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PUBLIC REVIEW DRAFT DKS 2440:2012

This Draft Kenya Standard has been prepared by the **Gases** Technical Committee in accordance with the procedures of the Bureau, and is now being circulated for public comments.

The Committee would appreciate any comments on this Draft Standard, which should be submitted before **2013-02-10** using the attached template. It will also be appreciated if those who have no specific comments to make but find the draft standard generally acceptable can notify us accordingly. **Absence of any reply or comments shall be deemed to be an acceptance of the technical contents of the draft Kenya standard and shall constitute an approval vote.**

Suggestions entailing amendments of the text should include wording preferred and the relevant clause number quoted against any comments made.

This draft standard is subject to change and should not be referred to or used as a Kenya Standard. All correspondence pertaining to this draft standard should be addressed to the Managing Director, Kenya Bureau of Standards for the attention of Mr. Samson Ombok.

Yours faithfully,

Samson Ombok

For: DIRECTOR

STANDARDS DEVELOPMENT AND INTERNATIONAL TRADE

High Purity Oxygen — Specification

PUBLIC REVIEW DRAFT, DECEMBER 2012

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Noble Gases International Ltd
Chemigas Ltd
Synergy Gases Ltd
Carbacid (CO₂) Ltd
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Kenya Science Teachers College
Ministry of Roads and Public Works – Materials Branch
Kenya Industrial Estates
Kenya Medical Association
Kenyatta National Hospital
Ministry of Medical Services
Kenya Bureau of Standards — Secretariat

REVISION OF KENYA STANDARDS

In order to keep abreast of progress in industry, Kenya Standards shall be regularly reviewed. Suggestions for improvements to published standards, addressed to the Managing Director, Kenya Bureau of Standards, are welcome.

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KENYA STANDARD

High Purity Oxygen — Specification

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Forward

This standard has been prepared by the Technical Committee on Gases under the guidance of the Standards Projects Committee, and it is in accordance with the procedures of the Kenya Bureau of Standards.

High purity oxygen covered in this standard is intended for use in general laboratory application in analytical instruments. Oxygen is used in analytical instruments such as AAS. Furnace atmospheres require low moisture levels to maintain instrument sensitivity and impurities limits. This standard covers characteristics touching on their purity, packaging and marking.

The standard also specifies limits impurities on water, nitrogen and total hydrocarbons (THC) as methane.

Any enrichment of the atmosphere by oxygen enhances rates of combustion. The effects are greatly increased atmospheres containing more than 25 per cent of oxygen and great care should be taken using oxygen in a confined space to prevent increase in concentration. It is essential that oil be excluded from any cylinder, container, valve or equipment coming into contact with clothing. The analytical procedures given in the annexes are designed by for the determination of trace impurities. Special care and attention is needed in carrying them out.

During the development of this standard, reference was made to the following documents:

BOC SGEM_2006 Section 5_Pure gases specifications, High purity grade Oxygen

Purity plus speciality Gases and Equipment catalogue, Purity plus Zero Oxygen

EN 1089-3:2004 Transportable gas cylinders—Cylinder identification, Part 3: Colour coding

KS 647: 2010 Specification for industrial oxygen

Acknowledgement is hereby made for the assistance received from these sources.

High Purity Oxygen —Specification

1. Scope

This Kenya Standard specifies requirements and test methods for high purity oxygen. The requirements apply to oxygen as delivered. It does not apply to the gas intended for medicinal use or inhalational purposes.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this Kenya Standard. For undated reference, the latest edition of the normative document referred to applies.

KS 648, Industrial Acetylene gas— Specification

KS ISO 7225, Gas cylinders — Precautionary labels

KS 09-532, Specification for standard atmospheric conditions for test purposes

3 Terms and Definitions

For the purposes of this Standard the following terms and definitions shall apply.

3.1 STP

standard atmospheric temperature and pressure as per KS 09-532

4. Requirements

4.1 Description

The gas shall be colourless, odourless at ordinary temperatures and pressures and shall consist essentially of Oxygen O₂, which has a density of 1.3546 kg/m³ at a temperature of 23 °C and 760 mm Hg pressure. It shall be supplied as compressed gas or as a liquid for vaporization.

4.2 Purity

When tested in accordance with the method specified in annex B the product shall have a minimum of 99.9 % v/v Oxygen content, calculated on a dry basis.

4.3 Impurities

The product shall comply with the impurities limits given table 1.

