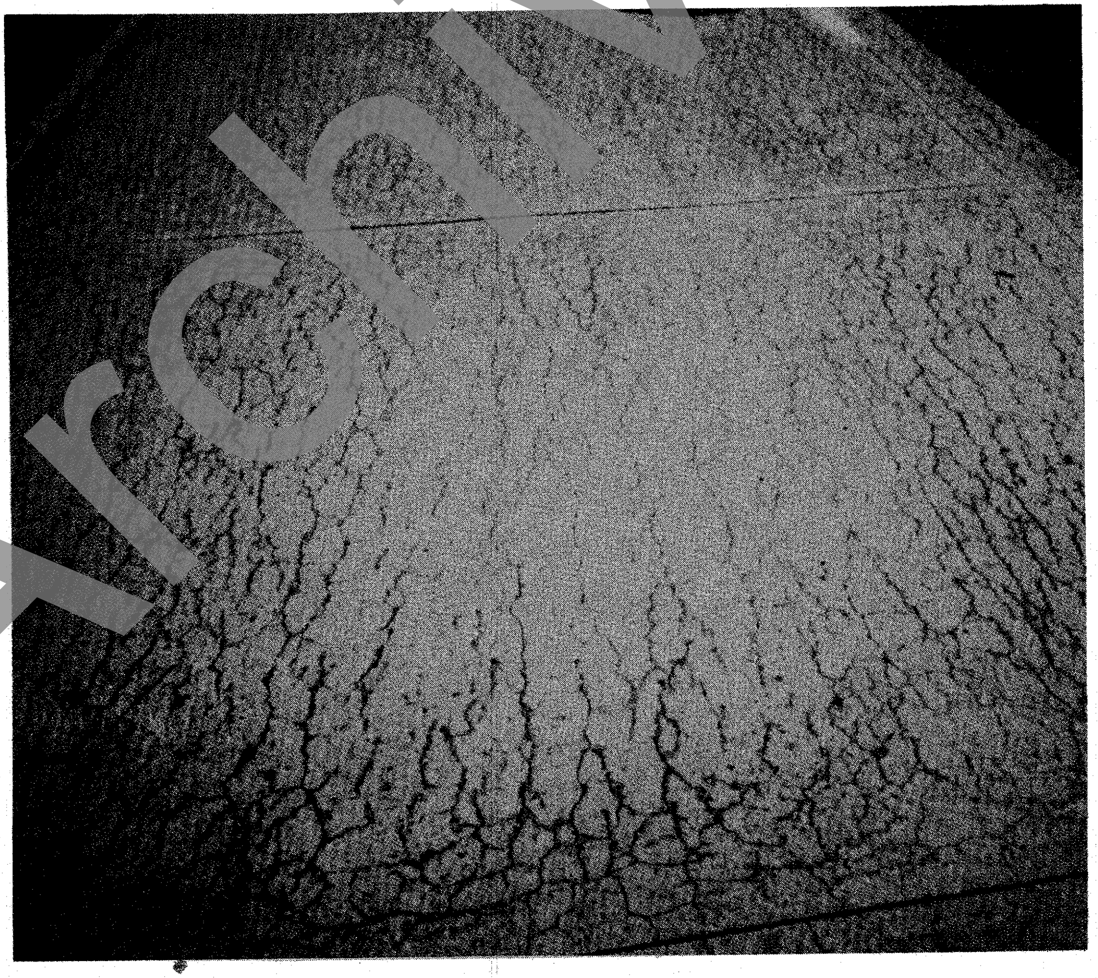
U.S. Department of Transportation
Federal Highway Administration

FHWA-SHRP SHOWCASE—WORKSHOP ON ALKALI-SILICA REACTIVITY IN HIGHWAY STRUCTURES

(INCLUDES SHRP PRODUCTS)



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FHWA-SHRP SHOWCASE

WORKSHOP ON ALKALI-SILICA REACTIVITY IN HIGHWAY STRUCTURES

(INCLUDES SHRP PRODUCTS)

Office of Engineering and
Office of Technology Applications
Federal Highway Administration
400 Seventh Street, SW
Washington, D.C. 20590

TABLE OF CONTENTS

TABLE OF CONTENTS (continued)

Appendices

TABLE OF CONTENTS (continued)

INTRODUCTION AND OBJECTIVES

This is the Federal Highway Administration's (FHWA) Strategic Highway Research Program (SHRP) Showcase Workshop on Alkali-Silica Reactivity (ASR). This program is one part of FHWA's implementation activities to disseminate information and showcase products developed under the SHRP program of Concrete and Structures.

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The goal of this Showcase is to educate people about ASR, and offer them solutions to address or avoid the problems it causes. This information from the SHRP study C-202 on ASR summarizes the following products:

1) "Eliminating or Minimizing Alkali-Silica Reactivity," Report SHRP-C-343. (SHRP Product 2011)

2) "Alkali-Silica Reactivity: An Overview of Research," Report SHRP-C-342. (SHRP Product 2011)

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5) Rapid Mortar Bar Test to Identify Potentially Expansive Reactive Aggregates. (SHRP Product 2009)

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This Showcase instructs in the identification of both innocuous and deleterious ASR in portland cement concrete. Procedures are explained for material testing and selection to avoid ASR in new concrete construction. Methods also are outlined to treat existing concrete pavement and structures to mitigate and minimize the effects of ASR.

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ACKNOWLEDGMENTS

This document is part of the Federal Highway Administration's (FHWA) Strategic Highway Research Program (SHRP) efforts to implement the products of the Alkali-Silica Reactivity (ASR) Contract SHRP-87-C-202, "Eliminating or Minimizing Alkali-Silica Reactivity." This work was conducted under the 1991 Intermodal Surface Transportation Efficiency Act which authorized FHWA additional funds to implement SHRP. Funding was provided by the FHWA's Office of Technology Applications (OTA), and coordinated by Ted Ferragut, ex-officio FHWA Division Chief, and Suneel Vanikar, FHWA's Concrete and Structures Program Manager.

Many people contributed to this document. It was prepared by David Stark, Principal Investigator, of Construction Technology Laboratories, Inc. (CTL), under contract with the FHWA, with contributions by CTL petrographers David Vollmer and Ronald Sturm, and Donald Campbell, formerly of CTL. Roger Surdahl, ASR Project Manager, from the FHWA's Office of Engineering, provided technical instructions to the team at CTL, with advice and guidance from the ASR Expert Task Group (ETG) listed below. We recognize that opinions of the individuals listed here may vary on some issues presented here, however each of them was invaluable in preparing this final document.

Roger Surdahl, ETG Secretary, FHWA, HNG-23
Donald H. Campbell, formerly of Construction Technology Laboratories, Inc.
James Dunne, FHWA, Region 3
Ted Ferragut (ex-officio FHWA, HTA-20) American Concrete Pavement Association
Tony Fiorato, Portland Cement Association
Steve Forster, FHWA, HNR-20
Colin Lobo, National Aggregate Association/National Ready Mix Concrete Association
James Hoblitzell, FHWA, HNG-32
Dr. Terence C. Holland, Master Builders, Inc.
Dan Johnston, South Dakota Department of Transportation
David G. Lambert, FMC Lithium Division
Steve Lane, Virginia Transportation Research Council
Vernon J. Marks, Iowa Department of Transportation
Marcia Simon, FHWA, HNR-20
David Stark, Construction Technology Laboratories, Inc.
A. Haleem Tahir, AASHTO Materials Reference Laboratory
Margaret Thomson, Pennsylvania Department of Transportation
Suneel N. Vanikar, FHWA, HTA-21
Paul Virmani, FHWA, HNR-10
Gerald Voigt, American Concrete Pavement Association
Hugh Wang, Master Builders, Inc.
Tom Weil, W. R. Grace

FHWA-SHRP Showcase Workshop
on
Alkali-Silica Reactivity

(Includes SHRP Products)

SCHEDULE

Day 1

Session 1 - Overview of Alkali-Silica Reactivity (ASR)

Objective: Provide an overview of ASR and the purpose of the Showcase, particularly targeted for management personnel who cannot attend the full workshop.

Time	Activity	Speaker
12:30-12:40	Welcome	FHWA/State DOT
12:40-1:00	Introduction - Objectives Scope of workshop	R. Surdahl/ D. Stark
1:00-1:45	Brief overview of concrete distress - D-cracking, spalling, popouts, ASR. Definition and historical perspective of ASR, local and nationwide occurrence of ASR, manifestations	D. Stark
1:45-2:15	ASR State-of-the-Practice, Solutions Role of SHRP SHRP products	D. Stark
2:15-2:45	Break	

Session 2 - Manifestations, Mechanisms, and Identification of ASR in Concrete

Objective: Demonstrate the causes of ASR, and show methods to indicate and identify ASR.

Time	Activity	Speaker
2:45-3:15	Lecture - ASR mechanisms and manifestations Factors affecting distress	D. Stark
3:15-4:00	Demonstration - Identifying ASR in concrete A) Crack patterns B) Stereomicroscope C) Petrographic microscope	D. Vollmer

4:00-5:00	Lecture and demonstration - Uranyl acetate-UV light method to identify possible ASR gel deposits A) Safety and handling of uranyl acetate-UV light method B) Hands-on use of the uranyl acetate-UV light method	D. Stark and D. Vollmer

Day 2

Session 3 - Field Trip to Identify ASR in Structures

Objective: Illustrate crack patterns due to ASR and other causes in field concrete, and demonstrate the uranyl acetate-UV light method on highway structures.

Time	Activity	Speaker
7:30-8:00	Description and history of the highway structures	DOT representative
8:00-8:45	Travel to field site	
8:45-11:15	Inspection of structures Hands-on use of the uranyl acetate-UV light method	D. Stark D. Vollmer DOT representative
11:15-12:00	Return to classroom	
12:00-1:00	Lunch	

Session 4 - Fluorescence and Microscopic Techniques to Identify ASR and Potentially Reactive Rock Types in Concrete

Objective: Demonstrate the microscopic techniques to identify potentially reactive rock types used in concrete.

Time	Activity	Speaker
1:00-1:30	Examine cores from structures inspected in Session 3	D. Stark and D. Vollmer

1:30-2:30 Lecture and demonstration - Petrographic examination of concrete and reactive rocks — D. Vollmer

- A) Classification of rocks
- B) ASTM C 295 "Petrographic Examination of Aggregates for Concrete"
- C) Use of powder mounts and thin sections to examine reactive rocks
- D) Problems with aggregates (other than ASR)

2:30-3:15 Demonstration - Fluorescence Microscopy — D. Vollmer

- A) Fluorescence in minerals
- B) Fluorescent epoxy for microcrack detection
- C) Visual aids for illustration

3:15-3:30 Break

<u>Session 5</u> - Rapid Immersion Test Method for Potential ASR of Aggregates

Objective: Describe the rapid immersion test method to identify potentially deleteriously reactive aggregate.

3:30-4:30 Lecture - Rapid immersion test (ASTM P 214 and C 1260) to identify potential for expansive ASR in concrete — D. Stark

- A) Potential for deleterious ASR
- B) Safe cement alkali level
- C) Acceptance testing - supplementary cementitious materials

Day 3

Session 6 - Preventing ASR in New Construction

Objective: Present options to use with fresh concrete to prevent expansion due to ASR.

Time	Activity	Speaker
8:00-9:30	Lecture - Controlling ASR in new concrete A) Avoid reactive aggregate B) Reduce cement alkali levels C) Use suitable mineral admixture 1) Fly Ash 2) Silica fume 3) Ground volcanic glass 4) Ground granulated blast furnace slag 5) Blended cements D) Use lithium compounds 1) Mechanism 2) Test results 3) Method of introduction 4) Safety precautions	D. Stark

Session 7 - Local Region Perspective of ASR

Objective: Present local region information and views by State highway and industry representatives on occurrences of ASR.

Time	Activity	Speaker
9:30-9:45	Lecture - local ASR problems	DOT representative
9:45-10:00	Lecture - local materials	Industry representative
10:00-10:15	Lecture - local materials	Industry representative
10:15-10:30	Break	

Session 8 - Combating ASR in Existing Concrete, Summary and Discussion, and Questions and Answers

Objective: Describe approaches to take with existing hardened concrete to mitigate expansion due to ASR, recommend a sequence of tests and equipment to be used to control ASR, and summarize information presented in the workshop.

Time	Activity	Speaker
10:30-11:15	Lecture - Combating ASR in existing concrete A) Drying B) Restraint C) Lithium treatment D) Methacrylates E) Overlays F) Remaining Life	D. Stark
11:15-11:30	Summary and closeout discussion	D. Stark
11:30-11:50	Questions and answers	D. Stark and D. Vollmer
11:50-12:00	Closing remarks	R. Surdahl
12:00-1:00	Lunch	

Session 9 - Regional Technical Working Group

Objective: Evaluate the workshop information, recommend specifications, and identify needs for further studies. Participation to include FHWA Regional Engineer (secretary), FHWA, State DOT, and designated industry representatives.

Time	Activity	Speaker
1:00-4:00	Discuss workshop; evaluate information and techniques; reach consensus on procedures, specifications, and on regional implementation activities.	As designated

EXECUTIVE SUMMARY

INTRODUCTION

This is the Federal Highway Administration's (FHWA) Strategic Highway Research Program (SHRP Showcase Workshop on Alkali-Silica Reactivity (ASR). This program is one part of FHWA's implementation activities to disseminate information and showcase products developed under the SHRP program of Concrete and Structures. In showcasing the SHRP ASR products, the objectives are to:

1) Instruct in the recognition of ASR in existing portland cement concrete structures.

2) Describe and recommend procedures for testing and identifying materials that will be resistant to expansive ASR in new construction.

3) Propose methods that can mitigate the continued development of ASR and its deleterious effects in existing highway structures.

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WHAT IS ASR?

ASR is a two-step physico-chemical reaction in portland cement concrete between alkalies and silica or silicates, in the presence of moisture. The first step is a reaction between the alkalies and silica to form an hydrophilic expansive ASR gel. In the second step, the gel absorbs moisture, swells, and creates an internal pressure that may crack the concrete. This is known as deleterious ASR.

WHAT DOES ASR LOOK LIKE IN THE FIELD?

ASR may cause cracks in concrete that define small polygonal areas, or extend primarily in the direction of greatest restraint to expansion. Since internal pressure will give way in the plane of least resistance, the cracks will generally run in the same direction as the unrestrained edges of the pavement or structure. At times the ASR gel flows into the cracks where it can be seen as a whitish deposit. Figure 1 illustrates the crack pattern. Another characteristic typical of ASR is darkened rims that form within reacted aggregate particles, as shown in Figure 2. ASR gel also may be seen in the rim area.

HOW CAN ASR BE IDENTIFIED?

The existence of ASR can be suspected through visual observation of crack patterns, gel deposits, and past history of material sources. ASR can be detected in the field through the use of an ultraviolet (UV) light kit in combination with uranyl acetate solution. After exposing a section of concrete, the section is treated with the solution. If it is present, the ASR gel will absorb the solution and fluoresce to a green or yellow color under the UV light as shown in Figures 3 and 4. Confirmation of the presence of ASR, however, must be done in the laboratory using petrographic procedures.

HOW IS ASR AVOIDED?

ASR can be avoided in new concrete with proper material selection. This is done using the past history of materials, or by testing new sources for ASR. New sources can be evaluated by determining expansions of material combinations with a rapid mortar bar test initially developed in South Africa. Variations of this test exist: AASHTO TP14, ASTM C 1260 (formerly ASTM P 214), or the South African Mortar Bar Test. When expansion exceeds 0.10% (or 0.08% recommended by SHRP) then remediation measures must be taken with the material combinations. Typical measures include selecting cement with safe alkali contents, using non-reactive aggregates, replacing a percentage of the cement with certain pozzolans, or using a chemical admixture such as a lithium compound. The latter is an experimental procedure developed under SHRP. Guide specifications should be developed to establish a system for the proper use of materials to avoid future deleterious ASR.

WHAT OPTIONS ARE AVAILABLE FOR TREATING EXISTING ASR?

ASR in existing structures creates dilemmas where resolutions are less obvious. Drying the concrete and keeping it below 80% relative humidity stops further ASR expansion. Applying restraint to counteract expansive ASR forces also can prevent further expansion. High density methacrylates can bond crack faces together, thereby possibly stiffening the concrete member. Applying lithium compounds to the concrete is another method being tried experimentally to stop further ASR damage. A final option is to reconstruct the structure using mitigation measures for new concrete.

CONCLUSION

ASR is a form of concrete distress that can be avoided or managed with proper care. With the necessary resources, and adequate assistance in identification, mitigation, training, and support, ASR can be avoided. While immediate solutions do exist, addressing ASR is really a long-term solution.

CONTACTS FOR FURTHER INFORMATION

The Federal Highway Administration Division Office in your area will provide you with further information, or contact the Federal Highway Administration, Office of Technology Applications, Engineering Applications Division (HTA-20), 400 Seventh Street, S.W., Washington, D.C., 20590.

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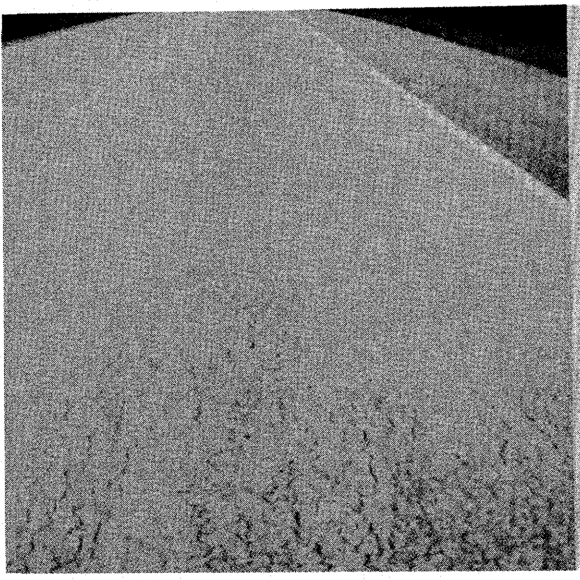


Fig 1 ASR has a typical crack pattern as shown in this pavement.

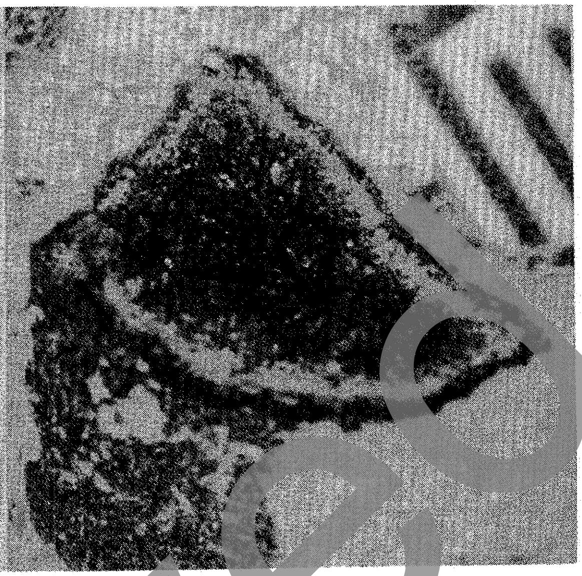


Fig 2 Darkened rims within this aggregate indicate the presence of ASR. White ASR gel also is present in the aggregate. Scale, 1 div. = 1 mm.

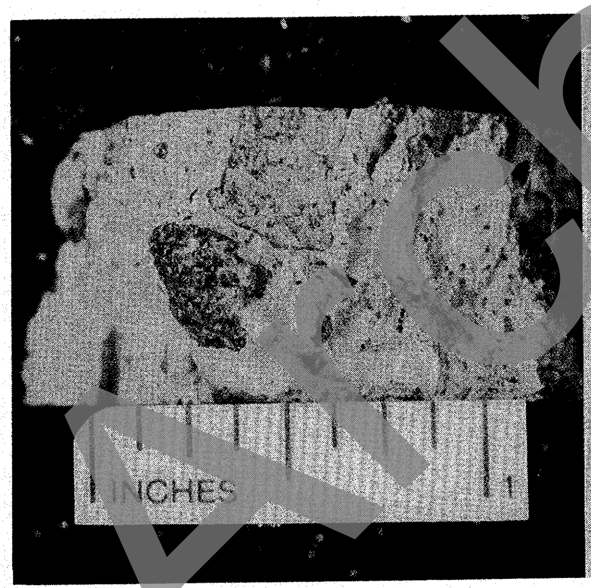


Fig 3 Original untreated concrete surface.

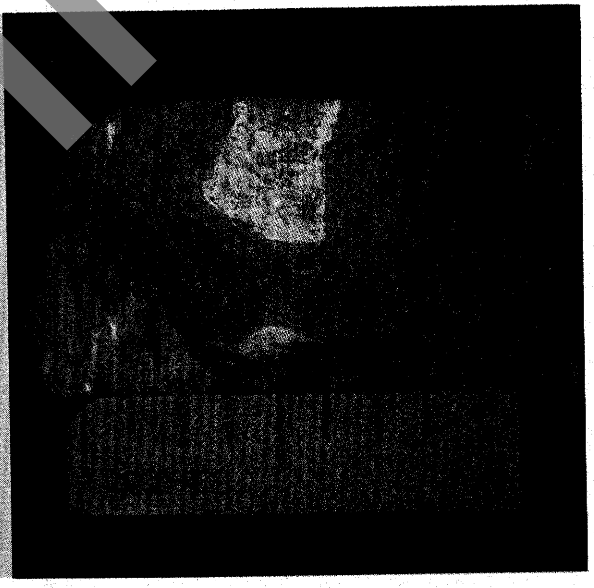


Fig 4 Concrete surface treated with uranyl acetate solution and viewed in UV light.

SESSION 1 - OVERVIEW OF ALKALI-SILICA REACTIVITY (ASR)

Notes

I. Welcome

II. Introduction - Objectives, scope and schedule of the workshop.

III. Overview of concrete distress and typical manifestations.

A) ASR - map or longitudinal cracking occurs in joint areas and central portions of columns and pavement slabs. Distress is due to an expansive reaction between certain types of aggregates and the alkaline pore solutions in the concrete. Crack pattern is influenced by restraint (Figs 1-1 through 1-4).

B) D-cracking - cracks occur adjacent and subparallel to joints, intermediate transverse cracks, and free edges of pavement slabs. Distress due to freeze-thaw failure of coarse aggregate (Fig 1-5).

C) Popouts - isolated inverted shallow, cone-shaped breakouts of near-surface concrete due to freeze-thaw failure or oxidation of iron sulfide minerals in aggregate particles (Fig 1-6).

IV. Occurrence and historical perspective (See Appendix A).

A) ASR is reported to have developed to one degree or another in most states.

B) ASR was first diagnosed as a source of distress in concrete highway structures in California in the late 1930's. In 1930's and 1940's, ASR also was identified in dams in Virginia and elsewhere, and in highways in the Kansas-Nebraska area.

1) Distress was characterized as "abnormal" cracking.

2) Laboratory and field observations revealed the following (Fig 1-7).

a) Certain rock types were deleteriously reactive.

b) Cement alkali contents less than 0.60% as equivalent Na_2O seemed to prevent expansive ASR.

c) Use of fly ash and natural pozzolans in concrete could prevent expansive ASR.

3) By the early 1950's, ASTM C 227 (mortar bar test) and C 289 (Quick Chemical Test) were accepted as methods to identify potential for expansive ASR.

V. ASR State-of-the-Practice Solutions

A) By the 1970's and 1980's, it was found that the 0.60% limit for the alkali content of the cement, ASTM C 227 and C 289, and the use of certain pozzolans did not always eliminate potential for expansive ASR in highway and other field structures.

B) Role of SHRP

1) In 1988, a 5-year project, sponsored by SHRP was initiated to develop means to eliminate or minimize ASR in concrete highway structures. CTL was the prime contractor.

2) Objectives of this project were as follows:

   a) Investigate fundamental aspects of ASR mechanisms.

   b) Develop rapid and reliable test methods to identify potential for deleterious ASR of aggregates and cement-aggregate combinations.

   c) Develop means to mitigate expansive ASR in existing concrete.

3) SHRP products from this project were as follows:

   a) Report SHRP-C/VWP-92-601, "Alkali Aggregate Reactions in Concrete: An Annotated Bibliography," 1939-1991

   b) Report SHRP-C-342, "Alkali-Silica Reactivity: An Overview of Research (Appendix B)

   c) Report SHRP-C-343, "Eliminating or Minimizing Alkali-Silica Reactivity (Appendix O)

d) Report SHRP-C-315, "Handbook for the Identification of Alkali-Silica Reactivity in Highway Structures" (Appendix F)

e) Equipment to identify ASR in concrete, using the uranyl acetate-UV light method

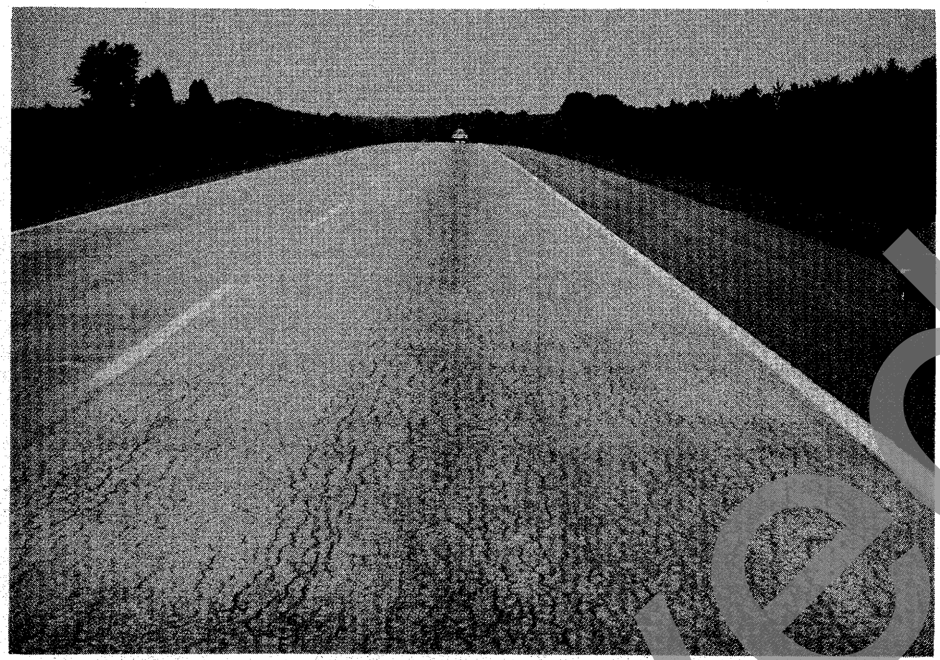


Fig 1-1 Severe longitudinal cracking due to ASR in a continuously reinforced pavement.

Fig 1-2 More generalized map cracking due to ASR in a jointed pavement.

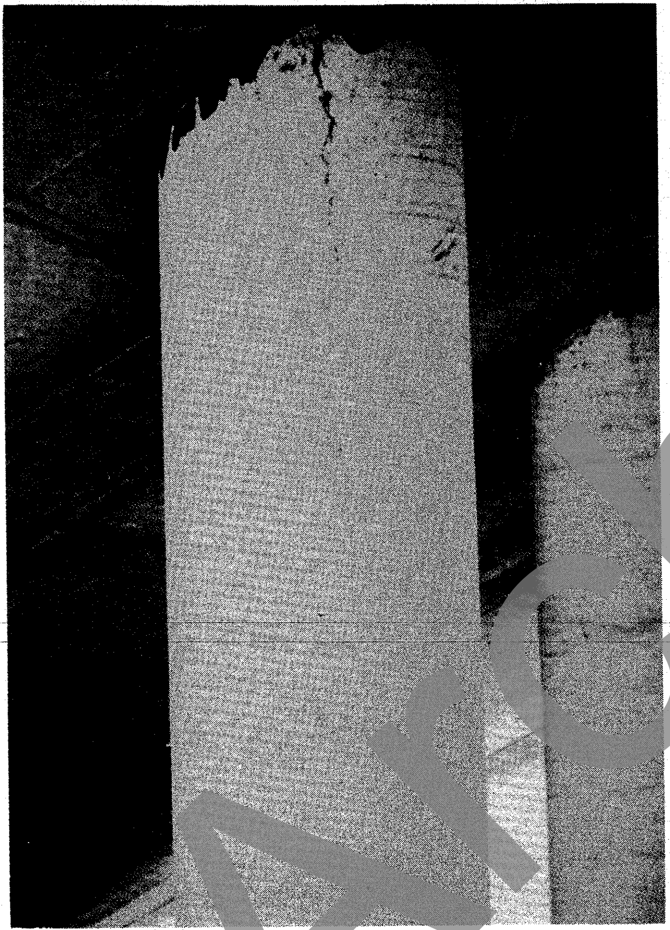


Fig 1-3 Vertical cracking due to ASR in a bridge column.

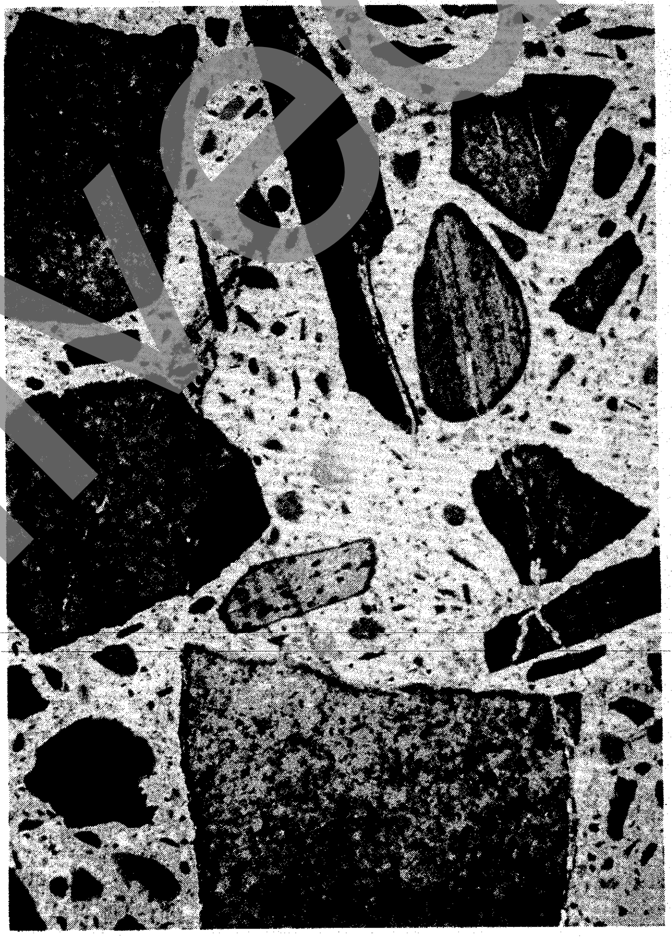


Fig 1-4 Cracking due to ASR in coarse aggregate particles. Reaction rims and ASR gel also are present in the aggregate.

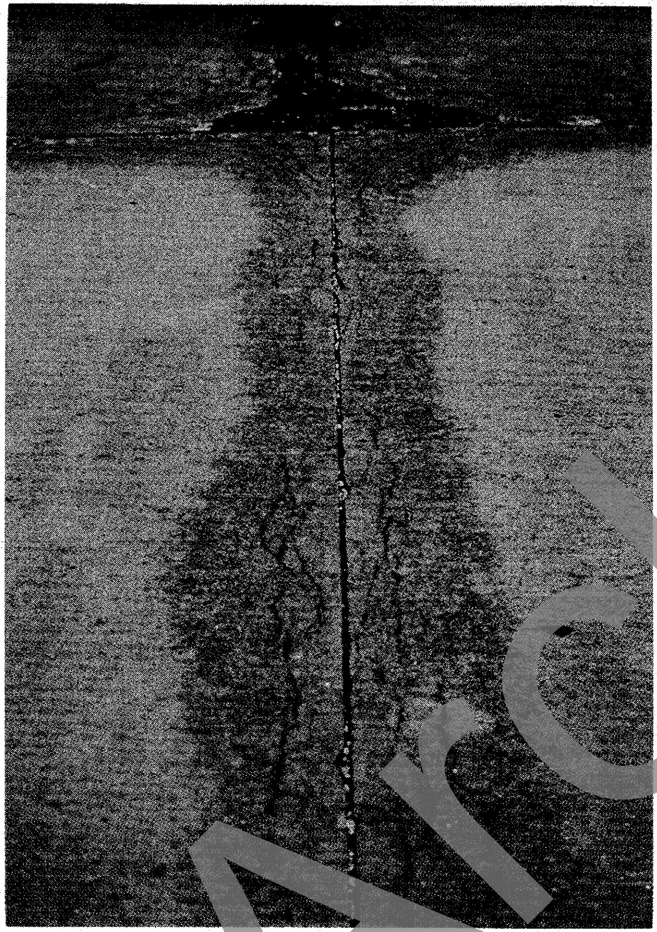


Fig 1-5 Early D-cracking which may be confused with early cracking due to ASR at transverse joint.

Fig 1-6 Popout due to freezing and thawing in a chert coarse aggregate particle.

OBSERVATIONS IN EARLY ASR INVESTIGATIONS

1. Only certain rock types reacted deleteriously
2. Expansive ASR could be prevented by using portland cement with less than 0.6% alkali as equivalent Na_2O
3. Portland cement-pozzolan combinations could prevent expansive ASR

Fig 1-7 Major observations in the early investigations of ASR.

SESSION 2 - MANIFESTATIONS, MECHANISMS, AND IDENTIFICATION OF ASR IN CONCRETE

Notes

I. Occurrence and Distribution

A) Potentially deleteriously reactive aggregates cover a wide range of composition (Fig 2-1).

B) In the United States, reactive rock types vary significantly in composition and relative reactivity from region to region (Fig 2-2).

Atlantic Seaboard - Maine to Georgia:
Reactive types: primarily metamorphic, such as gneisses, schists, granites, quartzites, metagraywackes and metavolcanics, chert.
Characteristic: reactive with high alkali cements.

Southern states - Florida to Texas:
Reactive types: primarily chert and quartzite, together with some opaline and chalcedonic carbonates and shales.
Characteristic: reactive with high alkali cements.

Midwest states - Ohio to Minnesota and Missouri:
Reactive types: opaline to chalcedonic carbonates, shales, and sandstones.
Characteristic: reactive with high alkali cements.

Great Plains states - North Dakota to Oklahoma and Colorado:
Reactive types: opaline shales,

quartzites, siliceous limestones, volcanics, chert.
Characteristic: reactive with low to high alkali cements.

Basin and Range states - Montana to Arizona:
Reactive types: glassy to cryptocrystalline rhyolite to andesite volcanics, chert.
Characteristic: reactive with low alkali cements.

Pacific Coast states - Washington to California:
Reactive types: glassy to cryptocrystalline rhyolite to andesite volcanics, chert, opaline sedimentary rocks.
Characteristic: reactive with low alkali cements.

II. Manifestations of ASR (Figs 2-3 to 2-6).

A) ASR is a process that may produce expansion and cracking in concrete. Not all ASR causes distress. Severity of cracking depends on (Fig 2-7):

1) extent of reaction - for a given concrete, the greater the degree of reaction, the greater the expansion.

2) composition of ASR gel - gels with higher alkali contents have, in general, greater capacity to expand.

3) internal restraint to expansion - adequate restriction to flow of ASR gel is needed to cause expansion of concrete. The resulting crack pattern is determined by differential magnitude and direction of movement of concrete.

4) degree of drying near surface increases the strain gradient, therby increasing width of cracks already formed by ASR.

B) Crack pattern that develops depends on (Fig 2-8).

1) geometry and restraint - non-uniform restraint alters the crack pattern, e.g., longitudinal restraint in pavement results in expansion in the transverse direction and in predominantly longitudinal cracking.

2) temperature and drying - higher temperatures accelerate and increase expansion. Drying can prevent expansion but exacerbate subsequent ASR after wetting by concentrating alkalies in the concrete pore solution.

3) traffic loading - can cause cracking and breaking out of concrete already weakened by cracking due to ASR.

4) crack propagation - growth of cracks increases the frequency and severity of cracking.

III. Mechanism of ASR and Expansion

A) These are three requirements for expansive ASR to occur (Fig 2-9):

Reactive silica in aggregate

Sufficient available alkali

Moisture

B) In simplified form, ASR is a two-step process (Appendix B and Fig 2-10).

STEP 1 - ALKALI + SILICA GEL = REACTION PRODUCT

Note: hydroxyl (OH$^-$) ion is the actual specie that attacks the aggregate. It's negative charge is electrically balanced primarily by the alkali ions sodium (Na$^+$) and potassium (K$^+$) in solution. Therefore, the greater the concentration of alkalies, the greater the OH$^-$ concentration, and the more aggressive is the solution.

STEP 2 - GEL REACTION PRODUCT + MOISTURE = EXPANSION

C) In highway structures, moisture availability is controlled primarily by climatic conditions. Desert exposures result in cyclic conditions favorable to expansion due to ASR. In these exposures, drying can be sufficient to remove moisture otherwise available for absorption by ASR gel reaction products. Slabs-on-grade, even in desert areas, have a continuous supply of subbase moisture available for ASR (Figs 2-11 and 2-12).

IV. Identifying ASR by the uranyl acetate-UV light method (Appendix C).

A) The presence of gel reaction product is indisputable evidence that ASR has occurred. However, its mere presence does not mean that ASR has caused distress. The uranyl acetate method can rapidly reveal the presence of ASR gel. This procedure was developed by Drs. Hover and Natesaiyer at Cornell University.

B) Certain safety precautions must be observed in the use of this method (See Appendix D).

C) Step-by-step instructions in the use of this procedure are given below.

1) Laboratory examination of 3-in. + diameter concrete core.

a) Produce freshly fractured surfaces in core by hammering loose several pieces of concrete.

b) Rinse fractured surfaces with deionized or distilled water and <u>blot</u> (do not wipe) with cloth rag. Deionized or distilled water is recommended to ensure that contaminants or other undesirable dissolved substances are not introduced into the concrete.

c) Apply several drops of uranyl acetate solution to still-damp freshly fractured surface with specimen in sink or plastic pan. A 100- to 500-ml plastic squeeze bottle with fine plastic works well for this purpose. Allow pieces to stand for 3 to 5 minutes.

d) Gently rinse treated surfaces with fine soft stream of deionized or distilled water, blot lightly to damp condition with fine cloth. It is important to rinse thoroughly to remove uranium ion that may have lodged elsewhere.

e) Examine treated surface in dark using UV light. Darkened room or viewing box is satisfactory for observations.

f) Look for localized areas with light yellow-green fluorescence in UV light. These are likely to be found within aggregate particles, either over entire cross section of particles or in peripheral regions. Fluorescent linings often will occur in air voids or in cracks in the concrete. All are examples of probable ASR gel deposits (Figs 2-13 and 2-14). Other secondary deposits, such as ettringite, likely will be dissolved by the uranyl acetate solution because it is acidic, having a pH of about 3.

g) Due to insufficient rinsing after treatment, a faint general yellowish fluorescence may be observed throughout the cement paste matrix. Or, uranyl acetate solution may be retained around the edges of the treated area, or run down other surfaces. This fluorescence should not be considered diagnostic of ASR gel.

2) Examination of in-situ concrete surfaces on field structures.

a) Prepare an in-place concrete surface by bushhammering with an impact drill, an area about 4 x 6 in. approximately 1/4-in. deep. The depth of bushhammering should be sufficient to expose a representative distribution of coarse aggregate particles (Figs 2-15 and 2-16).

b) Rinse prepared surface with deionized water and blot to dampened condition with cloth rag.

c) Treat prepared surface with a few drops of uranyl acetate solution. For vertical surfaces, hold a small cloth rag along the bottom edge of the treated area to catch any solution that drips below treated area (Fig 2-17).

d) Place viewing box with mounted UV light source over treated area, examine, and evaluate as in the laboratory.

e) When finished, bushhammer the treated surface layer and catch chips and powder with small hose to vacuum cleaner placed adjacent to bushhammer fitting. Continue cleaning procedure until the concrete no longer displays the greenish fluorescent tinge of uranium residue in UV light and the concrete surface displays the contrast of normal colors that appeared before treatment (Fig 2-18).

V. Microscopical Detection of ASR in Concrete

The microscopic examination of concrete is probably the surest means of confirming the development of ASR and is strongly recommended as an essential part of any assessment of concrete to determine or confirm causes of distress. Guidelines provided herein concentrate on ASR (See Appendix E).

A) Two types of microscopes are commonly used: stereomicroscope and a polarized-light microscope. Some are relatively small and portable, others are too heavy or delicate for field use. Both require a light source for optimum use.

B) Other equipment needed for laboratory examination of concrete is given in ASTM C 856. Required equipment includes diamond saws, lapping wheels, abrasives, and various refractive index liquids, glass microscope slides, cover slips, and other items.

C) For optimum use of microscopes, the investigator should be qualified by education or experience to operate the equipment and correctly identify the many features of ASR and associated petrographic aspects of aggregate and concrete.

VI. Utilization of the Stereomicroscope

A) Examination with a stereomicroscope involves broken surfaces, core sides, and lapped and polished cross sections of in-service concrete or laboratory specimens (Fig 2-19).

1) Hardened ASR gel deposits occur in voids and cracks and oozing from the sides of the core or saw-cut surface. Look for white to colorless accumulations in thin layers on void or crack surfaces. They may have the appearance of a hardened clear film showing an abundance of polygonal cracks (like checking on glazed pottery). Hardened gel is brittle, breaking into particles with conchoidal fractures. Gel-soaked paste is unusually translucent and appearing to be wet.

2) Microcracks typically radiate from within the reacting aggregate particle into the paste. Microcracks may or may not be coated with gel, which can be washed out by water during saw cutting. Microcracks typically are 0.01 to 1.0 mm wide, and short (2 to 10 mm long). The cracks may narrow from the aggregate center toward the periphery of the particle.

   a) Microcracks are best observed on polished surfaces using a bright illumination and magnifications up to 100x. Observation is aided by wetting the polished surface with glycerin or propylene glycol.

   b) Microcracks restricted to the cement paste may be caused by drying shrinkage, thermal stresses, freezing and thawing, or other processes and have nothing to do with ASR.

3) ASR gel and ettringite are common in cracks. ASR gel is normally noncrystalline, somewhat glassy in appearance, of widely varying refractive index, and irregular in particle shape, often displaying conchoidal fracture. It is generally isotropic with no apparent color in the petrographic microscope.

   Ettringite is easily recognized and displays elongated, almost needlelike habit, is crystalline and possesses different black and gray colors during rotation of the petrographic microscope stage under crossed polarizing prisms. Its refractive indices are 1.462 and 1.465.

4) Reaction Rims are generally darkened and translucent in appearance within the aggregate and occur at the paste-aggregate interface.

   a) Reaction rims can be seen on freshly broken surfaces as well as sawcut polished sections, sometimes better one way than the other.

   b) Width of a reaction rim is normally 1 to 2 mm. It can be confused with or combined with ordinary geologic weathering rims; crushed quarry rock will not have weathering rims and crushed gravel commonly shows weathering rims truncated by fractured surfaces produced in crushing.

VII. Utilization of the Polarized-Light Microscope

A) Sometimes called the "Petrographic Microscope," due to its development largely in the field of rock examination (petrographic geology).

B) For an ASR evaluation, two types of microscopical preparations are typically used: <u>immersion mounts</u> (finely ground powders of aggregates, mortar, concrete, or ASR gel immersed in a liquid of known refractive index, n) on a glass microscope slide, or <u>thin sections</u> (20- to 30-micrometer thick slices of concrete, mortar, or aggregates mounted on a glass microscope slide with epoxy resin).

1) Both preparations allow one to utilize transmitted plane-polarized light through the particles or the thin section, the observed optical effects providing information for identification of the particles or mineral grains.

2) The immersion mount may include powder from the reactive aggregates, the associated gel, and cement paste.

3) The thin section is normally made from a 24 x 46 mm block cut from the lapped and polished slab, and attempts to retain the reactive aggregate and gel intact and undisturbed. This is easier if the sample has been epoxy-impregnated (Fig 2-20).

4) Optical characteristics of ASR gel particles (Figs 2-21 and 2-22):

a) Conchoidal fracture.

b) Porcelaneous appearance, white to brown, colorless, possibly "dusty" or cloudy.

c) Index of refraction (*n*) — approximately 1.46 to 1.53, variable, "n" can be considered as the inverse of the velocity of light and is used to identify particles mounted in liquids of know refractive index. The same procedure can be used to determine the refractive indices of various minerals or secondary compounds in concrete such as ettringite. Such investigations require a working knowledge of optical mineralogy and crystallography.

d) The gel particle is optically isotropic; that is, in cross-polarized light the gel remains dark. Gel may show a slight change to gray in this mode of observation due to strain. Inclusions (tiny particulate material) in the gel may be anisotropic. The gel may have crystallized, forming a myriad of tiny interlocking, anisotropic crystals.

POTENTIALLY REACTIVE MATERIALS

Highly Reactive

Opal and Chalcedony in:
Limestone, Chert, Shale, and Sandstone

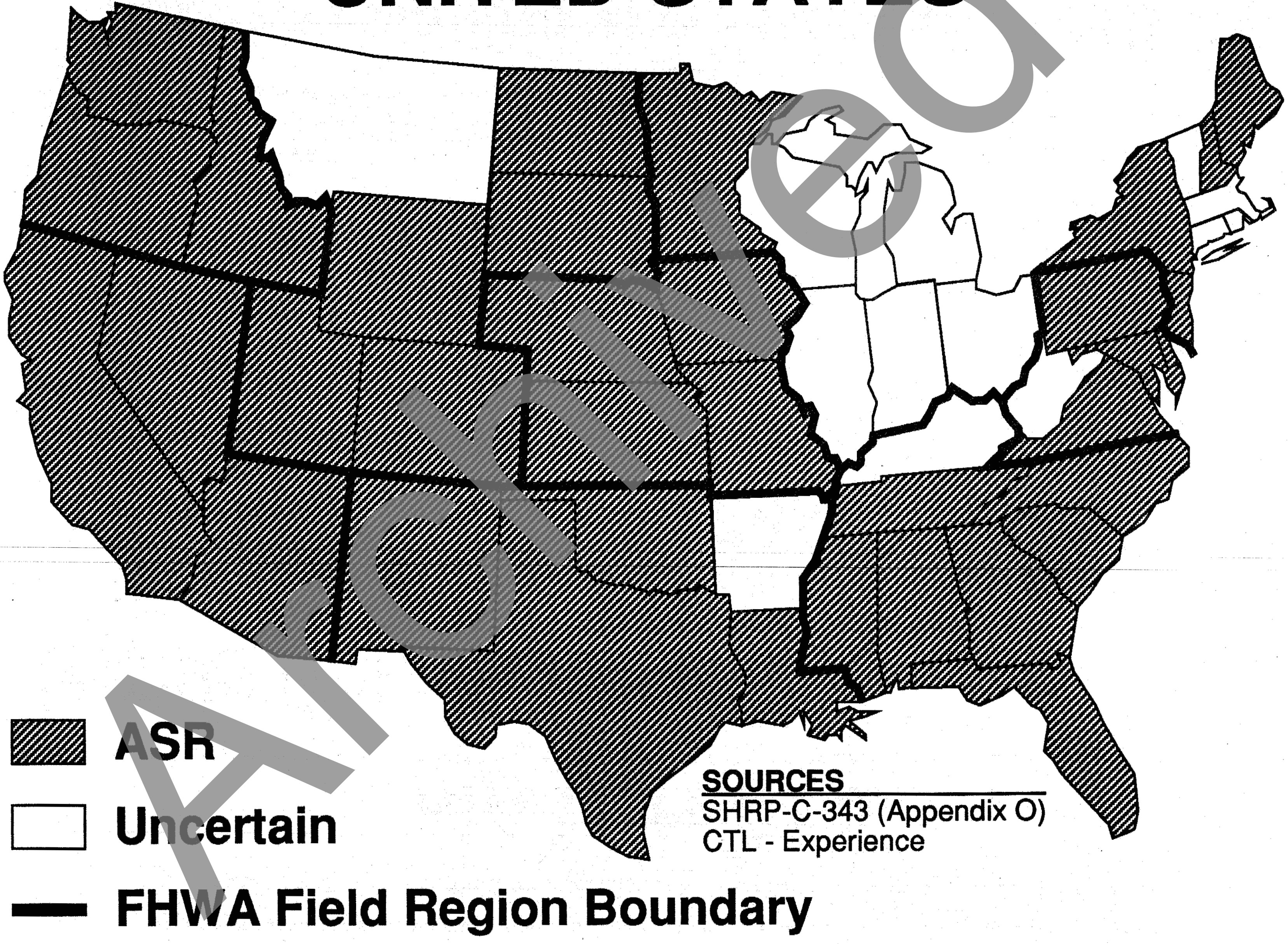
Glassy Volcanics:
Andesite and Rhyolite

Slowly Reactive

Microcrystalline and Strained Quartz in:
Schist, Metagraywacke, Granite Gneiss, and Quartzite

Fig 2-1 Known ASR-reactive rocks and minerals.

OCCURRENCE OF ASR IN THE UNITED STATES



SOURCES
SHRP-C-343 (Appendix O)
CTL - Experience

Fig 2-2 Occurrence of ASR in the United States. Map shows only the geographical distribution of known or reported occurrences of ASR and does not reflect severity of its development.

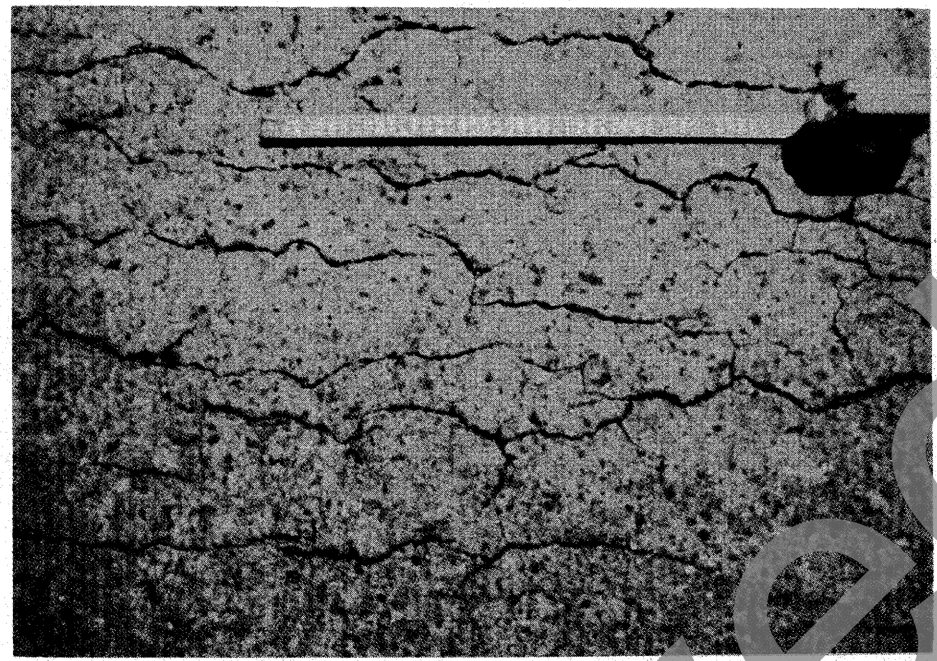


Fig 2-3 Closeup of surface cracking due to ASR in jointed pavement.

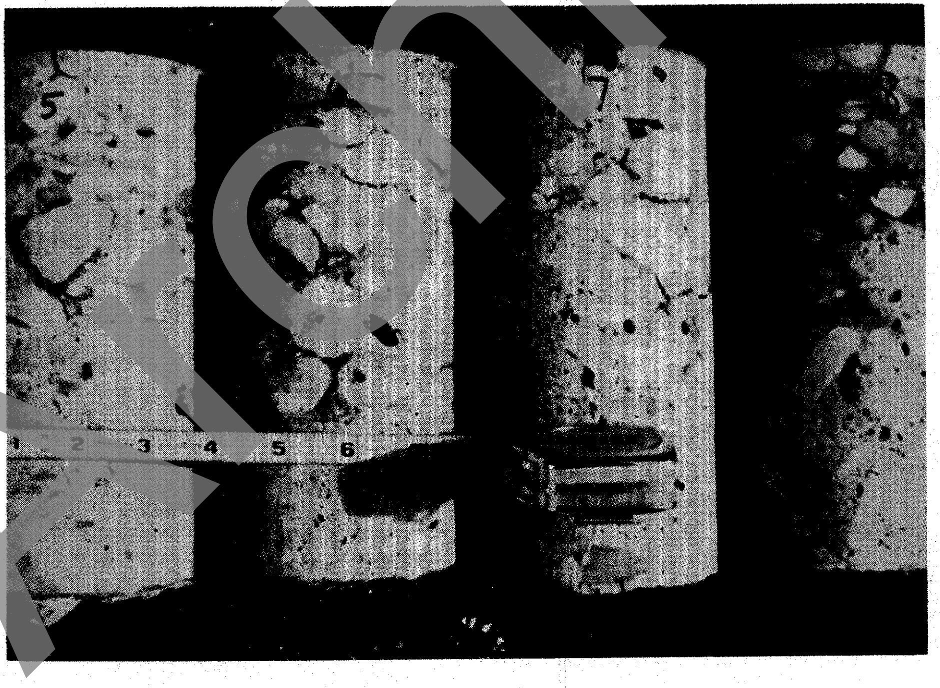


Fig 2-4 Extent of cracking due to ASR in jointed pavement shown above.

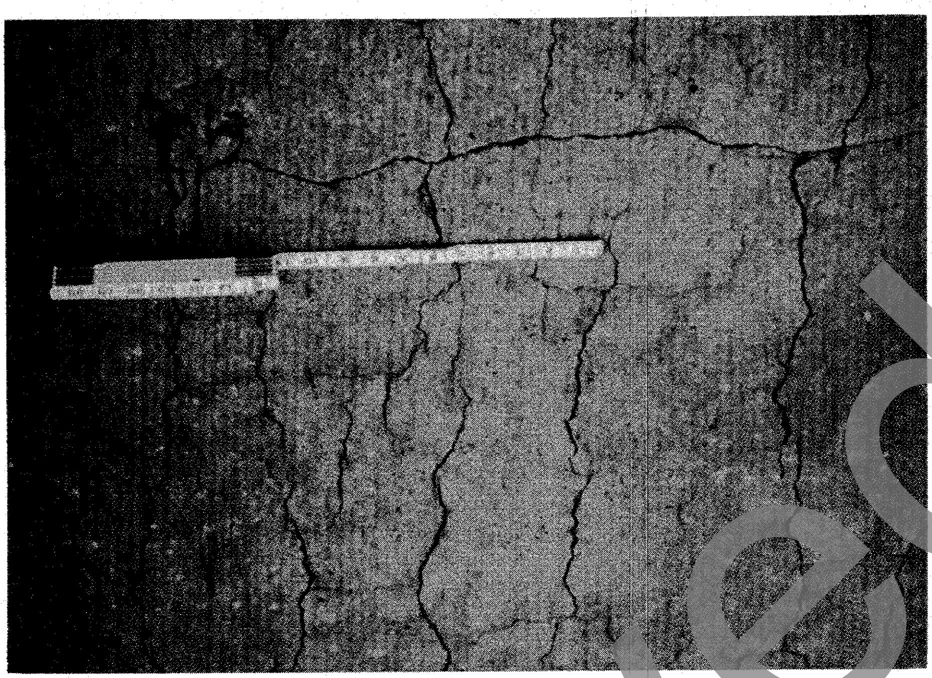


Fig 2-5 Closeup of cracking due to ASR in continuously reinforced pavement. Longitudinal cracks in pavement run top to bottom in photo.

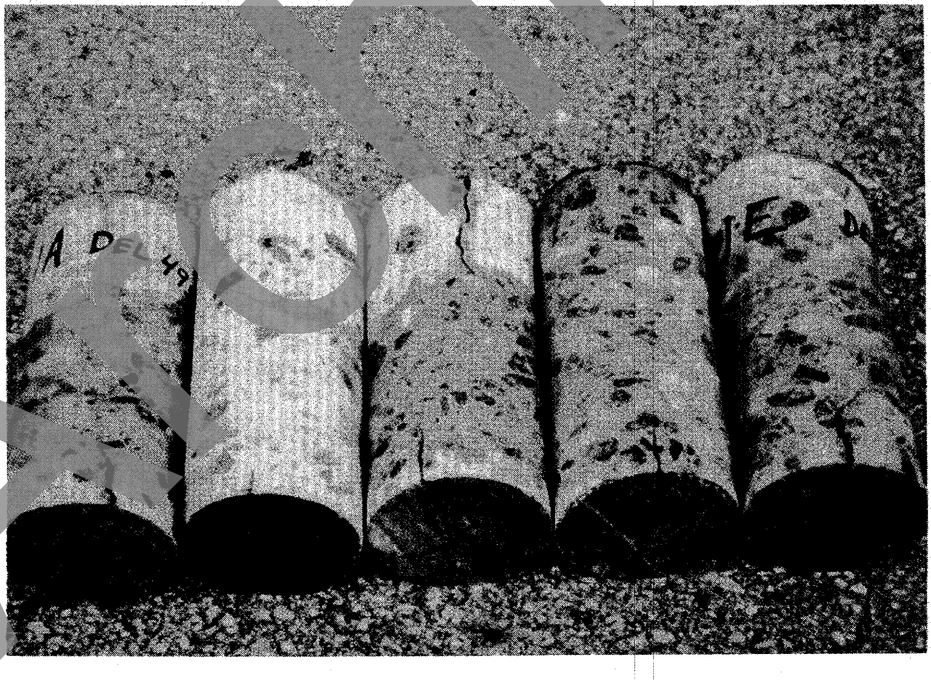


Fig 2-6 Cores, showing cracking due to ASR in continuously reinforced pavement shown above.

