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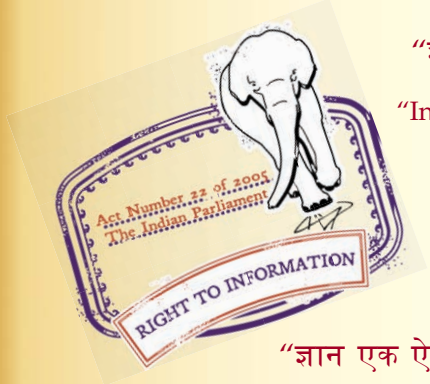

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METHOD OF SUB-DIVISION OF GROSS SAMPLE OF POWDER USED FOR DETERMINATION OF PARTICLE SIZE

UDC 620·168·36 : 621-492 : 66-492

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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February 1969

Indian Standard

METHOD OF SUB-DIVISION OF GROSS SAMPLE OF POWDER USED FOR DETERMINATION OF PARTICLE SIZE

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METHOD OF SUB-DIVISION OF GROSS SAMPLE OF POWDER USED FOR DETERMINATION OF PARTICLE SIZE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 18 November 1968, after the draft finalized by the Sieves, Sieving and Other Sizing Methods Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Accurate sampling for particulate material is an essential prerequisite for determination of size distribution, especially when using methods in which the test portion may be relatively small in amount.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by basing the standard on B.S. 3406: Part I : 1961 'Methods for the determination of particle size of powders: Part I Sub-division of gross sample down to 0·2 ml' issued by the British Standards Institution.

1. SCOPE

1.1 This standard describes the following methods of sub-division of gross sample down to the required quantity for further analysis for determination of particle size distribution:

a) Reduction by halving from over 1 500 to 500 litres,

b) Reduction by coning and quartering from 1 000 or less to 25 litres,

c) Reduction by turntable type sample divider from over 1 500 to 1 litre,

d) Reduction by slotted cone type sample divider from over 1 500 to 1 litre,

e) Reduction by large oscillating hopper type sample divider from 100 to 1 litre,

f) Reduction by grid type sample divider from 50 litres to 5 millilitres, and

g) Reduction by small oscillating hopper type sample divider from 1 litre to 0·2 millilitres.

1.2 This standard also covers method for checking the performance of sample divider.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions and those given in IS : 4124-1967* shall apply.

2.1 Increment — The quantity of material taken by a single motion of a sampling instrument, such as a scoop.

2.2 Gross Sample — The sum total of the increments.

2.3 Laboratory Sample — The portion of the gross sample which is delivered to the laboratory for determination of particle size distribution.

2.4 Analysis Sample — The portion of the laboratory sample which is used in the size analysis apparatus.

3. METHODS OF SUB-DIVISION

3.1 Methods described in **3.1.1** to **3.1.8** may be used for sub-division of gross sample to the required quantity.

3.1.1 *Method I Reduction by Halving from Over* 1 500 *to* 500 *Litres*

3.1.1.1 *Procedure*

a) Shovel the material into a conical pile over a smooth hard surface, each shovelful being placed on the top of the preceding one, while depositing material, care shall be taken that the pile is formed by depositing material uniformly and systematically so that apex of the cone is not displaced.

b) A long pile is then formed as given below:

1) Fill the shovel with material from the base of the cone and spread it along a ribbon to a length of two to three metres. The initial width of the ribbon being equal to the width of the shovel.

2) Take the next shovelful from the base of the cone from some other point and spread it over the top of the first shovel but in an opposite direction.

3) Repeat the procedure until one long pile is formed. The width of the pile will depend upon the angle of repose of the material.

*Glossary of terms relating to powders.

c) Discard half the pile formed above in the following manner:

1) Take a shovelful from one end of the pile from the bottom and put it to one side.

2) Take next shovelful adjacent to the first from the pile by advancing along the side of the pile to a distance equal to the width of the shovel. Discard this shovelful.

