

Designation: C856 - 11 C856 - 13

Standard Practice for Petrographic Examination of Hardened Concrete¹

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.
- 1.3 Annex A1 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.
 - 1.4 The purposes of and procedures for petrographic examination of hardened concrete are given in the following sections:

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- 1.5 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.
- 1.6 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

¹ This practice is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregatesand 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.

TABLE 1 Visual Examination of Concrete (1)6

Coarse Aggregate	+ Fine Aggre	gate	+ Matrix	+ Air	+ Embedded Items
Composition: Maximum dimension, ^A in. or mm, in the range> d>					
Type:	Туре:		color, by comparison with National Research Council Rock Color Chart (1963)	more than 3 % of total,	Type, size, location; kinds of metal; other items
1 Gravel 2 Crushed stone 3 Mixed 1 and 2	1 Natural sand 2 Manufactured san 3 Mixed	d	color distribution:	predominantly in spherical voids? less than 3 % of total,	
4 Other (name) 5 Mixed 1 + /or 2 + /or 4 If Type 1, 2, or 4, homogeneous	4 Other (name) 5 Mixed 1 + /or 2 + /	or 4	2 even 3 gradational changes	abundant nonspherical voids?	
or heterogeneous	homogeneous or heterogeneous			voids and mortar?	
Lithologic types Coarse aggregate more than 20, 30, 40, or 50 % of total				voids empty, filled, lined, or partly filled	
Fabric: Shape	distribution	١	distribution	shape	voids below horizonta
Distribution Packing Grading (even, uneven,	particle shape	as per-		distribution grading (as perceptible) parallelism of long axes of	or low-angle reinforcement
	grading	ceptible			
	preferred orientation)			
excess, or deficiency of size or sizes) Parallelism of flat sides or long axes of exposed				irregular voids or sheets of voids: with each other; with flat sides or long axes of coarse aggregate	
sections, normal to direction of placement + /or parallel to formed and finished surfaces ^B					
Condition: Does it ring when hit lightly with a	Dog	CUM	ent Previo	Overale 2 Herry distribute do	clean or corroded? Are cracks associated

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?

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

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 photos⁴

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.

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

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



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 percep-	Lithologic types and miner-	Color	Grading
tible	alogy as perceptible	Fracture around or through aggregate	Proportion of spherical to nonspherical
Surface texture	Shape	Contact of matrix with aggregate:	Nonspherical, ellipsoidal, irregular, disk
Within the piece:	Surface texture	close, no opening visible on sawed	shaped
Grain shape	Grading	or broken surface; aggregate not	Color change from interior surface to
Grain size extreme range observed, mm	Distribution	dislodged with fingers or probe;	matrix
Median within range _ to _ mm		boundary openings frequent,	Interior surface luster like rest of ma-
Textureless (too fine to resolve)		common, rare	trix, dull, shining
Uniform or variable within the piece		Width	Linings in voids absent, rare, common,
From piece to piece:		Empty	in most, complete, partial, colorless,
Intergranular bond		Filled	colored, silky tufts, hexagonal tab-
Porosity and absorption ^B		Cracks present, absent, result of spec-	lets, gel, other
If concrete breaks through aggregate,		imen preparation, preceding spec-	Underside voids or sheets of voids un-
through how much of what kind?		imen preparation	common, small, common, abundant
If boundary voids, along what kind of		Mineral admixtures ^C	
If boundary voids, along what kind of		Supplementary Cementitious Materials ^C	
aggregate? All? All of one kind? More		Contamination	
than 50 % of one kind? Several kinds?		Bleeding	
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 x _, or sucks in water that is dropped on it.

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3. Terminology

3.1 Definitions:

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

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 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.

^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.



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	Determinations by compressive tests and static modulus of cores; Test Method C805 for qualitative determination; Test Method C597 (2).
	570 80 Reduction in Modulus	
	Temperature,	· ·
	430 50	
	760 70	

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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.
- 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 a mineral admixture, 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.

TABLE 4 Outline for Examination of Concrete in Thin Sections

Coarse and Fine Aggregate

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).

Relict Cement Grains and Hydration Products

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 C₃S and C₂S). 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 vellow 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.

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Characteristics of Cement Paste

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 SO₃ 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.

- 5.2.7 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.
 - 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.

