

## **CHLORIDE DIFFUSION IN CONCRETE SPECIFICATIONS A CONTRACTUAL MINEFIELD**

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### **ABSTRACT**

This paper examines some of the test methods considered by the Concrete Institute of Australia, Z13 Recommended Practice "*Performance Criteria for Concrete in Marine Environments*". In particular the paper deals with real specifications where the test methods (or variants of them) have been part of the contract documents governing contracts performed by the authors' organisation over recent years. The paper seeks to demonstrate the dangers for specifiers in not fully understanding the nature, methodology and purpose of tests chosen for the specification. Further, the matter of time is highlighted as it relates to material pre-qualification, available time allowed by the contract to establish benchmarks and the real relevance of the tests to the design life of the structure. Finally, the paper deals with the huge potential for contractual dispute and litigation where short-term tests are specified indiscriminately without due reference to the outcomes actually being sought.

Keywords: Chloride, contract risk, correlation, coulomb, diffusion, dispute, interpretation, precast concrete, steam curing, time for tests, validity

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## INTRODUCTION

The problem with specifications for concrete durability in marine environments is that in all instances specifiers have demanded the very best irrespective of design life cycle, the relative importance of the structure and the cost associated in achieving the very best. Clearly, not all structures exposed to marine environments warrant such premium specification.

In the past it had been thought that concrete specified by strength for structural considerations alone (AS CA2-1963<sup>(1)</sup> and AS 1480-1974<sup>(2)</sup>) would render the concrete in the structure durable. In other words, little consideration for durability design was afforded to the concrete during the design process. Consequently, designers using these codes often did not recognise that higher strength F<sub>c</sub> values and other design measures should be specified for adequate durability design.

A whole generation of designers consequently believed that if the 28-day compressive strengths were met, the concrete had the required quality. Of critical importance, and yet not recognised, was the quality of the cover concrete (25-75 mm) extending from the outer surface to the depth of the closest steel reinforcement.

The publication in 1988 of AS 3600<sup>(3)</sup> addressed durability design with the introduction of Exposure Classification concepts. Factors including climate, location, proximity to the ocean, aggressive environments and chloride thresholds were recognised as important parameters in defining exposure classifications. Appropriate levels of minimum binder content, max binder/water ratio, compressive strength, type and duration of curing are used to define the durability characteristics for a given exposure class.

The 1994 amendments to AS 3600 provided little change to durability design despite the fact that many engineers and practitioners were concerned whether the provisions in AS 3600 and AUSTROADS bridge design specification were adequate for concrete durability in a marine (classification C) environment. Emerging and project specific construction specifications introduced and extended test methods and acceptance / rejection criteria for concrete well beyond the recognised codes in an attempt to formulate provisions which would assure the design life, up to 100 years plus.

## BACKGROUND TO THE CIA MARINE DOCUMENT

From a specialist supplier and contractor viewpoint, the inclusion of electrical resistance testing of concrete such as the ASTM C 1202<sup>(4)</sup> test in project specifications and contract documentation has the potential to bring about a large, dis-proportionate transfer of risk to constructors and suppliers. This is because conformance testing of concrete is based on short-term electrical tests which have not been correlated with long-term salt-water ponding tests. Researchers and specifiers have been testing the market-place for acceptance of ion migration theories for durability design and contractors, not surprisingly, are

concerned about the likelihood of contractual disputes and legal liability issues that might arise from these specification types.

These concerns were part of the genesis of the CIA marine document *“Performance Criteria for Concrete in Marine Environments”* and, accordingly, the major specifiers of concrete for marine applications in Australia were surveyed. These findings were compared with a limited review of international specifications. Australian specifications were critically examined to establish any consistency of approach. The review included documents prepared by Public Authorities, Major Consultancies, Australian Standards and Industry Associations. Limited overseas experience was also reviewed and all specifications were assessed against AS3600 and Austroads specification requirements. Durability parameters specified include concrete properties in terms of sorptivity, apparent volume of permeable voids (AVPV), compressive strength, rapid chloride ion penetration (RCIP), chloride diffusion, water absorption, drying shrinkage, soluble salts, type and duration of curing, surface treatment and crack size.

