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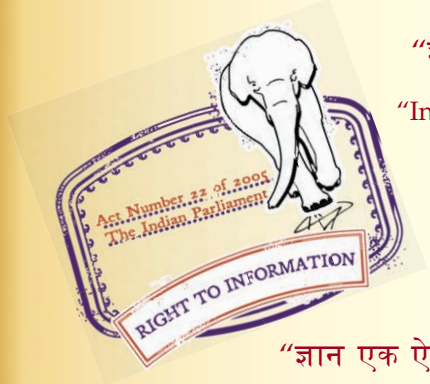
IS 7182 (1973): Methods of tests for aluminium collapsible tubes [MTD 32: Metal Containers]

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# METHODS OF TESTS FOR ALUMINIUM COLLAPSIBLE TUBES

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5

*July* 1974

# *Indian Standard*

# METHODS OF TESTS FOR ALUMINIUM COLLAPSIBLE TUBES

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# *Indian Standard*

# METHODS OF TESTS FOR ALUMINIUM COLLAPSIBLE TUBES

## 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 20 September 1973, after the draft finalized by the Metal Containers Sectional Committee has been approved by the Marine, Cargo Movement and Packaging Division Council.

**0.2** Following tests are recommended to assess the functional quality of collapsible tubes:

a) Collapsibility test

b) Leakage test ( test for closure effectiveness )

c) Lacquer curing test:

1) Power of adhesion test

2) Flexibility test

d) Lacquer porosity test

e) Lacquer-product compatibility test

f) External decoration test:

1) External decoration — Product compatibility test

2) Flexibility test

g) Metal particles test

**0.3** For the purpose of routine quality control of incoming supplies, the user and the manufacturer are advised to reach a prior agreement on quality standards and sampling procedures using IS : 2500 ( Part I )-1963* and IS : 2500 (Part II)-1965† respectively for attribute and measurable quality characteristics listed above.

**0.4** The leakage test ( *see* 3 ) is applicable only for tubes having the caps design to give leak-proof closure.

*Sampling Inspection Tables: Part I Inspection by attributes and by count of defects.

†Sampling Inspection Tables: Part II Inspection by variables for percent defective.

**0.5** As regards the lacquer porosity test ( *see* **5** ) extensive tests using the methods under **5.1** and **5.2** are being done with a view to determine whether the requirements of this test are attainable with the indigenous raw materials and till such a time a definite decision is taken, deviations from this test are not considered for rejection of supplies.

**0.6** The external decoration test ( *see* **7.2** ) may be considered only when decorations are specified to be alkali and soap resistant, such as toothpaste and shaving cream.

**0.7** The metal particle test ( *see* **8** ) is primarily meant for long nozzle eye ointment tubes.

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## 1. SCOPE

**1.1** This standard prescribes methods of tests and requirements for assessing the functional quality of aluminium collapsible tubes.

## 2. COLLAPSIBILITY TEST ( TEST FOR DEGREE ANNEALING )

**2.0 Principle** — A standard load is allowed to fall on the empty tube and the resistance offered to this load is measured.

**2.1 Apparatus** — The apparatus used shall be as detailed in Fig. 1. For tube of diameter 13·5 mm, the auxiliary base plate as shown in the figure shall be inserted in the apparatus.

**2.2 Procedure** — Place the tube on the groove, or in the case of tube of diameter 13·5 mm on top of the auxiliary base plate, on the base of the apparatus with the open end touching the stopper. Release the shutter and allow to fall upon the tube. Read on the scale the position of the top of the shutter.

**2.3 Acceptable Quality Levels** — When collapsible tubes are tested in this manner, the minimum and maximum limits for collapsibility readings which can be used for fixing suitable acceptable quality level ( AQLS ) between the manufacturer and the user shall be as shown below:

| *Diameter of Tube*, mm | *Collapsibility Readings*, mm | |
|---|---|---|
| | *Min* | *Max* |
| 13·5 | 7 | 10 |
| 16 | 4 | 6 |
| 19 | 5 | 8 |
| 22 | 6 | 10 |

| *Diameter of Tube*, mm | *Collapsibility Readings*, mm | |
|---|---|---|
| | *Min* | *Max* |
| 25 | 11 | 15 |
| 30 | 16 | 20 |
| 35 | 21 | 25 |
| 40 | 24 | 28 |
| 45 | 30 | 35 |

NOTE 1 — Use supplementary base plate for tube diameter 13·5 mm ( *see* Fig. 1 ).

