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JOB 146

MONSANTO CHEMICAL COMPANY
W. G. KRUMMRICH PLANT LABORATORY

STANDARD ANALYTICAL METHOD

DATE ISSUED	PAGE	NUMBER
3/2/65	1 OF 4	[illegible]
WRITTEN BY: CAM	APPROVED BY: [illegible]	
ATTACHMENTS:		

PRODUCT: Arochlors, Pyranols and Related Products

TEST: Dielectric Constant and Power Factor

THIS FORM ☒ SUPERSEDES METHOD 11,608 ☐ REVISES INSTRUCTIONS 11,751 ISSUED ON 1/21/66

SCOPE

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

EQUIPMENT

a. See Page 2 for the equipment list and connecting diagram.

b. 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-651874-2.

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

c. Oven, constant temperature. Fisher Jr. Cat. No. 13-261-10, modified to include complete inner wall lining of .032" aluminum, installation of motor and blower. Temperature remains constant to 100°C (±0.5°C).

The oven is also provided with General Radio Co. type 938 binding posts and insulators arranged to match terminals from the capacitance and resistivity bridges. The oven is mounted directly to the side of the cabinet containing the capacitance and power factor apparatus. This permits short, direct connections. Terminals and leads are inside the oven for connecting to the test cell.

d. A 150 watt heater is attached to the underside of the Capacitance Bridge shield to hold temperature about 20°C above ambient. Required to hold relative humidity below 40%.

Balance Capacitor - General Radio 1409F

Oscillator - General Radio 1302-A

Bridge - General Radio 716-C

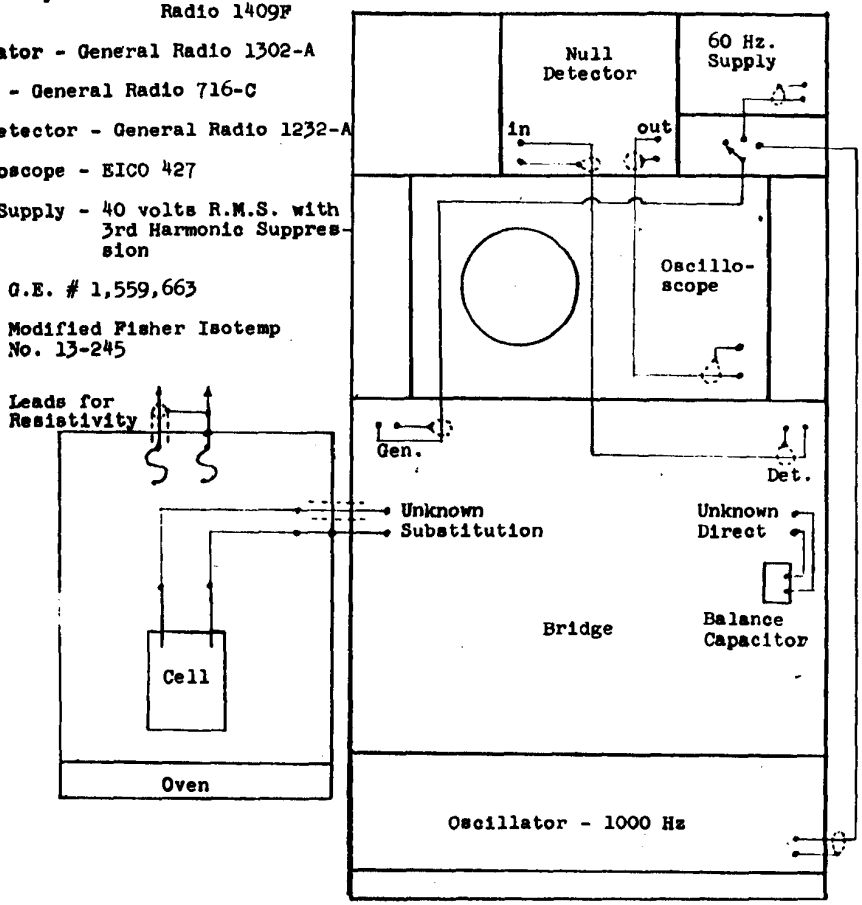
Null Detector - General Radio 1232-A

Oscilloscope - EICO 427

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

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

Oven - Modified Fisher Isotemp No. 13-245



ELECTRICAL PROPERTY APPARATUS

MGNS 076955

e. The remaining units are used as received after interconnection as shown on page 2.

f. 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

1. 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 and power factor at 1000 Hz (cycles). Turn this switch to the 60 position for measurements at 60 Hz (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.

5. For 60 Hz (cycle) measurements, turn the filter frequency knob to 20-200 C 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 Hz (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 compatable 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 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 Hz (cycle) measurements and to "1 Kc" for 1000 Hz (cycle) measurements.

