



SLOVENSKI STANDARD
SIST ISO 157:1998

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Coal -- Determination of forms of sulfur

Charbon -- Détermination de la teneur en différentes formes de soufre

Ta slovenski standard je istoveten z: **ISO 157:1996**

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ICS:

73.040 Premogi Coals

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INTERNATIONAL
STANDARD

ISO
157

Second edition
1996-06-01

Coal — Determination of forms of sulfur

Charbon — Détermination de la teneur en différentes formes de soufre

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Reference number
ISO 157:1996(E)

ISO 157:1996(E)**Foreword**

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 157 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 157:1975), of which it constitutes a technical revision.

Annex A of this International Standard is for information only.

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Introduction

For many purposes, a knowledge of the total sulfur content of a coal is sufficient, but for certain work it is also necessary to know how the sulfur is distributed between the coal substance and the mineral matter. In particular, such information may be required in connection with coal classification and cleaning.

Sulfur is usually present in coal in three forms, viz:

- a) sulfate sulfur, i.e. the sulfur present in the form of inorganic sulfates;
- b) pyritic sulfur, i.e. the sulfur present in the form of pyrites and marcasite;
- c) organic sulfur, i.e. the sulfur present in the form of organic sulfur compounds.

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Coal — Determination of forms of sulfur

Section 1: General

1.1 Scope

This International Standard specifies methods of determining the sulfate and pyritic sulfur contents of coals, including brown coals and lignites, and of calculating the amount of organic sulfur present.

ISO 5069-2:1983, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis.*

1.2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 331:1983, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method.*

ISO 334:1993, *Solid mineral fuels — Determination of total sulfur — Eschka method.*

ISO 351:1995, *Solid mineral fuels — Determination of total sulfur — High temperature combustion method.*

ISO 1015:1992, *Brown coals and lignites — Determination of moisture content — Direct volumetric method.*

ISO 1170:1977, *Coal and coke — Calculation of analyses to different bases.*

ISO 1988:1975, *Hard coal — Sampling.*

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method.*

1.3 Principle

The procedure described in this International Standard utilizes the differential solubilities of sulfates and pyrites in dilute hydrochloric and nitric acids under reflux conditions, such that each can be taken in solution successively and determined directly.

A general schematic representation of the procedure is given in figure 1.

