



THE PETROGRAPHIC DIAGNOSIS OF POTENTIALLY DELETERIOUS AGGREGATES

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ABSTRACT

Specifications for aggregates make statements such as 'The aggregate shall contain no opal', or 'Chert shall not be present outside the range 2% to 60% of the aggregate'. Such statements imply that the specifier has taken into account the standard deviation of the determination - the user is not permitted to introduce error calculations in determining agreement with the percentage specified (e.g. see ASTM C 670). Standards for the description of such an aggregate (ASTM C-295 and BS 812 part 104) give instructions for calculating the amount of aggregate that must be tested. These calculations are based on the maximum aggregate size and the amount of constituent considered to be significant. The calculations of standard amounts to be analysed do not properly allow specifications to be met. In practice the formula given in the British Standard is incorrect for an amount as low as 2%. Proof that 'the aggregate contains no opal' is regarded as not feasible. The standards also fail to deal with many issues concerning the description of the aggregate even where the roughly 800 particles making up the 6 kilograms suggested in BS812 part 104 are identified. Even if the formulae are taken to be correct it would not be possible to argue that if enough particles were examined and correctly identified a given specification could be met. This is because the methods of examination recommended take no account of source variability or internal particle variability. Consideration must be given to the geology of the source material and methods must be devised for control of such considerations. Even if a standard aggregate test gives a result of 0% for a specified component it remains possible for that component to be present in the aggregate used. This paper elaborates reasons for these arguments and attempts to initiate discussion of possible resolutions of the problems.

Keywords: Alkali-reactive aggregates, recognition, specifying to avoid, rock names, thin section petrography, confidence limits, tolerance limits.

INTRODUCTION

Aggregate testing with respect to the potential for the occurrence of AAR has been described in diverse standards and various reviews. ASTM.C-295 provides a widely used and valued standard for aggregate description and interpretation when coupled with ASTM.C-294. BS812, Part 104 outlines another method for the description of aggregate when used along with various other standards. The Concrete Society (1999) has revised an earlier technical report describing specification to avoid AAR and the BRE Digest 330 (1997) also provides a review aiding the specifier in trying to eliminate the potential for reaction. At the time of writing, RILEM and British Standards for the petrographic evaluation of aggregate with respect to AAR (BS 7943) are about to appear. Putting these and other reviews together reveals important differences and shows that all must be treated with caution. In a similar way a great diversity of standard tests have been developed including Concrete Prism and Accelerated Mortar Bar Tests that, though similar, have major and minor differences.

The problems encountered include the following.

- How much aggregate should be tested in a representative sample?
- Where and when should thin section and other microscopic and special techniques be employed?
- How are rock types in aggregate to be named?
- How are rocks to be identified as potentially reactive?
- Must the rock fragment or sand grain be wholly of a given rock type, or is it to be mainly of that type?
- If lists of safe rocks, reactive rocks, rocks of low, high, or normal reactivity, are provided, what happens with respect to rocks not on the list?
- How much of a given rock is required before a given level of reaction is encountered?
- How is the distribution of the reactive component in the aggregate influencing the potential for reaction?
- How is a potentially damaging rock concentration to be specified and how it is to be avoided?
- How can a history of observation of a given aggregate be used to improve confidence in its acceptance or rejection?
- What is the basis for specifying that a given concentration of a particular component is acceptable or not acceptable?

We have carried out experiments on the determination of rock proportions in aggregate and evaluated the numerical relationships by computer modelling. Stock piles were made containing mixtures of easily recognised rocks in the 5 to 10 mm sieve size range. Mixtures of white dolomite, brown flint, and black gabbro up to a maximum of 35 % by mass in a host of red granite were made and sampled. For each mixture 10 samples were taken each of about 800 g - this being the amount indicated in BS812 part 104 as appropriate where it is wished to determine an amount of 20% with upper and lower relative bounds of 10% at the 95% confidence level. The upper and lower bounds in practice were found to be asymmetric - with the asymmetry increasing as the amounts became smaller. Finding nothing of a given component means that some small but significant amount can be present. The formula given in BS812 part 104 cannot be used where the amount sought is likely to be small or very large. It was also found that where the stockpile was made by deliberately mixing two different aggregate mixtures by pouring one upon the other as might happen in a stockpile. The results showed no indication of inhomogeneity but the mean

result was greatly in error. Details of all these complex tests and the computer modelling will be given elsewhere (Howarth and French in preparation).

