

**Ministry of Defence  
Defence Procurement Agency, ADRP2  
Abbey Wood  
Bristol  
BS34 8JH**

## **OBSOLESCENCE NOTICE**

All DTD specifications were declared obsolescent from 1<sup>st</sup> April 1999. All DTD 900 series approvals also lapsed at that time. The standards will no longer be updated but will be retained as obsolescent documents to provide for the servicing of existing equipment.

### **Further Guidance**

The aim in declaring the specifications obsolescent is to recognise that the documents are not being updated and thus should be used with care by both purchaser and supplier. For example, a specification could contain valid technical information but may also contain type approval clauses that contradict procurement policy and/or use materials that do not comply with environmental legislation. The obsolescent specification can still be used as a basis for a purchase provided that the supplier and purchaser agree suitable changes to the specification within the purchase order/contract.

For the DTD 900 system, each specification has provided an MoD approved material and process. For these items, the declaration of obsolescence will constitute the termination of both the extant MoD approval and the continuing MoD assessment that had underpinned those approvals. Again, the technical content of the document remains valid and can be used by both purchaser and supplier as a basis for a contract but an acceptable (to the parties) approval/assessment procedure would be required.

**Aerospace Material Specification  
POLYVINYL BUTYRAL FOR AIRCRAFT  
TRANSPARENCIES**

Note 1. This specification is one of a series issued by the Procurement Executive, Ministry of Defence, either to meet a limited requirement not covered by an existing British Standard (Aerospace Series) or to serve as a basis for inspection of material, the properties and uses of which are not sufficiently established to warrant submission to the British Standards Institution for standardisation.

Note 2. The use of this specification will be reviewed periodically. Whenever possible it will be offered to BSI for issue as a British Standard (Aerospace) or to AECMA as the basis of a European Standard (Aerospace series).

Note 3. The tests employed in this specification are chosen for their reproducibility and ability to control the properties of the material. They are not intended to be simulated service tests which, because of variability of test conditions, may be unsatisfactory for control purposes.

Note 4. This specification calls for the use of substances and/or test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves either the supplier or the user from statutory obligations related to health and safety at any stage of manufacture or use.

This specification has been devised for the use of the Ministry of Defence and its contractors in the execution of contracts for the Ministry and, subject to the Unfair Contract Terms Act 1977, the Ministry will not be liable in any way whatever (including but without limitation negligence on the part of the Ministry, its Servants or agents) where the specification is used for other purposes.

Note 5. Information on the status and currency of this specification can be obtained from Specifications Section, Materials & Structures Department, Royal Aircraft Establishment, Farnborough, Hants.

**SECTION 1  
Scope**

The specification defines the requirements for the supply of plasticised polyvinyl butyral sheet material known hereafter as PVB, suitable for laminating aircraft transparencies. There are four types of plasticised PVB used in aircraft glazing, as follows:

Type 1 PVB plasticised with 19 parts per hundred by weight of DHA

TYPE 2 PVB plasticised with 36 parts per hundred by weight of plasticiser consisting of 82% by weight of DHA and 18% by weight of BBP.

Type 3 PVB plasticised with 21 parts per hundred by weight of DBS.

Type 4 PVB plasticised with 37.5 parts per hundred by weight of DBS.

NB. DHA is the abbreviation for di-hexyl adipate, BBP for benzyl butyl phthalate and DBS for di-n-butyl sebacate.

**SECTION 2  
Related Documents**

Reference is made in this document to the following:

BS 903            Methods of testing vulcanized rubber.  
                    Part A2 Determination of tensile stress: strain properties.

BS 5214          Testing machines for rubbers and plastics.  
                    Part 1 Constant rate of traverse machines.

BS 1752          (ISO 4793) Laboratory sintered or fritted filters.

The related documents listed are those applicable at the date of publication of this specification unless otherwise stated. The applicability of the documents must be confirmed by all users of the specification. The Quality Assurance Authority will supply, on request, information concerning any changes that may be necessary due to cancellation, replacement, supersession or amendment of any related document.

**SECTION 3  
Supply**

3.1 The materials shall be supplied in sheet form, free from folds, stains and inclusions of foreign matter, in lengths of the required width and wound on to a rigid and compatible core.

