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Indian Standard
SPECIFICATION FOR CARBON DIOXIDE
(Second Revision)

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

Gr 5

March 1967

Indian Standard
SPECIFICATION FOR CARBON DIOXIDE
(Second Revision)

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Indian Standard
SPECIFICATION FOR CARBON DIOXIDE
(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 9 September 1966, after the draft finalized by the Industrial Gases Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first issued in 1956 and subsequently revised in 1963. In the first revision the limits for acid and sulphur dioxide, phosphine and hydrogen sulphide in the material were prescribed. Also for carbon monoxide, a limit test was prescribed in place of the requirement of 0·5 percent laid down earlier. The requirement of moisture was prescribed at 0·1 percent for all the end uses.

0.2.1 After the first revision, there was further consideration on the basis of suggestions from the industries that two grades of carbon dioxide, covering most of the end uses, were adequate. This revision had been made to cover the necessary requirements for the two grades of the material.

0.3 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 test for carbon dioxide.

1.2 This standard does not prescribe the requirements of carbon dioxide for medical use, which are covered by the Indian Pharmacopoeia.

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

2. GRADES

2.1 The gas shall be of two grades, namely:

a) Grade 1, suitable for use as a reagent and for welding purposes; and

b) Grade 2, suitable for beverages, fire extinguishers, refrigeration and general commercial purposes.

3. REQUIREMENTS

3.1 The material shall be odourless.

3.2 When tested according to the methods prescribed in Appendix A, the material shall comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR CARBON DIOXIDE

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST (REF TO CL No. IN APPENDIX A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Carbon dioxide, percent by volume, *Min*	99·7	99·0	A-2
ii)	Carbon monoxide, ppm, *Max*	10	10	A-3
iii)	Acid and sulphur dioxide	To pass test	To pass test	A-4
iv)	Phosphine and hydrogen sulphide	To pass test	To pass test	A-5
v)	Moisture, percent by volume, *Max*	0·001	0·1	A-6
vi)	Arsenic, mg/normal m^3, *Max*	0·5	0·5	A-7
vii)	Oil, ppm by weight, *Max*	5	5	A-8

4. PACKING AND MARKING

4.1 The cylinders shall conform to the Gas Cylinder Rules 1940, of the Government of India, with such modifications or relaxations or both, as may be ordered from time to time by the Chief Inspector of Explosives, Government of India, or other duly constituted authority.

4.2 The packing, marking, painting, labelling and transport of cylinders shall be in accordance with the requirements of the Gas Cylinder Rules 1940 with such modifications or relaxations or both as may be ordered from time to time by the Chief Inspector of Explosives, Government of India, or other duly constituted authority. The cylinders shall also be marked as shown in Fig. 2 of IS : 1260-1958*.

4.3 The cylinders may also carry suitable tags, bearing the ISI Certification Mark for the contents of the cylinders.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Scale of Sampling

5.1.1 In any consignment all cylinders charged during one work shift from one charging manifold shall be grouped together to constitute a lot.

5.1.2 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this specification.

5.2 Method of Sampling

5.2.1 The number of cylinders to be selected from each lot shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CYLINDERS TO BE SELECTED

LOT SIZE N (1)	SAMPLE SIZE n (2)
Up to 25	3
26 " 65	4
66 " 110	5
111 " 180	7
181 " 300	10

5.2.2 The cylinder shall be selected at random and to ensure randomness of selection a random number table, as agreed to between the purchaser and the supplier, shall be used. In case such a table is not available, the

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*Code of symbols for labelling of dangerous goods. (Since revised and split into various parts).

following procedure is recommended for use:

> Starting from any cylinder in the lot, count them as 1, 2, 3……, up to r and so on, where r is the integral part of N/n (N being the number of cylinders in the lot and n the number to be selected as sample). Every rth cylinder thus counted shall be withdrawn to constitute a sample, till the required number of cylinders is obtained.

5.3 Number of Tests

5.3.1 From each of the cylinders selected according to **5.2.2** two separate samples of the gas shall be drawn.

5.3.2 All the samples of the gas shall be tested individually for all the requirements given in **3.1**, **3.2** and Table 1.

5.4 Criterion for Conformity

5.4.1 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 **3** and Table 1.