## SPECIFICATION SURVEYS

Reproduced from the CIA marine document are Tables 1, 2 and 3 which summarise the specification review data. Table 1 illustrates mix design and curing requirements in specifications while Table 2 lists the frequency of specific performance tests.

Table 3 combines aspects of mix design and performance testing from seven international sources.

**TABLE 1 – Survey Results**  
Mix Design and Curing Specifications

Specification	Units	No. of Specs where Referenced	Range Specified
<b>Binder Content</b>	(kg/m <sup>3</sup> )	8/11	400-470
Cement Content	(kg/m <sup>3</sup> )	1/11	
<b>Binder Composition</b>		8/11	
<b>Max W/B</b>		8/11	0.35-0.40
Superplasticiser		3/11	
Commercial integral waterproofers		2/11	
Slump	(mm)	2/11	
Minimum Curing	(days)	10/11	7-14

**TABLE 2 – Survey Results**  
Tests for Establishing Concrete Performance Under Marine Conditions

Specification	Units	No. of Specs where Referenced	Range Specified
Sorptivity	(mm)	1/11	
Minimum Strength	(MPa)	9/11	45-60
RCIP	(coulombs)	2/11	
Chloride Diffusion Coefficient	(m <sup>2</sup> /s)	1/11	
Modified BS1881 (7 days)	(%)	1/11	

**TABLE 3 - Marine Environment Specifications on Overseas Projects**

Specification	Unit	Norway	Canada	Finland	Channel Tunnel, U.K.
Comp. Strength	MPa	x	x	x	
Max. w/b ratio		x	x	x	x
C <sub>3</sub> A Content		x			
Binder Content	kg/m <sup>3</sup>	x		x	x
Chloride diffusion co-efficient	m <sup>2</sup> /s	x	x		
Cover to rebar	mm	x	x	x	

Inspection of this data shows conclusively that in Australia, a prescriptive approach to conformance is based on minimum strength, binder content, cover and minimum curing in at least 8 of the 11 (i.e. 73%) of specifications surveyed.

Performance testing based on chloride ion diffusion and rapid chloride ion penetration (RCIP) account for less than 10% in Australia and up to 50% in overseas specifications.

Clearly, there has been a general reluctance for specifiers to commit themselves and their clients to specification requirements that extend beyond the provisions of current codes of practice. This is not, we would suggest, due to ignorance, but rather due to the contractual certifications required for project delivery. The time required to realise reliable results, costs and uncertainty of specifying diffusion based testing together with the widespread legal view that the codes reflect current knowledge and, therefore, acceptable practice all mitigate against deviation from the codes. For these reasons specifiers tend to specify concrete for marine conditions on a prescriptive basis. This approach provides a practical way for suppliers and contractors to demonstrate compliance at the quality control level of project management.

## PERFORMANCE CRITERIA IN SPECIFICATIONS

Irrespective of the method and quantum of test(s) specified under a contract (strength and cover, sorptivity, absorption and the like) for marine environments, the contractor must consider the implications of material pre-qualification on the one hand and quality control compliance during the performance of the work on the other.

When the specifier is considering the method of specification, it should be clear that peripheral issues surrounding any testing become very important and can lead to contractors pricing the work as-they-see-it. If not properly specified, the seeds for dispute between the parties will have been sown even before a winning bid is awarded.

Factors such as duration of any test, the relative ease, cost, degree of accuracy and consistency must be considered so that the specifier makes appropriate use of testing within the contractual framework. For instance, it may be unreasonable

to require a two-month material pre-qualification period during a one-month tender period. As well, tests must be reliable and repeatable. Any acceptance criteria nominated must be quoted in the context of the precision of the test method. The risks associated with compliance must be well balanced and any consequences of non-compliance must be clearly described in the specification.

