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ScienceDirect
Cement and Concrete Research 38 (2008) 247–258
Cement and Concrete Research

Bridging the gap between research and standards

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Received 15 August 2007; accepted 10 September 2007

Abstract

Standards were developed and continued to exist to allow commerce. Minimum specifications are set for materials, and standard test methods are used to determine compliance with the specification limits. Materials specifications for cements and other concrete ingredients are referenced in concrete specifications, which are in turn referenced in building codes. A common complaint about standards is that they are not responsive to new research findings and lag behind new developments in materials and construction practices. However, standards do evolve over time as needs are seen to improve or add new tests methods or to set new specification limits. Another complaint is that historically prescriptive specifications should be switched to performance-based specifications so as not to limit development of alternative materials and construction methods. It should be noted that many standards writing organizations such as ASTM and CSA develop consensus standards, where volunteer members provide a balanced representation of a range of different interests, typically including both Producers and Users of a standard as well as General and Governmental agency interests. As a result of this balance, the wishes of one group alone can not unilaterally change standards against the majority interests. In this contribution, some both historical and recent developments in standards relating to cementitious materials are addressed, along with some thoughts on future directions. While some international standards are mentioned, it is acknowledged that this paper is largely limited to discussion of North American standards.

Keywords: Cement; Concrete; Composition; Sulfate resisting cement; Standards

1. Geoff Frohnsdorff and his role in promoting research on standards

Geoff Frohnsdorff was asked and accepted to co-author this paper. Unfortunately, shortly afterwards his health deteriorated and he died on March 5, 2006. A former chair of ASTM committee C01 on Hydraulic Cements, Geoff was a long-time proponent of advancing standards for cement and concrete through advancement of the material science basis for standards and use of modeling through such tools as the Virtual Cement and Concrete Testing Laboratory (VCCTL) being developed at NIST. He was a visionary, and his efforts are behind the success of the group of modelers at NIST now led by efforts of E. Garboczi and D. Bentz. Even on November 27, 2005, shortly before his death, he provided a memo along with a series of documents to the ASTM C01.92 Long-Range Planning subcommittee. Several of his stated objectives were: (a) that C01 standards should be science-based, user-friendly, with an emphasis on performance, preferably with ability to predict performance, but with prescriptive alternatives, (b) develop improved characterization techniques for cements and other concrete materials, (c) the need for putting cement and concrete data into database formats, (d) the need to develop databases, e.g., crystallographic, thermodynamic (solubilities, surface free energies), cement characteristics and performance, degradative reactions, reaction kinetics — should include potential cementing materials such as phosphates, aluminosulfates, carbonates, plumbates, and reactive fluids such as CO_2, sulfuric acid, phosphoric acid, and (e) the need for validated models, using the databases to predict performance. In his memo he regretted that his deteriorating health would prevent him from being at the next ASTM meeting.

Together with Jim Clifton (also since deceased) Geoff also organized and hosted an international workshop at NIST on *Cement and Concrete Standards of the Future* in October 1995, where issues such as performance standards, durability standards, and modeling were discussed [1].

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doi:10.1016/j.cemconres.2007.09.012

2. Recent developments in North American cement standards

2.1. Background

Cement standards have evolved from relatively simple requirements in the early 20th century, typically related to setting time, strengths at different ages, volume stability as well as prescriptive limits on chemical components. For example the 1927 version of the Canadian Engineering Standards Association (now CSA) A5 *Standard Specification for Portland Cement* had limits on allowable residue on a 75 μm sieve, Vicat setting time, a visual soundness test conducted on a pat of cement paste after steaming at 98–100 °C for 24 h, minimum tensile strengths of mortar briquettes at 7 and 28 days, as well as chemical limits on LOI, Insoluble Residue, SO_3, and MgO. Standards evolved from one type of cement to many types based on special performance objectives (e.g. low-heat, high-early strength, sulfate resistance) or based on composition (e.g. slag blended cement). In North America, the largest changes in cements have occurred due to increased alite contents and finenesses (resulting in higher-early strengths, which necessitated higher calcium sulfate additions, as well as changes in particle size distributions (due to use of more efficient separator circuits). While, as shown in Table 1, ASTM C 150 sulfate levels allowed in cements were increased several times as cement compositions and finenesses changed to allow better optimization of sulfate contents, more recently it was decided to leave the SO_3 limits alone, but allow them to be exceeded provided that it could be demonstrated that the optimum SO_3 content was above the stated limit (typically using ASTM C 563). In this case a 14-day mortar bar expansion test (ASTM C 1038, which had been previously adopted by CSA) was also required to be performed to show that the level of SO_3 in the cement would not result in adverse expansions from internal sulfate attack. Recent activity has been directed to widen the scope of the C 563 optimum sulfate test to allow for optimization of strength at ages other than one day). For example, in California, due to the arid climate, cements are typically optimized to reduce drying shrinkage, in order to meet the state-imposed shrinkage limits.

Table 1
Evolution of sulfate limits in ASTM C 150

Cement type	Type I	Type II	Type III	Type IV	Type V
1941 max SO_3 (%)	2.0	2.0	2.5	2.0	2.0
1946					
If $C_3A \leq 8\%$, max $SO_3\%$	2.0	2.0	2.5	2.0	2.0
If $C_3A > 8\%$, max $SO_3\%$	2.5	–	3.0	–	–
1953 all SO_3 limits dropped and C 265 0.50 g/L SO_3 limit adopted					
1955 SO_3 limits re-instituted but raised (C 265 was dropped)					
If $C_3A \leq 8\%$, max $SO_3\%$	2.5	2.5	3.0	2.3	2.3
If $C_3A > 8\%$, max $SO_3\%$	3.0	–	3.0	–	–
1960					
If $C_3A \leq 8\%$, max $SO_3\%$	2.5	2.5	3.0	2.3	2.3
If $C_3A > 8\%$, max $SO_3\%$	3.0	–	4.0	–	–
1971					
If $C_3A \leq 8\%$, max $SO_3\%$	3.0	3.0	3.5	2.3	2.3
If $C_3A > 8\%$, max $SO_3\%$	3.5	–	4.5	–	–
1978a — same table limits but a note was added that if optimum SO_3 by C 563 is higher than limits, the limits can be exceeded by up to 0.5% if C 265 value is less than 0.50 g/L at 24 h					
1989 — same table limits but C 265 was dropped and C 1038 expansion of 0.020% at 14 days was adopted. Also the 0.5% extra SO_3 limit was dropped.					

