INTERNATIONAL STANDARD

ISO 7404-2

Second edition
2009-10-01

Methods for the petrographic analysis of coals —

Part 2: Methods of preparing coal samples

Méthodes d'analyse pétrographique des charbons —

Partie 2: Préparation des échantillons de charbon

Reference number
ISO 7404-2:2009(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

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 2.

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.

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.

ISO 7404-2 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*.

This second edition cancels and replaces the first edition (ISO 7404-2:1985), which has been technically revised.

ISO 7404 consists of the following parts, under the general title *Methods for the petrographic analysis of coals*:

— *Part 1: Vocabulary*[1)]

— *Part 2: Methods of preparing coal samples*

— *Part 3: Method of determining maceral group composition*

— *Part 4: Methods of determining microlithotype, carbominerite and minerite composition*[1)]

— *Part 5: Method of determining microscopically the reflectance of vitrinite*

1) Parts 1 and 4 of this International Standard will be available under the original title, *Methods for the petrographic analysis of bituminous coal and anthracite*, until the revisions of these documents have reached the stage at which they are publicly available.

Introduction

Petrographic analyses have been recognized internationally as important in the context of the genesis, vertical and lateral variation, continuity, metamorphism and usage of coal. The International Committee for Coal Petrology (ICCP) has made recommendations concerning nomenclature and analytical methods and has published an extensive handbook that is continuously updated, describing in detail the characteristics of a wide range of coals. The text of this part of ISO 7404 agrees substantially with the text of the handbook and incorporates many useful comments made by members of the ICCP and by member bodies of ISO/TC 27, *Solid mineral fuels*.

Petrographic analyses of single-seam coals provide information about the rank, the maceral and microlithotype compositions and the distribution of mineral matter in the coal. The reflectance of vitrinite is a useful measure of coal rank and the distribution of the reflectance of vitrinite in a coal blend. Together with a maceral group analysis, it can provide information about chemical and technological properties of the coal and coal blend. Various other applications, like the characterization of bulk samples, cargoes, etc., and the precise determination of different rank vitrinites in complex coal blends are in use.

ISO 7404 (all parts) is concerned with the methods of petrographic analysis currently employed in characterizing coal in the context of its technological use and establishes a system for petrographic analysis.

The method is applicable for low-, medium- and high-rank coals.

The varied petrographic composition and hardness of coal and the type and amount of included mineral matter does not permit the formulation of a precise procedure that can be applied with equal success to all types and ranks of coal. For example, a successful preparation method for use with medium- and high-rank coals might not be applicable among low-rank coals. Within these limits, therefore, this part of ISO 7404 allows the operator to apply individual skills and experience to the preparation of a satisfactory polished surface. Nevertheless, recommended procedures that have been found applicable to a variety of coals, are given in the Annex A, which is for information only.

Many processes are involved between the mining of the coal and its preparation for industrial use. Petrographic analysis can be required at any stage on samples from the coal seam *in situ,* from borehole cores, on the raw product from the colliery, on the products from the preparation plant, or on the final product. The amount and size distribution of the coal being investigated thus varies widely and it is important to ensure that the sample obtained for petrographic analysis is fully representative.

Methods for the petrographic analysis of coals —

Part 2:
Methods of preparing coal samples

1 Scope

This part of ISO 7404 specifies methods for preparing a polished particulate block from a sample of crushed coal for analysis by reflectance microscopy. These methods can also be applied to the preparation of a polished, embedded lump of coal.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references the latest edition of the referenced document (including any amendment) applies.

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 7404-1, *Methods for the petrographic analysis of bituminous coal and anthracite — Part 1: Vocabulary*[1)]

ISO 18283, *Hard coal and coke — Manual sampling*

ICCP International Handbook of Coal Petrography

3 Definitions

For the purpose of this document, the definitions given in ICCP International Handbook and in ISO 7404-1 apply.

4 Principle

A representative sample of air-dried coal is crushed to a specified particle size and mixed with a suitable binder. The mixture is formed into a particulate block, one face of which is ground and polished to give a relief-free and scratch-free surface for analysis by reflectance microscopy.

