BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

PART C 1. DETERMINATION OF SURFACE RESISTIVITY OF INSULATING SOFT VULCANIZED RUBBER AND EBONITE

B.S. 903 : Part C 1 : 1956

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BRITISH STANDARDS INSTITUTION

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The following B.S.I. references relate to the work on this standard:—
Committee reference RUC/10/6 Draft for comment CV(RUC) 5411

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*Federation of British Rubber and Allied Manufacturers' Associations
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BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

Part C 1. Determination of surface resistivity of insulating soft vulcanized rubber and ebonite

FOREWORD

This British Standard has been published under the authority of the Rubber Industry Standards Committee. In deciding to issue a revision of the 1950 edition, it has also been considered desirable to publish B.S. 903 in separate parts and the present part replaces Section 33.2 of 1950. The main differences are the inclusion of an electrometer method, the provision of an alternative test voltage and the alteration of the layout.

The group of parts in which the prefix letter ' C ' is used covers methods of testing the electrical properties of rubber and ebonite. Further parts in this group have been issued as follows:—

Part C 2. Determination of volume resistivity of insulating soft vulcanized rubber and ebonite.

Part C 3. Determination of permittivity and power factor of insulating soft vulcanized rubber and ebonite.

SECTION 1 DEFINITION

For the purposes of this British Standard the following definition shall apply:

Surface resistivity. The electrical resistance between opposite edges of a square of the surface of the material after a given period of application of d.c. electric stress.

SECTION 2 SUMMARY AND EXPLANATORY NOTE

The resistance of the surface of a test piece is determined by measuring the current passing under an applied d.c. potential between electrodes in intimate contact with the surface under test and separated from one another by a standard distance. Surface leakage is always associated with some leakage through the volume of the material, and conditions are chosen to minimize this.

It has been observed that the surface resistivity may not be independent of the length of the leakage path. For standard measurements, therefore, the dimensions of electrodes are specified. The concentric electrodes provide uniform leakage paths and the guard electrode on the other face of the test piece reduces the effects of leakage other than across the test surface.

Two sizes of test piece and electrode are specified; the larger size is recommended for materials of very high surface resistivity.

Method A provides for the use of a galvanometer and is suitable where the surface resistivity is less than about 3×10^{13} ohms, when 500 V and large electrodes are used.

Method B is an electrometer method suitable for the measurement of surface resistivities from about 10^{10} to 10^{16} ohms, the upper limit being attainable only when 500 V and large electrodes are used.

Method C covers the range of Method A, but if sufficiently high value resistors are used it can cover a somewhat higher range. It can be used in conjunction with Method B without requiring additional major items of equipment.

If there is uncertainty as to the probable magnitude of the resistivity of the test piece, an attempt should be made to use either Method A or Method C first.

Methods B and C are methods which eliminate stray currents which may otherwise arise through differences in potential between the guarded and guard electrodes during the test. Additional details will be found in Ref. 1. A suitable variation of Method B is outlined in Ref. 2.

SECTION 3 TEST PIECE

3.1. Soft rubber. The test piece shall be a disk with smooth surfaces not less than 100 mm in diameter, or not less than 200 mm in diameter according to the size of electrodes to be used, and $1{\cdot}25 \pm 0{\cdot}2$ mm in mean thickness. In comparative tests the mean thickness of individual disks shall not vary by more than 0·2 mm.

3.2. Ebonite. The test piece shall be a disk with smooth surfaces not less than 100 mm in diameter, or not less than 200 mm in diameter according to the size of electrodes to be used, and not exceeding 3·5 mm in mean thickness. Test pieces used for comparative tests shall be as nearly as practicable of the same thickness.