APPENDIX A

(*Clause* 3.2)

ANALYSIS OF CARBON DIOXIDE

A-1. QUALITY OF REAGENTS

A-1.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

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

A-2. DETERMINATION OF CARBON DIOXIDE

A-2.1 Apparatus — The apparatus consists of the following parts assembled as shown in Fig. 1.

A-2.1.1 *Pipette (D)* — Semet Solvay modification of hempel pipette for the absorption of carbon dioxide.

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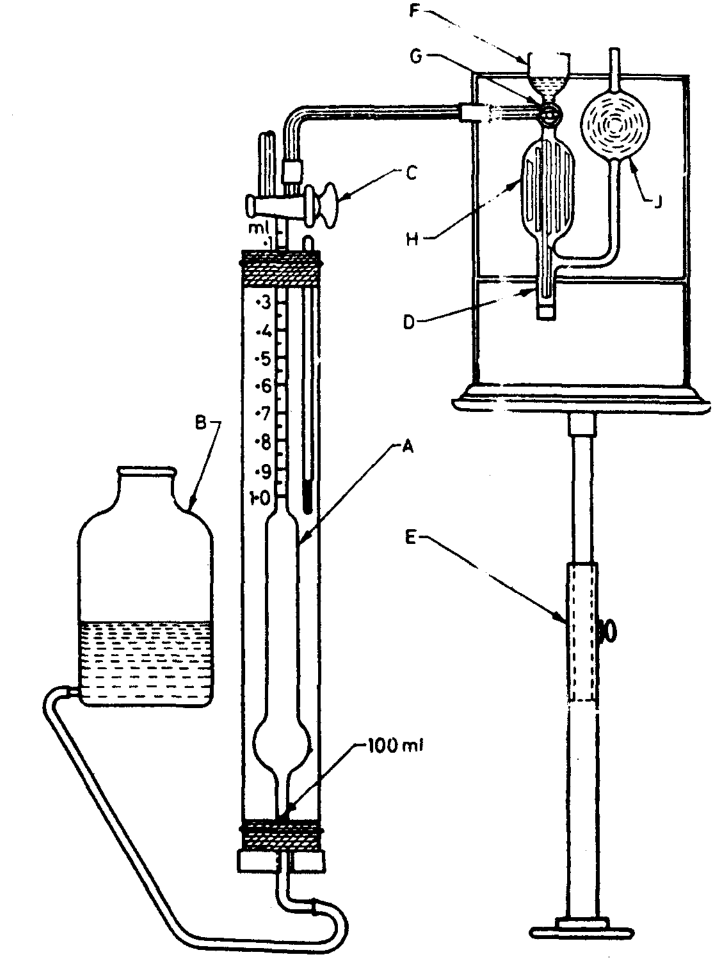
*Specification for water, distilled quality (*revised*). (Since revised).



A Burette
B Levelling bottle
C Three way stopcock
D Pipette
E Stand
F Cup
G Three way stopcock
H Pipette bulb
J Bulb

FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF CARBON DIOXIDE

A-2.1.2 *Burette* (*A*) — of 100 ml capacity with a graduated tube (at the top) of capacity 1·0 ml graduated in 0·05 ml beginning at the three way stopcock *C*. One branch of stopcock *C* communicates with the pipette *D* while the other branch with the sample to be analysed. The burette is enclosed in a jacket filled with water.

A-2.1.3 *Levelling Bottle* (*B*) — filled with acidulated water, saturated with the gas to be analysed and coloured red with methyl orange, to facilitate reading.

A-2.2 Reagents

A-2.2.1 *Acidulated Water* — containing 0·5 percent by weight of sulphuric acid.

A-2.2.2 *Potassium Hydroxide Solution* — Dissolve 360 g of potassium hydroxide in water and dilute to one litre.

A-2.3 Procedure

A-2.3.1 *Preparation of Apparatus* — Make sure that the temperature of water in the jacket and the levelling bottle *B* is about the same as the room temperature. Grease the stopcock *C* of the burette *A*, and test the burette and connections for gas tightness. To do this, connect *B* to the burette *A* with a rubber tubing and draw in 100 ml of air closing the stopcock *C* and reading the volume. Raise the levelling bottle *B* to the height of *C* and keep it in that position for 3 minutes. Large leaks will be observed by the meniscus gradually rising while smaller leaks will be disclosed by again reading the volume of air at the end of 3 minutes.