NOTE 2 — This test is not applicable to tubes of diameter greater than 45 mm.

## 3. LEAKAGE TEST ( TEST FOR CLOSURE EFFECTIVENESS )

**3.0 Principle** — The tube is tightly closed and placed in an inverted position. Water absorbed by the filter paper placed around the base of the nozzle will indicate leakage.

**3.1 Procedure** — Fill the tightly closed tube with water. Wipe the external surface of the tube thoroughly to make it dry. Place the tube in an inverted position with a filter paper fixed at the base of the cap. Allow the tube to stand for one hour.

**3.2 Requirement** — The filter paper shall not show any absorption of water at any time during the one hour test period.

## 4. LACQUER CURING TEST

### 4.1 Power of Adhesion Test

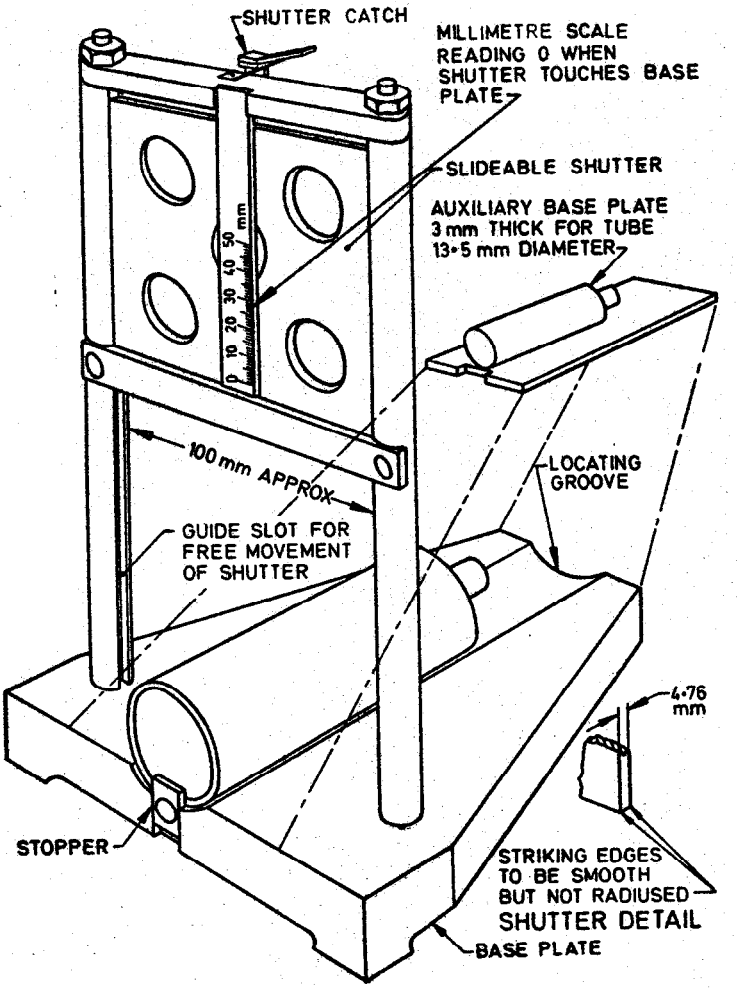
**4.1.0** *Principle* — The curing of lacquer layer and its power of adhesion to the internal tube surface is determined by rubbing the lacquered surface with cotton wool soaked in acetone.

**4.1.1** *Procedure* — Split a tube along the length and flatten it. Dip cotton wool in acetone and rub it on lacquered surface of the flattened tube 20 times.

**4.1.2** *Requirement* — Lacquer shall not lift from the surface and the cotton wool shall remain colourless.

### 4.2 Flexibility Test

**4.2.0** *Principle* — This test determines flexibility, that is, resistance to flakes cracking of internal lacquer.




| | |
|---|---|
| Height bottom of shutter from base plate | 108 mm |
| Groove depth | $5 \pm 0{\cdot}2$ mm |
| Groove radius | 32 mm |
| Distance between stopper and plane of shutter | 32 mm |
| Shutter weight | $70 \pm 1$ g |

FIG. 1 COLLAPSIBILITY TEST APPARATUS

**4.2.1** *Procedure* — Cut the tube from the open end and make it flat. Fold the tube in such a manner that the internal lacquered surface is on the outside. Sharpen the fold by passing it between the finger and finger nail. Repeat the procedure three times at different places of the tube.