PETROGRAPHY

Identification and Classification of Rocks

The analysis of an aggregate by hand picking or by point counting of the polished surfaces or thin sections requires identification of the rock types that are to be counted. In a mixed sand and gravel a number of lithologies might be identified. This number will not be absolute but will depend upon the knowledge and experience of the petrographer, on instructions received or agreed, and the purpose for which the petrography is carried out. The methods being used will also affect the way in which the rocks are named. Standards try to provide fresh classifications of rocks or introduce a short and usually inadequate glossary of rock names. A granite quarry might have variants including aplite and pegmatite, show various degrees and types of alteration, and various textures and structures. There will be rafts and xenoliths, and contrasting sheets and mineral veins. The analysis of crushed granite might therefore become very time consuming according to the number of rock types to be recognised. The rock name also conveys almost nothing bearing on the potential for reaction or instability and is regarded as unimportant for the diagnosis of potential problems in service. For the general classification of aggregates there is a need for a set of simple and generally applicable rock names which will be useful to the trade but this bears little relationship to the recognition of potentially deleterious features.

Conversely, there might be specifically reactive components in crushed rocks and in sands and gravel that might or might not be distinguished in the examination. For example, reactive chert occurs in Danish sands (Jensen et al 1957). Similar reactive components occur in some fine aggregate which is sea-dredged off the south coast of England. The reactive component is cryptocrystalline and has in some instances been described as opaline. Other cherts have none of this material. The reactive component is easily recognised by X-ray methods but some doubt must remain as to whether the distinction can be made reliably by the method of hand picking advocated for example in BS812, Part 104 for the general description of aggregates. Similarly, granite might have thin zones of cataclasite, tuffsite, siliceous veins, or strained quartz in some fragments. It might be that the hand specimen description of the rock would not effect a separation of rocks into those containing one or more of these features and those without special characteristics. In thin section analysis it would be possible to effect such separations, but the number of different materials to be recognised might become very high, so that the analysis would become very protracted with large sample sizes being required.

Lists of Rocks

Reviews of alkali-aggregate reactivity may provide lists of rocks which are regarded as potentially producing low levels of reactivity, while other lists indicate high levels of reactivity. Problems arise for both the engineer and petrographer when rocks in an aggregate do not occur in the lists. Rocks exist, may be widespread, may be common, but may not be described as either highly reactive, of low reactivity, or normally reactive. For example, in northern England there are some quarries producing aggregates which include meta-argillite. These rocks are reactive, but are not found in any of the lists. Similarly, while quartz is included in one list, quartzite is not.

Interpretation of the meaning of the name quartzite is always subject to ambiguity and in some cases might correspond with a rock that is of low reactivity and in others a rock of high reactivity. Further, if a list of rocks is provided grouping rocks as of low reactivity and one turns up which has moderately high reactivity, then the whole list is called into question. In Concrete Society Technical Report No.30 for example, limestone is among the rocks considered to be of low reactivity, so too is slate. Both experiments and field occurrences show that slate and limestone can exhibit a high degree of reactivity. Bituminous limestones in north-west England have generated cracking in important structures.

