Designation: C856 – 14

Standard Practice for Petrographic Examination of Hardened Concrete[1]

This standard is issued under the fixed designation C856; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This practice outlines procedures for the petrographic examination of samples of hardened concrete. The samples examined may be taken from concrete constructions, they may be concrete products or portions thereof, or they may be concrete or mortar specimens that have been exposed in natural environments, or to simulated service conditions, or subjected to laboratory tests. The phrase "concrete constructions" is intended to include all sorts of objects, units, or structures that have been built of hydraulic cement concrete.

NOTE 1—A photographic chart of materials, phenomena, and reaction products discussed in Sections 8 – 13 and Tables 1-6 are available as Adjunct C856 (ADJCO856).

1.2 The petrographic procedures outlined herein are applicable to the examination of samples of all types of hardened hydraulic-cement mixtures, including concrete, mortar, grout, plaster, stucco, terrazzo, and the like. In this practice, the material for examination is designated as "concrete," even though the commentary may be applicable to the other mixtures, unless the reference is specifically to media other than concrete.

NOTE 2—Appendix X1 outlines an uranyl acetate method for identifying locations where alkali-silica gel may be present. It is a requirement that the substances in those locations must be identified using any other more definitive techniques, such as petrographic microscopy.

1.3 The purposes of and procedures for petrographic examination of hardened concrete are given in the following sections:

	Section
Qualifications of Petrographers and Use of Technicians	4
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1.4 The values stated in inch-pound units are to be regarded as the standard. The SI units in parentheses are provided for information purposes only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* A specific hazard statement is given in 6.2.10.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C125 Terminology Relating to Concrete and Concrete Aggregates

C215 Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens

C227 Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)

C342 Test Method for Potential Volume Change of Cement-Aggregate Combinations (Withdrawn 2001)[3]

C441 Test Method for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction

C452 Test Method for Potential Expansion of Portland-Cement Mortars Exposed to Sulfate

C457 Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete

C496/C496M Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens

C597 Test Method for Pulse Velocity Through Concrete

C803/C803M Test Method for Penetration Resistance of Hardened Concrete

C805 Test Method for Rebound Number of Hardened Concrete

C823 Practice for Examination and Sampling of Hardened Concrete in Constructions

[1] This practice is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.65 on Petrography.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

***A Summary of Changes section appears at the end of this standard**

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TABLE 1 Visual Examination of Concrete (1)[5]

Coarse Aggregate	+ Fine Aggregate	+ Matrix	+ Air	+ Embedded Items
Composition:				
Maximum dimension,[A] in. or mm, in the range> *d*>				
Type: 1 Gravel 2 Crushed stone 3 Mixed 1 and 2 4 Other (name) 5 Mixed 1 + /or 2 + /or 4 If Type 1, 2, or 4, homogeneous or heterogeneous	Type: 1 Natural sand 2 Manufactured sand 3 Mixed 4 Other (name) 5 Mixed 1 + /or 2 + /or 4 If Type 1, 2, or 4, homogeneous or heterogeneous	color, by comparison with National Research Council *Rock Color Chart* (1963) color distribution: 1 mottled 2 even 3 gradational changes	more than 3 % of total, predominantly in spherical voids? less than 3 % of total, abundant nonspherical voids? color differences between voids and mortar?	Type, size, location; kinds of metal; other items
Lithologic types Coarse aggregate more than 20, 30, 40, or 50 % of total			voids empty, filled, lined, or partly filled	
Fabric:				
Shape Distribution Packing Grading (even, uneven, excess, or deficiency of size or sizes)	distribution particle shape grading preferred orientation } as perceptible	distribution	shape distribution grading (as perceptible) parallelism of long axes of irregular voids or sheets of voids: with each other; with flat sides or long axes of coarse aggregate	voids below horizontal or low-angle reinforcement
Parallelism of flat sides or long axes of exposed sections, normal to direction of placement + /or parallel to formed and finished surfaces[B]				
Condition: Does it ring when hit lightly with a hammer or give a dull flat sound? Can you break it with your fingers? Cracks? How distributed? Through or around coarse aggregate? With cores or sawed specimens, did the aggregate tear in drilling or sawing? Crack fillings? Surface deposits? If air dry, are there unusually wet or dry looking areas? Rims on aggregate?				clean or corroded? Are cracks associated with embedded items?

[A] A substantial portion of the coarse aggregate has maximum dimensions in the range shown as measured on sawed or broken surfaces.
[B] Sections sawed or drilled close to and parallel to formed surfaces appear to show local turbulence as a result of spading or rodding close to the form. Sections sawed in the plane of bedding (normal to the direction of placement) are likely to have inconspicuous orientation. Sections broken normal to placement in conventionally placed concrete with normal bond tend to have aggregate knobs abundant on the bottom of the upper piece as cast and sockets abundant on the top of the lower piece as cast.

C1012 Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution
C1260 Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method)
E3 Guide for Preparation of Metallographic Specimens
E883 Guide for Reflected–Light Photomicrography

2.2 *ASTM Adjuncts:*
Adjunct C856 (ADJCO856) A chart of 27 photos[4]

3. Terminology

3.1 *Definitions:*

For definitions of terms used in this practice, refer to Terminology C125.

[4] Available from ASTM International Headquarters. Order Adjunct No. ADJC0856. Original adjunct produced in 1995.

4. Qualifications of Petrographers and Use of Technicians

4.1 All petrographic examinations of hardened concrete described in this practice shall be performed by or under the technical direction of a full time supervising petrographer with at least 5 years experience in petrographic examinations of concrete and concrete-making materials. The supervising concrete petrographer shall have college level courses that include petrography, mineralogy, and optical mineralogy, or 5 years of documented equivalent experience, and experience in their application to evaluations of concrete-making materials and concrete products in which they are used and in cementitious-based materials. A resume of the professional background and qualifications of all concrete petrographers shall be available.

4.2 A concrete petrographer shall be knowledgeable about the following: concrete-making materials; processes of

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TABLE 2 Outline for Examination of Concrete with a Stereomicroscope (1)

NOTE 1—*Condition*—When it is examined at 6 to 10× under good light, the freshly broken surface of a concrete in good physical condition that still retains most of its natural moisture content has a luster that in mineralogical terms is subtranslucent glimmering vitreous.[A] Thin edges of splinters of the paste transmit light; reflections appear to come from many minute points on the surface, and the quality of luster is like that from broken glass but less intense. Concrete in less good physical condition is more opaque on a freshly broken surface, and the luster is dull, subvitreous going toward chalky. A properly cured laboratory specimen from a concrete mixture of normal proportions cured 28 days that has shown normal compressive or flexural strength and that is broken with a hammer and examined on a new break within a week of the time that it finished curing should provide an example of concrete in good physical condition.

Under the same conditions of examination, when there is reasonable assurance that the concrete does not contain white portland cement or slag cement, the color of the matrix of concrete in good physical condition is definitely gray or definitely tan, except adjoining old cracks or original surfaces.

Coarse Aggregate	Fine Aggregate	Matrix	Voids
Lithologic types and mineralogy as perceptible	Lithologic types and mineralogy as perceptible	Color	Grading
Surface texture	Shape	Fracture around or through aggregate	Proportion of spherical to nonspherical
Within the piece:	Surface texture	Contact of matrix with aggregate:	Nonspherical, ellipsoidal, irregular, disk-shaped
Grain shape	Grading	close, no opening visible on sawed or broken surface; aggregate not dislodged with fingers or probe; boundary openings frequent, common, rare	Color change from interior surface to matrix
Grain size extreme range observed, mm	Distribution	Width	Interior surface luster like rest of matrix, dull, shining
Median within range _ to _ mm		Empty	Linings in voids absent, rare, common, in most, complete, partial, colorless, colored, silky tufts, hexagonal tablets, gel, other
Textureless (too fine to resolve)		Filled	Underside voids or sheets of voids uncommon, small, common, abundant
Uniform or variable within the piece		Cracks present, absent, result of specimen preparation, preceding specimen preparation	
From piece to piece:		Supplementary Cementitious Materials[C]	
Intergranular bond		Contamination	
Porosity and absorption[B]		Bleeding	
If concrete breaks through aggregate, through how much of what kind?			
If boundary voids, along what kind of aggregate? All? All of one kind? More than 50 % of one kind? Several kinds?			
Segregation			

[A] Dana, E. S., *Textbook of Mineralogy,* revised by W. E. Ford, John Wiley & Sons, New York, N. Y., 4th ed., 1932, pp. 273–274.
[B] Pore visible to the naked eye, or at × _, or sucks in water that is dropped on it.
[C] Dark solid spheres or hollow-centered spheres of glass, or of magnetite, or some of glass and some of magnetite, recognizable at magnification of × 9 on sawed or broken surfaces. Other mineral admixtures with characteristic particles visible at low magnification are recognizable. Ground surface of concrete containing portland blast-furnace slag cement are unusually white near-free surfaces but retain greenish or blue-greenish patches, and slag particles can be seen with the stereomicroscope or polarizing microscope.

batching, mixing, handling, placing, and finishing of hydraulic-cement concrete; the composition and microstructure of cementitious paste; the interaction of constituents of concrete; and the effects of exposure of such concrete to a wide variety of conditions of service.