## ISSUES WITH CONTRACT SPECIFICATIONS

It is rare that specifications for use in construction contracts for marine structures are written in a concise, enforceable manner. Frequently, standard prescriptive specifications are adopted and amended to add some particular requirement for chloride ingress and/or electrical test limitation. It is even more rare for specifications to provide the time necessary for all the specified pre-requisites to be complied with. In BOOT, D&C or similar contracts, time (or the lack thereof) for development of trial mixes and related compliance criteria becomes of critical importance. The time available between award of the work and commencement of concrete manufacture often renders it impossible to comply with the specification.

To this end, the recommendation in the CIA Marine document<sup>(5)</sup> (Z13) should remedy the situation when the principles of pre-qualification of materials and concrete mixtures become more widespread and specifiers realise the need to provide time if acceptable and meaningful outcomes are to be achieved. This matter needs to be urgently addressed otherwise considerably more dispute between contracting parties will emerge than is currently the situation. Listed below are four specification extracts that details the compliance criteria for contracts recently completed with which the authors have been involved. Clearly, each of those specifications has the potential to result in dispute because of lack of understanding by the specifier of either the test or standard relating to the test.

### Specification 1

Exposure Classification	C
Concrete Strength at 28 days	50
Concrete cover	60
Shrinkage (56 days)	600
Maximum chloride penetration	$1 \times 10^{-12} \text{ (m}^2 \text{ / Second)}$

### Specification 2

The concrete shall be 1000 coulombs	
Concrete Strength at 28 days	50
Concrete cover	50

### Specification 3

Class of concrete	S50
Slump	80-100
Air content	2% +/- 1.5%
Cement type	GB (may contain slag/fly ash/silica fume)
Shrinkage (56 days)	550

Max W/C ratio	0.40
ASTM C1202 modified 28days	<1000 coulombs (1 test / month irrespective of volume)
ASTM C1202 modified 56 days	<500 coulombs
Curing	7 days

#### Specification 4

Specified exposure class, strength, cover, and shrinkage to prescriptive limits.

Contract award date to first placement of concrete under the contract = 6 weeks.

During this 6 week period, - trial mixes + 28-Day curing + 56-Day chloride ponding tests + 2 weeks approval period was specified. Total time required by contractor = 17 weeks.

Clearly impossible of performance.

Extensions of time were not allowable due to contract conditions.

Concrete deemed conforming only if 28-day coulomb values obtained during progress of the work of the contractor  $\leq 90\%$  of the coulomb value determined at the 56-day trial mix stage. For part of the works add calcium nitrite and perform RCIP test on that concrete as well.

#### ISSUES WITH CHLORIDE DIFFUSION LIMITS IN SPECIFICATIONS



In an attempt to move away from the prescriptive approach for durability design, researchers and specifiers have focused on comparing the material resistance to the anticipated deterioration process encountered in marine conditions. In the case where special concrete mixes with very low water/binder (0.29-0.30) ratios, high cement content and super-plasticisers are employed, very little information exists on the likely long-term resistance of such concrete to the rate of chloride ingress over time. In this context the most important material resistance is the chloride diffusion coefficient. The determination of coefficients is a

complex and time-consuming procedure.

**Figure 1** Apparatus for establishing chloride diffusion coefficient

Although chloride penetration into concrete follows a non-steady state process, current practice is to immerse the concrete specimens in saline solutions at constant salt concentrations for various time intervals ranging from 28, 35, 56, and 90 days depending on the specification. After immersion the chloride profile is determined by taking samples from the exposed face at various depths to determine the chloride diffusion coefficient Figure 1 using Fick's second law by curve fitting. The NORDTEST Method NT Build 443-1995<sup>(6)</sup> is one such test for this property.