5 Reagents and materials

5.1 Binder, used to hold the particles of crushed coal together as a particulate block, or to embed a lump of coal.

The properties of the binder shall be such that

a) there shall be no chemical reaction with the coal or immersion oil;

b) for liquid binders such as polyester resin, the curing temperature required to make the particulate block should not exceed 100 °C and a temperature of less than 60 °C is preferable;

c) for thermoplastic mounting materials such as polymethylmethacrylate (PMMA) powder, a temperature of about 120 °C is required for proper annealing;

d) the surfaces of the coal particles should be easily wetted and there shall be good penetration of pores and cracks;

e) the coal particles should be held securely during grinding and polishing;

f) there should be a marked contrast with the coal particles when immersed in oil and focused under the microscope;

g) the hardness should be comparable with that of the coal so that a flat, relief-free and scratch-free surface can be obtained by grinding and polishing;

h) there should be no large volume changes during curing, which can cause possible damage to the coal particles;

i) the viscosity of liquid binder should be such that the tendency of coal grains to segregate due to density and size is minimized.

5.2 Mould release agent, that does not affect the coal and mounting compound, nor damage the mould.

5.3 Grinding abrasives, consisting of silicon carbide papers of decreasing grain size, [53,5 µm (240 grit or P280), 23,6 µm (400 grit or P800), 16,0 µm (600 grit or P1200)].

Metal-bonded, diamond-impregnated 15 µm grinding disks may be used as a substitute for the smaller-grain-size silicon carbide paper.

5.4 Polishing abrasives, consisting of metal oxide powders, colloidal silica suspension, or diamond pastes of decreasing grain size.

A polishing abrasive having a maximum particle size not exceeding 0,05 µm shall be used for the final polishing stage.

NOTE The number of polishing stages depends on the grain size of the abrasive used at the final stage of grinding and on the grain size of the polishing abrasives available. It is recommended that aluminium oxide powders be used throughout and that an abrasive having a maximum particle size of 0,3 µm be used for the penultimate polishing stage.

5.5 Lap cloths, made of cotton, silk or synthetic fabric with a minimum of nap.

6 Apparatus

6.1 Test sieve, having an aperture 1,00 mm, in accordance with the requirements of ISO 3310-1, with a suitable lid and receiver.

6.2 Grinding mill or **mortar and pestle**, suitable for crushing 0,3 kg to 0,45 kg of coal to pass through the test sieve (6.1), with the minimum production of fines.

The grinding mill may be manually or electrically operated.

6.3 Press, for use when pressure is required during curing, for example when using PMMA.

It shall be capable of producing a pressure of up to 21 MPa [2)] and may be a simple hand operated lever, a torque-wrench, or a hydraulic press.

2) 1 MPa = 10^6 N/m^2 = 145 psi.

6.4 Moulds, to hold the mixture of coal and binder during the curing process.

In simple moulding, these may be made from heavy-gauge aluminium foil, but reusable moulds may be made from silicone rubber, flexible plastic, aluminium or steel. For pressure moulding, a cuboid or cylindrical steel mould equipped with a removable base and cap or other means of removing the block from the mould after curing; see Note. The interior surfaces of the metal mould should have a ground finish.

Metal moulds for use in pressure moulding shall be capable of withstanding double the pressure normally applied in making the particulate block. The internal dimensions of the mould shall be such that the face of the block that will be polished has a surface area of at least 500 mm^2.

NOTE For reflectance analysis, if the coal is deficient in vitrinite, it can be necessary to make more than one block of minimum size.

6.5 Pelletizing machine, consisting of an automatic mounting press that can be pre-programmed for mould size, type of binder (thermosetting, thermoplastic), heating time, cooling time, initial temperature, and curing pressure.

These have been found to be time-saving and produce blocks of consistent quality. By employing a spacer in a tall mould, two pellets can be made during one cycle.

6.6 Containers, disposable, suitable for mixing the required amounts of coal and binder.

NOTE Wax-coated containers are unsuitable for liquid binders.

6.7 Machine for grinding and polishing, either with stationary or rotating laps for manual polishing, or automatic grinder/polishers which have been found to save time, equipped with interchangeable lapping discs for each of the grinding and polishing stages.

The machine should be fitted with a contra-rotating specimen holder of the type in which the specimen is held rigidly and is not free to rotate independently of the holder. The specimen holder should have a means of varying the load on the specimen.

6.8 Sample cleaner, consisting of a means of cleaning the surface of the particulate block between the successive stages of grinding and polishing.

Jets of tap water and distilled water are essential and, in addition, an ultrasonic cleaning bath is desirable. If necessary, a water filter should be used to remove solid particulates from the water supply before it is used in cleaning and polishing.

6.9 Desiccator.

7 Procedure

7.1 Preparation of the coal sample for making a particulate block

7.1.1 Sample

Obtain a representative sample of the coal being examined. For most purposes, it is convenient to take this sample after the first stage in the preparation of the laboratory sample for general analysis in accordance with the requirements of ISO 18283.

7.1.2 Drying

Air-dry the sample (7.1.1) in accordance with the requirements of ISO 18283 to facilitate crushing and sample division and to avoid interference with the curing of the binder in the preparation of the particulate block.

