International Standard 7404/2

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Methods for the petrographic analysis of bituminous coal and anthracite —
Part 2: Method of preparing coal samples

Méthodes d'analyse pétrographique des charbons bitumineux et de l'anthracite — Partie 2: Préparation d'échantillons de charbon

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Methods for the petrographic analysis of bituminous coal and anthracite — Part 2: Method of preparing coal samples

0 Introduction

0.1 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 describing in detail the characteristics of a wide range of coals. The text of this International Standard 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 a single coal 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, can provide information about some important chemical and technological properties of the blend.

This International Standard is concerned with the methods of petrographic analysis currently employed in characterizing bituminous coal and anthracite in the context of their technological use. It establishes a system for petrographic analysis and comprises five parts, as follows:

Part 1: Glossary of terms.

Part 2: Method of preparing coal samples.

Part 3: Method of determining maceral group composition.

Part 4: Method of determining microlithotype composition.[1)]

Part 5: Method of determining microscopically the reflectance of vitrinite.

For information on the nomenclature and analysis of brown coals and lignites, reference should be made to the *International Handbook of Coal Petrography* published by the ICCP.[2)]

0.2 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 which can be applied with equal success to all types and ranks of coal. Within these limits, therefore, this part of ISO 7404 allows the operator to apply his individual skill and experience to the preparation of a satisfactory polished surface. At the same time a recommended procedure, which has been found applicable to a wide variety of coals, is given in the annex.

Many processes are involved between the mining of the coal and its preparation for industrial use. Petrographic analysis may be required at any stage on samples from the coal seam *in situ* or from borehole cores, the raw product from the colliery, the products from the preparation plant or 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.

1 Scope and field of application

This part of ISO 7404 specifies a method for preparing a polished particulate block from a sample of crushed coal, for analysis by reflectance microscopy using white light. It does not apply to the preparation of polished particulate blocks for analysis using fluorescence microscopy techniques nor to the preparation of polished orientated lumps of coal.

2 References

ISO 1988, *Hard coal — Sampling.*

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: Glossary of terms.*

1) At present at the stage of draft.

2) The second edition (1963), together with the supplement issued in 1971, may be obtained from Professor D.G. Murchison, Organic Geochemistry Unit, Department of Geology, University of Newcastle, Newcastle-upon-Tyne, NE1 7RU, United Kingdom. The supplement issued in 1973 may be obtained from Centre national de la recherche scientifique, 15, quai Anatole-France, F-75007 Paris, France.

3 Definitions

For the purpose of this part of ISO 7404, the definitions of ISO 7404/1 apply.

4 Principle

The mixing of a representative sample of air-dried coal, crushed to a specified particle size, with a suitable binder. Formation of the mixture 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 Materials

5.1 Binder, used to hold the particles of crushed coal together as a particulate block. The properties of the binder shall be such that:

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

b) the temperature required to make the particulate block shall not exceed 100 °C and a temperature less than 60 °C is preferable;

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

d) the coal particles shall be held securely during grinding and polishing;

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

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

g) there shall be no large volume changes during curing which might cause possible damage to the coal particles.

5.2 Mould release agent, which does not damage the mould or affect the coal or binder.

5.3 Grinding abrasives. Silicon carbide papers of decreasing grain size.

5.4 Polishing abrasives. Metal oxide powders or diamond pastes of decreasing grain size. A metal oxide powder having a maximum particle size not exceeding 0,05 µm shall be used for the final polishing stage.

NOTE — The number of polishing stages will depend 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: cotton, silk or synthetic fabric with a minimum of nap.

6 Apparatus

6.1 Test sieve, of aperture 1,00 mm complying with the requirements of ISO 3310/1, with a suitable lid and receiver.

6.2 Grinding mill or **pestle and mortar,** suitable for crushing 0,3 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, capable of producing a pressure of up to 17 MPa.[1)]

This may be a simple hand operated lever, a torque-wrench or a hydraulic press.