4.3 Sample preparation shall be performed by concrete petrographers or trained technicians pursuant to instructions from and under the guidance of a qualified concrete petrographer. Aspects of the petrographic examination, such as the measurement of sample dimensions, photography of as-received samples, staining of sample surfaces, that do not require the education and skills outlined in 4.1, shall be performed by concrete petrographers or by trained technicians pursuant to instructions and under the guidance of a qualified concrete petrographer. The analysis and interpretation of the features that are relevant to the investigation and evaluation of the performance of the materials represented by the sample shall be made solely by concrete petrographers with qualifications consistent with those outlined in 4.1.

4.4 A concrete petrographer shall be prepared to provide an oral statement, written report, or both that includes a description of the observations and examinations made during the petrographic examinations, and interpretation of the findings insofar as they relate to the concerns of the person or agency for whom the examination was performed. Supplementary information provided to the petrographer on the concrete and concrete materials, conditions of service, or other features of the concrete construction may be helpful in interpreting the data obtained during the petrographic examinations.

4.5 This practice may form the basis for establishing arrangements between a purchaser of the consulting service and the consulting petrographer. In such cases, the purchaser of the consulting service and the consulting petrographer should together determine the kind, extent, and objectives of the examinations and analyses to be made, and may record their agreement in writing. The agreement may stipulate specific determinations to be made, observations to be reported, funds to be obligated, or a combination of these and other conditions.

5. Purposes of Examination

5.1 Examples of purposes for which petrographic examination of concrete is used are given in 5.2 – 5.5. The probable usefulness of petrographic examination in specific instances may be determined by discussion with an experienced petrographer of the objectives of the investigation proposed or underway.

5.2 *Concrete from Constructions:*

5.2.1 Determination in detail of the condition of concrete in a construction.

5.2.2 Determination of the causes of inferior quality, distress, or deterioration of concrete in a construction.

5.2.3 Determination of the probable future performance of the concrete.

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TABLE 3 Effects of Fire on Characteristics of Concrete

Characteristic	Causes and Effects	Ways of Investigation
Surface hardness	Dehydration to 100°C removes free water; dehydration is essentially complete at 540°C; calcium hydroxide goes to CaO at 450–500°C. Paste expands with thermal coefficient effect and then shrinks, cracks, decrepitates, and becomes soft **(2)**.	Beneath the softened concrete, which can be tested in accordance with Test Method C805, the concrete is probably normal if it has not undergone color change. Establish by coring for compressive tests, by wear tests (CRD-C 52) **(2)**, and by scratching with a knife.
Cracking	Perpendicular to the face and internal, where heating or cooling caused excess tensile stresses. In some new concrete, resembles large-scale shrinkage cracking; may penetrate up to 100 mm but may heal autogenously **(2)**.	Examination of the surface, ultrasonic tests, coring, petrographic examination **(2)**.
Color change—When concrete has not spalled, observe depth of pink color to estimate the fire exposure.	Concrete made with sedimentary or metamorphic aggregates shows permanent color change on heating. Color normal to 230°C; goes from pink to red from 290 to 590°C; from 590 to 900°C color changes to gray and then to buff **(2)**. For temperatures up to about 500°C temperature distribution is little affected by using carbonate rather than siliceous aggregate **(3)**. At 573°C low quartz inverts to high with 0.85 % increase in volume, producing popouts. Spalling over steel to expose one fourth of the bar at 790°C; white powdered decomposed hydration products at 900°C. Surface crazing about 290°C; deeper cracking about 540°C.	Color change is the factor most useful to the investigator; permits recognizing how deeply a temperature of about 300°C occurred **(3)**.
Aggregate behavior—Aggregate behavior affects strength, modulus, spalling, cracking, surface hardness, and residual thermal strains **(2)**.	Aggregates differ in thermal diffusivity, conductivity, coefficient of expansion. Heat transmission decreases from concrete made with highly siliceous aggregate, sandstone, traprock, limestone, lightweight aggregates **(2)**.	Changes on heating are often accompanied by volume change **(2).**
Spalling	Occurs subparallel to free face; followed by breaking off saucer-like pieces especially at corners and edges **(2)**.	
Note: Compressive strength and elastic modulus. For concrete at least 1-year old, strength will increase after cooling from 300°C if design strength was attained **(3)**.	Reduction in strength of concrete containing siliceous gravel after heating, then cooling and testing: Heated to Temperature ° C / Reduction, % 180 / 25 370 / 50 570 / 80 Reduction in Modulus Temperature,° C / Reduction, % 200 / 25 430 / 50 760 / 70	Determinations by compressive tests and static modulus of cores; Test Method C805 for qualitative determination; Test Method C597**(2)**.

5.2.4 Determination whether the concrete in a construction was or was not as specified. In this case, other tests may be required in conjunction with petrographic examination.

5.2.5 Description of the cementitious matrix, including qualitative determination of the kind of hydraulic binder used, degree of hydration, degree of carbonation if present, evidence of unsoundness of the cement, presence of supplementary cementitious materials, the nature of the hydration products, adequacy of curing, and unusually high water–cement ratio of the paste.

5.2.6 Determination whether alkali–silica or alkali–carbonate reactions, or cement–aggregate reactions, or reactions between contaminants and the matrix have taken place, and their effects upon the concrete.

5.2.7 Determination whether the concrete has been subjected to and affected by sulfate attack, or other chemical attack, or early freezing, or to other harmful effects of freezing and thawing.

5.2.8 Part of a survey of the safety of a structure for a present or proposed use.

5.2.9 Determination whether concrete subjected to fire is essentially undamaged or moderately or seriously damaged.

5.2.10 Investigation of the performance of the coarse or fine aggregate in the structure, or determination of the composition of the aggregate for comparison with aggregate from approved or specified sources.

5.2.11 Determination of the factors that caused a given concrete to serve satisfactorily in the environment in which it was exposed.

5.2.12 Determination of the presence and nature of surface treatments, such as dry shake applications on concrete floors.

5.3 *Test Specimens from Actual or Simulated Service*—Concrete or mortar specimens that have been subjected to actual or simulated service conditions may be examined for most of the purposes listed under Concrete from Constructions.

5.4 *Concrete Products:*

5.4.1 Petrographic examination can be used in investigation of concrete products of any kind, including masonry units, precast structural units, piling, pipe, and building modules. The products or samples of those submitted for examination may be either from current production, from elements in service in constructions, or from elements that have been subjected to tests or to actual or simulated service conditions.

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TABLE 4 Outline for Examination of Concrete in Thin Sections

