Project No. 141 R.

RESEARCH LABORATORY

Swann Research, Incorporated

Anniston, Alabama.

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# SUMMARY REPORT

# METHODS OF TESTING AROCLORS

By

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April 20, 1931

## I N D E X


Resistivity of Aroclors, Determination of.

Dielectric Constant of Aroclors, Determination of.

Power Factor of Aroclors, Determination of.

Viscosity of Aroclors, Determination of.

Distillation Range of Aroclors, Determination of.

Color of Aroclors, Determination of.

Flash and Flame Point of Aroclors, Determination of.


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OBJECT:

To describe in detail methods used in testing Aroclors which are to be put to electrical uses.

SUMMARY:

Test methods are given for resistivity, dielectric constant, power factor, viscosity, distillation range, and color.

CONCLUSIONS:

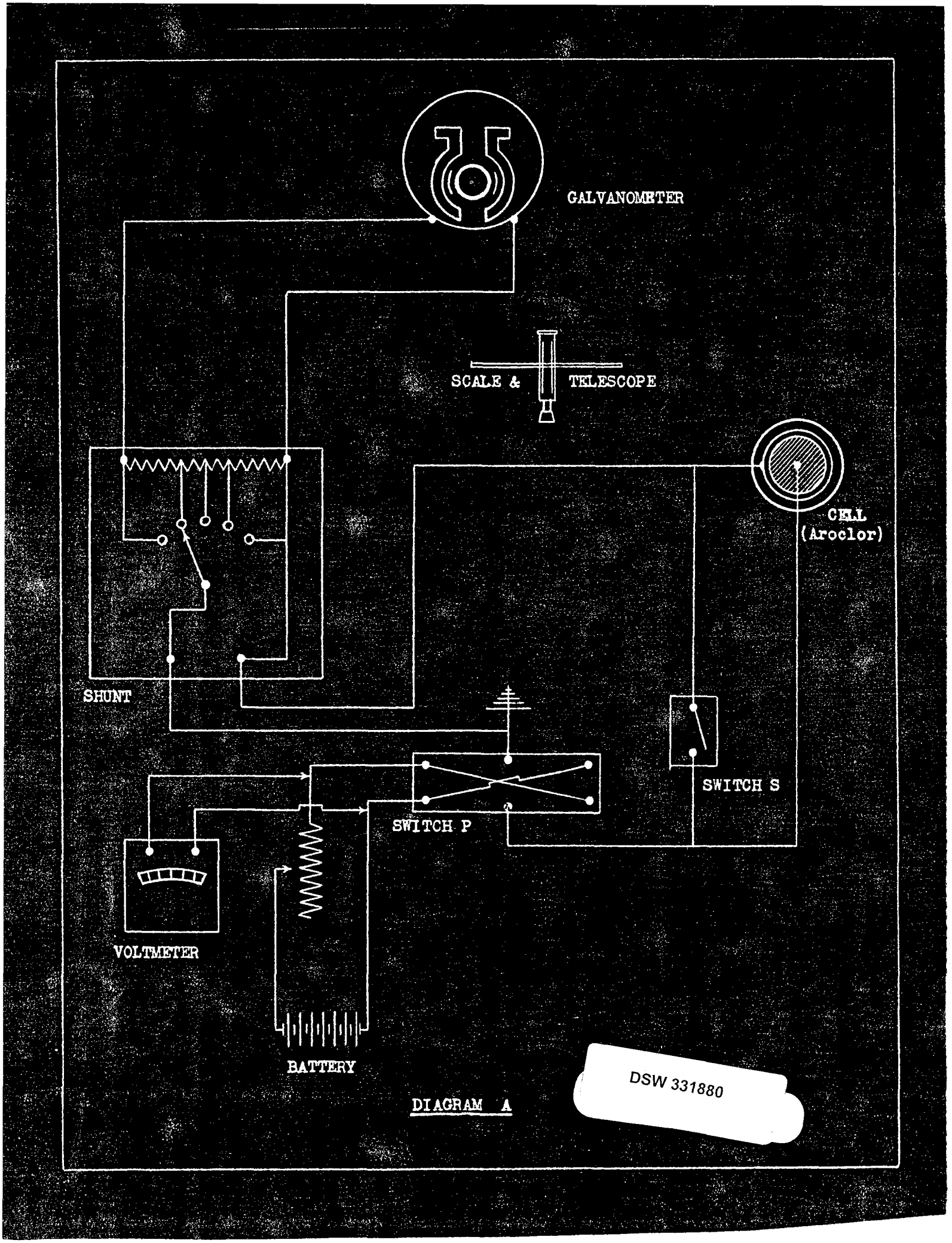
These methods are the ones used at present. They may be changed, or modified, later as warranted.