SECTION 4 ELECTRODES

The test piece shall be provided with electrodes and a guard plate as shown in Fig. 1 or 2; these shall take one of the following forms:

4.1. Graphite. This shall be applied before conditioning in the form of a colloidal suspension in water. It is convenient to dilute the graphite suspension with distilled water to the consistency of drawing ink and then to draw the circular outlines of the electrodes and guard plate on the surface of the test piece, afterwards painting the appropriate areas with the graphite suspension. Alternatively, the graphite may be applied by spraying, suitable stencils being used.

NOTE. The application of colloidal graphite to ebonite surfaces which have been exposed to light may increase the surface resistance by the interaction of the ammonia in the graphite suspension with the acidic decomposition products on the ebonite.

4.2. Metal films.* Metal films of adequate thickness deposited in vacuo either by sputtering or by volatilization shall be applied before conditioning.

4.3. Mercury. Mercury electrodes as shown in Fig. 2 shall be applied after conditioning.

When graphite or metal film electrodes are used, they and the guard plate shall be supplemented after conditioning by rigid brass backing plates which just cover the electrodes.

The electrodes shall be circular. The dimensions of the electrodes, guard plate, and backing plates, if any, shall be as follows (see Figs. 1 and 2) :—

	D_1	D_2	D_3 min.	D_4 min.
	cm	cm	cm	cm
Large electrodes	15·0	17·0	18	20
Small electrodes	5·0	7·0	8	10

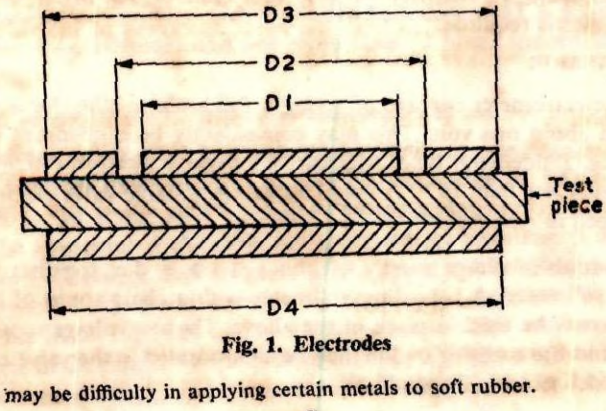

Fig. 1. Electrodes

* There may be difficulty in applying certain metals to soft rubber.

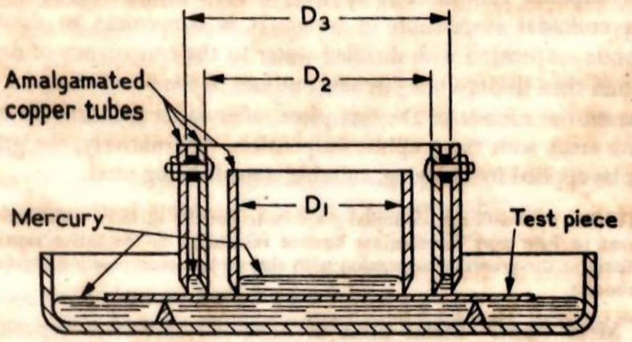

Fig. 2. Mercury electrodes

SECTION 5 APPARATUS

The apparatus required for all methods consists of:

5.1. A supply of 500 ± 50 V d.c. or 100 ± 10 V d.c. of adequate stability (see Appendix A). When a valve electrometer method is used to measure the current through the test piece, the positive pole of the voltage supply shall be earthed.

5.2. A voltmeter suitable for the above.

5.3. Screens and screened leads and a suitable switch for the high voltage.

In addition the following apparatus is required:

FOR METHOD A:

5.4. A galvanometer of suitable sensitivity together with a universal shunt.

To measure a resistivity of 3×10^{13} ohms a sensitivity of about 10^{-10} A/mm is required.

FOR METHOD B:

5.5. An electrometer capable of giving a full scale reading for a voltage less than about one volt. This may conveniently be a quadrant electrometer, e.g. Lindemann type. Alternatively a valve electrometer having a grid current less than 10^{-14} A may be used. A suitable valve electrometer is described in Appendix B.

