

BTS 357: 2021 IS 1747: 1972

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BHUTAN STANDARD
Specification for Industrial Nitrogen



ICS 71.100.20

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BHUTAN STANDARDS BUREAU

The National Standards Body of Bhutan

THIMPHU 11001

BTS 357: 2021 IS 1747: 1972

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NATIONAL FOREWORD

This Bhutan Standard which is identical with IS 1747: 1972 Specification for Industrial Nitrogen issued by the Indian Standards Bureau (IS) was adopted by Bhutan Standards Bureau by sub-committee on Medical and industrial gases (TC 05/SC 01) and Pharmaceutical and Traditional Medicines Technical Committee (TC 05) and approved by the Bhutan Standards Bureau Board (BSB Board) on, 2021.

The text of the IS Standard has been approved as suitable for publication as Bhutan Standard without deviation. Certain conventions are however, not identical to those used in Bhutan Standard.

Attention is particularly drawn to the following:

- a) Where the words “IS Standard” appear referring to this standard, they should be read as “Bhutan Standard”.
- b) Wherever page numbers are quoted, they are “IS (IS Standard)” page numbers.

For test methods, manufacturers may refer Annex I of this standard.

IS : 1747 - 1972
(Superseding IS : 3551 - 1965)
(Reaffirmed 2010)

Indian Standard
SPECIFICATION FOR NITROGEN
(First Revision)

Sixth Reprint AUGUST 2007
(Including Amendment No 1)

UDC 661 938

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

Gr 7

March 1973

AMENDMENT NO. 2 DECEMBER 2008
TO
IS 1747 : 1972 SPECIFICATION FOR NITROGEN
(First Revision)

(Page 26, Annex B, Table 2) — Substitute the following for the existing:

Table 2 Number of Cylinders to be Selected
(Clause B-1.2)

Lot Size <i>N</i> (1)	Number of Cylinders to be Selected <i>n</i> (2)
Up to 25	3
26 to 50	5
51 to 100	8
101 to 200	15
201 to 300	20
301 and above	25

(CHD 6)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 MAY 1984

TO

IS: 1747-1972 SPECIFICATION FOR NITROGEN

(First Revision)

Alteration

[Page 4, Table 1, Sl No. (vi)] - Substitute the following for the existing matter:

(1)	(2)	(3)	(4)	(5)
vi)	Water vapour, mg/m ³ , Max	5.6	-	A-7'

(CDC 35)

Indian Standard
SPECIFICATION FOR NITROGEN
(*First Revision*)

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(Continued on page 2)

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(Continued from page 1)

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Indian Standard
SPECIFICATION FOR NITROGEN
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 3 October 1972, after the draft finalized by the Industrial Gases Sectional Committee had been approved by the Chemical Division Council

0.2 Two Indian Standards have been issued for nitrogen, namely, IS : 1747-1960 Specification for nitrogen, technical and IS : 3551-1965 Specification for pure nitrogen (dry). This standard amalgamates the two Indian Standards.

0.3 Pure nitrogen caters mainly to the rigorous requirements of the incandescent lamp filling industries. Some of its other special uses are in research programmes involving work in inert atmosphere and in the manufacture of oxygen-labile organic chemicals and pharmaceuticals. Technical nitrogen is intended for general industrial uses.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for nitrogen (gas).

2. GRADES

2.1 There shall be two grades of the material, namely:

- a) *Technical* (tech), and
- b) *Pure*.

*Rules for rounding off numerical values (*revised*).

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3. REQUIREMENTS

3.1 The gas, when tested according to the methods prescribed in Appendix A, shall conform to the requirements given in Table 1. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR NITROGEN

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST (REF TO CL No. OF APPENDIX A)
		Pure Grade	Terh Grade	
(1)	(2)	(3)	(4)	(5)
i)	Oxygen, percent by volume, <i>Max</i>	0.001	0.5	A-1 and A-2
ii)	Hydrogen, parts per million, <i>Max</i>	5	—	A-3
iii)	Carbon dioxide, parts per million, <i>Max</i>	5	—	A-4
iv)	Other carbon compounds, determined as CO ₂ , parts per million, <i>Max</i>	5	—	A-5
v)	Carbon monoxide, parts per million, <i>Max</i>	Nil	—	A-6
vi)	Moisture, g/m ³ , at 21 kg/cm ² , <i>Max</i>	0.01	—	A-7

4. PACKING AND MARKING

4.1 The gas shall be supplied compressed in cylinders. The design of the cylinders, pressure of gas in the cylinders, packing, marking, painting, labelling and transport of cylinders shall be in accordance with the Gas Cylinder Rules, 1940, with such modifications as may be ordered from time to time by the Chief Inspector of Explosives, Government of India or other duly constituted authority.

4.1.1 The product may also be marked with Standard Mark

4.1.2 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards

5. SAMPLING

5.1 **Preparation of Test Samples** — The method for drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

A P P E N D I X A

(Clause 3.1)

METHODS OF TEST FOR NITROGEN

A-0. QUALITY OF REAGENTS

A-0.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

A-1. DETERMINATION OF OXYGEN (FOR PURE GRADE)

A-1.0 General—Two methods for the determination of oxygen in pure grade nitrogen are described. Either of these may be used for general routine purposes, but in case of a dispute Method 2 shall be used.

A-1.1 Method 1

A-1.1.0 Principle—A sample of gas is bubbled through an alkaline solution of pyrocatechol in complete absence of oxygen. The colour developed in the solution due to the reaction of pyrocatechol with the oxygen in the sample of gas is matched with the colour slides in the standard disc.

A-1.1.1 Description of Apparatus—The assembly of apparatus shall be as illustrated in Fig. 1.

A-1.1.1.1 Standard discs—available from the suppliers of scientific apparatus.

A-1.1.2 Reagents

A-1.1.2.1 Pyrocatechol solution—Bubble oxygen-free nitrogen (from tested certified cylinders) for half an hour through 1 000 ml of water contained in bottle *D*. Divide the water into two portions, one of 900 ml in bottle *D* and the other of 100 ml in a small conical flask. Keep both the portions bubbling with oxygen-free nitrogen. To the 900 ml of water in bottle *D* add 7 g of pyrocatechol, maintaining the nitrogen bubbling. To the 100 ml of water in the conical flask add 6 g of ferrous ammonium sulphate, 1 g of zinc and 2 ml of concentrated sulphuric acid, still maintaining the nitrogen bubbling, and warm the solution gently so that a brisk evolution of hydrogen occurs. When most of the zinc has dissolved, add the solution quickly to the solution in bottle *D*, transferring the remaining zinc as well. Replace bottle *D* quickly in the set through the coupling *M* where this solution as well as that in bottle *E* is kept bubbling through with oxygen-free

*Specification for water, distilled quality (revised).

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nitrogen continuously to prevent the entry of atmospheric oxygen. After leaving the reagents, the nitrogen will escape through *K* at atmospheric pressure.

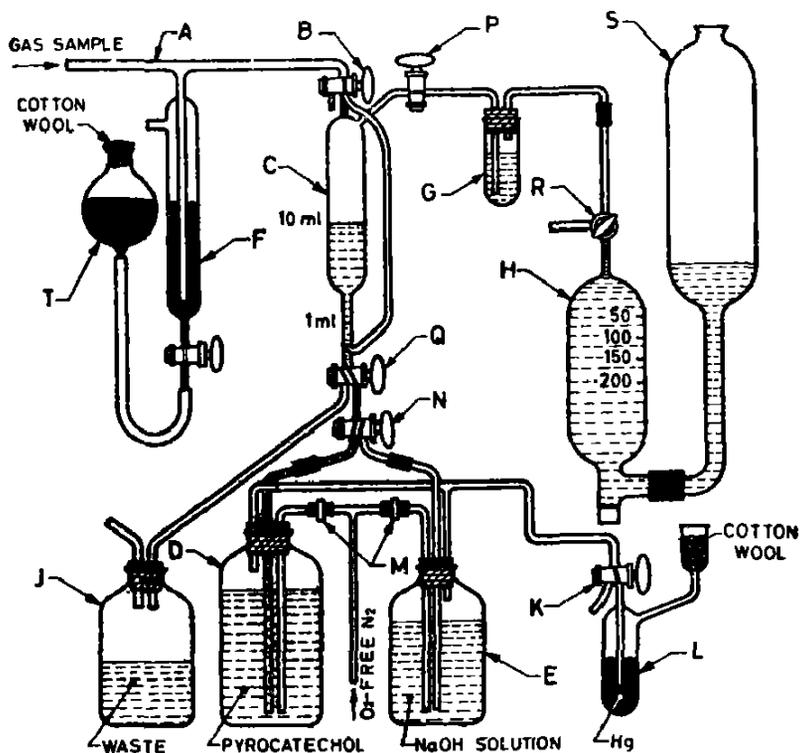


FIG. 1 APPARATUS FOR THE DETERMINATION OF OXYGEN IN NITROGEN PURE

A-1.1.2.2 Sodium hydroxide solution—Take 500 ml of water in a beaker and bubble, fairly vigorously, oxygen-free nitrogen through it. Add 250 g of sodium hydroxide in small portions to the water while nitrogen is bubbled through. When the solution is cool, pour it into bottle *E* and attach to the set through the coupling *M*.

