RILEM PETROGRAPHIC METHOD: PRACTICAL USE AND COMPARISON WITH OTHER PETROGRAPHIC METHODS IN USE

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Abstract
This paper discusses the principles between the new RILEM TC ARP – 1 Petrographic method and other petrographic methods in use around the world. The methods discussed are the American standard method ASTM C 295, British standard method BS 812: Part 104, European standard EN 932-3, Danish method TI B 52, Norwegian petrographic method and a draft Dutch method. A survey by Rilem has shown that petrographic methods are used in most countries where Alkali Aggregate Reaction has been accepted as a concrete problem. Correlation tests in Norway have shown that grain counting gives higher content of alkali reactive aggregates and that crushing of coarse aggregates not influence the overall content of alkali reactive aggregates in samples. Round robin tests in Norway have shown that the precision of test results are acceptable when operators are experienced and educated to perform the petrographic analyses.

Keywords: Concrete, Aggregates, Alkali silica reaction, Petrography, Microscopy,

1 Introduction

Natural and artificial aggregates are the most important constituent in many building materials e.g concrete, mortar and roads. Natural aggregates are processed from natural deposits of sand, gravel or crushed bedrock and artificial aggregates are in most cases waste materials (e.g. slag). The method to describe and classify aggregates is the petrographic analysis. The petrographic analysis is a systematic description method for rocks, minerals and other constituents, usually in hand specimens, thin sections or by use of other analytical methods (e.g. XRD- analysis). The purpose with the petrographic analysis is to obtain information on one or all of the material characteristics: *Geometrical–mechanical-physical-and chemical properties, impurities, contamination and very important the rock and mineral content*. The rock and minerals constituents are in many cases conclusive for the end use of aggregate. This because some constituents might give performance and durability problems in concrete, *e.g. sulphate attack, swelling problems, Alkali Aggregate Reaction (AAR) and in rare cases Alkali Carbonate Reaction (ACR).* The petrographic analysis can be qualitative or quantitative or both:

- The qualitative examinations identify and describe some or all of the constituents but the proportion of the constituents are not established.
- The quantitative examination determines some or all of the constituents in the samples.

To obtain information on the rock and mineral composition of aggregates two quantitative methods generally are used, namely grain counting method and point counting method:

- The grain counting method is used on one or all "coarser" size fractions where aggregate particles are divided into individual rock/mineral groups by hand sorting. The result is given as particle percent of total counted particles or weight percent of total weight.
- The point counting method first moulds selected size fractions into thin sections. By use of a petrographic microscope and a point counter device traverses in two directions are carried out to form a virtual orthogonal grid that covers the whole thin sections. Individual particles are classified and grouped. Results are given as volume percent.

The paper discusses principles of the two quantitative methods in use and the new petrographic method by the Rilem Technical Committee "ARP". Important issues discussed are correlation between the two quantitative methods, the effect of crushing coarse aggregates, accuracy of test results and nomenclature for rock and minerals.

2 Principles of petrographic methods

2.1 ASTM C 295. Petrographic Examination of Aggregates for Concrete

ASTM C 295 [1] is probably the most used petrographic method in the world. The method is used to assess undeveloped quarries, natural gravel and sand, drilled cores, ledge rock, crushed stone and manufactured sand. Both qualitative and quantitative analyses are carried out by assumed qualified personnel. Natural sands and gravels are examined in the following way:

- First visual examination for exterior coatings
- Assessment of rock and mineral constituents by dividing the material into sieved fractions. From each fraction a minimum of 150 particles shall be identified and sorted into rock types by visual examination and then counted. The stereoscopic microscope should be used for the examination of fine-grained rocks. Fine-grained and glassy materials which may be susceptible for reaction with alkali's should additionally be examined by the petrographic microscope (immersion oil slides). For aggregate sizes of less than 600 micron, these shall be fractionated into smaller sieve sizes and analysed by the stereoscopic microscope or "as required" mounted into immersion oil slides for examination and counting under the petrographic microscope. Grain thin sections mounted in epoxy can be used for materials finer than 300 micron.
- Sieve fractions are classified according to "condition" and categorised as either: 1) fresh or dense, 2) moderately weathered, 3) very weathered, or alternatively as: a) dense, or b) porous.

