



भारत सरकार
GOVERNMENT OF INDIA
रक्षा मंत्रालय
MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश
JOINT SERVICES SPECIFICATION

ON

NITROGUANIDINE (PICRITE) GENERAL AND SPECIAL
DCAN

Nitroguanidine (Picrite) General	-	1376-000 010
Nitroguanidine (Picrite) Special	-	1376-000 011

मानकीकरण निदेशालय
रक्षा उत्पादन विभाग
रक्षा मंत्रालय
'एच'-ब्लॉक, निर्माण भवन डाकघर
नई दिल्ली-११००११

DIRECTORATE OF STANDARDISATION
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LIST OF MEMBERS ASSOCIATED WITH FORMULATION OF THIS STANDARD

1. This Joint Services Specification has been approved by Shri RS Gauba, Sc 'G', Associate Director, PO-II, DRDO, Chairman, Armament Standardisation Sub-committee by circulation.
2. The representatives of following organisations have been present/consulted in approving the document:

S. No.	Organisations
1.	Programme Office-II, DRDO Orgn, New Delhi
2.	ADGWE/GS (WE-2/3), New Delhi
3.	Dte of Arty (GS/Artillery-5), New Delhi
4.	Dte Gen of Naval Armt, Naval HQ, New Delhi
5.	Dte of Armt & Safety Eqpt, Air HQ, New Delhi
6.	DGEME, Army HQ, New Delhi
7.	DGNAI, Naval HQ, New Delhi
8.	DGAQA, JD (Armt) Gp, New Delhi
9.	CQA (ME), Pune
10.	CQA (Amn), Pune
11.	CQA (SA), Ichapur, West Bengal
12.	CQA (W), Jabalpur
13.	HEMRL, DRDO, Pune
14.	ARDE/DRDO Orgn, Pune
15.	Ammunition Factory, Pune
16.	Secretary ASSC

RECORD OF AMENDMENTS

Amendment		Amendment pertains to S. No./Para No./ Column No.	Authority	Amended by	Signature & Date
No.	Date			Name & Appointment (In Block Letters)	

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0. FOREWORD

0.1 This Joint Services Specification has been prepared by the Armament Standardisation Sub Committee on the authority of the Standardisation Committee, Ministry of Defence.

0.2 This specification has been approved by the Ministry of Defence and is mandatory for use by the Defence Services.

0.3 This JSS 1376-03 : 2016 (Third Revision):

- a) was prepared in the year 1984.
- b) was revised in the year 2002 & 2012 and supersedes the same.

0.4 This specification would be used for Manufacture, Supply and Quality Assurance of Nitroguanidine (Picrite) General and Special.

0.5 Quality Assurance Authority for the item covered in this specification is the Controller, Controllerate of Quality Assurance (Military Explosives), Aundh Road, Pune-411 020. Enquiries regarding technical parameters shall be addressed to the Quality Assurance Authority, while other enquiries shall be referred to:

The Director,
Directorate of Standardisation,
Ministry of Defence,
'H' Block, Nirman Bhawan PO,
New Delhi-110 011.

0.6 Non-registered users can obtain the following on payment:

a) Copies of IS from:

Bureau of Indian Standards,
Manak Bhawan,
9, Bahadur Shah Zafar Marg,
New Delhi-110 002.

or
their regional/Branch offices.

b) Copies of JSSs/JSGs from:

The Director,
Directorate of Standardisation,
Standardisation Documents Centre,
Ministry of Defence,
Room No. 05, 'J' Block,
Nirman Bhawan PO,
New Delhi-110 011.

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0.7 Indian Standard (IS) specifications are available free of cost for registered users on:

Directorate of Standardisation Website:

www.ddpdos.gov.in

For registration visit our website.

0.8 This specification holds good only for the supply order for which it is issued.

0.9 Directorate of Standardisation Website - All the approved JSSs/JSGs are available on the Directorate of Standardisation Website ***www.ddpdos.gov.in***. Defence Organisations desirous of accessing a copy of this document are requested to approach the Directorate of Standardisation for obtaining user id/password to access the website.

1. SCOPE

This specification is meant to govern Manufacture, Supply and Quality Assurance of Nitroguanidine (Picrite) General and Special suitable for use in the manufacture of propellants.

2. RELATED SPECIFICATIONS/DOCUMENTS

2.1 Reference is made in this specification to:

<i>S. No.</i>	<i>Specification/ Document No.</i>	<i>Nomenclature</i>
a)	IS 138 : 1992 (Third Revision) AMD 1 Reaffirmed 2014	Ready Mixed Paint, Marking, for Packages and Petrol Containers-Specification
b)	IS 196 : 1966 (Revised) AMD 1 Reaffirmed 2016	Atmospheric Conditions for Testing
c)	IS 4668 : 1985 (First Revision) AMD 2 Reaffirmed 2016	Specification for Ammonium Nitrate for Explosives
d)	JSS 6810-95 : 2013 (Third Revision)	Guanidine nitrate
e)	IND/ME/Stencil/20	Stencil for Making on Wooden Drum Plywood for Picrite
f)	IND/ME/49	Drawing for Drum Plywood Picrite
g)	IND/ME/50	Drawing for Bag Polythene
h)	IND/ME/878 : 2015	Dicyandiamide

2.2 Copies of the IND/ME specification are obtainable on payment from:

The Controller,
Controller ate of Quality Assurance (Military Explosives),
Aundh Road,
Pune-411 020.

3. MATERIAL/FINISH

3.1 The Nitroguanidine (Picrite) General and Special shall consist essentially of Nitroguanidine ($\text{NH}_2 \text{C NH NH NO}_2$). It shall be supplied in form of a white crystalline powder.