1.4 Preparation of the test sample

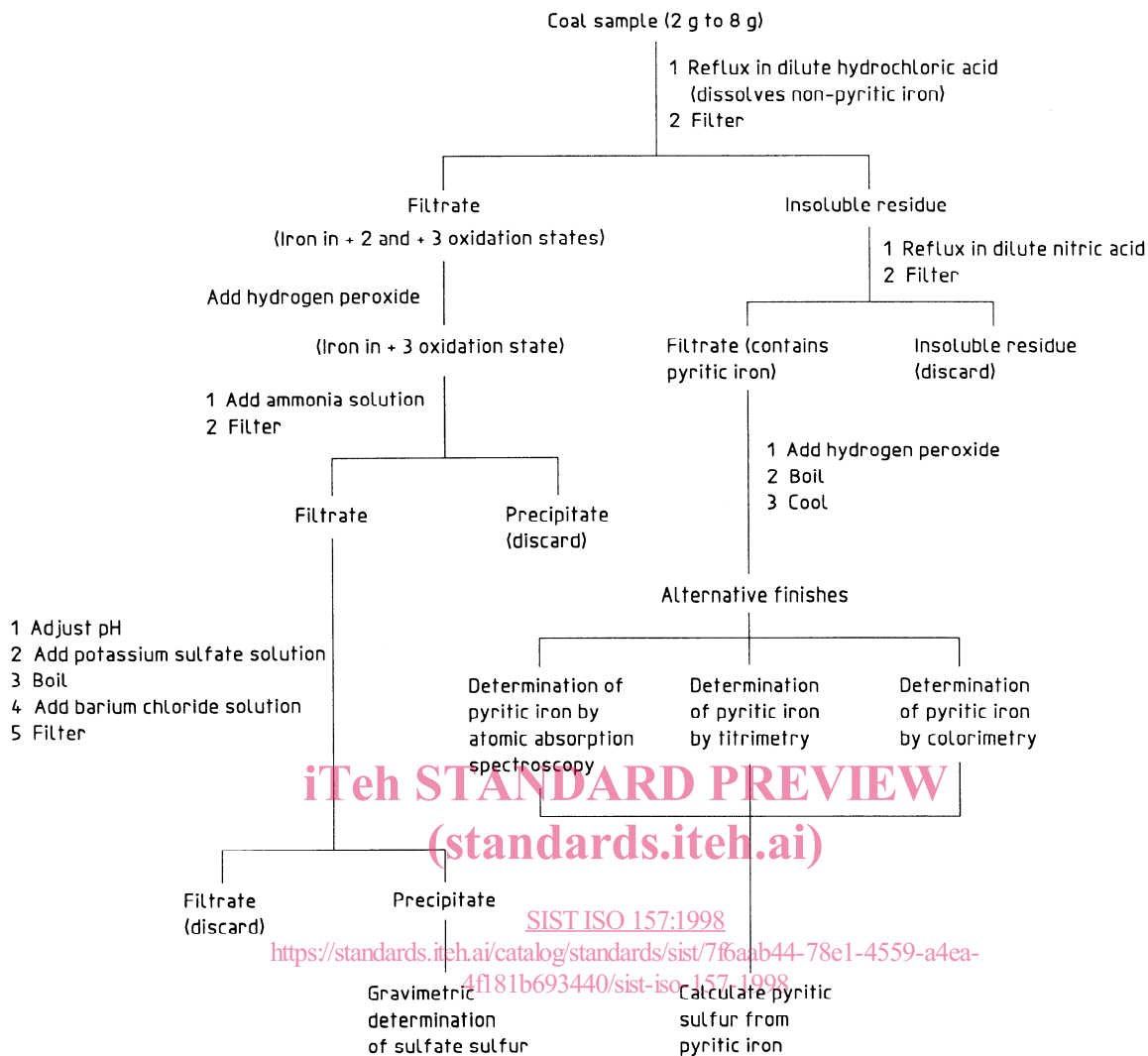
The test sample is the general analysis test sample prepared in accordance with ISO 1988 or ISO 5069-2, as appropriate.

Grind about 25 g of the sample to pass a sieve of aperture 75 μm and ensure that its moisture content is in equilibrium with the laboratory atmosphere, exposing it if necessary in a thin layer for the minimum time required to achieve equilibrium.

Before commencing the determination, thoroughly mix the test sample for at least 1 min, preferably by mechanical means.

If the results are to be calculated other than on an air-dried basis (see 6.1) then, after weighing the test portion (see 2.4), determine the moisture content using a further portion of the test sample by the method described in ISO 331, ISO 1015 or ISO 5068, as appropriate.

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NOTE — Organic sulfur = Total sulfur – (sulfate sulfur + pyritic sulfur)

Figure 1 — General schematic representation of the procedure

Section 2: Separation of sulfate and pyritic sulfur

2.1 Principle

The test portion is extracted with dilute hydrochloric acid to bring both the sulfate sulfur and the non-pyritic iron into solution. The pyritic iron and sulfur remain in the residue which is separated by filtration.

2.2 Reagents

WARNING — Care must be exercised when handling the reagents, many of which are toxic and corrosive.

During the analysis, unless otherwise stated, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent quality.

2.2.1 Hydrochloric acid, concentrated, approximately 36 % (m/m).

2.2.2 Hydrochloric acid, dilute, approximately 15 % (m/m).

Dilute 420 ml of the hydrochloric acid (2.2.1) to 1 litre with water.

