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Testing concrete

Part 211: Procedure and terminology for the petrographic examination of hardened concrete

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to ii, pages 1 to 20, an inside back cover and a back cover.

Foreword

Publishing information

This part of BS 1881 is published by BSI Standards Limited, under licence from The British Standards Institution, and came into effect on 30 November 2016. It was prepared by Subcommittee B/517/1, *Concrete production and testing*, under the authority of Technical Committee B/517, *Concrete*. A list of organizations represented on this committee can be obtained on request to its secretary.

Information about this document

The Applied Petrography Group (APG) set up a working party to develop a guidance document relating to the petrographic examination of hardened concrete. This guidance document was published on the Applied Petrography Group website as *APG Special Report 2* [1].

Further discussions around this document between members of the APG and other interested persons in the UK and abroad has led to the development of this part of BS 1881.

Use of this document

It has been assumed in the preparation of this part of BS 1881 that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its methods are expressed as a set of instructions, a description, or in sentences in which the principal auxiliary verb is "shall".

Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.

Requirements in this standard are drafted in accordance with *Rules for the structure and drafting of UK standards*, subclause **J.1.1**, which states, "Requirements should be expressed using wording such as: 'When tested as described in Annex A, the product shall ...'". This means that only those products that are capable of passing the specified test will be deemed to conform to this standard.

Where words have alternative spellings, the preferred spelling of the Shorter Oxford English Dictionary is used (e.g. "organization" rather than "organisation").

Contractual and legal considerations

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

Introduction

BS 1881-211 gives a method for the petrographic examination of hardened concrete.

When applied to concrete samples, petrographic examination refers to a detailed study primarily carried out by visual and optical microscopy and the documentation of observations derived from that examination. The documented report might contain observations only, or could also include conclusions and recommendations for those requesting the study.

It is not intended that such an examination would form part of the routine assessment of the suitability of hardened concrete products. However, the procedures can provide unique and valuable information regarding the ingredients, properties and condition of hardened concrete samples. Such findings can be used in isolation, or as part of a broader programme of investigative studies.

In all cases, the procedures outlined are intended to be performed by an appropriately qualified concrete petrographer or, where specialist support procedures are used, an appropriately qualified materials technologist.

In certain cases, other analytical techniques, including but not limited to, chemical analysis, scanning electron microscopy and X-ray diffraction, might be desirable to augment the petrographical examination.

1 Scope

This British Standard describes procedures and terminology for the examination of hardened concrete and mortar. The procedures are intended to be performed by an appropriately qualified or experienced concrete petrographer.

The principal application of the method described requires the use of a polarizing petrological microscope for the examination of concretes containing Portland and other cements. The petrographic procedures outlined are applicable to the examination of samples of all types of hardened hydraulic cement-based materials, including concrete, mortar, grout, cement and gypsum-based plaster, render, terrazzo, and similar materials.

NOTE In this British Standard, the material for examination is designated as "concrete", even though the commentary is also applicable to the other mixtures, unless the reference specifically excludes a material other than concrete.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS EN 480-11, *Admixtures for concrete, mortar and grout -– Test methods – Part 11: Determination of air void characteristics in hardened concrete*

3 Terms and definitions

For the purposes of this British Standard, the following terms and definitions apply.

3.1 macroscopic examination
initial examination of the sample in hand specimen with the aid of a hand lens or stereomicroscope in order to determine the general features of the concrete

3.2 microscopic examination
examination of a prepared thin section using transmitted plane and crossed polarized light in order to provide microstructural information

3.3 thin section
slice of concrete impregnated with dyed resin and mounted on a glass slide

NOTE 1 The concrete is ground to a thickness of between 20 µm and 30 µm, and is sufficiently thin for light to be transmitted through it.

NOTE 2 Customarily thin sections have an area of (50 × 70) mm.

4 Principle

Petrographical examination involves the visual recognition of the constituents within the concrete to determine the condition of the various materials, to address concerns relevant to wider investigations of a structure, or to facilitate laboratory investigations. A petrographical examination is important to highlight features within the concrete that might already, or might later, influence the behaviour of the material under service conditions. A nomenclature is provided in Annex A.

5 Apparatus

5.1 Apparatus for the examination of samples

5.1.1 *Hand lens or stereoscopic microscope*, capable of magnifications up to × 40.

5.2 Apparatus for the petrographic examination of concrete samples

5.2.1 *Polarizing petrological microscope,* capable of high-resolution at magnifications up to × 500 and fitted with a digital camera and a means of calibrating the scale of images.

5.2.2 *Point-counting apparatus,* for use with the petrological microscope, e.g. an electrical or mechanical stage linked to a counting device, such that operation of any one of the counters automatically moves the thin section one further step along the traverse.

5.2.3 *Image analysis software (if required).*

5.3 Apparatus for detailed study

5.3.1 *Petrological microscope*, with appropriate filters and light source providing the ability to carry out fluorescence observations in transmitted light.