Coarse and Fine Aggregate	Relict Cement Grains and Hydration Products	Characteristics of Cement Paste
Mineralogy, texture, fabric, variable or homogeneous. Grading; excess or deficiency of sand sizes is to be judged after examination of a series of thin sections. Grain size and nature of internal boundaries in aggregate. Classification of coarse and fine aggregate. Natural mineral aggregate or crushed stone; natural or manufactured fine aggregate. Bond with matrix; peripheral cracks inside the borders of aggregate grains; internal cracks. General microfractures if one can establish that they existed before thin-sectioning. *Alkali - carbonate reactions*—If the coarse aggregate is a carbonate rock or rocks, are there rims or partial rims depleted in calcium hydroxide? Partly dolomitic rocks that have reacted sometimes are bordered with paste free from calcium hydroxide along the dolomitic portion while the paste along the limestone portion is normal. See other comments in Column 3. *Alkali - silica reaction*—Does the aggregate contain particles of types known to be reactive (chert, novaculite, acid volcanic glass, cristobalite, tridymite, opal, bottle glass)? If quartzite, metamorphosed subgraywacke, argillite, phyllite, or any of those listed in the sentence above, are there internal cracks inside the periphery of the aggregate? Has the aggregate been gelatinized so that it has pulled off during sectioning leaving only a peripheral hull bonded to the mortar? (This last phenomenon also occurs in concrete with air-cooled slag aggregate, where it indicates reaction between cement and slag.) Cracks that appear to be tensile and to narrow from the center toward the border of the particle are also evidence of alkali - silica reaction **(4)**.	In concrete over 2 years old and normally cured, the only residual cement grains are those that were largest, which may be composed of several constituents or be of alite or belite (substituted C_3S and C_2S). The latter two may be bordered by one or two layers of gel having different indexes of refraction, or by a layer of calcium hydroxide. The largest relict grains may be truly unhydrated and retain the low (dark gray) birefringence of alite in distorted quasihexagonal sections and the visible birefringence to first-order yellow of the lamellar twins in rounded grains of belite. Interstitial aluminoferrite appears as prismatic grains ranging in color from brown to greenish brown to reddish brown and having a high refractive index and pleochroism masked by the color of the grain. Tricalcium aluminate is usually not recognized in thin section because the cubic form is isotropic or because it hydrates early in the hydration history of the concrete forming submicroscopic ettringite or tetracalcium aluminum sulfate hydrate or other tetracalcium aluminum hydrates with or without other anions. These may be visible in voids in older concrete but are best discriminated by X-ray diffraction. Cements from different sources have different colors of aluminoferrite and the calcium silicates have pale green or yellow or white shades. It should be possible to match cements from one source.	Normal cement paste consists in plane transmitted light of pale tan matter varying somewhat in index of refraction and containing relict unhydrated cement grains. In concrete sectioned at early age or not adequately cured, the paste contains unhydrated cement grains ranging down to a few micrometres in maximum size with an upper limit as large as 100 µm in maximum diameter if the cement was ground in open-circuit mills or was deliberately ground to low surface area to reduce the heat of hydration. With crossed polars, normal paste is black or very dark mottled gray with scattered anhedral poikilitic crystals or small segregations of calcium hydroxide and scattered relict grains of cement. In concrete of high water–cement ratio and siliceous aggregate, the calcium hydroxide crystals are as large as the maximum size of residual cement grains, about 100µ m. In concrete of lower water–cement ratio, higher cement content, and either siliceous or carbonate aggregate, the maximum size of calcium hydroxide crystals is considerably smaller. Regardless of water–cement ratio and type of aggregate, calcium hydroxide crystals occupy space tangential to the undersides of aggregate particles. Where all the aggregate is carbonate rock the maximum size of calcium hydroxide is smaller than in comparable concrete with siliceous aggregate. (Calcium hydroxide is probably epitaxial on calcite.) Cement paste in concrete that has been subjected to prolonged acid leaching is low in calcium hydroxide which is present as recrystallized virtually anhedral grains precipitated near the exterior surfaces. In concrete over 2 or 3 years old made with Type I, II, or III cement, some ettringite is to be expected as rosettes in air voids. This is a normal phenomenon; to demonstrate sulfate attack it must be established chemically that the SO_3 content of the concrete is greater than would be supplied by the original sulfate content of the cement. Ettringite in voids is not ettringite that has damaged concrete although it may accompany submicroscopic ettringite in the paste that has damaged the concrete.

5.4.2 Determination of features like those listed under concrete from constructions.

5.4.3 Determination of effects of manufacturing processes and variables such as procedures for mixing, molding, demolding, consolidation, curing, and handling.

5.4.4 Determination of effects of use of different concrete-making materials, forming and molding procedures, types and amounts of reinforcement, embedded hardware, etc.

5.5 *Laboratory Specimens*—The purposes of petrographic examination of laboratory specimens of concrete, mortar, or cement paste are, in general, to investigate the effects of the test on the test piece or on one or more of its constituents, to provide examples of the effects of a process, and to provide the petrographer with visual evidence of examples of reactions in paste or mortar or concrete of known materials, proportions, age, and history. Specific purposes include:

5.5.1 To establish whether alkali–silica reaction has taken place, what aggregate constituents were affected, what evidence of the reaction exists, and what were the effects of the reaction on the concrete.

5.5.2 To establish whether one or more alkali–carbonate reactions have taken place, which aggregate constituents were affected and what evidence of the reaction or reactions exists, and the effects of the reaction on the concrete properties.

5.5.3 To establish whether any other cement– aggregate reaction has taken place. In addition to alkali–silica and alkali–carbonate reactions, these include hydration of anhydrous sulfates, rehydration of zeolites, wetting of clays and reactions involving solubility, oxidation, sulfates, and sulfides (see Refs (**1-17**)).[5]

5.5.4 To establish whether an aggregate used in a test has been contaminated by a reactive constituent when in fact the aggregate was not reactive.

[5] The boldface numbers in parentheses refer to a list of references at the end of this standard.

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TABLE 5 Characteristics of Concrete Observed Using Microscopes

Characteristic	Type of Microscope		
	Stereomicroscope	Petrographic	Metallographic
Aggregate:			
Shape	X	X	X
Grading	X	...	...
Distribution	X	...	...
Texture	X	X	X
Composition	X	X	...
Rock types	X	X	...
Alteration	X	X	...
degree	X	X	
products	X	X	...
Coatings	X	X	...
Rims	X	X	X
Internal cracking	X	X	...
Contamination	X	X	
Concrete:			
Air-entrained or not	X	X	X
Air voids	...	...	...
shape	X	X	X
size	X	X	X
distribution	X	...	...
Bleeding	X	...	...
Segregation	X	...	...
Aggregate-paste bond	X	X	X
Fractures	X	X	X
Embedded items			
size	X	...	...
shape	X	...	...
location	X	...	...
type	X	...	...
Alteration			
degree and type	X	X	X
reaction products			
location	X	X	X
identification	X[A]	X	...
Nature and condition of surface treatments	X	X	...
Paste:			
Color	X	X	...
Hardness	X	...	X
Porosity	X	...	X
Carbonation	X	X	...
Residual cement			
distribution	...	X	X
particle size	...	X	X
abundance	...	X	X
composition	...	X	X
Supplementary cementitious materials	X[B]	X	X
size	...	X	X
abundance	X	X	X
identification	X	X	X
Compounds in hydrated cement	X[C]	X	X
Contamination			
size	X	X	X
abundance	X	X	X
identification	...	X	X[D]

[A] Secondary ettringite can sometimes be recognized by crystal habit and silky luster.
[B] Fly ash can be detected by color and shape when dark spheres are present. In concrete that has not oxidized the presence of slag may be inferred from the green or blue color of the paste.
[C] Ettringite and calcium hydroxide in voids may be recognized by their crystal habits.
[D] Magnesium oxide and calcium oxide should be identifiable in polished section.

5.5.5 To establish the effects of a freezing and thawing test or other physical or mechanical exposure of concrete on the aggregate and the matrix.

5.5.6 To establish the extent of reaction, the nature of reaction products, and effects of reaction produced in exposure to a chemically aggressive environment such as in Test Method C452 or Test Method C1012.

5.5.7 To determine the characteristics of moist-cured concrete that has not been subjected to chemical attack or cement–aggregate reaction or freezing and thawing.

5.5.8 By comparison with appropriate laboratory specimens, a petrographer may be able to substantiate the existence of a particular reaction in concrete or determine that the reaction cannot be detected.

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TABLE 6 Secondary Deposits in Concrete[A]

Compound and Mineral Equivalent	Indexes of Refraction	Form and Occurrence
Calcium carbonate ($CaCO_3$); calcite	ω = 1.658 ε = 1.486	Fine-grained, white or gray masses or coatings in the cement paste, in voids, along fractures, or on exposed surfaces; very common
Calcium carbonate ($CaCO_3$); aragonite	α = 1.530 β = 1.680 γ = 1.685	Minute, white prisms or needles in voids or fractures in concrete; rare
Calcium carbonate ($CaCO_3$); vaterite	o = 1.544–1.550 E = 1.640–1.650	Spherulitic, form-birefringent, white encrustations on moist-stored laboratory specimens (vaterite A); also identified in sound concrete from structures by X-ray diffraction (α-vaterite); common **(5)**
6-calcium aluminate trisulfate-32 hydrate $\{Ca_6[Al(OH)_6]_2\cdot 24H_2O\}(SO_4)_3\cdot 2H_2O$ **(6)**; ettringite	ω = 1.464–1.469[B] ε = 1.458–1.462	Fine, white fibers or needles or spherulitic growths in voids, in the cement paste, or in fractures; very common **(1, 5)**
Tetracalcium aluminate monosulfate-12-hydrate ($3CaO\cdot Al_2O_3\cdot CaSO_4\cdot 12H_2O$)	ω = 1.504 ε = 1.49	White to colorless, minute, hexagonal plates in voids and fractures; very rare **(5)**
Tetracalcium aluminate-13-hydrate ($Ca_4Al_2(OH)_{14}\cdot 6H_2O$)	ω = 1.53 ε = 1.52	Micalike, colorless, pseudohexagonal, twinned crystals in voids; very rare **(7)**
Hydrous sodium carbonate ($Na_2O\cdot CO_2\cdot H_2O$); thermonatrite	α = 1.420 β = 1.506 γ = 1.524	Minute inclusions in alkalic silica gel; rare **(5)**
Hydrated aluminum sulfate ($2Al_2O_3\cdot SO_3\cdot 15H_2O$); paraluminite	α = 1.463 ± 0.003 β = 1.471 γ = 1.471	Occurring in cavities in intensely altered concrete; very rare **(7)**
Calcium sulfate dihydrate ($CaSO_4\cdot 2H_2O$); gypsum	α = 1.521 β = 1.523 γ = 1.530	White to colorless crystals in voids, in the cement paste, or along the surfaces of aggregate particles in concrete or mortar affected by sulfate or seawater attack; unusual
Calcium hydroxide ($Ca(OH)_2$); portlandite	ω = 1.574 ε = 1.547	White to colorless, hexagonal plates or tablets in the cement paste, in voids, along fractures; ubiquitous in concrete
Magnesium hydroxide ($Mg(OH)_2$); brucite	ω = 1.559 ε = 1.580	White to yellow, fine-grained encrustations and fillings in concrete attacked by magnesian solutions or seawater; unusual **(8, 9)**
Hydrous silica ($SiO_2\cdot nH_2O$); opal	η = 1.43 varies with water content	White to colorless, finely divided, amorphous; resulting from intense leaching or carbonation of cement paste; unusual in recognizable proportions
Alkalic silica gel ($Na_2O\cdot K_2O\cdot CaO\cdot SiO_2$)	η = 1.46–1.53	White, yellowish, or colorless; viscous, fluid, waxy, rubbery, hard; in voids, fractures, exudations, aggregate; common **(10, 11)**
Hydrated iron oxides ($Fe_2O_3\cdot nH_2O$); Limonite	opaque or nearly so	Brown stain in fractures and on surfaces; common
Thaumasite $\{Ca_6[Si(OH)_6]_2\cdot 24H_2O\}(SO_4)_2(CO_3)_2$ **(6)**	ω = 1.504 ε = 1.468± 0.002[B]	Prismatic, hexagonal; capable of growing in continuity with ettringite; in sewer pipe subject to sulfate attack, in grout, in some pavement **(12)**
Syngenite ($K_2Ca(SO_4)_2\cdot H_2O$	α = 1.501 **(13)** β = 1.51 γ = 1.51	Found in cavities and zones peripheral to slate particles, in fibrous form **(14)**
Hydrotalcite $Mg_{3/4}Al_{1/4}(OH)_2(CO_3)_{1/8}(H_2O)_{1/2}$ **(6)**	ω = 1.510 ± 0.003 ε = 1.495± 0.003	Foliated platy to fibrous masses **(15, 6)**

