INTERNATIONAL STANDARD 609

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Coal and coke – Determination of carbon and hydrogen – High temperature combustion method

Charbon et coke – Dosage du carbone et de l'hydrogène – Méthode par combustion à haute température

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 609 and found it technically suitable for transformation. International Standard ISO 609 therefore replaces ISO Recommendation R 609-1967 to which it is technically identical.

ISO Recommendation R 609 was approved by the Member Bodies of the following countries :

Australia
Austria
Belgium
Bulgaria
Canada
Chile
Czechoslovakia
Denmark
Germany
India
Italy
Japan
Netherlands
New Zealand
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Switzerland
U.A.R.
United Kingdom
U.S.A.

The Member Bodies of the following countries expressed disapproval of the Recommendation on technical grounds :

France
Turkey*
U.S.S.R.

No Member Body disapproved the transformation of ISO/R 609 into an International Standard.

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* Subsequently, this Member Body approved the Recommendation.

Printed in Switzerland

INTERNATIONAL STANDARD **ISO 609-1975 (E)**

Coal and coke — Determination of carbon and hydrogen — High temperature combustion method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a high temperature combustion method of determining the total carbon and the total hydrogen in hard coal, brown coal, lignite and coke; the results include the carbon in the carbonates and the hydrogen combined in the moisture and in the water of hydration of silicates. A determination of moisture is carried out at the same time, and an appropriate correction is applied to the hydrogen value obtained by combustion. A determination of carbon dioxide may also be made and the total carbon value corrected for the presence of mineral carbonates.

An alternative method for the determination of carbon and hydrogen, involving combustion at a lower temperature (the Liebig method) is given in ISO 625[1].

2 PRINCIPLE

The sample is burned in a rapid current of oxygen at a high temperature in an impervious tube; all the hydrogen is converted to water and all the carbon to carbon dioxide. These products are absorbed by suitable reagents and determined gravimetrically. Oxides of sulphur and chlorine are retained by a silver gauze roll at the outlet end of the tube.

3 REAGENTS

All reagents shall be of analytical reagent quality. Distilled water shall be used throughout.

3.1 Magnesium perchlorate, anhydrous (anhydrone), less than 1,2 mm in size and preferably within the size range 1,2 to 0,7 mm.

NOTE — Regeneration of the magnesium perchlorate shall not be attempted, owing to the risk of explosion. When exhausted, the magnesium perchlorate shall be washed down the sink with a current of water.

3.2 Soda asbestos, preferably of coarse grading, for example 3,0 to 1,5 mm, and not finer than the grading 1,2 to 0,7 mm, and preferably of self-indicating type.

3.3 Alumina, finely divided, approximately 0,1 mm in size.

3.4 Sodium borate solution, 0,05 N.

3.5 Hydrogen peroxide, 1 % neutral solution.

3.6 Pure silver gauze, approximately 1 mm mesh, made of wire approximately 0,3 mm in diameter.

3.7 Oxygen, hydrogen-free, preferably prepared from liquid air and not by electrolysis. Electrolytically prepared oxygen shall be passed over red-hot copper oxide before use to remove any trace of hydrogen.

4 APPARATUS

4.1 Two purification trains, one for absorbing water vapour and carbon dioxide from the oxygen used for the combustion and the other for similarly treating the air used for sweeping out the absorption train before and after a determination. Assemble each train using the following reagents in the order stated :

1) Magnesium perchlorate (3.1) for absorbing water,

2) Soda asbestos (3.2) for absorbing carbon dioxide,

3) Magnesium perchlorate (3.1) for absorbing the water evolved in the reaction between carbon dioxide and soda asbestos.

The purification trains shall be large enough to render frequent recharging unnecessary even with continuous use.

4.2 Combustion assembly

4.2.1 Furnace, capable of carrying a tube approximately 25 mm outside diameter and heating it over a length of approximately 125 mm to over 1 250 °C and up to a maximum of 1 350 °C. A temperature-distribution curve for a typical furnace is shown in figure 1. Suitable furnaces are, for example,

a) molybdenum or tungsten wire wound;

b) platinum or platinum-rhodium wire wound;

c) heated by silicon carbide rods.

