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IS 5182-10 (1999): Methods for Measurement of Air Pollution, Part 10: Carbon Monoxide [CHD 32: Environmental Protection and Waste Management]



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(पहला पुनरीक्षण)

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 10 CARBON MONOXIDE

(First Revision)

ICS 13.040.20;17.060

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

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Air Environmental Protection Sectional Committee had been approved by the Chemical Division Council.

Devices and techniques for determining the concentration of pollutants in the atmosphere are important for the assessment of ambient air quality, establishing hazardous levels in the environment, determining the effectiveness of ameliorating measures and appraisal of contamination from a process or source. Carbon monoxide is of interest as an air contaminant because of its known toxic properties. It is produced mainly from the incomplete combustion of carbonaceous fuels. The exhaust gases from motor vehicles constitute a large source of carbon monoxide in an urban area. Other sources are the combustion products of fuels used for power, steam and heating purposes.

Carbon monoxide is not known to participate in secondary atmospheric reactions. Its primary effect on human health is dependent on its great affinity for hemoglobin, the oxygen transporting pigment of the blood. Carbon monoxide tends to combine with hemoglobin 210 times as readily as does oxygen, thus effectively preventing its important function of transporting the oxygen from the lungs to the tissues. However, the combination of carbon monoxide with hemoglobin is spontaneously but slowly reversible, and the blood tends to be cleared of carbon monoxide in exponential fashion with a half time of 3 to 4 hours.

Carbon monoxide occurs in unpolluted urban atmospheres in low concentrations, usually in the range of less than 1 part per million of air by volume. The first symptoms of carbon monoxide intoxication are headache, dizziness and lassitude. In most of the persons symptoms are first observed at carbon monoxide concentrations of about 100 mg/l, provided the exposure is sufficiently prolonged. It has been estimated that for each 1 mg/l of carbon monoxide with which the human body is in equilibrium, 0.16 percent of the body's hemoglobin will be inactivated by the formation of carboxy-hemoglobin.

Carbon monoxide is so common a pollutant that exposure to it occurs in many occupations, like garage workers, traffic policemen, railway engine and yard staff. Cigarette smoke also contains a considerable amount of carbon monoxide. Although adaptation to long term low level carbon monoxide exposure occurs, it seems likely that the addition of 5 percent of carboxy-hemoglobin by community air pollution to that caused by these other exposures may be sufficient to pose a substantial risk to the health of sensitive persons. Such a level might be produced by exposure of 30 mg/l, of the carbon monoxide in polluted air for 4 to 6 hours or exposure to 120 mg/l for one hour. A number of methods are available for the analysis of carbon monoxide. Larger proportions of carbon monoxide are usually determined by volumetric gas analysis, carbon monoxide being either oxidized to carbon dioxide with hot copper oxide or absorbed in a reagent containing cuprous salts. Small amounts of carbon monoxide are determined by some colorimetric reaction or by oxidizing to carbon dioxide and determination of either carbon dioxide or one of the other products of the reaction. In air pollution studies the normal range of carbon monoxide concentration would fall between 1 to 100 mg/l. Consequently, methods for the detection of short averages and maximum concentrations of carbon monoxide in the ambient air must be sensitive and accurate in the microchemical range. Other desirable features for a method should include a high degree of specificity, ease of operation and calibration and minimum interference from the other contaminants present in the air sample.

Oxidation with iodine pentoxide and colour reaction with palladous salts and ammonium molybdate are most commonly used methods for the determination of carbon monoxide in polluted air samples. The iodine pentoxide method developed by American Gas Association Laboratory is an accurate and convenient method. Besides, non-dispersive infrared absorption method and gas chromatography method are also gaining importance because of ease of operation, accuracy of test results, etc.

The indicator tube method developed by the National Bureau of Standards, USA, is based upon a colour reaction with palladous salt and ammonium molybdate. Highly purified silica gel impregnated with ammonium molybdate and a solution of palladium or palladium oxide digested in sulphuric acid forming palladium silicomolybdate is exposed to carbon monoxide. A molybdate blue is formed, the depth of colour in the detector tube varying from faint green to blue in proportion to the amount of carbon monoxide present in the air being tested. The indicator tubes being available now commercially, the carbon monoxide can be determined quickly and accurately.

