SELECTION OF SILICEOUS AGGREGATE FOR CONCRETE

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ABSTRACT

Alkali-aggregate expansive reactions are one of the potentially serious degradation problems that could affect the structural stability of underground concrete structures for disposing of low-level radioactive waste (LLW). It appears that all aggregates react to some degree with alkalies in cement. In the majority of cases the reactions are beneficial (e.g., increasing the bond between aggregate and hydrated cement paste) or innocuous. In some cases, however, the reactions result in the formation of expansive products which can cause serious cracking of the concrete.

This report deals with the selection of siliceous aggregates to avoid deleterious alkali-aggregate expansions. Current practices used to prevent expansive alkali-silica reactions and the standard test methods used to identify reactive aggregates are first discussed. Then the results of a study on using a new alkali-silica reactivity test to select siliceous aggregates for use in the concrete of LLW storage structures are presented. It is recommended that siliceous aggregates, selected for constructing underground vaults for disposal of LLW, have an expansion of less than 0.10 percent using the new test.

KEYWORDS: Aggregate; alkalies; alkali-aggregate reaction; concrete; durability; siliceous aggregate; testing.

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1. INTRODUCTION

1.1 Background

Alkali-aggregate expansive reactions are one of the potentially serious degradation problems that could affect the structural stability of underground concrete structures for disposing of low-level radioactive waste (LLW). In these reactions, alkali hydroxides in the concrete react with certain minerals in aggregates, in the presence of moisture, to form expansive products. If sufficient amount of expansive product is formed, cracking of the concrete could occur.

It appears that all aggregates react to some degree with alkalies in cement. In the majority of cases the reactions are beneficial (e.g., increasing the bond between aggregate and hydrated cement paste) or innocuous. In some cases, however, the reactions result in the formation of expansive products which can cause serious cracking of the concrete. The expansion is attributed to the imbibition of water by the reaction products. When cracking occurs its effects can range from being cosmetic (in most cases) to resulting in structural failure (uncommon) of concrete. Cracking of concrete even though not causing structural collapse can significantly increase its permeability. Alkali-aggregate reactions may proceed rapidly (several months to a few years) or much slower (e.g., early signs observed only after some 20 to 25 years (1,2,3)).

Expansive alkali-aggregate reactions are known to occur with certain siliceous aggregates and certain limestone aggregates. Alkali-silica reactions are the most common and well studied aggregate reactions and has been found to occur in numerous places in the United States (4) with the greatest abundance of alkali-silica rocks being found in the western states (5). However, some slow reacting siliceous aggregates also have been found in the eastern states. Alkali-carbonate reactions have occurred in some midwestern and eastern states (5).

This report deals with the selection of siliceous aggregates to avoid deleterious alkali-aggregate expansions. The selection of limestone aggregate will be addressed by a future report. Current practices used to prevent expansive alkali-silica reactions and the standard test methods used to identify reactive aggregates are first discussed. Then the results of a study of the feasibility of using the new alkali-silica reactivity test to select siliceous aggregates for use in the concrete of LLW storage structures are presented.

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2. CURRENT PRACTICES

Current practices to prevent expansive alkali-silica reactions and standard tests to identify reactive aggregates are briefly described. Their applications to ensuring the durability of underground concrete structures for disposing of LLW are also addressed.

2.1 Prevention of Alkali-Aggregate Expansive Reactions

Methods for preventing alkali-aggregate expansive reactions are given in detail in the ACI Guide to Durable Concrete (5). Recommendations based on these methods are given in expert system format in Durcon (6). Obviously, the best method for preventing alkali-aggregate reactions is to use a nonreactive aggregate. Another obvious method is to keep concrete dry, which may be virtually impossible to accomplish for many years when the concrete is buried in the ground.