**4.2.2** *Requirement* — The lacquer coating must not peel off when the sharpened fold is rubbed with the fingers.

## 5. LACQUER POROSITY TEST

### 5.1 Current Through-Flow Method

**5.1.0** *Trinciple* — This test is designed to assist in the quantitative determination of the porosity of the internal coating by determining the amount of flow of electric current which is proportional to the area of the pores on the surface.

**5.1.1** *Apparatus* — The diagram of the instrument is given in Fig. 2 and the wiring diagram to make clear the construction of instrument is given in Fig. 3.

**5.1.1.1** The instrument shall be capable of giving readings within three ranges of current, namely, 0-5, 0-50 and 0-500 mA with a range selector switch for selecting the appropriate range.

**5.1.1.2** The apparatus shall have three fuses to protect the instrument. The 1A fuse situated on the back of the instrument, next to the mains on-off switch is the mains fuse, and the 50mA and 500mA fuses on the front panel protect the ammeter from being overloaded.

**5.1.2** *Test Solution* — The solutions of following composition shall be used for the test:

| | |
|---|---|
| Copper sulphate ( CuS04. $5H_20$ ) | 1·0 percent ( *m*/*v* ) |
| Glacial acetic acid | 0·05 percent ( *m*/*v* ) |
| Sodium dioctylsulphosuccinate | 0·005 percent ( *m*/*v* ) |
| Distilled water | 98·945 percent ( *m*/*v* ) |

**5.1.3** *Procedure*

**5.1.3.1** Ascertain the correct mains voltage and adjust the transformer tapping accordingly. Connect the 3-pin plug to the mains supply and switch on the mains switch, indicated by the glow of red lamp on the front panel. Check the circuit by switching on 5, 50 and 500mA positions. Switch off the check switch, and the instrument is ready for use.

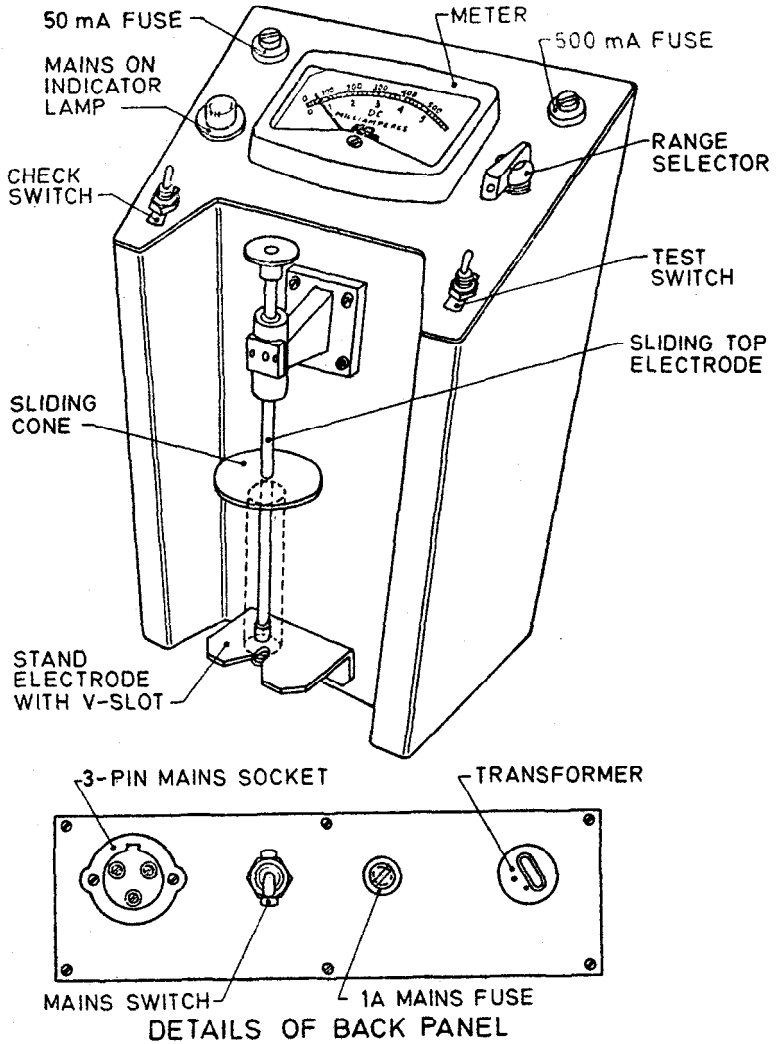



FIG. 2 INSTRUMENT FOR TESTING CONTINUITY OF INTERNAL COATING

**5.1.3.2** Set the range selector switch to 500mA before commencing the readings in order to safeguard the meter from overloading. Fit the tube to be tested with cap and fill the tube to within 25 mm of the open end with the test solution. Fit the nozzle shoulder portion of the tube to the V-slot in the stand electrode and lower the sliding top electrode into the

