TECHNICAL SPECIFICATION

ISO/TS 4667

First edition
2022-08

Coal — Determination of the thermal stability and thermal fragmentation

Reference number
ISO/TS 4667:2022(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 27, *Coal and coke*, Subcommittee SC 5, *Methods of analysis*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Coal gasification is an important technology for clean coal conversion, which involves many factors. The thermal stability of coal is one of the essential parameters for the gasification industry and its relevant coal trade.

The thermal stability of coal is a measure of how much a coal retains its original size under high-temperature reaction during the process of gasification or high-temperature combustion. The coal with a better thermal stability can be gasified or burnt in its original size without being broken, or while being only a little bit broken, during the process of gasification or combustion, while the coal with a poor thermal stability will be broken into smaller piece or into powder. When using a coal with poor thermal stability for the gasification or combustion, the materials being taken from the furnace will increase, causing the resistance to increase, which in turn, affects the gasification or combustion and decreases the efficiency of the gasification. So, the thermal stability of coal is a very important property highly concerned by the gasification or combustion industry of utilizing the sized coal for gasification or combustion.

The opposite property of thermal stability of coal is the thermal fragmentation. Thermal fragmentation has a highly significant negative linear correlation with thermal stability.

This document is based on GB/T1573 and on an alternative laboratory method to determine thermal fragmentation of coal sources during pyrolysis in the gasification process given in Reference [3] including two parameters of thermal stability (TS_{+6}) and thermal fragmentation (TF), both of which can be used for guiding the coal gasification of fixed bed.

Coal — Determination of the thermal stability and thermal fragmentation

1 Scope

This document specifies methods for the determination of thermal stability and thermal fragmentation of coal. It is applicable to brown coal and lignites, anthracites and bituminous coal with a caking index of zero (determined by ISO 15585).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

ISO 18283, *Coal and coke — Manual sampling*

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
thermal stability
TS_{+6}
dimensional stability of a solid mineral fuel heated under specified conditions

[SOURCE: ISO 1213-2:2016, 3.227]

3.2
thermal fragmentation
TF
dimensional fragmentation of coal heated under specified conditions

3.3
Ergun index
Sauter mean diameter
SMD
diameter of a sphere that has the same volume/surface area ratio as a particle of interest

4 Principle

4.1 Thermal stability

The coal sample with a size fraction of 6 mm to 13 mm is heated out of contact with air at (850 ± 15) °C for 30 min. Determine the mass and sieve the residues. Take the percentage by mass of the residue over 6 mm to the sum of the masses of each size fraction of the residues as the thermal stability index, and take the percentages by mass of 3 mm to 6 mm and -3 mm respectively to the sum of the masses of each size fraction of the residues as the secondary indexes of the thermal stability.

4.2 Thermal fragmentation

The coal sample with a size of 6 mm to 13 mm is heated out of contact with air at (850 ± 15) °C for 30 min. Determine the mass and sieve the residues. Take the percentage by the Ergun index before testing minus Ergun index after testing to the Ergun index before testing as the thermal fragmentation.

5 Apparatus

5.1 Furnace, with thermocouple and pyrometer, heated by electricity, a zone of 100 mm × 230 mm maintained at a uniform temperature of (850 ± 15) °C. An exhaust hole and a small opening for inserting thermocouple are provide on the back wall of the furnace. The uniform temperature zone, thermocouple and pyrometer shall be checked on a yearly basis to ensure accuracy of measurement.

5.2 Vibrating sieving machine, reciprocating, amplitude (40 ± 2) mm, frequency (240 ± 20) min^{-1}.

5.3 Round hole sieves, in square shape, matching to vibrating sieving machine, 13 mm,10 mm, 8 mm, 6 mm, 3 mm and 1 mm apertures respectively, with a cover and bottom plate.

5.4 Top-loading balance, with a resolution of 0,01 g and maximum capacity of 1 kg.

5.5 Crucible and lid (see Figure 1), made of porcelain or corundum with volume of 100 cm^3, designed to not allow oxygen to access coal.

5.6 Crucible stand, made of the metal materials with heat-resistant of over 900 °C. The stand can hold 5 crucibles to 10 crucibles based on the size of the uniform temperature zone in furnace.