Recognition of Reactive Material

Examination of field and laboratory concretes exhibiting reaction at various levels provides a basis for the recognition of those characteristics of rocks that confer reactivity. These features have been reviewed in a previous conference (French 1991). The starting point must be to agree with Mather (1975) that all rocks can be reactive and to perhaps add that all rocks could include some material that might be reactive under specific conditions. At very high alkali levels ($7 \text{ kg/m}^3 \text{ Na}_2\text{O}$ equivalent), even the flint of south east England is found to be significantly reactive. Under less stringent conditions numerous lithic components can take part in reactions and may occur in a great range of rocks. Cryptocrystalline quartz for example can occur in a wide range of igneous, metamorphic and sedimentary lithologies. The rock type is immaterial, it is the presence of the specific and localised mineral assemblage and texture that confers reactivity. The attribution of potential for reaction or non-reaction to a specific rock name fails to recognise the difficulty attached to the widespread distribution of potentially reactive material. Some names might relate to special geological occurrences - trondjemite, ijolite, charnockite, jacupirangite, kentallenite and so on. Such names are not found in the lists. Other names found in the lists might be loosely and generally used and might or might not exhibit reaction. Andesite might be reactive because of the presence of glass. Other andesites might be stable. Granite is mostly stable but might be reactive because of the presence of tuffisite, cataclasite or strained quartz. Granophyres can be entirely stable or moderately reactive. The draft for BS7943, the standard for the description of rocks with respect to alkali-aggregate reaction describes granite as of low reaction and mentions some of the features that might lead to the development of some reaction. However, a number of possible features that could lead to reaction are not included in this. It is concluded that however careful the author, it is nearly impossible to derive a scheme of nomenclature for rock types that will allow lists to be drawn up of rocks that are safe or of low reactivity in comparison with other lithologies that are of high reactivity. What is important is the presence, abundance and distribution of reactive components in the diverse lithological particles that might be present.

Structures and Textures of Rocks

Specification of permitted amounts of supposed potentially reactive material such as chert must take into account the distribution of the material within the aggregate. The reactive component might be not available to the alkalis because of the structure of the rock, or might be dispersed in the aggregate as a widespread uniformly distributed available ingredient. Opal occurring as a matrix to a sandstone or veins in a granite or patches in chert would be conducive to reaction. In some aggregates in the United Kingdom it is found that the presence of a few highly strained quartz grains, dispersed in sandstones confers reactivity. The recognition of the rock as a sandstone does not automatically confer any interpretation with respect to the degree of reactivity. Nor does the recognition of the highly strained quartz, unless it can be seen that the preparation of

the aggregate allows access to the reactive component for the alkalis in the pore fluid. It is therefore necessary to both identify potentially reactive components and to discuss the potential for reaction in the light of the distribution of the potentially reactive material. It is found for example, that crushed greywacke tends to provide a higher potential for reaction than aggregate found as natural gravels. Meta-argillites show a very similar feature. Perhaps the process of crushing has something to do with this, and the structure of the aggregate, including the presence of a crack within the aggregate, might be significant.

Distribution of Rock in Concrete

The distribution of reactive components in the concrete is also important in the diagnosis of potential for reaction. Reactive rocks are not spaced out uniformly, but are here absent, there abundant. However carefully the mixing of the cement, water and coarse and fine aggregates is carried out, there will be a chaotic distribution of the various particles. The mixing process does not guarantee that there will be uniformity in the distribution of the reactive component in the various fragments present in the concrete. If it is supposed that an aggregate contains about 10% of reactive material in rocks within the size range 5 to 10 mm, there will be places in the structure cast from this mixture which will have little or no reactive component. Other places may have significant concentrations of the component exceeding 35 particles in a slice through the concrete 100 mm square. Where the abundance is appropriate, the reaction becomes significant. Three or four reactive cherts in a coarse sand close together, tend to accentuate expansion created through crack propagation. Consideration must be given to the way in which the reactive component will be distributed: 2% of reactive material, very well dispersed and finely divided, may be beneficial, 2% of the same reactive material in grains 5 to 7 mm across might be very damaging. Slate dust has been found to reduce the expansion due to the presence of reactive slate.