tube. The sliding cone height shall be adjusted automatically, the cone holding the tube coaxially with the top electrode. Press the test switch and check the appropriate range for test as quickly as possible. Switch the range selector to the range and take the reading after 5 seconds. The same solution shall not be used more than six times.

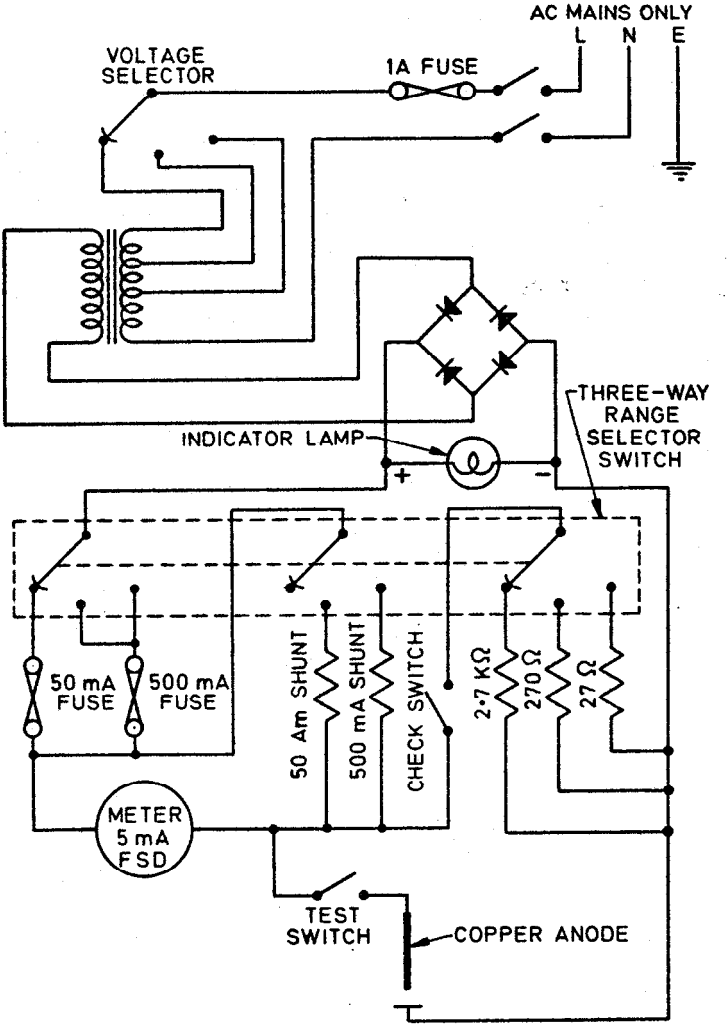



FIG. 3 WIRING DIAGRAM

**5.1.4** *Requirement* — Lacquered tubes when tested in this manner shall comply with the requirements specified below:

| *Tube Diameter*, mm | *Permissible Current Flow*, mA, *Max* |
|---|---|
| 13·5 | 75 |
| 16 | 80 |
| 19 | 85 |
| 22 | 90 |
| 25 | 95 |
| 30 | 100 |
| 35 | 105 |
| 40 | 110 |

NOTE 1 — These values of current should be regarded as upper specification limits when variable sampling inspection plans are agreed between the manufacturer and the user according to the procedure given in IS : 2500 (Part II)-1965*.

NOTE 2 — The permissible current value for dia 45 mm will be included at later stage.

## 5.2 Electrolytic Deposition of Copper Method†

**5.2.0** *Principle* — The pores in the lacquer layer are detected by electrolytic method with the help of direct current of a certain voltage and ampere. Spongy copper is deposited on the pores from the copper sulphate solution.