INTRODUCTION:

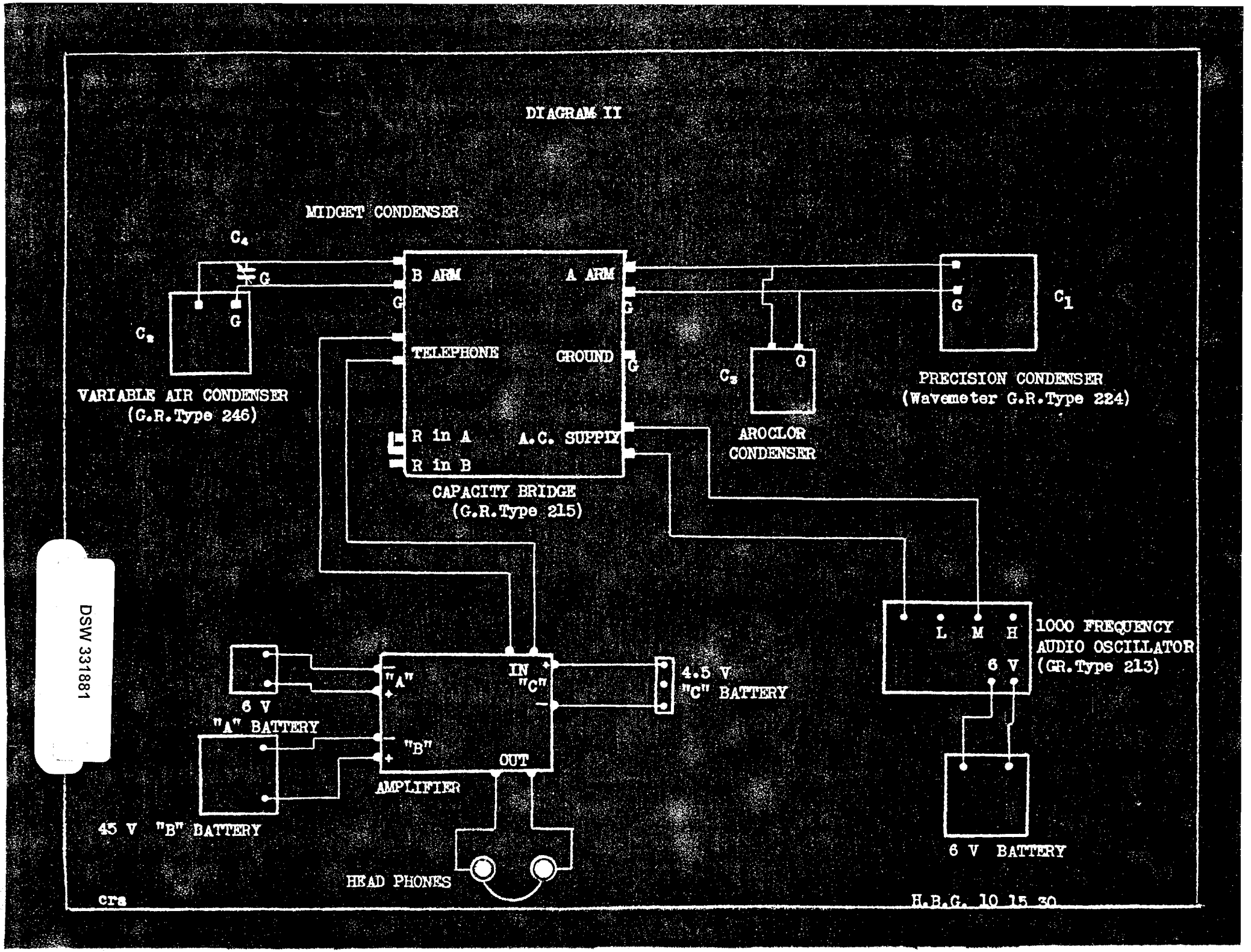
This report serves to put on record methods of testing which have been developed and used during the study of electrical properties of Aroclors.

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DIAGRAM II
MIDGET CONDENSER
$C_4$
G
$C_2$
G
VARIABLE AIR CONDENSER
(G.R.Type 246)
B ARM
A ARM
G
G
TELEPHONE
GROUND
G
R in A
R in B
A.C. SUPPLY
CAPACITY BRIDGE
(G.R.Type 215)
$C_3$
G
AROCLOR
CONDENSER
G
$C_1$
PRECISION CONDENSER
(Wavemeter G.R.Type 224)
L M H
6 V
1000 FREQUENCY
AUDIO OSCILLATOR
(GR.Type 213)
6 V
"A" BATTERY
"A"
"B"
IN
"C"
OUT
AMPLIFIER
4.5 V
"C" BATTERY
45 V "B" BATTERY
HEAD PHONES
6 V BATTERY
crs
H.B.G. 10 15 30
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## PART I

RESISTIVITY:

The circuit used for this measurement is shown in Diagram A. A working drawing of the test terminals is shown.