A-1.1.3 Procedure—Turn taps *Q* and *N* off so that the liquid does not rise into *C*. Turn *K* so that nitrogen bubbles through mercury and a pressure is obtained in *D* and *E*. Pour water into *S* with tap *R*, open the atmosphere until water level is just below tap *R*. Fill to half bubbler *G* with water. Fill the bubbler *L* to a height of 62 mm with mercury.

Run mercury into reservoir *T* and raise *T* until the mercury reaches the fixed mark at *F*. (This height of mercury gives a speed of approximately 15 ml/minute.) Connect the cylinder or gas supply to be tested to *A* by means of a flow regulating valve and glass or copper tubing, any junctions being made with rubber pressure tubing and as short as possible. Turn *B* so that the gas from the cylinder passes to the atmosphere and open carefully the cylinder valve and the flow regulating valves. When the connections have been thoroughly blown out with gas, reduce the rate of flow to about 2.5 litres per hour and turn *B* so that the gas passes into *C* through the sintered disc. Open the tap *P* and allow the gas to pass to the atmosphere through tap *R*. There should always be a slight excess of gas bubbling through the mercury in *F*. Allow the gas to pass until *C* is thoroughly washed out. By turning caps *N* and *Q*, force sodium hydroxide solution into *C* to the 1-ml mark. Turn the tap *N* to connect *C* to the pyrocatechol reservoir and force the solution into *C* to the 10-ml mark. Shut taps *Q* and *N*. If the solution in *C* is slightly coloured, it is ready for test, but if it is substantially coloured, run it carefully to a waste-bottle by closing tap *P*, and opening tap *Q* to waste. Refill *C* to the 10-ml mark as indicated above. If the solution is still appreciably coloured, the process shall be repeated until an almost colourless solution is obtained. Match the colour of the solution in *C* as quickly as possible with a suitable standard, on a colour disc using a matching plate and turn tap *R* immediately so that the gas passes into the measuring burette *H*. When 100 or 200 ml of the gas have passed in, rematch *C* against suitable standard.

A-1.1.3.1 If the gas contains appreciable quantities of oxygen, it may only be necessary to use 100 ml of gas to obtain a suitable colour change.

NOTE 1 — The set does not function satisfactorily with oxygen concentrations above 0.005 percent

NOTE 2 — The rate of flow should be approximately 15 ml/minute, and this should be checked during a test by noting the time for 200 ml of gas to collect in *H*. If there is any appreciable deviation from this rate, mercury in *F* should be raised or lowered until the correct speed is obtained.

NOTE 3 — The set will function satisfactorily at a temperature 15-20°C. Above this temperature the 'blanks' are likely to be high.

NOTE 4 — If after a test the increase in colour of the solution in *C* is slight, a further test may be carried out using the same solution.

NOTE 5 — After shutting off *P*, refilling *H* with water, and turning *B* to connect the gas supply to the atmosphere, the test cylinder is replaced by another cylinder and the procedure given in **A-1.1.3** repeated.

NOTE 6 — When the set is not in use, *C* shall always be empty and isolated from the atmosphere and the gas pressure in *D* and *E* released by turning *K* so that the nitrogen bubbles through the reagents direct to the atmosphere.

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A-1.1.4 Calculation

$$\text{Oxygen, percent by volume} = \frac{y - x}{v} \times 0.01$$

where

y = figure on colour standard after passing the gas sample;

x = figure on colour standard before passing the gas sample;
and

v = volume of gas passed, that is 100 or 200 ml.

NOTE—A figure '8' on colour Standard denotes 0.0008 ml of oxygen. Thus if 100 ml test gas causes a change from figure '8' to figure '12' then

$$\text{Number of ml oxygen} = 0.0012 - 0.0008 = 0.0004$$

$$\text{Therefore, percentage of oxygen in gas} = 0.0004 = \frac{0.0004}{100} \times 100 = 0.0004$$

A-1.2 Method 2

A-1.2.0 Principle — The oxygen in the test gas reacts with the silver cathode in the galvanic cell contained in the apparatus to produce hydroxyl ions which oxidize metallic cadmium to cadmium hydroxide. This generates a current which flows in the external circuit consisting of a galvanometer connected across the electrodes. The magnitude of the current is a measure of the amount of oxygen in the gas.

A-1.2.1 Apparatus

A-1.2.1.1 Hersch oxygen meter or one of equivalent accuracy

A-1.2.1.2 Description of Hersch oxygen meter — The apparatus consists of the following principle components

- a) *A flow regulating valve*
- b) *Calibrating unit*— consisting of a purifier containing manganous oxide which eliminates all oxygen in the test gas and a calibrator which adds a small predetermined amount of oxygen to the test gas, generated electrolytically by a controlled amount of current supplied by a voltage stabilized rectifier unit; and
- c) *Cell assembly* — consisting of a silver-cadmium galvanic cell containing 32-percent potassium hydroxide solution as electrolyte

A-1.2.1.3 Calibration of the apparatus — The procedures for calibration are briefly described below. The detailed instructions of the manufacturers shall be followed:

- a) *Calibration for 0-10 ppm by volume range* — Turn the sensitivity knob fully clockwise before calibration. See that the flow selector valve

is at 'calibrate' and the flow is adjusted to the appropriate setting for the gas being tested. Note the galvanometer reading obtained (cell zero blank).

Turn the current control knob clockwise until the milliammeter reads '2'; this represents an oxygen input of 2 ppm by volume at the 0-10 range setting.

The galvanometer pointer will start moving almost immediately and after a minute or two will settle down at a new reading. The difference between this reading and the cell zero blank represents the instrument's response at maximum sensitivity to a change in oxygen concentration of 2 ppm by volume.

To adjust so that a change of 2 ppm response of two divisions on the galvanometer is registered, the sensitivity control of the 'meter XI' shall be operated to bring the galvanometer to a new reading calculated as under'

$$\text{New reading} = \frac{\text{Reading at maximum sensitivity} \times 2}{\text{Reading at maximum sensitivity} - \text{Zero blank}}$$

Example:

Assuming zero blank before adjustment = 0.5

Reading at 2 ppm before adjustment = 4.5

Adjust reading at 2 ppm to $\frac{4.5 \times 2}{4.5 - 0.5} = 2.25$

The zero blank reading at the reduced sensitivity will be 0.25 ppm.

The instrument is now calibrated at a sensitivity of 'One division equals 1 ppm' and the zero blank of 0.25 division will necessitate a deduction of 0.25 ppm from test readings.

When calibration is completed, make sure that the calibrating current is turned back to zero.

- b) *Calibration for 0-100 ppm by volume range*— For use with gas where the oxygen concentration is over 10 ppm, set the range switch, at 'meter $\times 10$ '. The figures on the calibrating current ammeter will now represent 20, 40, 60, 80 and 100 ppm.

As the response of the instrument is not completely linear over this range, the calibration shall be carried out by plotting a curve showing galvanometer readings against oxygen input. Plot four or five points, including zero 'O₂'. The oxygen concentration of the gas, as shown by the galvanometer reading on 'Test' shall then be determined by reference to this curve.

