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R 1994

HARD COAL

DETERMINATION OF OXYGEN

1st EDITION

November 1971

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BRIEF HISTORY

The ISO Recommendation R 1994, *Hard coal – Determination of oxygen,* was drawn up by Technical Committee ISO/TC 27, *Solid mineral fuels,* the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1994, which was circulated to all the ISO Member Bodies for enquiry in June 1970.

The Draft was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

- Australia
- Belgium
- Canada
- Chile
- Czechoslovakia
- Denmark
- France
- Germany
- Greece
- India
- Italy
- Japan
- Netherlands
- New Zealand
- Poland
- Portugal
- South Africa, Rep. of
- Spain
- Sweden
- Switzerland
- Thailand
- Turkey
- U.A.R.
- United Kingdom
- U.S.A.
- Yugoslavia

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1994 November 1971

HARD COAL

DETERMINATION OF OXYGEN

INTRODUCTION

This ISO Recommendation follows methods developed by Schütze (1), Unterzaucher (2), and Oita and Conway (3) for the direct determination of oxygen in organic compounds. Modifications have been incorporated to avoid errors due to the presence of moisture in coal. Oxygen is evolved (as water and carbon dioxide) from the mineral matter associated with the coal when the sample is pyrolysed and, to reduce errors from this source, coals containing more than 5 % of ash and all samples of unknown characteristics should be demineralized (4) before analysis.

1. SCOPE

This ISO Recommendation describes semi-micro methods for the direct determination of oxygen in hard coal.

2. PRINCIPLE

The sample is dried at 105 to 110 °C in a stream of nitrogen and then pyrolysed under conditions in which the organic matter leaves an oxygen-free char. The volatile products, containing the organically bound oxygen and also water and carbon dioxide from any mineral matter, are decomposed with either pure carbon or platinized carbon to convert the oxygen to carbon monoxide. The carbon monoxide is oxidized to carbon dioxide and determined by a titrimetric (5) or a gravimetric procedure.

3. REAGENTS

3.1 *Nitrogen*. The nitrogen used for the pyrolysis should contain not more than 10 ppm of oxygen. If nitrogen of this purity is available commercially, further purification is not necessary if the total "blank" is within the limits specified in section 6.

3.2 *Alternative reagents for converting the volatile pyrolysis products to carbon monoxide*

(*a*) *Pure carbon*, 0.7 to 2.0 mm (6), ash content not exceeding 0.01 %.

The carbon should be ignited to dull red heat in an inert atmosphere to remove any oil before being placed in the pyrolysis tube.

Carbon containing up to 0.05 % ash can usually be purified as follows :

Heat enough carbon to pack the pyrolysis tube and to provide a sample for the determination of ash to dull red heat in an inert atmosphere to remove any residual oil. Allow to cool and digest the residue with hydrochloric acid ($d = 1.18$) at incipient boiling for 1 hour. Allow the carbon to settle and decant the liquid through a hardened filter paper in a Buchner funnel. Wash the carbon several times by decantation, then transfer it to the filter and continue until the washings are free from chloride. Dry the cake first by suction, then in an oven at 120 °C.

Crush and sieve the dried cake carefully to obtain the maximum yield of 0.7 to 2.0 mm material. Determine the ash content of the granules.

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(1) Schütze, M.Z., Anal. Chem., 1939, **118**, 258.

(2) Unterzaucher, J., Analyst, 1952, **77**, 584.

(3) Oita, I.J. and Conway, H.S., Anal. Chem., 1954, **26**, 600.

(4) See ISO Recommendation R 602, *Determination of mineral matter in coal.*

(5) Blom. L., Zeit. Anal. Chem., 1962, **189**, 91.

(6) To avoid too great a resistance to gas flow, it is important that material < 0.7 mm should be removed by sieving. Excessive resistance to gas flow may necessitate increasing the maximum size to 3 mm.

or

(*b*) *Platinized carbon* 0.7 to 2.0 mm ⁽¹⁾, containing about 50 % of platinum.

