
British Standard Methods of test for
Petroleum and its products
Part 357. Permittivity of bitumen

(Identical with IP 357/83)

Méthodes d'essai du pétrole et des produits pétroliers
Partie 357. Permittivité des bitumes

Prüfmethoden für Erdöl und Erdölprodukte
Teil 357. Dielektrizitätskonstante von Bitumen

Foreword

BS 2000 comprises a series of test methods for petroleum and its products that have been published previously by the Institute of Petroleum (IP) and have been accorded the status of British Standards. Each method should be read in conjunction with BS 2000 : Part 0 'General introduction', which gives details of the BSI/IP agreement for publication of the series, lists more than 60 methods scheduled to be published in this way in 1982 and 1983, and provides general information on safety precautions, sampling and other matters.

The numbering of the other Parts follows that of the corresponding methods published in 'IP Standards for petroleum and its products, Part 1 Methods for analysis and testing'; BS 2000 : Part 357 is thus identical with IP 357/83.

IP 357 describes both a 'preferred' method and an 'alternative' method; in making reference to this Part of this British Standard it may be necessary to make clear whether the reference is specific to one of the methods.

It is intended to issue further revisions when necessary in line with any technical changes that may be made in subsequent editions of this IP method.

Caution. Attention is drawn to the safety precautions mentioned in Part 0 of this British Standard and also to the specific hazards mentioned in clause 6.

NOTE. The titles of the publications referred to in this Part of this British Standard are listed on the inside back cover.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Method (identical with IP 357/83)

INTRODUCTION

Two methods for the determination of the permittivity of bitumen are described. Both methods provide results with a precision adequate for specification purposes but the preferred steel cell method is more precise than the glass cell method. Also the steel permittivity cell is much more robust than the glass cell, and the steel cell method is easier to use. It should be noted that the permittivity obtained using the glass cell method is, on average, 0.004 lower than that obtained using the steel cell method.

PREFERRED METHOD – PERMITTIVITY USING A STEEL CELL

1. SCOPE

1.1. This method describes a procedure for determining the permittivity (see definition in 2) of petroleum bitumens of grades 35, 40 HD, 50 and 70 penetration as defined in BS 3690:1970, 'Bitumens for Road Purposes'.

2. DEFINITION

2.1. *Permittivity* – For the purpose of this test the permittivity of a bitumen is the ratio of the capacitance of a capacitor having that material as dielectric to the capacitance of the same capacitor having air as the dielectric.

3. SUMMARY OF METHOD

3.1. The permittivity is determined, under specified conditions, by measuring the capacitance of a bitumen sample contained in a concentric-electrode double capacitor.

4. APPARATUS

4.1. *Universal Bridge*¹ – The bridge shall have a source of 1592 Hz (10^4 rad/s), at least four-figure resolution, an accuracy of $\pm 0.1\%$ in the range 1 to 10 pF and a discrimination of at least 0.0001 pF.

4.2. *Permittivity Cell*² – The cell shall be constructed to comply with the requirements of Fig. 1. The material used to insulate the current electrodes from the body of the cell shall be stable at 200°C, and shall not be affected by bitumen or the cleaning solvents; the insulation shall also effectively prevent the ingress of bitumen and the cleaning solvents into the construction joints. Before the voltage electrode is fitted the internal faces of the current electrodes shall be carefully cleaned to ensure the complete removal of any of the insulation from these faces. Before fixing the voltage electrode in position, the threads of the mounting system shall be lightly but completely coated with an insulating sealing material that is resistant to bitumen and solvent, and remains stable at 200°C; Loctite pipe seal has been found satisfactory. The voltage electrode shall be mounted sufficiently rigidly such that a 5 kg load, at right angles to the axis of the electrode, shall not cause a deflection of more than 0.5 mm. The cell shall not leak when filled with the cyclohexane.

The electrical resistance between the current electrodes and the body of the cell shall be more than 10 MΩ. The electrical resistance between the voltage electrode and the body of the cell shall be more than 1 MΩ.

