

MONSANTO CHEMICAL COMPANY
W. O. KRUMHOLTZ PLANT LABORATORY
STANDARD ANALYTICAL
METHOD

DATE ISSUED 1-21-66	PAGE 1 OF 10	NUMBER 11,608
WRITTEN BY CAM		APPROVED BY DCA
ATTACHMENTS		

PRODUCT	Aroclors, Pyranols and Related Products
TEST	Dielectric Constant and Power Factor
THIS FORM	☒ SUPERSEDES METHOD 11,608-57 ☐ REVISES INSTRUCTIONS 11,751-53
	ISSUED ON 1/10/57 2/26/53

SCOPE

This method is used to measure the dielectric constant and power factor of dielectric fluids.

EQUIPMENT

- See Page 2 for the equipment list and connecting diagram.
- The GE test cell is described as below.

Test electrodes, concentric nickel cylinders with feet. The inner electrode is 2.8" O.D. x 3.25" high. The outer electrode is 3.0" I.D. x 3.25" high. General Electric Co., Pittsfield, Mass., Cat. No. 1,559,663, Drawing No. M-651874.

Spacer plate, General Electric Co., Drawing No. M-651874-25.

800 ml. beaker - Set the spacer plate in the beaker with the concentric rings up. Set the concentric cylinders in the appropriate rings.

- Oven, constant temperature, Fisher Isotemp, Fisher Scientific Co., St. Louis, Mo., Cat. No. 13-245, or equivalent. This oven is modified to include inner-wall connectors and set to maintain a temperature of 100 ($\pm$ 0.5) $^{\circ}$ C.

The equipment is modified by drilling holes in the top for mounting two General Radio type 938 binding posts and insulators spaced 3/4 inch on centers. The holes are drilled one in front of the other.

One inch clearance holes are cut in the oven's inner lining directly under the binding post holes. A threaded coupling is used to connect the two General Radio 938 binding posts end to end. This enables the use of banana connectors inside and outside the oven.

The top (outside) connector will now fit a General Radio type 274-NL patch cord. The free end fits the "UNKNOWN DIRECT" terminals on the General Radio type 716-C bridge.

The inside connector will take General Radio Co. type 274-P plugs. Leads terminating in alligator clips will be soldered to these plugs. The alligator clips will in turn attach to the test cell.

The binding post nearest the back of the oven is also grounded to the oven's outer shell.

ELECTRICAL PROPERTIES TESTING APPARATUS

Oscillator - General Radio 1302-A

Bridge - General Radio 716-C

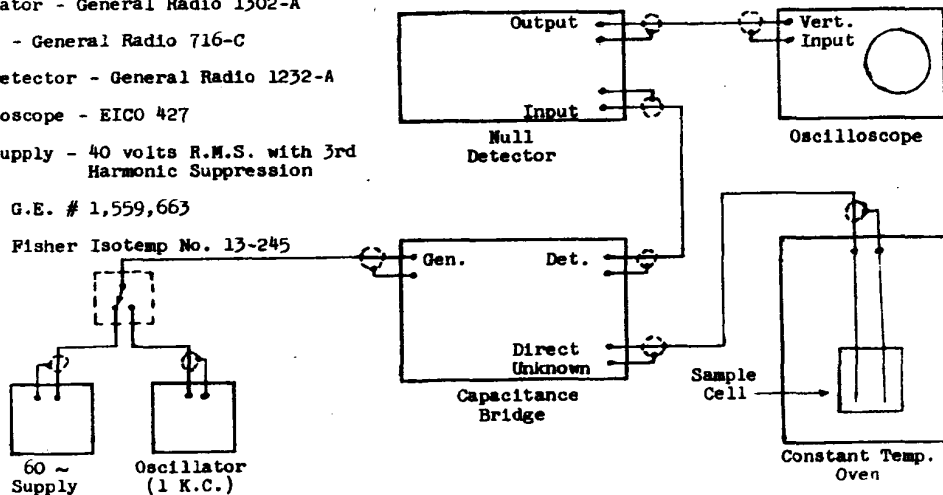
Null Detector - General Radio 1232-A

Oscilloscope - EICO 427

60 ~ Supply - 40 volts R.M.S. with 3rd Harmonic Suppression

Cell - G.E. # 1,559,663

Oven - Fisher Isotemp No. 13-245



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EQUIPMENT (Cont'd.)