SEVERITY OF DISTRESS DUE TO ASR DEPENDS ON:

1. **Extent of the reaction**
2. **Composition of the ASR gel**
3. **Restraint to expansion**

Fig 2-7 Factors determining severity of distress due to ASR.

CRACK PATTERN DUE TO ASR DEPENDS ON:

1. Geometry and restraint
2. Temperature and drying
3. Loading (traffic, etc.)
4. Crack propagation

Fig 2-8 Factors influencing the nature of the crack pattern due to ASR.

REQUIREMENTS FOR EXPANSIVE ASR

1. Reactive silica
2. Sufficient alkali
3. Availability of moisture

Fig 2-9: Three requirements for ASR.

REACTION STEPS IN ASR

Step 1.

Alkali + Silica = Gel reaction product

Step 2.

Reaction product + Water = Expansion

Fig 2-10 Reaction steps in ASR.

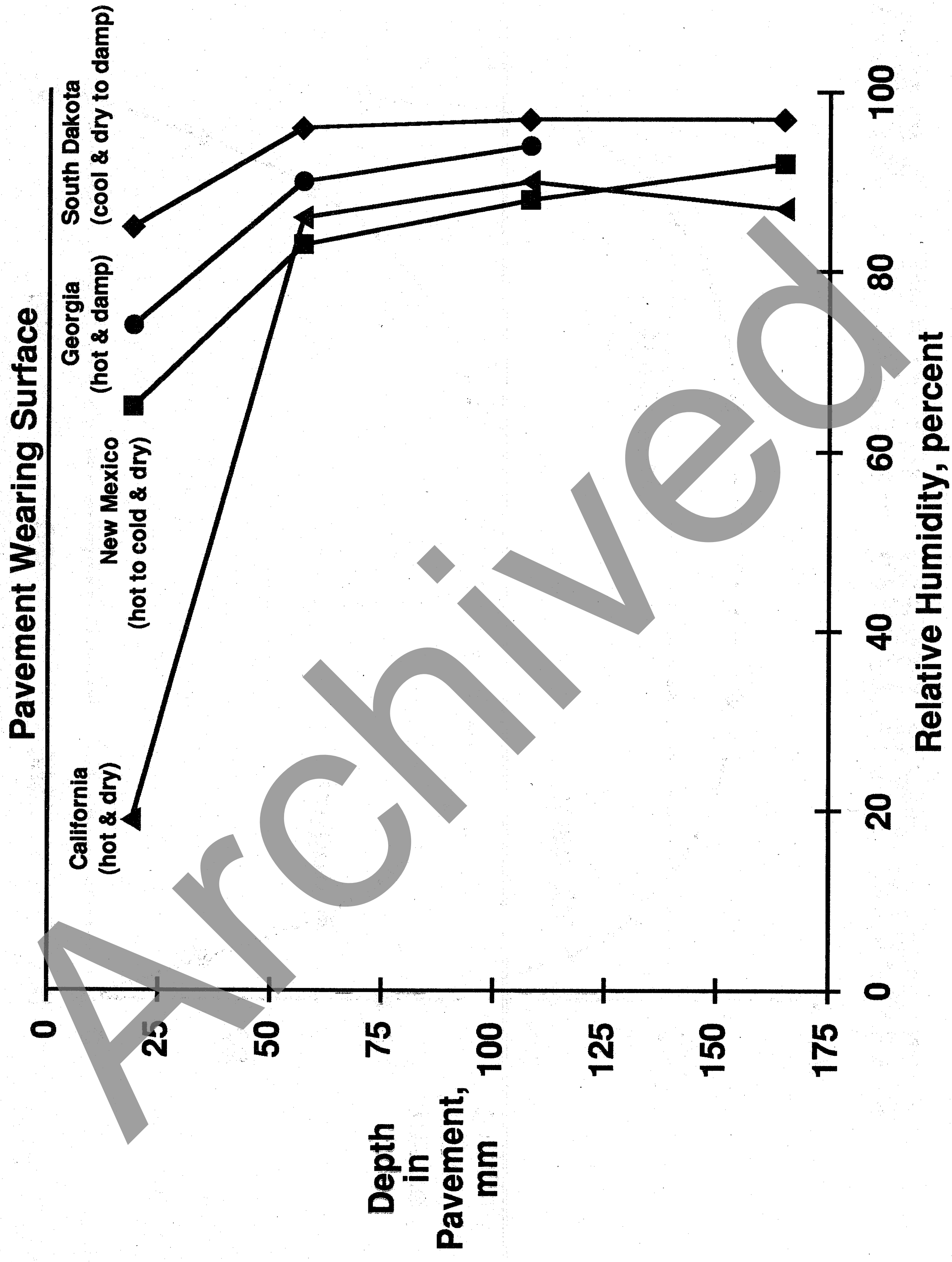


Figure 2-11 Relative Humidity of pavement concrete in different states and climatic conditions.

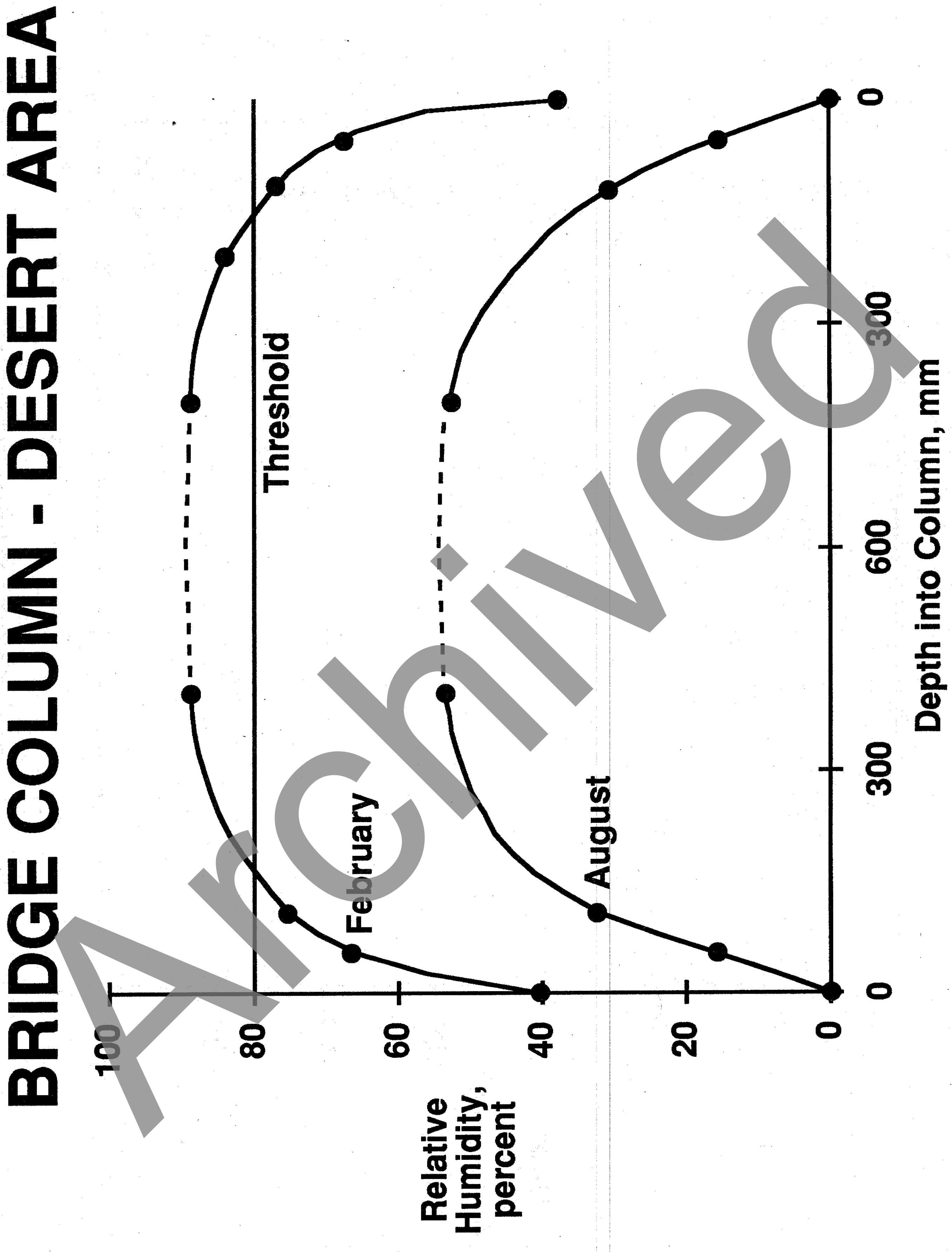


Fig 2-12 Relative humidity in bridge column located in hot desert area in California.

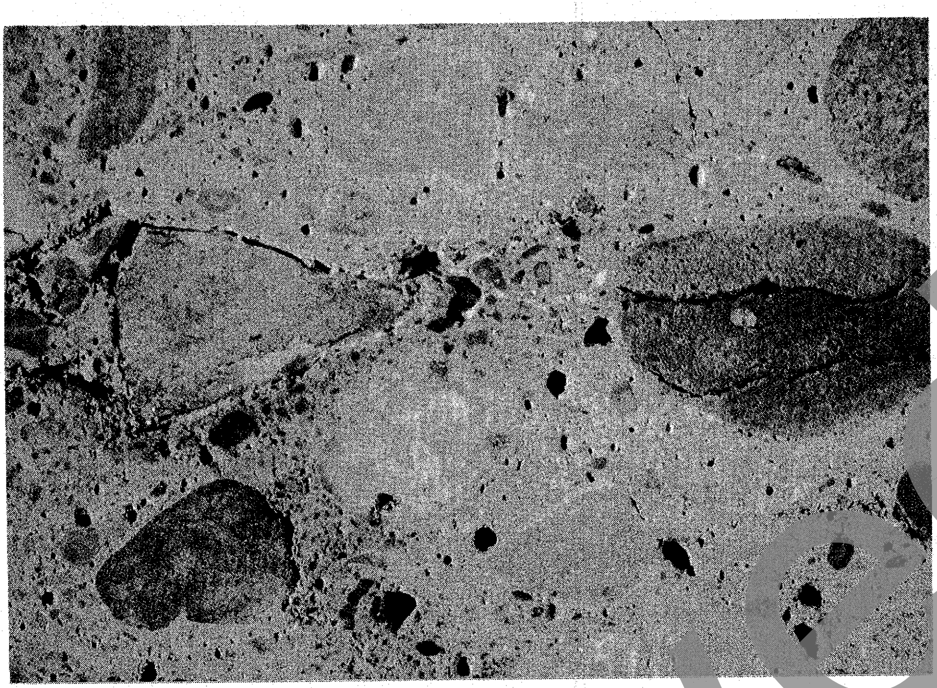


Fig 2-13 Sawed surface of concrete viewed in ordinary light.

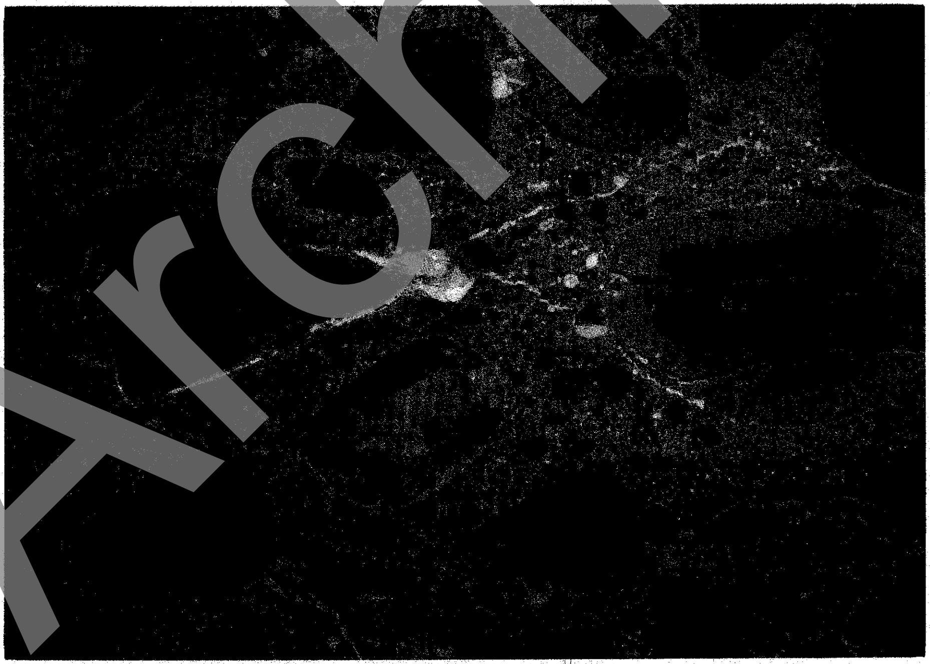


Fig 2-14 View of same surface as above, as seen in UV light after uranyl acetate treatment. Yellowish-green areas are deposits containing ASR gel.

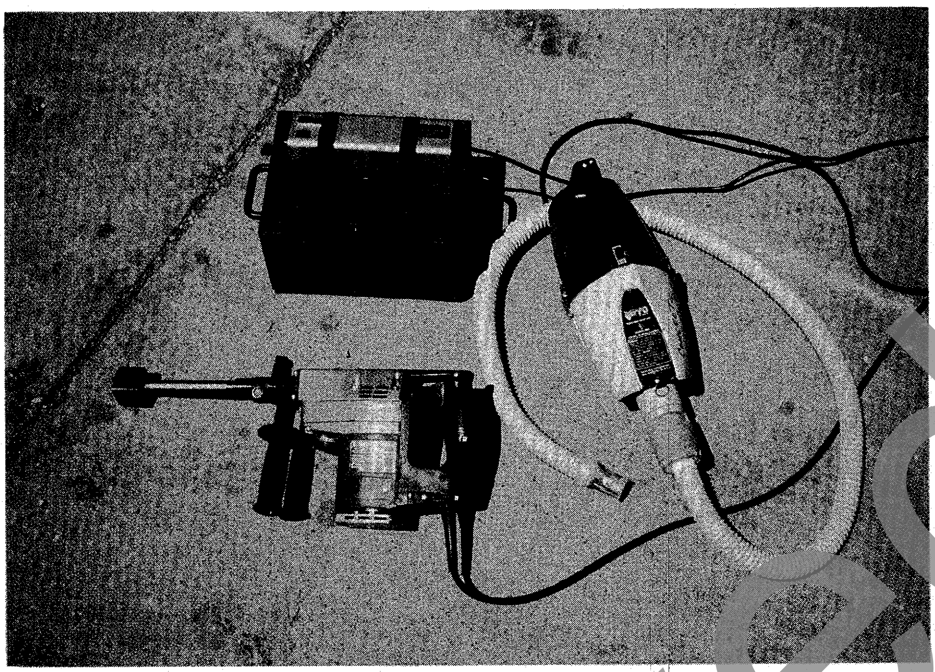


Fig 2-15 Impact drill, UV light-viewing box, and vacuum used in preparing and removing test surfaces. Drill is fitted with bushhammer.

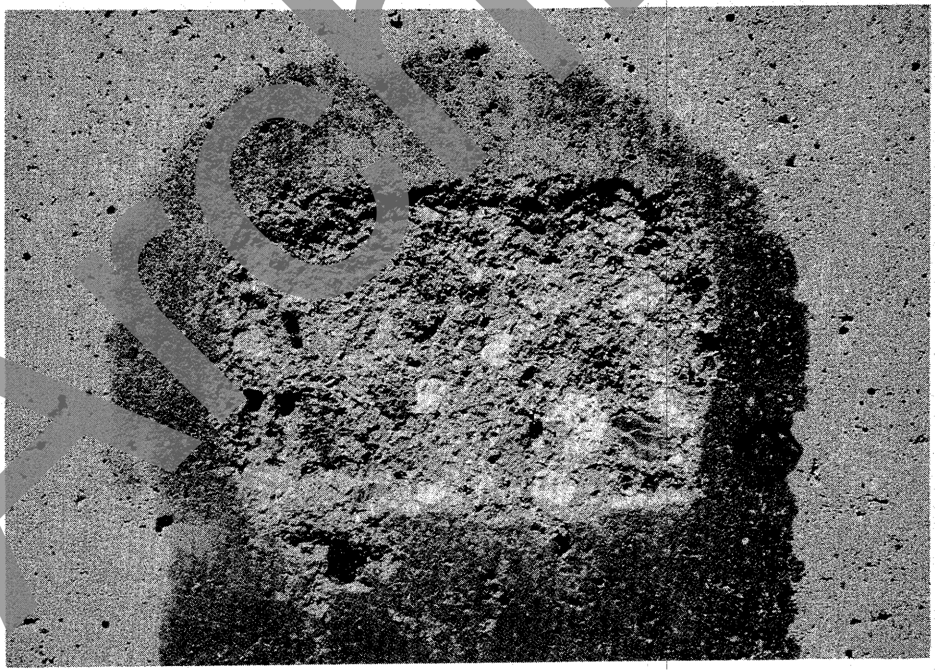


Fig 2-16 Bushhammered surface rinsed and ready for uranyl acetate application.

Fig 2-17 Drops of uranyl acetate solution being applied to vertical surface using plastic squeeze bottle. Note cloth held against concrete bushhammered surface to absorb excess solution.

Fig 2-18 Bushhammering treated surface to remove uranyl acetate contaminated layer. Note hose attached to vacuum to collect powder and chips generated by bushhammering.

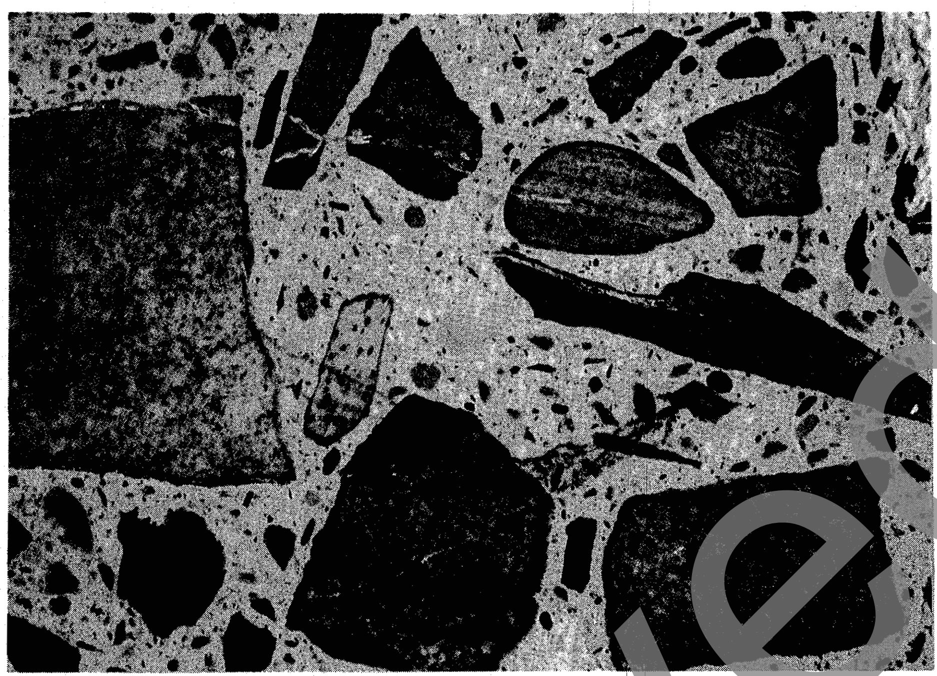


Fig 2-19 Lapped surface of concrete showing reaction rims, microcracks, and ASR gel in phyllite aggregate particles, (1.5x).

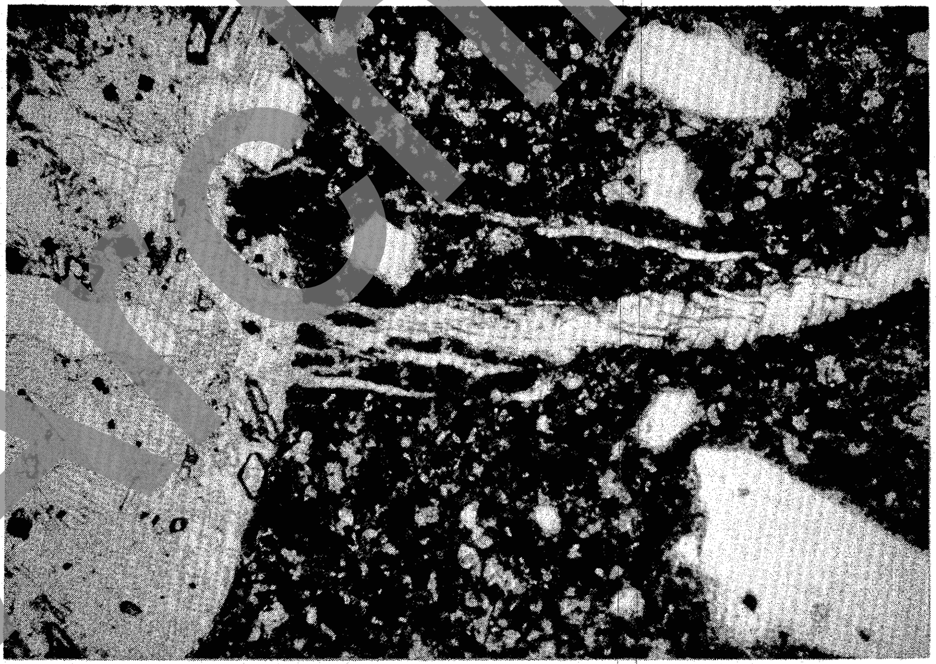


Fig 2-20 Thin section of concrete showing microcrack extending from volcanic aggregate particle (left) into cement paste matrix. Yellow fluorescent filling in crack is ASR gel, (100x).

Fig 2-21 ASR gel in powder mount in petrographic microscope. Particles are isotropic and have conchoidal fracture, (100x).

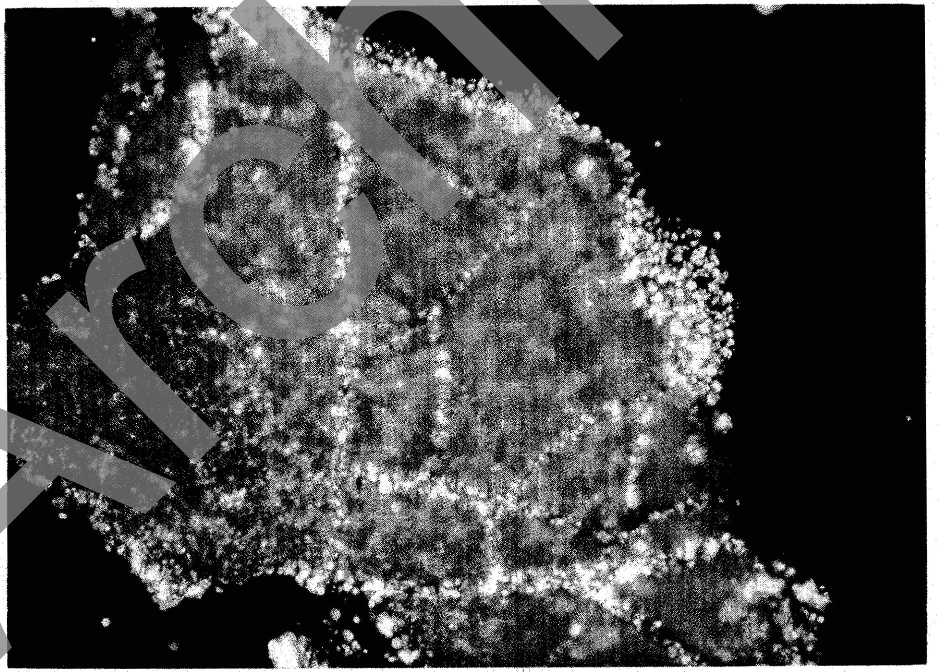


Fig 2-22 Partially crystallized ASR gel in powder mount in petrographic microscope, (100x).

GLOSSARY OF TERMS

Alkali — Sodium and potassium occurring in constituents of concrete and mortar, usually expressed in chemical analyses as the oxides Na_2O and K_2O.

Alkali-aggregate reaction — Chemical reaction in either mortar or concrete between alkalies (sodium and potassium) from portland cement or other sources and certain constituents of some aggregates; under certain conditions, deleterious expansion of the concrete or mortar may result.

Alkali-carbonate reaction — The reaction between the alkalies (sodium and potassium) in portland cement and certain carbonate rocks, particularly certain calcitic dolomite and dolomitic limestones, present in some aggregates; the process and products of the reaction may cause abnormal expansion and cracking of concrete in service.

Alkali-silica reaction — The reaction between the alkalies (sodium and potassium) in portland cement and certain siliceous rocks or minerals, such as opaline chert, strained quartz, and acidic volcanic glass, present in some aggregates. The products of the reaction can cause expansion of the concrete.

Anisotropic — In optical mineralogy, the ability of a material to doubly refract light. Characteristic of all nonopaque crystalline substances, except those belonging to the isometric system, which are isotropic.

Becke line — A line of high-intensity light formed around a translucent (or transparent) particle mounted, for example, in a refractive-index liquid on a glass microscope slide, and illuminated from below, due to concentration by split reflection or refraction.

Calcite — A mineral having the composition calcium carbonate ($CaCO_3$) and a specific crystal structure; the principal constituent of limestone, chalk, and marble; used as a major constituent in the manufacture of portland cement.

Carbonation — Reaction between carbon dioxide and a hydroxide to form a carbonate, especially in cement paste, mortar, or concrete. The reaction with calcium compounds produces calcium carbonate.

} entire cylce
carbon monoxide

Cement, low-alkali — A portland cement that contains a relatively small amount of sodium or potassium or both; in the United States a cement containing not more than 0.6 percent Na_2O equivalent, i.e., percent Na_2O + (0.658 x percent K_2O).

Chert — A very fine grained siliceous rock characterized by hardness and conchoidal fracture in dense varieties, the fracture becoming splintery and the hardness decreasing in porous varieties, and in a variety of colors; it is composed of silica in the form of chalcedony, cryptocrystalline or microcrystalline quartz, or opal, or combinations of any of these.

Ettringite — A mineral, high sulfate calcium sulfoaluminate ($3\ CaO \cdot Al_2O_3 \cdot 3\ CaSO_4 \cdot 30\text{-}32\ H_2O$) occurring in nature or formed as a cement hydration product or by sulfate attack on mortar and concrete; the product of the principal expansion-producing reaction in expansive cements; designated as "cement bacillus" in older literature.

Fly ash — The finely divided residue resulting from the combustion of ground or powdered coal and which is transported from the firebox through the boiler by flue gases.

Fluorescence — The emission of visible light by a substance exposed to ultraviolet light.

Fluorescence microscopy — That branch of microscopy utilizing ultraviolet (or near ultraviolet) light, producing fluorescence, thus facilitating microscopical observations.

Gel — In alkali-silica reaction, the clear to white gelatinous reaction product of variable composition, largely SiO_2, CaO, and alkalies.

Granulated blast-furnace slag — The glassy, granular material formed when molten blast-furnace slag is rapidly chilled, as by immersion in water.

Igneous rocks — Rocks formed from a molten state.

Isotropic — In optical mineralogy, those minerals that only singly refract transmitted light; the property of a substance to transmit light with equal velocity in all crystallographic directions (opposite of anisotropic).

Lithium — An element of the alkali metal group that is the lightest metal known and never found uncombined in nature. It is not considered an alkali in cement and concrete terminology.

Map cracking — 1) Intersecting cracks that extend below the surface of hardened concrete; caused by shrinkage of the drying surface of concrete which is restrained by concrete at greater depths where either little or no shrinkage occurs; vary in width from fine and barely visible, to open and well-defined. 2) The chief symptom of alkali aggregate reaction between alkalies in cement and mineral constituents in aggregate within hardened concrete; due to differential rate of volume change in different portions of the concrete; cracking is usually random and can be on a fairly large scale, and in severe instances the cracks may reach a width of 0.50 in.

Mohs scale — Arbitrary (quantitative) units, ranging from 1 through 10, by means of which the scratch hardness of a mineral is determined; each unit of hardness is represented by a mineral that can scratch any other mineral having a lower-ranking number; the minerals are ranked from talc or 1 (the softest), upward through gypsum or 2, calcite or 3, fluorite or 4, apatite or 5, orthoclase or 6, quartz or 7, topaz or 8, corundum or 9, and diamond or 10 (the hardest).

Metamorphic rocks — A rock which has been altered by heat or intense pressure, causing new minerals and structures to be formed.

Natural pozzolan — Either a raw or calcined natural material that has pozzolanic properties (e.g., volcanic ash or pumice, opaline chert and shales, tuffs, and some diatomaceous earths).

Opal — A mineral composed of amorphous hydrous silica ($SiO_2 \cdot H_2O$).

Opaline chert — Chert containing a major portion of opal.

Petrography — The branch of petrology dealing with description and systematic classification of rocks aside from their geologic relations, mainly by laboratory methods, largely chemical and microscopical; also, loosely, petrology or lithology.

Petrology — The science of rocks, treating of their origin, structure, composition, etc., from all aspects and in all relations. (See also **Petrography**.)

Polarized-light microscope — A microscope producing plane-polarized light, that is, light that vibrates in only one plane.

Powder mount (Immersion Mount) — Particles embedded or immersed in a liquid (or other material such as hardened epoxy resin on a glass microscope slide; a thin cover glass is placed on top of the particles.

Pozzolan — A siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties.

Reactive aggregate — Aggregate containing substances capable of reacting chemically with the alkalies and hydroxyl ion in the pore solution of portland cement concrete or mortar under ordinary conditions or exposure, resulting in some cases in harmful expansion, cracking, or staining.

Refraction — A change of direction of a ray of light (or other radiation) when it passes from one medium to another of different optical density.

Refractive index — Ratio of the speed of light in air to that in the substance.

Sedimentary rocks — Rocks formed as a result of accumulation of sediment in water or air; the sediment may be particulate (as in sand grains or clay) or a chemical (or biochemical) precipitate.

Silica fume — Very fine noncrystalline silica produced by electric arc furnaces as a byproduct of the production of metallic silicon or ferrosilicon alloys; also known as **Condensed silica fume**.

Thin section — A thin translucent slice of a substance (rock, mineral, bone, concrete, etc.) mounted beneath a cover slip on a glass microscope slide.

Ultraviolet light — Radiation beyond the visible spectrum at its violet end; having a wavelength shorter than those of visible light and longer than those of X-rays.

Uranyl acetate solution, $UO_2(C_2H_3O_2)_2 \cdot 2H_2O$ — A solution containing uranium acetate that is applied to concrete to detect possible alkali-silica gel reaction products.

X-ray diffraction — A characteristic reflection and refraction of X-rays by substances having a regular arrangement of atoms; a phenomenon used to identify substances having such structure.

X-ray fluorescence — Characteristic secondary radiation emitted by an element as a result of excitation by X-rays, used to yield chemical analysis of a sample.

Appendix V

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Appendix W

GUIDE TO

ALKALI-AGGREGATE REACTIVITY

DEVELOPED BY

MID-ATLANTIC REGIONAL TECHNICAL COMMITTEE

June 1993

Copies of this publication can be obtained by writing to:

Mid-Atlantic Regional Technical Committee
900 Spring Street
Silver Spring, MD 20910

Foreword

Mid-Atlantic Regional Technical Committee

Even though alkali silica reactivity was first recognized in the late 1930s as a problem causing concrete deterioration, it was not identified in the Mid-Atlantic Region until 1960. In 1980, some specifiers began to specify low alkali cement for concrete structures. Due to the relatively few low alkali cements that were available in our area, the Maryland Aggregates and the DC Ready Mixed Technical Committee organized an Alkali Silica Seminar which was held on January 12, 1990. About 120 people attended that seminar. As a result of this seminar, the first Mid Atlantic Regional Technical Committee meeting was held on February 9, 1990 and the following objective was adopted.

Mid Atlantic Regional Technical Committee

Objective: To provide a forum for specifying agencies, concrete materials specifiers, and producers to meet and discuss problems of mutual interest related to concrete construction and develop guide specifications and practices which address these concerns in a mutually satisfactory manner. The first issue which the group will address is that of alkali-silica reactivity. Other topics may be proposed for consideration by any interested party.

For purposes of this document, the Mid-Atlantic Region includes the states from Pennsylvania to South Carolina. The Committee consists of materials suppliers from Pennsylvania to South Carolina; State Department of Transportation representatives from Maryland, Delaware, Pennsylvania, Virginia, District of Columbia, and North Carolina. There has also been support from the Federal Highway Administration. The focus of the Committee is to develop information about concrete characteristics that are unique to the Mid-Atlantic Region. To date, alkali-silica reactivity has been the principal concern of the Committee; however, other items may be discussed by the Committee.

All meetings have been open to any interested party. In 1990, meetings were held monthly and Committee members have had an opportunity to share technical information and learn together. Field trips were made to inspect structures affected by the alkali-silica reaction.

We have also been fortunate to be able to have presentations from international experts and researchers such as Doug Hooton, Dave Stark, Chris Rogers, Jan Skalny, and Muhammad Bhatty.

The Committee has developed a "Guide Specification" and a "Guide to Alkali-Silica Reactivity." These documents represent the committee's concept of the present state of the knowledge. It is apparent that there are certain characteristics of alkali-silica reactivity that are unique to this region. In

What are these?

developing this material, the Committee focused on limiting this to alkali-aggregate reactivity in the Mid-Atlantic Region.

This "Guide" is designed to be used by knowledgeable parties to develop a better understanding of how alkali-aggregate reactivity affects concrete structures. The "Guide" also serves as a resource document to the "Guide" Specification. It is hoped that with the use of this "Guide," the reader will be better prepared to comprehend and control alkali-aggregate reactivity in concrete structures.

Task Group Members	Company Name
Steve Lane, Chairman	VA Trans. Research Council
Dave Stokes	DE Dept of Transportation
John D. Marchant	U. S. Ash Corporation
Joseph Mazzarella	ESSROC Materials, Inc.
Patrick Reardon, Jr.	Northeast Cement Shippers Assn.
James W. Yingling	Genstar Stone Products

Member Name	Company Name
Bailey, Bill	Virginia DOT
Barbour, Ronald	U. S. Ash Corporation
Bhatty, Muhammad	Capitol Cement Corporation
Brooks, Paul D.	Independent Cement Corporation
Call, B. M.	Roanoke Cement
Chua, Teck	Virginia Concrete Co.
Cook, Russell	Lehigh Portland Cement Co.
Diedrich, Joseph E.	Dixie Cement Company
Dunne, Jr., James P.	FHWA-Region 3
Eichelberger, Terry	Laurel Sand & Gravel Inc.
Ellis, Ed	MONEX Resources
Figliola, Anthony A.	A. G. Kurtz & Sons
Forster, Stephen	Federal Highway Administration
Gapinski, Gary	Scan Cem Industries
Hancock, James	Virginia DOT
Heckel, Ronald	Genstar Stone Products
Hergert, David	Mays Landing Sand & Gravel Co.
Hooton, Doug	University of Toronto
Howe, Richard	Consultant
Kalcheff, Ignat V.	D. C. Dept of Public Works
Kalendek, David	Maryland SHA
Karasopoulos, Theodore H.	Maine DOT
Koss, George	J. T. M. Industries
Lamond, Joseph F.	Consultant
Mackow, Richard	J. T. M. Industries
McCall, W. Calvin	Blue Circle Cement, Inc.
Neal, Robert	Lehigh Portland Cement Co.
O'Brien, William	ESSROC Materials, Inc.
Peters, Fran	Capitol Cement Corporation
Pryor, C. A.	National Stone Association
Rose, Jere	Blue Circle Atlantic
Skalny, Jan	Consultant
Steel, Richard	Virginia DOT
Stokes, David	Delaware DOT
Thomson, Margaret	Pennsylvania DOT
Tyson, Sam	American Coal Ash Assn.
Weber, Jack	Jack Weber Associates
Weisner, John	Maryland DOT
Wherely, David	The Arundel Corporation
Woolwine, Jr., Sam	W. W. Boxley Co.

GUIDE ON ALKALI-AGGREGATE REACTIVITY

MID-ATLANTIC REGIONAL CONCRETE TECHNICAL COMMITTEE

PREFACE

This guide presents information on the phenomenon of alkali-aggregate reactivity (AAR) in hydraulic cement concrete. It will present simple, practical, and well-established approaches for dealing with AAR. Separate chapters are devoted to the mechanism of alkali-silica reactivity (ASR) and alkali-carbonate reactivity (ACR); the identification of ASR and ACR in structures; test methods for evaluating aggregates, aggregate-cement combinations and aggregate-cement-pozzolan combinations; and procedures for minimizing the potential for ASR or ACR distress in concrete. A selected list of references is included.

1.0 INTRODUCTION:

1.1 ***History:*** The phenomenon of alkali-silica reactivity was first recognized in the late 1930s when the California Division of Highways (now CALTRANS) investigated abnormal cracking of a number of concrete structures in Southern California. The director of that agency, Thomas E. Stanton, wrote an article for the February 1, 1940, issue of Engineering News-Record that gave the first correct description of ASR.

The phenomenon of alkali-carbonate reactivity was first identified in 1956 by E.G. Swenson of the Division of Building Research, National Research Council of Canada. In Highway Research Record 45 (TRB, 1964), Sherwood and Newlon report on the occurrence of alkali-carbonate reactive aggregates in Virginia.

1.2 ***Background:*** Portland cement concrete is the most versatile and widely used construction material. Virtually any structure, for which a form can be built, can be constructed of concrete. Concrete provides strength, durability, fire resistance, and thermal properties that are utilized by architects and engineers in the design and construction of residential, commercial, and industrial structures worldwide. These structures can provide a service life free of significant deterioration when the proper combinations and proportions of concrete materials are specified and used, and when proper construction practices are followed.

Chemical reaction of aggregates and alkalies in concrete can result in deterioration and reduced service life of concrete. This reaction involves certain mineral constituents of the aggregates and soluble alkali compounds which significantly increase the pH of the pore solution in the concrete.

In order for this reaction to take place, the concrete must be subjected to sufficient moisture in service. The alkaline components are usually associated with alkalies in portland cement, but may also be contributed by the aggregates and admixtures in concrete. External sources such as deicing chemicals or cleaning compounds may also supply alkalies.

Alkali-aggregate reactivity was previously thought to occur in isolated instances and in limited geographical areas. It was not of major concern to owners, specifiers, contractors, or material suppliers. However, in recent years the presence of AAR has been identified in areas previously thought to be AAR free. In the Mid-Atlantic Region of the United States, concretes produced from materials previously thought to be nonreactive are exhibiting classical signs of AAR.

The main purpose of this guide is to present current knowledge and experience on AAR, as well as methods for minimizing its occurrence. It is intended for use by owners and individuals responsible for the design, construction, and maintenance of concrete structures where the potential for AAR is of concern.

Additional research is needed to develop a better understanding of the mechanisms of AAR. New and/or improved test methods need to be developed to provide more accurate and rapid identification of potentially reactive aggregates, and to evaluate cement-aggregate and cement-pozzolan-aggregate combinations. Research into various aspects of ASR is an ongoing effort at many universities and governmental agencies around the world. One such project was conducted as part of the Strategic Highway Research Program (SHRP)[1]. New developments may require revisions to this guide.

1.3 ***Scope:*** This guide presents current knowledge and experience on the phenomenon of alkali-aggregate reactivity. It also addresses applicable, currently available industry-accepted test procedures and measures that are intended to reduce the occurrence of AAR in concrete. This guide was developed for use in the Mid-Atlantic Region which consists of South Carolina, North Carolina, Virginia, Maryland, Delaware, New Jersey, Pennsylvania, and the District of Columbia.

The Mid-Atlantic Region contains a number of examples of distress related to alkali-aggregate reactivity in structures and pavements. It is recognized that aggregate types and constituents will vary as aggregate deposits vary from one geographic area to another; consequently, although portions of this guide may apply to conditions in other areas, the characteristics of the particular concrete-making materials available and the existing environmental and service conditions should be evaluated to determine whether this guide is appropriate in whole or in part.

This guide is divided into parts. Part A deals with alkali-silica reactivity; Part B with alkali-carbonate reactivity.

PART A: ALKALI-SILICA REACTIVITY

2.0 MECHANISM OF THE ALKALI-SILICA REACTION

Concrete, in a basic sense, is composed of two components: aggregates and cementitious paste. The paste is composed of the products of the hydration reaction between the cementitious materials and water.

Although appearing to be solid (discounting entrained and entrapped air voids) the cementitious paste actually consists of a matrix of solid material with an internal network of interconnected pores. The volume of these pores and their interconnectedness depends on factors such as water-cement ratio, curing regime, and the cementitious materials used.

Water can enter and move through this network of pores, dissolving soluble salts of calcium and alkali metals (sodium and potassium). The dissolution of these salts increases the hydroxide ion (OH⁻) concentration or pH of the resulting pore solution. In portland cement paste, the hydration reaction produces sufficient calcium hydroxide to saturate the pore solution. In solutions saturated with calcium hydroxide, the maximum pH is about 12.5; however, increasing amounts of alkali will increase the pH. Consequently, the soluble alkali content of a concrete's cementitious paste affects the upper limit of pH or alkalinity of the pore solution.

does dissolution of calcium increase pH.

are there other things that would cause the pH to rise?

The ASR process begins with the reaction of hydroxide ions from a highly alkaline pore solution with silica in the aggregate. This reaction produces a gel which may cause the aggregate and the concrete to crack. This cracking is also believed to be associated with expansion of the gel due to water absorption. Depending on the aggregate mineralogy and reaction conditions, even very small amounts of gel may cause cracking. On the other hand, alkali-silica gel may be present in some concretes without having caused significant distress.

3.0 FACTORS AFFECTING THE POTENTIAL FOR ASR RELATED DISTRESS

In order for ASR related distress to occur in concrete, three factors are required:

1) The presence of reactive forms of silica in the aggregate
2) A sufficiently high alkali content in the concrete pore solution
3) Sufficient moisture

Other factors may affect the development of ASR related distress. Among these are: admixtures, deicing salts, mineral additives, concrete permeability, and conditions of placement and curing.

3.1 ***Reactive Silica:*** Silica, SiO_2, occurring in a variety of forms, is a common constituent of many aggregates. In neutral and acidic environments, the various forms of silica are chemically very stable. As a consequence of this stability, silica forms the bulk of the natural sands and gravels in this region. However, in highly alkaline environments, that typically occurs in concrete, the solubility of silica increases dramatically.

The reactivity of silica, in addition to being affected by the alkalinity or pH, is also affected by the form, grain size, or crystalline structure of the silica.

Descriptions of the various forms can be found in ASTM C 294. Amorphous, or noncrystalline and poorly crystalline silica, such as volcanic glasses and opal, are the most reactive forms. The high temperature minerals, tridymite and cristobalite, and chalcedony, a fibrous form occurring in some cherts, are also fairly reactive. The above forms can all be classed roughly as rapidly-reactive and typically will be classified as potentially deleterious by traditional tests such as ASTM C 227 and ASTM C 289. These rapidly-reactive forms often exhibit the pessimum effect, where the maximum expansion occurs when the aggregate contains a certain proportion of the reactive element. At proportions greater or less than the pessimum, the expansion is less. For example, an opal content in aggregate of about 5% (the pessimum amount) yields the maximum expansion. Greater or lesser amounts of opal yield lower expansions; whereas for some other minerals, maximum expansion is achieved when the aggregate consists of 100% of that mineral. With the exception of chalcedony which may be present in chert, these forms of silica are extremely rare in the natural aggregates of the Mid-Atlantic region. More slowly reacting forms of silica include metamorphically strained quartz, and microcrystalline quartz. These forms have been found to cause deleterious reactivity in concrete, although it may take longer to develop than with rapidly-reactive aggregates.

what forms of silica are found in mid Atlantic region.

Generally the aggregates in the Mid-Atlantic Region may not be classified as potentially deleterious by ASTM C 227 or ASTM C 289 but may exceed limits, indicating potential reactivity, by ASTM proposed Method P 214 (ASTM Book of Standards Vol. 04.02, 1991). These forms of silica are quite common in most of the igneous and metamorphic rocks of this region. They are a primary constituent of our quartzites, sandstones, and sand and gravel deposits. Chert, composed of microcrystalline quartz and possibly chalcedony, is a common constituent of some sands and gravels and can be found in some of the carbonate rocks. While unstrained quartz, also common in many rocks in the region, appears to be the least reactive form of silica, it has also been associated with deterioration due to ASR.[2] The greater the porosity of the aggregate due to either its natural internal structure or fractures induced by crushing, the greater the extent of the reaction due to the increased surface area of silica available for reaction.

3.2 ***Alkalies:*** The potential for ASR increases with the alkali content of the concrete. This is because the hydroxide ion concentration (alkalinity, pH) increases as the alkali content increases. Portland cement is usually the primary source of alkalies in concrete. Although these alkalies are reported as Na_2O and K_2O, the readily soluble portions in the cement generally occur as alkali sulfates. Other internal sources include mineral and chemical admixtures; some aggregates (typically glassy volcanic rocks); and mix water, especially if seawater is used. Recycled ready-mixed wash water that conforms to ASTM C 94 should not contribute significant amounts of alkali when used in normal batching amounts. Deicing salts and seawater are external sources which can supply alkalies to the pore solution.

3.3 ***Moisture:*** Water, as a constituent of the pore solution in concrete, is necessary for the alkali-silica reaction to proceed. Concrete which is allowed to dry after proper curing and which will remain dry in service is unlikely to be affected by ASR. However, concrete members with large cross-sections may retain enough residual moisture to cause reactivity even though they are in a dry environment.

Concrete which is in contact with the ground or water or is periodically wetted will most likely contain sufficient moisture to cause reactivity. Moisture in excess of 80% relative humidity in the concrete is necessary to cause the gel to swell.[3] A certain level of protection can be provided to concrete by decreasing the permeability, and thus the availability of external moisture to the system.

3.4 ***Pozzolans and Ground Granulated Blast-Furnace Slag:*** Fly ash, ground granulated blast furnace slag (GGBFS), and silica fume can be effective in preventing deleterious expansions resulting from ASR. The primary constituent in each of these materials is amorphous or glassy silica. Because these materials are extremely fine grained, they react very rapidly with the hydroxide ions in the pore solution forming a compound similar to that formed by the hydration of portland cement. This compound fills in spaces between the hydrated cement grains reducing the permeability of the concrete. It effectively ties up the alkalies thus reducing the potential for ASR.

↗
how does it type up the alkalis?

Lower permeability inhibits the migration of ions and the ingress of alkalies, chlorides and moisture in the concrete. By slowing these processes, the potential for ASR is reduced.

3.5 ***Others:*** Chloride ions seem, in some manner, to exacerbate ASR. The most likely sources of chlorides are deicing salts, seawater and some chemical admixtures.

4.0 IDENTIFICATION OF ASR DISTRESS IN CONCRETE

There are two commonly used ways to identify the occurrence of ASR distress in a concrete structure: visible signs on the structure itself and petrographic analysis of the concrete samples from the structure. Of the two, the most reliable method is the petrographic analysis. A recently developed gel fluorescence test may be helpful in identifying possible gel deposits within the concrete.

4.1 *Outward Manifestations:* Visible signs of ASR-induced distress include expansion and exudations of gel. Signs of expansion include cracks, closing of joints, and displacement of members in severe cases. The cracks will be either longitudinal or map-type, depending on the geometry and restraint of the structure; and in severe, well-developed cases, will evolve into mostly map-type cracks. In continuously-reinforced pavements, the longitudinal nature of the cracks enables them to be easily distinguished from D-cracking in which the cracks run parallel to any restraining border. Often gel will have exuded from the cracks, and is colored from white to dark gray. The dark gray coloring comes from dirt and pollution from the environment. A very good photographic guide is the SHRP handbook by Stark[4].

4.2 ***Petrographic Analysis:*** ASTM C 856 outlines the procedures for performing a petrographic analysis of hardened concrete. This practice can be used to help answer a number of questions about the concrete being examined. With respect to ASR distress, there are two relevant questions: Is there evidence that siliceous elements in the aggregate have reacted with alkalies in the pore solution? And, is there evidence that this reaction has resulted in expansion?

In order to positively identify the occurrence of ASR in concrete, it is necessary to find evidence of the gel reaction product. The gel may be found associated with any of the elements present in concrete, filling and/or lining cracks in the aggregate and paste, coating and/or filling voids in the paste, and possibly even exuding from surface cracks in the concrete.

Depending on its composition, the gel will typically be transparent to translucent or milky white in appearance. The gel may be fluid, viscous, rubbery, waxy, or hard. As it dehydrates, the gel may exhibit drying shrinkage cracks or become powdery. Over an extended period of time the gel may begin to carbonate and crystallize under certain conditions, masking its original gel-like character.

Broken or sawn pieces of concrete can be examined for the presence of gel deposits. Material suspected of being gel can be removed and examined in grain mounts with a polarizing microscope to determine its optical characteristics. The index of refraction of alkali-silica gel is usually between 1.46 and 1.53, and it is white, yellowish or colorless[5]. Fresh gel will be isotropic, but as it begins to crystallize or carbonate, it may become slightly birefringent. The presence of gel may be detected using the gel fluorescence test described in Section 4.3, but the character of the fluorescing material should be determined petrographically. Other, more sophisticated, equipment may provide positive identification of alkali-silica gel.

When specimens are obtained, they should be wrapped in plastic film or otherwise protected from the atmosphere to maintain the moisture condition and to guard against carbonation. When observing the sample while it dries from a moist condition, areas containing gel may be inferred where the specimen is slow to dry. If the sample has dried, it can be rewetted.

Specimens to be examined for analysis of cracking should be sawed or cored, since broken pieces may contain cracks induced by the breaking process. A detailed crack analysis can be performed on finely lapped surfaces, using a stereoscopic microscope. Areas determined to warrant more detailed analysis can be thin-sectioned for examination using a polarizing microscope. Thin section blanks can be epoxy-impregnated, preferably with a colored dye for ease in identifying pre-existing cracks and gel deposits. Thin section analysis will also permit a detailed mineralogic description of the aggregates. Scanning electron microscopy (SEM) is a sophisticated technique permitting a more detailed view of the microstructure and composition.

4.3 *Gel Fluorescence Test:* The gel fluorescence test[4] is a quick and useful test for helping to detect the presence of ASR gel in concrete. It involves spraying a freshly exposed surface with a solution of uranyl acetate followed by viewing under ultraviolet illumination. The uranyl ion replaces alkali ions in the gel which is then visible as brightly fluorescing areas under the ultraviolet illumination. Areas where carbonation of the paste has occurred will also fluoresce but to a lesser degree with this method and may be confused with ASR gel. It should also be noted that the presence of ASR gel in concrete is not necessarily a positive indication that ASR damage has occurred. This is one reason for cautioning that petrographic analysis yields the only final proof that ASR has caused the concrete distress. However, with proper training and practice, the method can be a very useful supplementary diagnostic tool. When using uranyl acetate, it is important to consider the requirements for proper disposal of treated samples as well as the safety of personnel during its use.

what causes the flourescence?

5.0 EVALUATION OF MATERIALS AND ENVIRONMENT

5.1 *Aggregates:* There are several methods for evaluating the potential alkali-silica reactivity of an aggregate. They include:

ASTM C295 Guide for Petrographic Examination of Aggregates for Concrete

ASTM C289 Test Method for Potential Reactivity of Aggregates (Chemical Method)

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

ASTM P214 Proposed Test Method for Accelerated Detection of Potentially Deleterious Expansion of Mortar Bars Due to Alkali-Silica Reaction (1990,1991)

Whenever possible, the results of the above tests should be used in conjunction with information obtained from the service record of the aggregates over a period of years in similar concrete exposed to comparable service conditions.

5.1.1 Section 3.1 of ASTM C295 notes that the specific procedures employed in the petrographic examination of a sample will depend upon the purpose of the examination.

In the case of investigations related to ASR, it is important to identify the presence and percentage of potentially reactive constituents in the sample. Suggestions on the maximum percentage of a particular reactive constituent in the aggregate sample necessary to consider it potentially reactive can be found in various sources[6,7]. However, such suggestions often do not adequately address the forms of reactive silica most likely to be encountered in the Mid-Atlantic region. These forms are microcrystalline, strained, or microfractured quartz and may be present in a variety of rock types and sand and gravel deposits. For assessing the ASR potential of aggregates, the Mid-Atlantic Regional Technical Committee ASR Specification contains the following limits:

CONSTITUENT	LIMIT
Optically strained, microfractured or microcrystalline quartz	5 %
Chert or chalcedony	3 %
Tridymite or cristobalite	1 %
Opal	0.5 %
Volcanic glass	3 %

Attention must be paid to proper sampling of aggregate sources so that the sample is representative of the material to be supplied for the production of portland cement concrete. See ASTM D75 for additional guidance on proper sampling procedures.

5.1.2 ASTM C289 is a quick (two-day) test in which crushed aggregate is treated with hot NaOH solution for a day and the reduction in alkalinity of the solution is plotted on a graph versus the dissolved silica in the solution. Figure 2 of C289 delineates areas of reactive, potentially reactive, and innocuous materials based on service records. Unfortunately, the areas described in this figure <u>do not</u> properly identify many of the reactive aggregates in this region.

5.1.3 ASTM C227 involves the making of mortar bars and storing them at 100°F and 100% relative humidity for three months or longer and measuring the linear expansion. Expansion criteria for evaluating the reactivity of aggregates are given in the appendix to ASTM C33, Specification for Concrete Aggregates. In addition to the limitations listed in Section 3.3 of C227, the major drawback is that many of the aggregates believed to be reactive in service in this region have not expanded significantly under the test conditions.

5.1.4 ASTM P214 (V. 04.02, 1991) is a rapid test (16 days) which utilizes mortar bars stored at an elevated temperature (176°F) in a highly alkaline solution. P214 lists criteria for judging reactivity of the aggregates, suggesting that expansions greater than 0.20% after 16 days (14 days in the solution) indicate reactivity, and expansions less than 0.10% indicate a non-reactive aggregate. Recent experience in testing aggregates from the Mid-Atlantic region indicates that the expansion limit in P 214 should be lower than 0.20% at 16 days. The Mid-Atlantic Regional Technical Committee Specification uses an expansion limit of 0.10% to classify aggregates as reactive. However, this limit may not detect all reactive aggregates, and there have been suggestions of lower limits.[1]

5.1.5 Field service records can provide significant information for use in evaluating the potential performance of an aggregate. In evaluating field service records, important considerations are: how similar is the aggregate in the field concrete to the aggregate proposed for use; alkali content of cement; other sources of alkalies; and environmental factors affecting the moisture content of the concrete.

Unfortunately, field records often will be less than complete with respect to these items or will differ in some or many respects from the service conditions for which the aggregate is being evaluated. Consequently, the information they provide must be used judiciously in conjunction with the other information available about the aggregate in question, the other materials involved, and the intended service conditions in evaluating the potential performance of the aggregate.

5.2 *Cement*

5.2.1 Portland cement is a hydraulic cement consisting essentially of calcium silicates with calcium sulfate as an interground addition. ASTM C150 is the standard specification for portland cement and lists eight distinct types.

Alkalies are present in varying amounts in all portland cements, primarily in readily soluble sulfate compounds. In cement chemical reports, the alkali content is calculated on an oxide basis and reported as Na_2O equivalent. A limit of 0.60% Na_2O equivalent is listed in the optional chemical requirements of C150 for cement used with aggregate which could be potentially deleteriously reactive. However, some research indicates that the 0.60% limitation on the alkali content of the cement may not always provide protection from ASR. Several factors may play a role in such cases: (a) alkalies are concentrated in the concrete due to cyclical wetting and drying; (b) alkalies are contributed from other ingredients; (c) alkalies are contributed from external sources; (d) high cement content.

5.2.2 Blended cements are produced by uniformly and intimately blending one or more types of fine materials with portland cement. The blending materials may be fly ash, silica fume or other pozzolans, or ground granulated blast-furnace slag. ASTM C595 is the Standard Specification for Blended Hydraulic Cements. ASTM C595 includes an optional mortar expansion requirement using test method C 441 to evaluate its effectiveness in preventing expansions due to ASR. Some problems have been recognized with this procedure and further information is provided in Section 5.7.1 of this guide. The use of a blended cement is an attractive option to a concrete supplier with limited bin storage space.

5.3 *Pozzolans:* ASTM C618 defines a pozzolan as a siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties. Requirements are included in C 618 regarding the evaluation of the effectiveness of a pozzolan in preventing ASR. However, certain problems have been recognized and further information is provided in

Section 5.7.1.

5.3.1 Fly ash is the finely divided residue from the combustion of ground or powdered coal. ASTM C618 lists two types of fly ash, Class F and Class C. The predominate fly ash available in this region is Class F fly ash, which is a by-product of burning anthracite or bituminous coals. It has a low calcium content and is available throughout the United States. Class C fly ash, a by-product of burning lignite or subbituminous coal, is common in the mid-western and western states, and is both pozzolanic and cementitious. Some Class C fly ash may contain more than 10% lime.