Because the alkali-silica reaction is sensitive to the pH of the pore solution, designing a low-alkali containing concrete is often an effective measure. The alkali content can be kept low by the use of "low-alkali" cement (maximum of 0.60% equivalent sodium oxide according to ASTM C 150 (7)). However, for underground concrete, external sources of alkalies must be considered. For example, alkali salts in penetrating groundwater could react with calcium hydroxide, formed by the hydration of cements, to form alkali hydroxides. The reaction involving sodium sulfate is:

$$Na_2SO_4 + Ca(OH)_2 --> 2NaOH + CaSO_4. \tag{1}$$

Certain pozzolans, such as silica fume and fly ash, have been found effective in reducing expansion due to alkali-aggregate reactions to an acceptable level. They appear to reduce expansion by reducing the alkalinity of the concrete pore solution and by reducing the permeability of the concrete (8). However, their effectiveness depends on their alkali contents. For example, it has been observed that use of a high alkali containing silica fume material increased the mortar-bar expansion compared with the neat portland cement (9). Also, it is not known if they can prevent alkali-aggregate reactions in concrete which may be in contact with groundwater containing alkali salts for hundreds of years.

2.2 Determination of Aggregate Reactivity

Decisions on the potential alkali-silica reactivity of an aggregate are sometimes based on its service records performance. Several decades of recorded satisfactory performance should be available before predicting that aggregates will be free from

expansive reactions. Even then, testing of the alkali reactivity of the aggregate, to be used in constructing concrete vaults for LLW disposal, is recommended.

Laboratory investigation of the potential reactivity of silica-containing aggregates usually consist of petrographic examination, mortar bar test, and chemical test, or their combination.

2.2.1 Petrographic Examination. ASTM C 295 (10) provides a standard practice for the petrographic examination of aggregates. It can give information on the potential reactivity of an aggregate based on identification of the minerals present. If present in detectable quantities, presence of the well-known reactive minerals may be identified by an experienced petrographer. However, some minerals can be present in small quantities which escape detection but which are sufficient to result in concrete cracking (11).

2.2.2 Mortar-bar Test for Potential Reactivity. The ASTM C 227 (12) mortar bar test is most generally relied upon to indicate potential alkali reactivity. The test usually is completed within a year, sometimes six months of testing is adequate. However, certain metamorphic siliceous rock could require longer test times (possibly up to 3 years) and elevated temperatures to give evidence of expansion (13). If carried out for a sufficient testing period, the test is usually reliable in determining if an aggregate is potentially reactive, although some reactive aggregates appear to pass the test (14). The test does not always give a reliable estimate of the extent of expansive reaction that could occur in field concrete with a potentially reactive aggregate (14).

2.2.3 Chemical Test for Potential Reactivity. This test (ASTM C 289 (15)) is used primarily for a quick evaluation of the potential reactivity of an aggregate. The method measures the amount of silica dissolved in a concentrated sodium hydroxide solution and the reduction in alkalinity. The data is then correlated with aggregate reactivity using standard curves. Results can be obtained in a few days. The results usually correlate with aggregate behavior in the mortar bar expansion test, but it is not suitable for predicting the amount of expansion occurring in concrete due to alkali-aggregate reactions (8).

2.2.4 Combination of the Methods. The U.S. Corps of Engineers' Engineering Manual (16) concludes "that none of the available means of evaluation can be relied upon independently, or collectively, to provide an unquestionably positive answer to the problem of potentially harmful reactivity of an aggregate whose constituents include materials capable of reacting with the alkalies in portland cement." The results of the petrographic

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examination with corroborating evidence from the mortar-bar test
are considered to be the most reliable indicators of potential
reactivity. It is further stated that "Spurious results are
possible with the chemical test when certain substances are
present in the aggregate being tested." Thus the chemical test
should be used in conjunction with the petrographic examination.

Based on the analyses of the above standard test methods, it
appears they do not provide a basis for predicting the long-term
expansive characteristics of concrete containing siliceous
aggregates buried underground.

3.   NEW ACCELERATED ALKALI REACTIVITY TEST

3.1 Need for New Test

Based on the above discussion of test methods it appears that
correlations between accelerated laboratory expansion or chemical
reactivity tests and field expansions of concrete caused by
alkali-aggregate reactions have not been adequately established.
Also, it appears that the current standard tests may not always
identify reactive siliceous aggregates, at least not within the
prescribed testing times. A silica-containing aggregate which
passed the ASTM C 227 test, but is known from its field
performance to be reactive, was included in the present
laboratory investigation of a new test method.