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*Indian Standard***METHODS FOR MEASUREMENT OF AIR POLLUTION****PART 10 CARBON MONOXIDE***(First Revision)***1 SCOPE**

1.1 This standard describes the details of the following five methods for the measurement of carbon monoxide in air:

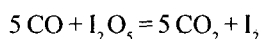
- a) Iodine Pentoxide Method;
- b) Indicator Tube Method;
- c) Non-dispersive Infrared Absorption Method;
- d) Gas Chromatography Method; and
- e) Catalytic Oxidation Method.

1.2 The indicator tube method is recommended for routine use.

The iodine pentoxide method, non-dispersive infrared absorption method, gas chromatography method and catalytic oxidation method are recommended for use as reference methods where laboratory facilities are available.

2 IODINE PENTOXIDE METHOD**2.1 Principle**

The basis of this method is the following reaction between carbon monoxide and iodine pentoxide within 135°C to 150°C to yield carbon dioxide and iodine vapour:



The concentration of carbon monoxide in the air sample may be determined by analysis of the amount of either carbon dioxide or iodine produced. The air sample should be free from any iodine vapours or carbon dioxide. Substances, such as water vapour, unsaturated hydrocarbons, aldehydes and other organic compounds which may react with the iodine pentoxide or the liberated iodine could cause interference. It is, therefore, necessary to pass the air sample through an elaborate purification train consisting of activated carbon, an efficient drying agent, a chromic acid wash tower and some solid absorbent to remove carbon dioxide.

The dry air is then passed through a U-tube containing iodine pentoxide. Free iodine is liberated according to the above reaction if carbon monoxide is present. The iodine liberated in the reaction is absorbed in potassium iodide solution and titrated with standard sodium

thiosulphate solution.

2.2 Apparatus

The apparatus may be easily fabricated (*see* Fig. 1). It consists of a chromic acid scrubber, a drying tube containing phosphorus pentoxide and potassium hydroxide, a U-tube packed with iodine pentoxide maintained at correct temperature (145°C), and bubbler to absorb either carbon dioxide in baryta or iodine in potassium iodide solution or a known volume of standard thiosulphate solution.

2.3 Reagents**2.3.1 Iodine Pentoxide**

Analytical grade iodine pentoxide shall be used. One tube may need about 150 g.

2.3.2 Chromic Acid

Dissolve 70 g of finely ground potassium dichromate in 1 000 ml of concentrated sulphuric acid while heating cautiously under a hood; cool and decant. Permit this solution to stand and then decant the supernatant reagent.