**5.2.1** *Test Solution* — The solution of following composition shall be used for the test:

| | |
|---|---|
| Copper sulphate ( $CuS04.5H_20$ ) | 5·0 parts ( *m/v* ) |
| Glacial acetic acid | 0·5 parts ( *m/v* ) |
| Distilled water | 94·5 parts ( *m/v* ) |

**5.2.2** *Source of Current* — Direct current of 6 volts and a maximum of 2·5 amperes from a battery charger shall be used.

**5.2.3** *Procedure* — Split open the tube and flatten it. Bend up about 1 cm of each of the four sides and squeeze the corners together in such a manner that an internally lacquered through is formed. The size of the trough should be constant for all tubes of the same size. Fill the trough with the test solution. Connect the negative pole of the current with the

*Sampling inspection tables: Part II Inspection by variables for percent defective.

†The test may be carried out if agreed between the purchaser and supplier and it is not a mandatory test.

aluminium side of the tube. Bend a copper wire approximately 4 mm in diameter so as to run parallel to the lacquer surface and extend over at least half the length of the trough. Attach the wire to the positive pole and immerse into the test solution for 10 seconds.

A dark deposit of copper is formed at porous points. To evaluate the porosity in the neck and shoulder region, screw the cap on and cut the tube round up to a height of about 2 cm above the shoulder, leaving a small projection to connect the negative pole. Fill the tube with solution and dip the copper wire inside almost to the nozzle.

5.2.4 *Requirements* — If no copper is deposited, the tube is entirely free from pores. The maximum number of porosity deposits for each diameter of tube shall be as given below:

| *Tube Diameter*, mm | *Maximum Number of Porosity Deposits of Size 0·5* mm *Diameter and Above* |
|---|---|
| 13·5 | 5 |
| 16 | 10 |
| 19 | 10 |
| 22 | 15 |
| 25 | 15 |
| 30 | 20 |
| 35 | 20 |

## 6. LACQUER—PRODUCT COMPATIBILITY TEST

**6.0 Principle** — The lacquer is brought in contact with the product to be packed and maintained at a fixed temperature and time. The lacquer is visually examined for any damage. With this test, a suitable control test in a stoppered neutral glass test tube may be carried out simultaneously.

**6.1 Procedure** — Take ten tubes for testing. Fill the product in tightly stoppered tubes. Crimp the open end of the tubes properly. Subject these tubes to 45°C in an oven for a period of 72 hours. After this period the tubes are allowed to cool to room temperature and then are carefully cut open lengthwise.

**6.1.1** *Product Compatibility* — The contents of the tube should not show any discolouration, change in odour, gas formation or signs of decomposition compared to control sample kept in the sealed neutral glass test tube and subjected to identical conditions at the same time.

**6.1.2** *Lacquer Compatibility* — After the observation in **6.1.1** is recorded, the contents are washed from the spread open tube body with water at about 45°C. The lacquer coat is dried with cotton wool avoiding rubbing and scratching.

6.1.3 *Requirement* — There shall be no visible signs of softening with scratching, lifting, peeling of lacquer when the lacquered surface is examined in each tube.

NOTE 1 — The test given in 6 is primarily meant to detect only gross irregularities in the quality of lacquer or lacquering process. The manufacturer and the purchaser are advised to conduct long term storage trials to examine the compatibility of the product with the lacquer in a more realistic manner.

NOTE 2 — These spread open tubes may be used in carrying out lacquer curing test (4) and lacquer porosity test (5) if agreed to between the user and supplier.

## 7. EXTERNAL DECORATION

### 7.1 External Decoration — Product Compatibility Test

7.1.0 *Principle* — The external coating is brought in contact with an alkaline solution and kept at a fixed temperature for a fixed time. The coating is examined for any damage.

7.1.1 *Procedure* — Take ten tubes for testing. Prepare 2 percent solution of pure sodium hydroxide in water and keep at 40°C. Fold the bottom portion of the tube so as to cover the undecorated portion. Immerse the tubes in the solution for 15 minutes. Wash the tubes after removal from the alkaline solution.

7.1.2 *Requirement* — There shall be no blistering or loosening of external decoration or bleeding of coating or inks to any noticeable extent.

NOTE — The test described in 7·1 is for general testing of decorated collapsible tubes. For testing specific product compatibility, ten tubes should be tested. Bottoms of the tubes should be folded and crimped and bodies smeared with the respective products and kept horizontally for 48 hours at 40°C and 90 percent relative humidity. On washing the tubes after 48 hours, they should not show any sign of loosening of the external decoration or bleeding of coating or inks to any noticeable extent.