Because controlling the alkali contents of cements and concrete
may not be possible if alkali-containing ground water is present,
it becomes necessary to identify or develop a test that will
indicate with reasonable assurance that an aggregate will be
unreactive, in the presence of alkali hydroxides at the
concentrations to be expected for concrete buried in the ground
(17). Such a test will assist in the development of a basis for
predicting the service life of underground concrete vaults in the
sense that concrete made with a non-reactive aggregate will not
experience alkali-aggregate expansive reactions.

3.2 Conceptual Testing Approach

One approach for testing the reactivity of siliceous aggregates
involves the determination of their chemical reactivity with
alkali hydroxides by preparing concrete with, or exposing it, to
a higher alkali content than that of the concrete to be used in
actual construction. The concrete would be exposed to elevated
temperatures and moisture and, after a specified testing time,
the occurrence of expansive reactions determined, directly or
indirectly (17). A direct determination could involve the
microscopic examination of the aggregate for reaction products.

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The presence of reaction products would indicate that the aggregate is reactive. Because almost all aggregates containing siliceous minerals may show some reaction in this test, aggregates of concretes which have been in service for many years without expansion occurring due to alkali-aggregate reactions could be subjected to the test and the extent of reaction determined. This would provide baseline data for evaluating the results of testing potentially reactive aggregates. In the indirect method of determining reactivity, the expansion of concrete immersed in an alkali hydroxide solution at elevated temperatures could be measured. An accelerated test based on the indirect method has been developed by Oberholster and Davies (14). This method is both a Canadian Standards Association (18) and an American Society for Testing and Materials (ASTM) proposed standard test method (19).

3.3 New Accelerated Testing Method

The new accelerated test method for determining if a silica containing aggregate is reactive involves the following five steps (14, 19): (i) the crushing of aggregates; (ii) preparation of mortar bars using the crushed aggregate; (iii) stripping the molds after one day and immersing the specimens in water which is then heated to 80 °C; (iv) after 24 hours of immersion, the specimen lengths are measured and then they are immersed in a 1N sodium hydroxide (NaOH) solution maintained at 80°C; and (v) measuring expansions for 14 days. At least three intermediate readings are made after the initial reading.

The test results are interpreted as follows (19): an expansion of 0.20% after 14 days of immersion in 1N NaOH indicates a potentially deleterious expansion; an expansion of less than 0.10% is indicative of innocuous behavior; and if the expansion is between 0.10 and 0.20% the results are considered to be inconclusive. If the expansions are between 0.10% and 0.20%, the proposed ASTM test method recommends that supplementary information be developed to confirm that the expansion is actually due to alkali reactivity. Suggested sources of such information include: (1) petrographic examination of the aggregate according to ASTM C 295 (10); (2) examination of the specimens after test, according to ASTM C 856 (20), to identify the products of alkali reactivity; and (3) tests of the aggregate for potential reactivity by chemical methods, according to ASTM C 289 (15).

3.3.1 Laboratory Testing

The practicability of the new accelerated test method was investigated by using it to evaluate the alkali susceptibility of selected aggregates.

3.3.1.1 Materials

A Type I portland cement was used in the study. Its oxide composition is given in Table 1. The cement was obtained from the ASTM-sponsored Cement and Concrete Reference Laboratory at the National Institute of Standards and Technology.

Three natural aggregates and one synthetic aggregate were used. The natural aggregates are from Canada and were tested because, for two of them, their long-term field performances are well documented. Also, the natural aggregates have been included in an interlaboratory study (21). The aggregates are briefly described in Table 2; a more detailed description is provided by Rogers (22). Pyrex glass meeting the requirements of ASTM C 441 (23) was the synthetic aggregate.

Mortar bars, 25 x 25 x 285 mm, were prepared by the procedures given in ASTM P 214 (19), with a water-to-cement ratio of 0.5 and an aggregate-to-cement ratio of 2.25.

3.3.1.2 Testing Procedures

The testing procedures were essentially the same as those given in the proposed ASTM test method (19). Some of the testing practices adapted by the National Institute of Standards and Technology laboratory and observations made on testing conditions are presented.

Nalgene containers[1,2] were used to immerse the test bars in either water or the sodium hydroxide, NaOH, solution. A silicone high vacuum grease was used to seal the container lid to minimize loss of solution. There were two bars and 1711 g of the 1N NaOH solution in each container, which resulted in about a 10 mm cover

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[1] Nalgene Instrument /Pipet Sterilizing Pan, Cat. No. 6910-0618, with approximate inside dimensions of 60 mm deep x 121 mm wide x 432 mm long, were found satisfactory.