2.3.3 Potassium Iodide Solution

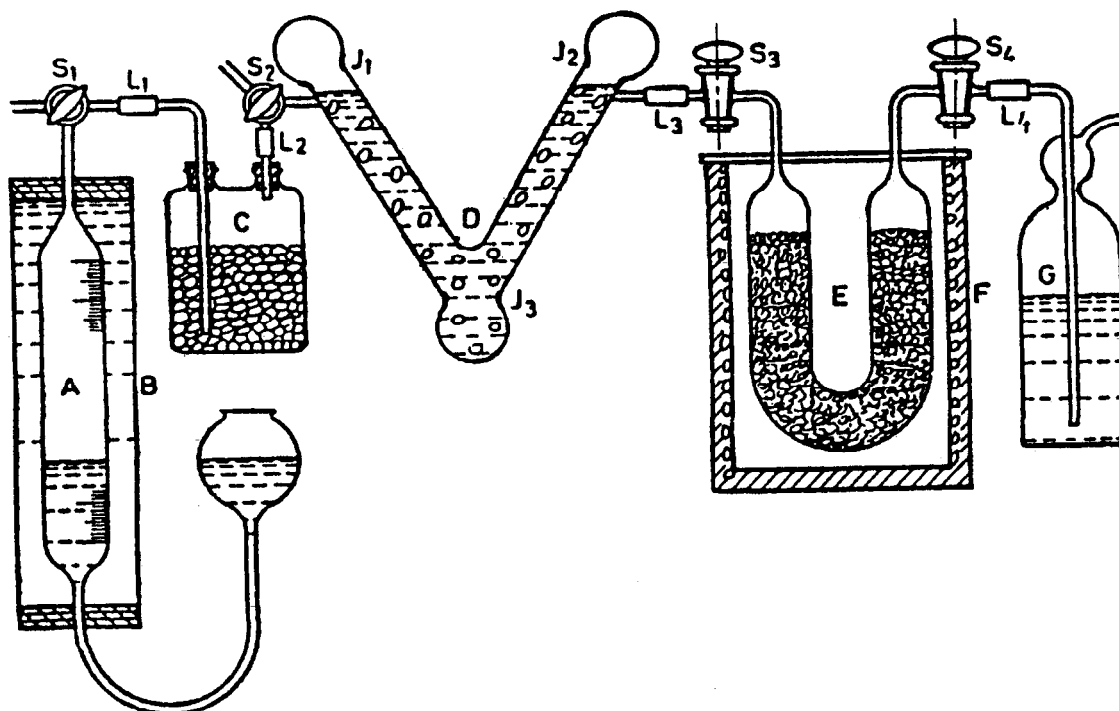
This shall be prepared fresh before an analysis has to be done. Dissolve about 10 g of potassium iodide in 100 ml of water. The reagent shall be stored in a dark coloured bottle. Test the reagent for the presence of free iodine by use of starch and reject if the solution gives a positive test.

2.3.4 Starch Indicator Solution

Fresh solution shall be prepared. Suspend 2 or 3 g of starch in a few millilitres of water and make a paste. Add this to 200 ml of water, heat just to boiling and cool to room temperature.

2.3.5 Sodium Thiosulphate Solution – 0.001 N.

Weigh out 24.8 g of reagent grade sodium thiosulphate and 2 to 3 g of borax crystals. Dissolve this in 1 litre of water. This is an approximately 0.1 N solution. Taking 10 ml of this solution and diluting it to 1 litre would give approximately 0.001 N solution. Standardize either with potassium dichromate solution or with iodine solution at least once a week.



- A - Sampling burette (500 ml)
 B - Water jacket
 C - Chromic acid wash tower containing chromic acid and glass beads
 D - Drying tube: The Y - tube contains alternate layers of phosphorus pentoxide and glass wool with a layer of potassium hydroxide pellets at the inlet side of G . It is sealed with paraffin wax
 E - Iodine pentoxide tube having alternate layers (about 15 mm) of iodine pentoxide and glass wool
 F - Furnace
 G - Potassium iodine trap
 Stopcocks - S_1 , S_2 , S_3 and S_4
 Rubber tube joints - L_1 , L_2 and L_4
 Standard joints - J_1 , J_2 and J_3

FIG. 1 APPARATUS FOR DETERMINATION OF CARBON MONOXIDE

2.3.5.1 Sodium thiosulphate solution is not stable, and is affected by oxygen, carbon dioxide and micro-organisms. For this reason it is essential to use boiled water in its preparation and to include a preservative like borax.

2.3.5.2 Standardization of thiosulphate solution

a) With potassium dichromate

Dissolve 0.5 g of potassium iodide and 1 g of sodium bicarbonate in 30 ml of water in 125 ml glass stoppered long necked flask. Then add concentrated hydrochloric acid slowly, swirling the flask, until no more carbon dioxide is generated and add about 1 ml more of acid. Now add 20 ml of 0.001 N potassium dichromate solution. Swirl the mixture very gently. Wash the sides of the flask with a few millilitres of water and allow this rinse to

form a layer above the acid dichromate solution without mixing. Stopper the flask and allow to stand for about 10 minutes in dark. Mix thoroughly, remove the stopper and run in the 0.001 N sodium thiosulphate solution to be standardized from a volumetric burette until the solution becomes light yellow. Add a few drops of starch indicator solution and continue the titration until the bright blue colour has disappeared and only the pale green colour of the chromic chloride remains. Add the last few drops very cautiously. Calculate the normality of the sodium thiosulphate solution.

b) With Iodine

Place 10 ml of standard 0.001 N iodine solution in a 125-ml long necked flask. Titrate with 0.001 N sodium thiosulphate solution to be standardized until yellow colour of the iodine solution is very

faint. Add a few drops of starch indicator solution, mix by swirling, and continue the titration adding the sodium thiosulphate solution drop by drop until the blue colour is discharged. Calculate the normality of the sodium thiosulphate solution.

