

RILEM Recommended Test Method AAR-1: Detection of potential alkali-reactivity of aggregates – Petrographic method

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The text presented hereafter is a draft for general consideration. Comments should be sent to the TC Chairman: Dr. Philip Nixon, Building Research Establishment (BRE), Garston, Watford WD2 7JR, UK; Tel.: +44 923 664 239; Fax: +44 923 664 010; Email: nixonP@bre.co.uk, by 31 January 2004.

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1. SCOPE

Petrographic analysis shall always be the first step in the assessment of the potential alkali-reactivity of concrete aggregates as stated in RILEM AAR-0 entitled, 'Outline Guide to the Use of RILEM Methods in Assessments of Aggregates for potential alkali-reactivity'.

This RILEM method specifies a general procedure for the petrographic examination of concrete aggregates, to identify rock types and minerals that might react with hydroxyl ions from the concrete pore solution. The method, including sampling, is intended to be used for the routine examination of natural aggregates including sand, coarse gravel, all-in and crushed rock aggregates, in testing laboratories, rather than by expert petrographers for research purposes. The method can be also used to quantify the amounts of various mineral and rock types where required. The main objective of the RILEM petrographic method is to determine a classification of a particular aggregate in terms of alkali-reactivity. As a result of undergoing the RILEM petrographic examination an aggregate should be classified as one of the following:

- Very unlikely to be alkali-reactive Class I
- Alkali-reactivity uncertain Class II
- Very likely to be alkali-reactive Class III

Acceptance and experience with reactive constituents differ between countries, and thus, final assessment of reactive constituents should where possible follow national or regional experiences, recommendations and specifications. RILEM AAR-0 also gives guidance on the further testing that may be undertaken on the basis of the findings of the AAR-1 petrographical examination.

It is very important that the petrographic analysis is carried out by a qualified geologist or petrographer with experience of materials used for concrete and good local knowledge of alkali-reactive aggregates and minerals.

2. DEFINITIONS

2.1 Alkali-aggregate reaction (AAR)

Chemical-physical expansive reaction in mortar or concrete between reactive mineral phases in aggregates and alkali hydroxide and/or calcium hydroxide in the pore solution from the cement paste or from external sources. The reaction can presently be divided in two types: Alkali-Silica Reaction (ASR) and Alkali-Carbonate Reaction (ACR). Worldwide, the occurrence of ASR is much more common that the occurrence of ACR.

2.2 Alkali-reactive aggregates

Alkali-reactive aggregates are aggregates that have been shown to react with alkali hydroxide and/or calcium hydroxide, usually derived mainly from the cement paste pore solution of a concrete, creating an internal expansive reaction within the aggregate particle which in more extreme cases can severely crack the surrounding concrete. Generally reactive aggregates can be grouped into fast acting reactive siliceous aggregates, slower or late reactive siliceous aggregates, or alkali-reactive carbonate aggregates.

2.3 All-in aggregate

This is an aggregate or a sample of aggregate consisting of both coarse (>4 mm) and fine (\leq 4 mm) size fractions.

2.4 Critical proportion

This is the content of reactive constituents within a total aggregate volume, beyond which it has been shown to develop a deleterious level of expansion induced by AAR. This proportion can vary depending on the criteria being employed to classify a concrete mix as potentially deleterious.

2.5 Cryptocrystalline

Cryptocrystalline rock texture too small to be resolvable under the petrographic microscope (< 0.004 mm or 4μ m).

2.6 Grain/crystal size

It is the size of a crystal or grain in a particular rock particle or mineral.

2.7 Innocuous constituent

This is a material, which is known not to be potentially reactive with respect to deleterious AAR.

2.8 Microcrystalline

Microcrystalline rock texture so fine-grained that a petrographic microscope is needed to resolve individual crystals (0.004 mm - 0.062 mm or 4 μ m - 62 μ m).

2.9 Monomictic

Monomictic aggregates essentially comprise particles of one rock or mineral type.

2.10 Pessimum behaviour

It is characterized by the development of differing levels of AAR expansion in concretes containing different contents of a reactive constituent. A maximum level of expansion will occur at a particular content of the reactive constituent known as the 'pessimum'. Decreasing levels of expansion will develop for contents of the reactive constituents away from the 'pessimum'. The 'pessimum' may differ for differing potentially reactive constituents.

2.11 Petrography

Petrography is the systematic description of rock, mainly with the unaided eye and by means of microscopical methods. The petrographic examination of aggregates describes the structure, texture and mineral composition of the rocks and minerals forming the various particles.

2.12 Polymictic

Polymictic aggregates comprise particles of many different rock and mineral types.

2.13 Potentially reactive constituent

This is a constituent, which in some circumstances has been previously shown to demonstrate deleterious levels of expansion induced by AAR.

2.14 Qualitative examination

It is an examination in which some or all of the constituents of a sample are identified and described, and the approximate relative proportions of those various constituents are estimated.

2.15 Quantitative examination

It is an examination in which the amounts and proportion of the constituents of a sample are determined with a required degree of accuracy.

2.16 Size of aggregate particles

This test method designates fine aggregate to consist of particles less than or equal to 4 mm, whilst coarse aggregate consists of particles larger than 4 mm.

3. PRINCIPLES

The RILEM petrographical examination method describes the visual recognition and quantification techniques for rocks and mineral constituents of aggregate sources with special emphasis on their potential for alkali reactivity. The procedures being followed are summarised in flow chart A.1.1. It assumes the petrographer is competent to recognize, unambiguously by visual examination and/or thin-section examination, the complete suite of minerals, both non-reactive and potentially reactive, likely to be encountered in an aggregate sample, based upon field experience.

Two techniques are described; the choice of usage is usually dependent on the particle size of the aggregate (or quantity of aggregate sample) and complexity being investigated. It is assumed that both techniques would be employed in most circumstances:

- Technique 1, which is primarily used for coarse aggregate particles, in which the constituents of the various particles can be unequivocally determined by visual inspection.
- Technique 2 is used to identify the various constituent minerals of an aggregate and quantify them by point-counting within a thin-section.

Both techniques should give a full petrographic composition of rock and mineral constituents of the material concerned. For aggregates where all the mineral constituents cannot be determined by either technique, or the petrographer suspects the presence of reactive constituents such as finely disseminated opal, complementary analysis techniques are recommended including chemical analysis, X-ray diffraction analysis, staining and the gel-pat test (see Annex 6).

4. APPARATUS

4.1 **Preparation of samples**

4.1.1 The sieves used shall comply with ISO 3310.

4.1.2 A sample divider (*e.g.* riffle-box or rotated divider) appropriate to the maximum particle size to be handled, *e.g.* according to prEN 932-1.

4.1.3 Balances capable of weighing the test portions to an accuracy of 0.1%.

4.2 Technique 1 (Coarse aggregate route see flow chart A.1.1)

4.2.1 Hand lens.

- 4.2.2 Pen knife or similar steel probe.
- 4.2.3 Stereoscopic microscope (*e.g.* x10 to x80).

4.2.4 Reagent: diluted hydrochloric acid approx. 10% by volume of concentrated HCl.

4.3 Technique 2 (Fine aggregate route see flow chart A.1.1.)

4.3.1 Laboratory crushing equipment used to reduce the particle-size to a maximum of 4 mm, if required. The crusher must be cleaned thoroughly before use.

4.3.2 Apparatus and machinery suitable for the preparation of $25-30 \mu$ m-thick geological thin-sections. This should include the ability to prepare thin-sections impregnated with epoxy resin containing a fluorescent dye. This technique not only consolidates friable materials, but can also be used to characterise the internal porosity of the aggregate constituents where necessary.

4.3.3 A petrological (*i.e.* polarizing) microscope capable of high-resolution at various magnifications up to at least x500. Fluorescence microscopy requires the microscope to be fitted with filters: an excitation band pass filter (*e.g.* Leitz BG12, max transmission 400 nm) and a suppression

long pass filter (e.g. Leitz K510, max transmission 500-550 nm).

4.3.4 A point-counting apparatus for use with the petrological microscope. A commonly used type is an electromechanical stage linked to a counting device, such that operation of any one of the counters automatically moves the thin-section one further step along the traverse. Alternatively a counting graticule placed within the eyepiece of the microscope can be used.

5. PREPARATORY INFORMATION

Subject to availability, information on known potentially reactive aggregates may be usefully obtained before assessing the sample.

5.1 Geological information

This information may be in the form of one or more of the following:

• Geological map (*e.g.* 1/50000 scale) locating the extraction site with the respect to all the major lithological units.

• Borehole data containing the major lithological units.

• Detailed macroscopical and microscopical descriptions of units, including rock and mineral composition. It is of prime importance that these descriptions include the presence of minerals considered as potentially reactive. The descriptions should use standardized nomenclature of rock and minerals (see clause 5.3).

• Structural information *e.g.* about joints, faults and thrust planes.

• Glacial and fluvial processes showing transport directions.

• Weathering and other climatic effects on the aggregate.

• Mapping and research information from the published literature.

It is recommended that where practicable the petrographer should visit the site of extraction and advise on the sampling locations.

