A Collaborative Study for the Development of a Standard Critical Chloride Threshold Test Method

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1. INTRODUCTION

1.1. Problem Statement

One of the most important and costly deterioration mechanisms affecting reinforced concrete structures is the corrosion of the embedded steel reinforcement. Steel reinforcement is unlikely to corrode in good quality concrete due to the formation of a thin protective oxide film (passive film) in the highly alkaline concrete pore solution [1]. Steel bars remain passive as long as the high alkalinity of concrete pore solution is sustained. However, the passive film can break down causing initiation of active corrosion due to two major causes; presence of chlorides at the steel-concrete interface above a threshold amount and carbonation [2]. Carbonation is the decrease of alkalinity in concrete environment and is caused by reactions of carbon dioxide (CO_2) penetrating into the concrete from the atmosphere. Chlorides may penetrate concrete from external sources such as seawater and deicing salts or may be admixed with concrete ingredients during concrete production. The amount of chlorides at the steel-concrete interface necessary to initiate corrosion is referred to as the critical chloride threshold (C_{crit}) [3, 4]. The C_{crit} for steel reinforcement in concrete is thought to be a distribution of values dependent on the quality of the constructed concrete, constituent materials and their proportions, exposure environment, and the laboratory test method used.

Over the last several decades chloride-induced corrosion of reinforcement in concrete has been studied extensively and numerous laboratory test methods have been developed to quantify the C_{crit} [5]. A literature survey reported that 29 authors published a wide range of different C_{crit} values based on over 20 different test methods, criterion, and/or materials. The C_{crit} data collected and published with these different laboratory test methods in the literature exhibits significant scatter and varies between 0.1 and 3.1% by weight of binder [4]. Important differences between the test methods used and differences between the physical and chemical properties of materials used in different studies contribute to the large scatter of published data. Development of a standard laboratory critical chloride threshold test method to decrease the variability in the reported C_{crit} data is necessary. This study developed a framework for a standard laboratory test method and evaluated two different test methods developed within this framework by two different groups. Test methods were evaluated through a round robin test with participation of multiple laboratories.

1.2. Objectives of the Study

A large part of C_{crit} variability reported in the literature is caused by differences in used test methods and by physical and chemical differences of the tested materials. The goal of this project is to develop a standard laboratory test method to determine the critical chloride threshold, C_{crit}, of a cementitious system with an embedded reinforcement. To reach this goal, this study had three objectives;

- The first objective of this study was to develop a framework for a standard laboratory test method. Alternatives for important components of a standard test method, such as exposure conditions and others, were evaluated based on literature by a subgroup of ACI 222 corrosion committee members to develop the framework.
- The second objective was to evaluate two different test methods developed within the established framework using a set of materials procured from the same source. Multiple laboratories participated in a round-robin test to perform the two test methods using materials supplied from one source to reach this objective.

- The third objective was to recommend one of the evaluated test methods as a standard laboratory C_{crit} determination method based on observed variability and repeatability.

1.3. Research Significance

The large variability of published C_{crit} data and a lack of standard laboratory test method creates significant difficulties for the concrete industry. Researchers cannot compare C_{crit} values obtained from their research to a standard value. Practitioners evaluating the condition of reinforced concrete structures for maintenance and rehabilitation cannot make a reliable assessment based on measured chloride contents. Service life modelling of reinforced concrete structures are extremely important for making important decisions early on during the design of structures and also for making maintenance decisions during the service life of structures. Large variability in reported C_{crit} values cause large uncertainties in the service life models including the one developed by ACI 365.

The amount of chlorides included in fresh concrete through its constituent materials should be limited to a value significantly lower than the C_{crit} of the system. These limits are called allowable chloride limits, C_A. A recent review of ACI documents indicates that ACI publishes at least five different C_A limits in 12 different documents (6 codes and specifications, and 6 guides and reports) and there is little consensus among the different ACI committees. This variability in C_A limits is a direct result of variability of reported C_{crit} values. This lack of guidance and consensus also leads to challenges in the concrete industry. The standardized laboratory test method and data set established through this study will be instrumental in the solution of these different important issues.

1.4. Background

Total variability observed in C_{crit} data has four main sources as shown in Figure 1; random variability, inter-laboratory variability, materials, and test method used to determine the C_{crit} value. The random variability refers to the fact that every specimen evaluated in a test sample will be slightly different and not exactly the same. The mean C_{crit} value calculated from C_{crit} values of all the specimens in a sample should be a good approximation of the true C_{crit} value of the evaluated system. In general the larger the sample size, the better the approximation. Inter-laboratory variability is the reproducibility or the variability among results obtained by different laboratories performing the same test on the same system. Physical and chemical properties of materials procured from different sources can also have a significant effect on the variability of test results, even if these materials satisfy the requirements of the same ASTM classifications, e.g., Type I/II cements obtained in various parts of the country from different manufacturers [6].

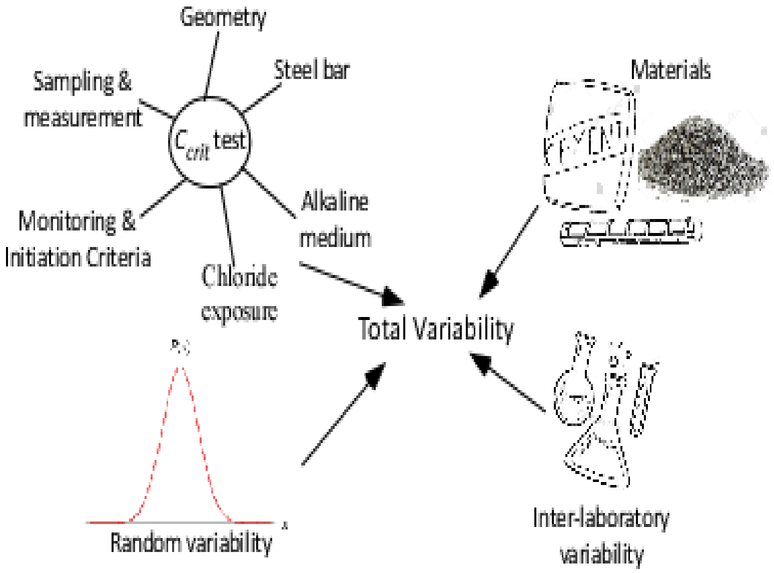


Figure 1: Four sources of variability

There are 6 aspects of a laboratory C_{crit} test method that can affect the results significantly;

steel sample type and condition, alkaline medium, chloride exposure scheme, corrosion initiation criteria, method of chloride content sampling and determination, and sample size and geometry. There are multiple options for each of these aspects and the test methods used in the literature are different from each other in one or multiple of these aspects [7]. Development of a standard laboratory C_{crit} test requires design of all these 6 aspects. The ideal developed standard laboratory test method should;

- provide low random variability
- provide low intra- and inter-laboratory variability
- be simple and inexpensive to perform
- provide results in a reasonable time period.

In 2009 a RILEM technical committee (TC) 235-CTC was formed to develop a reliable test method capable of delivering data with acceptable measurement uncertainty [7]. The committee worked on this until 2015 and proposed a test method which was evaluated with a round robin test. Eight laboratories performed tests in accordance with the developed test procedure. It should be noted that although the materials they used matched the same specifications, they were not obtained from the same source and each laboratory obtained their materials from different sources. Figure 2 shows the test setup used for the RILEM round robin test. The TC 235 decided to use concrete with 0.6 in (16 mm) maximum size of aggregate as alkaline medium. Steel samples were subjected to a pre-rusting procedure to prevent variability that may result from variability of as-received surface conditions. Steel samples were placed vertically in 6 in (150 mm) cubic molds to prevent void formation around the bars that can affect corrosion. Concrete samples were later cut to leave a 0.4 in (10 mm) cover, leaving the top section of sample as shown in the figure to decrease chloride exposure to the steel sticking out of the concrete. After a drying procedure, samples were exposed to 3.3% NaCl solution. The open circuit potential of samples were monitored to determine initiation of corrosion. A potential drop from its initial passive level by at least 150 mV in 24 hours or less, that remained at the lower level over a period of 7 days was considered the indication of corrosion initiation. Once corrosion initiation was detected, acid soluble total chloride content at the depth of the rebar was determined by grinding areas adjacent to the rebar. This description shows how the RILEM committee made decisions about the earlier discussed 6 aspects of a laboratory method. A more detailed discussion on how these decisions were made is in a paper published in RILEM technical letters [7].