A suitable method of preparation is as follows :

Dissolve 5 g of platinum in aqua regia and evaporate the solution to near dryness. Add 5 ml of hydrochloric acid ($d = 1.18$) and again evaporate to near dryness. Continue adding 5 ml portions of hydrochloric acid and evaporating to near dryness until nitrous fumes are no longer evolved and then evaporate the solution to dryness. Moisten the residue with 2 ml of hydrochloric acid and add sufficient water to dissolve the platinic chloride. Add 5 g of ignited carbon that contains not more than 0.01 % of ash (see clause 3.2(*a*)) and more water, if necessary, to form a paste. Mix thoroughly and evaporate to dryness on a water bath, mixing frequently with a glass spatula or rod to ensure a homogeneous product. Dry the resulting cake at 150 °C.

Break the cake into pieces and insert them into a 10 to 12 mm internal diameter quartz tube. Pass dry nitrogen (3.1) through the tube and heat the tube slowly to red heat, in stages, using a Bunsen burner. Begin heating the tube at the inlet end and when this is hot move the burner very slowly along the tube until all the contents have been ignited. Replace the nitrogen with hydrogen and heat again in a similar manner in a stream of hydrogen. Repeat heating in hydrogen in this way until the effluent gas is free from hydrogen chloride as shown by its neutrality to litmus. Replace the hydrogen with nitrogen and allow the contents of the tube to cool to room temperature in a stream of nitrogen.

Remove the platinized carbon, and crush and sieve the product carefully to obtain the maximum yield of 0.7 to 2.0 mm material.

3.3 *Copper gauze*

3.4 *Soda asbestos*, 0.7 to 2.0 mm ⁽¹⁾.

3.5 *Dry magnesium perchlorate*, 0.7 to 2.0 mm ⁽¹⁾ ⁽²⁾.

3.6 *Potassium iodide/cotton wool.*

Dissolve 160 g of potassium iodide in water and dilute to 250 ml. Impregnate 100 g of cotton wool with this solution and dry at 80 °C in a stream of nitrogen. Store in a dark bottle.

3.7 *Alternative reagents for converting carbon monoxide to carbon dioxide*

(*a*) *Schütze reagent.* Dry 30 g of pure granular silica gel (1 to 2 mm) at 200 °C for 1 hour and transfer it to a 250 ml round-bottomed flask fitted with a ground glass cone having an aperture of approximately 25 mm ⁽³⁾. Add slowly, with frequent shaking, 25 ml of sulphuric acid ($d = 1.84$). Then add, in about 1 g portions, 30 g of finely ground iodine pentoxide; after the addition of each portion of iodine pentoxide thoroughly mix the contents of the flask by shaking. Close the flask with a socket that has a tube through which the flask can be evacuated. Immerse the flask to the ground joint in an oil bath at 200 °C and evacuate to a pressure less than 0.05 bar for 2 hours. Allow the flask to cool under vacuum and transfer the reagent to a dry glass bottle that has a well-ground glass stopper. Store the bottle in a desiccator to prevent the reagent from absorbing moisture.

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(1) To avoid too great a resistance to gas flow, it is important that material $<$0.7 mm should be removed by sieving. Excessive resistance to gas flow may necessitate increasing the maximum size to 3 mm.

(2) The regeneration of magnesium perchlorate should not be attempted owing to the risk of explosion. When exhausted it should be washed down the sink in a stream of water.

(3) See ISO Recommendation R 383, *Interchangeable conical ground glass joints* : 29/32 or 34/35.

or

(*b*) *Anhydro-iodic acid* (HI_3O_8).

If anhydro-iodic acid suitable for the determination of carbon monoxide cannot be obtained commercially, it may be prepared by the following method :

Add an excess of iodic acid or iodine pentoxide to a boiling mixture of 3 volumes of nitric acid ($d = 1.4$) and 2 volumes of water. Boil the solution with the undissolved excess for 1 hour to obtain a saturated solution. Filter the hot solution immediately, by suction, through a sintered glass filter (pore size 15 to 40 μm). Leave the filtrate to crystallize for a day. Transfer the crystals to a sintered glass Buchner funnel and wash them with cold nitric acid ($d = 1.4$). Dry the crystals at room temperature, first by aspirating clean air over them for 1 hour and then in a desiccator containing dishes of sodium hydroxide and phosphorus pentoxide.

or

(*c*) *Red mercuric oxide*, pellets about 2 mm diameter and 1 mm thick prepared from good quality commercial material.