NOTE Fluorescence requires an ultraviolet light source with appropriate safety provisions.

5.3.2 *Petrological microscope*, with the facility to work in reflected as well as transmitted light.

NOTE For many manufacturers, objective lenses are designed specifically for this purpose.

5.3.3 *Air void characteristics measurement equipment,* for the measurement of the air void characteristics of hardened concrete in accordance with BS EN 480-11.

6 Sampling and preparation of test specimens

The number of specimens required depends on the purpose of the examination; for deterioration investigations, all regions of a structure or element that exhibit different types of distress or exposure conditions shall be sampled. Additionally, at least one control core specimen shall be taken from an adjacent region, in the same member, where no deterioration is visible. Ideally for quality assurance or quality control purposes, a sample shall be taken for a selected volume of the concrete produced for a project.

Suitable specimens are diamond-cut core samples, lump samples or cast cube/cylinder/prisms; the type of specimen submitted for examination shall depend on the nature of the task and the structure from which it is to be taken. The size shall be appropriate for the scope of work. Care shall be exercised if using specimens that might be too small to allow comparison of a chosen region of deterioration with that of surrounding non-deteriorated concrete.

For all specimens, the following information (where available) shall be recorded, either on the core/lump sample or on a sampling sheet:

a) unique site reference;

b) date of sampling;

c) photographs of the location of the core or cut specimen;

d) reason for sampling location;

e) age of structure;

f) exposure conditions

g) method employed to extract specimen;

h) vertically or horizontally cut specimen;

i) arrow on the profile of the specimen indicating direction towards outer face;

j) arrow on the end of the specimen indicating way-up on specimens from a vertical surface; and

k) mix design.

Specimens shall only be deemed fit for purpose if, when related to the scope of work, they are of sufficient size and number, are uncontaminated, and are from appropriate locations. Any deficiencies in the specimen sizes shall be recorded and the observations stated as preliminary or inconclusive. In such cases, it shall be recorded that the report relates specifically to the specimens received and that any extrapolation is clearly identified.

Care shall be taken when sampling not to wash out any deposits that might be present or induce any new cracking or damage to a specimen. The outer surface shall also be protected from damage during sampling as many features of interest are contained within this zone.

Specimens shall be wrapped in cling-film and placed in labelled sealed polythene bags before being placed into rigid containers suitable for secure transportation.

NOTE For early age concrete samples, it might be appropriate to wrap samples in moist paper towels placed in a sealed plastic bag to prevent premature drying of actively hydrating cement. This is not necessary for most concrete of 1 month or older.

Specimens shall be adequately padded and placed in a rigid container for shipping to prevent mechanical damage in transit.

7 Preparation of thin and polished thin sections

7.1 General

One or more thin sections shall be prepared depending on the size of the sample, the features observed and the purpose of the examination.

NOTE 1 Details of the technique needed to prepare a thin section is given in Annex B and supplementary techniques are given in Annex C.

A thin section is a portion of material mounted on a slide and mechanically reduced to a thin slice (0.025 ±0.005) mm in thickness, and normally protected by a slide cover. For special purposes (observations with reflected light microscope for the determination of opaque minerals or microprobe analysis), polished blocks, slabs, sections or polished thin sections shall be prepared. Polished blocks and polished thin sections shall have one side polished with alumina polishing paste (5 μm to 12 μm grade) and diamond paste (6 μm, 3 μm and 1 μm). The polished side shall remain uncovered.

The thin section shall typically measure approximately (70 × 50) mm, but larger or smaller sections may be used as appropriate.

NOTE 2 Smaller thin section sizes might require additional thin sections to be prepared to ensure that the total area of thin section available for study is representative of the sample.

7.2 Preparation of thin sections

*COMMENTARY ON **7.2***

This subclause provides a description of a method for thin section preparation commonly employed in the UK. However, many other procedures and types of equipment are available. For guidance on the apparatus and machinery to be used, see Annex D.

For all methods employed, the following precautions shall be taken.

a) Excessive heating involving temperatures of >45 °C shall be avoided during the thin section preparation.

b) Exposure to air shall be minimized as carbonation can take place very rapidly in freshly ground and polished concrete surfaces, and carbonation results in the loss of valuable information in the finished thin section.

c) Exposure to water shall be kept to a minimum in order to avoid secondary hydration and the loss of water-soluble compounds from the hardened cement paste.

7.3 Preparation of polished specimens

Large-area polished surfaces can be examined with a binocular microscope to obtain valuable information about the concrete. As large an area of the specimen as is possible shall be provided – ideally representing the full length of the core or area of cut surface and including the parts of the specimen not examined in thin section. The method of polishing shall typically involve the use of glass or cast iron lapping plates and abrasive slurry, finishing with a surface ground, and polished using alumina polishing pastes ending at 12 μm grade or finer.

