IS 1350-3 (1969): Methods of test for coal and coke, Part III: Determination of sulphur [PCD 7: Solid Mineral Fuels]

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IS : 1350 (Part III) - 1969
(Reaffirmed 2000)

Indian Standard

METHODS OF TEST FOR COAL AND COKE

PART III DETERMINATION OF SULPHUR

(First Revision)

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

Gr 4 *July* 1970

AMENDMENT NO. 1 JUNE 1973

TO

IS : 1350 (Part III)-1969 METHODS OF TEST FOR COAL AND COKE

PART III DETERMINATION OF SULPHUR

(First Revision)

Alterations

(*Page* 5, *clause* **5.1.2**, *informal table*) — Substitute the following for the existing informal table:

Expected Sulphur Content, Percent by Mass	*Quantity of Sample* g
0·1 and up to and including 2·5	1
Above 2·5 and up to and including 5	0·5
Above 5 and up to and including 10	0·25

NOTE — Coals containing more than 10 percent sulphur are rare. Therefore, in abnormal cases, mass of sample and the method adopted shall have to be suitably modified.

(*Page* 6, *clause* **5.1.4.1**, *Note*) — Delete.

(*Page* 6, *clause* **5.1.4.4**) — Substitute the following for the existing clause:

' **5.1.4.4** *Barium chloride solution* — Dissolve 100 g of pure crystalline barium chloride ($BaCl_2.2H_2O$) in water, dilute to one litre, add 0·5 ml of hydrochloric acid and filter. '

(*Page* 6, *clause* **5.1.4.7**) — Delete.

[*Page* 7, *clause* **5.1.5.4** (i), *line* 2] — Delete the word ' fluted, '.

(*Page* 8, *clause* **5.1.5.5**, *line* 2) — Substitute the word ' dry ' for the words ' on a silica plate '.

(*Page* 9, *clause* **5.2.3.7**, *last sentence*) — Substitute the following for the existing matter:

' Add 0·5 ml of hydrochloric acid and filter before use through a close-textured double acid-washed filter paper (Whatman No. 42) or a filter paper pad. '

[*Page* 11, *clause* **5.2.4.1** (i), *line* 2] — Delete the word ' fluted '.

Addendum

(*Page* 11, *clause* **5.2.4.2**, *line* 11) — Add the words ' containing 1 g of ammonium chloride per litre ' between the words ' water, ' and ' pierce '.

(CDC 14)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 JUNE 2011
TO
IS 1350 (PART 3) : 1969 METHODS OF TEST FOR COAL AND COKE

PART 3 DETERMINATION OF SUPHUR

(First Revision)

(*Page* 3, *clause* **0.4**, *line* 1) — Delete the words “ISO/R157 ‘Determination of forms of sulphur in coal’ and”.

(*Page* 3, *clause* **1.1**, *line* 2) — Delete the words ‘and forms of sulphur’.

(*Page* 9, *clause* **5.2**) — Delete the whole clause.

(PCD 7)

Reprography Unit, BIS, New Delhi, Indi

Indian Standard

METHODS OF TEST FOR COAL AND COKE

PART III DETERMINATION OF SULPHUR

(First Revision)

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Indian Standard

METHODS OF TEST FOR COAL AND COKE

PART III DETERMINATION OF SULPHUR

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part III) (First Revision) was adopted by the Indian Standards Institution on 29 December 1969, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Chemical Division Council.

0.2 IS: 1350 was originally issued in 1959. Later a study of various recommendations made by International Organization for Standardization (ISO), as well as the experience gained with the adoption of these methods in the country necessitated the present revision. This standard is now issued in three parts as follows to cover separate methods in individual parts:

Part I Proximate analysis,
Part II Determination of calorific value, and
Part III Determination of sulphur.

0.3 To keep in line with the suggestions of ISO to avoid adoptions of alternative methods, the titrimetric method of sulphur determination and procedure for alkaline extraction of the same have been omitted from this standard.

0.4 This standard is based on ISO/R157 'Determination of forms of sulphur in coal' and ISO/R334 'Determination of total sulphur in coal by the Eschka method'.

0.5 In reporting the result of a test or analysis 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*.

1. SCOPE

1.1 This standard (Part III) prescribes the methods of test for coal and coke relating to the determination of total sulphur and forms of sulphur.

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

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definition shall apply.