TABLE 5 Characteristics of Concrete Observed Using Microscopes

01		Type of Microscope		
Characteristic	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		
	X	X		
degree				
products	X	X	•••	
Coatings	X	X		
Rims	X	Χ	X	
Internal cracking	X	X	•••	
Contamination	X	X		
Concrete:				
Air-entrained or not	X	Χ	X	
Air voids				
shape	 X	 X	 X	
·				
size	X	Χ	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	Teh Standards			
degree and type	1ch Standa _x us	X	X	
reaction products				
location	://standardxiteh	X	X	
identification		\mathbf{x}		
Nature and condition of surface treatments	V V	X		
	^	^	•••	
laste:	cument Preview			
Color	CHILCHI I I CALCAA	Χ		
Hardness	X	•••	X	
Porosity	X		X	
Carbonation	X	X		
Residual cement				
distribution		- 1 - 0 X	X	
particle size standards.iteh.ai/catalog/standa	ards/sist/2c102740-6e33-4200-81fc	-e5d5f9 x 1ca0e/a	stm-c856 x 13	
abundance		X	X	
composition	 VP	X	X	
Mineral admixtures	X B	×	×	
Supplementary cementitious materials	<u>X</u> B	<u>X</u>	<u>X</u>	
size		$\frac{X}{X}$	$\frac{X}{X}$	
abundance	X	X	X	
identification	X	X	X	
Compounds in hydrated cement	χ^c	X	X	
Contamination	^	Α	^	
	V	V	V	
size	X	X	X	
abundance	X	Χ	X	
identification	***	X	X^D	

^A Secondary ettringite can sometimes be recognized by crystal habit and silky luster.

- 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:

^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.



TABLE 6 Secondary Deposits in Concrete^A

Compound and Mineral Equivalent	Indexes of Refraction	Form and Occurrence
Calcium carbonate (CaCO ₃); calcite	$\omega = 1.658$ $\varepsilon = 1.486$	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	$\alpha = 1.530$	Minute, white prisms or needles in voids or fractures in
($\beta = 1.680$	concrete; rare
	$\gamma = 1.685$,
Calcium carbonate (CaCO ₃); vaterite	o = 1.544 - 1.550	Spherulitic, form-birefringent, white encrustations on
	E = 1.640–1.650	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) $_6$] $_2$ ·24H $_2$ O}(SO $_4$) $_3$ ·2H $_2$ O (6); ettringite	$\omega = 1.464 - 1.469^{B}$ $\varepsilon = 1.458 - 1.462$	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-	$\omega = 1.504$	White to colorless, minute, hexagonal plates in voids and
$Al_2O_3\cdot CaSO_4\cdot 12H_2O)$	$\epsilon = 1.49$	fractures; very rare (5)
Tetracalcium aluminate-13-hydrate ($Ca_4Al_2(OH)_{14}\cdot 6H_2O$)	$\omega = 1.53$	Micalike, colorless, pseudohexagonal, twinned crystals in
	$\varepsilon = 1.52$	voids; very rare (7)
Hydrous sodium carbonate (Na ₂ O·CO ₂ ·H ₂ O);	$\alpha = 1.420$	Minute inclusions in alkalic silica gel; rare (5)
thermonatrite	$\beta = 1.506$	
	$\gamma = 1.524$	
Hydrated aluminum sulfate (2Al ₂ O ₃ ·SO ₃ ·15H ₂ O);	$\alpha = 1.463 \pm 0.003$	Occurring in cavities in intensely altered concrete; very
paraluminite	$\beta = 1.471$	rare (7)
Coloium cultate dibudusta (CoCO, OLL O), surroum	$\gamma = 1.471$	White to coloulose emistale in voide in the coment poets
Calcium sulfate dihydrate (CaSO ₄ ·2H ₂ O); gypsum	$\alpha = 1.521$ $\beta = 1.523$	White to colorless crystals in voids, in the cement paste, or along the surfaces of aggregate particles in concrete
	$ \gamma = 1.525 $ $ \gamma = 1.530 $	or mortar affected by sulfate or seawater attack; unusual
Calcium hydroxide (Ca(OH) ₂); portlandite	$\omega = 1.574$	White to colorless, hexagonal plates or tablets in the
	ε = 1.547	cement paste, in voids, along fractures; ubiquitous in concrete
Magnesium hydroxide (Mg(OH) ₂); brucite	$\omega = 1.559$	White to yellow, fine-grained encrustations and fillings in
	$\epsilon = 1.580$	concrete attacked by magnesian solutions or seawater; unusual (8, 9)
Hydrous silica (SiO ₂ ·nH ₂ O); opal	$\eta = 1.43$	White to colorless, finely divided, amorphous; resulting
	varies with water control	from intense leaching or carbonation of cement paste; unusual in recognizable proportions
Alkalic silica gel (Na ₂ O·K ₂ O·CaO·SiO ₂)	OCUM $e^{\eta} = 1.46-1.53$	White, yellowish, or colorless; viscous, fluid, waxy, rubbery, hard; in voids, fractures,
		exudations, aggregate; common (10, 11)
Hydrated iron oxides (Fe ₂ O ₃ ·nH ₂ O); 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)	$\Delta S T = 1.504 \\ \epsilon = 1.468 \pm 0.002^{B}$	Prismatic, hexagonal; capable of growing in continuity with ettringite; in sewer pipe subject to sulfate attack, in
Syngopita (K Ca/SO) (H ols. iteh. ai/catalog/sta	ndards/sist/2c102740-6e33-4	grout, in some pavement (12)
Syngenite (K ₂ Ca(SO ₄) ₂)·H ₂ O	$\alpha = 1.501 (13)$	Found in cavities and zones peripheral to slate particles, in
	$\beta = 1.51$	fibrous form (14)
Hydrotaleita Ma Al (OH) (CO) (H O) (6)	$\gamma = 1.51$	Foliated platy to fibrous masses (15.6)
Hydrotalcite Mg _{3/4} Al _{1/4} (OH) ₂ (CO ₃) _{1/8} (H ₂ O) _{1/2} (6)	$\omega = 1.510 \pm 0.003$ $\varepsilon = 1.495 \pm 0.003$	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.