Mercuric oxide having the following specification has been found suitable :

Assay :	not less than 99.3 % (dry)
Loss on drying :	not more than 0.3 % at 150 °C
Mercurous salts :	not more than a trace
Chloride :	not more than 0.25 %
Nitrate :	no reaction
Non-volatile residue :	not more than 0.1 %

Alternatively, red mercuric oxide may be prepared as follows :

Dissolve 140 g of mercury in about 100 ml of nitric acid ($d = 1.4$). Evaporate the solution to dryness in a porcelain dish. Heat the dish at about 400 °C in an efficient fume cupboard until no yellow crystals of mercuric nitrate [$Hg(NO_3)_2$] remain.

3.8 *Silver wire*, 0.1 to 0.2 mm diameter.

3.9 *Pyridine*

Pyridine having the following specification has been found suitable :

C_5H_5N :	not less than 99.0 %
Density at 20 °C :	0.981 to 0.983 g/ml
n_D^{20} :	1.5090 to 1.5100

Not less than 95 % distils at 114 to 117 °C

Maximum limits of impurities :

– water-insoluble matter :	nil
– non-volatile matter :	0.002 %
– chloride :	0.0005 %
– copper :	0.0002 %
– ammonia :	0.002 %
– oxygen absorbed from permanganate :	0.0008 %
– water :	0.1 %

If sufficiently pure pyridine is not available, re-distil with a column of 15 to 20 theoretical plates. Using a reflux ratio of 100, distil and reject the first 10 %. Reduce the reflux ratio to 10 and distil and collect the next 75 % for use.

3.10 *Ethanolamine*

3.11 *Thymol blue indicator*, 0.4 % solution in pyridine (3.9).

3.12 *Sodium methoxide*, 0.05 N solution.

Dissolve 1.15 g of sodium in 500 ml of methanol and dilute with pure pyridine to 1 litre. Store the solution so as to exclude carbon dioxide.

Weigh, to the nearest 0.1 mg, about 50 mg of benzoic acid. Place 15 ml of pyridine (3.9) in a 100 ml conical flask and add 2 drops of the thymol blue indicator (3.11). With nitrogen passing through the flask to exclude air, neutralize the contents with the sodium methoxide solution, dissolve the weighed benzoic acid in the contents of the flask and titrate with the sodium methoxide solution to a blue end-point. Calculate the normality of the sodium methoxide as follows :

$$N = \frac{m}{122.1\ V}$$

where

N is the normality of the sodium methoxide solution;

m is the mass, in milligrammes, of benzoic acid taken;

V is the volume, in millilitres, of sodium methoxide solution used in the titration.

4. APPARATUS

The general arrangement of the apparatus is shown in Figure 1. The components should be constructed with ground glass joints for connecting one to the other, except for the tube or vessel in which the carbon dioxide produced from the oxygen is absorbed.

4.1 *Nitrogen purification train*. A typical purifying train (see Figure 2) consists of :

(*a*) a pressure regulator containing heavy (medicinal quality) paraffin; a cylinder 300 mm high and 50 mm in diameter is suitable;

(*b*) a vertical tube about 120 mm high and 30 mm internal diameter containing dry magnesium perchlorate (3.5);

(*c*) a vertical quartz tube about 300 mm long and 15 mm internal diameter containing porous copper heated to approximately 500 °C. Porous copper is produced by reducing copper oxide in wire form with hydrogen diluted with nitrogen at the lowest temperature at which reduction occurs, usually between 200 and 250 °C;

(*d*) a bubbler, containing heavy (medicinal quality) paraffin, to provide a visual indication of the flow rate of the nitrogen;

(*e*) a U-tube about 150 mm high and 15 mm internal diameter, the first one-third of which contains soda asbestos (3.4), the next one-third magnesium perchlorate (3.5) and the final one-third a mixture of 2 parts by volume of phosphorus pentoxide powder and 1 part by volume of 0.7 to 2.0 mm glass chips.