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[1] ISO 625, *Coal and coke — Determination of carbon and hydrogen — Liebig method.*

Type c) has the lowest initial cost and has proved satisfactory in use.

NOTE – Furnaces of the type normally used for the determination of carbon or sulphur in steel are not suitable because of the absence of the auxiliary section to maintain the silver gauze roll at the correct temperature.

4.2.2 Combustion tube, approximately 20 mm internal diameter and approximately 700 mm long, made of refractory aluminous porcelain which is not permeable to gases at 1 400 °C.

4.2.3 Combustion boat of iron-free unglazed porcelain, approximately 60 mm long, 12,5 mm wide and 10 mm deep, capable of withstanding a temperature of 1 350 °C.

NOTE – Boats should not blister, discolour or change in mass on heating in oxygen at 1 350 °C for 3 h. A suitable boat lasts for about 10 to 20 determinations and should then be discarded because of the accumulation of fused ash. For high ash coals, it may be convenient to line the boat with alumina before adding the sample, in order to prevent fusion of the ash to the boat.

4.3 Absorption train, for absorbing the water and carbon dioxide evolved by the combustion of the sample. Midvale tubes (figure 2), which provide a large area of reaction, are used in order to reduce the back-pressure in the apparatus and so obviate the danger of leakage through the rubber sleeve carrying the pusher. Assemble the train using the following reagents in the order stated :

1) Magnesium perchlorate (3.1) for absorbing the water evolved during the combustion

2) Soda asbestos (3.2) for absorbing carbon dioxide

3) Magnesium perchlorate (3.1) for absorbing the water evolved in the reaction between carbon dioxide and soda asbestos.

Place glass wool above and below the absorbents to prevent the carryover of dust by the rapid flow of oxygen and the cracking of the soda asbestos tube by the heat of reaction. A typical absorption train with details of the packing is shown in figure 3. A is the absorber for water; B and C are the absorbers for carbon dioxide, C serving as a control to indicate when the packing in B is in need of replacement. Any water released in B by the reaction between soda asbestos and carbon dioxide is absorbed in C.

4.4 Two flowmeters, one capable of measuring rates of flow up to 300 ml/min and the other to 250 ml/min.

4.5 U-gauge to measure the resistance of the system.

NOTE – The normal back-pressure on the system is 5 to 7 mbar.

4.6 Heat-resisting stopper (acrylonitrile or chloroprene) for connecting the absorption train to the combustion tube.

4.7 Silica pusher about 450 mm long, with a disk end 12 mm in diameter for pushing the boat into the furnace, made from 6 mm diameter silica rod flattened at one end, or from a 6 mm diameter silica tube sealed and flattened similarly.

The pusher passes loosely through a glass T-piece, one end of which fits into the rubber stopper which closes the inlet end of the combustion tube, the other being sealed with a rubber sleeve[1], through which the pusher slides. Oxygen is admitted through the limb of the T-piece. The pusher is marked from the disk end for convenience in ascertaining the position of the boat in the combustion tube during pushing.

4.8 Balance sensitive to 0,1 mg.

5 PREPARATION OF SAMPLE

The sample used for the determination of carbon and hydrogen is the analysis sample ground to pass a sieve of 0,2 mm aperture. If necessary expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere.

Before commencing the determination, mix the air-dried sample of coal or coke thoroughly for at least 1 min, preferably by mechanical means. A small portion of the sample shall be re-exposed to the air for a few hours and again mixed before weighing out the quantity required for the determination.

A determination of moisture shall be made immediately on the remainder of this small sample.

6 PREPARATION OF APPARATUS

6.1 Preparation of the combustion tube

Insert the combustion tube into the furnace so that it projects 75 mm at the exit end. Wrap this projecting portion with asbestos string to prevent condensation of water in the tube. Insert the rubber stopper carrying the silica pusher at the inlet end of the combustion tube and connect the purified oxygen supply to the limb of the glass T-piece.