2.1 Air-Dried Coal — The sample of coal which, for the purposes of analysis, is exposed to the atmosphere of the laboratory to bring it in equilibrium with the humidity conditions prevailing in the laboratory, so that the sample does not lose or gain weight during weighing.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water conforming to IS : 1070-1960* shall be employed in test.

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

4. SAMPLING

4.1 Methods of Sampling — Methods of sampling shall be as prescribed in IS : 436 (Part I)-1964† for coal and IS : 436 (Part II)-1965‡ for coke.

4.2 Preparation of Samples for Test

4.2.0 *General* — Besides the laboratory samples of coal and coke to be used for the analysis, special samples are recommended for moisture.

4.2.1 The samples prepared in accordance with IS : 436 (Part I)-1964† and (Part II)-1965‡ shall be received in sealed containers and shall consist of the following:

a) Analysis sample of about 300 g of air-dried coal or coke, ground to pass 212-micron IS Sieve; and

b) Special moisture sample of 1 kg of coal or 2·5 kg of coke, crushed to pass IS Sieve 12·5 mm (C) to be sent in duplicate.

4.2.2 Where air-drying has been adopted in the preparation of the samples, the percentage loss of moisture in this operation shall be recorded on the label together with the method of sampling used.

4.2.3 Samples received in the laboratory, if already ground to pass the 212-micron IS Sieve, shall be re-sieved to verify that all the material passes through this sieve, and then air-dried for 24 hours and mixed and bottled as above. Should a sample of coal or coke be transported to any distance between the point of preparation and the laboratory under conditions in which it may be shaken down and may cause segregation to

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

†Methods for sampling of coal and coke: Part I Sampling of coal (*revised*).

‡Methods for sampling of coal and coke: Part II Sampling of coke (*revised*).

any degree, the entire contents of the bottle shall be re-mixed before analytical work is started. The use of mechanical devices is recommended.

4.2.4 Certain coals may be found to give irregular results on analysis. In such cases, and for analytical work only, the analysis sample should be further ground to pass 125-micron IS Sieve and re-mixed. This final grinding will normally be found to ensure reasonable agreement between repeat determinations. The normal method of mixing coal in the laboratory is to roll it on a sheet of rubber cloth and turn it over at intervals with a large spatula or flat spoon. This method is not entirely satisfactory, since the movement of the coal tends to cause segregation. The difficulty may be overcome in part by spreading the mixed sample in a thin layer and filling the analysis bottle by means of small increments taken with the spoon from places spaced uniformly over the surface of the layer. This method of filling is particularly necessary when more than one bottle is being filled. A laboratory mechanical mixer is, however, preferable.

5. DETERMINATION OF SULPHUR

5.0 General — Total sulphur in coal and coke is determined gravimetrically by the Eschka method given under **5.1**. The forms in which sulphur is present are determined by the method given under **5.2**.

5.1 Determination of Total Sulphur

5.1.1 *Outline of the Method* — The sample of coal is heated in intimate contact with Eschka's mixture in an oxidizing atmosphere to remove combustible matter and to convert the sulphur to sulphate. This is then extracted and determined by gravimetric method by precipitation as barium sulphate with barium chloride.

5.1.1.1 A separate determination of moisture in the air-dried material should be done at the same time as sulphur determination.

5.1.2 *Quantity of Sample for Analysis* — The weight of sample required for the determination of total sulphur varies according to the expected sulphur content of the material as given below:

Expected Sulphur Content, Percent by Weight	*Quantity of Sample* g
0·1 and up to and including 5	1
Above 5 and up to and including 10	0·5
Above 10 and up to and including 20	0·25
Above 20	0·1

5.1.3 *Apparatus*

5.1.3.1 *Crucible or capsule* — of porcelain or platinum, 50 ml capacity.

5.1.3.2 *Silica plate* — flat, 6 mm thick of silica or other suitable insulation material which can be inserted into and fitted easily in the muffle furnace.

5.1.3.3 *Muffle furnace* — an electrically heated muffle furnace capable of holding about six capsules in a substantially uniform hot zone at 800 ± 25°C with an air change of about five times per minute.

NOTE — A gas-heated furnace may be used if appropriate precautions are taken.

5.1.3.4 *Air-oven* — capable of being maintained at 130 ± 10°C.