6.4 Metal mould, to hold the mixture of coal and binder during the curing process, with an ejector ring and plunger or other means of removing the block from the mould after curing. (See note 1.)

The mould 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 to be polished has a surface area of at least 600 mm^2.

NOTES

1 The interior of the mould and the surface of the plunger should have a ground finish.

2 For reflectance analysis two blocks of minimum size may be necessary if the coal is deficient in vitrinite. The mould may be either cuboid or cylindrical provided that the block produced fits the holder of the grinding and polishing apparatus being used.

3 Figure 1 gives an example of the dimensions of a mould, plunger and ejector ring used to produce a block 40 mm in diameter.

6.5 Disposable containers, suitable for mixing the required amounts of coal and binder.

NOTE — Wax coated containers are unsuitable.

6.6 Grinding and polishing machine, with interchangeable lapping discs for each of the grinding and polishing stages.

NOTE — The machine should be fitted with a contrarotating 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.

1) 1 MPa = 10^6 N/m^2

6.7 Sample cleaner. Some means of cleaning the surface of the particulate block between the successive stages of grinding and polishing shall be available. Jets of tap water and distilled water are essential and, in addition, an ultrasonic cleaning bath, if available.

NOTE — If necessary a cleaning filter should be used to remove solid particulates from the water supply before use in cleaning and polishing.

6.8 Desiccator.

7 Procedure

7.1 Preparation of coal sample for making particulate block

7.1.1 Subsample

Obtain a representative subsample of the coal to be examined. For most purposes it will be 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 1988.

7.1.2 Drying

Air dry the subsample (7.1.1) in accordance with the requirements of ISO 1988 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.

NOTE — 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 to an upper size of 1,00 mm with minimum production of fines. If a pestle and mortar (6.2) is used, sieve and grind the oversize repeatedly until all the coal just passes the specified size.

7.1.4 Sample division

Divide the subsample using a riffle or small rotary sample divider to obtain a laboratory sample of 50 to 100 g of coal in accordance with the requirements of ISO 1988. The laboratory sample may be stored in a screw-topped jar prior to analysis.

7.2 Preparation of particulate block

The object is to prepare a particulate block of suitable thickness in which particles of coal are evenly dispersed in the resin such that at least 60 % of the cross-sectional area of the polished surface is coal.

NOTES

1 This percentage will reduce the time of analysis and any tendency towards the segregation of particles due to size and density.

2 The precise procedure for preparing a particulate block will depend on the type of binder, mould and press 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 experiment.

3 An elevated temperature is used to speed the rate of cure of the binder. When rapid curing is not required, curing may be carried out at ambient temperature provided that adequate time is allowed and all voids are eliminated.

4 A recommended procedure is given in the annex.

7.3 Preparation of polished surface of particulate block

Grind and polish one end face of the particulate block using a grinding and polishing machine (6.6) and a series of abrasives of decreasing particle size. The block may be held manually or by means of the specimen holder.

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 a metal oxide powder having a maximum particle size not exceeding 0,05 µm.

Thoroughly wash the surface of the block under a strong jet of water (6.7) after each stage of grinding and polishing. Immersion of the block in distilled water in an ultrasonic cleaning bath (6.7) is recommended for removing the debris remaining after the grinding stages. The removal of all traces of polishing abrasive from the block is essential and this may be achieved by wiping the surface with clean lens tissue or cotton wool whilst washing the surface under a strong jet of water. After the final washing rinse with a jet of distilled water. Dry the particulate block in a stream of clean air.

NOTES

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

2 A recommended polishing and grinding procedure is given in the annex.

7.4 Examination of the polished surface

Examine the polished surface with a dry objective lens at a magnification of approximately X 100 to X 250. The surface shall fulfil the following requirements:

a) the prepared surface shall be flat and substantially free from relief;

b) the particles on the surface shall be substantially free from pits;

c) the surface shall be substantially free from fine scratches;

d) the surface shall 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 requirement d) only, repeat the washing procedure detailed in 7.3. If, after further rinsing with distilled water and drying in a stream of clean warm air, the surface still does not meet all the requirements, repeat the procedures beginning at the grinding stage. Polishing defects and acceptable surfaces are illustrated in figure 2. (See the note.)

