# A COMPARISON OF LABORATORY TESTING METHODS FOR EVALUATING POTENTIAL ALKALI-REACTIVITY IN THE ST. LAWRENCE LOWLANDS (QUEBEC, CANADA).

Benoit Fournier* and Marc-André Bérubé**
* CANMET, 405 Rochester Street, Ottawa (Canada), K1A OG1
** Département de Géologie, Université Laval, Québec, Canada, G1K 7P4

Seventy-one limestone and dolostone aggregate samples representative of the different sedimentary rocks being exploited in the St. Lawrence Lowlands (Quebec, Canada) have been subjected to various AAR laboratory tests. Acceptance limit criteria are proposed based on the behavior of these aggregates under standard testing conditions and on field performance. A decision chart is presented for determining the potential reactivity of concrete aggregates in the St. Lawrence Lowlands.

## INTRODUCTION

Since the pioneering work of Stanton and co-workers in the 1940's, a variety of laboratory test methods have been developed for assessing the potential reactivity of concrete aggregates. All those test methods were developed to predict, as adequately and rapidly as possible, what might happen to concrete structures incorporating an aggregate over a 50-year time period under field exposure conditions. In fact, most of these test methods can only determine if the aggregate investigated has a potential for deleterious reactivity in an alkaline, basic environment such as the concrete pore solution. To what extent the aggregate will react deleteriously in the field will then depend on a large number of factors: the exposure conditions of the structure, total alkali content of the concrete mixture, the mixture proportioning, the design of the structure, etc. This paper compares the results of a series of laboratory alkali-aggregate reactivity (AAR) tests made with carbonate aggregates from different areas of the St. Lawrence Lowlands with the evidence of AAR in field concrete structures containing the same aggregates. The purpose is to establish practical reaction limit criteria for quality control purposes.

## MATERIALS AND METHODS

The characteristics of the carbonate aggregates investigated have been described by Fournier and Bérubé (1). Details of the various mortar tests performed in this study are given in Table 1. A modified version of the ASTM C227 Mortar Bar Method was applied to a number of selected aggregates. Bars were made at a constant 0.50 w/c with the total alkali content of the mixture raised to 1.25% $Na_2O$ equivalent by adding NaOH to the mixture water. The bars were then stored at 38°C in plastic pails where wicks have been removed to reduce the risks of alkali leaching (Rogers and Hooton (2), (3)). Three other series of ASTM C 227 type mortar bars were made and subjected to various curing conditions in the laboratory (Table 1). These include immersion in a 1N NaOH solution at 38°C and 80°C, and steam curing in an autoclave at 130°C (0.172 MPa). To serve as a reference for the mortar bar tests, concrete prisms 75 x 75 x 300 mm in size were made with a 0.50 w/c, using a cement content of 350 kg/m$^3$. A normal ASTM Type 1 high alkali cement was used, while the total alkali content of the mixture was raised to 1.25% $Na_2O$ equivalent by adding NaOH to the mixture water (1). The prisms were cured for 1 year at 38°C and R.H. > 95%; length change measurements and petrographic examinations were made periodically to monitor expansion and the development of cracking.

## RESULTS AND DISCUSSION

### Expansion Limits

Within the past five years, the Accelerated Mortar Bar Test (AMBT) (Oberholster and Davies (4), ASTM C-9 Proposal P 214 (5)) has become the most widely used rapid AAR test in Canada. A number of studies have indicated that a 14-day, 0.15% expansion limit would probably apply to most Canadian aggregates; however, Grattan-Bellew (6) suggests that expansion limit criteria based on aggregate types might be more reliable and realistic. Figures 1A and 1B show the 14-day expansion values obtained in the AMBT plotted against the 6-month and the 1-year expansion values, respectively, measured in the Concrete Prism Test. The results are shown according to the stratigraphic association of the corresponding rocks (1). An extensive field survey program performed over the past five years has indicated that the current condition of concrete structures in various parts of the St. Lawrence Lowlands is adequately represented by a 6-month, 0.04% concrete prism expansion limit criterion for distinguishing reactive or potentially reactive aggregates from non-reactive ones (Fig. 1A) (Fournier et al. (7), Fournier and Bérubé (8)). It was also suggested that 1 year expansion values might help to reveal the behavior of a few marginally reactive aggregates in concrete structures subjected to very severe exposure conditions. From these results and from those of the concrete prism test, Fournier and Bérubé (1) have suggested using a 14-day, 0.10% accelerated mortar bar expansion as a safe acceptance limit criterion for non-reactive carbonate aggregates (Fig. 1A). Increasing the limit criterion to 0.15% will lead to the acceptance of a few aggregates with a known deleterious field performance but a number of aggregates with a satisfactory field record. On the other hand, field performance observations in the St. Lawrence Lowlands have also indicated that the majority of carbonate aggregates causing accelerated mortar bar expansions > 0.25% at 14 days (most of them corresponding to fine-grained and dark-grey Black River and Trenton limestones) have performed deleteriously in concrete structures subjected to conditions that promote AAR (1,8).