3.2 The material shall be free from visible impurities and any foreign matter.

4. MANUFACTURE

4.1 The Nitroguanidine (picrite) General & Special shall be manufactured by a process which has received authoritative approval. The Quality Assurance Authority shall be informed regarding the process used and shall be given prior notification of any proposed deviation therefrom. All deviations from the approval process, however slight, shall be recorded immediately and all material affected shall be set aside pending the decision of the Quality Assurance Authority.

4.2 Nitroguanidine (Picrite) General

Picrite lots with specific surface between $12,000 \text{ cm}^2/\text{cm}^3$ and $16,000 \text{ cm}^2/\text{cm}^3$ and between $22,000 \text{ cm}^2/\text{cm}^3$ and $25,000 \text{ cm}^2/\text{cm}^3$ will be so blended that the specific surface of the blended material will be within the limits specified in Clause **6.4**, Table, S. No. 11 (i).

4.3 Nitroguanidine (Picrite) Special

4.3.1 Picrite lots intended for Naval propellants will be blended in accordance with the procedure agreed to by DGNAI for achieving the specific surface within the limits specified in Clause **6.4**, Table S. No. 11 (ii).

4.3.2 Picrite lots intended for special land service propellants will be manufactured to the specific surface limit specified in Clause **6.4**, Table, S. No. 11 (ii).

5. PRE-INSPECTION OF STORES/CONSIGNMENT

5.1 Manufactures/contractors must satisfy themselves that the stores are in accordance with the terms of contract and fully conform to the required specification, carrying out a thorough pre-inspection of each lot before actually tendering the same for inspection to the Quality Assurance Officer nominated under the terms of the contract. A declaration by the contractor that a necessary pre-inspection has been carried out on the stores tendered will be submitted along with the challan. The declaration will also indicate the method followed in carrying out pre-inspection showing the features checked/tested and will have the test certificate attached to the challan/declaration.

5.2 If the Quality Assurance Officer finds that the pre-inspection of the consignment as required above has not been carried out, the consignment is liable for rejection.

6. QUALITY ASSURANCE

6.1 Inspection

6.1.1 The material and its packages in which it is contained shall be subjected to inspection by and to the final approval of the Quality Assurance Officer/Quality Assurance Authority.

6.1.2 Samples of the material and the packages in which it is contained may be taken from any portion of the batch/lot/consignment.

6.2 Sampling

6.2.1 Normally two representative samples each 150 g free of all charges shall be drawn from each batch/lot/consignment of manufacture. However the number of sample and quality to be drawn shall be at the discretion of the Quality Assurance Officer/Quality Assurance authority.

6.2.2 All precautionary measures for handling of the store as stated in the standing instructions of the section shall be strictly complied with. In absence of these, instruction from the Quality Assurance Authority/Quality Assurance Officer shall be sought.

6.3 Criteria for Conformity

6.3.1 If, on examination, any sample is found not to conform to this specification, the whole batch/lot/consignment shall be rejected.

6.3.2 The foregoing provisions shall apply equally to prime contractors and to sub-contractor, if any.

6.4 Test Requirements

Samples taken from any portion of batch/lot/consignment shall conform to Clause 3 above and in addition shall conform to the test requirements shown in the following table:

<i>S. No.</i>	<i>Characteristics</i>	<i>Passing Standard</i>	<i>Test Method</i>
a)	Volatile matter, percent by mass	0.25 <i>Max</i>	APPX 'A'
b)	Acidity, calculated as Sulphuric acid, percent by mass	0.06 <i>Max</i>	APPX 'B'
c)	pH of water extract	4.5 <i>Min</i> 8.0 <i>Max</i>	APPX 'C'
d)	Water soluble impurities, per cent by mass	1.0 <i>Max</i>	APPX 'D'
e)	Water insoluble impurities, percent by mass	0.20 <i>Max</i>	APPX 'E'
f)	Chlorides calculated as sodium chloride (NaCl), percent by mass	0.10 <i>Max</i>	APPX 'F'
g)	Sulphates, Calculated as Sodium sulphate (Na ₂ SO ₄), percent by mass	0.20 <i>Max</i>	APPX 'G'
h)	Nitrates Calculated as Sodium nitrate (NaNO ₃), percent by mass	0.10 <i>Max</i>	APPX 'H'
j)	Ash content, percent by mass	0.30 <i>Max</i>	APPX 'J'
k)	Nitroguanidine (Picrite) content, percent by mass	98.5 <i>Min</i>	APPX 'K'
m)	Specific surface cm ² /cm ³ (Particle size), micrometer:		APPX 'L'
	1) Nitroguanidine (Picrite) General	3.75 <i>Max</i> (Corresponding to 16,000 cm ² /cm ³) 2.72 <i>Min</i> (Corresponding to 22,000 cm ² /cm ³)	

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Test Requirements (Concluded)

<i>S. No.</i>	<i>Characteristics</i>	<i>Passing Standard</i>	<i>Test Method</i>
	2) Nitroguanidine (Picrite) Special	3.33 <i>Max</i> (Corresponding to 18,000 cm ² /cm ³) 2.72 <i>Min</i> (Corresponding to 22,000 cm ² /cm ³)	APPX 'L'
n)	Iron compounds calculated as Iron (Fe), percent by mass	0.02 <i>Max</i>	APPX 'M'
p)	Grit, percent by mass	0.03 <i>Max</i>	APPX 'N'

NOTES

1. Samples shall be taken in bottles fitted with rubber or waxed cork stoppers. Use of screw stoppers is strictly prohibited.
2. Wherever applicable the tests shall be carried out under standard atmospheric conditions specified in IS 196.

7. WARRANTY

The stores supplied against the contract shall deem to have been warranted against defective material and performance by the contractor/manufacturer for a period of 12 months from the date of receipt of the stores at the consignee's end and if during this period any of the stores supplied is found defective, the same shall be replaced by the manufacturer/supplier/contractor, free of all charges, at the consignee's premises.