The following photomicrographs are shown in figure 2.

1 particles showing high relief indicated by dark peripheral shadows;

2 particles satisfactorily polished without relief or other defects;

3 particles with coarsely pitted surfaces unsatisfactory for measurement of reflectance;

4 smear tracks across the surface of the block;

5 an unacceptable polish due to the meshwork of coarse and fine scratches;

6 a satisfactorily polished surface.

Photomicrographs 1 to 4 are viewed with a dry objective lens. Photomicrographs 5 and 6 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 may 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 (6.8) 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

A surface which has been exposed to air or oil shall be re-polished according to 7.3 before re-examination.

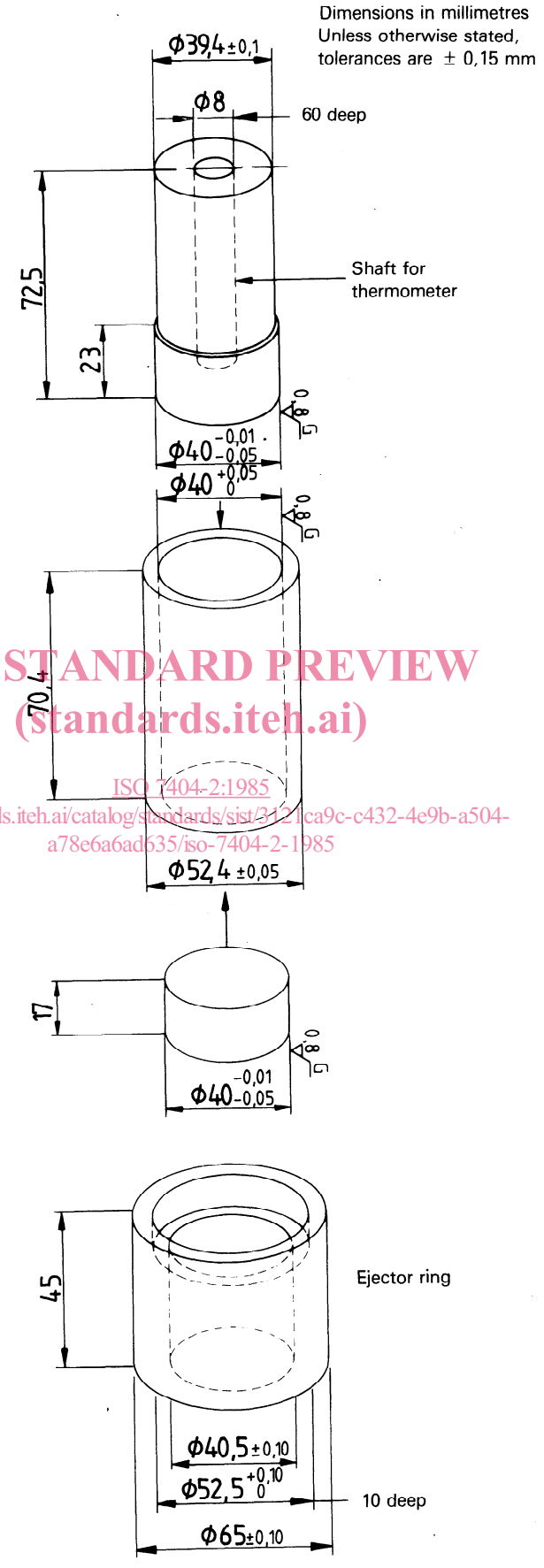

Figure 1 — Mould suitable for preparation of particulate block

Annex

A recommended procedure for the preparation of a polished particulate block from a sample of crushed coal for petrographic analysis

A.1 General

This annex describes the materials and apparatus needed to carry out the procedure for the preparation of a particulate block from a sample of crushed coal and the grinding and polishing of one end face. The procedure has the merit of being quick and of producing a polished surface to the required standard on coals of different rank with reasonable consistency.