Nomenclature for naming rock and minerals are given in **ASTM C 294 [2]** and assessment for alkali reactivity in **ASTM C 33 [3]**.

2.2 BS 812: Part 104. Qualitative and quantitative petrographic examination of aggregates

BS 812: Part 104 [4] is a relative new British standard. A similar test method was suggested as European Standard **EN 932-4 [5]** but was withdrawn as European standard by the CEN TC 154 committee. It also assumed that qualified personnel carry out the BS 812: Part 104 analysis. The method is used to assess natural gravel and sand, crushed rock and artificial aggregates. Both qualitative and quantitative analyses are carried out. The quantitative analysis is carried out on bias samples of such a size that an absolute error of +/- 10% is obtained. Coarse and fine aggregates are fractionated into specified sieve sizes and weighted. From each size fraction particles are hand sorted and subdivided into "groups of purely nomenclatural interest" according to **BS 812:Part 102 [6]**. The finer fractions can eventually be sorted by use of a stereo microscope. When required, thin sections of selected particles of each constituent shall be prepared for verification purposes. The size fractions of sand finer than 1.18 mm shall be mounted into a thin section and point counted. Results are calculated for each constituent of each size fraction for both bias samples. Results are given as average weight percent together with the combined standard deviation, confidence limit and relative error. Assessment for alkali reactivity is given in **BS 7943 [7]**.

2.3 EN 932:Part 3 Procedure and terminology for simplified petrographic description

The European standard **EN 932-3 [8]** is used for natural sand, gravel and crushed rock aggregate. It is pointed out that the procedure is not suitable for detailed petrographic study of aggregates for specific end use. Qualified personnel only should carry out the examination. The method is generally qualitative even grain counting should be carried out. However, no guidance on grain counting or precision is given. Individual particles and the aggregates as a whole should be described according to preferred nomenclature given in an Annex or in a simplified way e.g. as sedimentary rock.

2.4 TI B 52 Petrographic investigation of sand

TI B 52 [1] is a Danish petrographic method, which has been used in Denmark since the early 1980's. The method is used only on natural sand for assessment of potential alkali reactivity. There is no requirement for the personnel's qualifications. The method is carried out the following way.

- A representative sample (minimum 1-kg) is sieved into the size fractions 0-2mm and 2-4mm. One fluorescence impregnated thin section from each fraction is prepared.
- Rock and minerals are verifyed and counted by point counting. At least 1500 points for the 0-2 mm fraction and at least 1000 points for 2-4 mm fraction has to be estimated.

Average results from the two size fractions (0-4 mm) are given as volume % dense flint (innocuous) and volume % porous chalcedony flint and opal flint. The reactive content is the sum of porous chalcedony flint and opal flint.

2.5 Norwegian petrographic method(s)

Before 1993 a modified ASTM C 295 petrographic method was used in Norway for assessment of natural sand, gravel and crushed rocks. Grain counting of few selected size fractions was performed. In the early 1990's alkali silica reaction was accepted as a concrete problem in Norway and thin sections were then prepared for verification of grouped rock types in the same way as in BS 812:Part 104. Alkali reactive aggregates were identified according to a preliminary negative list by **Jensen [10]**, and a limit value on 20 volume percent alkali reactive aggregates were recommended **NB 19 [11]**. Because many Norwegian aggregates are dense and fine grained and impossible to identify by normal visual methods a point counting method was introduce in 1993, as described in the following:

- For natural sand and manufactured sand representative sub samples of the 1-2 mm size fraction and 2-4 mm fraction are moulded into thin sections, one thin section of the 1-2 mm fraction and 2 thin section of the 2-4 mm fraction.
- For coarse gravel and crushed rock the fragments or particles are stepwise crushed down. Sub samples of the 2-4 mm fraction is separated and moulded into 2 thin sections for point counting.
- Point counting is carried out on the thin sections. Minimum 1000 points are counted on each size fraction. All major rock types and minerals are recorded and grouped. Results are given as average percent of rock – and minerals of total counted points.

The sum of all identified alkali reactive rock types is used for assessment of alkali reactivity. In cases where the sum is higher than 20% by volume, the aggregates is classified as “alkali reactive”, otherwise it is regarded as innocuous **Lindgaard et al [12]**.