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The cell zero blank and the instrument leak figure shall normally be ignored when using this range.

A-1.2.2 Procedure — Pass the test gas through the instrument at a flow rate of 22.5 litres per hour with the flow selector valve at purge for 15 minutes. An inlet gas pressure of at least 28.44 kg/cm² is necessary. If the pressure of the gas is lower than this value, compress the test gas by a booster pump so that pressure is raised to this value or even higher.

Having calibrated the instrument, ??? the flow selector valve to test and readjust the flow, if necessary. After about four minutes, read the scale

A-1.2.3 Calculation and Report — The oxygen content as parts per million by volume is given directly by the linear scale of the instrument in the case of oxygen content being in the range of 0-10 ppm. If the oxygen content is above this range it shall be obtained by reference to the curve prepared as in A-1.2.1.3(b). From this value deduct the 'zero blank' figure and the leak figure which is the leading obtained by using an oxygen free gas such as oxygen-free hydrogen. Report the oxygen content as percent by volume.

A-2. DETERMINATION OF OXYGEN (FOR TECHNICAL GRADE)

A-2.1 Apparatus

A-2.1.1 The assembly of apparatus shall be as shown in Fig. 2.

A-2.2 Reagents

A-2.2.1 Pyrogallol Solution — Dissolve 350 g of pyrogallol in 1 000 ml of water. Keep the stock solution in amber coloured bottles.

A-2.2.2 Potassium Hydroxide Solution — Dissolve 1 000 g of potassium hydroxide in 1 000 ml of water.

NOTE — Alkali purified by alcohol should not be used since solutions evolve ??? carbon monoxide.

A-2.3 Procedure

A-2.3.1 Pour 140 ml of pyrogallol solution and 100 ml of potassium hydroxide solution into Woulfe's bottle *A* through its opening *J*. Immediately after introducing the solutions, close the opening with rubber ??? and shake the bottle to mix the solutions. Having charged bottle *A* with the solutions, fill measuring burette *C* with water by raising levelling bottle *D*, closing one way tap *G* and ??? three way tap *E*, to ??? 2. When the water in the burette reaches the zero mark, turn ??? tap *F* to position 1 and replace bottle *D* in set.

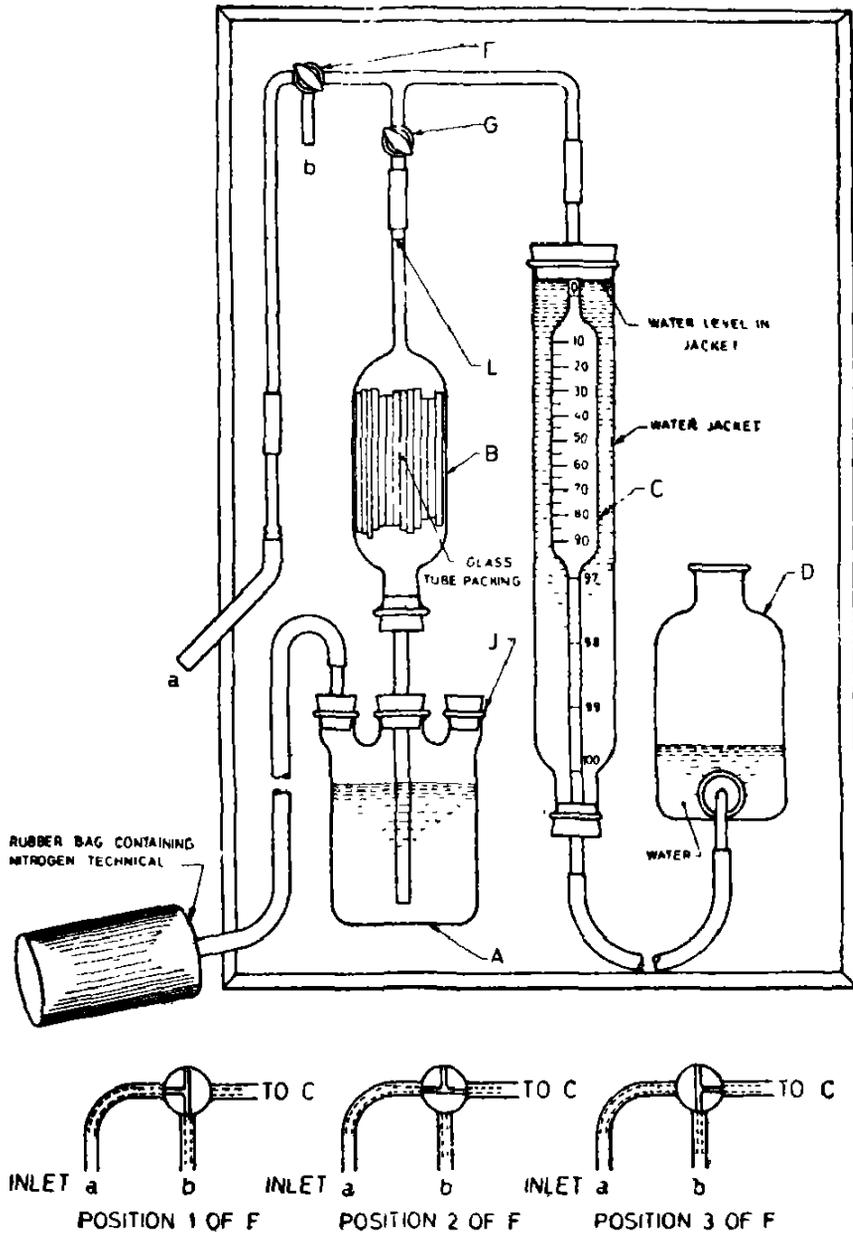


FIG. 2 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF OXYGEN AND OTHER ACTIVE CONTAMINANTS

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A-2.3.1.1 The burette *C* shall be capable of measuring to an accuracy of 0.05 ml.

A-2.3.2 Open one-way tap *G* and lower bottle *D* to draw the liquid into absorption pipette *B* from bottle *A*. When burette *C* becomes full gas, close one-way tap *G* and turn three-way tap *F* to position 2 and raise bottle *D* until burette *C* is full of water again. Turn three-way tap *F* to position 1 and repeat the foregoing operations until the level of the solution reaches mark *L*. Perfect control of the level may be obtained by raising or lowering bottle *D* slowly.

A-2.3.3 Close tap *G* and finally expel any gas from burette *C* and turn tap *F* to position 1.

A-2.3.4 Connect the supply of gas to inlet of a three-way tap *F*, the tap being in position. 2, purge the inlet tube with the sample and then turn tap *F* to position 2. Bring the level of water in bottle *D* in line with graduation mark 100 on burette *C*. Turn tap *F* to position 3 and raise water bottle *D* to expel all the gas to atmosphere. Again turn tap *F* to position 2 and lower water bottle *D* in line with graduation mark 100 on burette *C*. Repeat this process of purging burette *C* and the manifold connections up to *G* and *F* once again.

A-2.3.5 Turn tap *F* to position 1 again. Allow water to drain down the sides of burette *C* and read the volume keeping water level same in burette *C* and bottle *D*. Open tap *G*, and raise bottle *D* until burette *C* is just full of water. Close tap *G*, replace bottle *D* in set and leave gas to be tested in absorption pipette *B* for a few minutes so that oxygen and other active contaminants are absorbed from it.

A-2.3.6 Open tap *G* and lower bottle *D* until the level of the solution again reaches mark *L*. To ensure complete absorption, raise the bottle again until burette *C* is full of water, close tap *G*, replace bottle *D* in set for another few minutes. Open tap *G*, and lower bottle *D* until the level of the solution again reaches mark *L*. Close tap *G* and finally allow water to drain in the burette *C* and read the volume of gas, keeping the water level same in *C* and *D*.

A-2.3.7 Repeat the steps outlined in **A-2.3.5** and **A-2.3.6** until two consecutive readings agree.

A-2.4 Calculation

$$\text{Oxygen, percent by volume} = \frac{V - V_1}{V} \times 100$$

where

V = initial volume of gas (see **A-2.3.5**), and

*V*₁ = volume of gas after absorption (see **A-2.3.7**).