[A] The literature and private reports include data on many unidentified secondary compounds in concrete; these are not included in the tabulation. Indexes of refraction of common mineralogic types are taken from standard works on mineralogy.
[B] Higher and lower indexes of refraction have been recorded for naturally occurring ettringite **(13)** and thaumasite **(12)**, but it is not known that the naturally occurring minerals and compounds found in hydrated cement are of the same composition.

6. Apparatus

6.1 The apparatus and supplies employed in making petrographic examinations of hardened concrete depend on the procedures required. The following list includes the equipment generally used. Equipment required for field sampling is not listed. Any other useful equipment may be added.

6.2 *For Specimen Preparation:*

6.2.1 *Diamond Saw*—Slabbing saw with an automatic feed and blade large enough to make at least a 7-in. (175-mm) cut in one pass.

6.2.2 *Cutting Lubricant,* for diamond saw.

6.2.3 *Horizontal Lap Wheel or Wheels,* steel, cast iron, or other metal lap, preferably at least 16 in. (400 mm) in diameter, large enough to grind at least a 4 by 6-in. (100 by 152-mm) area.

6.2.4 *Free Abrasive Machine,* using abrasive grit in lubricant, with sample holders rotating on a rotating table. This type of grinding machine greatly increases the speed of preparation of finely ground surfaces.

6.2.5 *Polishing Wheel,* at least 8 in. (200 mm) in diameter and preferably two-speed, or a vibratory polisher.

6.2.6 *Hot Plate or Oven,* thermostatically controlled, to permit drying and impregnating specimens with resin or wax for preparing thin sections, ground surfaces, and polished sections.

6.2.7 *Prospector's Pick or Bricklayer's Hammer,* or both.

6.2.8 *Abrasives*—Silicon carbide grits, No. 100 (150-μm), No. 220 (63-μm), No. 320 (31-μm), No. 600 (16-μm), No. 800 (12-μm); optical finishing powders, such as M-303, M-204, M-309; polishing powders as needed.

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6.2.9 *Plate-glass Squares,* 12 to 18-in. (300 to 450-mm) on an edge and at least ⅜ in. (10 mm) thick for hand-finishing specimens.

6.2.10 *Suitable Medium or Media,* for impregnating concrete and mounting thin sections plus appropriate solvent. Canada balsam, Lakeside 70 cement, and flexibilized epoxy formulations have been used.

6.2.10.1 **Warning—**Flexibilized epoxies form strong bonds but have higher indexes of refraction than Canada balsam or Lakeside 70 and are toxic. Do not allow to touch the skin; plastic gloves shall be worn, and the work shall be done under a hood so as not to breathe the fumes.

6.2.11 *Microscope Slides*—Clear, noncorrosive, glass approximately 24 mm wide and at least 45 mm long. Thickness may need to be specified to fit some thin section machines.

6.2.12 *Cover Glasses,* noncorrosive and preferably No. 1 (0.18-mm) thickness.

6.3 *For Specimen Examination:*

6.3.1 *Stereomicroscope,* providing magnifications in the range from 7× to 70× or more.

6.3.2 *Dollies*—Small, wheeled dollies with flat tops and with tops curved to hold a section of core assist in manipulating concrete specimens under the stereomicroscope.

6.3.3 *Petrographic Microscope or Polarizing Microscope,* for examinations in transmitted light, with mechanical stage; low-, medium-, and high-power objectives such as 3.5×, 10×, and 20 to 25×; 43 to 50× with numerical aperture 0.85 or more; assorted eyepieces having appropriate corrections and magnifications for use with each of the objectives; micrometer eyepiece; condenser adjustable to match numerical aperture of objective with highest numerical aperture to be used; full-wave and quarter-wave compensators, quartz wedge, and other accessories.

6.3.4 *Metallographic Microscope,* with vertical illuminator, mechanical stage, metallographic objectives of low, medium, and high magnification, and appropriate eyepieces to provide a range of magnifications from about 25× to 500×. Reflected polarized light should be available and appropriate compensators provided. Some polarizing microscopes can be equipped with accessories for metallographic examination, if the tube can be raised or the stage lowered to give adequate clearance for the vertical illuminator and the thicker specimens usually employed.

6.3.5 *Eyepiece Micrometer*—Eyepiece micrometers calibrated using a stage micrometer are useful for measuring particles of aggregate, cement grains, calcium hydroxide and other crystals, and crack widths.

6.3.6 *Stage Micrometer,* to calibrate eyepiece micrometers.

6.3.7 *Microscope Lamps*—Many modern polarizing microscopes have built-in illuminators which are convenient and satisfactory if, with the condenser, they can be adjusted to fill the back lens of the objective of highest numerical aperture with light. If the microscope requires a separate illuminator, tungsten ribbon-filament bulbs in suitable adjustable housings are satisfactory. Many kinds of illuminators are available for stereomicroscopes; some can be mounted on the microscope, some stand on their own bases; choice is a question of adequacy of illumination for the tasks intended. Focusable illuminators are preferred.

6.3.8 *Needleholders and Points*—In addition to pin vises and needles from laboratory supply houses, a No. 10 sewing needle mounted in a handle or a selection of insect pins from size 00 to size 4 are useful for prying out reaction products.

6.3.9 *Bottles with Droppers,* for acid, water, and other reagents applied during examination.

6.3.10 *Assorted Forceps,* preferably stainless steel, including fine-pointed watchmaker's forceps.

6.3.11 *Lens Paper.*

6.3.12 *Refractometer, and Immersion Media,* covering the range of refractive indexes from 1.410 to at least 1.785, in steps not larger than 0.005. Stable immersion media, calibrated at a known temperature and of known thermal coefficient, are preferable and should be used in a temperature-controlled room. A thermometer graduated in tenths of a degree Celsius should be used to measure air temperature near the microscope stage so that thermal corrections of refractive index can be made if needed.

7. Selection and Use of Apparatus

7.1 Laboratories should be equipped to provide photographs, photomacrographs, and photomicrographs to illustrate significant features of the concrete. While ordinary microscope lamps are sometimes satisfactory for photomicrography in transmitted and reflected light, lamps providing intense point or field sources, such as tungsten ribbon-filament bulbs, or zirconium or carbon arcs, are highly desirable. For much useful guidance regarding photomicrography, especially using reflected light, see Guide E883.

7.2 The minimum equipment for petrographic examination of concrete where both specimen preparation and examination are completed within the laboratory consists of a selection of apparatus and supplies for specimen preparation, a stereomicroscope preferably on a large stand so that 6-in. (152-mm) diameter cores can be conveniently examined, a polarizing microscope and accessories, lamps for each microscope, and stable calibrated immersion media of known thermal coefficient. Specimens for petrographic examination may be obtained by sending samples to individuals or firms that offer custom services in preparing thin or polished sections and finely ground surfaces. It is more convenient to prepare specimens in house, and their prompt availability overrides their probably greater cost.