CALIBRATION OF APPARATUS

Electrode constant. The figure is obtained by calculating the mean average area of the two cylinderical electrodes. For the electrodes (test terminals) shown in the drawing, this constant is 736. This figure means that the electrodes are equivalent to two flat plates 736 cm. sq. spaced 1 cm. apart.

CURRENT LEAKAGE IN CIRCUIT:

Leakage within the test circuit must be tested for at frequent intervals. This is done by opening the circuit at the test electrodes, (switch S also open), setting the shunt on unity and closing switch P. The deflection of the galvanometer, after 30-60 sec., must not be more than 2 mm. scale reading. If the deflection is greater than this all insulating surfaces (switches, shunt, etc.) must be carefully cleaned.

PREPARATION OF ELECTRODES:

Before use, the electrodes glass plate and beaker must be very carefully cleaned and dried. This is best accomplished by cleaning them first with a small brush and benzene to remove grime. They should then be washed with hot distilled water, dried and washed in carbon tetrachloride. For cleaning after use in Aroclor a benzene wash should be used to remove all Aroclor. They should then be washed thoroughly with clean carbon tetrachloride and dried at 100°C for at least one hour. Care must be used not to touch any surface of the electrode, plate, or beaker (which is to come in contact with the liquid to be tested) with the fingers, after the last washing.

FILLING OF ELECTRODES:

After drying for at least one hour at 100°C, and with the beaker and electrode still hot, the liquid to be tested is poured in. Care must be used to make sure that the lip of the container, from which the liquid is poured, is clean.

MEASUREMENT OF RESISTIVITY:

As soon as the liquid to be tested has reached the desired temperature, resistance measurements may be made. It is present practice to make these measurements exactly four hours after the liquid is poured over the electrodes. The cell is connected in, by means of the screw clips, ten to fifteen minutes before measurements are to be made. The center electrode is then carefully placed so that the space between it and the outer electrode is



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equal at all points. With switch "S" open the shunt is set on the lowest ratio (.001) and switch "P" closed. Unless the approximate resistivity of the material being tested is known great care must be used here to prevent too great a deflection of the galvanometer. The short ratio is then decreased until the galvanometer scale reading is greater than 10 mm. but less than 200 mm. Switch P is then opened and switch S closed. The voltage from the battery is then set at exactly 500 by means of the variable resistance. The voltmeter must not be kept in the circuit during measurements.

At the end of the four hour peroid switch S is opened and P closed to either side. Exactly sixty seconds after closing P the deflection of the galvanometer is read, P opened and S closed. Three minutes later S is opened and P closed to the other side and a reading taken after sixty seconds. Four such readings are taken, the average of which is used to calculate the resistivity.

EXAMPLE:

Deflection: Right 50.52, left 48,50, Average 50.
Shunt setting: 0.01
Voltage: 500
Cell constant: 736
Galvonometer sensitivity: $1 \times 10^{-10}$ amps/mm (scale 1 meter distant).

Using Ohms' law

$$R = E/I$$

$$R = \frac{500 \times 0.01 \times 736}{50 \times 10^{-10}} = 73.6 \times 10^{10} \text{ ohms}$$

By convention resistivity is reported as ohms resistance times $10^9$ per cm.$^3$ (two plates 1 cm$^2$ spaced 1 cm apart). By this the above result becomes $736 \times 10^9$.

C A U T I O N: MAKE SURE THAT SWITCH S IS OPEN BEFORE CLOSING SWITCH P, otherwise a direct short circuit is made. Also, if the inner electrode should touch the outer electrode the shunt, or galvanometer, or both, will be burned out.

POWER FACTOR AND DIELECTRIC CONSTANT:

Diagram B shows the details of the circuit used for these measurements. A two-stage amplifier is used now, however, in place of the single-stage one shown. The instruction sheet which comes with an amplifier tube will show such a circuit.



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The Aroclor condenser, $C_x$ in diagram, of about 250 m.m.f. capacitance is carefully cleaned and dried and connected in. With its plates full in (in air), the precision air condenser, $C_1$, is set at any desired point and its capactance recorded. A-arm, consisting of $C_1$ and $C_x$ are now balanced by $C_3$ and $C_4$ (midget condenser for fine adjustment) of B-arm. This point is reached when the head-phones become silent. With all condensers filled with air the decade resistance should not be required. The plates of $C_x$ are now turned completely out and silence obtained in the phones by adjusting $C_1$. During this adjustment $C_2$ and $C_4$ must not be changed. The difference in capactance of $C_1$ before and after change of setting of $C_3$ gives the capacitance of $C_x$.