A-3. DETERMINATION OF HYDROGEN

A-3.0 Principle—The sample of gas is passed through a heated tube containing palladium chloride. The hydrogen in the gas reacts with palladium chloride to produce hydrochloric acid gas which is bubbled through water set at pH 9 to which an indicator is added. The time in minutes for successive changes of pH to 8.5, 8.0 and 7.0 will, by reference to the calibration chart, give the hydrogen concentration for a test gas flow of 5 litres per hour.

A-3.1 Apparatus—The assembly of apparatus shall be as shown in Fig. 3.

A-3.1.1 The test gas enters the apparatus at *A*. At stopcock *B* the gas may be led either to bubbler *C*, which is merely a visual gas flow indicator, or on to stopcock *D* through which the gas passes through the bypass tube to stopcock *E*. From *E* the gas passes through the drying tube *G* filled with activated alumina, the oxygen removing tube *H* filled with manganous oxide to the palladium chloride tube *J*, and then to the bubbler *K*, which contains indicator solution from reservoir *N*. The gas finally passes through a spray tap *L* containing glass wool to flowmeter *M*, and then to atmosphere.

A-3.1.2 There are three furnaces or heaters in the set, each controlled by a suitable electrical device. They are as follows:

- a) Heater *F* at 250°C is used to heat a copper oxide tube to obtain a hydrogen-free gas stream for blanking the set (to show that the set does not register hydrogen when none is present);
- b) Heater round the palladium chloride tube *J*; and
- c) Heater *R* which is used for regeneration of alumina and manganous oxide tubes.

A-3.1.3 The indicator solution stored in *N* is rendered free from carbon dioxide by passing nitrogen in at *S*, through *P* (which is packed with soda asbestos or soda lime) and then through the indicator solution in *N*. Carbon dioxide trap *T* also contains soda asbestos or soda lime. Mercury tube *Q* is a safety device so that if nitrogen is turned on while *X* is shut or if *P* becomes blocked, there is an exit for the gas.

A-3.1.4 Preparation of Apparatus—Switch on all heaters and adjust voltage regulators to the following temperature:

Palladium chloride heater	190-200°C
Copper oxide heater <i>F</i>	240-260°C
Regenerator heater <i>R</i>	250-280°C

A-3.1.4.1 It may take some hours to find the correct setting for each. When these readings have been found they should be carefully noted. A further setting of 480 to 500°C is required for the regenerator heater.

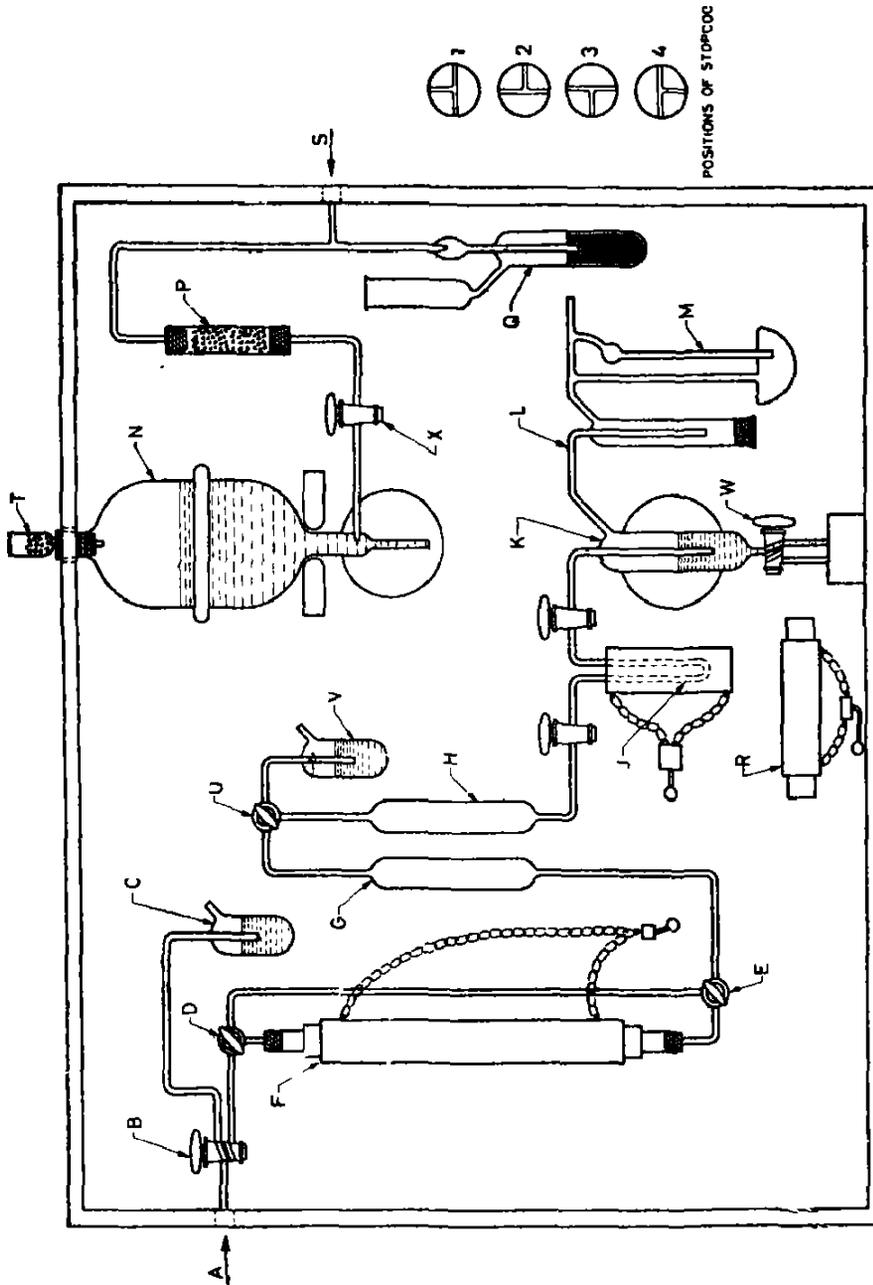


FIG. 3 LAYOUT OF MODIFIED COLORIMETRIC HYDROGEN TESTING SET

Care should be taken that this does not exceed 500°C for the regenerator heater, otherwise softening of the glass of the manganous oxide tube may occur. These figures once found need only be checked at intervals of several weeks. The dial controlling the palladium chloride set temperature should not be altered once set at 180 to 200°C. The set should be switched off by a wall switch.

A-3.1.5 Connect alumina tube *G* and manganous oxide tube *H* to the apparatus using red rubber pressure tubing, and turn stopcock *U* so that the stopcock bore is in position 1 (Fig. 3), and both stopcocks on palladium chloride tube *J* are shut.

A-3.1.6 Shut stopcock *X*, and pour 500 ml of water into reservoir *N* and add 6 ml of Universal indicator. Connect a nitrogen supply to *S* and turn on slowly. When the nitrogen starts to bubble through the mercury in *O*, open stopcock *X* and remove *T*, and continue passage of nitrogen until the solution is yellow-green in colour, that is, pH is 7. When this point is reached, add 0.1N barium hydroxide solution drop by drop to the reservoir, until the solution is a blue colour, matching the solution pH 9 in a 10-ml test-tube.

A-3.1.7 A cylinder of known hydrogen concentration is necessary. The cylinder should also contain a small amount of oxygen, and may be prepared by sucking a measured amount of hydrogen and oxygen into a evacuated cylinder of known capacity and filling with oxygen-free argon or nitrogen.

A-3.2 Reagents

A-3.2.1 *Activated Alumina* — 2 to 1 mm size.

A-3.2.1.1 *Preparation of alumina for use* — Insert alumina tube in the regenerator heater and pass dry nitrogen or argon through it at 50 litres per hour for 2 hours. Remove alumina tube, stop both ends with rubber tubing and glass plugs, and leave to cool. Treat spare tube in the same way.

A-3.2.2 *Manganous Oxide* — granular, 850 to 425 micron.