SPECIFICATION

ASTM standards C-670 and E-177 define the use of statistical terms such as precision, accuracy and bias, and place the knowledge and application of such statistical information firmly with the specifier. The user of a procedure must not add measures of precision to a specified amount. Where a specification gives a definite limiting weight or volume percentage of a potentially reactive aggregate, the specifier must know the precision of the method by which this amount is to be found and how the test is to be carried out. The user of the specification must also know the precision of the test method and will use this knowledge to establish the level of confidence to be attached to the results so that the appropriate number of tests and conditions for the acceptance or rejection of an aggregate can be established. Strictly the specifier should ask for a given value not to be exceeded by, or not to be less than, some value at a particular confidence level found by a specified method. In some descriptions of the evaluation of the potential for reaction in the UK, various figures have been given for use in specifications that require interpretation. These include, for example the following limits.

- Chert if present must exceed 60% of the total aggregate.
- Chert must be less than 2% of the total aggregate.
- The aggregate will be found to have low reactivity if 95% is drawn from a particular list of supposedly non-reactive rocks.
- No opal shall be present.

- Aggregate combinations containing more than 10% crushed greywacke are considered to be of high reactivity.

The difficulty of deciding such limits is recognised by the Concrete Society in their Technical Report No.30 (Third Edition) where it says that the specifier may consider setting a specific limit to an aggregate content of below 3% for a particular project. Here the report recognises that difficulties arise in specifying such low limits because the accuracy of the method of measurement for compliance must be taken into account and that the concentrations of reactive minerals in natural deposits can vary widely from point to point. The technical report states it may prove prohibitively expensive to categorise all or part of a deposit with adequate confidence. It must be agreed that where even huge amounts of aggregate are examined and analysed, the reliable determination of small amounts of potentially reactive materials is virtually impossible or at least unrealistic.

How is it feasible to meet a specification which says there shall be no opal? A test method has been included in draft BS7943 issued in 1999. This standard is a guide to the interpretation of petrographical examinations for alkali-silica reactivity. The method for opal detection is based on the gel pat procedure. This method remains statistically based and therefore all that can be said is that the amount of opal is below the detection limit of the specified method. It is necessary to define the detection limit and to adjust the experimental parameters to provide a detection limit acceptable to the specifier. At least 10 gel pat samples may be required unless an earlier test demonstrates reactivity. The amount of opal can also be estimated by an X-ray procedure in which the aggregate is analysed by X-ray diffraction before and after treatment with an alkaline solution. The diffraction lines due to opal can then be recognised and their magnitude determined. Again, the detection limit of this method must be specified.

Precision of Aggregate Analysis - Hand Sampling

Suppose a specification calls for the chert content of an aggregate to be less than 2% by mass of aggregate, then the method must be such that 2% is not exceeded at perhaps the 95% confidence level. The petrographer might follow the procedure given in BS812, Part 104. A sample might be taken of for example a 5 to 10 mm sieve size range. The amount taken would depend on the specified precision and limiting amount. BS812 part 104 gives a formula for calculating the amount of aggregate required for the test. If this formula is applied so as to give a relative error of 10%, then the amount to be examined is 9.8 kg which amounts to approximately 10,000 particles of aggregate. Two such samples must be analysed in full. To be below 2% at the 95% confidence level the result must be about 1.6% or less. If the confidence level is not specified, then it may be necessary to assume that a higher confidence level may be required amounting to certainty. Examination of the formulae provided for calculating the amount and the standard deviation show that the standard deviation is considered to be symmetrical about the mean. In addition, there are constants in the formulae which must have dimensions. The constants must include therefore an estimate of the density of the rock which appears to have been assumed to be 2500 kg/m³. If the individual components in the aggregates vary in density, or the density as a whole varies substantially from 2500 kg/m³, then the amount calculated will be correspondingly in error. It is also apparent both theoretically and from tests that as the weight fraction of component of the aggregate, or better the number of particles of the particular component, departs from 50% so the upper and lower bounds attached to possible results become increasingly asymmetrical. Where

the critical amounts are found to be low, such as 1 or 2% then the upper and lower bounds are strongly asymmetrical.