2.2. Re-thinking the split between specifications for cement and supplementary cementing materials

A problem that developed for historical reasons in both CSA and ASTM standards was the separation of standards for pozzolans and slag from those for cement. This division is considered to have been largely political as the cement industry in the 1950's to the 1970's was not interested in supplementary cement materials added separately at a concrete plant since they were seen, as a threat to cement markets. In fact, at ASTM the standards for pozzolans, slag and silica fume are still under jurisdiction of the concrete standards committee C09 rather than the cement standards committee C01. From a logical point of view, all of these materials should be considered as part of the cementitious materials, In recognition of this, in both CSA and ACI standards the term water to cement ratio (w/c) for concrete mix design has almost totally been replaced by water to cementitious materials ratio (w/cm) (without use of an efficiency factor concept as has been adopted in some countries).

In the 1970–80's with increasing awareness of energy issues, the cement companies in North America became involved with marketing SCM's and at CSA this resulted in more interest for integration of these standards into an overall cementitious materials standard.

In order to accomplish this objective, buy-in was needed from both user interest and general interest members in addition to producer members, so it was decided to move forward in stages. As a first stage, in 1998 a compendium volume was published which contained all of these individual standards together in one volume.

Once this was accepted, the next task was to integrate all of these standards into one document, as well as harmonizing the associated test methods. This task was completed with the issue of CSA A3000 in 2003 [2].

As well, the historical numeric designations of cement types (Types 10, 20, 30, 40, 50 which were similar to ASTM C 150 Types I, II, III, IV, and V, respectively) were altered to six designations based on desired performance. These were: GU: General Use, MH: moderate heat of hydration, LH: Low heat of hydration; MS: Moderate sulphate resisting, HS: highly sulphate resisting, and HE: High-early strength.

In addition, the blended cement specifications, which had been based on individual SCM's and replacement tests, were re-designated on the same basis as the Portland cements and with specification requirements altered to meet the same performance characteristics as their Portland counterparts. These blended cements are designated with the same symbols as those tested above except that the letter 'b' is appended (e.g. GUb) and on mill certificates, the SCM type and target replacement level is provided. The standard also allows blended cements to be comprised of binary, ternary or even quaternary mixtures as long as they meet the performance requirements. This has already resulted in the production of Portland-slag-silica fume as well as Portland-fly ash-silica fume ternary-blended cements.

The associated section of the CSA A3000 standard on SCM's [2] also allows for production of blended-SCM's for use at concrete plants. For example, in Ontario a high-alkali Class C fly ash has been successfully blended with a slag for many years, where the high calcium and alkali fly ash works synergistically to accelerate the slag hydration and the slag hydration utilizes the alkali, which reduces the risk of AAR associated with the utilization of the fly ash on its own.

2.3. Minor limestone additions

Similar to the European EN 197 and other international cement standards, since 1983 CSA has allowed 5% limestone to be interground or blended with clinker. Recently, new activity has been initiated due to interest in consideration of limestone cements (with 10 to 15% limestone) such as are currently allowed in EN197-1 [3].

In the US, after 3 attempts over a 20 year period, in 2004 ASTM C150 finally allowed up to 5% limestone to be used in Portland cements. However, many state highway agencies use American Association of State Highway Officials (AASHTO) standards rather than ASTM, and the members who govern the AASHTO M85 cement specification (AASHTO cements use the same basic numbering system as in ASTM C 150 for cement types) is expected to adopt 5% limestone in 2007 after significant work by a joint ASTM-AASHTO harmonization task group since 2005 to minimize differences in these cement standards including the limestone issue. Related to harmonization, due to significant cross-border movement of cementitious materials, there is current activity to harmonize or at least reduce the number of differences between ASTM and CSA cement standards.

In some respects this harmonization of North American cement standards resembles the EN 197 harmonization of individual European country national standards. Prior to EN 197, the myriad of individual national standards had been a barrier to trade. While the EN standard still contains 27 types of common cement (Portland and blended), few of which are produced in any one country, at least the test methods and requirements have been harmonized, in order to allow trade.

2.4. Performance-based cement specifications

Also, in the process of revising the CSA cementitious materials standards, there was a deliberate attempt to move towards performance-based tests and limits rather than the traditional prescriptive requirements. The CSA A3000 is not totally a performance standard but the number of prescriptive requirements has been reduced, and often they are accompanied by a performance requirement. Several examples of this exist: (a) the MgO limit is also matched by the ASTM C 151 autoclave limit for paste bar expansion, (b) the SO_3 limits can be over-ridden by the ASTM C 1038 sulfate expansion limit. This is also true in ASTM C 150. In addition, while ASTM C 150 has optional low-alkali limits for cements (0.60% Na_2O equivalent) there are no limits on alkali content of CSA cements, since the ASR concern is covered in the A23.1 concrete standard, where the alkali loading of concrete mixture is considered instead (cement alkali content multiplied by the cement content of the concrete, expressed in kg/m^3).

In the USA, the ASTM C 595 blended cement specification is a prescriptive-based specification while ASTM C 1157 is a pure performance specification for any hydraulic cement (either blended or Portland) and has no restrictions on composition or raw materials. However, even after more than a decade, this pure performance specification is not widely accepted or used, in large part due to user/owner concerns that the performance requirements in this specification may not be adequate to cover all performance concerns, whereas there is an historical record of performance for cements meeting ASTM C 595 and C 150 prescriptive-based specifications. As mentioned previously, the CSA A3000 approach is somewhere in between, with performance requirements being used where there is a comfort level by all members. In the author's opinion it is possible that it would be more successful in the US to try and move ASTM C 150 and C 595 specifications towards performance and, where possible, to minimize differences with the requirements in C 1157.

2.5. Direct phase determinations

While the shortcomings of the Bogue equations for prediction of the true phase compositions of cements have been well known for decades, they continue to be used in cement specifications around the world. While direct methods of measurement of phase compositions have been used, including point counting of polished and stained clinkers using optical microscopy and X-ray diffraction, until recently there were no standard methods. To address this, ASTM C 1365 was adopted in 1998 for direct determination of phase compositions of both clinker and cement by X-ray diffraction (XRD) analysis. The detailed methods used by a laboratory for XRD analysis, including Rietveld analysis, are calibrated using NIST Certified Reference Materials (CRM) for three different clinkers covering a range of compositions (NIST also maintains numerous CRM for cements) The method includes precision data for alite (C_3S), belite (C_2S), aluminate (C_3A, both orthorhombic and cubic), ferrite phases (C_4AF), periclase (MgO), gypsum, hemi-hydrate, anhydrite, arkanite (K_2SO_4) and calcite, and the precision of the phase determinations were determined from interlaboratory test programs. With many plants becoming equipped with XRD and Reitveld analysis capabilities, this method will see increased use and may eventually replace the Bogue equations, except possibly for internal plant control purposes.