TABLE 1 - Characteristics of the various mortar bar tests performed in this study.

Common features:

| | |
|---|---|
| Agg. particles: | Washed, grading according to ASTM C 227 |
| Cement: | High alkali ASTM Type 1 (1.0% $Na_2O$ equivalent) |
| W/C: | Fixed at 0.50. |
| Agg./cement: | 2.25 : 1 (3 bars per mix). |
| Bars size: | 25 x 25 x 285 mm. |

Characteristics specific to each method:

| "Modified" ASTM C 227 | Bars immersed in 1N NaOH at 38°C | NBRI Method (AMBT) | Autoclave test |
|---|---|---|---|
| Alkali content (mix): 1.25% | Alkali content (mix): 1.0% | Alkali content (mix): 1.0% | Alkali content (mix): 3.5% |
| Curing: 38°C, R.H. > 95% | Curing: 1N NaOH at 38°C | Curing: 1N NaOH at 80°C | Curing: steam curing for 5 hours at 130°C. |
| Container: 22-litre plastic pail, without wicks; (12 bars / pail). | Container: 4.2-litre plastic vessel (3 bars / vessel). | Container: 4.2-litre plastic vessel (3 bars / vessel). | Container: Autoclave. |
| Measurements up to 1 year. | Measurements up to 1 year. | Measurements up to 28 d. | Readings at room t°, before and after steam curing. |

A grey zone is observed in the upper left portion of Fig. 1A and 1B, for a number of aggregates that have induced excessive mortar bar expansion compared to the concrete prism test results. This applies often to dolomite-bearing Beekmantown, Chazy and Black River aggregates, for which this method seems to be too severe (1). Determining the potential alkali-aggregate reactivity of such aggregate types, as well as argillaceous dolomitic limestones susceptible to alkali-carbonate reaction, for which the AMBT is not effective, will thus require further testing in the laboratory, preferably using the Concrete Prism Test. Even if some of the aggregates falling in the upper left portion of Fig. 1A induced concrete prism expansion slightly > 0.04% at 1 year (Fig. 1B), most of them have a satisfactory field record. This would suggest increasing the concrete prism acceptance limit criterion to 0.06% when using the 1-year expansion values (Fig. 1B).

Figures 2A and 2B summarize the results of accelerated mortar bar tests performed on the 71 carbonate aggregates being studied. Expansion limit criteria of 0.04% and 0.06% were used at 6 months (A) and 1 year (B), respectively, so as to outline reactivity zones. Figures 2A and 2B also show the expansion limits proposed by Hooton (9) (0.15% at 14 days and 0.33% at 28 days) for distinguishing non-reactive and marginally reactive aggregates from reactive ones. Using the 1-year concrete prism expansion values as a reference for the AMBT results lowers the acceptance limit for non-reactive aggregates (even considering a 0.06% concrete prism expansion limit criterion instead of 0.04%) while leaving the other limit criterion unchanged (Fig. 2B). Three of the nine marginally reactive aggregates that have expanded less than 0.04% at 6 months but more than 0.04% at 1 year in the Concrete Prism Test were still expanding less than 0.10% after 28 days in the AMBT. The reason for such a behavior is still under investigation. Hooton (9), Shayan (10) and Bérubé and Fournier (11) have also reported cases of aggregates such as strained quartz-bearing gneisses and quartzites, quartzitic (Potsdam type) sandstones, and phyllites that did not expand significantly in the AMBT but showed deleterious field performance. It has thus been suggested that the curing period in the AMBT be extended to 28 or 56 days to catch these aggregates. In this study, since no deleterious field performance for the three marginally reactive aggregates mentioned above have yet been reported, and since lowering the acceptance limit will penalize a number of non-reactive aggregates, it is suggested that the chart presented on Fig. 2A be used for potential alkali-reactivity determination with carbonate rocks of the St. Lawrence Lowlands.