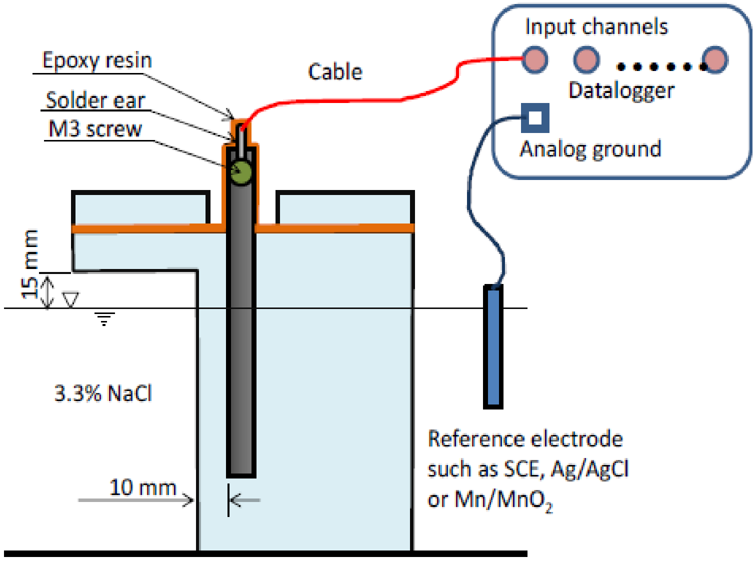


Figure 2: Test setup developed by RILEM TC 235-CTC

Only three out of the eight participating labs reported corrosion initiation of samples between 14 and 180 days during the round robin testing and other laboratories didn't report initiation in different testing durations varying between 290 and 800 days. The measured average C_{crit} values of the three laboratories varied between 0.94 and 1.59% by mass of cement. Chloride content values measured from samples that did not initiate corrosion varied between 0.5 and 2.8% by mass of cement. The developed laboratory test method was considered unsuccessful due to majority of samples not activating even in long testing durations. The pre-rusting procedure used to prepare the steel samples was preventing a drop in steel potential masking initiation of local corrosion. Not enough drying of samples before testing and surface densification due to calcite formation were among other possible causes for delaying initiation of corrosion. Although the RILEM study was not successful, they recommended to keep the test conditions as close to the conditions in real structures as possible for future test development attempts.

In Fall of 2017 the ACI committee 222 on corrosion of metals in concrete established a task group, TG1, to work on the development of a standard laboratory C_{crit} test method. TG1 met for the first time in March 2018 at Salt Lake City, UT to start developing a framework and started discussions on each of the six aspects of the test method. In May 2018 TG1 members met online to finalize the framework for a standard laboratory C_{crit} test method. The details of the developed framework are provided in Chapter 2. Considering how one decision made about the steel conditioning caused the whole round robin test effort of RILEM to be unsuccessful, the ACI committee decided to develop two different test methods based on the developed framework for redundancy. Dr. David Trejo from Oregon State University and Dr. Neal Berke from Tourney Consulting group developed two laboratory test methods and presented them to the TG1 during their online meeting in August 2018. After discussions at the TG1 meeting, the two groups were asked to revise and clarify certain parts of their prposed test methods by September 2018. The revised test methods were presented to ACI 222 committee during the Las Vegas ACI convention in October 2018. Both proposed test methods used mortar as an alkaline test medium as requested by the developed framework. At this meeting the 222 committee decided to include both methods in the round robin testing. It was also decided to add a third test method developed by Dr. Carolyn Hansson from University of Waterloo in the testing program. Different from the first two test methods, the third test method used a synthetic mortar solution as the alkaline medium. It was decided to perform this test method on the same materials used for the round robin testing to create an additional data set. In case both test methods provided significantly different C_{crit} values with low random variability, the results of the third test could be used to make a choice between the two test methods.

It should be noted that all materials used in this round robin testing were procured from the same source and distributed to the participating labs for testing. This should eliminate the variability in test results due to different materials. Use of developed test procedures on the same materials by different labs provided data on random variability and inter-laboratory variability for the chosen test methods.

1.5. Organization of Report

Chapter 1 provides the problem statement and the objectives of this study with a background on the development of the laboratory test methods included in this study. Chapter 2 provides the details of the laboratory test method framework developed by the ACI 222 TG1. Chapter 3 provides the details of the three methods that were included in this study; two test methods that were included in the round robin test and the third in-solution test method. This chapter also provides information on the materials and sample preparation procedures of the round robin test. Chapter 4 provides the test results obtained from three tests methods using the materials obtained from one source. Chapter 5 provides conclusions and recommendations regarding the use of developed test methods as a standard laboratory C_{crit} test method.

2. Standard Laboratory C_{crit} Test Method Framework

Design of a laboratory C_{crit} test method requires making decisions at least on six aspects of the test method that can affect the results. These aspects are:

- Steel sample type and surface condition
- Alkaline medium
- Chloride exposure scheme
- Sample size and geometry
- Corrosion monitoring and initiation criteria
- Method of chloride content sampling and determination

The first task of TG1 was to make decisions on these aspects based on observations in the literature and member's personal experiences to develop the necessary framework. The decisions made by TG1 and the reasoning are listed below.

2.1. Steel Type and Surface Condition

TG1 decided that ASTM A615 grade 60 bars in as-received and degreased condition should be used to evaluate the test methods in the round robin test. In the as-received condition steel surface exhibits different zones; bare steel, steel covered with mill scale and/or pre-existing rust layers. Chemical and electrochemical reactions that will take place when these zones come into contact with the high pH pore solution are different [8]. Many C_{crit} studies in the literature used steel bars that were highly polished to remove the mill scale and pre-existing rust aiming to decrease variability. However studies have shown that polishing the steel surface can significantly increase the C_{crit} values required to initiate corrosion [9, 10]. On the other hand, a study carried out by RILEM TC 235-CTC on steel samples collected from 9 different countries showed that in as-received condition, the surfaces of the steel bars were significantly different and they showed significant differences in electrochemical behavior depending on the amount of red/brown rust present on the surface [11]. This observed variability was the reason why the RILEM study trying to develop laboratory C_{crit} test decided to pre-rust their samples after applying a chemical cleaning. However, this decision turned out to be problematic, causing masking of corrosion initiation of the samples [7]. TG1 decided to use steel samples in as-received conditions to keep the test conditions as close as possible to the conditions in real structures. The variability of as-received surface conditions was not seen as a problem, since the round robin test was going to use the steel from the same heat of the same manufacturer minimizing the variability. ASTM A615 grade 60 bars are the most commonly used reinforcement bars in North America for typical applications.

2.2. Alkaline Medium

TG1 decided to use mortar similar to the one defined in ASTM C109 as the alkaline medium of the standard test method. Multiple corrosion studies in the literature tested steel in alkaline solutions, either saturated $Ca(OH)_2$ solutions or simulated concrete pore solutions, instead of mortar or concrete [12–18]. In solution testing eliminates the long time necessary for chlorides to penetrate the concrete cover but different studies have shown that chloride threshold for active rebar corrosion is lower and the corrosion rates are higher for steel tested in saturated $Ca(OH)_2$ than in a pore solution of the same composition as expressed from mature concrete [4, 19]. When considering mortar vs. concrete as alkaline medium, mortar was selected due to two reasons. The first reason was related to variability in chloride ingress due to existence of coarse aggregates. A study evaluating the spatial variability of chloride in concrete within homogeneously exposed areas stated that the local, microscopic chloride concentration may deviate from the average, macroscopic concentration by ± 20-70 % [20]. The same study concluded that while other factors such as exposure conditions, water binder ratio, and binder type moderately influence the chloride variability, the primary cause for dispersing the chloride front was the presence of coarse aggregates. The second reason for choosing mortar over concrete was the possibility of using smaller cover depths when coarse aggregates are not used. As stated earlier, penetration of chlorides through the cover may take a long time and decreasing the cover depth can help accelerate the test significantly.