5.6. A variable voltage supply of about 0–1·5 V d.c. together with a suitable voltmeter. A recording voltmeter with a chart speed of at least 6 in/min may be used in place of the above. The low voltage supply and control and the earthing switch may be incorporated in the valve electrometer as described in Appendix B.

5.7. A suitable earthing switch of very high insulation resistance (see Ref. 1).

5.8. A set of capacitors of known values with air insulation for the lower values and mica or polystyrene insulation for the higher values (see Ref. 1). All the capacitors used shall have a minimum insulation resistance of 10^{10} ohm microfarads.

5.9. If the voltmeter of Item 5.6 is not a recording instrument, a metronome or other audible timing device is useful to aid the operator to obtain the rate of change of voltage.

FOR METHOD C: *Items 5.5 to 5.7 together with*

5.10. A set of known resistances of about 0·001 times the resistances which are to be measured. High stability carbon resistors are available. 10^9 ohms is the highest value which may conveniently be used in this method unless the lead capacitances can be kept very small and the small test piece is used, in which case the upper limit is about 10^{10} ohms.

SECTION 6 PROCEDURE

6.1 Conditioning of samples and test pieces. Tests should not be carried out less than 24 hours after vulcanization, and for accurate comparisons between different rubbers it may be necessary to ensure that these are tested at substantially the same interval after vulcanization.

Except when it is required to study the effect of exposure to light, samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing, and care should be taken to avoid handling and contamination of the surface.

Before conditioning and applying the electrodes each test piece shall be wiped carefully with absorbent paper or with a soft cloth.

The test pieces shall then be conditioned at a temperature of 20 ± 2°C in a neutral atmosphere with a relative humidity of 65 ± 5 per cent, for not less than 18 hours and not more than 72 hours immediately before the test.

NOTE. See note under 7, Temperature and humidity of test.

6.2. Measurement of surface resistance. The surface resistance of the test piece shall be measured with a potential difference of 500 ± 50 V d.c. or 100 ± 10 V d.c. after 1 minute's electrification.

The apparatus shall be set up in accordance with Fig. 3. It is of the utmost importance that all parts of the apparatus connected directly to the guarded electrode should be adequately screened from leakage currents in all methods and from any electric fields which may induce charges in the case of Methods B and C. The insulation resistance between this screened system and its screen should, in the case of Method A, be sufficiently high to prevent shunting of the galvanometer and, in the case of Methods B and C, should be as high as possible.

In all methods, a check shall be made that any residual charge on the test piece at the beginning of the test is negligible.

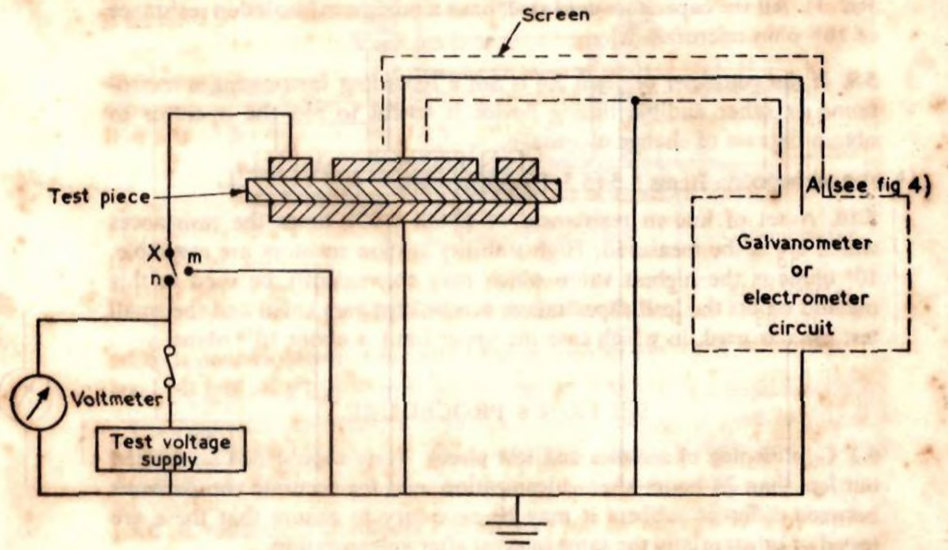

Fig. 3. Electrical connections and electrodes for surface resistivity test

METHOD A

Principle. In this method the current is measured by a galvanometer.