CALIBRATION OF AROCLOR CONDENSER ($C_x$)

| $C_1$ Scale Reading | | mmf (from Table I) |
|---|---|---|
| Plates in (air) | 1800.0 | 1064.0 |
| Plates out | 2192.4 | 1298.6 |
| | Capacitance of $C_x$ | 234.6 |
| Plates out | 1200.0 | 707.0 |
| Plates in (air) | 810.0 | 472.9 |
| | | 234.1 |
| Plates in (air) | 1400.0 | 826.0 |
| Plates out | 1794.5 | 1060.7 |
| | | 234.7 |
| Plates in (air) | 1000.0 | 586.0 |
| Plates out | 1390.5 | 820.4 |
| | | 234.4 |
| Plates in (air) | 200.0 | 110.0 |
| Plates out | 593.2 | 343.9 |
| | | 233.0 |

Average 234.3 m.m.f.

This value is that of the total change of capacitance of $C_x$ in air.

$C_x$ is now filled with the liquid to be measured and the same procedure repeated. The ratio of the change of capacitance of $C_x$ in air to its change of capacitance in Aroclor gives the dielectric constant.

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## DIELECTRIC CONSTANT OF AROCLOR 1254

(Research Reserve Sample)

| | Scale Reading | Capacitance |
|---|---|---|
| Plates in | 200 | 110.0 |
| Plates out | 2208.3 | 1307.9 |
| | | 1197.9 |

$$K = \frac{1197.9}{234.3} = 5.11$$

| | | |
|---|---|---|
| Plates in | 200 | 110.0 |
| Plates out | 2205 | 1306.6 |
| | | 1196.6 |

$$K = \frac{1196.6}{234.3} = 5.11$$

| | | |
|---|---|---|
| Plates in | 110.0 | 110.0 |
| Plates out | 2226.0 | 1318.3 |
| | | 1208.3 |

$$K = \frac{1208.3}{234.3} = 5.15$$

Average K = 5.12

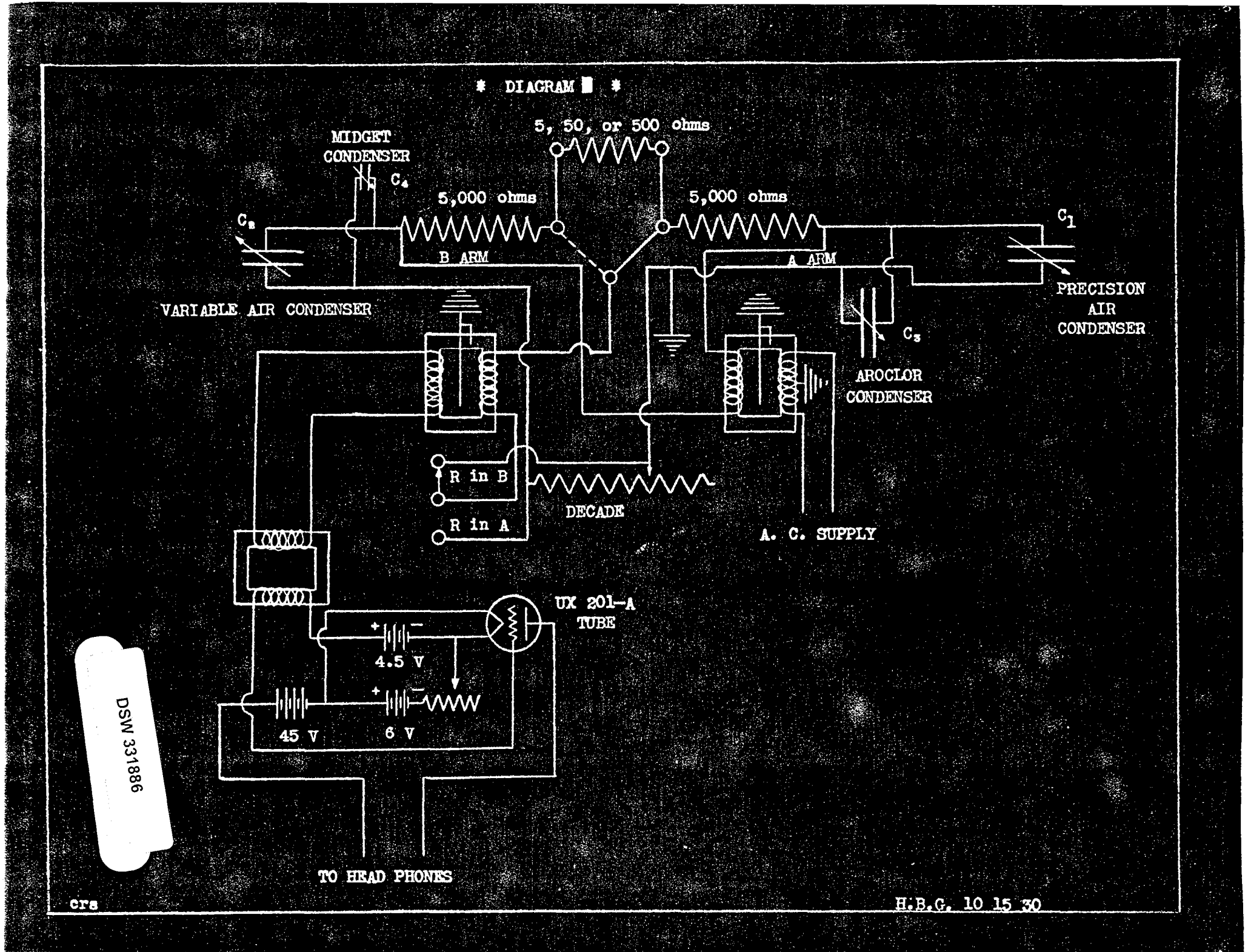
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Condenser $C_1$ must not be used below scale reading 200 or above scale reading 2400 as its calibration curve is not a straight line below and above these points. It will be found that the decade resistance must be used when $C_2$ is filled with Aroclor in order to obtain silence in the phones. This added resistance, however, can be neglected in the calculation of the dielectric constant. Great care must be used to remove all air from between the plates of $C_2$. Measurement of the dielectric constant of Aroclor 1242 requires a smaller condenser at $C_2$ than that required for Aroclor 1254.