2.6 Rilem TC ARP-1. Petrographic method

Rilem TC ARP-1 [13] (formerly Rilem TC 106-1) is a new international procedure for petrographic examination of concrete aggregates: to quantify the amounts of various mineral and rock types, and to identify rock types and minerals, which might react with alkalis. The method is intended to be used for the examination of natural aggregates including sand, coarse gravel, all-in and crushed rock aggregates. The method can be used for both qualitative and quantitative determination of rock and mineral compositions and it is very important that a qualified geologist or petrographer carries out the analysis. The petrographic analysis may be carried out by two procedures namely, procedure 1: "Simplified petrographic analysis" or procedure 2: "Examination and point counting on thin sections".

- Procedure 1 is similar but modified according to the ASTM C 295 method and used where alkali reactive rock/mineral constituents are easily identified by the naked eye or under a stereoscopic microscope. Procedure 1 is used where it is relatively easy to group aggregate constituents on the basis of colour, shape or texture and identify alkali reactive constituents. It may be necessary to study thin sections of selected aggregates to enable full identification of the material in a similar way as in BS 812: Part 104.
- Procedure 2 shall be employed when alkali reactive rock/mineral constituents cannot be identified by procedure 1. Procedure 2 is similar to the Norwegian point counting method. In cases the aggregate contain porous flint, thin sections should be prepared by vacuum impregnation with epoxy containing the fluorescent dye (e.g. Hudson Yellow). The porosity of constituents may then be identified under the petrographic microscope by UV-light in a similar way as the Danish TI B 52 method. Guidance on minimum number of standard size thin sections used in the methods to obtain satisfactory precision is given. It is recommended that a minimum of 1000 points exclusive points falling onto the resin are counted for the 2-4 mm fraction (two thin sections) and 1000 points on the 0-2 mm or 1-2 mm fraction (one thin section). However, when target values are low or higher precision is required the number of grains/points to be analysed can be read from charts on absolute and relative errors by **Howarth [14]**, which have been included in an annex.

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 method an aggregate should be classified as either: *"Unlikely to be alkali-reactive"*, *"uncertain alkali-reactive"* or *"very likely to be alkali-reactive"*. 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.

2.7 Dutch point counting method

The Dutch draft petrographic method for assessing the potential alkali-silica reactivity of aggregates is based to a large extent on Rilem ARP–1 **Larbi [15]**. The method is intended for use within the assessment of natural sand, gravel, crushed fine - and coarse natural aggregates and recycled aggregates. The crushing procedure of coarse aggregates is more comprehensive than recommended in the Rilem procedure, more thin sections are used and minimum 3000 points are counted in those thin sections. This is undertaken, because the potentially reactive constituents in many cases are detected at levels of less than 2 % by volume.

3 Rilem survey of AAR test methods in use

In 1990 the Rilem committee TC 106 (now replaced by committee ARP) undertook a worldwide survey (questionnaires to committee members) on test methods for alkali reactivity in use. This survey has since been revised several times, last time being in 1998 **Sims [16]**. The survey now covers 23 countries and data from a further 4 countries are awaited. Table 1 gives the answers from petrographic methods in use grouped into grain counting methods, point counting methods and unknown procedures to the authors knowledge. It has to be mentioned that other test methods are used in the countries but not given in table 1.

Table 1 Rilem TC 106 survey of test methods in use, **Sims [16]**

COUNTRY	GRAIN COUNTING	POINT COUNTING	UNKNOWN PROCEDURES
Argentina	**ASTM C 295**		
Australia	**ASTM C 295**		
Belgium		**Belgium method**	
Canada	**ASTM C 295**		
Cyprus*			
Denmark		**Ti-B52**	
France			**Petrographic method**
Germany			**Separation of +1mm**
Hong Kong	**ASTM C 295**		
Iceland	**ASTM C 295**		
India			**IS-2386:Pt8**
Ireland	**ASTM C 295**		
Italy			**UNI 8520 part 4**
Japan			
Netherlands		**Dutch method**	
New Zealand			**Petrographic exam.**
Norway		**Norwegian method**	
Romania			**Macro-micro method**
Russia			**GOST 8269.0-97**
South Africa			**SABS 1083**
Sweden	**ASTM C 295**	**Norwegian method**	
UK	**BS 812-104**		
USA	**ASTM C 295**		

*Note that only Cyprus does not presently use a petrographic methods for assessment of alkali reactivity (but uses other methods not shown in table 1).