3) Take the next shovelful in the manner as described for (2) above and mix it with the first shovel.

4) Repeat the procedure by taking shovelful, always advancing along and then around the pile in the same direction and discarding alternate shovelful so that the pile is decreased gradually and uniformly.

d) Repeat the procedure until about 500-700 litres of material is retained.

3.1.1.2 *Precautions* — The method shall be carried out in a closed space, free from draughts and air currents, to prevent possible loss of fine particles.

3.1.2 *Method II Reduction by Coning and Quartering from* 1 000 *or less to* 25 *Litres*

3.1.2.1 *Procedure*

a) Shovel the material into a conical pile over a smooth hard surface, each shovelful being placed on the top of the preceding one. While depositing material, care shall be taken that the pile is formed by depositing material uniformly and systematically so that the apex of the cone is not displaced.

b) Flattened the cone with the back of the shovel. While flattening, the shovel shall be given rotary motion. Care shall be taken that the flattened cone has uniform diameter and height.

c) The pile shall then be divided into quarters with the help of a sheet-metal cross made with four blades joined together at the centre at right angles to each other. The height of the blade should be greater than the height of the cone. The metal cross shall be placed centrally on the heap and pressed down to pass through the material.

d) After quartering as above, two diagonally opposite quarters will be removed and the space occupied by them cleaned keeping the metal cross in position.

e) The remaining two quarters shall then be mixed, coned and quartered again using the same method till the remaining material is between 25 and 50 litres.

3.1.2.2 *Precautions* — The method shall be carried out in an enclosed space, free from draughts or air currents, to prevent possible loss of fine particles.

3.1.3 *Method III Reduction by Turntable Type Sample Divider from Over* 1 500 *to* 1 *Litre*

3.1.3.1 *Apparatus* — The apparatus consists of the following parts (*see* Fig. 1):

a) *Conical feed hopper* — stationary, fed by a shovel of width less than its top diameter, and fitted with an interchangeable orifice at the bottom. The size of the orifice is such that the time taken by the sample to pass through is not less than one minute. The feed hopper should be able to be readily lifted or pivoted for access to the receiver. The hopper may be on the vertical axis of the receiver, feeding via a distribution cone, or off-centre when no such cone is required.

b) *Receiver* — rotating, fixed on a turntable rotating at a constant speed not less than 30 rev/min, containing removable segmented sample containers each covering at least $\frac{1}{24}$ of the area of the turntable.

c) *Segments for collecting samples* — The receiver should contain only those segments which are needed to collect the required amount of sample. A useful set of segments comprises of three $\frac{1}{4}$ two $\frac{1}{12}$ and one $\frac{1}{24}$ segment. Such a set will give $\frac{1}{2}$, $\frac{1}{4}$, $\frac{1}{6}$, $\frac{1}{8}$, $\frac{1}{12}$ and $\frac{1}{24}$ fractions. These segments should be made with a lip on one side to clip on to the adjacent segment of the diameter strip (clipping prevents dust passing to the receiver).

3.1.3.2 *Procedure* — The following procedure shall be adopted for reduction of sample:

a) A suitable hopper orifice shall be selected to give a total time of flow of at least one minute.

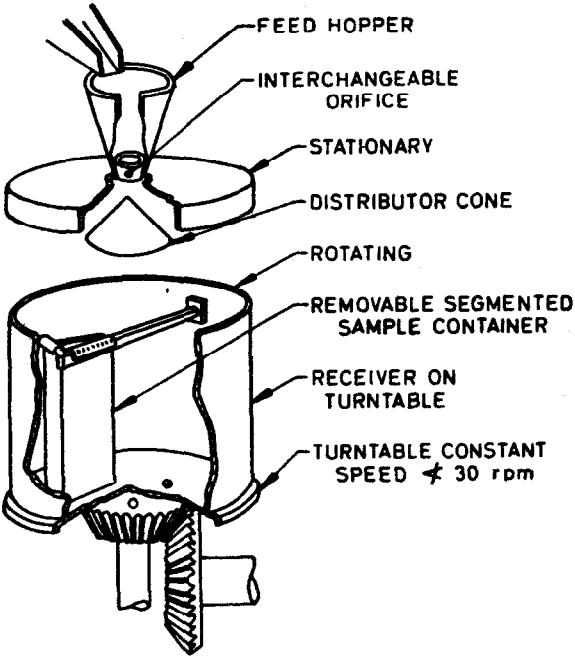
b) The requisite segments shall be inserted in the receiver.