A-2.3.2 Turn the stopcock *C* so that the burette *A* communicates with the atmosphere through the left hand capillary and raise the levelling bottle *B* to force the air out of the burette *A*. When the water reaches the top of the capillary, close *C* by turning the stopcock through 90 degrees and then connect the left hand capillary with the vessel containing the sample of gas to be analysed. Lower *B*, open the connection to the sample reservoir and then open *C* drawing in about 25 ml of the gas. Close *C* while the gas is still entering. Then turn the stopcock *C* to connect the burette *A* with the right hand capillary. Drive out and discard the gas contained in the burette *A* as it usually contains some air. When water emerges from the top of the capillary, turn *C* to connect again with the gas sample reservoir, lower *B* and draw into the burette *A* slightly more than 100 ml of gas. Close *C* and disconnect it from the gas sample reservoir. Allow the burette to drain for 30 seconds. Then raise *B* until the meniscus is at 100 ml mark. Pinch the rubber tubing with one hand and with the other open *C* momentarily to the air through the right hand capillary tube and allow the excess gas to pass out bringing the gas in the burette *A* to atmospheric pressure. Immediately close *C* and bring the water in *B* and in burette *A* exactly to the same level. Read and record the volume of gas. The volume of gas should be 100 ml in the burette *A*.

A-2.3.3 Place the pipette *D* on the stand *E* and adjust height. Fill the pipette *D* by pouring potassium hydroxide solution through a funnel inserted in the open stem of the bulb *J* so that the meniscus of the potassium hydroxide solution is near the lower edge of the stopcock *G*. Before connecting the pipette *D* to the right hand capillary of the burette *A* put a little acidulated water in cup *F* of the pipette *D* and by turning the three way stopcock *G* allow acidulated water to fill the capillary tube completely, and then close stopcock *G*. Connect the pipette *D* with the burette *A* with a rubber tube, squeezing the rubber tube between the thumb and forefinger and slipping it over the capillary of the burette without introducing air. Raise *B* sufficiently to put the gas in the burette under slight pressure. Open *C* to connect to the right hand capillary and then cautiously turn *G* of the pipette to allow the gas to drive the water in the capillary back into cup *F*. When the gas reaches stopcock *G* turn it to communicate with pipette bulb *H* and pass gas into the pipette *D* by raising *B* until the water reaches the top of the burette *A*. Allow it to remain for 5 to 7 seconds and then lower *B* and return all the gas to the burette *A*. Repeat this process 3 to 4 times. Lower *B* and draw the gas back into the burette until the potassium hydroxide solution reaches its initial mark on the capillary below *G*. Turn *G* to connect with *F* and draw acidulated water from the cup through the capillary to *G* and close *C*. Allow the burette to drain for 30 seconds and take the reading of the burette. Repeat this operation by again transferring the gas to the pipette to ensure complete absorption. Two consecutive readings should agree within 0·05 ml.

A-2.4 Calculation

$$\text{Carbon dioxide, percent by volume} = \frac{100\,(V - V_1)}{V}$$

where

V = volume in ml of the gas sample taken for the test, and

V_1 = volume in ml of the gas after absorption.

A-3. DETERMINATION OF CARBON MONOXIDE

A-3.1 Method — A known volume of the gas is passed through hot iodine pentoxide. Any carbon monoxide present is thus oxidized to carbon dioxide which is estimated by absorption in baryta solution.

A-3.2 Apparatus — The apparatus shall consist of the following components assembled as shown in Fig. 2.

A-3.2.1 *Mercury Filled Bye-Pass Safety Valve* (*A*)