4 Grain counting versus point counting

In 1993 a grain counting method (modified ASTM C 295) was replaced by a new point counting method used for commercial testing for alkali reactivity in Norway. At the same time test results obtained by the grain counting method were not considered valid as documentation on alkali reactivity according to the Norwegian "Declaration – and approval arrangement for concrete aggregates" **NB 19 [11]**. Figure 1 shows test results of alkali reactive rocks obtained by the two methods. Grain counting and point counting were carried out on 29 different samples taken from the same quarry with one year intervals between each sampling. Note that an "acceptable" correlation exists and that the grain counting surprisingly obtains significantly higher values than the point counting method.

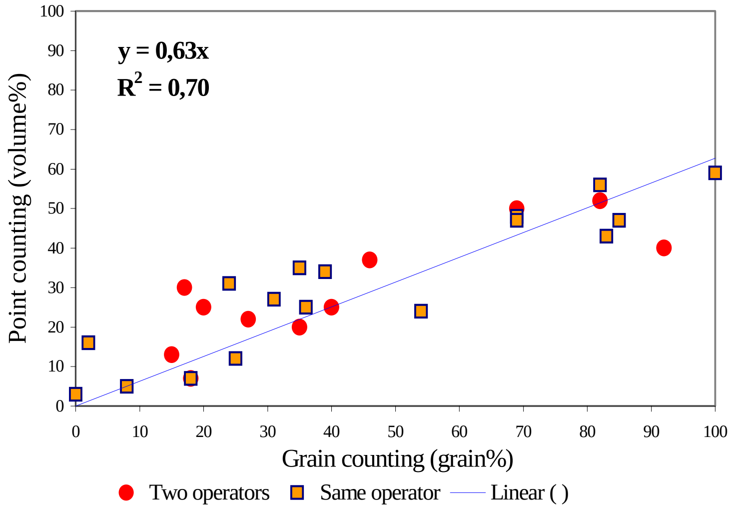

Figure 1: Reactive aggregates (%) from the same quarry analysed by grain - and point counting.

5 Effect of crushing

Examination of coarse aggregates by the point counting methods as described in the Norwegian and Rilem techniques can require that the aggregate particle size is initially reduced by crushing before mounting within a the thin sections. Figure 2 shows the correlation of alkali reactive rocks by point counting of 37 sand - and coarse aggregates sampled from the same quarry in Norway. The samples are taken from the same batch or different batches with one year intervals and the coarse aggregates have been crushed before being mounted into thin sections. Note that a good correlation exists

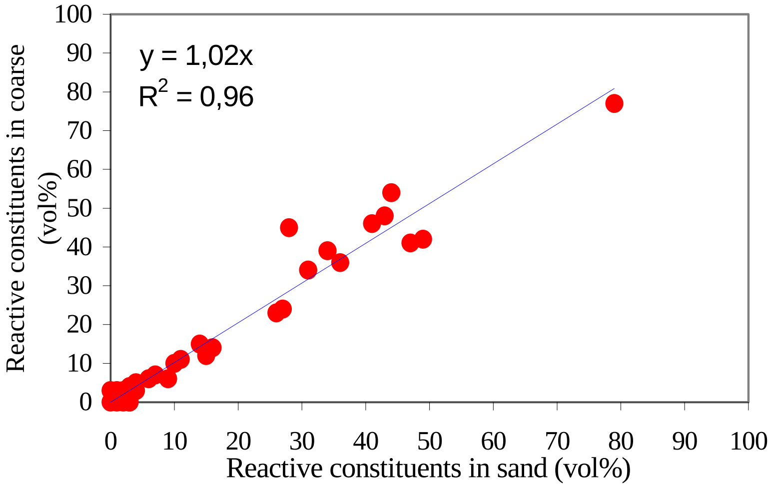

Figure 2: Alkali reactive rocks from the same quarry determined in coarse aggregates and sand

between the coarse fraction and sand from the same quarries. It looks like the crushing, which has been carried out in a series of steps, gives the same content of alkali reactive rocks compared to sand, which only have been sieved. Moreover, no significant difference on the reactive constituent content seems to occur between fine sand and coarse aggregates in Norwegian natural aggregates.