2.3. Chloride Exposure Scheme

C_{crit} test methods used in the literature can be classififed into two major groups; natural tests and accelerated methods [21]. Natural tests do not use any external electrical action that can change the electrochemical reactions at the surface of the reinforcement during depassivation. In natural tests chlorides may be penetrating from the external environment or may be mixed in during casting (admixed chlorides). Penetration of the chlorides from the external environment can be by pure diffusion where samples are kept saturated or by a combination of capillary suction and diffusion [4]. Capillary suction is achieved by drying the samples at the surface. Many studies in the literature used cycling wetting and drying to accelerate chloride migration similar to the procedure described in ASTM G109 test method [22, 23]. RILEM TC 235-CTC in their effort used an initial drying period followed by a continuous chloride solution exposure [7]. Admixed chlorides may accelerate the test and provide a more homogeneous distribution of chlorides in the system however they may also prevent steel from building a passive layer. Admixed chlorides may also accelerate cement hydration and cause increases in the porosity of concrete, increase the conductivity of the pore solution, and change the amount of chemically bound chlorides [19]. Accelerated test methods can be divided into two groups; potentiostatic methods and migration methods [21]. Potentiostatic methods polarize the steel to a predetermined and constant potential, typically more noble than -200 ± 50 mV vs SCE, where the passive layer does not vary significantly [24]. For potential more cathodic than -200 ± 50 mV C_{crit} were found to increase progressively. Migration methods use an external potential gradient to accelerate the ingress of chlorides. The steel sample being evaluated can be between the two electrodes used to apply the external potential [25], or the steel sample can be embedded together at the same level in concrete with the anode used to create the external potential [26]. Application of an external potential may cause migration of other ions present in the pore solution and may change the composition of the pore solution affecting the C_{crit} value. TG1 decided to use a natural test setup to keep the test conditions as close as possible to the conditions in real structures and to use a combination of initial drying and/or cycling wetting and drying to accelerate the test after the mortar samples were wet cured. Samples were to be exposed to an external chloride solution similar to the one used in ASTM C1556 which uses a NaCl solution. Majority of C_{crit} studies in the literature used NaCl to evaluate chloride induced corrosion instead of *KCl* or $CaCl_2$ [4]. Studies have shown that chloride binding in the case of $CaCl_2$ can be significantly higher compared to NaCl and may induce greater corrosion rates [27,28].

2.4. Sample size and Geometry

TG1 decided not to put any restrictions on the sample size and geometry and to leave the decision to the groups developing the test methods within the developed framework. Similarly, a minimum or maximum cover depth was also not determined. A recent study evaluating the effect of sample size on C_{crit} values measured in laboratory studies stated that small laboratory samples can tolerate higher chloride concentrations than larger specimens and also exhibited higher variability [29, 30]. The study explained this size effect by inhomogeneities at the steel surface and indicated that increasing the specimen size will increase the probability for the presence of conditions favoring corrosion initiation at lower chloride concentrations. Although TG1 did not put any restriction on the specimen size, the consensus was to prefer smaller laboratory specimens for ease of application in the laboratory. However, considering the expected increased variability, TG1 recommended testing of minimum of 10 samples for each laboratory test method. Another important aspect of a C_{crit} test method is the cathode to anode surface area ratio. Several authors studied the impact of this factor and stated that the measured macro-cell corrosion currents increased with increasing cathode to anode ratio up to a limit macrocell current value [31, 32]. Although there is no indication that C_{crit} value depends on the cathode to anode ratio, TG1 recommended a minimum 2:1 ratio in the test setups to generate large enough macro-cell currents to be easily detected, if the test method uses macro-cell current as an initiation criteria.

2.5. Corrosion monitoring and initiation criteria

There are various methods in the literature that were used to monitor and determine initiation of corrosion, such as steel potential, linear polarization, electrochemical impedance spectroscopy (EIS), macro-cell current, and others. Steel potential, E_{corr}, is a commonly used parameter to assess the condition of steel in concrete. When chlorides reach the threshold level and initiate corrosion of steel by damaging the passive film, the potential of the steel due to equilibrium of corrosion reactions should decrease which establishes the basis of recommendation of ASTM C876 [19]. There are other factors that may affect steel potential such as oxygen availability, resistivity of the environment etc. However, in a laboratory where samples are kept in a stable environment and data collection is performed with a controlled procedure, a drop in the potential should mainly be related to a switch from passive to active corrosion state of the steel. It should be noted that the drop in steel potential does not provide information on the rate of corrosion although multiple researchers have found good agreement between potential values and corrosion rates measured by polarization resistance method [33, 34].

Measurement of macro-cell current between the steel that is being evaluated and another electrode to determine initiation of corrosion is also a common method used in the literature [35–38]. Macro-cell current experiments are designed to expose the steel being evaluated to a corrosive environment and have it electrically connected, typically over a resistor, to another steel that is being protected from corrosion. Flow of current (macro-cell current) can be detected over the resistor between the steel bars, when corrosion is initiated on the steel exposed to the corrosive environment. The steel exposed to the corrosive environment acts as an anode and the steel protected from corrosion acts as a cathode. ASTM G109 test (and modifications of it), which was designed to evaluate chemical admixtures, was used by many researchers for macro-cell corrosion studies [23, 39]. Studies using macro-cell current became popular because they can be measured directly without expensive equipment [19]. It should be noted that these experimental setups typically ignore micro-cell corrosion where cathodic and anodic areas develop at adjacent locations on the same steel. There are studies that have shown that micro-cell corrosion can be the dominant process [40, 41]. TG1 decided that the developed tests should at the least use steel potential and/or macro-cell current to monitor corrosion. The committee also recommended that if a macro-cell current setup is being used that the resistance between the anode and cathode be measured to calibrate the capacity of the resistor being used in the setup. A potential difference is measured across the resistor which is further used to calcuate the macro-cell current.

Another issue reported in the literature when corrosion initiation is being monitored using steel potential is the depassivation/repassivation of some samples in a test group. [23, 42]. In this case, samples can exhibit either a sudden or slow drop over time in potential which is followed with an increase in potential indicating repassivation. TG1 recommended that the initiation criteria determined in the developed tests should be satisfied for at least two consecutive measurements before stopping the tests to ensure that initiation of a stable corrosion process was detected.

2.6. Method of Chloride Content Sampling and Determination

After the initiation of corrosion is determined, the chloride concentration at the steel concrete interface needs to be measured to determine the C_{crit} value. TG1 decided that a minimum of 10 g powder sample should be collected from samples at a depth between the top and bottom of the evaluated steel bar. As mentioned earlier different studies in the literature have shown spacial variability of chlorides along the steel concrete interface at chloride initiation [20, 29]. A recent study used a macro-cell current type setup to evaluate C_{crit} values in fly ash containing concrete specimens and determined the chloride contents along the bar separating the exact area of the steel where corrosion products were present [43]. The average chloride content in the area where corrosion products were present was 1.8 times higher than the average of all the other measurements along the steel. Collecting powder samples, and large enough powder samples, to analyze exactly from the point of observed corrosion is very difficult. Therefore TG1, aimed to get an average chloride content at the determined depth and requested the sample to be a combination of powder samples collected at least from three different locations along the bar. TG1 also decided to determine the total acid soluble chloride content of the collected powder samples following ASTM C1152, using potentiometric titration.

There are multiple alternatives for each of the six aspects of a laboratory C_{crit} test method and each alternative and combination of alternatives have their own advantages and disadvantages. The objective of TG1 was not to be too restrictive in the development of the framework but to set a wide boundary to make sure that the proposed test methods will be comparable with similar assumptions. TG1 also decided that the used mortar mixture should be further characterized by casting additional samples for determination of pH, surface and bulk electrical resistivity. There are multiple methods in the literature that were used to determine the pH of cementitious mixtures [44]. TG1 recommended the use of an in situ leaching (ISL) method that was first described by Sagues et al. [45]. A small cavity is drilled in the surface of the sample and a small and known amount of deionized water is added. The pH of the solution is monitored until an equilibrium is reached. A study compared pH results obtained by the ISL method and pore water extraction methods and concluded that the methods give comparable results, the difference being less than 0.2 pH units [46].