5.1.4 *Reagents*

5.1.4.1 *Eschka's mixture* — Mix two parts by weight of light, calcined magnesia with one part of anhydrous sodium carbonate. The mixture should entirely pass through 212-micron IS Sieve and its bulk density, shall be not greater than 0·50 g/ml. To determine the bulk density, place 50 g of the mixture in a 250-ml graduated cylinder, hold the cylinder in a vertical position and tap it gently 100 times on a bench or table top. Measure the volume occupied by the mixture.

NOTE — If light calcined magnesia is not available, pure calcium oxide may be used.

5.1.4.2 *Concentrated hydrochloric acid* — *see* IS: 265 - 1962*.

5.1.4.3 *Potassium sulphate solution* — Dissolve 0·006 0 g of potassium sulphate in water and dilute to one litre. Ten millilitres of this solution are equivalent to 0·008 0 g of barium sulphate or 1·033 ml of 0·1 N sodium thiosulphate solution.

5.1.4.4 *Barium chloride solution* — Dissolve 100 g of pure crystalline barium chloride ($BaCl_2 . 2H_2O$) in water and dilute to one litre.

5.1.4.5 *Methyl red indicator* — Dissolve 0·1 g of methyl red in 60 ml of rectified spirit or methylated spirit and dilute to 100 ml with water.

5.1.4.6 *Ammonia solution* — specific gravity 0·90.

5.1.4.7 *Hydrogen peroxide* — 30 percent (*w/v*) or 100 volume.

5.1.5 *Procedure*

5.1.5.1 *Preparation of solution* — Cover the bottom of the 50 ml crucible uniformly with 0·5 g of Eschka's mixture. Weigh accurately the appropriate quantity of the material (*see* 5.1.2) crushed to pass 212-micron IS Sieve and mix it intimately with 2·5 g of Eschka's mixture in an evaporating basin or other suitable vessel and brush into the crucible. Level the

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*Specification for hydrochloric acid (*revised*).

contents by tapping gently on the bench and cover uniformly with one gram of Eschka mixture. Place the charged crucible into the cold muffle furnace. Raise the temperature to 800 ± 25°C in about one hour and then heat for a further 90 minutes. Alternatively, place the charged crucible on the silica plate and place in the hot muffle furnace and keep for 90 minutes. Withdraw the plate with its crucible and cool.

NOTE — Weigh out the total of 4 g of Eschka's mixture and remove from this the 0·5 g and 1 g portions required for the bottom and top layers. For this purpose, calibrate a small glass tube, for each batch of Eschka's mixture, to deliver 0·5 and 1 g. The layer of Eschka mixture below the coal mixture reduces attack of the porcelain surface so that extraction of sulphate with hot water is complete, even when the surface deteriorates. Cracking of porcelain is prevented if they are slowly cooled on removal from the muffle by insertion in supports of light porous firebricks.

Transfer the ignited mixture as completely as possible from the crucible to a beaker containing 25 to 30 ml of water. If unburnt particles are present, reject the determination. Wash out the crucible thoroughly with about 50 ml of hot distilled water and add the washings to the contents of the beaker.

5.1.5.2 *Extraction by acid* — Place a cover-glass on the beaker, and then carefully add sufficient concentrated hydrochloric acid (17 ml will normally be required) to dissolve the solid matter, warming the contents of the beaker to effect solution. Boil for 5 minutes to expel carbon dioxide and filter through a filter pad or medium textured double acid-washed filter paper, collecting the filtrate in a 400-ml conical beaker. Wash the residue on the filter pad or filter paper with four 20-ml portions of hot distilled water. To the combined filtrate and washings, add 2 to 3 drops of methyl red indicator and then add cautiously ammonium hydroxide solution until the colour of the indicator changes and a trace of precipitate is formed. Add sufficient amount of concentrated hydrochloric acid to just redissolve the precipitate and then add one millilitre in excess.

5.1.5.3 *Precipitation of barium sulphate* — If necessary, make up the volume of the solution to approximately 300 ml with water. Cover the beaker and heat until the solution boils and then reduce the heating slightly until ebullition ceases. Then add within approximately 20 seconds, 10 ml of barium chloride solution from a pipette held such that the barium chloride falls into the middle of the hot solution which is being agitated. Keep the solution just below boiling point, for 30 minutes (*see* **5.1.5.6**).