The NORDTEST test procedure uses a profile grinder to grind out dust from a specimen for analysis at depth increments 0.5mm to 1mm. For every depth increment approximately 5 grams are collected for analysis Figure 2.



**Figure 2**  
Profile grinding

There seems to be general agreement that where proper correlation of results of saline ponding tests can be established with short-term electrical tests, then both diffusion limits and resistance limits are acceptable inclusions into project specifications. However, the issue is not as simple as it seems. Recent research<sup>(7)</sup> has shown that, theoretically, the use of Fick's law in the prediction of chloride penetration is not appropriate. It is valid only for non-ionic diffusants through a homogeneous inert medium. Chloride penetration into concrete is clearly an ionic transportation process involving other ions in the pore solution.

As well, the binder composition determines the time required for chemical reaction in the formation of hydration products. The rate of chloride diffusion is therefore also dependent on the completeness of the hydration process. Appendix A<sup>(8)</sup> shows the field results of diffusion coefficients ( $D_e$ ) after 28 and 56 days immersion in a saline solution and chloride profiles determined in accordance with the NORDTEST procedure for the recent M5-East project in Sydney.

Tests were performed on samples cured under standard 28-day moist curing conditions in the laboratory and also on 1-day steam cured plus 27-day air curing conditions.

The diffusion coefficient nominated in the specification at age 56-days was achieved. The rapid chloride penetrability test (ASTM C1202) requirement at age 28-days was, however, not achieved. This is because reliable correlation had not been established and it was not until age 84-days that technical compliance for chloride penetrability was achieved. Quality control compliance, albeit outside the limitations imposed by the specification became a matter of dispute. Because of the issues raised, it is not surprising the CIA document states in its conclusion *"there is significant debate as to the appropriateness of a diffusion coefficient obtained from a 28-56 day test to provide a reasonably accurate estimate of design life"*.

One important conclusion from this testing is that when concrete is properly steam cured in accordance with RTA specification Part B80 to achieve a 24 hour maturity of 350 deg centigrade hours, 28-day and 56-day diffusion coefficients are obtained that are comparable to those from standard laboratory tests after standard 28-day water curing. This clearly demonstrates the superior durability properties of steam-cured precast concrete. This is because in-situ concrete structures can not be cured nearly as effectively as standard 28-day water curing under laboratory conditions.

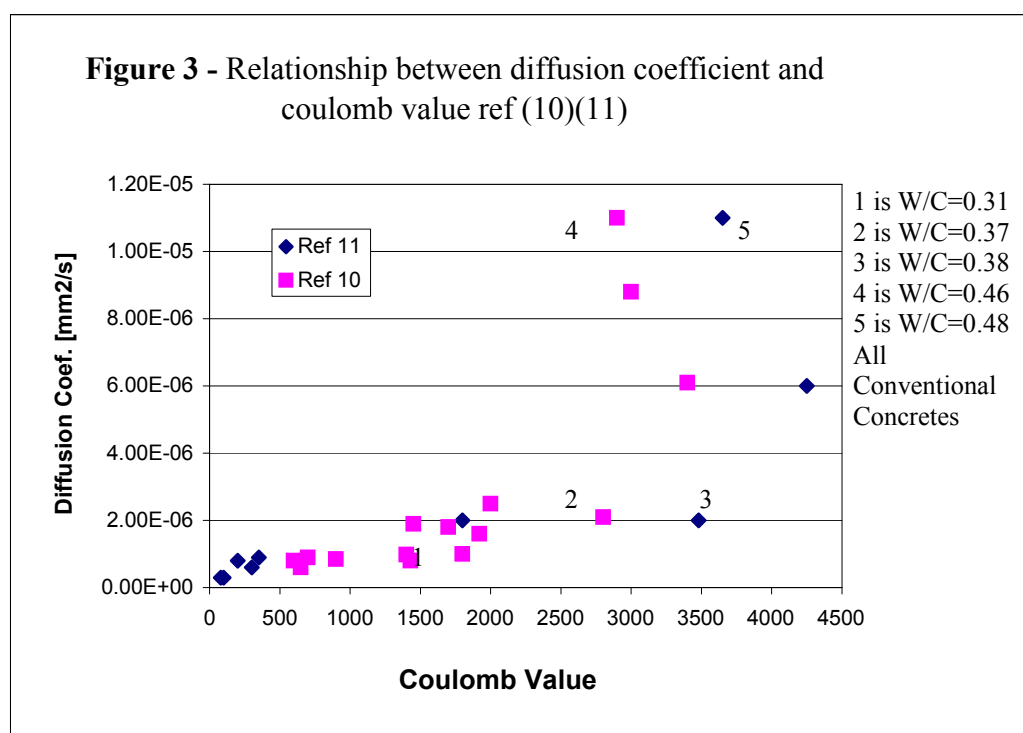
## ISSUES WITH THE RCIP (COULOMB) TEST IN SPECIFICATIONS