The capacitances of the lower and upper capacitors, when the cell is filled with air and the capacitances determined as described in 8.2.2, shall be within the range 1.05 ± 0.05 pF.

4.3. *Thermometer IP 38C* – The thermometer shall have been calibrated within the past year.

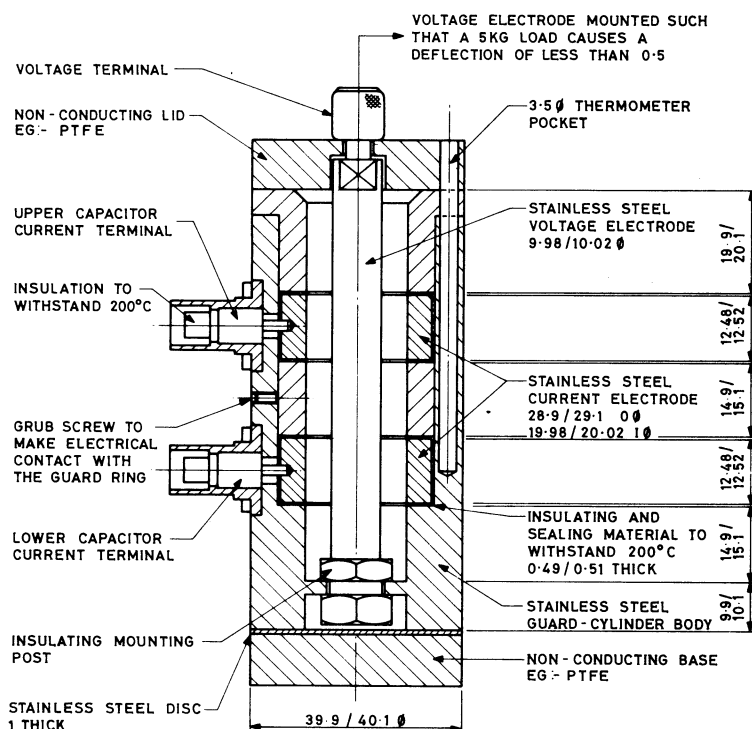
4.4. *Platinum-resistance Thermometer*³ – The thermometer shall be provided with a probe suitable for inserting in the cell body. The maximum error of the thermometer shall not exceed $\pm 0.15^\circ\text{C}$ at 25°C.

The thermometer shall be checked for accuracy against the IP 38C thermometer immediately before carrying out a permittivity test. This shall be done by immersing the thermometer bulb and probe, side by side, in a water bath controlled at $25 \pm 0.1^\circ\text{C}$.

NOTE to 4.3. The detailed specification for thermometer IP 38C is given in BS 2000 : Part 0 : Addendum 1.

¹ A suitable bridge can be obtained from Wayne Kerr Co., Durban Road, South Bersted, Bognor Regis, West Sussex. It is listed as a Universal autobalance bridge Model B642.

² A suitable cell and screened leads can be obtained from ERA Technology Limited, Cleeve Road, Leatherhead, Surrey KT22 7SA.



NOTE to Figure 1. All dimensions are in millimetres.

Fig. 1. Steel permittivity cell.

4.5. *Temperature-controlled Cabinet* - The cabinet shall be of appropriate internal dimensions (see Note 1), to accommodate the cell and a cooling coil; space to accommodate a small container of cyclohexane would be advantageous.

NOTE 1: Internal dimensions that have been found satisfactory are: 500 mm wide $\times$ 350 mm high and 350 mm deep.

The cabinet shall be fitted with an inner and outer door, the inner door being transparent. Forced-air circulation and the necessary controls shall be provided to maintain the air temperature in the cabinet at $25 \pm 0.25^\circ\text{C}$. When ambient temperature is above 22°C it will be necessary to provide a means of cooling the air in the cabinet, e.g. by passing cold water through a cooling coil placed in the cabinet.