6.2 Preparation and location of the silver gauze roll

The roll of silver gauze (3.6), through which passes a stout silver wire provided with a loop for convenience of withdrawal, should be 75 mm long and of sufficient diameter to ensure a close sliding fit in the combustion tube. Insert the silver gauze roll into the exit end of the combustion tube to a distance of about 75 mm. With the furnace at its working temperature of 1 350 °C, connect a

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[1] This sleeve should be changed periodically to avoid loss by leakage.

bubbler containing a 1 % solution of the hydrogen peroxide (3.5) to the exit end of the combustion tube by means of a heat-resisting stopper. Burn 0,5 g of a coal containing 1 to 2 % of sulphur by the procedure described below. Determine the amount of oxides of sulphur passing the silver gauze roll by titration of the hydrogen peroxide with the sodium borate (3.4). Move the silver gauze roll in 5 mm stages and repeat the test until a position is found where the volume of the sodium borate (3.4) corresponding to the end point is not more than 0,2 ml, which will represent at least 99 % retention of the oxides of sulphur by the silver gauze roll. Note its exact location.

NOTE – The silver gauze roll can absorb sulphur equivalent to 7 % of the sample; for higher sulphur contents, the mass of the coal sample should be proportionally reduced. The roll should be periodically cleaned by boiling in water (to remove silver sulphate), in concentrated ammonia solution (to remove silver chloride) and finally in water, after which it should be dried. The total sulphur content of samples examined between two cleanings of the roll should not exceed 0,035 g.

7 PROCEDURE

Raise the furnace to its working temperature of 1 350 °C and confirm that the silver gauze roll is in its correct position. Admit the oxygen (3.7) to the apparatus about 30 min before the temperature reaches 1 350 °C and adjust the rate of flow to 300 ml/min.

Spread about 0,5 g of the sample evenly over the bottom of a dried boat and weigh accurately. Cover with about 0,5 g of the alumina (3.3) (with care, 0,5 g of alumina can be made to blanket completely the sample in the boat). Connect a previously weighed absorption train and insert the charged boat into the combustion tube to a position such that its centre is 240 mm from the centre of the hottest zone. With the silica pusher fully withdrawn, replace the rubber stopper and continue to pass the oxygen (3.7) at a rate of 300 ml/min. At the end of each of the next six one-minute periods push the boat forward about 40 mm, withdrawing the silica pusher each time to prevent distortion; after the last push the centre of the boat should be at the centre of the hottest zone (see note 1).

Allow the boat to remain in the hottest zone for a further 4 min. Disconnect the absorption train, connect it to the purification train and purge it for 10 min with purified air at a rate of 200 to 250 ml/min. Disconnect the absorbers A, B and C, close the taps and stopper the central side-arms and allow to cool (see note 2).

Wipe and remove the guard stoppers and weigh the absorbers (see note 3).

A bent nickel-chromium wire can be used to withdraw the heated boat, which is conveniently pulled on to a thick sheet of asbestos.

NOTES

1 Certain coals which liberate volatile matter at a high rate in the early stages of heating may give a carryover of carbon particles. For such coals, the rate of pushing shall be reduced according to the following procedure :

> Insert the charged boat so that its centre is 240 mm from the centre of the hottest zone. At the end of the first minute, move the boat forward 40 mm. At the end of each of the next eight minutes move the boat forward by 20 mm, and at the end of the tenth minute move the boat forward by 40 mm. Allow the boat to remain in the hottest zone for a further 4 min.

For unreactive cokes, for example those with less than 0,5 % of volatile matter, the rate of pushing shall also be reduced as described above.

2 Midvale tubes used for the absorption of carbon dioxide cool slowly and 60 min should be allowed before weighing.

3 When not connected to the apparatus, the Midvale tubes should be protected from atmospheric contamination by closing the taps and fitting guard stoppers of plugged rubber tubing to the open limbs. It is usual to weigh the absorption tubes without guard stoppers, after wiping.

8 EXPRESSION OF RESULTS

The total carbon C, in the sample as analysed[1], expressed as a percentage by mass, is given by the formula

$$27{,}29\frac{m_2}{m_1}$$

where

m_1 is the mass, in grams, of sample taken;

m_2 is the increase in mass, in grams, of absorbers B and C.