4.2 *Pyrolysis tube assembly*. The pyrolysis tube should be of 10 ± 1 mm internal diameter and made from the purest available clear quartz, free from striae and other visible defects.

Two typical designs for the inlet end of the pyrolysis tube are shown. The design shown in Figure 3(*a*) incorporates an auxiliary tube with which it is possible to exclude air while the sample is being inserted. The sample is both dried and pyrolysed between the nitrogen inlet tube and the carbonaceous packing and this section must be sufficiently long to accommodate any heater used for drying the sample and also for pyrolysing the sample. A suitable removable heater for drying the sample is shown in Figure 4.

In the design shown in Figure 3(*b*), air admitted when the sample is inserted is removed, together with the moisture, before the sample is pyrolysed. The distance between the nitrogen inlet and the cap of the pyrolysis tube should be sufficient to accommodate the heater used to dry the sample and the distance between the nitrogen inlet and the carbonaceous packing sufficient for pyrolysing the sample.

The sample may be pyrolysed using either gas or electric heating, but electric furnaces are preferable for heating reagents.

Conversion of the oxygen in the volatile pyrolysis products to carbon monoxide may be achieved either with pure carbon (3.2(*a*)) or with platinized carbon (3.2(*b*)). If pure carbon (3.2(*a*)) is used, the packed length should be 190 to 200 mm. Rolled platinum gauze plugs 5 mm long are used to retain the packing in position. A furnace should be provided to heat the carbon to 1125 ± 25 °C.

If platinized carbon (3.2(*b*)) is used, the packing should consist of two 45 to 50 mm sections separated and retained by 5 mm long platinum gauze plugs. A furnace should be provided to heat the platinized carbon to 940 °C, but the normal operating temperature is 910 ± 10 °C.

Either reducing agent should be followed by a roll of copper gauze (3.3), 50 to 60 mm long, heated by a suitable furnace to 900 °C.

4.3 *Carbon monoxide purification tube*, 190 to 210 mm long and 10 to 15 mm internal diameter. A length of 130 to 140 mm is packed with soda asbestos (3.4) to remove acidic gases (HCl, H_2S, etc.), followed by about 60 to 70 mm packed with dry magnesium perchlorate (3.5) to remove water produced by reaction of the acidic gases with the soda asbestos.

4.4 *Carbon monoxide oxidation tube*, 10 ± 1 mm internal diameter and 220 to 250 mm long (depending on which of the alternative agents is used). The tube is packed with one of the following series of reagents in the order given in each case :

(*a*) (1) 15 to 20 mm packed with potassium iodide/cotton wool (3.6);

(2) 100 to 110 mm packed with the Schütze reagent (3.7(*a*));

(3) 35 to 40 mm packed with potassium iodide/cotton wool (3.6);

(4) 45 to 50 mm packed with dry magnesium perchlorate (3.5).

Each separated and retained by plugs of glass wool 5 mm long.

or

(*b*) (1) 130 to 140 mm packed with anhydro-iodic acid (3.7(*b*)), heated to 120 °C;

(2) 35 to 40 mm packed with potassium iodide/cotton wool (3.6);

(3) 45 to 50 mm packed with dry magnesium perchlorate (3.5).

Each separated and retained by plugs of glass wool 5 mm long.

or

(*c*) (1) 140 to 150 mm packed with red mercuric oxide (3.7(*c*)), retained by 5 mm plugs of glass wool and heated to 200 °C;

(2) 45 to 50 mm empty (and at room temperature) to allow mercury to condense;

(3) 35 to 40 mm packed with a plug of silver wire (3.8) to absorb mercury vapour.

4.5 *Apparatus for determining the carbon dioxide produced in the oxidation tube (4.4)*

4.5.1 *Titrimetric finish.* Suitable apparatus for absorbing and titrating the carbon dioxide is shown in Figure 5.