Permeability, diffusion and absorption are the important physical processes controlling chloride penetration into concrete during ponding tests. However, electrical resistivity appears to be the primary factor controlling charge passed during the RCIP test. The sensitivity of these two tests can cause significant variations in the ranking of the apparent permeability of various concretes.

Therefore, it is of concern to contractors that where the ASTM C1202-97 RCIP (Rapid Chloride Ion Penetration) or “Coulomb” test is specified for quality control testing, that the results of tests will generally **not** have been correlated to long-term salt-water ponding tests because of the lack of time under the contract. The RCIP test method is applicable for material pre-qualification only to types of concrete where correlations have been established between this test procedure and long-term chloride ponding procedures.

It has been established that proper and reliable correlations do not exist between the six-hour RCIP test results and 28, 56 and 90-day ponding test results. Accordingly, it has been recommended by the Precast / Prestressed Concrete Institute<sup>(9)(10)</sup> of North America that long-term ponding tests for the purpose of correlation should be of minimum duration of 1 to 2 years based on 2.5 year tests conducted by the Virginia Department of Transportation (VDOT), USA.

The data points in Figure 3<sup>(10)(11)</sup> are the combined results of a diffusion study for concrete, the subject of testing in Ref (10) and (11). Concrete mixes were prepared with and without calcium nitrite, and W/C ranging from 0.31 to 0.48 and silica fume contents of 7.5% to 15%. The data from the 24 concretes in Figure 3 show that there is a significant change in the relationship at a coulomb level of around 2500 and not at the 1000 coulomb level as is implied in Table 1 of ASTM C1202 specification. This shows the effectiveness of a low w/c in decreasing the chloride permeability of concrete.





Very low diffusion coefficients of  $2.0$  to  $1.0 \times 10^{-6}$  mm<sup>2</sup>/second were achieved even though coulomb values up to 2500 were reported. This data shows that very low permeability concretes may be the subject of non-conformance and rejection where a limit of 1000 coulomb has been specified, yet values up to say, 2500 coulombs will provide equivalent high quality, low permeability concrete.

Further, the RCIP test is not ion specific<sup>(12)</sup> and, as a result, several concurrent processes are likely to be involved within the influence of an electric field. Because the test is not ion specific, it follows that the test is not a good indicator of concrete quality when various binder types are used, and in particular, where supplementary cementitious materials are used. Results can also produce misleading results when corrosion inhibitors such as calcium nitrite have been admixed into concrete. The results using this test can indicate higher coulomb values, that is, lower resistance to chloride ion penetration than from tests on identical concrete mixes without calcium nitrite. Conversely, the addition of silica fume will lower the coulomb values. Clearly, the contractor is exposed to considerable risk if the RCIP test is specified for conformance testing at the quality control level of a project.