Figure 3 shows the expansion values obtained after 5 hours of steam curing in the autoclave test plotted against the 14-day expansion values obtained in the AMBT, for a number of aggregates. A 0.15% autoclave expansion limit criterion has been proposed to differentiate reactive or potentially reactive aggregates from non-reactive ones (Fournier et al. (12)). Empty and filled symbols correspond to samples that have caused concrete prism expansion < 0.04% and > 0.04%, repectively, at both the 6-month and 1-year time period. Grey symbols represent aggregates that induced concrete prism expansion < 0.04% at 6 months and > 0.04% at 1 year; these are considered as marginally reactive. The autoclave test performed in this study was found to give results as good as or even better than the AMBT while taking only three days to complete (12).

Figure 4 shows the 1-year expansion values obtained in the "modified" ASTM C 227 Mortar Bar Method (see Table 1) plotted against the 14-day expansion values obtained in the AMBT. Empty and filled symbols correspond to samples that have caused concrete prism expansion < 0.04% and > 0.04%, repectively, at both the 6-month and the 1-year time period. Grey symbols represent aggregates that have induced concrete prism expansion < 0.04% at 6 months and > 0.04% at 1 year; these are considered as marginally reactive. A grey zone is observed for ASTM C 227 expansion values falling in the interval 0.05 to 0.10%, while 1-year expansion values > 0.10% correspond to reactive aggregates. The same 0.10% expansion limit criterion for reactive aggregates and 0.05 - 0.10% expansion "grey interval" are also observed for mortar bars immersed for 6 months in 1N NaOH at 38°C (Fig. 5A). Using the 1-year expansion values for the latter group of bars simply shifts the grey zone up to the 0.10 - 0.20% expansion interval (Fig. 5B). Figures 4 and 5 indicate that relatively useful and reliable limit criteria could be drawn from the results obtained with the "modified" ASTM C227 Mortar Bar Method and with mortar bars immersed in 1N NaOH at 38°C. However, the lapse of time required to obtain these results (1 year in the former case and 6 months in the latter) does not allow them to compete with the more realistic and reliable Concrete Prism Test.

Figures 3 to 5 indicate that the expansive behavior of the various carbonate aggregates investigated in this study was rather consistent when determined through the various mortar bar tests performed despite the major differences in the curing conditions used. However, as illustrated on Fig. 1A and 1B and reported elsewhere (1,8,12), much more dispersion in the data has been observed when the expansion values measured on mortar are compared to those obtained from the concrete prism test or in the field. Indeed, a number of siliceous and argillaceous limestone aggregates from the eastern part of the St. Lawrence Lowlands (Zone 1 on Figs. 1A and 1B) were observed to give anomalously high expansion in the AMBT (and also in the various mortar bar tests performed) with respect to the expansion values obtained for the highly reactive aggregates from the Trois-Rivières and Ottawa areas (Zone 2). The reason for this behavior is still not known, but is real; moreover, the same group of aggregates also produced abnormally high dissolved and corrected dissolved silica values (Sc and Sc*, measured on the insoluble residues of the carbonate rocks) (Fournier and Bérubé (13)) and amounts of gel in a modified Gel Pat Test (Fournier and Bérubé (14)). Such abnormally high expansions found with mortar bar test, compared to field performance or concrete prism test results, have also been reported for a number of silicate rocks (11). Since time is unfortunately often the major factor dictating the choice of AAR test to be performed, great care should be taken when estimating the potential reactivity of an aggregate or the expansion it could generate in concrete based on accelerated mortar bar test results (1,12).