4.5.2 *Gravimetric finish.* A convenient design of absorption tube is shown in Figure 6. The absorption tube is followed by a guard tube containing dry magnesium perchlorate (3.5) and soda asbestos (3.4).

A balance sensitive to 0.01 mg is necessary for weighing the absorption tube.

4.6 *Platinum boat*, about 15 mm long, 4 mm wide and 4 mm deep, which has a lug pierced with a hole attached at one of its upper ends to facilitate removal from the apparatus.

5. SAMPLE

The coal used for the determination is the analysis sample, ground to pass a sieve of 0.2 mm aperture. Before commencing a determination, thoroughly mix the air-dried sample of coal for at least 1 minute, preferably by mechanical means.

6. PREPARATION OF THE APPARATUS

Assemble the components of the apparatus, charged with the appropriate reagents as indicated in sections 3 and 4, in the order shown in Figure 1 (1). Use low vapour pressure grease and wax that are impervious to atmospheric oxygen, carbon dioxide and water vapour to seal the ground joints. Use tubing impervious to water vapour and carbon dioxide (2) to connect the absorption vessel or tube to the oxidation tube; the ends of the glass tubes inside the sleeve should touch.

Adjust the nitrogen flow rate to 25 ml/min. Check that the apparatus is gas-tight, for example by pressurizing the apparatus to the pressure provided by the regulator in the nitrogen purification train (Figure 2(*a*)) and observing that the flow of nitrogen through the bubbler (Figure 2(*d*)) ceases. Heat the reagents to their working temperatures and heat the section of the pyrolysis tube in which the sample will be pyrolysed to a minimum temperature of 900 °C. Sweep out the apparatus with nitrogen for at least 15 hours.

Carry out a blank determination as described in section 7 but omitting the sample. If the blank is equivalent to more than 0.12 mg of oxygen, i.e. more than 0.15 ml of 0.05 N sodium methoxide solution (titrimetric finish) or more than 0.33 mg of carbon dioxide (gravimetric finish), sweep out the apparatus with nitrogen for a further period and then re-check the blank (3). When satisfactory blanks are obtained, check the performance of the apparatus using a pure organic compound (4) (5). If the result is within 0.2 % of the theoretical oxygen content, the apparatus is ready for use (6).

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(1) The reagents used in the titrimetric procedure contain pyridine, which necessitates housing the apparatus in well-ventilated surroundings.

(2) Polyethylene or polytetrafluorethylene tubing is suitable for connecting the absorption vessel when the titrimetric finish is used, and butyl rubber or natural rubber impregnated with wax for the absorption tube in the gravimetric finish.

(3) It may be necessary to sweep with nitrogen for a few days before a satisfactory blank is obtained.

(4) Acetanilide, anthraquinone, benzoic acid, 2-naphthol, octadecanol, sucrose and vanillin are suitable.

(5) The mass of sample taken should be such that the blank is equivalent to an oxygen content not exceeding 0.25 %.

(6) Some batches of platinized carbon need a higher temperature than 920 °C, If the values are only slightly low, raise the operating temperature to 940 °C and again check the performance of the apparatus.

7. PROCEDURE

Heat the platinum boat in nitrogen to the pyrolysis temperature and allow it to cool in nitrogen. Weigh, to the nearest 0.1 mg, 20 to 50 mg (1) of the sample into the boat.

7.1 Pyrolysis tube of the form shown in Figure 3(*a*)

7.1.1 *Titrimetric finish*. With nitrogen passing through the apparatus at about 25 ml/min, allow about 12 ml of the pyridine (3.9) to flow slowly from the reservoir to the absorption vessel so that pyridine does not enter the jet. Add about 100 mg of the ethanolamine (3.10) and 2 drops of the thymol blue indicator solution (3.11). Wait 2 to 3 minutes for the contents of the vessel to mix and then neutralize the solution with the 0.05 N sodium methoxide solution (3.12) to the blue end point. Close stopcock S_2 (Figure 5) at the entrance to the absorption vessel (2).