5.2 Reactive aggregates

Any available information on previous alkali-reactivity assessments of the quarry or rocks/lithological units should be made available to the analyst, including materials such as recommendations, specifications, petrographic descriptions of reactive aggregates, field experiences, laboratory results and other information of importance for the assessment of reactivity.

Alkali-reactive aggregates consist of many different rock and mineral types. There is evidence that apparently similar rock types can vary greatly in reactivity in practice depending on their genesis and geographical location. For several rock types, there are reactive and non-reactive varieties according to differences in the detailed mineralogical composition or mixture. Consequently it is not reliable to classify genetic rock types as reactive or nonreactive and each aggregate with a lithology known to have some association with ASR should be examined closely by technique 2 to determine their possible alkali-reactivity (see flow chart A.1.1 Interpretation and classification). Acceptance criteria for various types of reactive aggregate and the test methods used thus vary between countries, and guidance should be obtained from national practice and recommendations.

<u>NOTE</u>: Annexes A.1.2 and A.1.3 give tables summarizing experience on the potential alkali-reactivity of various rock types and mineral constituents.

Annex A.1.4 gives references on potentially alkalireactive aggregates. Examples of petrographic descriptions of some alkali-reactive rock and mineral types are given in Annex 5.

5.3 Nomenclature

Internationally acknowledged nomenclature for geological classification of rock and minerals shall be used where possible, as summarized within the Elsevier's mineral and rock types chart (Lof, P., 1983). When local names of aggregates are in common use, this should also be given in brackets. Annex 4 gives suggestions for names used by this RILEM petrographic method.

6. SAMPLING AND LABORATORY PREPARATION

6.1 Sampling

Sampling should be carried out in accordance with the procedures described in prEN 932-1, ASTM D75 or similar standards. The aggregate sample shall be taken from processed material and shall be representative of the aggregates to be used in the concrete.

<u>NOTE</u>: Care must be taken to ensure that sampling is representative. For sampling in undeveloped or nonproducing quarries, where processed materials are not available, rock samples should be taken in such a way that they are representative. Samples should be taken from each lithological strata (not less than 25 kg for each stratum), or by means of cores drilled through the entire depth of material expected to be exploited. Additional samples may be required when the rock facies exhibit variations within the face of the quarry.

The minimum size of samples is given in Table 1 and Annex 2.

Table 1 - Minimum size of sample for dispatch to			
laboratory			
Maximum particle	Minimum mass to be received		
size (mm)	in the laboratory (kg)		
50	200		
40	100		
20	15		
10	5		
4	1		

6.2. Qualitative examination of laboratory sample

Examine the laboratory sample to ascertain the general characteristics of the aggregates. Distinguish between natural sand, coarse gravel and crushed rock, and record

these details. Where aggregates have been produced in part by crushing of a coarse gravel (or boulder deposits), or where aggregates, especially fine aggregates, have been produced by blending natural and manufactured rock materials, state that the aggregate type is "blended". The apparent presence of potentially deleterious (other than potentially alkali-reactive) constituents should be recorded, such as: mica, organic matter (coal, wood), fine material (*e.g.* silt, clay), particle coatings, encrustation and sulfides.

6.3 Splitting and sieving

Reduce the laboratory sample to a suitable test portion by quartering or preferably by the use of a sample divider and sieving. The order of splitting and sieving depends on the mass of the sample and the fractions to be examined.

<u>NOTE 1</u>: Where required, the sample may be washed and dried before sieving. Note that washing might remove fine and low-density reactive materials. Similarly it may be necessary to wash out any adhering dust from the sample. Washing should take place over a 0.125 mm sieve. Where required, a representative sample of <0.125 mm fraction should be kept for further analysis.

<u>NOTE 2</u>: The size of the required test portion varies according to the nature of the aggregate, its composition and the stipulated precision. An estimation can be made using Annex 2 which shows the relation between number of particles and mass in several size fractions. Annex 3 shows a chart for the relation between number of particles (or points counted), the proportion of constituent under analysis (*e.g.* reactive particles) and the absolute and relative errors within 95% confidence limits.

<u>NOTE 3</u>: All-in samples are to be divided into two separate test portions, a fine aggregate (equal to or smaller than 4 mm) and a coarse aggregate (greater than 4 mm).

<u>NOTE 4</u>: Sieves should be carefully cleaned before use to remove contaminants of constituents from previously sieved samples.

7. PETROGRAPHIC ANALYSIS

7.1 Assumptions

7.1.1 Overall considerations

The RILEM petrographic method using either or both techniques described in section 3 is based on the following assumptions:

• Basic information on alkali-reactivity of various rocks and minerals, as well as petrographic descriptions of such materials that are known and available to the investigator.

• Alkali-reactive constituents and minerals are visible either by the unaided eye or stereoscopic microscope or thin-section examination.

• The list and acceptance of alkali-reactive rocks and minerals, limit values and pessimum relations may vary between countries. Therefore, guidance shall be obtained from national practices and recommendations where available.

Annex 7 gives guidance on how to obtain knowledge on AAR and reactivity of aggregates when not already established.

7.1.2 Alkali-reactive constituents which are amorphous or cryptocrystalline

In the case of some reactive volcanic rocks, chert (flint) and certain limestones the reactive constituents are amorphous or cryptocrystalline. In such cases thin-section examination may not be sufficient for qualitative and quantitative analyses and should be usefully supplemented by other test methods.

<u>NOTE 1</u>: For aggregates containing porous flint, with amorphous constituents and/or cryptocrystalline quartz, it has been found that the more reactive porous particles can be identified by increased fluorescence and can therefore be quantified by thin-section examination and point-counting.

<u>NOTE 2</u>: Annex 6 includes short descriptions of some of the more common supplementary analysis methods for the assessment of alkali-reactivity.

7.2 General method

The RILEM petrographic method is carried out by two mutually beneficial techniques; first a standard petrographic examination of the aggregate particles followed by the use of a detailed microscopical examination of thin-sections which may incorporate point-counting. The first technique is used where alkali-reactive rock/mineral constituents are easily identified by the unaided eye or stereoscopic microscope or where it is relatively easy to group aggregate constituents on the basis of mineralogy, colour, shape or texture. In such circumstances it may not be necessary to study thin-sections of selected aggregates to enable fuller identification of the material. When alkali-reactive rock/mineral constituents cannot be identified with complete confidence, then the second technique should always be employed. (See flow diagram Annex A.1.1.).

7.3 Procedure for aggregate particle separation technique

The petrographic examination should be carried out on the dominant sized fraction of the fine aggregate and the coarse aggregate respectively. The minimum number of particles to be examined and counted depends on the percentage of rock/minerals of interest and the confidence limits required set down within clause 7.3.2. This method can be best used for coarse gravel, all-in and crushed rock aggregates.

<u>NOTE 1</u>: Where the aggregate sample contains rock/mineral with varying densities and hardnesses, separation and differences in constituents between various size fractions may occur. In such samples, more than one size fraction should be examined.

<u>NOTE 2</u>: Where there is doubt over the identification of a particular rock/mineral type it is recommended that thin-sections of representative particles are to be prepared to assist in the identification (*e.g.* dense and fine grained rock types).

<u>NOTE 3</u>: For the classification nomenclature of aggregates, see Annex 4.

Examination of aggregate particles should be carried out by use of hand lens or stereoscopic microscope and in some samples by scratching for hardness or the use of diluted hydrochloric acid for identifying carbonate minerals. Some particular aggregate staining methods are useful for identification of certain constituents (see Annex 6.4). Aggregate particles from each size fraction are divided into individual rock/mineral groups by hand sorting. As a minimum requirement, this grouping must include one group designated as innocuous aggregates and one group designated as potentially reactive (uncertain) aggregates. Aggregate particles may be divided into more specified groups of constituents based on an assessment of grain form, colour, texture rock/mineral type, and/or classification of the aggregates. In samples where the reactivity of certain aggregates is difficult to assess, a group designated, as potentially reactive (uncertain) aggregates should be included.

7.3.1 Counting and calculation of results for initial particle separation technique

Aggregate particles, in each group, are counted and their percentages are calculated.

<u>NOTE 1</u>: When more than one size fraction is counted, the average of all the counted fractions shall be calculated. More precise results are obtained by correlating each size fraction with the mass % of the fraction.

<u>NOTE 2</u>: Alternatively, results can be given as mass percentage by weighing each aggregate group and relating these to the total size fraction.

7.3.2 Precision of particle counts

The minimum number of particles to be examined and counted depends on the percentage of rock/minerals of interest and the confidence limits required.

<u>NOTE</u>: Annex 2 gives the relationship between masses and number of particles in the size fractions. Annex 3 shows a chart for estimating errors of counted particles of interest versus total counted particles valid for the 95 % confidence interval.

7.4 Technique for point-counting of thinsection samples

Where the particle separation technique cannot be easily undertaken or requires further clarification owing to a complex composition range or indeterminable constituents, the pointcounting of thin-sections shall then be employed.

7.4.1 Crushing of samples

For coarse aggregate intended to be examined by the pointcounting method in a thin-section, crushing can be used to establish the amounts of specific potentially reactive constituents within the whole aggregate. However, this crushing procedure is not mandatory. With this technique a representative sample has to be selected and the whole sample has to be crushed. Crushing shall be applied in steps to avoid the production of flaky particles and unnecessary disintegration of constituents. Sieve the crushed sample and use the 2-4 mm fraction for preparation of thin sections.