6 Accuracy

6.1 Grain counting method BS 812: Part 104

According to BS 7943:1999 precision trials between 8 laboratories in UK have been carried out on two common gravel types in Southern England and gave the following results

- Thames flint gravel: average 6.7%, repeatability 2.3% and reproducibility 2.3%
- Essex flint gravel: average 75.6%, repeatability 6.9% and reproducibility 9.9%

Definitions of reproducibility and repeatability are not given but might be according to BS 5497-1.

6.2 Norwegian point counting method

In 1995 a "parallel testing" between SINTEF and NGU (The Norwegian Geological Survey) was carried out on the same thin sections of 47 samples from the most important natural aggregate deposits in Northern Norway. Before testing, personnel from NGU underwent an intensive 2 weeks education program allow them to classify Norwegian alkali reactive aggregates in thin sections. Results of the testing gave a correlation coefficient (R^2) on 99 and the absolute error for the method was suggested to be +/- 3% by **Jensen [17]**. In 1998 a round robin test was carried out between 6 laboratories within Norway. Before testing a one-day introduction course about point counting and identification of alkali reactive Norwegian rock types was undertaken. The results of the round robin test were very disappointing, with an insufficient low correlation between the various laboratories (R^2 varied from 0,84 to 0,34) **Jensen [18].** In 2000 a second round robin test was carried out in Norway using anonymous thin sections from the North Norway project **Wigum [19]**. Figure 3 shows results obtained by laboratories no 1 to 6 versus the average volume % obtained by the North Norway project.

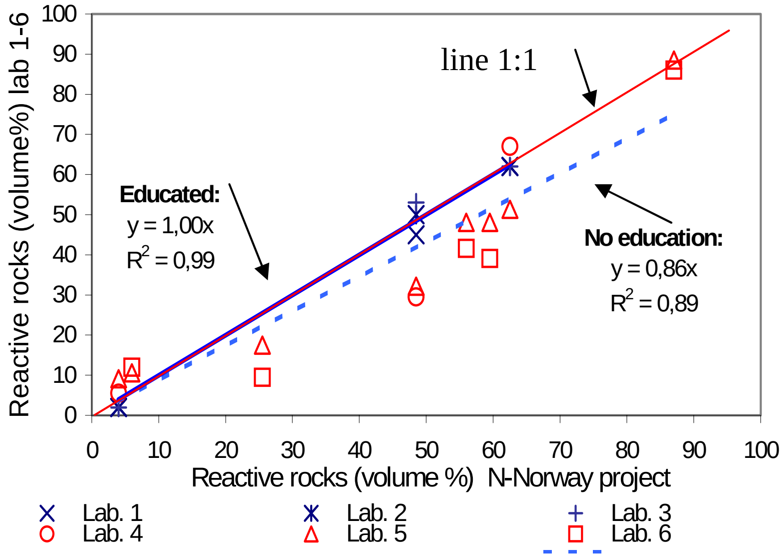

Figure 3: Results from round robin test: laboratory no 1 to 6 versus the North Norway project

Operators 1, 2 and 3 were all well educated to use the method and had experience to perform the point counting whereas operators 4, 5 and 6 not were educated in the use of this method (but with geological insight). However, all the operators had participated in the first round robin test in 1998. The difference from suggested right value (average vol. % by the North Norway project) results varied from *–2 vol. % to 4 vol. %* for the educated operators, and from *–6 vol. % to 20 vol. %* for the non-educated operators. Note that educated operators obtain a very good correlation (R^2 = 0.99) and the non-educated operators obtains a lower correlation (R^2 = 0.89).

6.3 Danish TI B 52 point counting method

In 1986 **Brandt [20]** reported the reproducibility and repeatability by the TI B 52 method. Material from the same natural sand quarry was parted into sub samples and analysed by 2 operators. One operator had experience with the point counting method and the other had no experience with the method, but had geological insight. The average result of recorded reactive constituents (porous chalcedony flint and opal flint) from the 7 sub samples (thin sections) was calculated to be 1.5 vol. % and standard deviation to be 0.43 %. The minimum and maximum values obtained by operator 1 varied from 0.9 vol. % to 2.2 vol. %, and by operator 2 from 0.8 vol. % to 2.0 vol. %. Figure 4 shows the results by the two operators. Note the relative large variation of results and rather low correlation between operators in the light of the Danish limit values on 1.0 vol. % respectively 2.0 vol. %.