Rate of expansion

Figures 6A to 6D show the "normalized" rates of expansion for reactive aggregates in the various mortar bar and concrete prism tests performed in this study, expressed in the percentage of the critical expansion value reached as a function of time. The so-called "critical expansion values" are those obtained at 14 days in the AMBT, and at 1 year in the other three tests. The minimum and maximum percentage values obtained at each specified time are also given on figs. 6A to 6D. Relatively small differences between the maximum and minimum percentage values are observed at each particular time for all the three mortar bar tests, which indicates that the expansion rates are similar for most of the different reactive carbonate rocks tested under the particular curing conditions used. Fournier and Bérubé (1) have shown that variations in the expansion rates are much more pronounced for the non-reactive carbonate aggregates tested in this study; but, a good indication of the 14-day and even the 28-day expansion values in the AMBT can be obtained after only 7 days of testing (Fig. 7). Most of the potentially deleterious samples investigated induced mortar bar expansion in excess of 0.05% after 7 days, with those producing 7-day expansion values > 0.15% also reaching expansions > 0.25% after 14 days. Rates of expansion in the concrete prism test are much more variable from one aggregate to another (larger minimum-maximum differences on Fig. 6D). This suggest that the rate at which a reactive aggregate will expand in the concrete prism test is influenced much more by parameters such as the inherent reactivity, porosity and permeability of the aggregate particles (other testing conditions and concrete mixture characteristics being constant) than in mortar bar tests. Figure 6E shows the average percentage values of the 4 tests presented on Figs. 6A to 6D plotted on the same graph. Very similar average percentage values are observed for the 3 mortar bar tests performed, which suggests that: [1], even if the reaction-expansion processes are highly accelerated in the AMBT, the rate of expansion within the 14-day suggested test period is similar to those obtained in less severe and long-term mortar bar tests; and [2], the high-temperature curing conditions used in the AMBT do not promote a misleading behavior. These remarks apply also to the autoclave mortar bar test performed in this study, for which a 5-hour steam curing period is used to accelerate AAR processes.

Figures 6C, 8A and 8B show the normalized rates of expansion obtained in the AMBT for the reactive carbonate rocks investigated in this study, and those obtained for a number of very reactive to marginally reactive silicate rocks and gravels tested by Ouellet (15) and Mongeau (16), respectively. The percentage values obtained for the silicate rocks (Fig. 8A) vary slightly more in the first few days than those calculated from the reactive carbonate rocks tested (Fig. 6C). The average percentage values for those two groups of rocks are, however, almost identical. The gravel aggregates gave a much more variable behavior (Fig. 8B). This is possibly related to the various proportions and the variable inherent reactivity (which is related to the petrographic nature) of reactive particles within gravel samples.

## Decision Chart

The results of the various AAR tests performed in this study combined with the condition survey of a large number of concrete structures have permitted drawing the decision chart presented on Fig. 9 for determining the potential alkali-reactivity of carbonate aggregates in the St. Lawrence Lowlands. Petrographic examination is an essential first step to any AAR testing program. Even if it rarely permits, by itself, the ready classification of an aggregate as non-reactive or reactive, petrographic examination is often crucial when selecting the testing method(s) to be used. Fournier and Bérubé (1,8) have indicated that the geological association of the aggregate source, as well as petrographic characterization of the aggregate sample, may be used as a screening parameter for determining potential AAR in the St. Lawrence Lowlands. In addition, an insoluble residue content of 5% was also found to be a good screening criterion for non-reactive aggregates. The results presented in this papers and others (1,8,12) suggest that the potential alkali-aggregate reactivity of St. Lawrence Lowlands carbonate aggregates can be reliably evaluated in the laboratory using at least two of the following test methods: the AMBT, Autoclave Mortar Bar Test and Concrete Prism test. The revised version of the Canadian Standards on AAR (in preparation) suggests using the AMBT as a tool for accepting but not rejecting aggregates. The results obtained in this study have indicated that accelerated mortar bar expansions < 0.10% at 14 days generally correspond to non-reactive aggregates, while expansions greater than this value would require further testing using the Concrete Prism Test. Field performance observations in the St. Lawrence Lowlands have shown that aggregates inducing accelerated mortar bar expansion > 0.25% at 14 days generally behave deleteriously in concrete structures subjected to conditions promoting AAR. Such a limit criterion might sometimes be applied directly without making the Concrete Prism Test, provided the source of the aggregate and its petrographic nature strongly supports the AMBT results. The autoclave test was found as effective as or even better than the AMBT for properly identifying non-reactive and reactive aggregates (Bérubé and Fournier 17)); however, the former is only an experimental test, so its use at present is limited. The chart proposed on Fig. 9 for potential alkali-reactivity determination is based on specific types of concrete aggregate, and its application to other rock or aggregate types may lead to misleading results.