3.2 The gauges normally supplied shall be as follows:

Type	Gauge	Tolerance
1	0.38, 0.51 and 0.63 mm	+0.05 mm, -0.025 mm
2	0.38, 0.51 and 0.76 mm	” ”
3	0.63 mm	” ”
4	0.63 mm	” ”

## SECTION 4 Requirements

### 4.1 Plasticiser content

4.1.1 The parts per hundred (pph) referred to in Section 1 shall not vary by more than  $\pm 2$  pph. For example Type 1 shall have  $19 \pm 2$  parts of DHA to every 100 parts by weight of PVB resin. The plasticiser shall be evenly dispersed throughout the sheet.

4.1.2 The determination of plasticiser content shall be made either by the method given in Appendix 1 or by a similar method employing a Soxhlet extractor which produces similar results.

In the case of dispute the method in Appendix 1 shall be used.

### 4.2 Stress-strain properties

These properties shall be determined only for Type 1 interlayer material which is used in certain aircraft transparency designs where it may be called upon to act as a load-bearing ply. The values of ultimate tensile strength and elongation at break shall be determined using a tensile testing machine meeting the requirements of BS 5214 Part 1.

In order to measure accurately the cross sectional area of the test pieces, the material shall be press polished between parallel glass plates using the intended laminating cycle. Dumb-bell specimens as described in BS 903 Part A2 shall be preconditioned prior to testing, for a period of at least 24 hours at  $23 \pm 1^\circ\text{C}$  and a controlled humidity corresponding to 0.45% moisture content of the material. They shall then be tested in this condition.

The mean ultimate tensile strength derived by measurement of three specimens shall be not less than 26 MPa and the elongation at break  $190 \pm 10\%$ .

### 4.3 Clarity

All test specimens referred to in the following sub-paragraphs are to the design given in Appendix 2.

4.3.1 Light transmission - the visible light transmission of test specimens shall not be less than 85% when viewed at normal incidence using the apparatus and method described in Appendix 3.

4.3.2 Yellowness; this only applies to Type 1 and shall be determined by the transmittance at  $425 \pm 1$  nm as measured using a spectrophotometer covering the visible light range. The transmittance shall not be less than 60% at  $425 \pm 1$  nm.

4.3.3 Haze - this applies to all Types 1-4 and shall be determined using the test procedure specified in Appendix 4. The haze shall not exceed 3%.

### 4.4 Moisture content

The moisture content shall not exceed 0.45% by weight as determined immediately after opening the container in a room maintained at no higher than 24% RH at  $18\text{-}20^\circ\text{C}$ .

## SECTION 5 Qualification

5.1 Before any manufacturer's materials are supplied as complying with the specification the manufacturer should obtain qualification approval. Application for this approval shall be submitted to the DG/DQA/TS/MC, Building EI35/1, Royal Arsenal East, London SE18 6TD.

5.2 When applying for qualification approval the manufacturer shall submit the following:

- (i) Evidence that the materials comply with all the requirements of this specification.
- (ii) Samples of the material at least 1 m square for which approval is sought.
- (iii) Details of the composition of the interlayer. All data will be regarded as confidential.
- (iv) Details of the available sizes and thickness with tolerances.

5.3 In certain cases it may be necessary for the windscreen manufacturer to apply for approval on behalf of the interlayer manufacturer.

5.4 Any proposed change by the manufacturer in either the composition as stated in 5.2 (iii) or in the production process shall be notified to the Qualification Approval Authority. In addition he shall notify the purchaser, and the purchaser's design authority of the proposed change. If these authorities consider the change to be significant the product shall be designated a new product which shall require specific and separate approval. The changed material, if approved, shall be given a new trade designation or symbol.

## SECTION 6 Routine Quality Control

6.1 Interlayer supplied to this specification shall comply with all the requirement of this specification.

### 6.2 Acceptance tests

The customer is recommended to carry out the following acceptance tests upon receipt of the material.

On 1 roll from each type of material supplied:

Plasticiser content (clause 4.1)

On each roll of material:

Moisture content (clause 4.4)

On three rolls from each consignment of each type, or on each roll if there are 3 or less rolls in the consignment:

Stress/strain properties (clause 4.2)

Light transmission (clause 4.3.1)

Yellowness (clause 4.3.2)

Haze (clause 4.3.3)

In the event of failure to meet the requirements of any test, several metres of material shall be removed from the roll and two further samples taken for test. If both samples pass all requirements, the roll shall be accepted. If one or both lots of samples fail, the roll shall be rejected and all rolls in the consignment for that type of material shall then be tested.

- 6.3 The Quality Assurance Authority named in the contract may at any time require the manufacturer to test for compliance with any or all of the requirements of this specification.