c) The turntable shall now be rotated at a constant speed.

d) Gross sample shall now be put into the hopper.

e) When whole of the sample has passed into the receiver, turntable is stopped and the desired sample is withdrawn. The rest of the sample shall be discarded.

NOTE — In case the gross sample is in excess of the volume of the receiver, it may require further operations, each operation filling full volume of the receiver. Fractions from all *n* operations are combined and procedure repeated again till the required amount is obtained.



NOTE — Material shall be any suitable metal.

Dimensions — For a final sample of 1 litre, a convenient size for the receiver is about 30 cm diameter and the same height, the sample container being about $\frac{3}{4}$ of this height. If required the linear dimensions may be increased up to $1\frac{1}{2}$ times these values, above which handling becomes cumbrous.

FIG. 1 TURNTABLE TYPE SAMPLE DIVIDER

3.1.3.3 *Precautions*

a) No component of the apparatus which is in contact with the powder shall be made of plastics.

b) The method shall be conducted in an enclosed space, free from draughts or air currents, to prevent loss of fine particles.

3.1.4 *Method IV Reduction by Slotted Cone Type Sample Divider from Over* 1 500 *to* 1 *Litre*

3.1.4.1 *Apparatus* — The apparatus consists of the following parts (*see* Fig. 2):

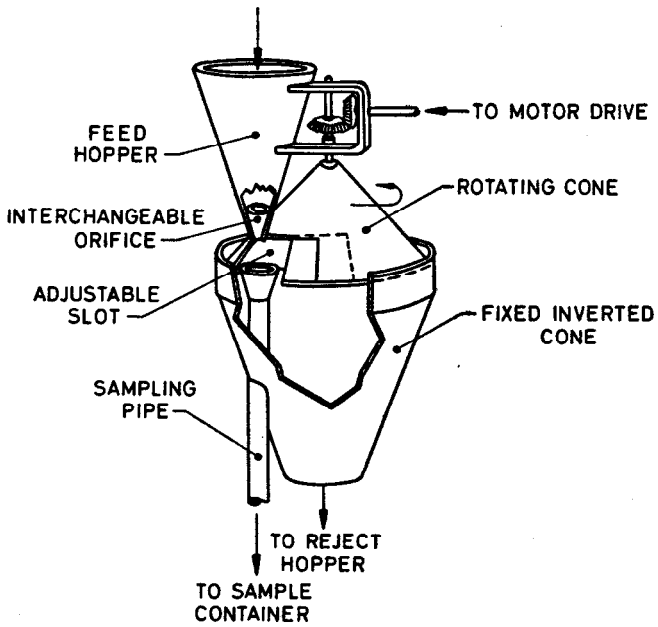
a) *Conical feed hopper* — stationary, fed by a shovel of width less than its top diameter and fitted with an interchangeable orifice at the

bottom. The size of the orifice is such that the time taken by the sample to pass through is not less than one minute.

b) *Sampling pipe* — stationary, placed just below the feed hopper so that any material passing through the feed hopper passes down the sampling pipe.

c) *Rotating cone* — rotating at a speed not less than 30 rev/min is placed between the hopper and the sampling pipe. The cone is provided with adjustable slot or slots. The material from the hopper passes down the sampling pipe whenever slot of the cone is between them, otherwise the material is diverted away.

d) *Fixed inverted cone* — is provided surrounding the rotating cone and collects material from the surface of the later.