3. Experimental Work

After the development of the framework by TG1 as described in Chapter 2, two test methods were proposed to TG1 by two different groups. The first test method, referred to as the OC_{crit} test method, was developed by Dr. David Trejo from Oregon State University. The second test was developed by Dr. Neal Berke from Tourney Consulting Group and is referred to as the ASTM type test method because of its similarity to the ASTM G109 test setup. Both methods were developed considering the requirements of the TG1 framework. With approval of the ACI 222 committee, TG1 decided to test both methods in a round robin test setup with participation of multiple laboratories. To limit the variability of results, all materials used for testing were procured, processed, and shipped to the participating laboratories by University of Missouri - Kansas City (UMKC). Table 1 shows the participating laboratories for both test methods. Although it didn't meet the test framework developed by TG1, a third test method developed by Dr. Carolyn Hansson from the University of Waterloo was also added to the test program. This test was not performed by multiple laboratories but only at University of Waterloo on the same set of materials that were tested by the OC_{crit} and ASTM type test methods. In case, both test methods provided different results for the same set of materials and both test methods provided low inherent variability and inter-laboratory variability, the third test could provide an additional data point to make a recommendation. Detailed descriptions of the OC_{crit} test method and the ASTM type test method are provided in Appendix A and B of this report, respectively. Some parameters of the ASTM type test method were modified for the round robin test and these modifications are listed in Appendix B as well. This chapter will briefly describe all three test methods and also provide information on the materials and their processing performed at UMKC before they were shipped to different labs.

Table 1: Laboratories participating in the study

OC_{crit}	**ASTM Type**
Oregon State University(OSU)	Tourney Consulting
University of Missouri-Kansas City(UMKC)	University of Missouri-Kansas City(UMKC)
University of Kansas(KU)	University of Kansas(KU)
CTL group	CTL group

3.1. Materials

Cement: Portland cement that meets the requirement of ASTM C150 Type I/II was used as the only source of cementitious material. To minimize variability, all cement used in this research by different laboratories was procured from the same production batch of the Ash Grove Cement Company. The XRF analysis of the procured cement is presented in Table 2. The alkali content of cement (calculated by $Na_2O + 0.658K_2O$) was 0.5%.

Table 2: Chemical composition of the cement

Oxide	**% amount**
SiO_2	21.32
Al_2O_3	386
Fe_2O_3	2.98
CaO	62.36
MgO	2.03
SO_3	2.65
Na_2O	0.18
K2O	0.49
TiO_2	0.23
P_2O_5	0.07
Mn_2O_3	0.1
SrO	0.24
LOI	3.75
Total	100.26
C_3S	54
C_2S	20
C_3A	5
C_4AF	9

Steel: #5 (16 mm) ASTM A615 grade 420/60 steel reinforcement from the same production heat of CMC Steel Oklahoma was procured for the round robin testing [47]. The chemical composition of the steel reinforcement is presented in Table 3. As discussed in framework development, no surface treatment was applied to the steel except degreasing. Steel specimens were degreased by immersing them in xylene filled ultrasonic baths for 15 minutes after they were cut to the required lengths for the test methods. An oil-cooled band saw was used to cut the 10 feet long bars to prevent heating.

Table 3: Chemical composition of steel

Components	% by Mass
C	0.30%
Mn	0.99%
P	0.013%
S	0.042%
Si	0.19%
Cu	0.24%
Cr	0.16%
Ni	0.11%
Mo	0.052%
V	0.009%
Sn	0.009%
Al	0.002%
N	0.0144%
Carbon Eq A6	0.53%

Mortar mix design: TG1 initially recommended the use of the standard mortar defined in the ASTM C109 method for compressive strength testing of hydraulic cement mortars [48]. The standard mortar has a water to cement ratio (w/c) of 0.485 and sand to cement ratio (s/c) of 2.75. However, after initial trials of casting OC_{crit} samples, the workability of this mortar was found to be too low. The mixture was modified to have a w/c of 0.42 and s/c of 1.375. After an online discussion with participating labs, it was decided to use the same modified mortar for both test methods to have comparable results. After casting of ASTM type samples with the modified mortar at UMKC, Tourney Consulting, and University of Kansas, it was observed that the modified mortar was causing settlement and shrinkage issues. Therefore, ASTM type samples were retested at UMKC with modified mortar designs using s/c of 2.0 and 2.75 and CTL performed the last set of ASTM type samples using a mortar with s/c of 2.0. The three mixture designs used in the study with different s/c ratios are shown in Table 4. Standard silica sand matching the requirements of ASTM C778 was used as fine aggregates for all mortars.

Table 4: Mortar mix design for different s/c values

s/c	Cement, Ib/yd^3(kg/m^3)	Sand, Ib/yd^3(kg/m^3)	water, Ib/yd^3(kg/m^3)
1.375	1328 (788)	1826 (1084)	558(331)
2.0	1118(663)	2235(1327)	469(279)
2.75	941(558)	2586(1534)	395(234)

3.2. OC$_{crit}$ test Setup

The OC$_{crit}$ test setup, developed at Oregon State University, is a macro-cell current type test method. Detailed description of the test setup is provided in Appendix A. This section provides a brief overview of the method. The test method uses a dog-bone shaped mortar anode specimen with a fully embedded 5.5 in (140 mm) steel bar as shown in Figure 3. The steel bar located between the thicker ends of the specimen has a thin, uniform mortar cover with a radial cover thickness of approximately 0.19 in (4.8 mm). The thickened ends were designed so that the steel reinforcing bar would not corrode at the ends as a result of chloride exposure. Chlorides are expected to reach the steel-mortar interface in the thin mortar cover region faster compared to the ends of the steel bar. This setup is expected to prevent galvanic and/or crevice corrosion when the ends of steel bars are not completely embedded in mortar or protected with epoxy coating.

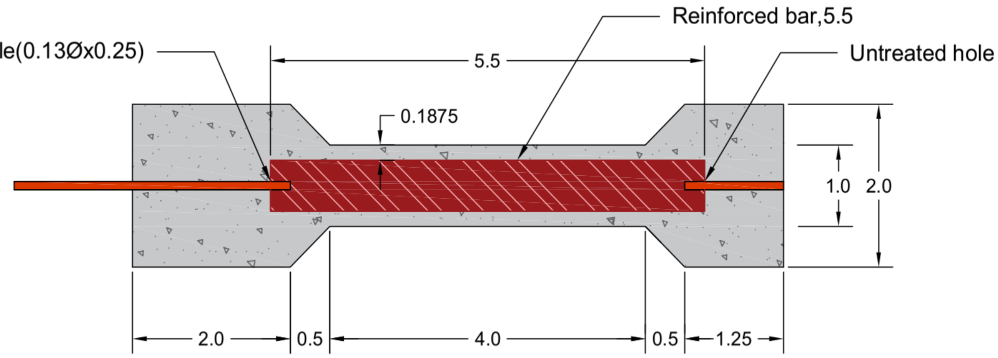
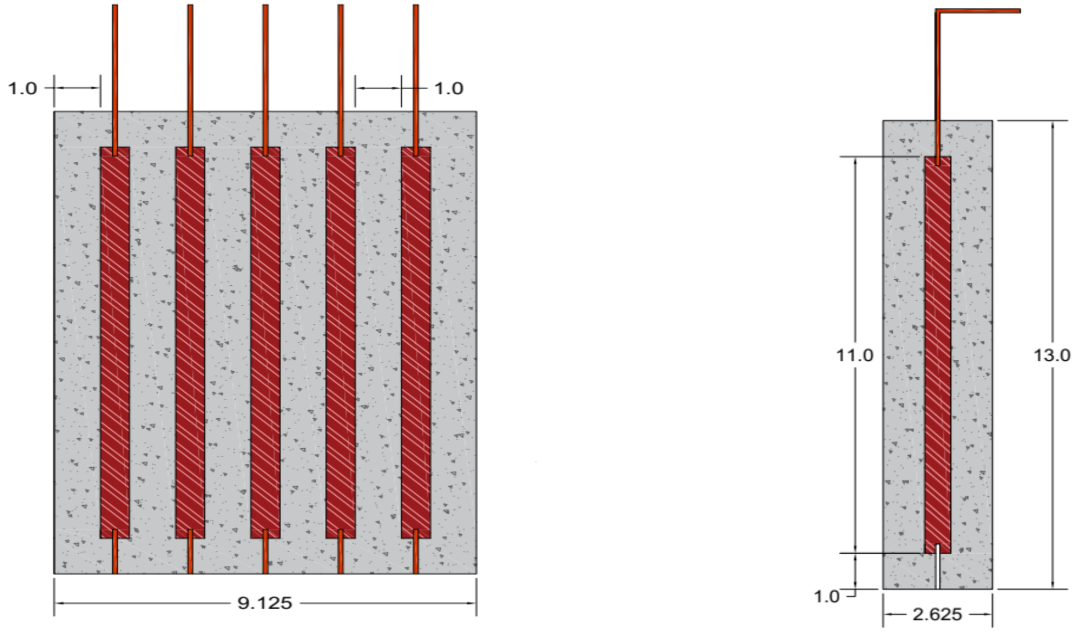