For cement clinker, an optical microscopy method using reflected light on polished then stained or etched surfaces was also standardized as ASTM C 1356 in 1996. Quantitative analysis of alite, belite, aluminate, ferrite, free lime, periclase, and alkali sulfate is determined using a point-counting method, and according to the precision statement, the standard deviation of measurements is 0.71% and the 95% confidence interval for two determinations on samples from the same material is 2.58%.

A number of plants use optical microscopy not only for phase determination on clinkers, but also to determine kiln burning conditions using other indicators such as the size and color of crystals.

2.6. Conduction calorimetry

Heat of solution tests such as ASTM C 186 for determination of the heat of hydration have been used since at least the 1930s. They have typically been used to assess heat of hydration of cements at fixed ages such as 7 and 28 days. This gives very limited information about rate of heat evolution, and the test is both time consuming and results are sensitive to test details. Conduction calorimeters, which give continuous heat evolution data, have been used as research tools for decades, but only recently has robust equipment together with user-friendly analysis software, suitable for standardization, been commercialized and adopted by the industry. This has recently led to an ASTM draft standard for use of conduction calorimetry. This test, once adopted, has the potential to become widely used by both cement users as well as producers.

2.7. Chemical shrinkage

The phenomenon of chemical shrinkage was first noted by LeChatelier and results from the fact that the absolute volumes of cement hydration products are smaller than the sum of the volume of their constituents (cement plus water), and that the amount of measured chemical shrinkage is directly proportional to the amount of hydration. While a useful research tool, until recently its application to standards was not appreciated. Based on the methodology developed by Geiker [4], a method for chemical shrinkage measurement was standardized by ASTM in 2005.

ASTM C 1608 is intended for characterizing the rate of hydration of cement paste using chemical shrinkage measurements. While not currently used in specifications, the results of this test are being used as input data for the CEMHYD3D hydration model developed at NIST [5], which is starting to be used to predict standard cement test results using “virtual testing”.

2.8. Sulfate resistance tests

Current standards for sulfate resistance in North America currently include restrictions on water-cementitious materials ratio, minimum strength, prescriptive limits on cement composition, and expansion limits when cementing material combinations are used in mortar bar tests. From the 1920’s even before the chemical reactions were understood, it was realized that low w/cm and low unit water contents of concrete mixtures were essential to obtaining resistance to the actions of sulfate soils. Shortly afterwards, the negative role of cement C_3A was realized, and the first sulfate-resistant cements were formulated in the 1930’s [6]. The first standard performance test for evaluating cements was ASTM C 452, but it was found not to be suitable for evaluation of blended cements and supplementary cementing materials. This led to the development of ASTM C 1012. There is no performance standard for testing concrete, due to the extended time it would take, so limits on w/cm and strength have been maintained.

While these measures were thought to provide protection against sulfate deterioration, since the 1990’s, a number of sulfate-related problems have been identified which may not be adequately addressed in current standards. These include delayed ettringite formation, sulfate salt crystallization, and thaumasite sulfate attack.

As stated above, the standard performance test for evaluating a sulfate-resisting Portland cement (SRPC) has been the ASTM C 452 14 day mortar bar expansion test developed by Lerch [7]. In this test, the SO_3 content of a Portland cement is raised to 7.0% using Terra Alba gypsum and mortar bars are cast. These bars are stored in water from 1 to 14 days at 23 °C and the expansion is measured. The ASTM C 150 expansion limit for SRPC (Type V) is 0.040% while in CSA A3001, it is 0.035% (Type 50 or HS) and 0.045% for moderate sulfate resisting (Type 20 or MS) cement.

ASTM C 452 was first published in 1960 but was not adopted in the C150 specification until 1971. The original SRPC optional test limit of 0.045% was reduced to 0.040% in 1985. (There had been a previous attempt in the 1950’s to develop a lean mortar bar test [8] but it was dropped due to poor reproducibility).

While this test has proven useful for testing sulfate resisting Portland cement it was found not to be valid for evaluation of blended cements or combinations of Portland cement and supplementary cementing materials. The reason for this is that with the excess SO_3 mixed into the mortar bars, the sulfate attack would start immediately and certainly before the pozzolanic and/or hydraulic reactions of fly ashes, slags, silica fumes and natural pozzolans had initiated. The admixed gypsum greatly accelerates the test since it eliminates the slow diffusion stage of external sulfate attack, but in reality, the bulk of cementitious materials in concrete have a chance to hydrate before being exposed to external sulfates in service. Therefore, to address this problem, the ASTM C01.29 sulfate resistance subcommittee, guided by K. Mather and T. Patzias, developed a new test method, C 1012 in the 1970’s which was first published in 1984. In this test, mortar bars are cured until a strength of 20 MPa is achieved, then they are exposed to 50 g/L Na_2SO_4 solution at 23 °C and expansion is measured. This test method had been adopted for blended cements (ASTM C 595, C 1157 and CSA A3001) and for evaluating mixtures of Portland cement with supplementary cementing materials (ASTM C 989, C 1240 and CSA A3001). The expansion test limits that have evolved [9,10] are 0.10% at 6 months for high sulfate resistance (this can be superseded by meeting a limit of 0.10% at 12 months). For moderate sulfate resistance the limit is 0.10% at 6 months. The ACI C201.2R guide to durability has adopted similar limits to qualify sulfate-resistant cementitious combinations for moderate and severe exposures, but also has included a very-severe exposure class where a limit of 0.10% at 18 months is required. These limits will be adopted in the 2008 version of the ACI 318 building code.