### **SECTION 7 Storage and Life**

- 7.1 The refrigerated material in suitable containers (see clause 8.1), shall be kept with the rolls in a vertical attitude, in a store maintained at 3 to 10°C. Automatic temperature recording shall be installed. The requirement of Clause 4.4 shall be met following a period of storage in the containers up to one year from date of manufacture.
- 7.2 Material supplied to this specification and stored in accordance with 7.1, shall satisfy all the requirements of this specification within the storage life declared by the manufacturer.

### **SECTION 8 Packaging and Identification**

- 8.1 The interlayer material shall be supplied wound on to a core, the whole being wrapped in a sealed metal foil, surrounded by a strong cardboard carton for protection. Labels describing the contents and instructions for storage shall be attached to the outer wrapping.
- 8.2 The core shall be accurately aligned with, and project well beyond the material at each end, and the internal diameter shall not be less than 75 mm.
- 8.3 Sheet material shall be delivered in refrigerated containers embodying a metallised heat sealing plastic to ensure compliance with clause 4.4. The temperature of refrigeration shall be within the range 3 to 10°C.
- 8.4 Roll identification  
The wrapping of each roll shall bear the following information:
- (a) Type of material;
  - (b) Roll number, date of manufacture and any other identification required for an unambiguous traceability to a particular production batch.

- 8.5 Certificate of Conformity

A certificate shall be sent with each consignment. The Certificate of Conformity shall contain the following information:

- (a) Type;
- (b) Roll numbers and date of manufacture;
- (c) Moisture content;
- (d) Gauge.

## APPENDIX 1 DETERMINATION OF TOTAL PLASTICISER CONTENT

### 1. Sample Preparation

The determination shall be carried out in triplicate. Dry the specimens over phosphorus pentoxide in a desiccator until constant weight is obtained. During drying, contact between the interlayer material must be avoided to ensure complete removal of moisture. Interlayer material which has been incorporated into a laminated assembly shall be removed from between the surface sheets before analysis.

### 2. Total Plasticiser Content

#### 2.1 Procedure

Cut the sample into small pieces (3 mm square) and accurately weigh about 0.75 gram ( $W_1$ ). Place the weighed sample in a Soxhlet extractor of approximately 7 ml capacity and extract for 16 hours with 25 ml anhydrous diethyl ether. Maintain the ether level during the extraction. Filter the ether extract through a G3 sintered glass crucible to BS 1752, into a vacuum filter flask. Wash the Soxhlet extractor with two 10 ml portions of anhydrous diethyl ether and filter into the vacuum flask. Weigh accurately a 100 ml beaker ( $W_2$ ), completely transfer the filtered extract into the weighed beaker and slowly remove the diethyl ether using a vacuum desiccator, vacuum oven, or a steam bath. Allow the beaker containing the extracted plasticiser to cool to room temperature in a desiccator and reweigh ( $W_3$ ).

#### 2.2 Calculation

$$\text{Total plasticiser content (\%)} = 100 \frac{(W_3 - W_2)}{W_1}$$

Report the mean value of three determinations.

#### 2.3 Infra-red spectrum

##### 2.3.1 Apparatus

An infra-red spectrophotometer covering the range 2.5-15  $\mu\text{m}$ . (4000-667  $\text{cm}^{-1}$ ).

##### 2.3.2 Procedure

Use one of the extracts obtained from the total plasticiser determination to prepare a capillary film. Record the infra-red transmittance over the range 2.5-15  $\mu\text{m}$  of the film.

The sample spectrum shall not differ significantly from that of the reference spectra of DHA shown in figure 1, of DHA/BBP in figure 2 and of DBS in figure 3.

## APPENDIX 2 CONSTRUCTION OF SPECIMENS FOR CLARITY TESTS

Specimens for light transmission (clause 4.3.1) yellowness (Clause 4.3.2) and haze (Clause 4.3.3) each about 100 mm X 100 mm, shall be made up by laminating the thickest PVB material available (to give  $10.0 \pm 0.5$  mm final interlayer thickness) with facing glasses. Normally 3 mm thick float glass (Pilkington - UK) would be used for this purpose, but glasses from an alternative source may be used with adjustment of the thickness to ensure that the white light transmission (as determined by the method of Appendix 3) of each glass thickness is not greater than 89.5%.