[2] Materials or equipment are identified in this paper to adequately specify the experimental procedure. Such identification does not imply and endorsement by the National Institute of Standards and Technology.

of solution over the top of the bars. To maintain the appropriate ratio of masses of mortar and solution, two bars were always placed in each container, even if one of the bars had broken during stripping. The bars were supported by thin shims, made of the same material as that of the containers. The weight of the container, its lid and vacuum grease, and contents (two bars, NaOH solution, and shims) was monitored during the test period to determine the loss of any water, which could result in an increase in the NaOH concentration. It was found that, as much as 9 percent, by mass of the solution, was lost over the 14 day immersion-testing period.

A Mitutoyo IDC Series 543 Digimatic Indicator[2], with a digital display and having a readout to within 1×10^{-5} in (2.5×10^{-4} mm) was used to measure the length of the bars. Use of the indicator permitted measurements of bar lengths to be made about 15 seconds after their removal from the NaOH solution. Rapid length measurement is advisable because it was found that while making measurements, the temperature of the NaOH solution decreased by as much as 2 to 3 C/min when the container was removed from the oven and its lid removed.

Stainless steel gauge studs were used and did not appear to have chemically reacted with the NaOH solution. A non-corrosive spray-applied tetrafluoroethylene telomer mold release agent was used. It did not always perform satisfactorily, as several bars broke while being stripped from the molds at an age of 1 day.

For each aggregate tested, there were either one or three batches made. With each batch, 4 bars were cast; in some cases, a bar broke during stripping the bars from their molds, leaving 3 bars to be tested. The bars were immersed in a 1N NaOH solution maintained at 80 C and their lengths were measured on at least five days during the 14 day immersion test.

3.3.2 Results and Discussion

The average expansions generally increased with time for three of the aggregates, as shown in Fig. 1 where the data for one batch of each aggregate is presented. Table 3 gives the percent expansion values for individual bars for each of the batches (A, B, and C) and the average, standard deviation, and coefficient of variation for the test results from each batch. With the exception of the Nelson aggregate, the coefficient of variations within a batch were considered acceptable, ranging from 1.03 to 18.2%. The magnitude of the coefficient of variations from the Nelson aggregate has little significance because the average expansions were small, between 0.00270 and 0.0142%, and relatively small variations appear large compared with the near-

zero average values. A small experimental error (e.g., difference in temperature of specimens) could significantly affect the within-batch coefficient of variation for the Nelson aggregate. The coefficient of variations for among-batch data for the Spratt and Sudbury aggregates are 1.95 and 8.90%, respectively, indicating that the test is reasonably reproducible. The high among-batch coefficient of variation for the Nelson aggregate, 65.1%, was again attributed to a low expansion.

Based on the test results, the Spratt aggregate, the Sudbury aggregate, and the Pyrex glass are classified as being highly alkali reactive, while the Nelson aggregate is classified as being innocuously reactive. The findings for the natural aggregates are consistent with observations on their field performances (22). For example, the Sudbury aggregate has been used in over 26 highway structures and nearly all of them have exhibited damage due to alkali-aggregate reaction. This aggregate, however, has been found to pass the ASTM C 227 mortar bar expansion test (22).

The test procedures are straightforward and relatively inexpensive equipment is needed to perform the test.   Caution must be exercised in working with hot sodium hydroxide solution and the precautions given in ASTM P 214 (19) should be followed.

4. RECOMMENDATIONS FOR SELECTING AGGREGATES CONTAINING SILICA

In selecting silica-containing aggregates for constructing concrete structures for disposal of low-level radioactive waste, it is recommended that potential aggregates be subjected to both the ASTM C 227 mortar bar test (12) and the ASTM proposed standard test, ASTM P 214 (19), described in Section 3 of this paper. ASTM C 227 should be used in parallel with the proposed test method, until the proposed test becomes a standard test method. ASTM P 214 appears to be more reliable than ASTM C 227 (14). An aggregate should pass both tests to be accepted, if it fails either test it should be rejected.