Figure 3: Cross sectional view of anode specimen (dimensions in inches)

A mortar prism with 5 completely embedded 11 in (280 mm) long steel bars is used as a cathode. The cathode specimen is designed to test five anode specimens simultaneously. The mortar cover around the steel bars is 1 in (25.4 mm) as shown in Figure 4. This setup provides a minimum of 2:1 cathode to anode surface ratio.

The anode and cathode specimens are connected by insulated wires and the solutions surrounding the anode and cathode are connected through a salt bridge to complete the corrosion cell as shown in Figure 5. Anode specimens are placed in separate containers and partially immersed in a saturated lime solution with 3.3% NaCl (2% chloride). The cathode specimens are partially immersed in saturated $Ca(OH)_2$ solution. The container with the cathode specimen is placed on an elevated platform such that the solution level in the cathode container is above the solution level in the anode container. This is done to prevent the contamination of solution in cathode container with chlorides.



(a) Front view (b) Side view

Figure 4: Cross sectional view of cathode specimen (dimensions in inches)

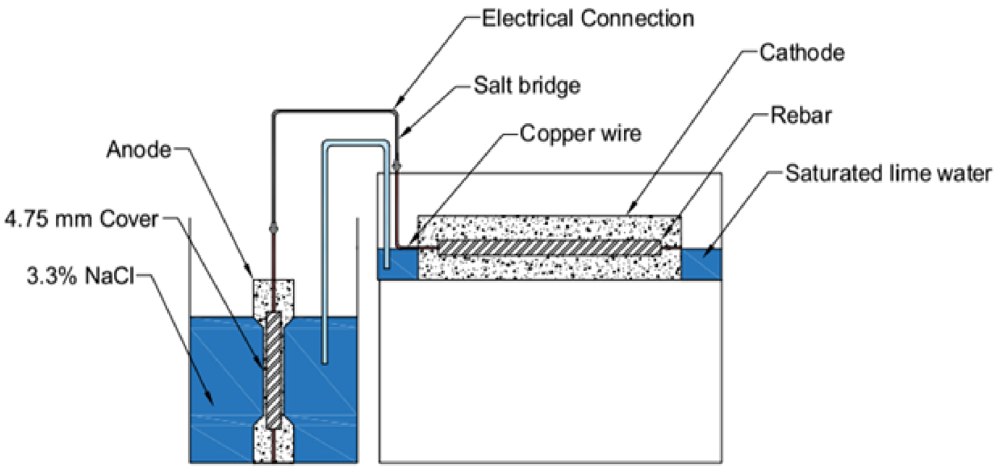
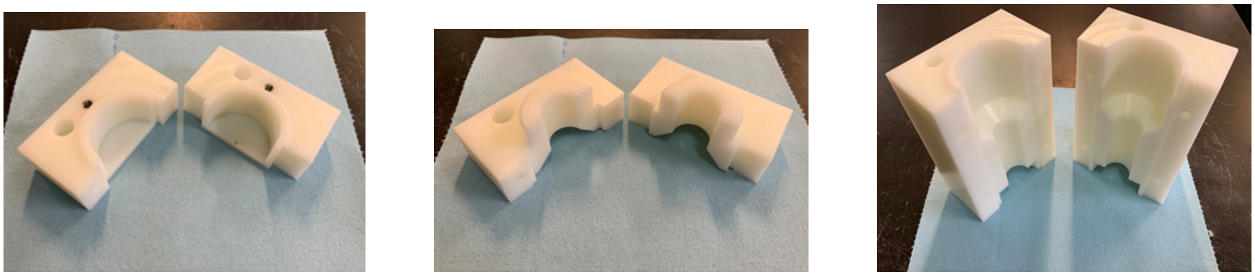


Figure 5: Schematic setup for OC_{crit} test method

The anode specimens were cast using specially produced 3-part plexiglass molds as shown in Figure 6 together with the steel sample. The bars were drilled and tapped at the bottom and top to attach copper wires which were used to center the bars in the molds during casting and for electrical connections later during the test. Due to their intricacy these molds were expensive to produce ($550 per mold). A set of five molds were produced at Oregon State University and the same set was shipped to UMKC, University of Kansas, and CTL group sequentially for casting of samples. Processing of all steel bars which included cutting, drilling, tapping, and degreasing were done at UMKC. Steel samples ready to be tested were shipped to the participating labs together with cement, sand, copper wires, tygon tubing and chemicals for salt bridges and molds.



(a) Bottom (b) Middle (c) Top

(d) Steel with copper wire

Figure 6: Parts of anode mold and steel specimen

UMKC performed the OC$_{crit}$ test twice, each time with a set of 10 samples. The use of 3 parts molds to cast the anode specimens was found to be a difficult task that required practice to obtain samples without large bugholes on the surface. At the conclusion of testing of the first set of samples, two samples were found to have their steel off-center. This was caused by bent copper wires during the casting that were used to hold the steel at center. Therefore, for the second set the procedure was slightly modified and copper wires were replaced with threaded 5-40 stainless steel rods as shown in Figure 7.

The specimens were cast using the mortar mixture with w/c of 0.42 and s/c of 1.375 as

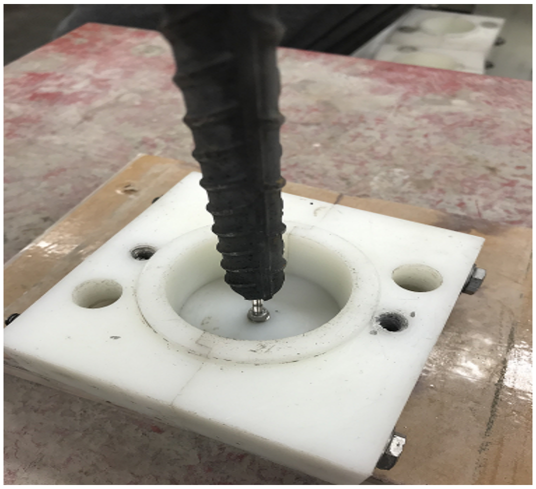


Figure 7: Stainless steel rod and nuts used to hold the steel

described earlier in the materials sections following the ASTM C305 procedure [49]. During casting specimens were consolidated using a mortar shake table. Five specimens were cast at a time, and covered with plastic sheets and wet burlap for 24 hrs. After demolding, the samples were transferred to a curing room for 27 days and cured at 72 °F and 100% relative humidity. After the curing period, the anode and cathode specimens were connected and placed in their respective solutions and the steel potential of the anodes was monitored daily using a Cu-$CuSO_4$ electrode and a high accuracy multimeter as shown in Figure 8. Testing was stopped when a sample exhibited a steel potential less than -350 mV for two consecutive readings.

The solutions were replaced weekly and the salt bridges were replaced at every three weeks. Most of the samples showed initiation of activation before three weeks as described in the results and discussion chapter. Activated specimens were washed with deionized water and dried in an environmental chamber (70 °F, 50% RH) for more than 24 hrs. Figure 9 shows the schematic process for the OC_{crit} test.