Pass nitrogen into the auxiliary tube at about 50 ml/min. Remove the stopper from the pyrolysis tube and fit the auxiliary tube. Using the magnet, slide the boat holder into the auxiliary tube. Insert the boat containing the sample into the holder and immediately slide it into the pyrolysis tube, remove the auxiliary tube and replace the stopper in the pyrolysis tube.

Pressurize the apparatus to the pressure provided by the regulator in the nitrogen purification train (Figure 2(*a*)). Open stopcock S_2 (Figure 5). Heat the boat to 105 to 110 °C in about 10 minutes. Titrate the carbon dioxide produced from the moisture in the sample continuously, to the blue end point. Maintain the boat at 105 to 110 °C until the production of carbon dioxide ceases (about 20 minutes), thus showing that the sample is dry (3). Read the burette, pyrolyse the sample at a minimum temperature of 900 °C for 20 minutes and titrate the carbon dioxide continuously to the blue end point. Sweep out the apparatus for a further 10 minutes and complete the titration (4).

7.1.2 *Gravimetric finish*. Pass nitrogen into the auxiliary tube at 50 ml/min and insert the boat into the pyrolysis tube as described in clause 7.1.1. Dry the sample by heating to 105 to 110 °C in the pyrolysis tube in a 25 ml/min stream of nitrogen for at least 20 minutes.

While the sample is being dried, wipe the absorption tube (5) with a damp chamois leather, polish it with a dry chamois leather and place it in the balance case for about 20 minutes to attain equilibrium with the atmosphere. Open and immediately close one of the stopcocks of the absorption tube and weigh the tube to the nearest 0.01 mg.

Connect the absorption tube to the apparatus, pyrolyse the dried sample for 20 minutes and sweep out the apparatus for a further 10 minutes (4). Close the stopcocks of the absorption tube, disconnect it and replace the guard tube.

Polish and weigh the absorption tube using the same procedure as for the initial weighing.

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(1) The mass of sample taken should be such that the blank is equivalent to an oxygen content not exceeding 0.25 %.

(2) This stopcock must be closed when the pyrolysis tube is opened to insert the sample, to prevent the reagent in the absorption vessel flowing into the beak, where carbon dioxide would be absorbed but not titrated.

(3) The moisture content of the sample may be calculated from the volume of sodium methoxide used. One millilitre of N sodium methoxide is equivalent to 18.02 mg of water.

(4) Alternatively, the pre-drying may be omitted and the total oxygen in the air-dried sample, including that present as moisture, determined by heating for 10 minutes at 105 to 110 °C and 20 minutes at a minimum temperature of 900 °C followed by 10 minutes sweeping out. The moisture is determined separately (see ISO Recommendation R 331, *Determination of moisture in the analysis sample of coal by the direct gravimetric method*, or ISO Recommendation R 348, *Determination of moisture in the analysis sample of coal by the direct volumetric method*) and the oxygen present as moisture deducted from the total oxygen (water contains 88.81 % of oxygen).

(5) Freshly packed absorption tubes should be thoroughly swept with nitrogen before being used.

7.2 Pyrolysis tube of the form shown in Figure 3(*b*)

7.2.1 *Titrimetric finish*. Close the stopcock S_2 (Figure 5). Remove the cap from the pyrolysis tube and insert the boat containing the sample followed by the iron-cored glass "slug". Open stopcock S_1 (Figure 3(*b*)) and replace the cap. Position the boat between the cap and the nitrogen inlet. Heat the boat in the stream of nitrogen to 105 to 110 °C for 20 minutes to dry the sample. Close stopcock S_1.

Open stopcock S_2. Fill the absorption vessel and neutralize the solution as described in clause 7.1.1.

Using the magnet, push the boat with the iron-cored glass "slug" to the pyrolysis position between the nitrogen inlet and the carbonaceous packing and withdraw the "slug" to the cap. Pyrolyse the sample at a minimum temperature of 900 °C for 20 minutes and titrate the carbon dioxide continuously to the blue end point. Sweep out the apparatus for a further 10 minutes and complete the titration.