One of the industry concern with C 1012 is the length of time required to obtain results. However, it is difficult to avoid this since sulfates must diffuse inwards to react with aluminate compounds before any expansion can occur. During development of this test, the original strength of 25 MPa before sulfate exposure was reduced to 20 MPa in order to shorten the test. In hindsight, this was counter-productive in terms of balancing the concept of allowing these blended materials to react with respect to the saving of only a few days in a six month test. As well, the test is not necessarily directly relevant to other types of sulfate salts than sodium sulfate. For example, magnesium sulfate typically results in softening and cracking of the bars without as much expansion. Therefore, visual indications of damage need to be recorded as well as expansion if other sulfate salts are substituted in this test. The original test allowed for a mixture of sodium and magnesium sulfates but it was found that this caused problems in some cases, so sodium sulfate was adopted as the standard solution.

While the pH of the solutions in C 1012 are not controlled, in interlaboratory testing, it was found that the pH of the Na_2SO_4 storage solutions rapidly rose to 12.8–13.0 whereas the sodium–magnesium sulfate mixture only resulted in pH of about 10. Brown [11] and Clifton et al. [12] have advocated pH controlled tests, this may not model the situation of stagnant sulfates in contact with concrete foundations but it would better model flowing water situations, as in pipes.

None of the sulfate resistance tests including C1012 model the situation of evaporative transport, or wick action, of sulfates into the specimens. This situation, which can occur in tunnels, culverts, slabs on grade or in foundations subject to wetting/drying cycles, can pull sulfates in much faster than by diffusion. The sulfates then precipitate as salts as the water evaporates near the drying surface [13]. These salts can build up in pores and undergo phase changes due to changes in temperature and relative humidity, resulting in expansive pressures. A common reversible transformation is that between thenardite ($NaSO_4$) and mirabilite ($NaSO_4 \cdot 10H_20$), which if occurring in the capillary pores, can cause progressive surface damage. There may be some scope to develop test methods to model this scenario in general, but not all possible situations could be modeled. This issue may better be addressed in concrete standards, as is typically done now, by use of maximum w/cm limits.

The ASTM C 1012 test was developed to show the susceptibility (or resistance) of a cementitious system to attack by external sulfate solutions. It was not intended to model all or any field conditions for concrete nor to evaluate concrete quality.

It is interesting to note that C 1012 expansion limits are more severe than those for C 452 when used to evaluate SRPC. There are several cases where SRPC has passed the 0.040% 14 day limit when tested by C452 but failed the 12 month C 1012 limit of 0.10%. In part this was due to adopting the C 1012 limits based on the time to reach 0.10% expansion plotted against the C_3A limits for Moderate and High Sulfate resistant cements, and then taking a best-fit relation: this would mean that 50% of cements would fail the requirement. For all SRPC's tested by the author since 1977, all these have exceeded 0.10% expansion after exposure periods of between 7 and 20 months (some of the author's data is shown in Table 2) and the bars start to crack and ravel at the edges. This illustrates the point that SRPC's are only sulfate resistant and not immune to attack. This may be in part due to the role of the ferrite phase. Therefore, steps must also be taken to make good quality concrete to slow sulfate penetration.

Table 2
Performance in C 1012 Tests

C_3A	% @ 6 months	% @ 12 months	Time to >0.10%
(A) Moderate (MS) PC Performance in C1012 Tests			
5.9	0.074	0.294	~7 months
5.5	0.044	0.119	~11 months
7.4	0.117	0.517	5 months
7.3	0.072	0.235	8 months
7.9	0.076	0.291	~7 months
Limit	0.050	–	–
(B) SRPC (HS) Performance in C1012 Tests			
2.0	0.037	0.063	18 months
2.1	0.032	0.061	18 months
~2.0	0.052	0.113	11 months
3.8	0.060	0.273	7 months
1.4	0.037	0.061	20 months
Limit	0.050	0.100	–

Brown, Hooton, and Clark [14] examined 150×300 mm SRPC concrete (w/cm=0.45) cylinders exposed to either Na_2SO_4 or $MgSO_4$ for 22 years and found them to show microcracking and formation of ettringite and thaumasite.

Since the field performance of Types II and V cements in concrete have been documented for over 50 years, by adopting the concept of "equivalence" the C 1012 test can be used to evaluate blended cement systems versus Type II or Type V performance (assuming equal quality concretes). This is the concept used in ASTM C 1157 and in CSA A3001 (formerly A362 and A23.5). It is interesting to note that the author has tested several Portland-SCM combinations which have not exceeded 0.10% expansion in over 10 years of test exposure [9,15].

In the European Standard EN197-1 [3], there is currently no test method for evaluating the sulfate resistance of a Portland or blended cement, due to lack of agreement on a common test method, in spite of several European countries previously having had test methods. As a result, in 2006 the CEN committee drafted an amendment A2 to EN197-1:2000 to simply prescriptively allow a family of seven types of cement for use in sulfate resistant applications (not yet adopted). These types include three CEM I cements with either 0, 3, or 5% Bogue C_3A in the clinker, as well as slag cements CEM III/B (66–80% slag), CEM III/C (81–95% slag), and pozzolan cements CEM IV/A (20–35% pozzolan), CEM IV/B (36–55% pozzolan).

The thaumasite form of sulfate attack has become a subject of increased interest [16–18] with numerous papers published based on the International conference on thaumasite in 2002 [19], but currently there are no standards specifically related to its prevention.

2.9. Trends to high-alkali cements and use of CKD

Cement kiln dust (CKDs) are the particulate and combustion gas precipitates that are collected in the pyro-process. Most cement plants return all or a portion of the CKD to the kiln as raw feed. A large percentage of the CKDs that are not returned to the pyro-process, however, are placed in landfills. While the cement industry has reduced its energy consumption through design of more efficient cement plants, concerns with alkali–silica reaction often result in concrete specifications requiring low-alkali cement in concrete. Production of low-alkali cement, often, requires changes in production practices at cement plants resulting in the need to remove a higher percentage of CKDs from the cement manufacturing process than normal. Not only can this result in the need to landfill more CKDs (i.e. increasing solid waste), but it is a waste of raw materials and the energy invested in pyro-processing of the material. Also, in many cases, CKDs are removed from the kiln to prevent instability problems, but these CKDs could potentially be re-introduced in finish mills for production of cement. However, CKDs vary widely so it is important to characterize each source of CKD and to determine its influence on the performance of the cement when used in concrete [20].

The cement industry is under pressure to reduce greenhouse gas (GHG) emissions and solid waste in the form of cement kiln dusts. Since GHG CO_2 emissions are directly related to the fossil fuel consumption and the calcining of limestone raw materials, anything that can be done to reduce the need for limestone in clinker or the clinker factor in cement will have a direct impact.