<u>NOTE 1</u>: In some circumstances the crushing of a coarse aggregate prior to examination has been found to improve

the identification of the various constituents or forms of particular minerals within an aggregate particle. For example where determination of the porosity of chert particles within a coarse aggregate is required.

<u>NOTE 2</u>: For aggregate samples containing particles of widely different hardness, crushing may produce and accumulate certain rock and/or mineral constituents in the 2-4 mm fraction relative to uncrushed material. For such samples, an additional thin- section including materials from the <2 mm fraction shall be prepared and point-counted. In samples where representative test portions cannot be obtained, the point-counting method is not recommended.

7.4.2 Procedure for determination

A representative test portion of each size fraction is to be prepared by quartering or dividing. The test portion shall be cast with epoxy resin to provide the specimen(s) for preparation of the thin-sections. Table 2 gives guidance on preparation of thin-sections with size 50 x 30 mm for the different crushed size fractions.

Table 2 - Combination of thin-sections to be prepared with					
certain size fractions					
Size	Minimum	Suggested number of 50 x 30 mm thin-sections			
fraction	number of	Fine Fine Coarse			
mm	particles	aggregates	aggregates *	aggregates	
2-4	300	2	2	2	
1-2	800		1		
<2	1500	1		(1) **	

* Generally for slowly expansive aggregates (crystalline)

** In case crushing has accumulated certain rock/minerals compared to the uncrushed material

<u>NOTE 1</u>: Thin-sections should where possible be prepared by vacuum impregnation with an epoxy resin containing a fluorescent dye. The porosity of constituents may then be identified under the petrographic microscope by fluorescence.

<u>NOTE 2</u>: To obtain an adequate number of particles in a 50 x 30 mm thin-section it is recommended that more than one thin-section is used. Alternatively a larger thin-section may be used. The number of analyses required is therefore dependent on the particle size of the constituent to be detected and the accuracy/reliability of the result (see Annex 3).

<u>NOTE 3</u>: Depending on the materials used the number of particles in such a thin section is about 1500 for the <2 mm fraction, about 800 for the 1-2 mm fraction and about 150 for the 2-4 mm fraction. If other thin section sizes are used then the total number of particles should be not less than those suggested above in Table 2 for each particle size.

7.4.3 Point-counting

The point-counting is carried out by traverses in regular increments in two directions to form a virtual orthogonal grid. It is important that point-counting covers the whole thinsection. During the point-counting, the operator must identify and group all rocks and minerals located under the cross hairs at each point on the grid.

<u>NOTE 1</u>: A minimum of 1000 points (excluding points falling on to resin) should be counted for the <2 mm, 1-2 mm, or 2-4 mm fractions respectively.

<u>NOTE 2</u>: The number of points may significantly exceed the number of particles, as several points may be counted across some larger particles.

Point-counting aims to identify and quantify both the reactive and the non-reactive rock and mineral particulate constituents present within an aggregate so that the content of reactive constituents can be determined. All cross points placed within one particle *e.g.* a sandstone should therefore be recorded as sandstone. When particles consist of more than one type of rock *e.g.* sandstone with quartz vein the cross point falling on to the sandstone should be recorded as sandstone, and cross points falling on to quartz vein should be recorded as quartz-vein material.

7.4.4 Calculation of results

The numbers of points counted for each rock/mineral group are divided by the total number of points counted excluding points within the resin. The average percentage is calculated and results from each rock/mineral group are given as volume percentage rounded to whole numbers (integers). As a minimum requirement, results are to be given as volume percentages of potentially reactive (uncertain) and non-reactive constituents.

<u>NOTE</u>: Volume percentage compositions of aggregates maybe taken as equivalent to mass percentages where the densities of the various constituents are reasonably similar.

7.4.5 Precision

The accuracy and precision of the point-counting method depend on several parameters. These include the representative nature of the test portion, the ability of the petrographer to obtain the correct classification and grouping of rocks and minerals, the number of particles in the thin-sections, the number of points counted, and the proportion of constituents

of interest within the sample. Annex 3 includes a chart for estimating the statistical precision of the point-counting method.

8. CLASSIFICATION

The procedure described in Section 7 results in a quantitative petrographic analysis for the sample under investigation, whereby each particulate constituent has been petrologically (or mineralogically) identified, its relative proportion determined and its alkali-reactivity status (innocuous or uncertain) established. According to the objective set out in Section 1, this information can now be used to classify the aggregate sample, for the purposes of the AAR assessment, into one of the following categories (see Flow Chart in Annex A.1.1):

- Very unlikely to be alkali-reactive Class I
- · Alkali-reactivity uncertain Class II
- Very likely to be alkali-reactive Class III

Class II and Class III aggregate might when appropriate further be grouped into alkali-silica reactive aggregates, alkalireactive carbonate aggregates or a combination of both. Acceptance and experience with reactive constituents differ between countries, and thus, final assessment and classification should where possible follow national or regional experiences, recommendations and specifications.

When available, use local guidance and/or experience to assist with this classification.

9. REPORT

The report shall state that the petrographic examination was carried out in accordance with this

RILEM method. Any deviation of procedure from the method shall be reported. State clearly in the report that the results and assessment relate specifically to the samples submitted and examined. Every effort should be made to ensure that persons without of petrographic experience examination methods can easily understand the conclusions of the report. Reference can be made to classification and interpretation sources such as those presented in Annex 1.

The following information shall be included in the report.

- Name and address of the petrographer's laboratory.
- Identification number on the test report.
- Name and address of the organization and/or person who ordered the test.
- Location of the sample (name of quarry or any other related information *e.g.* map grid references).
- Method of sampling (date and person responsible) or date of delivery to the laboratory.
- Amounts of sample received at the testing laboratory.
- Type of aggregate and size fraction received (*e.g.* natural sand, gravel, and crushed coarse rock).
- Observations from the initial examination, if relevant.
- Examination technique or techniques employed.
- Number and types of fractions examined and number of particles/points counted.
- Major rock and mineral composition identified.
- Results shall be given as average relative constituent proportions of the whole material. Also being shown if appropriate as: volume percent or mass percent. Other ways of calculations of results shall be given.
- Estimated errors.
- The reactivity status of each constituent.
- The reactivity class (and sub-class when appropriate) for the aggregate sample.
- Explanation for allocation of the reactivity class of the aggregate sample.
- References *e.g.* recommendations, specifications and literature from which guidance was obtained.
- Name of petrographer, signature and date of report.

Annex A.1.1 - Flow chart for petrographical examination of aggregates for the assessment of AAR potential



In cases of uncertainty, carry out this classification using the following definitions:

Class I aggregate samples contain no detectable known reactive rock types and reactive siliceous constituents according to Tables A.1.2 and A.1.3.

Class II aggregate samples cannot be unequivocally classified as either Class I or Class III.

Class III aggregate samples contain detectable reactive rock types and reactive siliceous constituents according to the Tables A.1.2 and A.1.3 in an amount assessed to cause damage in concrete.

<u>NOTE</u>: The critical limit value varies widely according to each reactive rock type, reactive siliceous constituent and local conditions and construction circumstances.

In the case of Classes II and III aggregate samples, additionally sub-classify the material according to the

siliceous and/or carbonate nature of the potentially reactive constituents, using the following definitions:

Classes II-S & III-S aggregate samples contain particulate constituents judged to be associated with alkali-silica reactivity (ASR).

Classes II-C & III-C aggregate samples contain

particulate constituents judged to be associated with alkali-reactive carbonates (ACR).

Classes II-SC & III-SC aggregate samples contain particulate constituents judged to be variously or both associated with alkali-silica reactive (ASR) and alkalireactive carbonates (ACR).