8 Preliminary examination

8.1 Macroscopic examination

The specimens shall be examined with a binocular microscope as received and their dimensions and main features shall be recorded using photographs and drawings to appropriate scales. The features observed shall include the following:

a) the presence and position of reinforcement;

b) the extent to which reinforcement is corroded;

c) the nature of the external surfaces of the concrete;

d) the features and distribution of macro and fine cracks;

e) the distribution, size range and type of the aggregate;

f) the type and condition of the cement paste;

g) any superficial evidence of deleterious processes affecting the concrete; and

h) the nature of any exudations on surfaces, voids or in cracks.

8.2 Polished surfaces

The features recorded shall include the following:

a) the size, shape and distribution of coarse and fine aggregate;

b) the coherence, colour and porosity of the cement paste;

c) the distribution, size, shape and content of voids;

d) the composition of the concrete in terms of the volume proportions of coarse aggregate, fine aggregate, paste and voids;

e) the distribution and orientation of fine cracks and microcracks; and

NOTE A common procedure is to stain the surface with a penetrative dye, so that these cracks can be more easily seen. Microcrack frequency may be as measured along lines of traverse across the surface and the orientation of the traverses recorded.

f) the relative abundance of rock types in the coarse aggregate.

After a period of storage of at least 24 h in humid conditions, the sample shall be examined with the aid of a binocular microscope for the possible presence of gel or other exudations.

8.3 Broken surfaces

After the specially prepared surfaces and sections are completed, the remainder of the core shall be examined with a binocular microscope. In particular, the pieces shall be broken to produce fresh surfaces. These surfaces shall allow the contents of voids to be studied and the nature of aggregate surfaces or crack surfaces to be investigated.

9 Microscopic examination

The features recorded shall include the following:

- details of the rock types present in the coarse and fine aggregate and, in particular, structures seen within those components and their degree of weathering;
- details of the aggregate properties, such as the degree of strain in any quartz;

- the size, distribution and abundance of phases in the cement paste, including the occurrence of calcium hydroxide (portlandite) and the amount of residual unhydrated clinker;
- the presence of cement replacement phases, such as slag or fly ash; and

  NOTE 1 These can usually be recognized, and undispersed micro-silica can be identified and in some cases quantified (although the amounts of other phases cannot be accurately quantified). The presence of calcium aluminate cement can be detected and the type of cement clinker can commonly be identified.
- any products of deterioration of either the cement paste or the aggregate.

  NOTE 2 For example, alkali-silica reactivity (see BS 7943).

10 Methods for the estimation of the original water to cement ratio of concrete

10.1 Water/cement ratio

The water/cement ratio in hardened concrete may be estimated petrographically. The determination shall be made using control samples of similar composition and type to the sample under examination, and the petrographer shall be aware of the many factors that can hinder an accurate measurement of the water/cement ratio.

NOTE 1 Water-reducing admixtures can affect the quantities of unhydrated cement and portlandite crystal size and abundance. Leaching and other forms of concrete deterioration can affect paste porosity as well as the abundance of portlandite and unhydrated cement grains. Also, low temperatures slow the hydration and reduce crystal size, with a concomitant reduction in strength and increase in porosity.

NOTE 2 Despite the many complicating factors, the advantage of carrying out this determination petrographically is that it is usually possible to tell whether there are any additional factors that could contribute to the uncertainty in the measurement of water/cement ratio.

10.2 Petrographic assessment of the original water/cement ratio

The water/cement ratio (w/c) shall be estimated petrographically, provided the hardened concrete is unaffected by deterioration.

NOTE If the concrete is affected by secondary deterioration, it is unlikely that the water cement ratio can be estimated.

A petrographic assessment of the original water/cement ratio of a fluorescent dye-impregnated concrete shall be based on the following types of information.

- Comparison with reference concrete samples made with a known water/cement ratio.
- Assessment of the amounts of unhydrated cement. The number and proportion of unhydrated cement clinker particles varies inversely with the original water/cement ratio.
- Measurement of the porosity of the cement hydrates. The porosity of the cement hydrates increase with increasing water/cement ratio. With this method it is critical that impregnation with fluorescent dye is carefully controlled and that precautions are taken to avoid grinding away impregnated concrete prior to sectioning.
- The saturated surface-dry density of the concrete (see BS EN 12390-7) can be used to calculate the water/cement ratio of the concrete if the porosity of the aggregate can be reliably judged.

- The amount, size and distribution of calcium hydroxide in the paste. Concretes with a low water/cement ratio tend to develop only limited proportions of coarsely crystalline calcium hydroxide. The extent to which calcium hydroxide is separated into layers on aggregate surfaces and occurs in voids and on void surfaces varies with the original water/cement ratio.
- An assessment of the extent to which the porosity and portlandite distribution of the concrete has been modified since its placement.

11 Petrographic estimation of mix proportions

11.1 Measurement of volume proportions

A minimum area of (100 × 100) mm shall be used to measure the composition concrete containing coarse aggregate. For concrete and mortar containing fine aggregate and cement paste, thin sections or sections of (70 × 50) mm shall be used.