There are not many non-carbonate-associated sources of calcium with the exception of industrial by-products such as iron blast-furnace slag, which in a patented process has been used to replace some of the limestone raw material. However, it is possible to maximize the use of calcined raw materials, by reducing or re-using the cement kiln dust that is removed from the process during Portland cement clinker production. The amount of CKD created varies from zero to approximately 15% by mass of clinker depending on the cement kiln process and the composition of raw materials and fuels being used.

While some plants either do not produce CKD or are able to recycle it back into the kiln, some processes involve removing or by-passing CKD in order to control the kiln operation. Dust may be removed based on chloride, alkali, and/or sulfate control to prevent for example, kiln ring formation, plugging of preheaters, or to produce low-alkali cement. If dust is removed to avoid problems with clinker production, it is possible to introduce some CKD into the finish grinding circuit. However, if the reason is to be able to produce low-alkali cement, then use of the CKD is not possible.

2.10. Trend to blended cements

In North America, blended cements have not been as popular as the use of separate additions of supplementary cementing materials at concrete plants, however, in many countries, the opposite is true. There are arguments on both sides. Proponents of blended cements claim that sulfate contents and performance can be better optimized. On the other hand, even though more silos maybe required, many concrete producers like the flexibility of being able to produce concretes with a range of different performance characteristics by combining different amounts of specific SCM with cement, in the same way they combine different chemical admixtures as well. In urban areas, large ready mixed and precast concrete plants have become very sophisticated, and technical staff often have the expertise to control these more complex concretes. However, this is not always true nor is it typically true in smaller rural concrete operations.

Partly as a result of international ownership of much of the North American cement industry, and in part due to efforts by the cement industry to reduce its Green House Gas emissions, by reducing the clinker content of cements, there has been recent pressure to adopt more blended cements. When performance-based specifications such as ASTM C 1157 are adopted, other components than pozzolans and slag can also be added, which may further reduce the clinker content of the final product. However, in terms of real environmental benefit, unless blended cements are able to be made with higher levels of SCM than can be accomplished when introduced as an ingredient at a concrete plant, there will be little overall environmental benefit to the adoption of blended cements. For example, related to the use of High-Volume SCMs in concrete, given its limited use, it would be easier for a concrete producer to be able to produce such concretes with separate additions of SCM rather than through use of a high-volume SCM-blended cement.

However, according to Herfort [21], the main challenge for future standards goes beyond the increased use of traditional SCMs. Whilst only about 25% of potential sources of slag and fly ash are utilized in cement and concrete production in North America, utilization has reached saturation point in many parts of Europe, which is why limestone, at least in terms of volume, is the most common clinker replacement material in European cements today. This trend is bound to continue as steel and electricity producers begin to reduce their own CO_2 emissions (and waste materials).

2.11. Standards for pozzolans and slags

2.11.1. Background

Natural pozzolans were used early in the 20th century for reducing heat of hydration in mass concrete (and had unintentional beneficial effects on preventing what was later identified as alkali–silica reaction (ASR) in some dams built by the US Bureau of Reclamation. The first ASTM standards for pozzolans (currently ASTM C 618 is the Specification for Fly Ash and Natural Pozzolans, with C 311 including the associated test methods) developed when coal power fly ashes became available and were also initially used for low-heat purposes in mass concrete applications. While some blended cements had been made with ground granulated iron blast-furnace slag (just referred to as slag) combined with either Portland cement or lime, they were not widely used or promoted in the US, unlike the European experience. However, with the Standard Slag Cement plant (now totally owned by Lafarge) near Hamilton Ontario starting production of separately ground slag in 1976, followed by Atlantic Cement's (now Lafarge) granulation and separate grinding facility at Sparrow's Point Maryland, USA around 1981, use of slag in concrete has gained popularity. In the late 1990's the number of slag granulation and grinding facilities in North America multiplied and its use as a separate addition has grown from 1.1MT in 1996 to over 3.2 MT in 2006 in the US with approximately 0.3MT being used in 1.2MT of slag-blended cements meeting ASTM C 595 [22].

Silica fume is covered by ASTM C 1240 and only applies to silica fumes from silicon or ferro-silicon furnaces where SiO_2 contents are at least 85%.

2.11.2. CSA fly ash F CI CH-role of CaO as opposed to ASTM

The ASTM C 618 specification for fly ash and natural pozzolans was developed in the 1950s and in it, fly ashes have been divided into two Classes, F and C, depending on coal type and on chemical composition of the fly ash itself. The compositional parameter which is still used is the sum of the oxides of $SiO_2+Al_2O_3+Fe_2O_3$. If this sum is at least 70% of the total, then the fly ash is considered Class F, whereas C ashes only need the sum to total 50%. While this parameter may have value, many of the performance issues with the use of fly ash in concrete, such as sulfate resistance and control of expansion due to alkali–silica reaction are more related to the fly ash CaO content and also the sodium plus potassium oxide content [23]. In recognition of this, in 1998, the CSA specification for Supplementary Cementing Materials (then designated A23.5) was changed from the ASTM classification system to one where fly ashes are categorized based on CaO content. Three classes of fly ash were created [2]: Class F with up to 8% CaO content, Class CI with 8 to 20% CaO content, and Class CH with >20% CaO content. Where a certain fly ash straddles one of these limits, the predominant CaO content governs, and individual CaO values are allowed to exceed the limit by up to 2%. While still prescriptive, it is thought that this system better describes how the fly ash is likely to perform in concrete. The ASR guide in CSA further separates fly ashes based on ranges of equivalent alkali content. While moving to such a classification system has been discussed at ASTM, there is no move to change at the time of writing.

3. Performance of cementitious binder systems

One of the problems with trying to develop performance standards for cements, is the fact that not only is cement just one ingredient in a concrete product, but that many other components in the cement paste fraction of concrete influence performance of the binder system; these typically include one or more SCM's and various chemical admixtures. Therefore, the results of some performance tests for cement (e.g. time of set), become somewhat irrelevant in such increasingly complex paste systems. For example, something as simple as setting time of cement will not likely bear any relation to the setting time of the paste system, let alone to concrete in various construction environments. Thus, in some respects, cement standards might be better left as manufacturing standards using expedient chemical content and prescriptive limits.