Precision of Aggregate Analysis - Thin Section

Suppose that the specification calls for the figure of 2% chert to relate to the finished concrete and to be found by analysing test cubes by the method of point counting. Such specifications have been applied in practice. In the past this problem has been treated using the chart given by van der Plas and Tobi (1965) - (reproduced in BS 812 part 104 and Harris and Sym 1990). If a count totalling 2000 points is carried out then a relative error of about 24% might be read from the chart and the 2% present might be cited as $2 \pm 0.5\%$ at the 95% confidence level. However, this chart has been shown to be based on statistical misconception and does not address the question of placing an upper bound on the proportion of a constituent which might reasonably be expected to be present, but has not been detected in a sample. These issues have been discussed in detail in Howarth (1998) who provides both graphical and exact computational methods for calculation of the correct confidence bounds. For example, the 95% confidence interval of the point count analysis using 2000 points will be asymmetrical and the upper bound value will be about 0.7%, while the lower will be close to 0.5%. The value of 0.7% will therefore apply to the cited example. If the result is to be less than 2% at the 95% confidence level, the maximum amount found would need to be 1.3% which has an upper bound of about +0.6% at the 95% confidence level. However, the 2000 points counted could relate to 10 pieces of rock or 1000 pieces of rock. The area of the concrete tested by the point count method must be determined by the size of the aggregate in question. The spacing might be about the mean size of the aggregate so that for a 5 to 10 mm aggregate at 2000 points, an area about 30 centimetres square would need to be analysed. Consideration therefore needs to be given to the grading curve of the aggregate and the number of particles contributing to the determination of the amount to be decided. It is concluded that it is unrealistic to expect specifications of the type listed above to be confirmed numerically by analysis of samples of aggregate. It is not possible to say there will be no opal and immensely difficult to say that there will be less than 2%.

Because of inherent sampling error, the reliability of the result for a given single sample the precision of the result can be improved and the method simplified if the analysis is carried out on separated sub-samples. Combining the results of the analysis by the method of point counting of three or more sub-samples will always be superior to counting the same total number of points on one sample. The same relationship applies to particle counting. However, the result obtained for the samples analysed by either weighing fragments or by point counting provide data relating only to the bulk of the sample tested. Analyses of samples serially collected can permit projection of the results and provide a basis for calculating a maximum expectation of the amounts of a particular ingredient (Howarth and French 1998 and in preparation). The question then becomes how can the result be extrapolated to future samples. This involves the concept of a tolerance interval, which specifies a statistical interval in which one can expect, with stated confidence, that a specific proportion of the individual in the population from which the statistical sample was taken can be expected to lie. This differs from a confidence interval which specifies an interval within which an unknown parameter of the sampled population, in the present case the proportion of a given constituent, can be expected to lie with stated confidence. This is reviewed in Howarth and French (in preparation) and Howarth and French (1998).

CONCRETE AND MORTAR TESTS

Concrete Prism and Mortar Bar Tests that have been used for many years have yet to yield very consistent and reliable results. The more the procedures are studied the worse their inherent precision appears to be. ASTM C1260 appears at first sight to be a well-defined test that should have very good reproducibility but the results of the round robin reported in Rogers *et al* (1996) suggests the opposite. Reasons for the possible range of results have been reviewed by Wigum *et al* 1998. Some of the factors are the variation in amount of reactive aggregate in relation to the pessimum in each bar, the variation in bulk composition of the mortar, and variation in aggregate grading and composition through riffing. Despite these difficulties, it seems essential that the petrographic examination should indicate where these tests would be appropriate. It is also essential that the test bars should be studied in thin section for evidence relating to the cause of expansion and the identity of any reactive component.

ROUTINE FOR PETROGRAPHIC EVALUATION

It is clear from the foregoing analysis that if rock types are to be identified and their proportions established with an appropriate level of confidence an unrealistically large sample size may be required. However it is more important to recognise where potentially reactive materials are present than to establish their abundance with a high degree of precision. The specification should avoid statements that cannot be met in a routine petrographic study. In order to circumvent the statistical problems it is proposed that aggregates should be examined for their potential for AAR and to provide a basic general description in the following four stages.