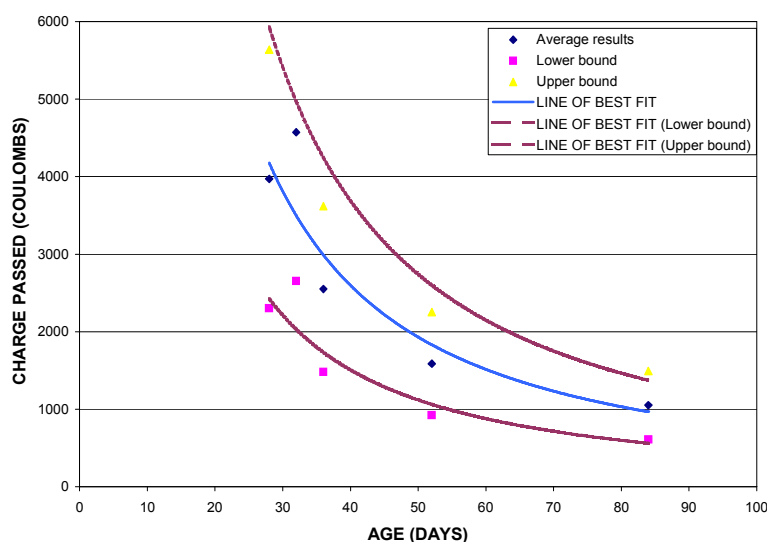
As well, there is concern regarding the precision and repeatability of results. These procedures are subject to testing error and variation to the property being tested in a similar manner to compression testing. The specification should therefore state a characteristic value and allow statistical acceptance similar to that provided in AS 1379<sup>(13)</sup> for the sampling and testing of concrete for compliance. This would require knowledge of the standard deviation usually achieved in laboratory and field-testing for the particular test. For example, acceptance criteria for the compressive strength of concrete is based upon a 95% confidence limit (ie 5% defective) in the normal binomial distribution of results where the dispersion or variance<sup>(14)</sup> about the mean value is:

$$S^2 = \sum (x_i - x_{av})^2 / (n-1) \quad \text{for summation limits } i = 1 \text{ to } n \quad \text{and therefore;}$$

the mean strength required is  $x_{av} = F'c + 1.65(S)$

However, most specifications which nominate the RCIP test to determine the rate of ion migration state single, lower bound limits and do not recognise that statistical variance about a mean value will occur.

Figure 4 shows the recent actual chloride permeability of a Classification C (marine concrete) mix with a w/c=0.30 containing cement plus flyash. The 28-day charge passed of 4000 coulombs would indicate that this concrete has high chloride permeability, yet at age 84-days the charge passed is 1050 coulombs. This example indicates the time effect in



**Figure 4** **SCI MIX 184**  
 Rapid Chloride Permeability to ASTM C1202-97  
 Trendline calculated by method of least squares fit

assessment for both  $D_e$  and the C1202 test. On the basis of 28-day testing the concrete would be deemed non-conforming under the contract. ASTM C1202 states that for single-operator laboratory precision, the coefficient of variation of a single test result has been found to be 12.3%. The coefficient of variation between laboratories has been found to be 18%. This means that the results of two properly conducted tests by the same operator or different laboratories on concrete samples from the same batch and of the same diameter can differ by more than 42% and 51% respectively. This is a significant statement that should alert specifiers for the need to introduce tolerance limits into the acceptance criteria.

Figure 4 also shows the upper and lower bound coulomb values for the noted precision.

For any one of the above concerns a contract dispute is liable to result between the parties where a single figure coulomb value has been specified. Accordingly, the test is not appropriate for use as a quality control test in contracts and must not be specified that way.