- d. The remaining units are used as received after interconnection as shown on page 2.
- e. Thermometer, -1 to 101°C., 0.1° division, 610 mm, Fisher Cat. No. 15-000, or equivalent.

REAGENTS

- a. Tri Sodium Phosphate, 10% solution, Tri Sodium Phosphate, AR grade or equivalent. Make the 10% solution immediately prior to use.
- b. Trichlorobenzene, Commercial Grade.
- c. Methanol, 99.9+% Pure, Carbon and Carbide Co., or equivalent.
- d. Benzene - ACS grade.

SAFETY

Chlorinated benzenes and diphenyl are irritating to the skin. The vapors of the hot materials are irritating to the eyes and upper respiratory system. If accidental contact is made with the eyes, flush immediately with a large amount of water for at least 15 minutes and get immediate medical attention. Wash other affected areas with soap and water. Benzene and methanol are flammable.

The voltages used in the determination of power factor and dielectric constant are low and present no unusual shock hazard. If it is necessary to open cabinet or remove any component, the line plug must be removed.

Most measurements are made at 100°C. Therefore, gloves should be worn when making connections to the test cells when they are in the oven.

DETERMINATION

- I. Adjustment of Controls on Electrical Apparatus
 - A. On Panel No. 1 (Top Panel, Amplifier and Null Detector)
 - 1. Turn the 4-way (main power) switch on the upper right hand side to the 1000 position to determine the Dielectric Constant at 1000 cycles. Turn this switch to the 60 position for measurements at 60 cycles.
 - 2. Allow the equipment to warm up for at least 10 minutes.
 - 3. Turn the gain control to the middle of the red area.
 - 4. Turn the toggle switch to LOG position.

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DETERMINATION (Cont'd.)

1. A. 5. For 60 cycle measurements, turn the filter frequency knob to 20 - 200C position and then adjust the filter tuning dial for a maximum meter reading. This will be very close to a dial reading of 6. For 1000 cycle measurements, turn the filter frequency knob to the 200C-2KC position. Then adjust the filter tuning dial for a maximum meter reading. This will be very close to a dial reading of 10.

B. On Panel No. 2 (Oscilloscope)

CAUTION: Do not allow a high intensity spot to remain stationary on the screen for any length of time.

1. Turn "OFF PHASE" control to the 12 o'clock position (relative to the oscilloscope).
2. Turn the "ITEN" control to about the 12 o'clock position (relative to the oscilloscope).
NOTE: Always use the minimum trace intensity compatible with easy visibility.
3. Adjust the "FOCUS" and "ASTIG" controls for a sharp line. Use the vertical and horizontal positioning controls to center the image.
4. Set the "SWEEP SELECTOR" to $\frac{10K}{100K}$.
5. Set the "SWEEP VERNIER" to 8.
6. Set the "VERT. ATTEN." v/cm to 0.1.
7. Set the "VERTICAL GAIN" to 4.
8. Set the "VERT. AC-DC" to AC.
9. Set the "HORIZONTAL GAIN" to about 2 1/2.
10. Set the "SYNC SELECTOR" to (-).

C. On Panel No. 3 (Capacitance Bridge)

1. Turn "RANGE SELECTOR" switch to "100c" for 60 cycle measurements and to "1 Kc" for 1000 cycle measurements.
2. Turn "METHOD SWITCH" to direct.
3. Turn "DISSIPATION FACTOR" selector switch to "0".

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DETERMINATION (Cont'd.)

1. D. On Panel No. 4 (Oscillator)

1. Disregard this panel for measurements at 60 cycles.
2. On 1000 cycle measurements, depress the No. 10 "MULTIPLY BY" button.
3. Set "FREQUENCY DIAL" to 100.
4. Turn "OUTPUT" dial so that pointer is at the end of the arrow.
5. Depress the "UNBAL. 5000 OHMS" button.