Table A.1.2 - Rock types, definitions and potentially alkali-reactive mineral constituents and reported reactivity in countries				
Rock Type (similar/associated rock types)	Definition	Potentially alkali-silica reactive mineral constituents	Country	
Andesite	Fine grained volcanic rock of intermediate composition	Glass or devitrified glass, cristobalite and tridymite. Opaline or chalcedonic veination or vugh-fillings	RA, CDN, IS, J, NZ, RO, CIS, USA, ADN	
Argillite (slate/claystone, phyllite)	Diagenetic sedimentary rock or weakly metamorphosed argillaceous rock with or without cleavage	Microcrystalline or cryptocrystalline quartz	CDN, J, N, SA, USA	
Basalt (dolerite)	Fine-grained basic volcanic rock	Glass or devitrified glass. Opaline or chalcedonic veination or vugh-fillings	RA, AUS, IS, SA, ADN	
Chert (<i>flint</i>)	Sedimentary rock occurring in or derived from chalk. Flint is chert of Cretaceous Age.	Chalcedonic silica and microcrystalline or cryptocrystaline quartz. Some varieties may contain opaline silica	B, CDN, DK, F, D, I, J, NL, CIS, GB	
Dolomite	Carbonate sedimentary rock containing more mineral dolomite than calcite	Finely disseminated quartz, opal or chalcedony. Fine dolomite crystals (might cause ACR)	A, CDN, CH, USA	
Gneiss (granulite, schist)	High-grade metamorphic rock exhibiting banding	Highly-strained quartz and/or poorly crystalline boundaries between quartz grains. Microcrystalline or cryptocrystalline quartz. Opaline or chalcedonic veins	CDN, IND, I, SA, USA	
Granite (granodiorite, quartz-diorite)	Coarse to medium grained acid igneous rock	Highly-strained quartz or microcrystalline quartz. Opaline or chalcedonic veination or vugh-fillings	RA, AUS, CDN, HK, IND, I, RO, SA, GB	
Greywacke (gritstone)	Detrital non-calcareous sedimentary rock character- ristically containing rock fragments and mineral grains in a very fine-grained matrix	Microcrystalline or cryptocrystalline quartz, especially in some meta-greywacke materials Not all greywackes are alkali-reactive	CDN, D, J, N, SA, GB	
Hornfels	Fine-grained, thermally metamorphosed rock	Microcrystalline or cryptocrystalline quartz	J, N, SA	
Limestone (marl, chalk)	Carbonate sedimentary rock containing calcite as the dominant mineral. Marl has a high clay content	Finely disseminated quartz, opal, or chalcedony Associated 'Chert' or 'Flint'	B, CDN, CY, F, I, NL, N, RO, CIS, TR, GB, USA	
Mylonite (cataclasite, breccia)	Dynamic metamorphosed rock with crushed and reduced grain sizes of quartz (due to cataclasis). Mylonite has foliated texture. Cataclasite and breccia have massive textures.	Strained, recrystallized quartz, cryptocrystalline and microcrystalline quartz, glassy material.	N, S, SA, USA	
Quartzite	Sedimentary <i>(orthoquartzite)</i> or metamorphic <i>(metaquartzite)</i> rock consisting predominantly of quartz	Highly-strained quartz and/or poorly crystalline boundaries between quartz grains. Microcrystalline or cryptocrystalline quartz	RA, AUS, CDN, IND, I, N, SA, GB, USA	
Rhyolite (dacite, porphyry)	Fine-grained to glassy acid volcanic rock	Glass or devitrified glass. Tridymite. Cristobalite. Opaline or chalcedonic veination or vugh-fillings. Microcrystalline or cryptocrystalline quartz	AUS, B, CDN, F, D, IS, J, CIS, NZ, N, S, TR, GB, USA	
Sandstone and siltstone	Detrital sedimentary rock. The grains are most commonly quartz, but fragments or grains of almost any types of rock or mineral are possible	Highly-strained quartz. Some types of rock cement notably opaline silica, chalcedonic silica, microcrystalline or cryptocrystalline quartz	RA, B, CDN, D, J, N, RO, CIS, SA	
Tuff (ignimbrite)	Detrital rock consisting of consolidated volcanic ash	Glass or devitrified glass. Cristobalite. Highly- strained quartz. Microcrystalline or cryptocrystalline quartz. Opaline or chalcedonic variation or variable fillings	RA, AUS, CDN, HK, J, TR	

Annex A.1.2 -Potentially alkalireactive rock types

Table A.1.2 gives rock types, definitions and potentially alkali-reactive mineral constituents and reported reactivity in countries.

Annex A.1.3 -Potentially alkalireactive siliceous constituents

Table A.1.3 gives potentially alkali-silica reactive mineral constituents. The tables are principally intended as guidance to the petrographer.

Notes on Tables A.1.2 and A.1.3:

Rock types which have been metamorphosed or altered in varying degree are often written with the prefix "**meta**" *e.g.* metasandstone, meta-rhyolite etc. Any rock types containing **sufficient alkali-reactive consti**-

tuents may be reactive *e.g.* opaline

silica and chalcedony veining of an inert rock aggregate source. Rocks containing some forms of strained quartz, and/or exhibiting poorly crystallised grain boundaries and/or microcrystalline or cryptocrystalline quartz between contiguous quartz grains, are sometimes found to be alkali-silica reactive. Undulatory extinction of quartz in rocks (when examined under a petrological microscope) suggests the presence of associated microcrystalline or cryptocrystalline quartz, but the measurement of undulatory extinction angles cannot be used to quantify the degree of reactivity and is not recommended.

Rocks containing significant proportions of **volcanic glass** of rhyolitic composition and/or **devitrified volcanic glass** have been found to be alkali-reactive in some parts of the world, including Japan and the USA. Not all **greywacke** rocks are alkali-reactive. Earlier suggestions that the phyllosilicate minerals (including the chlorite, vermiculite, mica and clay mineral groups) in greywacke and related argillitic rocks might participate in an 'alkali-silicate' reaction have not been confirmed by later work. Reactivity in greywacke seems to relate to the presence of extremely fine-grained quartz.

Certain types of **dolomitic limestone** (or calcitic dolomite) have been reported to be expansively reactive in North America, China and recently in Austria. The reaction patterns (*e.g.* alkali-silica gel is absent) differ from alkali-silica reaction and are therefore collectively termed 'alkali-carbonate' reactions. A number of factors have been associated with the development of deleterious levels of alkali carbonate reaction expansion. Deleterious carbonate rocks are usually impure argillaceous dolomitic limestone. Additional details on specific textural, compositional and crystal size criteria for potentially reactive dolomitic limestones may be found in the references given in appendix A.1.5.

Impure limestones or dolomites with sufficient contents of disseminated microcrystalline or cryptocrystalline quartz might prove to cause damage from alkali-silica reactivity.

In some other countries, highly altered or exceptionally weathered **feldspars** in aggregates have been considered to release additional alkalis within concrete.

Cristobalite is rare in natural aggregates on a world basis, but is common in volcanic rocks of younger geological ages in Japan, New

Table A.1.3 - Potentially alkali-reactive siliceous constituents				
Mineral Type Occurrence		Alkali-silica reactive components		
Chalcedony, Chalcedonic silica	Very fine-grained quartz with a distinctive fibrous microstructure	Chalcedony is reactive		
Cristobalite	Very high temperature form of silica (not quartz) occurring as a metastable constituent in some acid volcanic rocks and as a low temperature form in some sandstones, containing siliceous fossils and in some flints with a lower density than quartz.	Cristobalite is reactive		
Opal or Opaline silica	Essentially hydrated amorphous silica and disordered forms of cristobalite	Opaline silica is highly reactive.		
Quartz	The most common crystalline form of silica	Highly-strained quartz is possibly reactive. Some microcrystalline or cryptocrystalline forms are reactive.		
Volcanic Glass Non-crystalline phase within some volcanic rocks, may be 'devitrified' to very fine or incipient crystals.		Some volcanic glass (rhyolitic glass, hydrated rhyolitic glass) or devitrified volcanic glass is reactive. Tridymite or cristobalite might be present when devitrified		
High temperature form of silica occurring as a metastable constituent in some acid volcanic rocks and as a low temperature form in some sandstones, containing siliceous fossils and in some flints with a lower density than quartz.		Tridymite is reactive		
Artificial Glass	Alkali-silica-rich glass phases (e.g. Pyrex)	Disordered opaline phases		

Zealand and Iceland. An artificial aggregate, manufactured specifically for use in ASR experiments, largely comprises cristobalite formed by heating flint (CFC = calcined flint cristobalite). The presence of low temperature sedimentary derived forms of cristobalite and tridymite has also been reported in some localities in Germany. Some countries where examples of AAR involving particular rock types have been reported are indicated in the right-hand column. It is stressed that these are examples only and that the listing is incomplete. The following international (vehicle registration) codes have been used: PA = Accepting ALS = Acuttering A = Causting A = Ca

RA = Argentina, AUS = Australia, A = Austral *, B = Belgium, CDN = Canada, CH = China*, CY = Cyprus, DK = Denmark, F = France, D = Germany, HK = Honk Kong, IS = Iceland, IND = India, IRL = Ireland, I = Italy, J = Japan, NL = Netherlands, NZ = New Zealand, N = Norway, RO = Romania, CIS = Russia, SA = South Africa, S = Sweden *, TR = Turkey*, GB = United Kingdom, USA = United States, ADN = Yemen*.

* Information about reactivity of rock types not from RILEM ARP survey

Annex A.1.4 - References on alkali-reactive aggregates

International AAR Conference Proceedings

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• 2nd ICAAR, Reykjavik: Symposium on AAR, preventative measures, Icelandic Building Research Institute, Iceland, 1975.

• 3rd ICAAR, Wexham Springs, London: The effects of alkalis on the properties of concrete. Edited by A.B. Poole, 1976.

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Annex A.1.5 - References describing the characteristics of alkali-reactive carbonate aggregates

- Buck, A.D., 'Potential alkali reactivity of carbonate rock from six quarries', Miscellaneous paper No. 6-867, U.S. Army Engineer, Waterways Experimental station, Corps of Engineers, 1969.
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RILEM TC 191-ARP is currently developing guidance for the assessment and testing of carbonate aggregates. Further information for the petrographic assessment of carbonate rock material will be appended to a future edition of AAR-1. A RILEM test procedure that is specific to carbonate aggregates will be designated AAR-5.