Check for residual charge in test piece. The switch X shall first be connected in the m position and any current δI due to residual charge in the test piece shall be noted (see Note).

Measurement procedure. The switch X shall be connected to n and after an interval of one minute the current I through the galvanometer shall be noted. The switch X shall then be thrown to the m position.

The surface resistance of the test piece, $R = \frac{V_H}{I}$

where V_H is the voltage applied to the test piece.

NOTE. If δI is an appreciable fraction of I, the switch X should be left in the m position for sufficient time for δI to become negligible on repeating the test.

METHOD B

Principle. In this method the rate of charge of a capacitor by the test piece current is determined by measuring the rate at which one plate of the capacitor must change in potential to hold the other plate (on to which the test piece current flows) at zero potential as indicated by a high impedance null instrument.

Setting up current measuring equipment. The circuit diagram of the current measuring equipment is given in Fig. 4. (The resistors may be omitted if it is not desired to use Method C in conjunction with Method B.)

Switch K (Fig. 4) shall be closed and switch X (Fig. 3) set to position m. The test piece shall be inserted, potentiometer P being set to give zero reading on voltmeter V. The electrometer shall be adjusted to the null position.

Check for residual charge in test piece. With a small capacitor of value C_r in circuit, switch K shall be opened and potentiometer P adjusted to hold the electrometer indicator at the null position. If, owing to residual charges on the test piece, this requires a continuous change of the voltage V, the rate of change $(dV/dt)_r$ should be noted (see Note 1).

Measurement procedure. Switch K shall be closed. Switch X shall then be set to position n, thus applying the voltage to the test piece, and the time noted.

Switch K shall then be opened, the electrometer being maintained at the null position by adjustment of potentiometer P, using a capacitor with a value C_m such that the rate of change of V may conveniently be estimated over a period of not more than 15 seconds. This selection shall be effected within 30 seconds of applying the voltage to the test piece. Switch K shall be closed when capacitors are being changed.

With switch K open, the rate of change of voltage $(dV/dt)_m$ necessary to hold the electrometer indicator in the null position shall be measured 1 minute after applying the voltage to the test piece. In practice this may be interpreted as $\Delta V/\Delta t$ where Δt is a finite time not greater than 15 seconds and whose mid-point is the required period after applying the voltage to the test piece. Switch K shall then be closed.

Switch K shall then be opened, potentiometer P having been set to give zero voltage, and the electrometer indicator shall be allowed to drift from the null position. It shall drift at least 10 times the greatest accidental deviation from its null position which may have occurred during the operation described in the preceding paragraph, showing that stray leakages are negligible. The switch K shall then be closed and the switch X returned to the m position.

The surface resistance of the test piece shall be calculated from the formula:

$$R = \frac{V_H}{C_m(dV/dt)_m} \times 10^{12} \text{ ohms}$$

where V_H = voltage applied to test piece and C_m is expressed in μμ F.