A-3.2.2.1 *Preparation of manganous oxide for use* — Adjust regenerator heater *R* at 480-500°C. Insert manganous oxide tube and pass a stream of hydrogen at about 20 litres per hour for 4 to 5 hours. The heater is then turned off and oxygen-free nitrogen or argon passed through the tube at about 3 litres per hour while it cools. When removing after cooling, both ends are sealed quickly with rubber tubing and glass plugs as the oxide now absorbs oxygen rapidly from the atmosphere and becomes useless. The regenerated manganous oxide in the tube should be grey-green in colour and when only 18 to 25 mm of the grey-green zone is left, the tube should be regenerated. The spare tube should be treated in the same way.

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A-3.2.3 Soda Asbestos — 10 micron to 1.14 mm size.

A-3.2.4 Palladium Chloride — The material needs treatment to obtain the correct granular size, as fine powder obstructs the gas flow.

A-3.2.4.1 Dissolve palladium chloride in concentrated nitric acid and evaporate to dryness. Dissolve the residue in concentrated hydrochloric acid and evaporate to dryness again. Repeat with hydrochloric acid and then break up the residue into granular particles of size about 1.5-3 mm. The tube supplied should last for months, but as a precaution there should be a few spares. A fresh tube, when first used will give copious amounts of hydrogen chloride vapour before settling down to a satisfactory 'check' and 'blank'.

A-3.2.5 Universal Indicator

A-3.3 Procedure — Switch on the wall switch (this will switch on palladium chloride heater) and adjust copper oxide heater dial for 250°C. After one hour connect check cylinder to inlet at *A* and by turning *B*, pass gas through bubbler *C*. Adjust gas flow visually to about 5 litres per hour. Turn stopcocks *D* and *E* to positions 1 and 2, respectively, and then turn *B* so that the gas passes through *D*, *E*, *G* and *U* and out through bubbler *V*. This operation purges the oxygen from the alumina tube. This oxygen would considerably shorten the life of the manganous oxide tube. After 5 minutes, open both taps on palladium chloride tube *J*, and turn stopcock *U* through 90° anti-clockwise to position 3, so that the gas is now passing through *G*, *H*, *J*, *K*, *L* and *M* to atmosphere. This rate of flow should be adjusted to 5 litres per hour. (The upper mark on the flowmeter indicates 5 litres per hour for argon and the lower mark for nitrogen.) Admit 10 ml of indicator solution to bubbler *K* slowly by means of stopcock *W*. If the solution is admitted too rapidly some of it may be forced up the gas tube which should not occur. The colour of the solution will probably turn to red-orange or green. Let the gas pass for 15 to 20 minutes. Discharge the liquid and recharge several times, if necessary, until a good match with the pH 9 colour is obtained. Start stopwatch and note the time in minutes for colour change to pH 7. By reference to the calibration chart the hydrogen content of the mixture may be obtained. If the colour change is slow, the time may be taken for pH 8 or 8.5. The hydrogen content obtained for the check cylinder should be within 2 parts per million of the known figure. A low figure is usually explained by incomplete removal of oxygen and a high figure by a high rate of gas flow. When a reasonable figure has been obtained, the copper oxide furnace should be switched into the circuit by turning stopcock *D* through 90° anti-clockwise to position 3, and stopcock *E* through 90° anti-clockwise to position 4. (It is advisable when first using the copper oxide tube to blow the gas for ten minutes through *V* to atmosphere. While doing this, the stopcocks on the palladium chloride tube *J* should be shut.) A result of 1 ppm hydrogen or less should now be obtained.

A-3.3.1 Repeat the procedure given in A-3.3 with the sample introduced at inlet A.

A-4. DETERMINATION OF CARBON DIOXIDE

A-4.0 Principle—A known volume of the gas is passed through baryta solution and the amount of carbon dioxide absorbed is estimated from the amount of baryta solution neutralized.

A-4.1 Apparatus—The apparatus consists of two spiral absorbers, R_1 and R_2 , placed in series, R_2 being provided with soda-lime tube as shown in Fig. 4.

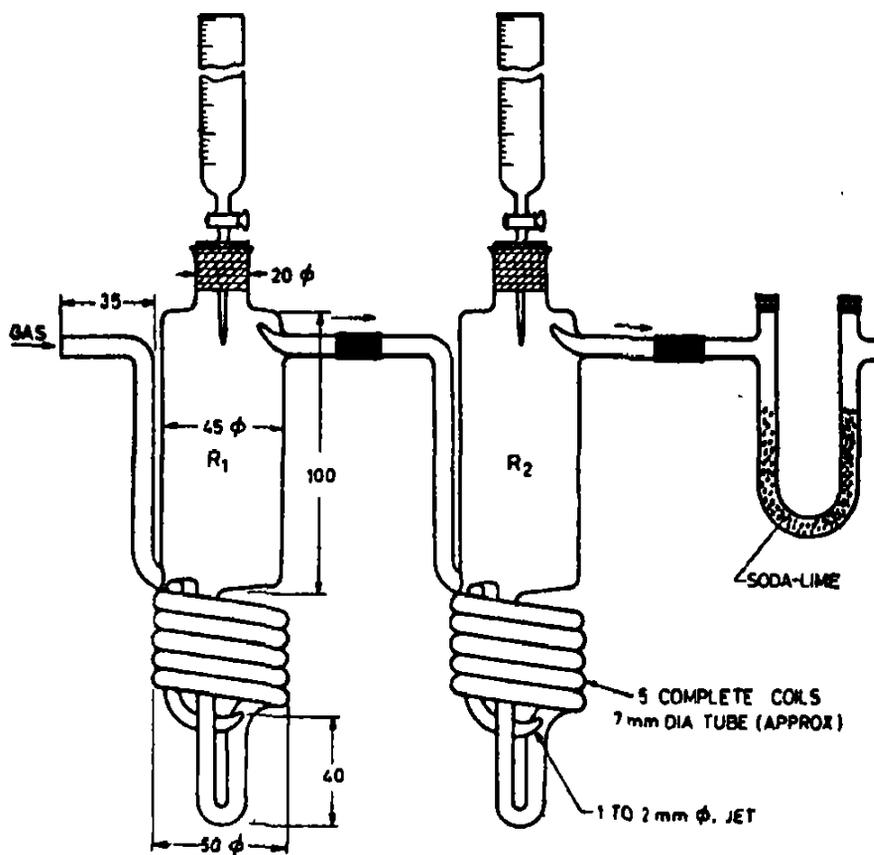


FIG. 4 SPIRAL ABSORBERS FOR THE DETERMINATION OF CARBON DIOXIDE

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A-4.2 Reagents

A-4.2.1 *Standard Hydrochloric Acid*— 0.02 N.

A-4.2.2 *Phenolphthalein Indicator*— Dissolve 0.50 g of phenolphthalein in 100 ml of rectified spirit (see IS : 323-1959*).

A-4.2.3 *Barium Hydroxide Solution*— Dissolve about 4 g of barium hydroxide in 2 000 ml of water in a large flask. Close the flask and shake until the crystals have completely disappeared and a light, insoluble powder of barium carbonate remains. Allow the solution to stand for two days, until the barium carbonate has completely settled; siphon it into a bottle through which a current of air (free from carbon dioxide) has been passed for 2 hours previously. Connect this bottle with a soda-lime tube and with a burette as shown in Fig. 5.

A-4.3 Procedure— Take 25 ml of baryta solution in each absorber, R_1 and R_2 , and add water to about half their height. Pass the gas through the spiral absorbers at the rate of about 5 litres per hour. Measure the rate of flow of the gas by a suitable device and record the temperature and atmospheric pressure at regular intervals. Pass 50 litres of gas, keeping the temperature and pressure of the gas constant. Titrate individually the baryta solution in both the absorbers with standard hydrochloric acid using phenolphthalein indicator and find out the total volume of standard hydrochloric acid used in the two titrations.

A-4.3.1 Carry out a blank titration with an equal volume of baryta solution used in the two absorbers.

A-4.4 Calculation

$$\text{Carbon dioxide, parts per million, by volume} = \frac{623.97 (V_1 - V_2) (273 + t) N}{p}$$

where

$V_{???$ = volume in ml of standard hydrochloric acid used for the blank titration of baryta solution,

V_Y = volume in ml of standard hydrochloric acid used for baryta solution after absorbing carbon dioxide,

$???$ = average temperature in degrees Celsius,

N = $???$ of the standard hydrochloric acid, and

p = average pressure in millimetres of mercury,

???