NOTE 1 The thin section is preferable, for example where large quantities of dust are present.

Air void content shall be assessed using a (100 × 100) mm slab in accordance with BS EN 480-11.

The volume proportions shall be measured by point counting using a mechanical stage (as described in the "modified point count procedure" in ASTM C457), or by appropriate automated methods. This method may be used to obtain information on the air void content and spacing factor, but if the full air void characteristics are required, the traverse line method in BS EN 480-11 shall be used. The amount of coarse aggregate may also be assessed by this means if a distinction can be made between coarse and fine aggregate.

NOTE 2 The results obtained usually represent the sample with reasonable accuracy, but might not represent the concrete.

11.2 Estimation of the composition of the concrete in terms of weight fractions

The composition of the concrete in terms of weight fractions shall be calculated from the measured volume proportions of aggregate and binder using assumed densities for the aggregates and binder and using the petrographically measured water/binder ratio. The method of calculation shall be as follows.

a) Petrographic measurement of the volume proportions of aggregate, paste and void.

b) Estimation of aggregate density from a petrographic identification of the rock types in the aggregate.

c) Petrographic measurement of water/binder ratio.

d) The aggregate content in kg/m^3 is given by:

Aggregate content (kg/m^3) = (aggregate density) × (vol.% of aggregate)

e) Cement content is given by the equation:

Cement content (kg/m^3) = (10 × paste vol.%) / [w/c + (1 000/cement density kg/m^3)]

NOTE The area measured should be of sufficient size to be representative of the composition of the sample.

An example of this calculation is given in Annex E.

12 Report

The report of the petrographical examination shall include:

a) unique identification number for the report;

b) number, title and date of issue of this British Standard;

c) name and address of the test laboratory;

d) name and address of the client;

e) any pertinent information regarding the structure or history or usage of the structure;

f) name of the person or organization that carried out the sampling;

g) date of delivery of the specimen;

h) date(s) of preparation of the thin sections and the date of the examination;

i) number and dimensions of the thin sections;

j) macroscopic and microscopic description of the concrete;

k) photography of the thin section under the following light conditions as appropriate:

   1) crossed polarized; and

   2) plain polarized light;

l) any deviation from this British Standard and the reasons for any such deviation;

m) remarks – to comprise all observed features of the concrete, including mineralogy, texture, fabric and relative proportions of ingredients;

   NOTE 1 An example of a template suitable for the detailed description of a specimen is given in Annex F.

   NOTE 2 In addition, attention should be drawn to those features that might indicate mixing, placement and finishing practices, concrete quality and performance under service conditions.

n) glossary of descriptive terms used; and

o) if requested by the client, interpretation of the nature of the concrete ingredients, the original concrete mix proportions, workmanship and construction practices, and the type and causes of concrete deterioration or distress during its service life may be attempted, based on the features observed. In addition to the petrographic examination, this may make reference to desk study information, site observations and/or results of physical and chemical tests made on the samples (if such information is made available by the client). Recommendations based on such interpretations may be given, if applicable, and requested by the client. Recommendations are typically supplemented by guidance from concrete technologists and engineers.

Annex A (informative)

Nomenclature

The following list is intended to provide explanations of simple petrographic terms most commonly used in concrete petrography.

- *Addition:* Mineral additions include fly ash, ground granulated blastfurnace slag (ggbs) and silica fume. The addition of finely divided material used in concrete in small quantities in order to improve certain properties or to achieve special properties. These include nearly inert additions and pozzolanic or latent hydraulic additions.
- *Admixture:* Material added during the mixing process in a quantity not more than 5% by mass of the cement content to modify the properties of the mix in the fresh and/or hardened state. Further information on the types of admixtures can be found in BS EN 934 (all parts). Admixtures are not generally detected by thin section examination but the effects can be observed, as in the case of air entraining admixtures.
- *Aggregate:* Aggregate forms between 60% – 80% by volume of concrete and comprises a fine fraction (typically <5 mm), commonly of natural sand or crushed rock, and a coarse fraction comprising gravel or crushed rock (typically 10 mm – 20 mm). Artificial aggregates are used in lighter weight concretes and are a by-product of industrial processes. Recycled aggregate is derived from material previously used in construction.
- *Alite:* An impure tricalcium silicate, termed C_3S in the Bogue Notation, with a composition approximating $3CaO.SiO_2$ forming euhedral pseudo hexagonal crystals when unhydrated.
- *Alkali Aggregate Reaction (AAR):* This is a broad term reflecting chemical exchanges between particular minerals within the aggregate particles and compounds of alkali metals, typically sodium and potassium, present in the cement paste and in pore fluids. The more commonly observed Alkali Silica Reaction and Alkali Carbonate Reaction are included here.
- *Alkali Carbonate Reaction (ACR)*: This is a process that results in the formation of a "de-dolomitized" rim developing around the aggregate. In some examples, expansion of the concrete has been reported but the exact cause is unclear.
- *Alkali Silica Reaction (ASR)*: This is the more widely reported of alkali aggregate reactions and results from the reaction between aggregate containing various forms of silica and the alkalis within cement paste. In the UK, chalcedonic silica containing microcrystalline quartz and highly reactive opaline silica are the most common contributors to ASR. It results in cracking of the aggregate, the formation of gel and subsequent cracking of concrete. (See BRE Digest 330 [2] for further information.)
- *Anhydrous Portland cement phases*: Portland cement contains four principal phases, tricalcium silicate, β-dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite.
- *Autogenous shrinkage*: This shrinkage occurs during the hydration and hardening of the cement and is independent of environmental factors. It is a result of the chemical hydration producing a lesser volume than the anhydrous clinker plus water, and self-dessication resulting from the consumption of water during the hydration process. Microcracks develop at the interfacial zone around aggregate particles and in the cement paste between aggregate particles. It is most common in high strength or high performance concrete with low water/cement ratios.