Table 1 - Impurity limits

SL NO.	Characteristic	Requirement	Test method
1.	Nitrogen ppm , max	500	Annex C
2.	Total hydrocarbons as Methane, ppm, Max	0.5	
3.	Water ppm , max	25	Annex D

5. Sampling

5.1 Samples of compressed oxygen shall be taken from a steel cylinder containing the gas direct to appropriate apparatus for all determinations. The valve and connecting lines shall be carefully purged before taking a sample.

5.2 Samples of liquid oxygen shall be taken in accordance with the method described in Annex A or by any alternative method that can be shown to ensure complete vaporization of the sample.

6 Packing and marking

6.1 Packaging

The product shall be supplied as a compressed gas or liquid at cryogenic temperature, in appropriate steel cylinders/containers complying with relevant Kenya standards. Valves or taps shall not be lubricated with oil or grease.

6.2 Marking

6.2.1 Cylinder

Each cylinder/container shall have labels which are clearly and indelibly marked with the following information:

- a) The words " High Purity Oxygen";
- b) The name or registered trade mark and address of the manufacturer;
- c) Purity;
- d) The impurities limits in table 1, as applicable;
- e) Batch number;
- f) Date of filling;
- g) Net Weight of contents in Kg for product in liquid form
- h) Filling pressure (at STP) for product in gaseous form;
- i) Cautionary note — see Annex E

6.2.2 Colour and chemical formula

Each cylinder shall in addition to the markings in 6.2.1, be painted with the colour black and clearly and indelibly marked with the chemical formula corresponding to oxygen.

6.2.3 Precautionary labels

Each cylinder shall in addition to the markings in 6.2.1, be clearly and indelibly marked with precautionary labels as specified in KS ISO 7225.

PUBLIC REVIEW DRAFT, DECEMBER 2012

Annex A

Treatment Of Liquid Samples

A1. OUTLINE OF METHOD

The liquid is forced under pressure into an evaporating coil. By use of small bore tubing complete evaporating of the liquid is ensured, and hence the gas produced has the same composition as the liquid.

CAUTION: Liquid oxygen requires careful handling and all flames should be extinguished in the vicinity during sampling operations, and care taken to avoid spilling it on to clothing. Liquid oxygen boils at -183°C so precautions should be taken to prevent it coming into contact with the skin and eyes.

A2. APPARATUS

The apparatus is shown in Figure 1 and comprises:

- (a) *Mercury lute* — Arranged so that the head of mercury can easily be adjusted.
- (b) *Dewar flask* — Of copper designed to withstand an internal pressure of 68.9 kPa and with a capacity of about 2½ litres, fitted with a screw-on brass cap carrying a bursting disk set at 34.5 kPa and two connections of 1.5 mm internal diameter drawn copper tubing.
- (c) *Evaporator* — Consisting of a 500-mm length of 1.5 mm internal diameter drawn copper tubing formed into a spiral and immersed in water at 50°C to 60°C .

A3. PROCEDURE

Cool the dewar flask by introducing a small quantity of the liquid oxygen and after a few minutes empty the flask and discard the liquid.

Immediately introduce more liquid oxygen until the flask is about two-thirds full. With stopcock T_1 open, screw on the brass cap and make the connections as shown in Figure 1. Control the rate of gas flow to the analysis apparatus by adjusting the control valve and the head of mercury in the lute.