7.3 X-ray diffraction, X-ray emission, differential thermal analysis, thermogravimetric analysis, analytical chemistry, infrared spectroscopy, scanning electron microscopy, energy or wavelength dispersive analysis, and other techniques may be very useful in obtaining quick and definite answers to relevant questions where microscopy will not do so. Some undesirable constituents of concrete, some hydration products of cement, and some reaction products useful in defining the effects of different exposures, and many contaminating materials may not be identified unless techniques that supplement light microscopy are used. (**18**, **19**).

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8. Samples

8.1 The minimum size of sample should amount to at least one core, preferably 6 in. (152 mm) in diameter and 1 ft (305 mm) long for each mixture or condition or category of concrete, except that in the case of pavement the full depth of pavement shall be sampled with a 4 or 6-in. (102 or 152-mm) core. Broken fragments of concrete are usually of doubtful use in petrographic examination, because the damage to the concrete cannot be clearly identified as a function of the sampling technique or representative of the real condition of the concrete. Cores smaller in diameter than 6 in. can be used if the aggregate is small enough; in deteriorated concrete, core recovery is much poorer with 2⅛-in. (54-mm) diameter core than with 6-in. diameter core. While it is desirable in examination and testing to have a core three times the maximum size of aggregate, this circumstance is a rare occurrence when concrete with aggregate larger than 2 in. is sampled, because of the cost of large bits and the problems of handling large cores.

8.2 *Samples from Constructions*—The most useful samples for petrographic examination of concrete from constructions are diamond-drilled cores with a diameter at least twice (and preferably three times) the maximum size of the coarse aggregate in the concrete. If 6-in. (152-mm) aggregate is used, a core at least 10 in. (250 mm) in diameter is desirable; usually a 6-in. diameter core is the largest provided.

8.2.1 The location and orientation of all cores, including cores or core lengths not sent to the laboratory, should be clearly shown; and each core should be properly labeled. For vertically drilled cores, the elevation or depth at top and bottom of each section should be shown, and core loss and fractures antedating the drilling should be marked. For cores taken horizontally or obliquely, the direction of the vertical plane and the tops and bottoms should be marked. A field log should be provided.

8.2.2 Broken pieces of concrete from extremely deteriorated structures or pieces removed while preparing for repair work are sometimes used for petrographic examination. The samples will be more useful if their original locations in the structure are clearly described or indicated in a sketch or photographs.

8.2.3 The information provided with the samples should include:

8.2.3.1 The location and original orientation of each specimen (see Practice C823),

8.2.3.2 The mixture proportions of the concrete or concretes,

8.2.3.3 Sources of concrete-making materials and results of tests of samples thereof,

8.2.3.4 Description of mixing, placing, consolidation, and curing methods,

8.2.3.5 Age of the structure, or in case of a structure that required several years to complete, dates of placement of the concrete sampled,

8.2.3.6 Conditions of operation and service exposure,

8.2.3.7 The reason for and objectives of the examination,

8.2.3.8 Symptoms believed to indicate distress or deterioration, and

8.2.3.9 Results of field tests such as measurements of pulse velocity (Test Method C215), rebound hammer numbers (Test Method C805) or probe readings (Test Method C803/C803M).

8.3 *Samples from Test Specimens from Natural Exposures, Concrete Products, and Laboratory Specimens:*

8.3.1 Information provided should include: materials used, mixture proportions, curing, age of concrete when placed in service or test, orientation in exposure, present age, condition surveys during exposure, characteristics of the natural or laboratory exposure, and method of manufacture of concrete products. Large concrete products may be sampled like constructions; smaller ones may be represented by one or more showing the range of condition from service or fabrication or both.

8.3.2 The exposure of laboratory specimens should be described with test results, age at test and available test results on the aggregates, hydraulic binders, and admixtures used. This information should accompany test specimens from natural exposures and concrete products or samples therefrom, if available.

9. Examination of Samples

9.1 *Choice of Procedures*—Specific techniques and procedures employed in examination of a sample depend on the purpose of the examination and the nature of the sample. Procedures to be used should be chosen after the questions that the examination is intended to answer have been clearly formulated. The procedures should be chosen to answer those questions as unequivocally and as economically as possible. The details that need to be resolved will be dictated by the objectives of the examination and will vary for different situations. Consequently, the selection and location of specimens from the samples submitted for examination should be guided by the objectives of the study. Test Method C457 should be referred to for those relevant subjects not described here.

9.2 *Visual Examination and Outline of Additional Examination*—A petrographic examination of concrete, mortar, or cement paste should begin with a review of all the available information about the specimen or specimens, followed by a visual examination of each sample. An outline of information that can be obtained is given in Table 1. That study should be followed by an examination using a stereomicroscope (see Table 2 and the section on Visual and Stereomicroscopic Examination). In some cases, further study is unnecessary, and a report can be prepared. In other cases, specimens are chosen during the visual and stereomicroscope examination for further processing and additional stereomicroscope study, more detailed examination using the petrographic or metallographic microscopes or by X-ray diffraction and other instrumental methods, and for other chemical or physical tests. Methods for specimen preparation are outlined in the Specimen Preparation Section. Tables 2-4 summarize characteristics of concrete conveniently observed with stereomicroscopic, petrographic, and metallographic microscopes. Examination using a stereomicroscope is outlined in the Visual and Stereomicroscopic Examination Section. Examination of fire-damaged concrete is outlined in Table 3; using a polarizing microscope in the Polarizing Microscope Examination Section and Table 4; and

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using a metallographic microscope in the Metallographic Microscope Examination Section. During each kind of study, the petrographer should note specific examinations to be made in detail, later, and may recognize the need to reexamine specimens. Observations possible using different kinds of microscopes are shown in Table 5; properties of some relevant compounds are listed in Table 6.

9.3 *Photographs*—Photographs and images should be maintained to illustrate features of the examined specimens, such as as-received conditions before they are altered, and important macro-and micro-features of prepared lapped sections, polished sections, fractured surfaces, thin sections, and immersion mounts. Photographs should have a scale or reference to scale.

10. Specimen Preparation

10.1 *Preparation for Visual and Stereomicroscope Examination:*

10.1.1 Diamond-drilled cores, formed or finished surfaces, freshly broken surfaces, or old crack surfaces should be examined in the condition received. It is sometimes helpful to have drilled surfaces and formed and finished surfaces wetted to increase contrast.

10.1.2 Diamond saw cuts should be oriented with relation to significant features of the concrete, either normal to the bedding directions in conventional concrete, or normal to a formed or finished surface, or to a crack or crack system, in order to reveal the structure and fabric of the concrete and the extent of alteration outward from the crack.

10.1.3 It is useful to prepare at least one sawed surface by grinding it with progressively finer abrasives (as described in Test Method C457) until a smooth matte finish is achieved and to select areas on the matching opposing surface for preparation of thin sections and specimens for optical, chemical, X-ray diffraction, or other examinations.

10.1.4 Specimens obtained by diamond drilling are not ordinarily damaged in the process; however, weak concrete damaged by chemical attack, an alkali–aggregate reaction, freezing and thawing, or several of these, will give poor core recovery with many fractures if it is drilled with a 2⅛-in. or 54-mm bit and barrel while it will give essentially complete recovery if drilled with a 6-in. (152-mm) diameter bit and barrel. This difference is particularly important in petrographic examinations made during condition surveys of old structures. Weakened concrete may also break during sawing. The removal and preparation of specimens for laboratory studies usually involves the application of force and sometimes the application of heat to the specimen.

10.1.5 The effects of force can be minimized during specimen preparation by using thicker slices and making only one cut parallel to the long axis of a core section. Fractured or fragile concrete can be supported by partially or completely encasing it in plaster, epoxy resin, or other reinforcing media before sawing.

10.1.6 Heat used while impregnating concrete with thermoplastic wax or resin will cause cracking if the concrete is heated while it is wet, and will alter the optical properties of some compounds, such as ettringite. Artifacts may therefore be produced and compound identification made difficult. These artifacts may be mistaken as original features. Care must therefore be used in evaluating a particular feature and indexing it as original in the specimen, or produced during the removal of the specimen from the structure or during laboratory processing.

10.1.7 When alkali–carbonate reactions are suspected and rims around crushed carbonate aggregate are seen, it is useful to etch a sawed or ground surface in 6 *N* or weaker hydrochloric acid to see if peripheral rims on coarse aggregate particles are more or less susceptible to etching than the interior of the particle. Since etching destroys the surface, this step should not be taken until all other examinations of the surface have been completed. Etching the ground surface for 30 s in 10 % hydrochloric acid is an appropriate procedure.

10.2 *Preparation of Immersion Mounts*—Immersion mount samples are prepared for examinations using the petrographic microscope. This type of examination provides versatility because materials can be immersed in liquids having different refractive indices. Detailed knowledge of the use of the capabilities of the petrographic microscope is required to properly examine immersion mounts.

10.2.1 Immersion mounts are useful for observing and identifying a variety of aggregate components, residual and relict portland cement particles, the calcium hydroxide component of cement hydration, components of blended cements, supplementary cementitious materials, components resulting from chemical alteration of cementitious components and of aggregates, secondary deposits from exposure of concrete to a variety of chemicals, and for confirming identifications by other methods.