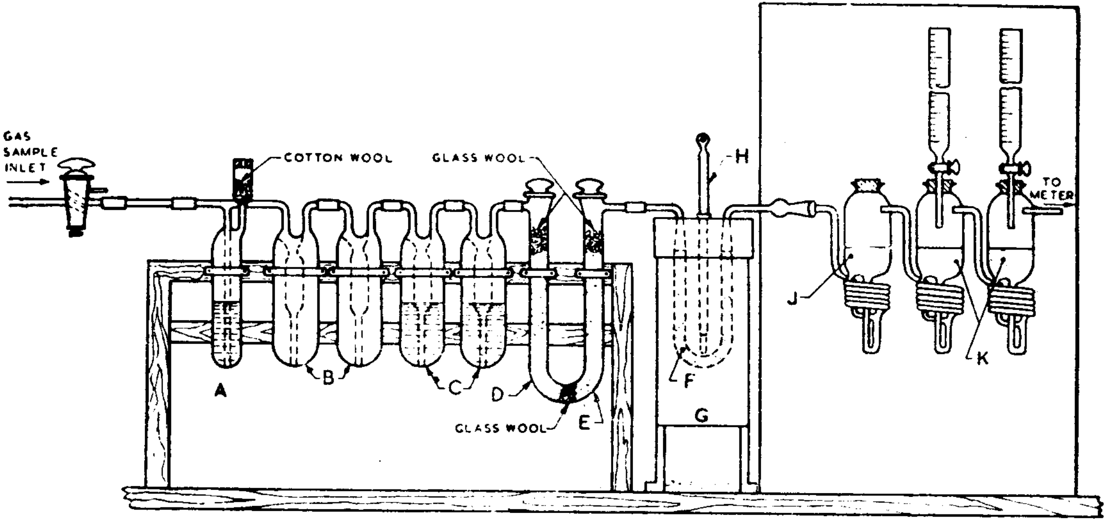


Fig. 2 Assembly of Iodine Pentoxide Apparatus for the Determination of Carbon Monoxide

A-3.2.2 *Gas Scrubbers* (*B*) — Containing 40 percent potassium hydroxide solution.

A-3.2.3 *Gas Bubblers* (*C*) — containing saturated solution of chromic acid (*see* IS : 559-1954*) in concentrated sulphuric acid (*see* IS : 266-1961†).

A-3.2.4 *U-Tube* — one limb (*D*) containing soda asbestos and the other limb (*E*) containing magnesium perchlorate.

A-3.2.5 *U-Tube* (*F*) — containing iodine pentoxide.

A-3.2.6 *Hot Bath* (*G*) — either air or oil, electrically heated.

A-3.2.7 *Thermometer* (*H*) — reading up to 200°C, graduated to 0·5°C.

A-3.2.8 *Absorber* (*J*) — containing potassium iodide solution.

A-3.2.9 *Carbon Dioxide Absorbers* (*K*) — two, each containing baryta solution and fitted with a burette containing standard hydrochloric acid.

A-3.3 Reagents

A-3.3.1 *Standard Hydrochloric Acid* — approximately 0·02 N.

A-3.3.2 *Baryta 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. 3.

A-3.3.3 *Iodine Pentoxide*

A-3.3.4 *Potassium Iodide Solution* — Dissolve 5 g of potassium iodide crystals in 100 ml of water.

A-3.3.5 *Phenolphthalein Indicator* — Dissolve 0·50 g of phenolphthalein in 100 ml of rectified spirit (*see* IS : 323-1959‡).

A-3.4 Procedure

A-3.4.1 Take 25 ml of baryta solution in each carbon dioxide absorber *K* and add water to about half its height. Maintain the temperature of iodine pentoxide bath at 150°C. Flush the apparatus with about 5 litres of nitrogen. Pass the carbon dioxide mixed with an equal volume of pure nitrogen at the rate of about 5 litres per hour. Measure the rate of flow of

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*Specification for chromium trioxide (chromic acid), analytical reagent. [Since superseded by IS : 330 - 1963 Specification for chromium trioxide. (*first revision*.]

†Specification for sulphuric acid (*revised*).

‡Specification for rectified spirit (*revised*).