NOTE — Material shall be any suitable metal.

Dimensions — For a final sample of 1 litre, a convenient size for the rotating cone is 10 cm maximum diameter.

FIG. 2 SLOTTED CONE TYPE SAMPLE DIVIDER

3.1.4.2 *Procedure* — The following procedure shall be adopted for reduction of sample:

a) A suitable hopper orifice shall be selected to give a total time of flow of at least one minute.

b) The slots shall be adjusted.

c) The rotating cone shall then be rotated at the fixed speed.

d) Gross sample shall then be put into the hopper.

e) The procedure shall be repeated for a part of the gross sample that has passed through the sampling pipe and has collected in the sampling container until the desired size is reached.

3.1.4.3 *Precautions*

a) No component of the apparatus which is in contact with the powder will be made of plastics.

b) The method shall be conducted in an enclosed space, free from draughts or air currents, to prevent loss of fine particles.

3.1.5 *Method V Reduction by Large Oscillating Hopper Type Sample Divider from* 100 *to* 1 *Litre*

3.1.5.1 *Apparatus* — Large oscillating hopper type sample divider consists of the following parts (*see* Fig. 3 and 4).

a) *Oscillating hopper* — The hopper oscillates at about 200 cycles per minute. Oscillation is achieved by means of a crank turned by an electric motor through belt drive.

b) *Receiving hopper* — The material from the oscillating hopper is divided into two parts and is collected into two receiving hoppers.

c) *Interchangeable orifice* — The oscillating hopper is fitted with an interchangeable orifice to control rate of flow of material into the receiving hopper.

3.1.5.2 *Procedure* — The following procedure shall be adopted for reduction of the sample:

a) A hopper orifice is selected to give a total flow time for the gross sample of at least a minute.

b) The relative position of the oscillating and receiving hoppers is adjusted until the material received by the two receiving hoppers is equal in quantity to within 2 percent.

c) The oscillation is started.

d) The gross sample is shovelled into the oscillating hopper.

e) When it has passed into the receiver, the material from one hopper is discarded, that from the other is again divided as above. Next time the material from the other hopper is discarded, the discard

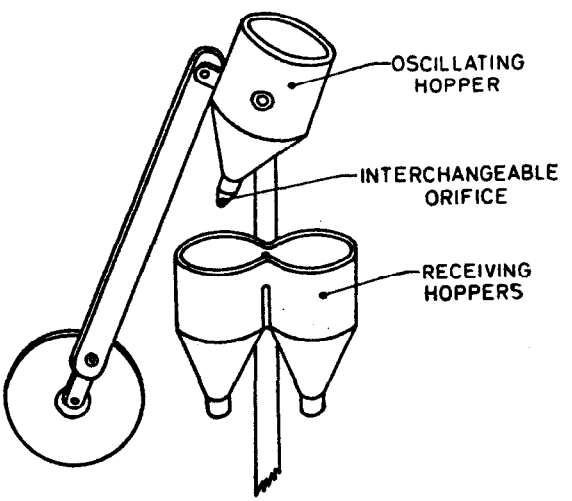
and re-division being made on the material from the alternate hoppers, until the required fraction is obtained.

3.1.5.3 *Procedure with slurries* — Slurries may also be divided by this method, using the same procedure. A small orifice will be needed.

3.1.5.4 *Precautions*

a) No component of the apparatus which is in contact with the powder or slurry as the case may be, will be made of plastics.

b) The method shall be conducted in an enclosed space, free from draughts or air currents, to prevent loss of fine particles. The appearance of dust on parts of the apparatus outside the collecting zone indicates that loss of fine particles is occurring and steps should be taken to remove the cause.