Dimensions in millimetres

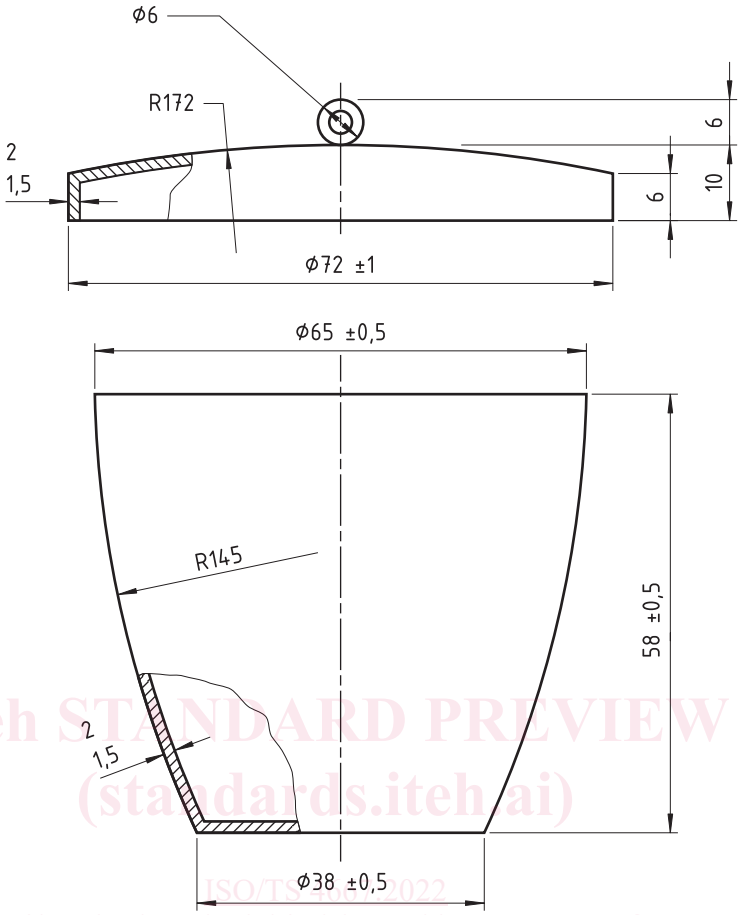

Figure 1 — Schematic of the crucible and lid

6 Preparation of test sample

The test sample, 2,0 kg with size of 6 mm to 13 mm, shall be prepared (stage crushed) in accordance with ISO 18283 or ISO 13909-4.

The sample shall be equilibrated with the laboratory atmosphere by exposure in a thin layer on a tray. Exposure time shall be kept to a minimum.

The test sample, with less than 6 mm removed, shall be thoroughly mixed before dividing into two portions.

7 Procedure

7.1 Thermal stability

7.1.1 Extract a subsample with a volume of 500 cm^3 from each of the two portions of the test sample (Clause 6) by using crucibles (5.5). Determine each subsample mass to the nearest 0,01 g and keep the mass difference between two subsamples to not more than 1 g, which is tested in different runs.

7.1.2 Distribute each subsample into 5 crucibles (5.5), each of which is about 100 cm^3 and cover them with crucible lids, then place them on the crucible stand (5.6).

7.1.3 Quickly insert the stand with samples into the furnace at 850 °C. Close the furnace door and heat for 30 min. The furnace temperature will drop when the samples are initially placed into the furnace, the furnace shall recover to (850 ± 15) °C within 8 min, then maintain a temperature of (850 ± 15) °C, otherwise discard this test. Heating time (30 min) includes temperature recovery time (8 min).

NOTE The initial temperature of 850 °C can be adjusted to guarantee that the furnace temperature of (850 ± 15) °C is regained within 8 min after insertion of the cold stand and its crucible(s).

7.1.4 Take out the stand with samples and allow them cool to room temperature. Determine the total mass of the residues to the nearest 0,01 g.

For those samples that are agglomerate after being heated, stop the experiment and illustrate it in the report.

7.1.5 Nest the sieves of 6 mm and 3 mm aperture and the bottom plate on the vibrating sieving machine (5.2). Transfer the residue of known mass to the 6 mm aperture sieve, and affix the cover.

7.1.6 Start the sieving machine and sieve for 10 min.

7.1.7 Determine the mass of the residue mass of the size fractions of +6 mm, 6 mm to 3 mm and -3 mm to the nearest 0,01 g.

7.1.8 The total mass of the residues before sieving and the sum of the mass of each size fraction of residue coke after sieving should not differ by more than 1 g, otherwise discard this test.

7.2 Thermal fragmentation

7.2.1 Extract a subsample with a volume of 500 cm^3 from each of the two portions of the test sample (Clause 6) by using crucibles (5.5). Determine each subsample mass to the nearest 0,01 g and keep the mass difference between two subsamples to not more than 1 g, which is tested in different runs.

7.2.2 Separate each subsample manually by using the sieves of 10 mm, 8 mm, 6 mm apertures (5.3). In the process of sieving, care shall be taken to prevent further breakage of the coal. The sieving shall be carried out in such small increments as to permit satisfactory contact between the individual pieces of coal and allow to pass without force. Determine the mass of the coal remaining on each sieve and record the mass of each specific particle size distribution of 13 mm to 10 mm, 10 mm to 8 mm, 8 mm to 6 mm, then recombine each particle size coal together for testing.

7.2.3 Distribute each subsample into 5 crucibles (5.5), each of which is about 100 cm^3, and cover them with crucible lids then place them onto the crucible stand (5.6).

7.2.4 Quickly insert the stand with samples into the furnace at 850 °C. Close the furnace door and heat for 30 min. The furnace temperature will drop when the samples are initially placed into the furnace, the furnace shall recover to (850 ± 15) °C within 8 min, then maintain the temperature, otherwise discard this test. Heating time (30 min) includes temperature recovery time (8 min).

NOTE The initial temperature of 850 °C can be adjusted to guarantee that the furnace temperature of 850 °C ± 15 °C is regained within 8 min after insertion of the cold stand and its crucible(s).

7.2.5 Take out the stand with sample and allow them cool to room temperature, Determine the total mass of the residues to the nearest 0,01 g.

For those samples that are agglomerate after being heated, stop the experiment and illustrate it in the report.