2. Turn "METHOD SWITCH" to SUBST.

3. Turn "DISSIPATION FACTOR" selector switch to "0".

D. On Panel No. 4 (Oscillator)

1. Disregard this panel for measurements at 60 Hz (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. Position the cell so that the thermometer hole in the oven top is over the center of the cell.

4. Connect the clip on the lead from the oven GROUND connection to the terminal on the outer cylinder of the cell.

5. a. Adjust the position of the oven HIGH clip so it is close to (about 1/4") but not touching the terminal of the inner cylinder.

b. Remove the thermometer from the cell.

6. To Balance the Bridge --

Adjust the CAPACITANCE and DISSIPATION FACTOR dials alternately until a sharp balance is obtained on both, adjusting the DISSIPATION SWITCH if necessary. This condition will be indicated on the oscilloscope when a sharp narrow trace is observed.

6. (Cont.)

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

CAPACITANCE value is the sum of the readings of the dial and vernier.

DISSIPATION FACTOR value is the sum of the reading of the dial and switch.

A. Balance the bridge and record the CAPACITANCE value. Call this value X.

7. Connect the clip from the HIGH lead to the inner cylinder of the cell.

8. Again balance the bridge and record the CAPACITANCE value. Call this value X'.

9. Remove the beaker containing the cell from the oven and fill it with benzene (ACS grade) to a level 0.737 inches (ca. 3/4 inch) above the top of the concentric cylinders of the cell.

10. Adjust the temperature of the benzene to 25°C. while stirring with a thermometer.

11. Replace the cell in the oven (at 25°C) and make the same electrical connections as in Steps 4 and 7. DO NOT interchange connections.

12. Balance the bridge again, recording the CAPACITANCE value. Call this value Y.

13. Calculate the cell constant by the following equation:

Cell Constant, $K = \frac{B - A}{2.27 - 1.0}$, (this is usually around 70 uuf).

Where $A = X - X'$
$B = X - Y$
2.27 is constant for benzene
G (cell lead constant) = A - K

14. Tabulate the System Constant (A), the Cell Lead Constant (G), 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.

(A) - The System Constant is the air capacitance of the entire system.

(K) - The Cell Constant is the air capacitance of the measurement portion of the cell alone, that is that part of the cell completely surrounded by the material being tested.

(G) - The Lead Constant is the capacitance of the leads to the cell not covered by the test material.

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. Position the cell so that the thermometer hole in the oven top is over the center of the cell.

4. Connect the clip on the lead from the oven GROUND connection to the terminal on the outer cylinder.

5. a. Adjust the position of the oven HIGH clip so it is close to (about 1/4") but not touching the terminal of the inner cylinder.

b. Allow 15 minutes for the cell to reach temperature equilibrium inside the oven.

c. Remove the thermometer from the top of the oven before taking any measurements on the bridge. This is important.

d. Make all the adjustments on the electrical apparatus as directed in Part I of this method.

6. Balance the bridge and record the readings. Call the CAPACITANCE value C^s and the DISSIPATION value D.

7. Connect the HIGH clip to the terminal of the inner cylinder.

8. Again balance the bridge and record the readings.

9. This checks cell element alignment and spacer as following example:

	capacitance
reading from step 6	997.0
reading from step 8	922.0
total cell capacitance	75.0

If value is the same as "A" in cell constant determination, cylinder alignment is acceptable.

	dissipation factor
reading from step 6	-0.041
reading from step 8	-0.039
dissipation factor difference	0.002

Dissipation factor difference should be less than 0.004 when teflon spacer is used.

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 4 and 7. 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. Balance the bridge and record the readings.

Call CAPACITANCE value C
DISSIPATION value D^a

16. CALCULATIONS

$$\text{Dielectric Constant} = \frac{C^a - C - G}{K}$$

Where C^a Capacitance value from step 6.
C Capacitance value from step 15.
G Cell lead value from part II, step 13.
K Cell constant from part II, step 13.

$$\% \text{ Power Factor} = \frac{C^a}{\Delta C}\left(D^a - D\frac{f}{Fo}\right)$$

Where C^a = CAPACITANCE value from step 6.
ΔC = Step 6 minus step 15 - G.
D^a DISSIPATION value from step 15.
D DISSIPATION value from step 6.
f TEST frequency.
Fo FREQ from Range Selector Switch.
Δ Stands for change in.
G Cell lead valve from part II step 13.

NOTE: Dissipation factor is numerically equal to power factor within 2% for all values less than 20%.

17. PRECISION

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

a. Capacitance readings are precise to ±2 uuf x multiplier reading (±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 ±0.0005 or ±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.

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 G.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.