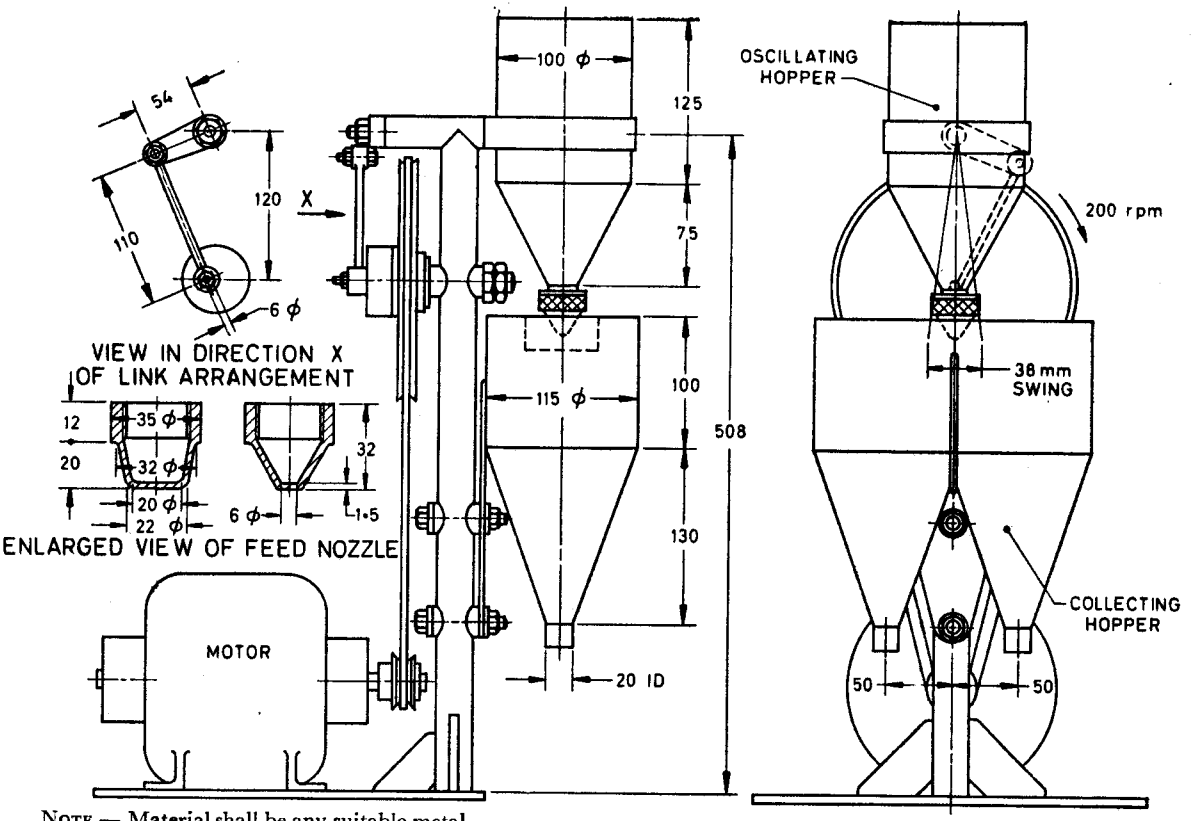


NOTE — Material shall be any suitable metal.

Dimensions for large type — For a final sample of 1 litre, the oscillating hopper should be about 100 mm diameter, and 125 mm cylindrical depth plus 75 mm conical depth. Its nozzle should swing through an arc about 40 mm long. The receiving hoppers should be of roughly the same dimensions.

Dimensions for small type (*Method VII*) — For a final sample of 0·2 ml, the oscillating hopper should be about 50 mm diameter, and 40 mm cylindrical depth plus 25 mm conical depth. A convenient size for the receiving hoppers is 25 mm diameter, 25 mm cylindrical depth and 20 mm conical depth.

FIG. 3 OSCILLATING HOPPER SAMPLE DIVIDER



NOTE — Material shall be any suitable metal.

All dimensions in millimetres.