8. PACKAGING

8.1 Nitroguanidine (Picrite) General and Special shall be packed in sound, clean dry polythene bag conforming to drawing No. IND/ME/50. The bag shall be securely closed and placed inside wooden drum to drawing No. IND/ME/49. Stenciling on the drum shall be as per drawing No. IND/ME/Stencil/20. Alternatively any other approved packages can be used.

8.2 The inclusion of any foreign matter or impurities in any of the packages will render the whole consignment liable to rejection.

9. MARKING

9.1 All packages containing the material shall indelibly and legibly be marked with following details:

- a) Nomenclature and specification number of the material.
- b) * Name and address of the consignee.
- c) * A/T or SO number and date.
- d) * Consignment number.

- e) Batch No. and date of manufacture.
- f) Gross and net mass.
- g) Consecutive number of package and total number of packages in consignment.
- h) * Date of supply.
- j) * Manufacturer's initials or recognised trademark.
- k) Station unit to be stenciled.

* **NOTE** - Not applicable when the store is manufactured by Ordnance Factory.

9.2 A government "Explosive Group D and U.N Hazard Division 1.1.D marking of approved pattern shall be affixed to each container by means of a prescribed label. Alternatively the marking may be applied by means of a stencil.

9.3 The paint used for marking shall conform to IS 138 and to the satisfaction of the Quality Assurance Officer/Quality Assurance Authority.

9.4 In addition to the above, the Quality Assurance Officer/Quality Assurance Authority may suggest more marking/identification suitable at the time of inspection.

10. DEFENCE CATALOGUE NUMBER

The Defence Catalogue Number allotted to are as under:

<i>S. No.</i>	<i>Nomenclature</i>	<i>DCAN</i>
a)	Nitroguanidine (Picrite) General	1376-000 010
b)	Nitroguanidine (Picrite) Special	1376-000 011

11. SAFETY OF OPERATIONS

11.1 Nothing in this specification shall relieve the supplier/contractor of his responsibility for the safety of operations in the manufacture, storage, transit or use of this store.

11.2 Safety certificate NO. SC/Pyro/65/57 is applicable (available on application to CQA (ME), Aundh Road, Pune-411 020).

12. SUGGESTIONS FOR IMPROVEMENT

Any suggestion for improvement in this particular document shall be forwarded to:

The Director,
Directorate of Standardisation,
Ministry of Defence,
'H' Block, Nirman Bhawan PO,
New Delhi-110 011.

DETERMINATION OF VOLATILE MATTER

A-1. Weigh a clean, dry and tared moisture dish with cover (M_1), transfer about 5 g sample in the dish with cover and weigh accurately (M_2). Remove the cover and heat the sample in an oven at $100^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 2 hours. Cover the dish, cool it in a desiccator at room temperature and weigh (M_3).

A-2. Express the loss in mass as percent on the original sample and record it as volatile matter.

$$\text{Volatile matter, percent by mass} = \frac{M_2 - M_3}{M_2 - M_1} \times 100$$

DETERMINATION OF ACIDITY

B-1. Weigh approximately 2.5 g sample, transfer to a 150 ml bottle which has previously been heated at 80°C and add 100 ml of freshly boiled distilled water which has been allowed to cool 80°C. Immerse the bottle up to its neck in a water bath maintained at 80°C ±2°C, removing it for shaking at intervals of 3 minutes. As soon as solution is complete, remove and cool rapidly in running cold water.

B-2. Calculate the Acidity as Sulphuric acid in the sample as follows:

$$\text{Acidity as Sulphuric acid, percent by mass} = \frac{4.9 (V_1 - V_2) \times N \times f}{M}$$

where,

V_1 = Burette reading for sample titration.

V_2 = Burette reading for blank titration.

N = Normality of Sodium Hydroxide solution.

M = Mass of sample taken.

f = Factor of Sodium hydroxide (NaOH) Solution.

DETERMINATION OF pH

C-1. Weigh approximately 10 g sample, transfer to a 400 ml bottle and dissolve in 200 ml freshly boiled distilled water which has been cooled to about 80°C. Stopper the bottle and immerse in a water-bath 80°C \pm 2°C until the sample has dissolved. Shake at intervals to hasten solution. As soon as solution is complete, cool rapidly in cold running water.

C-2. Determine the pH using a glass electrode and pH meter. Prior to the determination adjust the pH meter to pH 4.0 and pH 9.2 with standard buffer solutions.

APPX 'D'
(Clause 6.4)

DETERMINATION OF WATER SOLUBLE IMPURITIES

D-1. Weigh 5 g sample, transfer to a stopper flask of about 150 ml capacity and add 100 ml of distilled water. Shake vigorously for few minutes and immerse the flask in water bath maintained at $20^{\circ}\text{C} \pm 0.2^{\circ}\text{C}$ for four hours. During this period remove the flask periodically and shake it continuously for 1 minute. Filter the solution through Whatman filter paper No. 54. Pipette 20 ml of filtered liquid and transfer it to a tared evaporating dish. Evaporate the solution to dryness on a boiling water bath. Further dry the evaporating dish with residue by keeping inside a water oven maintained at $90^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for half an hour. Cool the dish in a desiccator containing Calcium chloride, until it is at constant mass.

D-2. Apply a correction factor of 0.054 g of sample for the dissolved Nitroguanidine (Picrite).

$$\text{Water soluble impurities, percent by mass} = \frac{(M_1 - 0.054) \times 100 \times 100}{V \times M}$$

where,

V = Volume in ml of solution pipetted out in the evaporating dish.

M = Mass of sample taken.

M_1 = Mass of residue.

DETERMINATION OF WATER INSOLUBLE IMPURITIES

Dissolve 5 g sample in 200 ml of boiling distilled water. Filter hot through a tared Gooch crucible and wash with 100 ml of boiling distilled water. Dry for 1 hour at 110°C and cool in a desiccator to constant mass.

$$\text{Water insoluble impurities, percent by mass} = \frac{(M_2 - M_1)}{M} \times 100$$

where,

M = Mass of sample taken.

