

**A Study of New Mexico Concrete and Aggregate:
Alkali-Aggregate Reactions**

by

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Thesis

**Submitted in Partial Fulfillment of the Requirement for
the Degree of Master of Science
in Geology**

**New Mexico Institute of Mining and Technology
Socorro, New Mexico**

August 1998

ACKNOWLEDGMENTS

Much thanks to Dr. George Austin, Dr. David Norman, and Dr. Andrew Campbell for their guidance and support on this project as well as throughout my experience at NMIMT.

I would like to extend thanks to William Barringer, of the New Mexico State Highway and Transportation Department for funding and support for this project. Additional support and information was given by Gordon McKeen and Lary Lenke of the Alliance for Transportation Research, a division of the University of New Mexico. Joe Barela and Bryce Simmons of the New Mexico State Highway and Transportation Department also provided background information on cement and concretes, as well as allowing the accelerated mortar bar tests to be completed at the Central Materials Laboratory in Santa Fe. Also in Santa Fe, I would like to thank John Garcia, Joel Archuleta and Gary Apodaca for running the mortar bar tests. I thank William Carey and George Guthrie for the information on the staining method for the identification of the ASR gel. The city of Gallup has provided information about concrete in the area as well as allowing concrete sampling. Thanks also to the Physical Plant at New Mexico Institute of Mining and Technology for allowing me to sample the concretes on campus and for providing information about the concretes. Nelia Dunbar has provided help and training in using the scanning electron microprobe, and Bill Macintosh has allowed use of the drill core equipment.

Also, thanks to Michiel Heynekamp and Bill Lefevre for their computer knowledge and expertise.

ABSTRACT

Many of the concrete structures in New Mexico are deteriorating in 5 to 10 years instead of lasting the 30 to 50 years they are designed to last. There has been much research by state engineers to slow or stop this deterioration. Controversy exists about what reactions are causing the distress in the concretes. The purpose of this study is to (1) examine existing concrete to identify which reactions are causing the distress and examine the mineralogy of the aggregate in the concrete, and (2) examine local aggregate that is going into concrete to identify the aggregate mineralogy, characteristics, and reactivity in concrete.

Concrete is made up of cement, aggregate, water, and additives/admixtures. The cement is basically a calcined mixture of alumina, silica, iron oxide, and calcium oxide. The heating of these raw materials creates clinker compounds, each with their own properties that add to the concrete. Aggregate is added for extra strength and as a filler. It is designed to be a nonreactive ingredient, however, reactions can occur between the aggregate and cement paste that cause deterioration. Additives and admixtures are added to a concrete to produce a specific property, or to add cementitious material. The two main reactions that can cause a concrete to decay include the alkali-silica reaction (ASR) that occurs between alkali hydroxides from the cement and the siliceous aggregate, and the delayed ettringite formation (DEF) that forms ettringite crystals from an excess of sulfate.

Concrete samples were taken from a number of structures including sidewalks, parking lots, drainage ditches, and a railroad trestle. They were analyzed using the scanning electron microprobe (SEM), petrographic microscope, and a chemical staining test. The aggregate was collected from several pits and quarries in New Mexico and analyzed using x-ray diffraction, the petrographic microscope, and an accelerated mortar bar expansion test. The concrete samples contain varying amounts of potentially reactive aggregate including chert/chalcedony, rhyolite, andesite, and quartz. However, all that have reactive aggregate do not show the ASR gel. There are three explanations for the discrepancy. First, additives or admixtures may have been used in the mixture to counteract ASR, second, there may not have been enough time for the reaction to occur, or third, there may have been an inadequate amount of water available to start the reaction. Ettringite formation does not seem to be a problem for the New Mexico concretes that were sampled. The aggregate sampled contains a large percentage of potentially reactive aggregate types. Reactive particles include rhyolite, andesite, chert/chalcedony, quartz, and weathered material. The mortar bar expansion tests correlate directly to the amount of reactive aggregate in the sample.

Concrete deterioration appears to be caused mainly by ASR. Reactive aggregate needs to be used in conjunction with admixtures or additives. More work needs to be done to completely understand the mechanisms and causes of the concrete deterioration in New Mexico and elsewhere.

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INTRODUCTION

Many of the concrete structures built in New Mexico in the past 20 years have problems with premature deterioration. These structures, which were designed to last 30 to 50 years, need replacement after only 5 to 10 years. A great deal of research by the New Mexico State Highway and Transportation Department and other engineering groups has been conducted to find a way to slow or stop concrete decay by adding chemicals and other substances to the concrete mixture. As a result, today's concrete is made with numerous additives that did not exist many years ago. Much controversy remains about various deleterious reactions as the principle causes of the deterioration. Many types of aggregate or aggregate pits are labeled as having reactive material without much proof or experimentation on why the aggregate from a certain pit may be reactive in concrete.

Purpose of study

As a geologist, my goal with this project is (1) to examine existing concrete to see what kind of aggregate it contains as well as what type of deterioration, if any, it has undergone; and (2) to examine the aggregate that is currently being used in New Mexico concrete to compare the aggregate's mineralogy, characteristics, and reactivity in concretes. This research will provide more insight into the geological reasons why New Mexico concrete is not lasting as long as it should, and help in the labeling of aggregate pits with potentially reactive aggregate.

BACKGROUND

Concrete

Concrete used to build bridges, highways, and building foundations is a mixture of cement, water, and aggregate, both sand-size and gravel-size. Numerous additives and admixtures are also added to produce specific properties. In order to understand the reactions that occur in a hardening concrete, the chemistry and properties of the cement and aggregate must first be understood.

Cement

Cement manufacture

Cement is the most expensive ingredient in concrete, but usually only amounts to about 10% of the mixture. Cement is made from five basic ingredients, including calcium and iron oxide, alumina, silica, and gypsum. Each of the first four comes from a variety of raw materials. The most common sources of calcium are limestone, chalk, marl, and marine shells. Alumina and silica generally are supplied by clay, shale, slate, or blast-furnace slag (Bouge, 1947). Iron oxide comes from magnetite, hematite, or bottom ash—a byproduct from coal-fired power plants. The alkali elements from raw materials must be kept to a minimum to ensure a cement with around 0.5 to 1.3% total alkalis, to limit adverse reactions with silicious aggregate that could occur in the concrete mixture. Most cements are limited to the American Society of Testing and Materials (ASTM) specified 0.6% alkalis. Other impurities are allowed, but must be limited to strict minimum percentages to maintain the correct properties of the cement. For example, magnesium

oxide is restricted to under 5%, and free lime (CaO) is kept under 2%, to maintain soundness. However, the free lime content must exceed 0.5% to preserve correct setting time.

The proportioned raw materials are combined and heated to approximately 2700°F in a large (usually 100–500 ft long) rotating, inclined kiln (Fig. 1). This length and temperature allows the chemical changes to occur that produce the clinker “minerals” (man-made compounds) in the last few 10s of feet of kiln. At the end of the kiln, the clinker is broken up and spread out on a 40-ft-long grate with an upward air-flow for cooling. This stage is very important to keep the chemistry from changing. The chemical changes that make the clinker are established and have reached an equilibrium during the heating process. If cooling is too fast or slow, the ratios of compounds may change due to the disruption of the equilibrium (Soroka, 1979). At a too fast cooling rate, the 20–30% liquid phases of two of the clinker minerals can quench to a glass instead of maintaining their composition. At a slower rate, each mineral will have a chance to crystallize (Austin and Barker, 1998). The correct cooling rate will ensure the correct ratios of all the clinker compounds that crystallize. After cooling, gypsum is added, all the materials are ground to a fine powder in a ball mill, and are bagged, stored, or shipped.

Cement chemistry

The clinker “minerals” include tricalcium silicate, or C_3S , called alite ($3CaO \cdot SiO_2$); dicalcium silicate, or C_2S , called belite ($2CaO \cdot SiO_2$); tricalcium aluminate, or C_3A , ($3CaO \cdot Al_2O_3$); and tetracalcium aluminoferrite, or C_4AF , called celite ($4CaO \cdot Fe_2O_3 \cdot Al_2O_3$)

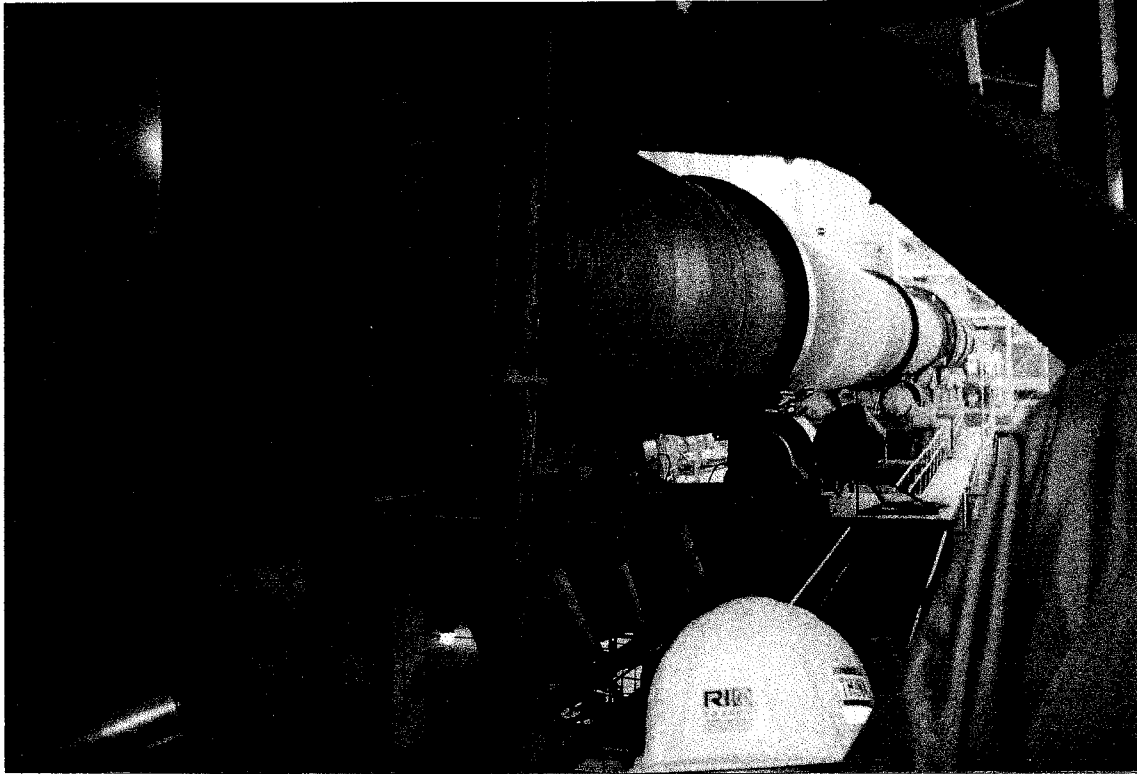


Figure 1. Rotating, inclined cement kiln at the Rio Grande Portland Cement Corporation in Tijeras, New Mexico. Diameter of kiln is approximately 15–20 ft.

(Soroka, 1979). The “minerals” are not exact chemical compositions since they are man-made and may contain impurities, but are often referred to as such for simplicity.

Similarly, C_3S , C_2S , C_3A , and C_4AF are commonly used as designations, not chemical formulas. These compounds have several phases of different crystal structure, each of which are stable at different temperatures.

Each “mineral” has its own properties and contributes differently to the strength and composition of the final concrete (cement + aggregate + water) product. C_3S is perhaps the most important, as it is most abundant in the common types of cement. It hydrates in about six hours, and has maximum strength in a few days (Bouge, 1947; Soroka, 1979). C_2S will hydrate only about 50% in 28 days and will continue to build strength over many years. Some research indicates that the C_2S continues to hydrate and harden for at least 50 years after initial mixing (Soroka, 1979). C_3A will instantaneously hydrate. Gypsum is added to cement clinker to form the mineral ettringite ($Ca_6Al_2(SO_4)_3OH_{12} \cdot 26H_2O$) during hydration. Ettringite initially coats the C_3A grains, but breaks up over time allowing hydration of C_3A . This reaction is important to delay the instant set to allow time to lay and work with the concrete. The C_4AF hydrates in minutes, but imparts no significant strength to concrete. Its main properties are to give the cement a gray color and to lower the clinkering temperature by acting as a flux (Soroka, 1979).

Aggregate in concrete

Aggregate in portland cement concrete is used to supply added strength and to act as a filler. The aggregate should be non-reactive in the mixture. This is not always the

case, as some aggregate can react with the cement to form expansive gels in the highly alkaline environment. Aggregate can be reactive for several reasons. An unstable or microcrystalline quartz phase, residual clay on the surface of particles, and certain other minerals associated with the aggregate itself can make it reactive in the alkaline cement-and-water solution. According to Kosmatka and Panarese (1988), many rocks and minerals, particularly some forms of quartz, will react with potassium and sodium ions in solution in hydrating cement to form an unstable gel (see Table 1). Some will react faster than others due to the increased surface area of finer particles, which can give alkaline solutions more access to unsatisfied charges. Particles can be made more reactive by weathering, crushing, or the presence of residual clays on particles surfaces.

Table 1. Potentially deleterious rocks and minerals in concrete.
After Kosmatka and Panarese, 1988, Table 4-5.

andesite
argillite
certain siliceous limestones and dolomites
chalcedonic cherts
chalcedony
cherts
cristobalite and tridymite
dacite
glassy or cryptocrystalline volcanics
granite gneiss
graywacke
metagraywacke
opal and opaline shales
phyllite
quartzite
rhyolite
schists
siliceous shales
strained quartz and certain other forms of quartz
synthetic and natural siliceous glass

Additives and admixtures in concrete

Along with cement, water, gravel, and sand, certain materials are added to concrete to produce specific properties. Generally, either the material will be involved in the reactions and add to the product (additives), or it will be involved in the chemical reactions, but only change the outcome slightly (admixtures) (Taylor, 1997). Additives have some cementing properties of their own (act as a pozzolan) and can replace some of the cement in the concrete mixture. They are sometimes added during the grinding of the clinker minerals.

Additives include natural pozzolans, ground granulated blast-furnace slag, flyash, and silica fume. Natural pozzolans include volcanics, mainly highly siliceous ash and tuff, zeolites, and some heat-treated clays. Expansion due to deleterious reactions can be decreased, and strength can be increased by replacing some of the sand with pozzolan in the concrete mixture (Taylor, 1997). Blast-furnace slag, when cooled quickly, will form a glass that has hydraulic cement properties (it will harden under water). The slag is ground and added to cement, and when hydrated, it will form calcium silicate hydrate gel (C-S-H, the main constituent of concrete) (Taylor, 1997). Flyash is a byproduct of a coal-burning power plant, and consists of glass, iron oxide, aluminum oxide, and quartz. When a low-carbon flyash, less than 15% carbon, is hydrated in a concrete mixture, it will produce C-S-H by contributing silica and aluminum oxide. In addition, it can reduce the effects of sulfate attack, which can cause concrete distress. Lithium salts, such as lithium carbonate, lithium hydroxide, lithium fluoride, and lithium chloride, have been used in conjunction with flyash to help combat concrete expansion. Silica fume, a glass, is produced in an

electric furnace during the process of reducing quartz to manufacture silicon. The fine-grained silica fume particles that have pozzolanic properties can create a denser cement paste, thereby increasing the compressive strength and lowering the permeability (Taylor, 1997).

Admixtures are added to the concrete mixture, but do not have hydraulic cement properties. They can include air entrainers, water reducers, accelerators, and retarders. These materials generally are added at the time when the cement, water, and aggregate is mixed together.

Air entrainers are generally fatty acids, sodium salts, or other organic materials. Their main purpose is to provide freeze/thaw resistance by allowing spaces for the expansion of freezing water without stressing the concrete. Typically, air voids should be about 3–6% by volume of the concrete and be in the range of 10–250 μm in diameter to be effective against freeze/thaw damage (Rixon and Mailvaganam, 1986; Taylor, 1997). Water reducers, typically calcium and sodium lignosulfonates, are used to increase the workability of a concrete at a lower water/cement ratio. Superplasticizers are water reducers in the extreme, in that they allow for greater workability, such as self-leveling and pumping of concretes (Rixon and Mailvaganam, 1986; Taylor, 1997). Accelerators such as calcium chloride, calcium formate, and triethanolamine act to increase the rate of hardening, or decrease setting time, in concretes. Typically, they are used in colder winter months, but also are added to counteract a water reducer's retardation effect (Rixon and Mailvaganam, 1986). Organic materials such as sucrose, calcium citrate, and calcium lignosulfonate act as retarders to slow the setting of concretes, mainly in hot weather

(Taylor, 1997). Other specialty admixtures can be used for specific properties and under specific conditions.

Chemical reactions in concrete

Normal hydration chemistry

During the exothermic reaction of a hydrating cement, the clinker minerals change chemically as they react with water. One of the major compounds formed is calcium hydroxide ($\text{Ca}(\text{OH})_2$), which is produced from the reaction of water, C_3S , and C_2S . Also from the reaction of clinker minerals and water, hydroxyl ions and calcium silicate hydrate gel are formed (C-S-H). Other ions in the cement paste solution originating from the clinker minerals and gypsum include Ca^{+2} , Na^+ , K^+ , and SO_4^{-2} (Stark and others, 1993). Pore solution chemistry evolves due to reactions between ions and the minerals formed.

Alkali-silica reaction

A normal pore solution in cement contains a significant amount of sodium and potassium hydroxide after about a day of hydration (Stark and others, 1993). Normally, the C-S-H gel that forms absorbs a small amount of the alkalis from solution. However, if the cement hydrate solution has too large an amount of sodium and potassium, a very alkaline solution with high amounts of hydroxyl ions is created that can chemically break down siliceous aggregate sometimes used in concrete. According to Stark and others (1993), the result is an alkali-silica reaction (ASR), which forms an alkali-rich silicate hydrate gel (C-N+K-S-H). This gel will then swell in the presence of water and can cause

concrete to crack and deteriorate. A gel with a CaO content of less than about 53% and a SiO₂ content greater than about 40% will swell.

The concentrations of ions in solution and their reactions are altered by external and internal sources of additional ions. Additives and admixtures, i.e., silica fume, flyash, pozzolans, water reducers, and accelerators, and external chemicals like deicer salts and sulfates from soils, can cause changes in concentrations of ions or changes in pH that affect ASR. The additives like flyash and pozzolans have been reported to decrease the severity of ASR by promoting non-swelling, high-calcium gel formation. The pozzolanic additives act as sacrificial silica by reacting with, and consuming, the alkalis in solution allowing for the reaction product to become richer in calcium. This is different than the reaction with aggregates, because many siliceous aggregates contain alkalis that can be released during the breakdown of the aggregate. In this case, the reaction continues to produce an alkali-rich solution. Some external chemicals like sea and deicer salts tend to increase ASR by supplying additional alkalis into the concrete through micro-cracks and pores (Stark and others, 1993). Many aspects of the alkali-silica reaction are still not completely understood; however, the basic ingredients, alkalis, silica, and water, must be present for it to occur.

Delayed ettringite formation

A second concrete deterioration problem may be due to delayed ettringite formation (DEF). Some ettringite formation during initial hydration is needed in order to delay the instantaneous hydration of C₃A, but ettringite that forms later in a hardened

concrete may cause damage. If ettringite crystals grow in a site with no space to expand, except to create their own space, the crystals can strain and crack the concrete structure. In order for ettringite to grow, there needs to be an abundance of sulfate, either from internal or external sources. This may be supplied internally from an excess of gypsum, impurities from the cement raw materials, or from aggregate, or externally from a surrounding soil.

METHODS

To characterize and study New Mexico's concrete deterioration problems, samples of existing concrete structures were examined to determine the mineralogy of aggregate used and to document any reactions that may be causing deterioration. Aggregate from several New Mexico quarries and pits was collected to determine mineralogy, geology, and reactivity in concrete.

Concrete

Sample sites

The concrete samples were taken from a number of sources in New Mexico and range in age from 1923 to 1997 (Table 2). Samples 1 to 11 were taken from the New Mexico Institute of Mining and Technology (NMIMT) campus in Socorro and include sidewalks, driveways, and parking lots. Samples 12 to 17 were taken from the Gallup area, and include a railroad trestle support, a drainage ditch and an arroyo spillway. Samples 18 to 26 were taken from a section of Gibson Boulevard, in Albuquerque. A soil

sample was taken to determine the pH of the area for all concretes sampled near a soil.

Records of the alkali concentration of the cement used or the source of the aggregates for the concretes could not be located. Therefore, a low-alkali percent (<0.6%) was assumed for the cements dated after the early 1970s, because most cements made in New Mexico after the early 1970s had a low alkali content. The earlier cements may have higher alkali contents, but no records confirm that. Petrographic analysis was used to determine aggregate mineralogy. Drainage and moisture conditions were noted for each site (Table 2).

Table 2. Concrete sample core locations, age, visual distress, drainage conditions, and pH of surrounding soils.

Sample	Location	Structure	Age	Distress	Drainage Conditions	Soil pH
1	Soc., G. Austin's house	sidewalk	1960s	none	sprinkler daily, drains	8.03
2	Soc., G. Austin's house	sidewalk	1960s	none	sprinkler daily, drains	8.03
3	Soc., McCutcheon Ave.	sidewalk	1936	none	runoff from lot, drains	NA
4	Soc., NMT, library	sidewalk	1988	map cracking	sprinkler daily, poor drainage	7.77
5	Soc., NMT, Brown Hall	sidewalk	1988	popouts	sprinkler daily, drains	8.12
6	Soc., NMT, Brown Hall	sidewalk	1988	popouts	sprinkler daily, drains	8.12
7	Soc., NMT, Brown Hall	sidewalk	1988	popouts	sprinkler daily, drains	8.12
8	Soc., NMT, Golf course	parking lot	1993	none	rain runoff only, drains	7.96
9	Soc., NMT, Workman	sidewalk	1997	none	rain runoff only, drains	7.76
10	Soc., NMT, Weir Hall	sidewalk	1939	map cracking	rain runoff only, poor drainage	NA
11	Soc., NMT, Gym	driveway	1988	popouts	sprinkler daily, puddles	7.53
12	Gal., train trestle	drainage	1923	none	rain runoff only, drains	NA
13	Gal., train trestle	drainage	1923	none	rain runoff only, drains	NA
14	Gal., train trestle	drainage	1923	none	rain runoff only, drains	NA
15	Gal., drainage channel	wall	1977-78	none	rain runoff only, drains	NA
16	Gal., spillway	drainage	1965-68	none	rain runoff only, drains	NA
17	Gal., spillway	drainage	1965-68	none	rain runoff only, drains	NA
18	Alb., Gibson Blvd.	street	1979	none	rain runoff only, drains	NA
19	Alb., Gibson Blvd.	street	1979	none	rain runoff only, drains	NA
20	Alb., Gibson Blvd.	street	1979	none	rain runoff only, drains	NA
21	Alb., Gibson Blvd.	street	1979	none	rain runoff only, drains	NA
22	Alb., Gibson Blvd.	street	1979	none	rain runoff only, drains	NA
23	Alb., Gibson Blvd.	street	1979	none	rain runoff only, drains	NA
24	Alb., Gibson Blvd.	street	1979	none	rain runoff only, drains	NA
25	Alb., Gibson Blvd.	street	1979	none	rain runoff only, drains	NA
26	Alb., Gibson Blvd.	street	1963	map cracking	rain runoff only, drains	NA

Note: Soc.= Socorro, NA = data not available, NMT = New Mexico Institute of Mining and Technology, Gal.= Gallup, Alb.= Albuquerque.

Soil samples

The soil pH was measured using the procedure for noncalcareous soils measured in water, at the New Mexico Bureau of Mines and Mineral Resources (NMBMMR) Chemistry Laboratory.

Sample preparation

All concrete samples were drilled cores of 1-in. diameter, approximately 2–4 in. long. A hand-held, electric drill, converted to receive power from a 12-volt battery, with a water connection for cooling the bit was used. The cores were cut using a small water-cooled saw into circular sections for petrographic thin sections and electron microprobe slabs. The thin section samples were sent to Spectrum Petrographics, Inc., in Winston, Oregon, to be made into 30- μm thin sections. The SEM slabs were attached to 1-in. round glass slides with epoxy, the surface was polished with a diamond wheel from 60-grit down to 2 μm diamond grit, and they were carbon-coated for the microprobe. A second set of microprobe slabs was left with the sawn surface and coated with carbon.

Scanning electron microprobe analysis

The polished samples were analyzed using a Cameca SX-100 high-vacuum scanning electron microprobe (SEM) using backscatter electrons (BSE). Some elemental maps were constructed of areas believed to be ettringite, or other crystalline phases. Sawn samples were analyzed using secondary electrons (SE). Some areas were analyzed to determine chemical composition using wave dispersive spectrometer analysis (WDS).

Petrographic analysis

Petrographic analysis of thin sections of some of the core samples was done according to the specifications of ASTM C856-83 *Standard Practice for Petrographic Examination of Hardened Concrete* (see Appendix A), with the exception of the initial core-sample size (ASTM, 1988). The procedure includes point-counting to identify the amounts of air voids, hardened cement paste, and the aggregate mineralogy.

Chemical staining

The procedure for identifying ASR gel using a set of chemical stains was developed by George Guthrie and William Carey from Los Alamos National Labs, New Mexico (Appendix B); (Guthrie and Carey, 1997). The chemicals stain soluble potassium and calcium ions that are associated with the ASR gel. The chemical test for soluble potassium, a saturated solution of sodium cobaltinitrite ($\text{Na}_3\text{Co}(\text{NO}_2)_6$), was used on the concrete samples. In this test, the solution was applied to a sample previously rinsed with distilled water, and allowed to react for 30 seconds. Then the sample was rinsed with distilled water and allowed to dry.

Aggregate

Geology of sample locations

Aggregate samples were collected from two pits in the Rio Grande Valley, a quarry in Edgewood, a pit in the Silver City area, and a quarry near Grants (Fig. 2, Table 3). Specifically, these are the Placitas, Santa Ana, Edgewood, Mimbres, and Tinaja

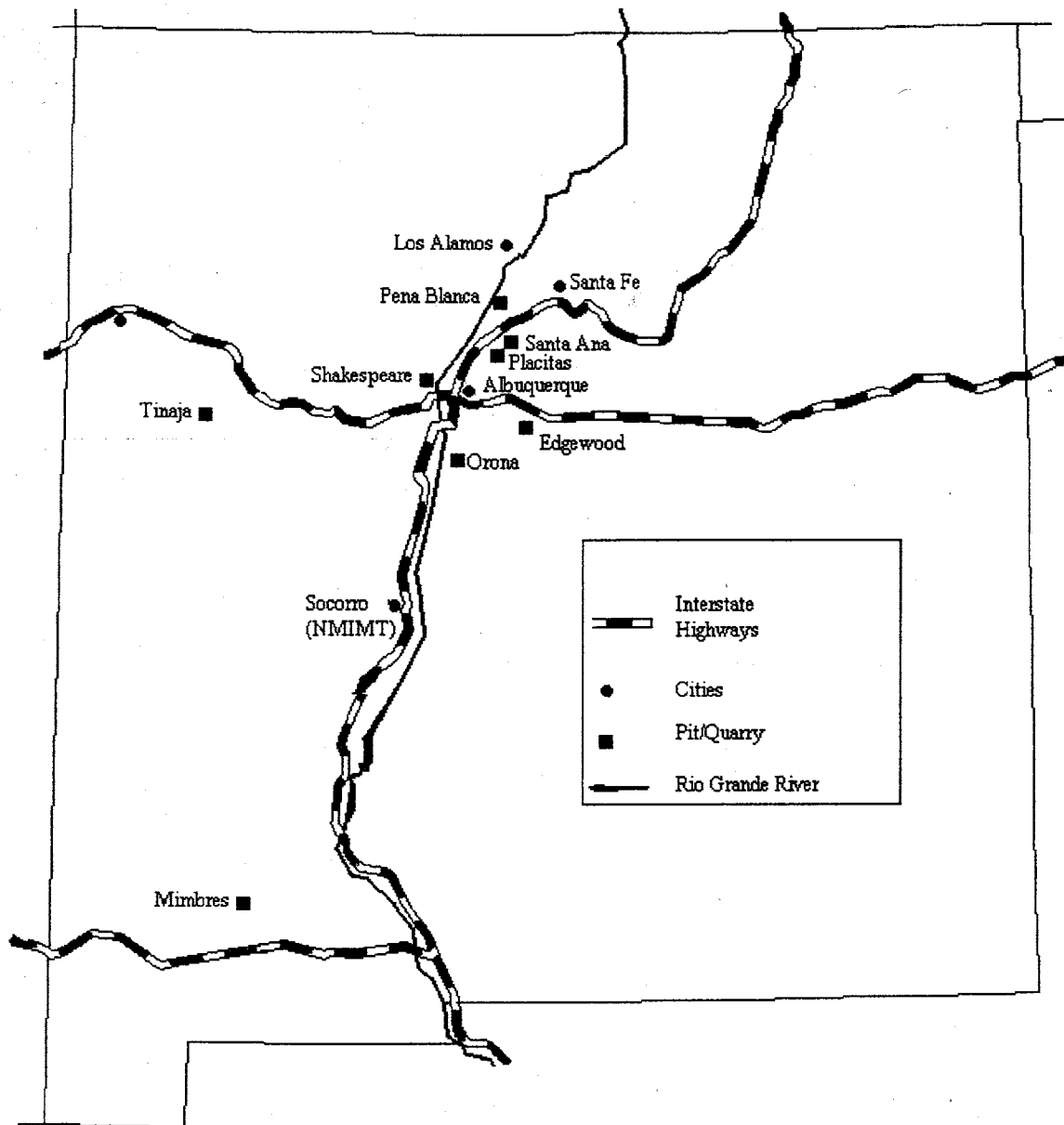


Figure 2. Map of New Mexico showing pits or quarries where either samples were taken or material was inspected.

operations. Several other pits in the Albuquerque area were examined to visually compare their mineralogy, and include Peña Blanca, Shakespeare, and Orona pits. The Placitas, Santa Ana, Edgewood, and Peña Blanca pits are owned by Western Mobile, Shakespeare is owned by Cal-Mat, but is no longer producing aggregate, and the Orona pit is worked by JHT Construction Co, Inc..