## CONCLUSIONS

a) The Accelerated Mortar Bar Test and the proposed Autoclave Mortar Bar Test can be used as preliminary methods (concurrently with petrographic examination) to determine the potential alkali-reactivity of carbonate aggregates produced in the St. Lawrence Lowlands. In general, carbonate aggregates with less than 5% insoluble residue or which produce expansion values < 0.10% after 14 days in the AMBT, or < 0.15% in the autoclave test, can be considered as non-reactive aggregates. The condition survey of concrete structures in the St. Lawrence Lowlands has indicated that aggregates which cause expansion > 0.25% in the corresponding time-limit proposed for either the AMBT or the Autoclave mortar bar methods have behaved deleteriously in concrete structures subjected to conditions promoting AAR.

b) Reliable results were obtained with the ASTM C 227 Mortar Bar Method provided that [1], the total alkali content of the mixture was raised to 1.25% ($Na_2O$ equivalent) by adding NaOH to the mixture water, and [2], containers with no absorbent materials were used. Mortar bars immersed in 1N NaOH solution at 38°C were found to reach the 1-year ASTM C 227 expansion level between 140 and 168 days. In addition, the 14-day expansion values obtained with the AMBT were found to correlate well with the 5-hour expansion values measured in the Autoclave Mortar Bar Test.

c) Care should be taken in estimating the degree or extent of expansion to be expected in concrete elements subjected to field exposure conditions when using the expansion values measured in the AMBT (and the autoclave test).

d) Similar "normalized" rates of expansion (i.e., the percentages of the "critical expansion values" reached as a function of time) were found for the three mortar bar tests made in this study. This similarity suggests that: [1], even if the reaction-expansion processes are highly accelerated in the AMBT, the rate of expansion within the 14-day suggested test period is similar to those obtained in less severe and long-term mortar bar tests; and [2], the high-temperature curing conditions used in the AMBT do not promote a misleading behavior.

e) A good indication of the 14-day and even the 28-day expansion values measured in the AMBT test, and consequently of the potential alkali-reactivity of the investigated carbonate aggregates, can be obtained after only 7 days of immersion in the NaOH solution.

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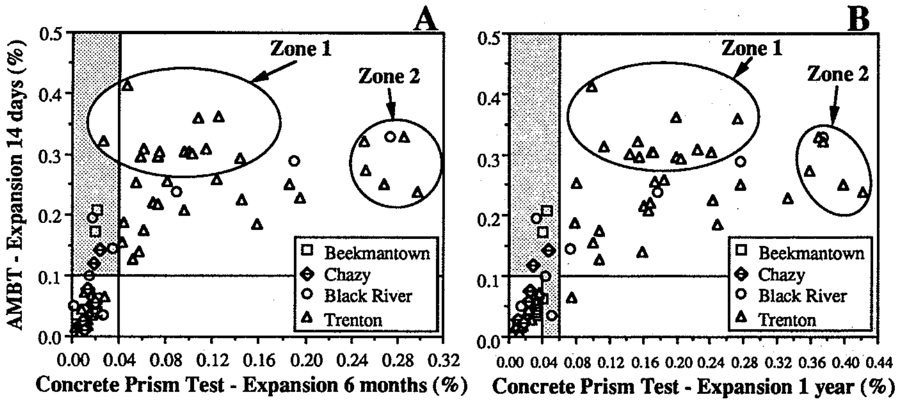


Figure 1 - Expansion values at 14 days in the AMBT as a function of the 6- month (A) and 1-year (B) expansion values measured in the Concrete Prism Test. Results are grouped according to the geological association of the samples.