FIG. 4 LARGE OSCILLATING HOPPER SAMPLE DIVIDER

3.1.6 *Method VI Reduction by Grid Type Sample Divider from* 50 *Litres to* 5 *Millilitres*

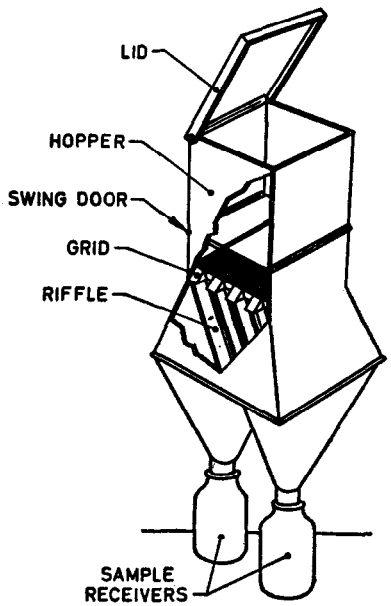
3.1.6.1 *Apparatus* — Grid type sampler consists of the following parts (*see* Fig. 5, 6 and 7):

a) *Grid* — with 64 passages, 8 row each of 8 passages. Alternate passages in each row being inclined in the opposite direction.

b) *Riffle* — is provided below the grid with nine sections as shown in Fig. 5.

c) *Hopper* — is provided with a lid to avoid loss of dust as shown in Fig. 5.

d) *Swing doors* — are provided for even distribution of material over the grid.



NOTE — Chromium plated sheet brass is suitable as material since it provides a good surface to allow the powder to slide quickly through. A convenient thickness is about 0·9 mm.

Dimensions — For a final sample of 5 ml, a convenient grid has 64 (8×8) apertures, each of 11 mm square.

