TECHNICAL SPECIFICATION

ISO/TS 11725

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Solid mineral fuels — Determination of nitrogen — Semi-micro gasification method

Combustibles minéraux solides — Détermination de la teneur en azote — Méthode semi-micrométrique par gazéification

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. 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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years with a view to deciding whether it should be confirmed for a further three years, revised to become an International Standard, or withdrawn. In the case of a confirmed ISO/PAS or ISO/TS, it is reviewed again after six years at which time it has to be either transposed into an International Standard or withdrawn.

Attention is drawn to the possibility that some of the elements of this Technical Specification may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 11725 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

Annex A of this Technical Specification is for information only.

Introduction

A method for the determination of nitrogen in coals, ISO 333, has been in use for many years, but experience has shown that it is unsuitable for cokes and some high carbon content coals and chars which require long reaction times with the potential loss of nitrogen from the system before completion of the test. The method described in this Technical Specification, based on JIS M 8813, addresses that shortcoming and is applicable to all solid fuels.

It has been prepared as a Technical Specification since there is at present little experience with the method outside Japan, where it was developed and tested.

Solid mineral fuels — Determination of nitrogen — Semi-micro gasification method

1 Scope

This Technical Specification specifies a method of determining the nitrogen content of hard coals, brown coals and lignites, cokes and chars by a semi-micro gasification method.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this Technical Specification. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this Technical Specification are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 687, *Coke — Determination of moisture in the general analysis test sample*

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

ISO 1988, *Hard coal — Sampling*

ISO 2309, *Coke — Sampling*

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

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

ISO 9411-1, *Solid mineral fuels — Mechanical sampling from moving streams — Part 1: Coal*

ISO 9411-2, *Solid mineral fuels — Mechanical sampling from moving streams — Part 2: Coke*

3 Principle

A known mass of the sample is mixed with a flux and pyrolyzed at temperatures up to 1 000 °C in a silica tube through which steam is passing. Ammonia, which is formed from the nitrogen present, is absorbed in boric acid solution and determined by titration with sulfuric acid.

4 Reagents

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

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

4.1 Boric acid solution, saturated

Dissolve 60 g of boric acid in 1 l of hot water, cool and allow to stand for three days before decanting the clear solution.

4.2 Sodium hydroxide, 250 g/l solution

Dissolve 250 g of sodium hydroxide in water and dilute to 1 l; mix thoroughly.

4.3 Ammonia, approximately 0,17 g/l solution

Dissolve 0,535 g of ammonium chloride in 30 ml of water. Transfer the solution to a steam distillation apparatus as described in 5.4. Transfer 4 ml of the boric acid (4.1) to the receiver. Add 150 ml of the sodium hydroxide solution (4.2) to the distillation flask and pass steam through the apparatus whilst maintaining the temperature at approximately 125 °C.

When approximately 250 ml of distillate have been collected, cease the distillation. Dilute the distillate to 1 l with water and mix well.

4.4 Helium, purity > 99,8 %

4.5 Soda-lime, powdered

Granular soda-lime (NaOH solution absorbed on CaO) may be crushed using a porcelain pestle and mortar.

4.6 Activated alumina, neutral, pore size approximately 9 nm

4.7 Silica fibre, diameter 1 µm to 5 µm

4.8 Sulfuric acid, standard volumetric solution, concentration $c(H_2SO_4) = 0{,}005$ mol/l

4.9 Mixed indicator solution

— solution A — dissolve 0,125 g of 2-(4-dimethylaminophenylazo) benzoic acid, sodium salt (methyl red), in 100 ml of water.

— solution B — dissolve 0,083 g of 3,7-bis(dimethylamino)phenothiazine-5-ylium chloride (methylene blue), in 100 ml of water. Store in a dark glass bottle.

Mix equal volumes of solutions A and B. Store in a dark glass bottle. Discard the mixed solution after 1 week.