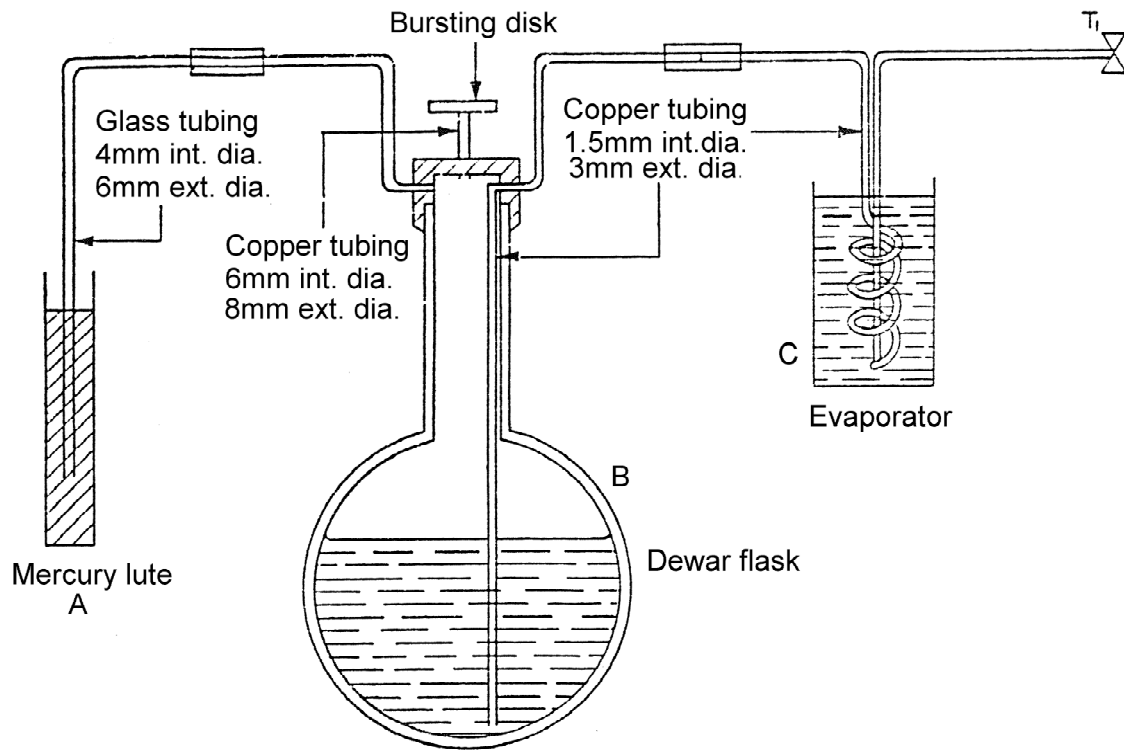


FIG. 1 — Apparatus For Treatment Of Liquid Samples

Annex B

Determination Of Oxygen Content

B1. OUTLINE OF METHOD

The oxygen content is determined by measuring the volume of sample before and after absorption of the oxygen by copper in the presence of ammoniacal ammonium chloride solution.

B2 APPARATUS

This apparatus is shown in Figure 2 and comprises of:

- (a) Levelling bottle.
- (b) Graduated burette and stopcock, as shown in Figure 3, fitted with a two-way burette stopcock (T_1).

NOTE: In graduating the burette the lowest point of the liquid meniscus should be adjusted to the top edge of the graduation mark. The meniscus can be clearly defined by folding a strip of black paper round the vessel, the top edge of the paper being horizontal and not more than 1 mm below the mark on which the setting is to be made. The meniscus, so shaded, is viewed against a white background. When reading the burette sufficient time should be allowed for all the liquid to drain from the walls.

The same technique should be used in making all readings of the burette.

- (c) Container for copper wire, as shown in Figure 4.
- (d) Reservoir.
- (e) Gas inlet, with three-way stopcock (T_1).
- (f) Flute, sealed with a head of about 50 mm of water.
- (g) Glass tube, 6 mm, outside diameter and 260 mm long.

B3.1 Ammoniacal Ammonium Chloride Solution — Dissolve 550 g of ammonium chloride in 1 250 mL of water and add 750 mL of concentrated ammonia solution, $d = 0.88$.

Freshly prepared solution in contact with the copper spirals is initially very pale in colour, and the absorption of oxygen in the determination may be incomplete, leading to errors in test results. Before it is used for a determination it should be aged to a deep blue colour by carrying out several dummy operations. The solution loses its efficiency with use and the apparatus may be recharged by the method given in Note 1 at the end of this appendix.

B3.2 Copper Wire Spirals — 200 mm lengths of copper wire, diameter 0.9 mm, wound into open spirals of about 6 mm diameter and about 20 mm long.

B4. PROCEDURE

B4.1 Preparation of the Apparatus — Fill container C with the copper spirals and connect to the rest of the apparatus as shown in Figure 2. Lubricate the stopcocks with the minimum amount of a suitable vacuum stopcock grease (silicone greases are unsuitable).