## APPENDIX 3 DETERMINATION OF WHITE LIGHT TRANSMISSION

### 1. Scope and Field of Application

This method specifies the determination of visible light transmittance of planar sections of transparent plastics employing a defined light source and a photometer with a spectral response corrected to approximate that of photopic vision.

### 2. Definition

Visible light transmittance is defined as the energy flux of an emerging beam of light compared with that of the incident parallel beam falling upon the specimen under examination.

### 3. Apparatus

The apparatus, as shown in Figure 4, consists of a voltage-stabilised light source with associated optical system to produce a collimated parallel beam, specimen holder and photometer all rigidly mounted on a convenient optical bench.

#### 3.1 Light Source

3.1.1 The light source shall be a gas-filled tungsten filament lamp of photometric quality operating at a colour temperature of  $2855 \pm 100\text{K}$ . The power supply to this lamp should be stabilised to ensure short-term constant light output.

3.1.2 The light source is combined with an optical system to produce a parallel light beam of area at least  $1 \text{ cm}^2$  and of approximately circular cross-section.

#### 3.2 Integrating Sphere Photometer

3.2.1 The integrating sphere may be of any diameter exceeding 150 mm so long as the total port area does not exceed 2 per cent of the internal reflecting area of the sphere. The axis of the irradiating beam shall pass

through the centre of the entrance port and the centre of the integrating sphere. The photocell shall be positioned on the sphere 90° from the entrance port.

3.2.2 The light beam shall not be vignetted at the entrance port of the integrating sphere.

3.2.3 Reflecting surfaces - the surfaces of the interior of the integrating sphere, and baffles shall be of substantially equal reflectance, matt, and highly reflecting throughout the visible wavelength region. A highly reflecting matt sphere paint should be used.

### 3.3 Photocell

3.3.1 The radiant flux within the sphere shall be measured by a photo-electric cell, the output measurements of which shall be proportional within  $\pm 0.5$  per cent to the incident flux over the range of intensity used. Spectral conditions for source and receiver must be constant throughout the test of each specimen. The design of measuring instrument shall be such that there shall be a zero reading when the sphere is dark.

3.3.2 The spectral response of the photocell shall be corrected to approximate that of photopic vision.

3.4 The specimen shall be mounted normal to the beam.

## 4. Test Specimens

Specimens shall be described in Appendix 2.

## 5. Procedure

5.1 The apparatus shall be set up and allowed sufficient time to reach thermal equilibrium before measurements are made.

5.2 A reading (a) of the measuring instrument is made without the transparent specimen in place.

5.3 A second reading (b) is made with the specimen placed between the collimator and integrating sphere and adjacent to the entrance port thereof.

5.4 The procedure shall be repeated for each of two additional specimens.

## 6. Expression of Results

The percent visible light transmittance is calculated from:

$$\text{Percent visible light transmittance} = 100 \frac{b}{a}$$

## 7. Test Report

The test report shall include:

7.1 Individual values and average value of percent visible light transmittance for three specimens.

7.2 Average value of thickness of these three specimens.

## APPENDIX 4 DETERMINATION OF HAZE

### 1. Scope and Field of Application

This standard specifies the determination of haze of planar sections of transparent plastics employing a defined light source and a photometer with a spectral response corrected to approximate that of photopic vision.

### 2. Definition

Haze is defined as the scatter of light from an accumulation of tiny particles within the material, and/or from very small defects on the surface. This can lead to an obscuration of the view through the material or the spreading of an image beyond its proper limits.

### 3. Apparatus

The apparatus, as shown in Figure 5, consists of a voltage-stabilised light source with associated system to produce a collimated beam, a specimen holder and photometer mounted on a convenient optical bench such that the specimen may be moved on the optical axis of the system between light source and photometer.

#### 3.1 Light Source

3.1.1 The light source shall be a gas-filled tungsten filament lamp of photometric quality, operating at a colour temperature of  $2855 \pm 100\text{K}$ . The power supply to this lamp shall be stabilised to ensure short-term constant light output.

3.1.2 The light source is combined with an optical system to produce a parallel light beam of area at least  $1 \text{ cm}^2$  and of approximately circular cross-section.

#### 3.2 Integrating Sphere Photometer

3.2.1 The integrating sphere may be of any diameter exceeding 150 mm so long as the total port area does not exceed 2 percent of the internal reflecting area of the sphere. The axis of the irradiating beam shall pass through the centre of the entrance port and the centre of the integrating sphere. The photocell shall be positioned on the sphere 90° from the entrance port.