10.2.2 Powders and fine chips for immersion mounts can be prepared by: (1) pulverization of samples from which specimens may be taken; (2) using a sharply pointed probe for removing specimens from small areas of aggregate and paste, material in aggregate sockets, voids, and cracks; and (3) scrapings from fracture and formed surfaces.

10.2.3 In immersion mounts, individual fragments are usually in random orientation so that the identification of principal refractive indices of a material can be determined in addition to data about other optical characteristics. Portions of powdered material, chips, or scraping are placed on a glass slide and immersed in an immersion liquid of known refractive index. A cover slip is used on top of the preparation. Based upon refractive indices and other optical properties, specific identifications can be made of unknown compounds.

10.2.4 Refractive index liquids available provide refractive index values to at least three decimal places (see 6.3.12). Liquids accurate to two decimal places can be used if the petrographer is knowledgeable about Becke line colors or other techniques.

10.3 *Preparation of Thin Sections*—The detailed description of thin-section preparation is beyond the scope of this practice. There are many laboratories that provide this service if in-house facilities are not available. The procedure includes slicing the concrete into 1⁄16-in. (2-mm) thick wafers if the concrete is strong and thicker slices if it is not. It may be necessary to impregnate the concrete with a resin before slicing to prevent disintegration. Diluted flexibilized epoxy resins or

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thermoplastic resins have been used successfully. The thin concrete slices are then mounted on glass slides with either flexibilized epoxy, Canada balsam, or Lakeside 70, and ground on laps using progressively finer abrasive until a thickness of 30 µm or less is obtained; thickness not greater than 20 µm is required for detailed examination of the paste in transmitted light. It is usually necessary to check the thickness of the section by the use of birefringent colors of common minerals in the aggregate, such as quartz or feldspar, during the final grinding stages. A cover glass is placed on the cleaned, prepared section and secured with Canada balsam or other media.

10.3.1 Semiautomatic thin-section making machines are available which prepare the original surface of the blank for mounting, trim the excess thickness of the blank after mounting, and grind the section to 50 to 100 µm, leaving little thickness to be removed by hand lapping.

10.4 *Preparation for Examination with the Metallographic Microscope:*

10.4.1 The preparation of specimens for examination with the metallographic microscope is described in Practice E3.

10.4.2 The procedures described here are intended as guides only. Methods should be used that are appropriate to the varieties of microscopical techniques to be employed, and to specimen condition and composition. It is impossible to provide instructions to suit every possible situation.

11. Visual and Stereomicroscope Examination

11.1 If there is more than one specimen, arrange them in logical order to represent: position in the structure and differences in materials, proportions, and exposure, or combinations of these. Photographs and sketches of significant features should be made before specimens are altered. Tables 1 and 2 list some features to be observed during visual and stereomicroscopic examination.

11.2 *Concrete from Constructions* (Core Log):

11.2.1 Fit fragmented cores together and determine if any pieces are missing. Measure cores to verify field data and prepare a diagramatic log of each core, if necessary. The log should be made to a scale to show relevant features. The log may be used to show fresh and old fractures, reacted particles, reaction products, changes in size or type of coarse and fine aggregates, distribution of coarse aggregate, honeycomb, segregation of components, cold joints or lift or course boundaries, location and direction of steel or other embedded items, changes in color of paste, and other significant features. Notes should be taken and suitably referenced in the log. If cores have been suitably packed so as to preserve the moisture content as drilled (which may or may not be the actual in-place moisture content), specimens should be carefully handled to preserve the moisture content and avoid breakage. Tests that may be made of specimens in the as-received moisture condition include compressive strength, static or dynamic modulus of elasticity, air content by high-pressure meter, permeability, and freezing and thawing.

11.2.2 During the visual examination, general comparison of cores representing different conditions, materials, and extent and nature of deterioration should be made and recorded. The specimens should be sorted into comparable groups based on condition or location of regions designated for more detailed examination. In some cases, during the visual examination or the stereomicroscope examination, one or more specimens from each group may be selected for more detailed studies. If a reduced number of specimens is to be selected for detailed examination, the selection should be made after careful visual and stereomicroscope examinations have been used to define one or more factors that characterize each group.

11.3 *Specimens from Natural Exposures*— These specimens should be laid out in logical order, either by materials, proportions, age, or combinations of these, and compared with respect to composition and condition. Significant features for more detailed examination should be marked and noted. Table 3 shows features of fire-damaged concrete.

11.4 *Concrete Products:*

11.4.1 The samples may consist of complete units when they have relatively small dimensions (such as tile, block, or brick), or portions removed from units by coring or sawing. The samples should be organized and oriented in a manner appropriate to the objectives of the examination. So, for example, portions taken from a single product unit should be grouped together, and each sample should be described by visual observation and measurement of dimensions in relation to markings or labels. The following information should be recorded or clearly marked on the samples: location in the product unit (top, side, end, interior, etc.); vertical and horizontal directions as cast or molded; outer and inner ends; position in the product unit during curing period; position of the sample relative to highly stressed portions of the concrete adjacent to tendon anchorages; and location in the unit as put in place in construction, etc. Sketches or photographs of pertinent features should be recorded to show such conditions as cracking, staining, chemical deposits, presence of foreign matter, segregation, surface defects, and the like.

11.4.2 Procedures such as those set forth in 11.1 and 11.2 are applicable in the examination of concrete products. The investigation may be directed toward features resulting from the specific manufacturing operations involved, such as mixing, molding, demolding, curing, and any prestressing procedures. For example, lack of uniformity might originate in incomplete mixing, improper consolidation during molding, or incomplete or nonuniform curing. Surface defects and poor appearance may result from improper application of form-release agents, possibly giving rise to inhibition of hydration of the cement in a near-surface zone. Features like these can be detected and described by visual and microscopical examination of formed or molded surfaces, sawed and lapped surfaces, or fracture surfaces across the sample.

11.5 *Laboratory Specimens*—These should be laid out in logical order and compared with respect to composition and condition, and to features influenced or expected to be influenced by the test process.

11.6 *Stereomicroscope Examination*—The stereomicroscope examination reveals additional details at magnifications from 5× to 150× (see Tables 1 and 2). The stereomicroscope examination and visual examination are often carried on

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alternately. Examinations at low magnifications reveal characteristics of formed, finished, deteriorated, broken, sawed, or ground surfaces. Old cracks may be opened and their surfaces examined to detect reaction products and secondary alteration. Old cracks are frequently of different color than the mass of the concrete, and appear blurred by secondary deposits.

11.6.1 Freshly broken surfaces may follow structural weaknesses and reveal significant features that have not been masked by secondary alteration.

11.6.2 Sawed and ground surfaces are examined to detect fine cracks and trace networks of cracks that are not perceptible on drilled or sawed surfaces, to examine filling and partial filling in voids and cracks, and to detect rims on aggregate particles that may indicate chemical reactions between the cement and the aggregate. Rims on gravel or natural sand should be presumed to be a result of weathering in the deposit, unless samples of aggregates used are available and show that rims were not present before the aggregate was used in the concrete. Rims produced in the concrete on particles of sand and gravel are absent or relatively thin and faint at locations where the particle is in contact with an air void. Rims produced by weathering may mask rims produced by alkali–silica reaction. Rimmed crushed stone in concrete usually indicates alteration in the concrete, as alkali–silica reaction or alkali–carbonate reaction **(10, 20, 21)**. Pale rims in mortar bordering coarse aggregate **(5, 19)** and pale areas in the mortar may be gel-soaked paste **(4)** or highly carbonated paste adjoining carbonate aggregate that has undergone an alkali–carbonate reaction.

11.6.3 Deposits in old cracks may include calcite or ettringite or calcium hydroxide, or combinations of these compounds, or more unusual crystalline substances, but sometimes include sand, silt, clay, and freshwater or saltwater organisms. Such organisms may provide evidence that a part of a structure that is not ordinarily submerged has been submerged.

11.6.4 Quantitative determination of constituents of concrete can be made by microscopical point-count or linear-traverse procedures in general accord with the requirements of Test Method C457. A total analysis may include the proportional amounts of coarse aggregate, fine aggregate, cementitious matrix, and air voids. The differentiation of coarse and fine aggregate is not accurate unless the two fractions are distinguishable lithologically, since the maximum dimension of the particles usually is not intersected by the prepared surface. Likewise, the proportion of individual rock types can be determined quantitatively by these methods, such as, the content of unsound or reactive constituents in the coarse or fine aggregate or both; proportions of lightweight and normal weight aggregate; and the abundance of granular contaminating substances.