FIG. 5 GRID TYPE SAMPLE DIVIDER

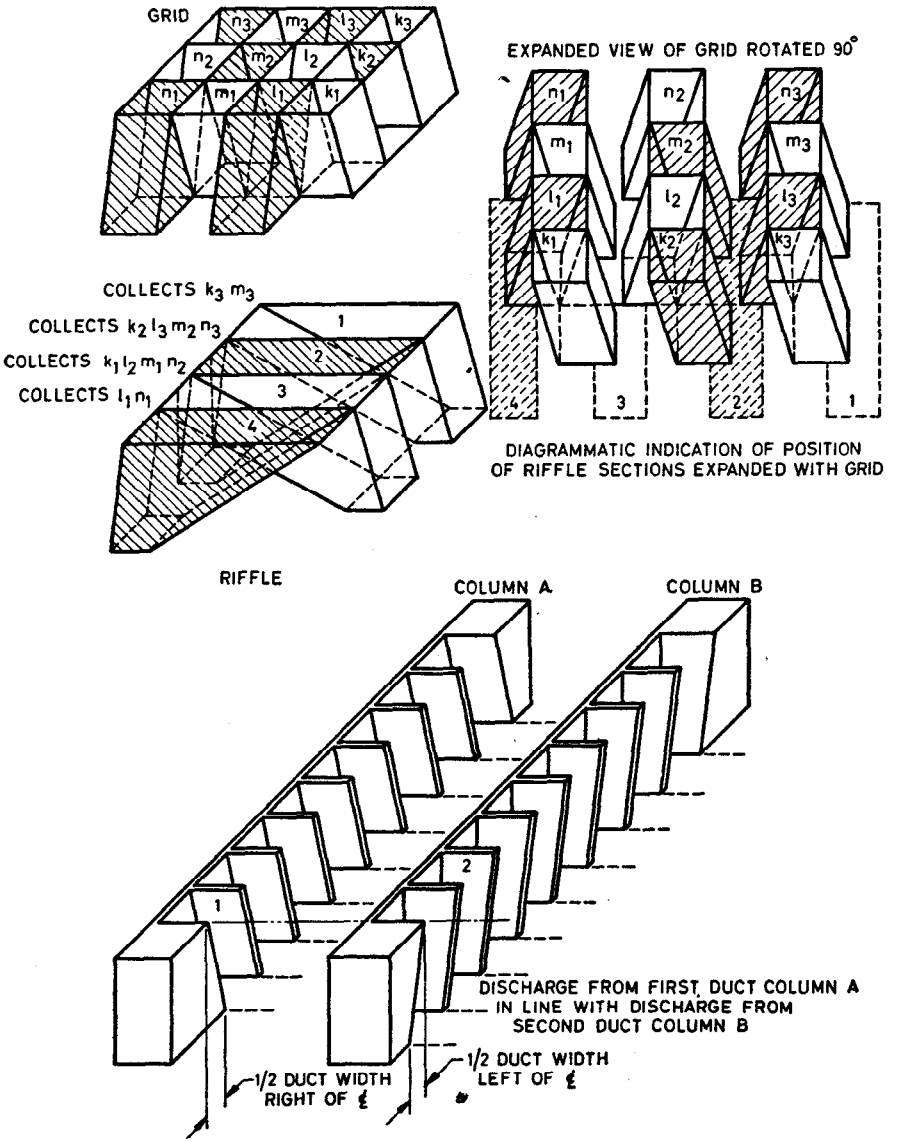
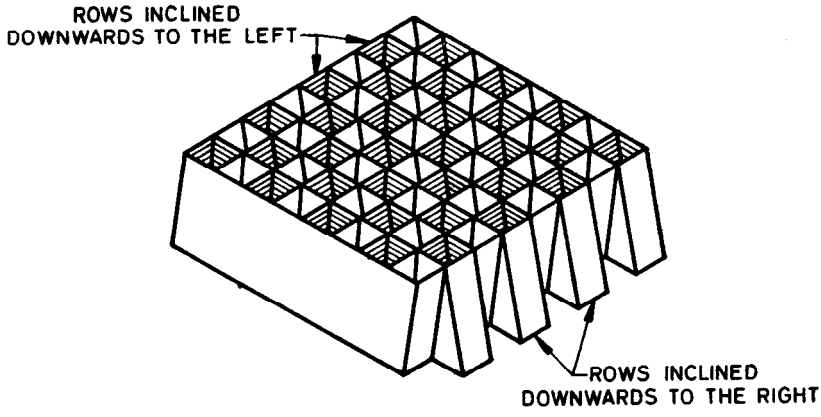
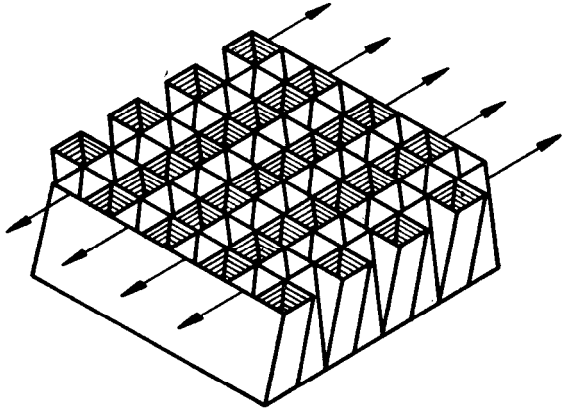


FIG. 6 GRID TYPE SAMPLE DIVIDER — ARRANGEMENT OF GRID



ARRANGEMENT OF ALTERNATE BLACK AND WHITE SQUARES AT ENTRANCE TO GRID



GRID INVERTED TO SHOW ALTERNATE BLACK AND WHITE LINES AT EXIT OF GRID

FIG. 7 GRID TYPE SAMPLE DIVIDER — VIEWS OF GRID

3.1.6.2 *Procedure* — The following procedure is adopted for reduction of sample:

a) The gross sample is fed into the hopper and levelled. The lid is then closed and swing doors opened.

b) The material now passing through the grid gets collected into receivers. The material in one of the receivers is discarded and in the other receiver taken for further sub-division.

c) The process is repeated till the required amount is obtained.