3.2.2 The irradiating light beam shall not be vignetted at the entrance port of the integrating sphere.

3.2.3 Reflecting surfaces - the surfaces of the interior of the integrating sphere and baffles shall be of substantially equal reflectance, matt, and highly reflecting throughout the visible wavelength region. A highly reflecting matt sphere paint should be used.

### 3.3 Photocell

3.3.1 The radiant flux within the sphere shall be measured by a photo-electric cell, the output measurements of which shall be proportional within  $\pm 0.5$  percent to the incident flux over the range of intensity used. Spectral conditions for source and receiver must be constant throughout the test of each specimen. The design of measuring instrument shall be such that there shall be a zero reading when the sphere is dark.

3.3.2 The spectral response of the photocell shall be corrected to approximate that of photopic vision.

3.4 The specimen shall be mounted normal to the beam.

## 4. Test Specimens

Specimens are to be as described in Appendix 1.

## 5. Procedure

5.1 The apparatus shall be set up and allowed sufficient time to reach thermal equilibrium before measurements are made.

5.2 A reading (b) is made with the specimen placed between the collimator and integrating sphere and at a distance equal to the diameter of the sphere from the entrance port thereof.

5.3 A further reading (c) is made with the specimen in contact with the entrance port of the integrating sphere where both incident and scattered light are received within the sphere. The relative positions of light source, collimator and sphere are not altered during the sequence of measurements.

5.4 The procedure shall be repeated for each of two additional specimens.

## 6. Expression of Results

The percent haze is calculated from:

$$\text{percent haze} = \frac{100(c-b)}{c}$$

## 7. Test Report

The test report shall include:

7.1 Individual values and average value of percent haze for these three specimens.

7.2 Average value of thickness of these three specimens.

### APPENDIX 5

#### DETERMINATION OF MOISTURE CONTENT OF POLYVINYL BUTYRAL

The moisture content of PVB may be determined by the Karl Fischer titration or the coulometric/electrolyte methods, both of which are accurate but lengthy.

For the routine determination of moisture in PVB materials, a reflectance type near infra-red moisture analyser has been found suitable. This method uses the ratio of a non-absorbing to an absorbing wavelength, typically 1.93 and 1.80  $\mu\text{m}$ . Moisture analysers of this type must be calibrated against one of the above methods. A daily calibration, using at least three reference standards, covering the range 0.2-0.5% moisture content must be performed. Three determinations shall be carried out and no result shall vary more than  $\pm 0.03\%$  from the mean.

Reference standards of known moisture content may be purchased or alternatively prepared by conditioning samples of PVB in a humidity controlled cabinet. Different moisture contents may be obtained by varying the relative humidity of the conditioning cabinet. On removal from the cabinet, the standard should not be touched with bare hands and must be immediately heat sealed in laminated metal foil bags to maintain a constant moisture content. The moisture contents of standards prepared by this technique must be determined by the Karl Fischer or coulometric electrolytic methods.

No results on these standards should vary from the mean of the three determinations by more than  $\pm 0.02\%$ .

Approved for Issue,

Dr D K Thomas,  
Head of Materials & Structures Dept  
Royal Aircraft Establishment,  
Farnborough.