12. Petrographic Microscope Examination

12.1 *Characteristics of Good Concrete Thin Sections*—Features that characterize good, thin sections of concrete or mortar are as follows: the mounted lower surface is free of abrasive and has been ground to a smooth matte finish ordinarily obtained by finishing the surface with optical alumina or equivalent; the lower surface of the blank is entire; air voids have complete peripheries; there is a sharp boundary between the void and surrounding mortar; deposits in voids are preserved; and aggregate that has been observed under the stereomicroscope to be entire when the blank was selected has remained entire and microfractures have not been produced in micro fsectioning. It is very difficult to avoid production of micro fsectioning when the aggregate is principally quartzite and quartz, if it is desired to reduce the section to a thickness of 30 µm so that the brightest birefringence color of quartz is first-order white. Some cracks will be formed and some loss of paste which is softer than quartz aggregate will occur.

12.1.1 The desirable thickness of the thin sections ranges from about 40 to 20 µm. Thinner sections may be required for detailed examination of the cement paste matrix. It is sometimes necessary to give up normal thickness and use a thicker section to preserve fragile deposits in voids such as alkali–silica gel, ettringite, calcium hydroxide, calcite, aragonite, or thaumasite. When it is desired to preserve the contacts between mortar and coarse aggregate, it may be preferable to leave the section thicker than normal with the highest birefringence color of the quartz pale first-order yellow. In normal concrete, the material bordering the aggregate consists of amorphous cement gel, with calcium hydroxide crystals quite evenly distributed but with slight concentrations along the undersides of coarse aggregate and fine aggregate, if the section is cut parallel to the placement direction. In cases of alkali–silica reaction, there is usually a zone depleted in or free of calcium hydroxide surrounding reacted aggregate particles; sometimes gel is present surrounding the aggregate or soaking the paste so that it is completely dark with crossed polars and brown grading hazily to more normal paste in plane polarized light. It is fairly easy to recognize gel-soaked paste; it is possible with experience to recognize depleted calcium hydroxide around a reacted aggregate particle, so long as the fine aggregate or any crusher dust present does not contain much colorless mica. Thin flakes of muscovite can be confused with thin tablets of calcium hydroxide when both are viewed on edge; they can be distinguished if the higher index of the mica is observed.

12.1.2 It is sometimes preferable to lose part of the section or crack the quartz in order to distinguish among quartz (birefringence 0.009), calcium hydroxide (birefringence 0.027), and calcite (birefringence 0.172). When the three materials occur together, the quartz particles will be shaped like sand grains or rock fragments, while calcium hydroxide will be present either as tablets tangential to aggregate or as poikilitic crystals in the paste enclosing residual cement grains or areas of gel. The birefringence of calcium hydroxide is three times that of quartz; the birefringence of calcite is 6.4 times that of calcium hydroxide; and birefringence of aragonite is 5.7 times that of calcium hydroxide. While aragonite is fairly uncommon in concrete, calcite is common as a product of carbonation and as an ingredient of aggregate. At normal thickness of 30 µm, the highest birefringence of calcium hydroxide viewed parallel to the cleavage is bright first-order yellow with an occasional orange or first-order red area. Calcite in the same section has high-order white birefringence except in very minute grains in which it is very thin, approximately rhombic in shape, and the birefringence color is

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first-order white. The birefringence relations discussed in this paragraph are the basis that makes possible much of the interpretation of concrete thin sections.

12.2 *Choice of Areas for Thin Sections of Concrete*—Areas from which thin sections are to be prepared usually should be chosen after examining the sawed or sawed and ground surface with a stereomicroscope. Selection of the area may depend on the features to which the examination is directed, or the choice may be affected in the case of highly deteriorated concrete by the desire to choose a volume strong enough to endure sectioning even after impregnation. In that second case, an area of mortar with coarse aggregate at the corners or along the sides may be the successful choice. If it is desired to examine the aggregate to compare it with aggregate of a known source to establish whether the two probably came from the same deposit or quarry, coarse aggregate should be chosen in sufficient number to cover the range of varieties present and their physical condition. If the concrete has been subjected to alkali–silica or alkali–carbonate reactions, areas selected for sectioning should include coarse aggregate with cracks inside the periphery but surrounding the center and cracks in the middle of the particle which narrow toward the border which may or may not contain gel in the case of alkali–silica reaction. Reaction-rimmed particles may be chosen for sectioning in the case of either kind of reaction, but it is ordinarily a futile effort; the rims distinct to the naked eye or the low power of the stereomicroscope may not be visible in thin section.

12.2.1 Features visible in the examination of thin sections of concrete are shown in Table 4.

13. Paste Features

13.1 Many physical and mineralogical properties of the paste are influenced, or are in part governed by, *w/c* and *w/cm*, extent of curing, fineness and composition of the portland cement and other cementitious materials, pozzolans, mineral and chemical admixtures, and fine material from aggregates (See Terminology C125). Among these properties, with respect to the evaluation of unaltered paste, are: (a) color, hardness density, and porosity (**22-25**); (b) texture of fracture surfaces (**22 and 23**); (c) size, abundance, color, and mineralogy of non-pozzolanic, and residual and relict pozzolanic and cementitious materials (**22-24, 26**); (d) size, abundance, and morphology of the calcium hydroxide component of cement hydration as may be affected by pozzolanic materials (**22 and 23, 26**); (e) degree of hydration of cementitious materials (**22-28**); (f) color intensity of dye-impregnated pastes (**24,26**); (g) nature of aggregate-paste bond; (h) rate of absorption of water droplets (**24**); (i) depth of carbonation (**23**); (j) magnitude of bleeding and segregation of concrete-making components (**23, 27**); and (k) paste micro hardness (**25**).

13.2 The *w/c* and *w/cm* and their uniform distribution throughout concrete may be affected by a number of influences that include: (a) the amount of original water, free water introduced by aggregates, and tempering and retempering water; (b) absorption of water by aggregates; (c) efficiency of mixing batch water and free water from aggregates; (d) bleeding and vibration after concrete placement; and (e) water lost due to evaporation and to absorption by the subbase.

13.3 Because of the variable nature of pastes, age of pastes, and exposure to a variety of external influences, there is no generally accepted standard procedure that employs microscopical methods for determining the *w/c* or *w/cm* of hardened concrete. A procedure for estimating *w/c* and *w/cm* (**29**) was critically evaluated in a SHRP study, but was not recommended by SHRP for inclusion in Practice C856.

14. Report

14.1 The report of the examination should include the following:

14.1.1 Location and orientation of the samples in the construction or products or type of specimen,

14.1.2 History of the samples insofar as is available,

14.1.3 Physical and chemical tests made on the samples, with their results,

14.1.4 Description of the samples and a report on mixture proportions, if available or if estimated, workmanship, construction practice, and original quality of the concrete in the constructions, insofar as such information is available, and

14.1.5 Interpretation, insofar as possible, of the nature of the materials and the chemical and physical events that have led to the success or distress of the concrete.

15. Keywords

15.1 aggregates; air voids; alkali-silica reaction; analysis; deterioration; examination; fire effects; hardened concrete; microscopy; paste; petrographic; secondary deposits

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APPENDIX

(Nonmandatory Information)

X1. A TECHNIQUE FOR DETECTING ALKALI-SILICA GEL SUBJECT TO CONFIRMATION BY OTHER METHODS

X1.1 Scope

X1.1.1 This technique has been used for detecting alkali-silica gel resulting from reactions of forms of silica in aggregates with alkalies in portland-cement paste. The technique involves treating the surface of conditioned concrete with a solution of uranyl-acetate and observing the treated surface exposed to short-wave ultraviolet light.

X1.1.2 This technique is one of a number of procedures that may be used to identify if alkali-silica reactions have occurred in hardened concrete. The test is ancillary to more definitive petrographic examinations and physical tests for determining concrete expansion. The petrographic examinations include identification of alkali-silica gel and the reactive aggregate component(s), as may be supplemented by electron microscopy and ancillary elemental probe analysis. The physical tests may include length change tests of prisms from the concrete.

X1.1.3 Fly ash, silica fume, and other materials that possess pozzolanic properties can react and produce secondary products similar to the gel produced by alkali-silica reaction (ASR). If these products are uniformly distributed, their effect is to create a uniform background on which the more localized and concentrated alkali-silica gel usually can be distinguished. Opal-containing materials and possibly other components of rocks, however, will also be detected. Localized concentrations of secondary ettringite have also been reported to fluoresce. Secondary ettringite is widespread as a component of concrete affected by many processes.

X1.2 Cautions

X1.2.1 Uranyl-acetate is a hazardous material that requires special handling and disposition. Ultraviolet light is hazardous to eyes and skin. Appendix X1 does not address the safety problems of handling, use, and disposition of uranyl-acetate and uranyl-acetate-treated concrete. Appendix X1 does not address the use and all cautions to be used regarding ultraviolet light applications and exposure to the eyes and skin.

X1.2.2 Before using uranyl-acetate and ultraviolet light involved in this technique, it is the responsibility of the user to establish adequate safety, health, and disposition of materials practices and procedures for handling uranyl-acetate and ultraviolet radiation. It is also the responsibility of the user to conform to all local, state, and federal regulatory requirements.