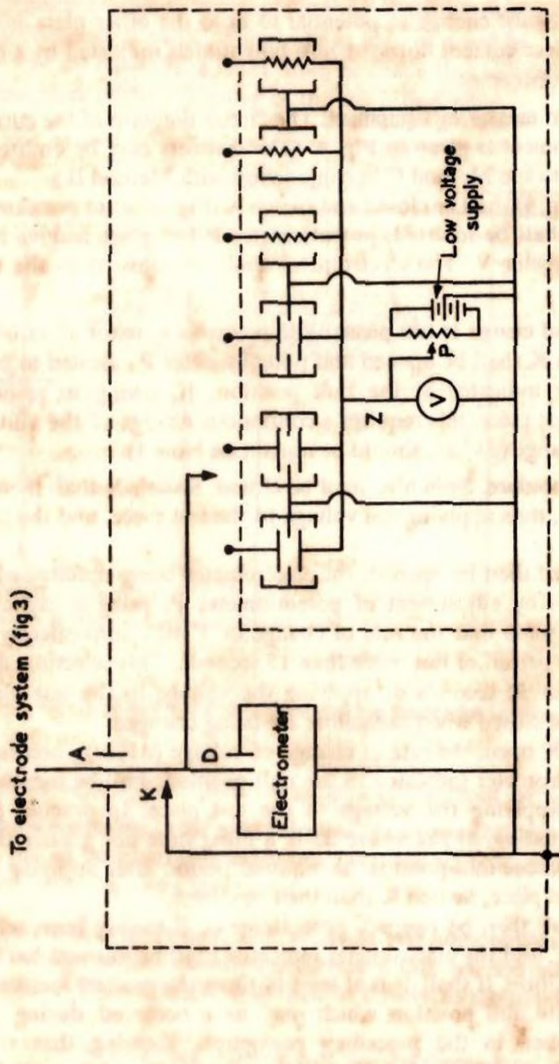

Fig. 4. Current measuring equipment Methods B and C

NOTE 1. A check should be made that the effects of residual charge measured at the beginning of the test have not caused appreciable errors, i.e.

$C_r\,(\mathrm{d}V/\mathrm{d}t)_r$ should be negligible compared with $C_m\,(\mathrm{d}V/\mathrm{d}t)_m$

If this is not so, the test should be repeated after a lapse of time such that $C_r\,(\mathrm{d}V/\mathrm{d}t)_r$ is sufficiently reduced

NOTE 2. Periodically, any capacitors used should be checked for leakage between the plates. This may be done by disconnecting the leads external to those shown in Fig. 4, adjusting potentiometer P to give a voltage V of the order of the maximum ever used, opening switch K and holding the electrometer indicator at the null position by adjusting potentiometer P. The value of $\mathrm{d}V/\mathrm{d}t$ must be negligible compared with the value of $(\mathrm{d}V/\mathrm{d}t)_m$ occurring during any test.

METHOD C

Principle. In this method the test piece current is passed through a resistor of known value, of which the end remote from the test piece is adjusted to a measured potential which just maintains the near end at zero potential as indicated on a high impedance null instrument. The current is then known from the resistance and the measured voltage (which is equal and opposite to that across the resistor).

Setting up current measuring equipment. The circuit diagram of the current measuring equipment is given in Fig. 4. The capacitors may be omitted. The resistors may all be encased in one earthed screen if desired and a switch may be employed to select the resistor provided it is screened from the rest of the circuit and has an insulation resistance more than 10 times the value of the highest resistance used. The low voltage supply and control are incorporated in the valve electrometer as described in Appendix B.

Switch K shall be closed and switch X set to position m. The test piece shall be inserted, the potentiometer P being set to give zero reading on the voltmeter V. The electrometer indicator shall be adjusted to the null position.

Check for residual charge on test piece. With a high resistance in circuit, switch K shall be opened and potentiometer P adjusted to return the electrometer to the null position. The voltage V_r required shall be noted (see Note).

Measurement procedure. Switch K shall be closed. The switch X shall then be set to position n, thus applying the voltage to the test piece, and the time noted.

Switch K shall then be opened and a resistance with a value R_m selected such that on adjustment of potentiometer P to maintain the electrometer at the null position a suitable voltage is indicated on voltmeter V. If in the selection of this resistance, the resistance connection is completely open circuited, switch K shall be closed whilst changing resistors.