### 7.2 Flexibility Test

7.2.0 *Principle* — This test determines the adhesion and resistance to flex-cracking of the coating.

7.2.1 *Procedure* — Cut the tube from the open end and make it flat. Fold the tube in such a manner that the external surface is on the outside. Sharpen the fold by passing it between the finger and finger nail. Repeat the procedure three times at different places of the tube.

7.2.2 *Requirement* — The coating on the external surface shall not peel off when the fold is rubbed with the finger.

## 8. METAL PARTICLE TEST

**8.0 Principle** — It includes filling the tube with molten eye ointment basis and cooling at a fixed temperature overnight. The ointment is squeezed out of the tubes and passed over a special filtering medium. The metal particles collected over the filter are observed through a magnifying glass and particles counted.

**8.1 Apparatus**

**8.1.1** *Extrusion Apparatus* — A suitable apparatus is shown in Fig. 4 and 5. It consists of block of about 190 mm × 100 mm made of a hard material (for example, phenol formaldehyde laminated or polymethyl methacrylate) 25 mm thick, through one short end of which is drilled longitudinally a 12·5 mm diameter hole so that it can be mounted on a standard laboratory retort stand. The other short end of the block should be curved in such a way that the spring roller moves smoothly over it when the handle is pulled vartically downwards. A slotted stop is required at the lower end of the roller's travel, the nozzle of the tube under test being held in this slot whilst the closed end of the tube is gripped between the roller and the block.

Apart from the block all parts should be made of brass with the exception of the roller bearing and the springs which should be steel.

NOTE 1 — In use of the handle should be pulled down gently to avoid the possibility of the ointment being forced past the roller or bursting the tube.

NOTE 2 — Care should be taken to avoid scoring the outside of the tube during the extrusion of the ointment.

**8.1.2** *Metal Bacteriological Filter* — With a 4·25 cm Whatman No. 541 filter paper supported on a suitable perforated plate in place of the standard sintered carbon disc.

**8.1.3** *Magnifying Glass* — With a graticule of 1 mm squares one of which is sub-divided into 0·2 mm squares.

**8.2 Procedure** — Select a sample of 50 tubes from the lot to be tested, and clean each tube by vibration and/or 'blowing'. Fill the tubes with molten eye ointment basis by any suitable method (*see* Note), close the open end of the tube by a double fold, and allow the filled tubes to cool overnight at room temperature (15-20°C).

NOTE — Precautions should be taken to ensure that metal particle are not introduced into the ointment base during the filling operations or from any other source.

**8.2.1** Heat the filtering apparatus in an oven at 80°C for 10 minutes or by circulation of hot water in a heating jacket surrounding the filter.

**8.2.2** Remove the caps from the cooled tubes and apply uniform pressure, either manually or by means of the apparatus described in **8.1** or other suitable machine, to the closed end of each tube in such a manner

that the time taken to express as much of the basis as possible through each nozzle is not less than 20 seconds in each case. Collect the extruded basis from the fifty tubes in the heated filter, applying suction to the stem of the filter in order to draw the molten basis through the filter paper. When all the melted basis has been removed, wash the walls of the filter and the filter paper with three successive 30 ml portion of chloroform, allow the filter paper to dry, and immediately mount it between glass for examination.