Class F fly ashes generally have a lesser amount of available alkalies and contain larger amounts of silica and alumina which make them more effective than Class C fly ash in minimizing the alkali-silica reaction. Most Class C fly ashes will require significantly higher replacement levels than Class F fly ashes to be effective in reducing expansion caused by ASR. When used in lesser amounts, higher expansions may occur than if the Class C fly ash had not been used; ie., a pessimum effect may be observed.

5.3.2 Natural pozzolans (ASTM C618, Class N) are raw or calcined shales, opaline cherts, volcanic ashes, tuffs, and pumicites. Normally, natural pozzolans are not available in the Mid-Atlantic Region.

5.3.3 Silica fume, also referred to as microsilica or condensed silica fume, is a byproduct of the production of silicon or ferrosilicon alloys in an electric arc furnace. It is essentially silicon dioxide in an extremely fine, noncrystalline form and is used as a cement replacement in high strength and/or low permeability concrete. In slurried or densified powder form it is effective in reducing expansions caused by ASR in concrete. There is some concern, however, that in pelletized form, silica fume may behave as a highly reactive aggregate if the pellet is not completely broken down during mixing.

is this true? why doesn't sf cause ASR

5.4 Ground granulated blast-furnace slag (GGBFS) is a cementitious and pozzolanic material having its own hydraulic cementing properties. GGBFS consists essentially of calcium silicates and calcium aluminates. "Standard Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortars," ASTM C989, classifies GGBFS into three grades based on their performance in the slag activity test: Grades 80, 100, and 120. Because of the strength reduction at 28 days with Grade 80 GGBFS, only Grades 100 or 120 should be used as a cement replacement to reduce ASR. Requirements are included in C 989 regarding the evaluation of the effectiveness of GGBFS in preventing ASR. Problems have been recognized and further information is provided in Section 5.7.1.

5.5 ***Chemical Admixtures:*** Commonly used admixtures have not been identified as being a significant contributor to ASR. However, some evidence exists that chlorides ions can increase the potential for expansion of ASR gel. Tests using one to two percent $CaCl_2$ by weight of cement increased expansions by as much as 30% for certain cement-aggregate combinations.[8]

ASTM C1017 does include a note that admixtures containing relatively large amounts of total alkalies may contribute to the alkali-silica reaction. Compliance with the requirements of the specification does not assure acceptability when used with potentially alkali-reactive aggregates and some cements.

5.6 Most approaches for avoiding ASR focus on the materials used to produce concrete. However, the environmental conditions of service should not be ignored. ASR needs at least an 80% relative humidity in the concrete to develop. Concretes exposed to the elements or in contact with the ground, such as pavements, sidewalks, bridge decks, and retaining walls typically exceed this level. Most interior concretes of limited cross-sectional area will not retain sufficient moisture to support deleterious ASR. Exposure to external sources of alkalies or chlorides such as deicing salts or seawater may constitute a severe service environment.

5.7 Testing and Evaluation of Materials: Care should be taken that each material considered for use is tested and evaluated in accordance with the appropriate ASTM Standard by a laboratory fully qualified to do the work.

5.7.1 ASTM C441 is a test method for evaluating the effectiveness of mineral admixtures in reducing expansions caused by ASR. The procedure is similar to C227 but uses pyrex glass as a standard reactive aggregate and expansions are measured at 14 days. ASTM C618 and C 989 use a criteria of a maximum 0.02% expansion at 14 days for qualifying pozzolans and GGBFS. It is recommended that this limit not be used because of difficulties in reducing expansions to or below this value. A suggested alternative is to determine the expansion of a control mixture using a low-alkali cement. Test a second mix with the job cement and pozzolan and compare the expansion. The mixture being tested should have an expansion equal to or less than the control mixture to be considered as effective as the control mixture in preventing expansion due to ASR (see Section 5.2.1).

Some researchers have also proposed using the P 214 method to test the effectiveness of mineral admixtures[9,1]. The mineral admixture to be tested is used to replace a portion of the cement in the P 214 mortar using the proposed aggregate. Bars are fabricated and tested according to P 214. Stark[1] proposes that expansions of 0.08% or less at 16 days be considered to indicate that the mineral admixture is effective in controlling ASR.

6.0 MINIMIZING POTENTIAL FOR ASR-RELATED DISTRESS

When it has been determined that an aggregate source is to be considered reactive, through either laboratory analysis or field performance records, and the service conditions for the proposed concrete structure are conducive for deleterious ASR to occur, a number of alternative approaches are available to reduce ASR potential.

Currently recognized options are:

6.1 Use a blended hydraulic cement (Section 5.2.2). The use of a blended hydraulic cement (Type IS or IP) is believed to be an effective measure in controlling deleterious ASR expansion in concrete. Adjustments to mix proportions or curing regime may be necessary to achieve desired setting and strength gain characteristics during cold weather. If accelerating admixtures are used, it is recommended that they be ASTM C 494 non-chloride type.

6.2 Use portland cement and fly ash (Section 5.3.1). The use of Class F fly ash constituting at least 15% to 25% by weight of the total cementitious content of concrete is believed to be an effective measure in controlling deleterious ASR expansion. Class C fly ash may need to be a higher percentage of the total cementitious material and in some cases may not be effective. Adjustments to mix proportions or curing regime may be necessary to achieve desired setting and strength gain characteristics during cold weather. If accelerating admixtures are used, it is recommended that they be ASTM C 494 non-chloride type. Segregated bin storage space will be necessary for fly ash utilization.

6.3 Use portland cement and GGBFS (Section 5.4). The use of GGBFS constituting at least 25% by weight of the total cementitious content is believed to be an effective measure in controlling deleterious ASR expansion. Adjustments to mix proportions or curing regime may be necessary to achieve desired setting and strength gain characteristics during cold weather. If accelerating admixtures are used, it is recommended that they be ASTM C 494 non-chloride type. Additional segregated bin space will be required for GGBFS.

6.4 Use portland cement and silica fume (Section 5.3.3). The use of silica fume constituting at least 5% by weight of the total cementitious content is believed to be an effective measure in controlling deleterious ASR expansion. The use of silica fume will add significant costs and will require the use of a high-range water reducer. Concrete setting times may be retarded. Silica fume is generally available in slurry form for dispenser additions methods or an additional plant bin may be used for dried forms of silica fume. Curing and prevention of premature surface drying are critical in nature and likely to require special construction procedures.

6.5 Use a nonreactive aggregate (Section 5.1). While the region offers the ready mixed concrete producer a choice of aggregate sources, increased haul distance will adversely affect the cost of the aggregate. The selection of aggregate is based on lowest cost for a given level of quality. In reviewing an aggregate's service record, the user is reminded that although aggregates may be identified as being produced by a specifically named source, the character of those products may not match the materials that were produced some years earlier.

In the case of sand and gravel operations, it would not be uncommon for an ASTM C33 aggregate to be produced from a blend of feed materials from a variety of pits. As old locations are mined-out, new sites are developed.

In the production of crushed stone, the character of the rock may change at different elevations or locations in the quarry.

6.6 Use a low-alkali portland cement (Section 5.2.1). The use of a portland cement having a maximum of 0.60% total alkalies, calculated as Na_2O equivalent, can be an effective measure of protection against ASR. However, the availability of low-alkali cement in certain parts of this region is limited.

6.7 Several factors may reduce the effectiveness of any of the options outlined in 6.1 through 6.6 for preventing ASR. Among these are the heavy usage of deicing salts, marine exposures, high cement factors, and the migration of and concentration of alkalies due to repeated cycles of wetting and drying.

PART B: ALKALI-CARBONATE REACTIVITY

7.0 MECHANISM OF ALKALI-CARBONATE REACTIVITY

The expansive alkali-carbonate reaction occurs between the hydroxide ions in the alkaline pore solution in concrete and carbonate aggregates which are characterized by the presence of discreet crystals of dolomite. The reaction is believed to involve dedolomitization, producing calcium carbonate and alkali carbonate, while releasing magnesium ions which combine with hydroxide ions to form brucite. In a subsequent process, the alkali carbonate reacts with calcium hydroxide to form calcium carbonate and alkali hydroxide. Thus the alkalies which originally were involved in attacking the dolomite are released back into the pore solution for further assault. The expansion which accompanies this reaction, while still not completely understood, is believed to be related to the swelling of the interstitial clay minerals caused by association with the alkaline pore solution[10].

8.0 FACTORS CONTRIBUTING TO ALKALI-CARBONATE REACTIVITY

8.1 *ACR ROCK:* Rocks which are susceptible to ACR seem to exhibit a characteristic lithology. They consist of roughly equivalent amounts of calcite and dolomite with an insoluble residue of 10% to 30% consisting primarily of clay minerals. In hand specimens, the rocks are usually dark, fine grained, and exhibit conchoidal fracture. The distinctive microtexture, identifiable in thin section, is of rhombs of dolomite, roughly 25 microns across, floating in a fine-grained matrix of calcite and clay.

Two factors which directly affect the amount of expansion associated with concrete containing ACR rock are the maximum size of the aggregate particles and the percentage of ACR rock in the aggregate. As either factor increases, the amount of concrete expansion increases.

8.2 *Alkalies:* Expansion is directly affected by the alkali content of the pore solution. The primary source of alkalies is the cement, but other mixture components, and deicing salts may contribute alkalies as well.

8.3 *Moisture:* Moisture is necessary for the reaction to occur. Water provides the vehicle for the alkali hydroxides necessary to drive the dedolomitization reaction, and it is absorbed by the clay minerals resulting in expansion. Concrete which will remain completely dry in service is unlikely to become distressed by ACR.

9.0 IDENTIFICATION OF ALKALI-CARBONATE REACTIVITY DISTRESS IN CONCRETE

9.1 Alkali-carbonate reactivity causes expansion of concrete and, as such, the outward manifestations of distress are similar to that of alkali-silica reactivity or any other process which causes differential volume change in a mass. Most evident will be map or pattern cracking. If the concrete member is subject to restraint in a particular direction, the cracking will align itself in that direction. Signs of expansion include closing of joints, offsets or misalignments of adjacent members, rotation of bridge deck rocking supports, crushing of concrete, and pavement blowups.

9.2 In order to establish alkali-carbonate reactivity as the cause of expansive distress in concrete, it is necessary to perform a petrographic examination. The procedures for performing such an analysis are outlined in ASTM C 856. Unlike alkali-silica reactivity, alkali-carbonate reactivity does not produce a readily recognizable reaction product. Consequently, an examination of distressed concrete for alkali-silica gel will quickly determine if ASR is involved. This examination can be performed on broken or sawn samples and is described in Section 4.

In the absence of gel, a detailed study of the cracking is required to determine its origins. Specimens should be sawn or cored since broken pieces may have fractures induced by the breaking process. The detailed crack analysis can be performed on finely lapped surfaces using a stereoscopic microscope. Cracks which originate in carbonate aggregate particles and radiate out through the cement paste are indicative of alkali-carbonate reactivity. Areas which contain such cracked aggregate particles should be thin-sectioned for detailed examination using a polarizing microscope. Thin section blanks can be epoxy-impregnated using a colored dye to aid in identifying pre-existing cracks. This examination permits a detailed description of the lithologic character of the aggregate and is definitive in establishing alkali-carbonate reactivity as the cause of the distress. In order to clearly distinguish the ACR texture, it is preferable to examine thin sections less than 20 microns in thickness.

10.0 EVALUATION OF MATERIALS AND ENVIRONMENT

10.1 ***Aggregates:*** The ASTM Standard Specification for Concrete Aggregates, C 33, places certain restrictions on the use of aggregates containing constituents reactive with alkalies. In its appendix, C 33 contains information on evaluating aggregates for potential reactivity. It should be noted that ASTM C 289, C 227, and P 214 are not appropriate for determining the potential for alkali-carbonate reactivity. A petrographic examination of an aggregate is the first step in the evaluation process. Guidance on performing such examinations is given in ASTM C 295 and the Handbook of Concrete Aggregates[11]. Alkali-carbonate reactive rock has been found to exhibit a distinctive set of compositional and micro-textural characteristics. These characteristics are roughly equivalent amounts of calcite and dolomite with a substantial portion of insoluble residue consisting primarily of clay. The dolomite occurs as rhombic crystals in a much finer grained matrix of calcite and finely disseminated clay. This texture is recognizable in thin sections less than 20 microns in thickness. Hand specimens of the reactive rock are usually dark, fine grained, and exhibit conchoidal fracture.

Rocks suspected of being reactive on the basis of compositional and textural characteristics should be subjected to further testing to determine if they expand in the presence of alkalies. The rock cylinder test, ASTM C 586, provides a rapid method which indicates whether the rock will expand in alkaline solutions.

This method should be used in conjunction with other evidence to form an opinion on the potential reactivity of an aggregate. It should not be used for acceptance or rejection of the aggregate. Guidance on interpreting the results are given in section 10 of C 586. Since the test uses a small sample, it is important that the sample be representative of the aggregate in question. Appropriate sampling techniques can be found in C 295.

The concrete prism test, ASTM C 1105, is a method to determine the potential for alkali-carbonate reactivity in concrete. The method can be used to evaluate job materials and provides a means for testing various combinations of aggregates, gradations, and cements. The test is however, time consuming, requiring 6 to 12 months for results.

Field service record can provide significant information for use in evaluating the potential performance of an aggregate. In evaluating field service records, important considerations are: aggregate size; proportion of questioned aggregate; degree of similarity of the aggregate in the field concrete to the aggregate proposed for use; alkali content of cement; other sources of alkalies; and environmental factors affecting the moisture content of the concrete.

Unfortunately, field records often will be less than complete with respect to these items or will differ in some or many respects from the service conditions for which the aggregate is being evaluated. Consequently, the information they provide must be used judiciously in conjunction with the other information available about the aggregate in question, the other materials involved, and the intended service conditions in evaluating the potential performance of the aggregate.

10.2 ***Cementitious Materials:*** See Section 5.2.1.

10.3 ***Environment:*** For alkali-carbonate reactivity to occur, there must be a sufficient amount of alkali available from the cement or other sources and there must be sufficient moisture available. Interior concretes of limited cross-sectional area will likely remain dry to an extent that deleterious ACR will not occur.

11.0 MINIMIZING POTENTIAL FOR ALKALI-CARBONATE REACTIVITY DISTRESS

When concrete is to be placed in a service environment whose conditions are conducive to alkali-carbonate reactivity and the aggregate to be used is potentially reactive, several factors should be considered to reduce the risk of deleterious expansion. The amount of expansion is directly related to the amount of reactive rock in the aggregate.

11.1 If carbonate rocks are stratified, the layers containing the reactive lithology can be identified, and they can be avoided in the quarrying process.

11.2 Reactive rock can be blended with nonreactive rock to produce non-expansive aggregate.

11.3 The amount of expansion is directly related to the top size of the aggregate. Reduce the size of the reactive rock.

11.4 The amount of expansion is directly related to the alkali content of the cement. Use a low-alkali cement. ASTM C 33 indicates a limit 0.6% (Na_2O equiv). Others indicate that alkali contents lower than 0.4% (Na_2O equiv) may be necessary to prevent expansion with some aggregates[12].

11.5 The use of mineral admixtures does not appear to be effective in controlling alkali-carbonate reactivity.

With these factors in mind, material combinations can be proposed and then evaluated using C 1105 prior to acceptance. Supplementary information can be found in the appendix to C 1105.

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Appendix T

GUIDE SPECIFICATIONS FOR CONCRETE SUBJECT TO ALKALI-SILICA REACTIONS

DEVELOPED BY

Copies of this publication can be obtained by writing to:

Mid-Atlantic Regional Technical Committee
900 Spring Street
Silver Spring, MD 20910

developing this material, the Committee focused on limiting this to alkali-aggregate reactivity in the Mid-Atlantic Region.

This specification is designed to be used by owners, transportation engineers, structural engineers and others to provide safeguards against the occurrence of ASR failures. As with all specifications, this specification should be used only by qualified professionals who are competent to evaluate the significance and limitations of the specifications and who will accept responsibility for the application of its requirements to the structure under consideration.

Task Group Members	Company Name
Richard Gaynor, Chairman	National Ready Mixed Concrete Assoc.
Bernie Igusky	Allentown Cement Co.
Joseph F. Lamond	Consultant
Robert Neal	Lehigh Portland Cement Co.
David L. Wherley	The Arundel Corp.

Member Name	Company Name
Bailey, Bill	Virginia DOT
Barbour, Ronald	U. S. Ash Corporation
Bhatty, Muhammad	Capitol Cement Corporation
Brooks, Paul D.	Independent Cement Corporation
Call, B. M.	Roanoke Cement
Chua, Teck	Virginia Concrete Co.
Cook, Russell	Lehigh Portland Cement Co.
Diedrich, Joseph E.	Dixie Cement Company
Dunne, Jr., James P.	FHWA-Region 3
Eichelberger, Terry	Laurel Sand & Gravel Inc.
Ellis, Ed	MONEX Resources
Figliola, Anthony A.	A. G. Kurtz & Sons
Forster, Stephen	Federal Highway Administration
Gapinski, Gary	Scan Cem Industries
Hancock, James	Virginia DOT
Heckel, Ronald	Genstar Stone Products
Hergert, David	Mays Landing Sand & Gravel Co.
Hooton, Doug	University of Toronto
Howe, Richard	Consultant
Kalcheff, Ignat V.	D. C. Dept of Public Works
Kalendek, David	Maryland SHA
Karasopoulos, Theodore H.	Maine DOT
Koss, George	J. T. M. Industries
Lane, Stephen	VA Trans. Research Council
Mackow, Richard	J. T. M. Industries
Marchant, John D.	U. S. Ash Corporation
Mazzarella, Joseph	ESSROC Materials, Inc.
McCall, W. Calvin	Blue Circle Cement, Inc.
O'Brien, William	ESSROC Materials, Inc.
Peters, Fran	Capitol Cement Corporation
Pryor, C. A.	National Stone Association
Reardon, Jr., Patrick W.	Northeast Cement Shippers
Rose, Jere	Blue Circle Atlantic
Skalny, Jan	Consultant
Steel, Richard	Virginia DOT
Stokes, David	Delaware DOT
Thomson, Margaret	Pennsylvania DOT
Tyson, Sam	American Coal Ash Assn.
Weber, Jack	Jack Weber Associates
Weisner, John	Maryland DOT
Woolwine, Jr., Sam	W. W. Boxley Co.
Yingling, James	Genstar Stone Products

SPECIAL SUPPLEMENTARY PROVISIONS:
GUIDE SPECIFICATION FOR CONCRETE SUBJECT TO ALKALI-SILICA REACTIONS

1.0 SCOPE

The special provisions stated herein are for use in modifying a specification for concrete: subjected to (1) wetting or extended exposure to humid conditions or in contact with moist ground in service, (2) made with a potentially reactive aggregate, and (3) made with a high alkali cement as defined in ASTM C 150. In such instances, the potential for detrimental alkali-silica reactions in concrete exists, and these special provisions are applicable to aggregates found in the middle Atlantic region of the United States (Note 1). These provisions do not apply to concrete that will be continually dry in service.

Note 1. See the "Guide to Alkali-Aggregate Reactivity" for a discussion on alkali-carbonate reactions that may occur with certain calcitic dolomites and dolomitic limestones.

2.0 REFERENCED DOCUMENTS

2.1 *American Society for Testing and Materials (ASTM):*

C 33 Specification for Concrete Aggregates

C 150 Specification for Portland Cement

C 227 Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Standard)

C 294 Descriptive Nomenclature of Constituents of Natural Mineral Aggregates

C 295 Standard Guide for Petrographic Examination of Aggregates for Concrete

C 441 Standard Test Method for Effectiveness of Mineral Admixtures or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction

C 595 Specification for Blended Hydraulic Cements

C 618 Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete

C 823 Standard Practice for Examination and Sampling of Hardened Concrete in Construction

C 856 Standard Practice for Petrographic Examination of Hardened Concrete

C 989 Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortars

P 214 Proposed Test Method for Accelerated Detection of Potential Deleterious Expansion of Mortar Bars Due to Alkali-Silica Reaction

P 227 Proposed Standard Practice for Evaluation Criteria of Hydraulic Cement Testing Laboratories

C 1077 Practice for Laboratories Testing Concrete and Concrete Aggregates for Use in Construction and Criteria for Laboratory Evaluation

2.2 ***American Association of State Highway and Transportation Officials (AASHTO)***

M 307 Standard Specification for Microsilica for Use in Concrete and Mortar

3.0 TERMINOLOGY

3.1 ***Alkalies***: Sodium and potassium, expressed analytically as oxides, are generally derived from portland cement, but may be derived from admixtures and, in some instances, aggregates. Alkalies may also be available to concrete from external sources such as deicing salts. Alkali content is expressed as Na_2O equivalent and calculated as:

$$(Na_2O + 0.658\ K_2O)$$

3.2 ***Pozzolan***: In this guide specification, pozzolanic materials are considered to include: fly ash, ground granulated blast-furnace slag, and silica fume (microsilica).

3.3 ***Alkali-Silica Reaction***: A chemical reaction in concrete between alkalies and certain siliceous aggregates and some calcareous aggregates containing certain forms of silica. The products of this reaction, under certain conditions, may cause deleterious expansion within the concrete.

4.0 SUBMITTALS

Initial submittals include the information required in the following subparagraphs. Whenever the sources of material change or the relevant properties of the materials change, a resubmittal of the relevant information shall be made demonstrating compliance with the applicable provisions.

4.1 ***Aggregates***: Laboratory test reports shall be submitted by the supplier of the aggregates and shall contain data on mineralogy and potential reactivity with alkalies as specified herein.

4.2 ***Portland Cements***: Laboratory test reports shall be submitted by the supplier of portland cement and shall contain data on the alkali content. See 3.1.

4.3 ***Blended Hydraulic Cements***: Laboratory test reports shall be submitted by the supplier of blended hydraulic cement demonstrating compliance with section 7.0. The statement shall include data on the alkali content of the portland cement constituent and sources of cement and percentages of pozzolan used in the manufacture of the blended cement.

4.4 ***Pozzolans***: Laboratory test reports shall be submitted by the supplier of pozzolanic materials demonstrating compliance with the requirements of section 7.0. Include the minimum weight percent pozzolan and alkali contents of the cements used in 7.0.

4.5 ***Chemical Admixtures***: Laboratory test reports shall be submitted by the supplier of chemical admixtures which shall include information on the chloride ion content and alkali content expressed as $(Na_2O + 0.658\ K_2O)$. Test reports are not required for air-entraining admixtures used at dosages less than 2 oz per 100 lbs of cement or nonchloride chemical admixtures used at maximum dosages less than 5 oz per 100 lbs. of cement. Both the chloride ion and total alkali content of the admixture are to be expressed in percent by weight of cement for a stated or typical dosage of the admixture, generally in fluid ounces per 100 lb of cement.

4.6 *Laboratories*: Laboratories supplying test reports or data, as required in this guide specification, shall provide evidence that the laboratory is properly equipped and qualified to perform the test method(s).

Note 2. See ASTM C 1077 and P 227.

5.0 AGGREGATES

5.1 *Evaluation of Aggregates*: Aggregates for use in concrete shall conform to the requirements of ASTM C 33. Testing and evaluation for alkali-silica reaction in ASTM C 33 Appendix X1 shall be as hereinafter specified.

Note 3. The criteria contained in Appendix X1 of ASTM C 33 may not identify many of the aggregates in the Mid-Atlantic region which are susceptible to alkali-silica reaction.

5.1.1 Petrographic analysis in accordance with ASTM C 295 shall identify the constituents of the fine and coarse aggregate. Fine and coarse aggregate containing more than the following quantities of constituents shall be considered potentially reactive:

(a) Optically strained, microfractured, or microcrystalline quartz exceeding 5.0% (a common constituent of granite and granite gneiss).
(b) Chert or chalcedony exceeding 3.0 %
(c) Tridymite or cristobalite exceeding 1.0 %
(d) Opal exceeding 0.5%
(e) Natural volcanic glass in volcanic rocks exceeding 3.0 %

5.1.2 Additionally, aggregate determined to be potentially reactive by ASTM C 295 may be further evaluated by ASTM Proposed Method P-214. Aggregate sources which exhibit P 214 mean mortar bar expansion at 16 days greater than 0.10% shall be considered potentially reactive.

Note 4. The classification of an aggregate as potentially reactive may be appealed if additional tests or service record data supporting the reclassification are acceptable to the specifier.

5.1.3 Tests of ASTM C 227 mortar bars may be used to confirm potential reactivity of fine or coarse aggregate, but not to classify an aggregate as "non-reactive". The aggregate is considered "reactive" when expansion is greater than 0.10% at six months when ASTM C 227 Mortar bars are made with a cement with an alkali content greater than 0.80%.

Note 5. Some of the Mid-Atlantic region aggregates that have produced alkali-silica reactions in service do not produce excessive expansion in ASTM Method C 227; however, aggregates that expand excessively in C 227 should be classified as potentially reactive. The quick chemical test method ASTM C 289 has limitations and should not be used to evaluate aggregates in the Mid-Atlantic region.

5.1.4 Aggregates that have shown evidence of reactivity in service shall be considered potentially reactive regardless of the results of 5.1.1 or 5.1.2. Determination of reactivity in structures must include:

(a) Petrographic analysis of cores by ASTM C 856.

(b) Visual examination of cracking and expansion of elements of the structure.

(c) Evaluation of available data, including construction records – if any.

5.2 Aggregate determined to be potentially reactive with alkalies shall only be used in concrete with cementitious materials conforming to the requirements of Section 6.

6.0 CEMENTITIOUS MATERIALS

6.1 One of the following three options shall be used to reduce alkali silica reactions if:

a) the aggregates have been determined to be reactive with alkalies and,

b) the concrete will be subjected to wetting or extended exposure to humid conditions or in contact with moist ground in service.

6.2 <u>Option A</u>. Use a combination of a pozzolan with a portland or a blended cement, which meets the requirements of the following subsections of Section 6.2.

6.2.1 The alkali content of the portland cement proposed for use under this option shall not be more than 0.05% alkali greater than that of the cement used in the "Test mixture" to evaluate the effectiveness of the pozzolan. See 7.2.2.

6.2.2 If a blended cement is used with additional pozzolan, under this option, the blended cement must conform to ASTM C 595, Types IP, I(PM), IS or I(SM).

6.2.3 Fly ash or natural pozzolan shall conform to ASTM C 618, including the optional chemical requirement of a maximum of 1.5% available alkali. The required minimum fly ash or pozzolan content shall be that determined to be "effective" in Section 7 but not less than 15% for Class F fly ash or 25% for Class C fly ash.

Note 6. Pozzolan percentages shall be calculated as a percent by weight of cement plus pozzolan.

6.2.4 Slag shall conform to ASTM C 989 Grade 120 and Grade 100. Silica fume shall conform to AASHTO M 307. The required minimum slag or silica fume content shall be that determined to be effective in Section 7, but not less than 25% for slag or 5% for silica fume. See Note 6.

6.3 <u>Option B</u>. Use a blended hydraulic cement which has been demonstrated to be effective in controlling expansion as specified in Section 7 and which conforms to ASTM C 595 Types IS or IP.

6.4 <u>Option C</u>. Use a low alkali portland cement which conforms to ASTM C 150 and which has a total alkali content not greater than 0.60%.

7.0 EFFECTIVENESS OF A POZZOLAN IN CONTROLLING EXPANSION

The limits on expansion referenced in ASTM Standards C 595, C 618, and C 989 are not applicable. The effectiveness of a pozzolan or blended cement in controlling deleterious expansion shall be determined by comparative testing outlined below.

7.1 *Criteria for Effectiveness*:

7.1.1 Pozzolans: The 14-day expansions of the "test mixture" made with the pozzolan and the selected high alkali cement shall be no greater than the expansion of a "control mixture" made with a low alkali cement with total alkalies between 0.50 and 0.60 percent.

7.1.2 Blended Cements: The expansion of a "test mixture" made with blended hydraulic cement meeting the requirements of ASTM C 595, Types IP and IS, shall be no greater than the expansion of a "control mixture" made with a low alkali cement with total alkalies between 0.50 and 0.60 percent.

7.2 *Test Procedure*: Mix "control" and "test" mixtures using 900 g of the Pyrex Glass aggregate specified in ASTM C 441. The amount of mixing water shall be that needed to produce a flow of 100 to 115 as determined in ASTM Method C 109.

7.2.1 Control Mixture: The control mixture shall be made with 400 g of cement conforming to ASTM C 150 with total alkalies of 0.50 to 0.60%.

7.2.2 Test Mixture: The test mixture shall be made with:

(a) an ASTM C 150 cement with a total alkali content exceeding 0.80% and a quantity of pozzolan not be less than that required in 6.2.3. The total weight of cement plus pozzolan shall be 400 g;

(b) or a blended cement C 595 Types IS or IP;

(c) or a blended cement Types IS, IP , ISM, or IPM with additional pozzolan. The pozzolan used in the manufacture of the blended cement shall be included in the calculation of the percent pozzolan for compliance with the minima required in 6.2.3. Pozzolan percentages are calculated as a percent of the total weight of cement plus pozzolan.

7.3 *Specimen Preparation*: Except for proportioning of mortar, prepare, store and measure specimens, and calculate expansion in accordance with sections 7, 8 and 9 of ASTM C 227-87. Measure the bars at an age of 14 days and calculate expansion from the length at 24 ± 2 hr. Express the expansion of the test mixture as a percent of the control mixture.

Appendix U

SESSION 3 - FIELD TRIP TO IDENTIFY ASR IN STRUCTURES

Notes

This field trip is intended to familiarize observers, first-hand, with crack patterns due to ASR in highway structures, and to better relate field manifestation of ASR to microscopic observations in the laboratory. In particular, comparison of UV light observations both in the laboratory and in the field will be demonstrated. The SHRP "Handbook for The Identification of Alkali-Silica Reactivity in Highway Structures" (Appendix F) can be used and should be brought on the trip.

During the inspections, the nature of the crack pattern should be observed keeping in mind evidence of expansion that has developed in response to restraint and interaction with the environment.

I. - Visual Inspection of Crack Patterns

A) Inspections will be made of highway structures previously identified as possibly exhibiting ASR. Structures with other forms of cracking may be inspected and probable causes discussed to help in distinguishing ASR. Inspections should be made keeping the following factors in mind:

1) Environmental effects

a) Moisture - must be available for expansion to occur. Sources: subbase, atmospheric precipitation.

1. Relative Humidity (RH) in concrete must be at least 80% to sustain expansion due to ASR. This condition will exist continuously in pavement slabs, and cyclically in bridge decks in hot arid climates.

2. Shrinkage due to atmospheric drying can accentuate cracking due to ASR.

b) Restraint - affects development of crack patterns.

1) Minor to no restraint - crack pattern tends to be equidimensional, similar to that seen on dried mud flats.

2) Major restraint - causes most prominent cracks to form parallel to the direction of maximum restraint. In both jointed and CRC pavement, most prominent cracking tends to be longitudinal due to restraint of abutting pavement slabs (See Appendix F).

Crack pattern tends to be more uniformly developed in joint areas because of lesser restraint in the upper half of slabs. In bridge structures, cracking varies from longitudinal to random or uniform pattern in response to restraint by reinforcing steel and bridge girders.

II. - Uranyl Acetate - UV Light Method in Field on Field Structures

A) The uranyl acetate - UV light method will be demonstrated on in-place prepared surfaces on pavement concrete and bridge structures. Use the procedure described in Session 2, IV-2.

B) ABIDE BY ALL SAFETY PRECAUTIONS DETAILED IN THE WORKSHOP MANUAL AND SAFETY PRECAUTIONS SHEET (Appendix D).

III. - Hands-on Application of Uranyl Acetate-UV Light Method in Classroom.

A) Return to classroom for hands-on detection of possible ASR gel in concrete cores procured from the structures inspected.

1) Use uranyl acetate-UV light method and observe the safety precautions described in Session 2, IV-C-1 and Appendix D.

IV. - Wearing Apparel

A) Attendees should be dressed appropriately for outdoor inspection. As necessary, include protection from direct sunlight, and safety shoes suitable for climbing embankments. The inspections will not be carried out in the rain, and will be rescheduled as time permits. Lecturing and discussions will continue in the classroom during the time period originally scheduled for the field trip.

SESSION 4 - FLUORESCENCE AND MICROSCOPICAL TECHNIQUES TO IDENTIFY ASR AND POTENTIALLY REACTIVE ROCK TYPES IN CONCRETE

Notes

I. Examination of cores taken from structures inspected during the field trip.

- A) Visual examination in plain light for crack patterns.
- B) Examination of freshly fractured surfaces using the uranyl acetate-UV light method. Use safety precautions described in Session 2.

II. Common Rock-forming Minerals (Fig 4-1 and 4-2). Representative samples of aggregates from any source to be used for portland cement concrete should be subjected to petrographic examination. The examination, by a competent petrographer, will reveal not only characteristics pertaining to ASR, but also other features that may affect the performance of the concrete. This session describes features that relate particularly to ASR (Appendix G, ASTM C 294, and Appendix H, ASTM C 295).

III. Classification of Rocks

- A) Igneous Rocks, solidified from molten material and classified on the basis of mode of occurrence and mineral constituents, including quartz, feldspar, and other minerals (Fig 4-3).
- B) Sediments and sedimentary rocks - particulate materials transported by water, wind, ice, gravity, and chemically precipitated deposits formed on the earth's surface; cemented (Fig 4-4).

C) Metamorphic Rocks, formed by modification of pre-existing rocks due to heat and pressure, and classified on basis of mineralogy and structure, i.e., foliation, banded, or massive (Fig 4-5).

D) Miscellaneous Aggregates

1) Recycled concrete

2) Mine tailings

IV. Sampling for Petrographic Examination

A) ASTM C 295 "Standard Practice for Petrographic Examination of Aggregates for Concrete" requires sampling according to ASTM C 75, "Practice for Sampling Aggregates (Appendix I and Fig 4-6).

B) Sample quarry rocks and pit materials at all major changes if detailed petrographic analysis is required.

C) Sample size (weight) determined according to particle dimensions (see tables in C 295 and D 75); quartered and sieved.

V. Petrographic Examination

A) Demonstration of prepared specimens and microscope slides of reactive rocks and ASR effects (powder mounts and thin sections) with stereomicroscope and petrographic microscope(Figs 4-7 to 4-12).

B) Demonstration - Fluorescence Microscopy (Fig 4-13 and 4-14).

1) Procedure for microcrack examination using a fluorescent impregnant, epi-fluorescence microscopy, and polished sections (Appendix J).

CONSTITUENTS OF AGGREGATES

1. Minerals
2. Igneous rocks
3. Sedimentary rocks
4. Metamorphic rocks

Fig 4-1 Major constituents of aggregates.

MINERALS

Silica

Silicates

Carbonates

Sulfates

Iron Sulfide

Iron Oxide

Fig 4-2 Mineral consitiuents of aggregates.

IGNEOUS ROCKS

Granite

Diorite

Gabbro

Pegmatite

Rhyolite

Andesite

Basalt

Volcanic glass

Fig 4-3 Igneous rocks commonly used as aggregate.

SEDIMENTARY ROCKS

Sandstone

Shale

Limestone

Chert

Conglomerate

Graywacke

Fig 4-4 Sedimentary rocks commonly used as aggregate.

METAMORPHIC ROCKS

Gneiss

Schist

Metagraywacke

Quartzite

Marble

Fig 4-5 Metamorphic rocks commonly used as aggregate.

PROCEDURE

1. Dry sieve the sample in accordance with ASTM C 136
2. For sands, wash the sample to collect <75μm (No. 200) particles (ASTM C 117)
3. Microscopically examine and identify at least 150 particles in each sieve fraction and calculate the percentages

Fig 4-6 Procedures in the petrographic examination of aggregate.

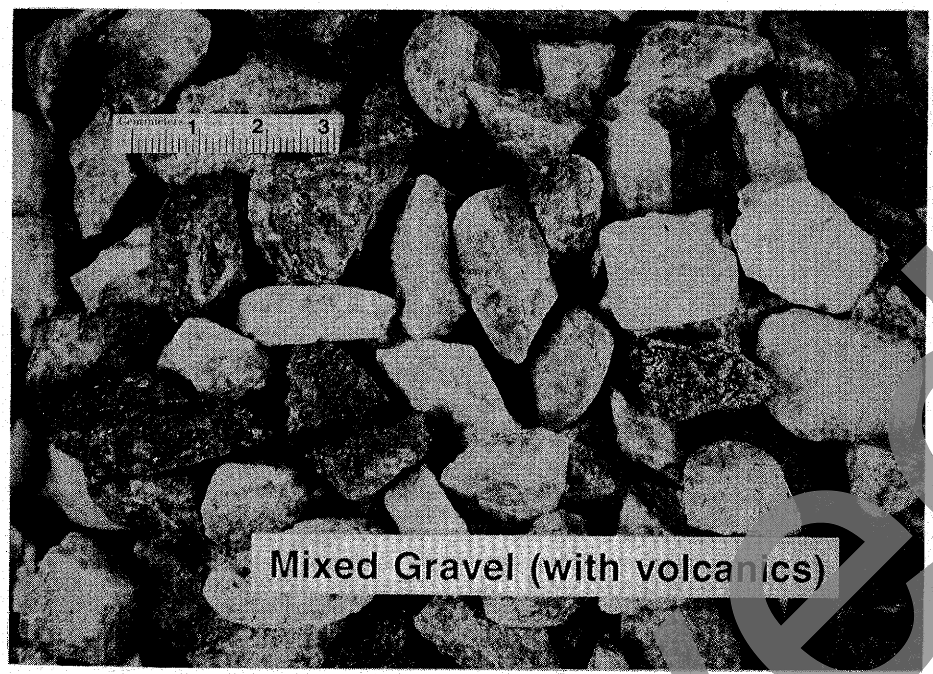


Fig 4-7 Gravel containing mixture of igneous, metamorphic, and sedimentary rocks.

Fig 4-8 Thin section of devitrified ash-flow tuff, a reactive constituent of the gravel shown in Fig 4-11, plane-polarized light, 100x.

Fig 4-9 Crushed chalcedonic limestone.

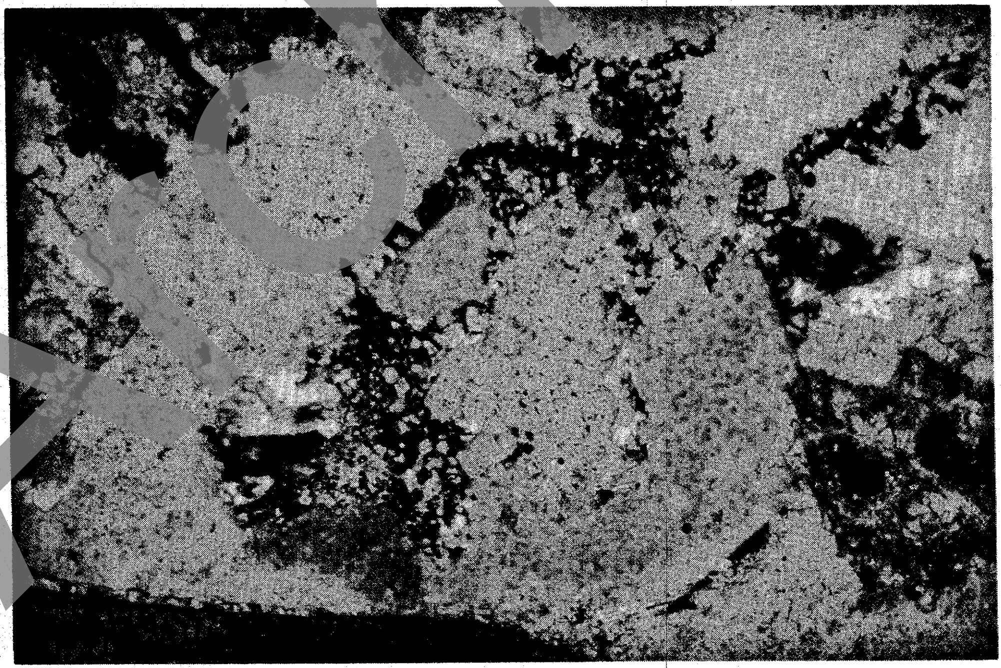


Fig 4-10 Thin section of crushed chalcedonic limestone, showing the reactive chalcedony. Cross-polarized light, 100x.

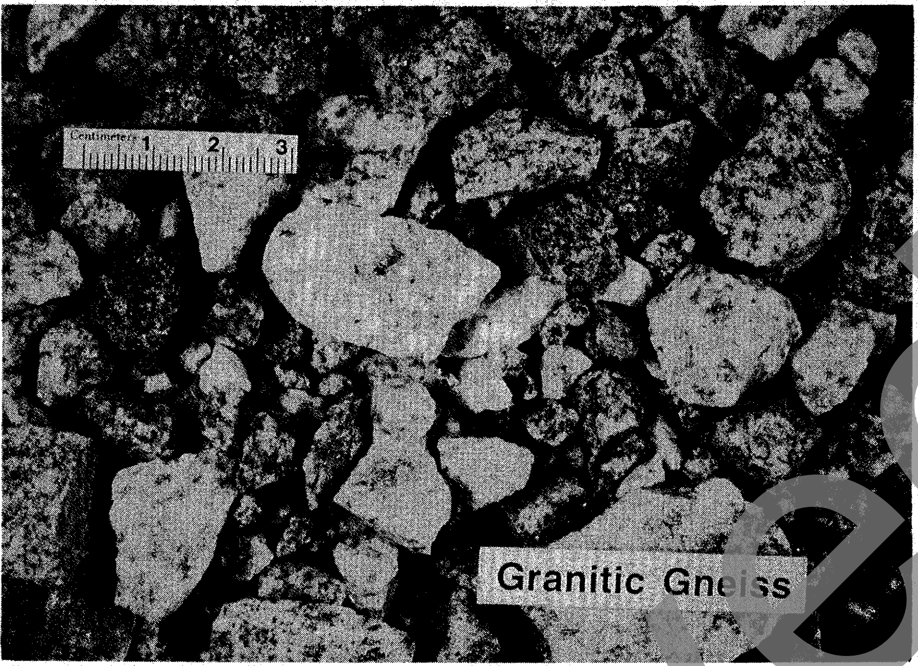


Fig 4-11 Crushed granite gneiss.

Fig 4-12 Thin section of crushed granite gneiss showing zone of sheared reactive quartz. Cross-polarized light, 100x.

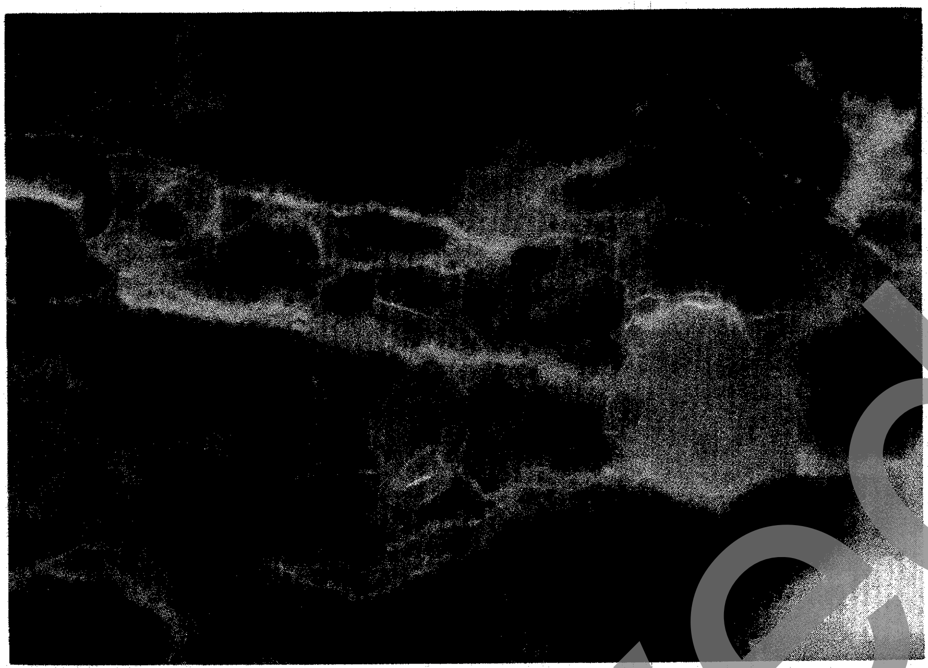


Fig 4-13 Lapped surface of concrete impregnated with fluorescent epoxy. Microcracks are revealed by green color, as revealed in UV light. Width of field: 1.4 mm.

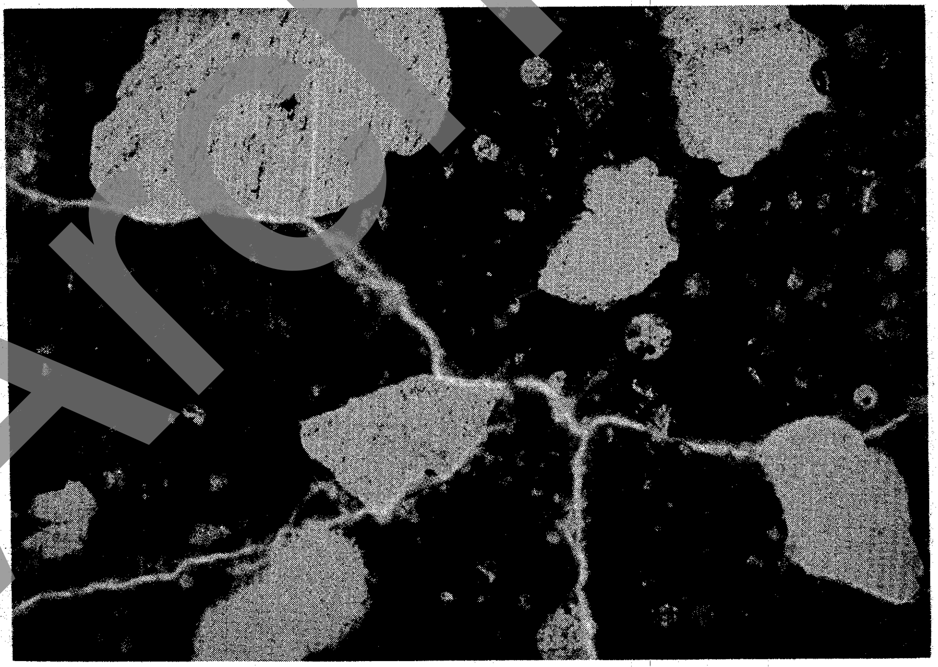


Fig 4-14 Thin section of high strength concrete containing silica fume and fly ash. Microcracks are revealed by yellow-green fluorescent color, as seen in UV light. Yellow areas are aggregate or air voids. Width of field: 1.4 mm.

SESSION 5 - RAPID IMMERSION TEST METHOD FOR POTENTIAL ASR OF AGGREGATES

Notes

I. Need for Test

A) ASTM C 227 Mortar Bar Test

In this test, mortar bars containing the test aggregate and cement are stored over water in sealed containers at 100°F (38°C). Expansions are calculated for ages up to 6 months or more. This test is too slow and too lenient and allows potentially deleteriously reactive aggregate or cement-aggregate combinations to pass the test (Appendix K and Figs 5-1 and 5-2).

B) ASTM C 289 Quick Chemical Test

In this test, a No. 50 to No. 100 sieve size aggregate sample is immersed in 1N NaOH solution at 176°F (80°C) for 24 hours. The amount of silica in solution is then plotted against the reduction in alkalinity of the solution. This test, although rapid, also is too lenient. (Figs 5-3 and 5-4)

C) ASTM C 295 Petrographic Examination

This procedure is subjective and is dependent on the capability of the petrographer. Petrographic examinations should be run on materials from all sources of aggregate, regardless of any suspected limitations in performance.

II. New Test Method (Fig 5-5)

A) The rapid immersion test method, ASTM C 1260, (Appendix L) and (modified ASTM P 214) AASHTO TP14 (Appendix M), is recommended to determine the potential of an aggregate for deleterious ASR. It was developed by Oberholster and Davies at NBRI in South Africa (Appendix N), and was previously known as ASTM P 214. It has the following advantages (Fig 5-6).

1) Although ASTM C 1260 is overly severe as it stands, adjusting the NaOH concentration of the immersion solution appears to have the potential to determine cement alkali level with which the aggregate can be safely used. This aspect of the test is still under development by the Portland Cement Association. (Appendix O, SHRP-C-343, Section 3.3.4.).

2) The capability and quantity of mineral admixtures to minimize expansive ASR, regardless of whether added or as an admixture or in blended cement may be determined (Appendix O, SHRP-C-343, Section 3.3.5., and Appendix P). This aspect of testing currently is under ASTM consideration.

3) The method requires a test period of only 16 days, is not labor intensive, and does not require expensive equipment.

III. Standard Methodology

A) Process aggregate per ASTM C 1260 (same gradation as ASTM C 227).

B) Cast three companion mortar bar test specimens.

C) Cure 1 day in molds at 23 ± 1.7°C in a moist cabinet..

D) Take comparator reading then transfer to immersion in water brought to 80 ± 2.0°C.

E) Take comparator reading at 24 hr and transfer to test solution. This reading is the "zero" reading to which subsequent readings are referenced. Read to nearest 0.0001 in. (0.0025 mm).

F) Transfer specimens, without allowing drying of the specimens, to the NaOH test solution maintained at 80 ± 1.0°C.

G) Take subsequent comparator readings at 7 and 14 days after the zero reading.

H) Calculate expansions in percent as difference between the 7- or 14-day reading and the "zero" reading. Calculate to the nearest 0.001%.

I) Expansions as low as 0.08% at 14 days have been found for certain aggregates that have caused expansive ASR in field structures. This also is the maximum expansion obtained for innocuous aggregates.(Appendix O and Figs 5-7 through 5-9).

J) Petrographic examination to identify the reacting particles in those mortar bars showing expansion is optional.

IV. Adjustments to Determine Maximum Safe Cement Alkali Level.

A) NaOH concentration of the immersion solution is adjusted to correspond to cement alkali level (Appendix O, SHRP C-343, Section 3.3.4).

B) The procedure otherwise is the same as given in the standard methodology.

C) The test failure criterion is variable and depends on NaOH concentration of the immersion solution. The curve in Fig 5-10 represents a projected test criterion, based on a limited number of tests. Refinement of this criterion is underway.

V. Evaluation of mineral admixtures and blended cements

A) Mineral admixtures can be evaluated in this procedure. This includes determining quantity required relative to cement to prevent deleterious expansion (Figs 5-11, 5-12, 5-13, and Appendix O).

ASTM C 227 LIMITATIONS

1. The test is too slow
2. The test is too lenient

Fig 5-1 Limitations of ASTM C 227.

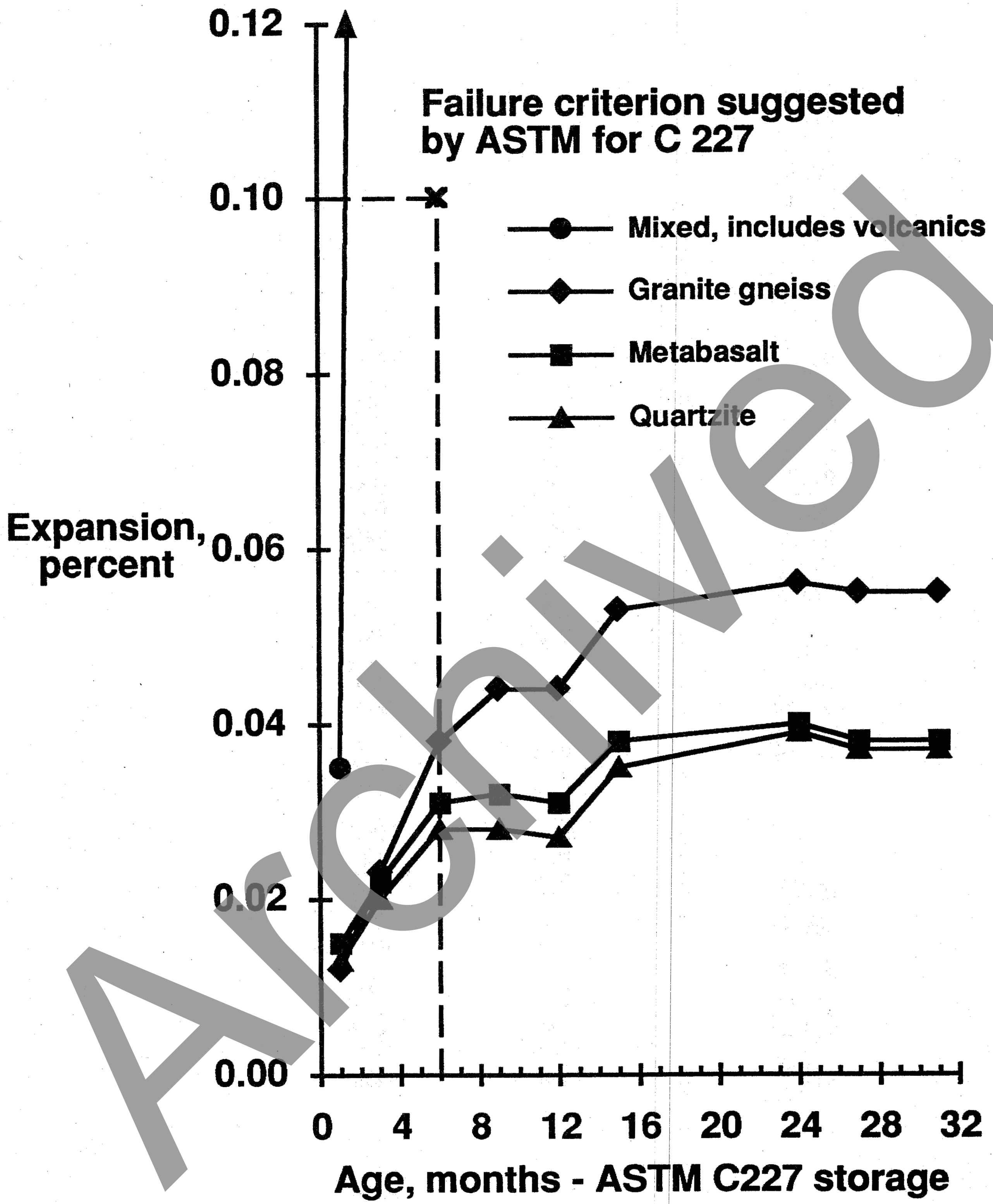


Fig 5-2 Results for SHRP aggregates evaluated in accordance with ASTM C 227 (Mortar-Bar Method). All aggregates are deleteriously reactive in field structures.

ASTM C 289 LIMITATIONS

1. The test is too lenient and does not properly identify certain potentially deleteriously reactive aggregates

2. The test can not indicate the adequacy of specific cement alkali reductions and admixtures in preventing expansive ASR

Fig 5-3 Limitations of ASTM C 289.

ASTM C 289 RESULTS

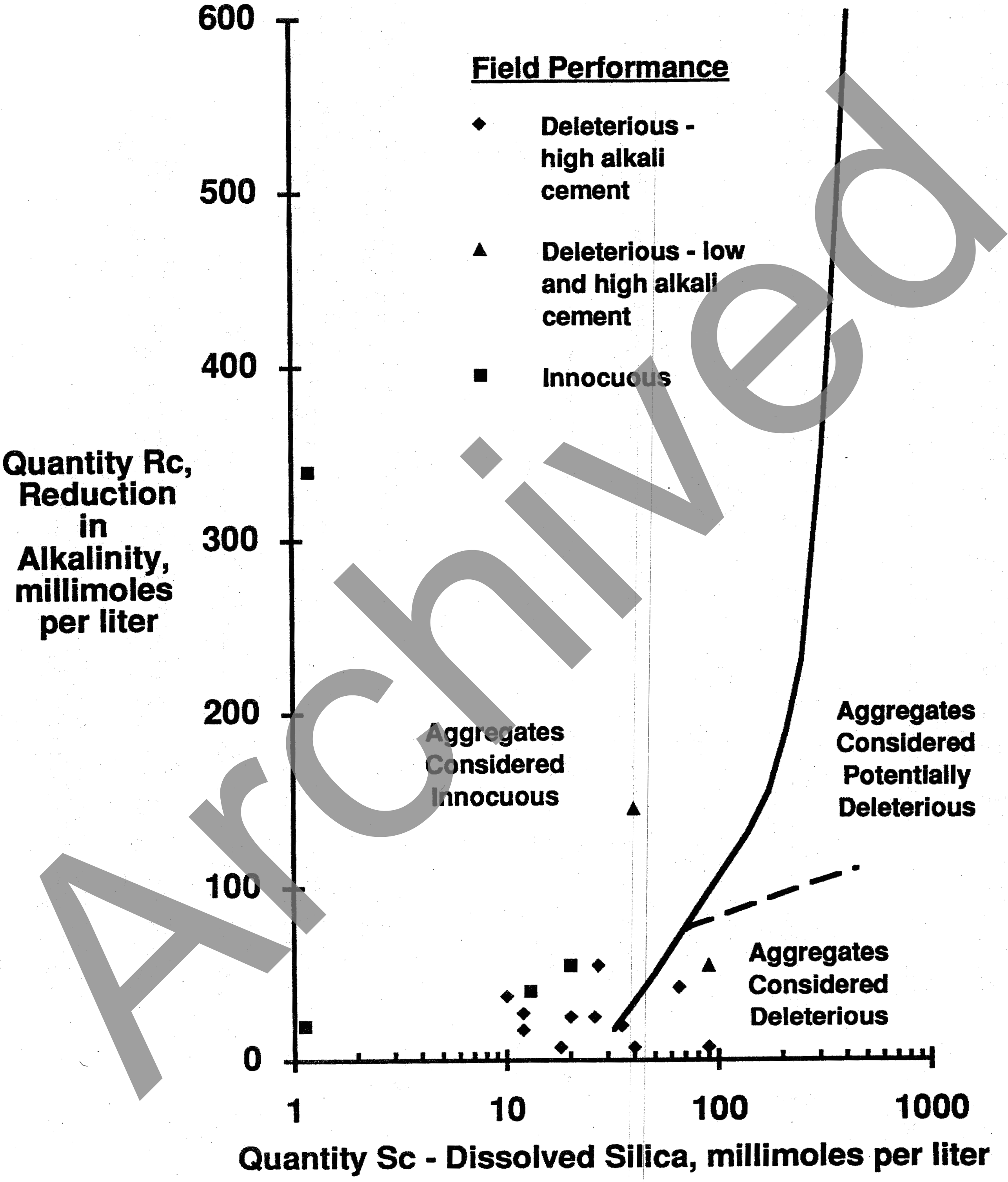


Fig 5-4 Results for SHRP aggregates evaluated in accordance with ASTM C 289.