After drying, the thin mortar layer around the stem of activated anode specimens was broken and collected as shown in Figure 10. This mortar was ground until all mortar powder could pass

(a) (b)

Figure 8: OC_{crit} samples being tested

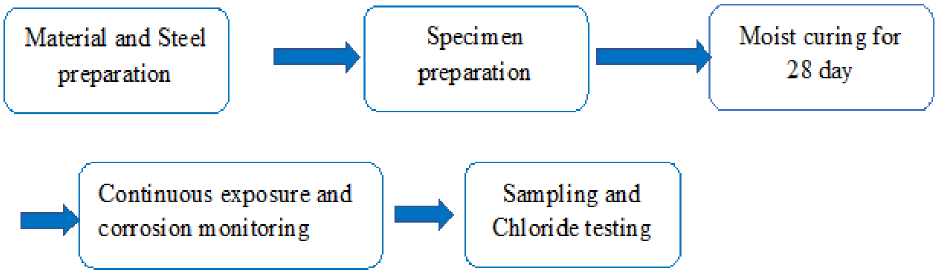


Figure 9: Schematic setup for OC_{crit} test method

sieve No. 20. The acid soluble total chloride content of mortar powder was determined following the ASTM C1152 [50]. It should be noted that the determined chloride content, Cl_{test} is the average chloride content of the whole 0.19 in (5 mm) thick cover around the steel. The critical chloride content, Cl_{crit}, which is the chloride content at the steel mortar interface, was calculated using the Cl_{test} and equation 1. This is a statistically developed equation by the researchers at Oregon State University. To develop this equation, the chloride content of a set of activated samples were determined at 0.04 in (1 mm) thick mortar slices around the steel as shown in Figure 11. The chloride content of the last slice (layer 5) at the steel mortar interface, Cl_{crit}, was correlated to the average chloride content of the whole cover, Cl_{test}. The set of samples in the preliminary study used for the development of this equation activated in the range of 10 to 70 days.

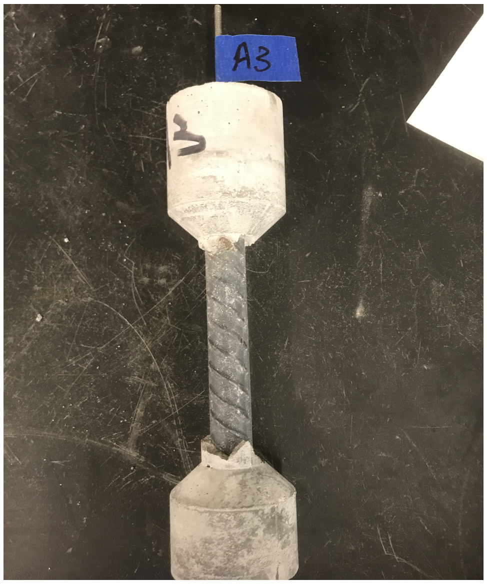


Figure 10: Sample collection form stem of OC$_{crit}$ specimens

$$Cl_{crit} = (0.492 + 0.004 \times T_{act}) \times Cl_{test} \tag{1}$$

Where:

Cl$_{crit}$ is critical chloride threshold,

T$_{act}$ is time to activation, and

Cl$_{test}$ is the average percent chloride content in the whole cover

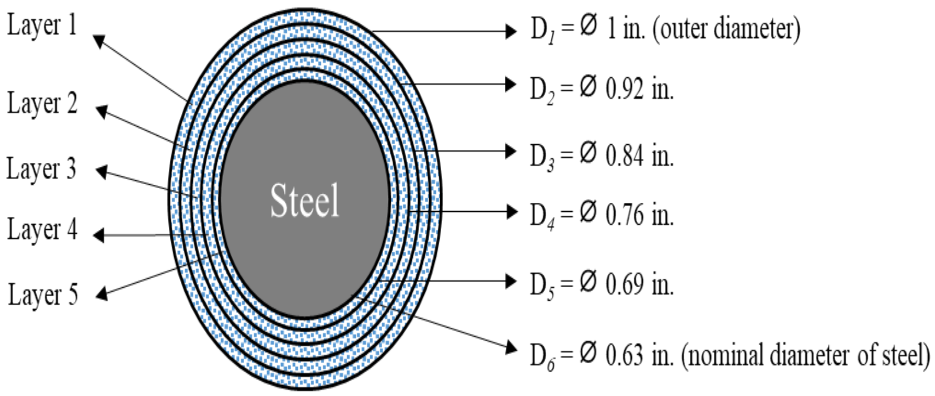


Figure 11: An illustration of the cross section of OC_{crit} specimen in the thin cover region and the different depth increments considered for chloride profiles (25.4 mm = 1 in.)

Salt Bridge: The detailed OC_{crit} procedure provided in Appendix A refers to a procedure developed by Kahrs et. al. [51] for the preparation of salt bridges but does not provide detailed instructions. Therefore, the procedure used in this study following this reference is described below:

1. 4.5 gram of agar, 30 gram of potassium chloride (KCl), and 100 gram of water created enough gel to fill a 3 ft (0.9 m) long flexible Tygon tube with inner diameter of 0.25 in (6.4 mm).

2. Agar and KCl mixture is heated until it gets to the consistency of a syrup.

3. The semisolid gel is poured into the Tygon tube placed in a tub of boiling water for approximately 4 hours.

4. The salt bridge is taken out of the boiling water and allowed to cool at room temperature. If the gel does not fully fill the tube, i.e., contains voids, the salt bridge is discarded. Prior to using the salt bridge, it should be checked for electrical conductivity using a multimeter.

3.3. ASTM type test Setup

The ASTM type test uses 6 x 6 x 5.5 in (150 x 150 x 140 mm) mortar prisms with two layers of reinforcement similar to the ASTM G109 test samples as shown in Figure 12. This mortar test obtained through modification of the ASTM G109 setup was evaluated at an initial study with the participation of four laboratories [52]. Due to different materials and procedures being used at different laboratories, this study did not evaluate the variability of the test between the labs but focused on the ability of the test to differentiate critical chloride content in various systems. It was shown that the effect of inhibitors could be measured using the developed mortar test. The mortar samples have an integrally cast 0.75 in (19 mm) high dam on the top to pond a solution. Different from the earlier study, the mortar cover above the top reinforcement in this study is 0.5 in (13 mm). All reinforcement bars are 9 in (229 mm) long and there are two bars at the bottom layer. The samples are exposed to a 3.3 % NaCl solution on a weekly cycle of 4 days wet and 3 days dry. This wetting and drying cycle increases the speed of chloride penetration into the mortar using a combination of diffusion and capillary suction. The two layers of reinforcement are connected over a 10 Ohm resistor with an accuracy of 1%. When the chloride content reaches the critical chloride threshold at the top reinforcement layer, corrosion initiates causing a drop in the potential of the top bar and an increase in the current flowing between the layers of reinforcement. In the established macro-cell, the top reinforcement is the anode and the bottom layer is the cathode, providing a minimum of 2:1 cathode to anode surface ratio. It should be noted that the macro-cell current measured between the layers of reinforcement does not provide information on the extent of micro-cell corrosion that can take place on the top reinforcement.