Measurements of dielectric constant are now made at 100°C. After filling condenser $C_2$ with Aroclor to be tested it should be left in an oven at 100° for four hours.



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POWER FACTOR:

This property of a dielectric varies very markedly with temperature and with frequency of the current. It is determined with the apparatus in Diagram B by removing condenser $C_1$ and, balancing $C_2$ (with plate in) against $C_3$ (Be sure that midget $C_4$ is turned completely out.), using the decade resistance.

Power factor = $(C\ R\ 2\ W\ F)\ 10^{-10}$

Where C is the capacitance of $C_2$ (read from curve given in Table II), R the resistance read from the decade and F the frequency of the A. C. Supply. This expression gives the power factor in percentage.

## PART III

VISCOSITY:

This measurement is made at 212°F using a Saybolt Standard Viscosimeter.

Set the instrument level on a work bench or table with the three legs of the jacket stand touching the outside edge of the base block. Fill the bath vessel with pale engine oil (of about 350 to 400°F flash) so that the bath vessel will be full after this oil is heated and the turn-table cover adjusted. Place the turn-table cover in position. Place perforated brass disc in position by means of rod with threaded end, being sure that the convex side of the disc is towards the bottom of the tube. Insert the bath thermometer into the holes of the Tees to which the stirring paddles are attached. The bulb of the thermometer should rest in the lower Tee. Insert the electric heater into the receptacle.

Connect the attachment plug of the extension cord to a lamp socket. Be sure that button switch is off when heater is out of bath. The heater will burn out in a few minutes if not covered by liquid when current is on. Heat the bath slowly, gradually approaching the temperature specified for the test. To obtain this gradual heating, alternately connect and disconnect the heater by means of the two-button switch. Do not alternately immerse and remove the heater from the bath, because with the heater removed and the current on the heater will burn out. Keep the temperature uniform throughout the bath by means of a small stirrer. Do not raise the temperature of bath above highest graduation on bath thermometer.



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Clean the inner tube with some of the oil to be tested by means of plunger. The perforated disc above referred to may be left in during this cleaning. Place the small cork-stopper into the lower outlet of the oil tube, just enough to make an air-tight closure, but not so as to touch the small orifice of the oil tube. Strain the oil to be tested from cup, through strainer, into the oil tube until it overflows into the overflow cup, up to and above the upper edge of the oil tube. See that the bath is at the proper temperature to maintain the desired test temperature in the oil-tube and keep this bath temperature constant during test. Bring the oil in the tube to the desired test temperature using the oil-tube thermometer as a stirrer. This temperature should be uniform throughout the tube during the test. Remove the thermometer from the oil tube.