APPARATUS FOR ASTM C 1260 TEST

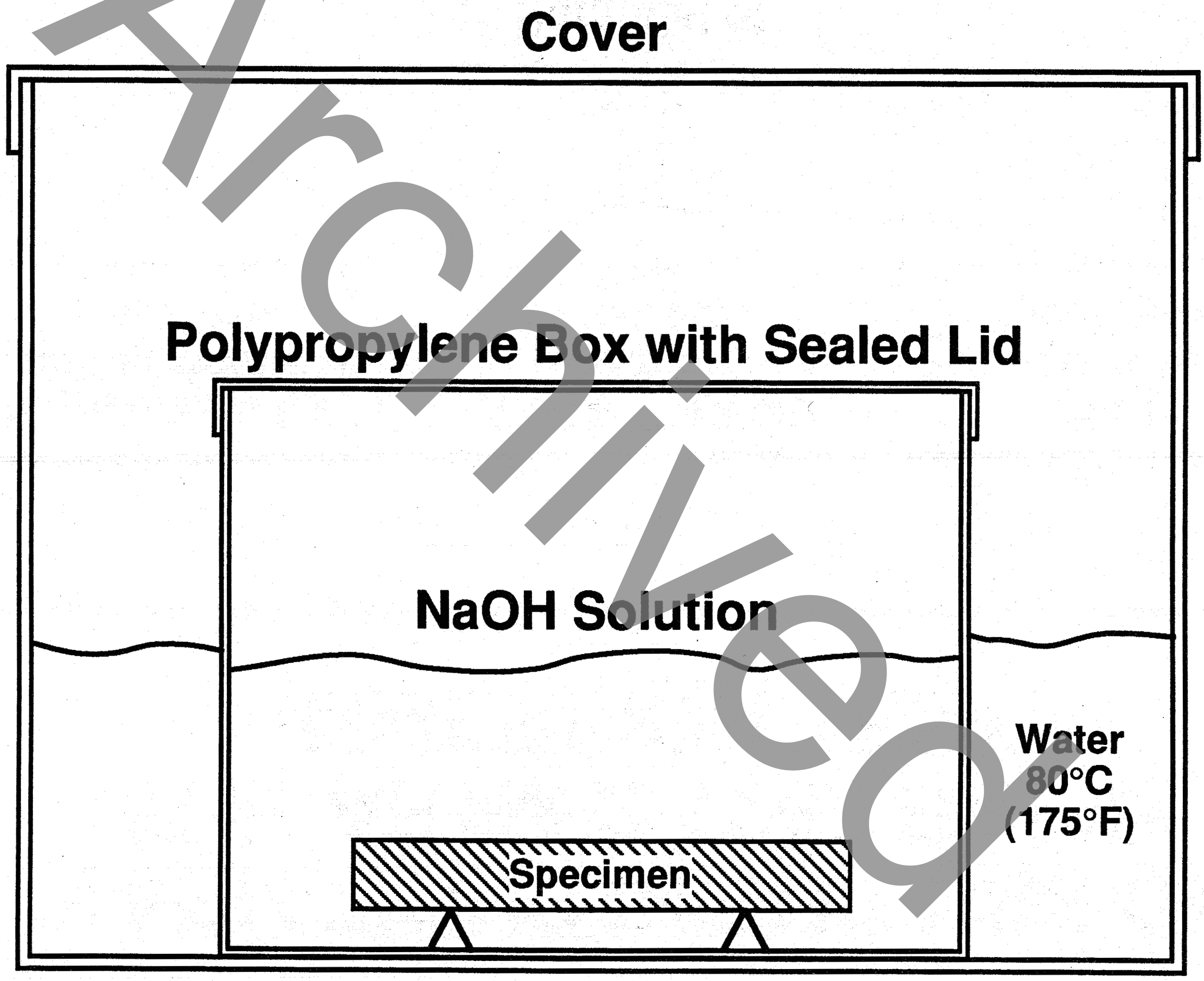
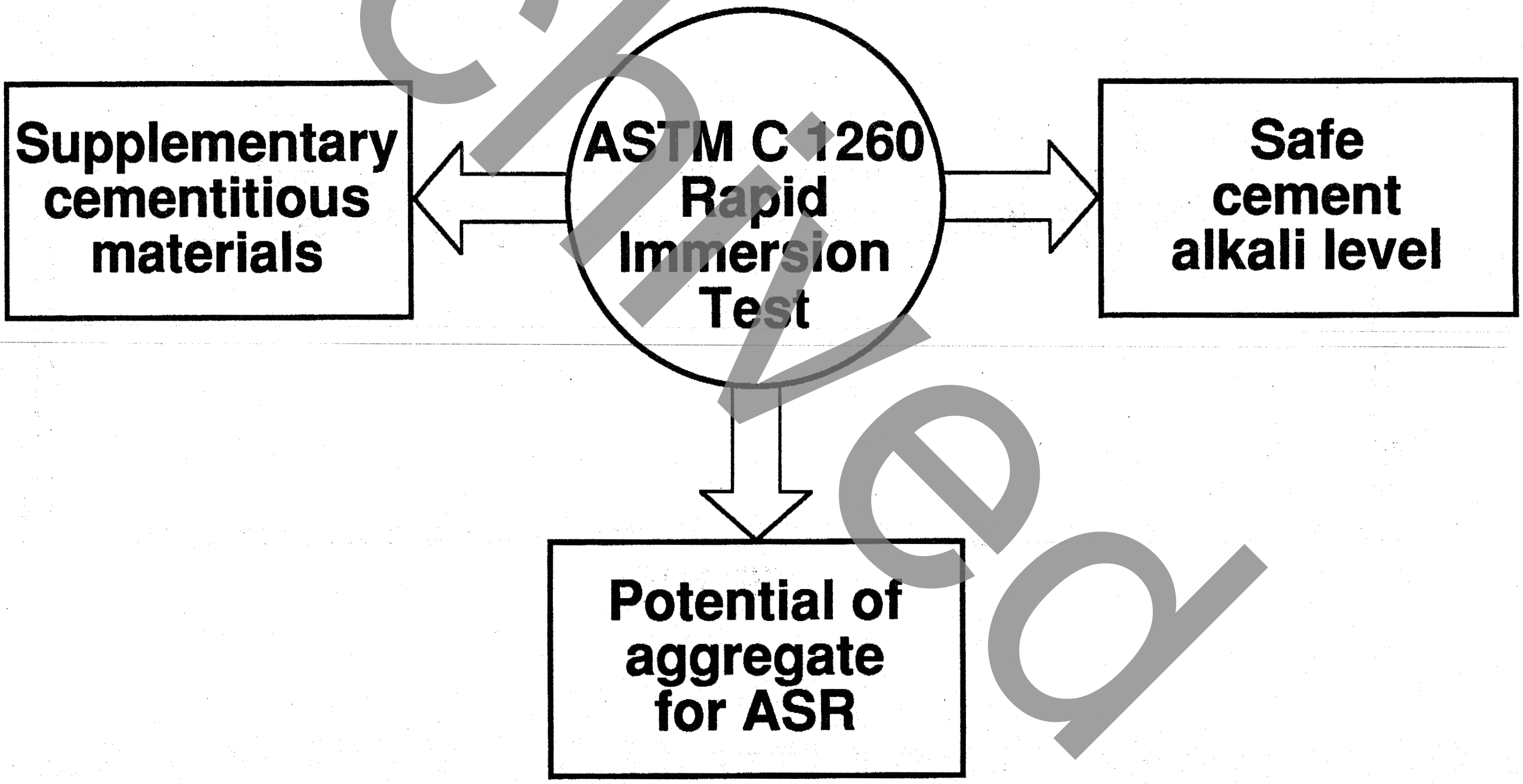


Fig 5-5 Example of the apparatus for conducting ASTM C 1260 tests.

VARIATIONS IN THE USE OF THE ASTM C 1260 RAPID IMMERSION TEST



ASTM C 1260 Rapid Immersion Test

Supplementary cementitious materials

Safe cement alkali level

Potential of aggregate for ASR

Fig 5-6 Applications of the rapid immersion test to identify deleterious cement-aggregate combinations.

ASTM P 214 TEST RESULTS

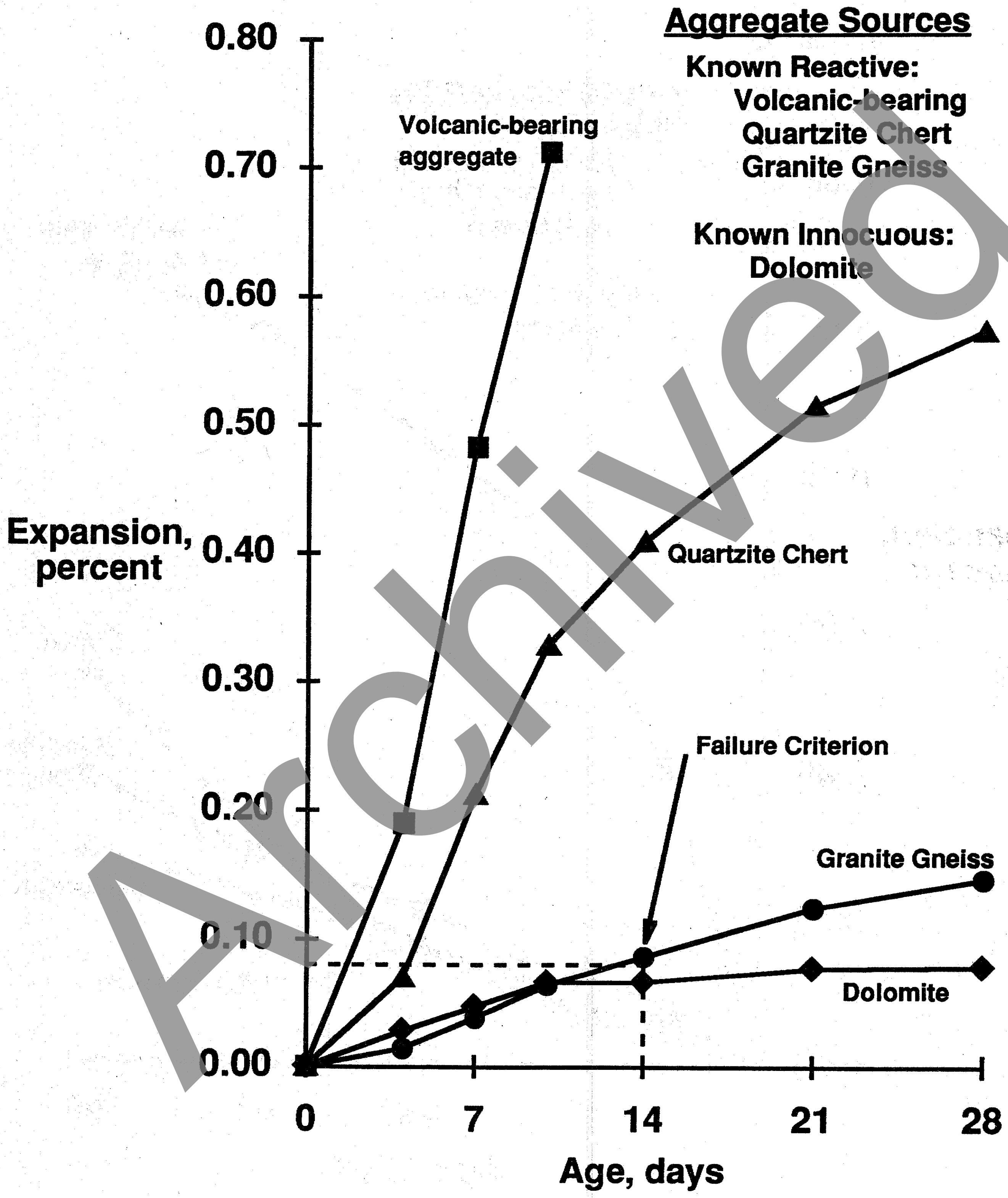


Fig 5-7 Results for ASTM P 214 tests (w/c = 0.50). Failure criterion shown is not that for ASTM C 1260.

ASTM P 214 TEST RESULTS

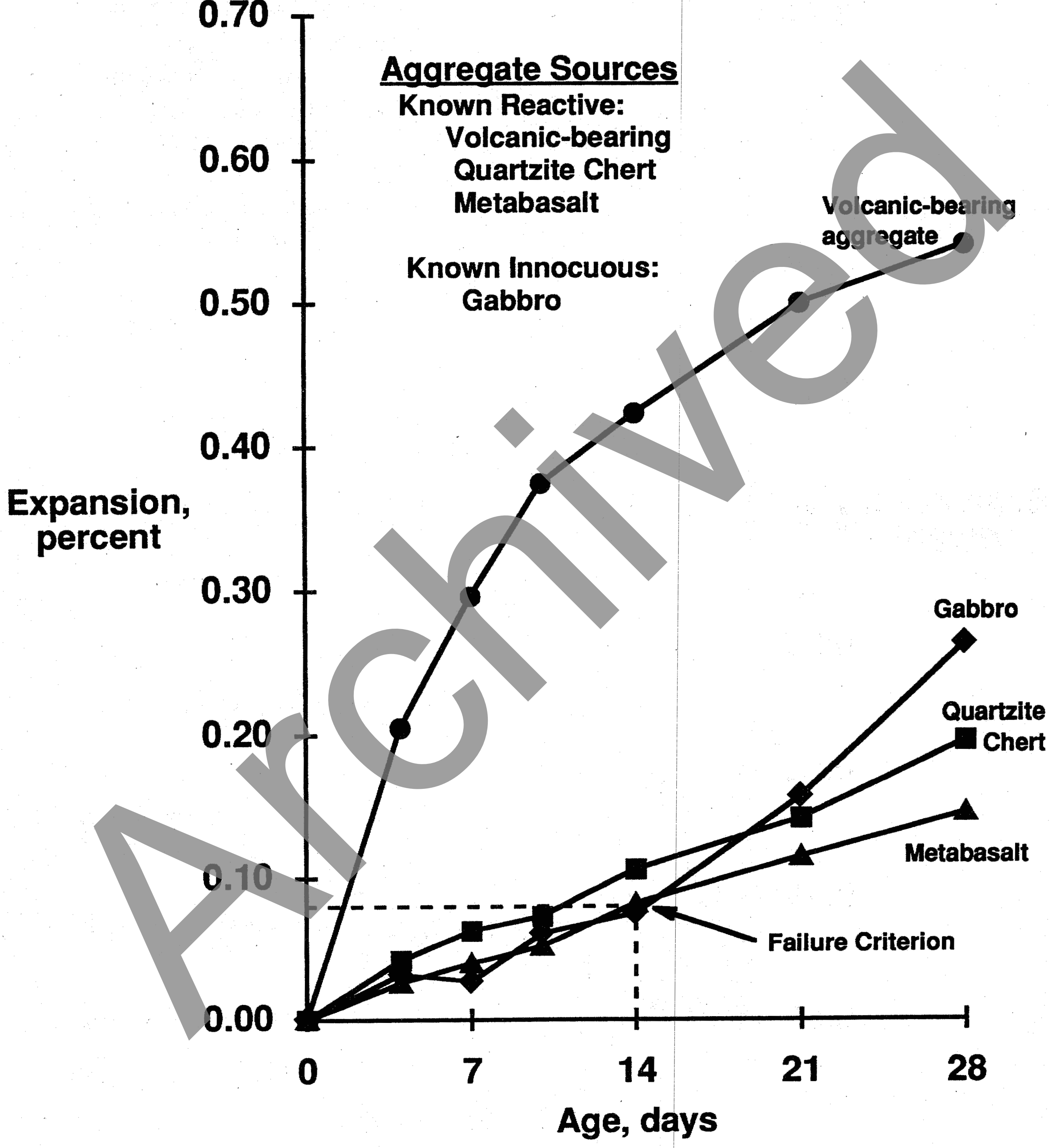


Fig 5-8 Results for ASTM P 214 tests (w/c = 0.50). Failure criterion shown is not that of ASTM C 1260.

ASTM P 214 TEST RESULTS

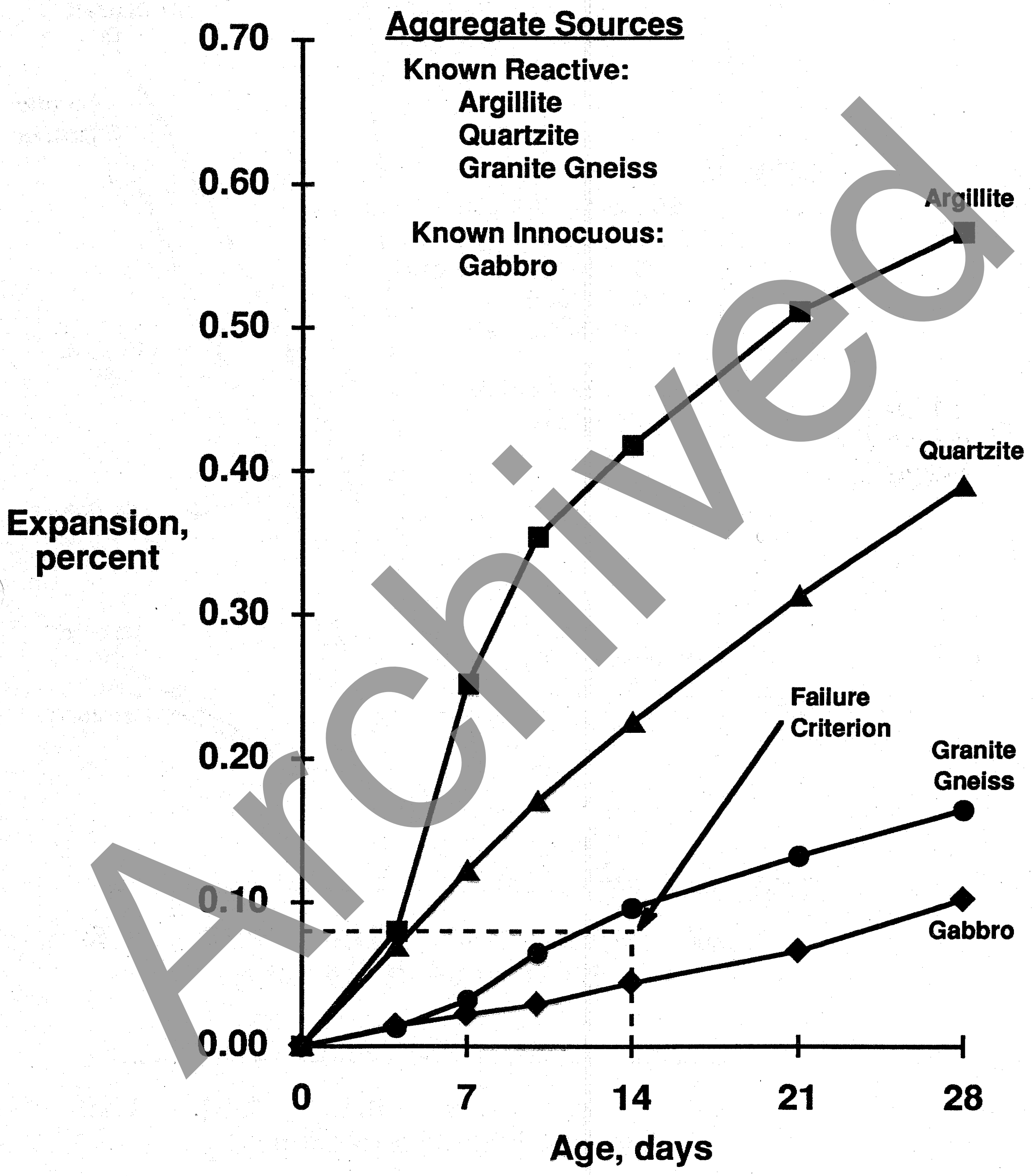


Fig 5-9 Results for ASTM P 214 tests (w/c = 0.50). Failure criterion shown is not that for ASTM C 1260.

MODIFIED ASTM P 214 TEST RESULTS

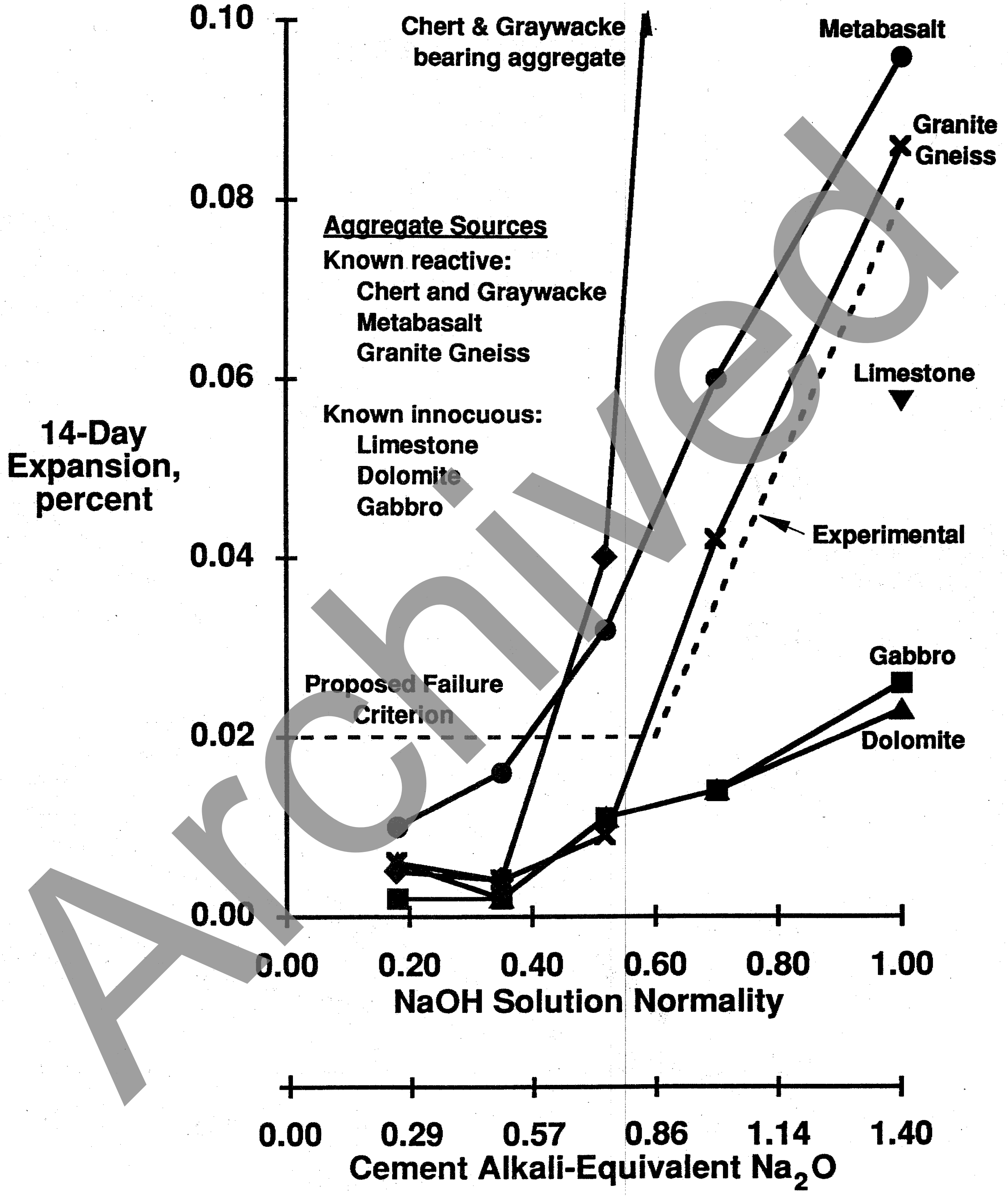


Fig 5-10 Proposed failure criterion for different NaOH concentrations (cement alkali levels).

MODIFIED ASTM P 214 TEST RESULTS

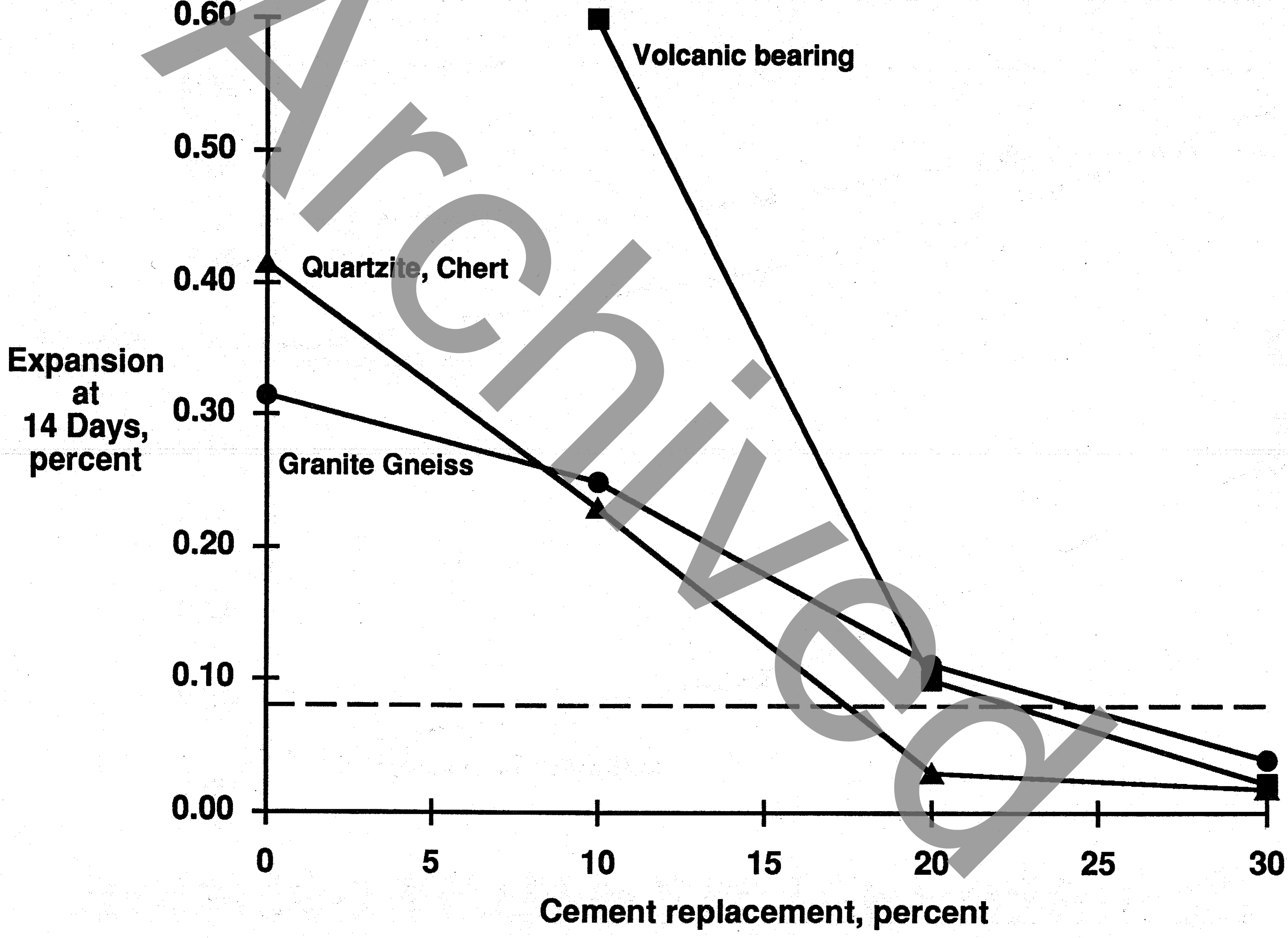


Fig 5-11 Modified ASTM P 214 test results using a low lime (Class F) fly ash (w/c = 0.50).

MODIFIED ASTM P 214 TEST RESULTS

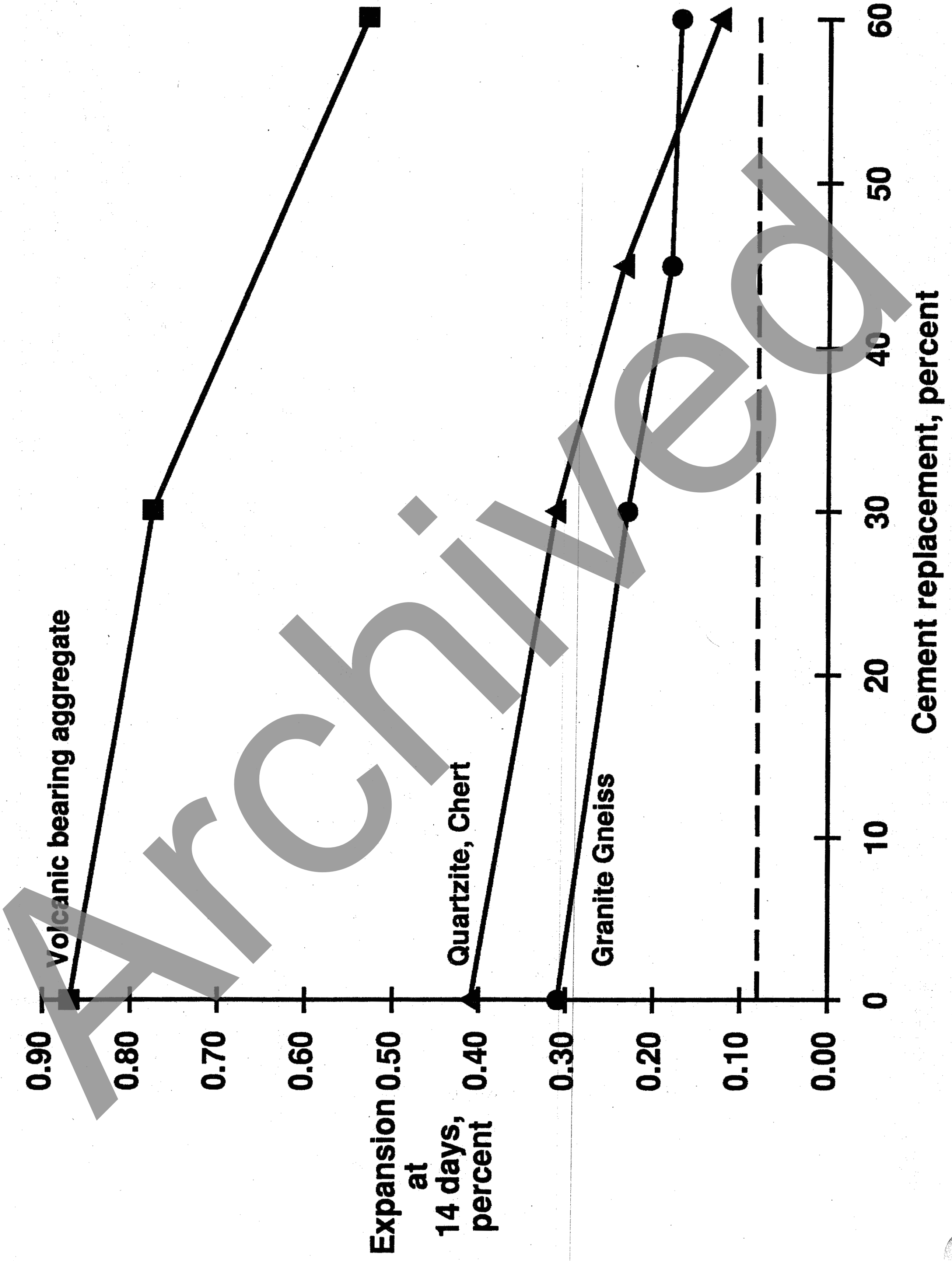


Fig 5-12 Modified ASTM P 214 test results using a high lime (Class C) fly ash (w/c = 0.50).

MODIFIED ASTM P 214 TEST RESULTS

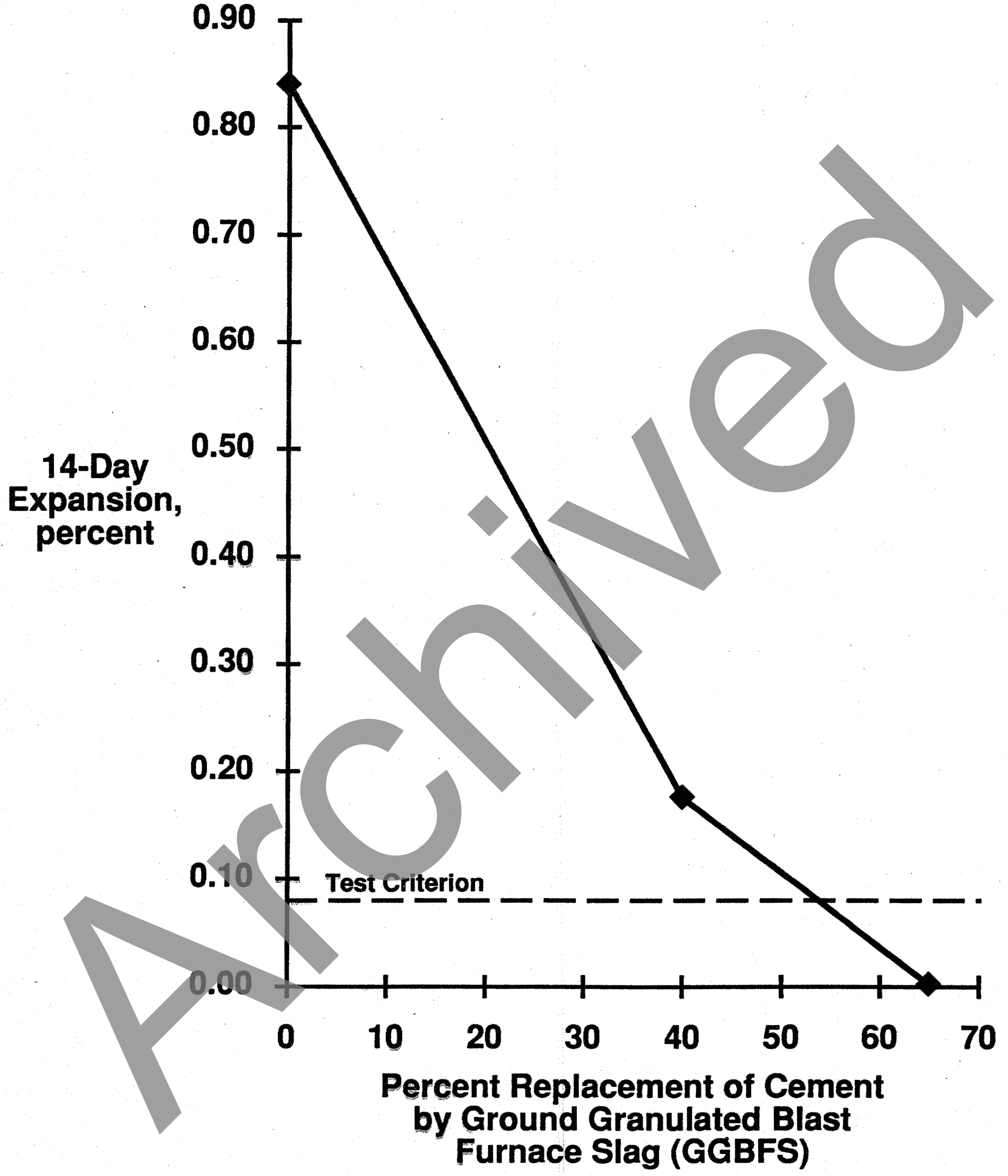


Fig 5-13 Modified ASTM P 214 test results for ground granulated blast furnace slag as cement replacement (w/c = 0.50).

SESSION 6 - PREVENTING ASR IN NEW CONSTRUCTION

Notes

I. Introduction

Several options based on local feasibility are available to prevent deleterious ASR in future construction as described below (Fig 6-1). Three of these, A, B, and C, have long-standing usage and are still applicable today. The fourth approach, the introduction of lithium compounds into concrete, D, represents SHRP development of an idea first presented in the early 1950's, and is still in the experimental stage in field installations.

II. Methods

A) Avoid the use of potentially deleteriously reactive aggregate (Fig 6-2).

This precaution includes an understanding of the nature of aggregates and requires suitable laboratory testing and careful review of field service record. The latter requires that all pertinent data, particularly cement alkali level, be identified. Environmental conditions and age also are important. Records retained by the specifier or owner, and by various materials suppliers should be reviewed. Where candidate structures for review of service record are considered, it is advantageous to procure concrete cores for petrographic examination.

B) Reduce cement alkali to safe level (Fig 6-3).

In this approach, the object is to reduce the hydroxyl ion concentration in the pore solution in the concrete, thereby rendering the solution less aggressive on aggregate particles. Accordingly, resulting reaction products, if any, will have higher calcium and lower alkali contents with little or no capacity to expand with absorption of moisture. Different maximum cement alkali levels will be acceptable for different aggregates and concrete mixture proportions. This may be estimated using the rapid immersion test but the test would not take into account possible effects of other sources of alkali, such as deicer salts or seawater..

The ASTM suggested specification of 0.60% equivalent Na_2O maximum for cement where deleteriously reactive aggregate will be used may be overly restrictive and unnecessary in some cases and inadequate in others.

C) Use suitable mineral additions (Fig 6-4).

Additions of fly ash, silica fume, volcanic glass, and ground granulated blast furnace slag have been successfully used to prevent deleterious ASR. They have been added to concrete mixtures as substitutions for cement, as additions to the cement, and as components of blended cement. Mechanisms by which they operate appear to be different.

1) They may reduce paste permeability sufficiently, as proposed for ground granulated blast furnace slag, to prevent relatively rapid migration of alkali to aggregate reaction sites.

2) Mineral admixtures such as fly ash, silica fume, and volcanic glass react with calcium hydroxide released during cement hydration to produce additional calcium silicate hydrate (CSH) (Fig 6-5). The calcium content of the fly ash is important because lower-lime CSH products have greater capacity to retain alkali, thereby more effectively reducing potential for expansion. High lime fly ashes (> 10 to 15% CaO) may not result in sufficiently low lime C-S ratios in CSH products to control expansion until cement replacement levels greater than 50% are reached. Lower (< 5%) CaO fly ashes appear to have good potential to control expansions due to ASR at cement replacement levels of about 20%. The quantity of mineral admixture required to control expansion due to ASR varies with pozzolan characteristics. It is hoped that this quantity and can be determined routinely, using the rapid immersion test after additional verification of the procedure.

D) Lithium Compounds

1) Laboratory data have indicated that small additions to fresh concrete of soluble lithium compounds can be extremely effective in preventing expansions due to ASR (Fig 6-6). Lithium hydroxide (LiOH) was recommended by SHRP. However, lithium carbonate (Li_2CO_3) or lithium fluoride (LiF) also have been very effective in controlling expansion due to ASR. The quantity of $LiOH \cdot H_2O$ (lithium hydroxide monohydrate) required is about equivalent, by mass, to the Na_2O equivalent of the cement (Fig 6-7).
For example, for use with a cement with 0.60% Na_2O equivalent, 0.60% $LiOH \cdot H_2O$ by mass of cement should be used (Appendix O, SHRP C-343, Chapter 4).

2) To insure the most uniform dispersion, $LiOH \cdot H_2O$ can be dissolved in the mix water prior to introduction of cement and aggregates.

3) Lithium hydroxide is highly alkaline and should be treated as such. Provisions should be made to prevent inhalation or contact of the solution on bare skin (Appendix Q).

4) FHWA considers the use of lithium compounds experimental in new construction and their effectiveness yet to be fully evaluated.

PREVENT ASR IN NEW CONSTRUCTION

1. Avoid reactive aggregate
2. Use a cement with a safe alkali level
3. Use a suitable mineral admixture or blended cement
4. Use a lithium-based admixture

Fig 6-1 Methods of preventing deleterious ASR in new construction.

AVOID A SOURCE OF REACTIVE AGGREGATE

1. Evaluate the field service record
2. Petrographic examination
3. Rapid immersion test

Fig 6-2 Recommended actions to avoid the use of potentially deleterious reactive aggregate.

USE A CEMENT WITH A SAFE ALKALI LEVEL

1. Modified ASTM P 214 tests

2. Safe alkali level varies with materials and mix proportions

3. Do NOT assume 0.60% alkali level is safe

4. Service record is useful

Fig 6-3 Precautions to use in the selection of a cement with a safe alkali level.

USE SUITABLE MINERAL ADDITION

1. Fly ash, Ground volcanic glass, Granulated blast furnace slag, Heat-treated clay, or Silica fume
2. Can be used as a separate addition to concrete or as a component in blended cement
3. Can be evaluated by the modified ASTM P 214 test
4. Must meet ASTM C 618, C 595, C 989, or C 1240 criteria

Fig 6-4 Considerations in the use of mineral additions.

PORTLAND CEMENT - FLY ASH REACTIONS

$$C_3S + C_2S + H_2O \longrightarrow CSH + Ca(OH)_2$$

↓

Average C:S > 1.5

$$\left.\begin{matrix} CSH \\ Ca(OH)_2 \\ \text{Fly Ash} \end{matrix}\right] + H_2O \longrightarrow \text{Additional CSH}$$

↓

Average C:S < 1.5

Fig 6-5 Mechanism by which fly ash may prevent expansive ASR.

LITHIUM COMPOUND ADDITION

1. Lithium hydroxide monohydrate ($LiOH \cdot H_2O$) used at a rate of 1:1 Na_2O equivalent (%) of cement

2. Predissolve in mix water for best dispersion in concrete mixture

3. CAUTION - Product is caustic

Fig 6-6 Use of lithium compounds to prevent expansive ASR in new construction.

MODIFIED ASTM P 214 TEST RESULTS

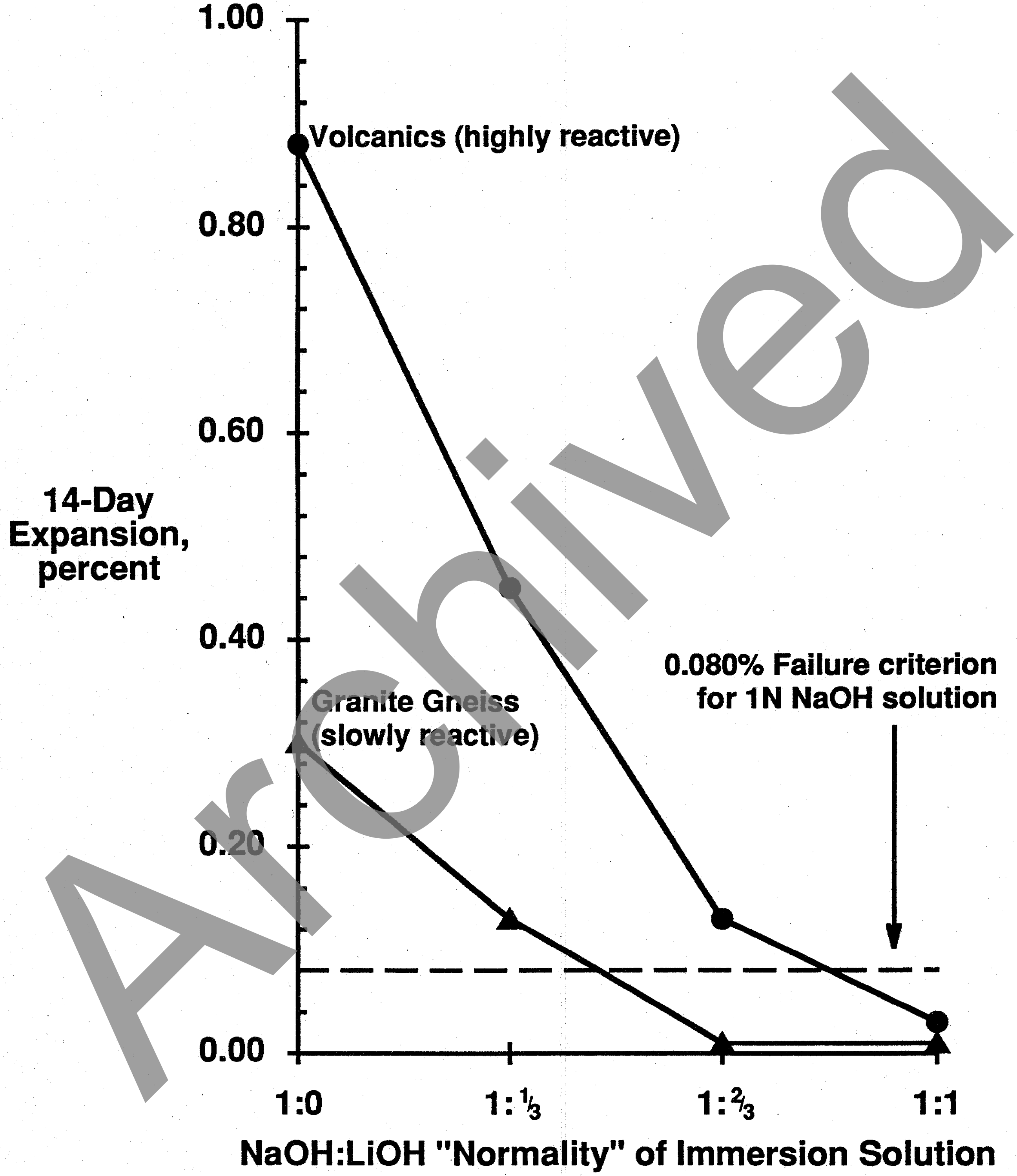


Fig 6-7 Results with LiOH added to 1 Normal NaOH solutions.

SESSION 7 - LOCAL REGION PERSPECTIVE OF ASR

Notes

This session consists of local perspectives of ASR. Participation by attendees describing local problems and resolution of ASR, is encouraged.

I. Perspective on ASR by local state highway representatives.

II. Perspective presented by a local industry representative.

III. Perspective presented by a local industry representative.

SESSION 8 - COMBATING ASR IN EXISTING CONCRETE, SUMMARY AND DISCUSSION, AND QUESTIONS AND ANSWERS

Notes

I. Combating ASR in existing hardened concrete

A pressing need in the highway industry is preventing future expansion due to ASR in existing structures. Several approaches are described below (Fig 8-1 and Appendix O, SHRP C-343, Chapter 5.0). They include interfering with the mechanism of ASR. and dealing with symptoms of ASR.

II. Interrupt Mechanisms

A) Drying - The internal relative humidity of concrete must exceed about 80% referenced to 70 to 75°F before water in the concrete is available for absorption and swelling by ASR gel(Fig 8-2). Below this level, water is sufficiently tightly adsorbed on surfaces of cement hydration products that it cannot be absorbed by ASR gel with resultant swelling. Heating is the most efficient means of drying concrete. In pavements, this method may not be effective because of ready access of subbase moisture to the concrete. In elevated structures, such as bridge deck slabs, the concrete could be sufficiently dried artificially, but retaining the concrete in this dried condition is difficult if not impossible. Sealants that "breathe" may prevent intrusion of bulk water, such as rainfall or deicer solution, but would allow penetration by water vapor, which could raise internal RH levels to near 100%. Bridge columns, for example, could be dried, but groundwater could wick upward through capillary action, thereby rewetting internal concrete to levels exceeding 80% RH. Coatings purported to seal concrete also have been found to transmit water vapor. Cyclic drying and wetting influenced by sealants may exacerbate ASR by first concentrating alkaline solutions and intensifying chemical attack on the aggregate, then allowing penetration of water vapor into the concrete with subsequent absorption and swelling by ASR gel. Thus, drying techniques would appear to require repeat applications to prevent expansion due to ASR over prolonged periods of time (Appendix O, SHRP C-343, Section 5.4).

B) Restraint - External triaxial restraint of 1.72 to 2.07 mPa (275 to 300 psi) or greater appears to be sufficient to prevent expansion due to ASR (Fig 8-3). In this approach, expansive forces due to ASR are balanced by creep, thereby resulting in no increase in volume. Uniaxial or biaxial restraint, such as imposed by dead load or embedded reinforcing steel, is always present in field structures, as evidenced by crack patterns that resulted primarily from expansive ASR. In these cases, triaxial restraint was not sufficient to prevent expansion. (Appendix O, SHRP C-343, Section 5.6).

This approach could be used for bridge columns, for example, in which the columns are wrapped cylindrically while the dead load of the bridge deck provides the required vertical restraint. Applying steel plates to flat surfaces of columns, and squeezing the columns on opposite sides also would be another approach (this method has been used apparently successfully).

C) Lithium treatment - Lithium-bearing solutions introduced into hardened concrete appear to be capable of reducing or preventing expansions due to ASR. The quantity needed is presumed to be equivalent to the alkali content of the cement, as prescribed for fresh concrete (Appendix O, SHRP C-343, Section 5.1 and Fig 8-4). The major difficulty with this approach at the present time is the rate at which solutions penetrate concrete by capillary action. Because of the extremely low permeability of mature highway concrete, ponding times for full-depth penetration may be prohibitively long.

Besides its intrinsically low permeability, concrete is normally rather highly saturated in field exposures, thereby further impeding lithium penetration. Under this circumstance, lithium concentration gradiant is virtually the only driving force by which the solution can permeate the concrete. Even utilizing existing surface cracks resulting from expansive ASR provides little increased penetrability of lithium ion. Alternate means might be developed that would enable more rapid penetration of lithium into existing concrete.

Thus far, only two experimental pavement sections have been treated - one in Delaware and one in Nevada. FHWA considers lithium treatment in hardened concrete an experimental procedure not yet fully evaluated.

III. Treat Symptoms

A) Treatment with high density weight methacrylate.

One approach is the application to pavement or bridge decks of a high density, high molecular weight, methacrylate to fill and bond together cracks present at exposed surfaces (Fig 8-5). This is expected to stiffen the concrete under load. Experience in combating the effects of ASR is limited presently to one experimental pavement in Nevada and in South Dakota (Appendix O, SHRP C-343).

Application on pavement concrete has been by brooming or squeeging on the liquid methacrylate immediately after mixing. Prior to curing the methacrylate, a fine sand layer was spread on the surface for temporary skid resistance. Handling time for the methacrylate product is of the order of one-half hour.

Performance at the Nevada site is monitored by FWD measurements and by static modulus of elasticity determinations on concrete cores. This technology is only several years old, so appraisal of this approach is still experimental.

B) Overlay - Placement of overlays on pavements would appear to be a means of rehabilitating a pavement affected by ASR. However, its effectiveness in extending pavement service life has not been systematically investigated. Undoubtedly, factors such as severity of distress due to ASR in the original concrete, intensity of continued ASR, and overlay design would need to be considered.

C) Remaining potential for expansion due to ASR

Determining the remaining potential for expansion due to ASR may have a decisive effect on approaches to combating ASR in existing concrete. If potential for expansive ASR is negligible or non-existent, the current condition of the pavement could be used in any planning rehabilitative measures. If major potential exists, then projection of future condition would be necessary. Unfortunately, a rapid and reliable test method for this purpose is not available. Such a method should be developed to facilitate planning the most economically feasible and timely approach to rehabilitation.

IV. Summary and Closeout Discussion

This section summarizes material covered in the workshop, and includes the following (Appendices R, S, and T):

A) Manifestations of distress

B) Mechanism of reaction

C) Petrographic recognition of reactive aggregates, reaction products, and associated distress

D) Testing potential for ASR

E) Options to preventing ASR in new construction

F) Means of combating ASR in existing structures

V. Questions and Answers

COMBAT ASR IN EXISTING CONCRETE

1. Dry the concrete
2. Apply lithium solution
3. External triaxial restraint
4. Apply high density methacrylate

Fig 8-1 Possible approaches to prevent distress due to ASR in existing structures.

DRYING TO PREVENT EXPANSIVE ASR

1. Internal relative humidity of concrete - less than 80%

2. Useful for elevated concrete

Fig 8-2 Application of drying to prevent distress due to ASR.

RESTRAINT TO EXPANSIVE FORCES

1. Must be triaxial

2. Minimum required pressure: 1.72 to 2.07 MPa (250 to 300 psi)

Fig 8-3 Requirements using restraint to prevent expansion due to ASR.

LITHIUM TREATMENT ON HARDENED CONCRETE

1. Use Lithium hydroxide monohydrate ($LiOH \cdot H_2O$)
2. Difficult to apply - Slow diffusion into the concrete
3. Effective where Sodium chloride (NaCl) deicer salts are used

Fig 8-4 Use of lithium compounds to prevent further expansive ASR in existing concrete.

HIGH DENSITY METHACRYLATE APPLICATION

1. Stiffens the concrete slab

2. Bonds opposite crack surfaces together

3. Penetrates 25 to 50 μm (0.001 to 0.002 inch) wide cracks

Fig 8-5 Use of methacrylate to increase the structural integrity of concrete affected by ASR.

SESSION 9 - REGIONAL TECHNICAL WORKING GROUP

Notes

I. Workshop discussion

II. Evaluate information and techniques.

III. Consensus on procedures

IV. Specifications

V. Needs for further studies

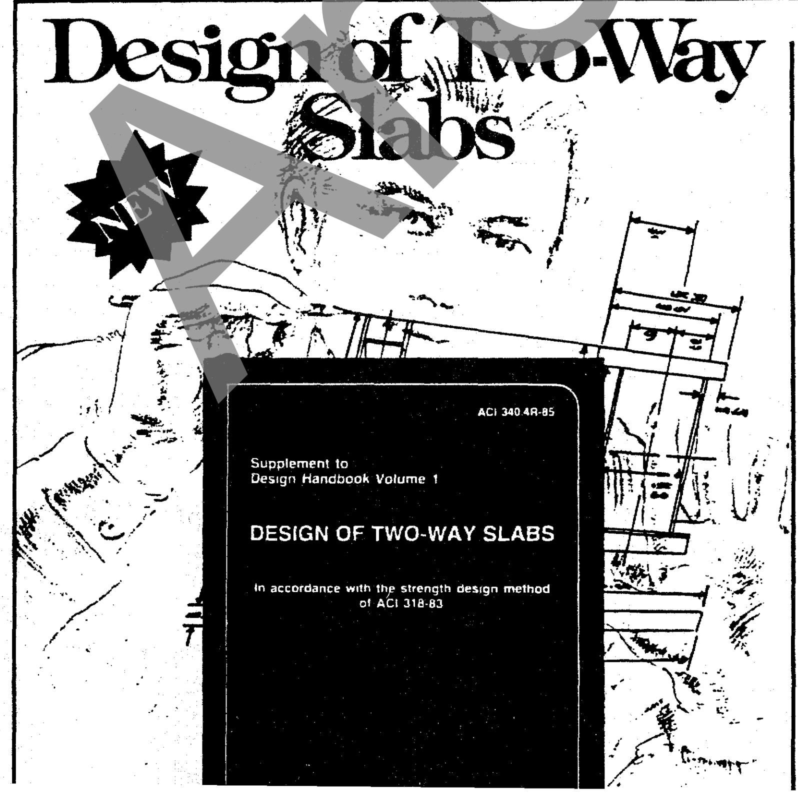
structures, even after more than 40 years under controlled environments of less than 60 percent relative humidity. One implication from this work is that residual concrete mix water may be the only moisture required for long-term expansive reactivity to occur.

Several test procedures conceived in the 1940s to identify the potential for deleterious reactivity are in use today as ASTM test methods. These include the ASTM C 227 mortar bar test and the ASTM C 289 quick chemical test for evaluating cement-aggregate combinations and unused aggregate, respectively. Limitations of these tests, such as chemical interference or extended time periods required to achieve results suggest the need for refinements and improvements, including a better understanding of acceptable cement alkali levels to permit more appropriate specifications for structures. Several possibilities are currently under development. These include rapid methods for identifying potentially reactive aggregate, the testing of concrete prisms instead of mortar bars, and utilizing the concept of total alkalinity of the concrete.

Several long standing methods that have been used to prevent expansive alkali-silica reactivity are still very effective today. Besides avoiding the use of potentially reactive aggregates, these include the use of natural and artificial pozzolans as cement additions or replacements, and the use of low-alkali cement. As noted earlier, the latter approach has not always been successful. A much better understanding of the mechanism by which pozzolans prevent expansive reactivity has been obtained in recent years from research carried out in different parts of the world.