M_1 = Mass of crucible.

M_2 = Mass of crucible and impurities.

APPX 'F'
(Clause 6.4)

DETERMINATION OF WATER SOLUBLE CHLORIDES

F-1. Weigh approximately 10 g sample, transfer to a 400 ml beaker and dissolve in 200 ml boiling distilled water. Cool the solution rapidly to approximately 5°C and filter with suction. Wash once with cold water. Transfer the filtrate to a suitable vessel and neutralize with sodium carbonate.

F-2. Titrate the solution with 0.1 N Silver nitrate solution using Potassium chromate as the indicator.

F-3. Calculate water soluble Chlorides in the sample as follow:

$$\begin{array}{l} \text{Water soluble Chlorides as} \\ \text{Sodium chloride, percent by mass} \end{array} = \frac{5.846 \times V \times N \times F}{M}$$

where,

V = Burette reading for sample titration.

N = Normality of Silver nitrate solution.

M = Mass of sample taken.

F = Factor of Silver nitrate solution.

F-3. Alternate Method

F-4. Instrument

Analytic Chloride Titrator Model No. 261, American Instrument Company, 8030, Georgia Avenue, Silver Spring Maryland, USA.

F-5. Reagents

- 1) *Acid Reagent* - Add 100 ml of glacial Acetic acid and 7.1 ml of 60 % Nitric acid (mass/mass) to 500 ml water, mix thoroughly and dilute to 1 litre.
- 2) *Gelatin Reagent* - Dissolve 6.2 g Gelatin reagent (mixture of Gelatin, Thymol blue and Thymol in proportion of 60:1:1 by mass) in little hot water and dilute to 1 litre.
- 3) Use Chloride free distilled water for all solutions and rinsing the electrodes.

F.6 Procedure

Weigh about 5g of sample accurately, dissolve in 60 ml of freshly boiled distilled water. Cool to room temperature and filter. Wash the contents in the filter paper with approximately 30 ml of distilled water. Take 1 ml of this solution in a titration viol, add 4 ml of acid reagent and 0.2 ml of Gelatin reagent and titrate according to the instructions of the instrument. Record timer reading (R_2). Similarly titrate 0.1 ml of about *0.9% (C1) Sodium chloride (AR) and record reading (R_1). Titrate blank with 4 ml acid reagent and 0.2 ml Gelatin reagent and record reading (b).

F.7 Calculate water soluble chlorides in the sample:

$$\begin{array}{l} \text{Water soluble Chlorides as} \\ \text{Sodium chloride, percent by mass} \end{array} = \frac{C1 (R_2-b) \times 10}{(R_1-b) \times \text{mass of sample}}$$

* The strength of the standard solution can be lowered down depending upon the capacity and range of the instrument.

APPX 'G'
(Clause 6.4)

DETERMINATION OF WATER SOLUBLE SULPHATES

G-1. Dissolve 5 g sample in 200 ml of boiling distilled water. Cool the solution rapidly to approximately 5°C and filter with suction. Wash once with cold water. To the filtrate add 1 ml of Concentrated Hydrochloric acid, heat the solution and add 1 ml of 10 per cent Barium chloride solution. Boil for approximately 5 minutes and allow to stand for 10 hours. Againboil and filter, collecting the precipitated Barium sulphate in a suitable tared gooch crucible. Dry the precipitate in a constant temperature oven at 100°C ±2°C followed by 1 hour in a muffle furnace at approximately 600°C. Cool in a desiccator and Weigh.

G-2. Calculate water soluble Sulphates in the sample as follows:

$$\begin{array}{l} \text{Water soluble Sulphate as} \\ \text{Sodium sulphate, percent by mass} \end{array} = \frac{60.86 M_1}{M}$$

where,

$$M_1 = \text{Mass of residue.}$$

$$M = \text{Mass of sample taken.}$$

DETERMINATION OF NITRATES

H-1. Weigh accurately about 5 g of sample and transfer to a 250 ml glass bottle. Add 150 ml distilled water and shake continuously for 1 hour in a mechanical shaker. Filter through a No. 54 what man filter paper. Discard the first 20 ml of filtrate and collect the filtrate. To 50 ml of the filtrate add 5 ml of 5% Sodium hydroxide solution and evaporate to dryness on the water bath. Moisten with 1 ml of water, then add 3 ml of Phenol sulphonic acid reagent and warm on the water bath. Add 30 ml of water, stir well and transfer to a nessler tube, using 20 ml of water to complete the transfer. Make alkaline with 15 ml of 50 % Sodium hydroxide solution. Make upto 100 ml, mix and match the colour developed with that obtained from known amounts of Potassium nitrate treated in the same manner.

H-2. Standard Colour Solution - Evaporate 1.5 ml, 0.6 ml and 0.3 ml of a solution containing 1.0 g of Potassium nitrate per litre of water to dryness in separate evaporating basins. To each of the evaporated residue slowly add 3 ml of Phenol sulphonic acid reagent and warm on a water bath. Add 30 ml of water, mix and transfer to a 100 ml Nessler cylinder using 20 ml of water to complete the transfer. Add 15 ml of 50% (m/m) Sodium hydroxide solution, adjust the volume to 100 ml with water and mix thoroughly. Alternatively transfer 30.0 ml, 2.9 ml and 1.2 ml of a solution containing 1.0 g of Potassium chromate per litre of water to separate 100ml Nessler cylinders. Adjust each volume to 100 ml with water and mix thoroughly.

H-3. Calculate the Nitrate content (as Sodium nitrate) and report as a per cent by mass of the sample as follows:

<i>Volume (ml) of Solution used in Preparing Standard Solution</i>		<i>Equivalent Percent of Sodium Nitrate</i>
<i>Potassium Nitrate</i>	<i>Potassium Chromate</i>	
1.5	30.0	0.05
0.6	2.9	0.02
0.3	1.2	0.01

H-4. Phenol - Sulphuric Acid Reagent - Prepare the reagent by adding 55.5 ml of concentrated Sulphuric acid to 4.5 ml of water containing 9 g of Phenol crystal. Store in a well stoppered bottle.