Table 3. Location of pits and quarries by Township and Range system.

Pit or quarry	County	Location	Owner
Shakespeare	Bernalillo	Sec. 34, T11N, R3E	Cal-Mat
Tinaja	Cibola	Sec. 4, T9N, R13W	C&E Concrete, Inc.
Mimbres	Luna	Sec. 12, T21S, R11W	SW Concrete & Paving, Inc.
Peña Blanca	Sandoval	Sec. 5, 6, T15N, R6E	Western Mobile NM
Placitas	Sandoval	Sec. 22, 23, 26, 27, 34, 35, T13N, R4E	Western Mobile NM
Santa Ana	Sandoval	Sec. 24, T13N, R4E	Western Mobile NM
Edgewood	Santa Fe	SW4, NW4, Sec. 35, T10N, R7E	Western Mobile NM
Orona	Valencia	W2, Sec. 31, T7N, R3E	JHT Construction Co, Inc.

The material at the Peña Blanca pit is a crushed river rock, which includes volcanics, quartz and quartzites, granites, and metamorphic rocks. Sand lenses with cross bedding are abundant, and the overburden is a silt layer. Several severely weathered or rotten rocks were identified, as were some with black coatings, probably manganese. According to Smith and Kuhle (1998), the area of the Peña Blanca pit includes terrace gravel deposits from the middle and upper Pleistocene. The first layer is up to ~100 ft thick with a lower cobbly gravel with sand lenses, a middle sand and silt with gravel lenses, and an upper gravel. The second layer is a 26-ft-thick gravel with basalt and hawaiite boulders. The breakdown of the terrace gravels given is 30–40% quartzite, 15–20% intermediate volcanics, 5–10% rhyolite, 10–15% granite/gneiss, 5–20% basalt, and

0–10% Bandelier tuff. The area also has alluvium deposited by tributaries to the Rio Grande, which is an Upper Pleistocene to Holocene poorly sorted gravelly sand.

The Shakespeare pit contains quartz, granite, and basalt, with weathered rock and a small amount of black organic coating. Most of the rock is sand-sized. Connell (1997) refers to the pit area as artificial fill and disturbed area. However, by extrapolating to the surrounding formations, the pit probably includes Rio Grande alluvium, specifically the alluvium of Menaul Boulevard from the upper Pleistocene. The material is a poorly consolidated pebble conglomerate and pebbly sand with a thickness of less than 9 ft. Also included in the alluvium of the Rio Grande, is the Edith Boulevard alluvium, which is middle Pleistocene. This is a poorly consolidated conglomerate, sand, and silty clay, which can be up to 40 ft thick. Part of the valley-floor and valley-border alluvium, Upper Holocene to historic, may be included. This is a stream and piedmont alluvium with unconsolidated sand, sandy clay loam, and gravel less than 50 ft thick. Finally, the upper Holocene to historic fan alluvium consists of unconsolidated sand, silt, and pebble to cobble conglomerate.

The Orona pit has a fair amount of chert, both light and dark, with quartz, gneiss, basalt, pegmatite, granite, and greenstone. Little to no coatings were found on the rock, but much sand-sized material and clay masses approximately 1.5 ft in diameter were identified.

Samples were collected from Placitas, Santa Ana, Edgewood, Mimbres, and Tinaja in November 1997, and a second set of samples was collected from Placitas, Santa Ana, Edgewood, and Tinaja in May 1988. In all cases, a representative sample was taken,

usually the same size material that would be used in a concrete (1-in.), from a product pile (Figs. 3 and 4). Each sample was collected and stored in a plastic 5-gal pail. Total sample weight was approximately 50–70 lbs.

At Placitas, the material is a rounded river gravel with numerous sand layers and several concentrated black layers of possible manganese (Figs. 5 and 6). This area has Upper Santa Fe Group, middle Pleistocene age. The material includes chert, quartz, pegmatite, volcanics (including basalts and scoria), with clay and caliche coatings. According to Connell (1998), the Placitas pit is in the Upper Santa Fe Group axial fluvial stratified sand and gravels including quartzite, volcanics, granite, basalt, and chert; the valley floor and valley border alluvium of unconsolidated sand, sandy clay, and gravel; and the piedmont alluvium of moderately consolidated sand and gravel. Samples taken from the pit were collected from the product pile after the material had been washed and screened. The samples consisted of ASTM #57 (approximately 1-in.) size collected both in November and in May, and ASTM C-33 (washed concrete sand) size collected in November.

The Santa Ana pit has reddish sand and silt with larger boulders up to 1.5 ft in diameter. The pit has a caliche layer overlaying much of the sands, and contains more silt layers than the Placitas pit, but is also contained within the upper Miocene to middle (?) Pleistocene Upper Santa Fe Group. Cather and others (1996), describe the pit material as axial river deposits, with ancestral Rio Grande sandstone, conglomerate, and mudstone deposits. The river deposits may include limestone, quartzite, granite, chert, volcanics, gneiss, sandstone, siltstone, schist, phyllite, and pumice. Samples were taken from the



Figure 3. ASTM # 57 ((1-in.) product pile at Edgewood pit. Material is unwashed.



Figure 4. Close up of product pile at Edgewood pit shown in Figure 3. Shovel is approximately 2.5 ft. tall.

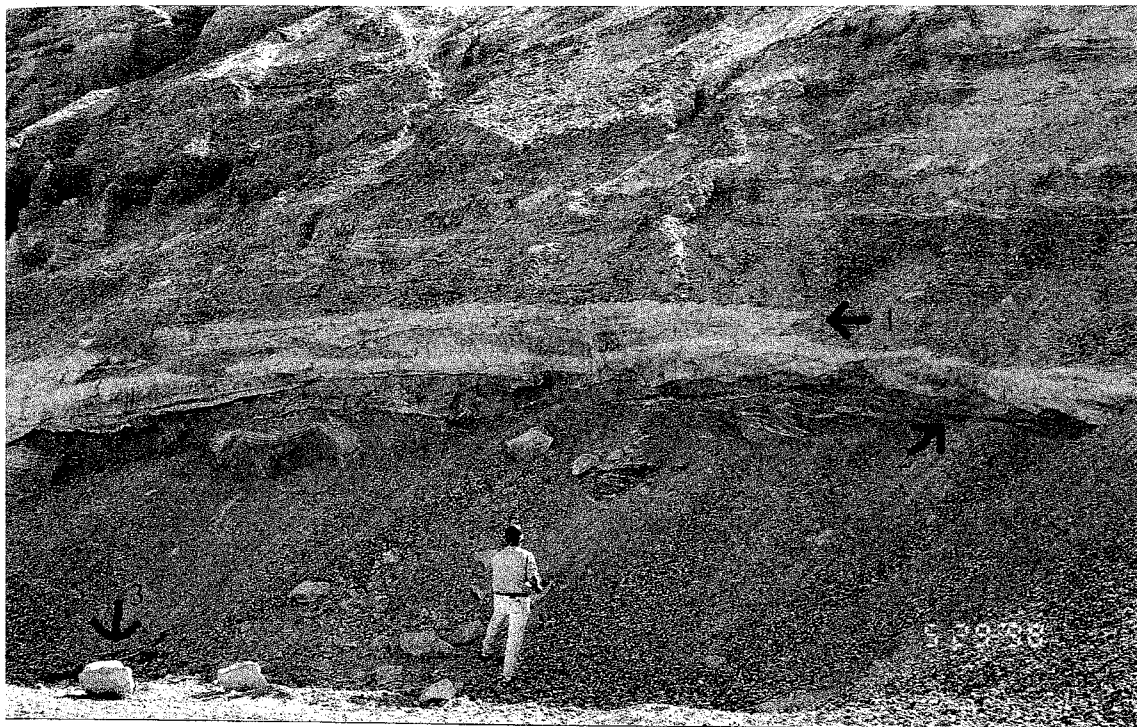


Figure 5. Placitas pit wall. Notice the large sand lens (1), the layers of black material (2), and the clay balls (3).



Figure 6. Close up view of the same pit wall.

product pile of unwashed but screened material. A bucket of ASTM #57 (1-in.) aggregate was taken both in November 1997 and in May 1998.

Samples taken from the Edgewood pit in November and May include a 3/8-in. blend of crushed limestone from the quarry and a sand transported to Edgewood from the Placitas pit. In addition, a crushed limestone sample of ASTM #57 size was taken from the product pile, at both sampling times (Fig. 3 and 4). Both the blended sand and crushed limestone and the large-sized crushed limestone material on the product piles were not washed. The limestone from the pit is part of the Pennsylvanian Madera Formation and is a dark gray crystalline limestone with some clay and reddish-orange iron staining.

The material from Tinaja is a very clean, white to pink, massive limestone, which is from the Upper Permian San Andres Limestone (Fig. 7). The material on the product piles was not washed. Samples were taken of the ASTM #57 sized crushed limestone both in November 1997 and in May 1998.

Sample preparation

Each 50–70 lb sample of aggregate was washed with tap water to remove the fine particles. The material washed from the aggregate was then sieved to separate the sand, coarse silt, and clay-sized material. Fine silt and clay material cannot be separated easily, so the coarse silt-size material was used to represent the silt-size fraction. In the case of the Placitas ASTM C-33 size and the Edgewood 3/8-in. blend, only coarse silt and clay-sized material was collected. The sand includes material that passed the No. 10 (2 mm)



Figure 7. Tinaja pit wall. Notice man on top of highwall for scale.

sieve and was retained on the No. 230 sieve (63 μm), the coarse silt was retained in the No. 400 (38 μm) sieve, and the fine silt and clay passed the No. 400 sieve.

The collected, washed material was prepared for x-ray diffraction (XRD) analysis. The material passing the No. 400 sieve was agitated in solution and allowed to settle for ten minutes. The $<2 \mu\text{m}$ clay-sized material was pipetted from the surface and oriented suspension slides were made. The sand-sized fraction was crushed with a mortar and pestle to pass a No. 100 sieve (150 μm), so bulk XRD analysis could be performed.

The washed aggregate was crushed using a large mill crusher and a smaller pulverizer in the NMBMMR Materials Lab. It was then sieved into five size fractions using industrial screens. The sizes include: passing a No. 4 (4.75 mm) and retained on a No. 8 (2.36 mm), passing a No. 8 and retained on a No. 16 (1.18 mm), passing a No. 16 and retained on a No. 30 (600 μm), passing a No. 30 and retained on a No. 50 (300 μm), and passing a No. 50 and retained on a No. 100 (150 μm) sieve. These aggregate splits were sent to the New Mexico State Highway and Transportation Department (NMSH&TD) Central Materials Laboratory for accelerated mortar bar expansion tests. A split of each size fraction was sent to Spectrum Petrographics to be made into petrographic thin sections.

X-ray diffraction analysis

X-ray diffraction (XRD) data on the material washed from the aggregate was obtained from 2° to $35^\circ 2\theta$ for the air-dried oriented clay samples on a Rigaku diffractometer with a $\text{CuK}\alpha$ tube. The oriented clay slides were then put in an ethylene

glycol chamber overnight and then run from 2° to $15^\circ 2\theta$, heated to 350°F , run from 8.5° to 9.8° and then 2° to $15^\circ 2\theta$. The bulk XRD analysis of sand and silt was run from 2° to $35^\circ 2\theta$ using an unoriented Vaseline mount. This data was then used for qualitative and semi-quantitative clay analysis, and the identification of other minerals present.

Mortar bar expansion tests

To test an aggregate for its reactivity in concrete, it is made into mortar bars that are cured and measured for expansion. The test used by the NMSH&TD Central Materials Laboratory in Santa Fe is the AASHTO (American Association of State Highway and Transportation Officials) Designation TP14, *Standard Test Method for Accelerated Detection of Potentially Deleterious Expansion of Mortar Bars Due to Alkali-Silica Reaction* (see Appendix C) (AASHTO, 1993). In this test, mortar bars (1 x 1 x 11.25 in.) are made in accordance with Section 7: Procedure For Mixing Mortars of AASHTO T162, *Standard Test Method for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency* (see Appendix C), using a washed and crushed aggregate and a low-alkali cement, with no admixtures (AASHTO, 1993). The bars are cured at 100% relative humidity and $21.3\text{--}24.7^\circ\text{C}$ for 26 hours, measured for length, then put into a tap water solution in a 80°C oven for 24 hours. The bars are then transferred to a 1.0 N sodium hydroxide (NaOH) solution maintained at 80°C , where they are measured regularly for 14 days. The mortar bar measurements are done in accordance to Section 6: Procedure of AASHTO M 210, *Apparatus for Use in Measurement of Length Change of Hardened Cement Paste, Mortar, and Concrete* (see Appendix C) (AASHTO, 1993).

An expansion of less than 0.10% is considered innocuous, and greater than 0.10% is a potentially deleterious expansion (Barela, 1997). The aggregate samples were sent to the NMSH&TD Central Materials Lab for this test to compare the reactivity and expansion of the different pits.

Petrographic examination

The thin sections of the crushed and sieved aggregate were examined using a petrographic microscope in accordance with ASTM C295-90, *Standard Guide for Petrographic Examination of Aggregates for Concrete* (see Appendix D) (ASTM, 1995). Each size fraction slide was point-counted to identify mineralogy, alteration, and weathering of aggregate particles.

RESULTS

Concrete

Soil pH

The pH of soil samples ranged from 7.53 to 8.12 (Table. 2). No observed correlation exists between the alkalinity of the surrounding soil and the condition of the concrete. While the worst concrete visually had nearby soils with a lower pH (sample 4 at 7.77), so did concretes that showed no distress (sample 8 at 7.96 and 9 at 7.76). One of the concretes that had severe popouts had a nearby soil with a higher alkaline pH (samples 5-7 at 8.12), but the other samples with a higher alkaline pH (samples 1-2 at 8.03) had no visual signs of distress.

Scanning electron microprobe

Using the SEM, many occurrences of ettringite were found, mostly growing in open air voids in the concrete. In one occurrence, a crack was lined with ettringite crystals (Fig. 8). The ettringite was confirmed chemically by the element mapping of Si, S, and Al on the suspected area. The mapping showed the crack area to be high in sulfur and aluminum and void of silica, which correlates with the chemical composition of ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3\text{OH}_{12}\cdot 26\text{H}_2\text{O}$). Most of the ettringite was in the form of elongated crystals, approximately 5–10 μm long (Fig. 9). However, in sample 12, from the Gallup train trestle support (poured in 1923), the ettringite was in the form of a fibrous mass which filled the air void (Fig. 10).

Gel in concretes has been identified by others using the SEM, and appears as a cracked area, looking very similar to mud cracking in clays (Stark and others, 1993; Marusin, 1997). Several occurrences of a gel-like mass were identified in the concrete samples using the SEM. An example of the gel can be seen in Figure 11 (same as Fig. 8), in the upper right hand corner of the BSE image. The gel area contained substantial calcium and silica, but no significant amount of potassium, which one would expect to find in an ASR gel. Therefore, this gel (and others) has been identified as a C-S-H gel. Other researchers have suggested that ASR gel sometimes forms in association with ettringite crystals. In most cases when ettringite was found, an element map of the area was made. In no case was there any appreciable amount of potassium or sodium found, which would be expected if ASR was present.

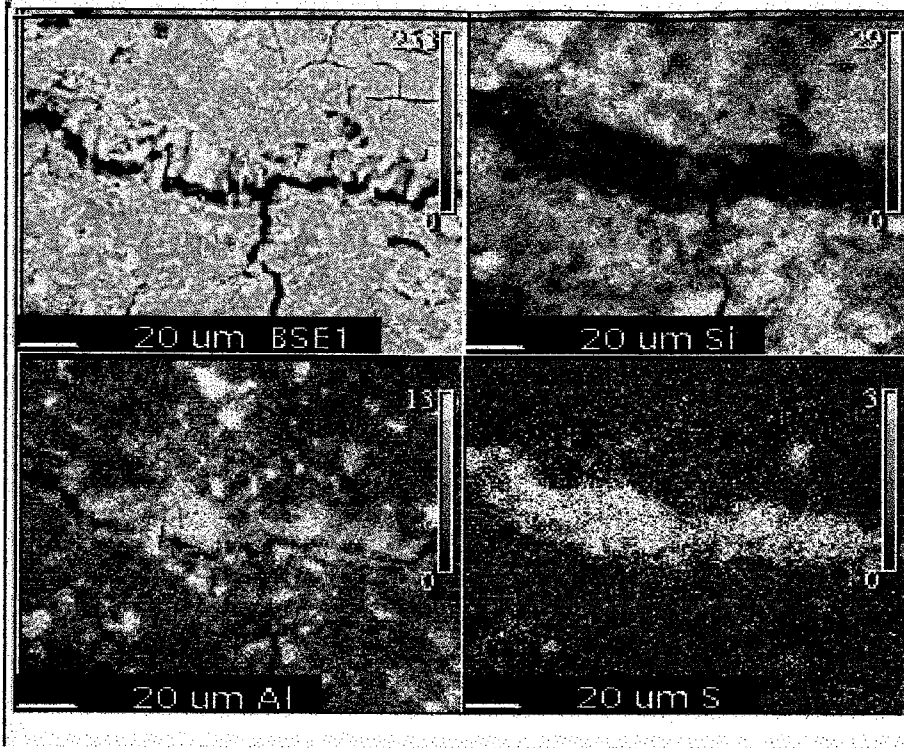


Figure 8. Ettringite lining a crack in sample 4. BSE image, and element maps of silica, aluminum, and sulfur included. Lighter areas in the element maps indicate more counts of the element are present.

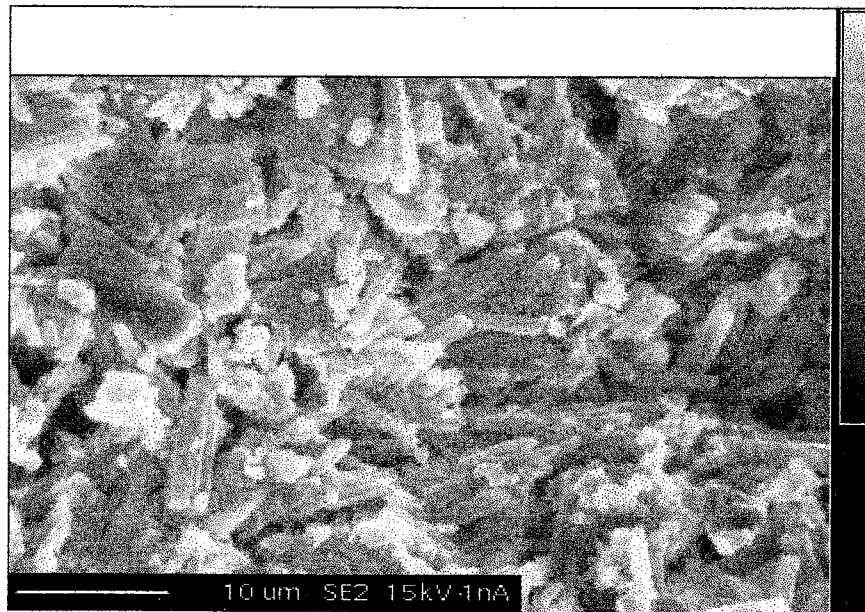


Figure 9. Secondary electron image of elongated ettringite crystals in air void (sample 4).

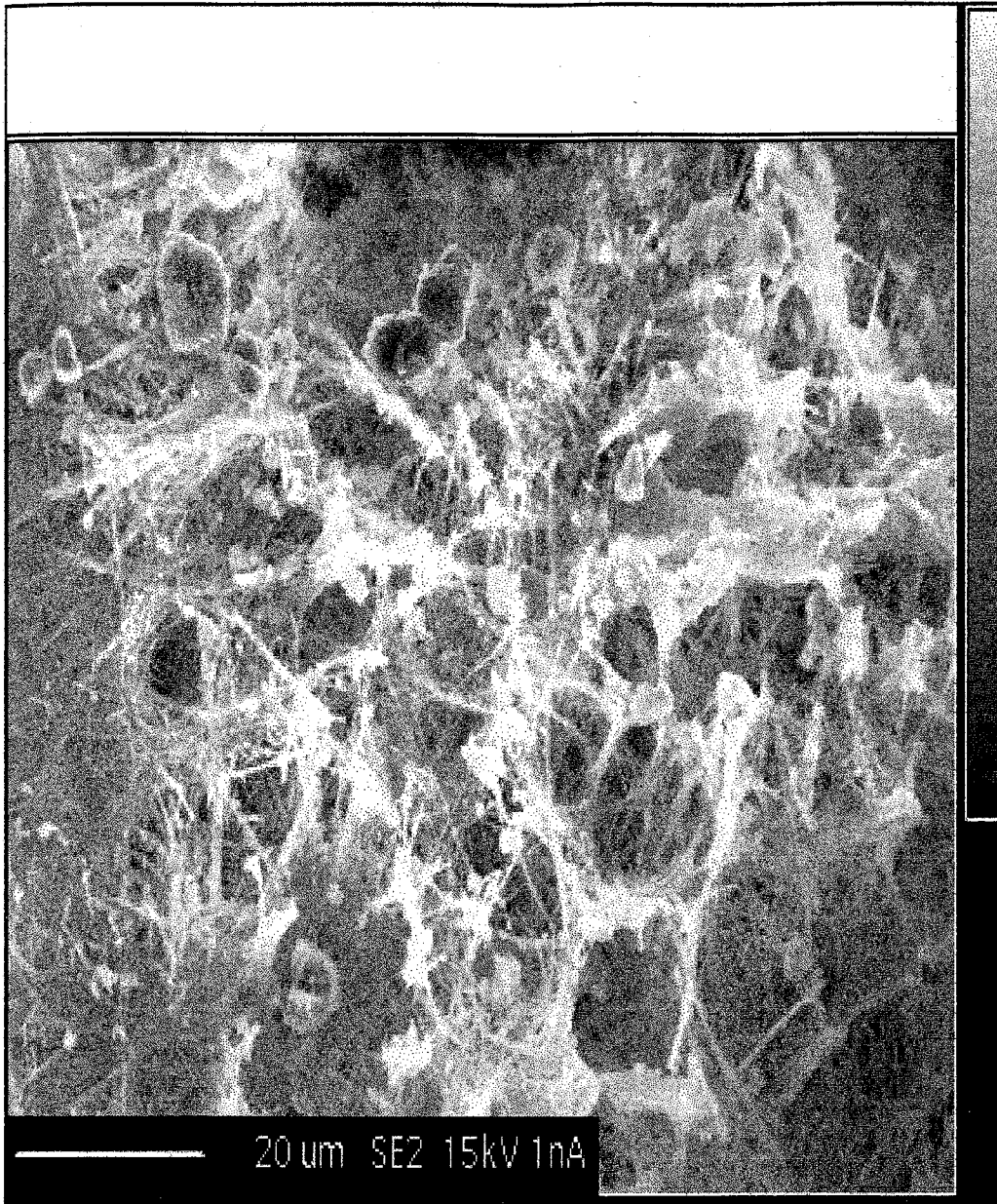


Figure 10. SEM image of fibrous ettringite in air void in 1923 sample (12).

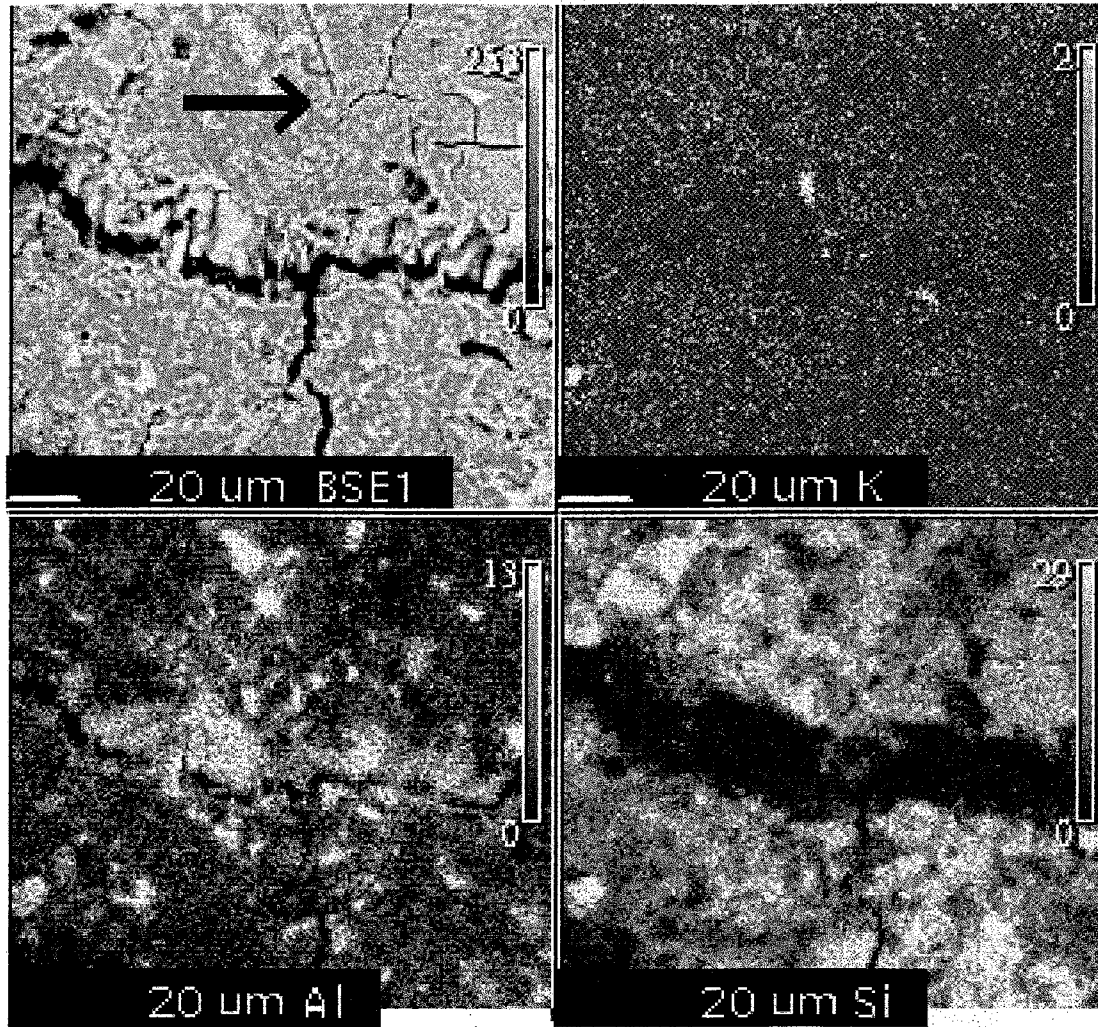


Figure 11. Gel can be seen in the upper right corner (arrow) in the image of sample 4. Element maps of potassium, aluminum, and silica are included with the BSE image. Lighter areas in the element maps indicate more counts of the element are present.

Petrographic analysis

Petrographic analysis is the standard test of the industry, and when performed to specifications, should give a representative analysis of the concrete characteristics.

However, because in the current study, the core size of the samples examined is smaller than the specifications, and only one thin section from each was examined, the results are only qualitative.

Many of the concretes contain large amounts of potentially reactive aggregate (Table 4). Sample 3, when adding the rhyolite, andesite, chalcedony/chert, and quartz, had over 75% potentially reactive aggregate. In comparison, the sample with the least amount of reactive aggregate (15) contained less than 10% reactive particles. Of the five samples with exceptionally large amounts of reactive particles, (rhyolite + andesite + chert/chalcedony + quartz) only two of them actually showed visual distress in the form of map cracking (sample 10, Fig. 12), or popouts (sample 11, Fig. 13) on the concrete surface. Although popouts generally do not indicate structural stress, they can be a sign of abundant chert particles.

Figure 14 is an example of a large piece of potentially reactive chalcedony found in sample 10. Several samples contained a very large amount of hardened cement paste, which is generally supposed to be a small portion (10–15%) of the concrete. This large number is most likely not representative of the whole sample due to the small core size and from looking at only one section per core.