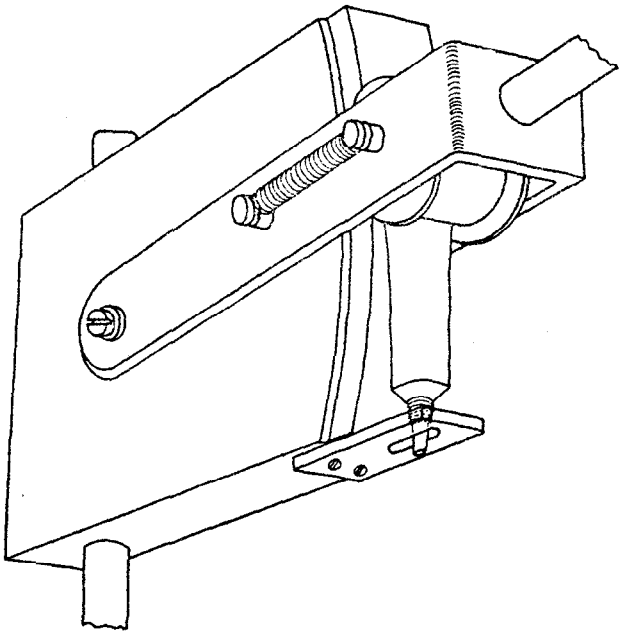


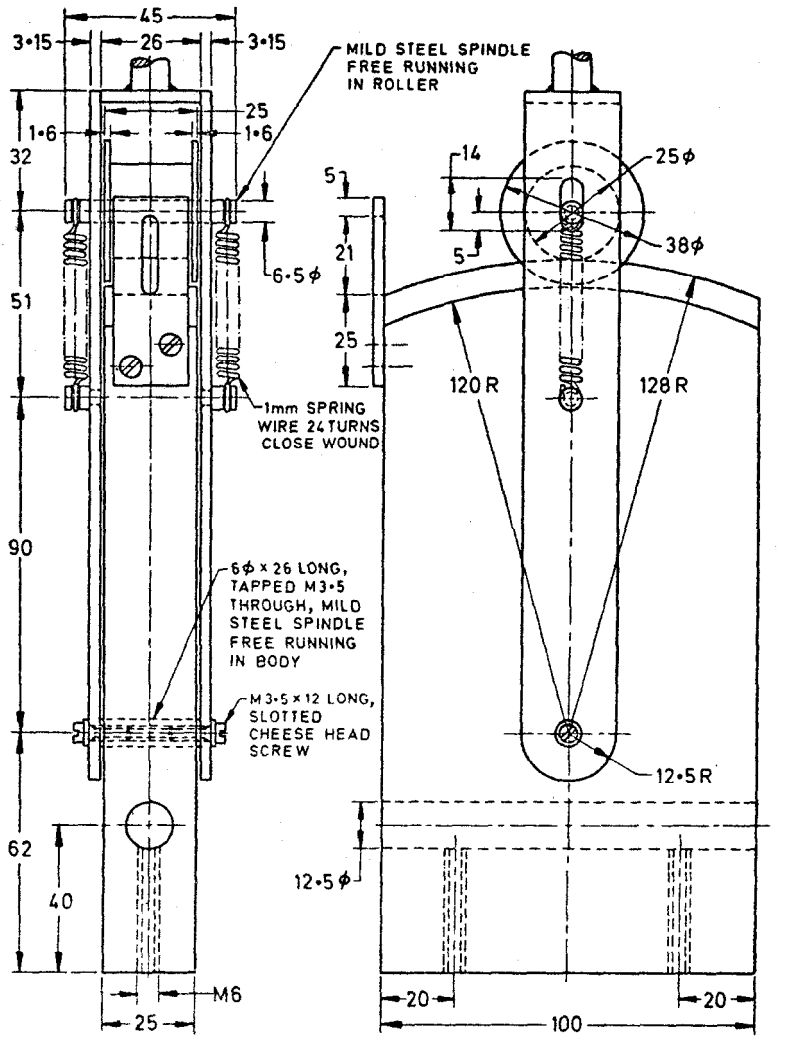
FIG. 4 EXTRUSION APPARATUS ASSEMBLY

**8.2.3** Examine the filter paper under oblique lighting with the aid of the magnifying glass and note the following:

a) The number of all metal particles 1 mm in length and longer,

b) The number in the range of 0·5 mm to less than 1 mm, and

c) The number in the range of 0·2 mm to less than 0·5 mm.




All dimensions in millimeters.

FIG. 5 DETAILS OF EXTRUSION APPARATUS

**8.2.4** Carry out two further examinations with the filter paper in two different positions, so that the lighting comes from different directions, and calculate the average number of metal particles counted in each of the three ranges specified.

**8.3 Expression of Results** — The metal particle content is assessed by giving each metal particle detected on the filter paper a score as follows and adding the scores together:

| | |
|---|---|
| Particles 1 mm and above | 50 |
| Particles 0·5 mm but less than 1 mm | 10 |
| Particles 0·2 mm but less than 0·5 mm | 2 |
| Particles less than 0·2 mm | Nil |

**8.3.1** If the total score is less than 100 points, the batch of tubes passes the test; if the total score is more than 150 points the batch fails the test. If the total score is between 100 and 150 points both inclusive, the test is repeated on a further sample of 50 tubes and the batch passes the test if the sum of the total scores in the two tests is less than 150 points.

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