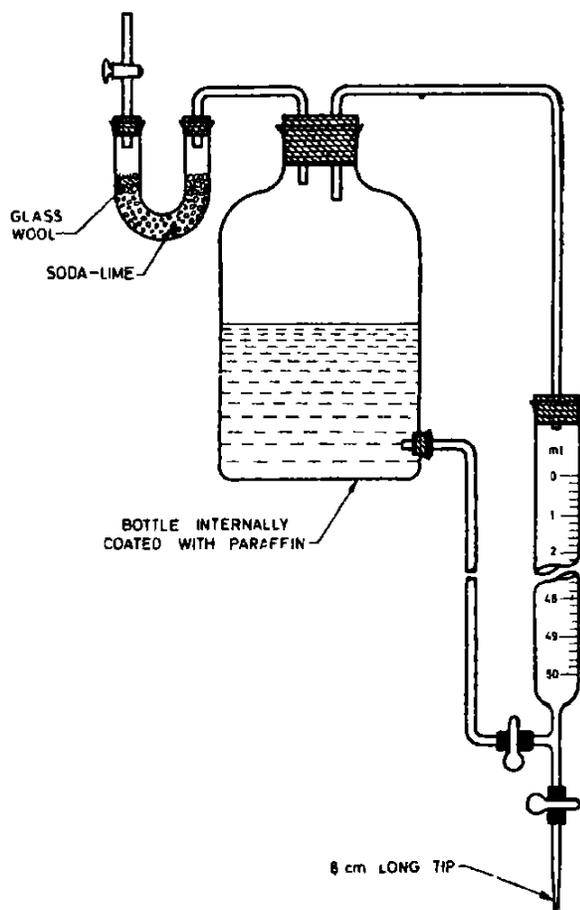


FIG. 5 BOTTLE FOR STORING BARIUM HYDROXIDE SOLUTION

A-5. DETERMINATION OF OTHER CARBON COMPOUNDS

A-5.0 Principle—A sample of the gas is passed over activated copper oxide catalyst at 600°C and the amount of carbon dioxide produced by the oxidation of hydrocarbons is titrated with standard barium hydroxide solution.

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A-5.1 Apparatus—The apparatus is the same as that used for the determination of carbon dioxide (A-4.1) with an additional furnace placed before the absorbers. This furnace holds the tube containing the activated catalyst.

A-5.2 Reagents

A-5.2.1 Activated Copper Oxide (???) Catalyst—This special catalyst is prepared as follows:

Grind together 99 percent by weight of copper oxide with one percent of ferric oxide. When the mixture is well ground and intimately mixed, add to eighty parts by weight of the mixture twenty parts of finely ground kaolin, mix the whole well to a stiff paste with water and squeeze it through a tube to form threads. Dry the threads, break them to convenient length, and fire them at a temperature not exceeding 600°C. Pack them into the reaction tubes so that the whole tube is full of catalyst. To regenerate the catalyst, circulate air through them at their working temperatures, higher temperatures shall not be used.

A-5.2.2 Standard Barium Hydroxide Solution — 0.05 N.

A-5.2.3 Standard Hydrochloric Acid — 0.002 N.

A-5.3 Procedure—Pass the gas sample through the soda asbestos tube on the inlet side at the rate of 3 to 5 litres per hour to absorb the free carbon dioxide from the sample. The gas then passes through the tube containing the catalyst which is heated to 600°C in an electric furnace. The combustion products then pass into the spiral bubblers in series each containing standard barium hydroxide solution. Measure the volume of the gas passed with a suitable meter placed in series. The actual gas sample that has to be passed through the apparatus depends upon the amount of carbonaceous compounds present in the original sample. Generally, for an accurate estimation, 50 litres of the gas are required. When the required volume of the gas sample has been passed, titrate the excess barium hydroxide solution in the bubbler for removing free carbon dioxide with standard hydrochloric acid and calculate the amount of free carbon dioxide. Titrate the excess barium hydroxide in the bubblers for absorbing the combustion products with standard hydrochloric acid. The carbonaceous matter shall be expressed as carbon dioxide.

A-5.3.1 Carry out a blank titration on 25 ml of the standard barium hydroxide solution.

A-5.4 Calculation

Other carbon compounds (excluding free carbon dioxide) expressed as carbon dioxide, parts per million by volume

$$= \frac{311.98 (V_1 - V_2) (273 + t) N}{V \times p}$$

where

V_1 = volume in ml of standard hydrochloric acid used for the blank titration of baryta solution,

V_2 = volume in ml of standard hydrochloric acid used for baryta solution after absorbing carbon dioxide,

t = average temperature in degrees Celsius,

N = normality of the standard hydrochloric acid,

V = volume in litres of gas passed, and

= average pressure in millimetres of mercury.

NOTE 1 — When the apparatus is first assembled, pass pure oxygen through the catalyst tube at 600°C until a satisfactory blank of few parts per million is obtained.

NOTE 2 — The apparatus should be checked occasionally with pure nitrogen containing a known amount of methane.

A-6. TEST FOR CARBON MONOXIDE

A-6.0 Principle— Carbon monoxide, if present in the test gas, reacts with palladium sulphite adsorbed on silica gel in the indicator tube and produces a blue-grey colour.

A-6.1 Apparatus

A-6.1.1 Indicator Tubes— These tubes are obtainable from suppliers of scientific apparatus.

A-6.1.2 The assembly of the apparatus consists of an inlet for the gas through a T-piece to a test-tube, then to a bubbler containing water which is connected to the indicator tube. The outlet of the indicator tube is connected to another bubbler containing water and then the gas escapes to atmosphere.

A-6.2 Procedure

A-6.2.1 Pour water in the first bubbler so that bottom of the glass tube dipping in it is covered by 75 mm of water. Introduce sufficient water in to the two bubblers so that the bottoms of the inlet tubes are covered by 12 mm of water.

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A-6.2.2 Connect the cylinder to be tested to T-piece with a piece of rubber tubing and adjust the gas flow so that the gas is bubbling freely up through the water in the test-tube. Observe that the gas is flowing through bubblers at equal rates. Immediately before the test, remove the indicator tube from the sealed envelope and connect between two bubblers. Allow gas to flow through the apparatus for four minutes. Compare the colour of the granules in the indicator tube with those in a sealed unused indicator tube. Absence of a brown ring or change of colour of the granules in the indicator tube indicates absence of carbon monoxide in the gas sample.

A-7. DETERMINATION OF MOISTURE

A-7.1 Method 1

A-7.1.0 Principle — Nitrogen at a fixed pressure of 21.0 kg/cm² is passed over a polished surface which is maintained at a desired temperature. When the dew point is reached a film of moisture or ice will be formed on the polished surface which sets as an opaque screen and a definite image of the lighted filament which is focussed on the surface becomes visible in the viewing device. The temperature at which a film of moisture is just formed (the dew point) under the experimental conditions is a measure of the moisture content.

A-7.1.1 Apparatus — All Ministry hygrometer or a similar one of equivalent accuracy.

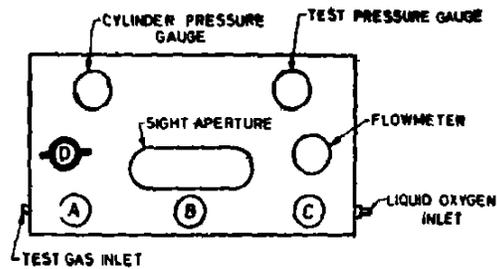
A-7.1.1.1 The essential features of the instrument are an electric supply system, an optical system and a liquid oxygen supply system. The assembly of the Air Ministry Hygrometer is illustrated in Fig. 6.

A-7.1.1.2 The manufacturer's instructions for handling the instrument shall be closely followed. Hygrometers having a built in photoelectric device to indicate the formation of a film of moisture may also be conveniently used.