2.4 Procedure

2.4.1 The iodine pentoxide tube shall be thoroughly purged from free iodine. This is accomplished by heating the tube to 200°C and passing nitrogen gas through stopcock S_3 till no colour change is observed on adding starch indicator to the potassium iodide solution in the trap. This may take as long as 5 days. Stopcocks S_2 and S_4 should then be greased and kept closed to avoid exposing the iodine pentoxide tube to moisture from the air.

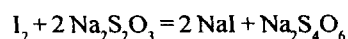
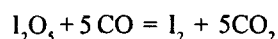
2.4.2 A blank determination shall be made before any analysis is to be performed. Fresh 10 percent potassium iodide solution is taken in the trap. Nitrogen is passed through stopcock S_2 for 30 minutes, keeping stopcocks S_3 and S_4 open. Stopcocks S_3 and S_4 are now closed and the solution in the potassium iodide trap titrated with standard sodium thiosulphate solution. The amount of free iodine thus obtained is considered the blank of the apparatus and should be subtracted from subsequent determinations.

2.4.3 Known volume of the air sample is taken in the sample burette. A through stopcock S_1 and allowed to stand until it attains the temperature of the water in the jacket B . Potassium iodide trap is filled with potassium iodide solution and attached to the apparatus. Stopcocks S_1 , S_2 , S_3 and S_4 are opened so that the gas burette is connected to the iodine pentoxide tube. The gas is then slowly pushed by raising the water level in the gas burette A . This takes approximately 7 to 10 minutes. When all the gas has been passed, stopcocks S_1 and S_2 are closed. The apparatus is again purged with nitrogen for about 30 minutes. Stopcocks S_3 and S_4 are closed. The potassium iodide trap is disconnected and its contents titrated for free iodine.

2.4.4 In case the concentration of carbon monoxide in the test sample exceeds 0.1 percent, only a small portion of the sample should be passed through the apparatus at a time in order to avoid the release of excessive amounts of free iodine, which may deposit in the form of crystals and cause choking. As the carbon monoxide concentration in atmospheric pollution normally falls much below this level, it is usual practice to pass the entire volume of the gas sample through the apparatus.

2.5 Calculation

2.5.1 If carbon monoxide is present in the sample being analysed, then iodine is evolved and titrated in accordance with the following equations:



Expressed numerically on mass basis:

$$140 \text{ g CO} = 253.8 \text{ g I}_2 = 496.4 \text{ g Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$$

Since a normal solution of iodine contains 0.127 g of iodine per millilitre, then according to the above equations the equivalent mass of sodium thiosulphate and carbon monoxide is 0.248 g and 0.07 g respectively.

Now volume of 0.07 g of carbon monoxide at standard temperature and pressure (STP) (0°C and 760 mm Hg) is given by:

$$\frac{22400 \times 0.07}{28} = 56 \text{ ml}$$

Hence, 1 ml of normal sodium thiosulphate solution, containing 0.248 g is equivalent to 0.07 g or 56 ml of carbon monoxide.

Therefore, volume of carbon monoxide in the sample analyzed is given by:

56 X Volume of thiosulphate in ml X normality of thiosulphate (where the volume of sodium thiosulphate used in ml is the volume to titrate the iodine liberated in the analysis of the sample).

$$\text{Carbon monoxide, } \mu\text{g/m}^3 = \frac{0.07 \times \text{Volume of thiosulphate} \times \text{Normality of thiosulphate} \times 10^9}{\text{Volume of gas sampled (litre)}}$$

3 INDICATOR TUBE METHOD

3.1 Principle

Carbon monoxide reduces yellow silicomolybdate to lower oxides. The colour changes from yellowish green to green and finally deep blue depending on the extent of reduction which again under identical conditions depends on concentration of carbon monoxide in air.