DETERMINATION OF ASH CONTENT

J-1. Weigh approximately 5 g sample in a tared porcelain crucible which has previously been ignited to 900⁰C. Add approximately 15 ml of melted paraffin wax and allow to stand for a few minutes so that the wax fully impregnates the sample. Place the crucible on a stand behind a shatter proof safety screen and warm gently from below with a Bunsen burner without permitting the flame to play into the crucible. When the mixture ignites, allow it to burn slowly. Reheat the crucible until all the paraffin has burned away. Place in a muffle at 900⁰C, cool in a dessicator and weigh. Make a blank determination on the Paraffin wax and apply the necessary correction.

J-2. From the corrected mass of residue calculate the ash content in the sample.

$$\text{Ash content, percent by mass} = \frac{M_1 - M_2}{M} \times 100$$

where,

M_1 = Mass of ash of the sample with Paraffin wax;

M_2 = Mass of ash Paraffin wax; and

M = Mass of sample taken.

NOTE - Preserve the Ash for the determination of Iron compounds.

**DETERMINATION OF NITROGUANIDINE (PICRITE)
CONTENT BY NITROMETER**

K-1. Air-dry a portion of the sample at a temperature not above 46°C for 8 hours and transfer approximately 1g of the material to a ground-glass stoppered weighing bottle (dimensions approximately 60 mm long by 25 mm diameter) which may be tared before or after the test. Dry the bottle and contents for 90 minutes at a temperature 100°C ±2°C, cool in a desiccator and weigh. Wash the cup of the generating bulb free of nitrogenous impurities and transfer the dried Nitroguanidine to the cup. From a graduated or small beaker, transfer 25 ml of Nitrogen free 95% ±0.5% Sulphuric acid in small portions to the weighing bottle and then to the generating bulb cup until sufficient quantity has been used to make an emulsion with the Nitroguanidine. Lower the mercury reservoir sufficiently to cause reduced pressure in the generating bulb. Open wide the upper stopcock and open the lower stopcock sufficiently to draw the Nitroguanidine into the generating bulb. Make successive rinsing with Sulphuric acid until all the Nitroguanidine has been drawn in with the 25 ml of Sulphuric acid. It will generally be found possible to manipulate the upper stopcock after the first time. Force out any air, drawn into the bulb, close the upper stop cock while leaving the lower one open and adjust the mercury reservoir so that the level of the mercury in the reservoir is from 32 mm to 38 mm below the level of the mercury in the generating bulb. Shake the generating bulb gently, keeping the lower end in a fixed position until most of the gas has been generated. Adjust the mercury reservoir so that the mercury in the generating bulb drops nearly to the lower shoulder, then close the lower stopcock and shake the bulb vigorously for 3 minutes. Replace the bulb on the rack, open the lower stopcock and adjust the mercury reservoir until the mercury in the generating bulb is at approximately the same height as the mercury in the reservoir. This will bring the pressure inside the generating bulb to approximately 101.3 Kpa and the solubility of the Nitric oxide gas in the Sulphuric acid will be normal. Close the lower stopcock and shake the bulb vigorously for an additional 3 minutes. Transfer the gas to the measuring tube and adjust the levels of the standardisation mark. Allow the gas to stand for approximately 20 minutes in order to permit equalization of temperature of the gas in the two tubes.

Adjust the levels of the mercury in the tubes so that they are the same and that the level of the mercury in the compensating tube is at the standardization mark. Use a leveling device, which will make possible the accurate adjustment of the levels. Note the reading on the measuring tube.

K-2. Calculate the Nitroguanidine content in the sample as follows:

$$\text{Nitroguanidine (Picrite), percent by mass} = \frac{7.430 R}{M}$$

where,

$$R = \text{Reading of measuring tube;}$$

M = Mass of sample taken; and

7.430 = Factor converting Nitro-nitrogen to Nitroguanidine.

**DETERMINATION OF AVERAGE PARTICLE SIZE/SPECIFIC SURFACE OF
NITROGUANIDINE (PICRITE) SAMPLE USING FISHER SUB-SIEVE SIZER**

L-1. Principles of Operation

L-1.1 The sub-sieve sizer operates on the air permeability principle for measuring the average particles size or specific surface of powders (Fig. 1 & 2). The principle employs the fact that particles in the path of a regulated air flow will affect that air flow in relationship to their size. More specifically, the principle finds its basis in the fact that a current of air flows more readily through a bed of coarse powder than through an otherwise equal bed of fine powder that is equal shape of bed, apparent volume and percentage of voids but by reason of difference in general coarseness of material measurement of average particles sizes are obtained.

L-1.2 The instrument eliminates most variables and resolves the formula into a series of curves which can be read directly from the calculator chart as average particles size.

L-2. Calibration of the Instrument - The National Bureau of Standards (NBS) sample No. 114, a powdered Portland cement of certified specific surface is the generally accepted primary standard to be used in calibrating the fisher sub-sieve size. NBS sample No. 114 has a relative density 3.15 and can be purchased directly from the National Bureau of Standards. When this sample is utilised as directed below, the pressure drop across it will correspond to that of the primary jewel orifice used to determine the average diameter engraved on the fisher sub-sieve sizer calibrator 14-313-7 as a secondary standard.

L-2.1 The specific surface of NBS sample No. 114 changes on exposure to air and moisture. Therefore, once the seal is broken the sample must be protected from atmospheric moisture and used within eight hours. The average particles size of some stable powdered material should be immediately determined as a secondary check.

L-2.2 The certificate packed with NBS sample No. 114 notes the mean particle diameter of the sample obtained through use of air permeability principle.