The total carbon less that present as carbon dioxide C_1, expressed as a percentage by mass, is given by the formula

$$C - 0{,}273\ CO_2$$

where CO_2 is the carbon dioxide, in per cent, in the sample as analysed, determined according to ISO/R 925.[2]

The total hydrogen H, in the sample as analysed[1], expressed as a percentage by mass, is given by the formula

$$11{,}19\frac{m_3}{m_1}$$

where m_3 is the increase in mass, in grams, of absorber A, less the moisture in the alumina used (see note).

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[1] Calculation to other bases is dealt with in ISO 1170.

[2] See ISO/R 925, *Determination of carbon dioxide in coal by the gravimetric method.*

The total hydrogen less that present as moisture H_1, expressed as a percentage by mass, is given by the formula

$$H - 0{,}111\,9\,M$$

where M is the moisture, in per cent, in the sample as analysed.

The results (preferably the mean of duplicate determinations, see clause 9) should be reported to the nearest 0,1 % for the carbon determination and to the nearest 0,01 % for the hydrogen determination.

NOTE — The total moisture in the alumina is determined by heating 1 g to 1 350 °C in the apparatus and absorbing the water in magnesium perchlorate (3.1).

Drying at 105 °C is unsuitable, since the water in the alumina is not liberated below 1 000 °C. Only one or two determinations of moisture need be carried out on each bottle of alumina (3.3).

9 PRECISION OF THE METHOD

	Maximum acceptable differences between results (calculated to the same moisture content)	
	Repeatability	Reproducibility
Carbon	0,25 % absolute	0,5 % absolute
Hydrogen	0,12 % absolute	0,25 % 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 representative portions taken from the same analysis sample, should not differ by more than the above values.