Annex 2 - Relation between number of particles and weight for typical normal weight aggregates

Number	Fractions				
of	1-2 mm	2-4 mm	4-8 mm	8-16 mm	16-32 mm
particles	g	g	g	kg	kg
100	1	5	40	0.4	2.5
200	2	10	80	0.7	5.0
300	2	15	120	1.1	7.5
400	3	20	160	1.4	10.0
500	4	25	200	1.8	12.5
600	4	30	240	2.1	15.0
700	5	35	280	2.5	17.5
800	6	40	320	2.8	20.0
900	6	45	360	3.2	22.5
1000	7	50	400	3.5	25.0
1100	8	55	440	3.9	27.5
1200	8	60	480	4.2	30.0
1300	9	65	520	4.6	32.5
1400	10	70	560	4.9	35.0
1500	10	75	600	5.3	37.5
1600	11	80	640	5.6	40.0
1700	11	85	680	6.0	42.5
1800	12	90	720	6.3	45.0
1900	13	95	760	6.7	47.5
2000	13	100	800	7.0	50.0

Nielsen, B.K. and Thyregod, P., 'Statistical calculations on unreliability on samples of gravel', Environmental Ministry – Preservation Government Report on resource survey 2. (in Danish), IMSOR – Research Report No. 27/84 (1984).

Annex 3 - Absolute and relative errors -Method of estimating the certainty of proportions by point-counting method

Figs. 2A and 2B, taken from [1] (see below), enable approximate two-sided 95% confidence bounds to be estimated graphically for any percentage P > 0 and total counts (N) in the range 25 to 5000. This technique can therefore be used to provide improved confidence bounds on percentages or proportions and estimate the number of points required to ensure the percentages of all components are simultaneously within a stated accuracy.



Fig. 2A – Half-width of exact lower two-sided 95 percent confidence bound on observed percentage (abscissa) 0 percent of aconstituent and upper two-sided 95 percent confidence bound on $observed percentage <math>50 \le p < 100$ percent corresponding to a total of N counts (ordinate).



Fig. 2B – Half-width of exact upper two-sided 95 percent confidence bound on observed percentage (abscissa) 0 percent of a constituent and uppertwo-sided 95 percent confidence bound on observed $percentage <math>50 \le p < 100$ percent corresponding to a total of N counts (ordinate).

Reference

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Example: A petrographic analysis by hand sorting of 1000 particles of natural aggregates counted 125 particles classified as chert which is 12.5 % of the total sample. In Figs. 2A and 2B it can be read that 1000 particles (or number of points) with 12.5 % constituents under analysis (chert) gives 95% confidence limits of $\pm 2.2\%$ (here symmetrically) or a 95% confidence range of 10.3 % to 14.7 %.

Annex 4 - Suggestions on nomenclature

This Annex gives suggestions for naming and classifying aggregates. It has to be remembered that the petrographic analysis is normally carried out on small fragments and aggregate particles compared to field conditions where additional geological information on a larger scale is available. Generally, the smaller particles/fragments give less information about the origin and genesis for naming and classifying the aggregate. For particles in the fine sand fraction naming and classifying are often only based on mineralogical composition rather than texture and mineralogical composition.

The following nomenclature suggests the use of common geological rock and mineral names, here called the *preferred name*, which are accepted by most geologists around the world. However, because some fragments/particles cannot always be satisfactory classified the rock should be given a *mineralogical name* based on the mineralogical composition. In other cases it is more convenient to name the fragment/particles with a *genetic name*. Any special or local names, mineralogy, crystal/particle size as well as other relevant supplementary information should be given in brackets after the name of the aggregate.

Table A.4.1 - Metamorphic rocks				
Genetic name Mineralogical name		Preferred name	Examples: supplementary*	
Metamorphic rock	Quartzose rock	Quartzite	highly strained	
Metamorphic rock	Quartzose rock	Quartz schist		
Metamorphic rock	Quartzo-feldspathic rock	Charnokite		
Metamorphic rock	Quartzo-feldspathic rock	Gneiss	biotite	
Metamorphic rock	Quartzo-feldspathic rock	Schist	mica, chlorite	
Metamorphic rock	Mafic-feldspathic rock	Granulite		
Metamorphic rock	Argillaceous rock	Phyllite	quartz	
Metamorphic rock	Argillaceous rock	Slate		
Metamorphic rock	Mafic rock	Greenstone		
Metamorphic rock	Mafic rock	Greenschist		
Metamorphic rock	Mafic rock	Eclogite		
Metamorphic rock	Mafic rock	Amphibolite		
Metamorphic rock	Ultramafic rock	Serpentinite	altered	
Metamorphic rock	Calcareous rock	Marble		
Metamorphic rock	Calc-silicate rock	Calcsilicate		
Cataclastic rock		Mylonite		
Cataclastic rock		Cataclasite		
Cataclastic rock		Breccia		
Contact		Hornfels	Microcrystal-	
metamorphic rock			line quartz	

	Table A.4.2 - Igneous rocks				
Genetic name	name Mineralogical Preferred Examples:				
	(chemical) name name		supplementary*		
Plutonic rock	Quartzitic rock	Quartzolite			
Plutonic rock	Quartzo-feldspathic rock	Granite	alkali-feldspar		
Plutonic rock	Quartzo-feldspathic rock	Granodiorite			
Plutonic rock	Quartzo-feldspathic rock	Tonalite	trondhjemite		
Plutonic rock	Feldspathic rock	Syenite	quartz syenite		
Plutonic rock	Feldspathic rock	Monzonite			
Plutonic rock	Feldspathic rock	Anorthosite			
Plutonic rock	Feldspathic rock	Diorite			
Plutonic rock	Mafic rock	Gabbro			
Plutonic rock	Mafic rock	Norite			
Plutonic rock	Feldspathoidal rock	Foidolite			
Plutonic rock	Ultramafic rock	Pyroxenite			
Plutonic rock	Ultramafic rock	Peridotite			
Plutonic rock	Ultramafic rock	Dunite			
Plutonic rock	Mafic rock	Dolerite	diabase		
Plutonic rock	Quartzo-feldpathic rock	Pegmatite			
Pyroclastic rock		Tuff			
Volcanic rock	Acid volcanic rock	Felsite			
Volcanic rock	Acid volcanic rock	Rhyolite	porphyritic		
Volcanic rock	Acid volcanic rock	Dacite			
Volcanic rock	Intermediate volcanic rock	Andesite	trachyandesite		
Volcanic rock	Intermediate volcanic rock	Trachyte			
Volcanic rock	Intermediate volcanic rock	Calite			
Volcanic rock	Basic volcanic rock	Basalt	tholeiitic, diabase		
Volcanic rock	Basic volcanic rock	Phonolite			
Volcanic rock	Basic volcanic rock	Tephrite			
Volcanic rock	Ultrabasic volcanic rock	Foidite			
Volcanic rock	Basic to acid volcanic rock	Lava			
Volcanic rock	Acidic volcanic rock	Ignimbrite	welded tuff		
Volcanic rock	Basic volcanic rock	Scoria	intermediate		
Volcanic rock		Pumice	acidic to		
			intermediate		
Volcanic rock	Volcanic glass	Obsidian	acidic to		
	-		intermediate		

genetic names for the main rock groups: metamorphic, igneous, sedimentary and artificial. Minerals should be named by their mineralogical names eventually with supplementary information in a bracket.

<u>NOTE 1</u>: All the clastic rock name classifications can be further developed using the dominant constituents, grain size (coarse, medium, fine), and purity observations.

<u>NOTE 2</u>: Carbonate rock types are usually also further classified by means of the Folk (1959, 1962) and Dunham (1962) classification techniques.

Other: Altered aggregates, which cannot be identified, should be named **altered rock**.

For natural aggregates (gravels) containing different rock and mineral types it is convenient to have more than one rock type in a "counting group". This reduces the number of aggregate groups required during the counting procedure. However, reactive rock and mineral types should not be grouped but recorded individually. Mineral particles should be counted and named by mineralogical name *e.g.* quartz.