- *Belite* (and felite): An impure dicalcium, termed C_2S in the Bogue Notation, with a composition approximating to $2CaO.SiO_2$. It is an important constituent of cement clinker with unhydrated sub-rounded grains commonly occurring in clusters.
- *Calcium Aluminate Cement (CAC)*: A broad term for the range of calcium aluminate cements that include rapidly setting repairs and grout concretes. Calcium aluminate cement and Ciment Fondu are manufactured by fusing low silica bauxite and limestone rather than shale or clay and limestone. Previously known as High Alumina Cement (HAC).
- *Carbonation*: Within a Portland type cement paste, this results from a reaction between the calcium hydroxide and cement hydrates with atmospheric carbon dioxide. In general terms it results in the formation of carbonate. Within high alumina or calcium aluminate cement concretes (HAC/CAC) a reaction with atmospheric carbon dioxide results in the formation of $CaCO_3$ and $Al(OH)_3$.
- *Cement (hydraulic binder):* Refers to finely ground inorganic material which, when mixed with water, forms a paste that sets and hardens by means of hydration reactions and processes and which, after hardening, retains its strength and stability even under water.
- *Chalcedony:* Chalcedony typically consists of 90% – 99% SiO_2, with a cryptocrystalline or fibrous structure where fibres are perpendicular to the free wall. Some claim chalcedony to be a mixture of amorphous and cryptocrystalline silica resulting from reaction in alkaline solutions.
- *Chert:* This is a general term for fine-grained silica deposits of organic biochemical, biogenic, volcanic or hydrothermal origin. Within the UK microcrystalline and cryptocrystalline silica commonly form nodules and layers within carbonate successions. Flint is similar to chert and the term is most specifically used for the nodules of microcrystalline silica found within Cretaceous Chalk.
- *Cracks:* Cracks can be open, closed or filled discontinuities in concrete, resulting after placement and can develop at any stage through the life of the concrete. The terms for crack width are by convention as follows: large cracks >1 mm wide, cracks 1 mm – 100 μm, fine cracks 100 μm – 10 μm, microcracks 10 μm – 1 μm and fine microcracks <1 μm.
- *Delayed Ettringite Formation* (DEF): This results from the initial suppression of ettringite formation and its subsequent expansive precipitation in moist conditions. It occurs within concrete where curing has been at elevated temperatures (65 °C – 75 °C). Ettringite characteristically forms continuous rims around aggregate particles.
- *Drying shrinkage cracks:* As the term suggests, these result from a volume loss in the concrete mass after hardening. Typically these radiate from aggregate surfaces and commonly form a rectilinear pattern that includes surface crazing. Cracks tend to have sharp well-defined linear margins.
- *Ettringite:* Calcium sulfoaluminate hydrate, $Ca_6Al_2(SO_4)_3.(OH)_{12}.26H_2O$, is a very common mineral within concrete. It is found in voids as a secondary precipitate resulting from leaching in moist conditions. Generally, ettringite in this form does not cause concrete deterioration. Sulfate attack resulting from ettringite precipitation is relatively rare, but can cause significant cracking and concrete deterioration.
- *Gibbsite*: Aluminium hydroxide, $Al(OH)_3$, is a mineral that occurs in bauxite, laterite and aluminous clays, and forms as a hydration product in high alumina cement.