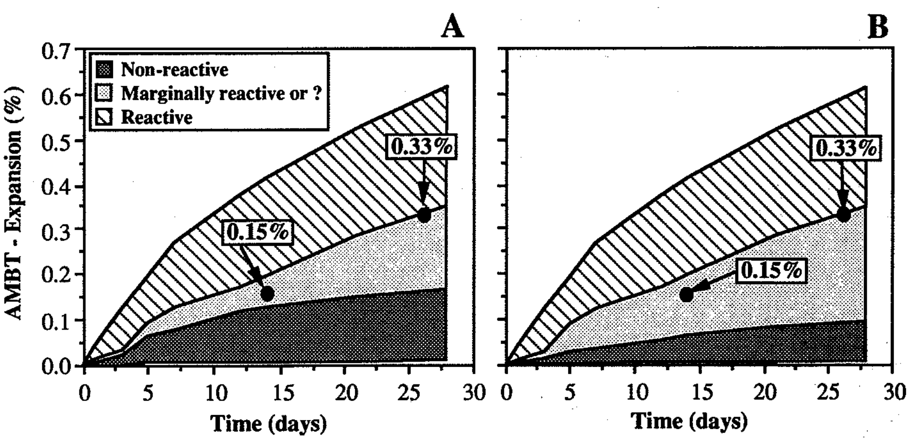


Figure 2 - Suggested "reactivity zones" for St. Lawrence Lowlands carbonate aggregates, as determined by the AMBT results. Concrete prism expansion limit-criteria of 0.04% at 6 months (A) and 0.06% at 1 year (B) were used to distinguish marginally reactive and reactive aggregates from non-reactive ones.

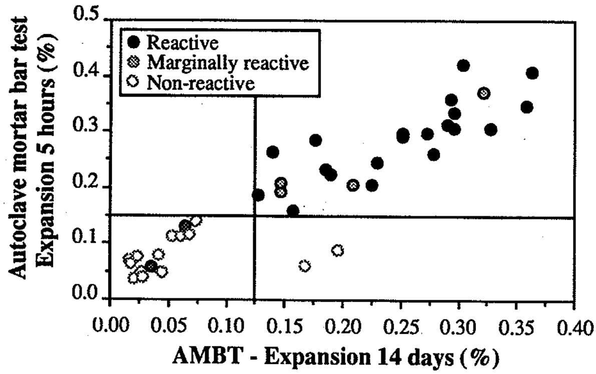


Figure 3 - Expansion values measured after 5 hours of steam curing (0.17MPa - 130°C) in the proposed autoclave test plotted against the 14-day expansion values obtained in the AMBT.

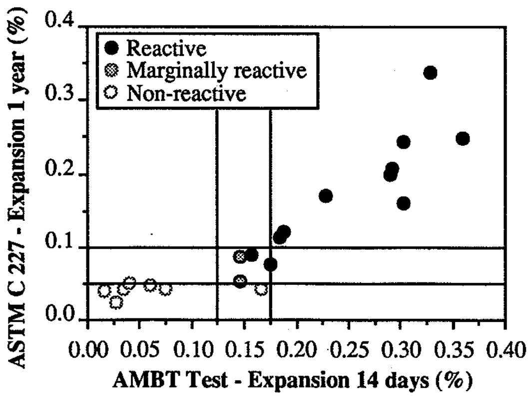


Figure 4 - Expansion values measured after 1 year in the ASTM C 227 Mortar Bar Method plotted against the 14-day expansion values obtained in the AMBT.

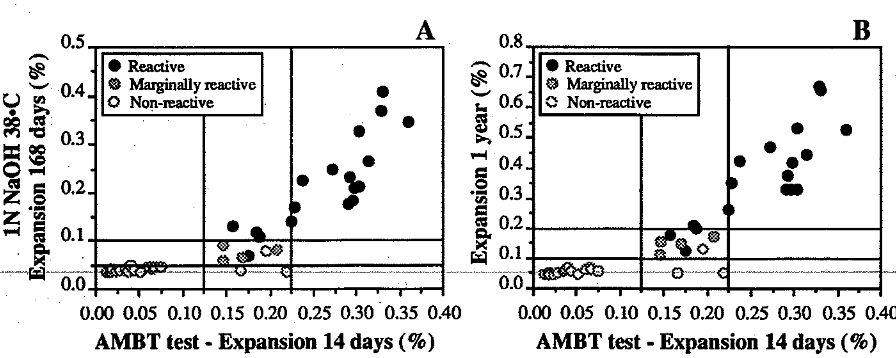


Figure 5 - Expansion values measured for mortar bars immersed for 168 days (A) and 1 year (B) in a 1N NaOH solution at 38°C plotted against the 14-day expansion values obtained in the AMBT.