Regarding the maximum acceptable limit for the ASTM P 214 test, the authors of this paper recommend that only aggregates with test expansions of less than 0.10% be selected for construction of underground concrete structures for disposal of low-level radioactive waste. Further, if any expansion measurement exceeds 0.10% during the test time, the aggregate should be rejected. The lower the expansion, the more assurance that an aggregate is nonreactive. If a choice exists between aggregates with expansions less than 0.10%, assuming that their other properties are adequate, the aggregate with the lowest expansion should be selected. With regards to an acceptable limit for ASTM C 227, it has been recommended that for critical structures, such as those

used for nuclear containment, that the maximum expansion limits be 0.05% at six months and 0.10% at one year (25).

5. RECOMMENDED RESEARCH

It is likely that the smaller the expansion of a siliceous aggregate in the ASTM P 214 test (19), the smaller the amount of expansive reaction potential in field concrete. Further research should be carried out to determine if the expansive limit of 0.10% will give a reasonable assurance of non-reactivity for aggregates of concrete used for LLW disposal. If it is decided that the 0.10% expansion level is too high, then a lower limit would need to be established.

While it appears that the degradation process simulated by the procedure in ASTM P 214 is similar to that occurring in concrete in the field (14), further verification research is recommended.

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Table 1. Chemical Composition of Type I Cement
Used in Mortar Bars

Constituent	No. of Testing Laboratories	Average (Percent)	S.D.[a]	C.V.[a]
SiO_2	136	19.96	0.22	1.10
Al_2O_3	149	6.11	0.28	4.63
Fe_2O_3	150	2.36	0.065	2.76
CaO	144	61.87	0.56	0.91
MgO	144	3.02	0.14	4.65
SO_3	134	3.22	0.10	3.14
Loss on Ignition	87	1.58	0.098	6.21
Na_2O	109	0.386	0.043	11.2
K_2O	147	0.747	0.039	5.17
Mn_2O_3	40	0.114	0.0093	8.15
P_2O_5	52	0.279	0.038	13.5
TiO_2	64	0.327	0.015	4.68

[a] S.D = standard deviation; C.V. = coefficient of variation.

Table 2. Description of Aggregates Used

SPRATT AGGREGATE (22)
The Spratt aggregate is from a horizontally bedded limestone quarry, containing 3 to 4% microscopic chalcedony and black chert with a conchoidal fracture. The rock consists of calcite and small amounts of dolomite with an insoluble residue of 10%. The insoluble residue consists of quartz with small amounts of illite and pyrite.

This rock has been used in some concrete construction where damage due to alkali-silica reaction is obvious. The performance of concrete structures made with these similar aggregates is generally unsatisfactory. Within ten years, cracking and distress due to an alkali-silica reaction is apparent.

SUDBURY AGGREGATE (22)
This aggregate is a crushed gravel. It has been used in over twenty-six highway structures. Nearly all structures show damage due to alkali-aggregate reaction. The gravel contains about 75% reactive particles which are: argillite, greywacke, sandstone, arkosic sandstone, quartzwacke, and quartzite.

A feature of this type of aggregate is that it does not produce excessive expansion in the mortar bar expansion test (ASTM C-227). Usually expansion testing in concrete prisms stored over water at 38°C will give expansion if the alkali content is sufficiently high (>5 kg/m^3). Another unusual feacture of this aggregate is the rather small amounts of visible alkali-silica gel in damaged concrete.

NELSON AGGREGATE
The Nelson aggregate is a "non-reactive" calcitic dolostone standard laboratory coarse aggregate. It was chosen because it was known to be essentially non-reactive.

PYREX GLASS AGGREGATE
The pyrex glass was obtained as a lump cullet, No. 7740, from Corning Glass Works, Corning, NY. The glass met ASTM C441 [23]. Lump cullet refers to waste glass from the manufacturing process, either to the residual melt left during a batch process or to broken pieces of finished glass, typically rods and tubes, that are added to the melt [24].