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 values

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

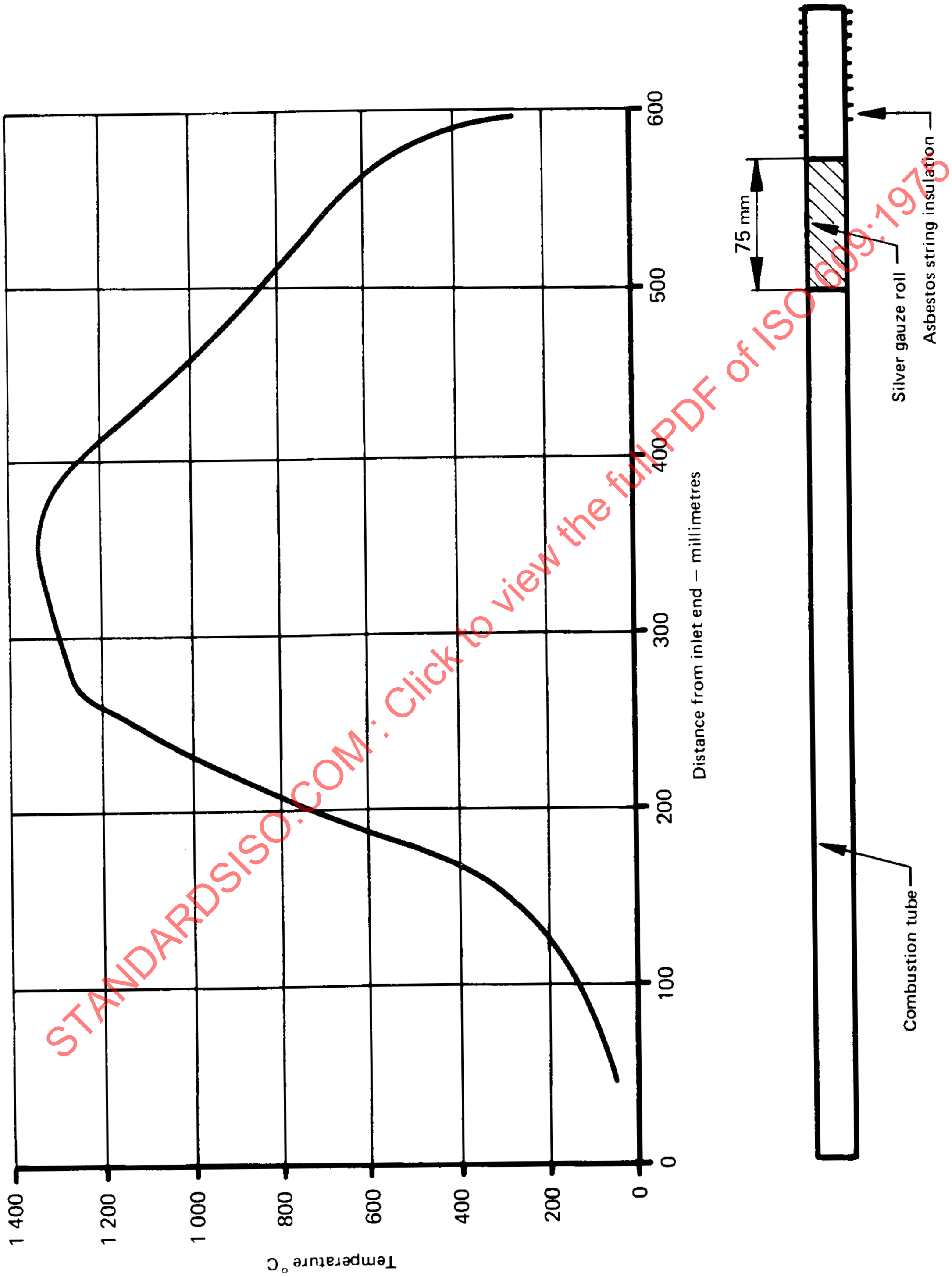