3.2 Interferences

Hydrogen sulphide, unsaturated hydrocarbons, moisture, etc, interfere but they may be removed by passing the sample through various absorbents provided with the indicator tubes.

3.3 Procedure

Follow the manufacturer's instructions carefully. Draw 250 ml of the sample at the prescribed rate (40 to 50 ml/min) through the tube by the aspirator provided. Compare the colour produced with the standard colours provided and calculate the concentrations.

4 NON-DISPERSIVE INFRARED ABSORPTION METHOD

4.1 Principle

Samples containing carbon monoxide in the range of 0 to 100 mg/l are analyzed on a non-dispersive infrared absorption gas analyser, namely, an electro-optical spectrophotometer with no spectral dispersion component. It may consist of a single or double source of infrared energy and one or more infrared detectors separated by an optical cell or cells through one or more of which the sample flows, whereby the specific spectral absorption of the component of interest is determined.

NOTES

1 Specific spectral sensitivity is obtained by either a selective source or a selective detector and may be improved further by filters in the light path.

2 The analyser shall be constructed to or be adjusted to select only the spectral bands at which carbon monoxide has its characteristic absorption, and the sample cell length shall be appropriate for the rated range of concentration.

3 Though the basic instrument recommended is for (0 to 100) mg/l range, for samples of higher mg/l range the instrument may be used with proper dilution and conditioning of the samples. The dilution shall meet all other conditions given in 4.3.

4.2 Apparatus

4.2.1 Infrared Gas Analyser

Any non-dispersive type, provided it meets the following requirements:

- a) The apparatus shall be constructed so as to be suitable for operating within the temperature range of 15°C to 40°C and in a relative humidity range of 0 to 90 percent. The apparatus shall embody facilities for the analysis of continuously applied sample or a discrete sample of volume of 2.0 litres. In the later case the purification train shall be of such a design and dead volume that 2.0 litres is adequate for its proper flushing out. The apparatus shall include facilities for the visual inspection of results and also for their recording. The scale shall be divided into steps of 1.0 percent full scale division (fsd).
- b) The 95-percent confidence limits of the repeatability shall be within ± 1.0 percent full scale division (fsd). The minimum detectable concentration shall be 1 mg/l or less;
- c) The cross sensitivities (or interference) of the analyser to carbon dioxide and to water vapour shall each be less than 1 percent full scale division (fsd);
- d) The maximum deviation between an actual instrument reading and the reading predicted

by a straight line between the upper and lower calibration points shall be 1 percent full scale division (fsd);

- e) Neither the zero drift nor the full scale drift shall be greater than ± 1 percent fsd in 24 hours, excluding change due to changes of atmospheric pressure;
- f) The response time of the analyser excluding the sample conditioning train shall be less than 40 sec to 90 percent of the final reading at a flow rate of 250 ml/min; and
- g) The apparatus shall be capable of meeting the requirements in 4.2.1(a) to 4.2.1(f) despite a variation of ± 10 percent in the nominal applied mains voltage. The requirement may be met by incorporating a voltage stabilizer either within the instrument or externally.

4.2.2 Pump

4.2.3 Flowmeter

4.2.4 Connecting Tubes

Stainless steel, teflon, polyvinyl chloride or polyethylene.

4.2.5 Arrangement of Apparatus

The analysing system consists of two major parts, namely, the sample conditioning train and the infrared gas analyser itself.

A pump is necessary to move the sample through the conditioning train and through the analyser. The pump may be sited before or after the analyser unit:

- a) *Pump after the analyser (suction system)* — If the pump is installed after the analyser, false readings and damage to the instrument by accidental overpressurizing are completely obviated. However, leak at any point may give rise to an error;
- b) *Pump before the analyser (pressure system)* — Minor leaks in the system do not invalidate the results of the analysis. The pump shall be of a type that will not contaminate the samples. Neoprene diaphragm and stainless steel below pumps have been found suitable.

In both cases it may be necessary to install a reducing valve between the pump and the analyser in order to prevent pressure pulses by the pump influencing the analyser.