The voltage V_m required to maintain the electrometer indicator at the null position after the required period of electrification shall be noted. The changes ΔV_m required to produce visible deflections of the electrometer indicator on either side of the null position shall be small compared with V_m, showing that stray leakages are negligible.

The surface resistance of the test piece shall be calculated from the formula:

$$R = \frac{V_H}{V_m} R_m \text{ ohms,}$$

where V_H = voltage applied to test piece.

NOTE. A check should be made that the effects of residual charge measured at the beginning of the test have not caused appreciable errors, i.e.

$\frac{V_r}{R_r}$ should be negligible compared with $\frac{V_m}{R_m}$

If this is not so the test should be repeated after a lapse of time such that $\frac{V_r}{R_r}$ is sufficiently reduced.

SECTION 7 TEMPERATURE AND HUMIDITY OF TEST

The temperature during the test shall be 20 ± 2°C, and the relative humidity 65 ± 5 per cent. After complete assembly the test piece shall be maintained under these conditions for at least 30 minutes immediately before the test is carried out.

NOTE. A temperature of 20 ± 2°C for conditioning and testing is not yet practicable for all countries. In tropical countries it is very difficult to maintain conditioning chambers or laboratories at this temperature, and an alternative temperature of 27 ± 2°C is therefore permitted.

SECTION 8 CALCULATION OF RESULT

Let R = surface resistance between electrodes in ohms,
D_2 = internal diameter of exterior electrode in centimetres,
and D_1 = diameter of interior electrode in centimetres.

$$\text{Surface resistivity } S = \frac{2\pi R}{\log_e \frac{D_2}{D_1}} \text{ ohms}$$

For the larger electrodes $S = 50{\cdot}3R$
For the smaller electrodes $S = 18{\cdot}7R$

SECTION 9 REPORT

The report shall state:

1. Surface resistivity in ohms.
2. Size of electrode.
3. Type of electrode.
4. Thickness of test piece.
5. Temperature of test.
6. Voltage used.

REFERENCE

1. R. H. Norman, *J. Sci. Instrum.*, 1950, 27, 200.
2. L. Hartshorn, *J. Sci. Instrum.*, 1936, 3, 1297.

APPENDIX A. H.T. VOLTAGE SUPPLY

The drift of the h.t. voltage must be so small that the value of $C_s \mathrm{d} V_H/\mathrm{d}t$ (where C_s is the capacitance of the test piece) is much less than the current being measured during the test. A battery of lead acid accumulators is probably the ideal if available. A battery of dry cells is adequate when new, but when it begins to deteriorate it may have a fairly steady rate of drift, either downwards or upwards according to whether a voltmeter is being or has been used to measure its voltage. Such a steady drift may cause unnoticed errors. It is probably most satisfactory (except perhaps for the highest end of the range covered by Method B) to use a stabilized mains operated power supply which need only be stabilized against input voltage changes. For really high stability it may be advantageous, where the power pack is operated from a.c. mains, to stabilize the a.c. voltage, rectify it and then stabilize the d.c. voltage. The main advantage of the stabilized power pack is that any fluctuations are generally of such short period that they may be detected from irregular motion of the electrometer indicator during the test.

APPENDIX B

DESCRIPTION AND OPERATION OF THE VALVE ELECTROMETER

General construction. The instrument (Fig. 5) should be completely enclosed in a light-tight earthed metal case. The lead to the grid of the valve should be air insulated and the terminal connected to it should have polystyrene or polythene insulation with fairly long surface leakage paths. The resistances of the three fixed resistors should have an accuracy of 2 per cent or better.

The input of the instrument at the point D must be well screened from the incorporated low voltage supply whose output is at the point Z. The switch K should be a contact hinged on an earthed part of the instrument which can make contact with the grid of the valve. The insulation between the lead from the grid to D and its screen should be air, except for any necessary supports which should be of polythene or polystyrene.