Several aspects of alkali-silica reactivity are currently in need of further study. These include better characterization of rock types such as quartzite, which may produce slow, long-term expansion, better understanding of the effects of alkalies from sources other than portland cement, evaluation of the remaining potential for expansive reactivity in existing structures and development of cost effective means of preserving structures already affected by alkali-silica reactivity.

WE Kunze



Appendix B

SHRP-C-342 "Alkali-Silica Reactivity An Overview of Research"
(See book attached)

Standard Method of Test
for

Rapid Identification of Alkali Silica Reaction Products in Concrete

AASHTO DESIGNATION: T 299-93
SHRP DESIGNATION: 2013

1. SCOPE

1.1 This test covers the rapid visual detection of the products of alkali silica reaction in portland cement concrete.

1.2 The values stated in acceptable metric units are to be regarded as standard.

1.3 This test involves hazardous materials, operations, and equipment. It does not purport to address all of the safety problems with its use. It is the responsibility of the user of this test to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

2. REFERENCED DOCUMENTS

2.1 *AASHTO Standards:*
M 231 Weighing Devices Used in the Testing of Materials.
T 198 Splitting Tensile Strength of Cylindrical Concrete Specimens.

3. SIGNIFICANCE AND USE

3.1 This test covers the identification of alkali silica reaction products in concrete samples by means of visual observation. Test results have shown that the products formed by the reaction of alkalis and a wide variety of reactive aggregates can be identified by this method. The amount and location of the products are indicators of the development of alkali silica reaction in the concrete sample tested. This test is not meant to be used to detect the products of alkali carbonate reaction in concrete.

3.2 This test is intended to be used as one in a series of tests to diagnose alkali silica reaction in a concrete structure. The results of this test will help to confirm or deny the presence of alkali silica reaction products in the concrete structure under investigation.

3.3 Sodium present in concrete can react with the reagent in this test to form a product that fluoresces with a greenish-yellow color and which can interfere with the interpretation of the results. However, sodium is dispersed rather uniformly in the hydrated cement matrix in concrete whereas the alkali-silica reaction products are formed particularly in and around the aggregate particles. Interference by sodium from the de-icing salts or seawater can be minimized by rinsing the test surface.

3.4 Care should be taken in interpreting the results of this test when it is used on concrete surfaces subjected to wearing, erosion and abrasion, because these processes may remove reaction products and, therefore, the absence of reaction products may not be indicative of the development of the reaction in the interior of the concrete. Care should also be taken in interpreting the results for concrete showing carbonation or containing fly ash or silica fume. Both fly ash and silica fume can react with alkalies in concrete to produce reaction products similar to those produced by ASR. The extremely fine nature and dispersion of fly ash and silica fume, however, results in a product that is well dispersed. The fluorescence of such products is generally faint and uniform, but may be influenced by the dispersion of fly ash and silica fume.

4. APPARATUS, REAGENTS, AND MATERIALS

4.1 Specimen conditioning apparatus

4.1.1 Plastic beaker—500 mL capacity

4.1.2 Plastic squeeze bottle (for water)—250 mL capacity

4.1.3 Disposable polyethylene gloves

4.1.4 Protective clothing (lab coat)

4.1.5 Absorbent paper towels

4.1.6 Enclosed space for spraying reagent application

NOTE 1—A fume hood, glove box, or disposable glove bags have been found to be suitable for this purpose.

4.2 Specimen treatment apparatus

4.2.1 Medicine dropper for applying reagent.

4.2.2 Sprayer for water: A small, hand held sprayer for spraying tap water.

4.3 Reagent (uranyl acetate solution)

4.3.1 Acetic acid solution (1N)

4.3.2 Glass boiling flask—250 mL capacity

4.3.3 Two polyethylene storage bottles with tight fitting cap—100 mL capacity

4.3.4 Polythylene volumetric flask—100 mL capacity

4.3.5 Uranyl acetate powder (ACS reagent grade)

4.3.6 Balance capable of weighing up to 10 g meeting the requirements of M2 for a Class "C" balance.

4.3.7 Uranyl acetate solution prepared as follows: Weigh out 5 gm of powdered uranyl acetate. Measure 100 mL of acetic acid solution using the 100 mL volumetric flask. (Caution: Use proper lab safety precautions in handling the uranyl acetate and the acetic acid.) Transfer the measured acetic acid solution from the volumetric flask to the boiling flask. Add the 5 g of powdered uranyl acetate to the boiling flask. Warm the mixture over a low flame until the powder is dissolved. Do not bring the

From Standard Specifications for Transportation Materials and Methods of Sampling and Testing, Part II - Tests, 1993.

mixture to a boil. Remove the flask from the burner, cover, and let it cool to room temperature. Store the cooled solution at room temperature in the polyethylene storage bottle with tight fitting cap. The reagent, when stored as described, will keep for at least one year.

4.4 Shortwave (254 nm) ultraviolet (UV) lamp with peak intensity of at least 1200 μW/sq.cm. at 15 cm (6 in.).

4.5 Viewing cabinet or a dark room. The viewing cabinet can be a box, approximately 38 cm × 30 cm × 30 cm (15 in. × 12 in. × 12 in.) in size, made of steel, wood, or cardboard with an opening at the top for placing the UV lamp.

4.6 Goggles that shield short wave ultraviolet light.

NOTE 2—Because of the toxic, carcinogenic, and radioactive nature of uranyl acetate, special caution should be taken in both preparation and application of the reagent. The use of the reagents should be kept to a minimum.

5. SPECIMENS

5.1 Obtain representative samples from the structure to be evaluated in the form of cores or slabs. The number of samples and the location of samples should be specified by the engineer responsible for the service evaluation of the structure. At any given location, however, samples shall be of such a size and shape that exposed interior surfaces for testing of at least 155 sq.cm. (24 sq.in.) can be obtained from the samples. The total area of 155 sq.cm. (24 sq.in.) can be obtained from more than one sample at the given location, if necessary.

5.2 Maintain the field moisture content of the test specimen. Place the samples in plastic bags for transport to the testing location.

5.3 Break or split the specimens to expose interior fractured surfaces of the concrete. If the specimens are in the form of cores, use the splitting tensile arrangement described in T 198 to expose interior surfaces of the specimens. Conduct the test immediately on exposure of interior surfaces.

NOTE 3—The interpretation of the results of the test are simplified if the test is conducted immediately.

6. PROCEDURE

6.1 Conditioning

6.1.1 Dampen specimen with a light spray of tap water. Proceed with the conduct of the test while the specimen is still wet.

6.1.2 Pre-screening for natural fluorescence.

6.1.3 Wear protective clothing including polyethylene gloves, lab coat, and UV filtering goggles.

NOTE 4—Short wave ultraviolet light can be harmful to the eye. Normal eye glasses or plastic goggles or face shield will absorb the harmful rays.

6.1.4 If a viewing cabinet is being used, place the ultraviolet lamp in the opening on the top to allow the light to shine directly on the specimen. Place the damp specimen inside the viewing cabinet. If a darkened room is being used, place the damp specimen on the table and turn off the lights in the room.

6.1.5 Turn on the short-wave ultraviolet light in the viewing cabinet or the darkened room and shine it on the damp specimen. Use UV filtering goggles and observe and note for future comparison whether any of the aggregates fluoresce. *Note the location and nature of any fluorescence.*

NOTE 5—Non-fluorescent aggregates will appear dark while cement paste and naturally fluorescent aggregates such as opal and some slags will fluoresce mildly.

6.2 Observation

6.2.1 Wear protective clothing including polyethylene gloves, lab coat, and UV filtering goggles.

6.2.2 Conduct the test only on interior fractured surfaces of concrete specimens. If the interior surface of the specimen is not damp, dampen it using the water sprayer filled with tap water.

6.2.3 Line the bottom of the glove box, glove bag, or fume hood with absorbent paper towels. Place the squeeze bottle, medicine dropper, 500 mL plastic beaker, and the dampened specimen inside the glove box, glove bag, or fume hood.

6.2.4 Place the specimen on a paper towel. Apply the uranyl acetate solution to the wet interior surface of the specimen with a medicine dropper. Do not apply uranyl acetate on any other surface except the interior surface of the concrete specimen. Attempt to apply a minimal amount of reagent in a controllable fashion to avoid excess.

6.2.5 Allow the uranyl acetate to absorb on the interior surface of the concrete for 60 seconds.

6.2.6 After 60 seconds, hold the specimen in a vertical position over the 500 mL plastic beaker and wash the interior surface with water three times to remove excess uranyl acetate. Use the squeeze bottle filled with water for washing. Collect the wash water from the specimen in the plastic beaker.

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

6.2.8 If a viewing cabinet is being used, place the ultraviolet lamp in the opening on the top to allow the light to shine directly on the specimen. Line the bottom surface of the viewing cabinet with paper towels. If a dark room is being used, line an area of a table top with paper towels.

6.2.9 Dampen the specimen with tap water again using the water sprayer. Place the specimen inside the viewing cabinet on the paper towel. If a dark room is being used, place the wet specimen on the paper towel on the table top in the dark room and turn off the lights in the room.

6.2.10 Turn on the ultraviolet lamp and shine the light on the treated specimen. The specimen surface will fluoresce with a bright greenish-yellow color.

6.2.11 The presence of bright fluorescing areas in and around aggregates indicates the presence of alkali silica reaction products in the sample. If the fluorescence is present around the coarse aggregates, count the number of aggregates with fluorescence associated with them. Also count the total number of exposed coarse aggregate particles in the test sample. This procedure is not practical if the fluorescence is associated with the fine aggregates.

NOTE 6—It may be helpful for comparison and confirmation purposes to develop a set of reference samples (and photos), both with and without alkali-silica reaction, for use when performing the test.

6.2.12 Note the presence of reaction products in voids and cracks, and in and around the aggregate.

NOTE 7—The presence of reaction product in cracks can be indicative of an advanced state of deterioration due to alkali silica reaction.

NOTE 8—Sodium in concrete reacts with uranyl acetate to form a product which fluoresces with greenish-yellow color. However, sodium is dispersed in the cement paste matrix in the concrete and only increases the generally uniform background fluorescence intensity. Evidence of alkali-silica reaction product should be searched in and around the aggregate particles where a distinct greenish-yellow fluorescence should be observed.

NOTE 9—Carbonated areas of concrete may also fluoresce in the test. The fluorescence due to carbonation products, however, is uniform and is normally only present at the edges of the test specimen. If the test is conducted on a freshly broken interior surface, as prescribed, the interference due to carbonation products are minimized in the interior of the specimen.

NOTE 10—Fly ash and silica fume can react with alkalis in cement and form reaction products which fluoresce in the test. Well dispersed fly ash and silica fume, however, cause only an increase in the generally uniform background fluorescence intensity in the paste. If the fly ash and silica fume are not well dispersed or are clumped together, fluorescence will be observed as discrete "points" randomly distributed over the specimen. The size of these "points" is normally very small and can be distinguished from the bright fluorescence of alkali silica reaction products derived from the coarse and fine aggregates.

7. REPORT

7.1 The report shall include the following:

7.1.1 Identification number of the specimen, its source (in terms of the structure, and the particular location in the structure from where it was obtained), its complete description (including presence and location of reinforcing steel, presence and thickness of overlay, and presence of visible cracks and other deterioration), any other relevant data provided (such as the age of concrete, composition, mix proportion, etc.), and the method used to break the specimen to obtain interior surfaces and the approximate area of interior surfaces tested.

7.1.2 Any variation in the test method from that given in this procedure.

7.1.3 Any occurrence of any natural fluorescence before treatment with the reagent, and test results reported as the presence or absence of fluorescing reaction products in the test specimen. If fluorescing reaction products are present, detail the presence or absence of reaction products in cracks and voids in the concrete. If the products are associated with coarse aggregate, indicate the total number of coarse aggregate particles in the test surface and the number of particles with fluorescing reaction products. This is done for qualitative purposes only and it does not reflect a quantitative analysis. Also note the presence or absence of fluorescence which may be associated with carbonation products at the edges of the test specimen.

8. PRECISION AND BIAS

8.1 No statement is made about either precision or bias of this test method since the results merely indicate the presence or absence of alkali-silica reaction product in concrete.

9. KEYWORDS

9.1 Alkali Silica Reaction, Concrete.

Appendix D

MALLINCKRODT

Material Safety Data Sheet

Mallinckrodt, Inc. Science Products Division, P.O. Box M Paris, KY 40361

Emergency Telephone Number: 314-982-5000

URANYL ACETATE

PRODUCT IDENTIFICATION:

Synonyms: Bis-(aceto)dioxouranum; uranium acetate; uranium oxyacetate

Formula CAS No.: 6159-44-0 (Hydrated)

TSCA CAS No.: 541-09-3 (Anhydrated)

Molecular Weight: 424.15

Chemical Formula: $UO_2(C_2H_3O_2)_2 \cdot 2H_2O$

Hazardous Ingredients: Uranyl acetate

PRECAUTIONARY MEASURES

WARNING! HARMFUL IF SWALLOWED OR INHALED. CAUSES KIDNEY AND LIVER DAMAGE. CAUTION! RADIOACTIVE MATERIAL

Avoid breathing dust.
Keep container closed.
Use with adequate ventilation.
Wash thoroughly after handling.

EMERGENCY/FIRST AID

If swallowed, induce vomiting immediately by giving two glasses of water, or milk if available and sticking finger down throat. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases call a physician.
SEE SECTION 5.

DOT Hazard Class: Radioactive Material

SECTION 1 Physical Data

Half life : 4.51x10^9 years.

Appearance: Fine, bright yellow crystals.

Odor: Odorless or slight acetic acid odor.

Solubility: 7.69g/100g water @ 15°C (59°F).

Boiling Point: Decomposes @ 275°C (527°F).

Melting Point: Loses water of hydration @ 110°C (230°F).

Density: 2.89

Specific Activity: ca. 0.2 microcuries/gm

Vapor Density (Air=1): No information found.

Vapor Pressure (mm Hg): No information found.

Evaporation Rate: No information found.

SECTION 2 Fire and Explosion Information

Fire:
Not considered to be a fire hazard.

Explosion:
Not considered to be an explosion hazard.

Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire.

Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

SECTION 3 Reactivity Data

Stability:
Stable under ordinary conditions of use and storage.

Hazardous Decomposition Products:
Oxides of carbon and slightly radioactive uranium.

Hazardous Polymerization:
This substance does not polymerize.

Incompatibilities:
Reducing agents.

SECTION 4 Leak/Spill Disposal Information

Clean-up personnel should wear protective clothing and respiratory equipment suitable for toxic, combustible, or explosive dusts. Sweep, scoop or pick up spilled material. Vacuuming or wet sweeping may be used to avoid dust dispersal. Radioactive material. Cleanup thoroughly and package all residues for recovery or return to supplier. Disposal must comply with CFR 10 Title 40.

Reportable Quantity (RQ)(CWA/CERCLA) : 5000 lbs.

Ensure compliance with local, state and federal regulations.

SECTION 5 Health Hazard Information

A. EXPOSURE / HEALTH EFFECTS

Inhalation:
Soluble uranium salts are moderately hazardous on inhalation. Coughing, sneezing and breathing difficulty may be expected as with other nitrates, and damage to kidneys and liver may occur after continued exposure.

Ingestion:
The toxicity rating is not high (slight to moderate) due to the low absorption rate of soluble uranium compounds. However, gastrointestinal discomfort with, probably, vomiting and diarrhea may follow sizeable ingestions. Kidneys and liver may be damaged, as well.

Skin Contact:
Mild irritation, reddening and possible soreness may be experienced in cases of prolonged exposure to moist skin.

Eye Contact:
Absorption of soluble uranium compounds through eye tissues is reported. No specific symptoms of eye irritation by uranyl acetate have been found although the reddening and pain due to chemical substances can probably be expected.

Chronic Exposure:
Principal hazards are kidney and liver damage resulting from prolonged contact and absorption. Radioactivity-induced tumors or malignancies are also possible.

Aggrevation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired liver or kidney function may be more susceptible to the effects of the substance.

B. FIRST AID

Inhalation:
Remove to fresh air. Get medical attention for any breathing difficulty.

Ingestion:
If swallowed, induce vomiting immediately by giving two glasses of water, or milk if available and sticking finger down throat. Call a physician immediately. Never give anything by mouth to an unconscious person.

Skin Exposure:
Remove any contaminated clothing. Wash skin with soap or mild detergent and water for at least 15 minutes. Get medical attention if irritation develops or persists.

Eye Exposure:
Wash eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

C. TOXICITY DATA (RTECS, 1986)

No LD50/LC50 information found relating to normal routes of occupational exposure.

SECTION 6 Occupational Control Measures

Airborne Exposure Limits:
-OSHA Permissible Exposure Limit (PEL):
0.05 mg/m^3 (TWA) for Soluble Uranium Compounds as U

-ACGIH Threshold Limit Value (TLV):
0.2 mg/m^3 (TWA) as U

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, "Industrial Ventilation, A Manual of Recommended Practices", most recent edition, for details.

Personal Respirators: ***(NIOSH Approved)***
If the TLV is exceeded, a dust/mist respirator with chemical goggles may be worn, in general, up to ten times the TLV. Consult respirator supplier for limitations. Alternatively, a supplied air full facepiece respirator or airlined hood may be worn.

Skin Protection:
Wear protective gloves and clean body-covering clothing.

Eye Protection:
Use chemical safety goggles. Contact lenses should not be worn when working with this material. Maintain eye wash fountain and quick-drench facilities in work area.

SECTION 7 Storage and Special Information

Keep in a tightly closed container. Protect from direct sunlight. Store in a cool, dry, ventilated area away from sources of heat or ignition.

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SAFETY PRECAUTIONS AND DISPOSAL OF URANIUM IN THE UV LIGHT-URANYL ACETATE METHOD TO IDENTIFY ALKALI-SILICA REACTION PRODUCTS IN CONCRETE

Introduction

The rapid screening method developed by Hover and Natesaiyer at Cornell University to detect alkali-silica reactivity (ASR) in concrete is being adopted by AASHTO as T-299. The procedure calls for the use of uranyl (uranium) acetate solution and ultraviolet (UV) light. Recognizing ASR by this technique depends on the ASR gel reaction product fluorescing under UV light after drops of the uranyl acetate solution are applied to freshly prepared surfaces. In this procedure, the radioactive uranium ion from the solution substitutes for potassium and sodium in the ASR gel. After rinsing with water, the uranium is retained in the gel and displays a light green to yellow fluorescence under UV light, thereby locating a likely deposit of ASR gel. Reaction products from other aggregate components or cement paste and the solution, as they naturally develop in the concrete, that may be misinterpreted as ASR gel, are not known. The expended solution cannot be reused. Use of both the uranyl acetate and the UV light require some caution. Guidelines for their use are described below.

Uranium Acetate

Uranyl (or uranium) acetate is made from naturally occurring uranium which is weakly radioactive (about 0.2 microcuries per gram for the acetate) and emits alpha particles. Radiation is usually not detectable from a sealed plastic bottle. The principal health hazard is the toxicity of uranyl acetate when working with the crystalline compound or its solution, not the radioactivity. The route of exposure with the greatest hazard is inhalation.

Handling and Use

Uranyl acetate is a yellow crystalline solid and generally is sold as the dihydrate $[UO_2(C_2H_3O_2)_2 \cdot 2H_2O]$. It should be kept in a tightly closed container, protected from direct sunlight, and stored in a cool, dry, ventilated area away from sources of heat or ignition. Properly stored, it is believed to have indefinite shelf life. Regulations allow laboratories and businesses to purchase and use uranyl acetate in limited quantities with minimal oversight. Check with the state agency responsible for radiation safety for the specific regulations in your area. Twenty-nine states have their own regulations while 21 states are federally regulated by the Nuclear Regulatory Commission (NRC). In many states, 15 pounds may be purchased at any one time but no more than 150 pounds can be purchased in a year. No more than 150 pounds should be stockpiled at any time. Users must comply with state or federal regulations concerning hazardous materials handling and employee training and notification.

It is recommended that the uranyl acetate and acetic acid solution be prepared and stored in properly labelled plastic bottles. The bottle containing the solution should be fitted with a plastic dropper. Either a chemist or trained laboratory technician should prepare the solution. The plastic bottle and dropper for the solution should be reused for subsequent batches so that the bottle does not have to be sent for disposal.

Principal routes of exposure are inhalation and ingestion; skin contact is a secondary route of exposure. In order to minimize exposure, the following procedures should be followed:

- Local exhaust ventilation provided by a good laboratory hood should be used as much as possible when handling the solid or solutions. Eyes should be protected with chemical and UV light safety goggles or glasses. Wear chemical safety gloves and either a lab coat or apron when handling the solid, the solution, or the treated concrete.

- Minimize inhalation of the solid and droplets of the solution. Prevent dust generation when handling the solid. Apply the solution by dripping on the concrete surface. Provide a tray to catch any excess solution that drips from the sample. Prevent splashing or any type of spray. Prevention of inhalation is especially important because the effects are cumulative.

- If good exhaust ventilation is not available, and the Threshhold Limit Value is exceeded in the work area, the National Institute of Occupational Safety and Health (NIOSH-) approved personal respirators should be used. Normal treatment of concrete with the solution in a good chemical hood or an outdoor field location should not require a respirator. Respirators should be used in the laboratory or in enclosed field locations if no hood is available. Check the Material Safety Data Sheet (MSDS) airborne exposure limits if in question.

Waste Disposal

Several types of uranium-contaminated matter result from the use of the uranyl acetata-UV light method for identifying possible ASR reaction products in concrete. These include the treated concrete, containers, and towels which, for disposal purposes, would be considered hazardous waste materials.

Discussions with experts and consultants in this field indicate that the minute quantities of uranium ion used in this procedure at any given time would allow for rinsing the solution down a sink and into the sewer system. The concentration of uranium so disposed of is determined by the total quantity of water flushed into the sewer system by a facility over a period of a day or a month. Assuming that tens of thousands of gallons of water per day are likely to be passed into the sewer system from a facility where the procedure is used, it appears evident that uranium ion can be disposed of through this system without contracting with hazardous waste brokerage firms.

To take advantage of this disposal approach, it is evident that uranium ion consumed in the procedure must be taken into solution. Following are recommended steps for achieving this condition.

Laboratory Testing

1. Application of solution - Excess solution (should be only a few drops) can be rinsed down a sink drain using tapwater.

2. Treated concrete sample - The fractured surface that has been treated can be dipped in a 5 to 10% hydrochloric acid solution, under a hood, and the solution rinsed down the sink drain using tapwater. This should be done until all traces of fluorescence are removed.

3. Blotting material - Cloth material should be used for all blotting of treated surfaces and for wiping up any spilled solution. The reason for using cloth instead of a paper product is the cloth can be washed free of uranium-bearing solution and the washwater then rinsed down a sink drain using tapwater.

By following the above procedures, it should be possible to dispose of all uranium-contaminated products without recourse to using costly hazardous waste disposal brokers.

Field Testing

1. Application of solution - Excess droplet quantities of uranium acetate solution applied to vertical or horizontal bushhammered surfaces should be caught with a cloth rag. For vertical surfaces, the drops should be applied at the top edge of the bushhammered area and the rag placed at the bottom edge of the area. This step should not be necessary on horizontal surfaces.

2. Removal of solution - After testing, the treated surface layer should be bushhammered off, at the same time holding a hose attached to a vacuum cleaner, just below the bushhammer fitting at the concrete surface. Powder thus caught by the vacuum cleaner later can be dissolved in hydrochloric acid and the resulting solution, containing uranium ion washed down a sink drain using tapwater.

3. Miscellaneous - Surgical or lightweight rubber gloves should be worn throughout both the laboratory and field applications to insure that the uranium solution does not deposit on the hands. When finished, the gloves can be washed and the washwater rinsed down the sink drain. This should complete disposal of uranium ion when using this procedure. Similarly, the bushhammer fitting can be washed to dispose of any uranium still adhering to the fitting.

Comparative Examples

To provide some perspective of the intensity of the alpha radiation emitted by the uranyl acetate solution used in this test procedure, the following comparisons are made. Ingestion of 1 ml of the 5% test solution corresponds to 5.9 mREM (milliroentgen-equivalent-man) units.

1) Smoking two packs of cigarettes per day corresponds to 8,000 mREM/year.

2) Annual average for "radiation workers," such as in hospitals, is 650 mREM/year.

3) Approximate exposure from one dental X-ray is 100 mREM.

4) Annual average radiation intake from food is 20 mREM/year.

5) Average annual radiation intake from highway construction, of each person in the United States, is 4 mREM/year.

Health Hazard Information

Inhalation:

Soluble uranium salts are moderately hazardous on inhalation. Coughing, sneezing, and breathing difficulty may be expected, and damage to kidneys and liver may occur after continued exposure.

First Aid - Remove to fresh air. Get medical attention for any breathing difficulty.

Ingestion:

The toxicity rating is not high (slight to moderate) due to the low absorption rate of soluble uranium compounds. However, gastrointestinal discomfort with vomiting and diarrhea may follow sizeable ingestions. Kidneys and liver may be damaged, as well.

First Aid - If swallowed, induce vomiting immediately by giving two glasses of water or milk, if available, and sticking a finger down the throat. Call a physician immediately. Never give anything by mouth to an unconscious person.

Skin Contact:

Mild irritation, reddening and possible soreness may be experienced in cases of prolonged exposure to moist skin.

First Aid - Remove any contaminated clothing. Wash skin with soap or mild detergent and water for at least 15 minutes. Get medical attention if irritation develops or persists.

Eye Contact:

Absorption of soluble uranium compounds through eye tissues is reported. No specific symptoms of eye irritation by uranyl acetate have been found although the reddening and pain due to chemical substances can probably be expected.

First Aid - Wash eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Chronic Exposure:

Principal hazards are kidney and liver damage resulting from prolonged contact and absorption. Radioactivity induced tumors or malignancies are also possible.

Aggravation of Pre-existing Conditions:

Persons with pre-existing skin disorders or eye problems or impaired liver or kidney function may be more susceptible to the effects of the substance.

Ultraviolet Light

This procedure requires the use of 254-nanometer wavelength UV light to observe fluorescence of the uranyl acetate-treated concrete surface. It is well known that UV radiation directed at the eyes is harmful. Thus, protective eyewear which absorbs UV light should always be worn when using the UV light source or when removing it from the viewing box. If a viewing box with mounted UV light source is used, the protective eyewear is not necessary since the glass plate through which the concrete is viewed absorbs any reflected UV light.

Appendix E

Appendix E

Designation: C 856 – 83 (Reapproved 1988)$^{\epsilon 1}$

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Standard Practice for Petrographic Examination of Hardened Concrete[1]

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.

$^{\epsilon 1}$ 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 Concrete[2]

C 215 Test Method for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens[2]

C 294 Descriptive Nomenclature of Constituents of Natural Mineral Aggregates[2]

C 295 Guide for Petrographic Examination of Aggregates for Concrete[2]

C 452 Test Method for Potential Expansion of Portland Cement Mortars Exposed to Sulfate[3]

C 457 Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete[2]

C 597 Test Method for Pulse Velocity Through Concrete[2]

C 637 Specification for Aggregates for Radiation-Shielding Concrete[2]

C 638 Descriptive Nomenclature of Constituents of Aggregates for Radiation-Shielding Concrete[2]

C 803 Test Method for Penetration Resistance of Hardened Concrete[2]

C 805 Test Method for Rebound Number of Hardened Concrete[2]

C 823 Practice for Examination and Sampling of Hardened Concrete in Constructions[2]

C 944 Test Method for Abrasion Resistance of Concrete or Mortar Surfaces by the Rotating Cutter Method[2]

C 1012 Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution[3]

E 3 Methods of Preparation of Metallographic Specimens[4]

E 883 Practice for Metallographic Photomicrography[4]

2.2 *ASTM Adjunct:*

Adjunct C 856 A chart of 27 photos[5]

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

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

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[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)[6]

Coarse Aggregate	+ Fine Aggregate	+ Matrix	+ Air	+ Embedded Items
Composition: Maximum dimension,[A] in. or mm, in the range >*d*> Type: 1 Gravel 2 Crushed stone 3 Mixed 1 and 2 4 Other (name) 5 Mixed 1 +/or 2 +/or 4 If Type 1, 2, or 4, homogeneous or heterogeneous Lithologic types Coarse aggregate more than 20, 30, 40, or 50 % of total	Type: 1 Natural sand 2 Manufactured sand 3 Mixed 4 Other (name) 5 Mixed 1 +/or 2+/or 4 If Type 1, 2, or 4, homogeneous or heterogeneous	color, by comparison with National Research Council *Rock Color Chart* (1963) color distribution: 1 mottled 2 even 3 gradational changes	more than 3 % of total, predominantly in spherical voids? less than 3 % of total, abundant nonspherical voids? color differences between voids and mortar? voids empty, filled, lined, or partly filled	Type, size, location; kinds of metal; other items
Fabric: Shape Distribution Packing Grading (even, uneven, excess, or deficiency of size or sizes) Parallelism of flat sides or long axes of exposed sections, normal to direction of placement +/or parallel to formed and finished surfaces[B]	distribution particle shape grading preferred orientation } as perceptible	distribution	shape distribution grading (as perceptible) parallelism of long axes of irregular voids or sheets of voids: with each other; with flat sides or long axes of coarse aggregate	voids below horizontal or low-angle reinforcement
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 sawe in the plane of bedding (normal to the direction of placement) are likely to have inconspicuous orientation. Sections broken normal to placement in conventionally place 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 cas

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 exami nation of concrete is used are given in 4.2 through 4.5. Th probable usefulness of petrographic examination in specifi instances may be determined by discussion with an experi enced petrographer of the objectives of the investigatio proposed or underway.

4.2 *Concrete from Constructions:*

4.2.1 Determination in detail of the condition of concret 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 performanc of the concrete.

4.2.4 Determination whether the concrete in a construc tion was or was not as specified. In this case, other tests ma be required in conjunction with petrographic examination.

4.2.5 Description of the cementitious matrix, includin qualitative determination of the kind of hydraulic bind used, degree of hydration, degree of carbonation if presen evidence of unsoundness of the cement, presence of mineral admixture, the nature of the hydration product adequacy of curing, and unusually high water - cement rat of the paste.

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[6] The **boldface** numbers in parentheses refer to the list of references at the e of this practice.

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

NOTE—*Condition*—When it is examined at 6 to 10× under good light, the freshly broken surface of a concrete in good physical condition that still retains most of i natural moisture content has a luster that in mineralogical terms is subtranslucent glimmering vitreous.[A] Thin edges of splinters of the paste transmit light; reflectior 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 conditic 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 norm 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 t 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 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 Surface texture Within the piece: Grain shape Grain size extreme range observed, mm Median within range _ to _ mm Textureless (too fine to resolve) Uniform or variable within the piece From piece to piece: Intergranular bond Porosity and absorption[B] If concrete breaks through aggregate, through how much of what kind? If boundary voids, along what kind of aggregate? All? All of one kind? More than 50 % of one kind? Several kinds? Segregation	Lithologic types and mineralogy as perceptible Shape Surface texture Grading Distribution	Color Fracture around or through aggregate Contact of matrix with aggregate: close, no opening visible on sawed or broken surface; aggregate not dislodged with fingers or probe; boundary openings frequent, common, rare Width Empty Filled Cracks present, absent, result of specimen preparation, preceding specimen preparation Mineral admixtures[C] Contamination Bleeding	Grading Proportion of spherical to nonspherica Nonspherical, ellipsoidal, irregular, dis shaped Color change from interior surface to matrix Interior surface luster like rest of matrix, dull, shining Linings in voids absent, rare, commor in most, complete, partial, colorless colored, silky tufts, hexagonal tablets, gel, other Underside voids or sheets of voids ur common, small, common, abundar

[A] Dana, E. S., *Textbook of Mineralogy*, revised by W. E. Ford, John Wiley & Sons, New York, N. Y. 4th ed., 1932, pp. 273–274.

[B] Pore visible to the naked eye, or at × _, or sucks in water that is dropped on it.

[C] Dark solid spheres or hollow-centered spheres of glass, or of magnetite, or some of glass and some of magnetite, recognizable at magnification of ×9 on sawed broken surfaces. Other mineral admixtures with characteristic particles visible at low magnification are recognizable. Ground surface of concrete containing portla 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 stereomicrosc 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, fro elements in service in constructions, or from elements th have been subjected to tests or to actual or simulated servi conditions.

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

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

4.4.4 Determination of effects of use of different concret making materials, forming and molding procedures, typ and amounts of reinforcement, embedded hardware, etc.

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

4.5.1 To establish whether alkali - silica reaction has tak place, what aggregate constituents were affected, what e dence of the reaction exists, and what were the effects of t reaction on the concrete.

4.5.2 To establish whether one or more alkali - carbona reactions have taken place, which aggregate constituer were affected and what evidence of the reaction or reactio exists, and the effects of the reaction on the concr properties.

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

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 coring 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, coring, petrographic examination **(4).**
Color change—When concrete has not spalled, observe depth of pink color to estimate the fire exposure.	Concrete made with sedimentary or metamorphic aggregates shows permanent color change on heating. Color normal to 230°C; goes from pink to red from 290 to 590°C; from 590 to 900°C color changes to gray and then to buff **(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).**	
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 — 50 570 — 80 Reduction in Modulus Temperature, °C — Reduction, % 200 — 25 430 — 50 760 — 70	Determinations by compressive tests and static modulus of cores; Test Method C 805 for qualitative determination; Test Method C 597 **(4).**

anhydrous sulfates, rehydration of zeolites, wetting of clays and reactions involving solubility, oxidation, sulfates, and sulfides (see Refs **1, 2,** and **3**).[6]

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 th equipment generally used. Equipment required for fiel sampling is not listed. Any other useful equipment may b added.

5.2 *For Specimen Preparation:*

5.2.1 *Diamond Saw*—Slabbing saw with an automati 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, o other metal lap, preferably at least 16 in. (400 mm) i diameter, large enough to grind at least a 4 by 6-in. (100 b 152-mm) area.

5.2.4 *Free Abrasive Machine*, using abrasive grit in lubr cant, with sample holders rotating on a rotating table. Th type of grinding machine greatly increases the speed c preparation of finely ground surfaces.

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

5.2.6 *Hot Plate or Oven*, thermostatically controlled, t permit drying and impregnating specimens with resin or wa for preparing thin sections, ground surfaces, and polishe 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
Mineralogy, texture, fabric, variable or homogeneous. Grading; excess or deficiency of sand sizes is to be judged after examination of a series of thin sections. Grain size and nature of internal boundaries in aggregate. Classification of coarse and fine aggregate. Natural mineral aggregate or crushed stone; natural or manufactured fine aggregate. Bond with matrix; peripheral cracks inside the borders of aggregate grains; internal cracks. General microfractures if one can establish that they existed before thin-sectioning. *Alkali - carbonate reactions*—If the coarse aggregate is a carbonate rock or rocks, are there rims or partial rims depleted in calcium hydroxide? Partly dolomitic rocks that have reacted sometimes are bordered with paste free from calcium hydroxide along the dolomitic portion while the paste along the limestone portion is normal. See other comments in Column 3. *Alkali - silica reaction*—Does the aggregate contain particles of types known to be reactive (chert, novaculite, acid volcanic glass, cristobalite, tridymite, opal, bottle glass)? If quartzite, metamorphosed subgraywacke, argillite, phyllite, or any of those listed in the sentence above, are there internal cracks inside the periphery of the aggregate? Has the aggregate been gelatinized so that it has pulled off during sectioning leaving only a peripheral hull bonded to the mortar? (This last phenomenon also occurs in concrete with air-cooled slag aggregate, where it indicates reaction between cement and slag.) Cracks that appear to be tensile and to narrow from the center toward the border of the particle are also evidence of alkali - silica reaction **(6)**.	In concrete over 2 years old and normally cured, the only residual cement grains are those that were largest, which may be composed of several constituents or be of alite or belite (substituted C_3S and C_2S). The latter two may be bordered by one or two layers of gel having different indexes of refraction, or by a layer of calcium hydroxide. The largest relict grains may be truly unhydrated and retain the low (dark gray) birefringence of alite in distorted quasihexagonal sections and the visible birefringence to first-order yellow of the lamellar twins in rounded grains of belite. Interstitial aluminoferrite appears as prismatic grains ranging in color from brown to greenish brown to reddish brown and having a high refractive index and pleochroism masked by the color of the grain. Tricalcium aluminate is usually not recognized in thin section because the cubic form is isotropic or because it hydrates early in the hydration history of the concrete forming submicroscopic ettringite or tetracalcium aluminum sulfate hydrate or other tetracalcium aluminum hydrates with or without other anions. These may be visible in voids in older concrete but are best discriminated by X-ray diffraction. Cements from different sources have different colors of aluminoferrite and the calcium silicates have pale green or yellow or white shades. It should be possible to match cements from one source.	Normal cement paste consists in plane transmitted light of pale tan matter varying somewhat in index of refraction and containing relict unhydrated cement grains. In concrete sectioned at early age or not adequately cured, the paste contains unhydrated cement grains ranging down to a few micrometres in maximum size with an upper limit as large as 100 µm in maximum diameter if the cement was ground in open-circuit mills or was deliberately ground to low surface area to reduce the heat of hydration. With crossed polars, normal paste is black or very dark mottled gray with scattered anhedral poikilitic crystals or small segregations of calcium hydroxide and scattered relict grains of cement. In concrete of high water - cement ratio and siliceous aggregate, the calcium hydroxide crystals are as large as the maximum size of residual cement grains, about 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.) Cement paste in concrete that has been subjected to prolonged acid leaching is low in calcium hydroxide which is present as recrystallized virtually anhedral grains precipitated near the exterior surfaces. In concrete over 2 or 3 years old made with Type I, II, or III cement, some ettringite is to be expected as rosettes in air voids. This is a normal phenomenon; to demonstrate sulfate attack it must be established chemically that the SO_3 content of the concrete is greater than would be supplied by the original sulfate content of the cement. Ettringite in voids is not ettringite that has damaged concrete although it may accompany submicroscopic ettringite in the paste that has damaged the concrete.

5.2.8 *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 ⅜ 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× to 70× 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×, 10×, and 20 to 25×; 45 to 50× with numerical aperture 0.85 or more; assorted eyepieces having appropriate corrections and magnification 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 othe accessories.

5.3.4 *Metallographic Microscope*, with vertical illuminator, mechanical stage, metallographic objectives of low medium, and high magnification, and appropriate eyepiece to provide a range of magnifications from about 25× to 500×. Reflected polarized light should be available and

TABLE 5 Characteristics of Concrete Observed Using Microscopes

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

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

[B] Fly ash can be detected by color and shape when dark spheres are present. In concrete that has not oxidized the presence of slag may be inferred from the gre 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 *Eyepiece Micrometer*—Eyepiece micrometers calibrated using a stage micrometer are useful for measuring particles of aggregate, cement grains, calcium hydroxide and other crystals, and crack widths.

5.3.6 *Stage Micrometer*, to calibrate eyepiece microm ters.

5.3.7 *Microscope Lamps*—Many modern polarizing m croscopes have built-in illuminators which are convenie and satisfactory if, with the condenser, they can be adjust to fill the back lens of the objective of highest numeri aperture with light. If the microscope requires a separ illuminator, tungsten ribbon-filament bulbs in suitable a justable housings are satisfactory. Many kinds of illumir

TABLE 6 Secondary Deposits in Concrete[A]

Compound and Mineral Equivalent	Indexes of Refraction	Form and Occurrence
Calcium carbonate ($CaCO_3$); calcite	ω = 1.658 ϵ = 1.486	Fine-grained, white or gray masses or coatings in the cement paste, in voids, along fractures, or on exposed surfaces; very common
Calcium carbonate ($CaCO_3$); aragonite	α = 1.530 β = 1.680 γ = 1.685	Minute, white prisms or needles in voids or fractures in concrete; rare
Calcium carbonate ($CaCO_3$); vaterite	o = 1.544–1.550 E =1.640–1.650	Spherulitic, form-birefringent, white encrustations on moist-stored laboratory specimens (vaterite A); also identified in sound concrete from structures by X-ray diffraction (α-vaterite); common **(7)**
6-calcium aluminate trisulfate-32 hydrate $\{Ca_6[Al(OH_6)]_2 \cdot 24H_2O\}(SO_4)_3 \cdot 2H_2O$ **(8)**; ettringite	ω = 1.464–1.469[B] ϵ = 1.458–1.462	Fine, white fibers or needles or spherulitic growths in voids, in the cement paste, or in fractures; very commo **(1, 7)**
Tetracalcium aluminate monosulfate-12-hydrate ($3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$)	ω = 1.504 ϵ = 1.49	White to colorless, minute, hexagonal plates in voids and fractures; very rare **(7)**
Tetracalcium aluminate-13-hydrate ($Ca_4Al_2(OH)_{14} \cdot 6H_2O$)	ω = 1.53 ϵ = 1.52	Micalike, colorless, pseudohexagonal, twinned crystals in voids; very rare **(9)**
Hydrous sodium carbonate ($Na_2O \cdot CO_2 \cdot H_2O$); thermonatrite	α = 1.420 β = 1.506 γ = 1.524	Minute inclusions in alkalic silica gel; rare **(7)**
Hydrated aluminum sulfate ($2Al_2O_3 \cdot SO_3 \cdot 15H_2O$); paraluminite	α = 1.463 ± 0.003 β = 1.471 γ = 1.471	Occurring in cavities in intensely altered concrete; very rare **(9)**
Calcium sulfate dihydrate ($CaSO_4 \cdot 2H_2O$); gypsum	α = 1.521 β = 1.523 γ = 1.530	White to colorless crystals in voids, in the cement paste, or along the surfaces of aggregate particles in concrete or mortar affected by sulfate or seawater attack; unusual
Calcium hydroxide ($Ca(OH)_2$); portlandite	ω = 1.574 ϵ = 1.547	White to colorless, hexagonal plates or tablets in the cement paste, in voids, along fractures; ubiquitous in concrete
Magnesium hydroxide ($Mg(OH)_2$); brucite	ω = 1.559 ϵ = 1.580	White to yellow, fine-grained encrustations and fillings in concrete attacked by magnesian solutions or seawater: unusual **(10, 11)**
Hydrous silica ($SiO_2 \cdot nH_2O$); opal	η = 1.43 varies with water content	White to colorless, finely divided, amorphous; resulting from intense leaching or carbonation of cement paste; unusual in recognizabl proportions
Alkalic silica gel ($Na_2O \cdot K_2O \cdot CaO \cdot SiO_2$)	η = 1.46–1.53	White, yellowish, or colorless; viscous, fluid, waxy, rubbery, hard; in voids, fractures, exudations, aggregate; common **(12, 13)**
Hydrated iron oxides ($Fe_2O_3 \cdot nH_2O$); Limonite	opaque or nearly so	Brown stain in fractures and on surfaces; common
Thaumasite $\{Ca_6[Si(OH_6)]_2 \cdot 24H_2O\}(SO_4)_2(CO_3)_2$ **(8)**	ω = 1.504 ϵ = 1.468 ± 0.002[B]	Prismatic, hexagonal; capable of growing in continuity wit ettringite; in sewer pipe subject to sulfate attack, in grout, in some pavement **(14)**
Syngenite ($(K_2Ca(SO_4)_2) \cdot H_2O$)	α = 1.501 **(15)** β = 1.51 γ = 1.51	Found in cavities and zones peripheral to slate particles, fibrous form **(16)**
Hydrotalcite $Mg_{3/4}Al_{1/4}(OH)_2(CO_3)_{1/8}(H_2O)_{1/2}$ **(8)**	ω = 1.510 ± 0.003 ϵ = 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 refractio 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 occurrin 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, calibrated at a known temperature and of known therma coefficient, are preferable and should be used in a temperature-controlled room. A thermometer graduated in tenths c a degree Celsius should be used to measure air temperatur near the microscope stage so that thermal corrections c refractive index can be made if needed.

6. Selection and Use of Apparatus

6.1 Laboratories should be equipped to provide photographs, photomacrographs, and photomicrographs to illustrate significant features of the concrete. While ordinar microscope lamps are sometimes satisfactory fc photomicrography in transmitted and reflected light, lamp providing intense point or field sources, such as tungste ribbon-filament bulbs, or zirconium or carbon arcs, ar highly desirable. For much useful guidance regardin photomicrography, especially using reflected light, see Practice E 883.

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 2⅛-in. (54-mm) diameter core than with 6-in. diameter core. While it is desirable in examination and testing to have a core three times the maximum size of aggregate, this circumstance is a rare occurrence when concrete with aggregate larger than 2 in. is sampled, because of the cost of large bits and the problems of handling large cores.

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 fiel log should be provided.

7.2.2 Broken pieces of concrete from extremely deteri rated structures or pieces removed while preparing for repa work are sometimes used for petrographic examination. Th samples will be more useful if their original locations in th structure are clearly described or indicated in a sketch photographs.

7.2.3 The information provided with the samples shou include:

7.2.3.1 The location and original orientation of ea specimen (see Practice C 823),

7.2.3.2 The mixture proportions of the concrete or co cretes,

7.2.3.3 Sources of concrete-making materials and resu of tests of samples thereof,

7.2.3.4 Description of mixing, placing, consolidation, a curing methods,

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

7.2.3.6 Conditions of operation and service exposure,

7.2.3.7 The reason for and objectives of the examinatic

7.2.3.8 Symptoms believed to indicate distress or deteri ration, and

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

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

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

7.3.2 The exposure of laboratory specimens should described with test results, age at test and available t results on the aggregates, hydraulic binders, and admixtu used. This information should accompany test specime from natural exposures and concrete products or samp therefrom, if available.

8. Examination of Samples

8.1 *Choice of Procedures*—Specific techniques and pro dures employed in examination of a sample depend on purpose of the examination and the nature of the samp Procedures to be used should be chosen after the questic that the examination is intended to answer have been clea formulated. The procedures should be chosen to ans those questions as unequivocally and as economically possible. The details that need to be resolved will be dicta by the objectives of the examination and will vary different situations. Consequently, the selection and locat of specimens from the samples submitted for examinat 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 chosen during the visual and stereomicroscope examination for further processing and additional stereomicroscope study, more detailed examination using the petrographic or metallographic microscopes or by X-ray diffraction and other instrumental methods, and for other chemical or physical tests. Methods for specimen preparation are outlined in the Specimen Preparation Section. Tables 2, 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*[2]—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 photomacrographs of features of interest on a core surface, a ground surface, or an old or new crack surface, at magnifications of 0.7× to 3× are particularly useful if the specimen will be destroyed during subsequent studies. A camera equipped with a long extension bellows and lenses of focal length from 6.5 to 2.9 in. (165 to 74 mm), and a ground-glass back, is desirable.

8.3.3 Photomacrographs at magnifications of at least 3× to 10×, and sometimes at higher magnifications, may be used to illustrate reaction products or growths on the specimen or significant crack patterns. Black and white photographs such as 4 by 5 in. (102 by 127 mm) taken at 1× magnification on fine-grained film with a long gray scale, convey a great deal of information and may be more useful than a photograph taken at a higher magnification on smaller format. As a general guide, the subject should be illuminated by oblique lighting at a low angle if the relief on the subject surface is low, and at a higher angle if the relief is high. The object of the oblique illumination is to reveal detail and contrast without obscuring features by long shadow Details can also be enhanced by the use of colored filte such as Wratten G or other yellow filters. Cross lighting fro two sides is sometimes successful in revealing detail ar obliterating confusing shadows.

8.3.4 Photomicrographs of thin sections, immersic mounts, and polished sections at magnifications from 2× an upper limit appropriate to the subject and dependent the quality of the equipment and the skill of the photogr pher may be used to illustrate textures, compounds, ar crack patterns.

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

9. Specimen Preparation

9.1 *Preparation for Visual and Stereomicroscope Exar nation:*

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

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

9.1.3 It is useful to prepare at least one sawed surface grinding it with progressively finer abrasives (as described Practice C 457) until a smooth matte finish is achieved a to select areas on the matching opposing surface for prepa tion of thin sections and specimens for optical, chemic X-ray diffraction, or other examinations.

9.1.4 Specimens obtained by diamond drilling are r ordinarily damaged in the process; however, weak concr damaged by chemical attack, an alkali - aggregate reactic freezing and thawing, or several of these, will give poor cc recovery with many fractures if it is drilled with a 2⅛-in. 54-mm bit and barrel while it will give essentially comple recovery if drilled with a 6-in. (152-mm) diameter bit a barrel. This difference is particularly important in pet graphic examinations made during condition surveys of structures. Weakened concrete may also break duri sawing. The removal and preparation of specimens laboratory studies usually involves the application of fo and sometimes the application of heat to the specimen.

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

9.1.6 Heat used while impregnating concrete with th moplastic wax or resin will cause cracking if the concrete heated while it is wet, and will alter the optical properties some compounds, such as ettringite. Artifacts may theref be produced and compound identification made diffic

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 anddeleterious 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 an differences in materials, proportions, and exposure, or com binations of these. Photographs and sketches of significan features should be made before specimens are altered. Table 1 and 2 list some features to be observed during visual an stereomicroscopic examination.

10.2 *Concrete from Constructions* (Core Log):

10.2.1 Fit fragmented cores together and determine if an pieces are missing. Measure cores to verify field data an prepare a diagramatic log of each core, if necessary. The lo should be made to a scale to show relevant features. The lo may be used to show fresh and old fractures, reacte particles, reaction products, changes in size or type of coars and fine aggregates, distribution of coarse aggregate, hone comb, segregation of components, cold joints or lift or cours boundaries, location and direction of steel or other en bedded items, changes in color of paste, and other significa features. Notes should be taken and suitably referenced i the log. If cores have been suitably packed so as to preser the moisture content as drilled (which may or may not be th actual in-place moisture content) specimens should be car fully handled to preserve the moisture content and avoi breakage. Tests that may be made of specimens in th as-received moisture condition include compressive strengt static or dynamic modulus of elasticity, air content b high-pressure meter, permeability, and freezing and thawin

10.2.2 During the visual examination, general compa ison of cores representing different conditions, materials, an extent and nature of deterioration should be made an recorded. The specimens should be sorted into comparab groups based on condition or location of regions designat for more detailed examination. In some cases, during th visual examination or the stereomicroscope examinatio one or more specimens from each group may be selected f more detailed studies. If a reduced number of specimens is be selected for detailed examination, the selection should made after careful visual and stereomicroscope examinatio have been used to define one or more factors that chara terize each group.

10.3 *Specimens from Natural Exposures*—These spec mens should be laid out in logical order, either by materia proportions, age, or combinations of these, and compar with respect to composition and condition. Significant fe tures for more detailed examination should be marked a noted. Table 3 shows features of fire-damaged concrete.

10.4 *Concrete Products:*

10.4.1 The samples may consist of complete units wh they have relatively small dimensions (such as tile, block, brick), or portions removed from units by coring or sawi The samples should be organized and oriented in a man appropriate to the objectives of the examination. So, f example, portions taken from a single product unit should grouped together, and each sample should be described visual observation and measurement of dimensions in re tion to markings or labels. The following information sho be recorded or clearly marked on the samples: location in t product unit (top, side, end, interior, etc.); vertical a horizontal directions as cast or molded; outer and in ends; position in the product unit during curing peri position of the sample relative to highly stressed portions the concrete adjacent to tendon anchorages; and location 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 saltwater organisms. Such organisms may provide evidence tha a part of a structure that is not ordinarily submerged ha been submerged.

10.6.4 Quantitative determination of constituents of concrete can be made by microscopical point-count or linea traverse procedures in general accord with the requiremen of Practice C 457. A total analysis may include the proportional amounts of coarse aggregate, fine aggregate cementitious matrix, and air voids. The differentiation o coarse and fine aggregate is not accurate unless the tw fractions are distinguishable lithologically, since the maximum dimension of the particles usually is not intersected b 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 th 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 o mortar are as follows: the mounted lower surface is free o abrasive and has been ground to a smooth matte finish ordinarily obtained by finishing the surface with optica alumina or equivalent; the lower surface of the blank i 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 microfracture 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 surroundi the center and cracks in the middle of the particle whi narrow toward the border which may or may not contain in the case of alkali - silica reaction. Reaction-rimm particles may be chosen for sectioning in the case of eith kind of reaction, but it is ordinarily a futile effort; the ri distinct to the naked eye or the low power of t stereomicroscope may not be visible in thin section.

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

12. Metallographic Microscope Examination (22)

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

13. Report

13.1 The report of the examination should include following:

13.1.1 Location and orientation of the samples in 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 sam with their results,

13.1.4 Description of the samples and a report on mi proportions, if available or if estimated, workman construction practice, and original quality of the concre the constructions, insofar as such information is avail and

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

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Appendix F

SHRP-C-315, "Handbook for the Identification of Alkali-Silica Reactivity in Highway Structures"

(See book attached)

Appendix G

Designation: C 294 – 86 (Reapproved 1991)$^{\epsilon 1}$

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Standard Descriptive Nomenclature for Constituents of Natural Mineral Aggregates[1]

This standard is issued under the fixed designation C 294; 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.

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.

$^{\epsilon 1}$ NOTE—Section 25, Keywords, was added editorially in April 1991.

1. Scope

1.1 This descriptive nomenclature provides brief descriptions of some of the more common, or more important, natural materials of which mineral aggregates are composed (Note 1). The descriptions provide a basis for understanding these terms as used to designate aggregate constituents. Many of the materials described frequently occur in particles that do not display all the characteristics given in the descriptions, and most of these rocks grade from varieties meeting one description to varieties meeting another with all intermediate stages being found.

NOTE 1—These descriptions characterize minerals and rocks as they occur in nature and do not include blast-furnace slag or lightweight aggregates that are prepared by the alteration of the structure and composition of natural material. Blast-furnace slag is defined in Definitions C 125. Information about lightweight aggregates is given in Specifications C 330, C 331, and C 332.

1.2 The accurate identification of rocks and minerals can, in many cases, be made only by a qualified geologist, mineralogist, or petrographer using the apparatus and procedures of these sciences. Reference to these descriptions may, however, serve to indicate or prevent gross errors in identification. Identification of the constituent materials in an aggregate may assist in recognizing its properties, but identification alone cannot provide a basis for predicting the behavior of aggregates in service. Mineral aggregates composed of any type or combination of types of rocks and minerals may perform well or poorly in service depending upon the exposure to which they are subjected, the physical and chemical properties of the matrix in which they are embedded, their physical condition at the time they are used, and other factors. Small amounts of minerals or rocks that may occur only as contaminants or accessories in the aggregate may decisively influence its quality.

2. Referenced Documents

2.1 *ASTM Standards:*

C 125 Terminology Relating to Concrete and Concrete Aggregates[2]

C 289 Test Method for Potential Reactivity of Aggregates (Chemical Method)[2]

C 330 Specification for Lightweight Aggregates for Structural Concrete[2]

C 331 Specification for Lightweight Aggregates for Concrete Masonry Units[2]

C 332 Specification for Lightweight Aggregates for Insulating Concrete[2]

3. Classes and Types

3.1 The materials found as constituents of natural mineral aggregates are rocks and minerals. Minerals are naturally occurring inorganic substances of more or less definite chemical composition and usually of a specific crystalline structure. Most rocks are composed of several minerals but some are composed of only one mineral. Certain examples of the rock quartzite are composed exclusively of the mineral quartz, and certain limestones are composed exclusively of the mineral calcite. Individual sand grains frequently are composed of particles of rock, but they may be composed of a single mineral, particularly in the finer sizes.

3.2 Rocks are classified according to origin into three major divisions: igneous, sedimentary, and metamorphic. These three major groups are subdivided into types according to mineral and chemical composition, texture, and internal structure. *Igneous rocks* form from molten rock matter either above or below the earth's surface. *Sedimentary rocks* form at the earth's surface by the accumulation and consolidation of the products of weathering and erosion of existing rocks. *Metamorphic rocks* form from pre-existing rocks by the action of heat, pressure, or shearing forces in the earth's crust. It is obvious that not only igneous but also sedimentary and metamorphic rocks may be weathered and eroded to form new sedimentary rocks. Similarly, metamorphic rocks may again be metamorphosed.

DESCRIPTIONS OF MINERALS

4. General

4.1 For the purpose of indicating significant relationships, the descriptions of minerals are presented in groups in the following sections.

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[1] This descriptive nomenclature 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.

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This standard has been extensively revised. The reader should compare this edition with the last previous edition for exact revisions.

[2] *Annual Book of ASTM Standards*, Vol 04.02.

5. Silica Minerals

5.1 **quartz**—a very common hard mineral composed of silica (SiO_2). It will scratch glass and is not scratched by a knife. When pure it is colorless with a glassy (vitreous) luster and a shell-like (conchoidal) fracture. It lacks a visible cleavage (the ability to break in definite directions along even planes) and, when present in massive rocks such as granite, it usually has no characteristic shape. It is resistant to weathering and is therefore an important constituent of many sand and gravel deposits and many sandstones. It is also abundant in many light-colored igneous and metamorphic rocks. Some strained or intensely fractured (granulated) quartz may be deleteriously reactive with alkalies in concrete.

