C_a = Air Capacitance of the test cell in air

at room temperature

F. Make Resistivity Measurements Using Type 1644A Megohm Bridge

1. Turn on bridge to discharge position, allow 10. min. warm up.
2. Connect leads from test cell to Megohm Bridge.
3. Zero resistance bridge while charging zero at 500 volts for 30 seconds.
4. Measure for 30 seconds.
5. Read 1 minute after applying bridge voltage.
6. Return switch to discharge position.
7. Record resistance reading.