The instrument is provided with internal monitoring of the battery voltages, the settings of switch S1 being:

1. Normal operation.
2. Anode voltage check.
3. Filament voltage check.
4. Bias voltage check.

Setting up the instrument. Before the instrument is switched on, switch S1 should be set to position 3 and switch K should be closed. Immediately after the instrument is switched on, the 30-ohm potentiometer should be adjusted until the micro-ammeter reads 170 μA, and then switch S1 should be set to position 1. The zero-set potentiometer should then be adjusted until the micro-ammeter reads 100 μA.

The instrument will then have a null point reading of 100 μA. It is advisable that this be checked frequently and the zero-set potentiometer be adjusted as necessary.

Replacement of batteries. The batteries should be replaced under any of the following circumstances:

Battery A. If the filament resistance cannot be adjusted to give a 170 μA reading of the micro-ammeter. (Switch S1 in position 3.)

Battery B. If the backing-off voltage available is less than 1·3 V.

Battery C. If the reading of the micro-ammeter is less than 170 μA on either the anode or bias voltage checks (positions S2 and S4) or if it is impossible to set the zero on the instrument to 100 μA with switch S1 in position 1 and with switch K closed.

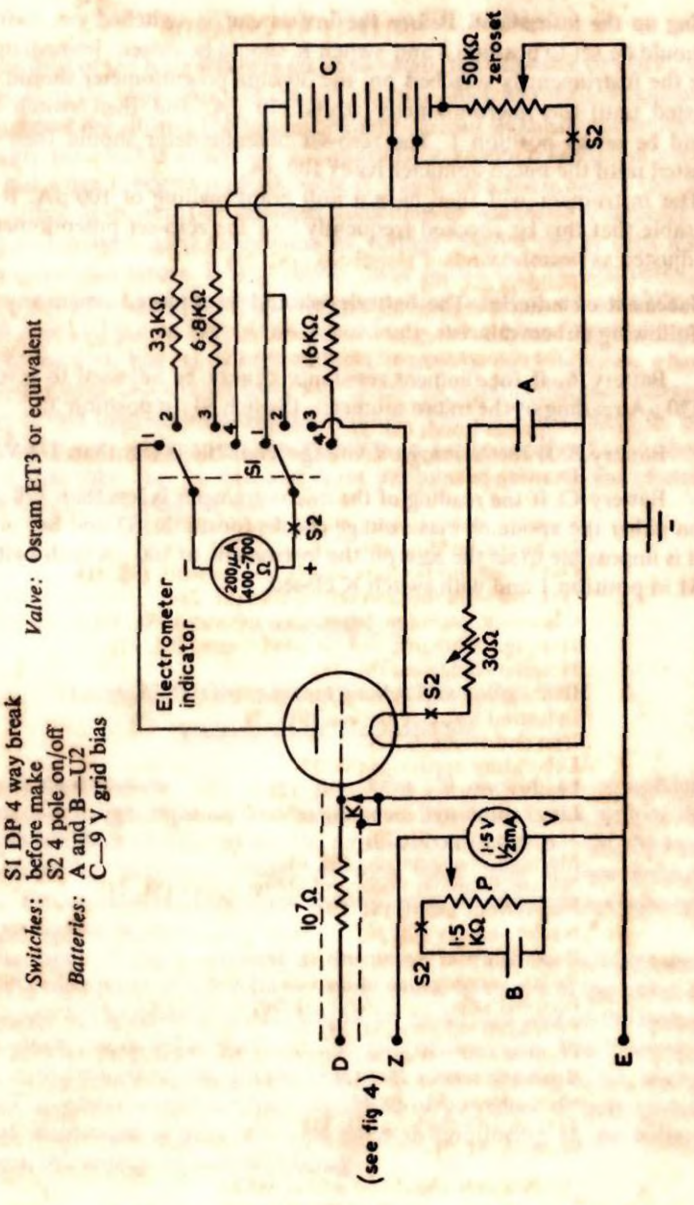

Fig. 5. Sketch of circuit for valve electrometer.

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