A.2 Materials

A.2.1 Binder. An epoxy resin of low viscosity, containing diglycidyl ethers and a liquid hardener.

WARNING — Contact between epoxy resins and the skin should be avoided. Rubber or disposable polyethylene gloves are recommended. Avoid inhaling fumes from the resin during mixing and curing steps by providing good ventilation.

The mixture of the epoxy resin and hardener should have a viscosity not exceeding 10 P[1] at 25 °C when freshly mixed and the curing time at 90 ± 5 °C should not exceed 30 min.

A.2.2 Mould release agent, silicone compound dissolved in white spirit which has an aromatics content less than 25 % (*V*/*V*).

A.2.3 Grinding abrasives, water-resistant adhesive-backed silicon carbide papers having a medium grain size of approximately 50 and 15 µm.

A.2.4 Polishing abrasives, aluminium oxide powders having a maximum particle size not exceeding 0,3 and 0,05 µm.

A.2.5 Lap cloths, synthetic cloths backed with water-resistant adhesive and with a minimum of nap.

A.3 Apparatus

A.3.1 Hydraulic press, capable of producing a pressure of up to 17 MPa.

A.3.2 Metal mould.

See 6.4.

A.3.3 Heating jacket, suitable for raising the temperature of the mould to 90 ± 5 °C.

A.3.4 Disposable containers.

See 6.5.

A.3.5 Grinding and polishing machine, with lapping discs having a diameter of 300 mm.

See 6.6.

A.3.6 Ultrasonic cleaning bath, as sample cleaner.

A.3.7 Desiccator, having a diameter of 20 to 25 cm.

A.3.8 Oven, capable of being maintained at 90 ± 5 °C.

A.3.9 Thermometer, mercury in glass type, covering the range 0 to 110 °C.

A.4 Procedure

A.4.1 Preparation of particulate block

Prepare about 10 ml of the epoxy resin and hardener (A.2.1) in the proportions recommended by the manufacturer and thoroughly mix in a disposable container (A.3.4) using a disposable wooden splint.

Place approximately 20 ml (26 g) representative of the crushed coal sample into another disposable container. Add a few drops of the prepared resin and hardener mixture and thoroughly stir these into the coal using a disposable wooden splint. Add further resin drop by drop until the mixture has the consistency of a stiff paste which will just adhere to the walls of the container.

Clean the plungers and internal surfaces of the mould (A.3.2) to remove any resin left from previous use and coat these surfaces with the mould release agent (A.2.2). Insert the lower plunger. Preheat the mould and plungers to a temperature of 90 ± 5 °C by using a heating jacket (A.3.3) or alternatively by placing them in a drying oven (A.3.8) maintained at 90 ± 5 °C. Fill the preheated mould with the coal and resin mixture, and insert the upper plunger. Place the mould and contents in a press (A.3.1) and apply a pressure of at least 14 MPa and not more than 17 MPa to the block for 3 to 5 s. Release and reapply the pressure and repeat the cycle several times to remove air bubbles entrapped during mixing. Maintain the pressure for at least 10 min to accommodate any shrinkage whilst the block hardens.

1) 1 P = 0,1 Pa·s = 0,1 N·s/m^2

Avoid the application of too great a pressure which may result in the cracking of the coal particles. Use the heating jacket to maintain the temperature of the mould at 90 ± 5 °C throughout this operation, and for at least a further 10 min. Check the temperature with a thermometer (A.3.9) placed in the shaft drilled into the plunger.

Eject the particulate block from the mould and label it. If the block is slightly soft when pressed with the thumbnail, place it in a drying oven maintained at 90 ± 5 °C for a few minutes until it is quite hard.