For this reason, ASTM formed a new joint C01/C09 joint subcommittee C01.48/ C09.48 on Paste Systems Performance in 2004 (chaired by the author) to evaluate potential test methods and standard practices that might be better suited to evaluate the fresh and early-age performance of the combination of all the ingredients in the cement paste system. For example, tests for workability, setting time, setting issues, as well stability of entrained air are some that may be developed or adapted for use with paste systems. Recently, a draft of a standard practice was balloted for "Monitoring hydration kinetics of hydraulic cementitious materials as cement mortar or paste using isothermal calorimetry". While the first draft received negative votes, it has been revised and will be re-balloted in 2007. This method can potentially be used to determine whether normal setting times and early-age hydration rates are being achieved, and it includes a rapid and cost effective, multi-sample, semi-adiabatic method which can be used at a concrete plant to troubleshoot and evaluate changes in admixture dosages and types on the particular cementitious materials being used. For a cement producer, this standard practice could be useful in determining whether their cement, when being used in typical cementitious materials-admixture concrete systems, has a sufficient sulfate content, which influences time of set and early rate of hydration.

Another concept being considered is to modify the ASTM C 359 test for early stiffening characteristics of mortars, for use in evaluating cementitious materials-admixture system variables. As well, a modification of the foam index test, originated by Dodson [24] for use in determining AEA dosages required for use with fly ash mixtures, has been proposed as a method for the same purpose, but applied to the entire cement paste systems.

4. Specifications for chemical admixtures

There are numerous types of chemical admixtures used in concrete and most concrete mixtures contain several chemical admixtures. They include water reducers (low, medium and high-range), retarders, accelerators, air-entrainers, and more recently de-foamers, thixotropic agents (viscosity-modifying admixtures), and corrosion inhibitors. Each of these types of admixtures are typically available in different chemical formulations. Therefore, for standardization purposes, the admixture standards, ASTM C 233 (air-entrainers), C 494 (water-reducers, retaders, accelerators, high-range water reducers, and combined purpose admixtures), C 1017 (admixtures for flowing concrete) and C 1582 (corrosion inhibitors) are pure performance standards. The admixture being evaluated has to show in concrete tests that (a) it acts as intended and also (b) that it does not alter other concrete performance requirements by more than stated limits (e.g. setting times, strengths, shrinkage, freezing and thawing resistance). In Canada, CSA standards for chemical admixtures have been dropped and CSA now references ASTM standards for chemical admixtures.

5. Developments in Concrete Standards

5.1. High-volume SCM use in concrete

The use of high volume replacements of cement by supplementary cementing materials has become popular for a number of applications including "green buildings" around the world. While supporting their use in principle, the CSA Concrete committee thought that some special considerations had to be made to prevent potential durability problems when using such concretes in severe exposures. CSA A23.1–04 [25] defines HVSCM concretes, as containing a level of SCM above that typically used in normal construction (About 75% of concrete in Canada contains some level of fly ash, slag or silica fume). Two categories of HVSCM concrete are defined:

$$\text{HVSCM-1}: \ \%\text{FA}/40 + \%S/45 > 1$$
$$\text{HVSCM-2}: \ \%\text{FA}/30 + \%S/35 > 1$$

where, FA = fly ash, and S = ground granulated blast-furnace slag.

The standard performance requirements for durability exposures are modified in some cases when HVSCM concrete is used. For example, when the concrete is exposed to freezing and thawing in which case the maximum w/cm values required to be reduced by 0.05 for HVSCM-1 in all exposure classes. For example, in concrete exposed to chlorides and freezing in a saturated condition (CSA C-1 Exposure) the maximum water-to-cementing materials ratio is normally 0.40, but for HVSCM-1 concrete this maximum value is reduced to 0.35. Also, to account for the potentially slower rate of strength gain, the minimum specified 28 day compressive strength requirements is changed to 56 days for HVSCM-1 concrete. To reduce the risk of carbonation-induced corrosion of reinforcement, for concrete exposed to moisture with <50 mm depth of cover, the maximum allowable w/cm for HVSCM-2 concrete is 0.45, and 0.40 for HVSCM-1 concrete. Finally, for severe exposure class categories such as C-1, HVSCM-1 concretes are required to be moist cured for 10 days (as compared to 7 days for other types of concrete). For less severe exposure classes, 7 days moist curing is required for all HVSCM concretes (compared to 3 days for other types of concretes). Currently, to the author's knowledge, there are no ASTM standards nor is there specific information in the ACI Building Code related to the use of HVSCMs in concrete.

Performance specifications can allow for innovation in the supply of concrete, including HVSCM concretes by providing flexibility in materials supply and concrete proportions. This can be used to allow use of more environmentally friendly concrete materials and proportions.

5.2. Concrete standards: specifying for durability and performance

There has been a recent surge in interest in sustainable development as well as "green buildings". Concrete structures already offer many advantages for sustainable development. They are almost exclusively made with local materials (reducing energy in transportation), most concretes already contain some level of recycled materials (supplementary cementing materials (SCM) which are by-product wastes of other industrial processes, and some chemical admixtures which are derived from pulp and paper wastes), the thermal mass of concrete building envelopes helps to reduce Heating and air-conditioning (HVAC) requirements, interior and exterior surfaces can be left untreated, and light-coloured, sun-exposed surfaces help reflect solar radiation. The sustainability of concrete structures can be further enhanced by using higher levels of cement-replacement materials, and by better design for durability performance (extending service life), even in severe environments. Sometimes standard specifications, which are often prescriptive in nature, can be an impediment to these latter issues, either by putting prescriptive restrictions on materials and concrete proportions, by not providing durability performance specifications, or by not allowing options based on performance tests.

There is a current trend away from prescriptive towards performance specifications in North America and around the world. Historically, prescriptive specifications are the norm and have been developed by local experience but are often conservative. They also often inhibit innovation since new materials and methods do not fit into the prescriptive mould. This is of significance when 'sustainable concretes', such as those containing High-Volume SCM's (HVSCM), are being considered (As discussed previously, CSA A23.1-04 makes provision for the use of HVSCM concretes). However, adoption of true performance-based specifications presupposes that we have a clear understanding of all the performance issues that can affect concrete. It also assumes that there are appropriate performance test methods in place to evaluate all of the performance issues for: concrete materials, fresh concrete, hardened concrete, and durability. It also assumes that performance can either be measured in time to affect the outcome, and/or can be used to pre-qualify concrete mixtures. Most parties to construction are familiar with testing for fresh and hardened properties of concrete, but the biggest challenges in this regard relate to requirements for durability.