microns

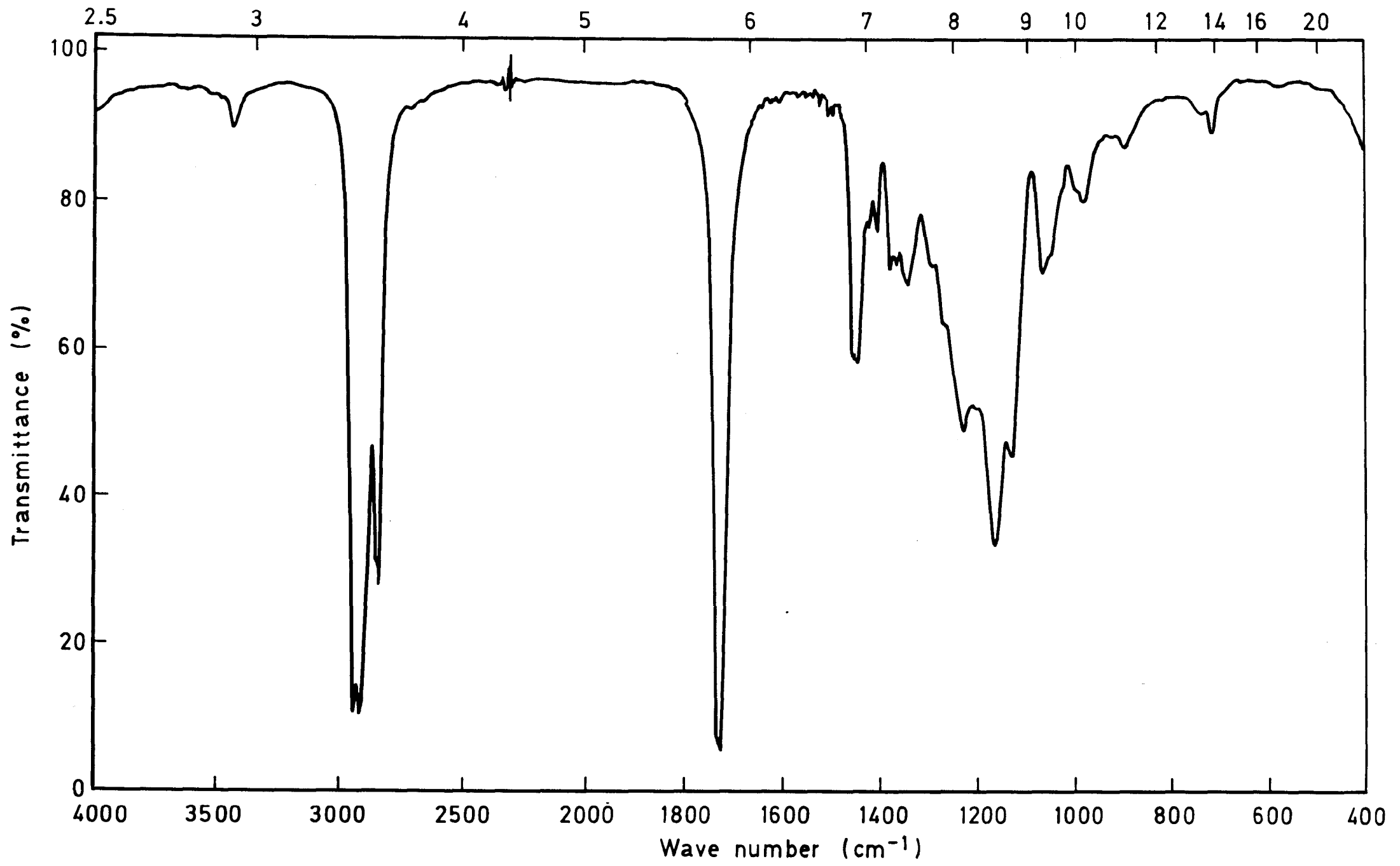