Introduce the ammoniacal ammonium chloride solution by the following series of operations:

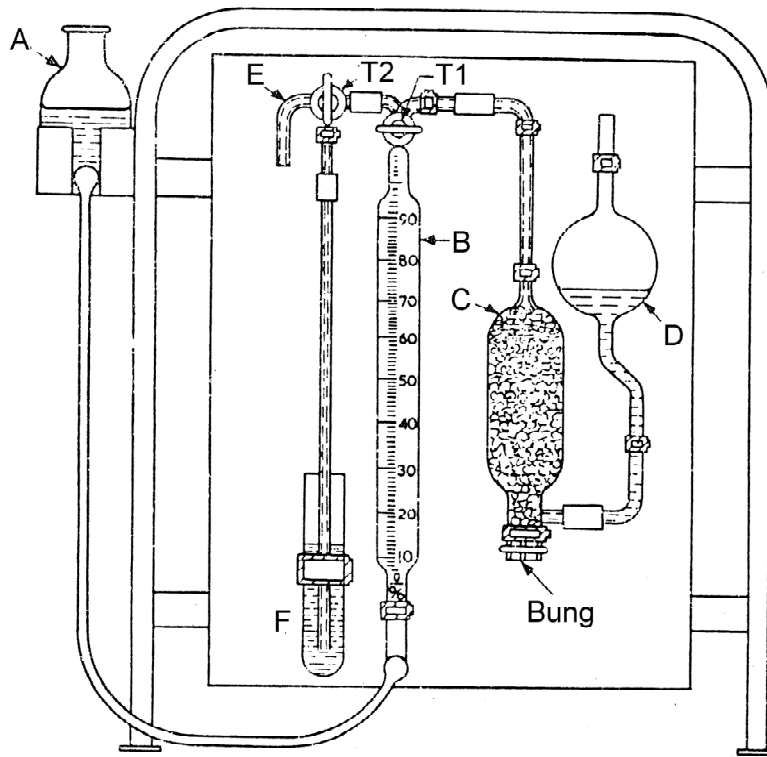


FIG. 2 — Apparatus Forthe Determination Of Oxygen Content

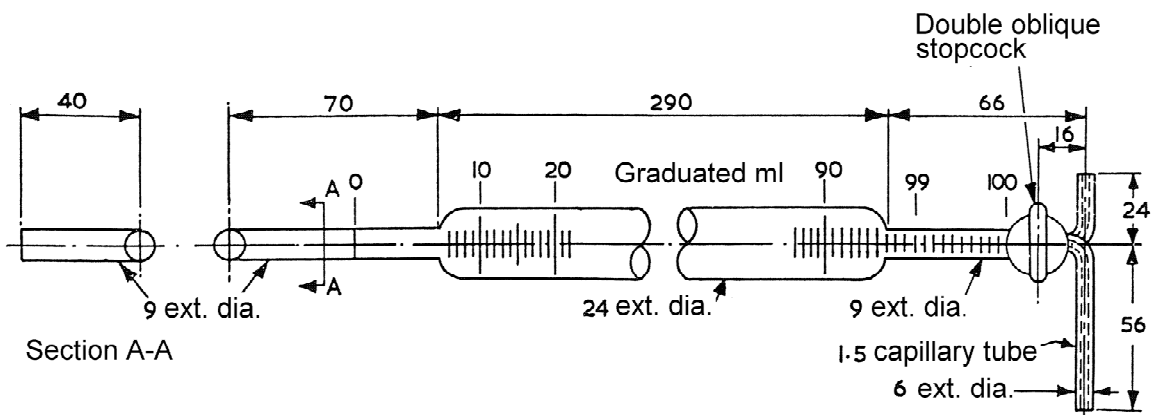


FIG. 3 — Graduated Burette For The Determination Of Oxygen Content

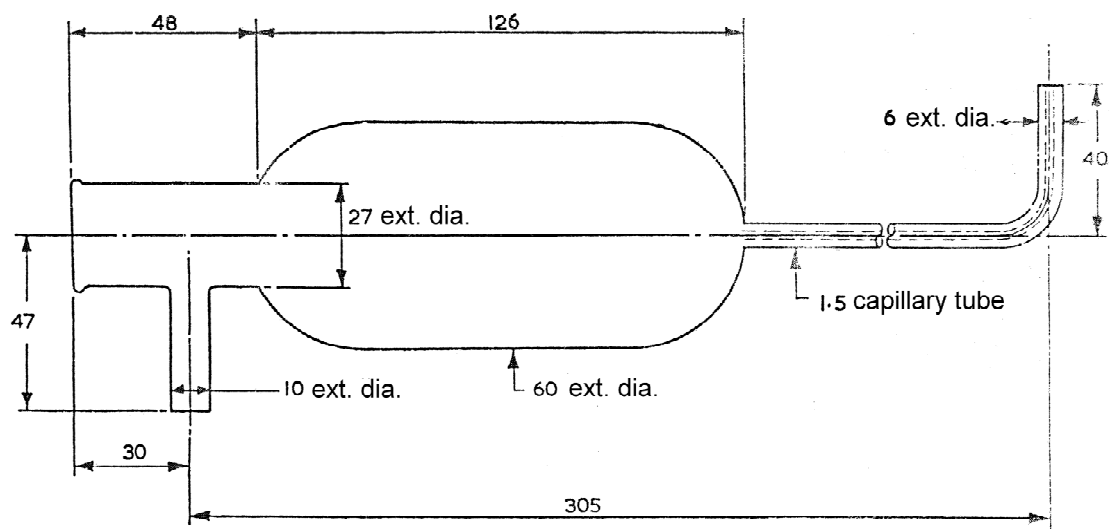


FIG. 4 — Copper Wire Container For The Determination Of Oxygen

- B4.1.1** Lower the reservoir A to the bench and disconnect the tube and invert the rest of the apparatus with stopcock T_1 closed.
- B4.1.2** Fill container C with solution and close it with a rubber bung.
- B4.1.3** Return the apparatus to the upright position.
- B4.1.4** Fill levelling bottle with the solution.
- B4.1.5** Operate stopcocks T_1 and T_2 so burette B is open to the atmosphere, and by raising A completely fill burette B with the solution.
- B4.1.6** Operate stopcock T_1 so that B is in communication with C, and keeping A raised, allow the solution to pass through C until reservoir D is about one-third full. Close stopcocks T_1 .
- A4.1.7** With A still raised, fill the capillary between stopcocks T_1 and T_2 with the solution. Close stop T_2 and return A to its resting position. Add more solution if necessary, until A is half full.
- B4.2 Determination** — Carry out the following sequence of operations:
- B4.2.1** Connect the sample gas supply, either from a cylinder or from the liquid evaporator to E and with stopcock T_2 closed, purge the tubing by allowing the gas to escape through the water tube F.
- B4.2.2** With the gas still bubbling through F, operate stopcock T_1 to connect B to E and lower the levelling bottle A to maintain the same liquid level in A and B until the level in B reaches the zero mark.
- B4.2.3** Raise A and expel all the gas from B through the tube. Lower A to refill B, as in **B4.2.2**. Close stopcock T_1 and rock the apparatus gently until absorption appears to be complete.