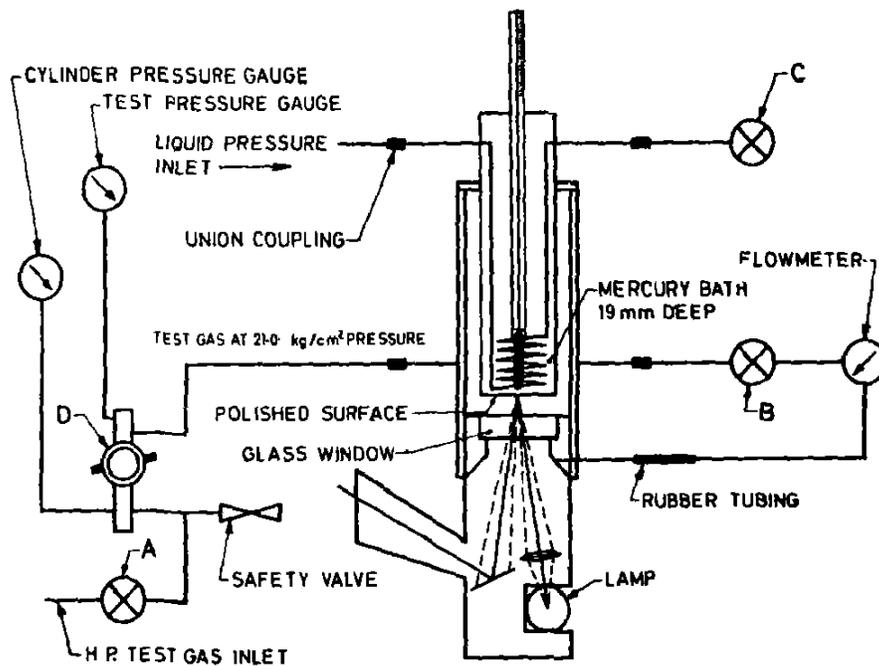
A-7.1.2 Procedure

A-7.1.2.1 Conditioning of the optical system — Remove the rubber bung from the orifice on top of apparatus and insert the unsharpened end of a lead pencil, 10 cm long, until the bottom of mercury chamber is reached. Release pressure on pencil and if the chamber contains any mercury, the pencil will rise and float on the surface of the liquid. The vertical movement of the pencil should be approximately 18 mm. If it is less than this, mercury should be added until the necessary height is obtained. When the correct amount of mercury has been added, insert the thermometer and metal case through the rubber bung supplied (with central hole) until the thermometer bulb rests in the mercury on the bottom of the

compartment. The bulb should project below the sheath supplied with the thermometer. When these instructions have been complied with, make sure that all the three panel valves, *A*, *B* and *C* in Fig. 6A, are shut and connect a 750-litre tested and certified cylinder to high pressure inlet on the left hand side of the set by means of the long adaptor supplied with the set and high pressure connecting tube. Open fully the cylinder valve with care and remedy the leaky joints. Open valve *A* slowly and adjust,



6A



6B

FIG. 6 AIR MINISTRY HYGROMETER

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if necessary, regulator *D* so that a pressure of 21.0 kg/cm² is indicated on test pressure gauge. The cylinder pressure will now be indicated on the cylinder pressure gauge. If the apparatus is in a satisfactory condition no leaks will be observed, but attention should be given to some of the joints and to the safety valve at the back of the set which may have to be adjusted until it just functions at 120 atm. The safety valve on the pressure regulator may also require adjustment so that it does not blow below a pressure of 21.0 kg/cm². Shut *A* and slowly open *B* until pressure in the apparatus falls to atmospheric, carefully noting that rate of flow as shown by flowmeter does not exceed 15 litres per minute. Following these adjustments it is necessary to purge the set free from moisture. Close the cylinder valve and slowly open *A* to vent the pressure in the connection. Then fully open both *A* and *B* very carefully, open the cylinder valve sufficiently to cause the flowmeter pointer to move 6 to 12 mm from its zero position. If gross contamination is absent the set is conditioned in a few minutes.

A-7.1.2.2 The valve of a cylinder to be tested should be momentarily opened and then closed to blow out any moist air in the neck before coupling the cylinder to the high pressure connecting tube. Open valves *A* and *B* fully and repeat the moisture purging operation described in **A-7.1.2.1**. Screw the adaptor into a ten-litre liquid oxygen vessel, making gas tight joint with the rubber washer supplied. Connect the liquid outlet tube (that is the tube reaching into the liquid in vessel) by rubber tubing to the liquid inlet valve on set using the adaptor supplied. This connection should be as short as possible and lagged with asbestos rope. The gas inlet of the adaptor on the liquid oxygen vessel should be connected by the special safety rubber tubing to a 2.8 m³ oxygen cylinder by means of a low pressure regulator set to a pressure of 0.70 kg/cm². When the valve on regulator on oxygen gas cylinder is opened slightly, gas passes from this cylinder and the pressure of the gas on the liquid oxygen in the vessel forces the liquid oxygen through the cooling coil in the apparatus when valve *C* (Fig. 6B) is opened. Open valve *C* fully so that the temperature falls to -27°C. This may be done in approximately 5 minutes. Valve *C* is then checked to maintain the temperature while *A* is closed momentarily to allow the cylinder valve to be opened more fully. *A* is then progressively opened until the flowmeter registers slightly more than the test rate of 15 litres (about 5 mm beyond the mark), then slowly close *B* until the test pressure gauge to the right registers 21.0 kg/cm², and the cylinder pressure gauge rises but does not exceed 30 atm. If these figures are not attained simultaneously, valves *A* and *B* are suitably adjusted. It is possible that regulator *D* may require re-adjustment to attain the 21.0 kg/cm² test pressure. It should be turned clockwise to increase the pressure. View the image of the lamp filament through the sight box. Allow the temperature to rise or go down by adjusting the valve *C*. As soon as an image becomes brighter than a faint image, the temperature shall be noted. Repeat by raising the temperature and then cooling again slowly.

Note the temperature of return of the bright image. Approach the same dew-point from cooler side also and note the temperature at which a bright image becomes faint.

A-7.1.3 Calculation and Report — From the chart supplied, calculate the moisture content corresponding to the dew-point obtained by averaging all readings. Report the moisture content in grams per cubic metre.

A-7.2 Method 2

A-7.2.0 Principle — A gas at atmospheric pressure is compressed and the pressure suddenly released, to cool the gas to the lower temperature. When the compression and expansion take place under adiabatic conditions, that is, without gain or loss of the original heat, the lower temperature upon expansion may be calculated by formula, provided the temperature and pressure before expansion are known.

A-7.2.1 Apparatus

A-7.2.1.1 Adiabatic dew-point meter — It consists essentially of the following components:

- a) *A sensitive thermometer*
- b) *Observation window*
- c) *Pressure ratio gauge* — It gives direct reading of the ratio of pressure of atmosphere and observation chamber.
- d) *A double acting type pump* — It keeps dry gas on both sides of the piston.
- e) *An operating valve* — When depressed, it allows the gas sample to expand rapidly and switches on the lamp simultaneously, illuminating the mist chamber.
- f) *A Purging valve* — It blows out all the air out of the apparatus before the test.

A-7.2.1.2 Dew-point calculator — A disc type calculator which converts the pressure ratios to dew-points on Celsius scale.

A-7.2.2 Procedure — Connect the source of gas or air to the inlet on the front panel of the dew-point meter and open the purging valve. Draw the gas through the instrument by means of its hand pump until the air originally present in it is purged. Close the purging valve and draw a sample of the gas into the instrument and compress the pump to a pressure above that of atmosphere. A pressure ratio gauge indicates the amount of compression and the temperature of the gas is read from the thermometer on the panel. Then release the pressure suddenly by depressing quickly the operating valve and watch the observation window of the expansion chamber for the appearance of mist. To enable the mist to be seen clearly a lamp below the chamber lights up simultaneously with the depression of the valve. If no mist appears repeat the test at slightly higher compression until mist is seen. Note the pressure ratio at this point. Repeat the test.

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A-7.2.3 Calculation and Report—Average the readings and calculate the dew-point from the dew-point calculator. The moisture content is calculated from a standard chart.

A P P E N D I X B

(Clause 5.1)

SAMPLING OF NITROGEN

B-1. SCALE OF SAMPLING

B-1.1 Lot—In any consignment, all cylinders charged during one work-shift from one charging manifold shall be grouped together to constitute a lot.