7.2.2 *Gravimetric finish*. Insert and dry the sample as described in clause 7.2.1.

While the sample is being dried, wipe and weigh the absorption tube (1) as described in clause 7.1.2 and connect it to the apparatus. Close stopcock S_1. Push the boat to the pyrolysis position, withdraw the "slug" to the cap, pyrolyse the sample and sweep out the apparatus as described in clause 7.2.1.

Close, detach, polish and weigh the absorption tube using the same procedure as for the initial weighing.

8. CALCULATION AND EXPRESSION OF RESULTS

8.1 Titrimetric method

The oxygen (O) in the coal analysed, expressed as a percentage, is given by the formula

$$\text{O} = \frac{1600\,N\,V}{m}$$

where

m is the mass of sample taken, expressed in milligrammes;

N is the normality of the sodium methoxide solution;

V is the volume of sodium methoxide used to titrate the carbon dioxide produced by the pyrolysis, corrected for any blank, expressed in millilitres.

8.2 Gravimetric method

The oxygen (O) in the coal analysed, expressed as a percentage, is given by the formula

$$\text{O} = \frac{36.36\,a}{m}$$

where

a is the increase in mass of the absorption tube resulting from the absorption of carbon dioxide produced by the pyrolysis, corrected for any blank, expressed in milligrammes;

m is the mass of sample taken, expressed in milliligrammes.

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(1) Freshly packed absorption tubes should be thoroughly swept with nitrogen before being used.

9. PRECISION OF THE METHOD

Oxygen	Maximum acceptable difference between results obtained	
	in the same laboratory (repeatability)	in different laboratories (reproducibility)
	0.3 % absolute	0.5 % absolute

9.1 Repeatability

The results of duplicate determinations carried out at different times in the same laboratory, by the same operator, with the same apparatus, on the same analysis sample, should not differ by more than the above value.

9.2 Reproducibility

The means of the results of duplicate determinations carried out in each of two laboratories, on representative portions taken from the same sample after the last stage of sample preparation, should not differ by more than the above value.