References on nomenclature

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- [10] Turner, F.J. and Verhoogen, J., 'Igneous and Metamorphic Petrology' (Mcgraw-Hill, New York, 1960).
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- [12] Winkler, H.G., 'Petrogene-

* when relevant, supplementary information to be given in brackets after the main name

Table A.4.3 - Sedimentary rocks				
Genetic name	Mineralogical name	Preferred name	Examples: Supplementary*	
Clastic rock		Conglomerate	monomictic, polymictic	
Clastic rock		Sandstone	lithic, glauconitic	
Clastic rock		Arenite		
Clastic rock		Siltstone	micaceous	
Clastic rock	Quartz sedimentary rock	Quartzite	ortho-, proto-	
Clastic rock	Quartz-feldspar sedimentary rock	Arkose	sericitic, sparagmite	
Clastic rock	Clay-feldspar sedimentary rock	Greywacke	dark green, volcanic,	
Clastic rock	Clay-quartz sedimentary rock	Quartzwacke	subgreywacke	
Clastic rock	Argillaceous sedimentary rock	Mudstone	claystone, marl,	
Clastic rock	Argillaceous sedimentary rock	Shale	sandy, calcareous	
Organic chemical rock	Carbonate sedimentary rock	Limestone	impure, silicified	
Organic chemical rock	Carbonate sedimentary rock	Calcareous tufa	travertine	
Organic chemical rock	Carbonate sedimentary rock	Dolomite	dolostone, argillaceous	
Organic chemical rock	Siliceous sedimentary rock	Chert	flint, chalcedony	
Organic chemical rock	Siliceous sedimentary rock	Opal	porous, fossilifereous	
Organic rock	Carbonaceous rock	Coal	lignite	
Chemical rock	Sulfate sedimentary rock	Gypsum	layered, nodular	
Chemical rock	Sulfate sedimentary rock	Anhydrite	evaporite	

* when relevant, supplementary information to be given in brackets after the main name

These tables list preferred names, mineralogical names and

Table A.4.4 - Artificial rocks					
Genetic	Examples:				
name	name		supplementary*		
Artificial rock	Slag	Blast-furnace slag	air-cooled, granulated		
Artificial rock	Slag	Steel slag			
Artificial rock	Glass	Glass	recycled, cullet,		
			foamed, Pyrex		
Artificial rock		Lightweight aggregate	Leca		
Artificial rock	Sintered fly ash	Lightweight aggregate	Lytag		

* when relevant, supplementary information to be given in brackets after the main

sis of metamorphic rocks' (Springer Verlag, Berlin, fifth edition, 1979).

Annex 5 - Some examples of petrographic descriptions of reactive aggregates (Class III)

A.5.1 Meta-rhyolite, Norway

<u>Texture</u>: The rock is green/red/dark grey, homogeneous, occasionally porphyric and laminated. Perlitic and spherolitic textures have been observed. Up to 4 mm large phenocrysts of feldspars and quartz are sometimes visible.

<u>Mineralogy</u>: The major constituents of rhyolite are matrix consisting of varying amounts of inter grown cryptomicrocrystalline feldspar, quartz and sericite.

Grain size¹: Average grain size of matrix is 13 - 20 microns.

<u>Reactive minerals</u>: Crypto-microcrystalline quartz (possible supplemented by alkali from neighbouring feldspar crystals). <u>Alterations</u>: Recrystallization of quartz, quartz veins.

Test methods: Thin section examination, XRD.

Concrete aggregate: Crushed rock and constituents in glacio-fluvial sediments.

<u>AAR</u>: Metamorphosed rhyolite has reacted in several concrete hydro power plants and dams in the Rjukan area (and other places in Norway).

A.5.2 Porous flint, Denmark

<u>Texture</u>: The colour of the rock varies from white to grey to brownish. Porous flint can be found as homogeneous particles and as particles, which consist of partly porous flint, and partly dense chalcedony flint (dense flint with a crust of porous flint).

<u>Mineralogy</u>: Porous flints consist of a matrix of porous opal or cryptocrystalline quartz with varying amounts of calcium carbonate (from 0 to approx. 45 weight %).

<u>Grain size</u>: from approx. 0.075 microns up to more than 100 microns. Average grain size in fine aggregate is 0.5 - 4 mm. <u>Reactive rock and minerals</u>: Opaline flint

<u>Test methods</u>: Petrographic examination of thin section using the Danish test method TI-B 52 and accelerated mortar bar test using the Danish test method TI-B51.

<u>Concrete aggregate</u>: Porous flint can be found in glacial till and other glacial-fluvial sediments.

<u>AAR</u>: Porous flint (found mainly in the sand fraction) has reacted in several concrete structures, such as bridges, swimming pools, car parks, balconies and marine structures, etc.

A.5.3 Silicified limestone, Belgium

<u>Texture:</u> the rock is a fine dark-grey argillaceous limestone; fossil debris are often visible. Sometimes, we observe dark cherts.

<u>Mineralogy</u>: It is a carbonated micrite or biomicrite containing many debris of fossils such as: numerous bryozoans (fenestellae), echinoïds (crinoïds, sea-urchins), brachiopods, trilobites, ostracods, some corals debris (syringopora) and seldom

foraminiferae. The limestone often contains thin clay layers with large amounts of organic matter; it is sometimes dolomitized. We observe a lot of silicification and often the silica partially replaces the calcite in the fossils.

<u>Reactive minerals</u>: Crypto-microcrystalline quartz showing sometimes a fibrous habit like chalcedony.

<u>Test methods</u>: An experienced geologist can recognize this type of limestone directly by petrography. The reactivity can be confirmed by expansion tests (with petrography control on expanded concrete or mortar).

<u>Concrete aggregate:</u> used for concrete purpose as crushed rock produced in quarries.

<u>AAR</u>: Silicified limestone has reacted in several concrete structures such as bridges and fluvial structures.

<u>NOTE</u>. Similar rock types are found in Eastern Canada where they have caused premature distress of different types of structures.

A.5.4 Greywacke/ Siltstone, United Kingdom

<u>Texture</u>: Dark grey to black coloured fairly homogeneous sedimentary rock, with well developed cleavage and lamination developed in many particles. Microcrystalline quartz bands are noted running preferentially with the inherent cleavage within the rock. Dense, low porosity material with occasional calcite veins.

<u>Mineralogy</u>: Fine grained material composed primarily of clastic quartz material within a matrix composed from lesser amounts of chlorite, microcrystalline quartz, muscovite, pyrite, calcite and undetermined clay minerals (sericite). There is some evidence for small amounts of organic material also being present. Rock has undergone low 'Chlorite grade' metamorphism. Cementing agents appear to be primarily silica and a lesser amount of carbonate.

<u>Grain size</u>: ranges from 1.15 mm down to 0.01 mm, with an average around 0.025 mm. Modest degree of variation between particles due to the rocks apparent fine grained turbidite origin.

<u>Reactive minerals</u>: Crypto-microcrystalline quartz found within siliceous bands and to a lesser degree within the fine grained matrix.

Test method: Petrographic examination of thin sections, SEM and XRD.

<u>Concrete aggregate</u>: This aggregate source is now rarely, if ever used in concrete as reports of its highly reactive nature are nationally well documented.

<u>AAR</u>: This aggregate has been identified as responsible for a number of moderate to severe cases of alkali-silica reaction within a number of structures in the north of England, *e.g.* Bridges and concrete road pavements (reference Sibbick and West 1992).

¹ In this circumstance and those below the grain size refers to grains within the aggregate's overall lithology.

A.5.5 Quartzite, France

<u>Texture</u>: this sedimentary rock is a white yellowish quartzite with a saccharoidal aspect.

<u>Mineralogy</u>: the rock is mainly composed of quartz grains. Their contours are irregular and show sometimes micrograins of quartz. Muscovite minerals are often observed, sometimes as a cementing agent.

<u>Grain size</u>: the grain size is varying from approximately 50 up to 200 microns.

Reactive minerals : intergranular microquartz.

<u>Test methods</u>: several expansion tests and one chemical test according to the French standards (P 18-585, P 18-587, P18-588, P 18-589 and P 18-590).

Concrete aggregate: used as sand and gravel.

A.5.6 Andesite, Japan

<u>Texture</u>: In Japan, crushed andesites from younger strata represent the major reactive aggregate, while dacite and rhyolite in the local sand-gravel deposits may also be reactive. These rocks range from dark- (glassy) to light-grey (crystalline or devitrified), from aphyric (*e.g.* sanukitoid, a glassy bronzite andesite) to porphyritic, and from fresh (unaltered zone) to moderately altered (smectite zone) in texture.

<u>Mineralogy</u>: Well-crystallized andesite and dacite contain interstitial silica minerals, such as cristobalite (rounded patches), tridymite (platy aggregation) and/or quartz (mosaic), in addition to the phenocrysts of feldspars and pyroxenes with or without hornblende. Glassy volcanic rocks rich in rhyolitic interstitial glass are scant in the silica minerals, while devitrified dacite and rhyolite consist mainly of cryptocrystalline quartz.

<u>Reactive minerals</u>: Cristobalite and tridymite have small pessimum proportions, while rhyolitic glass does not. In the large quarries and gravel deposits, a wide variety of glassy to well-crystallized andesites occur, which present intermediate pessimum proportions. Cryptocrystalline quartz in the devitrified rocks is less reactive, but may be slowly expansive in some conditions.

<u>Alterations</u>: Reactive andesite include: 1) unaltered rocks containing cristobalite, tridymite and/or rhyolitic glass, and 2) moderately altered rocks (smectite zone) in which smectite has replaced glass and mafic minerals. Older rocks (chlorite zone) in Japan are generally inert due to recrystallization of reactive minerals during diagenesis.

<u>Test methods</u>: Chemical test for screening, mortar bar test (JIS A 5308) for confirmation, but the latter misleads when the aggregate has a small pessimum proportion (it only specifies a mix of 100% aggregate). Petrography, XRD and phosphoric acid method to check cristobalite and tridymite. Accelerated concrete core expansion test (80°C, 1N NaOH: 5cm diameter, length 13 cm) confirms ASR potential of sand-gravel aggregates in the structure.

<u>Concrete aggregate</u>: Highly reactive glassy bronzite andesite (sanukitoid) is not used now, but crushed stone of common pyroxene andesite is popular, and sand-gravel deposits containing andesite, dacite and rhyolite, are used locally.