The IP 38C thermometer shall be placed in a position within the cabinet to enable the air temperature to be monitored accurately.

5. REAGENTS

5.1. *Cyclohexane* - dry, of analytical reagent quality.

5.2. *Cleaning Solvent* - any good solvent for bitumen that is suitable for cleaning the steel cells when they are still hot, e.g. white spirit.

5.3. *Dichloromethane* - complying with BS 1994.

NOTE to 5.2. White spirit complying with BS 245 is suitable.

6. SAFETY PRECAUTIONS

6.1. Cyclohexane, white spirit and dichloromethane are toxic and should be handled in an effective fume cupboard. In the presence of flames or red-hot heating wire dichloromethane can decompose into highly toxic gases.

6.2. Cyclohexane and white spirit are flammable.

7. QUANTITY OF BITUMEN REQUIRED FOR A TEST

7.1. A single test comprises two determinations made in the one cell. A test requires about 20 g of bitumen to fill the cell.

8. PREPARATION, OPERATION AND CALIBRATION OF THE APPARATUS

8.1. *Operation of the Universal Bridge* – Refer to the manufacturer's handbook for the complete instructions for the use of the universal bridge in the measurement of capacitance. Follow closely the instructions during all stages of calibration and testing to ensure the high accuracy required in determining the permittivity of bitumen.

8.1.1. *Standardization of the Bridge* – Select the 10 pF range. Disconnect the voltage terminal (*E*) at the bridge and standardize the bridge. Take particular note of the need to check and adjust, if necessary, the conductance (*G* term) of the bridge whenever a balance of the capacitance (*C* term) is required.

8.2. Calibration of the Cell:

8.2.1. *Cleaning the Cell* – Clean the cell very thoroughly. If necessary, remove any traces of bitumen using the chosen solvent, then invert the cell and allow the solvent to drain completely. Finally, rinse the inside of the cell, provided it is cold, with dichloromethane, and dry.

NOTE 2: Prolonged contact with dichloromethane is inadvisable because of the risk of attack to the material used to form the cell joints.

NOTE 3: The final rinse of the inside of the cell should not show any discoloration of the solvent.

8.2.2. *Calibration Procedure* – Place the cell in an oven maintained at $150 \pm 10^\circ\text{C}$ for at least 30 min, then remove the cell and allow to cool for about 60 min.

Place the cell in the cabinet maintained at $25 \pm 0.25^\circ\text{C}$ and make connections to the voltage terminal and the lower cell capacitor. Insert the probe of the platinum-resistance thermometer in the hole provided in the cell body.

At least 30 min after the cell was placed in the cabinet, disconnect the voltage terminal (*E*) at the bridge and standardize the bridge (see 8.1.1). Reconnect the voltage terminal to the bridge and, provided the platinum-resistance thermometer indicates a temperature of $25 \pm 0.1^\circ\text{C}$, balance the capacitance and the conductance. Record the capacitance as LC_a to 0.0001 pF. Connect the upper cell capacitor to the bridge, balance, and record the capacitance as UC_a to 0.0001 pF.

8.2.2.1. *Capacitance of the Cyclohexane-filled Cell* – Fill the cell with cyclohexane that has been stored in the cabinet, and place the lid of the cell in position. Place the filled cell in the cabinet and make the connections to the voltage terminal and the lower cell capacitor. Leave for at least 1 h with the probe of the platinum-resistance thermometer in position. When the platinum-resistance thermometer indicates $25 \pm 0.1^\circ\text{C}$, disconnect the voltage terminal (*E*) at the bridge and standardize the bridge. Reconnect the bridge and balance both the capacitance and the conductance. Note the conductance and if it exceeds 0.02 nS the cell either requires further cleaning or the cell joints require attention. To check whether further cleaning is all that is required, repeat the procedure for calibration of the cell, i.e. 8.2.

Provided the conductance check has been satisfied, note the capacitance LC_c and the conductance LG_c . After a further 5 min check that the capacitance has not changed by more than 0.0002 pF and then record the capacitance as LC_c to 0.0001 pF.