Some of the samples displayed a color change in the cement paste. Many of the air voids and cracks were surrounded by a light tan paste rather than the dark, almost black

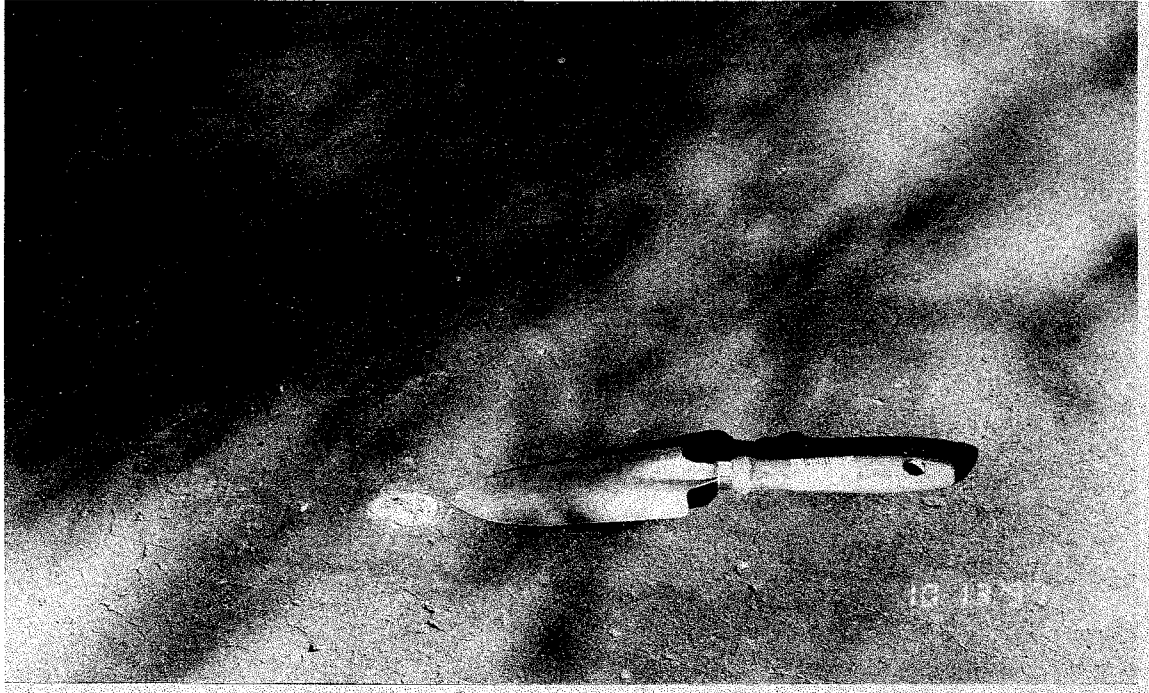


Figure 12. Map cracking in sidewalk, sample 10. The trowel is for scale, and is pointing to the core location.



Figure 13. Popouts in sample area 11. Trowel is pointing to the core location.

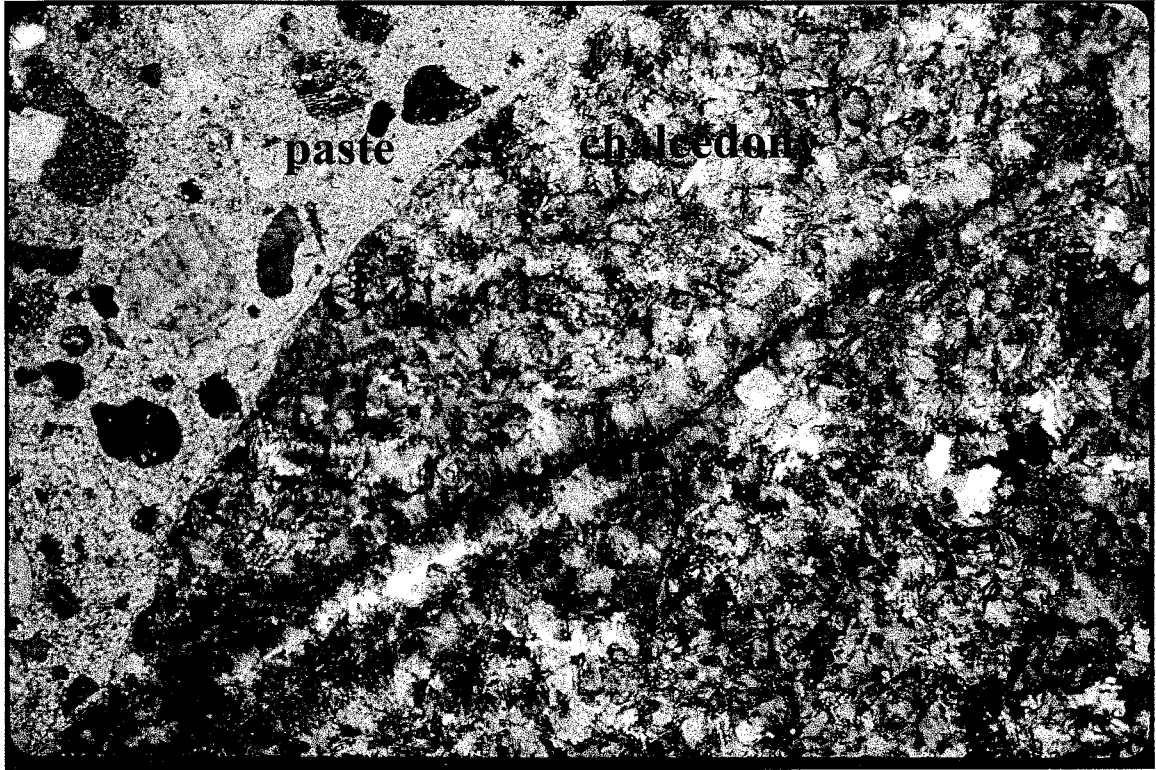


Figure 14. Large piece of chalcedony aggregate in concrete sample 10. Width of frame approximately 5 mm.



Figure 15. Map cracking on sidewalk where core sample 4 was taken. Core was taken near the yellow field notebook.

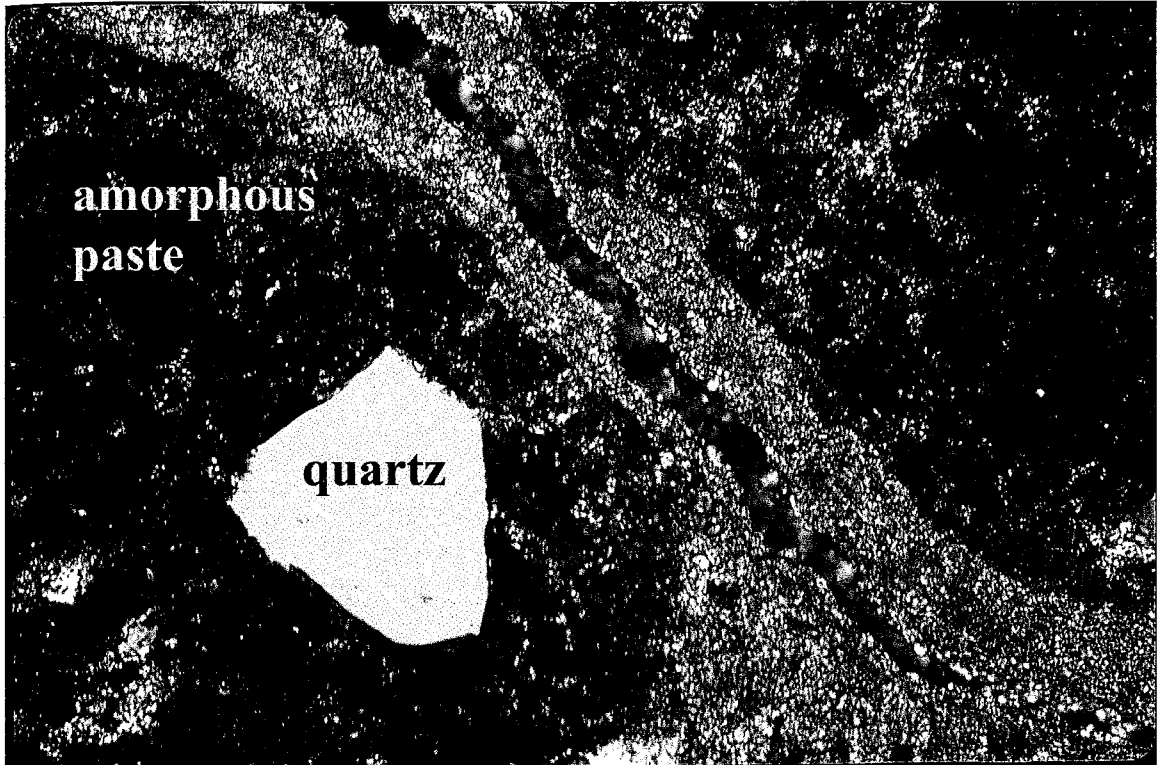


Figure 16. Crack in hardened cement paste lined by tan mineral in sample 4. Width of frame approximately 2–3 mm.

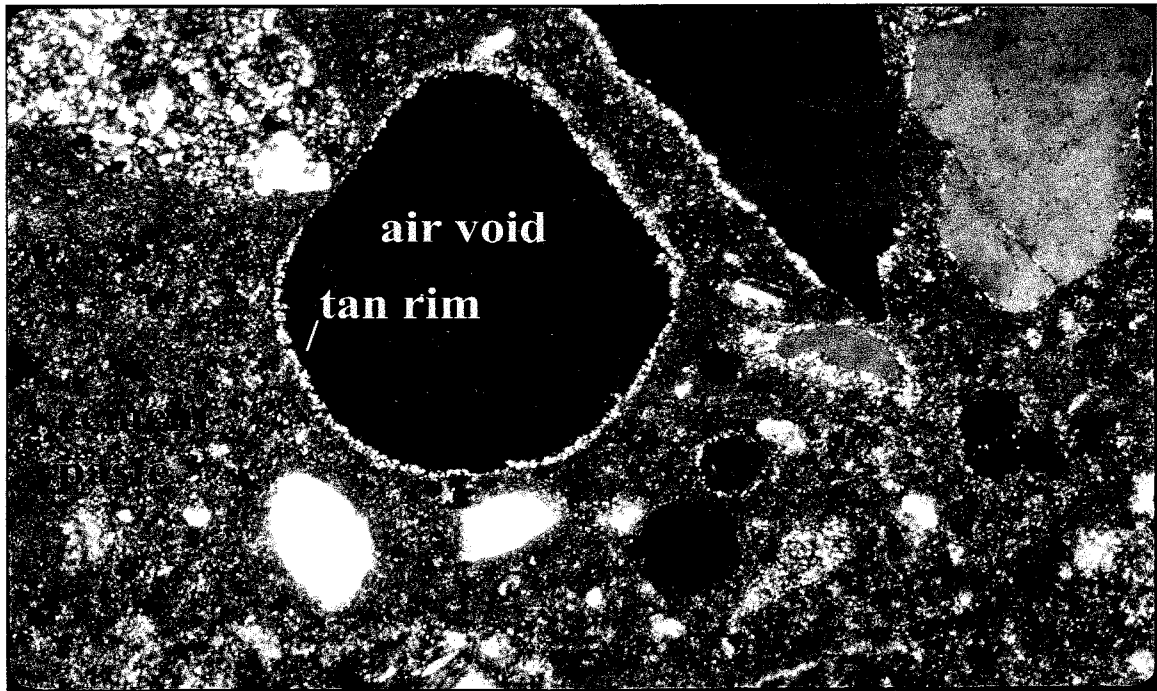


Figure 17. Air void with tan colored rim in concrete sample 11. Width of frame approximately 2–3 mm.

paste (Figs. 16 and 17). This tan phase is believed to be ettringite or perhaps a calcium hydroxide instead of the black, amorphous C-S-H gel. It is not possible to distinguish the ASR gel from the C-S-H gel in this analysis.

Table 4. Petrography results (ASTM C856-83) of concrete core samples, in percent.

Sample	1	3	4	5	6	8	9	10	11	14	15	16
Rhyolite and trachyte	1.8	66.6	0.0	6.9	5.4	7.4	41.6	7.6	7.7	1.1	1.1	1.3
Andesite	0.0	0.0	tr	0.0	0.0	0.0	0.0	5.0	15.6	0.0	0.0	tr
Dacite and basalt	42.9	tr	tr	tr	tr	0.0	2.0	5.0	1.3	1.4	tr	4.6
Chert and chalcedony	0.0	0.0	tr	tr	8.5	6.0	4.2	31.8	16.8	2.0	tr	4.1
Quartz	21.0	9.1	22.6	26.7	34.6	54.0	19.0	9.2	14.2	14.1	8.0	12.5
Feldspar	5.5	4.2	11.9	7.5	8.1	4.1	6.1	7.3	7.6	11.0	5.0	7.9
Ferro-magnesian	tr	1.9	9.6	1.3	1.7	2.1	tr	tr	tr	1.7	tr	0.6
Opaque (Fe-rich)	1.1	tr	tr	2.1	1.1	1.1	2.8	tr	1.3	0.0	tr	0.0
Limestone	tr	tr	3.2	23.4	2.6	tr	tr	1.1	1.3	2.8	53.8	27.0
Voids	tr	2.8	tr	1.8	2.0	5.0	1.2	3.0	1.9	5.9	3.8	9.0
Cement paste	25.3	14.2	50.9	29.8	35.1	19.6	21.3	29.4	31.6	59.9	25.6	32.4
*Sum	97.6	98.8	98.2	99.5	99.1	99.3	98.2	99.4	99.3	99.9	97.3	99.4
Reactive particles	22.8	75.7	22.6	33.6	48.5	67.4	64.8	53.6	54.3	17.2	9.1	17.9
Percent reactive particles	23.4	76.6	23.0	33.8	48.9	67.9	66.0	53.9	54.7	17.2	9.4	18.0

*Note: tr = trace, less than 1%, reactive particles = rhyolite + andesite + chert/chalcedony + quartz.

Chemical staining

The chemical test for soluble K^+ ions was used to try to identify the presence of ASR gel in the concrete samples. The sidewalk near the NMT library (near sample 4) was tested using sodium cobaltinitrite, the chemical that stains soluble potassium yellow. A 0.125-in. yellow halo was observed surrounding a crack <0.125-in. wide. Core sample 4 also tested positive for ASR. The results of the core chemical tests are given in Table 5.

The staining test results are interpreted on a relative scale. If the stain coloring was present, it was considered a positive result. This was then broken down into high, medium, or low, depending on the amount/brightness of the stain and where the stain

occurred. Several of the cores had a positive result. Five samples (2, 4, 20, 23, and 24) received a high positive rating. In these samples, the yellow stain showed up brightly in a cavity where an aggregate particle was, or as a halo surrounding an aggregate particle. Three samples received a medium positive rating (3, 10, and 25). The stain in these samples showed up as a halo on an aggregate particle or in a cavity, but not as brightly as in the high positive samples. The five samples that received a low positive rating (7, 8, 9, 15, and 21) had only slight staining as a halo or on the aggregate particles themselves. Negative ratings were given to 5 samples, (11, 13, 17, 19, and 26) in which there may have been a very slight stain on the surface of an aggregate particle, or no stain at all.

Table 5. Results of chemical staining test for soluble potassium, given in order of increasing age. NA = no data available.

Sample	Results	Comments	Age	Distress	Additives	% Reactive
13	neg.	slight stain on surface of granitic aggregate	1923	none	NA	NA
3	med pos.	halo on rhyolite/granitic aggregate; in cavity	1936	none	NA	76.6
10	med pos.	stain halo around aggregate; stains altered feldspar	1939	cracking	NA	53.9
2	hi pos.	stain on and surrounding rhyolite and quartz	1960	none	NA	NA
26	neg.	very slight stain on quartz aggregate	1963	cracking	NA	NA
17	neg.	slight yellow aggregate	1965	none	NA	NA
15	low pos.	slight stain around aggregates and altered feldspar	1977	none	NA	9.4
19	neg.	slight stain on quartz aggregate	1979	none	flyash	NA
20	hi pos.	stain in cavity and on quartz aggregate	1979	none	flyash	NA
23	hi pos.	stain on surface of aggregate and in cavity	1979	none	flyash	NA
21	low pos.	slight halo around quartz aggregate	1979	none	flyash	NA
24	hi pos.	stain in cavity	1979	none	flyash	NA
25	med pos.	stain in cavity	1979	none	flyash	NA
11	neg.	only stains altered feldspar in aggregate	1988	popouts	NA	54.7
7	low pos.	stain coats and forms halo on quartz or chert	1988	popouts	NA	NA
4	hi pos.	bright yellow in cavity, and halo on andesite	1988	cracking	NA	23.0
8	low pos.	stain halo on chert aggregate	1993	none	NA	67.9
9	low pos.	slight yellow stain in paste	1997	none	NA	66.0

Not all samples with large amounts of reactive material had a positive result (sample 11 at around 55% reactive particles was negative, and samples 8 and 9 at around 68 and 66% reactive particles were low positive). Not all samples that showed outward

distress had a positive result (sample 26 with map cracking, and 11 with popouts were negative). Not all old concretes had negative results (samples 3 from 1936 and 10 from 1939 were medium positive, and 2 from 1960 was high positive). Not all the samples from 1979 that were made with flyash (all are from a section in Gibson Boulevard) have consistent results. They vary from negative to high positive.

Aggregate

X-ray diffraction analysis

The XRD analysis of sediments washed from the aggregate collected in November shows that several of the samples contained large amounts of smectite, or illite/smectite mixed layer expandable clays (Table 6, Fig. 18). The Mimbres pit has the highest amount of smectite at 4 parts out of 10, and the highest amount of illite/smectite mixed-layered clays at 6 parts out of 10. Varied amounts of quartz, feldspars, calcite, and dolomite were present in all three size fractions—sand, silt and clay. The samples collected in May contained far less amounts of smectite and only one with 1/10 illite/smectite mixed-layer clay minerals (Edgewood 3/8-in.) (Table 7, Fig. 19). The non-clay minerals in the clay, silt and sand-sized fractions remained the same (calcite, feldspars, dolomite, and quartz) from November to May. None of the pits had duplicate results of clay mineralogy. All pits/quarries show much variability in the clay mineral analysis. Even the two different size fractions from Placitas (1-in. and concrete sand) showed variability during the same sampling time in November.

Table 6. Results of the x-ray diffraction of material washed from aggregate samples taken in November 1997. Note: Clay XRD results in parts out of ten, but may not add to ten due to rounding.

Clay mineralogy 11/97

PIT	Illite	Kaolinite	Smectite	I/S Mix	Other minerals (in decreasing abundance)
Ed 3/8-in.	4	4	2	0	albite, calcite Ed = Edgewood
Ed 1-in.	4	2	0	4	calcite, quartz PI = Placitas
PI C-33	2	3	2	3	calcite, quartz Tn = Tinaja
PI 1-in.	2	5	1	2	calcite, feldspar Mb = Mimbres
Tn 1-in.	4	5	0	2	calcite, dolomite SA = Santa Ana
Mb 1-in.	0	0	4	6	microcline
SA 1-in.	3	4	2	2	calcite, quartz

Silt mineralogy (in decreasing abundance)

PIT	Mineralogy
Ed 3/8-in.	quartz, albite, calcite
Ed 1-in.	calcite, quartz, feldspar
PI C-33	quartz, albite
PI 1-in.	quartz, albite, calcite
Tn 1-in.	calcite, feldspar, dolomite
Mb 1-in.	quartz, albite, plagioclase, dolomite
SA 1-in.	quartz, calcite, albite

Sand mineralogy (decreasing)

PIT	Mineralogy
Ed 3/8-in.	NA
Ed 1-in.	calcite, feldspar, quartz
PI C-33	NA
PI 1-in.	albite, quartz
Tn 1-in.	calcite, feldspar, dolomite
Mb 1-in.	quartz, albite, plagioclase
SA 1-in.	quartz, calcite, albite

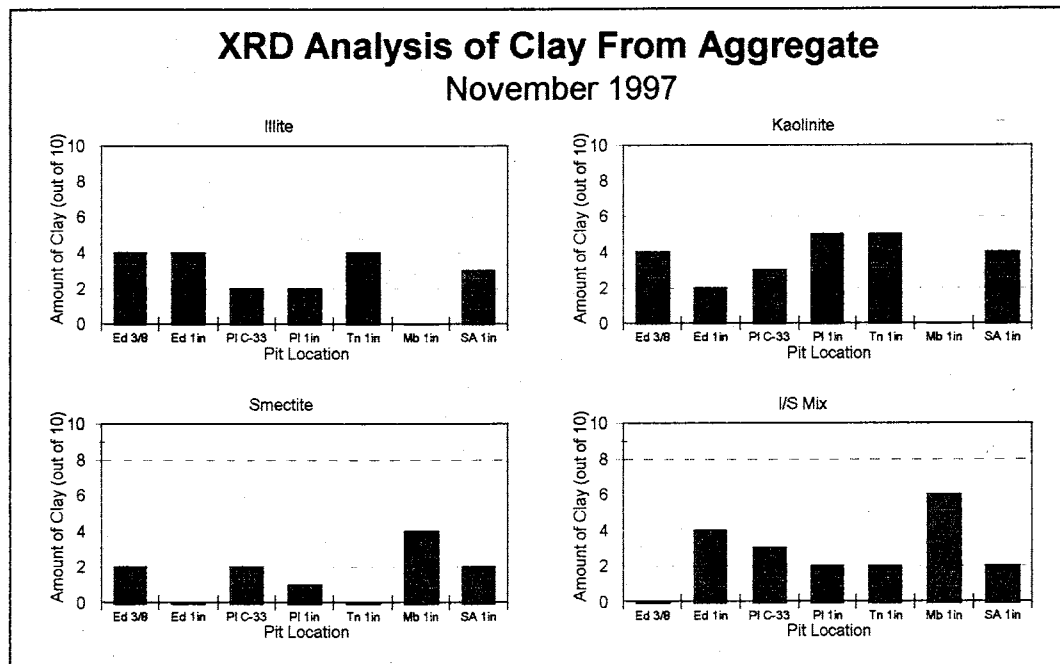


Figure 18. Results of XRD analysis of clay-size material washed from aggregate collected in November 1997.

Table 7. Results of the x-ray diffraction of material washed from aggregate samples taken in May 1998. Note: Clay XRD results in parts out of ten, but may not total 10 due to rounding, dol. = dolomite, NA = no data available.

Clay mineralogy 5/98

PIT	Illite	Kaolinite	Smectite	I/S Mix	Other minerals (in decreasing abundance)
Ed 3/8-in.	3	4	2	1	feldspar, calcite
Ed 1-in.	10	0	0	0	calcite, quartz
Pl 1-in.	4	4	2	0	quartz, feldspar, calcite
Tn 1-in.	2	9	0	0	calcite, dolomite, quartz
SA 1-in.	0	10	0	0	calcite, quartz

Ed = Edgewood
 Pl = Placitas
 Tn = Tinaja
 SA = Santa Ana

Silt mineralogy (in decreasing abundance)

PIT	Silt mineralogy (in decreasing abundance)
Ed 3/8-in.	quartz, feldspar, calcite
Ed 1-in.	calcite, quartz, feldspar
Pl 1-in.	quartz, feldspar, calcite
Tn 1-in.	calcite, quartz, dolomite, feldspar
SA 1-in.	quartz, calcite, feldspar

Sand mineralogy (decreasing)

PIT	Sand mineralogy (decreasing)
Ed 3/8-in.	NA
Ed 1-in.	calcite, quartz, feldspar,
Pl 1-in.	quartz, feldspar
Tn 1-in.	calcite, dolomite, quartz, feldspar
SA 1in	quartz, calcite, feldspar

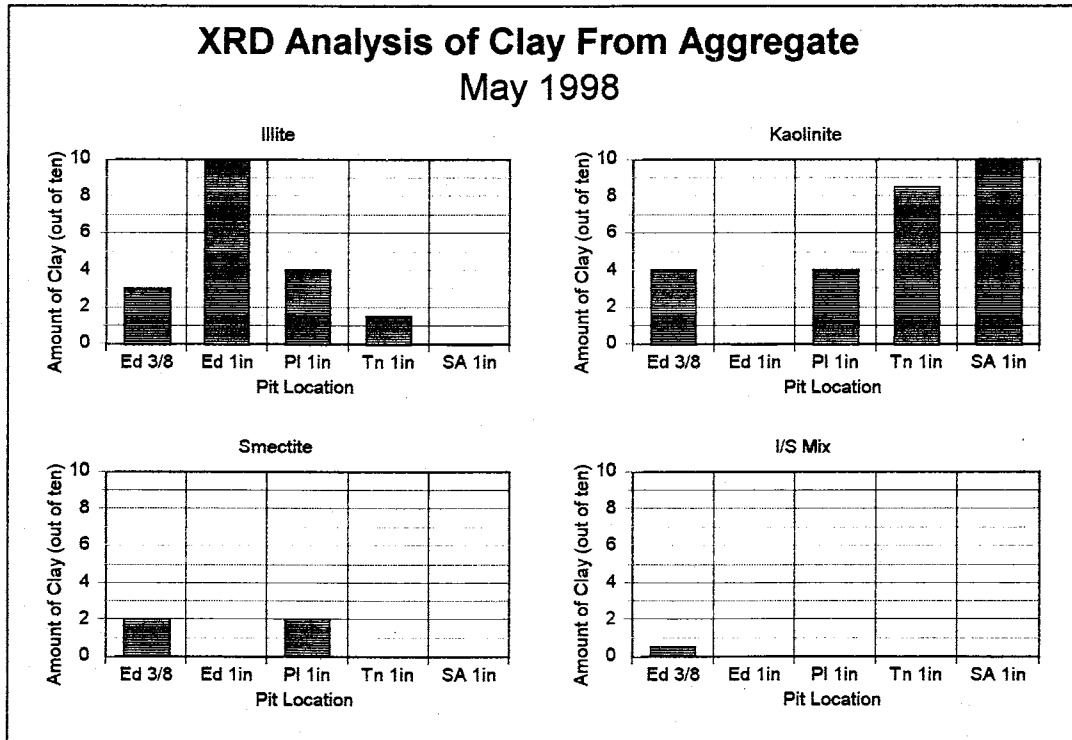


Figure 19. Results of XRD analysis of clay-size material washed from aggregate collected in May 1998.

Accelerated mortar bar expansion testing

In the November testing, the Tinaja aggregate had innocuous expansion of 0.01% (Table 8). All other pits/quarries were well above the limit of 0.1% and in the deleterious range. The results from the mortar bar testing of the material collected in May is similar. The Edgewood 3/8-in. and Santa Ana expansion was lower than in November, and the Edgewood 1-in. increased, but all still remain within the same categories.

Table 8. Results of accelerated mortar bar expansion tests. Shows who collected material, when, and what size was collected. Ed = Edgewood, Pl = Placitas, Tn = Tinaja, Mb = Mimbres, SA = Santa Ana. NA = no data available. <0.1 is innocuous, >0.1 is potentially reactive.

Pit or Quarry	NMSH&TD	Coose	Coose	Lenke	Lenke
	<Nov. 1997 Expansion	Nov. 1997 Expansion	May 1998 Expansion	Fall 1997 Expansion	Fall 1997 Expansion
Ed 3/8-in.	NA	0.973	0.882	NA	NA
Ed 1-in.	NA	0.373	0.407	NA	NA
Pl C-33	NA	0.880	NA	0.806	0.785
Pl 1-in.	1.340	0.998	0.986	NA	NA
Tn 1-in.	0.000	0.014	0.009	0.003	0.022
Mb 1-in.	0.520	0.567	NA	0.492	0.495
SA 1-in.	0.240	0.848	0.792	0.534	0.417

The test results from November and May were compared to a set of expansion tests that were completed before November 1997, by Joe Barela at the NMSH&TD Central Materials Laboratory. These expansion tests correlate to only some of the areas where this study's samples were collected, but are useful for comparison. They include the Placitas, Mimbres, Santa Ana, and Tinaja material (Table 8). The Tinaja material is completely innocuous at 0.00% expansion, and the Placitas is potentially deleterious at 1.34% expansion. While these results differ from the results from the samples run in November and May, they remain in the same categories.

Expansion tests were also completed on material collected by Lary Lenke of the Alliance for Transportation Research, a group affiliated with the University of New Mexico in Albuquerque. These aggregates were collected from November to December of 1997 and include the Placitas, Tinaja, Mimbres, and Santa Ana pits. These expansion test results are included in Table 8 for comparison.

Petrographic analysis

The petrographic analysis of the size fractions of each of the aggregate samples was used to determine the mineralogy of the particles as well as the percentages of weathered material (Table 9, Figs. 20 and 21). In November, the Tinaja 1-in. material was 99.7% limestone with only 1.5% weathered material, but other samples contained much more potentially reactive material. The Edgewood 1-in. material is a limestone (99.1%) but contains 7% weathered and/or iron stained material that may be reactive in ASR (Fig. 22). The other pits contain various amounts of chert/chalcedony as well as rhyolite, andesite, and weathered particles, all of which are potentially reactive (Figs. 23 and 24). The Mimbres 1-in., Placitas 1-in., Placitas C-33, and Edgewood 3/8-in. aggregate all consist of high amounts of chert/chalcedony, 37.5%, 27.8%, 20.9%, and 13.9%, respectively. Placitas 1-in. and Edgewood 3/8-in. in addition have large amounts of rhyolite, 10.9%, and 9.1%. The Edgewood 3/8-in. aggregate is 17.5% weathered particles, which is also a cause of reactivity.