While there are many types of aggressive exposures which might require a multitude of durability tests, the common element is that most aggressive exposures require that the permeability or fluid penetration resistance of concrete be minimized. Therefore adoption of one or more tests for penetration resistance is fundamental to ensuring durable concrete.

While there are many definitions of concrete performance specifications, the Canadian CSA A23.1-04 [25] defines it as follows: "A performance concrete specification is a method of specifying a construction product in which a final outcome is given in mandatory language, in a manner that the performance requirements can be measured by accepted industry standards and methods. The processes, materials, or activities used by the contractors, subcontractors, manufacturers, and materials suppliers are then left to their discretion. In some cases, performance requirements can be referenced to this Standard, or other commonly used standards and specifications, such as those covering cementing materials, admixtures, aggregates or construction practices".

A recent literature review was made of concrete performance standards from national and other recognized standards and specifications from around the world by Bickley et al. [26]. The following quotations provide a summary of some of the general findings from that review:

"It became clear that while there was an almost universal interest in performance, primarily for durability, there were few specifications that contained any pure performance criteria.

Most defined exposure conditions that pertained to each country and then tabulated concrete mixture contents and limits that studies had shown would result in the desired durability. These include maximum limits for water to cement or water to cementitious materials ratio limits, minimum cement contents and an acceptable range of air contents. There is an almost universal use of supplementary cementitious materials such as fly ash, granulated ground blast furnace slag and silica fume, as additions or in blended cements. All the specification documents assumed the use of statistical quality control to assure consistent conformity at the lowest cost."

"It also became clear that the term "performance specification" means many things to many different people. This is not necessarily because of any misinterpretation. This is because there is such a wide array of options and valid interpretations, making it imperative that the term be carefully defined in any given context. Parties could agree in principle to execute work under the performance specification umbrella, and yet have widely differing views about mutual expectations."

"A lack of reliable, consistent and standardized test procedures for evaluating concrete performance is frequently cited as a major barrier to the adoption of performance specifications. Some of the available tests can be expensive, take a long time to run and may not be as precise as desired. Short bid times and quick construction starts create a difficult situation for a concrete supplier faced with the need to develop a performance mixture and to perform prequalification testing. In a number of jurisdictions, such as State Highway Departments, some advanced tests have been site proven and then specified in subsequent years for pay items in contracts."

"On the other hand, in the face of an international mindset that says that testing technology has not yet caught up with performance philosophy, there are a wide range of tests that are available today, and have been used successfully on important concrete projects, and these tests methods can be called into action to support performance-based specifications. While some may complain that current tests are not ideal or are insufficiently accurate or precise, which of our everyday concrete quality tests are ideal? If a new test only has to be as accurate, as precise, or as meaningful as the slump test, there may be many new developments to choose from."

"The advent of performance specifications could significantly change the distribution and sharing of responsibility among owner, contractor and concrete supplier. It would be up to the owner (through design professionals) to clearly specify performance requirements together with the test procedures used for acceptance. In the case of true end-result specifications based on hardened, in-place concrete properties, the execution of these requirements would be the joint responsibility of contractor and concrete supplier. They would assume the risk involved and would have to work closely to determine the appropriate concrete mixture. Quality management programs would also be required from both since the successful installation of a concrete mixture would be imperative to achieving acceptance by the owner."

"The transition to performance specifications as another, complimentary way of doing business will require a dedicated educational effort, and advantages and disadvantages will have to be made concrete, so to speak. The motivation will have to come from clear benefits that can be shared at many levels of the industry, and not just because it is time for a change."

In that same review [26], the following keys to the concept of performance specifications were identified:

a) The ability of the specifications writer to discern the performance characteristics appropriate to the owner's intended use of the concrete.
b) The ability of the specifications writer to describe these performance characteristics clearly, unambiguously, and quantitatively so that performance can be evaluated.
c) The availability of reliable, repeatable test methods that evaluate the required performance characteristics (along with performance compliance limits that take into account the inherent variability of each test method).
d) The ability of the concrete producer–contractor team to choose combinations of materials, mixtures, and construction techniques to meet required characteristics so that projects can be planned and bid, risks and costs can be assessed, and materials and construction operations adjusted to comply with performance requirements.

As a first step towards developing a performance alternative for ACI 318 [27], a reformatting of durability concerns into exposure classifications was suggested [28]. A modification of this has been adopted for the 2008 version of the ACI 318 building code.

5.3. Durability tests

Most deterioration processes involve two stages. Initially, aggressive fluids (water, ionic solutions with dissolved salts, gases) need to penetrate or be transported through the capillary pore structure of the concrete to reaction sites (e.g., chlorides penetrating to reinforcement, sulfates penetrating to reactive aluminates) prior to the actual chemical or physical deterioration reactions. Therefore, a standard acceptance test or tests to measure rates of ingress of aggressive fluids, or a related rapid index test, is fundamental to the development of performance-based durability specifications. However, before tests are adopted in specifications, they must not only be shown to be useful and reliable, they must also be standardized and include precision data preferably based on interlaboratory evaluations, in order to develop confidence in the results and to be able to set realistic specification limits that take account of the test variability. Many tests have been proposed by various researchers, but only a few have been adopted in recognised standards [29,30]. Since most rigorous methods are too slow and expensive to be used beyond prequalification of mixtures, rapid index tests are typically used for quality control and in-place purposes. In CSA A23.1, the ASTM C 1202 'coulomb' permeability index test has been adopted for this purpose, although rapid chloride migration or water sorptivity tests maybe considered in future revisions.

Test methods related to measurement of various durability properties exist in various standards (e.g. ASTM, AASHTO, Corps of Engineers (CRD), and individual Departments of

Transport (DOT)) in North America and abroad. Limits based on some of these test methods are specified in ACI, CSA, Federal, and individual DOT specifications, amongst many others. It was stated [31] that in the USA alone there are over 2000 specifications for concrete. Each of these specifications employs different test methods and different test limits.

Another issue is that tests do not exist for all of the relevant durability or performance concerns, such as freezing and thawing with or without de-icer exposure, sulfate and other chemical resistance. As well, existing tests are not always rapid, accurate, or repeatable, nor do they necessarily adequately represent any or all of the exposure conditions in-situ. The lack of adequate performance-related test methods for concrete is one of the main factors that inhibit the move from prescriptive to performance specifications. A couple of examples related to specific durability issues are used to illustrate need for relevant test methods.