Draw off the oil from the overflow cup down to and below upper edge of the oil tube by means of the pipette. By this procedure the oil-tube is exactly filled. Place the receiving flask in the center hole of the base block so that it touches the block at one point, thus permitting the oil to strike the neck of the flask when the cork-stopper is withdrawn. This procedure prevents foaming, which tends to obscure the 60 cc mark on the receiving flask. Now with the oil and bath at the proper temperatures, snap the cork-stopper from the orifice with one hand and simultaneously start the watch with the other. Stop the watch when the bottom of the meniscus of the oil reaches the 60 cc mark on the receiving flask. The time in seconds required for the delivery of 60 cc is the Saybolt Viscosity of the oil at the test temperature.

Keep the oil tube covered when not in use. Clean the electric heater of the non-conducting black carbonaceous matter that collects on its surface, by using the heater to boil water and then rubbing with a cloth, when the current is off. Steam or hot water for heating or cold water for cooling the bath, may be introduced through the U-tube. Gas may also be used for heating.

## PART IV

FLASH AND FLAME POINTS:

The A.S.T.M Cleveland Open Cup is used for this test.

PROCEDURE:

The thermometer shall be suspended or held in a vertical position by the wire support. The bottom of the bulb shall be 1/4 in. from the bottom of the cup, and above a point half way between the center and back of the cup.



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The cup shall be filled with the oil to be tested in such a manner that the top of the menisous is exactly at the filling line at room temperature. The surface of the oil shall be free from bubbles. There shall be no oil above the filling line or on the outside of the apparatus.

The test flame shall be approximately 5/32 in. in diameter, the same size as the bead.

The device for applying the flame may be of any suitable type but it is suggested that the tip be approximately 1/16 in. in diameter at the end and that the orifice be 1/32 in. in diameter. If the device for operating the test flame be mounted in such a manner as to permit automatic duplication of the sweep of the test flame the radius ofswing shall be not less than 6 in.

The test flame shall be applied as the temperature read on the thermometer reaches each successive 5°F mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 6 in.) across the center of the cup and at right angles to the diameter passing through the thermometer. The test flame shall, while passing across the surface of the oil, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approximately one second.

The rate of heating of the oil shall be such that the temperature read on the thermometer increases not less than 9 nor more than 11°F per minute.

FLASH POINT:

The flash point shall be taken as the temperature read on the thermometer when a flash appears at any point on the surface of the oil. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame.

FLAME POINT:

After determining the flash point the heating shall be continued at the specified rate, and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn for a period of at least five seconds. The temperature read when this occurs shall be taken as the flame point.



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PRECAUTIONS:

The flash point and flame point tests shall be made in a room or compartment free from air drafts. The pperator shall avoid breathing over the surface of the oil. It is desirable that the room or compartment be darkened sufficiently so that the flash may be readily discernible.

## PART V

DISTILLATION RANGE:

(Applicable only to Aroclors that may be distilled at atmospheric pressure.)

This method is a modification of that standardized by the A.S.T.M. References -- "Tenative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment" (D20-28 T). A.S.T.M. Tenative standards, 1929,476; also Asphalts and Allied Substances by Abraham, 3rd Edition, Page 712.

APPARATUS:

The apparatus consists of a flask, condenser tube, shield, receivers and thermometers as specified.

FLASK: The distillation flask, Fig. 213, shall be a side neck distilling flask, having the following dimensions:

| | |
|---|---|
| Diameter of bulb, outside | 86 mm. ±1.5 mm. |
| Diameter of neck, inside | 22 mm. ±1.0 mm. |
| Diameter of tubulature, inside | 10.0 mm. ± 0.5 mm. |
| Length of neck | 43 mm. ±1.0 mm. |
| Distance, top of neck to tubulature | 25 mm ± 1.0 mm. |
| Length of tubulature | 220 mm. ±5.0 mm. |
| Angle of tubulature | 73 deg. ±1 deg. |

CONDENSER TUBE: The condenser tube shall be a suitable form of tapered glass tubing of the following dimension

| | | |
|---|---|---|
| Outside diameter of small end | 12.5 mm. | permissible variation ±1.5 m |
| Outside diameter of large end | 28.5 mm. | permissible variation ±3.0 m |
| Length | 360.0 mm. | permissible variation ±4.0 m |
| Length of tapered part | 100.0 mm. | permissible variation ±5.0 m |

SHIELD: A galvanized iron shield, lined with 1/8 in. asbestos, of the form and dimensions shown in Figure 214 shall be used to protect the flask from air currents and prevent radiation. The cover (top) may be of transite board made in two parts, or it may be of galvanized iron lined with 1/8 in. asbestos.

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RECEIVER: The distillates shall be collected in 100 cc glass graduates.

THERMOMETER: The thermometer shall be graduated in degrees centigrade.