5.2 **opal**—a hydrous form of silica ($SiO_2 \cdot nH_2O$) which occurs without characteristic external form or internal crystalline arrangement as determined by ordinary visible light methods. When X-ray diffraction methods are used, opal may show some evidences of internal crystalline arrangement. Opal has a variable water content, generally ranging from 3 to 9 %. The specific gravity and hardness are always less than those of quartz. The color is variable and the luster is resinous to glassy. It is usually found in sedimentary rocks, especially some cherts, and is the principal constituent of diatomite. It is also found as a secondary material filling cavities and fissures in igneous rocks and may occur as a coating on gravel and sand. The recognition of opal in aggregates is important because it reacts with the alkalies in portland-cement paste, or with the alkalies from other sources, such as aggregates containing zeolites, and ground water.

5.3 **chalcedony**—chalcedony has been considered both as a distinct mineral and a variety of quartz. It is frequently composed of a mixture of microscopic fibers of quartz with a large number of submicroscopic pores filled with water and air. The properties of chalcedony are intermediate between those of opal and quartz, from which it can sometimes be distinguished only by laboratory tests. It frequently occurs as a constituent of the rock chert and is reactive with the alkalies in portland-cement paste.

5.4 **tridymite and cristobalite**—crystalline forms of silica (SiO_2) sometimes found in volcanic rocks. They are metastable at ordinary temperatures and pressures. They are rare minerals in aggregates except in areas where volcanic rocks are abundant. A type of cristobalite is a common constituent of opal. Tridymite and cristobalite are reactive with the alkalies in portland-cement paste.

6. Feldspars

6.1 The minerals of the feldspar group are the most abundant rock-forming minerals in the crust of the earth. They are important constituents of all three major rock groups, igneous, sedimentary, and metamorphic. Since all feldspars have good cleavages in two directions, particles of feldspar usually show several smooth surfaces. Frequently, the smooth cleavage surfaces show fine parallel lines. All feldspars are slightly less hard than, and can be scratched by, quartz and will, when fresh, easily scratch a penny. The various members of the group are differentiated by chemical composition and crystallographic properties. The feldspars *orthoclase, sanidine*, and *microcline* are potassium aluminum silicates, and are frequently referred to as *potassium*
feldspars. The *plagioclase* feldspars include those th
sodium aluminum silicates and calcium aluminum sil
or both sodium and calcium aluminum silicates. This
frequently referred to as the "soda-lime" group, incl
continuous series, of varying chemical compositio
optical properties, from *albite*, the sodium alun
feldspar, to *anorthite*, the calcium aluminum feldspar
intermediate members of the series designated *olig*
andesine, labradorite, and *bytownite*. Potassium fel
and sodium-rich plagioclase feldspars occur typica
igneous rocks such as granites and rhyolites, w
plagioclase feldspars of higher calcium content are fo
igneous rocks of lower silica content such as diorite, g
andesite, and basalt.

7. Ferromagnesian Minerals

7.1 Many igneous and metamorphic rocks contai
green to black minerals that are generally silicates of i
magnesium, or of both. They include the minerals
amphibole, pyroxene, and olivine groups. The
common amphibole mineral is hornblende; the
common pyroxene mineral is augite; and the most co
olivine mineral is olivine. Dark mica, such as bioti
phlogopite, are also considered ferromagnesian mi
The amphibole and pyroxene minerals are brown to g
black and generally occur as prismatic units. Oliv
usually olive green, glassy in appearance, and usually a
Biotite has excellent cleavage and can be easily cleave
thin flakes and plates. These minerals can be fou
components of a variety of rocks, and in sands and g
Olivine is found only in dark igneous rocks where qu
not present, and in sands and gravels close to the c
source.

8. Micaceous Minerals

8.1 Micaceous minerals have perfect cleavage i
direction and can be easily split into thin flakes. The
minerals of the muscovite group are colorless to light
of the biotite group, dark brown to black or dark gre
the lepidolite group, white to pink and red or yellow;
the chlorite group, shades of green. Another
phlogopite, is similar to biotite, commonly has a pea
luster and bronze color, and less commonly is brownis
green, or yellow. The mica minerals are common and
in igneous, sedimentary, and metamorphic rocks, a
common as minor to trace components in many san
gravels. The muscovite, biotite, lepidolite, and phlo
minerals cleave into flakes and plates that are elasti
chlorite minerals, by comparison, form in elastic flak
plates. Vermiculite (a mica-like mineral) forms b
alteration of other micas and is brown and has a b
luster.

9. Clay Minerals

9.1 The term "clay" refers to natural material com
of particles in a specific size range, generally less than
(0.002 mm). Mineralogically, clay refers to a group of la
silicate minerals including the clay-micas (illites), the
group, very finely divided chlorites, and the swelling c
montmorillonites (smectites). Members of several g
particularly micas, chlorites, and vermiculites, occur b

the clay-size range and in larger sizes. Some clays are made up of alternating layers of two or more clay groups. Random, regular, or both types of interlayering are known. If smectite is a significant constituent in such mixtures, then fairly large volume changes may occur with wetting and drying.

9.2 Clay minerals are hydrous aluminum, magnesium, and iron silicates that may contain calcium, magnesium, potassium, sodium and other exchangeable cations. They are formed by alteration and weathering of other silicates and volcanic glass. The clay minerals are major constituents of clays and shales. They are found disseminated in carbonate rocks as seams and pockets and in altered and weathered igneous and metamorphic rocks. Clays may also be found as matrix, void fillings, and cementing material in sandstones and other sedimentary rocks.

9.3 Most aggregate particles composed of, or containing large proportions of clay minerals are soft and, because of the large internal surface area of the constituents, they are porous. Some of these aggregates will disintegrate when wetted. Rocks in which the cementing matrix is principally clay, such as clay-bonded sandstones, and rocks in which montmorillonite is present as a continuous phase or matrix, such as some altered volcanics, may slake in water or may disintegrate in the concrete mixer. Rocks of this type are unsuitable for use as aggregates. Rocks having these properties less well developed will abrade considerably during mixing, releasing clay, and raising the water requirement of the concrete containing them. When such rocks are present in hardened concrete, the concrete will manifest greater volume change on wetting and drying than similar concrete containing non-swelling aggregate.

10. Zeolites

10.1 The zeolite minerals are a large group of hydrated aluminum silicates of the alkali and alkaline earth elements which are soft and usually white or light colored. They are formed as a secondary filling in cavities or fissures in igneous rocks, or within the rock itself as a product of hydrothermal alteration of original minerals, especially feldspars. Some zeolites, particularly *heulandite*, *natrolite*, and *laumontite*, reportedly produce deleterious effects in concrete, the first two having been reported to augment the alkali content in concrete by releasing alkalies through cation exchange and thus increasing alkali reactivity when certain siliceous aggregates are present. Laumontite and its partially dehydrated variety *leonhardite* are notable for their substantial volume change with wetting and drying. Both are found in rocks such as quartz diorites and some sandstones.

11. Carbonate Minerals

11.1 The most common carbonate mineral is *calcite* (calcium carbonate, $CaCO_3$). The mineral *dolomite* consists of calcium carbonate and magnesium carbonate ($CaCO_3 \cdot MgCO_3$ or $CaMg(CO_3)_2$) in equivalent molecular amounts, which are 54.27 and 45.73 by mass %, respectively. Both calcite and dolomite are relatively soft, the hardness of calcite being 3 and that of dolomite 3½ to 4 on the Mohs scale, and are readily scratched by a knife blade. They have rhombohedral cleavage, which results in their breaking into fragments with smooth parallelogram shaped sides. Calcite is soluble with vigorous effervescence in cold dilute hydrochloric acid; dolomite is soluble with slow effervescence cold dilute hydrochloric acid and with vigorous effervesce if the acid or the sample is heated or if the sample pulverized.

12. Sulfate Minerals

12.1 Carbonate rocks and shales may contain sulfate impurities. The most abundant sulfate mineral is *gyp* (hydrous calcium sulfate; $CaSO_4 \cdot 2H_2O$); *anhydrite* (a drous calcium sulfate, $CaSO_4$) is less common. Gypsu usually white or colorless and characterized by a pe cleavage along one plane and by its softness, represe hardness of 2 on the Mohs scale; it is readily scratched b fingernail. Gypsum may form a whitish pulverulen crystalline coating on sand and gravel. It is slightly solub water.

12.2 Anhydrite resembles dolomite in hand specime has three cleavages at right angles; it is less solubl hydrochloric acid than dolomite, does not effervesce a slightly soluble in water. Anhydrite is harder than gyp Gypsum and anhydrite occurring in aggregates offer ris sulfate attack in concrete and mortar.

13. Iron Sulfide Minerals

13.1 The sulfides of iron, *pyrite*, *marcasite*, and *pyrrh* are frequently found in natural aggregates. Pyrite is fou igneous, sedimentary, and metamorphic rocks; marcas much less common and is found mainly in sedime rocks; pyrrhotite is less common but may be found in types of igneous and metamorphic rocks. Pyrite is yellow, and pyrrhotite bronze brown, and both ha metallic luster. Marcasite is also metallic but lighter in and finely divided iron sulfides are soot black. Pyrite is found in cubic crystals. Marcasite readily oxidizes wit liberation of sulfuric acid and formation of iron o hydroxides, and, to a much smaller extent, sulfates; and pyrrhotite do so less readily. Marcasite and certain of pyrite and pyrrhotite are reactive in mortar and con producing a brown stain accompanied by a volume in that has been reported as one source of popouts in con Reactive forms of iron sulfides may be recognize immersion in saturated lime water (calcium hydr solution); upon exposure to air the reactive varieties pr a brown coating within a few minutes.

14. Iron Oxide Minerals, Anhydrous and Hydrous

14.1 There are two common iron oxide minera Black, magnetic: *magnetite* (Fe_3O_4), and (*2*) red or r when powdered: *hematite* (Fe_2O_3); and one co hydrous oxide mineral, brown or yellowish: *g* ($FeO(OH)$). Another common iron-bearing mineral is weakly magnetic, *ilmenite* ($FeTiO_3$). Magnetite and il are important accessory minerals in many dark ig rocks and are common detrital minerals in sedi Hematite is frequently found as an accessory min reddish rocks. Limonite, the brown weathering proc iron-bearing minerals, is a field name for a variety of h iron oxide minerals including goethite; it frequently c adsorbed water, and various impurities such as collo crystalline silica, clay minerals, and organic matte presence of substantial amounts of soft iron-oxide m in concrete aggregate can color concrete various shades of yellow or brown. Very minor amounts of iron minerals color many rocks, such as ferruginous sandstones, shales, clayironstones, and granites. Magnetite, ilmenite, and hematite ores are used as heavy aggregates.

DESCRIPTIONS OF IGNEOUS ROCKS

15. General

15.1 Igneous rocks are those formed by cooling from a molten rock mass (magma). They may be divided into two classes: (*1*) plutonic, or intrusive, that have cooled slowly within the earth; and (*2*) volcanic, or extrusive, that formed from quickly cooled lavas. Plutonic rocks have grain sizes greater than approximately 1 mm, and are classified as coarse- or medium-grained. Volcanic rocks have grain sizes less than approximately 1 mm, and are classified as fine-grained. Volcanic rocks frequently contain glass. Both plutonic and volcanic rocks may consist of porphyries, that are characterized by the presence of large mineral grains in a fine-grained or glassy groundmass. This is the result of sharp changes in rate of cooling or other physico-chemical conditions during solidification of the melt.

15.2 Igneous rocks are usually classified and named on the basis of their texture, internal structure, and their mineral composition which in turn depends to a large extent on their chemical composition. Rocks in the plutonic class generally have chemical equivalents in the volcanic class.

16. Plutonic Rocks

16.1 **granite**—granite is a medium- to coarse-grained, light-colored rock characterized by the presence of potassium feldspar with lesser amounts of plagioclase feldspars and quartz. The characteristic potassium feldspars are orthoclase or microcline, or both; the common plagioclase feldspars are albite and oligoclase. Feldspars are more abundant than quartz. Dark-colored mica (biotite) is usually present, and light-colored mica (muscovite) is frequently present. Other dark-colored ferromagnesian minerals, especially hornblende, may be present in amounts less than those of the light-colored constituents. *Quartz-monzonite* and *granodiorite* are rocks similar to granite, but they contain more plagioclase feldspar than potassium feldspar.

16.2 **syenite**—syenite is a medium- to coarse-grained, light-colored rock composed essentially of alkali feldspars, namely microcline, orthoclase, or albite. Quartz is generally absent. Dark ferromagnesian minerals such as hornblende, biotite, or pyroxene are usually present.

16.3 **diorite**—diorite is a medium- to coarse-grained rock composed essentially of plagioclase feldspar and one or more ferromagnesian minerals such as hornblende, biotite, or pyroxene. The plagioclase is intermediate in composition, usually of the variety andesine, and is more abundant than the ferromagnesian minerals. Diorite usually is darker in color than granite or syenite and lighter than gabbro. If quartz is present, the rock is called *quartz diorite.*

16.4 **gabbro**—gabbro is a medium- to coarse-grained, dark-colored rock consisting essentially of ferromagnesian minerals and plagioclase feldspar. The ferromagnesian minerals may be pyroxenes, amphiboles, or both. The plagioclase is one of the calcium-rich varieties, namely labradorite, bytownite, or anorthite. Ferromagnesian minerals are usu ally more abundant than feldspar. *Diabase* (in Europea usage *dolerite*) is a rock of similar composition to gabbro an basalt but is intermediate in mode of origin, usually occu ring in smaller intrusions than gabbro, and having a mediu to fine-grained texture. The terms "trap" or "trap rock" a collective terms for dark-colored, medium- to fine-graine igneous rocks especially diabase and basalt.

16.5 **peridotite**—peridotite is composed of olivine ar pyroxene. Rocks composed almost entirely of pyroxene a known as *pyroxenites*, and those composed of olivine *dunites*. Rocks of these types are relatively rare but the metamorphosed equivalent, serpentinite, is more commor

16.6 **pegmatite**—extremely coarse-grained varieties of i neous rocks are known as pegmatites. These are *usual* light-colored and are most frequently equivalent to granite syenite in mineral composition.

17. Fine-Grained and Glassy Extrusive Igneous Rocks

17.1 **volcanic rock**—volcanic or extrusive rocks are th fine-grained equivalents of the coarse-and-medium-graine plutonic rocks described in Section 16. Equivalent types hav similar chemical compositions and may contain the sam minerals. Volcanic rocks commonly are so fine-grained th the individual mineral grains usually are not visible to th naked eye. Porphyritic textures are common, and the roc may be partially or wholly glassy or non-crystalline. Th glassy portion of a partially glassy rock usually has a high silica content than the crystalline portion. Some volcanic extrusive rocks may not be distinguishable in texture ar structure from plutonic or intrusive rocks that originated shallow depth.

17.2 **felsite**—light-colored, very fine-grained igneou rocks are collectively known as felsites. The felsite grou includes *rhyolite, dacite, andesite*, and *trachyte*, which a the equivalents of granite, quartz diorite, diorite, and syenit respectively. These rocks are usually light colored but the may be gray, green, dark red, or black. When they are da they may incorrectly be classed as "trap" (see 16.4). Whe they are microcrystalline or contain natural glass, rhyolite dacites, and andesites are reactive with the alkalies portland-cement concrete.

17.3 **basalt**—fine-grained extrusive equivalent of gabb and diabase. When basalt contains natural glass, the glass generally lower in silica content than that of the lighte colored extrusive rocks and hence is not deleteriously rea tive with the alkalies in portland-cement paste; howeve exceptions have been noted in the literature with respect the alkali reactivity of basaltic glasses.

17.4 **volcanic glass**—igneous rocks composed wholly glass are named on the basis of their texture and intern structure. A dense dark natural glass of high silica content called *obsidian*, while lighter colored finely vesicular glas froth filled with elongated, tubular bubbles is called *pumic* Dark-colored coarsely vesicular types containing more or le spherical bubbles are called *scoria*. Pumices are usua silica-rich (corresponding to rhyolites or dacites), where scorias usually are more basic (corresponding to basalts). high-silica glassy lava with an onion-like structure and pearly luster, containing 2 to 5 % water, is called *perli* When heated quickly to the softening temperature, perli puffs to become an artificial pumice. Glass with up to 10 % water and with a dull resinous luster is called *pitchstone.* Glassy rocks, particularly the more siliceous ones, are reactive with the alkalies in portland-cement paste.

DESCRIPTIONS OF SEDIMENTARY ROCKS

18. General

18.1 Sedimentary rocks are stratified rocks laid down for the most part under water, although wind and glacial action occasionally are important. Sediments may be composed of particles of preexisting rocks derived by mechanical agencies or they may be of chemical or organic origin. The sediments are usually indurated by cementation or compaction during geologic time, although the degree of consolidation may vary widely.

18.2 *Gravel, sand, silt,* and *clay* form the group of unconsolidated sediments. Although the distinction between these four members is made on the basis of their particle size, a general trend in the composition occurs. Gravel and, to a lesser degree, coarse sands usually consist of rock fragments; fine sands and silt consist predominantly of mineral grains; and clay exclusively of mineral grains, largely of the group of clay minerals. All types of rocks and minerals may be represented in unconsolidated sediments.

19. Conglomerates, Sandstones, and Quartzites

19.1 These rocks consist of particles of *sand* or *gravel,* or both, with or without interstitial and cementing material. If the particles include a considerable proportion of gravel, the rock is a *conglomerate.* If the particles are in the sand sizes, that is, less than 2 mm but more than 0.06 mm in major diameter, the rock is a *sandstone* or a *quartzite.* If the rock breaks around the sand grains, it is a sandstone; if the grains are largely quartz and the rock breaks through the grains, it is quartzite. Conglomerates, and sandstones are sedimentary rocks but quartzites may be sedimentary (*orthoquartzites*) or metamorphic (*metaquartzites*). The cementing or interstitial materials of sandstones may be quartz, opal, calcite, dolomite, clay, iron oxides, or other materials. These may influence the quality of a sandstone as concrete aggregate. If the nature of the cementing material is known, the rock name may include a reference to it, such as *opal-bonded sandstone* or *ferruginous conglomerate.*

19.2 **graywackes and subgraywackes**—gray to greenish gray sandstones containing angular quartz and feldspar grains, and sand-sized rock fragments in an abundant matrix resembling claystone, shale, argillite, or slate. Graywackes grade into subgraywackes, the most common sandstones of the geologic column.

19.3 **arkose**—coarse-grained sandstone derived from granite, containing conspicuous amounts of feldspar.

20. Claystones, Shales, Argillites, and Siltstones

20.1 These very fine-grained rocks are composed of, or derived by erosion of sedimentary *silts* and *clays,* or of any type of rock that contained clay. When relatively soft and massive, they are known as *claystones,* or *siltstones,* depending on the size of the majority of the particles of which they are composed. Siltstones consist predominantly of silt-sized particles (0.0625 to 0.002 mm in diameter) and are intermediate rocks between claystones and sandsto When the claystones are harder and platy or fissile, they known as *shales.* Claystones and shales may be gray, bl reddish, or green and may contain some carbonate mine (calcareous shales). A massive, firmly indurated fine-grai argillaceous rock consisting of quartz, feldspar, micaceous minerals is known as *argillite.* Argillites do slake in water as some shales do. As an aid in distinguisi these fine-grained sediments from fine-grained, foli metamorphic rocks such as slates and phyllites, it ma noted that the cleavage surfaces of shales are generally and earthy while those of slates are more lustrous. Ph has a glossier luster resembling a silky sheen.

20.2 Aggregates containing abundant shale are d mental to concrete because they can produce high shrinl but not all shales are harmful. Some argillites are alkali-s reactive.

20.3 Although aggregates which are volumetrically stable in wetting and drying are not confined to any cla rock, they do share some common characteristics. If the a matrix or continuous phase, it is usually physically and consists of material of high specific surface, freque including clay. However, no general relation has demonstrated between clay content or type of clay and volume change upon wetting and drying. Volumetri unstable aggregates do not have mineral grains of modulus interlocked in a continuous rigid structure ca of resisting volume change.

20.4 Aggregates having high elastic modulus and volume change from the wet to the dry condition contr to the volume stability of concrete by restraining the vo change of the cement paste. In a relatively few c aggregates have been demonstrated to contribute to un factory performance of concrete because they have relat large volume change from the wet to the dry cond combined with relatively low modulus of elasticity drying, such aggregates shrink away from the surrou cement paste and consequently fail to restrain its vo change with change in moisture content.

21. Carbonate Rocks

21.1 *Limestones* are the most widespread of carb rocks. They range from pure limestones consisting o mineral calcite to pure *dolomites* (dolostones) consisti the mineral dolomite. Usually they contain both miner various proportions. If 50 to 90 % is the mineral dolo the rock is called *calcitic dolomite.* Magnesium limest sometimes applied to dolomitic limestones and c dolomites but it is ambiguous and its use should be av Most carbonate rocks contain some noncarbonate imp such as quartz, chert, clay minerals, organic matter, gy and sulfides. Carbonate rocks containing 10 to 50 % sa *arenaceous* (or *sandy*) *limestones* (or *dolomites*); those taining 10 to 50 % clay are *argillaceous* (or *clayey* or *limestones* (or *dolomites*). *Marl* is a clayey limestone w fine-grained and commonly soft. Chalk is fine-textured soft, porous, and somewhat friable limestone, com chiefly of particles of microorganisms. Micrite is very textured chemically precipitated carbonate or a mech ooze of carbonate particles, usually 0.001 to 0.003 m size. The term "limerock" is not recommended.

21.2 The reaction of the dolomite in certain carbonate rocks with alkalies in portland cement paste has been found to be associated with deleterious expansion of concrete containing such rocks as coarse aggregate. Carbonate rocks capable of such reaction possess a characteristic texture and composition. The characteristic microscopic texture is that in which relatively large crystals of dolomite (rhombs) are scattered in a finer-grained matrix of micritic calcite and clay. The characteristic composition is that in which the carbonate portion consists of substantial amounts of both dolomite and calcite, and the acid-insoluble residue contains a significant amount of clay. Except in certain areas, such rocks are of relatively infrequent occurrence and seldom make up a significant proportion of the material present in a deposit of rock being considered for use in making aggregate for concrete.

22. Chert

22.1 **chert**—the general term for a group of variously colored, very fine-grained (aphanitic), siliceous rocks composed of microcrystalline or cryptocrystalline quartz, chalcedony, or opal, either singly or in combinations of varying proportions. Identification of the form or forms of silica requires careful determination of optical properties, absolute specific gravity, loss on ignition, or a combination of these characteristics. Dense cherts are very tough, with a waxy to greasy luster, and are usually gray, brown, white, or red, and less frequently, green, black or blue. Porous varieties are usually lighter in color, frequently off-white, or stained yellowish, brownish, or reddish, firm to very weak, and grade to tripoli. Ferruginous, dense, red, and in some cases, dense, yellow, brown, or green chert is sometimes called *jasper.* Dense black or gray chert is sometimes called *flint.* A very dense, even textured, light gray to white chert, composed mostly of microcrystalline to cryptocrystalline quartz, is called *novaculite.* Chert is hard (scratches glass, but is not scratched by a knife blade) and has a conchoidal (shell-like) fracture in the dense varieties, and a more splintery fracture in the porous varieties. Chert occurs most frequently as nodules, lenses, or interstitial material, in limestone and dolomite formations, as extensively bedded deposits, and as components of sand and gravel. Most cherts have been found to be alkali-silica reactive to some degree when tested with high-alkali cement, or in the quick chemical test (Test Method C 289). However, the degree of the alkali-silica reactivity and whether a given chert will produce a deleterious degree of expansion in concrete are complex functions of several factors. Among them are: the mineralogic composition and internal structure of the chert; the amount of the chert as a proportion of the aggregates; the particle-size distribution; the alkali content of the cement; and the cement content of the concrete. In the absence of information to the contrary, all chert should be regarded as potentially alkali-silica reactive if combined with high-alkali cement. However, opaline cherts may produce deleterious expansion of mortar or concrete when present in very small proportions (less than 5 % by mass of the aggregate). Cherts that are porous may be susceptible to freezing and thawing deterioration in concrete and may cause popouts or cracking of the concrete surface above the chert particle.

DESCRIPTIONS OF METAMORPHIC ROCKS

23. General

23.1 Metamorphic rocks form from igneous, sedimenta
or pre-existing metamorphic rocks in response to change
chemical and physical conditions occurring within
earth's crust after formation of the original rock. T
changes may be textural, structural, or mineralogic and m
be accompanied by changes in chemical composition. T
rocks are dense and may be massive but are more frequen
foliated (laminated or layered) and tend to break into pl
particles. Rocks formed from argillaceous rocks by dynar
metamorphism usually split easily along one plane indep
dent of original bedding; this feature is designated "pl
cleavage." The mineral composition is very variable
pending in part on the degree of metamorphism and in p
on the composition of the original rock.

23.2 Most of the metamorphic rocks may derive eit
from igneous or sedimentary rocks but a few, such
marbles and slates, originate only from sediments.

23.3 Certain phyllites, slates, and metaquartzites c
taining low-temperature silica and silicate minerals or hig
strained quartz may be deleteriously reactive when used w
cements of high alkali contents.

24. Metamorphic Rocks

24.1 **marble**—a recrystallized medium- to coarse-grai
carbonate rock composed of calcite or dolomite, or calc
and dolomite. The original impurities are present in the fo
of new minerals, such as micas, amphiboles, pyroxenes, a
graphite.

24.2 **metaquartzite**—a granular rock consisting essenti
of recrystallized quartz. Its strength and resistance to wea
ering derive from the interlocking of the quartz grains.

24.3 **slate**—a fine-grained metamorphic rock that is
tinctly laminated and tends to split into thin parallel lay
The mineral composition usually cannot be determined w
the unaided eye.

24.4 **phyllite**—a fine-grained thinly layered rock. M
erals, such as micas and chlorite, are noticeable and impa
silky sheen to the surface of schistosity. Phyllites are interm
diate between slates and schists in grain size and mine
composition. They derive from argillaceous sediment
rocks or fine-grained extrusive igneous rocks, such as felsi

24.5 **schist**—a highly layered rock tending to split i
nearly parallel planes (schistose) in which the grain is coa
enough to permit identification of the principal miner
Schists are subdivided into varieties on the basis of the m
prominent mineral present in addition to quartz or to qu
and feldspars; for instance, *mica schist. Greenschist* is a gr
schistose rock whose color is due to abundance of one
more of the green minerals, chlorite or amphibole, and
commonly derived from altered volcanic rock.

24.6 **amphibolite**—a medium- to coarse-grained da
colored rock composed mainly of hornblende
plagioclase feldspar. Its schistosity, which is due to para
alignment of hornblende grains, is commonly less obvi
than in typical schists.

24.7 **hornfels**—equigranular, massive, and usually to
rock produced by complete recrystallization of sediment
igneous, or metamorphic rocks through thermal metam

phism sometimes with the addition of components of molten rock. Their mineral compositions vary widely.

24.8 **gneiss**—one of the most common metamorphic rocks, usually formed from igneous or sedimentary rocks by a higher degree of metamorphism than the schists. It is characterized by a layered or foliated structure resulting from approximately parallel lenses and bands of platy minerals, usually micas, or prisms, usually amphiboles, and of granular minerals, usually quartz and feldspars. All intermediate varieties between gneiss and schist, and between gneiss and granite are often found in the same areas in which well-defined gneisses occur.

24.9 **serpentinite**—a relatively soft, light to dark green to almost black rock formed usually from silica-poor igneous rocks, such as pyroxenites, peridotites, and dunites. It may contain some of the original pyroxene or olivine but is largely composed of softer hydrous ferromagnesian minerals of the serpentine group. Very soft talc-like material is often present in serpentinite.

25. Keywords

25.1 aggregates; carbonates; clays; concrete; feldspars; ferromagnesian minerals; igneous rocks; iron oxides; iron sulfides; metamorphic rocks; micas; minerals; nomenclature; rocks; sedimentary rocks; silica; sulfates; zeolites

Appendix H

Designation: C 295 – 90

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Standard Guide for Petrographic Examination of Aggregates for Concrete[1]

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 guide[2] 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 Aggregates[3]

C 117 Test Method for Materials Finer than 75-μm (No. 200) Sieve in Mineral Aggregates by Washing[3]

C 136 Method for Sieve Analysis of Fine and Coarse Aggregates[3]

C 294 Descriptive Nomenclature of Constituents of Natural Mineral Aggregates[3]

C 702 Practice for Reducing Field Samples of Aggregate to Testing Size[3]

D 75 Practice for Sampling Aggregates[3]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

E 883 Guide for Metallographic Photomicrography[4]

3. Summary of Method

3.1 The specific procedures employed in the petrographi 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 optica 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 tha 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.

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

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[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.

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[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 03.01.

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 aggregates 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 aggreg
sample or samples. Flat, elongated, and thin chip-
particles in aggregate increase the mixing water requirem
and decrease concrete strength.

4.8 Petrographic examination should identify and
attention to potentially alkali-silica reactive and alk
carbonate reactive constituents, determine such constitu
quantitatively, and recommend additional tests to conf
or refute the presence in significant amounts of aggre
constituents capable of alkali reaction in concrete. This gu
is referred to in the Appendix of Specification C 33. Alk
silica reactive constituents found in aggregates include: o
cristobalite, tridymite, siliceous and some intermediate
canic glass, chert, glassy to cryptocrystalline acid volc
rocks, synthetic siliceous glasses, some argillites, phyll
metamorphic graywackes, rocks containing highly metam
phic quartz such as graywackes, phyllites, schists, gneis
gneissic granites, vein quartz, quartzite, and sandsto
Criteria are available in the mineralogic literature for ide
fying the minerals in the list above by optical propertie
X-ray diffraction, or both, and in petrographic and pe
logic literature for identifying the rocks in the list by min
composition and texture in thin section, sometimes assi
by X-ray diffraction for mineral composition. Potenti
deleterious alkali-carbonate reactive rocks are usually cal
eous dolomites or dolomitic limestones with clayey insolu
residues. Some dolomites essentially free of clay and s
very fine-grained limestones free of clay and with mi
insoluble residue, mostly quartz, are also capable of s
alkali-carbonate reactions, however, such reactions have
yet been found deleterious.

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

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

5. Apparatus and Supplies

5.1 The apparatus and supplies listed below compr
selection that will permit the use of all of the proced
described in this guide. All specific items listed have
used, in connection with the performance of petrogra
examinations, by the procedures described herein; it is
however, intended to imply that other items canno
substituted to serve similar functions. Whenever possibl
selection of particular apparatus and supplies should be
to the judgment of the petrographer who is to perform
work so that the items obtained will be those with the u
which he has the greatest experience and familiarity.
minimum equipment regarded as essential to the makin
petrographic examinations of aggregate samples are t
items, or equivalent apparatus or supplies that will serv

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*[5]—*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 *Plattner 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× to about 150×.

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 the
own thin sections.

5.3 It is necessary that facilities be available to th
petrographer to check the index of refraction of the imme
sion media. If accurate identification of materials is to b
attempted, as for example the differentiation of quartz an
chalcedony, or the differentiation of basic from intermedia
volcanic glass, the indices of refraction of the media need
be known with accuracy. Media will not be stable for ve
long periods of time and are subject to considerable variatic
due to temperature change. In laboratories not provided wi
close temperature control, it is often necessary to recalibra
immersion media several times during the course of a sing
day when accurate identifications are required. The equi
ment needed for checking immersion media consists of
Abbé refractometer. The refractometer should be equipp
with compensating prisms to read indices for sodium lig
from white light, or it should be used with a sodium a
lamp.

5.4 A laboratory that undertakes any considerab
amount of petrographic work should be provided wi
facilities to make photomicrographic records of such featur
as cannot adequately be described in words. Phot
micrographs can be taken using standard microscope lam
for illumination; however, it is recommended that whenev
possible a zirconium arc lamp be provided for this purpo
For illustrations of typical apparatus, reference may be ma
to the paper by Mather and Mather,[2] and manufacturers
microscopes equipped with cameras and photomacrograph
equipment may be consulted. Much useful guidance
garding photomicrography, especially using reflected light
found in Guide E 883.

6. Sampling

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

6.1.1 *Undeveloped quarries* should be sampled by me
of cores drilled through the entire depth expected to
exploited. Drilling of such cores should be in a direction
is essentially perpendicular to the dominant structural
ture of the rock. Massive material may be sampled by "
(53-mm (2⅛-in.) diameter) cores. Thinly bedded or com
material should be represented by cores not less than
mm (4 in.) in diameter. There should be an adeq
number of cores to cover the limits of the deposit prop
for the project. The entire footage of the recovered
should be included in the sample and accurate data give
to elevations, depths, and core losses.

6.1.2 *Operating quarries and operating sand and gr*
deposits, in which stock piles of the material produced
available, should be represented by not less than 45 kg (
lb) or 300 pieces, whichever is larger, of each size of mat

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[5] The 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.)	. . .	. . .	A
75 to 150-mm (3 to 6-in.)	. . .	. . .	300A
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.

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-μm (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.[6] 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 fo the purpose. In some instances, petrographic methods othe than microscopy, such as X-ray diffraction, may be require or might most rapidly serve to identify fine-grained roc materials.

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

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[6] Simpson, G. G., and Rowe, A., *Quantitative Zoology*, McGraw-Hill Co., In New York, NY, 1939, pp. 182–185.

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

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 containing substances known to react deleteriously in concrete, should be set aside to be examined with the petrograp microscope. If the question of reaction with the alka (sodium and potassium) of portland cement paste is imp tant in the examination of the sample, certain addition the procedure are indicated. If the coarser sand sizes cont fine-grained, possibly glassy igneous rocks, several typ particles of each variety of such rocks should be selected f more thorough examination. The petrographer should de mine the presence or absence of glass by crushing typ grains and examining them in immersion media, using petrographic microscope. In difficult or especially import cases, it may be necessary to break suspected grains an make immersion mounts of part of the grain and a t section of another part. Where the sand contains chert the potential reactivity of the chert is an important consic ation, a number of chert particles from the fractions retai on the 600-μm (No. 30) sieve should be set aside determinations of the index of refraction. Methylene b staining techniques may be useful in identifying the prese of smectite.[7]

9.1.2 *Sizes Finer than 600 μm (No. 30)*—The si fractions finer than the 600-μm (No. 30) sieve should each reduced in a sample splitter or by quartering to about 4 o g. The volume will usually be less than a level teaspoonful. some gradings the fractions retained on the 150-μm (100) and 75-μm (No. 200) sieves may be present in su small amounts that reduction is unnecessary. These sp should be further reduced on a miniature sample splitter by coning and quartering with a spatula on a clean sheet paper. The examination may be conducted as in 9.1.1. required, the petrographic microscope should be used. this purpose, a representative portion of each reduced sp should be mounted in immersion oil on a clean glass sl and covered with a clean cover glass. No entirely satisfact method of reducing a split to a predetermined number grains is known. The reduced split can be sampled spreading it in a thin layer on glass or clean paper, dragg the end of a dissecting needle moistened in immersion through the sample and transferring the grains that adhere the needle to a drop of immersion oil on a clean slide. If t is done carefully, a fairly representative sample will obtained. If the dissecting needle is made of magnetized ste a concentration of magnetic minerals may result. It is usu necessary to make several mounts of the 300-μm (No. and 150-μm (No. 100) sieve fractions to obtain at least 1 grains of each. The index of the immersion oil should selected to make the identification of the important cons uents as easy and as definite as possible. The use of immersion oil with an index of or just below the lower inc of quartz (1.544) is recommended. The slide should mounted on a petrographic microscope equipped with mechanical stage. Several traverses should be made, and ea grain that passes under the cross hair should be identifi and counted. Care should be taken to move the slide on t north-south adjustment between traverses so that no gra will be counted twice. Each sieve fraction passing t 600-μm (No. 30) and retained on the 75-μm (No. 200) sie

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[7] Higgs, Nelson B., "Preliminary Studies of Methylene Blue Adsorption a Method of Evaluating Degradable Smectite-Bearing Concrete Aggregate San *Cement and Concrete Research*, Vol. 16, 1986, pp. 528–534.

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, hardness[8], 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).[9] 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 examination of fine-grained igneous rocks, particular att tion should be directed to the nature of the ground-m This examination should include determination of the p ence or absence of opal, chalcedony, natural glass, swelling clays; if any of these are found the amount shoul estimated; if natural glass is found the type should determined.

11. Procedure for Examination of Ledge Rock

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

12. Procedure for Examination of Crushed Stone

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

13. Procedure for Examination of Manufactured Sand

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

CALCULATION AND REPORT

14. Calculation

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

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

14.1.2 Obtain the weight percent of the sieve fractic the whole sample (individual percentages retained on co utive sieves) from the grading of the sample as determine Method C 136.

14.1.3 By multiplying the percentage of the constitue the sieve fraction, determined as described above by percentage of the sieve fraction in the whole sample. tained as described above, calculate the percentage ir whole sample of that constituent in that size (weig percentage of constituents in sieve fraction, Table 2). convenient to calculate and record these percentage tenths.

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[8] Woolf, D. O., "Methods for the Determination of Soft Pieces in Aggregate," *Proceedings*, ASTM, Vol 47, 1947, p. 967.

[9] 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 (⅜-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	62	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 (⅜-in.)	4.75-mm (No. 4)	
A_1	8.7	13.0	8.9	2.1	32.7 64.6 (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 indic 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, suc 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 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]			
	19.0-mm (¾-in.)	12.5-mm (½-in.)	9.5-mm (⅜-in.)	4.75-mm (No. 4)	Condition 1	Condition 2	Condition 3	Totals
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	...	...	...	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 convenie 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 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 gra 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 *Significance of Tests and Properties of Concrete and Concrete-Making Materials, ASTM STP 169 B*, p. 197.

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 of the whole sample as traces. Table 3 is an exam constructed from the data obtained in Table 2. As convention, the total in each sieve fraction and the total the whole sample shall each be 100 % without the trac Difficulties in abiding by this convention can usually avoided by grouping minor constituents of little engineer 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 numeri limits may be applicable, to add that the material appe acceptable for use provided the applicable acceptance te are made and the results are within the appropriate lim The report should not, however, contain conclusions ot than those based upon the finding of the examination unl the additional data to support such conclusions are inclu in or with the petrographic report and the petrographer been authorized to analyze the other relevant nonpe graphic data.

15.3 The petrographic report should include recomm dations regarding any additional petrographic, chemi physical, or geological investigations that may be require evaluate adverse properties that are indicated by the pe graphic examination that has been performed. Supplem tary petrographic investigations might include qualitativ quantitative analysis of the aggregate or of selected port thereof by X-ray diffraction, differential thermal method other procedures that are directed to identification description of the constituents of the aggregate.

16. Precision and Bias

16.1 Those test methods that are used in connection this guide that have been standardized in ASTM are sub to having precision and bias sections. Those that have been standardized will each be provided with such a sect if and when standardized. None of the nonstandard procedures mentioned for optional use in this practice used in ways that lend themselves to the preparatio precision and bias statements.

Appendix I

Designation: D 75 – 87

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American Association State Highway and Transportation Officials Standard
AASHTO No.: T2

Standard Practice for Sampling Aggregates[1]

This standard is issued under the fixed designation D 75; 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.

This practice has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope*

1.1 This practice covers sampling of coarse and fine aggregates for the following purposes:

1.1.1 Preliminary investigation of the potential source of supply,

1.1.2 Control of the product at the source of supply,

1.1.3 Control of the operations at the site of use, and

1.1.4 Acceptance or rejection of the materials.

NOTE 1—Sampling plans and acceptance and control tests vary with the type of construction in which the material is used. Attention is directed to Practices E 105 and D 3665.

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

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 702 Practice for Reducing Field Samples of Aggregate to Testing Size[2]

D 2234 Test Method for Collection of a Gross Sample of Coal[3]

D 3665 Practice for Random Sampling of Construction Materials[4]

E 105 Practice for Probability Sampling of Materials[5]

E 122 Practice for Choice of Sample Size to Estimate the Average Quality of a Lot or Process[5]

E 141 Practice for Acceptance of Evidence Based on the Results of Probability Sampling[5]

3. Significance and Use

3.1 Sampling is equally as important as the testing, and the sampler shall use every precaution to obtain samples that will show the nature and condition of the materials which they represent.

3.2 Samples for preliminary investigation tests are obtained by the party responsible for development of the potential source (Note 2). Samples of materials for control of the production at the source or control of the work at the site of use are obtained by the manufacturer, contractor, or other parties responsible for accomplishing the work. Samples for tests to be used in acceptance or rejection decisions by the purchaser are obtained by the purchaser or his authorized representative.

NOTE 2—The preliminary investigation and sampling of potential aggregate sources and types occupies a very important place in determining the availability and suitability of the largest single constituent entering into the construction. It influences the type of construction from the standpoint of economics and governs the necessary material control to ensure durability of the resulting structure, from the aggregate standpoint. This investigation should be done only by a responsible trained and experienced person. For more comprehensive guidance, see the Appendix.

4. Securing Samples

4.1 *General*—Where practicable, samples to be tested for quality shall be obtained from the finished product. Samples from the finished product to be tested for abrasion loss shall not be subject to further crushing or manual reduction in particle size in preparation for the abrasion test unless the size of the finished product is such that it requires further reduction for testing purposes.

4.2 *Inspection*—The material shall be inspected to determine discernible variations. The seller shall provide suitable equipment needed for proper inspection and sampling.

4.3 *Procedure:*

4.3.1 *Sampling from a Flowing Aggregate Stream (Bins or Belt Discharge)*—Select units to be sampled by a random method, such as Practice D 3665 from the production. Obtain at least three approximately equal increments, selected at random from the unit being sampled, and combine to form a field sample whose mass equals or exceeds the minimum recommended in 4.4.2. Take each increment from the entire cross section of the material as it is being discharged. It is usually necessary to have a special device constructed for use at each particular plant. This device consists of a pan of sufficient size to intercept the entire cross section of the discharge stream and hold the required quantity of material without overflowing. A set of rails may

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[1] This practice is under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.30 on Methods of Sampling.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 05.05.

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

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

*** A Summary of Changes section appears at the end of this practice.**

be necessary to support the pan as it is passed under the discharge stream. Insofar as is possible, keep bins continuously full or nearly full to reduce segregation.

NOTE 3—Sampling the initial discharge or the final few tons from a bin or conveyor belt increases the chances of obtaining segregated material and should be avoided.

4.3.2 *Sampling from the Conveyor Belt*—Select units to be sampled by a random method, such as Practice D 3665, from the production. Obtain at least three approximately equal increments, selected at random, from the unit being sampled and combine to form a field sample whose mass equals or exceeds the minimum recommended in 4.4.2. Stop the conveyor belt while the sample increments are being obtained. Insert two templates, the shape of which conforms to the shape of the belt in the aggregate stream on the belt, and space them such that the material contained between them will yield an increment of the required weight. Carefully scoop all material between the templates into a suitable container and collect the fines on the belt with a brush and dust pan and add to the container.

4.3.3 *Sampling from Stockpiles or Transportation Units*—Avoid sampling coarse aggregate or mixed coarse and fine aggregate from stockpiles or transportation units whenever possible, particularly when the sampling is done for the purpose of determining aggregate properties that may be dependent upon the grading of the sample. If circumstances make it necessary to obtain samples from a stockpile of coarse aggregate or a stockpile of combined coarse and fine aggregate, design a sampling plan for the specific case under consideration. This approach will allow the sampling agency to use a sampling plan that will give a confidence in results obtained therefrom that is agreed upon by all parties concerned to be acceptable for the particular situation. The sampling plan shall define the number of samples necessary to represent lots and sublots of specific sizes. General principles for sampling from stockpiles are applicable to sampling from trucks, rail cars, barges or other transportation units. For general guidance in sampling from stockpiles, see the Appendix.

4.3.4 *Sampling from Roadway (Bases and Subbases)*—Sample units selected by a random method, such as Practice D 3665, from the construction. Obtain at least three approximately equal increments, selected at random from the unit being sampled, and combine to form a field sample whose mass equals or exceeds the minimum recommended in 4.4.2. Take all increments from the roadway for the full depth of the material, taking care to exclude any underlying material. Clearly mark the specific areas from which each increment is to be removed: a metal template placed over the area is a definite aid in securing approximately equal increment weights.

4.4 *Number and Masses of Field Samples:*

4.4.1 The number of field samples (obtained by one of the methods described in 4.3) required depends on the criticality of, and variation in, the properties to be measured. Designate each unit from which a field sample is to be obtained prior to sampling. The number of field samples from the production should be sufficient to give the desired confidence in test results.

NOTE 4—Guidance for determining the number of samples required to obtain the desired level of confidence in test results may be found in Method D 2234, Recommended Practice E 105, Recommended Practice E 122, and Recommended Practice E 141.

4.4.2 The field sample masses cited are tentative. The masses must be predicated on the type and number of tests to which the material is to be subjected and sufficient material obtained to provide for the proper execution of these tests. Standard acceptance and control tests are covered by ASTM standards and specify the portion of the field sample required for each specific test. Generally speaking, the amounts specified in Table 1 will provide adequate material for routine grading and quality analysis. Extract test portions from the field sample according to Method C 702 or as required by other applicable test methods.

5. Shipping Samples

5.1 Transport aggregates in bags or other containers so constructed as to preclude loss or contamination of any part of the sample, or damage to the contents from mishandling during shipment.

5.2 Shipping containers for aggregate samples shall have suitable individual identification attached and enclosed so that field reporting, laboratory logging, and test reporting may be facilitated.

TABLE 1 Size of Samples

Maximum Nominal Size of Aggregates[A]	Approximate Minimum Mass of Field Samples, lb (kg)[B]
Fine Aggregate	
No. 8 (2.36 mm)	25 (10)
No. 4 (4.75 mm)	25 (10)
Coarse Aggregate	
3/8 in. (9.5 mm)	25 (10)
1/2 in. (12.5 mm)	35 (15)
3/4 in. (19.0 mm)	55 (25)
1 in. (25.0 mm)	110 (50)
1 1/2 in. (37.5 mm)	165 (75)
2 in. (50 mm)	220 (100)
2 1/2 in. (63 mm)	275 (125)
3 in. (75 mm)	330 (150)
3 1/2 in. (90 mm)	385 (175)

[A] For processed aggregate the maximum nominal size of particles is the largest sieve size listed in the applicable specification, upon which any material is permitted to be retained.

[B] For combined coarse and fine aggregates (for example, base or subbase) minimum weight shall be coarse aggregate minimum plus 25 lb (10 kg).

APPENDIXES

(Nonmandatory Information)

X1. SAMPLING AGGREGATE FROM STOCKPILES OR TRANSPORTATION UNITS

X1.1 Scope

X1.1.1 In some situations it is mandatory to sample aggregates that have been stored in stockpiles or loaded into rail cars, barges, or trucks. In such cases the procedure should ensure that segregation does not introduce a serious bias in the results.

X1.2 Sampling from Stockpiles

X1.2.1 In sampling material from stockpiles it is very difficult to ensure unbiased samples, due to the segregation which often occurs when material is stockpiled, with coarser particles rolling to the outside base of the pile. For coarse or mixed coarse and fine aggregate, every effort should be made to enlist the services of power equipment to develop a separate, small sampling pile composed of materials drawn from various levels and locations in the main pile after which several increments may be combined to compose the field sample. If necessary to indicate the degree of variability existing within the main pile, separate samples should be drawn from separate areas of the pile.

X1.2.2 Where power equipment is not available, samples from stockpiles should be made up of at least three increments taken from the top third, at the mid-point, and at the bottom third of the volume of the pile. A board shoved vertically into the pile just above the sampling point aids in preventing further segregation. In sampling stockpiles of fine aggregate the outer layer, which may have become segr gated, should be removed and the sample taken from th material beneath. Sampling tubes approximately 1¼-i (30-mm) min by 6 ft (2-m) min in length may be inserte into the pile at random locations to extract a minimum five increments of material to form the sample.

X1.3 Sampling from Transportation Units

X1.3.1 In sampling coarse aggregates from railroad cars barges effort should be made to enlist the services of pow equipment capable of exposing the material at various leve and random locations. Where power equipment is nc available, a common procedure requires excavation of thre or more trenches across the unit at points that will, fror visual appearance, give a reasonable estimate of the characteristics of the load. The trench bottom should be approx mately level, at least 1 ft (0.3 m) in width and in depth belo the surface. A minimum of three increments from approx mately equally spaced points along each trench should b taken by pushing a shovel downward into the materia Coarse aggregate in trucks should be sampled in essentiall the same manner as for rail cars or barges, except fc adjusting the number of increments according to the size c the truck. For fine aggregate in transportation units, sampling tubes as described in X1.2 may be used to extract a appropriate number of increments to form the sample.

X2. EXPLORATION OF POTENTIAL AGGREGATE SOURCES

X2.1 Scope

X2.1.1 Sampling for evaluation of potential aggregate sources should be performed by a responsible trained and experienced person. Because of the wide variety of conditions under which sampling may have to be done it is not possible to describe detailed procedures applicable to all circumstances. This appendix is intended to provide general guidance and list more comprehensive references.

X2.2 Sampling Stone from Quarries or Ledges

X2.2.1 *Inspection*—The ledge or quarry face should be inspected to determine discernible variations or strata. Differences in color and structure should be recorded.

X2.2.2 *Sampling and Size of Sample*—Separate samples having a mass of at least 50 lb (approximately 25 kg) should be obtained from each discernible stratum. The sample should not include material weathered to such an extent that it is no longer suitable for the purpose intended. One or more pieces in each sample should be at least 6 by 6 by 4 in. (150 by 150 by 100 mm) in size with the bedding plane plainly marked, and this piece should be free of seams or fractures.

X2.2.3 *Record*—In addition to the general informatio accompanying all samples the following information shoul accompany samples taken from ledges or quarry faces:

X2.2.3.1 Approximate quantity available. (If quantity very large this may be recorded as practically unlimited.)

X2.2.3.2 Quantity and character of overburden.

X2.2.3.3 A detailed record showing boundaries and loca tion of material represented by each sample.

NOTE—X2.1—A sketch, plan, and elevation, showing the thickne and location of the different layers is recommended for this purpose.

X2.3 Sampling Roadside or Bank Run Sand and Grave Deposits

X2.3.1 *Inspection*—Potential sources of bank run san and gravel may include previously worked pits from whic there is an exposed face or potential deposits discovere through air-photo interpretation, geophysical exploration, c other types of terrain investigation.

X2.3.2 *Sampling*—Samples should be so chosen fror each different stratum in the deposit discernible to th sampler. An estimate of the quantity of the different mate rials should be made. If the deposit is worked as an open-fac bank or pit, samples should be taken by channeling the face vertically, bottom to top, so as to represent the materials proposed for use. Overburdened or disturbed material should not be included in the sample. Test holes should be excavated or drilled at numerous locations in the deposit to determine the quality of the material and the extent of the deposit beyond the exposed face, if any. The number and depth of test holes will depend upon the quantity of the material needed, topography of the area, nature of the deposit, character of the material, and potential value of the material in the deposit. If visual inspection indicates that there is considerable variation in the material, individual samples should be selected from the material in each well defined stratum. Each sample should be thoroughly mixed and quartered if necessary so that the field sample thus obtained will be at least 25 lb (12 kg) for sand and 75 lb (35 kg) if the deposit contains an appreciable amount of coars aggregate.

X2.3.3 *Record*—In addition to the general information accompanying all samples the following information should accompany samples of bank run sand and gravel:

X2.3.3.1 Location of supply.

X2.3.3.2 Estimate of approximate quantity available.

X2.3.3.3 Quantity and character of overburden.

X2.3.3.4 Length of haul to proposed site of work.

X2.3.3.5 Character of haul (kind of road, maximum grades, etc.)

X2.3.3.6 Details as to extent and location of material represented by each sample.

NOTE—X2.2—A sketch of plans and elevations, showing the thickness and location of different layers, is recommended for this purpose.

X3. NUMBER AND SIZE OF INCREMENTS NEEDED TO ESTIMATE CHARACTER OF UNIT SAMPLED

X3.1 Scope

X3.1.1 This appendix presents the rationale used by the responsible committee in the development of this practice.

X3.2 Descriptions of Terms Specific to This Standard

X3.2.1 *field sample*—a quantity of the material to be tested of sufficient size to provide an acceptable estimate of the average quality of a unit.

X3.2.2 *lot*—a sizable isolated quantity of bulk material from a single source, assumed to have been produced by the same process (for example, a day's production or a specific mass or volume).

X3.2.3 *test portion*—a quantity of the material of sufficient size extracted from the larger field sample by a procedure designed to ensure accurate representation of the field sample, and thus of the unit sampled.

X3.2.4 *unit*—a batch or finite subdivision of a lot of bulk material (for example, a truck load or a specific area covered).

X3.3 Test Unit, Size, and Variability

X3.3.1 The unit to be represented by a single field sample should neither be so large as to mask the effects of significant variability within the unit nor be so small as to be affected by the inherent variability between small portions of any bulk material.

X3.3.2 A unit of bulk material composed of graded aggregate or aggregate mixtures might consist of a full truckload. If it were possible, the entire load might be tested; as a practical matter, a field sample is composed of three or more increments chosen at random from the material as it is loaded or unloaded from the truck. Research has shown that such a procedure permits an acceptable estimate to be made of the average gradation that might be measured from 15 or 20 increments from the truck.

X3.3.3 Significant variability with a lot of material, where it might exist, should be indicated by statistical measures, such as the standard deviation between units selected at random from within the lot.

SUMMARY OF CHANGES

This section identifies the location of selected changes to this practice that have been incorporated since the last issue. For the convenience of the user, Committee D-4 has highlighted those changes that may impact the use of this practice. This section may also include descriptions of the changes or reasons for the changes, or both.

(1) Appendix X3 was added.

Appendix J

Procedure for Microcrack Examination Using a Fluorescent Impregnant, Epi-Fluorescence Microscopy, and Polished Sections

1. Saw, lap, and polish a 0.5- to 1.0-in.-thick slice of a concrete (cylinder, core, cube, etc.). Make sure that the prepared surface is thoroughly cleaned in isopropyl alcohol in a sonic cleaner (10 min.); rinse with forceful alcohol spray. This step assumes that none of the materials are appreciably dissolved in the isopropyl alcohol. Soap and water may also be used.

2. Allow specimen to dry on the slide warmer at temperature no higher then 50°C for at least 1 hour, preferably 8 hours.

3. Using a low-viscosity fluorescent epoxy, flood the prepared surface with the liquid and place in a vacuum bell-jar, allowing the vacuum to build up several times and releasing it over a 3-5 minute period. Gently warming the epoxy resin and hardener (prior to mixing) reduces viscosity, increasing penetration.

4. Withdraw the specimen from the vacuum and, using a razor blade or edge of a glass microscope slide, strike off the fluid epoxy from the prepared surface, leaving as little epoxy as possible, and allow the epoxy to harden on the slide warmer set at no higher than 50°C.

5. Using a rotary, diamond grinding plate (#600) grit) or adhesive-backed silicon-carbide paper, the prepared surface is lapped only to the point of removing the remaining streaks, droplets, and the thin coating of epoxy on the prepared surface, followed by forceful washing with an alcohol spray.

6. With coarse polishing cloths and 5μ alumina powder, the surface is given a rough polish (3-5 minutes) and again cleaned with a sonic cleaner followed by a forceful alcohol spray.

7. Thin lines are inscribed on the specimen with a fine-point permanent ink pen and each line is numbered. Spacing between the lines is roughly 5 to 10 mm, but other choices may be suitable.

8. Using a reflected-light microscope equipped with a 75-watt xenon lamp, or other suitable light, the appropriate wavelength is produced by passing the beam through an excitation filter (EX450-490), a barrier filter (BA520), and a dichroic mirror (DM455), all in a filter block. Magnifications range from 50X to 400X; 100X is routine. Protective eyewear and a plastic light shield panel mounted on the microscope are recommended while the test is in progress.

9. Moving the specimen by hand or mechanically so that the inscribed ink line remains in the field of view, the number of intersected cracks are counted. Thus, over a traverse distance of, say, 1500 mm (specimen area = 7500 mm^2), one might record 180 crack intersections, giving a "crack index" of 180/1500 = 0.12.

10. If the project demands very quick answers, one can use fingernail hardener colored with a fluorescent material. The mixture presently used is 4 parts fingernail hardener + 1 part acetone in which a few milligrams of the fluorescent powder have been dissolved.

    A. The fluorescent mixture is brushed on a lapped or polished, dry, concrete slab. The mixture dries very quickly (too fast for vacuum application), after which most of the excess hardener (a very thin layer) is removed with a razor blade.

    B. The specimen is briefly relapped using #600-grit silicon-carbide adhesive-backed paper on a rotary wheel or silicon-carbide powder on a coarse polishing cloth or other similar materials, followed by rough polishing with 5 µm alumina on the cloth. If a fingernail hardener is used, exposure to solvents such as isopropyl alcohol should be eliminated and only soap and water used in a forceful spray for cleaning. Relapping and polishing usually takes only 3 to 5 minutes, assuming the specimen surface has been properly scraped with the razor blade.

    C. Lines are inscribed on the specimen and "linear traverse" is conducted as previously described to determine the crack index, using the microscope in reflected light.

    D. The fingernail hardener mixture is less viscous than epoxy and is similar to various low-viscosity rapid-hardening cyanoacrylate esters with which I have had similar results. These materials are soluble in acetone and alcohol, consequently soap and water are used for cleaning. For air-void filling, the fluorescent fingernail hardener mixed with a fine-grained powder such as quick-setting plaster is applied trowel-fashion with a razor blade, leaving no excess on the polished surface. The technique shows promise for automated determination of air content via image analysis. In addition, epi-fluorescence techniques can be combined with transmitted, ordinary-light examination of thin sections.