5.1.5.4 Filter, using one of the following techniques:

i) By gravity, through an ashless fine-textured double acid-washed filter paper in a fluted, long-stemmed 60° funnel (Whatman No. 42 filter paper is suitable for this purpose).

or

ii) Under suction, using good quality asbestos in a Gooch crucible which has been dried for one hour at 130 ± 10°C and weighed.

Wash the filter with hot distilled water, using not more than 250 ml, until 20 ml of the washings give not more than a faint opalescence with silver nitrate solution.

5.1.5.5 *Ignition* — Place the wet filter paper in an ignited and weighed silica, porcelain or platinum capsule on a silica plate and insert into the muffle furnace at room temperature and raise the temperature to 800 ± 25°C. Keep the crucible at this temperature for 15 minutes. If a Gooch crucible is used, dry it for one hour at 130 ± 10°C. Cool and weigh.

5.1.5.6 It has been shown that with the excess of barium chloride used and under the specified conditions of precipitation, complete recovery of the barium sulphate can be made by filtering on the fine filter paper after 30 minutes. It is not necessary to stand for two hours or overnight. When using one gram of coal ten millilitres of 10 percent barium chloride solution give sufficient excess of barium ions even for coal containing up to 8 percent of sulphur.

5.1.5.7 Carry out a blank determination under the same conditions, but omitting the material. Pipette ten millilitres of the potassium sulphate solution to the filtrate after extraction. Deduct the weight of the barium sulphate found in the blank determination less the barium sulphate equivalent of the potassium sulphate added, from the weight obtained in the test determination.

5.1.6 Calculation

$$\text{Sulphur, percent by weight} = \frac{13{\cdot}74\,(A - B + 0{\cdot}008\,0)}{W}$$

where

A = weight in grams of barium sulphate found in the determination,

B = weight in grams of barium sulphate found in the blank determination, and

W = weight in grams of the material taken for the test.

5.1.7 *Precision* — The results of duplicate determinations should agree to the following limits:

Sulphur Content, Percent by Weight	*Precision*	
	Within Laboratory	Between Laboratories
Up to 2	± 0·02	± 0·05
2 to 10	± 0·04	± 0·05

5.2 Determination of Forms of Sulphur

5.2.1 *Method* — Sulphur is usually present in coal in three forms — as sulphates, as iron pyrites (FeS_2), and as sulphur in organic compounds. The amounts of sulphur so present are known as 'sulphate', 'pyritic' and 'organic' respectively. 'Sulphate' sulphur is determined by extracting coal with dilute hydrochloric acid and determining the sulphur in the extract. 'Pyritic' sulphur is insoluble in dilute hydrochloric acid, but it is quantitatively dissolved by dilute nitric acid under the experimental conditions described. It is conveniently determined by an indirect method, that is, by determining the amount of iron combined in the pyritic state and calculating the amount of sulphur associated with this iron. 'Organic' sulphur is calculated by deducting the sum of the percentages of 'sulphate' and 'pyritic' sulphur from the total sulphur in the coal as determined by the Eschka method (*see* **5.1**).

5.2.2 *Apparatus*

5.2.2.1 *Muffle furnace* — electrically heated, capable of holding about six capsules in a substantially uniform hot zone at 800 ± 25°C, and a silica plate 6 mm thick which can fit easily in the muffle.

5.2.2.2 *Air-oven* — capable of being maintained at 130 ± 10°C.

5.2.2.3 *Cold finger condenser* — of the shape and dimensions shown in Fig. 1.

5.2.3 *Reagents*

5.2.3.1 *Concentrated hydrochloric acid* — specific gravity 1·16 (*see* IS : 265-1962*).

5.2.3.2 *Dilute Hydrochloric acid* — approximately 5 N.

5.2.3.3 *Dilute hydrochloric acid* — approximately 0·5 N.

5.2.3.4 *Bromine water* — saturated solution.

5.2.3.5 *Ammonium hydroxide solution* — specific gravity 0·90.

5.2.3.6 *Methyl orange indicator* — Dissolve 0·5 g of methyl orange in 000 ml of water, and filter the cold solution if a precipitate separates.

5.2.3.7 *Barium chloride solution* — 10 percent. Dissolve 100 g of barium hloride dihydrate ($BaCl_2 . 2H_2O$) in water and make up the volume to 000 ml. Filter before use through a close-textured double acid-washed lter paper (Whatman No. 42) or a filter-paper pad.

5.2.3.8 *Dilute nitric acid* — approximately 2 N.