5.4. Guidance on use of standards

Since standards are required to be succinct and procedural, often users must seek guidance elsewhere related to developing an understanding of their use. As a result, the ASTM C09 Committee on Cement and Concrete Aggregates has sponsored a series of Manuals called STP 169, *Significance of Tests and Properties of Concrete and Concrete-Making Materials* [32]. The latest, STP 169D, was published in 2006 and explains concrete and concrete materials standards as well as test methods, explaining how they relate to concrete properties. It is a valuable adjunct to the ASTM Book of Standards as test methods and standards are usually written in very terse, mandating language, although appendices are sometimes included to provide explanatory and reference material.

Similarly, in Europe, RILEM technical committees have published similar monographs on single technical topics in the Materials and Structures journal.

6. General comments on test methods and the future of models in standards

While standard test methods and specifications are often viewed as “gospel” at least in a legal sense, in reality they are imperfect documents and are constantly being revised (or should be) to reflect new and relevant knowledge. On the other hand, some see standards as impediments to change and to the utilization of new materials and practices. However, if there is a need, and a person or group to champion a new test method or materials standard, then standards will be developed relatively quickly. Also if a material, process, is viewed as important to the construction community, then utilization of new material and products will proceed regardless of the standards and their use will positively influence the eventual standards development. One example is silica fume. It was being used around the world in concrete for years before the CSA A23.5 standard for silica fume became the first available national standard in 1983. Another example is Self-consolidating Concrete (SCC) where this type of concrete was in widespread use before any test method were written at least in North America (Note: At ASTM, the late Bryant Mather, a stickler for terminology and grammar, said that the term, “Self-Compacting Concrete” was incorrect since the concrete didn’t compact itself but rather consolidated without external effort).

A more current example, is Pervious Concrete, where ASTM initiated a new subcommittee in 2006, but where this type of product was already being used to drain surfaces and to reduce runoff to storm sewers.

On the other hand, as discussed earlier, the consensus standard process, while possibly slowing down the adoption of new test methods and standards, take advantage of obtaining input from Producer, User, and General Interest members. The requirement for precision data in ASTM (and more recently CSA) that would help ensure that all parties are able to discern the significance of the data being generated, and it helps in selecting realistic and statistically-based limits in specifications. As well, this process can prevent inadequately developed tests from being adopted and then subsequently causing problems.

The attributes of a good test method include, (a) reliability, (b) reproducibility, (c) that it addresses a relevant performance issue (d) uses realistic boundary conditions, and (e) it is rapid and as simple as practical without overly sacrificing the other criteria.

However, it must be realized that while a test may generally address a relevant performance concern, it typically cannot mimic all relevant boundary conditions and in-situ scenarios. For example, most sulphate resistance tests only measure the resistance of a cementitious binder (typically in a mortar sample) when fully immersed in a sulphate solution (e.g. ASTM C 1012 or the German DIN 1164). While these tests are relevant to the ettringite form of sulphate distress from attack on the cement aluminates, they do not relate to situations such as, (a) where the concrete is also exposed to evaporative transport (e.g. physical sulphate salt crystallization pressures such as from the reversible mirabolite–thenardite transformations), or (b) where concretes are exposed to cool temperatures and carbonation favour formation of thaumasite sulphate attack on the C–S–H matrix, or (c) where different types of sulphate salts and concentration levels than those tested are experienced. Many of these issues are currently being addressed by the Nanocem consortium in Europe, after the European Cement industry could not come to agreement on a test method for sulphate resistance in 2003 [33].

However, it is doubtful that a single test could address all of these issues, and testing for all possible exposures would be both cost and time prohibitive. Therefore, development of predictive models, combined with rapid index tests and making use of relevant performance databases may provide useful solutions eventually. Predictive models such as those being developed by teams at NIST, as well as universities such as Laval, Delft, Tokyo, and Aberdeen will likely shape the future of standard development, when used with specific materials characterization and science-based index tests.

For example, in one of the NIST models [34], cementitious material particle size distributions and XRD mineralogical compositions are already used to predict properties such as time of set and strength development. The STADIUM model [35,36] is being used to predict resistance to various forms of chloride and sulphate penetration, by modelling of the transport and interaction of multiple ionic species in solution with the solid phases. Life 365, developed by Thomas and Bentz [37], has been used by designers across North America to predict time-to-corrosion due to chloride ingress, using a default database of time-dependant chloride diffusion data (user-defined data can also be used as inputs). A number of other chloride ingress service life models are also being used in Europe [38].

7. Future trends in cement standards-performance indicators and modeling

As stated above, test methods, especially those related to durability issues, cannot be expected to mimic all possible exposures and boundary conditions. However, if test methods are based on good materials science and are combined with sufficient data bases, they can possibly be used as inputs into models where the influence of other exposures and boundary conditions can be predicted. This is likely the trend for future standards development. While many groups are involved with predictive modeling, NIST appears to be the most focused on use in support of standards development for cementitious materials [5]. For example, Bentz [34] has used a hydration model, CEMHYD3D, along with chemical shrinkage measurements to calibrate the rate of reaction of the cementitious material, was able to predict heat of hydration. In another example, using the same model, Bentz [39] was able to predict the influence of limestone fillers on cement hydration using materials properties and knowledge of how finely-ground limestone reacts with cement.

8. Summary

A number of issues have been raised in this paper but the list of topics covered is by no means comprehensive. As well, given the nature of a plenary paper, not all issues were dealt with in detail and have been summarized. While perhaps not as quickly as some would like, it is apparent that research has and continues to influence standards, and that standards respond to industry needs as new materials and construction methods develop. Adoption of methods for direct determination of cement compounds, conduction calorimetry, chemical shrinkage, as well as development of methods for evaluation of cementitious materials-admixture combinations are some examples where research has led to recent and current standards activity. Some of the biggest opportunities for future developments in standards will likely occur as predictive models become more sophisticated in their ability to predict performance of both cementitious materials and concrete, as more relevant and reliable performance tests are developed, and as industry moves to adopt their use.

Acknowledgements

I would like to thank Duncan Herfort, Paul Tennis, and Michelle Nokken for their comments on the draft paper and also John Bickley and Ken Hover for their contributions related to concrete performance standards.

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