- ***A walk over survey of quarry or pit.***

This allows recognition of the lithologies major and minor that will appear in the aggregate. Some thin section examination might be required. (Benefit will derive from the examination of concrete containing the aggregate where this is possible.)

- ***Examination of hand specimen sized samples after scalping and the initial crush.***

A selection of 100 to 200 hand specimens from the conveyor belt, washed and examined with the hand lens allows a detailed view of components likely to be seen in the prepared aggregate and their proportions to be established. It also provides a simple method of quality control in that new rocks appearing can be recognised early.

- ***Examination of a fine aggregate or crushed coarse aggregate in thin section.***

The aggregate is prepared as a thin section in resin with a maximum size of 5 mm after washing to remove dust. The section should contain about 6,000 particles. Main rock types will be identified and specific structures, textures and compositions likely to prove potentially deleterious recorded. Sampling of the stock pile must be carefully designed. It will usually prove best to sample over a period and to examine the material in batches. Estimates should be made of the proportions of rock fragments containing potentially reactive components using grain counts in traverses of the section. It is not considered necessary to count a large number of particles since the procedure is for the diagnosis of the presence of the potentially reactive components and not determination of statistically valid proportions.

- ***Examination of mortars in thin section.***

Where suspect material is encountered the fine aggregate or crushed coarse is made into a mortar and cast as briquettes measuring about 50 by 80 by 10 mm. These have the alkali content of the cement increased to 4% by weight of cement by adding alkali hydroxide. They are heated

in an autoclave at 120 °C for 4 hours at 0.1 MPa and on cooling are made into thin sections. Observations are made of the extent to which reaction has occurred in the formation of gel and microcracking. This procedure can also be adapted to quality control. Batches of 200 ml of mortar with a volume of paste of 40% and a water/cement ratio of 0.4 are required.

CONCLUSIONS

1. Statistical analysis of aggregate in the manner outlined in BS 812, Part 104 is meaningless from the point of view of diagnosis of aggregate quality or of the potential of the aggregate for reaction. The amount of rock that must be analysed is extremely high, especially where minor components are likely to promote damage. The extrapolation of data from one stockpile to another is unsafe unless a great deal of work is carried out from day to day, week to week and month to month. Only then is it possible to predict forward from existing data.

2. How many tests should be carried out? Irrespective of the type of test undertaken, if the result is classified as a pass or fail then the total number of tests (N) required, in order to have a stated confidence that the risk of future failure in the material under test will not exceed a stated value, may be very large. For example, how many tests should be carried out in order to have 95% confidence that if no failures have been observed in all N tests, the upper limit of the underlying risk of failure in the material sampled (Δ) will not exceed for example 1%? In this case N is given by $100(3/\Delta) = 300$ tests (see Howarth 1998). Clearly, long-term sequential sampling and testing of the source material is the only practical answer to such a demanding testing requirement, as was advocated by Howarth and French (1998). Lack of application of these principles has led to grossly over optimistic estimates of the level of control which is achievable in practice in meeting product specification.

3. The construction of lists of potentially reactive and potentially safe rocks is both unsatisfactory, unnecessary, and unsafe. No rock can be regarded from its name as truly of low reactivity or as not exhibiting reaction. Rather, the presence or absence of particular features throughout the aggregate may be used to infer potential for reaction or its absence. It is necessary to find out what is present in the aggregate and evaluate its distribution without recourse to impossibly large sets of samples.

4. Hand specimen diagnosis of rock types can be misleading and is potentially dangerous. The recognition of features that might lead to reaction can only be carried out through the observation of polished or thin sections. X-ray confirmation of the identity of particular phases might also be important as are studies of gel pats and mortars. Tests using prepared concretes and mortars should be followed by petrographic examination of the products.

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