5.2.3.9 *Standard sulphate solution* — Dissolve 0·600 g of potassium ulphate in water and make up the volume to 1 000 ml. Ten millilitres of nis solution are equal to 0·022 2 percent sulphur on 5 g of coal.

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*Specification for hydrochloric acid (*revised*).

5.2.3.10 *Hydrogen peroxide solution* — 30 percent (*w/v*) or 100 volume solution.

5.2.3.11 *Stannous chloride solution* — Dissolve 5 g of stannous chloride ($SnCl_2$) in 50 ml of concentrated hydrochloric acid, add 50 ml of water and some metallic tin.

5.2.3.12 *Mercuric chloride solution* — Add 6 g of mercuric chloride to 100 ml of water and shake for 10 minutes.

5.2.3.13 *Sulphuric-phosphoric acid mixture* — Carefully add 15 ml of concentrated sulphuric acid, sp gr 1·84 (*see* IS : 266 - 1961*), to 50 ml of water followed by 15 ml of phosphoric acid, sp gr 1·75, and then dilute to 100 ml.

5.2.3.14 *Sodium diphenylamine sulphonate indicator* — 0·2 percent solution in water.

5.2.3.15 *Standard potassium dichromate solution* — 0·0179 N (containing 0·8772 g of $K_2Cr_2O_7$ per litre).

5.2.4 *Procedure*

5.2.4.1 '*Sulphate*' *sulphur* — Before commencing the determination, mix thoroughly the sample, ground to pass 212-micron IS Sieve, preferably with a laboratory mechanical mixer. Accurately weigh about 5 g of the material and boil with 50 ml of dilute hydrochloric acid for 30 minutes in a 250-ml conical flask, into the neck of which is fitted a cold finger condenser. Filter the mixture through a medium textured double acid-washed filter paper (Whatman No. 41), wash six times with dilute hydrochloric acid using about 20 ml of acid in all. Discard the residue. Add one millilitre of bromine water to the filtrate, boil for 10 minutes to ensure that all iron is in the ferric state and that the bromine is boiled off. Precipitate the iron with ammonium hydroxide solution in a slow stream until a slight excess of ammonium hydroxide is present and then add 5 ml in excess. Filter on a fast toughened filter paper into a 250-ml beaker. Wash with hot water and preserve this precipitate for the determination of non-pyritic iron. To determine the 'sulphate' sulphur in the filtrate, add 2 to 3 drops of the methyl orange indicator and then add cautiously concentrated hydrochloric acid until the colour of the solution changes, and then add one millilitre of acid in excess. Cover the beaker and heat until the solution boils and then reduce the heating slightly until ebullition ceases. Add 10 ml of cold barium chloride solution from a pipette, with a delivery time of approximately 20 seconds, so that the barium chloride falls into the centre of the hot solution which is being agitated. Keep the solution just below boiling point for 30 minutes.

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*Specification for sulphuric acid (*revised*).

Filter using any of the following techniques:

i) By gravity, through an ashless fine-textured double acid-washed filter paper (Whatman No. 42) in a fluted long stemmed 60° funnel.

or

ii) Under suction, using asbestos in a Gooch crucible which has been previously dried for one hour at 130 ± 10°C and weighed.

Wash the filter with hot distilled water, using not more than 250 ml, until 20 ml of the washings give not more than a faint opalescence with silver nitrate solution. Place the wet filter paper in an ignited and weighed silica or platium capsule on the silica plate and insert into the muffle furnace for 15 minutes at 800 ± 25° C. If a Gooch crucible is used, dry it for one hour at 130 ± 10°C. Cool and weigh.

NOTE — Carry out a blank determination under the same conditions but omitting the material. Pipette 10 ml of the standard sulphate solution to the filtrate before precipitation of barium sulphate. The weight of the barium sulphate found in the blank determination, less the equivalent of the standard solution, is deducted from that obtained in the full determination.

Redissolve the iron precipitate obtained at the beginning of the test and determine the non-pyritic iron present in the sample by the method described for the determination of total iron under **5.2.4.2**.