L-2.3 The mean particle diameter listed on the NBS certificate can be calculated from the specific surface area of the sample by using the formula given at the end of this method.

L-3. The instrument can be calibrated by performing following procedure:

L.3.1 Perform steps (i) through (vii) as under in Clause **L-4.2** for the determination of optimum porosity point.

L.3.2 Set the calculator chart at 0.50 porosity.

L.3.3 Perform steps (ix) through (xii) as under in Clause **L-4.2** for the determination of optimum porosity point.

L-3.4 Read the particle size off the calculator chart. If it corresponds to the known particle diameter of the NBS sample the instrument is calibrated correctly and no further calibration is necessary.

L-3.5 If it does not correspond to the known particle diameter of the sample, remove the back panel of the instrument and turn the case so as to bring the right side forward.

L-3.6 With Range Control Selector (B of Fig. 1) in the 0.2 micrometre to 20 micrometre range, turn the end of high needle value until the meniscus in the outside manometer gives the same reading as the particle size of the NBS sample. Turning the end of the valve in a clockwise direction raises the meniscus and turning it in a counter clockwise direction lowers the meniscus. Then, allow at least 20 seconds for the instrument to stabilise and take another reading. If necessary, make further adjustment until chart reads exact average particle size of NBS sample. When this is done the instrument is calibrated correctly and is ready for actual particle diameter measurements.

L-4. Method for Determination of Optimum Porosity Point and Average Particles Diameter Specific Surface

L-4.1 Before operating the sub sieve sizer, study carefully the Instruction manual and become familiar with locations and functions of the apparatus. Adjust and check properly various settings/control like range control selector, manometer level control, manometer level, water level in the pressure regulator standpipe, flow of air rate of air bubbles arising from the air pump, color of the drying agent etc. before any measurement is done.

L-4.2 Procedure

- 1) Lay a filter paper disc (Whatman No. 1) over one end of the sample tube. Then with the perforated surface of the plug against the surface of the paper disc, push one of the two porous plugs about 2 cm into the tube forcing the paper to crimp around the edges.
- 2) Vertically place the tube in the sample tube stand with the open end up.
- 3) Weigh out (to 0.01 g) sample (vacuum dried) equal in g to the true density of the sample (1.76 g/ml).
- 4) Using the funnel, completely transfer the weighed sample in to the sample tube. Tap the side of the tube to settle the powder.
- 5) Lay a second filter paper disc over the open top of the sample tube and force the other porous plug & paper disc down into the tube.
- 6) Place the sample tube on the brass post with the lower plug touching the upper end of the post.
- 7) Using the rack and pinion control, lower the rack until the flat bottom end touches upper plug.

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- 8) Move the calculator chart to the right until the pointer is set at the porosity of 0.80.
- 9) Using the rack and pinion control, lower the rack until the tip of the pointer on the rack is set on the sample height curve on the chart.
- 10) Without moving the chart, raise the rack and remove the sample tube, being careful not to disturb the sample.
- 11) Mount the sample tube between the rubber cushioned supports just to the right of the brass-post and twist the clamp assembly control to the right until the tube is locked in to place, making an air tight seal at both ends of the tube.
- 12)
 - i) Adjust the pressure control selector until bubbles rise in the pressure regulator standpipe at the rate of two to three per second. The water level will rise above the water level mark as observed through the water level observation window. This is normal, however, does not mean that the calibration is incorrect. In addition the liquid level in the monometer tube will rise slowly and reach a maximum height within 30 seconds to several minutes depending upon the particle size.
 - ii) After the liquid in the monometer level reaches its maximum level, turn up the rack until the upper edge of the cross bar coincides with the liquid meniscus in the manometer.
- 13) Read the particle size on the chart and record the reading. The particle size is indicated by the location of the tip of the pointer with relation to the curves on line chart.
- 14) Move the calculator chart to the left to a porosity of 0.75 and repeat steps (ix to xiii), then moving the calculator chart by 0.05 porosity to the left each time, repeat steps (ix to xiii) a sufficient number of times until the chart runs off-scale at 0.40 or until the sample is packed as tight as possible.
- 15) Plot all readings on a sheet of graph paper and connect the points. The mid point of the plateau of the curve is the optimum porosity points or the point on which the porosity scale should be set to measure the specific surface at that particular density of the sample with the minimum degree of error. Measurement at this optimum porosity is mandatory to enable checking of results.
- 16) Determine average particle diameter (micrometer) of the sample at optimum porosity (Normally optimum porosity lies around 0.65 for Nitroguanidine (picrite) sample).
- 17) Calculate Specific surface of the sample using the following equation:

$$S_0 = \frac{6 \times 10^4}{dm}$$

where,

S_0 = Specific surface (cm^2/cm^3) of the sample at optimum porosity; and

d_m = Average particle diameter in micrometer taken from calculator chart on the instrument.

NOTE - Perspective of Operating principles is shown at figure 3.