The petrographic results from the materials sampled in May are similar, but are variable. The Santa Ana pit material is most variable with almost 15% less limestone and

more quartz and feldspar. The amounts of weathered materials as well as the other reactive particles differs in each analysis.

Table 9. Petrographic results of aggregate samples, in percent.

November 1997							
Percents	Mb 1"	Tn 1"	SA 1"	PI 1"	PI C-33	Ed 1"	Ed 3/8"
Rhyolite/Trachyte	3.4	0.0	6.6	10.9	8.8	0.0	9.1
Andesite	7.4	0.0	0.1	1.9	3.0	0.0	0.1
Dacite/Basalt	41.7	0.0	8.1	23.9	16.7	0.0	15.7
Chert/Chalcedony	37.5	0.1	8.8	27.8	20.9	0.4	13.9
Quartz	2.8	0.1	11.9	23.8	19.6	0.4	12.6
Feldspar	5.7	0.0	9.6	10.9	28.5	0.0	21.8
Ferromagnesian	0.3	0.0	0.0	0.4	0.6	0.0	0.4
Opaque	0.3	0.0	0.0	0.4	1.0	0.0	0.3
Limestone/Dolomite	0.9	99.7	55.0	0.1	0.9	99.1	26.1
Total	100	100	100	100	100	100	100
Weathered	8.2	1.5	12.7	12.1	9.1	7.0	17.5

May 1998							
Percents	Mb 1"	Tn 1"	SA 1"	PI 1"	PI C-33	Ed 1"	Ed 3/8"
Rhyolite/Trachyte	NA	0.0	4.7	6.3	NA	0.0	2.6
Andesite	NA	0.0	1.5	6.1	NA	0.0	3.7
Dacite/Basalt	NA	0.0	11.2	28.0	NA	0.0	18.5
Chert/Chalcedony	NA	0.1	7.0	19.3	NA	0.6	13.6
Quartz	NA	0.1	20.9	18.3	NA	0.0	13.5
Feldspar	NA	0.3	14.8	20.6	NA	0.0	27.6
Ferromagnesian	NA	0.0	0.5	0.9	NA	0.0	1.2
Opaque	NA	0.0	0.3	0.4	NA	0.0	0.9
Limestone/Dolomite	NA	99.5	39.1	0.1	NA	99.4	18.4
Total	NA	100	100	100	NA	100	100
Weathered	NA	1.9	12.5	9.2	NA	7.9	11.2

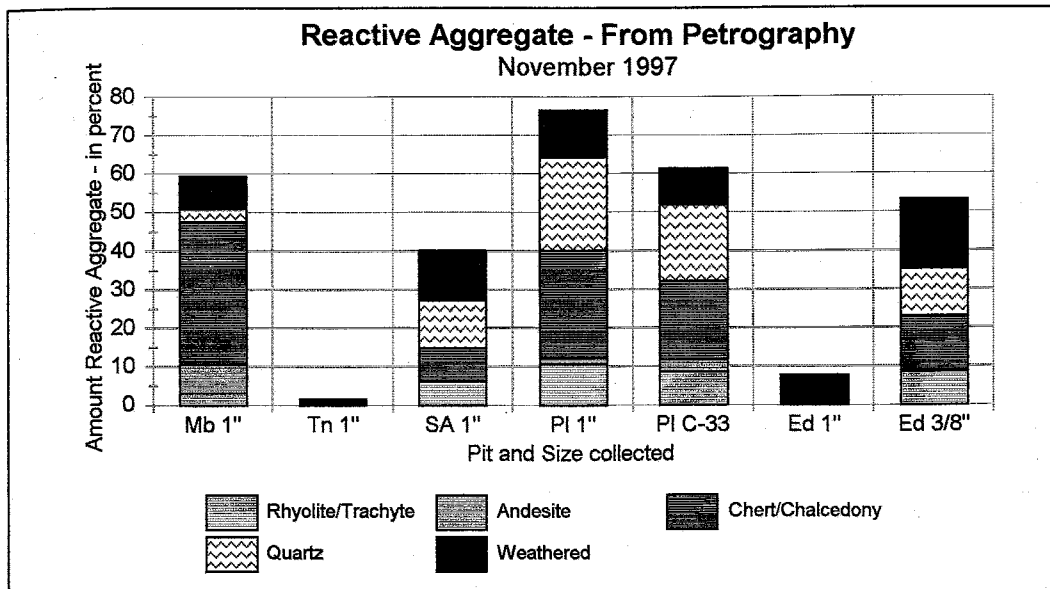


Figure 20. Results of petrographic analysis showing reactive particles of aggregate collected from pits and quarries in November 1997. Mb = Mimbres, Tn = Tinaja, SA = Santa Ana, Pl = Placitas, Ed = Edgewood.

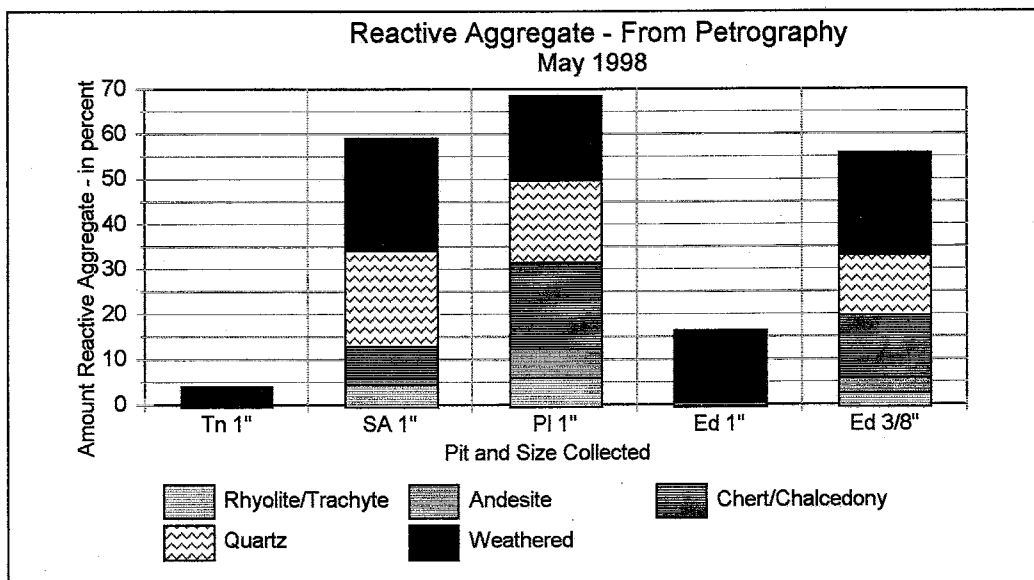


Figure 21. Results of petrographic analysis showing reactive particles of aggregate collected in May 1998. Tn = Tinaja, SA = Santa Ana, Pl = Placitas, Ed = Edgewood.

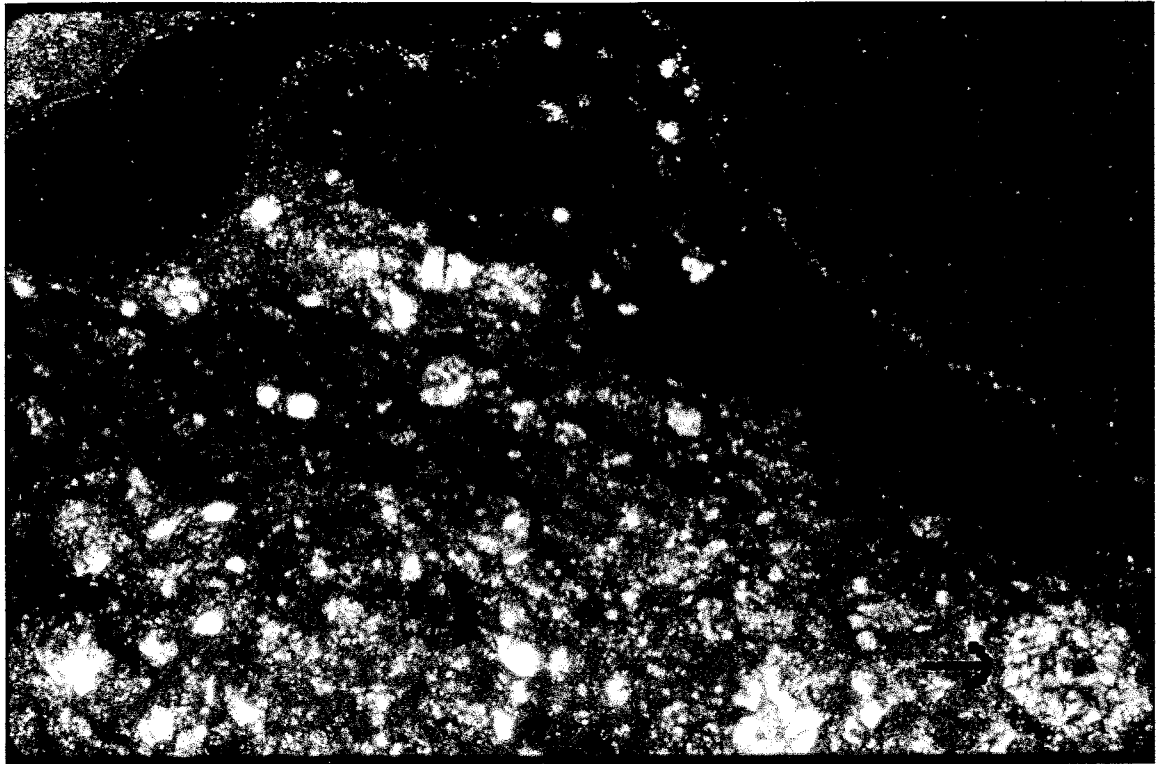


Figure 22. Limestone with iron staining in Edgewood sieve size -16+30 collected in November 1997. Notice small piece of crystalline quartz in bottom right corner (arrow). Width of frame approximately 2–3 mm.

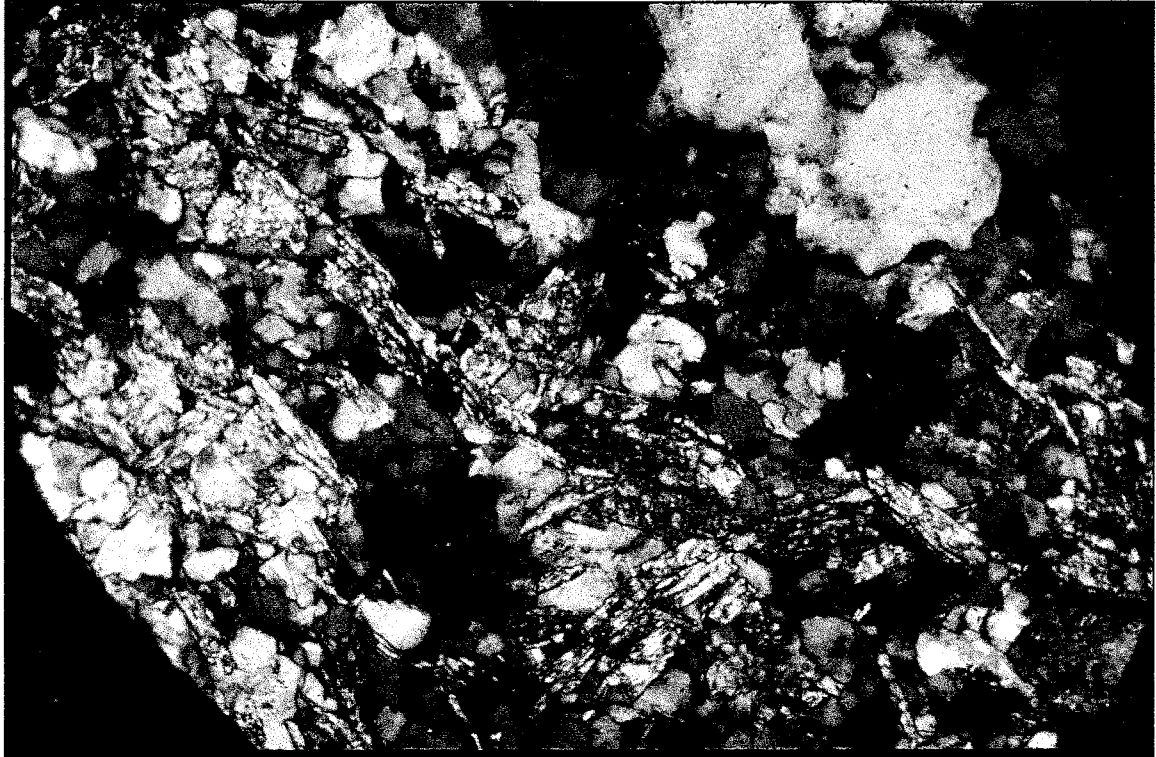


Figure 23. Altered granitic piece of aggregate in Edgewood sieve size -8+16. Width of frame approximately 2–3 mm.

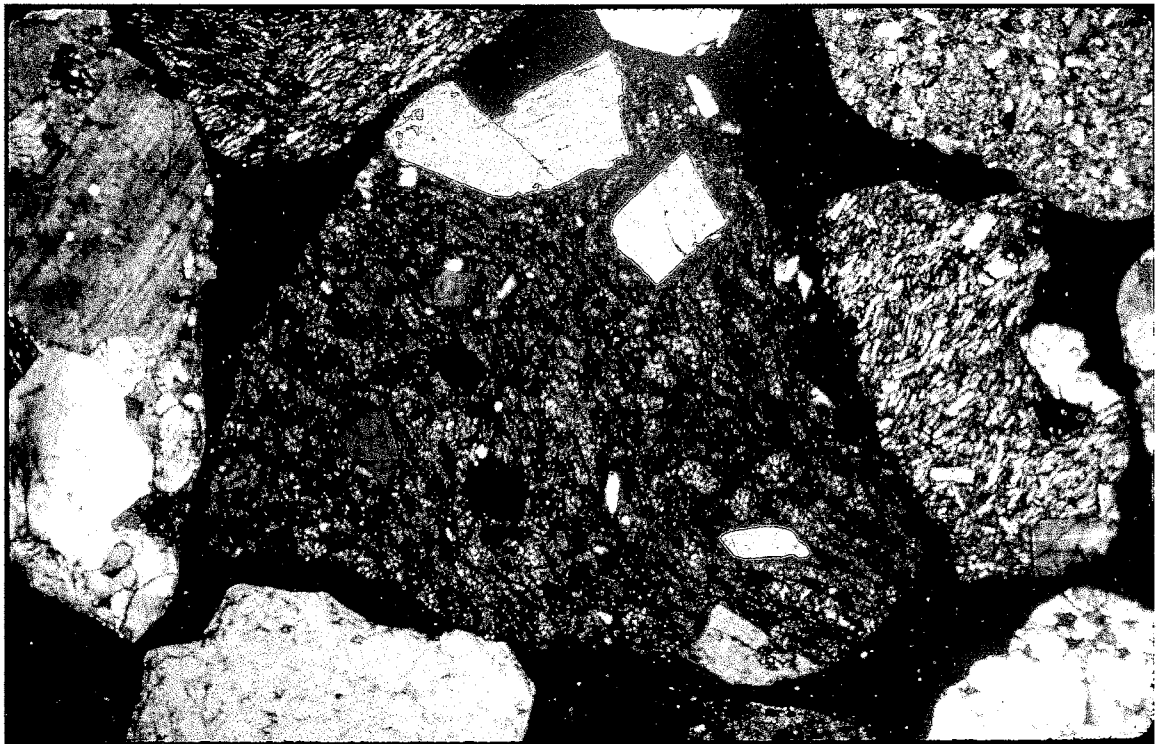


Figure 24. Potentially reactive aggregate including rhyolite, andesite, and quartz in Placitas sieve size -8+16. Blue color is due to the tungsten slide-film used. Width of frame is approximately 5 mm.

DISCUSSION

Concrete

Surrounding soil alkalinity and the presence of ettringite are two factors which do not seem to affect concrete deterioration. The soil pH of the area surrounding the concrete sampled is mildly alkaline, but has no apparent effect on whether the concrete is sound or deteriorated. It does not correlate with any of the other test results. Using the SEM, ettringite has been identified, but its presence shows no correlation with the concrete soundness. The ettringite in most cases does not seem to be causing any distress, as it is growing into open air void spaces. The only exception to that is in sample 4 in which the ettringite was found lining a crack. Even then, it cannot be said whether the crack was existing prior to the ettringite forming in it, or the ettringite formation caused the crack. Either way, the growing ettringite crystals now have the potential to further the splitting of the crack, causing distress to the concrete.

Even though no ASR gel was identified using the SEM, petrographic analysis identified large amounts of potentially reactive ASR particles. However, these results should be taken mainly qualitatively, because the core size was small and only one thin section per core was examined. The results of the petrographic analysis and the chemical staining tests do not correlate. When one test implies a distressing problem, the other tests seem to cancel it out. This discrepancy may be due to a number of reasons. First, there may be additives and admixtures in the concretes that affect the formation of the ASR gel. Even if there is a large amount of potentially reactive aggregate, it may be counteracted by additives that will slow or stop the reaction. This may be the case in

sample 11. It has poor drainage, contains a large amount of reactive particles (55%), and has popouts, but is negative in the chemical stain test. This concrete may contain additives that have not allowed ASR to occur. Second, there may be a time factor. Two samples, from 1993 and 1997, have large amounts of reactive particles, but do not show distress. The ASR gel may not have formed yet, but still may develop and distress the concrete in the future. Third, the drainage conditions may affect the formation of the ASR gel. Two of the samples are from the 1930s, and they may behave differently because of the drainage conditions. One of the samples shows no distress, has a large amount of reactive rhyolite, but is well drained. The other shows map cracking, has a large amount of chalcedony, but is not well drained. The availability of water is extremely important in the production of the ASR gel.

Each of these possible explanations has exceptions. Not all the visually distressed samples are subject to poor drainage, not all the poorly drained samples contain the ASR gel according to the chemical stain test, and not all the concrete with flyash has negative stain test results. This indicates that the deterioration and ASR gel presence may be due to more than what is suggested here, and very well may be a combination of these factors and others of which we are unaware.

The chemical staining test seems not to be a completely reliable test. The 1979 samples of Gibson Boulevard in Albuquerque were all from the same section of road, presumably poured at the same time, under the same conditions, with the same materials added. Each of the six samples tested with the chemical stain test showed different results. They varied from negative to high positive results. This variability either shows

that (1) the chemical stain test is not a reliable test and does not work all the time, or (2) the concrete itself is so variable, that doing the chemical stain test on just one section will not identify an ASR problem. Most likely, the second theory is correct, as shown by the variability in the other tests. Concrete is not homogeneous. What is in a sample of core will depend on where the core is taken. There is no way to tell what is under the surface of a concrete, therefore, this problem will continue to plague concrete researchers.

Aggregate

Both the x-ray diffraction of clay minerals washed off aggregate samples and the petrographic analysis of the aggregate samples shows evidence of potentially reactive particles (except the Tinaja pit). Expansive clays as well as other reactive particles such as chert, chalcedony, and rhyolite/andesite are the most likely cause of the reactivity in concrete.

The expansive mortar bar tests confirm that the aggregate sampled is reactive in ASR. The higher expansion rates correspond to higher amounts of reactive particles, including rhyolite, andesite, chert/chalcedony, quartz, and weathered particles. From this testing though, it cannot be said which, if any, of the rock/mineral types may be more reactive than others. The mortar bar test also has a question of relevancy and reliability to a real situation. Washing the aggregate before testing will remove any clay, silt, and sand-sized particles that may be on the surface of the aggregate, which are particles that will actually go into a concrete under most conditions. Also, crushing an aggregate will produce more surface area, creating unsatisfied charges, essentially making the aggregate

more reactive. The test, therefore is altering the aggregate's true reactivity by washing away fine particles and adding more by crushing. The mortar bar expansion tests are good for comparison, but they do not necessarily translate to a real situation.

The clays washed from the aggregate, as well as the aggregate mineralogy varied from November to May. The pits have variable mineralogy, as can be seen from the pictures taken of the pit walls (Figs. 5 and 6). This heterogeneity in the pit walls carries to the product piles, where the sampling occurred, which then carries to the expansion tests. However, while the results of the mortar bar expansion tests were variable, they still remained within the same categories of innocuous and potentially reactive, giving them some sense of reliability.

CONCLUSIONS

The concretes sampled are not deteriorating due to the formation of ettringite. The ASR gel is most likely the principle cause of the concrete deterioration, indicated by the presence of reactive rock and mineral types and positive chemical stain test results.

The problem of concrete deterioration is one with many variables. Each of the materials that go into making concrete have variability, as well as the processes employed while it is being mixed, poured, and cured. This results in a heterogeneous concrete. This is part of the reason why a single, straight-forward answer to the deterioration of New Mexico concrete could not be found.

An aggregate pit or quarry material can be generally characterized with the petrographic analysis. This test, along with the mortar bar expansion test presents an

overall reactivity in ASR. Once the aggregate pit material has been identified as reactive, proper measures must be taken to mitigate the deleterious ASR reaction, such as adding lithium compounds and/or flyash. Design engineers should know what type of material is being supplied for use in concrete, if it will be reactive, and how to counteract that reactivity.

FUTURE WORK

Many aspects of the alkali-silica reaction are yet to be understood. Many of the variables that play a role in the concrete reactions and deterioration have not been addressed by this project. New or improved testing methods that are more accurate need to be designed and implemented. By continuing to work on concrete problems, we will come to understand the mechanisms and causes of the deterioration.

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APPENDIX A

ASTM C856-83

Standard Practice for Petrographic Examination of Hardened Concrete

Designation: C 856 - 83 (Reapproved 1988)¹

Standard Practice for Petrographic Examination of Hardened Concrete¹

This standard is issued under the fixed designation C 856; 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 Note—Editorial changes were made throughout in February 1988.

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 —A photographic chart of materials, phenomena, and reaction products discussed in Sections 7 through 12 and Tables 1 through 6 are available as Adjunct C 856.

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

	Section
Qualifications of Petrographers	3
Purposes of Examination	4
Apparatus	5
Selection and Use of Apparatus	6
Samples	7
Examination of Samples	8
Specimen Preparation	9
Visual and Stereomicroscope Examination	10
Polarizing Microscope Examination	11
Metallographic Microscope Examination	12
Report	13

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 may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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 5.2.10.1.

2. Referenced Documents

2.1 ASTM Standards:

- C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete²
 - C 215 Test Method for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens²
 - C 294 Descriptive Nomenclature of Constituents of Natural Mineral Aggregates²
 - C 295 Guide for Petrographic Examination of Aggregates for Concrete²
 - C 452 Test Method for Potential Expansion of Portland Cement Mortars Exposed to Sulfate³
 - C 457 Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete²
 - C 597 Test Method for Pulse Velocity Through Concrete²
 - C 637 Specification for Aggregates for Radiation-Shielding Concrete²
 - C 638 Descriptive Nomenclature of Constituents of Aggregates for Radiation-Shielding Concrete²
 - C 803 Test Method for Penetration Resistance of Hardened Concrete²
 - C 805 Test Method for Rebound Number of Hardened Concrete²
 - C 823 Practice for Examination and Sampling of Hardened Concrete in Constructions²
 - C 944 Test Method for Abrasion Resistance of Concrete or Mortar Surfaces by the Rotating Cutter Method²
 - C 1012 Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution³
 - E 3 Methods of Preparation of Metallographic Specimens⁴
 - E 883 Practice for Metallographic Photomicrography⁴
- 2.2 *ASTM Adjunct:*
Adjunct C 856 A chart of 27 photos⁵

3. Qualifications of Petrographers

3.1 It is assumed that the examination will be made by persons qualified by education and experience to operate the

¹ This practice is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.63 on Petrography of Concrete and Aggregates.

Current edition approved Oct. 26, 1983. Published December 1983. Originally published as C 856 - 77. Last previous edition C 856 - 77.

² Annual Book of ASTM Standards, Vol 04.02.

³ Annual Book of ASTM Standards, Vol 04.01.

⁴ Annual Book of ASTM Standards, Vol 03.01.

⁵ Available from ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103. Request adjunct No. 12-308560-00.

TABLE 1 Visual Examination of Concrete (1)^a

Coarse Aggregate	+ Fine Aggregate	+ Matrix	- Air	+ Embedded Items
Composition				
Maximum dimension ⁴ or mm. in the range >>>				
Type:	Type:	color, by comparison with National Research Council Rock Color Chart (1963)	more than 3 % of total, predominantly in spherical voids?	Type, size, location; kinds of metal; other items
1 Gravel	1 Natural sand		less than 3 % of total, abundant nonspherical voids?	
2 Crushed stone	2 Manufactured sand	color distribution:	color differences between voids and mortar?	
3 Mixed 1 and 2	3 Mixed	1 mottled		
4 Other (name)	4 Other (name)	2 even		
5 Mixed 1 +/or 2 +/or 4	5 Mixed 1 +/or 2 +/or 4	3 gradational changes	voids empty, filled, lined, or partly filled	
If Type 1, 2, or 4, homogeneous or heterogeneous	If Type 1, 2, or 4, homogeneous or heterogeneous			
Litologic Types				
Coarse aggregate more than 20, 30, 40, or 50 % of total				
Fabric:				
Shape	distribution	distribution	shape	voids below horizontal or low-angle reinforcement
Distribution	particle shape	as	distribution	
Packing	grading	perceptible	grading (as perceptible)	
Grading (even, uneven, excess, or deficiency of size or sizes)	preferred orientation	ible	parallelism of long axes of irregular voids or sheets of voids: with each other; with flat sides or long axes of coarse aggregate	
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.

equipment used and to record and interpret the results obtained. In some cases, the petrographer will have had experience adequate to provide detailed interpretation of the materials' performance with respect to engineering and other consequences of the observations. In others, the interpretation will be made in part by engineers, scientists, or others qualified to relate the observations to the questions to be answered.

3.2 This practice may be used by a petrographer employed directly by those for whom the examination is made. The employer should tell the petrographer, in as much detail as necessary, the purposes and objectives of the examination, the kind of information needed, and the extent of examination desired. Pertinent background information, including results of prior testing, should be made available. If the petrographer is highly experienced, his advice and judgment should be sought regarding the extent of the examination, and the matters discussed in 3.3.

3.3 This practice may form the basis for establishing arrangements between a purchaser of a consulting service and the consultant. In such a case, the purchaser and the consultant should together determine the kind, extent, and objectives of the examination and analyses to be made, and should 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 or other conditions.

4. Purposes of Examination

4.1 Examples of purposes for which petrographic examination of concrete is used are given in 4.2 through 4.5. 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.

4.2 Concrete from Constructions:

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

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

4.2.3 Determination of the probable future performance of the concrete.

4.2.4 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.

4.2.5 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, the nature of the hydration products, adequacy of curing, and unusually high water - cement ratio of the paste.

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



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

NOTE—Condition—When it is examined at 6 to 10x 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. 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 $\frac{1}{2}$ to $\frac{1}{4}$ 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:		Mineral admixtures ^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 of one kind? More than 50% of one kind? Several kinds?			
Segregation			

^a Dana, E. S. *Textbook of Mineralogy*, 3rd ed., J. W. Ford, John Wiley & Sons, New York, N. Y., 4th ed., 1932, pp. 273-274.

^b Pore visible to the naked eye, or at 1x, 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 $\times 9$ 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.

4.2.6 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.

4.2.7 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.

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

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

4.2.10 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.

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

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

4.3 *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.

4.4 *Concrete Products*

4.4.1 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.

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

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

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

4.5 *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:

4.5.1 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.

4.5.2 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.

4.5.3 To establish whether any other cement - aggregate reaction has taken place. In addition to alkali - silica and alkali - carbonate reactions, these include hydration of

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 (4).	Beneath the softened concrete, which can be tested in accordance with Test Method C 805, the concrete is probably normal if it has not undergone color change. Establish by coning for compressive tests, by wear tests (CRD-C 52) (4), 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 (4).	Examination of the surface, ultrasonic tests, coning, petrographic examination (4).
Color change—When concrete has not scaled, 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 (4). For temperatures up to about 500°C temperature distribution is little affected by using carbonate rather than siliceous aggregate (5). 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 (5).
Aggregate behavior—Aggregate behavior affects strength, modulus, spalling, cracking, surface hardness, and residual thermal strains (4).	Aggregates differ in thermal diffusivity, conductivity, coefficient of expansion. Heat transmission decreases from concrete made with highly siliceous aggregate, sandstone, traprock, limestone, lightweight aggregates (4).	Changes on heating are often accompanied by volume change (4).
Spalling	Occurs subparallel to free face; followed by breaking off saucer-like pieces especially at corners and edges (4).	Determinations by compressive tests and static modulus of cores: Test Method C 805 for qualitative determination; Test Method C 597 (4).
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 (5).	Reduction in strength of concrete containing siliceous gravel after heating, then cooling and testing: Heated to Temperature,	
	°C	Reduction, %
	180	25
	370	50
	573	80
	Reduction in Modulus	Reduction, %
	Temperature, °C	
	200	25
	430	50
	750	70

anhydrous sulfates, rehydration of zeolites, wetting of clays and reactions involving solubility, oxidation, sulfates, and sulfides (see Refs 1, 2, and 3).⁹

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

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

4.5.6 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 C 452 or Test Method C 1012.

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

4.5.8 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.