2.2.3 Nitric acid, dilute, approximately 9 % (m/m).

Dilute 130 ml of concentrated nitric acid [approximately 70 % (m/m)] to 1 litre with water.

2.3 Apparatus

2.3.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

2.3.2 Graduated glassware, conforming to the requirements for class A in the International Standards prepared by ISO/TC 48, *Laboratory glassware and related apparatus*.

2.3.3 Cold-finger condenser, to fit loosely into the neck of a 250 ml conical flask (see figure 2).

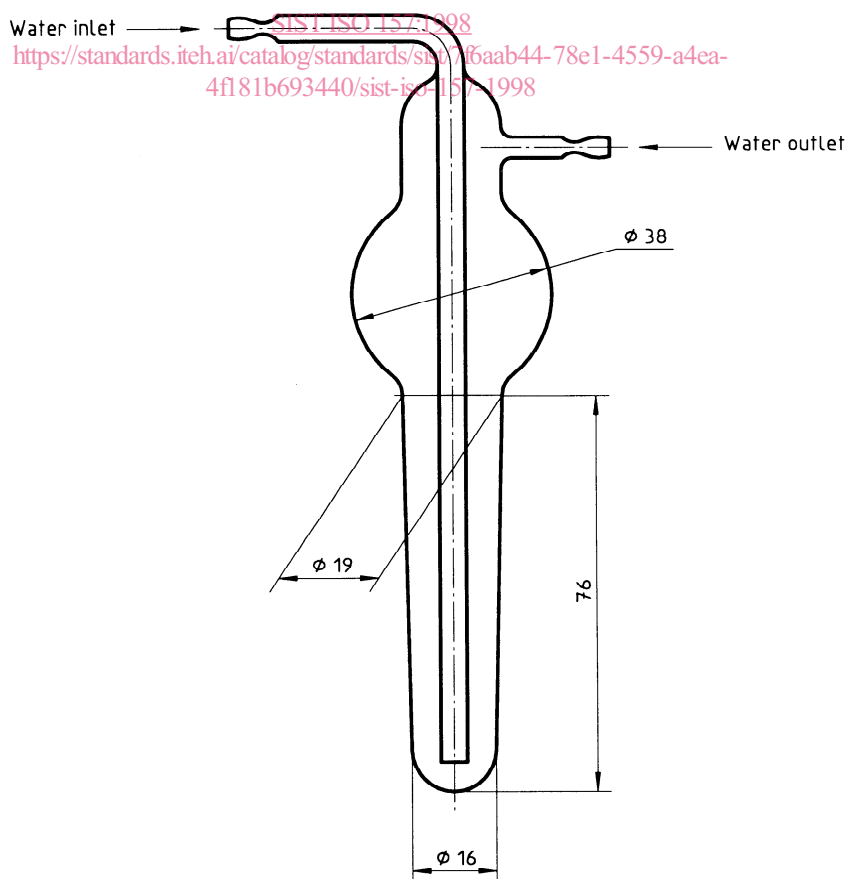


Figure 2 — Cold-finger condenser for the determination of sulfate and pyritic sulfur

2.4 Procedure

2.4.1 Test portion

Take a test portion of the mass given in table 1 for the expected total sulfur content, weighing to the nearest 0,1 mg.

Table 1 — Test portion

Total sulfur content % (m/m)	Mass of test portion g
< 0,7	8
0,7 to 2,0	5
> 2,0	2

2.4.2 Extraction

Transfer the test portion to a 250 ml conical flask, add 50 ml of the dilute hydrochloric acid (2.2.2) and fit the cold-finger condenser (2.3.3) into the neck of the flask. Boil for 30 min, ensuring that a slow stream of water is passing through the cold finger. Remove the condenser and thoroughly rinse back into the flask. Filter the mixture through a medium-textured, doubly acid-washed filter paper into a tall-form beaker. Wash the residue three times with the dilute hydrochloric acid (2.2.2) and a further three times with hot distilled water, using a total volume of approximately 30 ml. Retain the filtrate for the determination of sulfate sulfur (see Section 3). Immediately transfer the washed, undissolved residue and filter paper to a 250 ml conical flask, add 50 ml of the dilute nitric acid (2.2.3) and retain for the determination of pyritic sulfur (see Section 4).