4.10 Graphite, powdered, spectroscopy electrode purity

5 Apparatus

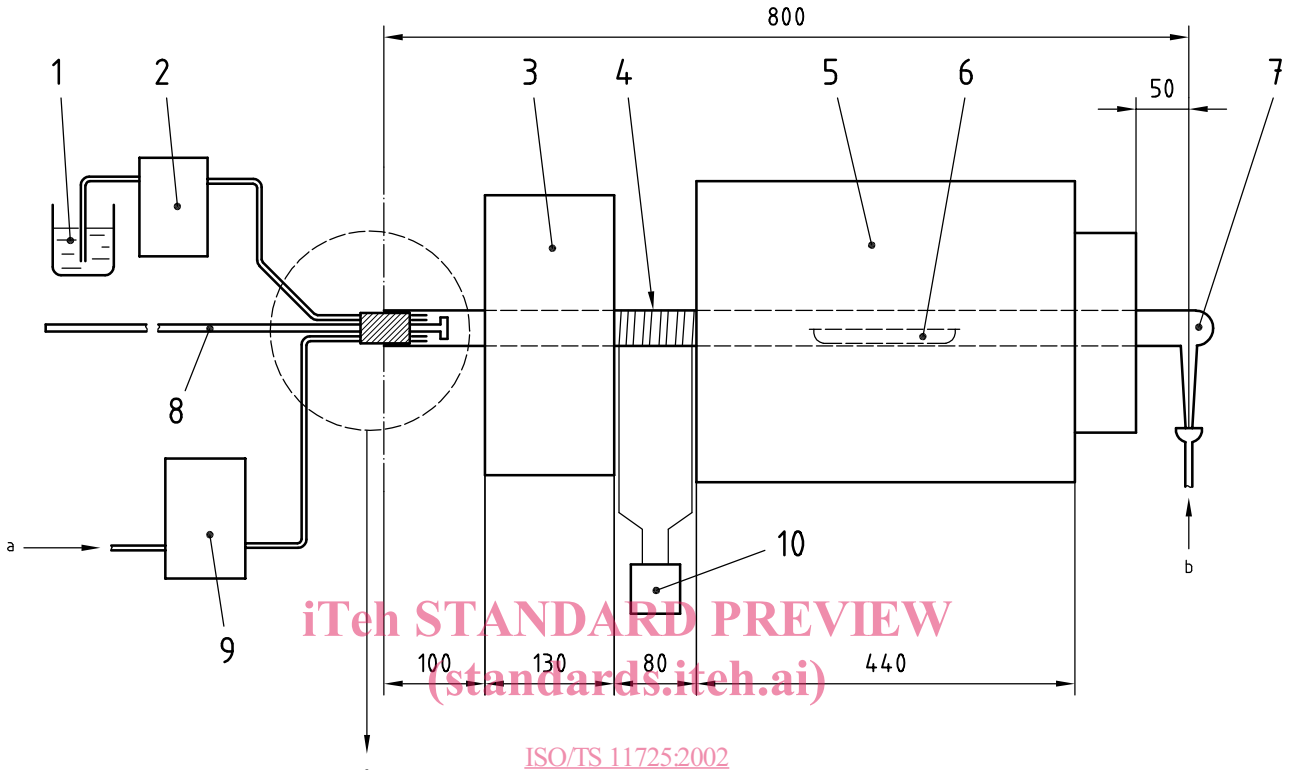
5.1 Analytical balance, capable of weighing to 0,1 mg

5.2 Graduated glassware, conforming to the requirements for Grade “A” in the International Standards prepared by ISO/TC 48, “Laboratory glassware and related apparatus”

5.3 Gasification apparatus

See Figure 1.

Dimensions in millimetres



Key

1	Water	6	Boat
2	Peristaltic pump	7	Pyrolysis tube
3	Steam-raising surface	8	Pusher rod
4	Pre-heater	9	Helium flow controller
5	Main furnace	10	Voltage regulator

a Helium in

b Entry to distillation flask (see Figure 3)

c See Figure 2

Figure 1 — Gasification apparatus

5.3.1 Steam-raising furnace, of sufficient capacity to maintain a portion of the pyrolysis tube (5.3.4) at about 450 °C.

5.3.2 Pre-heater, formed by wrapping insulated heating tape, of heat capacity 800 W at 100 V, supplied through a variable voltage regulator, around the pyrolysis tube. The tape shall completely cover the length of tube between the steam-raising furnace (5.3.1) and the main furnace (5.3.3).