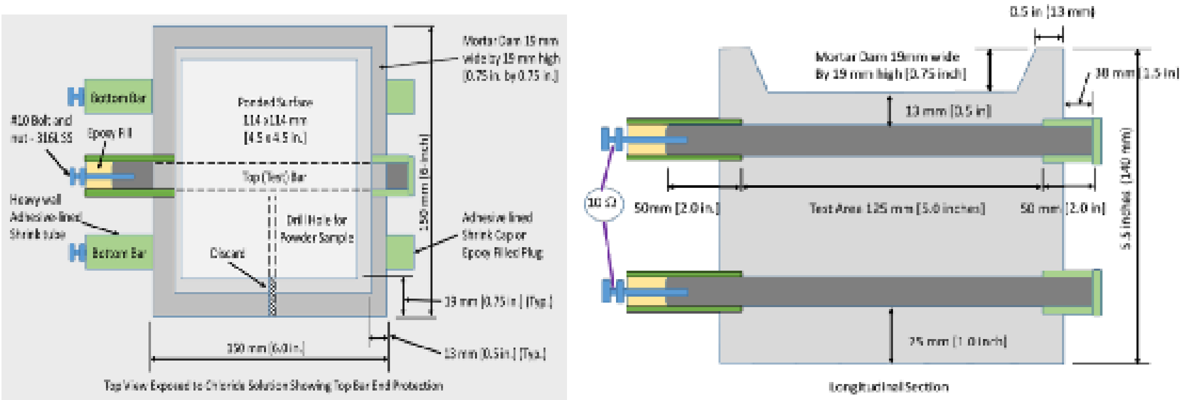


Figure 12: ASTM type sample

A set of 12 plywood molds were fabricated at UMKC for each lab participating in this study. Samples were cast upside down and were cured for 27 days at 72 °F and 100 % relative humidity. Figure 13 shows one of the produced molds before casting and a demolded sample. Prior to casting the samples, inside of the molds were taped using duct tape to avoid water absorption by the plywood. At the completion of 28 days of curing, samples were placed in an environmental chamber (72 ± 2 °F, 50% RH) for 14 days of drying. As stated earlier, all steel samples were cut, drilled, and tapped at UMKC before shipping them out to the participating labs. No surface treatment was applied to the steel samples except degreasing using xylene in an ultrasonoic bath. Two inches at each end of the steel specimen were protected against crevice corrosion by taping with electrical tape and covering with heat shrink tubes. One day before the start of testing by ponding the samples with chloride solution, the sides of the samples were coated with epoxy. Epoxy coating is applied to prevent diffusion of chlorides from the sides of samples during testing and handling.

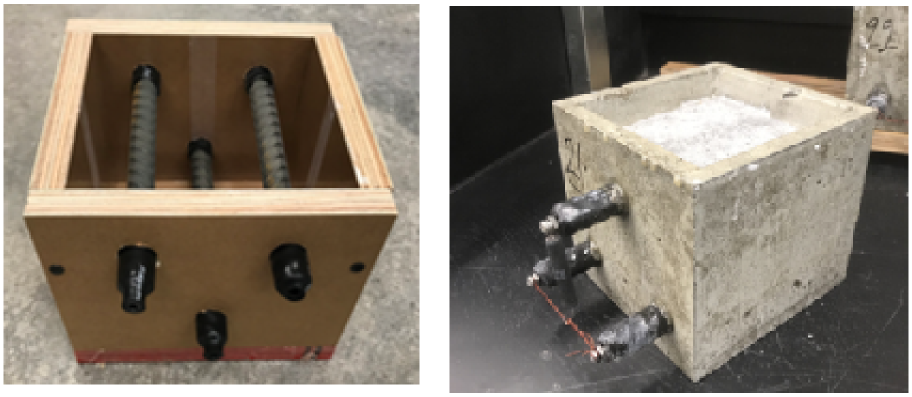


Figure 13: Mold used for ASTM Setup and demolded sample

At the end of every wet cycle, the potential of the top reinforcement was measured against a Ag-AgCl$_2$ reference electrode using a multimeter. The potential drop across the 10 Ohm resistors were measured at the same time to calculate the macro-cell corrosion current. The activation criteria was a drop greater than 100 mV to a value less than -230 mV or a macro-cell current greater than 1 μA. The test was stopped when either of these events were observed for two consecutive weeks. The test method also required measurement of instantaneous corrosion rate by linear polarization when activation criteria was observed for two cycles. However, linear polarization data was not used as part of the activation criteria. Therefore this measurement was not performed by some of the laboratories participating in the study.

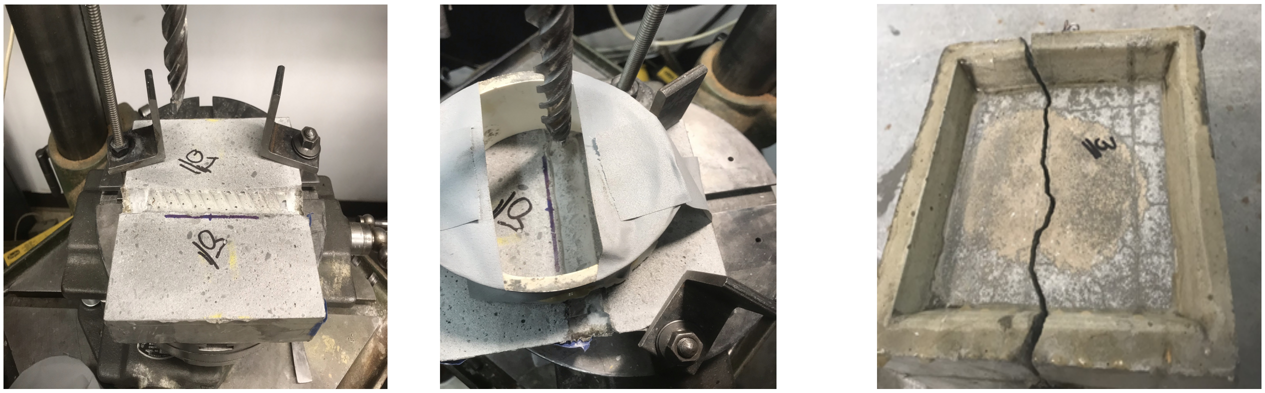
Upon detection of corrosion initiation, the wetting/drying cycles were stopped and the surface of the samples was washed with tap water to remove surface salt. After drying at room temperature, samples were cut at the mid level of top steel bar using a water cooled concrete saw as shown in Figure 14a. Mortar cover above the top steel bar with the trace of the steel bar was removed for chloride sampling (Figure 14b).

(a) Cutting samples at the level of top steel bar

(b) Mortar cover above the top steel bar

Figure 14: Steps for sample collection for ASTM type setup

Mortar powder was collected from the center 3 in (77 mm) section of the cover. Removed mortar cover samples were fixed on a moving platform of a drill press (Figure 15a). A flat-tipped drill bit was used to grind 0.2-0.35 oz (6-11 g) of mortar powder while moving the middle section of the mortar cover back and forth. The depth of drill bit penetration was limited to 0.1 in (2.5 mm). A PVC pipe with 3 in (77 mm) inner diameter was attached to the mortar cover to limit collection of powder samples to the mid section of the mortar cover. The plastic pipe was covered with duct tape during grinding to prevent loss of airborne fine powder (Figure 15b). Cutting and removing of the mortar cover above the top bar should be done gently due to low thickness and strength of the cover. The cover of some samples cracked during this process which prevented collection of mortar samples (Figure 15c).



(a) Sampling (b) Sample collection (c) Broken specimen

Figure 15: Steps for sample collection for ASTM setup

Considering the 28 days of curing at 100 % relative humidity and 14 days of drying of samples at 50 % humidity chamber, the ASTM type test takes at least 42 days to start the testing. Figure 16 shows the steps of the ASTM test method.

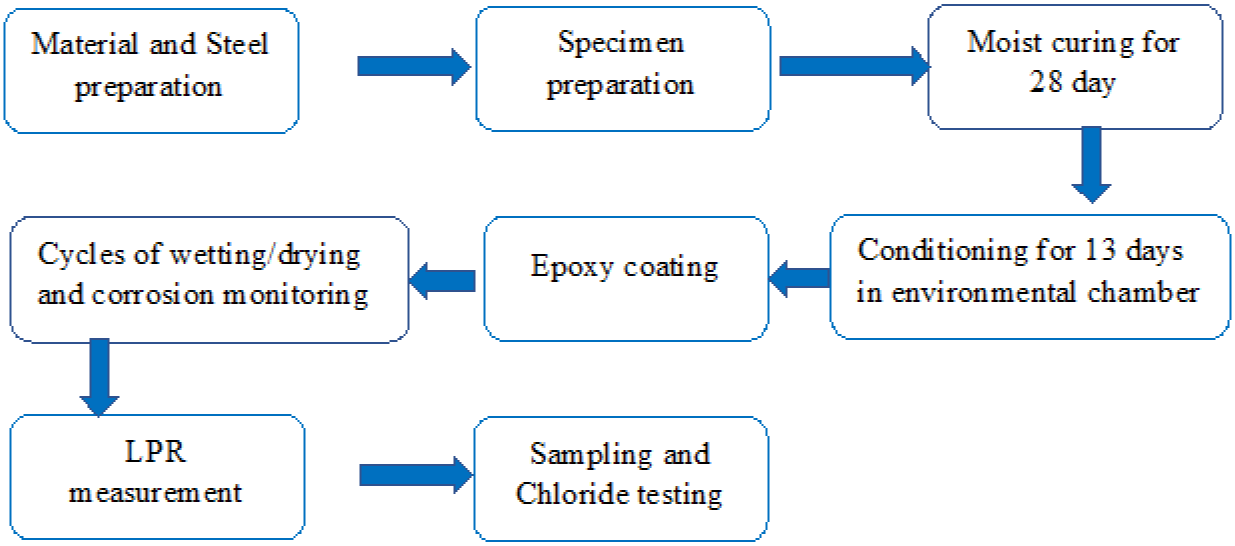


Figure 16: Steps of ASTM type test procedure

3.4. Testing for Fresh and Hardened Properties of Mortar

Fresh and hardened properties of mortar mixtures, such as flow and compressive strength, used in this study were measured to ensure that there was not a significant difference between different batches mixed to produce test samples. Resistivity and pH of mortar mixtures were also measured as requested by TG1 test framework to help evaluate the measured critical chloride threshold values.