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Section 3: Determination of sulfate sulfur

3.1 Principle

The sulfate sulfur extracted by dilute hydrochloric acid from the test portion is determined gravimetrically by precipitation with barium chloride.

3.2 Reagents

WARNING — Care must be exercised when handling the reagents, many of which are toxic and corrosive.

During the analysis, unless otherwise stated, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

3.2.1 Hydrogen peroxide, solution, approximately 30 % (*m/m*).

3.2.2 Methyl red, indicator solution.

Dissolve 1 g of 2-(4-dimethylaminophenylazo)benzoic acid, sodium salt (methyl red) in 1 litre of water.

3.2.3 Ammonia, concentrated, solution not less than 25 % (*m/m*).

3.2.4 Hydrochloric acid, concentrated, approximately 36 % (*m/m*).

3.2.5 Potassium sulfate, approximately 2 g/l solution.

Dissolve approximately 2 g of potassium sulfate in water and dilute to 1 litre.

3.2.6 Barium chloride, approximately 85 g/l solution.

Dissolve 100 g of barium chloride dihydrate in water and dilute to 1 litre. Filter before use through a close-textured, doubly acid-washed filter-paper or filter-pad.

3.2.7 Silver nitrate, approximately 17 g/l solution.

Dissolve 17 g of silver nitrate in water and dilute to 1 litre. Store in a dark glass bottle.

3.3 Apparatus

3.3.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

3.3.2 Graduated glassware, conforming to the requirements for class A in the International Standards prepared by ISO/TC 48.

3.3.3 Electrically-heated muffle furnace, capable of being maintained at $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$, with an adequate ventilation rate.

3.3.4 Crucible, of platinum, silica or glazed porcelain, of capacity approximately 25 ml.

3.3.5 Flat plate, 6 mm thick, of silica or other suitable refractory material, which fits easily into the muffle furnace (3.3.3).

3.3.6 Gooch crucible, of glazed porcelain or sintered glass.

3.3.7 Air oven, capable of being maintained at a temperature of $130\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$.

3.3.8 Filter cone, made of porcelain, of diameter 25 mm.

3.4 Procedure

To the filtrate from the hydrochloric acid extraction (see 2.4.2), add 5 ml of the hydrogen peroxide solution (3.2.1) and boil for 5 min to convert all the soluble iron to the + 3 oxidation state. While still hot, add 2 or 3 drops of the methyl red indicator solution (3.2.2) followed by the ammonia solution (3.2.3), drop by drop, until the solution is just alkaline (yellow colour) and then add a further 5 drops in excess. Filter the resultant precipitate on a toughened fast filter paper into a 250 ml beaker. Wash thoroughly with hot water, discarding the precipitate. Cautiously add the concentrated hydrochloric acid (3.2.4) to the filtrate, drop by drop, until the colour of the solution changes to pink and then add a further 1 ml in excess. The volume of the solution shall be between 150 ml and 250 ml.

Add 25,0 ml of the potassium sulfate solution (3.2.5) from a one-mark pipette. Cover the beaker with a watch glass, heat the covered beaker until the solution boils and then reduce the heating slightly until ebullition ceases. Add 10 ml of the cold barium chloride solution (3.2.6) from a pipette with a delivery time of approximately 20 s, held so that the barium chloride falls into the centre of the hot solution, whilst this is being agitated. Keep the solution just below boiling point, without agitation, for 30 min.

Filter the solution using one of the following techniques.

- By gravity through an ashless, close-textured, doubly acid-washed filter paper of diameter 100 mm to 125 mm. Carefully fold the filter paper and fit it into a fluted, long-stemmed 60° funnel, so that the stem remains full of liquid during the filtration.