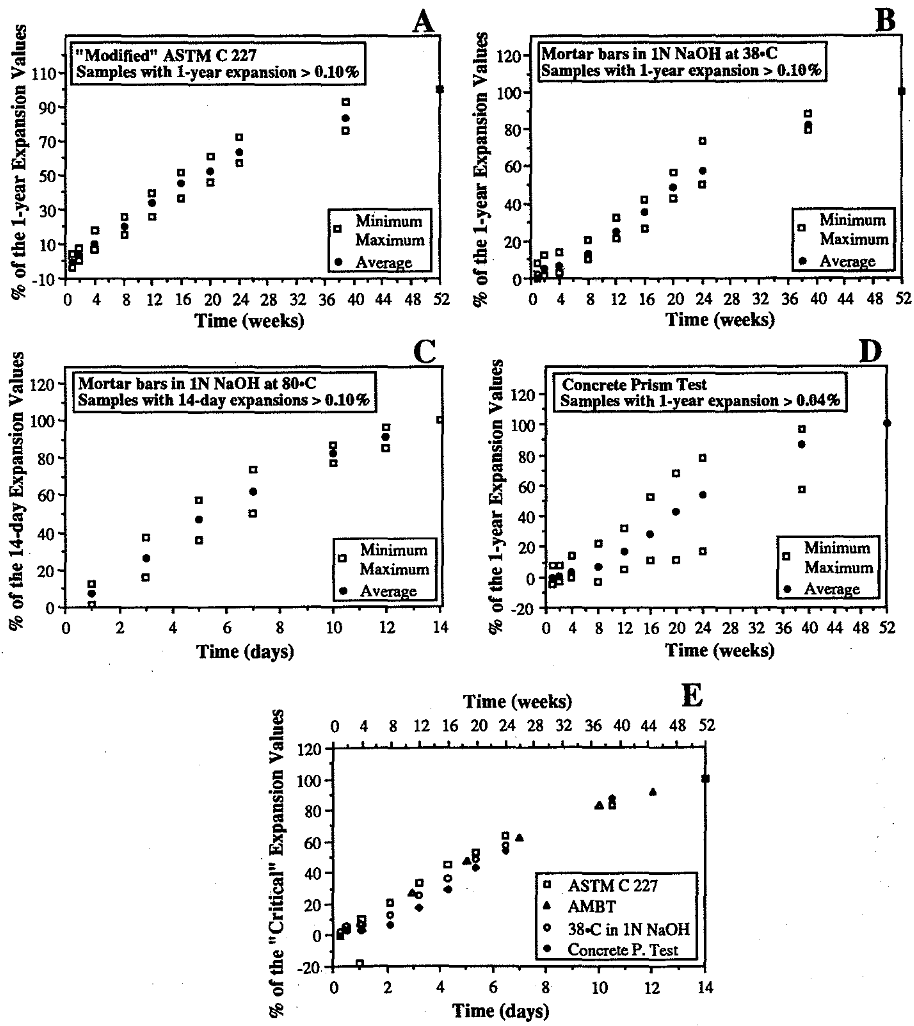


Figure 6 - Average "normalized" rate of expansion for the different mortar bar and concrete prism tests performed in this study, expressed as the percentage of the "critical" expansion value reached as a function of time. The maximum and minimum percentage values obtained at each time are also given.

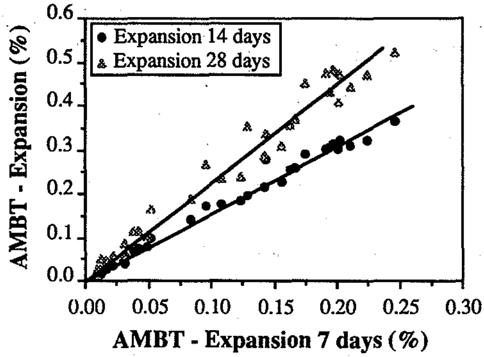


Figure 7 - Relation between the 7, 14 and 28-day expansion values obtained in the AMBT.