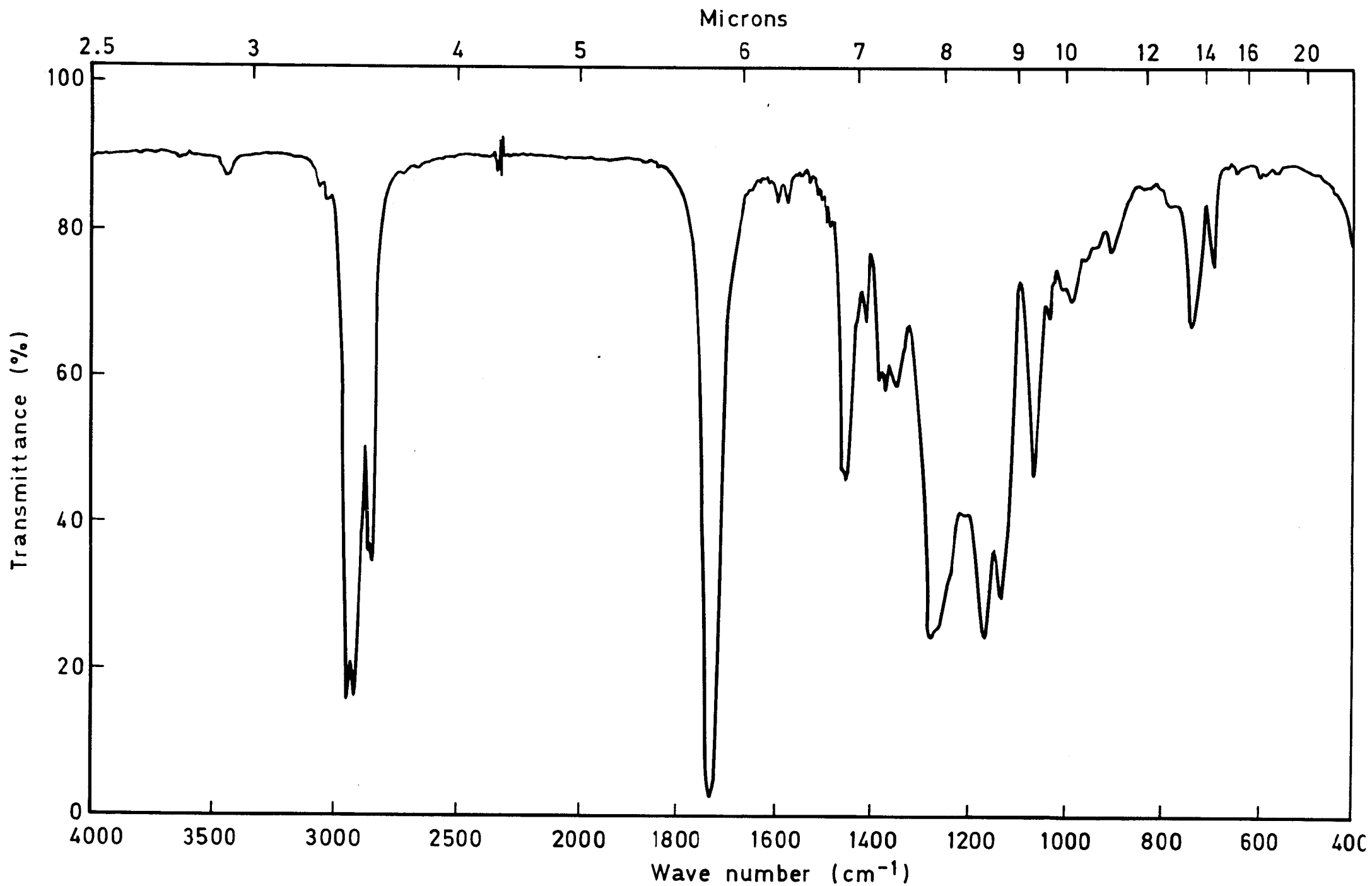
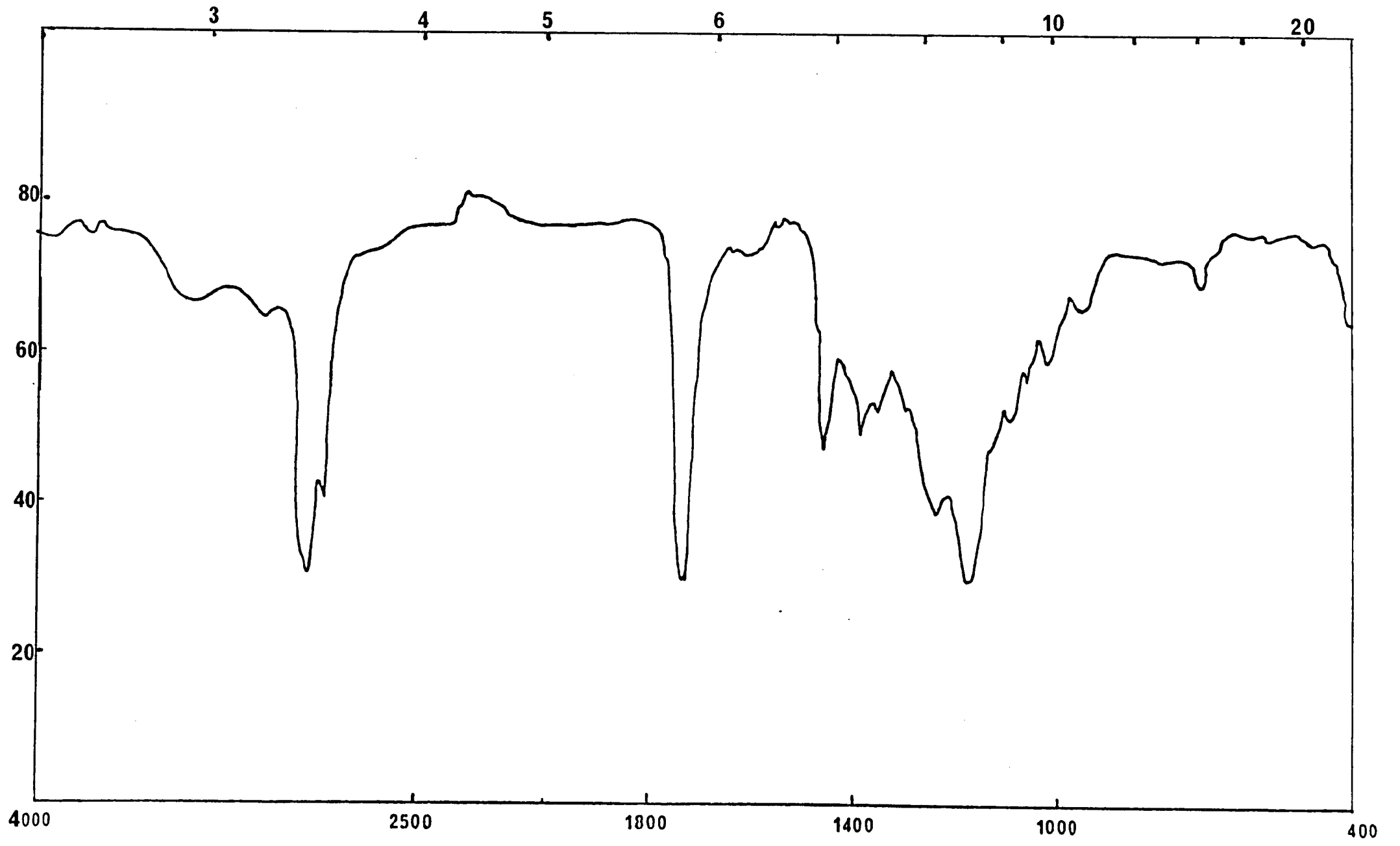