FIGURE 1 — **Typical temperature-distribution curve for furnace.**

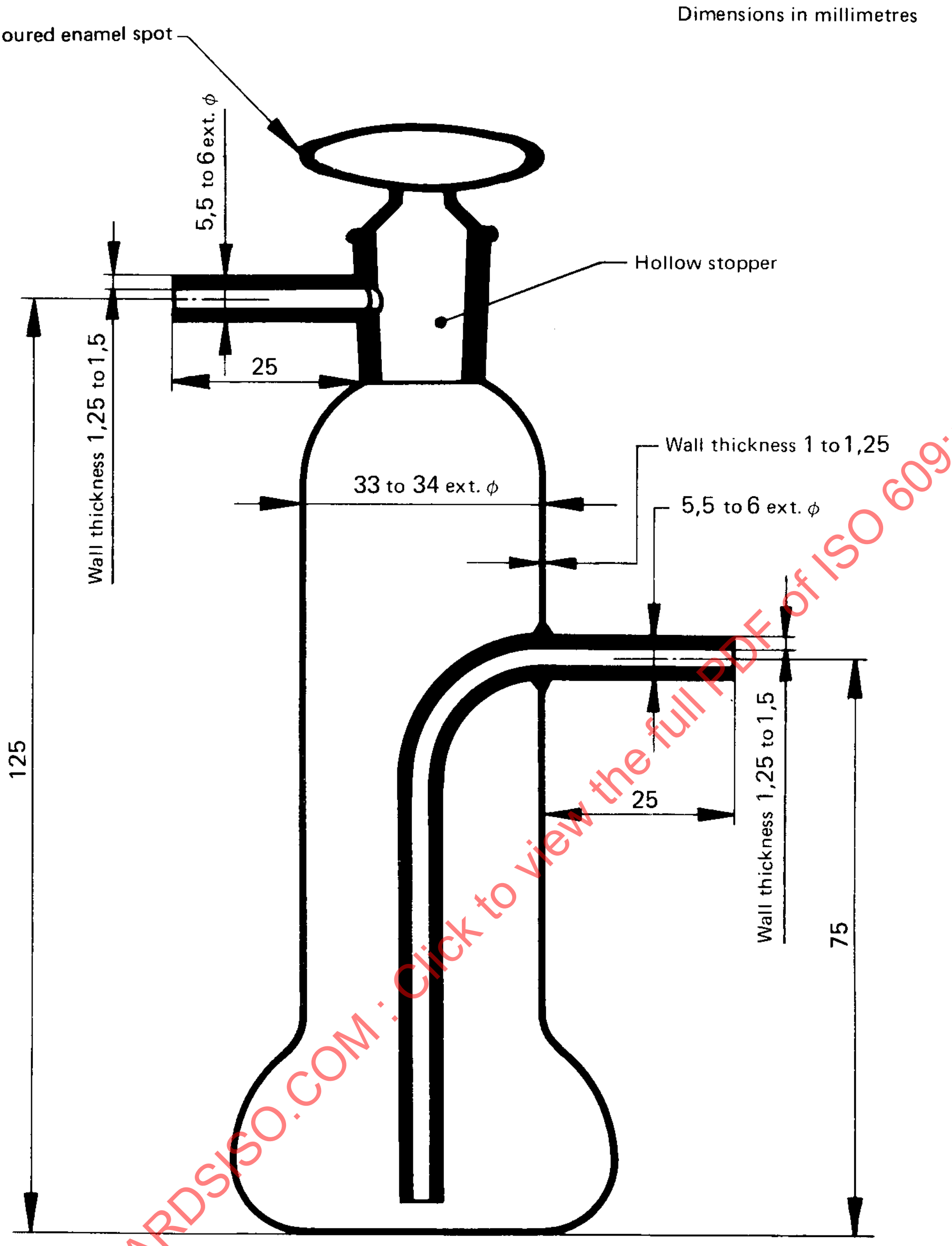

FIGURE 2 – **Midvale tube**

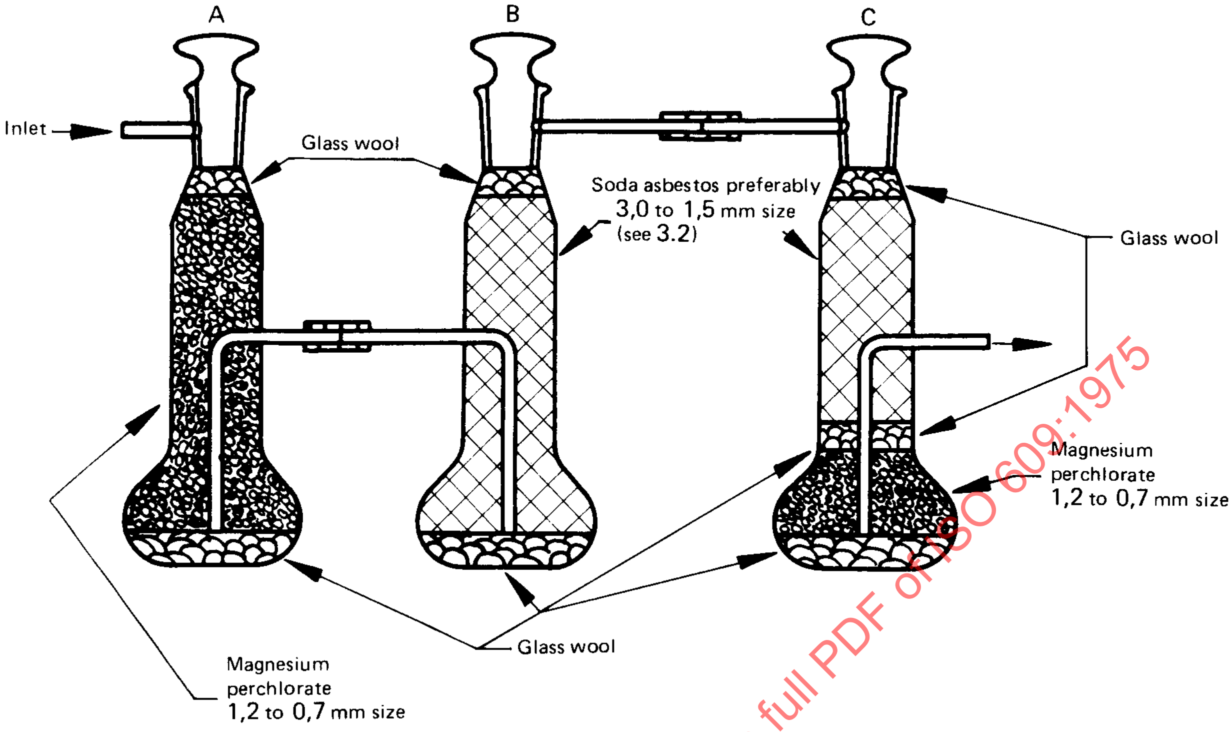

FIGURE 3 – **Absorption train**