BRITISH STANDARD

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Methods for the petrographic analysis of coals

Part 2: Methods of preparing coal samples

ICS 73.040; 75.160.10

National foreword

This British Standard is the UK implementation of ISO 7404-2:2009. It supersedes BS 6127-2:1982 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/16, Solid mineral fuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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

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Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
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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.

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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.

INTERNATIONAL STANDARD **ISO 7404-2:2009(E)**

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.

Annex A
(informative)

Examples of procedures for the preparation of a polished particulate block suitable for petrographic analysis from a sample of crushed coal

A.1 General

This annex describes the materials and apparatus used by many laboratories to make particulate blocks from samples of crushed coal, and the grinding and polishing of these blocks for examination. The procedures have the merit of being relatively quick and produce a polished surface to the required standard on coals of different ranks with reasonable consistency. PMMA powder is unsatisfactory for mounting low-rank coals.

A.2 Reagents and materials

A.2.1 Binders, consisting of one of the following:

A.2.1.1 Epoxy resin, **polyester resin**, and their **hardeners**, consisting of a low-viscosity epoxy resin and a liquid hardener, or a polyester resin and its 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[3)] at 25 °C when freshly mixed and the curing time at (90 ± 5) °C should not exceed 30 min, or gravity sedimentation of the grains can take place.

A.2.1.2 Polymethyl methacrylate powder.

A.2.2 Mould release agent, consisting of a silicone compound in a non-chlorofluorocarbon (CFC) aerosol propellant.

An aerosol mixture of canola oil, grain alcohol and lecithin, normally used as a non-stick cooking spray, is equally effective.

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

Metal-bonded, diamond-impregnated, 15 µm disks may be substituted for silicon carbide paper with the smaller grain size.

A.2.4 Polishing abrasives, consisting of an aluminium oxide powder having a maximum particle size not exceeding 0,3 µm or 0,05 µm, or colloidal silica suspensions having a maximum particle size of 0,05 µm.

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

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3) 1 P = 0,1 Pa·s = 0,1 N·s/m^2.

A.3 Apparatus

A.3.1 Hydraulic press, capable of producing a pressure of up to 21 MPa.

A.3.2 Mould; see 6.4.

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

A.3.4 Cooling wings, aluminium, capable of accelerating the cooling of the mould when using PMMA powder as mounting compound.

A.3.5 Containers, disposable; see 6.6.

A.3.6 Grinding and polishing machine; see 6.7.

A.3.7 Ultrasonic cleaning bath, capable of functioning as a sample cleaner.

A.3.8 Desiccator.

A.3.9 Oven, capable of being maintained at (90 ± 5) °C.

A.3.10 Thermometer, metal probe type, with a mechanical temperature indicator, or digital solid-state read-out covering the range 0 °C to 110 °C.

A.3.11 Automatic mounting press.

A.4 Procedures

A.4.1 Preparation of particulate block

A.4.1.1 Preparation of particulate block using polyester resin

Prepare about 10 mL of the polyester resin and hardener (A.2.1.1) in the proportions recommended by the manufacturer and thoroughly mix in a disposable container (A.3.5) using a stirring rod.

Place approximately 10 g representative of the crushed coal sample into a mould. Add an equivalent amount of activated polyester resin and thoroughly mix using a stirring rod. The coal/polyester resin mixture should fill the mould to a depth of 25 mm. Ensure that particles located at edges and corners of the mould are thoroughly mixed and wetted.

Particulate blocks made with liquid binders and cured in the absence of pressure under ambient conditions can show gravity (density) stratification, with the larger particles along the bottom or base of the block and finer material at the top.

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

A.4.1.2 Preparation of particulate block using epoxy resin and pressure moulding

Prepare about 10 mL of the epoxy resin and hardener (A.2.1.1) in the proportions recommended by the manufacturer and thoroughly mix in a disposable container (A.3.5) 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 additional resin drop by drop until the mixture has the consistency of a stiff paste that just adheres 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 a mould-release agent (A.2.2). Insert the lower plunger. Preheat the mould and plungers to a temperature of (90 ± 5) °C using a heating jacket (A.3.3) or alternatively by placing them in a drying oven (A.3.9) 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 but not more than 17 MPa to the block for 3 s 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.

Avoid application of too much pressure, which can result in cracking 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 an additional 10 min. Check the temperature with a thermometer (A.3.10) 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 hard.