Appendix K

David Stark[1]

Alkali-Silica Reactivity: Some Reconsiderations

REFERENCE: Stark, D., "**Alkali-Silica Reactivity: Some Reconsiderations,**" *Cement, Concrete, and Aggregates*, CCAGDP, Vol. 2, No. 2, Winter 1980, pp. 92–94.

ABSTRACT: Numerous instances have been recorded where the use of low alkali cement with certain volcanic aggregates has failed to prevent deleterious alkali-silica reactivity. Present testing using the ASTM Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method) (C 227) may fail to identify this reactivity because testing is normally done with only high alkali cements. It is recommended that concurrent testing be done, using a range of both high and low alkali cements, to determine that maximum safe cement alkali level for each aggregate in question. Although the traditional limit of 0.60% alkali has prevented alkali aggregate reaction with many reactive aggregates, there are certain volcanic aggregates that require even lower alkali or the use of suitable pozzolanic materials to avoid alkali aggregate reactivity or both.

KEY WORDS: cements, alkali aggregate reactions, andesite, bridges (structures), expansion, pavements, rhyolite, opal, low alkali cement, mortar bar tests

Field and laboratory studies over the past several years have shown that the use of low alkali cement with certain volcanic aggregates in the western United States has not been effective in preventing excessive expansion and concrete deterioration caused by alkali-silica reactivity. In most instances, the aggregates in question were judged to be potentially reactive by either petrographic examination, or by the ASTM Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method) (C 227), or by the ASTM Test for Potential Reactivity of Aggregates (Chemical Method) (C 289). Accordingly, specifications were established that require the use, without additional testing, of cements with 0.60% or less alkali, in the form of equivalent sodium oxide, with these aggregates.

Apparently, the basis for this type of specification dates back to work done in the 1940s and 1950s, where it was found that excessive expansion could be avoided in laboratory tests with reactive opaline materials when cement alkali levels did not exceed 0.60% [*1–3*]. Subsequently, this finding was taken to apply to other reactive aggregate types, including andesitic and rhyolitic volcanic materials, and the 0.60% cement alkali level was interpreted as a sharp breakpoint, below which excessive expansion could be avoided. That such reasoning can be misleading in some cases will be shown in the following review of field and laboratory work at the Portland Cement Association. Recommendations will be made for more appropriate testing and evaluation of aggregate materials.

Field Observations

Deleterious alkali-silica reactivity involving low alkali cements has been identified in pavements, bridge structures, sidewalks, and curb and gutter concrete. In these cases, cracking of the severity shown in Fig. 1 was observed five to eight years after placement of the concrete. In a few cases the initial appearance of cracking has been noted only two years after construction. Records indicated that cement alkali levels in these concretes ranged from 0.45 to 0.57% as sodium oxide. The reactive rock types occurred in various alluvial deposits and were glassy volcanic materials of andesitic to rhyolitic composition, which constituted from 1 to 10% of the total aggregate. In contrast to opal, which is essentially pure silica, these reactive volcanics were alkali calcium aluminosilicates, with the glassy, or noncrystalline, silicate matrix being the reactive component. Thus, the reactive aggregate in these cases differed in some respects from opal, on which is based much of the apparent justification for present requirements for the use of low alkali cements.

Laboratory Testing

Aggregate materials were obtained from a number of sources associated with the above instances of reactivity and tested according to ASTM Test C 227. These aggregates were sized according to the recommended gradation and combined with cements ranging in alkali content from 0.35 to 0.92%. Testing was extended beyond the six-month test period to more than 36 months. Periodic examinations were made of the mortar bars for evidence of reactivity. Typical results are shown in Figs. 2 to 4.

Figure 2 presents results for Aggregate A, which contains about 10% reactive volcanic material. Also shown is the prescribed test failure criterion of 0.10% expansion at six months. Results with the 0.92% alkali cement indicate failure according to the test criterion, as was previously known, but show the aggregate to be innocuous with the two low alkali cements. However, during the extended test period these low alkali cement-aggregate combinations exceeded the test criterion in only eight months, and eventually they reached expansion levels of 0.30% or more. It is important to note that the 0.57% alkali cement was from the same source as the cement used with this aggregate in field structures where deterioration has occurred because of reactivity. In normal practice, testing would have been done only with the high alkali cement, thus establishing the basis for prescribing, simply, low alkali

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Presented at the Symposium on Nonstandard Test Methods Useful in Determining Physical-Chemical Characteristics of Cements, held in San Diego on 12 Dec. 1979 by ASTM Committee C-1 on Cement and chaired by Albert W. Isberner of the Portland Cement Association.

[1] Principal research petrographer, Portland Cement Association, 5420 Old Orchard Rd., Skokie, Ill. 60077. Member of ASTM.

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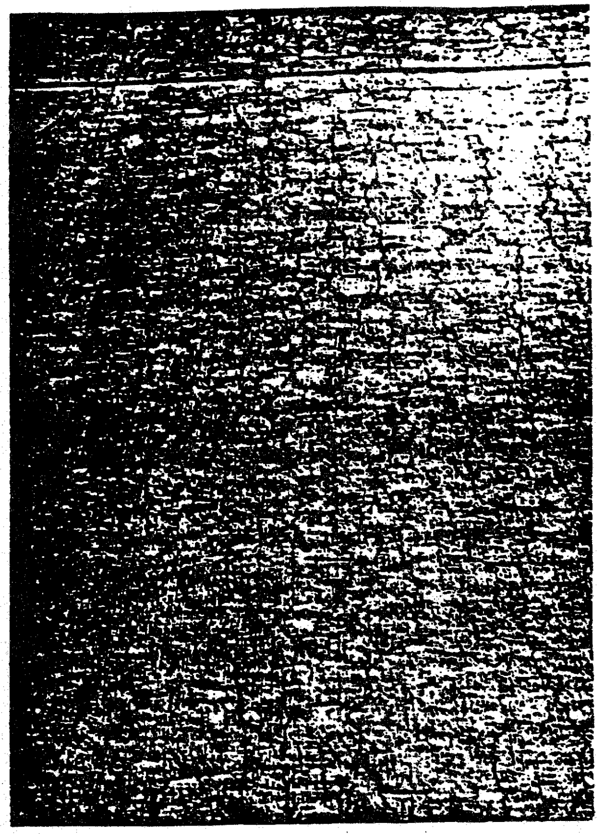

FIG. 1—*Map cracking caused by alkali-silica reactivity in pavement concrete.*

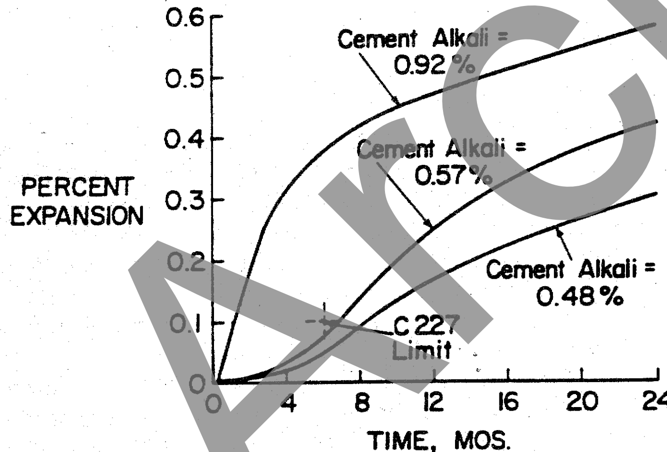

FIG. 2—*ASTM Test C 227 test results for Aggregate A.*

cement. If concurrent testing with low alkali cements had been done, and if the 0.10% failure criterion was moved to one year instead of six months, the potential deleterious reactivity of these low alkali cement-aggregate combinations would have been properly shown, thereby corroborating field performance.

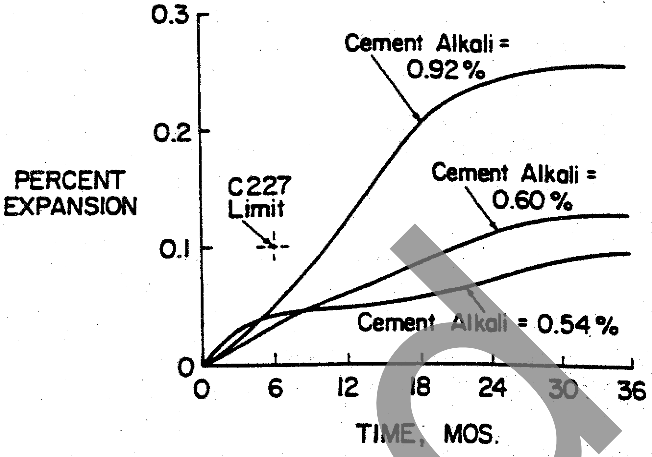

FIG. 3—*ASTM Test C 227 test results for Aggregate B.*

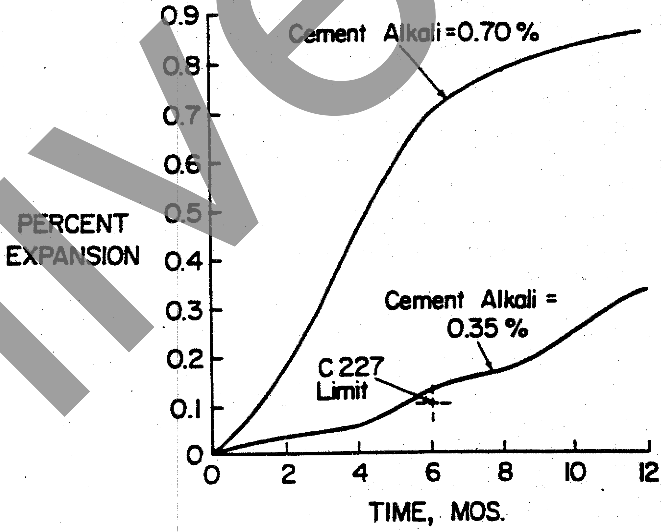

FIG. 4—*ASTM Test C 227 test results for Aggregate C.*

Results shown in Fig. 3 are for Aggregate B, which contains about 1% reactive andesitic volcanic material. The low as well as the high alkali cement-aggregate combinations easily met the existing failure criterion; however, low alkali cement-aggregate combinations have failed within eight years in pavement concrete. To identify the 0.92% alkali cement-aggregate combination as reactive would have required the reasonable test period of about ten months. To identify the 0.60 and 0.54% alkali cement-aggregate combinations would have required 21 and 36-month test periods, respectively. These longer test periods are impractical; thus a lower expansion criterion would have been necessary to identify the potential for harmful reactivity. For example, a 0.040% expansion level has been found to be meaningful for freeze-thaw testing of aggregates in concrete [*4*].

Figure 4 presents results for Aggregate C, which contains about 10% reactive glassy to cryptocrystalline volcanic andesites and rhyolites. Here, both the 0.70 and 0.35% alkali cement-aggregate combinations are indicated to be potentially reactive. In normal practice testing would have been done only with the high alkali cement, and thus a misleading recommendation would have been made to use low alkali cement even though it, too, is harmfully reactive. In this case, the present failure criterion would be acceptable, but concurrent testing with low alkali cement would have been necessary to reveal the full potential for expansive reactivity.

Discussion

The above data are typical for a larger group of aggregates that were tested according to ASTM Test C 227, for which the current test criterion and the normal practice of testing only with high alkali cements fails to characterize the full potential for reactivity as reflected in field performance. It is now evident that some adjustment is desirable in the existing test procedure, or in the failure criterion, to properly identify reactive cement-aggregate combinations. Given the present storage conditions specified in ASTM Test C 227 (specimens supported above water in sealed containers stored at 37.8° C [100°F]), at least two changes are recommended. First, the test period should be extended to twelve months and secondly, concurrent testing should be carried out with a range of both high and low alkali cements. The latter recommendation requires, most importantly, that the well-established, but erroneous, notion that the 0.60% alkali level represents a sharp breakpoint between innocuous and potentially deleterious reactivity for all aggregates regardless of exposure conditions be discarded.

That such is the case is perhaps better seen in Fig. 5, where cement-alkali level is plotted against time to 0.050% expansion for mortar bars stored under ASTM Test C 227 conditions. This expansion level was selected on the basis of its being the approximate limit above which microcracking was found to extend from reactive aggregate particles into surrounding mortar. Results are shown for Aggregates A, B, and C and for an otherwise innocuous sand that carried a 2% opal replacement, all in the 4.75-to 2.36-mm (# 4 to #8) sieve size. In Fig. 5, both the different curvatures of the individual plots (which indicate different sensitivities to unit changes in the alkali content of the cement) and the alkali range in which the time to 0.050% expansion changes significantly indicate that a single alkali level, such as 0.60%, cannot be meaningful for all aggregates even under the same storage conditions. Until these factors are taken into account, acceptance testing can only lead to further misleading recommendations for avoiding expansive reactivity in field structures. Thus, ASTM Test C 227, which does address itself to these possibilites, should be reconsidered with these recommendations in mind.

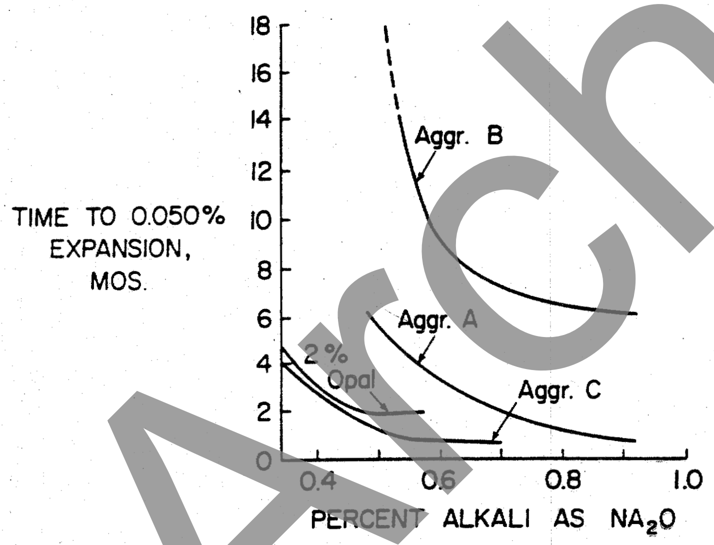

FIG.5—*Plot of ASTM Test C 227 test results showing time to 0.050% expansion.*

If testing of the type recommended above indicates cement alkali levels below those that are feasible in field construction, properly tested and accepted pozzolans or granulated slags should be considered as additives to the concrete to minimize expansive reactivity.

Summary

Field and laboratory observations indicate that deleterious alkali-silica reactivity may occur with certain glassy volcanic aggregates and low alkali cements. One approach to more completely identifying this potential is to use the ASTM Test C 227 test procedure but extend the test period to perhaps twelve months and to run concurrent tests with a range of high and low alkali cements. The work described here supports the concept that a single cement alkali level of 0.60%, although useful with many reactive aggregates, cannot be applied to all aggregates under the same or different exposure conditions.

References

[1] Stanton, T. E., "Expansion of Concrete Though Reaction Between Cement and Aggregate," *Proceedings of the American Society of Civil Engineers*, Vol. 66, 1940, pp. 1781-1811.

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[3] Powers, T. C. and Steinour, H. H.,"An Interpretation of Published Researches on the Alkali-Aggregate Reaction," *Journal of the American Concrete Institute. Proceedings*, Vol. 51, Feb. 1955, pp. 497-516, and April 1955, pp. 785-812.

[4] Klieger, P., Monfore, G., Stark, D., and Teske, W., "D-Cracking of Concrete Pavements in Ohio," Report No. OHIO-DOT-11-74, Final Report, Portland Cement Association, Skokie, Ill., Oct. 1974.

Appendix L

Designation: C 1260 – 94

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Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method)[1]

This standard is issued under the fixed designation C 1260; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method permits detection within 16 days of the potential for deleterious alkali-silica reaction of aggregate in mortar bars.

1.2 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.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* A specific precautionary statement is given in 5.3.1.

2. Referenced Documents

2.1 *ASTM Standards:*

C 109 Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)[3]

C 150 Specification for Portland Cement[2,3]

C 151 Test Method for Autoclave Expansion of Portland Cement[3]

C 227 Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)[2]

C 295 Practice for Petrographic Examination of Aggregates for Concrete[2]

C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency[3]

C 490 Specification for Apparatus for Use in Measurement of Length Change of Hardened Cement Paste, Mortar, and Concrete[2,3]

C 511 Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes[2,3]

C 856 Practice for Petrographic Examination of Hardened Concrete[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

D 1193 Specification for Reagent Water[5]

2.2 *CSA Standards:*

CAN/CSA-A23.2-14A-M90 Test Methods for Potential Expansivity of Cement-Aggregate Combination (Concrete Prism Expansion Method)[6]

3. Significance and Use

3.1 This test method provides a means of detecting the potential of an aggregate intended for use in concrete for undergoing alkali-silica reaction resulting in potentially deleterious internal expansion. It is based on the NBRI Accelerated Test Method (**1, 2, 3, 4**).[7] It may be especially useful for aggregates that react slowly or produce expansion late in the reaction. However, it does not evaluate combinations of aggregates with cementitious materials nor are the test conditions representative of those encountered by concrete in service.

3.2 Because the specimens are exposed to a NaOH solution, the alkali content of the cement is not a significant factor in affecting expansions.

3.3 When excessive expansions (see appendix) are developed, it is recommended that supplementary information be developed to confirm that the expansion is actually due to alkali-silica reaction. Sources of such supplementary information include: (1) petrographic examination of the aggregate (Practice C 295) to determine if known reactive constituents are present; (2) examination of the specimens after tests (Practice C 856) to identify the products of alkali reaction; and (3) where available, field service records can be used in the assessment of performance.

3.4 When it has been concluded from the results of tests performed using this test method and supplementary information that a given aggregate should be considered potentially deleteriously reactive, the use of mitigative measures such as low-alkali portland cement, mineral admixtures, or ground granulated blast-furnace slag should be evaluated (see last sentence of 3.1).

4. Apparatus

4.1 The apparatus shall conform to Specification C 490, except as follows:

4.2 *Sieves*—Square hole, woven-wire cloth sieves, shall conform to Specification E 11.

4.3 *Mixer, Paddle, and Mixing Bowl*—Mixer, paddle, and mixing bowl shall conform to the requirements of Practice C 305, 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.).

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[1] This test method is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.26 on Chemical Reactions.

Current edition approved March 15, 1994. Published May 1994. Formerly published as Proposal P 214.

[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 14.02.

[5] *Annual Book of ASTM Standards*, Vol 11.01.

[6] Available from National Standards of Canada, Canadian Standards Association, 178 Rexdale Blvd., Toronto, Ont. Canada M9W 1R3.

[7] The boldface numbers in parentheses refer to a list of references at the end of the text.

4.4 *Tamper and Trowel*—The tamper and trowel shall conform to Test Method C 109.

4.5 *Containers*—The containers must be of such a nature that the bars can be totally immersed in either the water or 1N NaOH solution. The containers must be made of material that can withstand prolonged exposure to 80°C (176°F) and must be inert to a 1N NaOH solution (see Note 1). The containers must be so constructed that when used for storing specimens, the loss or gain of moisture is prevented by tight-fitting covers, by sealing, or both (see Note 2). The bars in the solution must be placed and supported so that the solution has access to the whole of the bar; therefore, it should be ensured that the specimens do not touch the sides of the container or each other. The specimens, if stood upright in the solution, shall not be supported by the metal gage stud.

NOTE 1—The NaOH solution will corrode glass or metal containers.

NOTE 2—Some microwave-proof food storage containers made of polypropylene or high-density polythylene have been found to be acceptable.

4.6 *Oven, or Water Bath*—A convection oven or water bath with temperature control maintaining 80 ± 2.0°C (176 ± 3.6°F).

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 0.99N and 1.01N.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

5.3 *Sodium Hydroxide Solution*—Each litre of solution shall contain 40.0 g of NaOH dissolved in 900 mL of 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 ± 0.5 volumes of solution to 1 volume of mortar bars. The volume of a mortar bar may be taken as 184 mL.

5.3.1 **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 not less than 20°C (68°F) and not more than 27.5°C (81.5°F). The temperature of the mixing water, and of the moist closet or moist room, shall not vary from 23°C (73.4°F) by more than 1.7°C (3°F).

6.2 Maintain the relative humidity of the molding room at not less than 50 %. The moist closet or room shall conform to Specification C 511.

6.3 Maintain the storage oven or water bath in which the specimens are stored in the containers at a temperature of ± 2.0°C (176 ± 3.6°F).

7. Sampling and Preparation of Test Specimens

7.1 *Selection of Aggregate*—Process materials propos for use as fine aggregate in concrete as described in 7.2 wi a minimum of crushing. Process materials proposed for u as coarse aggregate in concrete by crushing to produce nearly as practical a graded product from which a sample c be obtained. Grade the sample as prescribed in 7.2. T sample shall represent the composition of the coarse aggr gate as proposed for use.

7.1.1 When a given quarried material is proposed for u both as coarse and as fine aggregate, test it only by selecti of an appropriate sample crushed to the fine aggregates siz unless there is reason to expect that the coarser size fractio have a different composition that the finer sizes and th these differences might significantly affect expansion due reaction with the alkalies in cement. In this case test t coarser size fractions in a manner similar to that employed testing the fine aggregate sizes.

7.2 *Preparation of Aggregate*—Grade all aggregates which this test method is applied in accordance with t requirements prescribed in Table 1. Crush aggregates which sufficient quantities of the sizes specified in Table 1 not exist until the required material has been produced. the case of aggregates containing insufficient amounts of o or more of the larger sizes listed in Table 1, and if no lar material is available for crushing, the first size in whi sufficient material is available shall contain the cumulati percentage of material down to that size as determined fr the grading specified in Table 1. When such procedures required, make a special note thereof in the test report. Af the aggregate has been separated into the various sieve siz wash each size with a water spray over the sieve to remc adhering dust and fine particles from the aggregate. Dry portions retained on the various sieves and, unless us immediately, store each such portion individually in a cle container provided with a tight-fitting cover.

7.3 *Selection and Preparation of Cement:*

7.3.1 *Reference Cement*—Use the portland ceme meeting the requirements of Specification C 150. In ad tion, the autoclave expansion in Test Method C 151 shall less than 0.20 %.

NOTE 5—The alkali content of the cement has been found to h negligible (3) or minor (5) effects on expansion in this test.

7.3.2 *Preparation of Cement*—Pass cement for use in t test through an 850-μm (No. 20) sieve to remove lu before use.

7.4 *Preparation of Test Specimens:*

7.4.1 *Number of Specimens*—Make at least three

TABLE 1 Grading Requirements

Sieve Size		Mass.
Passing	Retained on	
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

specimens for each cement-aggregate combination.

7.4.2 *Preparation of Molds*—Prepare the specimen molds in accordance with the requirements of Specification C 490 except, the interior surfaces of the mold shall be covered with a release agent (see Note 6). A release agent will be 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.

NOTE 6—TFE-fluorocarbon (Teflon[8]) tape complies with the requirements for a mold release agent.

7.4.3 *Proportioning of Mortar*—Proportion the dry materials for the test mortar using 1 part of cement to 2.25 parts of graded aggregate by mass. The quantities of dry materials to be mixed at one time in the batch of mortar for making three specimens shall be 440 g of cement and 990 g of aggregate made up by recombining the portions retained on the various sieves (see 7.2) in the grading prescribed in Table 1. Use a water to cement ratio equal to 0.47 by mass (see Note 7).

NOTE 7—Ruggedness tests indicated that mortar bar expansions were less variable at a fixed water to cement ratio than when gaged to a constant flow (3).

7.4.4 *Mixing of Mortar*—Mix the mortar in accordance with the requirements of Practice C 305.

7.4.5 *Molding of Test Specimens*—Mold test specimens within a total elapsed time of not more than 2 min and 15 s after completion of the original mixing of the mortar batch. Fill the molds with two approximately equal layers, each layer being compacted with the tamper. Work the mortar into the corners, around the gage studs, and along the surfaces of the mold with the 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 *Initial Storage and Reading*—Place each mold in the moist cabinet or room immediately after molds have been filled. The specimens shall remain 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 make an initial comparatory reading. Make and record the initial and all subsequent readings to the nearest 40.002 mm. Place the specimens made with each aggregate sample in a storage container with sufficient tap water to totally immerse them. Seal and place the containers in an oven or water bath at 80 ± 2.0°C (176 ± 3.6°F) for a period of 24 h.

8.2 *Zero Readings*—Remove the containers from the oven or water bath one at a time. Remove other containers only after the bars in the first container have been measured and returned to the oven or water bath. Remove the bars one at a time from the water and dry their surface with a towel paying particular attention to the two metal gage studs. Take the zero reading (see Note 8) of each bar immediately after drying, and read as soon as the bar is in position. Complete the process of drying and reading within 15 ± 5 s of removing the specimen from the water. After readings, leave the specimen on a towel until comparatory readings have been taken on the remainder of the bars. Place all specimens made with each aggregate sample in a container with sufficient 1N NaOH, at 80 ± 1.0°C (176 ± 3.6°F) for the samples to be totally immersed. Seal the container and return it to the oven or water bath.

NOTE 8—The reference bar should be read prior to each set of specimens since the heat from the mortar bars may cause the length of the comparator to change.

8.3 *Subsequent Storage and Measurement*—Make subsequent comparator readings of the specimens periodically, with at least three intermediate readings, for 14 days after the zero reading, at approximately the same time each day. If readings are continued beyond the 14-day period, take at least one reading per week. The procedure is identical to that described in 8.2 except that the specimens are returned to their own container after measurement.

9. Calculation

9.1 Calculate the difference between the zero comparatory reading of the specimen and the reading at each period to the nearest 0.001 % 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 % as the expansion for the combination for a given period.

10. Report

10.1 Report the following information:

10.1.1 Type and source of aggregate,

10.1.2 Type and source of portland cement,

10.1.3 Autoclave expansion and alkali content of cement as percent potassium oxide (K_2O), sodium oxide (Na_2O), and calculated sodium oxide (Na_2O) equivalent,

10.1.4 Average length change in percent at each reading of the specimens,

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

10.1.6 Any significant features revealed by examination of the specimens during and after test,

10.1.7 Amount of mixing water expressed as mass percent of cement,

10.1.8 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*—The between-laboratory precision has been estimated from an interlaboratory study involving six laboratories, each testing three aggregates with three cements (5). For values of expansion greater than 0.015 %, the between-laboratory precision has been found to be 9.55 % (1s %). Therefore, the results of two properly conducted tests in two different laboratories should differ by no more than 27.0 % of the mean expansion.

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.

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[8] Teflon is a registered trademark.

APPENDIX

(Nonmandatory Information)

X1. INTERPRETATION OF TEST RESULTS

X1.1 There is good agreement in the published literature **(1, 2, 6, 7, 8, 9)** for the following expansion limits:

X1.1.1 Expansions of less than 0.10 % at 16 days after casting are indicative of innocuous behavior in most cases (see Note X1.1).

X1.1.2 Expansions of more than 0.20 % at 16 days after casting are indicative of potentially deleterious expansion. (See 3.3.)

X1.1.3 Expansions between 0.10 and 0.20 % at 16 days after casting include both aggregates that are known to be innocuous and deleterious in field performance. Fo aggregates, it is particularly important to develop s mental information as described in 3.3. In such a situa may also be useful to take comparator readings until 2 **(7, 9)**.

NOTE X1.1—Some granitic gneisses and metabasalts ha found to be deleteriously expansive in field performance even their expansion in this test was less than 0.10 % at 16 days after **(9)**. With such aggregate, it is recommended that prior field perf be investigated. In the absence of field performance data, m measures should be taken as discussed in 3.4.

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Appendix M

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 TRANSPORTATION OFFICIALS
444 North Capitol Street, N.W., Suite 249
Washington, D.C. 20001

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

AASHTO Designation TP14[1]

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 this 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 Cube 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

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[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 supplementary 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 T162, 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°F) 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 ± 1.7°C (176 ± 3°F) 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 of 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 of solution to 1 volume of mortar bars.

> Note 2 - The volume of a mortar bar may be taken as 184 mL (11.25 in^3).

> 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 alkalies 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.

Table 1
GRADING REQUIREMENTS

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 Preparation of Test Specimens:

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 surfac 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 b mass. Mix 440 g of cement and 990 g of dry aggregate (made up by recombining the portions retained on t various sieves in the grading prescribed in Table 1) at one time to prepare a batch of mortar sufficient for maki 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 requiremer 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 mixi of the mortar batch. Fill the molds with two approximately equal layers, each layer being compacted with t tamper. Work the mortar into the corners, around the gage studs, and along the surfaces of the mold with t tamper until a homogeneous specimen is obtained. After the top layer has been compacted, cut off the mortar flu 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 specime in the molds for 24 ± 2 h. Remove the specimens from the molds and, while they are being protected from lo of moisture, properly identify and determine the initial length of each specimen using the length comparat 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 immer them. Seal the containers and place them in an oven maintained at 80.0 ± 1.7°C (176 ± 3°F) for a period of h.

8.3 Measure the comparator bar prior to measuring each set of specimens since the heat from the mortar bars m cause the length of the comparator to change. If the length of the comparator bar has changed, wait until t comparator has returned to room temperature before remeasuring the comparator bar. Remove the containers fro the oven one at a time. Remove other containers only after the specimens in the first container have been measur and returned to the oven. Remove the specimens one at a time from the water and dry their surface with a tow paying particular attention to the two metal gage measuring studs. Record the zero measurements of each specim immediately after drying, and as soon as the specimen is in position. Complete the process of drying and measuri within 15 ± 5 s of removing the specimen from the water. After measurement, leave the specimen on a tow until the remainder of the bars have been measured. Place each set of specimens in separate containers with t 1 normal NaOH solution, at 80.0 ± 1.7°C (176 ± 3°F) to totally emerse the samples. Seal the containers and retu them to the oven.

8.4 Undertake subsequent measurement of the specimens periodically, with at least three intermediate reading for 14 days after the zero reading, at approximately the same time each day. The measuring procedure is identic to that described in Section 8.3 except that the specimens are returned to their own container after measuremer

9. Calculation

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 that 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.

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Appendix N

CEMENT and CONCRETE RESEARCH. Vol. 16, pp. 181-189, 1986. Printed in the USA.
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AN ACCELERATED METHOD FOR TESTING THE POTENTIAL ALKALI REACTIVITY OF SILICEOUS AGGREGATES

R.E. Oberholster and G. Davies
National Building Research Institute
Council for Scientific and Industrial Research
Pretoria, South Africa

(Communicated by J.P. Skalny)
(Received Dec. 16, 1985)

ABSTRACT

The National Building Research Institute has developed a quick, reliable test which gives an indication of the relative alkali reactivity of opaline and of quartz-bearing aggregates. This paper describes the test method and discusses factors which can affect the precision of the results.

Introduction

Several methods are employed for the determination of the potential alkali reactivity of siliceous aggregates such as the 'gel pat test' of Jones and Tarleton (1), the 'quick chemical test' ASTM C 289 (2), the 'mortar prism test' ASTM C 227 (3), the 'concrete prism test', the 'rock cylinder test' ASTM C 586 (4) and a petrographic examination.

Of the methods used, the quick chemical test is the quickest and takes only one day to perform. Unfortunately, with this method, some of the quartz-bearing rocks such as greywacke, argillite, granite and quartzite which have a service record of being deleteriously reactive, often give results that classify them as non-deleterious. Petrographic examination for determining the presence of reactive constituents and undulatory extinction angles, although relatively quick, is very difficult to apply in the case of fine-grained rocks such as hornfels, and should in general only be used as a preliminary screening test. The gel pat test is fairly quick in the case of opaline aggregates, but slow where quartz-bearing aggregates are involved, unless the pats are kept at a temperature of 80°C; this test should also only be used for preliminary screening. The ASTM C 227 mortar prism test is probably the most widely used test for determining the potential alkali reactivity of cement-aggregate combinations. It has the following disadvantages:

(a) to obtain conclusive results a period of between three and six months is required in the case of opaline aggregates, and between six and twelve months in the case of quartz-bearing aggregates;

(b) the National Building Research Institute (NBRI) has found that the test criteria have to be adjusted for quartz-bearing aggregates;

(c) different cement-aggregate combinations have to be tested in the case of opaline aggregates to make provision for a pessimum effect.

During a test programme which involved five laboratories in different countries, only one laboratory (5) recorded deleterious expansion with a greywacke argillite aggregate in combination with high-alkali cements using the ASTM C 227 test, despite the service record of this aggregate which has proved deleteriously expansive in combination with high-alkali cements. This casts doubts on either the control which laboratories have over this test procedure or the applicability of the method.

The concrete prism test has the disadvantages mentioned in (a), (b) and (c) and, in addition, concrete prisms require much more space than the mortar prisms. The major advantage of this test is that the materials can be tested in the proportions used in practice.

It was found that the rock cylinder test takes at least eight weeks to give conclusive results and that significant expansions are only obtained if the cylinders are cored perpendicular to veins or the rock layers (6).

An accelerated mortar prism test, which involves storage in a saturated NaCl solution at 50°C, was proposed by Chatterji (7). Tests at the NBRI, however, indicated that this method did not significantly reduce the time required to obtain results with quartz-bearing aggregates.

The problems outlined, with the different tests, created a need for a quick, reliable test for opaline as well as for quartz-bearing aggregates, and one which could give an indication of the relative alkali reactivity of the aggregates.

The NBRI quick mortar prism test

Van Aardt and Visser (8) proposed a method which involved storage of mortar prisms prepared in accordance with ASTM C 227 in 1N NaOH at 80°C. Their results showed that expansions of more than 0.10 per cent within 10 days were obtained with potentially deleteriously reactive quartz-bearing aggregates.

However, no criteria were established to distinguish deleterious aggregates from innocuous aggregates.

Oberholster (9) investigated 28 quartz-bearing aggregates, 2 non-reactive reference aggregates and different percentages of Beltane opal. Using the method of van Aardt and Visser, results were related to those previously obtained with either the mortar prism or the concrete prism method, and with expansions in concrete beams and cubes exposed to natural environmental conditions. Provisional criteria for the method were determined.

The test has now been used for three years on a wide variety of aggregates. This paper describes the test method and presents data on the factors which can affect the precision of the results.

The method

The mortar prisms are prepared in accordance with ASTM C 227-81. The prisms are demoulded after 24 hours, then ~~immersed in water~~ in a closed container which is placed in an oven and kept at a constant temperature of 80°C. After 24 hours in the oven, the prisms are removed to a room with a temperature of 23°C, and before significant cooling takes place, their length is measured using a vertical comparator (dial-type strain gauge) accurate to 2 µm. This reading is used as the zero reading. After the initial reading, the prisms are immersed in a 1N NaOH solution kept at 80°C. This solution is stored in a tightly covered plastic container, large enough for the prisms to be totally immersed. Plastic is used because the caustic solution corrodes glass and metal. The prisms are then measured each working day over 14 days at a room temperature of 23°C, and their linear expansion calculated. The average expansion of the triplicate prisms for each day is then calculated; if none of the values differs from the mean by more than 15% the repeatability is considered satisfactory (3). The average expansion after 12 days is then taken as the reference value for assessing potential alkali reactivity.

Evaluation of the method

Before a method such as the NBRI accelerated test can be accepted, it is necessary to establish that the reaction has merely been accelerated and that no unrelated reaction, which could lead to erroneous results is occurring.

Although not unequivocal proof, the shape of the 'expansion vs time' curve should give some indication of whether the reaction has been merely accelerated or not. Linear expansions against time have respectively been plotted for a siliceous hornfels aggregate (Fig 1) and a non-reactive sand sample in which 30 per cent by mass Beltane opal has been substituted (Fig 2). In both figures, the results from both the NBRI accelerated test and the ASTM C 227 test data have been shown. All the curves (except the accelerated test with opal) show the three features described by Grattan-Bellew (10) as the induction phase, the main expansion phase and the late expansion phase. Opal samples, when subjected to the accelerated test, often lack the induction phase. These results are encouraging and would appear to indicate the efficacy of the NBRI accelerated test. However, more work is required for conclusive proof.

The linear expansions at 12 days (for the NBRI accelerated test) are plotted against those taken at 365 days (for the ASTM C 227 test) for the same set of quartz-bearing aggregates (Fig 3). A rather poor linear correlation (the correlation coefficient is 0.6696) is obtained. The reason for the poor fit is possibly the poor precision of the ASTM C 227 test.

The lower limit of expansion for deleterious aggregate subjected to the ASTM C 227 mortar prism test is taken by the NBRI as 0.05 per cent linear expansion after 365 days. This value is based on the correlation of results

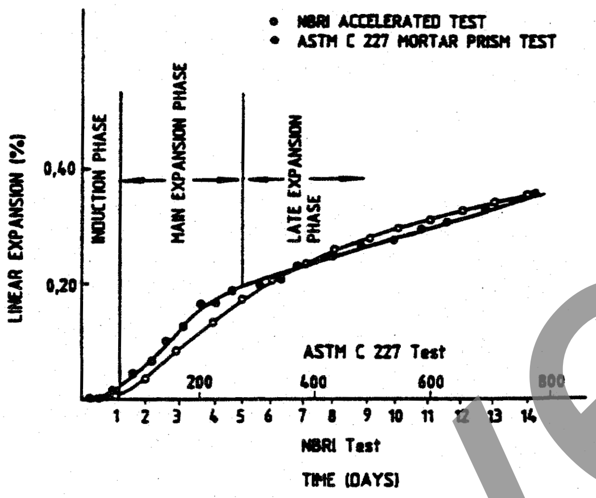

FIG 1

Expansions measured for a hornfels sample (ASTM test was done using a cement with an available alkali content of 1.08 per cent Na_2O equivalent)

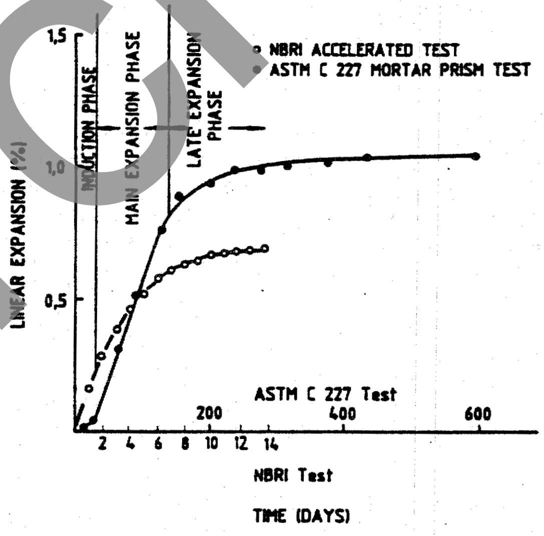

FIG 2

Expansion measured for a mortar prism in which Beltane opal was substituted for a non-alkali-reactive sand (ASTM test was done using a cement with an available alkali content of 1.08 per cent Na_2O equivalent)

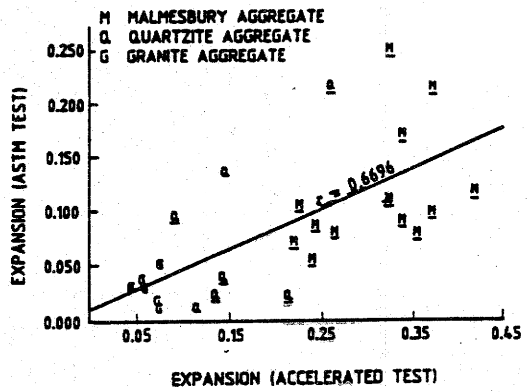

FIG 3

A plot of results from quartz-bearing aggregates subjected to the NBRI accelerated test and the ASTM C 227 test (using a cement with an available alkali content of 1.28 (11) and a total alkali content of 1.30 per cent Na_2O equivalent). The linear expansions were measured after 12 and 365 days respectively. Samples underlined are aggregates with a proven record of deleterious expansion in practice.

from long-term tests on concrete cubes exposed to natural conditions, and from the ASTM C 227 mortar prism test. Using this value and the regression equation ($y = 0.37x + 0.008$) from Fig 3, the lower limit of expansion for deleterious aggregate is calculated as 0.11 per cent at 12 days for the accelerated test.

The correlation between the results of the NBRI and ASTM tests is more problematic in the case of the opaline samples, because the pessimum effect is present when the ASTM test is undertaken, but apparently absent when the NBRI test is performed (Fig 4). Also, the expansion is considerably lower in the NBRI test compared with samples with the same opal content giving the pessimum value for the ASTM test.

The effect of different variables on expansion

Van Aardt and Visser (8) investigated the effect of temperature and alkali concentration on the expansion recorded for mortar prisms made with hornfels aggregate. At 40°C they found little difference between the rates of expansion of prisms immersed in 2N and 4N NaOH solutions, and those made with the same aggregate but in which alkali hydroxide was added to the cement and exposed under ASTM C 227-81 conditions. At 80°C, however, mortar prisms stored in alkali solution showed significantly increased rates of expansion.

Different concentrations were tried, namely, 0.25N, 0.5N, 0.75N, 1.0N and 2.0N solutions. The maximum expansion measured after 12 days was that of the

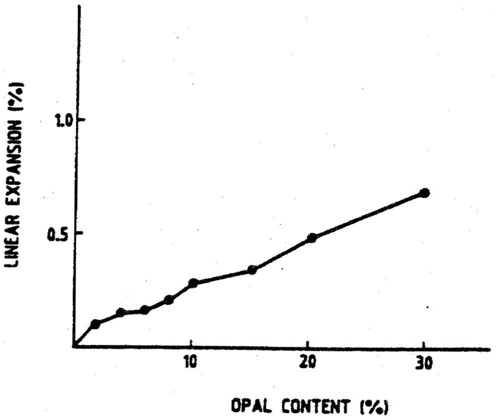

FIG 4

The variation in linear expansion with variation in opal content, for mortar prisms subjected to the NBRI accelerated test. Note the linear relationship.

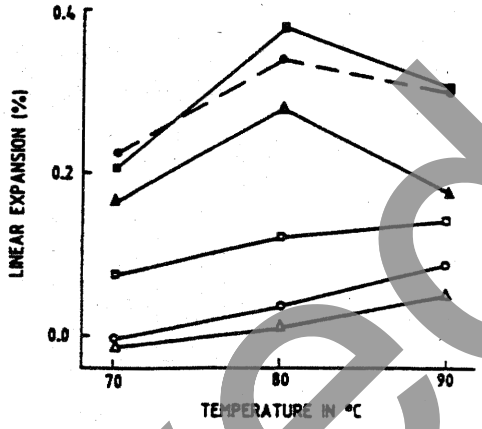

FIG 5

Linear expansion after 12 days for a suite of aggregates showing the effect of the variation in temperature on expansion during the NBRI accelerated test.

prisms in the 1N NaOH solution, while lower expansions were measured for prisms in the other solutions. Prisms in the 2N and 4N solutions initially expanded more quickly than the others but the expansion levelled off at a value below that obtained for prisms in the 1N solution.

Van Aardt and Visser (8) also found that prisms in KOH solutions of the same normality as NaOH, expanded less.

Additional experiments were undertaken to check the effects of both temperature and the various possible concentrations of the alkali solution on the precision of the test.

Six aggregates with alkali reactivities over the whole range of aggregates found in South Africa, were used in these experiments. Because no opaline aggregates have thus far been identified in South Africa only quartz- or silicate-bearing aggregates were used.

The effects of the different variables will be discussed in turn.

Temperature. The prisms made with the aggregates were immersed in 1N NaOH solution and stored in ovens at 70°C, 80°C and 90°C. The expansions at 12 days are compared in Fig 5.

As can be seen for samples which have expansion below 0.150 per cent after 12 days, the value increases progressively with temperature. For samples with linear expansions above 0.150 per cent, the expansion is highest at 80°C, followed by that at 90°C and then by that at 70°C. The variation between the results obtained at the different temperatures for the same aggregate can be

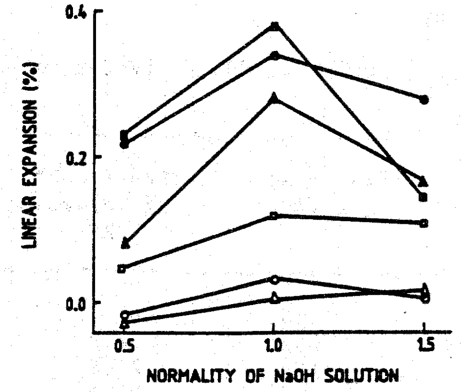

FIG 6

Linear expansion after 12 days for the same suite of aggregates used in Fig 5 showing the effect of the variation in NaOH concentration on expansion during the NBRI accelerated text.

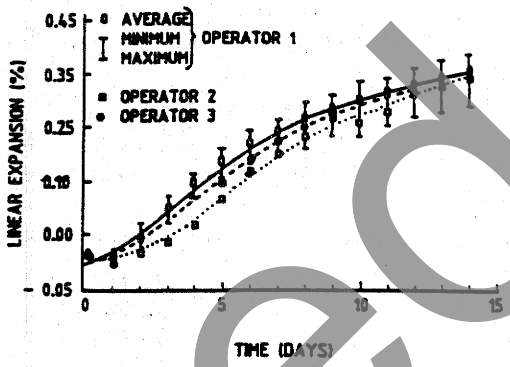

FIG 7

A plot of the linear expansion with time for a reactive hornfels aggregate used to test the precision of the NBRI accelerated test.

0.032 percentage points which, in relative terms, is well in excess of a 100 per cent variation in the case of less reactive aggregates having very low expansions. For the more reactive aggregates, the maximum variation between the values is 0.180 percentage points, or 47 per cent in relative terms.

NaOH concentration. The variation of the linear expansion with variation in concentration of the alkali solution, at 80°C, is shown in Fig 6. In this case the 1N solution gave the largest expansion for all but one aggregate. The maximum variation between expansions for a particular aggregate is 0.232 percentage points, or approximately 61 per cent in relative terms.

The results indicate that variations in both temperature and alkali concentration can affect the precision of the test significantly.

Precision. The precision of the method was tested using a reactive hornfels aggregate and only one operator. The average, the minimum and the maximum values as well as the coefficient of variation for seven tests on this aggregate over a 14-day period, are presented in Table 1. The coefficient of variation for this particular sample stabilises after the fifth day and is 9.7 per cent on the 12th day, the day the NBRI has decided to take as the reference day of the test.

The effect of different operators on the precision of the method was also tested in the NBRI laboratory. The results are presented in Fig 7 and show that the expansion obtained by operators 2 and 3 fall well within the minimum and maximum values obtained by the operator 1, after the seventh day.

Clearly, insufficient data are available for a rigorous statistical evaluation

TABLE 1

Statistical Data for Precision of Accelerated Test Method (7 Determinations)

Day	Av linear expansion %	Min linear expansion %	Max linear expansion %	Coefficient of variation %
1	0.009	0.002	0.013	41.6
2	0.035	0.018	0.052	32.3
3	0.084	0.066	0.109	21.0
4	0.134	0.113	0.156	12.5
5	0.177	0.155	0.200	10.0
6	0.208	0.181	0.226	7.8
7	0.235	0.197	0.252	8.2
8	0.258	0.211	0.280	9.4
9	0.278	0.228	0.301	9.7
10	0.296	0.245	0.321	8.7
11	0.313	0.252	0.341	9.8
12	0.327	0.267	0.361	9.7
13	0.342	0.278	0.379	10.0
14	0.354	0.289	0.392	10.0

of the method, but the results are encouraging and indicate that if care is taken, the test can give reasonably precise results for quartz-bearing aggregates.

Conclusions

Experience at the NBRI over a period of three years has shown the accelerated test proposed by van Aardt and Visser (8) to be a quick reliable test for the determination of the potential alkali reactivity of aggregates, as long as reasonable care is taken in the execution of the test.

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Appendix O

SHRP-C-343, "Eliminating or Minimizing Alkali-Silica Reactivity"

(See book attached)

Appendix P

CEMENT and CONCRETE RESEARCH. Vol. 17, pp. 97-107, 1987. Printed in the USA.
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USE OF THE NBRI ACCELERATED TEST TO EVALUATE THE EFFECTIVENESS OF MINERAL ADMIXTURES IN PREVENTING THE ALKALI-SILICA REACTION

G Davies and R E Oberholster
National Building Research Institute
Council for Scientific and Industrial Research
Pretoria, South Africa

(Communicated by J.P. Skalny)
(Received Oct. 31, 1986)

ABSTRACT. The NBRI accelerated test for determining the potential alkali reactivity of aggregates can also be used to assess the ability of mineral admixtures to prevent deleterious expansion in concrete, which is caused by the alkali-silica reaction of quartz-bearing aggregates. Results from the accelerated test, the ASTM C227 mortar prism test, and tests undertaken on concrete beams and cubes exposed outdoors, are compared and discussed.

Introduction

There is still uncertainty about the role that different admixtures play in the prevention of the deleterious expansion that results from the alkali-silica reaction. Mineral admixtures with a proven record in this regard are fly ash (FA), milled granulated blast-furnace slag (MGBS) and condensed silica fume (SF). Vivian (1) suggests that the pozzolan particles react very rapidly with alkalis in cement because of their reactive nature and fineness, thus leaving little unreacted alkali for later reaction with aggregate. Other workers (2,3) believe that the pozzolans react with $Ca(OH)_2$, thereby preventing the formation of the deleterious reaction product. Diamond (4) has demonstrated that the addition of pozzolan reduces the pH of the pore fluids in cement pastes. However, some mineral admixtures have been shown to be more effective than others, and in certain cases the mineral admixture has had little effect on the pH of the pore fluid and appeared to act as an inert diluent (5).

Regardless of what the exact role of mineral admixtures is in preventing deleterious alkali-silica reaction, the results of Diamond (5) cast doubt on the effectiveness of certain mineral admixtures.

ASTM C441-81 test (6) evaluates the effectiveness of mineral admixtures using mortar prisms made with high-alkali cement and Pyrex glass. Up to 25 per cent of the cement is replaced with a mineral admixture on a volume/volume basis.

Experience at the National Building Research Institute (NBRI) indicates that the ASTM C441-81 test is unreliable for such evaluation because of the poor reproducibility of results (7). The exact reason for this is not clear but poor reproducibility with this test has also been reported by other laboratories (8).

It is also possible to evaluate the effectiveness of mineral admixtures using the ASTM C227-81 test (9), which has the advantage that a particular mineral admixture can be tested with specific cements and aggregates. However, this test is rarely used because of the time it takes to obtain definitive results with quartz-bearing aggregates.

Work undertaken at the NBRI showed that the accelerated test described by Oberholster and Davies (10) can be used to determine, within 14 days, the effectiveness of any mineral admixture in preventing deleterious expansion due to the alkali-silica reaction in quartz-bearing aggregates. This test has the advantage that it can also be applied to specific aggregate/mineral admixture combinations.

Materials and methods

The following materials were used in the study:

(a) fly ash (FA) from ESCOM Grootvlei power station;
(b) condensed silica fume (SF) from Silicon Smelters, Pietersburg;
(c) milled granulated blast-furnace slag (MGBS) from ISCOR, Pretoria;
(d) calcined shale (CS);
(e) reactive Malmesbury greywacke/hornfels aggregate;
(f) low-alkali cement;
(g) calcium carbonate.

The chemical and physical properties of the mineral admixtures except the calcium carbonate, are presented in Table 1. The calcium carbonate was precipitated, chemically pure material with a relative density of 2.99.

The relevant details of the aggregates and cements used in the different experiments are presented in Table 2.

Results obtained with the accelerated test described by Oberholster and Davies (10) (see this reference for method) were compared with the results from ASTM C227 tests (11) and from beams (12,13) and cubes (13) exposed under natural conditions (field tests). In the case of the ASTM C227 test, the results which are used here are for prisms with an aggregate:cement ratio of 1.5:1 (rather than the 2.25:1 stipulated by ASTM), but apart from this, the ASTM C227 procedure was followed exactly. The effect of the lower aggregate/cement ratio was to increase the active alkali content of the mortar, thereby testing more rigorously the ability of the mineral admixture to reduce expansion. Further details of the ASTM C227 and field test results can be obtained from the relevant references. To simplify the comparisons, the materials used for the accelerated test were, where possible, the same or similar to those used for the ASTM C227 tests and the field experiments. Replacement of the cement by mineral admixture was undertaken on a volume/volume basis for all experiments except for the field experiments with concrete beams, where the replacement was done on a mass/mass basis. The volume percentages for the beams were calculated and these are used in the discussion to facilitate comparison with the other data.

TABLE 1
Chemical and physical properties of mineral admixtures used

	Mineral admixtures							
	SF^{1+}	SF^{2,3}	FA^{1}	FA^{2}	FA^{3}	MGBS^{1,2,3}	CS^{1,3}	CS^{2}
SiO_2	94.6	94.06	41.7	45.62	45.1	33.38	64.6	64.10
Al_2O_3	0.20	0.72	36.7	40.79	36.2	14.43	24.41	20.35
Fe_2O_3	0.05	0.08	2.63	2.99	2.58	0.31	5.23	9.43
MgO	0.86	0.47	1.97	1.63	1.43	15.40	0.13	1.23
CaO	0.14	0.13	10.67	2.19	8.42	31.14	0.50	0.36
Na_2O	0.20	0.23	1.03	2.92	0.67	1.90	0.11	0.56
K_2O	1.13	0.38	0.99	1.30	0.97	1.15	2.50	4.48
MnO	0.11	0.02	-	0.04	0.08	0.73	-	0.25
SO_3	-	0.42	1.13	0.81	0.87	0.16	0.03	0.08
P_2O_5	-	-	-	-	-	-	-	-
S	-	-	-	-	-	1.14	-	-
LOI	3.31	2.43	3.86	1.49	3.04	1.66	1.60	0.13
Total	100.60	98.94	100.68	99.78	99.36	101.40	99.11	100.97
Free CaO	-	-	1.09	-	-	-	-	-
Na_2O equivalent								
Total	0.94	0.48	1.68	3.78	1.31	2.66	1.76	3.51
Active*	-	0.14	-	0.49	0.66	0.21	0.64	0.81
Relative density	2.40	2.27	2.34	2.28	2.25	2.88	2.81	2.69
Specific surface (cm2/g)	-	37000	5700	4721	4557	3620	11000	6148
Pozzolanicity with $Ca(OH)_2$, MPa	10,74	9.81	10.9	7.59	6.94	5.08	5.24	4.50

* ASTM C311 : Section 18
+1 Used in the accelerated test
2 Used in the ASTM C227-81 test (11)
3 Used in the field experiments (12,13)

In an experiment to see what effect the dilution of cement has on the expansion measured during the accelerated test, the calcium carbonate was used as an inert diluent to replace cement. Oberholster and Westra (11) determined the effect of the dilution on expansion produced during the ASTM C227 test, by calculating the decrease in Na_2O equivalent of the binder as a whole for each substitution (assuming that the admixture provides no alkalis), and then blending a low-alkali cement with the high-alkali cement (used in the tests) in the proportions necessary to obtain the appropriate Na_2O equivalent.

Results

Before discussing the results it should be noted that the NBRI considers expansion in excess of 0.05 per cent to be deleterious in the case of the ASTM C227 test and the outdoor exposure experiments. For the accelerated test, Oberholster and Davies (10) considered an expansion in excess of 0.10 per cent at 12 days to be deleterious.

Graphs of expansion plotted against time for the accelerated, the ASTM C227,

TABLE 2
Details of the aggregate, cement and concrete samples used in the different experiments

Experiment	Aggregate		Cement			Concrete
	Sample Number	Type	Sample Number	Na_2O equivalent, % Total	Active*	kg of binder per m^3
Accelerated tests	A28	Malmesbury Metasediment	C49	0.27	0.17	-
ASTM C227 tests (11)	P5	Malmesbury Metasediment	H	0.97	0.79	-
Field experiments Beams (12)	A22	Malmesbury Metasediment	M	1.30	1.18	370
Field experiments Cubes (13)	P6	Malmesbury Metasediment	J	1.12	1.10	450

* ASTM C311 : Section 18

and the field tests, in which cement was replaced with various amounts of different admixtures, are presented in Figures 1 to 4. It is evident from the Figures that there is a reasonable correlation between the accelerated and ASTM C227 test results when using the different admixtures.

The results for the beams and cubes are limited in number, but they, too, correlate well with the laboratory tests once the dormant period (always evident in field experiments) is over.

Effectiveness of the different admixtures

The reduction in expansion effected by the different admixtures, as measured by the different test methods, is shown in Figures 5 to 7. All tests show that the silica fume is by far the most effective agent in reducing expansion due to the alkali-silica reaction, followed by the fly ash. The calcined shale and slag are shown to be ineffective at substitutions of less than 30 per cent.

There is a discrepancy between the accelerated test and the ASTM C227 test, in that the slag appears to be more effective than the calcined shale in the accelerated test while the opposite seems true in the ASTM C227 test. The discrepancy is most likely because different batches of calcined shale were used in the two tests (see Table 1), but the different effects that dilution of both the cement and active alkalis by the mineral admixtures themselves have on the results of the two tests also needs to be borne in mind.

As can be seen in Figure 5, substitution of cement by an inert diluent ($CaCO_3$ in this case) has little effect on the expansion measured in the accelerated test; in fact, it appears to increase expansion in the 0-20 per cent substitution range. In the ASTM C227 test, the dilution of the cement plays a significant role in decreasing expansion (see Figure 6). The reason for this discrepancy seems obvious since, in the case of the accelerated test, the alkali utilised by the reaction comes from an external source, the 1N sodium hydroxide solution in which the prisms are immersed, while in the case of the ASTM test, the alkali is derived mainly from the cement.