## CONCLUSIONS

The Concrete Institute Recommended Practice "*Performance Criteria for Concrete in Marine Environments*" has set out to review and put forward recommendations for acceptable performance criteria with respect to the whole of the design and construction process for concrete in marine environments. It has considered the appropriateness of test methods as applicable at the Design, Pre-qualification and Quality Control stages in a contract. The main conclusions that can be drawn from the document and the foregoing discussion are:

- (i) It is clear that the widely used performance criteria based on ion diffusion models are currently still in the experimental stage and are therefore not satisfactory for the basis of acceptance testing of production concrete
- (ii) That any form of short-term testing other than compressive strength may or may not be a reliable indicator of long-term performance
- (iii) That caution needs to be exercised in using performance criteria for marine environment exposures, especially where correlations with long-term test results are necessary but are not available
- (iv) That low (0.30-0.34) w/c concrete will be most effective in decreasing the chloride permeability of concrete.
- (v) That the coulomb values in Table 1 in the ASTM C 1202 document (reproduced as Table 7 in the CIA document) are misleading and should not be used for compliance criteria in specifications as the test was developed only for use as a rapid comparison between concrete mixes
- (vi) That concretes with coulomb values of around 2500 can have similar low permeability properties to concrete with 1000 coulombs
- (vii) That contractual disputes are likely where insufficient time is provided to allow a proper assessment of performance based testing in the pre-qualification of materials
- (viii) That contractual disputes are likely where inappropriate tests are specified for routine quality control acceptance criteria
- (ix) That the risk to the contractor is high where badly constituted specifications form part of the contract documents

Currently, controversy surrounds the specifying and use of emerging short-term durability based test methods to predict the long-term durability performance of concrete. It is a considered view that insufficient confidence can be placed on results obtained from such test methods.

From a contractual viewpoint, only **acceptable** criteria can be specified. The Recommended Practice document (Z13) recognises compressive strength as the **only** acceptable criterion for pre-qualification and quality control. Accordingly, in the immediate future, compressive strength should be the only quality control test that should be specified and used under a contract.

## APPENDIX – A

**Table 5**  
28-day average chloride diffusion coefficient (least squares best fit) for chloride profile shown in Figure 6a below

NORDTEST METHOD: NT BUILD 443 - 1995

**Client:** Structural Concrete Industries

**File No:** 410/99

Lab Sample No. 18845

Sample Number **SCI 57273-K -1**

Date of Casting 19-Oct-99

Date of Grinding 14-Dec-99

Background Chloride Content **C<sub>o</sub>** **0.001** %

Chloride Content at Surface **C<sub>s</sub>** **0.660** %

Exposure time in Salt Solution **t** **28** days

**t** 2419200 sec

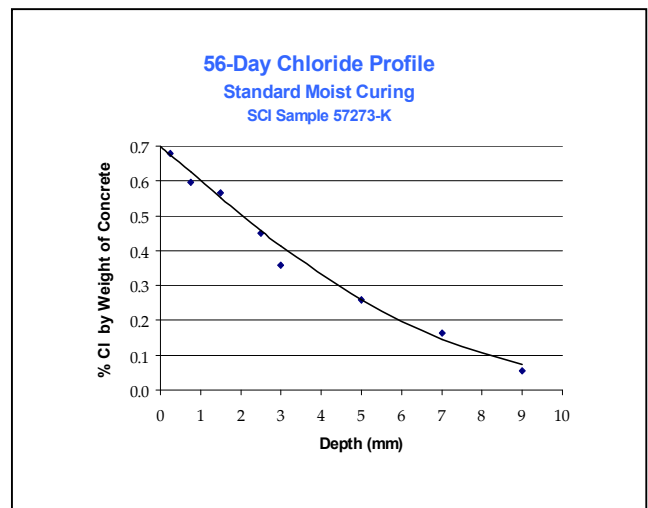
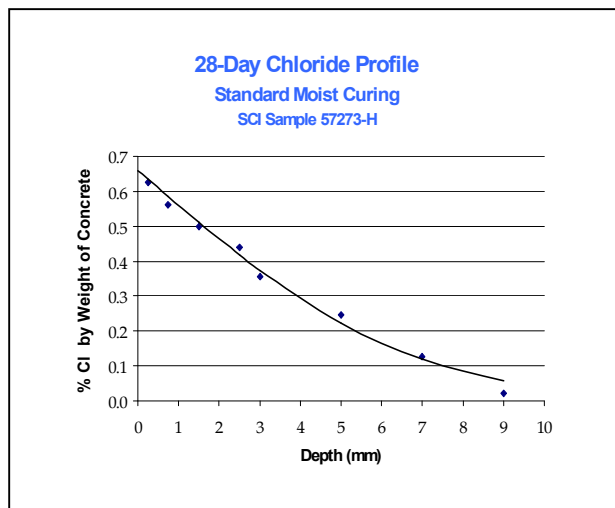
Depth, mm	% Cl by wt	Macros Eq	Trial Di
0.25	0.625	#VALUE!	5.66E-12
0.75	0.562	#VALUE!	4.50E-12
1.5	0.499	#VALUE!	5.08E-12
2.5	0.439	#VALUE!	5.98E-12
3	0.355	#VALUE!	6.00E-12
5	0.246	#VALUE!	5.83E-12
7	0.127	#VALUE!	7.92E-12
9	0.022	#VALUE!	3.91E-12