B-1.2 The number (n) of cylinders to be selected from each lot shall depend on the lot size (N) and shall be obtained from col 2 of Table 2 for the corresponding lot size in col 1.

TABLE 2 NUMBER OF CYLINDERS TO BE SELECTED

LOT SIZE	SAMPLE
N	n
(1)	(2)
Up to 50	5
51 to 100	8
101 „ 150	15
151 „ 300	20
301 and above	25

B-1.2.1 These cylinders shall be selected at random. To ensure the randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used (*see also* IS : 4905-1968*). In case such a table is not available, the following procedure shall be adopted:

Starting from any cylinder in the lot, count them as 1, 2, 3, . . . , etc, up to r and so on in one order, where r is the integral part of N/n . Every r th cylinder thus counted shall be withdrawn.

*Methods for random sampling.

B-2. TEST SAMPLES

B-2.1 One sample of gas shall be drawn from each cylinder selected as in **B-1.2** and **B-1.2.1** and shall be the individual test sample from each cylinder.

B-3. NUMBER OF TESTS

B-3.1 All the individual test samples from each lot prepared as in **B-2.1** shall be tested separately for all the requirements given in Table 1.

B-4. CRITERION FOR CONFORMITY

B-4.1 A lot shall be declared as conforming to the requirements of this specification if all the individual test results satisfy the relevant requirements given in Table 1.

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H No 15, Sector-3, PARWANOO, Distt Solan (HP)	173220	235 436
Plot No A-20-21, Institutional Area, Sector 62, Goutam Budh Nagar,	NOIDA 201307	240 2206
Patliputra Industrial Estate, PATNA	800013	226 2808
Plot Nos 657-660, Market Yard, Gultkdi, PUNE	411037	2427 4804
"Sahajanand House" 3 rd Floor, Bhaktinagar Circle, 80 Feet Road,	RAJKOT 360002	237 8251
TC No 2/275 (1 & 2), Near ??? Corporation of India, Kesavadasapuram-Ulloor Road,	Kesavadasapuram, THIRUVANANTHAPURAM 695004	255 7914
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Annex I

Gas Detector Tubes (Alternate Test Methods)

Gas detector tubes are cylindrical, sealed tubes consisting of an inert transparent material and are constructed to allow the passage of gas. They contain reagents adsorbed onto inert substrates that are suitable for the visualization of the substance to be detected and, if necessary, they also contain preliminary layers and/or adsorbent filters to eliminate substances that interfere with the substance to be detected. The layer of indicator contains either a single reagent for the detection of a given impurity or several reagents for the detection of several substances (monolayer tube or multilayer tube).

The test is carried out by passing the required volume of the gas to be examined through the indicator tube. The length of the colored layer or the intensity of a color change on a graduated scale gives an indication of the impurities present.

The calibration of the detector tubes is verified according to the manufacturer's instructions.

Procedure: Examine according to the manufacturer's instruction or proceed as follows:

The gas supply is connected to a suitable pressure regulator and needle valve. Connect the flexible tube fitted with a Y-piece to the valve and adjust the flow of gas to be examined to purge the tubing in order to obtain an appropriate flow (Figure 7). Prepare the indicator tube and fit to the metering, following the manufacturer's instructions. Connect the open end of the indicator tube to the short leg of the tubing and operate the pump by the appropriate number of strokes to pass a suitable volume of gas to be examined through the tube. Read the value corresponding to the length of the colored layer or the intensity of the color on the graduated scale. If negative result is achieved, indicator tubes can be verified with a calibration gas containing the appropriate impurity.

In view of the wide variety of available compressor oils, it is necessary to verify the reactivity of the oil detector tubes for the oil used. Information on the reactivity for various oils is given in the leaflet; the tube manufacturer must verify the reactivity and if necessary, provide a tube specific for this oil.

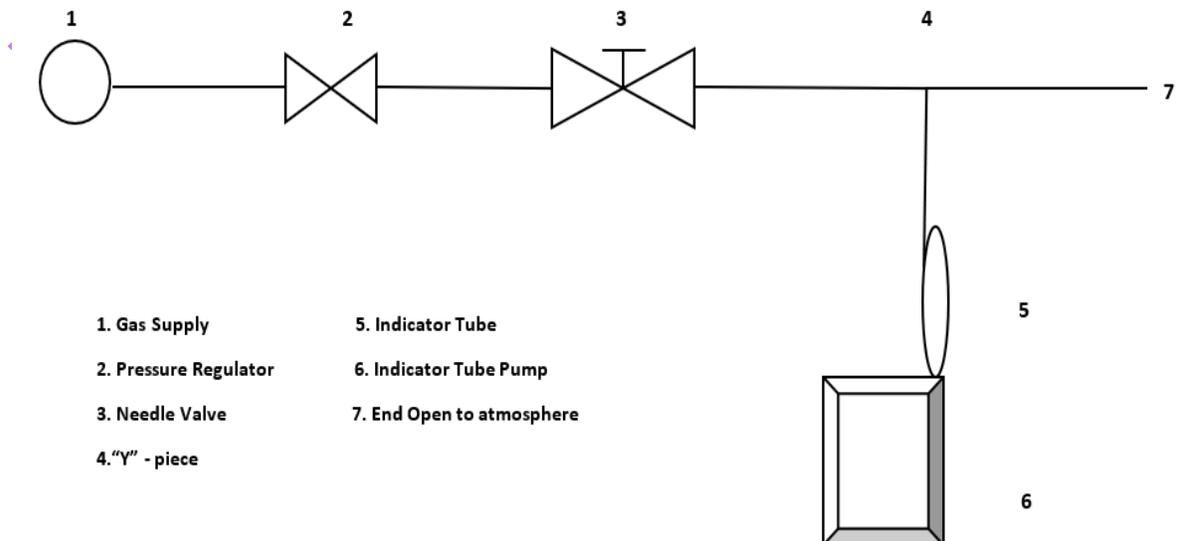


Figure 7: Apparatus for gas detector tube

Arsine detector tube

Sealed glass tube containing adsorbent filters and suitable supports for the gold salt or other appropriate indicator. The minimum value indicated is 0.25 ppm or less with a relative standard deviation of at most 20 percent.

Carbon Dioxide Detector Tube

Sealed glass tube containing adsorbent filters and suitable supports for hydrazine and crystal violet indicators. The minimum value indicated is 100 ppm with a relative standard deviation of at most 15 per cent.

Carbon Monoxide Detector Tube

Sealed glass tube containing adsorbent filters and suitable supports for di-iodine pentoxide, selenium dioxide and fuming sulfuric acid indicators. The minimum value indicated is 5 ppm or less, with a relative standard deviation of at most 15 per cent.

Hydrogen sulfide detector tube

Sealed glass tube containing adsorbent filters and suitable supports for an appropriate lead salt indicator. The minimum value indicated is 0.2 ppm or less, with a relative standard deviation of at most 10 per cent.

Nitrogen Monoxide and Nitrogen Dioxide Detector Tube

Sealed glass tube containing adsorbent filters and suitable supports for an oxidizing layer [Cr (VI) salt] and the diphenylbenzidine indicator. The minimum value indicated is 0.5 ppm with a relative standard deviation of at most 15 per cent.

Oil Detector Tube

Sealed glass tube containing adsorbent filters and suitable supports for the sulfuric acid indicator. The minimum value indicated is 0.1 mg/m³ with a relative standard deviation of at most 30 percent.

Phosphine detector tube

Sealed glass tube containing adsorbent filters and suitable supports for the gold salt or other appropriate indicator. The minimum value indicated is 0.2 ppm or less, with a relative standard deviation of at most 20 per cent.

Sulfur Dioxide Detector Tube

Sealed glass tube containing adsorbent filters and suitable supports for the iodine and starch indicator. The minimum value indicated is 0.5 ppm with a relative standard deviation of at most 15 per cent.

Water Vapour Detector Tube

Sealed glass tube containing adsorbent filters and suitable supports for the magnesium perchlorate indicators. The minimum value indicated is 67 ppm or less, with a relative standard deviation of at most 20 per cent.

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