A.4.1.3 Preparation of particulate block using PMMA powder and pressure curing

Use a 5 mL (3 g or 1 tsp) measuring spoon, and place three parts by volume representative of the crushed coal sample (9 g) and one part by volume (3 g) of the powdered PMMA in a container and thoroughly mix together by gentle shaking. Spray the internal surfaces of the mould and plungers with a mould-release agent (A.2.2). Insert the lower plunger and pour the coal and PMMA mixture into the mould. Gently tap the mould to ensure that the mixture is evenly distributed and level, and add about 1 g of the PMMA powder. Place a sample identification label on top of the PMMA, and add sufficient PMMA to just cover the label. Carefully insert the upper plunger without disturbing the PMMA, and place the mould and the contents in a press (A.3.1), equipped with a heating jacket (A.3.3) and apply a pressure of at least 1 MPa to the block. The heating cycle varies with the age and condition of the equipment used, but for a steel mould with an internal diameter of 25 mm to reach the annealing temperature (about 120 °C) of the PMMA can take between 10 min and 20 min. When the temperature of 120 °C is reached, remove the heating jacket from around the mould and replace it with the aluminium cooling wings. Release and reapply the pressure several times to release air bubbles entrapped during heating. Maintain a pressure of 21 MPa during the cooling cycle of the PMMA pellet, which lasts about 15 min.

If an automatic mounting press is being used, ensure that the temperature of the mould during the heating cycle does not greatly exceed a maximum temperature of 120 °C, and that the pressure applied during the cooling cycle does not greatly exceed 21 MPa.

A.4.1.4 Preparation of an embedded lump of coal using polyester resin

Place the lump of coal in a mould and activate an adequate volume of polyester resin so that the sample is completely covered by the liquid binder. The viscosity of the binder can be relatively high, especially if the sample of coal is porous. It is recommended that moulds containing very porous samples covered in polyester resin, be placed in a sealed container in which the air pressure can be reduced by a vacuum pump and returned to ambient several times prior to curing. This promotes the flow of polyester resin into the pores and spaces, thereby preserving the structural integrity of the sample.

A.4.2 Grinding procedure

The following procedure describes the use of a specimen holder with a semi-automatic polishing machine. However, manual grinding, using a similar sequence of steps to acquire a suitable surface for polishing, is acceptable.

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 (240 grit) with water as the lubricant. The laps should rotate at between 125 r/min and 150 r/min and the specimen holder should rotate in the opposite direction at between 30 r/min 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. Although this stage should normally be completed within 1 min, it can take considerably more time; see Notes 1 and 2.

Remove any particles of grit by washing the particulate block contained in the specimen holder under a strong flow of tap water. Immersion in distilled water in an ultrasonic cleaning bath (A.3.7) can ensure the removal of any trapped coarse grit.

Change to a silicon-carbide paper having a medium grain size of approximately 15 µm (600 grit), or to a metal-bonded 15 µm diamond-impregnated disk, 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 described in the previous paragraph.

Examine the ground surface using a microscope equipped with a dry objective lens of low magnification (approximately 10x) 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.

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

NOTE 1 The edge of the block can 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.

NOTE 2 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

The procedure in this subclause describes the use of a specimen holder with a semi-automatic polishing machine. However, manual polishing, using a similar sequence of steps to acquire a suitable surface for examination, is acceptable.

Prepare a slurry by adding 100 mL of water to 25 mL (five level teaspoons) of alpha aluminium oxide powder (A.2.4) having a maximum particle size of 0,3 µm, and pour into a squirt-bottle container. Alternatively, use a slurry of a watered-down, commercially prepared suspension of 0,3 µm alpha aluminium oxide powder. While the lap is stationary, saturate the lap cloth with water and pour the slurry over the surface of the cloth.

Apply the same pressure to the block that was used in the grinding stage and with the same lap speeds polish the blocks for 2 min. It is not necessary to add additional slurry or water during the 2 min polishing time. At the end of this period, and with the lap still rotating, flood the lap with 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 aluminium oxide powder by washing the particulate blocks contained in the specimen holder under a strong flow of water. Immersion in distilled water in an ultrasonic cleaning bath (A.3.7) can ensure removal of any trapped polishing abrasive.

Repeat the polishing procedure using a second lap charged with a slurry consisting of 100 mL of water and 25 mL of gamma aluminium oxide powder with a maximum particle size of 0,05 µm. Alternatively, use a slurry of a watered-down, commercially prepared suspension of 0,05 µm gamma aluminium oxide powder. While the lap is stationary, saturate the lap cloth with water and pour the slurry over the surface of the cloth.

Apply the same pressure to the block that was used in the grinding stage and with the same lap speeds polish the blocks for 2 min. It is not necessary to add additional slurry or water during the 2 min polishing time. At the end of this period, and with the lap still rotating, flood the lap with 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 polishing abrasive by washing the particulate blocks contained in the specimen holder under a strong flow of water. Immersion in distilled water in an ultrasonic cleaning bath (A.3.7) can ensure removal of any trapped polishing abrasive.

The final polishing procedure uses a third lap charged with a slurry of colloidal silica suspension, usually watered-down to the manufacturer's specifications, and with a particle size of 0,05 µm. While the lap is stationary, saturate the lap cloth with water and pour the slurry over the surface of the cloth.