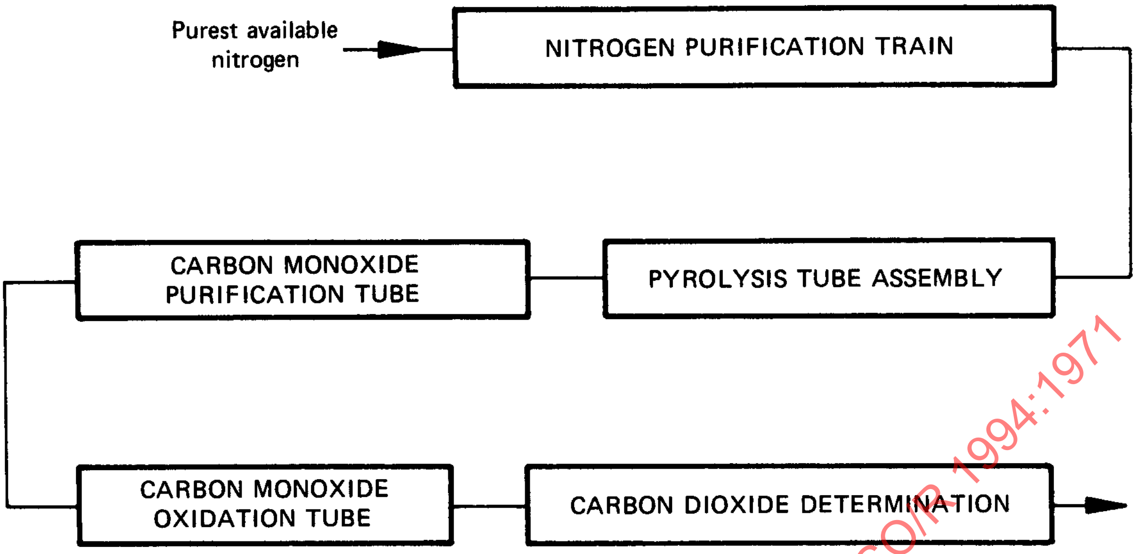

FIGURE 1 – General arrangement of apparatus

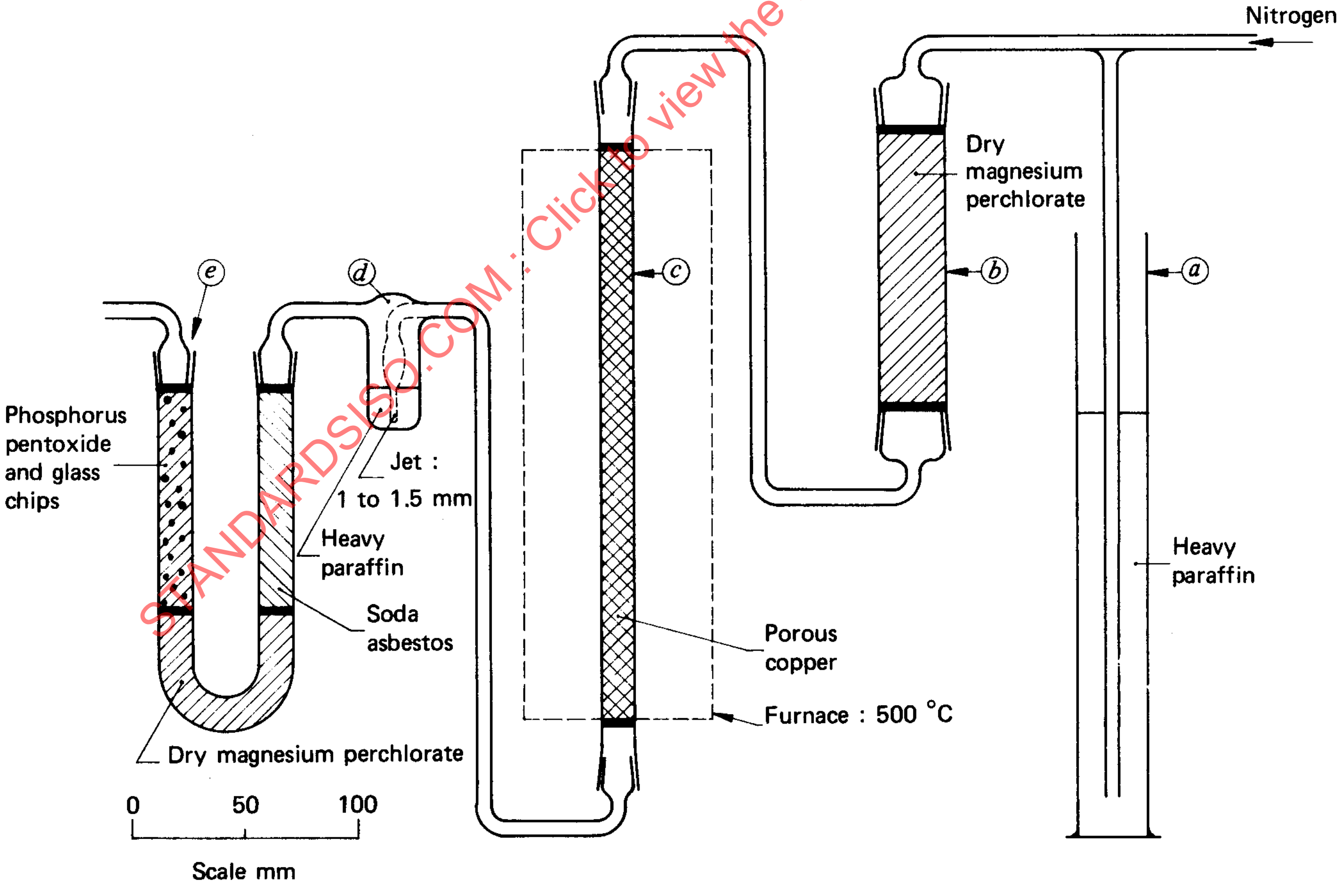

FIGURE 2 – Nitrogen purification train