7.1.3 Size reduction

Reduce the size of the particles to an upper limit of 1 mm.

The reduction in the size of the coarse particles should be carried out using a grinding mill (6.2) adjusted to give a product crushed with minimum production of fines. If a mortar and pestle (6.2) is used, sieve and grind the oversize repeatedly until all the coal passes the specified size.

7.1.4 Sample division

Divide the sample using a riffle or small rotary sample divider to obtain a laboratory sample of 50 g to 100 g of coal in accordance with the requirements of ISO 18283. The laboratory sample may be stored in a sealed container prior to analysis.

7.2 Preparation of particulate block

The objective is to prepare a suitably thick particulate block that, when polished, exposes a surface comprised of at least 50 % coal.

NOTE 1 A polished surface with this percentage of coal reduces the time of analysis and any tendency towards the segregation of particles due to size and density.

The precise procedure for preparing a particulate block depends on the type of binder, mould and whether a press is used. Provided that the materials and apparatus comply with the requirements of Clauses 5 and 6, the steps in the procedure may be chosen by the operator.

NOTE 2 In the case of blocks made with epoxy resin, an elevated temperature may be used to promote curing of the binder. When rapid curing is not required, curing can be carried out at ambient temperature.

7.3 Preparation of polished surface of particulate block

For cylindrical-shaped samples prepared by pressure moulding, grind and polish one end face of the particulate block, by hand or by using a grinding and polishing machine (6.7) and a series of abrasives of decreasing particle size. The block may be held manually or by means of a specimen holder.

Particulate blocks made with liquid binders and cured in the absence of pressure under ambient conditions may show gravity (density) stratification, with larger particles along the bottom or base of the block, and finer material at the top. Grind and polish the sides of such stratified blocks, that is, the surfaces perpendicular to the stratification.

Suitable materials for both grinding and polishing are described in 5.3 to 5.5, and A.2.3 to A.2.5. Carry out the final polish with an abrasive having a maximum particle size not exceeding 0,05 µm.

Thoroughly wash the surface of the block under a strong jet of water (6.8) after each stage of grinding and polishing. Immersion of the block in distilled water in an ultrasonic cleaning bath is recommended for removing the debris remaining after the grinding stages. The removal of all traces of polishing abrasive from the block is essential. After the final washing, rinse with a jet of distilled water and dry the particulate block in a stream of clean air.

NOTE 1 An electric hair-drier or fan-assisted warm-air chamber are both suitable for this purpose.

NOTE 2 Several recommended polishing and grinding procedures are given in Annex A.

7.4 Examination of the polished surface

7.4.1 Examine the polished surface using a microscope equipped with a dry objective lens at a magnification of approximately 100x to 250x. The polished surface should fulfil the following requirements.

a) It should be flat and essentially free from relief.

b) It should be substantially scratch-free, with little evidence of pitting.

c) It should be clean, free from smears and abraded material.

If the polished surface does not meet requirements a) to c), repeat the procedures detailed in 7.3 beginning at the grinding stage.

Give particular attention to the final stage of polishing and, if necessary, change the final polishing abrasive and/or the lap cloth.

If the surface fails only requirement c), repeat the washing procedure detailed in 7.3. If, after further rinsing with distilled water and drying in a stream of clean air, the surface still does not meet all the requirements, repeat the procedure beginning at the grinding stage. Polishing defects and acceptable surfaces are illustrated in Figure 1; see Note.

7.4.2 The following photomicrographs are shown in Figure A.1:

a) coal grains well polished without relief or other defects, in air;

b) coal grains well polished without defects, under oil immersion;

c) coal grains with smear tracks across the surface of the block;

d) coal particles with coarsely pitted surfaces unsatisfactory for the measurement of reflectance;

e) an unacceptable polish due to the meshwork of coarse and fine scratches.

Photomicrographs a) and d) are viewed with a dry objective lens. Photographs b), c) and e) are viewed with an oil immersion objective lens.

NOTE The appearance of very fine scratches on the polished surface of vitrinite is a common fault in polishing. These scratches can be seen more easily by altering the intensity of illumination or by using oblique illumination.

7.5 Storage prior to reflectance analysis

If the polished surface is satisfactory, remove the block from the holder. Store in a desiccator (6.9) for 15 h prior to reflectance analysis, unless it has previously been established that the reflectance of the coal is unaffected by moisture content.

7.6 Re-examination of a particulate block

Prior to re-examination for measurements, a surface that has been previously exposed to immersion oil and subsequently cleaned for storage shall be re-polished in accordance with 7.3.