X1.2.3 Protective clothing, including polyethylene gloves, laboratory coat, and other clothing as may be needed should be used when handling uranyl-acetate and other chemicals.

X1.2.4 Short-wave ultraviolet light can be harmful to the eyes and skin. Ultraviolet-filtering goggles or face shields that will absorb the ultraviolet radiation should be used.

X1.2.5 Uranyl-acetate is classed by the United States Government as a hazardous material requiring special handling and disposition. Handling, storage, and disposition of the uranyl-acetate solution and materials to which it is applied and contacts must be in conformance to applicable regulations.

X1.3 Qualification

X1.3.1 This technique can be used by an adequately trained technician.

X1.4 Apparatus, Reagents, and Materials

X1.4.1 *Specimen Conditioning Apparatus:*

X1.4.1.1 *Plastic Beaker*—500-mL capacity or larger,

X1.4.1.2 *Plastic Squeeze Bottle (for Water)*—Nominal 250-mL capacity,

X1.4.1.3 *Disposable Polyethylene Gloves,*

X1.4.1.4 *Protective Clothing* (for example, laboratory coat),

X1.4.1.5 *Absorbent Paper Towels,* and

X1.4.1.6 *Enclosed Space for Applying the Reagent*—A fume hood, glove box, or disposable glove bags have been found to be suitable for this purpose.

X1.4.2 *Specimen Treatment Apparatus:*

X1.4.2.1 *Non-Atomized or Non-Misting Type Applicator Unit for Reagent* (for example, a small, hand-held squeeze bottle for applying dilute reagent solutions), and

X1.4.2.2 *Applicator for Water*—A small hand-held squeeze bottle for applying dilute solutions.

X1.4.3 *Reagent (Uranyl-Acetate Solution),*

X1.4.3.1 *Acetic Acid Solution (1N),*

X1.4.3.2 *Boiling Flask*— Nominal 250-mL capacity,

X1.4.3.3 *Distilled or Deionized Water,*

X1.4.3.4 *Volumetric Flask*—Nominal 100-mL capacity,

X1.4.3.5 *Polyethylene Storage Bottle with Tight Fitting Cap*—Nominal 100-mL capacity,

X1.4.3.6 *Uranyl-Acetate Powder (ACS Reagent Grade),*

X1.4.3.7 *Balance*—Capable of weighing up to 10 g within 0.05 g, and

X1.4.3.8 *Uranyl-Acetate Solution (prepared as follows)*—Weigh 5 g of powdered uranyl-acetate. Measure 100 mL of acetic acid solution using a 100-mL volumetric flask. (**Warning—**Use proper safety precautions in handling the uranyl-acetate and the acetic acid. Transfer the measured acetic acid solution from the volumetric flask to the boiling flask. Add 5 g of powdered uranyl-acetate to the boiling flask. Warm the mixture over a low flame until the powder is dissolved. Do not bring the mixture to a rolling boil. Remove the flask from the burner, cover, and let it cool to room temperature. Store the cooled solution at room temperature in a tightly capped polyethylene storage bottle. The reagent, when stored as described, will keep for at least one year.)

X1.4.4 *Short-Wave (254-nm) Ultraviolet (UV) Lamp,*

X1.4.5 *Viewing Cabinet or a Dark Room,* and

X1.4.6 *Goggles*—for shielding short-wave ultraviolet light.

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X1.5 Specimens

X1.5.1 Samples to be evaluated can be of any size or shape. The surface area for testing should have a nominal area of 155 cm^2 (24 $in.^2$) or sufficiently large to be representative of the concrete. The size will be dictated in part by the maximum size of the aggregate. The method is applicable to field and laboratory-prepared samples. The latter includes samples tested using Test Methods C227, C342, C441, and C1260.

X1.5.2 Break the specimen to expose the interior of the concrete using any suitable method, such as the splitting techniques of Test Method C496/C496M. Immediately conduct the test on the freshly exposed surfaces.

X1.6 Conditioning and Pre-Screening Procedure

X1.6.1 *Conditioning and Pre-Screening:*

X1.6.1.1 Dampen the freshly broken specimen surface using the squeeze bottle filled with distilled or deionized water. Proceed with the test while the specimen is still damp.

X1.6.2 Pre-screen the specimen for natural fluorescence.

X1.6.2.1 Wear protective clothing including polyethylene gloves, laboratory coat, and UV-filtering goggles. Short-wave UV light can be harmful to the eyes and skin. Normal eyeglasses or plastic goggles or face shield will absorb the harmful rays.

X1.6.2.2 If a viewing cabinet is being used, place the UV lamp in the proper place on the viewing cabinet. Place the damp specimen inside the viewing cabinet. If a darkened room is being used, place the damp specimen on the table and turn off the lights.

X1.6.2.3 Turn on the short-wave UV light in the viewing cabinet or the darkened room, and shine it on the damp specimen. Use UV-filtering goggles and observe and note for future comparison whether any of the aggregates or other components fluoresce. Non-fluorescent aggregates will appear dark, while cement paste and naturally fluorescent aggregates, such as opal and some slags, will fluoresce mildly. Note the location and nature of any fluorescence.

X1.7 Uranyl-Acetate Treatment Procedure:

X1.7.1 Wear protective clothing including polyethylene gloves, laboratory coat, and UV filtering goggles.

X1.7.2 The test should be conducted only on freshly fractured interior surfaces. If the interior surface of the specimen is not damp, dampen it using the squeeze bottle filled with distilled or deionized water.

X1.7.3 Fill the squeeze bottle to about half its capacity with the prepared uranyl-acetate solution.

X1.7.4 Line the bottom of the glove box, glove bag, or fume hood with absorbent paper towels. Place the squeeze bottles containing the distilled or deionized water and reagent, the 500-mL plastic beaker, and the dampened specimen inside the glove box, glove bag, or fume hood.

X1.7.5 Place the specimen in a horizontal position on a paper towel. Apply the uranyl-acetate solution to the wet interior surface of the specimen. Direct the squeeze bottle nozzle on the specimen surface at all times. The nozzle should be kept approximately 25 mm (1 in.) away from the surface. The bottle pressure should be kept to a minimum to avoid misting and rebound from the specimen surface.

X1.7.6 Allow the uranyl-acetate to absorb on the interior surface of the concrete for 1 min.

X1.7.7 After 1 min, hold the specimen in a vertical position over the 500-mL plastic beaker and wash the treated surface three times with distilled or deionized water to remove excess uranyl-acetate. Collect the wash water from the specimen in the plastic beaker.

X1.7.8 Remove the specimen from the glove box, glove bag, or fume hood, and place it on a paper towel. Proceed with observation of the specimen immediately after the uranyl-acetate treatment is completed.

X1.8 Ultraviolet Exposure of Treated Surface

X1.8.1 If a viewing cabinet is being used, place the UV lamp at the proper place in the viewing cabinet. Line the bottom surface of the viewing cabinet with paper towels. If a dark room is being used, line an area of a table top with paper towels.

X1.8.2 Dampen the specimen again using the squeeze bottle filled with distilled or deionized water. Place the specimen inside the viewing cabinet on the paper towel. If a dark room is being used, place the wet specimen on the paper towel on the table top in the dark room and turn off the lights.

X1.8.3 Shine the UV light on the treated surface. Do not view, and do not allow anyone to view the UV light without eye protection.

X1.9 Identification of Alkali-Silica Gel

X1.9.1 Alkali-silica gel will fluoresce bright greenish-yellow, and usually occurs in and around aggregate particles, in voids, and in cracks. It has been reported that ettringite may similarly fluoresce.

X1.9.2 Carbonated areas of concrete also may fluoresce. The fluorescence due to carbonation products, however, is usually uniform and normally present along concrete surfaces exposed to the atmosphere. Carbonation may also be present where there is extensive deterioration or cracks, or both, which allow atmospheric carbon dioxide into the interior of the specimen.

X1.9.3 Some fly ash, silica fume, slag, and natural pozzolans can react and form reaction products that fluoresce. Well dispersed fly ash and silica fume and fine ground slag and natural pozzolans, however, cause only an increase in the generally uniform background fluorescence intensity of the paste. If these materials are not well dispersed or are clumped together, the fluorescence will be observed as discrete fine areas. The size of these areas is normally very small and usually is distinguishable from the bright fluorescence of gel derived from the coarse and fine aggregates.

X1.9.4 Identification of the presence of alkali-silica gel using this technique must be confirmed by other petrographic techniques, such as microscopical examinations of thin sections or powder mounts, or both.

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X1.9.5 Literature on the development and use of this technique is given in Refs (**30-36**).

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SUMMARY OF CHANGES

Committee C09 has identified the location of selected changes to this standard since the last issue (C856 – 13) that may impact the use of this standard. (Approved June 1, 2014.)

(1) Revised the mandatory Annex to a non-mandatory Appendix; revised and removed references to Appendix X1 throughout.

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