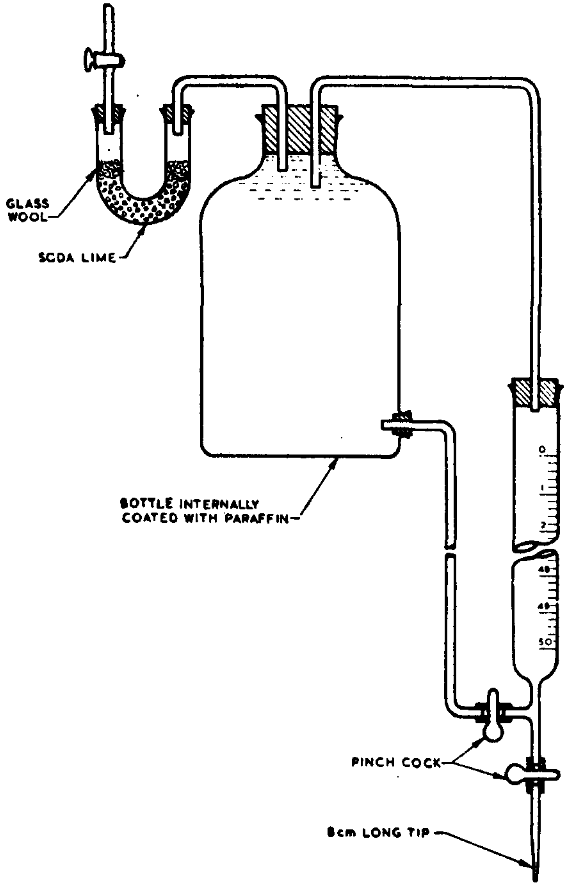


FIG. 3 BOTTLE FOR STORING BARYTA SOLUTION

the gas by a suitable device and record the temperature and atmospheric pressure at regular intervals. Pass 10 litres of the carbon dioxide, keeping the temperature and pressure of the gas constant. Titrate individually the baryta solution in both the absorbers with standard hydrochloric acid using phenolphthalein as indicator. Find out the total volume of standard hydrochloric acid used in the two titrations.

A-3.4.2 Carry out a blank titration with an equal volume of baryta solution used for absorption.

A-3.5 Calculation

$$\text{Carbon monoxide, ppm by volume} = \frac{3\,098{\cdot}0\,(V_1 - V_2)\,(273 + t)\,N}{}$$

where

V_1 = volume in ml of standard hydrochloric acid used for the blank titration of baryta solution in **A-3.4.2**,

V_2 = volume in ml of standard hydrochloric acid used for the titration of baryta solution in **A-3.4.1**,

t = average temperature in degrees centigrade,

N = normality of standard hydrochloric acid, and

p = average pressure in millimetres of mercury.

A-4. TEST FOR ACIDS AND SULPHUR DIOXIDE

A-4.1 Apparatus

A-4.1.1 *Nessler Tubes* — 50 ml capacity.

A-4.2 Reagents

A-4.2.1 *Methyl Orange Indicator Solution* — Dissolve 0·01 g of methyl orange in 100 ml of water.

A-4.2.2 *Standard Hydrochloric Acid* — 0·01 N.

A-4.3 Procedure — Take 50 ml of carbon dioxide-free cooled water in a vessel of such diameter that a hydrostatic column of 12 to 14 cm is produced. Pass carbon dioxide equivalent to 1 000 ml at normal temperature and pressure, through the water using a delivery tube having an orifice about 1 mm in diameter and extending to within 2 mm of the bottom of the vessel. Regulate the flow of gas so that about 15 minutes are taken for passing the gas. After passing the gas, transfer the liquid to a Nessler tube and add 0·1 ml of methyl orange, indicator solution. Carry out a control test in another Nessler tube, adding 50 ml of carbon dioxide free water, 1 mm of standard hydrochloric acid and 0·1 ml of methyl orange indicator solution.

A-4.3.1 The material shall be taken to have passed the test if the intensity of red colour of the test solution is not greater than that in the control test.

A-5. TEST FOR PHOSPHINE AND HYDROGEN SULPHIDE

A-5.1 Reagents

A-5.1.1 *Silver Nitrate Solution* — 10 percent.

A-5.1.2 *Ammonium Hydroxide* — 1 : 1 (*v/v*)

A-5.2 Procedure — Pass carbon dioxide equivalent to 1 000 ml at normal temperature and pressure through a mixture of 25 ml of silver nitrate solution, 5 ml of ammonium hydroxide and 20 ml of water.

A-5.2.1 The material shall be taken to have passed the test if no turbidity is produced and the solution is not darker than an equal quantity of test solution through which no gas has been passed.

A-6. DETERMINATION OF MOISTURE

A-6.1 Apparatus — The apparatus consists of the following parts assembled as shown in Fig. 4.