5. Apparatus

5.1 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.

5.2 For Specimen Preparation:

5.2.1 *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.

5.2.2 *Cutting Lubricant*, for diamond saw.

5.2.3 *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.

5.2.4 *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.

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

5.2.6 *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.

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

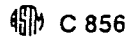


TABLE 4 Outline for Examination of Concrete in Thin Sections

Coarse and Fine Aggregate	Relict Cement Grains and Hydration Products	Characteristics of Cement Paste
<p>Mineralogy: texture, fabric, variable or homogeneous</p> <p>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.</p> <p>Natural mineral aggregate or crushed stone: natural or manufactured fine aggregate.</p> <p>Bond with matrix: peripheral cracks inside the borders of aggregate grains; internal cracks. General microstructures if one can establish that they existed before thin-sectioning.</p> <p>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.</p> <p>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 (6).</p>	<p>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.</p>	<p>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 anhydrous 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 100 μm. 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.)</p> <p>Cement paste in concrete that has been subjected to prolonged acid leaching is low in calcium hydroxide which is present as recrystallized virtually anhydrous grains precipitated near the exterior surfaces.</p> <p>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.</p>

5.2.8 *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.

5.2.9 *Plate-glass Squares*, 12 to 18-in. (300 to 450-mm) on an edge and at least $\frac{3}{8}$ in. (10 mm) thick for hand-finishing specimens.

5.2.10 *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.

5.2.10.1 **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.

5.2.11 *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.

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

5.3 For Specimen Examination:

5.3.1 *Stereomicroscope*, providing magnifications in the range from 7 \times to 70 \times or more.

5.3.2 *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.

5.3.3 *Polarizing Microscope*, for examinations in transmitted light, with mechanical stage; low-, medium-, and high-power objectives such as 3.5 \times , 10 \times , and 20 to 25 \times ; 45 to 50 \times 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.

5.3.4 *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 \times to 500 \times . Reflected polarized light should be available.

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	...
Composition	X	X	...
Rock types	X	X	...
Alteration	X	X	...
degree	X	X	...
products	X	X	...
Coatings	X	X	X
Rims	X	X	...
Internal cracking	X	X	...
Contamination	X	X	X
Concrete:			
Air-entrained or not	X	X	X
Air voids	X	X	X
shape	X
size	X
distribution	X
Bleeding	X	X	X
Segregation	X	X	X
Aggregate-paste bond			
Fractures			
Embedded items	X
size	X
shape	X
location	X
type	X
Alteration	X	X	X
degree and type	X	X	X
reaction products	X	X	X
location	X ^a	X	...
identification	X	X	...
Nature and condition of surface treatments			
Paste:			
Color	X	...	X
Hardness	X	...	X
Porosity	X	X	...
Carbonation			
Residual cement		X	X
distribution	...	X	X
particle size	...	X	X
abundance	...	X	X
composition	X ^b	X	X
Mineral admixtures	...	X	X
size	X	X	X
abundance	X	X	X
identification	X ^c	X	X
Compounds in hydrated cement			
Contamination	X	X	X
size	X	X	X
abundance	X	X	X ^d
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.

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.

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

5.3.6 *Stage Micrometer*. to calibrate eyepiece micrometers.

5.3.7 *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 illumina-

TABLE 6 Secondary Deposits in Concrete^a

Compound and Mineral Equivalent	Indexes of Refraction	Form and Occurrence
Calcium carbonate (CaCO ₃); calcite	n = 1.658 r = 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	n = 1.530 r = 1.680 γ = 1.685	Minute, white prisms or needles in voids or fractures in concrete; rare
Calcium carbonate (CaCO ₃); vaterite	n = 1.544-1.550 r = 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 (n-vaterite); common (7)
6-calcium aluminate trisulfate-32 hydrate (Ca ₆ [Al(CH ₃) ₂] ₂ ·24H ₂ O)(SO ₄) ₂ ·2H ₂ O (8); ettringite	n = 1.464-1.469 ^b r = 1.458-1.462	Fine, white fibers or needles or spherulitic growths in voids, in the cement paste, or in fractures; very common (1, 7)
Tetracalcium aluminate monosulfate-12-hydrate (3CaO·Al ₂ O ₃ ·CaSO ₄ ·12H ₂ O)	n = 1.504 r = 1.49	White to colorless, minute, hexagonal plates in voids and fractures; very rare (7)
Tetracalcium aluminate-13-hydrate (Ca ₄ Al ₂ (OH) ₁₄ ·5H ₂ O)	n = 1.53 r = 1.52	Mica-like, colorless, pseudohexagonal, twinned crystals in voids; very rare (9)
Hydrous sodium carbonate (Na ₂ O·CO ₂ ·H ₂ O); thermonatrite	n = 1.420 r = 1.506 γ = 1.524	Minute inclusions in alkalic silica gel; rare (7)
Hydrated aluminum sulfate (2Al ₂ O ₃ ·SO ₃ ·15H ₂ O); parakuninite	n = 1.463 ± 0.003 r = 1.471 γ = 1.471	Occurring in cavities in intensely altered concrete; very rare (9)
Calcium sulfate dihydrate (CaSO ₄ ·2H ₂ O); gypsum	n = 1.521 r = 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	n = 1.574 r = 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	n = 1.559 r = 1.580	White to yellow, fine-grained encrustations and fillings in concrete attacked by magnesian solutions or seawater; unusual (10, 11)
Hydrous silica (SiO ₂ ·nH ₂ O) opal	n = 1.43	White to colorless, finely divided, amorphous; resulting from intense leaching or carbonation of cement paste; unusual in recognizable proportions
Alkalic silica gel (Na ₂ O·K ₂ O·CaO·SiO ₂)	n = 1.46-1.53	White, yellowish, or colorless; viscous, fluid, waxy, rubbery, hard; in voids, fractures, exudations, aggregate; common (12, 13)
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 ₄ [Si ₄ (OH) ₁₂] ₂ ·24H ₂ O)(SO ₄) ₂ ·CO ₂ ·2H ₂ O	n = 1.504 r = 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 (14)
Syngenite K ₂ Ca(SO ₄) ₂ ·H ₂ O	n = 1.501 (15) r = 1.51 γ = 1.51	Found in cavities and zones peripheral to slate particles, in fibrous form (16)
Hydrotaalcite Mg ₃ Al ₂ (OH) ₂ (CO ₃) ₂ (H ₂ O) ₂ (8)	n = 1.510 ± 0.003 r = 1.495 ± 0.003	Foliated platy to fibrous masses (17, 8)

^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 (15) and thaumasite (14), but it is not known that the naturally occurring minerals and compounds found in hydrated cement are of the same composition.

tors 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.

5.3.8 *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.

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

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

5.3.11 *Lens Paper*

5.3.12 *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, cali-

brated 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.

6. Selection and Use of Apparatus

6.1 Laboratories should be equipped to provide photographs, photomicrographs, 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 Practice E 333.

6.2 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.

6.3 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).

7. Samples

7.1 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.

7.2 *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.

7.2.1 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.

7.2.2 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.

7.2.3 The information provided with the samples should include:

7.2.3.1 The location and original orientation of each specimen (see Practice C 823).

7.2.3.2 The mixture proportions of the concrete or concretes.

7.2.3.3 Sources of concrete-making materials and results of tests of samples thereof.

7.2.3.4 Description of mixing, placing, consolidation, and curing methods.

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

7.2.3.6 Conditions of operation and service exposure.

7.2.3.7 The reason for and objectives of the examination.

7.2.3.8 Symptoms believed to indicate distress or deterioration, and

7.2.3.9 Results of field tests such as measurements of pulse velocity (Test Method C 215), rebound hammer numbers (Test Method C 805) or probe readings (Test Method C 803).

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

7.3.1 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.

7.3.2 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.

8. Examination of Samples

8.1 *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. Practice C 457 should be referred to for those relevant subjects not described here.

8.2 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 mounted 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, 3, and 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.

8.3 Photography—It is frequently desirable to maintain a photographic record as illustrations for the report and for future reference. Photographs which may be useful include:

8.3.1 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.

8.3.2 Photographs and photomicrographs 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.

8.3.3 Photomicrographs 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.

8.3.4 Photomicrographs of thin sections, immersion mounts, and polished sections at magnifications from 2× 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.

8.3.5 The range of photographic techniques used should be adequate to provide a choice useful for illustrations for the report and for the record. The subjects may usually be selected during the visual and stereomicroscopic examinations.

9. Specimen Preparation

9.1 Preparation for Visual and Stereomicroscope Examination

9.1.1 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.

9.1.2 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.

9.1.3 It is useful to prepare at least one sawed surface by grinding it with progressively finer abrasives (as described in Practice C 457) 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.

9.1.4 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 2 3/8-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.

9.1.5 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.

9.1.6 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.

9.1.7 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.

9.2 *Preparation of Immersion Mounts*—Secondary products of nondeleterious and deleterious chemical processes may be observed during the stereomicroscopic examination. Finely pointed probes may be used to excavate and transfer them to microscope slides. The material is then covered with a cover glass, immersed in a refractive index liquid, and examined using a polarizing microscope to determine the optical characteristics of the product such as index of refraction, birefringence, and optical character.

9.3 *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 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.

9.3.1 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.

9.4 *Preparation for Examination with the Metallographic Microscope*

9.4.1 The preparation of specimens for examination with the metallographic microscope is described in Methods E 3.

9.4.2 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.

10. Visual and Stereomicroscope Examination

10.1 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.

10.2 *Concrete from Constructions (Core Log)*:

10.2.1 Fit fragmented cores together and determine if any pieces are missing. Measure cores to verify field data and prepare a diagrammatic 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.

10.2.2 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.

10.3 *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.

10.4 *Concrete Products*:

10.4.1 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.

10.4.2 Procedures such as those set forth in 10.1 and 10.2 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.

10.5 *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.

10.6 *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 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.

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

10.6.2 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 (12, 20, 21). Pale rims in mortar bordering coarse aggregate (7, 19) and pale areas in the mortar may be gel-soaked paste (6) or highly carbonated paste adjoining carbonate aggregate that has undergone an alkali - carbonate reaction.

10.6.3 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 water organisms. Such organisms may provide evidence that a part of a structure that is not ordinarily submerged has been submerged.

10.6.4 Quantitative determination of constituents of concrete can be made by microscopical point-count or linear traverse procedures in general accord with the requirements of Practice C 457. 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.

11. Polarizing Microscope Examination

11.1 *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 sectioning. It is very difficult to avoid production of microfractures 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.

11.1.1 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.

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

11.2 *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.

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

12. Metallographic Microscope Examination (22)

12.1 There are two advantages of examining concrete with a metallographic microscope: only one prepared surface is required; the polished surface enhances and allows etching residual cement. When the aim of the petrographic examination is particularly concerned with the composition and characteristics of the cement, polished sections that are produced by grinding and polishing a sawed piece of concrete are particularly useful in detection of unusual amounts of free CaO or MgO or their hydration products and other cement constituents. Although the coarser relict cement grains may be far from a representative sample, they can reveal characteristics that, if correlated with the behavior and history of the concrete, can be extremely helpful in interpreting its behavior. Table 5 lists constituents of concrete visible, after preparation, using different microscopes. Table 6 is a list of substances reported as occurring in concrete.

13. Report

13.1 The report of the examination should include the following:

13.1.1 Location and orientation of the samples in the construction or products or type of specimen.

13.1.2 History of the samples insofar as is available.

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

13.1.4 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

13.1.5 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.

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APPENDIX B

Chemical Staining Method For ASR Gel



PII S0008-8846(97)00123-3

A SIMPLE ENVIRONMENTALLY FRIENDLY, AND CHEMICALLY SPECIFIC METHOD FOR THE IDENTIFICATION AND EVALUATION OF THE ALKALI-SILICA REACTION

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(Received February 13, 1997; in final form July 9, 1997)

ABSTRACT

A rapid, dual staining method is described whereby reaction products associated with the alkali-silica reaction (ASR) are readily identified by their pink or yellow color following treatment in the laboratory or field. The method is based on both the compositional and physical characteristics of the ASR gel; hence, it provides greater information than non-chemical-specific techniques (such as the uranyl acetate method). In addition, the chemicals used in the staining method pose minimal health risks and are environmentally benign.

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Introduction

One of the principal mechanisms of premature degradation of concrete is alkali silica reaction (ASR), in which an alkali-rich silica gel forms around and within some reactive aggregate grains, in pores, along fractures, and within paste (1). This reaction can dramatically impact the strength and durability of a concrete structure and significantly shorten its lifetime.

ASR results from an interaction between high pH pore fluid, some aggregate constituents (e.g., poorly crystalline silica phases), and alkali cations (Na^+ and K^+) released by the cement and/or aggregate (2,3). As the alkali content of the pore fluid (and, hence, the pH) rises, the solubility of the silica phases increases, resulting in a release of silica to form an alkali-calcium-silica-hydrate gel (commonly referred to as "ASR gel"). Gel formation leads to an increase in volume relative to the reacting silica phases, which causes internal pressure in the concrete and eventually results in fractures. This process can be exacerbated by freeze-thaw cycles and by salting of road surfaces.

Methods for Identifying ASR Gel. Identification of ASR as the cause of a structure's demise is critical to a number of practical needs. The recognition that a particular aggregate is prone to ASR is probably the prime method used to ensure that ASR is avoided. Although several rapid tests (4,5) offer the ability to screen aggregates for their potential reactivity, field experience shows that these do not always accurately predict an aggregate's performance in a particular mix. Thus, identification of ASR in a degrading structure is the ultimate check of an aggregate's reactivity. In addition, potential treatment methods—such as the application of a lithium compound to a structure—are likely to be most effective if ASR can

be identified as early as possible. Finally, identification of ASR is an important component of the management of a transportation infrastructure (e.g., by allowing long-term scheduling and budgeting of repair and replacement). Hence, the identification of ASR has been the focus of a number of efforts, including the Strategic Highway Research Program (SHRP).

There are two principal methods used to identify ASR gel in a concrete structure: petrographic analysis and labeling with uranyl acetate. The most common type of petrographic analysis relies on morphological and optical identification of the ASR gel product using binocular and petrographic microscopes. Less common methods of petrographic analysis involve morphological and chemical identification using a scanning electron microscope (SEM). Each of these methods requires specialized training and equipment; hence, petrographic analysis is typically performed by a private laboratory, making ASR verification costly and time-consuming (requiring days to weeks or longer).

One recent outcome of SHRP (6) was the evaluation of a technique by Natesaiyer and Hover (7,8) in which uranyl acetate [$\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2$] is applied to a concrete surface and rinsed off, leaving uranyl (UO_2^{2+}) sorbed to the negatively charged ASR gel. The distribution of the uranyl ion is monitored by its yellow-green fluorescence under ultraviolet light, thereby showing the distribution of the ASR gel.

Development of the uranyl-acetate technique was an important contribution to the practical needs of individuals interested in identifying ASR products in the field or in their own laboratory. Nevertheless, there are several potential concerns with the uranyl-acetate method, including: personnel are exposed to uranium-containing solutions and uranium-contaminated solids; uranium contaminated wastes are generated; the method requires the use of an ultraviolet light source in nearly complete darkness; and, the method is somewhat non-specific for ASR gel because the uranyl ion is capable of binding to any negatively charged surface.

These first two issues are often believed to be insignificant because of the relatively small amount of uranyl acetate that is used. However, the amount of uranium present in a typical uranyl acetate test is significant from a regulatory standpoint. OSHA provides three values for assessing exposure to uranium compounds, the most relevant of which for short-term exposures is the ceiling limit (which is the concentration that should not be exceeded in an occupational setting). The ceiling limit for inhalation of uranium compounds is 0.6 mg U/m^3 . In comparison, the treatment of a concrete core with $\sim 5 \text{ ml}$ of 0.125 molal uranyl acetate solution ($5 \text{ gm}/100 \text{ mL}$ acetic acid) involves 150 mg U .* In other words, the ceiling limit may be reached by a person using the uranyl acetate method if only about 0.4% of the applied uranium becomes airborne in a meter-cubed of air. Some practices (e.g., vacuuming of a bush-hammered, treated surface) may result in short exposures at significant levels. In addition, environmentally responsible disposal of the uranium-contaminated liquid and solid waste generated by this method is costly.

The necessity of viewing the sample in ultraviolet light to see the presence of sorbed uranyl ions greatly restricts the analyst's ability to interpret the distribution of ASR gel. One cannot

*Even if uranyl acetate is made with depleted uranium, it is still both a radiological and a toxicological hazard. In fact, all isotopes of uranium are radioactive. The primary difference between natural and depleted uranium is the ^{235}U content (0.7 wt% vs. 0.2 wt%). This has only a minor effect on the radioactivity and no known effect on the toxicity.

observe simultaneously both the fluorescing gel and the aggregate, pore space, fractures, and past, because these latter components are difficult to observe in the UV light. Consequently, the uranyl acetate method does not help in a petrographic analysis aimed at understanding the extent and nature of ASR damage, unless one uses specialized petrographic equipment (8). In addition, the uranyl acetate method can be inconvenient to conduct in the field where it is difficult for the eyes to adjust from the brightness of daylight to the dimness of the ultraviolet viewing box.

The final issue—the non-specificity of the uranyl ion—raises the concern that the method can lead to false positives, particularly if the analysis is based solely on the presence of fluorescence. The non-specificity arises because the uranyl ion in solution will readily exchange with most cations associated with solids in the concrete (9), including K^+ , Na^+ , and Ca^{2+} . Any negatively charged surface (e.g., most silicates that are commonly present in concrete) can potentially sorb the uranyl ion by an ion-exchange process, so any negatively charged, high surface area material may test positive with the uranyl acetate technique. In addition, the non-specificity results in a loss of information concerning the nature of the ASR products (i.e., gels of different compositions are not differentiated). This non-specificity combined with the need to view the concrete under UV light limits the uranyl acetate method as a petrographic tool.

A New Method for Identifying ASR Gel. We report an alternative method for identifying ASR gel and its distribution in hand specimen using a dual staining technique (10). The method overcomes many of the limitations of the uranyl acetate method: the technique is based on environmentally friendly chemicals with relatively minor health concerns; the stains are chemically specific for ASR gel, and they are visible in normal light. The method allows ASR gel to be identified by individuals lacking extensive training in petrographic analysis while greatly improving the petrographic investigations by trained individuals. The method is capable of revealing the detailed reaction and distribution of ASR gel with respect to aggregate type, pore structure, and fractures. Finally, the method provides an overview of the abundance and distribution of two chemically distinct ASR gel products which are indistinguishable by both standard petrographic analysis and the uranyl-acetate technique.

The Staining Method

The staining method is based on two chemicals that are diagnostic for two components of gels associated with ASR: potassium and calcium.

Gel produced by ASR is typically enriched in both sodium and potassium (1,11), which are believed to originate largely from the cement. Most cements have potassium as their dominant alkali contaminant (12); likewise, ASR gels often have more potassium than sodium (e.g., Ref. 8; see also summary of reported compositions in Ref. 1). In our observations of ASR gels (primarily in samples from New Mexico), potassium is always the dominant alkali cation as determined by energy-dispersive spectroscopy (EDS) analysis. In contrast, typical calcium-silica-hydrate (CSH) gels produced by hydration of cement phases do not contain appreciable potassium. Consequently, one of the probes we selected for ASR gel was sodium cobaltinitrite, $Na_3Co(NO_2)_6$. Sodium cobaltinitrite is well known in analytical chemistry for use in the determination of the potassium content of a fluid (13), wherein the dissolved sodium cobaltinitrite reacts with potassium to form a $K_2NaCo(NO_2)_6 \cdot 6H_2O$ precipitate. It has also been used in the geological sciences for staining potassium feldspars

that have been etched in hydrofluoric acid (14-16). In the present method, sodium cobaltinitrite solution is allowed to react directly with the concrete surface (following a simple pre-wetting of the surface with deionized water but without an acid-etching step), ultimately resulting in the deposition of a yellowish precipitate—presumably $K_2NaCo(NO_2)_6 \cdot 6H_2O$ —on the surface of K-rich ASR gels.

Calcium is also a common constituent of both ASR and CSH gels; hence, we selected rhodamine B ($C_{28}H_{31}N_2O_3Cl$) as a stain for calcium, initially in an attempt to improve the contrast between the yellow-stained and non-yellow-stained areas. However, the rhodamine B did not appear to sorb to ordinary CSH but did sorb to a calcium-rich, potassium-poor silica gel that is apparently associated with ASR (as will be discussed below). In addition, rhodamine B may sorb to some but not all calcium carbonate. As discussed below, parts of some limestone aggregates and some layers within a travertine-like deposit were stained lightly by rhodamine B, but most calcium carbonate remained unstained. We do not know why only some calcium carbonate reacts with the rhodamine B (e.g., whether it reflects grain size, defect density, or some other property).

Both rhodamine B and sodium cobaltinitrite can be purchased as chemical reagents. The working solutions were prepared as saturated aqueous solutions (i.e., excess solid was present in the solutions). Sodium cobaltinitrite can be stabilized for longer periods when a small amount of acetic acid is added to the solution (17). Both simple aqueous solutions of sodium cobaltinitrite and solutions with weak acetic acid were tested, but no significant difference in behavior was found over the course of several weeks (i.e., both solutions remained effective as stains). Solutions were, however, prepared with deionized water in an attempt to minimize external sources of potassium.

Concrete samples were tested by briefly rinsing the surface to be examined with deionized water followed by treatment with the staining solutions. Each solution was applied to the surface in turn and allowed to remain on the surface for approximately 30-60 seconds followed by a thorough rinse of the surface with deionized water. The effect of differing staining sequences was examined, including the use of both stains simultaneously. The best results appeared to be obtained with the following sequence: rinse—sodium cobaltinitrite treatment—rinse—rhodamine B treatment—rinse. After final rinsing, stained regions could be seen clearly on the concrete surfaces; however, stained regions were most easily observed following drying of the surface. The intensity of the staining appeared to fade over the course of weeks to months; however, the original staining intensity could be achieved with a re-treatment of the concrete surface.

Results

The following observations are based on treatment of numerous samples from approximately 20 different concrete structures of various ages. The concrete structures include sections of highways and roads, bridges, and sidewalks exhibiting field conditions ranging from good to highly distressed. Cores were available from most localities, but bush hammering was used only on a few localities. Some samples were treated immediately following coring, whereas other samples were examined years after coring.

Yellow-Stained Regions. In hand sample, yellow staining was always associated with a "gel-like" material occurring as halos around some coarse aggregates, as precipitates in voids, and occasionally as precipitates on fracture surfaces (Figs. 1-2, 4). No other occur-

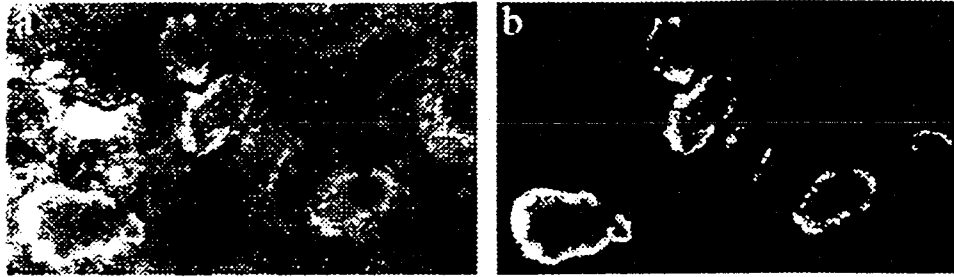


FIG. 1.

a) Optical scan of a treated concrete core from a highly distressed gutter at Los Alamos National Laboratory. Surface showed yellow-stained gel but no pink-stained gel. b) Processed image from (a) showing distribution of yellow staining. Images are approximately 47 mm along the bottom edge.

rences of yellow staining were observed (e.g., feldspars associated with coarse aggregates did not stain yellow). Some gel-like precipitates on fractures initially stained a faint yellow but were subsequently stained pink by rhodamine B. However, most regions that stained deeply yellow remained yellow following treatment with rhodamine B.

At higher magnification (i.e., with a 10 \times hand lens or at up to \sim 500 \times in a binocular microscope), the yellow staining was clearly associated with either a massive or granular precipitate. The massive precipitate had a distinctive gel-like morphology (smooth surface; desiccation cracks). The granular precipitate typically formed particles approximately 10–100 μ m across and appeared to consist of aggregates of tiny crystals that may have grown from the gel. The granular precipitates typically occurred toward the interiors of the reacting

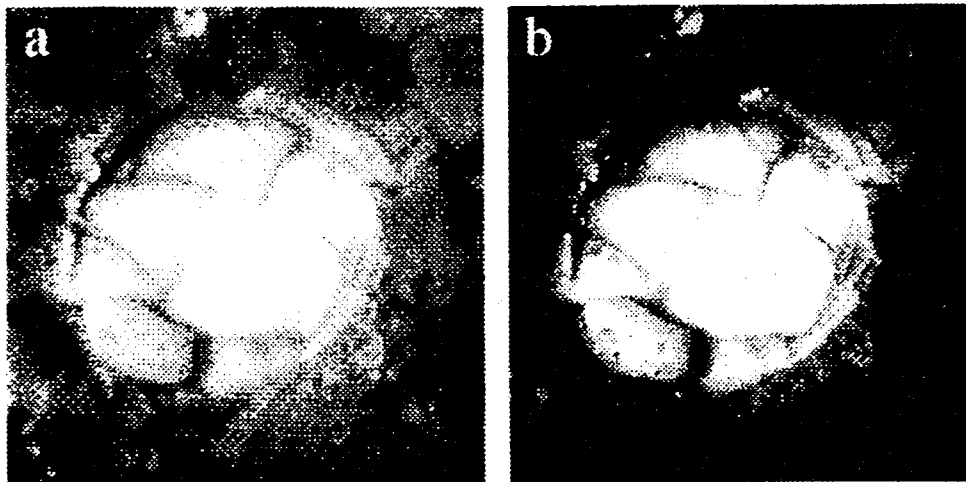


FIG. 2.

a) Photograph of a treated concrete core from US 20 in Iowa. A few voids were filled with yellow-stained gel, but most voids were empty; b) Processed image from (a) showing distribution of yellow staining. Images are approximately 1 mm along the bottom edge.

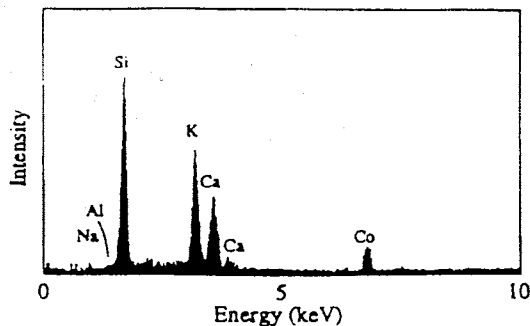


FIG. 3.

Representative chemical analysis of yellow-stained gel.

aggregates, whereas the massive precipitate occurred toward the rims. With the naked eye, the massive and granular precipitates could not readily be differentiated.

Samples of the massive material were examined in a petrographic microscope and found to be isotropic.

Samples of both the massive and granular materials that had stained yellow were hand picked for examination by EDS in the SEM.† All such materials were dominantly Si with large amounts of K and Ca (Fig. 3). In addition, they contained Co (from the stain), Na and, in some cases, minor Al or S. The relative amounts of the elements varied from sample to sample. No attempt was made to quantify these data, because of the general difficulties associated with EDS data and because of the problems associated with the geometry of the rough surfaces examined. Hence, it is difficult to determine from these data whether the $(Ca+K+Na)/Si$ ratio (or, perhaps, the $(Ca+2K+2Na)/Si$ ratio) is similar between the two gels (see also Fig. 5).

Based on these observations, we conclude that the yellow-stained materials were $K+Ca+Si \pm Na$ gels resulting from ASR.

Pink Staining Regions. Pink staining was generally less common than yellow staining and was typically found in samples from structures exhibiting a higher degree of deterioration. In all but two instances (discussed below under "Non-ASR-Related Pink-Staining Regions"), pink staining only occurred when extensive yellow staining was present in the sample.

In hand sample, pink staining generally occurred on fracture surfaces, immediately adjacent to a reacting aggregate (with a yellow-stained rim) (Fig. 4). In such examples, the pink staining occurred on patches of the surface that were coated with a white precipitate prior to staining, where the white precipitate also included the material comprising the "reaction rim" around reactive aggregates. However, the actual reaction rim always stained yellow (although some rims additionally exhibited a slight pinkish hue), and there was a sharp break between the reaction rim and the pink-stained precipitate covering the rest of the fracture surface. Prior

†Plots were obtained by EDS analysis in an SEM of stained, carbon-coated gels hand picked from concrete cores. We used a Be-windowed detector that is insensitive to X-rays from elements with atomic numbers less than F.