- *Gypsum*: Calcium sulfate dihydrate, $(CaSO_4).2H_2O$, most commonly forms by the evaporation of seawater in a range of environments. It causes deterioration in renders, mortars and concrete where these are exposed to sulfate bearing solutions, including seawater.
- *High Alumina Cement (HAC)*: See Calcium Aluminate Cement (CAC).
- *Hydrated cement paste:* The four main cement phases, C_3S (alite), C_2S (belite), C_3A (calcium aluminates) and C_4AF (ferrite), are hydraulic and when mixed with water form a range of hydrated phases and calcium hydroxide. The hydrated phases are generally termed calcium silicate hydrates or CSH and calcium aluminate hydrates or CAH.
- *Leaching*: Refers to the removal of compounds in solution. In Portland cement concrete such leaching can result in the depletion of Portlandite and other soluble components in cement paste.
- *Mortar*: A material used as a jointing, plastering or rendering agent, comprising fine aggregate, that can be natural sand and/or a synthetic material, and a bonding agent. Proprietary products may not all use cement as a binder.
- *Petrography:* This term refers to the detailed and systematic description of rocks in hand specimen and in thin section. Concrete petrography similarly relies on detailed observation of the material to determine the components, textures, and the presence of inadequate, deleterious or incompatible phases which can affect service performance.
- *Plastic shrinkage cracks:* Plastic shrinkage cracks develop before concrete hardening and commonly skirt aggregate particles. When viewed in cross section they taper from the outer surface inwards.
- *Pleochroite:* This fibrous, pleochroic phase is characteristic of Calcium Aluminate Cement (CAC) and is a complex solid solution containing calcium, aluminum, silica and magnesia with ferric and ferrous iron substitution.
- *Portlandite*: Portlandite, otherwise calcium hydroxide $Ca(OH)_2$, is a readily recognizable product of cement hydration. In general terms it forms coarser crystals in concrete with higher water content.
- *Pozzolana (pozzolanic materials):* Named after volcanic dust from Pozzuoli (Italy) this siliceous or siliceous and aluminous material, when finely ground, reacts with calcium hydroxide in the presence of water to form hydraulic compounds.
- *Sulfate attack:* This general term encompasses deterioration caused by the expansive precipitation of the sulfate minerals ettringite, gypsum and thaumasite.
- *Thaumasite:* Calcium carbonate silicate sulfate hydrate $[Ca_3Si(OH)_6.12H_2O](SO_4)(CO_3)$ forms a partial solid solution with ettringite, resulting in mixed ettringite/thaumasite crystals. The crystals form in response to cold wet conditions in concrete where external calcium silicate, calcium ions and sulfate ions are available. Typically, the temperatures favoured for formation are <15 °C and development has been recorded in buried concrete structures. Thaumasite might not always be readily distinguished from ettringite and ancillary XRD and SEM could be required. Thaumasite form of sulfate attack (TSA) results in the growth of thaumasite at the expense of the cement paste and leads to deterioration of the concrete.
- *Voids:* The term encompasses entrained air voids (<1 mm) and larger spaces with entrapped air that are usually present in concrete. Air voids can be intentionally entrained within a concrete mix with the use of an air entraining agent.

- *Water/cement ratio:* This refers to the ratio of the effective water content to cement content by mass in the fresh concrete.

Annex B (informative) Thin section preparation technique

B.1 Preliminary impregnation and cutting

In most cases it is possible to carry out initial cutting using a water-lubricated large-diameter diamond saw. However, in the case of very weak or friable samples such those affected by fire damage or sulfate attack, the concrete surface should be vacuum impregnated with resin in order to consolidate and stabilize it prior to any cutting being carried out. It is desirable that the final stages of cutting are carried out using a precision small-diameter saw in order to minimize the damage in the cut surface that needs to be removed before mounting on to a glass microscope slide.

B.2 Impregnation

In order to produce high-quality polished surfaces from concrete – particularly if this is very porous – the sample should be vacuum impregnated with a low-viscosity epoxy resin prior to polishing and grinding. The use of a fluorescent dye is recommended in order to assist the determination of primary porosity and microcracking. Coloured dyes can be useful for this purpose. The most effective means of impregnation is to place the sample in a vacuum chamber and evacuate before introducing the impregnating resin. The vacuum is released before the resin sets so that it is forced into the specimen as the external pressure rises.

B.3 Initial lapping

Prior to mounting the concrete specimen onto a glass slide, it is necessary to remove the damage produced in the surface of the sample during cutting. This is generally done using a combination of grinding and lapping to produce a high-quality optically flat surface that can be bonded on to the glass microscope slide.

B.4 Mounting onto glass slides

The flattened specimen should be fully cleaned, preferably using an ultrasonic cleaning bath and a solvent such as petroleum spirit. The polished surface should then be wiped over with a soft tissue using a solvent such as methylated spirits or acetone. The cleaned surface is then bonded on to a frosted glass slide using a UV-cured adhesive. It is important in mounting the specimen on to the glass that the thickness of the bond is controlled and is kept to a minimum beneath the specimen.

B.5 Removal of excess material

Once it is bonded on to the glass, the specimen is ready for the excess concrete to be cut off. This is done using a precision oil-lubricated diamond saw and when complete should leave a section thickness of the order of 1 mm.

B.6 Final lapping

The thin sample is then ground down in stages to a thickness of approximately 150 µm to 200 µm using diamond surface-grinding equipment lubricated by oil. Further lapping using a precision vacuum chuck is used to take the section to a thickness of about 40 µm. Typically the section is then finished by hand lapping. With some types of equipment it is possible to take the thin section down to its final thickness using grinding wheels impregnated with very fine diamonds.