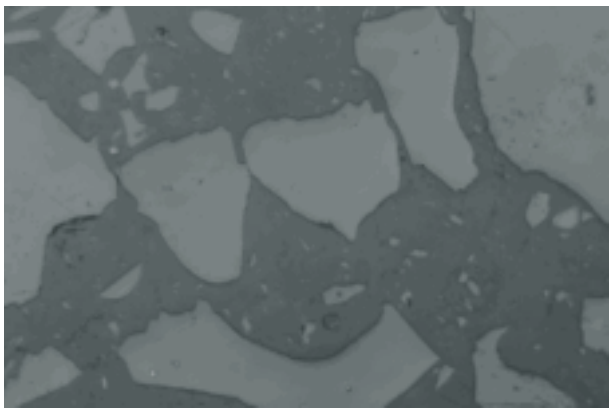
Apply the same pressure to the block that was used in the grinding stage and with the same lap speeds polish the blocks for 1 min. It is not necessary to add additional slurry or water during the 1 min polishing time. At the end of this period, and with the lap still rotating, flood the lap with water 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 by washing the particulate blocks contained in the specimen holder under a strong flow of water. Immersion in distilled water in an ultrasonic cleaning bath (A.3.7) can ensure removal of any trapped polishing abrasive.

Finally, rinse the blocks with distilled water, dry them in a stream of clean air and, without removing them from the specimen holder, proceed with the examination of the blocks as described in 7.4.

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

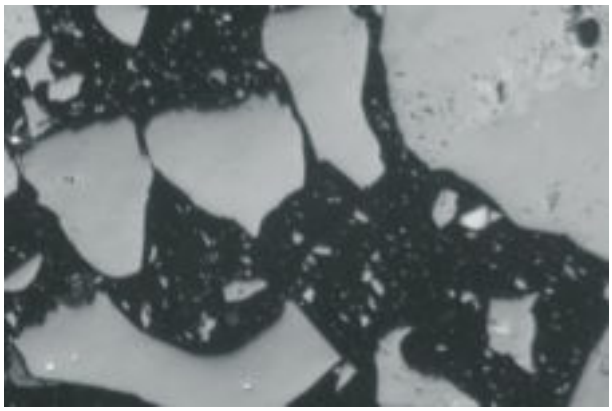
The type of lap cloth used in the final stage of polishing determines the ability to produce consistently satisfactorily polished coal surfaces. Operators should experiment with a variety of lap-cloth types in order to find those that are satisfactory.

The final polishing time should normally not exceed 1 min, but this is dependent on the pressure applied to the block, the speeds of rotation of the lap and the number of blocks in the specimen holder. Relief effects at the edges of the particles are 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, for similar coals.



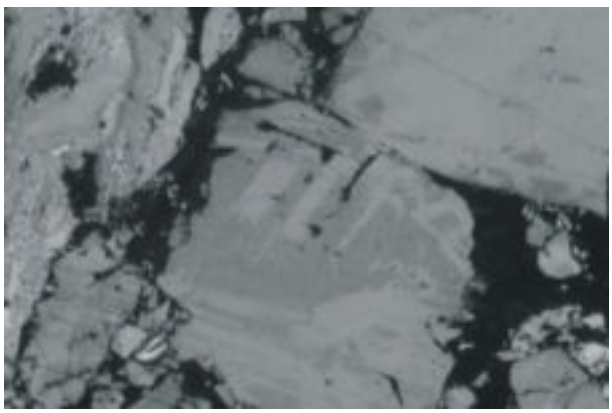
Grains of vitrinite that are substantially free from relief or scratching, observed in air. Dry objective lens. Image area approximately 600 µm by 400 µm

a) Photomicrograph 1



Grains of vitrinite that are substantially free from relief or scratching, observed under oil immersion. Oil immersion objective lens. Image area approximately 600 µm by 400 µm

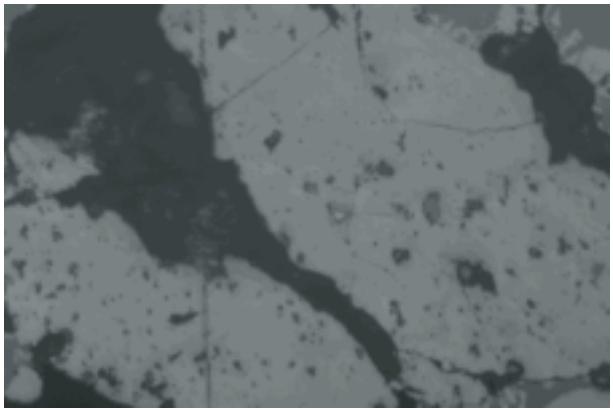
b) Photomicrograph 2



Smear tracks across the surface of some vitrinite grains. Oil immersion objective lens. Image area approximately 600 µm by 400 µm

c) Photomicrograph 3

Figure A.1 (*continued*)



Coarsely-pitted coal particles, unsuitable for analysis or measurement. Dry objective lens. Image area approximately 600 µm by 400 µm

d) Photomicrograph 4

The surface of this vitrinite grain is dissected by a meshwork of coarse and fine scratches that make it unusable for measurement. Oil immersion objective lens. Image area approximately 600 µm by 400 µm

e) Photomicrograph 5

Figure A.1 — Photomicrographs of particulate blocks viewed by reflected light showing a satisfactory polish and various polishing defects
(see 7.4)

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