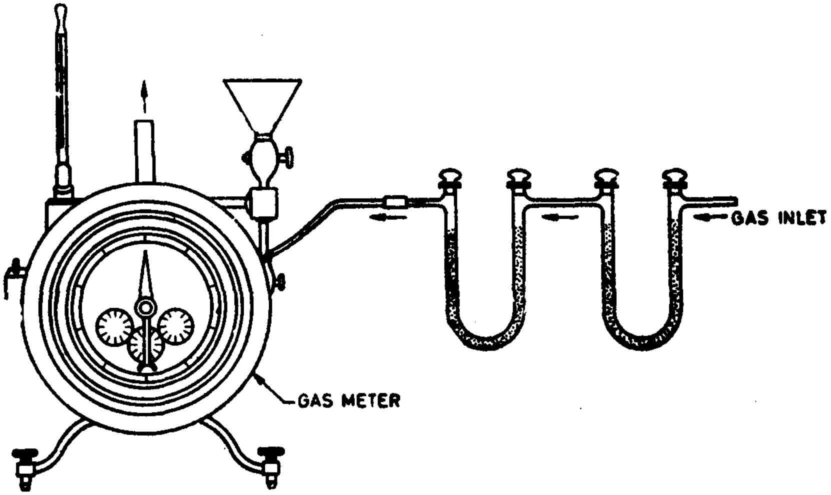


FIG. 4 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF MOISTURE

A-6.1.1 *Gas Meter* — accurate to 1 percent.

A-6.1.2 *Absorption Train* — three U-tubes containing phosphorus pentoxide connected in series to the gas meter. The one near to the gas meter will serve as a guard to prevent moisture from backing into the first two tubes.

A-6.2 Procedure — Connect the 3 tubes in series to the inlet of the gas meter and connect the free end of U-tubes to the gas sample. Allow the gas to flow through the train for a while to saturate the water in the gas meter with the gas as well as replacing the air in the first two U-tubes. Stop the gas flow, close the stopcocks of the U-tubes and weigh the first two U-tubes. Connect again, record the meter reading. Pass the gas through the train at the rate of 10 litres per hour for 4 to 5 hours. Close the gas supply and record the meter reading. Remove the first two U-tubes and weigh.

A-6.3 Calculation — Convert the volume of gas taken for the test to normal temperature and pressure. From the increase in weight, calculate the volume of moisture present on the basis that 1 g of water is equivalent to 1·244 litres of water vapour at normal temperature and pressure and express the result as percentage by volume.

A-7. TEST FOR ARSENIC

A-7.1 Apparatus — The apparatus shall consist of the following parts as shown in Fig. 5.

A-7.1.1 *Evolution Flask* — capacity 100 to 125 ml and scrubber with glass wool attached to the reaction flask by ground joint.

A-7.1.2 *Arsenic Absorber* — With a calibrated mark at 4 ml. This is connected to the scrubber by means of ball and socket joint through the side capillary tube of the absorber.

A-7.1.3 *Spectrophotometer* — with 10-mm cells.

A-7.1.4 *Dreschel Bottle* — with fitted tube.

A-7.1.5 *Gas Meter, Wet Type*

A-7.2 Reagents — Only arsenic free reagents should be used.

A-7.2.1 *Lead Acetate Solution* — Dissolve 10 g of lead acetate in 100 ml of water.

A-7.2.2 *Silver Diethyldithiocarbamate-Pyridine Solution* — Dissolve 1·0 g of pure dry silver diethyldithiocarbamate in 200 ml pyridine, store in amber coloured bottle.

A-7.2.3 *Potassium Iodide Solution* — Dissolve 15 g of potassium iodide in 100 ml distilled water.

A-7.2.4 *Standard Arsenic Solution* — Dissolve 1·320 g of arsenious oxide in the minimum volume of 1 M sodium hydroxide solution, acidify with dilute hydrochloric acid and make up to one litre in a volumetric flask. One millilitre contains 1 mg of arsenic. Dilute to get a solution containing 1 µg per ml.

A-7.2.5 *Concentrated Hydrochloric Acid*

A-7.2.6 *Stannous Chloride Solution* — Dissolve 40 g of stannous chloride ($SnCl_2 . 2H_2O$) in 100 ml concentrated hydrochloric acid.

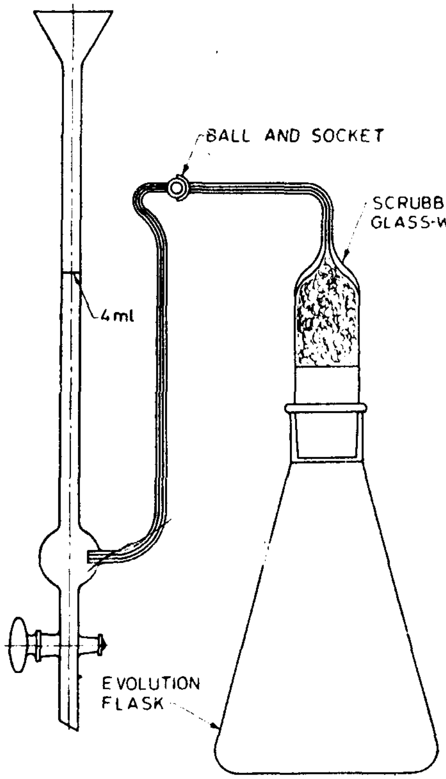


FIG. 5 ASSEMBLY OF APPARATUS FOR TESTING ARSENIC

A-7.2.7 *Zinc Powder* — 500 to 850 µ particle size.