<u>AAR</u>: Crushed andesite and gravels of andesite, dacite and rhyolite, produced extensive damage due to ASR in bridges, viaducts, culverts, sea walls, dams, debris barriers, retaining walls, pavements, tunnel linings, water-tanks, buildings, precast armour blocks, kerbs, etc.

A.5.7 Chert, Japan

<u>Texture</u>: Grey to white, occasionally red or black, homogeneous sedimentary rock with a vitreous luster. Chert consists of non-clastic, fine grained biogenic siliceous materials as radiolarian fossils or sponge spicules, and may grade through radiolarian shale into black mudstone. Derived from Mesozoic and Paleozoic strata.

<u>Mineralogy</u>: Siliceous materials consists mainly of crypto- to microcrystalline quartz with lesser amounts of chalcedony and vein quartz, with or without illite, chlorite, calcite, pyrite, iron oxides and organic materials as a pelitic impurity.

<u>Reactive minerals</u>: Cryptocrystalline quartz, microcrystalline quartz and chalcedony. The mixture of these silica minerals in the chert causes slower expansion with larger pessimum proportions than reactive andesites in Japan.

<u>Test method</u>: Thin section petrography, SEM observation, and determination of the crystallinity index of quartz based on XRD. Chemical test for screening, mortar bar test for confirmation, while the latter is not always sensitive to the gravel containing chert.

<u>Concrete aggregate</u>: Crushed stone and local sand-gravel deposits <u>AAR</u>: Generally slowly-expansive, but may be accelerated by steam curing and the use of de-icing salt. Damage to bridges, viaducts, retaining walls, debris barriers, buildings, as well as precast armour blocks, pedestrian slabs, kerbs, etc.

Annex 6 - Supplementary methods

A.6.1 Hydrochloric acid method

The method is used to disintegrate carbonate-rich aggregate samples for further analysis. Some carbonate rocks are alkalisilica-reactive; the reactive silica is usually disseminated in the cryptocrystalline mass of the matrix and not easily visible in thin section under the microscope. The hydrochloric acid method allows a determination of the acid-insoluble residue from the carbonate rocks. Optical microscopy, X-ray diffraction or chemical analysis can be used to identify the types and amounts of impurities such as argillaceous material, chert and opal. Other deleterious constituents such as sulfates and sulfides may also be assessed.

The procedures are as the following: 20% hydrochloric acid solution is added to 5 g representative sample from the carbonate rock. However, several samples should be used to improve the precision. To speed up the solubility of the carbonates, the sample is heated to 80°C and the acid added in small amounts until the carbonates are completely dissolved. After the reaction has ceased, the solution is filtered and the residues washed with distilled water. Sulfate impurities are removed by precipitation with barium chloride. Precise determination of the nature and amounts of non-carbonate impurities may be obtained using X-ray diffraction, scanning electron microscopy and chemical analysis.

The method is not recommended for determination of aggregates suspected of being alkali-carbonate reactive. This is because the reactive components are dolomite and not siliceous components.

Reference

[1] Dolar-Mantuani, D., 'Handbook of Concrete Aggregates, a Petrographic and Technological Evaluation', Noyes Publications, New Jersey, USA, 1983.

A.6.2 Phosphoric acid method

The method extracts silica minerals (cristobalite, tridymite and quartz) from the rock and dissolves other silicates, carbonates and oxides. It has been used in the environmental hygiene and cement industry in Japan, but is of great use in detecting reactive silica minerals in concrete aggregates.

The procedures are as follows: 25 ml of phosphoric acid solution is added to the representative 0.3 g of powdered rock sample (sieved <75 micrometer) in a conical beaker. Place the beaker (300 ml) on an electric heater (300W, Ni-Cr wire) so that boiling starts after 90 seconds, adjusting the voltage (near 80V). After 12 minutes of boiling, pour a hot water (60-70°C, 125 ml) and shake the beaker vigorously to dissolve syrup-like phosphoric acid.

Then, add a 10 ml of fluoboric acid solution (10% HBF₄) and shake vigorously, and add 10 ml of paper pulp suspension. Wash the inside of beaker (with 25 ml of water) and stand it for 1 hour. The solution is filtered, and the residue is washed with a diluted hot hydrochloric acid solution (1+9), then washed with hot water repeatedly to remove phosphoric acid before ignition. It is desirable to compare the content of the residue with the boiling of 14 minutes.

For the petrographic purpose, the residue should be ignited at low temperatures (600-800°C) and subjected to quantitative XRD analysis to determine the ratio of cristobalite, tridymite and quartz in the volcanic rocks. For sedimentary rocks, particularly argillaceous carbonate rocks, SEM observation is made to confirm the presence of reactive crypto- to microcrystalline quartz in the fine-grained matrix which is obscure in thin section. Examples of volcanic rocks from Iceland, New Zealand and Japan, and of carbonate rocks, are given by Katayama *et al.* [3].

References

- JCAS I-31-1996, 'Quantitative determination of quartz (phosphoric acid method)', Japan Cement Association Standard (in Japanese), 1996.
- [2] Katayama, T., Helgason, T.S. and Olafsson, H., 'Petrography and alkali-reactivity of some volcanic aggregates from Iceland', Proc.10th ICAAR, Melbourne, Australia, 1996, p. 377.
- [3] Katayama, T., Ochiai, M. and Kondo, H., 'Alkali-reactivity of some Japanese carbonate rocks based on standard tests', Proc.10th ICAAR, Melbourne, Australia, 1996, p. 294.
- [4] Katayama, T., St. John, D.A. and Futagawa, T., 'The petrographic comparison of some volcanic rocks from Japan and New Zealand – Potential reactivity related to interstitial glass and silica minerals', Proc.8th ICAAR, Kyoto, Japan, 1989, p. 537.
- [5] Sulcek, Z. and Povondra, P., 'Methods of decomposition in inorganic analysis' (CRC press inc., USA, 1992).

A.6.3 X-ray diffraction analysis (XRD)

XRD analysis is used for identification of crystalline materials. The principle is that powders of crushed crystals are exposed for monochromatic radiation with known wavelength. The X-ray is reflected (diffracted) by the planes of atoms making up the crystal structure. Because each mineral has different crystal lattice (D) the reflected X-rays at a given wavelength (here fixed) will reflect at one angle of incidence only. The crystal lattice is unique for each mineral type and can be calculated from the recorded angle of incidence according to Bragg's Law. The mineral identification is done by use of a strip chart (diffractogram) or by help of a computer program with a library of known patterns. XRD analysis is normally used for

qualitative identification of minerals but can also be used for semi-quantitative measurement when compared with standards.

A method for evaluating Japanese reactive chert aggregates by use of a crystallization index has been suggested. The method is based on comparison and calculations of peak heights. Specialized laboratories normally carry out XRD analyses.

References

- [1] Battey, M.H., 'Mineralogy for students' (Longham Group Limited, London, 1975).
- [2] Bloss, F.D., 'Crystallography and Crystal Chemistry' (Mineralogical Society of America, USA, 1994).
- [3] Morino, K., 'Alkali-aggregate reactivity of cherty rock', Proc.8th Int. Conf. on AAR, Kyoto, Japan, 1989, p. 501.
- [4] Nishiyama, T. *et al.*, 'A few remarks on Alkali-Reactive Chert Aggregates', Proc. 8th Int. Conf. on AAR, Kyoto, Japan, 1989.

A.6.4 Staining methods

Rock samples may be treated with chemical solutions to distinguish between different types of feldspars, carbonates and some other minerals. One method, which has been used in Denmark to distinguish between porous flint and chalk, is staining with a blue dye (methylene blue). Because porous flint contains many capillary pores the blue dye is sucked into the porous flint but not into the chalk. For further information on staining methods see [1] (below).

Reference

 Dolar-Mantuani, D., 'Handbook of Concrete Aggregates. A Petrographic and Technological Evaluation' (Noyes Publications, New Jersey, USA, 1983).

A.6.5 Gel-pat test

The method is used for identification of alkali-silica gel developed by aggregates under test. The method is presently incorporated into BS 7943 (1999) as an appendix. Its use is deemed necessary where there exists a potential for small quantities of opal within an aggregate sample, which might not be detectable during a conventional petrographic examination. Slower reactive materials will not be detected by this method.

The technique allows for the production of visible ASR gel on the surface of aggregate particles immersed in alkaline solution whilst embedded in a cement paste disc (or 'pat'). During storage at a constant temperature of 20°C, the specimen is inspected periodically and the appearance of any gel growth, occurring on the aggregate is recorded. The development of gel growths within a specified period of time indicates the probable presence of opaline silica or other exceptionally reactive forms of silica.

The gel-pat test is intended to provide a semiquantitative means of detecting highly alkali-reactive siliceous materials, such as opaline silica in aggregates.

References

[1] BS 7943, 'Guide to The interpretation of petrographical examinations for alkali-silica reactivity', British Standards Institution, London, UK, Annex B (1999) 7-14.

[2] Fournier, B. and Bérubé, M.A., 'Recent application of a modified gel pat test to determine the potential alkali-silica reactivity of carbonate rock', *Cement and Concrete Composites* **15** (1993) 49-73.