A flowmeter shall also be incorporated in the sample flow line, preferably before the analyser.

Connecting tubing will be required between the filter, pump, instrument, etc, as well as for the construction

of the sampling probe. Stainless steel, teflon, PVC and polyethylene have been found suitable for this purpose.

It is to be noted that the length of connecting tube may have to be kept to a minimum in order that the apparatus meets the performance requirements of 4.2.1

4.3 Sample Conditioning

4.3.1 Particulate Matter

Fine particulate matter causes scattering of the infrared beam, producing a positive interference and although small amounts may not affect the immediate reading they are objectionable because dust may accumulate on the optics of the instrument leading to reduced sensitivity and permanent impairment. To obviate these difficulties a filter capable of removing 99 percent of all particulate matter above $1.0\ \mu\text{m}$ is essential and these should be installed immediately prior to the analysis cell. The filters may be warmed to prevent condensation occurring within them. Filters with elements made of fibre glass or sintered metal are suitable. These filters should be cleaned or changed periodically in order to avoid building up pressure across the filtering membrane and thus affecting the flow of gas through the analysis cell.

To minimize the frequency of changing the filter it is normally advisable to incorporate a preliminary coarse filter for which a porosity of $10\ \mu\text{m}$ is suitable and this may be placed at the entrance to the sample conditioning train.

4.3.2 Water Vapour

Most ambient atmospheres contain sufficient water vapour to produce interference and the diurnal pattern of atmospheric humidity change may lead to variations in the degree of interference from hour to hour and day to day.

There are three methods for eliminating the interference by water vapour:

a) Humidification

Saturation of the sample to constant humidity is applicable in all cases but for adequate repeatability the temperature of the saturator shall be kept within $\pm 0.1^\circ\text{C}$.

NOTE—There may be long term deterioration of the optics of some instruments when continuously exposed to saturated sample streams.

b) Refrigeration

Refrigeration of the sample steam is always applicable but attention is drawn to the increase in dead volume of the apparatus which the refrigerated tubes entail. Conformity to the response time shall be confirmed experimentally.

A final column of self-indicating desiccant shall be put at the end of the purifying train to give warning of a failure of the refrigerator.

c) Desiccants

The use of drying agents is always permissible, but to achieve adequate desiccation capacity between servicing intervals low rates of flow and large containers have to be used. These do not only make substantial additions to the response time but also allow a degree of mixing and hence loss in sharpness in response to steep changes of concentration.

Desiccants tend to swell as they absorb water giving rise to increasing obstruction to the sample flow.

Desiccant towers and saturation vessels are typically constructed of glass and suitable desiccants include self-indicating magnesium perchlorate.

4.3.3 Carbon Dioxide

Optical filters are adequate for eliminating interference due to carbon dioxide up to 2 percent.

Samples that may contain more than this will require purification, for example, by the use of soda lime. In this case, the difficulties that arise from the use of drying towers for the removal of water vapour also occur and the precautions recommended in that case also taken.

4.3.4 Response Time

The response time of the analyser and the conditioning train together shall be 2 minutes to reach 90 percent of the final reading.

4.3.5 Changes due to Conditioning

For accurate analysis, it may be important to calculate the change in concentration brought about by the process of purifying the sample.

4.4 Mode of Operation

4.4.1 Continuous Analysis

Infrared analysers are normally used for continuous analysis and for this purpose flow rate between 100 ml per minute to 1 000 ml per minute are suitable.

4.4.2 Discrete Samples

When used to analyse discrete samples the whole system shall be thoroughly flushed with the sample before each analysis and the whole system shall therefore be of such a volume and design that 1 000 ml is adequate for this purpose.

4.5 Procedure

4.5.1 Warm-Up Period

Infrared analysers are normally operated on a continuous basis; however, an initial warm-up period is required and no readings shall be taken until the apparatus has reached steady temperature conditions and is giving a steady response. This shall not be more than 6 h.

When used to analyse discrete samples, similar precautions to ensure that the apparatus has reached temperature stability shall also be taken.