A-7.2.8 *Potassium Hydroxide Solution* — 0·1 N.

NOTE — All glassware used should be thoroughly cleaned with either hot concentra ted sulphuric acid or boiling concentrated nitric acid, followed by rinsing with distille water, and then with acetone.

A-7.3 Procedure

A-7.3.1 Impregnate the glass wool (purified) in the scrubber with lead acetate solution. Charge the absorption tube with 4·00 ml of silver diethyldithiocarbamate reagent.

A-7.3.2 Prepare a calibration curve by pipetting suitable aliquots of the diluted standard arsenic solution into a series of clean evolution flasks. Cover the range 0 to 10 µg of arsenic. To each add 5 ml of concentrated hydrochloric acid, 2·0 ml of 15 percent potassium iodide solution and 8 drops of stannous chloride solution. Swirl the contents of the flasks and allow them to stand for 15 minutes to ensure complete reduction. Add 5·0 g of zinc and insert the hydrogen sulphide scrubber immediately. The evolution of arsine is 99 percent complete in 30 minutes and virtually complete in 40 minutes. If necessary, dilute the liquid in the arsine absorber with pure pyridine to the 4-ml mark and pass gentle stream of air through the absorber to mix the solution. Transfer the absorbing solution to a 10-mm cell and measure the transmittance at 540 mμ in the spectrophotometer. Repeat the procedure with the remaining flasks. Plot the transmittance of each aliquot (less that of the blank) against its arsenic content in μg.

A-7.3.3 Pass 25 to 50 litres of carbon dioxide through 25 ml of 0·1 N potassium hydroxide solution in 100 ml dreschel bottle at the rate of 10 litres per hour. The gas is measured by the gas meter placed after the bubbler. The water in the gas meter is saturated with the gas prior to the experiment.

A-7.3.4 Take an aliquot from the treated potassium hydroxide in **A-7.3.3** to give about 5·0 μg of arsenic and follow the same procedure as in **A-7.3.2.** From the transmittance obtained at 540 mμ evaluate the arsenic content of the sample by reference to the calibration graph

A-8. METHOD FOR THE DETERMINATION OF OIL

A-8.1 Apparatus

A-8.1.1 *Two Gage Bubbers* — shown in Fig. 6.

A-8.1.2 *Dreschel Bottle* — 250 ml capacity.

A-8.1.3 *Flow Meter* — to measure 200 to 2 000 ml of carbon dioxide per minute.

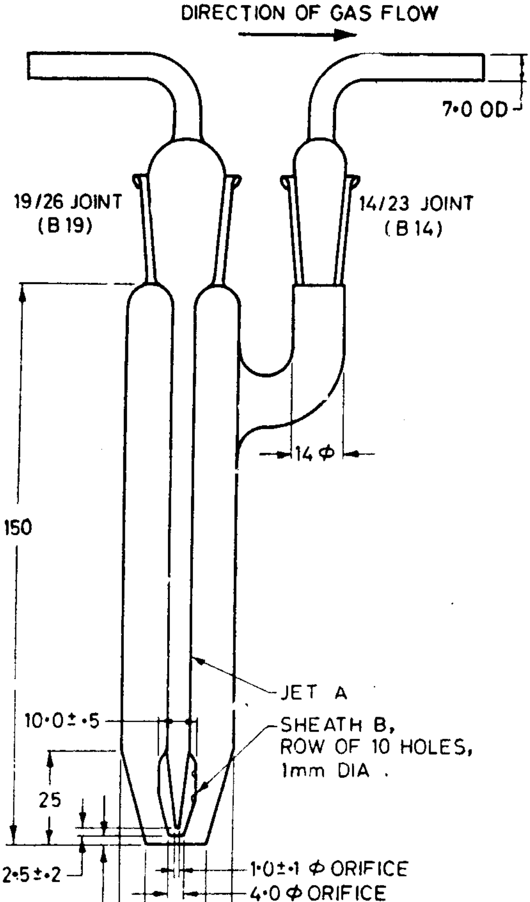
A-8.1.4 *Wet Gas Meter* — calibrated for 1 or 2·5 litres per revolution.