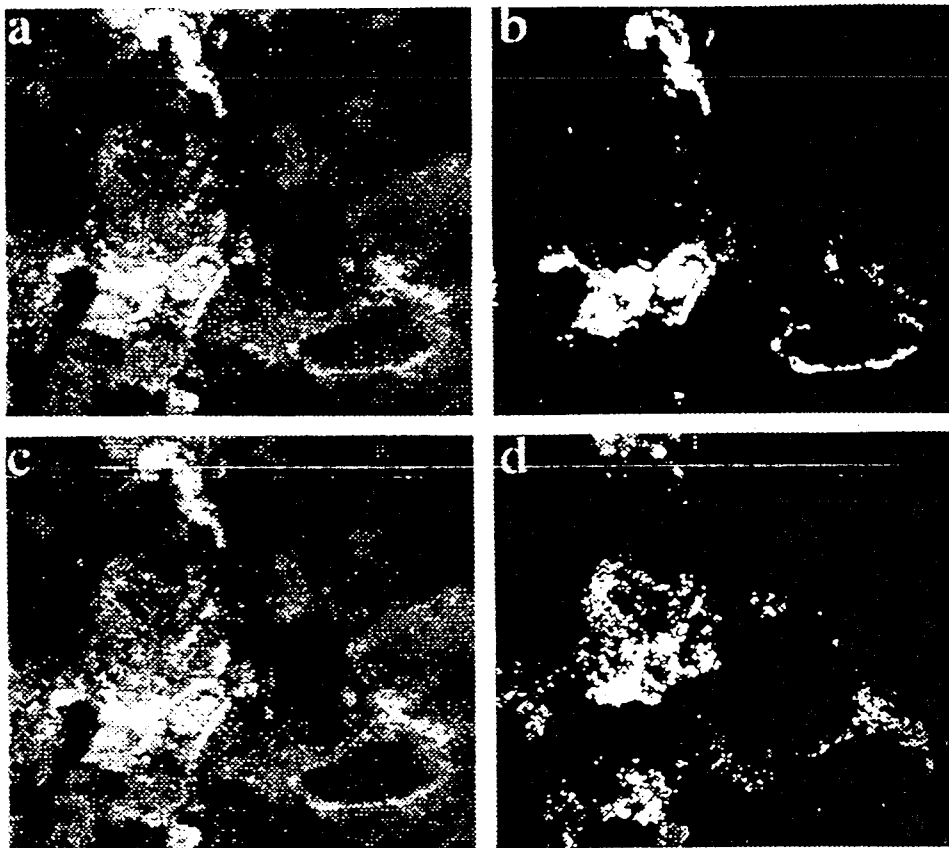


FIG. 4.

a & c) Optical scan of treated concrete during a highly stressed bridge along I-25 near Pecos, NM. Surface of fracture, yellow staining (gel around aggregate particles and pink-stained gel) portion of the fracture surface. Processed images from (a & c) showing distribution of yellow staining (b) processed image from (a & c) showing distribution of pink staining. Images are approximately 55 mm along the bottom edge.

to staining, no obvious difference could be observed between the white precipitate on the fracture surface and the white precipitate associated with the reaction rims. In some cases, the white precipitate adjacent to a reaction rim was stained yellow following sodium cobaltinitrite treatment but would then become pink following treatment with molybdate B.

At higher magnification, like the yellow staining, the pink staining was clearly associated with either a massive or granular precipitate that had inverted angles like the yellow-stained angles. An additional occurrence for the pink staining was within portions of the paste in some concrete samples. These pink-staining materials occurred in what appeared to be "normal" paste (i.e., no free CSH); however, in general, CSH did not stain pink in most concrete samples investigated.

Samples of both the massive and granular pink-stained materials were hand picked for

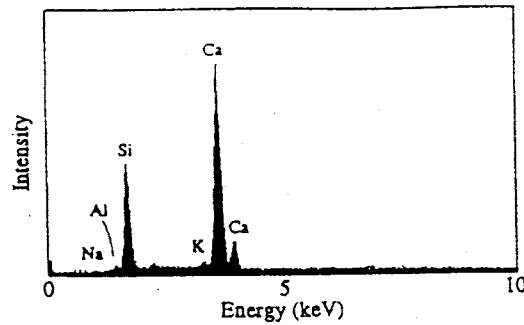


FIG. 5.

Representative chemical analysis of pink-staining gel.

examination by EDS in the SEM. All such materials were dominantly Si with large amounts of Ca (Fig. 5). In addition, they occasionally contained minor amounts of K and perhaps Na.

Based on these observations, we conclude that the pink-staining materials were alkali poor Ca+Si gels associated in some way with advanced ASR. Based on petrographic observations, it appears that these pink-staining gels are derived from the yellow-staining gels. The pink-staining gels may have formed directly from the alkali-rich (yellow) gels by leaching of the alkalis or by reacting with the cement paste or other components in the fractures (e.g., perhaps CO_2).

Non-ASR-Related Pink-Staining Regions. Two other examples of pink-staining materials were observed: some calcium carbonate and some "gel-free" fractures in two distressed cores.

Some occurrences of calcium carbonate were susceptible to rhodamine B. Although we have not undertaken any systematic investigation of staining of calcium carbonates, we noted two instances in which calcite was stained pink. In one instance, a travertine-like deposit (a layered deposit of calcium carbonate) had formed on the side of a highly distressed bridge with advanced ASR. Some layers within this deposit stained pink, whereas others did not. The other instance was in the case of a limestone aggregate used in a concrete exhibiting minor ASR and delayed ettringite formation (DEF). Parts of the limestone aggregate were lightly stained pink; however, much of the limestone aggregate remained unstained. (As an aside, it is possible, perhaps likely, that some of the "gels" we have investigated with our staining procedure have wholly or in part carbonated following exposure to the atmosphere; hence, other precipitates that we've observed to stain yellow or pink may in fact contain carbonates derived from gels.)

Another occurrence of pink staining was along some fracture surfaces of cores from a section of a concrete road that is exhibiting distress in the field. These particular cores showed no signs of ASR and had no occurrences of yellow-stained gel. The distress mechanism for these road sections remains undetermined, and we do not know why some fracture surfaces from these cores stained pink. The surfaces did not have any observable precipitate and were indistinguishable from other fracture surfaces that did not stain pink. (We did not collect the core, so we could not determine which fractures might have been associated with cracks in the concrete that were present prior to coring). One possibility is that these pink-stained

surfaces were coated with a very thin carbonate precipitate that was unobservable prior to staining.

Comparison With the Uranyl Acetate Method. We have compared our dual stain approach with the uranyl acetate method on several samples of concrete suffering from ASR as well as some apparently free of ASR. These comparisons have been made on matched fracture surfaces (i.e., where one face was stained by our approach and the other stained by uranyl acetate) and on individual surfaces that were treated by both methods (either uranyl acetate first followed by the dual staining treatment or vice versa). In all cases, our dual staining method identified the same materials that the uranyl acetate method tagged. Interestingly, the uranyl acetate method tagged both the pink- and yellow-staining materials; hence, the dual staining method provided greater information on the nature of the ASR in a given concrete (i.e., it identified two chemically distinct gels). This improvement relates directly to the non-specificity of the uranyl acetate technique, as discussed in the Introduction.

In addition to providing greater information, the dual staining method was superior to the uranyl acetate method because it augmented standard petrographic examination of the concrete. Unlike the uranyl ion (which requires a UV source and nearly complete darkness to be seen), these colored stains allowed the reaction products to be identified concurrent with identification of aggregate lithology and the gross morphology of the concrete. Furthermore, the stains can be seen in a binocular microscope or hand lens, which greatly improved our petrographic examination of cores.

The staining method was particularly useful in our examination of one core from a distressed highway in Iowa. This concrete had been examined by several experts, and the distress mechanism remains hotly debated. At issue is whether the concrete contains ASR or DEF and, if so, how much. Standard petrographic examination of the core would lead to conclude that most of the voids contain both a gel-like precipitate and ettringite. Following staining, however, the extent and distribution of pink- and yellow-stained gels was readily apparent, and ettringite (unstained) was readily differentiated from the ASR gels in the core as a whole.

Effect of Staining on Ettringite. One sample of a core from a highway section known to contain ettringite-filled vugs was examined by the staining procedure. Both pink- and yellow-stained gel were observed in the core following treatment. However, clearly identifiable ettringite crystals—fine needles seen with a binocular microscope; presence of Ca, Al, and S verified by energy dispersive spectroscopy (EDS) analysis in the scanning electron microscope (SEM)—did not stain either pink or yellow. Some voids containing a pink-stained gel covered with unstained ettringite crystals were also observed. We cannot rule out the possibility that microcrystalline ettringite might have stained pink, but we believe, based on the non-staining of the larger crystals, that this is probably unlikely. Furthermore, ettringite should not stain yellow, because it lacks potassium.

Preparation of Concrete Specimen. In our evaluations of this dual staining method, we concluded (like other workers) that bush hammered surfaces do not provide readily interpretable information on the nature of ASR in the particular structure. We evaluated the staining method on several bush hammered surfaces from structures for which we also had cores. Although some staining could be seen on bush hammered surfaces, the staining was not nearly as striking as staining on fractured surfaces from the cores. Moreover, it was clear

from the cores that in some cases ASR was more pronounced in the upper 0.1 m of the structure, whereas in other cases it was more pronounced in the lower portions of the structure. Hence, a bush hammered surface would not be expected to provide an adequate assessment of ASR within a concrete structure.

Another concern expressed by some workers is that method such as uranyl acetate must be applied to freshly fractured surfaces. In contrast, the dual staining method reported here appeared to work adequately both on freshly fractured surfaces and on aged (up to several years) surfaces.

Conclusions

The dual staining method described above provides a simple means for evaluating the extent and distribution of gel-related products associated with ASR. The method results in pink- and yellow-stained areas that give the observer a rapid indication of the degree to which ASR has affected the concrete and that are easy to interpret (even for someone not experienced in petrographic analysis).

For experienced petrographers, the method greatly improves the evaluation of ASR in a concrete, because the extent and distribution of ASR are readily determined and because the method provides additional information on the distribution of two different gels (a yellow-staining gel that is alkali-rich and a pink-staining gel that is alkali-poor). Like the uranyl acetate method, the dual staining method allows individuals to evaluate concrete for ASR in house and even in the field. Unlike the uranyl acetate method, the dual staining method does not require the use of a source of UV illumination in a light-tight environment and, more importantly, does not involve the use of uranium. In addition, the dual staining technique described above differs markedly from other staining techniques such as with cuprammonium sulphate (18) (which is non-chemical-specific, like uranyl acetate) or pH indicators such as phenolphthalein. Finally, false positives will be uncommon with this technique, because only materials that can cation-exchange potassium should stain yellow. Hence, common rock forming minerals such as feldspars, micas, and illite remain unstained. Less common minerals such as K-rich smectites and K-rich zeolites (most smectites and zeolites are not K-rich) may stain yellow. In general, however, these "false positives" should be easily recognized in hand specimen, particularly in light of the unique staining pattern (yellow within aggregates and pink surrounding aggregates) that appears to be typical of advanced ASR.

Acknowledgements

This work was supported by the Department of Energy through contract no. W-7405-ENG-36 to Los Alamos National Laboratory via a Laboratory-Directed Research and Development grant. We have benefited greatly from our interactions and discussions with a number of concrete experts, including Joe Barela, Wendell Dubberke, Steve Forster, David Gress, Ron Gorver, Vernon Marks, and Roger Surdahl. In addition, we wish to thank Robert Raymond for extensive discussions on ASR and for help on SEM examination; and Dave Vaniman and two anonymous reviewers for critical reviews of the manuscript.

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APPENDIX C

AASHTO Designation TP14

**Standard Test Method for Accelerated Detection of Potentially Deleterious
Expansion of Mortar Bars Due to Alkali-Silica Reaction**

AASHTO Designation T162-92

Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

AASHTO Designation M210-92

**Apparatus for Use in Measurement of Length Change of Hardened Cement Paste,
Mortar, and Concrete**

AASHTO Provisional Standard



Standard Test Method for Accelerated Detection of Potentially Deleterious Expansion of Mortar Bars Due to Alkali-Silica Reaction

AASHTO Designation TP14
Edition 1A

August 1993

This is Edition 1A of the provisional standard (PS) based on the technical substance provided by the Strategic Highway Research Program (SHRP) researchers. The PS was collated and formatted jointly by the AASHTO and SHRP Staffs.

This PS is being referred to the AASHTO Subcommittee on Materials (SOM) for a review, ballot and approval. If the approval process produces changes, an amended version of the PS shall be published as the second edition.

AMERICAN ASSOCIATION OF STATE HIGHWAY AND
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**Standard Test Method for
Accelerated Detection of Potentially
Deleterious Expansion of Mortar Bars
Due to Alkali-Silica Reaction**

AASHTO Designation TP14¹

1. Scope

1.1 This test method allows detection within 16 days of the potential for deleterious expansion of mortar bars due to the alkali-silica reaction.

1.2 This standard may involve hazardous materials, operations and equipment. This proposed standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the proposed standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. A specific precautionary statement is given in Note 3.

1.3 The values stated in SI units are to be regarded as standard. The values in inch-pound units are shown in parentheses, and are for informational purposes only.

2. Referenced Documents

2.1 AASHTO Standards

- M85 Specification for Portland Cement
- M92 Specification for Wire-Cloth Sieves for Testing Purposes
- M201 Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
- M210 Specification for Apparatus for Use in Measurement of Length Change of Hardened Cement Paste, Mortar and Concrete
- T106 Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cubic Specimens)
- T162 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

2.2 ASTM Standards

- C295 Practice for Petrographic Examination of Aggregates for Concrete
- C856 Practice for Petrographic Examination of Hardened Concrete
- D1193 Specification for Reagent Water

3. Significance and Use

3.1 This test method provides a means of detecting the potential of an aggregate used in concrete for undergoing alkali-silica reaction and resulting potentially deleterious internal expansion. It is based on the NBRI accelerated test method. [1][2][3]

3.2 When expansions greater than 0.10 percent are developed within 16 days from casting, it is recommended that supplementary information be developed to confirm that the expansion is actually due to alkali reactivity. Sources of such supplementary information include: (1) petrographic examination of the aggregate by ASTM C295 to

¹ This standard is based on SHRP Product 2009.

determine if known reactive constituents are present; and (2) examination of the specimens after tests by ASTM C856 to identify the products of alkali reactivity.

3.3 When it has been concluded from the results of tests performed using this test method and supplemental information that a given aggregate should be considered potentially deleteriously reactive additional studies, using alternative methods, may be appropriate to develop further information on the potential reactivity.

4. Apparatus

4.1 The apparatus shall conform to Specification M210, except as follows:

4.2 Sieve - Square hole, woven-wire cloth sieves, shall conform to the requirements of M92.

4.3 Mixer, Paddle, and Mixing Bowl - Mixer, paddle, and mixing bowl shall conform to the requirements of T16 except that the clearance between the lower end of the paddle and the bottom of the bowl shall be 5.1 ± 0.3 mm (0.20 ± 0.01 in.)

4.4 Tamper and Trowel - The tamper and trowel shall conform to Test Method T106.

4.5 Containers - The containers shall permit the test specimens to be totally immersed in either water or 1 normal NaOH solution. The containers shall be made of material that can withstand prolonged exposure to 80°C (176°) and shall be inert to a 1 normal NaOH solution. The containers shall be equipped with tightfitting covers, seals or both. The containers shall be constructed in a manner that permits test specimens to be supported, without using the gage studs, so that the solution has access to the whole test specimen, and the specimens do not touch the sides of the container or each other.

Note 1 - The NaOH solution will corrode glass or metal containers. Polypropylene containers are recommended.

4.6 Oven - A thermostatically controlled oven capable of maintaining a temperature of $80.0 \pm 1.7^{\circ}\text{C}$ ($176 \pm 3^{\circ}$) or a thermostatically controlled water bath capable of maintaining the same temperature range.

4.7 Moist Room or Closet - The moist closet or room shall conform to M201.

5. Reagents

5.1 Sodium Hydroxide (NaOH) - USP or technical grade may be used, provided the Na^+ and OH^- concentrations are shown by chemical analysis to lie between 0.99 and 1.01 normal.

5.2 Purity of Water - Unless otherwise indicated, water shall be reagent Type IV water conforming to ASTM D1193.

5.3 Sodium Hydroxide Solution - Each liter of solution shall contain 40.0 g of NaOH dissolved in 900 mL water, and shall be diluted with additional distilled or deionized water to obtain 1.0 L of solution. The volume proportion of sodium hydroxide solution to mortar bars in a storage container shall be 4.0 ± 0.5 volumes solution to 1 volume of mortar bars.

Note 2 - The volume of a mortar bar may be taken as 184 mL (11.25 in³).

Note 3 - Precaution - Before using NaOH, review: (1) the safety precautions for using NaOH; (2) first aid for burns; and (3) the emergency response to spills, as described in the manufacturer's Material Safety Data Sheet or other reliable safety literature. NaOH can cause very severe burns and injury to unprotected skin and eyes. Suitable personal protective equipment should always be used. These should include full-face

shields, rubber aprons, and gloves impervious to NaOH. Gloves should be checked periodically for pin holes.

6. Conditioning

6.1 Maintain the temperature of the molding room and dry materials at no less than 20°C (68°F) and no more than 27.5°C (81.5°F). Maintain the temperature of the mixing water, and of the moist closet or moist room, at 23.0 ± 1.7°C (73.4 ± 3.0°F).

6.2 The relative humidity of the molding room shall be not less than 50 percent.

6.3 Maintain the storage oven in which the specimens are stored in the containers at a temperature of 80.0° ± 1.7°C (176 ± 3°F).

7. Sampling and Preparation of Test Specimens

7.1 Selection of Aggregate - Process materials proposed for use as fine aggregate in concrete as described in 7.2 with a minimum of crushing. Process materials proposed for use as coarse aggregate in concrete by crushing to produce as nearly as practical a graded product from which a sample can be obtained. The sample shall have the grading as prescribed in 7.2 and be representative of the composition of the coarse aggregate as proposed for use.

7.1.1 When a given quarried material is proposed for use both as coarse and as fine aggregate, test the material only by selection of an appropriate sample crushed to the fine aggregate sizes, unless there is reason to expect that the coarser size fractions have a different composition than the finer sizes and that these differences might significantly affect expansion due to reaction with the alkalis in cement. In this case the coarser size fractions shall be tested in a manner similar to that employed in testing the fine aggregate sizes.

7.2 Preparation of Aggregate - Grade all aggregates in accordance with the requirements prescribed in Table 1. Crush aggregates for which sufficient quantities of the sizes specified in Table 1 do not exist until the required material has been produced. In the case of aggregates containing insufficient amounts of one or more of the larger sizes listed in Table 1, and if no larger material is available for crushing, the first size in which sufficient material is available shall contain the cumulative percentage of material down to that size as determined from the grading specified in Table 1. Note in the test report when such procedures are required. After the aggregate has been separated into the various sieve sizes, wash each size with a water spray over the sieve to remove adhering dust and fine particles from the aggregate. Dry the portions retained on the various sieves and, unless used immediately, store each portion individually in a clean container. Seal the container to prevent moisture loss or gain.

Passing Sieve Size	Retained On Sieve Size	Mass Percent
4.75 mm (No. 4)	2.36 mm (No. 8)	10
2.36 mm (No. 8)	1.18 mm (No. 16)	25
1.18 mm (No. 16)	600 μm (No. 30)	25
600 μm (No. 30)	300 μm (No. 50)	25
300 μm (No. 50)	150 μm (No. 100)	15

7.3 Selection and Preparation of Cement - Select a reference cement which meets the requirements of M85. Pass the reference cement through a 850-μm (No.20) sieve to remove lumps before use.

- 7.4.1 Prepare at least three test specimens for each cement-aggregate combination.
- 7.4.2 Prepare the specimen molds in accordance with the requirements of M210 except, cover the interior surface of the mold with a release agent.
- Note 4 – A release agent is acceptable if it serves as a parting agent without affecting the setting of the cement and without leaving any residue that will inhibit the penetration of water into the specimen. TFE-fluorocarbon tape complies with the requirements for a mold release agent.
- 7.4.3 Proportion the dry materials for the test mortar using 1 part of cement to 2.25 parts of graded aggregate mass. Mix 440 g of cement and 990 g of dry aggregate (made up by recombining the portions retained on various sieves in the grading prescribed in Table 1) at one time to prepare a batch of mortar sufficient for making three specimens. Use a water to cement ratio equal to 0.50 by mass.
- 7.4.4 Mix the mortar in accordance with the requirements of Practice T162 using water that meets the requirements of Section 5.2.
- 7.4.5 Mold test specimens with a total elapsed time of not more than 135 s after completion of the original mixing of the mortar batch. Fill the molds with two approximately equal layers, each layer being compacted with a tamper. Work the mortar into the corners, around the gage studs, and along the surfaces of the mold with a tamper until a homogeneous specimen is obtained. After the top layer has been compacted, cut off the mortar flush with the top of the mold and smooth the surface with a few strokes of the trowel.

8. Procedure

- 8.1 Place each mold in the moist cabinet or moist room immediately after it has been filled. Cure the specimens in the molds for 24 ± 2 h. Remove the specimens from the molds and, while they are being protected from loss of moisture, properly identify and determine the initial length of each specimen using the length comparator. Record all length measurements to the nearest 0.002 mm (0.0001 in).
- 8.2 Place the specimens of each aggregate sample in a storage container with sufficient tap water to totally immerse them. Seal the containers and place them in an oven maintained at $80.0 \pm 1.7^\circ\text{C}$ ($176 \pm 3^\circ\text{F}$) for a period of 2 h.
- 8.3 Measure the comparator bar prior to measuring each set of specimens since the heat from the mortar bars may cause the length of the comparator to change. If the length of the comparator bar has changed, wait until the comparator has returned to room temperature before remeasuring the comparator bar. Remove the containers from the oven one at a time. Remove other containers only after the specimens in the first container have been measured and returned to the oven. Remove the specimens one at a time from the water and dry their surface with a towel paying particular attention to the two metal gage measuring studs. Record the zero measurements of each specimen immediately after drying, and as soon as the specimen is in position. Complete the process of drying and measuring within 15 ± 5 s of removing the specimen from the water. After measurement, leave the specimen on a towel until the remainder of the bars have been measured. Place each set of specimens in separate containers with the 1 normal NaOH solution, at $80.0 \pm 1.7^\circ\text{C}$ ($176 \pm 3^\circ\text{F}$) to totally immerse the samples. Seal the containers and return them to the oven.
- 8.4 Undertake subsequent measurement of the specimens periodically, with at least three intermediate readings, for 14 days after the zero reading, at approximately the same time each day. The measuring procedure is identical to that described in Section 8.3 except that the specimens are returned to their own container after measurement.

9.1 Calculate the difference between the zero length of the specimen and the length at each period of measurement to the nearest 0.001 percent of the effective gage length and record as the expansion of the specimen for that period. Report the average expansion of the three specimens of a given cement-aggregate combination to the nearest 0.01 percent as the expansion for the combination for a given period.

Note 5 - When the mean expansion of the test specimens exceeds 0.10 percent at 16 days from casting (14 days from zero reading), it is indicative of potentially deleterious expansion. This value has been determined from tests of aggregate with known field performance in concrete. When the mean expansion of the test specimens is less than 0.10 percent at 16 days after casting, it is indicative of innocuous behavior.[4]

10. Report - The report shall include the following:

10.1 the type and source of aggregate,

10.2 the type and source of portland cement,

10.3 the alkali content of cement as percent potassium oxide (K_2O), sodium oxide (Na_2O), and calculated sodium oxide (Na_2O) equivalent,

10.4 the average length change in percent at each reading of the specimens,

10.5 any relevant information concerning the preparation of aggregates, including the grading of the aggregate when it differs from that given in 7.2,

10.6 any significant features revealed by examination of the specimens during and after test,

10.7 the amount of mixing water expressed as mass percent to cement,

10.8 the type, source, proportions and chemical analyses, including Na_2O and K_2O , of any pozzolans employed in the tests, and,

10.9 a graph of the length change data from the time of the zero reading to the end of the 16 day period.

11. Precision and Bias

11.1 Precision - Preliminary data indicate that for materials giving an expansion at 14 days in solution of greater than 0.10 percent, the average multi-laboratory coefficient of variation (for experienced laboratories) is 9.55 percent. Therefore, results of two properly conducted tests in different laboratories on the same material should not differ from each other by more than 27.0 percent of their average nineteen times in twenty.

Note: for further information see:

Hooton, R.D., "Interlaboratory Study of the NBRI Rapid Test Method and CSA Standardization Status" in Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity, Ministry of Transportation, Ontario Engineering Materials Report 92, March 1990, pp. 225-240.

11.2 Bias - Since there is no accepted reference material for determining the bias of this test method, no statement on bias is being made.

REFERENCES

- [1] Obserholster, R. E., and Davis, G., "An Accelerated Method for Testing the Potential Alkali Reaction of Siliceous Aggregates," *Cement and Concrete Research*, Vol. 16, 1986, pp. 181-189.
- [2] Davis, G., and Obserholster, R. E., "Use of the NBRI Accelerated test to Evaluate the Effectiveness of Mineral Admixtures in Preventing the Alkali Silica Reaction," *Cement and Concrete Research*, Vol. 17, 1987, pp. 97-107.
- [3] Davis G., and Obserholster, R. E., "An Interlaboratory Programme on the NBRI Accelerated Test to Determine the Alkali-Silica Reactivity of Aggregates," National Building Research Institute, C-1, Special Report BOU 92-1987, Pretoria, RSA, 1987, 16pp.
- [4] Hooton, R. D. and Rogers, C. A., "Evaluation of Rapid Test Methods for Detecting Alkali-Silica Reaction in Aggregates", *Proceedings of the Eighth International Conference on Alkali-Aggregate Reaction*, Vol. 1, 1989, pp. 439-444.

Standard Method of Test
for

Mechanical Mixing of Hydraulic Cement Pastes
and Mortars of Plastic Consistency

AASHTO DESIGNATION: T 162-93
(ASTM DESIGNATION: C 305-91)

1. SCOPE

1.1 This method covers the mechanical mixing of hydraulic cement pastes and mortars of plastic consistency.

1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems, 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.

2. SIGNIFICANCE AND USE

2.1 This method is intended for use in the mechanical mixing of pastes and mortars for the testing of hydraulic cements.

3. APPARATUS

3.1 *Mixer*—The mixer shall be an electrically driven mechanical mixer of the epicyclic type, which imparts both a planetary and a revolving motion to the mixer paddle. The mixer shall have a minimum of two speeds, controlled by definite mechanical means. (Rheostat adjustment of speed will not be acceptable.) The first, or slow speed shall revolve the paddle at a rate of 140 ± 5 rpm, with a planetary motion of approximately 62 rpm. The second speed shall revolve the paddle at a rate of 285 ± 10 rpm, with a planetary motion of approximately 125 rpm. The electric motor shall be at least 124 W ($1/3$ hp).¹ The mixer shall be equipped with the clearance adjustment

bracket as shown in Figure 1A or 1B (NOTE 1), which shall be used to maintain the clearance between the lower end of the paddle and the bottom of the bowl not greater than 2.5 mm but not less than 0.8 mm (the approximate diameter of a grain of 20–30 Ottawa sand) when the bowl is in the mixing position.

NOTE 1—When the bracket is in the proper position beneath the motor housing, the lugs are to the front and facing upward and the heads of the adjustment screws are to the rear and facing downward in the path of the sliding frame that holds the bowl. It is intended that the bracket be fastened at the front housing connection by inserting replacement screws of an appropriate size upward through the opening in each lug and into the existing threaded holes in the bottom of the motor housing. The original stops for the sliding frame are to be filed down if they prevent the frame from coming in contact with the adjustment screws.

3.2 *Paddle*—The paddle shall be readily removable, made of stainless steel, and shall conform to the basic design shown in Figure 2. The dimensions of the paddle shall be such that when in the mixing position the paddle outline conforms to the contour of the bowl used with the mixer, and the clearance between corresponding points on the edge of the paddle and the side of the bowl in the position of closest approach shall be approximately 4.0 mm but not less than 0.8 mm.

3.3 *Mixing Bowl*—The removable mixing bowl shall have a nominal capacity of 4.73 L, shall be of the general shape and comply with the limiting dimensions shown in Figure 3, and shall be made of stainless steel. The bowl shall be so equipped that it will be positively held in the mixing apparatus in a fixed position during the mixing procedure. There shall

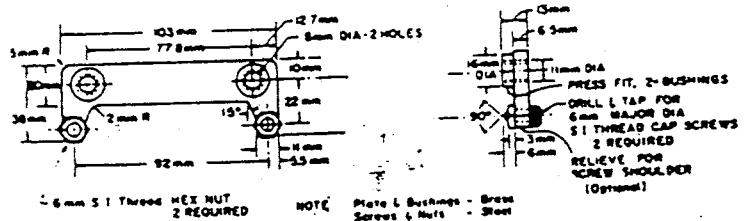


FIGURE 1A Clearance Adjustment Bracket

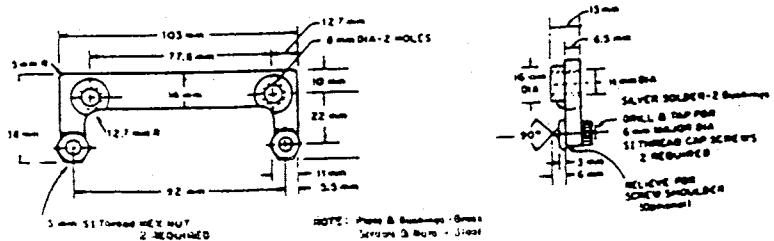


FIGURE 1B Clearance Adjustment Bracket

¹ The Model N-50 Mixer (less the clearance adjustment bracket), manufactured by the Hobart Corp., Troy, Ohio, is considered to conform to these requirements.