3.1.6.3 *Precautions*

a) No component of the apparatus which is in contact with the powder will be made of plastics.

b) The method shall be conducted in an enclosed space, free from draughts or air currents, to prevent loss of fine particles.

3.1.7 *Method VII Reduction by Small Oscillating Hopper Type Sample Divider from* 1 *Litre to* 0·2 *Millilitres*

3.1.7.1 *Apparatus* — The apparatus is similar as described in **3.1.5**, but of smaller size (*see* Fig. 8 on P 16).

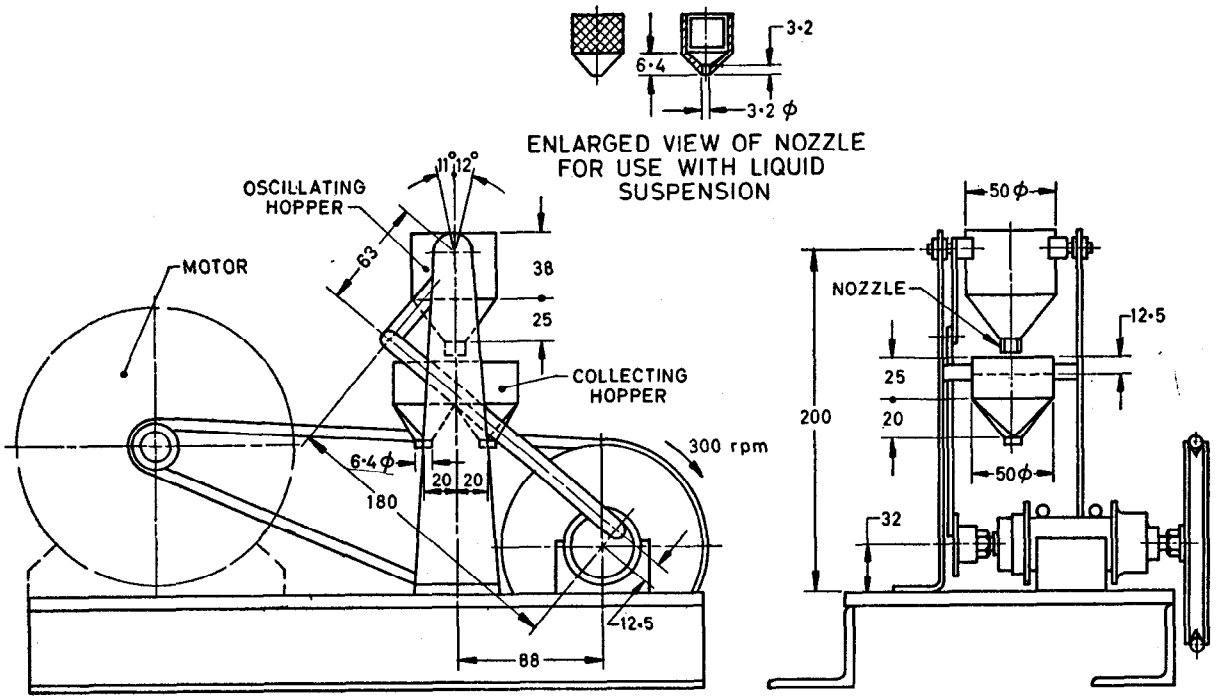
3.1.7.2 *Procedure — See* **3.1.5.2.**

3.1.7.3 *Procedure with slurries — See* **3.1.5.3.**

3.1.7.4 *Precautions — See* **3.1.5.4.**

3.1.8 *Method VIII Checking the Performance of Sample Dividers*

3.1.8.1 A simple method of checking the construction and use of apparatus for sub-division is to analyse chemically, fractions of finely ground mixtures of chalk and sand or dry salt and sand.



NOTE — Sheet brass is suggested as a suitable material.

All dimensions in millimetres.

FIG. 8 WORKING DRAWING OF SMALL OSCILLATING HOPPER SAMPLE DIVIDER

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