Table 3. Percent Expansion Values Based on 14 Day Immersion in 1 N NaOH Solution at 80°C

	Aggregate									Pyrex Glass		
	Spratt			Sudbury			Nelson			Batch A		
	Batch A	B	C	Batch A	B	C	Batch A	B	C			
Bar No.												
1	0.353	0.353	0.358	0.301	0.233	0.245	0.0147	0.00670	-0.00250	0.524		
2	0.372	0.354	0.364	0.308	0.332	0.237	0.0125	0.0112	0.00450	0.574		
3	0.385	0.361	0.374	0.263	0.242	0.222	0.0155	0.00950	0.00250	0.580		
4	--	0.358	0.374	0.250	0.237	--	--	0.0123	0.00630	0.602		
Within[a] Batch $\bar{x}$	0.370	0.357	0.367	0.281	0.261	0.235	0.0142	0.00993	0.00270	0.570		
s	0.0162	0.00367	0.00781	0.0286	0.0473	0.0116	0.00155	0.00244	0.00380	0.033		
CV (%)	4.38	1.03	2.13	10.2	18.1	4.92	10.9	24.6	141.	5.79		
Among[b] Batch $\bar{\bar{x}}$	0.365			0.259			0.00895			--		
$S_{\bar{x}}$	0.00710			0.0230			0.00583			--		
$CV_{\bar{x}}$(%)	1.95			8.90			65.1			--		

a $\bar{x}$, s, CV(%) = average, standard deviation, and coefficient of variation values respectively based on 3 or 4 individual 14 day expansion values.

b $\bar{\bar{x}}$, $S_{\bar{x}}$, $CV_{\bar{x}}$ (%) = average, standard deviation, and coefficient of variation respectively based on three $\bar{x}$ values for Batches A, B, and C.
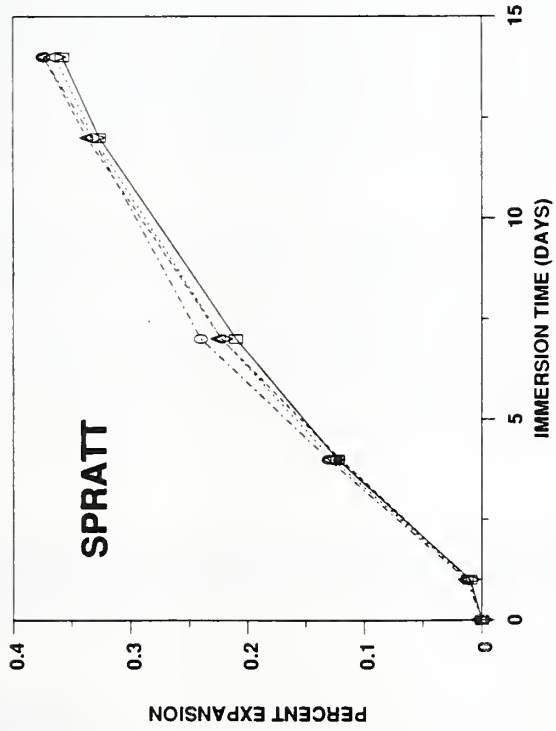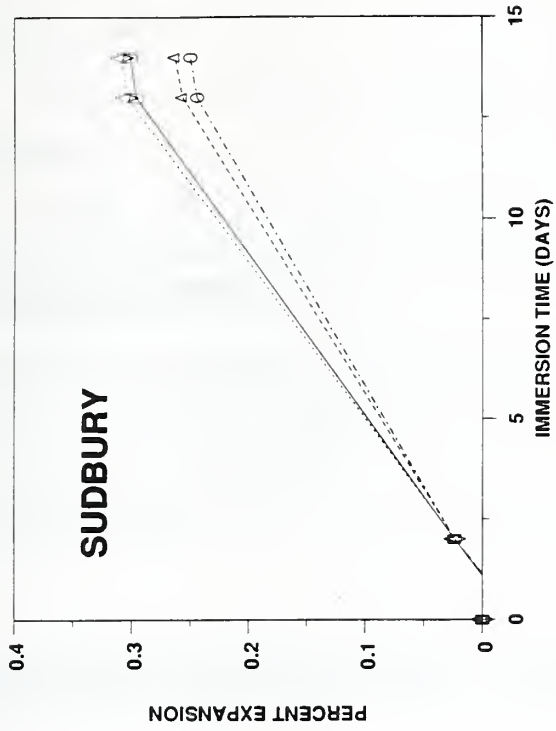
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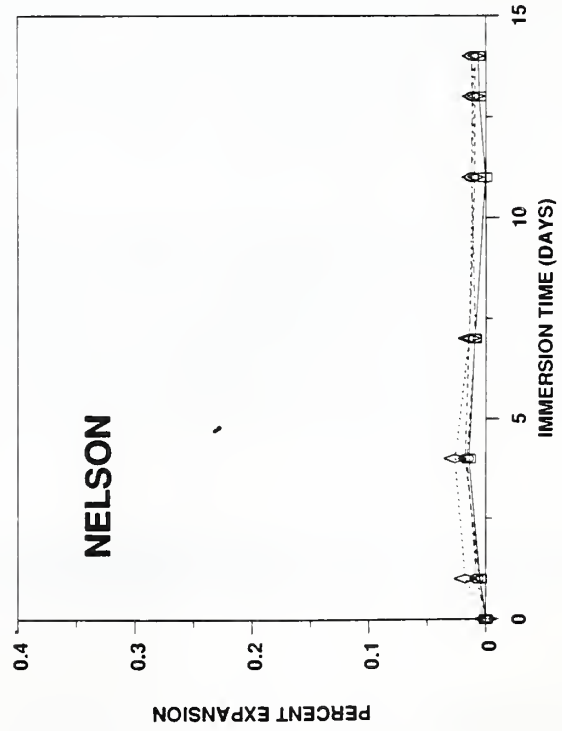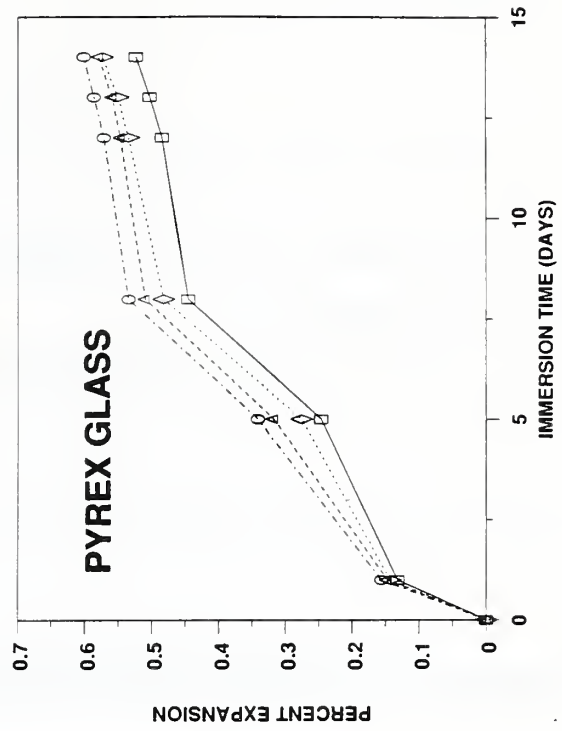Figure 1. The percent expansion versus the immersion time of the Spratt (a), Sudbury (b), Nelson (c), and Pyrex glass (d) aggregates in a 1N NaOH solution. Data from four mortar bars cast from one batch are shown.

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10. SUPPLEMENTARY NOTES

☐ DOCUMENT DESCRIBES A COMPUTER PROGRAM; SF-185, FIPS SOFTWARE SUMMARY, IS ATTACHED.

11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

Alkali-aggregate expansive reactions are one of the potentially serious degradation problems that could affect the structural stability of underground concrete structures for disposing of low-level radioactive waste (LLW). It appears that all aggregates react to some degree with alkalies in cement. In the majority of cases the reactions are beneficial (e.g., increasing the bond between aggregate and hydrated cement paste) or innocuous. In some cases, however, the reactions result in the formation of expansive products which can cause serious cracking of the concrete.

This report deals with the selection of siliceous aggregates to avoid deleterious alkali-aggregate expansions. Current practices used to prevent expansive alkali-silica reactions and the standard test methods used to identify reactive aggregates are first discussed. Then the results of a study on using a new alkali-silica reactivity test to select siliceous aggregates for use in the concrete of LLW storage structures are presented. It is recommended that siliceous aggregates, selected for constructing underground vaults for disposal of LLW, have an expansion of less than 0.10 percent using the new test.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

Aggregate; alkalies; alkali-aggregate reaction; concrete; durability; siliceous; aggregate; testing

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