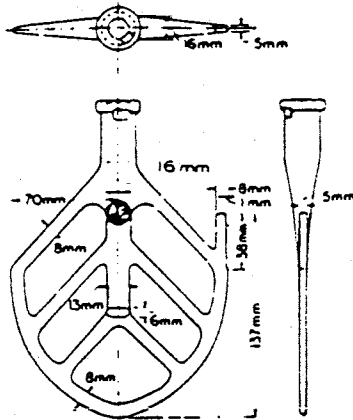


FIGURE 2 Paddle

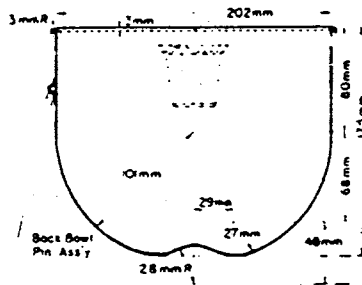
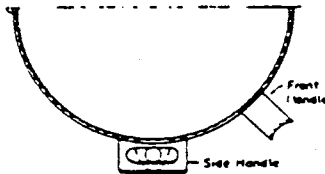


FIGURE 3 Mixing Bowl

be provided a lid, made of a nonabsorbing material not attacked by cement.

3.4 *Scraper*—The scraper shall consist of a semirigid rubber blade attached to a handle about 150 mm long. The blade shall be about 75 mm long, 50 mm wide, and tapered to a thin edge about 2 mm thick.

NOTE 2—A kitchen tool known as a plate and bowl scraper conforms to these requirements.

3.5 *Supplementary Apparatus*—The balances, weights, glass graduates, and any other supplementary apparatus used in measuring and preparing the mortar materials prior to mixing shall conform to the respective requirements for such apparatus as specified in the method for the particular test for which the mortar is being prepared.

4. TEMPERATURE AND HUMIDITY

4.1 The temperature of the room shall be maintained between 20 and 27.5°C (68 and 81.5°F), and the temperature of the dry materials, paddle, and bowl shall be within the above range at the time of test. The temperature of the mixing water shall not vary from 23°C (73.4°F) by more than $\pm 1.7^\circ\text{C}$ (3°F).

4.2 The relative humidity of the laboratory shall not be less than 50 percent.

5. MATERIALS, PROPORTIONING, CONSISTENCY

5.1 The materials and their proportions and quantities shall conform to the requirements contained in the particular method for which the paste or mortar is being prepared.

6. PROCEDURE FOR MIXING PASTES

6.1 Place the dry paddle and the dry bowl in the mixing position in the mixer. Then introduce the materials for a batch into the bowl and mix in the following manner:

6.1.1 Place all the mixing water in the bowl.

6.1.2 Add the cement to the water and allow 30 seconds for the absorption of the water.

6.1.3 Start the mixer and mix at slow speed (140 \pm 5 rpm) for 30 seconds.

6.1.4 Stop the mixer for 15 seconds and during this time scrape down into the batch any paste that may have collected on the side of the bowl.

6.1.5 Start the mixer at medium speed (235 \pm 10 rpm) and mix for 1 minute.

7. PROCEDURE FOR MIXING MORTARS

7.1 Place the dry paddle and the dry bowl in the mixing position in the mixer. Then introduce the materials for a batch into the bowl and mix in the following manner:

7.1.1 Place all the mixing water in the bowl.

7.1.2 Add the cement to the water; then start the mixer and mix at the slow speed (140 \pm 5 rpm) for 30 seconds.

7.1.3 Add the entire quantity of sand slowly over a 30-second period, while mixing at slow speed.

7.1.4 Stop the mixer, change to medium speed (285 \pm 10 rpm), and mix for 30 seconds.

7.1.5 Stop the mixer and let the mortar stand for 1½ minutes. During the first 15 seconds of this interval, quickly scrape down into the batch any mortar that may have collected on the side of the bowl; then for the remainder of this interval, cover the bowl with the lid.

7.1.6 Finish by mixing for 1 minute at medium speed (285 \pm 10 rpm).

7.1.7 In any case requiring a remixing interval, any mortar adhering to the side of the bowl shall be quickly scraped down into the batch with the scraper prior to remixing.

NOTE 3—Caution—The clearances between paddle and bowl specified in this method are suitable when using the standard mortar made with Ottawa sand. To permit the mixer to operate freely and to avoid serious damage to the paddle and bowl when coarser aggregates are used, it may be necessary to set the clearance adjustment bracket to provide greater clearances than those specified in Section 3.1.

*Standard Specification
for*

**Apparatus for Use in Measurement of Length Change of
Hardened Cement Paste, Mortar, and Concrete**

AASHTO DESIGNATION: M 210-92
(ASTM DESIGNATION: ASTM C 490-89)

1. SCOPE

1.1 This practice covers the requirements for the apparatus and equipment used to prepare specimens for the determination of length change in hardened cement paste, mortar, and concrete, the apparatus and equipment used for the determination of these length changes, and the procedures for its use.

1.2 Methods for the preparation and curing of test specimens, conditions of testing and curing, and detailed procedures for calculating and reporting test results are contained in applicable test methods.

1.3 The values stated in inch-pound units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

- 2.1 *AASHTO Standards:*
M 201 Specification for
Moist Cabinets,
Moist Rooms, and
Water Storage Tanks
Used in the Testing of
Hydraulic Cements
and Concretes
- 2.1 *ASTM Standards:*
C 1005 Specification for
Weights and
Weighing Devices for
Use in the Physical
Testing of Hydraulic
Cement

3. TERMINOLOGY

3.1 *Length Change*—an increase or decrease in the linear dimension of a test specimen, measured along the longitudinal axis, due to causes other than applied load.

4. SIGNIFICANCE AND USE

4.1 This practice is intended to provide standard requirements for apparatus common to many test methods used in connection with cement and concrete and standardized procedures for its use. The detailed requirements as to materials, mixtures, specimens, conditioning of specimens, number of specimens, ages at which measurements are to be made, interpretation of results, and precision and bias are left to be dealt with in specific test methods.

5. APPARATUS

5.1 *Weight and Weighing Devices*, shall conform to the requirements of ASTM C 1005.

5.2 *Glass Graduates*, of suitable capacities (large enough to measure the mixing water for paste and mortar mixtures in a single operation) shall be made to deliver the indicated volume at 20°C (68°F).

5.2.1 The permissible variation for graduates of 100 to 150-mL capacities shall be ± 1.0 mL, for graduates of 200 to 300-mL capacities shall be ± 2.0 mL, and for all larger graduates shall be ± 0.5 percent of the rated capacity.

5.2.2 These graduates shall be subdivided to at least 5 mL with the following exceptions:

5.2.2.1 The graduation lines may be omitted for the lowest 15 mL for a 150-mL graduate.

5.2.2.2 The graduation lines may be omitted for the lowest 25 mL for a 250-mL graduate.

5.2.2.3 The graduation lines may be omitted for the lowest 50 mL for a 500-mL graduate.

5.2.3 The main graduation lines shall extend at least three quarters of the way

around the graduate and shall be numbered.

5.3 *Molds*, shall have either one or two compartments and shall be constructed as shown in Figures 1 or 2. Molds for test specimens used in determining the length change of cement pastes and mortars shall provide for 1 by 1 by 11 $\frac{1}{4}$ -in. prisms having a 10-in. gage length, or for 25 by 25 by 285-mm prisms having a 250-mm gage length. Molds for test specimens used in the length change of concretes shall provide for prisms of the desired cross section having a 10-in. or 250-mm gage length. In some routine tests, 1 by 1 by 6 $\frac{1}{4}$ -in. specimens with a gage length of 5 in. or 25 by 25 by 106-mm specimens with a gage length of 125 mm are permitted, but in case of dispute, results obtained with specimens of 10-in. (250-mm) gage length shall govern.

5.3.1 The gage length shall be considered as the nominal length between the innermost ends of the gage studs. The parts of the molds shall be tight fitting and firmly held together when assembled, and their surfaces shall be smooth and free of pits. The molds shall be made of steel or other hard metal not readily attacked by the cement paste, mortar, or concrete. The sides of the molds shall be sufficiently rigid to prevent spreading or warping. For the molds shown in Figure 1, the tolerance on dimension A is ± 0.03 in. For the molds shown in Figure 2, the tolerance on dimension A is ± 0.7 mm.

5.3.2 Each end plate of the mold shall be equipped to hold properly in place, during the setting period, one of the gage studs shown in Figures 1 or 2. The gage studs shall be of American Iron and Steel Institute (AISI)¹ Type 316

¹ Details on this material are available from the American Iron and Steel Institute, 1133 15th St. N.W., Washington, DC 20005.

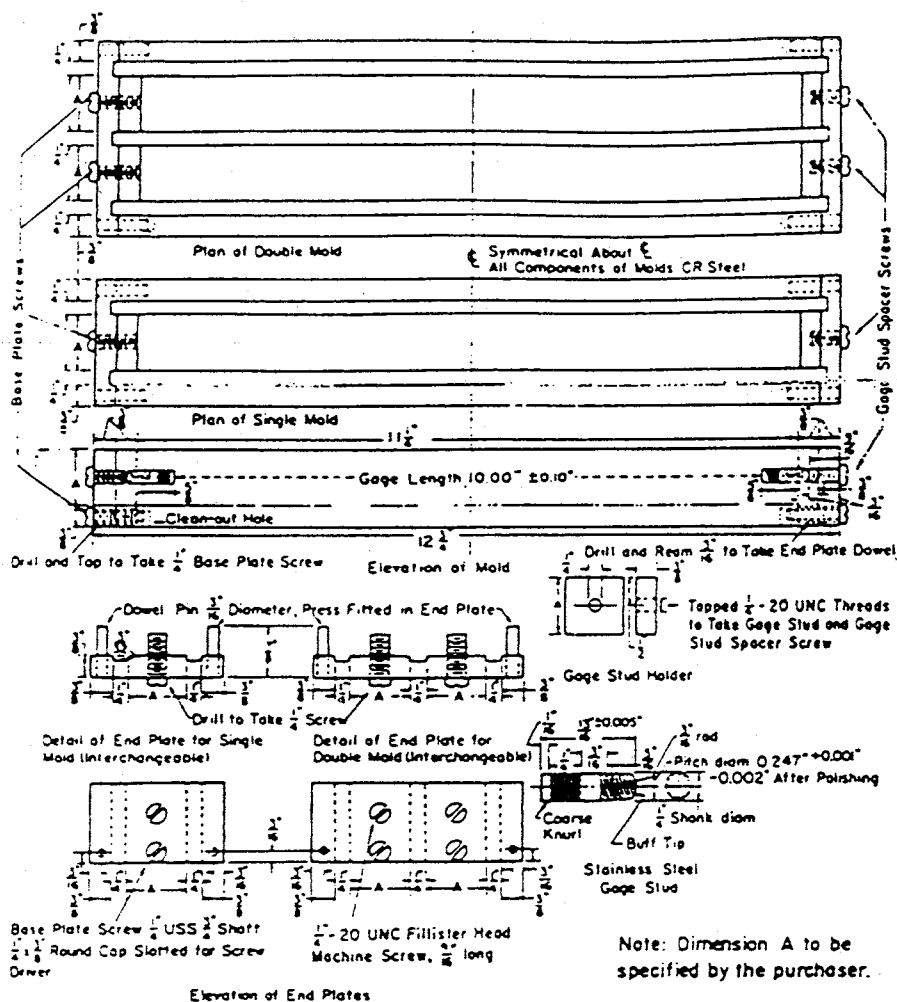


FIGURE 1 Molds (Inch-Pound Units)

stainless steel or other corrosion-resistant metal of similar hardness. Gage studs of Invar or similar metal shall be used when specimens are tested at widely different temperatures. To prevent restraint of the gage studs before demolding of the specimen, the device for holding the gage studs in position shall be so arranged that, if necessary, it can be partially or completely released after the compaction of the paste or mortar into place in the mold. The gage studs shall be set so that their principal axes coincide with the principal axis of the test specimen. For the molds shown in Figure 1, gage studs shall extend into the specimen $0.625 \pm$

0.025 in. and the distance between the inner ends of the gage studs shall be 10.00 ± 0.10 in.; and 10 in. shall be considered the gage length for calculating length change. For the molds shown in Figure 2, gage studs shall extend into the specimen 17.5 ± 0.5 mm and the distance between the inner ends of the gage studs shall be 250.0 ± 2.5 mm and 250 mm shall be considered the gage length for calculating length change.

5.4 *Length Comparator*, for determining length change of specimens, shall be designed to accommodate the size of specimen employed and to provide or permit a positive means of con-

tact with the gage studs and the convenient and rapid obtaining of comparator readings (Note 1).

5.4.1 The comparator for determining length changes of specimens produced in the molds shown in Figure 1 shall provide a dial micrometer of other measuring device graduated to read in 0.0001-in. units, accurate within 0.0001 in. in any 0.0010-in. range, and within 0.0002 in. in any 0.0100-in. range, and sufficient range (at least 0.3 in.) in the measuring device to allow for small variations in the actual length of various specimens. The terminals of the comparator shall be plane, polished and heat-

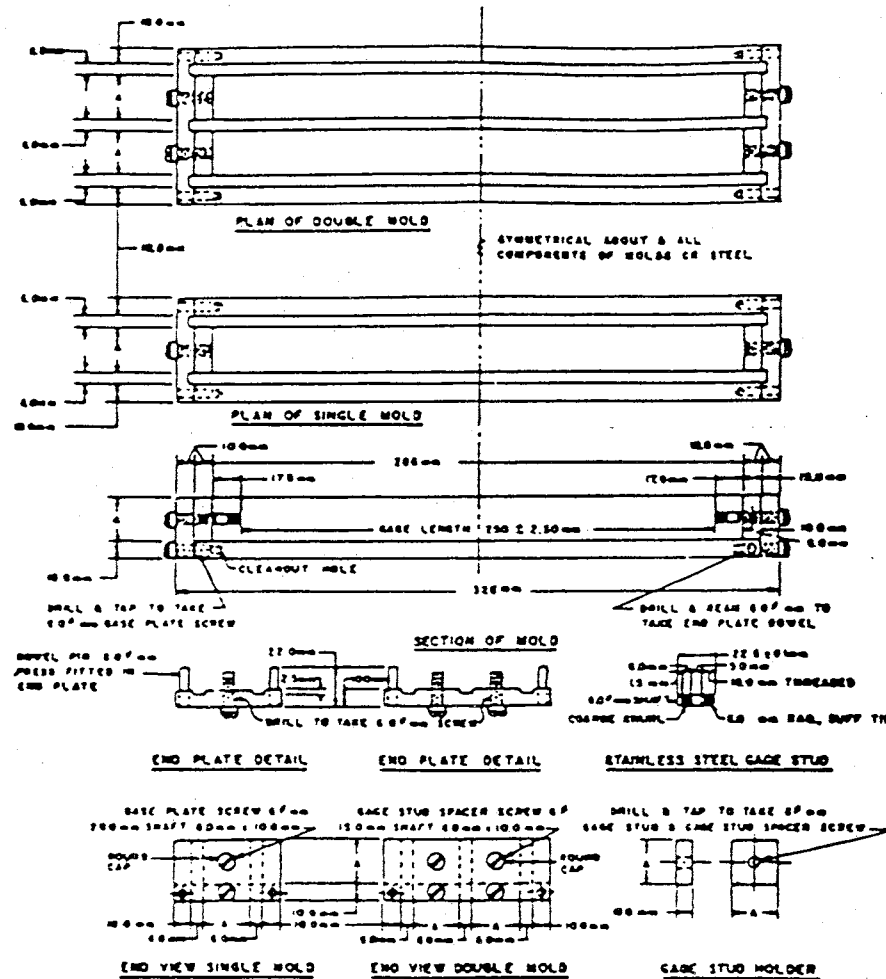


FIGURE 2 Molds (SI Units)

treated. They shall be fitted with collars held in place with set screws. The collars shall extend 0.062 ± 0.003 in. beyond the plane face of the terminal and have an inside diameter 0.02 in. greater than the average diameter of the portion of the gage studs that must fit into the collars.

5.4.2 The comparator for determining length changes of specimens produced in the molds shown in Figure 2 shall provide a dial micrometer or other measuring device graduated to read in 0.001 or 0.002-mm units, accurate within 0.002 mm in any 0.020-mm range, and sufficient range (at least 3.0 mm) in the measuring device to allow for small variations in the actual length of various specimens. The terminals of the compa-

razor shall be plane, polished and heat-treated. They shall be fitted with collars held in place with set screws. The collars shall extend 1.5 ± 0.1 mm beyond the plane face of the terminal and have an inside diameter 0.5 mm greater than the average diameter of the gage studs that must fit into the collars.

5.4.3 The design shall provide a means for checking the measuring device against a reference bar at regular intervals. The reference bar shall have an overall length of $11\frac{1}{8} \pm \frac{1}{16}$ in. or $6\frac{1}{8} \pm \frac{1}{16}$ in. (300 ± 1.5 mm or 170 ± 1.5 mm), whichever is appropriate for the specimen in use. The bar shall be of a steel alloy having a coefficient of thermal expansion not greater than two millionths

per degree Celsius. Each end shall be machined to the same shape as the contact end of a gage stud, and shall be heat treated, hardened, and then polished. The central 4 in. (100 mm) of the length of the reference bar shall be covered by a rubber tube with a wall at least $\frac{1}{8}$ in. (3 mm) thick to minimize the effect of temperature change during handling. The reference bar shall be provided near one end with a positioning mark.

NOTE 1—One type of instruments that has been found satisfactory for use with small crystals is shown in Figure 3. A horizontal comparator should be used with prisms with a cross section greater than 9 in.² or 58 cm².

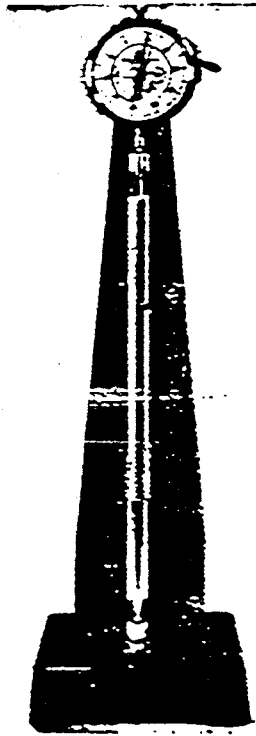


FIGURE 3 Type of Suitable Apparatus for Measurement of Length Changes

6. PROCEDURE

6.1 *Preparation of Molds*—Prior to the molding of specimens, if necessary, seal the outside joints of the mold and the contact lines of the molds and base plates with a microcrystalline wax. Thinly cover the interior surfaces of the mold with mineral oil. After this operation, set the gage studs, taking care to keep them clean, and free of oil, grease, and foreign matter.

6.2 *Use of Reference Bar*—Place the reference bar in the instrument in the same position each time a comparator reading is taken. Check the dial gage setting of the measuring device by use of the reference bar at least at the begin-

ning and end of the readings made within a half day when the apparatus is kept in a room maintained at constant temperature. Check it more often when kept in a room where the temperature is not constant.

6.3 *Obtaining Comparator Readings*—Rotate specimens slowly in the measuring instrument while the comparator reading is being taken. Record the minimum reading of the dial if the rotation causes a change in the dial reading. Place specimens in the instrument with the same end up each time a comparator reading is taken.

6.3.1 *Obtaining Comparator Readings of Specimens Stored Moist*—Clean the hole in the base of the comparator into which the gage stud on the lower end of the bar fits (this hold tends to collect water and sand and should be cleaned after every reading). Read and record the comparator indication of the length of the reference bar. Take one bar out of immersion, blot the pins, put the bar in the comparator, read, and record the indication. Return the bar to immersion and clean the hole in the base of the comparator. Take out the second bar and treat it in a like manner. Return the second bar to immersion, record the reading, and clean the hole in the base of the comparator. Continue the procedure until all bars have been read, returned to immersion, and the readings recorded, cleaning the hole in the bottom of the comparator each time. After reading the last bar, clean the hole in the comparator base and read and record the reference-bar indication. Blot only around the pins (Note 2).

NOTE 2—The purpose of the minimal blotting of the pins and no blotting of the bars is to avoid drying and shrinkage of the bars. It has been observed that if the pins are blotted, and the bar placed in the comparator and the dial read, and the bar is then wiped gently with a dry cloth, the bar will shrink measurably. Therefore, drying should be minimized.

7. CALCULATION OF LENGTH CHANGE

7.1 Calculate the length change at any age as follows:

$$L = \frac{(L_1 - L_0)}{G} \times 100$$

where:

- L = change in length at x age, percent,
- L_1 = comparator reading of specimen at x age minus comparator reading of reference bar at x age; in inches when using Figure 1 apparatus, in millimeters when using Figure 2 apparatus,
- L_0 = initial comparator reading of specimen minus comparator reading of reference bar at that same time; in inches when using Figure 1 apparatus, in millimeters when using Figure 2 apparatus, and
- G = nominal gage length, 10 when using Figure 1 apparatus, 250 when using Figure 2 apparatus.

7.2 Calculate length change values for each specimen to the nearest 0.001 percent and report averages to the nearest 0.01 percent.

8. TEMPERATURE, HUMIDITY, AND TIME

8.1 *Molding Room*—The temperature of the molding room and dry materials shall be maintained between 20 and 27.5°C (68 and 81.5°F). The relative humidity shall be not less than 50 percent. The temperature of the mixing water shall not vary from 23°C (73.4°F) by more than $\pm 1.7^\circ\text{C}$ (3°F).

8.2 *Moist Storage Facility*—The temperature and humidity of the air in the moist storage facility shall conform to the requirements of AASHTO M 201.

8.3 *Time*—Comparator readings shall be taken at specified time intervals or ages. All intervals and ages shall be met within ± 2 percent.

APPENDIX D

ASTM C295-90

Standard Guide for Petrographic Examination of Aggregate for Concrete



Standard Guide for Petrographic Examination of Aggregates for Concrete¹

This standard is issued under the fixed designation C 295; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This guide² outlines procedures for the petrographic examination of samples representative of materials proposed for use as aggregates in concrete.

1.2 This guide outlines the extent to which petrographic techniques should be used, the selection of properties that should be looked for, and the manner in which such techniques may be employed in the examination of samples of aggregates for concrete.

1.3 This guide does not attempt to describe the techniques of petrographic work since it is assumed that the guide will be used by persons who are qualified by education and experience to employ such techniques for the recognition of the characteristic properties of rocks and minerals and to describe and classify the constituents of an aggregate sample.

1.4 The rock and mineral names given in Descriptive Nomenclature C 294 should be used insofar as they are appropriate in reports prepared according to this guide.

1.5 *This standard does not purport to address the safety problems 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.*

1.6 The values stated in SI units are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

- C 33 Specification for Concrete Aggregates³
- C 117 Test Method for Materials Finer than 75- μ m (No. 200) Sieve in Mineral Aggregates by Washing³
- C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates³
- C 294 Descriptive Nomenclature of Constituents of Natural Mineral Aggregates³
- C 702 Practice for Reducing Field Samples of Aggregate to Testing Size³
- D 75 Practice for Sampling Aggregates⁴

¹ This guide is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.65 on Petrography of Concrete and Aggregates.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as C 295 - 54. Last previous edition C 295 - 85.

² This guide is based on the "Method of Petrographic Examination of Aggregates for Concrete," by Katharine Mather and Bryant Mather, *Proceedings*, ASTM, ASTEA, Vol. 50, 1950, pp. 1288-1312.

³ *Annual Book of ASTM Standards*, Vol. 04.02.

⁴ *Annual Book of ASTM Standards*, Vol. 04.03.

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵

E 883 Guide for Metallographic Photomicrography⁶

3. Summary of Method

3.1 The specific procedures employed in the petrographic examination of any sample will depend to a large extent on the purpose of the examination and the nature of the sample. In most cases the examination will require the use of optical microscopy. Complete petrographic examinations for particular purposes and to investigate particular problems may require examination of aggregates or of selected constituents by means of additional procedures, such as X-ray diffraction analysis, differential thermal analysis, infrared spectroscopy, or others; in some instances, such procedures are more rapid and more definitive than are microscopical methods.

3.2 Identification of the constituents of a sample is usually a necessary step towards recognition of the properties that may be expected to influence the behavior of the material in its intended use, but identification is not an end in itself. The value of any petrographic examination will depend to a large extent on the representativeness of the samples examined, the completeness and accuracy of the information provided to the petrographer concerning the source and proposed use of the material, and the petrographer's ability to correlate these data with the findings of the examination.

3.3 It is assumed that the examination will be made by persons qualified by education and experience to operate the equipment used and to record and interpret the results obtained. In some cases, the petrographer will have had experience adequate to provide detailed interpretation of the materials' performance with respect to engineering and other consequences of the observations. In others, the interpretation will be made in part by engineers, scientists, or others qualified to relate the observations to the questions to be answered.

4. Significance and Use

4.1 Petrographic examinations are made for the following purposes:

4.1.1 To determine the physical and chemical characteristics of the material that may be observed by petrographic methods and that have a bearing on the performance of the material in its intended use.

⁵ *Annual Book of ASTM Standards*, Vol. 14.02.

⁶ *Annual Book of ASTM Standards*, Vol. 03.01.

4.1.2 To describe and classify the constituents of the sample.

4.1.3 To determine the relative amounts of the constituents of the sample, which are essential for proper evaluation of the sample, when the constituents differ significantly in properties that have a bearing on the performance of the material in its intended use, and

4.1.4 To compare samples of aggregate from new sources with samples of aggregate from one or more sources, for which test data or performance records are available.

4.2 This guide may be used by a petrographer employed directly by those for whom the examination is made. The employer should tell the petrographer, in as much detail as necessary, the purposes and objectives of the examination, the kind of information needed, and the extent of examination desired. Pertinent background information, including results of prior testing, should be made available. The petrographer's advice and judgment should be sought regarding the extent of the examination.

4.3 This guide may form the basis for establishing arrangements between a purchaser of consulting petrographic service and the petrographer. In such a case, the purchaser and the consultant should together determine the kind, extent, and objectives of the examination and analyses to be made, and should 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 or other conditions.

4.4 Petrographic examination of aggregate considered for use in hydraulic cement concrete is one aspect of the evaluation of aggregate, but petrographic examination is also used for many other purposes. Petrographic examinations provide identification of types and varieties of rocks present in potential aggregates. However, as noted above, identification of every rock and mineral present in an aggregate source is not required.

4.5 The petrographic examination should establish whether the aggregate contains chemically unstable minerals such as soluble sulfates, unstable sulfides that may form sulfuric acid or create distress in concrete exposed to high temperatures during service, or volumetrically unstable materials such as smectites (also known as the montmorillonite-saponite group of minerals or swelling clays). Specifications may limit the quartz content of aggregates for use in concrete that may be subject to high temperature (purposefully or accidentally) because of the conversion to beta-quartz at 573°C (1063°F), with accompanying volume increase.

4.6 Petrographic examination should identify the portion of each coarse aggregate that is composed of weathered or otherwise altered particles and the extent of that weathering or alteration, whether it is severe, moderate, or slight, and should determine the proportion of each rock type in each condition. If the concrete in which the aggregate may be used will be exposed to freezing and thawing in a critically saturated condition, finely porous and highly weathered or otherwise altered rocks should be discriminated because they will be especially susceptible to damage by freezing and thawing and will cause the aggregate portion of the concrete to fail in freezing and thawing. This will ultimately destroy the concrete because such aggregates cannot be protected by adequately air-entrained sound mortar. Finely porous aggregate

near the concrete surface are also likely to form popouts, which are blemishes on pavements and walls.

4.7 Petrographic examinations may also be used to determine the proportions of cubic, spherical, ellipsoidal, pyramidal, tabular, flat, and elongated particles in an aggregate sample or samples. Flat, elongated, and thin chip-like particles in aggregate increase the mixing water requirement and decrease concrete strength.

4.8 Petrographic examination should identify and call attention to potentially alkali-silica reactive and alkali-carbonate reactive constituents, determine such constituents quantitatively, and recommend additional tests to confirm or refute the presence in significant amounts of aggregate constituents capable of alkali reaction in concrete. This guide is referred to in the Appendix of Specification C 33. Alkali-silica reactive constituents found in aggregates include: opal, cristobalite, tridymite, siliceous and some intermediate volcanic glass, chert, glassy to cryptocrystalline acid volcanic rocks, synthetic siliceous glasses, some argillites, phyllites, metamorphic graywackes, rocks containing highly metamorphic quartz such as graywackes, phyllites, schists, gneisses, gneissic granites, vein quartz, quartzite, and sandstone. Criteria are available in the mineralogic literature for identifying the minerals in the list above by optical properties or X-ray diffraction, or both, and in petrographic and petrologic literature for identifying the rocks in the list by mineral composition and texture in thin section, sometimes assisted by X-ray diffraction for mineral composition. Potentially deleterious alkali-carbonate reactive rocks are usually calcareous dolomites or dolomitic limestones with clayey insoluble residues. Some dolomites essentially free of clay and some very fine-grained limestones free of clay and with minor insoluble residue, mostly quartz, are also capable of some alkali-carbonate reactions, however, such reactions have not yet been found deleterious.