Following a similar procedure to that used for the lower cell capacitor, connect the upper cell capacitor to the bridge, balance, apply the conductance check, and if the conductance check is satisfied, note the capacitance as UC_c . After a further 5 min check that the indicated temperature is still within $25 \pm 0.1^\circ\text{C}$ and that the capacitance has not changed by more than 0.0002 pF, and record the value to 0.0001 pF.

If there is a change in the capacitance of more than 0.0002 pF when the checks are made after the 5 min delay, repeat the whole of the calibration procedure.

³ A suitable thermometer can be obtained from Jenway Ltd, Gransmere Green, Felstead, Dunmow, Essex. It is listed as a battery-operated digital Model 1004 LCD thermometer with a platinum-resistance probe Type 112/3.

8.2.3. *Calculation of the Equivalent Air Capacitance (EAC)* – Calculate the equivalent air capacitance of the lower cell capacitor, rounded to four decimal places, as

$$EAC_L = \frac{L C_c}{2.015} \text{ pF}$$

where 2.015 is the permittivity of cyclohexane at 25°C.

Similarly, for the upper cell capacitor:

$$EAC_U = \frac{U C_c}{2.015} \text{ pF}$$

9. PROCEDURE

9.1. Check on the air capacitance of the cell. Check the air capacitances $L C_a$ and $U C_a$ as described in 8.2.2.

If the air capacitances differ by more than 0.0001 pF from those previously determined, check the cleanliness of the cell and repeat.

9.2. *Treatment of Sample* – Ease the lid or other closure of the sample container and then place it in an oven maintained at the temperature indicated in Table 1 for the time indicated in Table 2.

TABLE 1. Oven temperature

Nominal penetration grade (BS 3690)	Temperature range, °C
70	145 ± 5
50	150 ± 5
35	155 ± 5
40 HD	160 ± 5

TABLE 2. Heating period

Approximate volume of sample, ml	Period of heating, h
750 ± 250	2 ± $\frac{1}{4}$
1500 ± 500	2½ ± $\frac{1}{4}$
2500 ± 500	3 ± $\frac{1}{2}$

NOTE 4: Normally the sample size will fall in the range 500 to 3000 ml. Should a sample of less than 500 ml be received it is recommended that this fact should be reported with the test result; it is also recommended that the period of heating should not exceed 1½ h for such small samples.

The treatment of samples larger than 3000 ml will require special consideration. If it is not practical to heat the whole sample to ensure its homogeneity, a part of the sample may be removed from the container with a warmed 'knife'. This material should then be treated by a process that is as similar as is possible to that described in 9.2.

NOTE 5: In the unlikely event of the sample showing any tendency to foam during the heating period, it is probable that there is water present in the sample. In such a case any permittivity test result would be suspect.

9.3. *Filling the Test Cell with Bitumen* – At least 60 min before the end of the specified bitumen heating period, place the clean cell in an oven maintained at the same temperature as that used in the treatment of the sample (9.2). At the end of the cell heating period, fill the cell to within 1 mm of the top with the hot bitumen; avoid, as far as possible, the introduction of any air bubbles into the test material.

Immediately after filling the cell replace it in the oven for 10 min, then remove the cell and allow to cool for at least 60 min.

9.4. *Determination of Permittivity* – After cooling the cell, place it in the cabinet maintained at 25 ± 0.25°C, insert the platinum-resistance thermometer probe in the hole provided, and make connections with the voltage terminal and the lower cell capacitor. Disconnect the voltage terminal (*E*) at the bridge and standardize the bridge. Reconnect the bridge, check that the cell temperature is 25 ± 0.1°C, then balance the capacitance and the conductance, and record the capacitance as $L C_b$ to 0.001 pF.

Connect the upper cell capacitor to the bridge, check the cell temperature, balance, and record the capacitance as $U C_b$ to 0.001 pF as required for the lower cell capacitor.