SAMPLE: The sample as received shall be thoroughly stirred and agitated, warming if necessary, to insure a complete mixture before the portion for analysis is removed.

PROCEDURE: (a) The flask shall be supported on a tripod or rings over two sheets of 20 mesh gauze, 150 mm. square, as shown in Figure 215. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the neck with the top of the bulb level with the lowest point of juncture of the tubulature and neck of the flask.

(b) The axis of the flask through the neck shall be vertical.

(c) One hundred cc of the sample shall be measured into the flask using the same graduate to be used for collecting the distillate. The apparatus shall then be assembled and heat applied so that the first drop comes over in from five to fifteen minutes. The distillation shall be conducted at a rate of three to five cubic centimeters per minute. The condenser tube shall be warmed whenever necessary to prevent accumulation of solid distillates. Temperature readings shall be made at the following points -- 1st drop 5 cc -- 10 cc and then ever 10 cc up to the end point.

(d) During the progress of the distillation the thermometer shall remain in its original position. Correction shall be made for the emergent stem of the thermometer.

## PART VI

COLOR:

This determination is made by comparison method. An oil sample bottle is filled with the Aroclor to be tested and matched with the N.P.A. standards to the nearest one-half.

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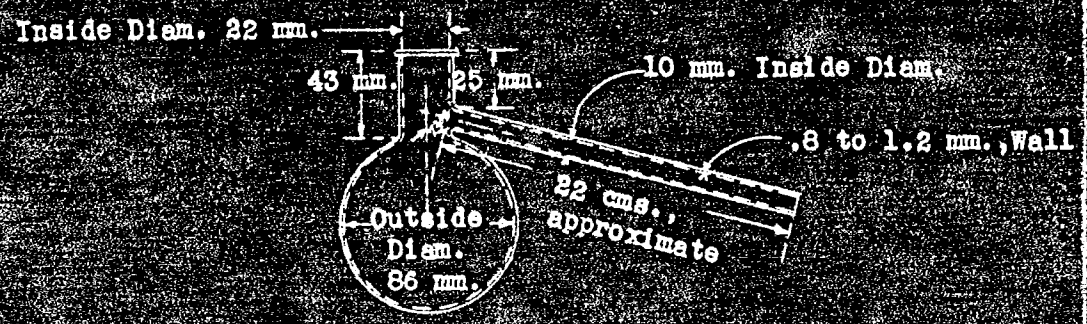
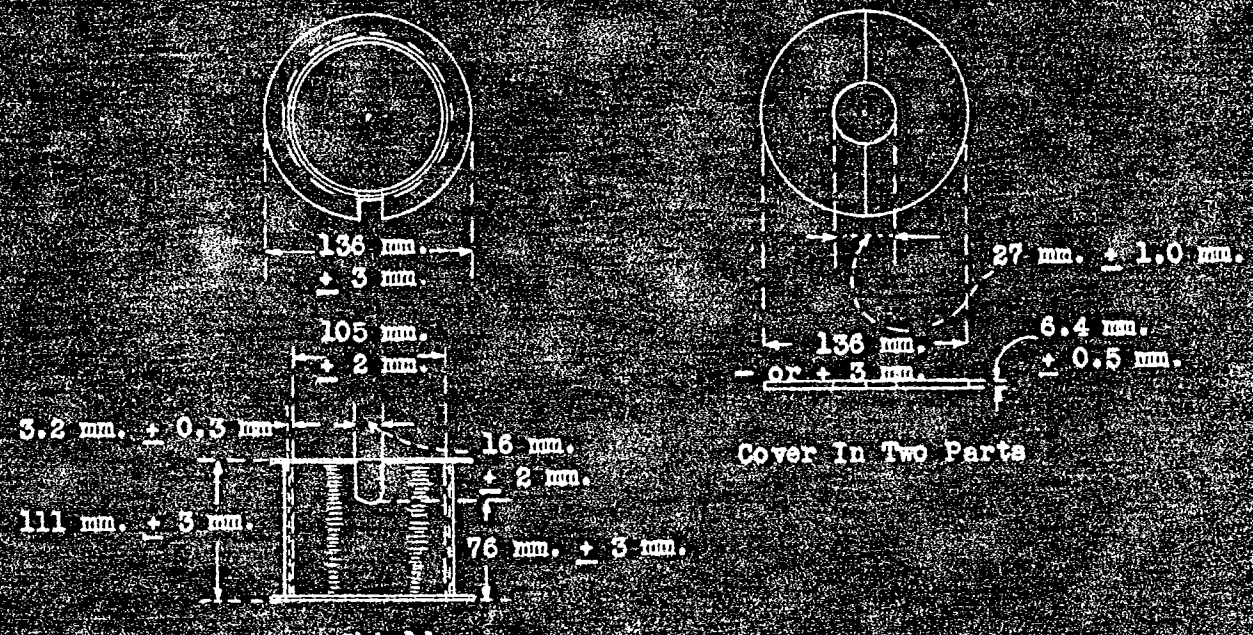



Figure 213. — Distillation Flask




Shield

Flanged open-end cylinder Made of 22 gage Galvanized Iron with 1/8" Asbestos Lining Rivited to Metal.