4.9 Petrographic examination may be directed specifically at the possible presence of contaminants in aggregates, such as synthetic glass, cinders, clinker, or coal ash, magnesium oxide, calcium oxide, or both, soil, hydrocarbons, chemicals that may affect the setting behavior of concrete or the properties of the aggregate, animal excrement, plants or rotten vegetation, and any other contaminant that may prove undesirable in concrete.

4.10 These objectives for which this guide was prepared will have been attained if those involved with the evaluation of aggregate materials for use in concrete construction have reasonable assurance that the petrographic examination results wherever and whenever obtained, as corrected, may confidently be compared.

5. Apparatus and Supplies

5.1 The apparatus and supplies listed below comprise a selection that will permit the use of all of the procedures described in this guide. All specific items listed have been used, in connection with the performance of petrographic examinations, by the procedures described herein; it is not, however, intended to imply that other items cannot be substituted to serve similar functions. Whenever possible the selection of particular apparatus and supplies should be left to the judgment of the petrographer who is to perform the work so that the items obtained will be those with the use of

which he has the greatest experience and familiarity. The minimum equipment regarded as essential to the making of petrographic examinations of aggregate samples are those items, or equivalent apparatus or supplies that will serve the same purpose, that are indicated by asterisks in the lists given below.

5.1.1 *Apparatus and Supplies for Preparation of Specimens:*

5.1.1.1 *Rock-Cutting Saw*,* preferably with 350-mm or larger diamond blade, and automatic feed.

5.1.1.2 *Horizontal Grinding Wheel*,* preferably 400 mm in diameter.

5.1.1.3 *Polishing Wheel*, preferably 200 to 300 mm in diameter.

5.1.1.4 *Abrasives*⁷—*Silicon carbide grit No. 100 (122 μm), 220 (63 μm), 320 (31 μm), 600 (16 μm), and 800 (12 μm); alumina M-305 (5 μm).

5.1.1.5 *Geologist's Pick or Hammer*.

5.1.1.6 *Microscope Slides*,* clear, noncorrosive, 25 by 45 mm in size.

5.1.1.7 *Canada Balsam*,* neutral, in xylene, or suitable low-viscosity epoxies, or Lakeside 70.

5.1.1.8 *Xylene*,*

5.1.1.9 *Mounting Medium*,* suitable for mounting rock slices for thin sections.

5.1.1.10 *Laboratory Oven*,*

5.1.1.11 *Plate-Glass Squares*,* about 300 mm on an edge for thin-section grinding.

5.1.1.12 *Sample Splitter with pans*,*

5.1.1.13 *Micro Cover Glasses*,* noncorrosive, square, 12 to 18 mm, 25 mm, etc.

5.1.1.14 *Platner Mortar*.

5.1.2 *Apparatus and Supplies for Examination of Specimens:*

5.1.2.1 *Polarizing Microscope*,* with mechanical stage; low-, medium-, high-power objectives, and objective-centering devices; eyepieces of various powers; full- and quarter-wave compensators; quartz wedge.

5.1.2.2 *Microscope Lamps** (preferably including a sodium arc lamp).

5.1.2.3 *Stereoscopic Microscope*,* with objectives and oculars to give final magnifications from about 6 \times to about 150 \times .

5.1.2.4 *Magnet*,* preferably Alnico, or an electromagnet.

5.1.2.5 *Needleholder and Points*,*

5.1.2.6 *Dropping Bottle*, 60-mL capacity.

5.1.2.7 *Petri Culture Dishes*.

5.1.2.8 *Forceps*, smooth, straightpointed.

5.1.2.9 *Lens Paper*,*

5.1.2.10 *Immersion Media*,* $n = 1.410$ to $n = 1.785$ in steps of 0.005.

5.1.2.11 *Counter*.

5.1.2.12 *Photomicrographic Camera* and accessories.

5.2 The items under Apparatus and Supplies include those used to make thin sections. Semiautomatic thin section machines are now available, and there are several thin-section makers who advertise in *Geotimes*, the *American*

Mineralogist, and other mineralogical or geological journals. Laboratories may find it reasonable to buy a thin-section machine or use a commercial thin-section maker. Remotely located laboratories have more need to be able to make their own thin sections.

5.3 It is necessary that facilities be available to the petrographer to check the index of refraction of the immersion media. If accurate identification of materials is to be attempted, as for example the differentiation of quartz and chalcedony, or the differentiation of basic from intermediate volcanic glass, the indices of refraction of the media need to be known with accuracy. Media will not be stable for very long periods of time and are subject to considerable variation due to temperature change. In laboratories not provided with close temperature control, it is often necessary to recalibrate immersion media several times during the course of a single day when accurate identifications are required. The equipment needed for checking immersion media consists of an Abbé refractometer. The refractometer should be equipped with compensating prisms to read indices for sodium light from white light, or it should be used with a sodium arc lamp.

5.4 A laboratory that undertakes any considerable amount of petrographic work should be provided with facilities to make photomicrographic records of such features as cannot adequately be described in words. Photomicrographs can be taken using standard microscope lamps for illumination; however, it is recommended that whenever possible a zirconium arc lamp be provided for this purpose. For illustrations of typical apparatus, reference may be made to the paper by Mather and Mather,² and manufacturers of microscopes equipped with cameras and photomicrographic equipment may be consulted. Much useful guidance regarding photomicrography, especially using reflected light, is found in Guide E 883.

6. Sampling

6.1 Samples for petrographic examination should be taken by or under the direct supervision of a geologist familiar with the requirements for random sampling of aggregates for concrete and in general following the requirements of Practice D 75. The exact location from which the sample was taken, the geology of the site, and other pertinent data should be submitted with the sample. The amount of material actually studied in the petrographic examination will be determined by the nature of the examination to be made and the nature of the material to be examined, as discussed below.

6.1.1 *Undeveloped quarries* should be sampled by means of cores drilled through the entire depth expected to be exploited. Drilling of such cores should be in a direction that is essentially perpendicular to the dominant structural feature of the rock. Massive material may be sampled by "NX" (53-mm (2 $\frac{1}{8}$ -in.) diameter) cores. Thinly bedded or complex material should be represented by cores not less than 100 mm (4 in.) in diameter. There should be an adequate number of cores to cover the limits of the deposit proposed for the project. The entire footage of the recovered core should be included in the sample and accurate data given as to elevations, depths, and core losses.

6.1.2 *Operating quarries and operating sand and gravel*

⁷The values given in micrometres are the approximate average grain size of commercial silicon carbide grit in the designated size classification.

TABLE 1 Minimum Sizes for Samples from Undeveloped Sand and Gravel Deposits

Sieve Size	Quantity		
	kg	(lb)	Pieces
Larger than 150-mm (6-in.)	4
75 to 150-mm (3 to 6-in.)	300 ^a
37.5 to 75-mm (1½ to 3-in.)	180	(400)	...
19.0 to 37.5-mm (¾ to 1½-in.)	90	(200)	...
4.75 to 19.0-mm (No. 4 to ¾-in.)	45	(100)	...
Finer than 4.75-mm (No. 4) ^b	23	(50)	...

^a Not less than one piece from each apparent type of rock.

^b Fine aggregate.

deposits, in which stock piles of the material produced are available, should be represented by not less than 45 kg (100 lb) or 300 pieces, whichever is larger, of each size of material to be examined. Samples from stock piles should be composed of representative portions of larger samples collected with due consideration given to segregation in the piles.

6.1.3 *Exposed faces of nonproducing quarries, where stock piles of processed material are not available, should be represented by not less than 2 kg (4 lb) from each distinctive stratum or bed, with no piece weighing less than 0.5 kg (1 lb), or by a drilled core as described above.*

6.1.4 *Undeveloped sand and gravel deposits should be sampled by means of test pits dug to the anticipated depth of future economic production. Samples should consist of not less than the quantities of material indicated in Table 1, selected so as to be representative of the deposits.*

NATURAL GRAVEL AND SAND

7. Procedure

7.1 *Selection of Samples for Examination*—Samples of gravel and natural sand for petrographic examination should be dry sieved in accordance with Method C 136 to provide samples of each sieve size. In the case of sands an additional portion should then be tested in accordance with Test Method C 117, with the wash water being saved and removed by drying in order to provide a sample of the material passing the 75- μ m (No. 200) sieve. The results of the sieve analysis of each sample made in accordance with Method C 136 should be provided to the petrographer making the examination and used in calculating results of the petrographic examination. Each sieve fraction should be examined separately, starting with the largest size available. Rocks are more easily recognized in larger pieces; the breakdown of a heterogeneous type present in the larger sizes may have provided particles of several apparently different types in the smaller sizes. Some important and easily confused types may be recognizable using the stereoscopic microscope if they are first recognized and separated in the larger sizes, but may require examination using the petrographic microscope if they are first encountered in the smaller sizes.

7.2 The number of particles of each sieve fraction to be examined will be fixed by the required precision of determination of the less abundant constituents. Assuming that the field sampling and laboratory sampling procedures are accurate and reliable, the number of particles examined, identified, and counted in each sieve fraction will depend on the required accuracy of the estimate of constituents present in small quantities. The numbers given in this method are

minimal. They are based on experience and on statistical considerations.³ It is believed that at least 150 particles of each sieve fraction should be identified and counted in order to obtain reliable results. Precise determinations of small quantities of an important constituent will require counts of larger numbers of particles. If the sample of a sieve fraction contains many more particles than need to be identified, the sample shall be reduced in accordance with one of the procedures in Practice C 702, so as to contain a proper number of particles for examination.

8. Procedure for Examination of Natural Gravel

8.1 *Coatings*—The pebbles should be examined to establish whether exterior coatings are present. If they are, it should be determined whether the coatings consist of materials likely to be deleterious in concrete (opal, gypsum, easily soluble salts, organic matter). It should also be determined qualitatively how firmly the coatings are bonded to the pebbles.

8.2 *Rock Types*—The sieve fraction should be sorted into rock types by visual examination. If all or most of the groups present are types easily identifiable in hand specimen by examination of a natural or broken surface, and by scratch and acid tests, no further identification may be needed. Fine-grained rocks that cannot be identified macroscopically or that may consist of or contain constituents known to be deleterious in concrete should be checked by examination with the stereoscopic microscope. If they cannot be identified by that means, they should be examined by means of the petrographic microscope. The amount of work done in identifying fine-grained rocks should be adapted to the information needed about the particular sample. Careful examination of one size of a sample, or study of information from previous examination of samples from the same source, will usually reveal the amount of additional detailed microscopical work required to obtain information adequate for the purpose. In some instances, petrographic methods other than microscopy, such as X-ray diffraction, may be required or might most rapidly serve to identify fine-grained rock materials.

8.3 *Condition*—The separated groups belonging to each rock type should be examined to determine whether a further separation by physical condition is necessary. If all of the particles of a rock type are in a comparable condition, that fact should be noted. More frequently, particles in several degrees of weathering will be found in a group. They should be sorted into categories based on condition and on the expectation of comparable behavior in concrete. The types of categories intended are: (1) fresh, dense; (2) moderately weathered; (3) very weathered; or (1) dense; (2) porous (or porous and friable). It usually is not practicable to recognize more than three conditions per rock type, and one or two may be sufficient. An important constituent present in larger quantities may sometimes require separation into four groups by condition. The conspicuous example is chert

³ Simpson, G. G., and Rowe, A., *Quantitative Zoology*, McGraw-Hill Co., Inc., New York, N.Y., 1939, pp. 132-135.

Dryden, A. L., Jr., "Accuracy in Percentage Representation of Heavy Mineral Frequencies," *Proceedings, U. S. Nat. Academy Sciences*, Vol. 17, No. 5, May 1931, pp. 233-238.

when it is the major constituent of a gravel sample. It may be present as dense, unfractured chert; as vuggy chert; as porous chert; and as dense but highly fractured chert. The determination of which of these four conditions characterizes a particle may be expected to have an important influence on prediction of the behavior of the particle in concrete.

8.4 Record:

8.4.1 Notes should be taken during the examination. Each rock type should be described; the relevant features may include the following:

- 8.4.1.1 Particle shape.
 - 8.4.1.2 Particle surface, texture.
 - 8.4.1.3 Grain size.
 - 8.4.1.4 Internal structure, including observations of pore space, packing of grains, cementation of grains.
 - 8.4.1.5 Color.
 - 8.4.1.6 Mineral composition.
 - 8.4.1.7 Significant heterogeneities.
 - 8.4.1.8 General physical condition of the rock type in the sample.
 - 8.4.1.9 Coatings or incrustations, and
 - 8.4.1.10 Presence of constituents known to cause deleterious chemical reaction in concrete.
- 8.4.2 Particle counts should be recorded so that tables can be made for inclusion in the report. When the examination has been completed, the notes should contain enough information to permit the preparation of tables and descriptions. Tables should be prepared showing the composition and condition of the samples by sieve fractions, and the weighted average composition, based on the grading of the sample as received and on the distribution of constituents by sieve fractions. Descriptions of constituent groups should be prepared containing the relevant features among those enumerated in the preceding list.

9. Procedure for Examination of Natural Sand

9.1 The procedure for the examination of natural sand is similar to that for the examination of gravel, with the modifications necessitated by the differences in particle size.

9.1.1 *Sizes Coarser than 600 μm (No. 30)*—Each sieve fraction present that is coarser than the 600- μm (No. 30) sieve should be reduced in accordance with one of the procedures in Practice C 702 until a split or splits containing at least 150 particles are obtained. The reduced sample of each sieve fraction should be examined, and its constituents identified and counted, using the stereoscopic microscope. It is convenient to spread out the sample in a flat-bottom glass dish such as a Petri dish and manipulate the grains with a forceps and dissecting needle. The identification of grains in the coarser sand sizes is often easier when the grains are just submerged in water. The submergence lessens reflection from the outer surfaces and may show diagnostic features that cannot be seen when the grains are dry. There are exceptions to this generalization. Where identification is difficult, the examination includes examination of the natural surface (dry and wet), examination of a broken surface (dry and wet), and scratch and acid tests. Only after all of these steps have been taken and the grain is still unidentified should the petrographer resort to the petrographic microscope. Grains that cannot be identified using the stereoscopic microscope, or that are suspected of consisting of or con-

taining substances known to react deleteriously in concrete, should be set aside to be examined with the petrographic microscope. If the question of reaction with the alkalis (sodium and potassium) of portland cement paste is important in the examination of the sample, certain additions to the procedure are indicated. If the coarser sand sizes contain fine-grained, possibly glassy igneous rocks, several typical particles of each variety of such rocks should be selected for a more thorough examination. The petrographer should determine the presence or absence of glass by crushing typical grains and examining them in immersion media, using the petrographic microscope. In difficult or especially important cases, it may be necessary to break suspected grains and to make immersion mounts of part of the grain and a thin section of another part. Where the sand contains chert and the potential reactivity of the chert is an important consideration, a number of chert particles from the fractions retained on the 600- μm (No. 30) sieve should be set aside for determinations of the index of refraction. Methylene blue staining techniques may be useful in identifying the presence of smectite.⁹

9.1.2 *Sizes Finer than 600 μm (No. 30)*—The sieve fractions finer than the 600- μm (No. 30) sieve should each be reduced in a sample splitter or by quartering to about 4 or 5 g. The volume will usually be less than a level teaspoonful. In some gradings the fractions retained on the 150- μm (No. 100) and 75- μm (No. 200) sieves may be present in such small amounts that reduction is unnecessary. These splits should be further reduced on a miniature sample splitter or by coning and quartering with a spatula on a clean sheet of paper. The examination may be conducted as in 9.1.1. As required, the petrographic microscope should be used. For this purpose, a representative portion of each reduced split should be mounted in immersion oil on a clean glass slide and covered with a clean cover glass. No entirely satisfactory method of reducing a split to a predetermined number of grains is known. The reduced split can be sampled by spreading it in a thin layer on glass or clean paper, dragging the end of a dissecting needle moistened in immersion oil through the sample and transferring the grains that adhere to the needle to a drop of immersion oil on a clean slide. If this is done carefully, a fairly representative sample will be obtained. If the dissecting needle is made of magnetized steel, a concentration of magnetic minerals may result. It is usually necessary to make several mounts of the 300- μm (No. 50) and 150- μm (No. 100) sieve fractions to obtain at least 150 grains of each. The index of the immersion oil should be selected to make the identification of the important constituents as easy and as definite as possible. The use of an immersion oil with an index of or just below the lower index of quartz (1.544) is recommended. The slide should be mounted on a petrographic microscope equipped with a mechanical stage. Several traverses should be made, and each grain that passes under the cross hair should be identified and counted. Care should be taken to move the slide on the north-south adjustment between traverses so that no grain will be counted twice. Each sieve fraction passing the

⁹ Higgs, Nelson B., "Preliminary Studies of Methylene Blue Adsorption as a Method of Evaluating Degradable Smectite-Bearing Concrete Aggregate Sands," *Cement and Concrete Research*, Vol. 16, 1986, pp. 523-534.

600- μm (No. 30) and retained on the 75- μm (No. 200) sieve should be examined. Ordinarily, the material passing the 75- μm (No. 200) sieve is mounted on a slide following the procedure described above, examined by means of the petrographic microscope, and its composition estimated. If an unusually large amount of this size is present, or if it contains constituents that may be expected to have an important effect on the suitability of the aggregate for the intended use, it should be counted. In this event, it is suggested that the fraction passing the 75- μm (No. 200) sieve be washed over the 45- μm (No. 325) sieve before being counted.

9.1.3 Grain thin sections using an epoxy as mounting medium also may be useful in classifying particles passing the 300- μm (No. 50) sieve.

DRILLED CORE, LEDGE ROCK, CRUSHED STONE, AND MANUFACTURED SAND

10. Procedure for Examination of Drilled Core

10.1 Each core should be examined and a log prepared showing footage of core recovered, core loss and location; location and spacing of fractures and parting planes; lithologic type or types; alternation of types; physical condition and variations in condition; toughness, hardness¹⁰, coherence; obvious porosity; grain size, texture, variations in grain size and texture; type or types of breakage; and presence of constituents capable of deleterious reaction in concrete. If the size of the core permits, the probability that the rock will make aggregate of the required maximum size should be considered. If the surface of the core being examined is wetted, it is usually easier to recognize significant features and changes in lithology. Most of the information usually required can be obtained by careful visual examination, scratch and acid tests, and hitting the core with a hammer. In the case of fine-grained rocks, it may be necessary to examine parts of the core, using the stereoscopic microscope, or to prepare thin sections of selected portions. Some considerations and procedures are more applicable to particular rock types than to others. Ordinarily, the layered rocks considered for concrete aggregate will be limestone, and occasionally metamorphic rocks, such as phyllite, gneiss, or schist. One of the most important questions arising in the examination of limestone is that of the presence, type, and distribution of argillaceous impurities. Limestones that contain intercalated thin beds of soft shale may make suitable sources of aggregate if the shale is so distributed that it does not prevent manufacture of the required maximum size, and if the shale can be eliminated or reduced in processing. Where argillaceous impurities are present, it should be determined whether they actually consist of clay minerals or of other minerals in clay sizes. If they do consist of clay minerals, it should be established whether the clay minerals include swelling clays (that is, smectites, also known as the montmorillonite-saponite group of minerals).¹¹ X-ray diffraction analysis is especially valuable in identification and

quantitative determination of clay minerals. Methylene blue staining techniques are useful in identifying smectite. In the examination of fine-grained igneous rocks, particular attention should be directed to the nature of the ground-mass. This examination should include determination of the presence or absence of opal, chalcedony, natural glass, and swelling clays; if any of these are found the amount should be estimated; if natural glass is found the type should be determined.

11. Procedure for Examination of Ledge Rock

11.1 The procedure used in examination should be the same as for core samples to the extent that the spacing of samples and size of the individual pieces allow. If the sample consists of a relatively large quantity of broken stone produced by blasting, it is desirable to inspect the whole sample, estimate the relative abundance of rock types or varieties present, and sample each type before further processing. Subsequent procedure should be the same as given below for crushed stone.

12. Procedure for Examination of Crushed Stone

12.1 The procedure for examination of crushed stone should be similar to that for core, except that necessary quantitative data should be obtained by particle counts of the separated sieve fractions obtained as described in the section on Natural Gravel and Sand.

13. Procedure for Examination of Manufactured Sand

13.1 The examination procedure should be similar to that for natural sand, with particular emphasis on the amount and extent of fracturing and the amount and nature of rock dust developed by the milling operations. If a sample of the rock from which the sand was produced is available, examination of it will provide useful information.

CALCULATION AND REPORT

14. Calculation

14.1 Calculate the composition of each sieve fraction of a heterogeneous sample and the weighted average composition of the whole sample as follows:

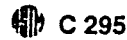
14.1.1 Express the composition of each sieve fraction by summing the total number of particles of that fraction counted, and calculating each constituent in each condition as a percentage of the total amount (as number of particles in percent, in each sieve fraction). It is convenient to calculate and record the percentages to tenths at this stage. An example of these calculations is given in the upper half of Table 2.

14.1.2 Obtain the weight percent of the sieve fraction in the whole sample (individual percentages retained on consecutive sieves) from the grading of the sample as determined by Method C 136.

14.1.3 By multiplying the percentage of the constituent in the sieve fraction, determined as described above by the percentage of the sieve fraction in the whole sample, obtained as described above, calculate the percentage in the whole sample of that constituent in that size (weighted percentage of constituents in sieve fraction, Table 2). It is

¹⁰ Woolf, D. O., "Methods for the Determination of Soft Pieces in Aggregate," *Proceedings, ASTM*, Vol. 47, 1947, p. 367.

¹¹ Carroll, Dorothy, "Clay Minerals: A Guide to Their X-ray Identification," *The Geological Society of America*, Special Paper 126, 1970.

TABLE 2 Calculation of Results of Particle Counts^a

Individual Percentage Retained on Sieve	Composition of Fractions Retained on Sieves Shown Below							
	19.0-mm (¾-in.)		12.5-mm (½-in.)		9.5-mm (¾-in.)		4.75-mm (No. 4)	
	17.4		32.6		29.5		20.5	
Constituents ^b	Number of Particles	%	Number of Particles	%	Number of Particles	%	Number of Particles	%
A ₁	250	50.0	200	40.0	150	30.0	50	10.0
A ₂	50	10.0	100	20.0	125	25.0	100	20.0
A ₃	10	2.0	50	10.0	75	15.0	100	20.0
B ₁	107	21.4	70	14.0	82	12.4	32	6.4
B ₂	76	15.2	53	10.6	19	3.8	87	17.4
B ₃	20	4.0	43	8.6	96	19.2
C ₁	5	1.0	5	1.0	20	4.0	20	4.0
C ₂	2	0.4	2	0.4	6	1.2	10	2.0
C ₃	5	1.0
Totals	500 ^c	100	500 ^c	100	500 ^c	100	500 ^c	100

	Weighted Percentages of Constituents in Each Sieve Fraction				Weighted Composition of Sample
	19.0-mm (¾-in.)	12.5-mm (½-in.)	9.5-mm (¾-in.)	4.75-mm (No. 4)	
A ₁	8.7	13.0	8.9	2.1	32.7 64.8 (Total A)
A ₂	1.7	6.5	7.4	4.1	19.7
A ₃	0.4	3.3	4.4	4.1	12.2
B ₁	3.7	4.6	3.7	1.3	13.3 31.8 (Total B)
B ₂	2.6	3.5	1.1	3.6	10.8
B ₃	...	1.3	2.5	3.9	7.7
C ₁	0.2	0.3	1.2	0.8	2.5 3.6 (Total C)
C ₂	0.1	0.1	0.3	0.4	0.9
C ₃	0.2	0.2
Total in sieve fraction	17.4	32.6	29.5	20.5	
Total in sample, condition 1					48.5
Total in sample, condition 2					31.4
Total in sample, condition 3					20.1

^a This table indicates a convenient method of setting up a work sheet for recording results and calculations. The results developed here are entered in the form indicated by Table 3. Table 3 is included in the petrographic report. Table 2 is not.

^b Letters (A, B, C) refer to the various constituents found, subscript numbers (1, 2, 3) refer to the various conditions in which each constituent has been found, such as relative degree of weathering.

^c The recommendation concerning the number of particles to be counted has been met; the selection of 500 particles per fraction for the example is to illustrate the calculation; it is not intended to suggest that a predetermined number of particles per fraction should be selected.

TABLE 3 Composition and Condition of an Aggregate Sample (Table Constructed from Calculations Shown in Table 2)^c

Constituents	Amount, as Number of Particles in Percent							
	In Fractions Retained on Sieves Shown Below ^a				In Whole Sample ^b			Totals
	19.0-mm (¾-in.)	12.5-mm (½-in.)	9.5-mm (¾-in.)	4.75-mm (No. 4)	Condition 1	Condition 2	Condition 3	
A	62	70	70	50	33	20	12	65
B	37	29	25	43	13	11	8	32
C	1	1	5	7	2	1	tr	3
Total	100	100	100	100	1	100
Weighted average, condition 1					48
Weighted average, condition 2					...	32
Weighted average, condition 3					20	...

^a Based on count of 500 particles in each sieve fraction. (The number of particles of each sieve fraction counted should be shown in the report and this may conveniently be done as a footnote to the table.)

^b Based on grading of the sample as received, and on the distribution of constituents by sieve fractions shown at the left above. (If the petrographic report forms part of a complete investigation of the sample, including a report of the grading, the grading need not be shown. If the petrographic report is to be submitted alone, the grading of the sample should be included with it.)

^c For other forms of presentation of results of petrographic analysis, see Tables 1-4 in Mielenz, R. C., "Petrographic Examination of Concrete Aggregates," Chapter 33, *Significance of Tests and Properties of Concrete and Concrete-Making Materials, ASTM STP 169 B*, p. 197.

convenient to calculate and record these percentages to tenths.

14.1.4 By adding the weighted percentages of each constituent in each sieve fraction, obtain the weighted percentage of each constituent in the whole sample (see under weighted composition of sample in Table 2).

14.1.5 Construct a table to show the composition of each

sieve fraction and the weighted composition of the whole sample. Report values to the nearest whole number. Report constituents amounting to 0.5 % or less of a sieve fraction or of the whole sample as traces. Table 3 is an example constructed from the data obtained in Table 2. As a convention, the total in each sieve fraction and the total in the whole sample shall each be 100 % without the traces.



Difficulties in abiding by this convention can usually be avoided by grouping minor constituents of little engineering importance. It is preferable to tabulate constituents known to react deleteriously in concrete so that their distribution will be apparent from inspection of the table, even though the amount in the whole sample or in any fraction is very small.

15. Report

15.1 The report of the petrographic examination should summarize the essential data needed to identify the sample as to source and proposed use, and include a description giving the essential data on composition and properties of the material as revealed by the examination. The report should record the test procedures employed, and give a description of the nature and features of each important constituent of the sample, accompanied by such tables and photographs as may be required. The findings and conclusions should be expressed in terms likely to be intelligible to those who must make decisions as to the suitability of a material for use as concrete aggregate.

15.2 When the sample has been found to possess properties or constituents that are known to have specific unfavorable effects in concrete, those properties or constituents should be described qualitatively and, to the extent practicable, quantitatively. The unfavorable effects that may be expected to ensue in concrete should be mentioned. When appropriate, it should be stated that a given sample was not found to contain any undesirable features. When such is the case it may also be appropriate, especially if the report of the petrographic examination is not accompanied by reports of

results of physical and chemical tests for which numerical limits may be applicable, to add that the material appears acceptable for use provided the applicable acceptance tests are made and the results are within the appropriate limits. The report should not, however, contain conclusions other than those based upon the finding of the examination unless the additional data to support such conclusions are included in or with the petrographic report and the petrographer has been authorized to analyze the other relevant nonpetrographic data.

15.3 The petrographic report should include recommendations regarding any additional petrographic, chemical, physical, or geological investigations that may be required to evaluate adverse properties that are indicated by the petrographic examination that has been performed. Supplementary petrographic investigations might include qualitative or quantitative analysis of the aggregate or of selected portions thereof by X-ray diffraction, differential thermal methods, or other procedures that are directed to identification and description of the constituents of the aggregate.

16. Precision and Bias

16.1 Those test methods that are used in connection with this guide that have been standardized in ASTM are subject to having precision and bias sections. Those that have not been standardized will each be provided with such a section, if and when standardized. None of the nonstandardized procedures mentioned for optional use in this practice are used in ways that lend themselves to the preparation of precision and bias statements.

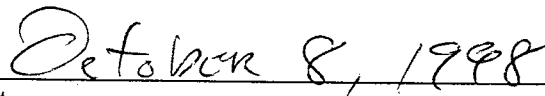
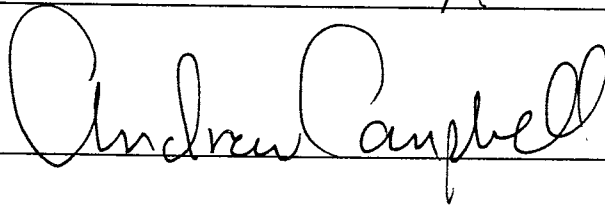
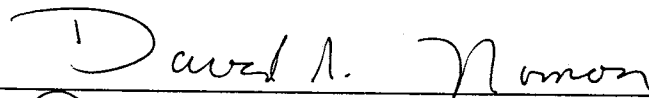
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This thesis is accepted on behalf of the faculty
of the institute by the following committee:



Advisor



Date