- 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. The technique in Annex A1 is helpful for identifying locations where alkali-silica gel may be present.
- 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, 16, and 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.
- 5.5.5 To establish the effects of a freezing and thawing test or other physical or mechanical exposure of concrete on the aggregate and the matrix.

^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.

⁵ The boldface numbers in parentheses refer to the list of references at the end of this practice.



- 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.

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 needed to perform the examinations in Annex A1 is listed therein. 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.
- 6.2.9 *Plate-glass Squares*, 12 to 18-in. (300 to 450-mm) on an edge and at least 3/8 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). The uranyl-acetate technique given in Annex A1 can be helpful in locating sites where alkali-silica gel may be present.

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 2½-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 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. A broad overview of possible locations where alkali-silica gel may be present can be obtained using the uranyl-acetate method outlined in Annex A1.
- 9.3 <u>PhotographyPhotographs</u>—It is frequently desirable to maintain a photographic record as illustrations for the report and for future reference. Photographs which may be useful include: 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.
- 9.3.1 Overlapping close-ups of a core or cores, laid out in sequence, including a dimensional scale. It is convenient to use a view camera at a constant distance using the same lighting in all photographs.
- 9.3.2 Photographs and photomacrographs of features of interest on a core surface, a ground surface, or an old or new crack surface, at magnifications of 0.7× to 3× are particularly useful if the specimen will be destroyed during subsequent studies. A camera equipped with a long extension bellows and lenses of focal length from 6.5 to 2.9 in. (165 to 74 mm), and a ground-glass back, is desirable.
- 9.3.3 Photomacrographs at magnifications of at least 3× to 10×, and sometimes at higher magnifications, may be used to illustrate reaction products or growths on the specimen or significant crack patterns. Black and white photographs such as 4 by 5 in. (102 by 127 mm) taken at 1× magnification on fine-grained film with a long gray scale, convey a great deal of information and may be more useful than a photograph taken at a higher magnification on smaller format. As a general guide, the subject should be illuminated by oblique lighting at a low angle if the relief on the subject surface is low, and at a higher angle if the relief is high. The object of the oblique illumination is to reveal detail and contrast without obscuring features by long shadows. Details can also be enhanced by the use of colored filters such as Wratten G or other yellow filters. Cross lighting from two sides is sometimes successful in revealing detail and obliterating confusing shadows.
- 9.3.4 Photomicrographs of thin sections, immersion mounts, and polished sections at magnifications from 2x to an upper limit appropriate to the subject and dependent on the quality of the equipment and the skill of the photographer may be used to illustrate textures, compounds, and crack patterns.