Fig 1 Di-Hexyl adipate





**Fig 2 Di-Hexyl adipate 82 wt % benzyl butyl phthalate 18 wt %**



Di-n-butyl sebacate (DBS)

FIGURE 3

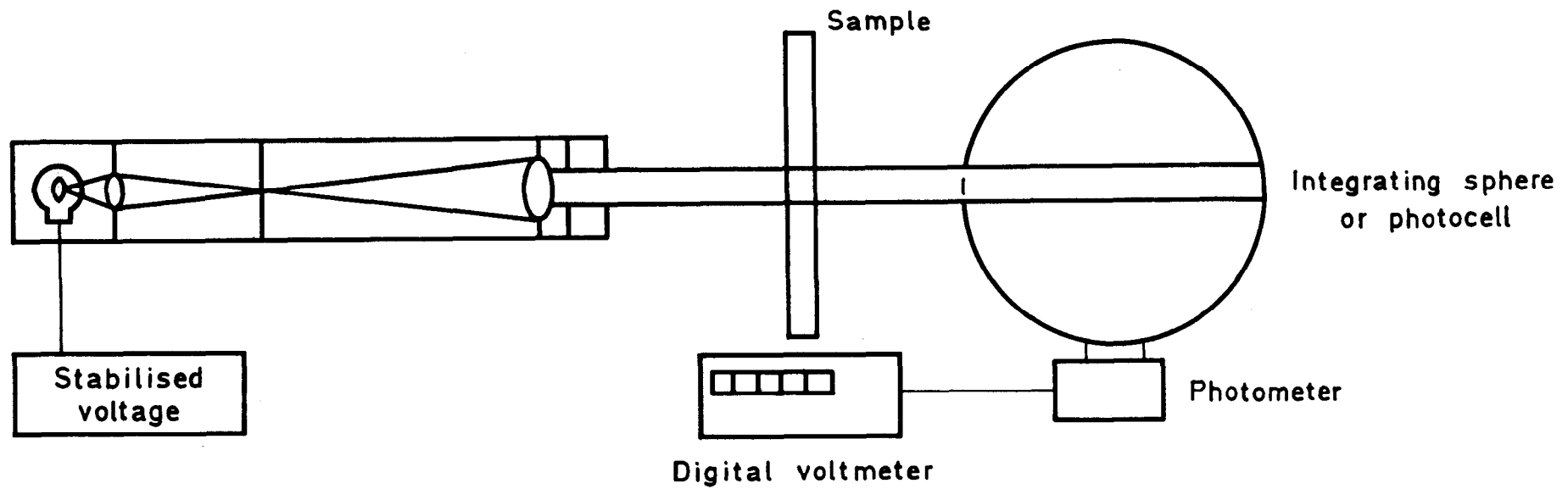


Fig 4 Measurement of in-line visual light transmission

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