A.4.2 Grinding procedure

Secure the particulate block in a specimen holder, apply a pressure of approximately 0,02 MPa to the block and grind one flat surface of the block using silicon carbide paper having a median grain size of approximately 50 µm and water as lubricant. The laps should rotate at between 125 and 150 r/min and the specimen holder should rotate in a contrary direction at between 30 and 60 r/min. Flush the abrasive surface with water whilst grinding and continue grinding until the exposed surfaces of the coal particles lie in a plane free from holes and cracks. The time for automatic grinding is dependent on such factors as the hardness of the coal, the state of wear of the silicon carbide papers, and the initial levelling of the blocks in the specimen holder. This stage should normally be completed within 1 min. (See notes 1 and 2.)

Remove any particles of grit by washing the particulate block contained in the specimen holder under a strong jet of tap water. Immersion in distilled water in an ultrasonic cleaning bath (A.3.6) will ensure removal of any trapped coarse grit.

Change to a silicon carbide paper having a medium grain size of approximately 15 µm and continue the grinding until the individual coal particles are clearly visible and the surface is smooth, free from deep scratches and has a slight polish. Cleanse the particulate block and specimen holder as in the previous paragraph.

Examine the ground surface using a dry objective lens of low magnification (approximately X 10) without removing the block from the specimen holder, and if deep scratches are present regrind the surface by the same procedure commencing with the 50 µm grain size silicon carbide paper. (See note 3.)

NOTES

1 Specimen holders normally accommodate several blocks and all the spaces should be filled before operating, with blanks if necessary.

2 The edge of the block should be manually chamfered on the silicon carbide paper of 50 µm grain size to avoid fragmentation of the edge during polishing with the consequent risk of damage to the prepared surface.

3 The silicon carbide papers are replaced when they are worn to an extent that suitably ground surfaces cannot be produced in the normal time required for the particular grinding stage.

A.4.3 Polishing procedure

Prepare a slurry by adding 10 ml of water to 10 ml of polishing alumina (A.2.4) having a maximum particle size of 0,3 µm, contained in a 25 ml measuring cylinder. While the lap is stationary, saturate the lap cloth with water and pour the whole of the slurry over the surface of the cloth.

Apply the same pressure to the block as was used in the grinding stage and with the same lap speeds polish the blocks for 2 min. No additional slurry or water need be added during the 2 min polishing time. At the end of this period, and with the lap still rotating, flood the lap with a jet of water to remove the slurry from the lap and the blocks. After washing the blocks on the lap for about 0,5 min, remove any residual particles of coal or alumina by washing the particulate blocks contained in the specimen holder under a strong jet of water. (See note 1.)

Repeat the polishing procedure using a second lap charged with a slurry consisting of 10 ml of water and 10 ml of polishing alumina with a maximum particle size of 0,05 µm. (See notes 2 and 3.)

Without removing the specimen holder, flush the revolving lap with water for about 0,5 min to remove the slurry. Whilst still contained in the specimen holder, remove any residual alumina by washing the particulate blocks under a strong jet of water whilst wiping the surfaces with cotton wool or lens tissue. Finally, rinse the blocks with a jet of distilled water, dry them in a stream of clean warm air and, without removing them from the specimen holder, proceed with the examination of the blocks as described in 7.4.

NOTES

1 After use, the laps should be washed with water and stored in a dust-proof container. The lap cloths should be replaced when they become worn or when unsatisfactory polished surfaces are produced.

2 The type of lap cloth used in the final stage of polishing determines the ability to produce consistently satisfactorily polished coal surfaces. Operators should experiment with a variety of lap cloth types in order to find one that is satisfactory.

3 The final polishing time should normally not exceed 2 min but is dependent on the pressure applied to the block, the speeds of rotation of the lap and the specimen holder, their dimensions and the distance between their centres. Relief effects at the edges of the particles will be minimized by keeping polishing times as short as possible. Using this type of equipment with the range of lap speeds and pressures specified it has been found that a satisfactory relief-free polish can be achieved. The precise conditions to achieve the standard of polish required should be determined for the particular machine used and once established they should not be varied for the subsequent production of polished particulate blocks.