5.2.4.2 *'Pyritic' sulphur* — The method involves the determination of total iron, 'pyritic' and 'non-pyritic', and the separate determination of non-pyritic iron. Weigh accurately about one gram of the material, ground to pass 212-micron IS Sieve. Add 50 ml of dilute nitric acid in an apparatus similar to that used under **5.2.4.1** and boil for 30 minutes. Filter through a medium-textured double acid-washed filter paper and wash six times with dilute nitric acid. Add to the filtrate two millilitres of hydrogen peroxide and boil for five minutes to destory any colouration arising from the decomposition of coal. Heat the filtrate to boil precipitate iron with ammonium hydroxide solution and filter (*see* **5.2.4.1**). Wash the precipitate with hot water, pierce the filter paper and wash with a fine jet of hot water into a 500-ml conical flask or beaker. Pour 10 ml of dilute hydrochloric acid on the filter in small portions to remove the last traces of iron, and give a final wash with hot water. If necessary, warm the solution to dissolve the iron hydroxide, the total bulk; preferably, not exceeding 20 ml. Heat to boil and add stannous chloride solution, drop by drop, from a pipette or dropping bottle, stirring until the yellow colour disappears. Add 5 drops in excess, cool rapidly to room temperature and immediately add 10 ml of mercuric chloride solution when a silky looking precipitate of mercurous chloride is formed. Add 15 ml of sulphuric-phosphoric acid mixture, dilute to 150 to 200 ml with water, add 5 drops of sodium diphenylamine sulphonate indicator and titrate with standard potassium dichromate solution. Near the end point the colour of the solution deepens to blue-green or, in the presence of a large amount of iron to greenish blue. Add the dichromate, drop by drop, until the colour changes to an intense violet blue. The volume in millilitres of potassium dichromate solution used is a measure of the percentage of total iron content of the sample.

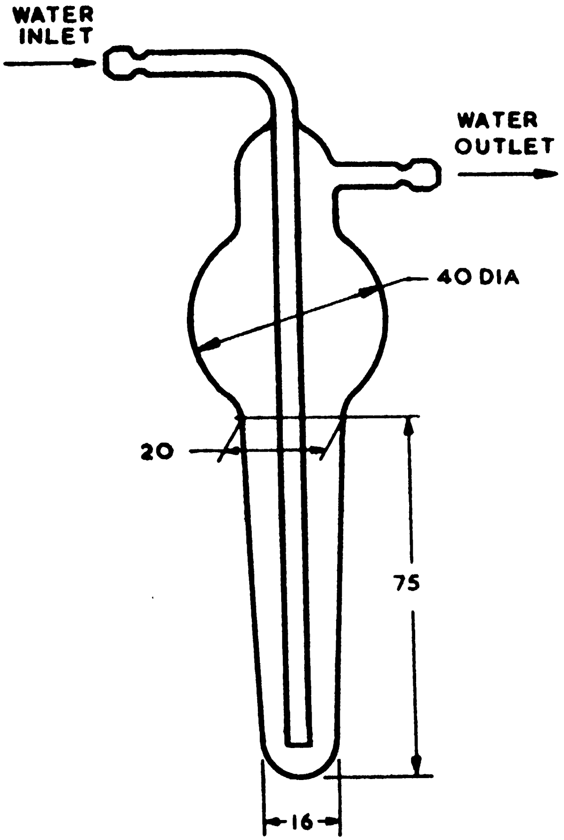

Fig. 1 Cold Finger Condenser

5.2.5 *Calculation and Reporting of Results*

a) 'Sulphate' sulphur, percent by weight $= \dfrac{13{\cdot}74\,(A - B)}{W}$

where

A = weight in g of barium sulphate found in the determination;

B = weigh in g of barium sulphate found in the blank determination, less that due to addition of standard sulphate; and

W = weight in g of material taken for the test under **5.2.4.1**.

b) 'Pyritic' sulphur, percent by weight $= \dfrac{U - V}{X - W}\ 0{\cdot}115$

where

U = volume in ml of standard potassium dichromate solution equivalent to the total iron (*see* **5.2.4.2**),

V = volume in ml of standard potassium dichromate solution equivalent to the non-pyritic iron (*see* **5.2.4.1**),

X = weight in g of the material taken for the test under **5.2.4.2**, and

W = weight in g of the material taken for the test under **5.2.4.1**.

c) *'Organic' sulphur* — Deduct the sum of the percentages of 'sulphate' and 'pyritic' sulphurs [*see* **5.2.5** (a) and (b)] from the percentage of total sulphur in the coal obtained in a separate determination by the Eschka method (*see* **5.1**) and report as 'organic' sulphur.

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