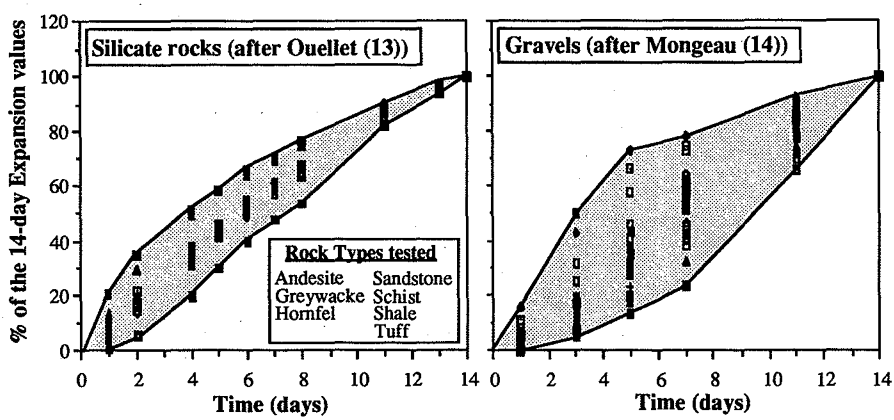


Figure 8 - Average "normalized" rate of expansion calculated from AMBT results obtained on a series of silicate rocks and gravels, expressed as the percentage of the 14-day expansion value reached as a function of time. The maximum and minimum percentage values obtained at each time are also given.

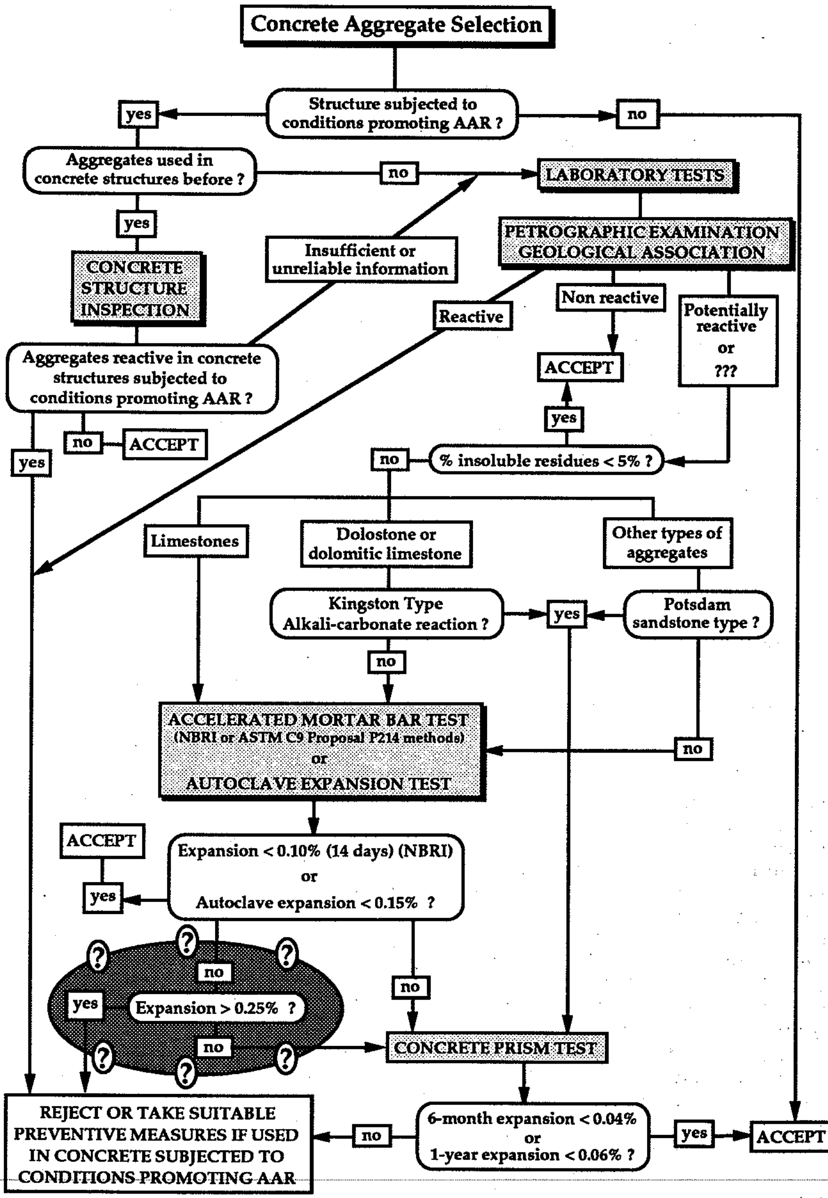


Figure 9 - Decision chart proposed for determining the potential alkali-reactivity of concrete aggregates in the St. Lawrence Lowlands.