Average D = 5.61E-12 m<sup>2</sup>/sec

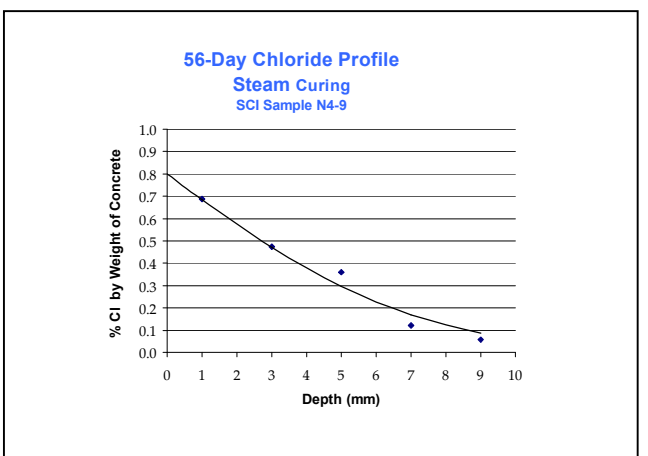
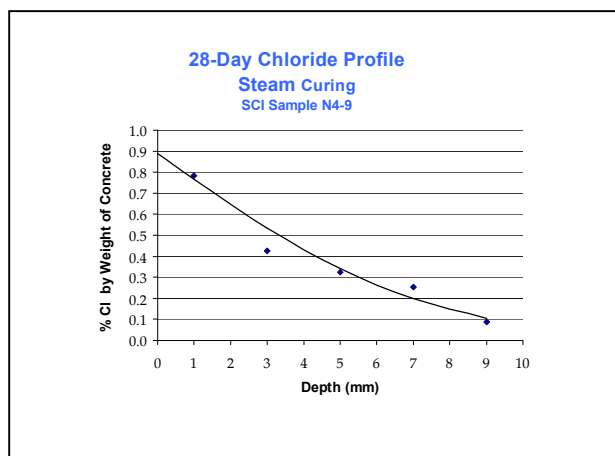
Best Fit Diffusion Coefficient **D** = 5.61E-12 m<sup>2</sup>/sec

Best Fit Chloride Content at Surface **C<sub>s</sub>** = Trial Di %

**Note:** 28 days standard water cured then submersed in salt solution for 28 days



**Figure 6a and 6b** – Chloride profile after 28-days and 56-days in salt solution after 28-day Standard water curing



**Figure 7a and 7b** – Chloride profile after 28-days and 56-days in salt solution after 1-day Steam curing + 27-day Air

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