4.5.2 Calculation of Results

Infrared analysers are calibrated variously in arbitrary units, in microvolts or in units of concentration and no one method of calculating results can be given. Infrared analysers which are not calibrated in units of concentration shall be accompanied with a precise statement of how a scale reading is to be converted to concentration of carbon monoxide.

4.6 Standardization

4.6.1 Preparation of Standard Gases

Infrared methods of gas analysis depend on calibration against gas mixtures of known composition. Method of preparing reference mixtures of standard gases shall be in accordance with the relevant Indian Standard. In the absence of these, they shall be prepared as agreed to between parties concerned.

4.6.2 Calibration Gases

Two standard gas mixtures are required for calibration purposes. One shall contain between 90 to 100 mg/l of carbon monoxide in air or nitrogen (span gas) and the other air or nitrogen with not more than 1 mg/l carbon monoxide (zero gas).

4.6.3 Analysis of Standard Gases

The span and zero gases shall be supplied to the instrument for calibration at the same temperature, humidity and pressure as will obtain when samples are to be analysed. Calibration mixtures shall be passed through the sample conditioning train.

5 GASCHROMATOGRAPHY METHOD

5.1 Principle

A sample of the air containing carbon monoxide is injected into the gas chromatograph where it is carried from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated. The separated constituents emerge from the end of the column one after the other and are

detected by suitable means whose response is related to the amount of a specific component leaving the column.

NOTE—The lower detection limit of this method is 10 mg/l and sample size is 10 ml.

5.2 Apparatus

5.2.1 Any gas chromatograph capable of being operated under conditions given below for resolving the sample into distinct peaks may be used. A typical gas chromatogram for carbon monoxide using such a chromatograph is given in Fig. 2.

5.2.2 Column

- Stainless steel, with activated charcoal of 30 to 35 mesh; 100 cm long and 4 to 6 mm in diameter. The column is activated at 150°C for 3 h; and
- Stainless steel, with molecular sieve 5 A (40 to 60) mesh; 110 cm long, 4 to 6 mm in diameter, activated at 300°C for 3 hours.

These two columns are in series, with the charcoal column on the injection side. The columns shall be activated at regular intervals as the retention time of carbon monoxide will decrease. Methane present in large concentrations will interfere.

5.2.3 Conditions

Temperature	50°C
Carrier gas	Hydrogen
Carrier gas flow	5 litres/hour
Sample loop	10 ml
Bridge current	350 mA
Chart speed	30 cm/hour

5.3 Procedure

Conduct the flow of carrier gas and inject the sample at injection port where it is well mixed with carrier gas. This is then led into the chromatographic column. The constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For efficient separation it is necessary that the column is maintained at the temperature prescribed in 5.2.3. As the sample enters the detector, it gives a signal corresponding to the amount of a particular constituent, carbon monoxide in this case, leaving the column. From the specific area under the peak corresponding to carbon monoxide, the quantity of carbon monoxide may be determined.

5.4 Calculation

Calculate the individual concentrations of the constituents on the basis of the peak areas on the chromatograph obtained with known amount of constituent, namely, carbon monoxide, using the same apparatus under identical conditions.

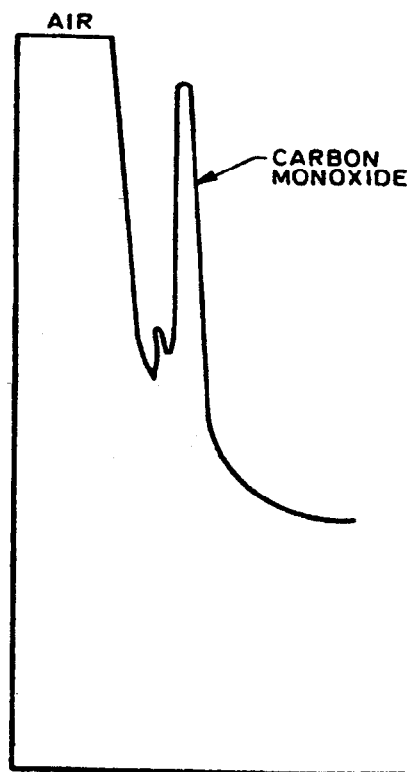


FIG. 2 TYPICAL GAS CHROMATOGRAM FOR CARBON MONOXIDE IN AIR SAMPLE

6 CATALYTIC OXIDATION METHOD

6.1 Principle

Air containing carbon monoxide is passed over a heated catalyst commercially known as Hopcalite and reacts to form carbon dioxide. The resultant rise in temperature due to exothermic nature of the reaction is a measure of carbon monoxide concentration. Glass bead thermistors embedded in hopcalite measure the temperature rise. Samples containing 0 mg/l to 500 mg/l carbon monoxide are measured by this method.