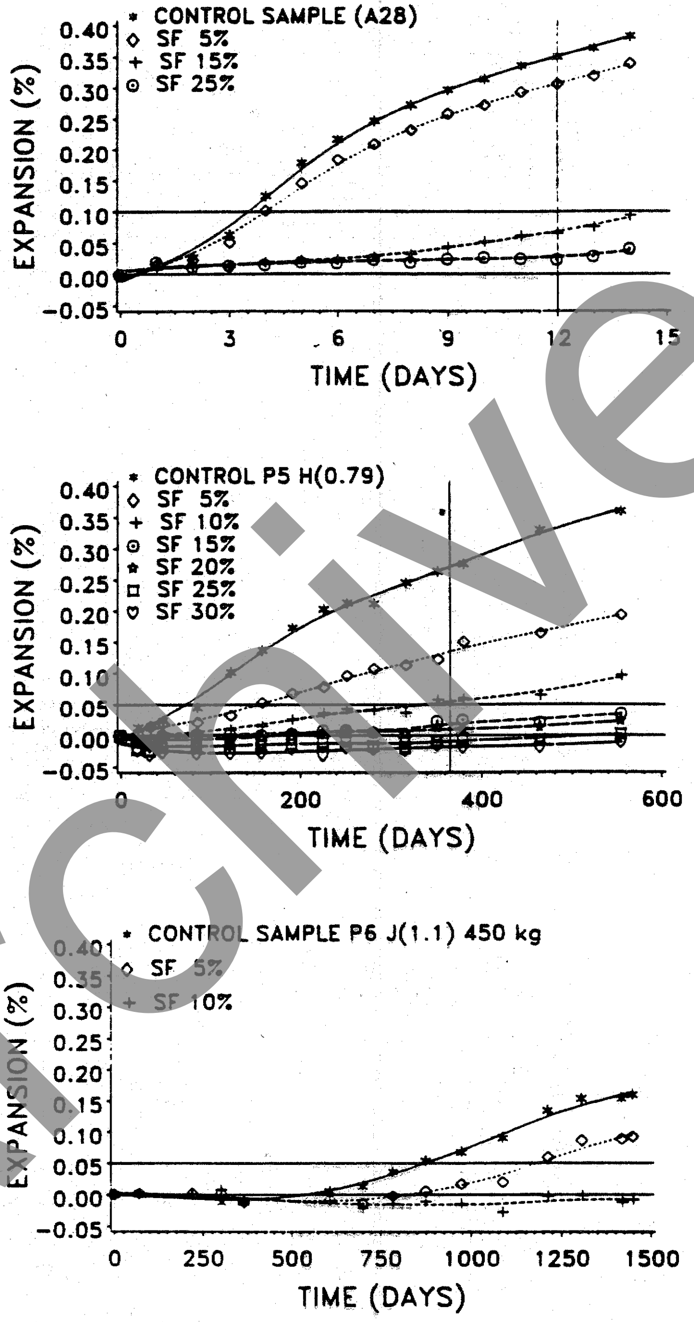

FIG 1
Expansion vs time graphs for three tests in which the cement was replaced by various proportions of silica fume. (a) The NBRI accelerated test, (b) the ASTM C227 mortar prism test and (c) field experiments using concrete cubes

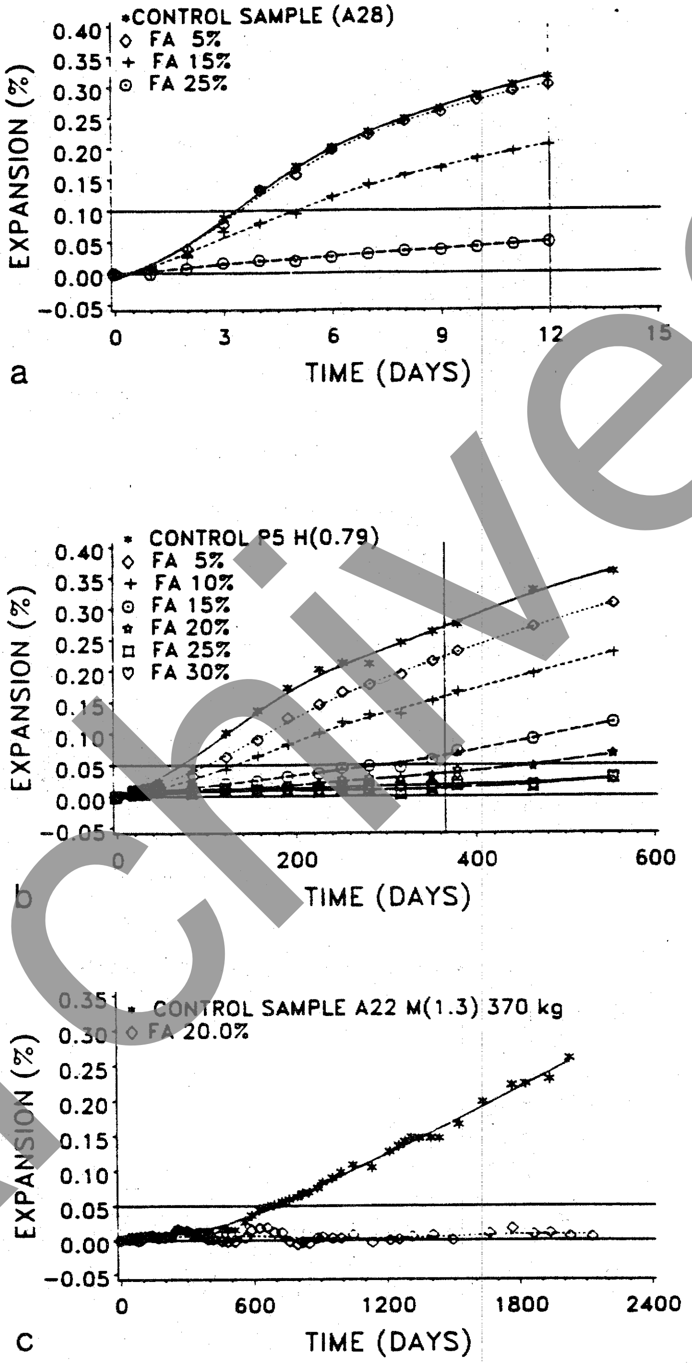

FIG 2
Expansion vs time graphs for three tests where the cement was replaced by various proportions of fly ash. (a) The NBRI accelerated test, (b) the ASTM C227 mortar prism test, and (c) field experiments using concrete beams

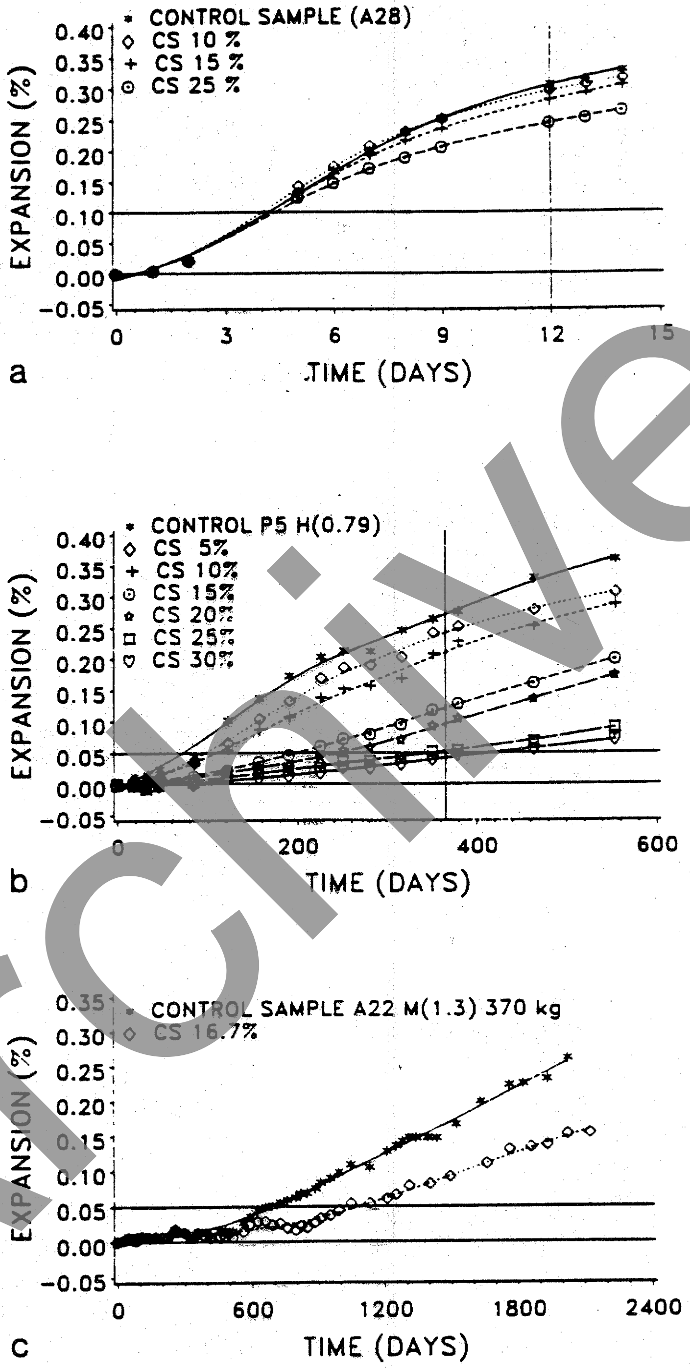

FIG 3
Expansion vs time graphs for three tests, where the cement was replaced by various proportions of calcined shale. (a) the NBRI accelerated test, (b) the ASTM C227 mortar prism test, and (c) field experiments using concrete beams

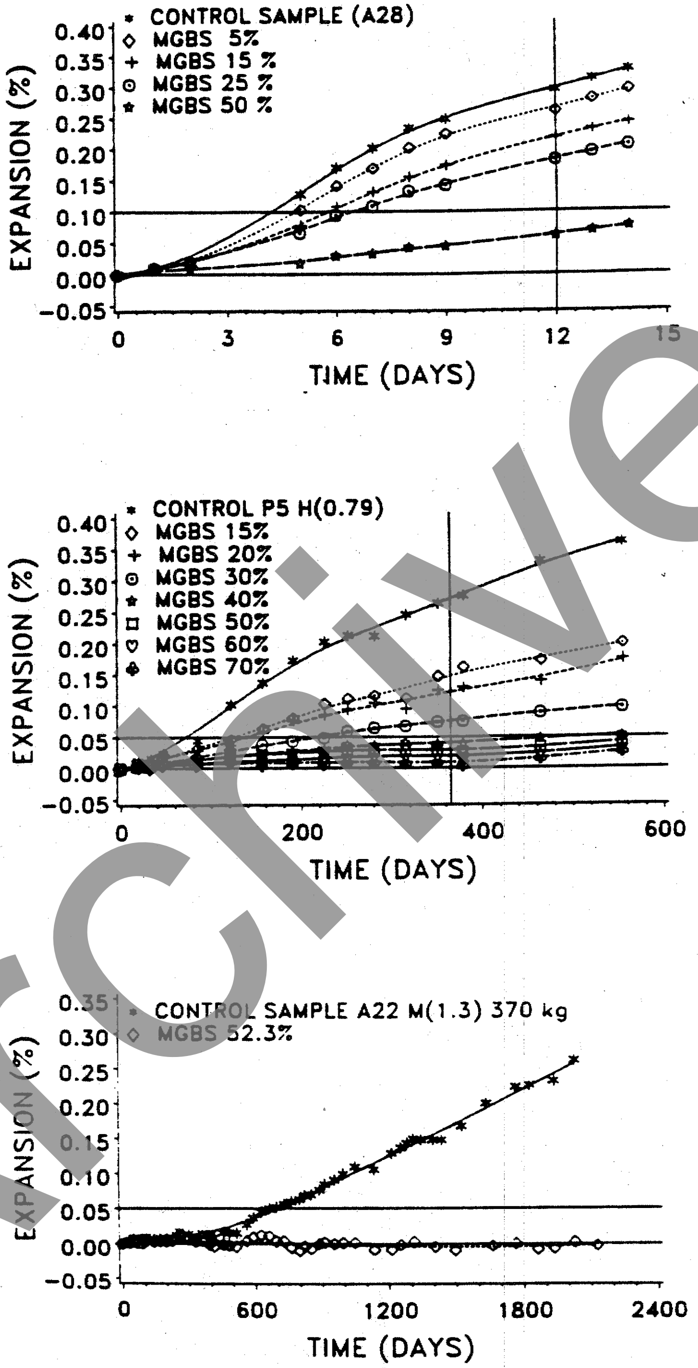

FIG 4
Expansion vs time graphs for three tests, where the cement was replaced by various proportions of slag (a) the NBRI accelerated test, (b) the ASTM C227 mortar prism test, and (c) field experiments using concrete beams

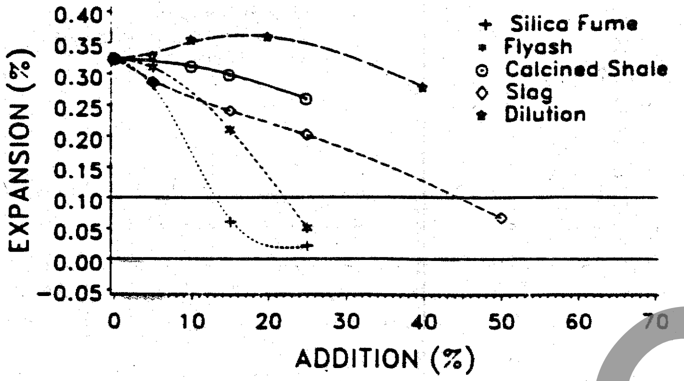

FIG 5
The effect on expansion (measured at 12 days) obtained with the NBRI accelerated test when different proportions of mineral admixtures are substituted for the cement. The dilution curve was obtained by substituting calcium carbonate for cement

Repeated tests at the NBRI have shown that the quartz-bearing, alkali-reactive aggregates of South Africa do not have a pessimum effect and, as a general rule, expansion increases with an increase in the active alkali content of the cement. Since the active alkali content of most of the mineral admixtures is less than that of the cement (0.79 per cent) - see Table 1 - the active alkali content usually decreases in the mortar as a whole when a mineral admixture is substituted for cement; this results in a decrease in the expansion due to the alkali-silica reaction.

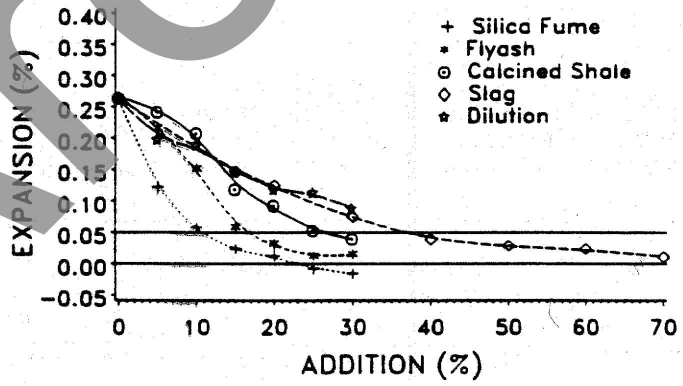

FIG 6
The effect on expansion measured at 352 days obtained with the ASTM C227 test, when different proportions of mineral admixtures are substituted for the cement (11). Note the difference between the dilution effect in this figure and that in Figure 5

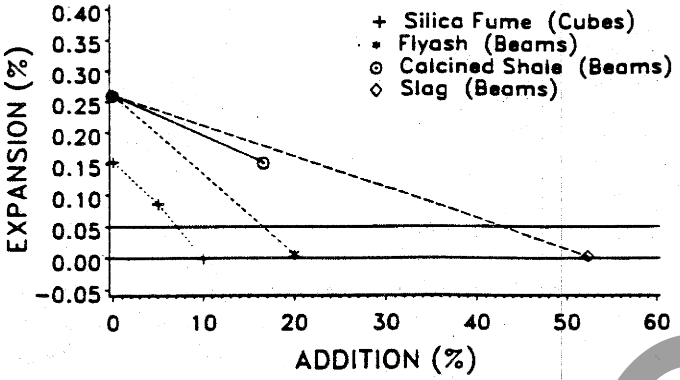

FIG 7
Expansion measured during field experiments using beam and cube samples in which cement was replaced by different mineral admixtures (12,13). Expansions used in this figure were measured at 2027 and 1308 days for the beams and cubes, respectively

The active alkalis in the mineral admixtures also cause different results in the two laboratory tests. Unpublished NBRI data indicate that active alkalis added to the mortar prism in the mix water do not affect the expansion measured in the accelerated test. In the ASTM test any active alkali introduced by the mineral admixture will affect the alkali content of the mortar as a whole, and therefore will influence expansion. The reduction in expansion in the ASTM test will therefore be an interplay between the ability of the mineral admixture to prevent the alkali-silica reaction and the amount of active alkali that is introduced to the system as a whole by the mineral admixture.

Irrespective of the discrepancy discussed above, it is still clear from the diagrams that the accelerated test can successfully be used to gauge the

TABLE 3
Estimated addition of different mineral admixtures required (volume/volume) to prevent deleterious expansion due to the alkali-silica reaction. Values are obtained from Figures 5, 6 and 7 (where necessary by extrapolation).

	Silica fume %	Fly Ash %	Slag %	Calcined shale %
Accelerated Test	13	22	45	50
ASTM C227-81 Test	11	16	37	26
Field Test	7	16	42	32

ability of different mineral admixtures to prevent deleterious expansion due to the alkali-silica reaction, provided the admixture does not contain excessive quantities of active alkalis.

In the accelerated test, a greater substitution of admixture is required to prevent deleterious expansion than in the ASTM C227 test, which has results very similar to those of the field experiments (see Table 3). The NBRI accelerated test therefore provides a 'worst case' result whereas the expansion measured during ASTM C227 and field tests will vary depending on the active alkali content of the binder (cement plus mineral admixture) as a whole.

Conclusions

The NBRI accelerated test method can successfully be used to screen different mineral admixtures for their effectiveness in preventing deleterious expansion due to the alkali-silica reaction, as long as the limitations of the test are borne in mind when evaluating the results. Because the accelerated test apparently represents a 'worst case', the amounts of mineral admixture required to prevent deleterious expansion in this test should represent the maximum required in practice, for a particular aggregate. The NBRI method is relatively easy to carry out and provides results in a short period of time.

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6. American Society for Testing and Materials. ASTM C441-81. Annual Book of ASTM Standards, Part 14, Philadelphia, Pa, (1981).
7. M.P. Brandt, R.E. Oberholster and W.B. Westra. Proc 5th Intern Conf on Alkali-Aggregate Reaction in Concrete, Paper S252/10, Cape Town (1981).
8. Discussion by S Diamond on Reference 7.
9. American Society for Testing and Materials. ASTM C227-81. Annual Book of ASTM Standards, Part 14, Philadelphia, PA, (1981).
10. R. E. Oberholster and G. Davies. Cement Concr Res, 16, 181 (1986).
11. R.E. Oberholster and W.B. Westra. Proc 5th Intern Conf on Alkali-Aggregate Reaction in Concrete, Paper S252/31, Cape Town (1981).
12. K.E. Putterill and R.E. Oberholster. CSIR Research Report BRR 626, Pretoria (1985).
13. R.E. Oberholster and G. Davies. Proc 7th Intern Conf on Alkali-Aggregate Reaction, Ottawa (1986).

Appendix Q

FMC® Material Safety Data Sheet

LIFETIME (TM) SL ADMIXTURE
MSDS Number: QS-MSD-130
Issued: 01/05/95
Replaces: New
Page: 1 of 5

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

MANUFACTURER:

FMC CORPORATION, LITHIUM DIVISION
P.O. BOX 795
BESSEMER CITY, NC 28016-0795
(704) 868-5300

EMERGENCY TELEPHONE NUMBERS

CHEMTREC:	(800) 424-9300	TRANSPORTATION
MEDICAL:	(303) 595-9048	POISON CENTER (CALL COLLECT)
FMC LITHIUM:	(704) 629-5361	PLANT EMERGENCY RESPONSE TEAM (PERT) (CALL COLLECT, 24 HOURS / EVERYDAY)

PRODUCT NAME: LIFETIME(TM) SL Admixture
TRADE NAME: Lithium hydroxide, monohydrate
SYNONYMS: None
CHEMICAL FORMULA: $LiOH.H_2O$
CHEMICAL FAMILY: Alkali
PRODUCT CAS NO.: 1310-66-3
PRODUCT USE: Chemical manufacturing
VERSION: USA/Canada

2. COMPOSITION/INFORMATION ON INGREDIENTS

COMPONENT	CAS NUMBER	WT %
Lithium hydroxide, monohydrate	1310-66-3	> 99

3. HAZARD IDENTIFICATION

EMERGENCY OVERVIEW:

Corrosive
Odorless, white crystals

HEALTH EFFECTS:

CORROSIVE to eyes (may cause blindness), skin, nose, throat and stomach. Continuous inhalation may cause lung damage.
(See Section 11. Toxicological Information).

4. FIRST AID MEASURES

EYES: Immediately flush with water for at least 15 minutes, lifting the upper and lower eyelids intermittently. See a medical doctor or ophthalmologist immediately.

SKIN: Immediately flush with plenty of water while removing contaminated clothing and/or shoes and thoroughly wash with soap and water. Obtain medical attention. Contact a medical doctor if necessary.

INHALATION: Remove to fresh air. If breathing discomfort occurs and persists, see a medical doctor. If breathing has stopped, give artificial respiration and see a medical doctor immediately.

INGESTION: Rinse mouth with water. Dilute by giving 1 or 2 glasses of water. Do not induce vomiting. Never give anything by mouth to an unconscious person. See a medical doctor immediately.

NOTES TO MEDICAL DOCTOR: Lithium hydroxide has high inhalation toxicity and corrosivity. Removal of exposure should be immediate, using copious water flushes and gastric lavage, if necessary. Treatment is otherwise symptomatic and supportive.

From Material Safety Data Sheet, for Lithium Hydroxide, Monohydrate.

5. FIRE FIGHTING MEASURES

PHYSICAL HAZARD: Non-combustible
EXTINGUISHING MEDIA: Any suitable for fire in surrounding area.
SPECIAL FIRE FIGHTING PROCEDURES: Wear full protective clothing and self-contained breathing apparatus (SCBA) approved for fire fighting. This is necessary to protect against the hazards of heat, products of combustion and oxygen deficiency. Do not breathe smoke, gases or vapors generated.
FLASH POINT: Not applicable
AUTOIGNITION TEMPERATURE: Not applicable
FLAMMABLE LIMITS IN AIR: UPPER: Not applicable
LOWER: Not applicable
HAZARDOUS COMBUSTION PRODUCTS: None
PROPERTIES CONTRIBUTING TO FLAMMABILITY: Not applicable
SENSITIVITY TO MECHANICAL IMPACT: Not applicable
SENSITIVITY TO STATIC DISCHARGE: Not applicable

(See Section 10. Stability and Reactivity).

NATIONAL FIRE PROTECTION CODE 704
FIRE (RED) - 0
HEALTH (BLUE) - 3
REACTIVITY (YELLOW) - 1
SPECIFIC HAZARD - None

6. ACCIDENTAL RELEASE MEASURES

PROCEDURE FOR RELEASE OR SPILL:

Sweep up and place in suitable transport container. Dispose of waste according to local, state and federal regulations.

Before cleanup measures begin; review the entire MSDS with particular attention to Section 3. Emergency Overview and Potential Health Effects; and Section 8. Recommended Personal Protective Equipment.

7. HANDLING AND STORAGE

HANDLING: Do not get in eyes, on skin or clothing. Avoid breathing dust. Wash thoroughly after handling.
STORAGE: Keep container closed. Store away from acids and water.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

ENGINEERING CONTROLS: Provide adequate ventilation.

RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT:
RESPIRATORY: When adequate ventilation is not available, wear a NIOSH/MSHA-approved respirator.
EYES: Safety glasses or goggles.
SPECIAL CLOTHING: Rubber gloves
OTHER: Quick-drench eyewash and safety shower.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION, CONTINUED

EXPOSURE LIMITS:

COMPONENT	CAS NO.	WT. %	TWA (ACGIH)	STEL (ACGIH)	PEL (OSHA)	CEILING (OSHA)
Lithium hydroxide, monohydrate	1310-66-3	> 99	-	-	-	-

Lithium hydroxide is considered to be similar to sodium and potassium hydroxides in its irritant/corrosion property. OSHA and ACGIH list a Ceiling TLV Limit of 2 mg/m^3 for these compounds. A Ceiling Limit (1 minute) of 1.75 mg/m^3 is recommended by the AIHA Workplace Environmental Exposure Level Guide, 1984.

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9. PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE:	White crystals	VAPOR DENSITY Air = 1:	Not applicable
ODOR:	Odorless	VAPOR PRESSURE:	Not applicable
MOLECULAR WEIGHT:	41.96	ODOR THRESHOLD:	Not applicable
BOILING POINT:	Not applicable	FREEZING/MELTING POINT:	470°C (878°F)
DENSITY OR SPECIFIC GRAVITY:	1.5 g/cc	SOLUBILITY IN WATER: % by wt. @ 25°C (77°F)	10
VOLATILES (%):	Not applicable	EVAPORATION RATE: Butyl Acetate = 1	Not applicable
WATER/OIL PARTITION COEFFICIENT:	Not applicable	PH (AS IS) @ 25°C:	Not applicable
		PH (1% solution) @ 25°C:	> 13

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10. STABILITY AND REACTIVITY

STABILITY:	Stable under normal storage and temperature conditions
CONDITIONS TO AVOID:	None
INCOMPATIBILITY WITH OTHER MATERIALS:	Acids, aluminum, zinc
HAZARDOUS DECOMPOSITION PRODUCTS:	None
HAZARDOUS POLYMERIZATION:	Will not occur

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11. TOXICOLOGICAL INFORMATION

ROUTES OF EXPOSURE

EYE:	Corrosive	
INHALATION:	Corrosive.	4 hour LC50 = 0.96 mg/L (rat) [Fund. Appl. Toxicol. 7:58 1986]
SKIN CONTACT:	Corrosive	
SKIN ABSORPTION:	Corrosive.	No data available.
INGESTION:	Corrosive.	No data available.

11. TOXICOLOGICAL INFORMATION, CONTINUED

EFFECTS OF OVEREXPOSURE

ACUTE EFFECTS: Lithium hydroxide is toxic by inhalation and corrosive to eyes (may cause blindness), skin, nose, throat and stomach.

CHRONIC EFFECTS: No data available.

CARCINOGENICITY: Product is not indicated to be carcinogenic.

NTP Annual:	Not Listed.
IARC Monograph:	Not listed.
OSHA 29 CFR Part 1910 Subpart Z:	Not listed.
ACGIH (Appendix A):	Not listed.

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12. ECOLOGICAL INFORMATION

Environmental toxicity testing of this product has not been carried out. The toxicity of the lithium ion in the aquatic environment is poorly defined. The hydroxide ion will affect the pH of the water depending on the buffering capacity of the receiving water.

13. DISPOSAL CONSIDERATIONS

Ensure disposal of material is in compliance with state, local and federal regulations.

14. TRANSPORTATION INFORMATION

U.S. DOT, INTERNATIONAL MARITIME, AND INTERNATIONAL AIR

Proper Shipping Name:	Lithium hydroxide, monohydrate
Classification:	8, Corrosive
Labels:	Corrosive, 8
UN Number:	UN 2680
Packing Group:	II

15. REGULATORY INFORMATION

STATE REGULATIONS

California Proposition 65: California Safe Drinking Water and Toxics Enforcement Act of 1986 requires that the Government of California develop a list of carcinogens and reproductive toxins and that no persons doing business shall knowingly expose any individual to a chemical known to the state to cause cancer or reproductive toxicity without first giving clear and reasonable warning to such an individual. FMC would like you to know that our Lithium Hydroxide, Monohydrate contains the indicated concentrations of chemicals which are listed by California as chemicals known to cause cancer (A) or reproductive toxicity (B).

Chemical	Concentration	Listed as A or B
Lithium carbonate	0.4 %	B

15. REGULATORY INFORMATION, CONTINUED

US FEDERAL REGULATIONS

TSCA, Inventory of Chemical Substances (40 CFR 710):
Lithium hydroxide is listed; the hydrated form is not required to be listed.

TSCA 12 (b) Export Notification (40 CFR 707.60):
This product is not subject to TSCA 12 (b) Export Notification requirements.

CERCLA (40 CFR 302.4):
Not listed

SARA Title III:
Section 302, Extremely Hazardous Substances (40 CFR 355):
Not listed
Section 313, Toxic Chemicals (40 CFR Part 372):
This product does not contain a toxic chemical subject to the reporting requirements of section 313 of Emergency Planning and Community Right-To-Know Act of 1986.

INTERNATIONAL REGULATIONS

Canadian (WHMIS) — Product Identification No.: 2680
Hazard Classification: Class E (Corrosive)
Disclosure List: Listed

SECTION 16. OTHER INFORMATION

This MSDS has been prepared to meet U.S. OSHA Hazard Communication Standard, 29 CFR 1910.1200 and Canadian WHMIS requirements.

Information Contact:

Manager, Marketing Services
FMC Corporation, Lithium Division
449 North Cox Road
P.O. Box 3925
Gastonia, North Carolina 28054-0020
704-868-5300

Lifetime is a trademark of FMC Corporation, Lithium Division.

FMC Material Safety Data Sheet

LIFETIME (TM) ADMIXTURE
MSDS Number: QS-MSD-121
Issued: 11/21/94
Replaces: 08/23/94

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

MANUFACTURER:

FMC CORPORATION, LITHIUM DIVISION
P.O. BOX 795
BESSEMER CITY, NC 28016-0795
(704) 868-5300

EMERGENCY TELEPHONE NUMBERS

CHEMTREC:	(800) 424-9300	TRANSPORTATION
MEDICAL:	(303) 595-9048	POISON CENTER (CALL COLLECT)
FMC LITHIUM:	(704) 629-5361	PLANT EMERGENCY RESPONSE TEAM (PERT) (CALL COLLECT, 24 HOURS / EVERYDAY)

PRODUCT NAME: LIFETIME(TM) Admixture
TRADE NAME: Lithium Hydroxide Solution
SYNONYMS: None
CHEMICAL FORMULA: $LiOH$
CHEMICAL FAMILY: Alkali
PRODUCT CAS NO.: 1310-65-2
PRODUCT USE: Research and Development Only
VERSION: USA/Canada

2. COMPOSITION/INFORMATION ON INGREDIENTS

COMPONENT	CAS NUMBER	WT %
Lithium hydroxide	1310-65-2	5 - 15
Water	7732-18-5	85 - 95

3. HAZARD IDENTIFICATION

EMERGENCY OVERVIEW:
Corrosive aqueous liquid

POTENTIAL HEALTH EFFECTS:
CORROSIVE to eyes (may cause blindness), skin, nose, throat and stomach. Continuous inhalation may cause lung damage.
(See Section 11. Toxicological Information).

4. FIRST AID MEASURES

EYES: Immediately flush with water for at least 15 minutes, lifting the upper and lower eyelids intermittently. See a medical doctor or ophthalmologist immediately.

SKIN: Immediately flush with plenty of water while removing contaminated clothing and/or shoes and thoroughly wash with soap and water. Obtain medical attention. Contact a medical doctor if necessary.

INHALATION: Remove to fresh air. If breathing discomfort occurs and persists, see a medical doctor. If breathing has stopped, give artificial respiration and see a medical doctor immediately.

INGESTION: Rinse mouth with water. Dilute by giving 1 or 2 glasses of water. Do not induce vomiting. Never give anything by mouth to an unconscious person. See a medical doctor immediately.

NOTES TO MEDICAL DOCTOR: Lithium hydroxide has high inhalation toxicity and corrosivity. Removal of exposure should be immediate, using copious water flushes and gastric lavage, if necessary. Treatment is otherwise symptomatic and supportive.

From Material Safety Data Sheet, for Lithium Hydroxide Solution. Copyright 1995 by the FMC Corporation, Lithium Division. Used by Permission.

5. FIRE FIGHTING MEASURES

PHYSICAL HAZARD: Non-combustible
EXTINGUISHING MEDIA: Any suited for fire in surrounding area.
SPECIAL FIRE FIGHTING PROCEDURES: Wear full protective clothing and self-contained breathing apparatus (SCBA) approved for firefighting. This is necessary to protect against the hazards of heat, products of combustion and oxygen deficiency. Do not breathe smoke, gases or vapors generated.
FLASH POINT: Not applicable
AUTOIGNITION TEMPERATURE: Not applicable
FLAMMABLE LIMITS IN AIR: UPPER: Not applicable
LOWER: Not applicable
HAZARDOUS COMBUSTION PRODUCTS: None
PROPERTIES CONTRIBUTING TO FLAMMABILITY: None
SENSITIVITY TO MECHANICAL IMPACT: Not applicable
SENSITIVITY TO STATIC DISCHARGE: Not applicable

(See Section 10. Stability and Reactivity).

NATIONAL FIRE PROTECTION CODE 704
FIRE (RED) - 0
HEALTH (BLUE) - 3
REACTIVITY (YELLOW) - 0
SPECIFIC HAZARD - None

6. ACCIDENTAL RELEASE MEASURES

PROCEDURE FOR RELEASE OR SPILL:

Contain spill with absorbant. Transfer or pump into a suitable container. Material may be neutralized with mild acid, such as acetic. Dispose of waste according to local and federal authorities.

Before cleanup measures begin; review the entire MSDS with particular attention to Section 3. Emergency Overview and Potential Health Effects; and Section 8. Recommended Personal Protective Equipment.

7. HANDLING AND STORAGE

HANDLING: Do not get in eyes, on skin or clothing. Wash thoroughly after handling.
STORAGE: Keep container closed. Store away from strong acids.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

ENGINEERING CONTROLS: Provide adequate ventilation.

RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT:
RESPIRATORY: When adequate ventilation is not available, wear a NIOSH/MSHA-approved respirator.
EYES: Chemical splash goggles with a faceshield.
SPECIAL CLOTHING: Rubber gloves; rubber clothing recommended.
OTHER: Quick-drench eyewash and safety shower.

5. FIRE FIGHTING MEASURES

PHYSICAL HAZARD: Non-combustible
EXTINGUISHING MEDIA: Any suited for fire in surrounding area.
SPECIAL FIRE FIGHTING PROCEDURES: Wear full protective clothing and self-contained breathing apparatus (SCBA) approved for firefighting. This is necessary to protect against the hazards of heat, products of combustion and oxygen deficiency. Do not breathe smoke, gases or vapors generated.
FLASH POINT: Not applicable
AUTOIGNITION TEMPERATURE: Not applicable
FLAMMABLE LIMITS IN AIR: UPPER: Not applicable
LOWER: Not applicable
HAZARDOUS COMBUSTION PRODUCTS: None
PROPERTIES CONTRIBUTING TO FLAMMABILITY: None
SENSITIVITY TO MECHANICAL IMPACT: Not applicable
SENSITIVITY TO STATIC DISCHARGE: Not applicable

(See Section 10. Stability and Reactivity).

NATIONAL FIRE PROTECTION CODE 704
FIRE (RED) - 0
HEALTH (BLUE) - 3
REACTIVITY (YELLOW) - 0
SPECIFIC HAZARD - None

6. ACCIDENTAL RELEASE MEASURES

PROCEDURE FOR RELEASE OR SPILL:

Contain spill with absorbant. Transfer or pump into a suitable container. Material may be neutralized with mild acid, such as acetic. Dispose of waste according to local and federal authorities.

Before cleanup measures begin; review the entire MSDS with particular attention to Section 3. Emergency Overview and Potential Health Effects; and Section 8. Recommended Personal Protective Equipment.

7. HANDLING AND STORAGE

HANDLING: Do not get in eyes, on skin or clothing. Wash thoroughly after handling.
STORAGE: Keep container closed. Store away from strong acids.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

ENGINEERING CONTROLS: Provide adequate ventilation.

RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT:
RESPIRATORY: When adequate ventilation is not available, wear a NIOSH/MSHA-approved respirator.
EYES: Chemical splash goggles with a faceshield.
SPECIAL CLOTHING: Rubber gloves; rubber clothing recommended.
OTHER: Quick-drench eyewash and safety shower.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION INFORMATION continued

EXPOSURE LIMITS:

COMPONENT	CAS NO.	WT. %	TWA (ACGIH)	STEL (ACGIH)	PEL (OSHA)	CEILING (OSHA)
Lithium hydroxide	1310-65-2	5-15	-	-	-	-
Water	7732-18-5	85 - 95	-	-	-	-

Lithium hydroxide is considered to be similar to sodium and potassium hydroxides in its irritant/corrosion property. OSHA and ACGIH list a Ceiling TLV Limit of 2 mg/m^3 for these compounds. A Ceiling Limit (1 minute) of 1.0 mg/m^3 is recommended by the AIHA Workplace Environmental Exposure Level Guide, 1984.

9. PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE:	Clear liquid	VAPOR DENSITY Air = 1:	Not available
ODOR:	Odorless	VAPOR PRESSURE:	Not applicable
MOLECULAR WEIGHT:	23.95 (LiOH)	ODOR THRESHOLD:	Not applicable
BOILING POINT:	ca. 101°C (214°F)	FREEZING/MELTING POINT:	-18°C (- 0.4°F)
DENSITY/SPECIFIC GRAVITY:	1.1 @ 20°C	SOLUBILITY IN WATER: % by wt. @ 25°C (77°F)	100
VOLATILES (%):	Not applicable	EVAPORATION RATE: Butyl Acetate = 1	As water
WATER/OIL PARTITION COEFFICIENT:	Not available	PH (AS IS) @ 25°C:	12.7
		PH (1% Solution) @ 25°C:	>12

10. STABILITY AND REACTIVITY

STABILITY:	Stable under normal storage and temperature conditions
CONDITIONS TO AVOID:	None
INCOMPATIBILITY WITH OTHER MATERIALS:	Acids, aluminum, zinc
HAZARDOUS DECOMPOSITION PRODUCTS:	None
HAZARDOUS POLYMERIZATION:	Will not occur

11. TOXICOLOGICAL INFORMATION

ROUTES OF EXPOSURE

EYE:	No data available for the formulation. LiOH: Corrosive
INHALATION:	No data available for the formulation. LiOH: 4 hour LC50 = 0.96 mg/L (rat) [Fund. Appl. Toxicol 7:58 1986]
SKIN CONTACT:	No data available for the formulation. LiOH: Corrosive
SKIN ABSORPTION:	No data available for the formulation.
INGESTION:	No data available for the formulation. LiOH: Corrosive

15. REGULATORY INFORMATION continued

US FEDERAL REGULATIONS

TSCA, Inventory of Chemical Substance (40 CFR 710):

This product is for research and development purposes only, and not for commercial use. Lithium hydroxide is listed.

TSCA 12 (b) Export Notification (40 CFR 707.60):

This product is not subject to TSCA 12 (b) Export Notification requirements.

CERCLA (40 CFR 302.4):

Not listed.

SARA Title III:

Section 302, Extremely Hazardous Substance (40 CFR 355):

Not listed.

Section 313, Toxic Chemicals (40 CFR 372):

This product does not contain a toxic chemical subject to the reporting requirements of section 313 of Emergency Planning and Community Right-To-Know Act of 1986.

INTERNATIONAL REGULATIONS

Canadian (WHMIS) — Product Identification No.: 2679
Hazard Classification: Class E (Corrosive)
Disclosure List: Listed

SECTION 16. OTHER INFORMATION

This MSDS has been prepared to meet U.S. OSHA Hazard Communication Standard, 29 CFR 1910.1200 and Canadian WHMIS requirements.

Since the previous issue, revisions were made for this MSDS in the following sections:
Section: 3, 9, 11

Information Contact:

Manager, Marketing Services
FMC Corporation, Lithium Division
449 North Cox Road
P.O. Box 3925
Gastonia, North Carolina 28054-0020
704-868-5300

Appendix R

Portland Cement Association

CONCRETE

INFORMATION

Guide Specification for Concrete Subject to Alkali-Silica Reactions

Developed by

Alkali-Silica Reactivity/Pavement Durability Task Group
Portland Cement Association

FOREWORD

Most aggregates are chemically stable in hydraulic-cement concrete, without deleterious interaction with other concrete ingredients. However, this is not the case for aggregates containing certain siliceous substances that react with soluble alkalies in concrete, resulting in detrimental expansion and cracking of concrete structures. Alkali-silica reactivity (ASR), which was first reported in 1940, is now known worldwide. Fortunately, most concrete is not affected by this reaction. Although the risk of catastrophic failure and the number of affected structures are low, ASR-induced cracking can exacerbate other deterioration mechanisms such as occur in frost, deicer, or sulfate exposures. ASR can be controlled by the methods presented in this guide specification.

This guide specification is modeled after the *Guide Specifications for Concrete Subject to Alkali-Silica Reactions*, developed in 1993 by the Mid-Atlantic Regional Technical Committee: a group of concrete users, material suppliers, consulting engineers, and state transportation engineers. This guide specification expands the principles of the Mid-Atlantic document to provide a national, rather than regional, approach to ASR. Because different regions have different needs and materials available, this guide specification provides several options for the user to control ASR.

This specification is designed to be used by owners, transportation engineers, structural engineers, and others to provide safeguards against the occurrence of ASR failures. As with all specifications, this specification should be used only by qualified professionals who are competent to evaluate the significance and limitations of the specification and who will accept responsibility for the application of its requirements to the structure under consideration.

From Concrete Information, Guide Specification for Concrete Subject to Alkali-Silica Reactions. March 1995 by the Portland Cement Association, Skokie, IL. Used by Permission.

March 1995

IS415

1.0 SCOPE

The special provisions stated herein are for use in modifying a specification for concrete made with a potentially reactive aggregate. In such instances, the potential for detrimental alkali-silica reactions in concrete exists, and these special provisions are applicable to aggregates found in North America.

Note 1: These provisions do not apply to alkali-carbonate reactivity and do not apply to concrete that will be continually dry in service. An internal relative humidity of less than 80% at 23°C (73°F) indicates insufficient moisture is available in the concrete for expansion due to ASR to occur.

2.0 REFERENCED DOCUMENTS

2.1 *American Society for Testing and Materials (ASTM)*:

C 33	Specification for Concrete Aggregates
C 109	Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)
C 150	Specification for Portland Cement
C 227	Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)
C 294	Descriptive Nomenclature of Constituents of Natural Mineral Aggregates
C 295	Guide for Petrographic Examination of Aggregates for Concrete
C 441	Test Method for Effectiveness of Mineral Admixtures or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction
C 595	Specification for Blended Hydraulic Cements
C 618	Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete
C 823	Practice for Examination and Sampling of Hardened Concrete in Construction
C 856	Practice for Petrographic Examination of Hardened Concrete
C 989	Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortars
C 1077	Practice for Laboratories Testing Concrete and Concrete Aggregates for Use in Construction and Criteria for Laboratory Evaluation
C 1157	Performance Specification for Blended Hydraulic Cement
C 1222	Practice for Evaluation of Laboratories Testing Hydraulic Cement
C 1240	Silica Fume for Use in Hydraulic Cement Concrete and Mortar
C 1260	Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method) (formerly P 214)

2.2 *Canadian Standards Association (CSA)*:

A23.2-14A	Potential Expansivity of Aggregates (Procedure for Length Change due to Alkali-Aggregate Reaction in Concrete Prisms)

3.0 TERMINOLOGY

3.1 *Alkalies*: Sodium and potassium, expressed analytically as oxides, are generally derived from portland cement, but may be derived from pozzolans, chemical admixtures, and, in some instances, aggregates. Alkalies may also be available to concrete from external sources such as deicing salts, soils, sea water, and industrial processes. Alkali content is expressed as percent Na_2O equivalent and calculated as:

$$(Na_2O + 0.658\ K_2O)$$

3.2 *Pozzolan*: In this guide specification, pozzolanic materials are considered to include: fly ash and raw or calcined natural pozzolan (ASTM C 618) and silica fume (ASTM C 1240).

3.3 *Slag*: Ground granulated blast-furnace slag (ASTM C 989).

3.4 *Alkali-Silica Reaction*: A chemical reaction in concrete between alkali hydroxides and certain siliceous aggregates and some calcareous aggregates containing certain forms of silica. The products of this reaction, under certain conditions, may cause deleterious expansion within the concrete.

4.0 SUBMITTALS

Initial submittals shall include the information required in the following subparagraphs. Whenever the sources or the relevant properties of the materials change, a resubmittal of the relevant information shall be made demonstrating compliance with the applicable provisions.

4.1 *Aggregates*: Laboratory test reports shall be submitted by the supplier of the aggregates and shall contain data on mineralogy (ASTM C 295) and potential reactivity with alkalies as specified in Section 5 herein.

4.2 *Portland Cements*: Laboratory test reports shall be submitted by the supplier of portland cement and shall contain the alkali content in percent expressed as Na_2O equivalent.

4.3 *Blended Hydraulic Cements*: Laboratory test reports shall be submitted by the supplier of blended hydraulic cement demonstrating compliance with Sections 6.0 and 7.0. The statement shall include data on the alkali contents of the portland cement and pozzolan or slag constituents and sources of cement and percentages of pozzolan or slag used in the manufacture of the blended cement.

4.4 *Pozzolans and Slags*: Laboratory test reports shall be submitted by the supplier of pozzolanic or slag materials demonstrating compliance with the requirements of Sections 6.0 and 7.0. The available alkali content of the pozzolan or slag shall be included in the test report.

4.5 *Chemical Admixtures*: Laboratory test reports shall be submitted by the supplier of chemical admixtures, which shall include information on the chloride ion content and alkali content expressed as Na_2O equivalent. Test reports are not required for air-entraining admixtures used at dosages less than 130 mL per 100 kg (2 fl oz per 100 lb) of cement or nonchloride chemical admixtures used at maximum dosages less than 325 mL per 100 kg (5 fl oz per 100 lb). Both the chloride ion and total alkali content of the admixture are to be expressed in percent by mass of cement for a stated or typical dosage of the admixture, generally in milliliters per 100 kg or fluid ounces per 100 lb of cement.

4.6 *Laboratories*: Laboratories supplying test reports or data, as required in this guide specification, shall provide evidence that the laboratory is properly equipped and qualified to perform the test method(s).

Note 2: See ASTM C 1077 and C 1222 for laboratory evaluation criteria.

5.0 AGGREGATES

A field performance history is the best method of evaluating the susceptibility of an aggregate to ASR. When evaluating past field performance, the following should be determined: (1) are the cement content of the concrete, the alkali content of the cement, and the water-cement ratio of the concrete the same or higher than proposed for future use, (2) is the field concrete at least 15 years old, (3) are the exposure conditions of the field concrete at least as severe as those proposed for future use, and (4) were pozzolans used in the field concrete? In addition, the current aggregate supply should be examined petrographically to ensure that it is representative of that used in the field concrete. When field history is not available, or when any evidence of ASR is indicated, the tests outlined below shall be used to evaluate the potential reactivity of aggregate.

5.1 *Evaluation of Aggregates*: Aggregates for use in concrete shall conform to the requirements of ASTM C 33. Testing and evaluation for alkali-silica reaction are outlined below. Testing of an aggregate need not be performed in the order listed.

Note 3. The ASR criteria contained in Appendix XI of ASTM C 33-92a may not identify some of the aggregates in North America that are potentially reactive.

5.1.1 Petrographic analysis in accordance with ASTM C 295 shall identify the constituents of the fine and coarse aggregate. Fine and coarse aggregate containing more than the following quantities of constituents shall be considered potentially reactive:

- (a) Optically strained, microfractured, or microcrystalline quartz exceeding 5.0% (a common constituent of granite and granite gneiss)
- (b) Chert or chalcedony exceeding 3.0%
- (c) Tridymite or cristobalite exceeding 1.0%
- (d) Opal exceeding 0.5%
- (e) Natural volcanic glass in volcanic rocks exceeding 3.0%

Note 4. The constituent limits are adopted from the *Guide Specification for Concrete Subject to Alkali-Silica Reactions* of the Mid-Atlantic Regional Technical Committee.

5.1.2 Aggregate shall be evaluated by ASTM C 1260. Aggregate sources which exhibit C 1260 mean mortar bar expansion at 14 days greater than 0.10% shall be considered potentially reactive.

Note 5. The limit of 0.10% is adopted from the lower limit in the Appendix of ASTM C 1260.

5.1.3 Aggregate considered potentially reactive by 5.1.1 or 5.1.2 may be further evaluated by CSA A23.2-14A. Aggregate sources, that exhibit mean concrete prism expansion at one year greater than 0.04%, shall be considered potentially reactive. Aggregate sources exhibiting expansions no more than 0.04% and demonstrating no prior evidence of reactivity in the field shall be considered nonreactive.

Note 6. The concrete prism test is adopted from Canadian practice and is the CSA recommended test for evaluating aggregates for potential reactivity. Extensive testing using this method indicates that most potentially reactive aggregates will be detected. The test method has also been correlated to field performance in Canada.

Note 7. The classification of an aggregate as potentially reactive may be appealed if additional tests or service record data supporting the reclassification are acceptable to the specifier. Discrepancies between field performance and test results do occur. For example, an aggregate may be declared potentially reactive by the above methods, yet have no evidence of ASR in field performance. This occurs because the tests in Sections 5.1.2 and 5.1.3 have artificially high alkali concentrations and relatively high temperatures to accelerate any potential reactivity. In most cases, field concretes would not experience such high alkali levels, and as long as the field alkali level remains below the threshold at which an aggregate would react, the risk of deleterious reactivity is minimized. This demonstrates the importance of using field performance data.

5.1.4 Aggregates that have shown evidence of reactivity in service shall be considered potentially reactive regardless of the results of Sections 5.1.1, 5.1.2, and 5.1.3. Determination of reactivity in structures must include:

- (a) Petrographic analysis of cores by ASTM C 856
- (b) Visual examination of cracking and expansion of elements of the structure
- (c) Evaluation of available data, including construction records

5.2 Aggregate determined to be potentially reactive with alkalies shall be used only in concrete with cementitious materials conforming to the requirements of Section 6.

6.0 CEMENTITIOUS MATERIALS

One of the following three options shall be used to control alkali-silica reactions.

6.1 *Option A*: Use a combination of a pozzolan or slag with a portland or a blended cement, which meets the requirements of the following subsections of Section 6.1.

- 6.1.1 If a blended cement is used with additional pozzolan or slag, under this option, the blended cement must conform to ASTM C 595 [Types IP, I(PM), P, IS or I(SM)] or ASTM C1157. The required minimum pozzolan or slag content shall be that determined to be "effective" in Section 7.
- 6.1.2 Fly ash or natural pozzolan shall conform to ASTM C 618, including the optional chemical requirement of a maximum of 1.5% available alkali. The required minimum fly ash or natural pozzolan content shall be that determined to be "effective" in Section 7.
- 6.1.3 Slag shall conform to ASTM C 989 Grade 120 and Grade 100. Silica fume shall conform to ASTM C 1240. The required minimum slag or silica fume content shall be that determined to be effective in Section 7.
- 6.1.4 The alkali content of the cement, pozzolan, or slag proposed for use under this option shall not be more than 0.05 percentage points greater than that of the corresponding materials used in the "Test Mixture" to evaluate the effectiveness of the pozzolan or slag in Section 7.2.3.2., unless Section 7.1 is used.

6.2 *Option B*: Use a blended hydraulic cement that has been demonstrated to be effective in controlling expansion as specified in Section 7 and that conforms to ASTM C 595 [Types I(PM), I(SM), P, IS or IP] or ASTM C 1157.

Note 8. Deleterious expansion of concrete due to ASR can be safely controlled by proper use of pozzolans, slags, or blended cements, and this is the preferred approach. However, if Options A and B cannot be applied because pozzolans, slags, and blended cements are not available, or other engineering concerns preclude their use, Option C can be used.

6.3 *Option C*: Select portland cement and other concrete ingredients to limit concrete alkali content based on proven field performance under similar conditions with the potentially reactive aggregate. For service conditions more severe than experienced in the past, such as increased exposure to external alkalies or increased concrete alkali content, this option may not apply. See Section 5.0 for guidance on field performance histories.

Note 9. Specifications in the United States have traditionally limited the alkali content in concrete through use of low-alkali cement. ASTM C 150 defines low-alkali cement as having a maximum alkali content of 0.60% equivalent Na_2O. However, higher alkali limits have been safely used with certain moderately reactive aggregates. Another alternative, the practice of limiting the alkali content of the concrete based on alkali from concrete ingredients, is accepted in Europe and Canada. In Canada and Europe when reactive aggregates are used in concrete containing less than 3 kg per cubic meter (5 lb per cubic yard) of alkali, deleterious expansion usually does not take place. In certain European countries, such as the United Kingdom, the effective alkali content is the summation of the total sodium oxide equivalent for portland cement, natural pozzolan, and silica fume, and a percentage of the total sodium oxide equivalent for fly ash and slag (17% for fly ash and 50% for slag). Restrictions on pozzolans are also included with this approach. For mass-concrete structures where slight expansion may be deleterious, for external sources of alkali, or for when severely reactive aggregate is used, the 3 kg alkali limit may not be effective. An alternative to the 3 kg limit is to determine the effective alkali burden from properly performing field structures in a locality and apply that historically established alkali limit to new structures when similar materials are used in a similar environment.

Note 10. Field and laboratory research indicates that the use of chemical inhibitors, such as lithium compounds, can control ASR, and represents another potential solution.

7.0 EFFECTIVENESS OF A POZZOLAN, SLAG, OR BLENDED CEMENT IN CONTROLLING EXPANSION

The limits on expansion referenced in ASTM Standards C 595, C 989, and C 1157 are not applicable. The effectiveness of a pozzolan, slag, or blended cement in controlling deleterious expansion shall be determined by one of the two options outlined below. Where applicable, different amounts of pozzolan or slag should be tested to determine the minimum effective dosage.

7.1 *Option A—Criteria for Effectiveness*:

7.1.1 *Pozzolans or Slags*: Pozzolan, slag and portland cement, or pozzolan, slag, and blended cement combinations that exhibit ASTM C 1260 mean mortar bar expansion at 14 days less than or equal to 0.10% shall be considered acceptable to control ASR for a particular job aggregate.

7.1.2 *Blended Cements*: Blended cement combinations that exhibit ASTM C 1260 mean mortar bar expansion at 14 days less than or equal to 0.10% shall be considered acceptable to control ASR for a particular job aggregate.

7.1.3 *Test Procedure*: Mix the pozzolan, slag, or blended cement combinations with the job aggregates and test in accordance with ASTM C 1260. Where applicable, different amounts of pozzolan or slag should be tested to determine the minimum effective dosage.

7.1.3.1 *Test Mixture*: The test mixture shall be made with 990 g of ASTM C1260 prepared job aggregate and one of the following:

(a) An ASTM C 150 cement and pozzolan or slag. The total mass of cement plus pozzolan or slag shall be 440 g;

(b) A blended cement. The total mass of blended cement shall be 440 g;

(c) A blended cement with additional pozzolan or slag. The total mass of blended cement and pozzolan or slag shall be 440 g.

7.2 *Option B—Criteria for Effectiveness*:

7.2.1 *Pozzolans or Slags*: The 14-day expansions of the "test mixture" made with the pozzolan or slag and the selected high-alkali cement shall be no greater than the expansion of a "control mixture" made with a low-alkali cement with total Na_2O equivalent alkalies between 0.50% and 0.60%.

7.2.2 *Blended Cements*: The expansion of a "test mixture" made with blended hydraulic cement shall be no greater than the expansion of a "control mixture" made with a low-alkali cement with total Na_2O equivalent alkalies between 0.50% and 0.60%.

7.2.3 *Test Procedure*: Mix "control" and "test" mixtures using 900 g of the Pyrex glass aggregate specified in ASTM C 441. The amount of mixing water shall be that needed to produce a flow of 100 to 115 as determined in ASTM Method C 109.

7.2.3.1 *Control Mixture*: The control mixture shall be made with 400 g of cement conforming to ASTM C 150 with total Na_2O equivalent alkalies of 0.50% to 0.60%.

7.2.3.2 *Test Mixture*: The test mixture shall be made with one of the following:

(a) An ASTM C 150 cement, with a total Na_2O equivalent alkali content exceeding 0.95% as manufactured, and pozzolan or slag. The total mass of cement plus pozzolan or slag shall be 400 g;

(b) A blended cement;

(c) A blended cement with additional pozzolan or slag. The pozzolan or slag used in the manufacture of the blended cement shall be included in the calculation of the percent pozzolan or slag. Pozzolan or slag percentages are calculated as a percent of the total mass or weight of cement plus pozzolan or slag.

7.2.4 *Specimen Preparation*: Prepare, store, and measure specimens, and calculate expansion in accordance with Sections 7.2, 8, and 9 of ASTM C 227-90. Measure the bars at an age of 14 days and calculate expansion from the length at 24 ± 2 hr. Express the expansion of the test mixture as a percent of the control mixture.

PCA R&D Serial No. 2001

Portland Cement Association *5420 Old Orchard Road, Skokie, Illinois 60077-1083 (708) 966-6200, FAX (708) 966-8389*

An organization of cement manufacturers to improve
and extend the uses of portland cement and concrete
through market development, engineering, research,
education, and public affairs work.

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Appendix S