- B4.2.4** Operate stopcock T_1 to connect B to C, and by raising A pass the sample from B to C. Close stopcock T_1 and rock the apparatus until absorption appears to be complete.
- B4.2.5** Lower and operate stopcock T_1 to connect C to B so as to pass any unabsorbed gas back into B, taking care that the connection capillary is filled with solution. Close stopcock T_1 .
- B4.2.6** Raise A until liquid levels in A and B are the same and read the volume of residual gas. (See note on reading meniscus given in **B2**).
- B4.2.7** Repeat operation of **B4.2.4**, **B4.2.5** and **B4.2.6** until a constant reading is obtained.

NOTE:1. When the absorption of oxygen becomes slow or deposits form in the ammoniacal ammonium chloride solution replace approximately half the solution in the apparatus by the following sequence of operations:

- (a) Operate stopcock T_1 so that B is connected to C and raise A until B is full of liquid.
 - (b) Close stopcock T_1 , remove the bung from C and allow the solution in C and D to run to waste.
 - (c) Replace the bung in C.
 - (d) Operate stopcock T_1 , to connect B to C and by raising A pass the solution from B to C until the latter is half full.
 - (e) Add fresh solution to A, and lower A to draw gas from C into B, taking care not to draw in air through D. Operate stopcocks T_1 and T_2 to connect B to atmosphere and expel the air from burette B by raising A.
 - (f) Repeat operations (d) and (e) until all levels are correct.
- 2 It is essential to keep C completely full of copper spirals as any fall in the level of the copper will slow down the rate of absorption of oxygen. To recharge with additional spirals close the top of D with a bung, lower A to the bench, and disconnect the tube, invert the apparatus and remove the bung from C. Add more spirals until C is full and then reassemble the apparatus.

Annex C

(Normative)

Determination of Nitrogen and total hydrocarbons (as methane)

C.1 Principle

This method covers the determination of Nitrogen and total hydrocarbons (as methane) content of oxygen by means of gas chromatography.