A-8.1.5 *Infra-Red Spectrometer and Accessories* — to measure at 3·46 μ.

A-8.2 Reagents

A-8.2.1 *Carbon Tetrachloride* — Distilled, analytical reagent quality. The infra-red absorption at 3·46 μ of each fresh supply of this reagent should be determined before the use to ensure that it is of suitable quality.

A-8.2.2 *Standard Oil Solution* — Dissolve 0·020 0 g of liquid paraffin, in carbon tetrachloride and dilute to exactly 100 ml with more carbon tetrachloride. One millilitre of this solution contains 200 μg of oil.



All dimensions in millimetres.

FIG. 6 GAGE HIGH SPEED BUBBLER FOR USE IN DETERMINATION OF OIL IN CARBON DIOXIDE

A-8.3 Procedure

A-8.3.1 About 800 to 1 000 g of material is needed for each determination. A set of small sampling cylinders of 1·5 to 2 kg capacity with one valve at each end may be used for containing this amount of gas

NOTE — A set of cylinders should preferably be retained for sampling for oil and not used for other purposes.

A-8.3.2 *Preparation of Test Solution* — Place 100 ml of the carbon tetrachloride in the dreschel bottle and 25 ml in each gage bubbler. Connect the weighed sampling cylinder, supported vertically with the dip tube at the top to the evaporator and thence to the absorption train, flow meter and wet gas meter in series using short rubber connections. Alternatively, for this determination the sample cylinder may be supported vertically with the dip tube valve at the bottom; when this is done, omit the evaporating device and connect the absorption train to the top valve of the cylinder so that gaseous carbon dioxide can be withdrawn.

A-8.3.3 Totally expand the contents of the cylinder through the absorption train at the rate of about 1 000 ml per minute and read the volume of gas passed; this serves as a check on the amount of carbon dioxide used for the test.

NOTE — Some evaporation of the carbon tetrachloride will take place. If this is excessive, stop the gas flow and refill the absorption bottles noting the volume of carbon tetrachloride added.

A-8.3.4 Reweigh the sampling cylinder taking care to use the same valve fittings as at the first weighing. Dismantle the lower cylinder valve and wash the inside of the cylinder with 25 ml of carbon tetrachloride. Wash the interior of the evaporating device and valves with carbon tetrachloride and combine this solution with the cylinder washings and the bubbler contents. Adjust the combined solutions to some suitable definite volume, after previous evaporation, if this is considered necessary for the particular infra-red spectrometer to be used. This is the test solution.

A-8.3.5 *Preparation of Control* — Evaporate a volume of carbon tetrachloride equal to the total volume (including any additions) of carbon tetrachloride used in the absorption train plus washings to the volume of the combined solutions and subsequently treat it in the same way as the test solution. If there is definitely no blank on the carbon tetrachloride, it is sufficient to prepare a control of volume equal to that of the final test solution.

A-8.3.6 *Preparation of Calibration Chart* — Prepare suitable dilutions of the standard oil solution to cover the range of values within which the weight of oil in the sample is expected to be found. Measure the optical densities of this series of solutions as described above. Prepare a calibration chart by plotting the weights of oil against the corresponding optical densities.

A-8.3.7 *Measurement of Infra-Red Absorption of the Test Solution* — Following the manufacturer's instructions for the operation of the particular instrument used, determine the optical densities of the test solution and control at the wave length of maximum absorption (approximately 3·46 μ). From the calibration graph compute the weights of oil present in the total volumes of both test solution and control.

A-8.4 Calculations

$$\text{Oil content, ppm by weight} = \frac{W_1 - W_2}{W_3}$$

where

W_1 = weight, in μg, of oil in test solution,
W_2 = weight, in μg, of oil in the control, and
W_3 = weight, in g, of sample taken.

INDIAN STANDARDS INSTITUTION

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones : 3 31 01 31, 3 31 13 75
Telegrams : Manaksanstha
(Common to all Offices)

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†Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola, CALCUTTA 700054	36 24 99
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