NOTES

1 The minimum detection limit for this method is ± 2 mg/l.

2 Though the basic instrument is recommended for 0 to 500 mg/l range, samples with higher concentration of carbon monoxide may be measured by proper dilution.

6.2 Apparatus

6.2.1 Gas Monitor

It shall meet the following specifications:

- Monitor should be suitable for operation within the temperature range of 15°C to 40°C and a relative humidity of 0 to 99 percent;
- The 95 percent confidence limits of repeatability shall be within ± 1.0 percent fsd. The minimum

detectable concentration shall be 2 mg/l or less;

- The maximum deviation between an actual instrument reading and the reading predicted by a straight line between the upper and lower calibration points shall be ± 1 percent fsd;
- Neither the zero drift nor the full scale drift shall be greater than ± 1 percent fsd in 24 hours, excluding changes due to changes of atmospheric pressure; and
- The response time of the monitor shall be less than 5 minutes for 90 percent of the final reading at a flow rate of 2 litres/min.

6.2.2 Pump

Any pump having a flow rate of 10 litres per minute is suitable.

6.2.3 Connecting Tubes

Tubes of teflon, polyvinyl chloride or polyethylene.

6.2.4 Arrangement of Apparatus

The analysing system may be considered as consisting of CO monitor and a pump for drawing the air sample continuously through the monitor. A needle valve and a flow meter fitted in the monitor control and measure the flow of sample air connecting tubes will be required between the sampling point, the instrument and the

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pump. Teflon or polyvinyl chloride or polyethylene tubes have been found suitable for this purpose.

6.3 Sample Conditioning

6.3.1 Particulate Matter

Coarse particles present in the gas stream can clog the flow meter and needle valve over a long period of time. To overcome these difficulties a filter capable of removing over 90 percent of 1 μm size particles is fitted at the inlet. This filter should be checked at weekly intervals when the instrument is continuously used and changed if it is clogged or begins to affect the flow in the instrument.

6.3.2 Water Vapour

Constituents of the air sample will not interfere if the instrument is zeroed with carbon monoxide free air whose composition is same as the composition of the sample to be analysed.

6.4 Mode of Operation

The instruments based on catalytic oxidation method are used for continuous monitoring of the sample gas. For this purpose a flow of 2 litres per minute or as specified by manufacturer, is maintained through the instrument.

6.5 Procedure

6.5.1 Warm-Up Period

Catalytic oxidation method instruments are operated on a continuous basis. However, an initial warm-up

period of 90 to 100 minutes is required. Air flow is maintained during this period and no measurements are made.

6.5.2 Calculation of Results

These instruments are direct reading instruments and carbon monoxide concentrations may be directly read on the meter.

6.6 Standardization

6.6.1 Preparation of Standard Gases

Catalytic oxidation method of gas analysis depends on calibration against gas mixtures of known composition. Method of preparing reference mixtures of standard gases shall be in accordance with the relevant Indian Standard. In the absence of these they shall be prepared as agreed to between parties concerned.

6.6.2 Calibration Gases

Two standard gas mixtures are required for calibration purposes. One shall contain between 90 to 100 mg/l of carbon monoxide in air (span gas) and the other air with not more than 1 mg/l carbon monoxide (zero gas).

6.6.3 Analysis of Standard Gases

The span and zero gases shall be supplied to the instrument for calibration at the same temperature, humidity and pressure as will obtain when samples are to be analysed.

(Continued from second cover)

In this revised version, an additional method, namely, catalytic oxidation method which is suitable for continuous monitoring, has been incorporated to be used as reference method.

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'.

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