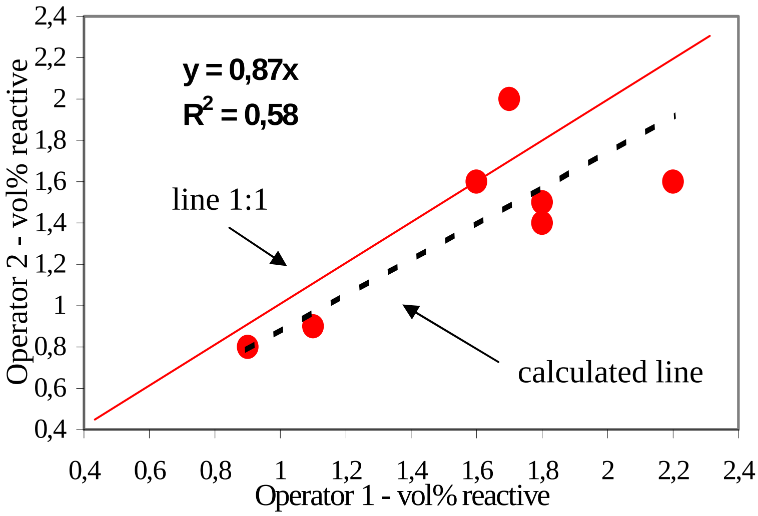

Figure 4: Reactive constituents in natural sand from the same Danish quarry obtained by two operators

7 Discussion and summery

The Rilem survey has shown that petrographic analyses for assessment of potentially alkali reactive aggregates are used in most countries where the Alkali Aggregate Reaction (AAR) problem have been identified. The grain counting method is often supplemented by a more detailed investigations by immersion oil slides (e.g. ASTM C 295) or thin sections (e.g. BS 812: Part 102) examined under the petrographic microscope. The point counting method is used generally for assessment of alkali reactivity when reactive constituents can not be identified by "normal" visual methods. The point counting of thin sections for assessment of potentially alkali reactivity is today used in an increasing number of countries e.g. Denmark, Norway, Belgium, The Netherlands and Sweden. The Rilem TC ARP–1 petrographic method combine both the grain counting method and point counting and is intended to be an International standard method. The draft Dutch petrographic method is primarily based on this new Rilem method. Tests in Norway have shown that grain counting gives higher content of alkali reactive rocks than point counting in thin sections, and that crushing does not influence the test results. Round robin tests in Norway have also shown that when operators are experienced and educated to use the petrographic method the precision is more acceptable and close to the optimum statistical error to be obtained according to charts by Howarth [14]. For lower limit values e.g. 2.0 vol. % as required in Denmark and The Netherlands it is necessary to analyse a rather higher number of grains and points (minimum 3000) to obtain an acceptable precision. An important item taken on by Rilem ARP-1 is nomenclature for the naming of rock and minerals. A complete guidance and naming list of aggregates is given in an annex. Classification of alkali reactive rock and minerals, nomenclature of aggregates and accuracy of petrographic methods are available in many countries. In the United Kingdom a group of material testing companies presently collaborate on a technical committee termed the 'Proficiency Scheme for Aggregate Petrographers' (PSAP) which is affiliated to the Geological Society of London. The ultimate aims for this group of companies would be an increased accuracy of aggregate petrographic analyses with respect to precision and nomenclature. In Norway material testing laboratories together with aggregate producers collaborate in the committee "FARIN" with the same goals as PSAP.

8 Conclusion

Petrographic methods for assessment of potential alkali reactivity of aggregates are used in most countries where Alkali Aggregate Reaction has been identified as a concrete problem. Generally quantitative petrographic analyses can be performed as grain counting or point counting. ASTM C 295 and BS 812:Part 104 specify grain counting as the preferred method. However, point counting is now specified as preferred method in an increasing number of countries in Northern Europe. The Rilem TC ARP–1 petrographic method includes both a grain counting procedure and a point counting procedure to be performed. To obtain reliable results it is very important that operators are qualified and educated to perform the petrographic analyses. This has been shown by round robin tests in Norway and other places.

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