Fig. 214. — Distillation Shield

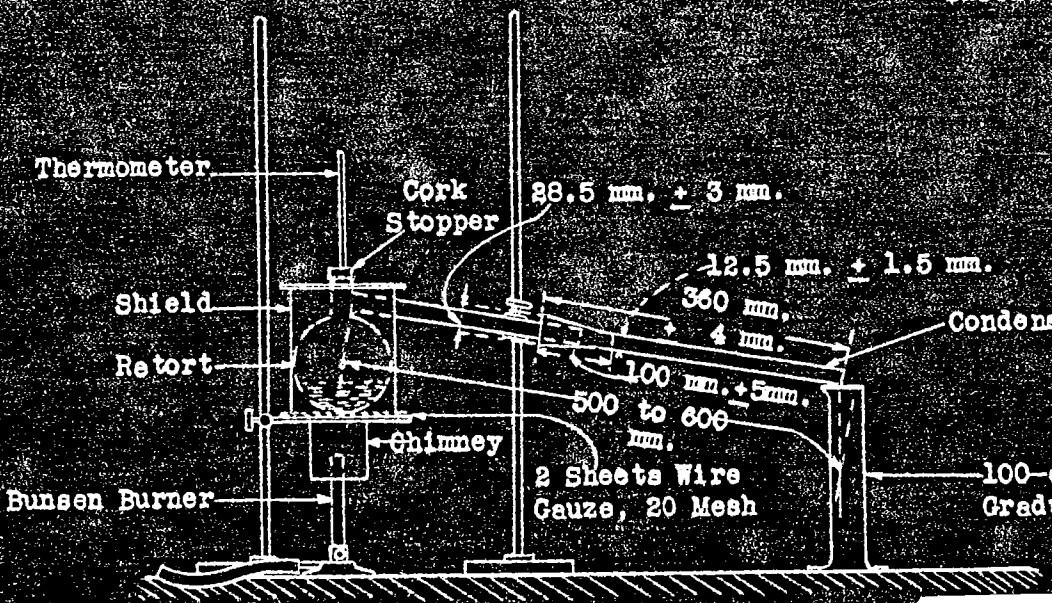



Fig. 215. — Distillation Apparatus Assembly.

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DIRECTIONS FOR TEST NO. 01-44-41

SUBJECT: Color of "Water White" Aroclors

METHOD: Comparison against A.P.H.A. Scale

## SCOPE

The method is applicable to Aroclors 1254, 1260, or others having color less than A.P.A. "1 ("Water").

## A.P.H.A. Standards

These color standards are acid solutions of potassium chloroplatinate and cobaltous chloride. The unit of color is that produced by 1 mg. of platinum per liter. The ratio of cobalt to platinum may be varied if necessary to match the hue. Because the hue of Aroclor 1254 and 1260 seems best matched without cobalt, this has been omitted in the preparation described.

Dissolve 1.245 g. of potassium chloroplatinate ($K_2PtCl^6$) containing 0.5 g. of platinum in water with 100 ml. of concentrated hydrochloric acid, and dilute to 1 liter with distilled water. This solution has a color of 500.

Prepare standards from 10 to 100 in steps of 10 by diluting 1, 2, 3 ml. etc. of the above solution with distilled water to 50 ml. in standard high form Nessler tubes. Protect the tubes from evaporation.

Refer to page 13 of "Standard Methods for the Examination of Water and Sewage" eighth edition (1936) for details of standards containing cobalt. Published by American Public Health Association, 1790 Broadway, New York.

## PROCEDURE

Fill a matching Nessler tube with Aroclor to a height equal to that in the standard tubes. Compare with standards by looking vertically downward through the tubes upon a white or mirrored surface placed at such an angle that light is reflected upward through the column of liquid. A color tube support (Fisher 7-065, 50 ml.) is convenient.

Inasmuch as the proportions of the standard color solution in the comparison tubes are such as to represent an aliquot part of a liter, the readings are direct as parts per million. Colors up to 100 are recorded to the nearest 5. Direct comparisons are satisfactory up to 100. Above this the sample should be diluted with carbon tetrachloride (maximum A.P.H. color – 5), and the color obtained – to nearest 10 – by multiplying by the dilution ratio.

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