B.7 Hand finishing

Using a petrological microscope to optically estimate the thickness, the thin section can be hand finished down to its final thickness of 25 µm – 30 µm. The birefringence of quartz present in the sample commonly provides a convenient way of judging the thickness of the thin section during this process.

B.8 Covering

The final section should be thoroughly cleaned on completion and then covered using a glass cover slip. This is to prevent carbonation and damage to the sample after preparation and is also important to reduce light scattering during the examination of the thin section.

Annex C (informative) Supplementary techniques

C.1 General

The techniques in this annex are optional, and their omission does not detract from many petrographic examinations. The principles of each technique are outlined and the potential enhancements that they offer some petrographic examinations are outlined.

C.2 X-ray diffraction

X-ray diffraction (XRD) is a procedure for the identification of crystalline components in a powdered sample. In controlled situations it can also provide semi-quantitative information on the crystalline components, and indicate the approximate levels of non-crystalline materials such as calcium silicate hydrates (CSH). It augments optical microscopy, which is limited by the small size and uneven distribution of hydrate minerals. The powdered paste samples should be protected from excessive heat or atmospheric carbonation. The technique is particularly valuable for the identification of sub-microscopic deleterious minerals.

C.3 Scanning electron microscopy

Scanning electron microscopy (SEM) uses an electron beam to image the sample and provides resolution of small components far greater than that accessible by optical microscopy. Higher magnifications, e.g. up to × 100 000 can be utilized for small-scale detailed examinations of specific regions of interest. Interactions between the electron beam and the sample produces images from backscattered and secondary electrons, plus X-rays. These different emissions can be separately detected to provide elemental contrast, topography and elemental chemical analysis [energy dispersive X-ray spectroscopy (EDX)] respectively. With properly prepared samples and with experienced analysis, the microstructure (type quantity and distribution of all components, including porosity) can be assessed.

Typical uses of SEM/EDX include porosity and Ca/Si ratio assessment of the paste, detailed examination of the aggregate/paste interfacial region, secondary deposits in cracks and pores, mapping of elements such as Na, K, S and Mg, and the chemical composition of regions of paste or discrete particles that might have a bearing on chemical deterioration and concrete durability.

C.4 Chemical analysis

Chemical analysis is commonly a supplemental procedure but it is essential to provide data relating to the initial and potentially altered chemical composition of the concrete in question. This can be useful both in terms of original mix verification and for potential durability issues such as corrosion of reinforcing bars or sulfate attack. A variety of methods can be used for chemical analysis and their descriptions/benefits might be found in other publications.

Physical testing data are also useful, providing information on the strength of the concrete, potentially from different areas of a structure that represent sound and suspect regions.

Annex D (informative) Apparatus and machinery for the preparation of thin sections

D.1 Diamond saws

Two diamond saws are desirable, one with a large-diameter cutting wheel (~500 mm diameter) and one with a small-diameter cutting wheel (<300 mm diameter). A narrow-bladed (<1 mm thick) oil-lubricated precision trim saw is also useful for cutting fragile specimens and those that might contain water-soluble components, but also for cutting off flattened concrete specimens mounted onto glass slides.

D.2 Vacuum impregnation equipment

A vacuum chamber capable of containing large concrete samples up to 5 kg with a means of introducing epoxy resin after the sample is evacuated should be used.

D.3 Polishing, lapping and grinding equipment

A very wide range of grinding and polishing equipment is available. The following types are in common use in the UK, but other equally suitable equipment is in use elsewhere in Europe and the USA. A petrographic preparation laboratory should have a combination of the following items of equipment.

- High-speed vertical-spindle grinding wheel with diamond abrasive bonded in brass that can be oil- or water-lubricated. This can be used for rapid grinding, and fine-diamond grinding wheels lubricated with oil are suitable for flattening impregnated concrete specimens prior to polishing.
- Lapping machines fitted with resin-bonded diamond abrasive pads. These can be used in the same way as the high-speed vertical-spindle grinding wheel. The coarser grades are suitable for rapid stock removal with the finer grades suitable for polishing. They can be used with either oil or water lubricants.
- Lapping machines with cast iron lapping plates and with a controlled abrasive slurry feed. Common abrasives include carborundum and aluminium oxide. Both oil and water form suitable grinding media. Water-lubricated laps are suitable for the production of large-area polished plates, and oil-lubricated laps can be used to flatten impregnated concrete specimens prior to mounting on glass for thin sectioning but also to retain water soluble components particularly for subsequent chemical analysis of the concrete.
- Vacuum chucks used in conjunction with an oil-lubricated lapping machine for the controlled grinding of thin sections to thicknesses down to about 40 μm.

- Plate glass sheet to be used for final hand finishing of thin sections using an oil/carborundum abrasive slurry.
- Polishing machines with felt pads for use with abrasive diamond pastes for polishing to a mirror finish suitable for reflected light examination and for quantitative SEM microanalysis.