A.6.6 Scanning electron microscopy

The electron microscope can be used for investigations of rocks and minerals, but is especially useful for determination of alkali-silica reaction in concrete. The principle involves high speed primary electron bombardment of a sample surface under vacuum. The reflected secondary electrons are determined by a detector and transformed to an electrical signal. The signal being dependent on the atomic number of analyzed material, density of the sample and the energy of primary electrons (fixed). To form an image of the sample, the electron beam is moved in a linear direction and scanning over the sample. By the use of a back scatter technique, the image appears with different grey tones where lighter areas consist of minerals with higher atomic numbers and darker areas with lower atomic numbers.

By analyzing the X-ray emitted from spots or smaller areas, analysis of elements can be determined by an energy dispersive detector (EDX). Electron microscopes can also be equipped with wave length dispersive detector systems (WDX) which often give more accurate results, especially for lighter elements, but may cause damage to cement hydrates and ASR gel, owing to the higher electric current applied (100 times that of EDX). These analytical SEMs are called EPMA.

The SEM system has been used extensively with silicified limestones in France to help determine the siliceous materials networks and morphology within such aggregate materials. Two different forms of siliceous material can be identified. The first form is recognised at the macroscopic level and consists of siliceous nodules (similar to chert). These have the texture and composition of chalcedony and/or opal and mainly consist of microcrystalline silica, commonly considered as having a high potential for alkali-silica reactivity. The second form of silica, called 'diagenetic silica' can only be observed at the microscopic level. In fact, after a slight acid attack of polished limestone sections (3 min in a HCl solution diluted to 1/200th), a fine silica 'grid' is visible under the SEM. This silica 'grid' cannot be observed under a conventional petrological microscope, since the thickness of the grid is between 4 and 10 µm.

These two types of siliceous material are present at different scales but are considered as the possible cause of the potential alkali-reactivity of certain limestones in France.

The above method is inappropriate for some clay-rich limestones, where the reactive silica is micro-quartz grains of less than 10 μ m size, which are embedded in the clay matrix. In this case, the phosphoric acid or a similar method may be applied (see A.6.2).

EPMA is also used in Japan to diagnose AAR in concrete, as well as to estimate the alkali content of the original cement used in the hardened concrete.

References

[1] Katayama, T., 'Petrographic diagnosis of alkali-aggregate reaction in concrete based on quantitative EPMA analysis',

Proc.4th CANMET/ACI/JCI Int. Conf. Recent Advances in Concrete Technology, Tokushima, Japan. ACI SP-179, 1998, p. 539.

- [2] Lawes, G., 'Scanning electron microscopy and X-ray Microanalysis' (John Wiley & Sons, London, UK, 1987).
- [3] Leroux, A., 'Les méthodes pétrographiques de l'étude de l'alcalis-réaction', *Bull. de l'Association Internationale de Géologie de l'Ingénieur* **44** (1991) 47-54.

A.6.7 Other useful references

- [1] Hewlett, P.C., 'Lea's Chemistry of Cement and Concrete', 4th edition (Arnold, London, UK, 1998).
- [2] Lawes, G., 'Scanning Electron Microscopy and X-Ray Microanalysis', (John Wiley & Sons, London, UK, 1987).
- [3] McConnell, D., Mielenz, R.C., Holland, W.Y. and Greene, K.T., 'Cement-aggregate reaction in concrete', *Journal of the American Concrete Institute* 19 (2) (1947) 93-128.
- [4] Neeb, P.R., 'Building raw materials, survey, investigation and use', Norwegian Geological Survey -Tapir, 1992 (in Norwegian).
- [5] Stark, A., 'Handbook for the identification of Alkali-Silica Reactivity in Highway Structures', SHRP Strategic Highway Research Program, National Research Council, Washington, D.C., USA, 1991.
- [6] Wigum, B.J., 'Alkali-aggregate reactions in concrete: properties, classification and testing of Norwegian cataclastic rocks', Doktor Ingeniöravhandling 1995:98, Institutt for Geologi og Bergteknikk, University of Trondheim, Norway, 1995, p. 227.

Annex 7 - Information on aggregate reactivity from existing concrete

Examination of existing concrete affected by AAR or containing the aggregate under investigation is the most reliable method identifying AAR and the reactivity of aggregates. Where the reaction is just beginning to develop, signs of the reaction may be difficult to observe and require experience to identify. RILEM TC 191-ARP is developing guidance for the diagnosis of AAR in hardened concrete and existing structures.

A.7.1 Examination of concrete structures

Map-cracking, signs of movement caused by volume expansion and deformation of concrete members may indicate that deleterious AAR is present. However, map cracking can occur with other processes *e.g.* drying shrinkage. Sometimes, the exudation of ASR gel can be observed on the surface of the concrete. In some cases, AAR can occur in the concrete without any sign of map cracking, expansion or deformation observed within the structure. Crack patterns can also be influenced by reinforcement.

To obtain information on the AAR and the reacted aggregates, it is necessary to drill cores from the structure and investigate the concrete under the microscope (microstructural analysis). It is recommended to drill a minimum of two cores from each structure. The cores should be long enough to permit concrete more than 5-10 cm beneath the original outer to be examined and to allow the dimensions of any overall crack pattern to be studied. Appropriate precautions should be taken during core sampling and subsequent sample preparation to ensure that evidence of AAR is retained [2] (Palmer, 1992).

A.7.2 Microstructural analysis

Microstructural analysis can be applied to polished half cores (sawn along the length axes) and thin-sections. It is advantageous to impregnate the concrete with epoxy containing fluorescent dye (*e.g.* Hudson Yellow) to highlight the microcracks and porosity. For the examination of polished half cores, a low power stereo microscope can be used. Examination with fluorescent light can be done using an UV-lamp.

Alkali-silica gel may occur as:

- Gel exudations on the surface of cores and on the sawn and polished surfaces of samples.
- Reaction rims around the aggregates on broken surfaces of samples.
- Gel fillings in air voids and cracks in the concrete (normally whitish colour in ordinary light)
- Gel impregnation of the cement paste around porous reacted aggregates and cracks filled with gel.
- Gel in cracks inside aggregates

Identification of reactive aggregates in concrete is indicated by the following:

- Gel situated at the margin of aggregates and reaction rims in the aggregates indicate that the aggregate has reacted in the concrete. Note that dark rims can be observed around some gravel particles that can be related to weathering and have nothing to do with AAR.
- The occurrence of reaction products located within the aggregate; indicates that the aggregate has reacted in the concrete.
- Cracks originating from within the aggregate and penetrating into the surrounding cement paste together with reaction product; indicate that the aggregate particle has reacted in the concrete.
- Gel impregnation of the cement paste around reacted aggregates (observed *e.g.* in porous flint and opal).

It should be noted that the occurrence of alkali-silica gel confirms the existence of an ASR in the concrete; it does not necessarily mean that the reaction causes or has caused any damage to the concrete. This can be evaluated by studying the crack pattern. Advice on this matter should be obtained from literature ([1, 2] below) or should be given by specialists within this field. Unless involved with the site investigation and the selection of core drilling locations, a petrographer should be careful not to reach unqualified conclusions from the laboratory study of concrete. Guidance on diagnosis and prognosis is being prepared by RILEM TC 191-ARP.

References

- Jensen, V., 'Alkali Aggregate Reaction in Southern Norway', Doctoral Thesis, University of Trondheim, Norway, 1993.
- [2] Palmer, D., 'The diagnosis of alkali-silica reaction, report of a working party', 2nd edition, British Cement Association, Slough (now Crowthorne), UK, 1992.

Annex 8 - Other applicable documents

• ASTM C 294, Standard descriptive nomenclature of constituents of natural mineral aggregates.

• ASTM C 295, Standard practice for petrographic examination of aggregates for concrete.

- ASTM D 75, Sampling aggregates.
- ASTM C 856, Standard practice for petrographic examination of hardened concrete.
- BS 812-102, Testing aggregates: Methods for sampling.

• BS 812-104, Testing aggregates: Method for qualitative and quantitative petrographic examination of aggregates.

• BS 7943, Guide to the interpretation of petrographical examinations for alkali-silica reactivity.

• ISO 3310, Parts 1 and 2: Test sieves - Technical requirements and testing.

• prEN 932-1, Test for general properties of aggregates: Part 1: method for sampling.

• prEN 932-2, Test for general properties of aggregates: Part 2: method for reducing laboratory samples to test portions.

• prEN 932-3, Test for general properties of aggregates: Part 3: method for description and petrography - simplified procedure.

• AAR-0, Outline Guide to the Use of RILEM Methods in Assessments of Aggregates for AAR potential, *Mater. Struct.* (in press).

• AAR-2, Alkali-aggregate reaction: A - TC 106-2 [now AAR-2] - Detection of potential alkali-reactivity of aggregates - the ultra-accelerated mortar-bar test, *Mater. Struct.* **33** (229) (2000) 283-289.

• AAR-3, Alkali-aggregate reaction: B - TC 106-3 [now AAR-3] - Detection of potential alkali-reactivity of aggregates - method for aggregate combinations using concrete prisms, *Mater. Struct.* **33** (229) (2000) 290-293.