C.2 Apparatus

C.2.1 Gas chromatograph, with thermal conductivity detector with stainless steel column (4m × 4.6mm) packed with molecular sieve 5A and using Ultra High purity Helium as the carrier gas. The carrier gas shall be purified further by passing through argon gas purifying system. Maintain the column at 60 °C and the detector at 130 °C.

Note Many other variables, such as column type, length, carrier flow, detector temperature, detector type and oven temperature, may be left open, and still provide a suitable system for determination of these impurities.

C.3 Reagents

C.3.1 Reference gas, commercially available calibration gas mixture of Nitrogen and methane in high purity oxygen.

C.4 Procedure

C.4.1 Set the GC conditions in accordance with the manufacturer's instructions. Calibrate the instrument by injecting a suitable volume of the reference gas (C.3.1).

C.4.2 Inject a suitable volume of both the test gas and the reference gas (C.3.1).

C.4.3 Adjust the volume, as well as the conditions specified above (C.2), to produce a peak response for the reference gas (A.3.1) that gives a height of not less than 35 % on the recorder.

C.4.4 Measure the areas of the peak responses obtained in the chromatograms from the injections of test gas and reference gas, and calculate the percentage content of each analyte by comparing with the peak response obtained from the reference gas (C.3.1).

C.4.5 The result of the calibration for hydrocarbons shall be expressed as total hydrocarbons calibrated as Methane.

Annex D

(Normative)

Method for the determination of water content

D.1 Principle

The gas is passed through a direct reading hygrometer.

D.2 Apparatus

D.2.1 A direct reading hygrometer – of one of the following types:

- a) cooled mirror dew point;
- b) electrolytic;
- c) capacitance;

D.3 Calibration

Ensure that the instrument has a current certificate of calibration demonstrating traceability to a physical standard held at the KEBS Metrology Laboratory or a recognized international body.

D.4 Procedure

Operate the direct reading hygrometer in accordance with the manufacturer's instructions. Keep all sample lines as short as practicable and, together with all ancillary equipment, ensure that they have only polytetrafluorethylene or stainless product in accordance with the manufacturer's instructions.

Record the water content that is displayed by the instrument.

Annex E

Caution Note On Oxygen

Oxygen can be supplied for industrial use in both gaseous and liquid form. Oxygen is applied in the gaseous phase only.

When oxygen supplied in liquid state it can cause cold 'burns' and make certain material sufficiently brittle to lead to structural.

Oxygen enrichment or deficiency poses hazards and should be avoided.

Oxygen enrichment increases the combustability of materials.

Hydrocarbon oil and grease should never be used to lubricate oxygen or enriched air equipment as they can ignite spontaneously and burn with explosive violence.

Many burning accidents which occur are triggered off by the lighting of a cigarette, therefore it is impossible to overemphasise the danger of smoking in oxygen enriched atmosphere or where oxygen enrichment can occur. In such areas smoking should be forbidden.

Oxygen deficiency leads to the following physiological hazards:

- loss of mental alertness.
- distortion of judgement.
- in a relatively short time considerable brain damage.

Oxygen enrichment can be avoided by taking the following precautions:

- ensure the equipment used has no leaks.
- in many metallurgical processes such as gauging, cutting, scarfing, thermic lancing, etc., employing oxygen, a surplus of the gas escapes into the atmosphere. Therefore the ventilation in areas where such processes are undertaken must be sufficient to ensure that oxygen enrichment does not occur.
- blowpipes must be used properly by correct section of the nozzles and gas pressures.
- Improper use of oxygen such as driving pneumatic tools, inflating vehicle tyres, rubber boats, etc., must be strictly forbidden.

Any oxygen deficiency of the atmosphere is best guarded against by careful attention to the following.

- avoid leakage of gases other than oxygen.
- avoid spillage of liquid gases other than oxygen.
- avoid vent outlets venting into the atmosphere.
- do not vent purging and cryogenic gases into the atmosphere.
- ensure that welding and cutting processes have sufficient workspace and ventilation.

The Industrial Cases Committee (IGC), and International Association of Companies who produce gases like oxygen, nitrogen and acetylene has published Document No. 8/76 entitled 'Prevention of accidents arising from enrichment of deficiency of the oxygen in the atmosphere'.

People who have special responsibility for safety or who are engaged in teaching or training others in the use of oxygen should study the complete IGC document in order to get more comprehensive information on the subject.