II. Determination of Constants for the Apparatus

1. Carefully assemble the cell which has been cleaned and dried within the last 8 hours. Refer to Section IV.
2. Place the cell assembly in the Fisher oven which has been adjusted to 25°C.
3. Connect the back wire inside the oven to the lead on the outer cylinder of the cell and the front wire to the lead on the inner cylinder of the cell.
4. Connect the coaxial cable from the capacitance bridge to the fitting on top of the oven.
5. Remove the thermometer from the top of the oven before going on with the test. This is important.
6. Make all adjustments on the electrical apparatus as directed in Part I of this method.
7. Adjust the "CAPACITANCE" and "DISSIPATION FACTOR" dial and switch if necessary alternately until a sharp balance is obtained, that is when the line on the oscilloscope is a minimum width. This will be a narrow line except when high dissipation factor reading is obtained. The line will still be sharply defined but may not narrow to a fine trace.

The capacitance balance usually must be made more closely than the dial can be read in order to obtain a dissipation factor balance to a tenth division on the dial.

Correct manipulation will cause a large change in width of oscilloscope trace for very small change in either dial.

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DETERMINATION (Cont'd.)

11. 8. Record the sum of the readings on the "CAPACITANCE" and vernier and call this value A.
9. Remove the beaker containing the cell from the oven and fill it with benzene (ACS grade) to a level 0.737 inches (or 1/4 inch) above the top of the concentric cylinders of the cell.
10. Adjust the temperature of the benzene to 25°C. while standing with a thermometer.
11. Replace the cell in the oven (at 25°C) and make the same electrical connections as in Steps 3 and 4. DO NOT change connections.
12. Balance the bridge again as in Step 7.
13. Record the sum of the readings on the "CAPACITANCE" and vernier and call this value B.
14. Calculate the cell constant by the following equation:
$$\text{Cell Constant, } K = \frac{B - A}{2.27 - 1.0} \quad (\text{this is usually around } 1.0)$$
15. Remove the cell from the oven and balance the bridge as in Step 7 with the "CAPACITANCE" and "DISSIPATION FACTOR" dials.
16. Record the sum of the readings on the "CAPACITANCE" and vernier and call this value F. (capacitance of conductors)
17. Calculate the CELL LEAD CAPACITANCE by the following equation:
$$\text{CELL LEAD CAPACITANCE, } G = A - F - K \quad (\text{this is usually around } 3 \text{ uuf.})$$

WHERE:

- A = CAPACITANCE OF ENTIRE SYSTEM IN AIR (SYSTEM CONSTANT)
G = CAPACITANCE OF THE CELL LEADS (CELL LEAD CONSTANT)
F = CAPACITANCE OF CABLE AND WIRES WHICH CONNECT THE CELL AND CELL LEADS TO THE PLATES (CONNECTOR CONSTANT)
K = CAPACITANCE OF THE CELL ALONE (THE CELL CONSTANT)

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DETERMINATION (Cont'd.)

II. 17. (Cont'd.)

Tabulate the System Constant (A), the Cell Lead Constant (G), the Connector Constant (F), and the Cell Constant (K) on a piece of stiff paper and post them near the instrument where they can be easily referred to for comparison and calculations.

These constants must be checked at least once every three months and in all cases where the Dielectric Constant and/or Power Factor are out of specification.

III. Measurement of Dielectric Constant and Power Factor on Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends.

A. Test Run on Cell to Determine Whether it is Clean and Properly Aligned.

1. Carefully assemble a cell which has been cleaned and dried within the past 8 hours.
2. Adjust the oven control to hold at a temperature of 100°C.
3. Place the empty cell assembly in the oven and connect the back wire inside the oven to the lead on the outer cylinder of the cell, and connect the other (front) wire to the lead on the inner cylinder.
4. Connect the coaxial cable from the capacitance bridge to the fitting on top of the oven.
5. Allow 15 minutes for the cell to reach temperature equilibrium inside the oven.
6. Remove the thermometer from the top of the oven before taking any measurements on the bridge. This is important.
7. Make all the adjustments on the electrical apparatus as directed in Part I of this method.
8. Balance the bridge by rotating the "CAPACITANCE" and "DISSIPATION FACTOR" dials on Panel No. 3 until the wide vertical band on the oscilloscope screen is adjusted to a minimum width.

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- III. A. 9. Record the sum of the readings on the "CAPACITANCE" dial and vernier and compare this value with the SYSTEM CONSTANT determined in Part II of this method.