D.4 Ovens and heating plates

Elevated temperatures of curing are required for some types of epoxy resin. The curing of such resins should be carried out at temperatures not exceeding 45 °C. Ovens and plates of this type are also suitable for drying specimens prior to vacuum impregnation with epoxy resin. Electric heating plates facilitate curing but should not exceed 45 °C.

D.5 Cleaning equipment

An ultrasonic cleaning bath is useful for cleaning impregnated and polished surfaces.

D.6 Consumables

Common materials used in the UK for the preparation of the concrete thin sections include the following.

- Low-viscosity epoxy resin for vacuum impregnation of concrete. Some resins cure exothermically and need to be kept cool during curing if used in large volumes. Some need slightly elevated temperatures (not more than 45 °C) in order to cure.
- Fluorescent dye that can be dissolved in epoxy resin.
- Coloured dyes that can be dissolved in epoxy resin.
- Solvents for cleaning purposes, such as petroleum spirit, acetone and methylated spirits.
- Coolant other than water, such as cutting oil.
- Ultraviolet-curing adhesive for mounting and covering thin sections.
- Carborundum abrasive of various grades. One of the most commonly used grades is F600.

NOTE General guidance on the equipment needed for making geological thin sections (that are essentially similar to those involving concrete) and the various procedures to be employed is given in Allman and Lawrence, 1972 [3]. This is a procedure requiring considerable skill and experience.

Annex E (informative) Example of the method of calculation of concrete composition from volume proportions measured by point counting

a) Results of point counting (excluding voids) and petrographic examination for water/cement ratio:
   - coarse aggregate: 40%;
   - fine aggregate 30%;
   - paste: 30%; and
   - petrographically measured water/cement (w/c) ratio: 0.50.

b) Assumed densities based on the petrographic identification of the aggregate types:

- coarse aggregate density (siliceous gravel): 2 620 kg/m^3;
- fine aggregate density (siliceous sand): 2 620 kg/m^3; and
- cement density assuming Portland cement: 3 140 kg/m^3.

c) Calculation of aggregate contents:

Equation 1: Aggregate content (kg/m^3) = (aggregate density) × (vol.% of aggregate)

Using the above formula gives coarse and fine aggregate contents of 1 048 kg/m^3 and 786 kg/m^3 respectively.

d) Calculation of cement content:

Equation 2: Cement content (kg/m^3) = (10 × paste vol.%) / [w/c + (1 000/cement density kg/m^3)]

The above example gives a cement content of 367 kg/m^3.

e) Calculation of water content:

Equation 3: Water content (kg/m^3) = w/c ratio × cement content kg/m^3.

The above example gives a water content of 183 kg/m^3.

Annex F
(informative)

Example of a report template for the detailed description of the concrete specimen

Sample no.		Lab ref.	
As-received details	Sample description		
	Dimensions		
	Sample surface condition		
	Comments		
Sample photograph			
Sample sketch			

Sample no.		Lab ref.	
Aggregate details	Coarse aggregate	Major	
		Minor	
		Trace	
		Cement/ aggregate bond	
		Remarks	
	Fine aggregate	Major	
		Minor	
		Trace	
		Cement/ aggregate bond	
		Remarks	
Concrete features	Apparent cement type and matrix details		
	Mineral additions		
	Air void details and compaction		
	Microporosity and apparent water/cement ratio		
	Carbonation		
	Portlandite		
	Other concrete details		
	Reinforcement		
	Evidence of cracking		
	Exudations and deposits		
	Other		

Bibliography

Standards publications

For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ASTM C457, *Standard test method for microscopical determination of parameters of the air-void system in hardened concrete*

BS 7943, *Guide to the interpretation of petrographical examinations for alkali–silica reactivity*

BS EN 934 (all parts), *Admixtures for concrete, mortar and grout*

BS EN 12390-7, *Testing hardened concrete – Part 7: Density of hardened concrete*

Other publications

[1] APPLIED PETROGRAPHY GROUP. *Special Report 2. A code of practice for the petrographic examination of concrete.* London: APG, July 2010. [1)]

[2] BUILDING RESEARCH ESTABLISHMENT. *Digest 330. Alkali-silica reaction in concrete.* Watford: BRE, August 2004.

[3] ALLMAN, M. and LAWRENCE, D.F. *Geological laboratory techniques*. Dorset: Blandford Press, 1972.

Further reading

ASTM C856-14, *Standard practice for petrographic examination of hardened concrete.*

CONCRETE SOCIETY. *Technical Report 32. Analysis of hardened concrete: a guide to tests, procedures and interpretation of results.* Camberley: The Concrete Society, 2014.

CONCRETE SOCIETY. *Technical Report 71. Concrete petrography: an introductory guide for the non-specialist (second edition).* Camberley: The Concrete Society, 2010.

[1)] Available online at: <http://www.appliedpetrographygroup.com/files/code_of_practice_concrete.pdf> [last viewed 7 November 2016].

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