SUB SIEVE SIZER

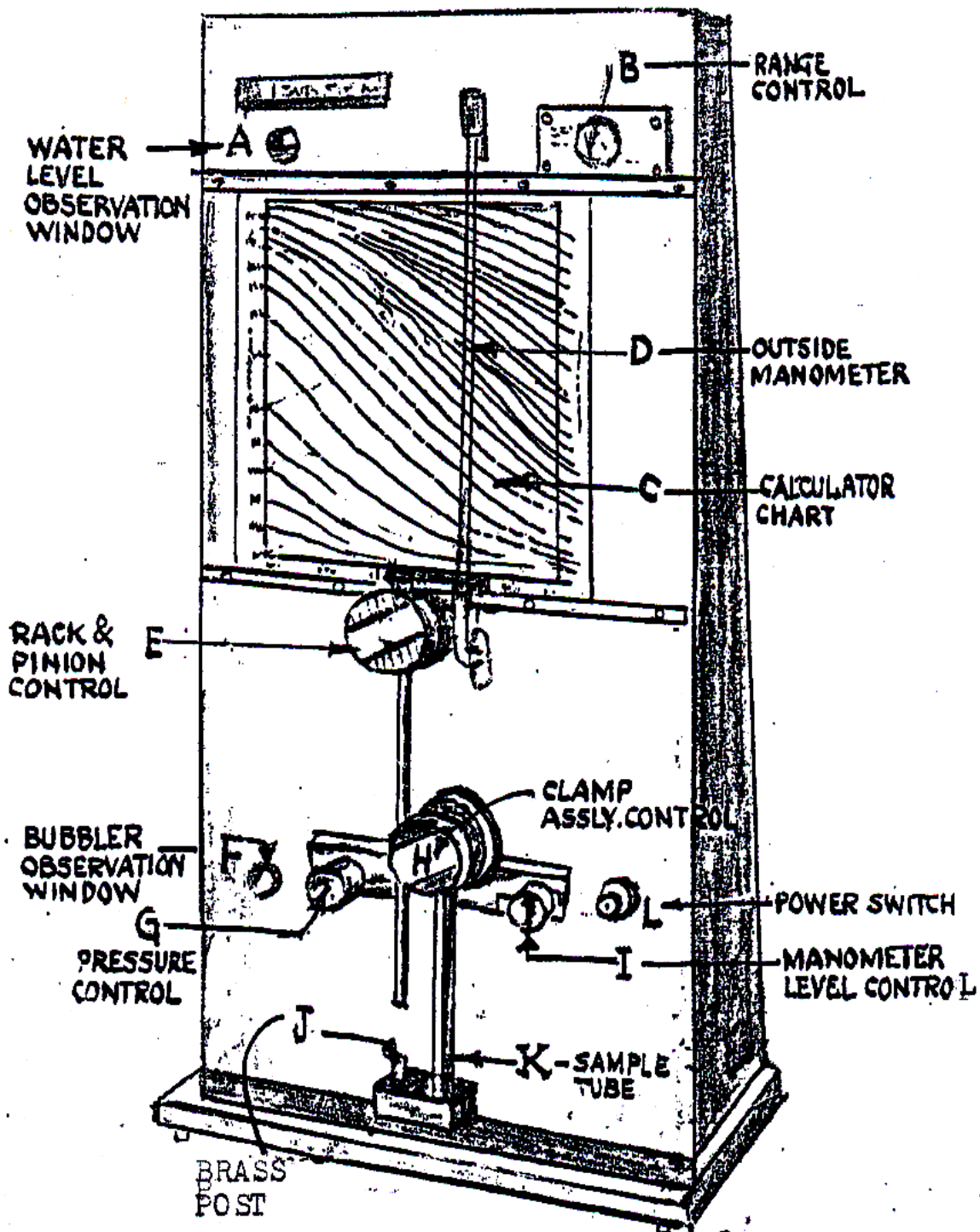


Fig. 1 : Control Panel

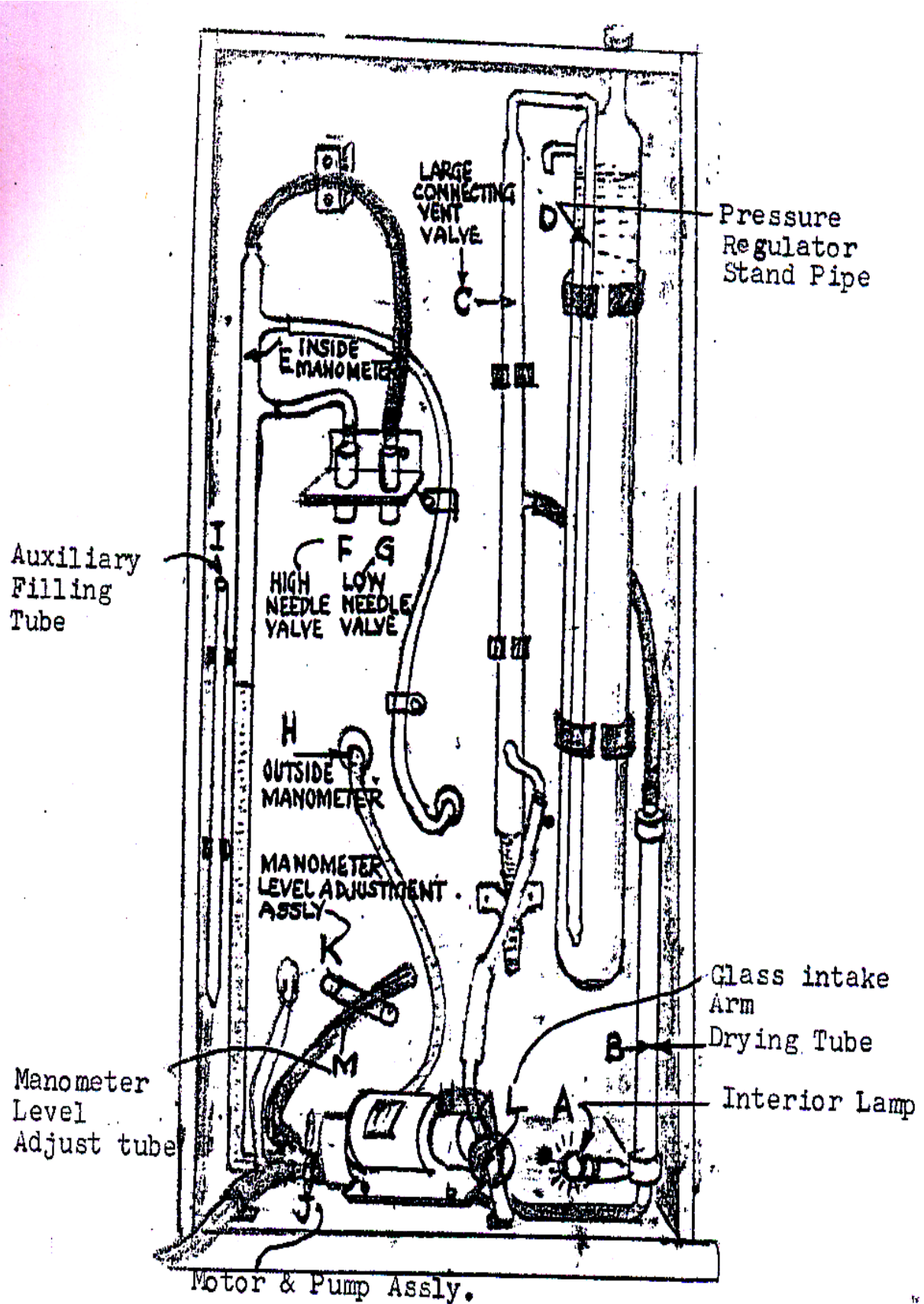


Fig. 2 : Back Removed

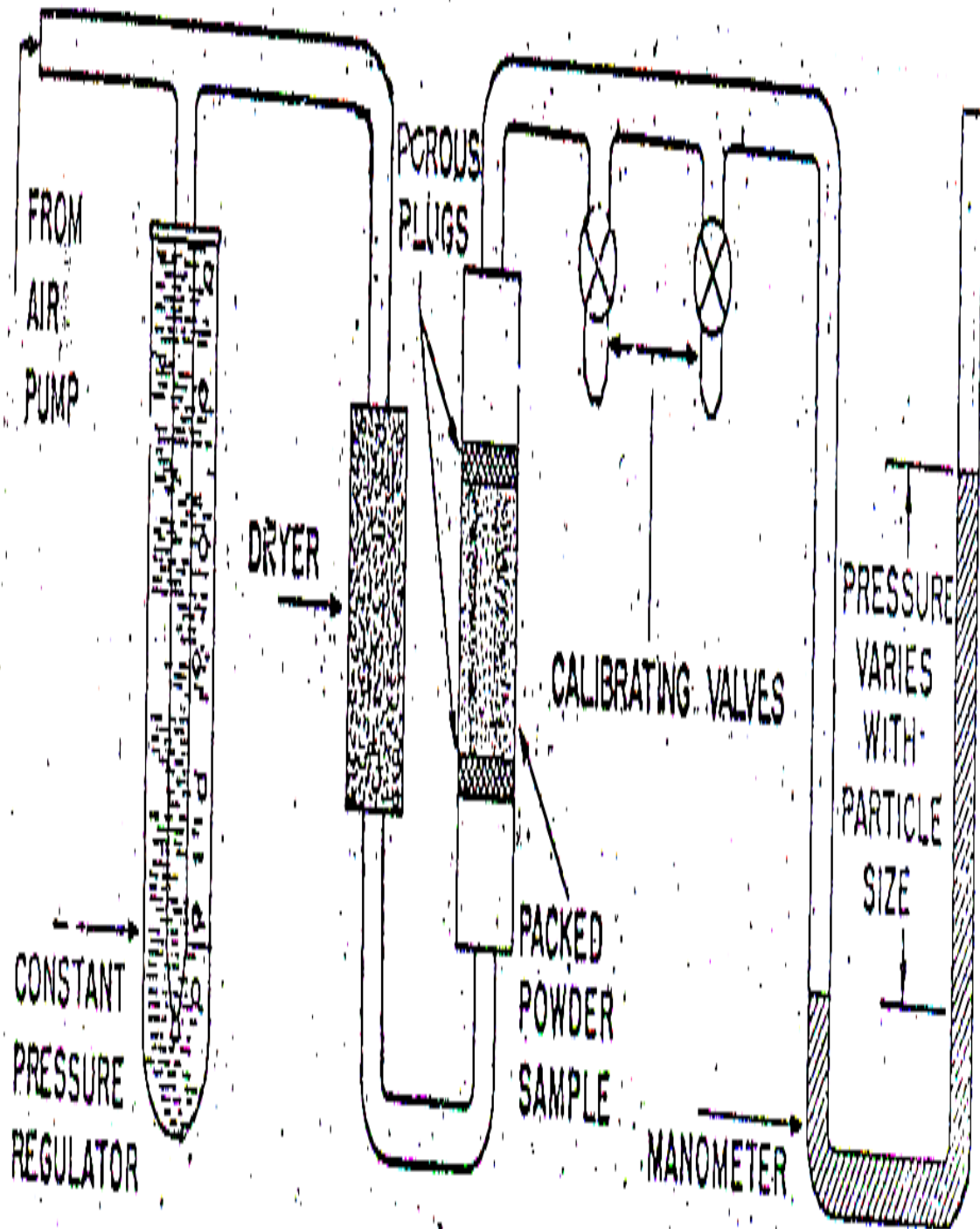


Fig. 3 : Perspective of Operating Principles

DETERMINATION OF IRON COMPOUNDS

Treat the ash obtained at (APPX 'J') of 5g sample in dilute Hydrochloric acid (HCl) and estimate the Iron content by the standard Colorimetric method.

DETERMINATION OF GRIT

N-1. Weigh 10 g sample, transfer in a 500 ml conical flask and dissolve it in 200 ml hot distilled water. Continue heating on water-bath/hot plate till all sample is dissolved. Decant the liquid carefully and again pour about 150 ml of water. Heat and decant it. Repeat till picrite is taken out by decantation process. Finally let the content be retained to about 10 ml to 15 ml. Add 5 ml aquaregia and heat till all soluble impurities are dissolved. Dilute the content with water and filter through a dry and weighted 63sintered crucible. Wash the contents of the crucible by distilled water and finally with Acetone. Dry the crucible at 100°C ±2°C and weigh to constant mass.

N-2. Calculate grit content in percent by mass.