IMPORTANT: If the value obtained in Step 9 does not agree with the System Constant A (Part II) within 5 uuf, the cell must be re-cleaned, re-dried, re-assembled, and the test run for the System Constant must be repeated.

NOTE: Although the above test run must be made prior to each analysis, the value obtained in Step 9 is not to be used in calculations but is to be used only as a check on the cleanliness and alignment of the cell.

B. Procedure for Testing Materials

10. Remove the cell from the oven and fill the beaker with the material to be tested to a level 0.737 inches (ca. 3/4 inch) above the cylinders of the cell.
11. Adjust the temperature of the sample to 100°C. (use hot plate)
NOTE: Stir sample continuously with a thermometer while adjusting the temperature.
12. Place the cell and sample in the oven and make the same connections from the cell to the bridge as in Steps 3 and 4. DO NOT interchange connections.
13. Allow fifteen minutes for the cell to reach temperature equilibrium inside the oven.
14. Remove thermometer from the oven before taking a measurement. This is important.
15. Make the adjustment of controls on the electrical apparatus as directed in Part I of this method.
16. Adjust the "CAPACITANCE" and "DISSIPATION FACTOR" dial and switch if necessary alternately until a sharp balance is obtained, that is when the line on the oscilloscope is a minimum width. This will be a narrow line except when high dissipation factor reading is obtained. The line will still be sharply defined but may not narrow to a fine trace.

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DETERMINATION (Cont'd.)

- III. B. 17. Record the sum of the readings on the "CAPACITANCE" dial and vernier, and call this value X.
18. Record the sum of the readings on the "DISSIPATION FACTOR" dial and switch. Call this value X.

CALCULATIONS

$$\text{Dielectric Constant} = \frac{X - F - G}{K}$$

Where: X = Capacitance reading from Step 17.
F = Connector Constant (Determined in Part II)
G = Cell Lead Constant (Determined in Part II)
K = Cell Constant (Determined in Part II)

$$\% \text{ Power Factor} = \frac{f}{f_0} \times D$$

Where: f = Test Frequency (60 cycles or 1000 cycles)
f₀ = Frequency of "Range Selector" on Panel No. 3
D = Dissipation Factor reading from Step 18.

NOTE: When D (dissipation factor) is less than 0.1, the dissipation and power factors differ by less than 0.0005. Therefore, for our measurements, power factors and dissipation factors are equal.

PRECISION

(Reference: General Radio Manual for Model 716-C Capacitance Bridge)

- a. Capacitance readings are precise to ± 2 uuf $\times$ multiplier reading ($\pm 0.2\%$ of full scale for each range) when the dissipation factor is less than 0.01.
- b. Dissipation Factor (Power Factor) readings are precise to ± 0.005 or $\pm 2\%$ of the dial reading whichever is larger, for values less than 0.1 for D (Dissipation Factor).

IV. Cell Cleaning

A. The Electrodes Cleaning Procedure

1. Remove the electrodes from the tested material while hot and allow them to drain.
2. Place the electrodes in hot TCB for 10 minutes.
3. Wash with unheated TCB.

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- IV. A. 4. Rinse twice with methanol and twice with tap water.
5. Place the electrodes in hot 10% T.S.P. solution. Soak and heat for 10 minutes.
6. Wash thoroughly with tap water.
7. Place the electrodes in hot tap water for 10 minutes.

CAUTION: After Step 6 -- DO NOT TOUCH THE ELECTRODES WITH HANDS!

8. Wash with distilled water twice.
9. Dry in drying oven for at least two hours at 120°C.

B. The C. E. Cell Cleaning

1. Reclean the Cell before use, if more than 8 hours have elapsed since the previous cleaning.
2. Follow the procedure for the electrodes starting at Step 4.

C. Cleaning of the Accessories

1. Apply the same cleaning procedure as given for the electrodes (Steps 1 to 8) to prepare the glass spacer and beaker for next test.
2. Clean the thermometer in the same manner as the electrodes, except for Step 8.
3. Place the wet thermometer (after Step 7) directly in position in the Temperature Heating Unit (Modified Fisher Isotemp Oven) and allow to dry.

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