3.4.1. Flow test

The flow of hydraulic mortar mixtures shown in Table 4 of section 3.1 was determined following the standard procedure in ASTM C1437 using the standard flow table and mold described in ASTM C230. Although, the procedure in ASTM C1437 requires 25 drops of the flow table in 15 seconds, for the mortar mixture with s/c of 1.375 the flow was recorded at 10 drops as described in ASTM C185. This method was modified because the flow of this mortar was larger than the standard flow table. The flow of mixtures with s/c of 2.0 and 2.75 were recorded at 25 drops.

3.4.2. Compressive strength

The compressive strength of the mortar mixtures were measured using 2 in (52 mm) cubes following the standard procedure described in ASTM C109 [48] at 7 and 28 days. An average of 3 specimens was calculated for each mixture.

3.4.3. Mortar pH measurement

The pH of mortar mixtures with different s/c values was measured using an in situ leaching method described by Sagues et al. [45]. Additional 4 x 8 in (100 x 200 mm) cylindrical samples were cast from each mixture to determine their pH after 28 days of curing. A 0.5 in (12.7 mm) diameter hole was drilled at the top of the samples to a depth of 1.5 in (38 mm) as shown in Figure 17. A plexiglass washer was attached to top of the sample around the drilled hole to hold a rubber stopper. The hole was filled with deionized water to a height of 0.5 in (12.7 mm) and covered with the rubber stopper to prevent evaporation. The pH of water in the hole was measured using a combination glass pH electrode over time until it came to an equilibrium with the sample.

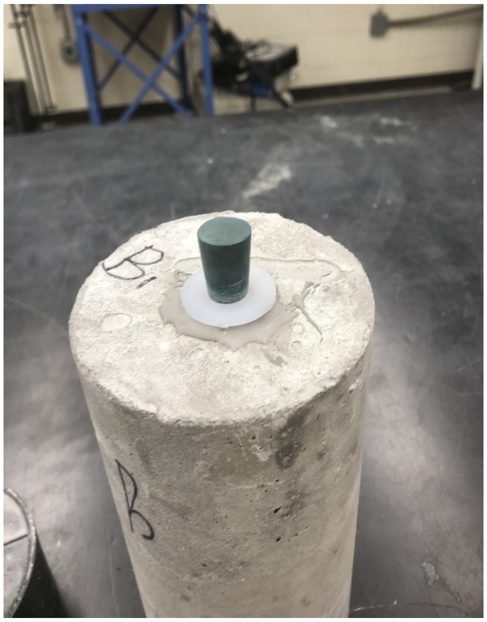


Figure 17: pH specimen

3.4.4. Resistivity

Bulk and surface resistivity of mortar mixtures was measured after 28 days of curing using additionally cast three 4 x 8 in (100 x 200 mm) cylindrical samples. One of the samples was cut to 6 in (150 mm) to measure its bulk resistivity following the ASTM C1760 procedure [53]. Bulk resistivity was measured using a Germann Instruments Proove'it system for rapid chloride permeability testing and a modified cell to hold a 6 in (150 mm) sample. Equation 2 was used to calculate the bulk resistivity based on the measured current after 1 minute.

$$\sigma = K \times \frac{I_1}{V} \times \frac{L}{D^2} \tag{2}$$

Where: σ is bulk electical conductivity, mS/m, I_1 is current at 1 min, mA, V is applied voltage,V, L average length of specimen,mm D is average diameter of specimen,mm, and K is conversion factor which is 1273.2

The remaining two samples were used to measure surface resistivity following the procedure outlined in AASHTO T 358-15 [54] using a Resipod resistivity meter as shown in Figure 18 .

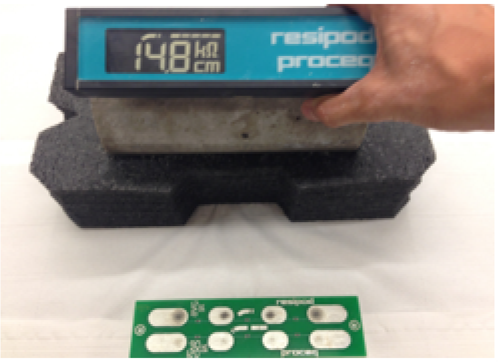


Figure 18: Surface resistivity measurement

3.5. In-solution Test - University of Waterloo

This test method was added to the test program to evaluate the same steel and cement used in this study, however was not a part of the round robin test. Unlike the OC_{crit} and ASTM type setup, this test method used a synthetic pore solution as the alkaline medium. TG1 added this test method to the program to help make a recommendation with a third set of data, in case both test methods evaluated in the round robin test produced very different results with low variability.

The synthetic pore solution was prepared by analysing pore water extracted from cement paste samples with admixed chlorides at five different levels. The same cement that was used in the round robin test was used to prepare the cement paste samples. The admixed chloride contents were 0, 0.5, 1, 2, and 3 % by mass of cement and were prepared by adding NaCl to the mixing water. Four 2 x 4 in (50 x 100 mm) samples were cast for each salt content. The cylinder moulds were capped and all samples were slowly rotated around the cylindrical axis for 24 hours to prevent bleeding. They were then kept in their molds for an additional 27 days. The molds were stripped 28 days after casting and samples were placed in the compression system shown in Figure 19 to extract their pore solution. Pressure was slowly applied via the piston and as the pores were squeezed, the pore solution was collected in a syringe attached to the drain. The syringe tip contained a 0.45 µm filter to prevent cement particles entering the body of the syringe.

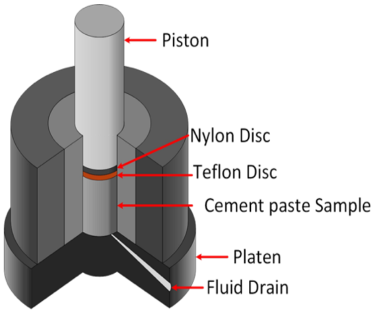


Figure 19: System for pore solution expression

The pH levels of the extracted solutions were recorded immediately and the solutions were kept in the refrigerator until all paste cylinders had been tested. Each solution was analysed using Ion chromatography (IC) and Inductively coupled plasma (ICP - both Optical Emission Spectrometry and Mass Spectrometry). Synthetic pore solutions were prepared with reagent grade chemicals, NaOH, KOH, $Ca(OH)_2$, $CaSO_4.2H_2O$, NaCl, and distilled water. As a result of the analysis, researchers were also able to establish the relation between admixed chloride contents and measured chloride contents in the extracted pore water solutions. 5 in (127 mm) long steel samples, supplied by UMKC, were placed in individual testing solutions in a three-probe electrochemical cell shown in Figure 20. The steel specimens were epoxied at both cut ends and an insulated electrical wire was connected to one end.

The bars were allowed to passivate in their synthetic pore solutions for two weeks. After the bars have reached full passivation (determined by a non-varying corrosion potential), chlorides and sulphates were added as NaCl and $CaSO_4.2H_2O$ and the solutions were stirred for one

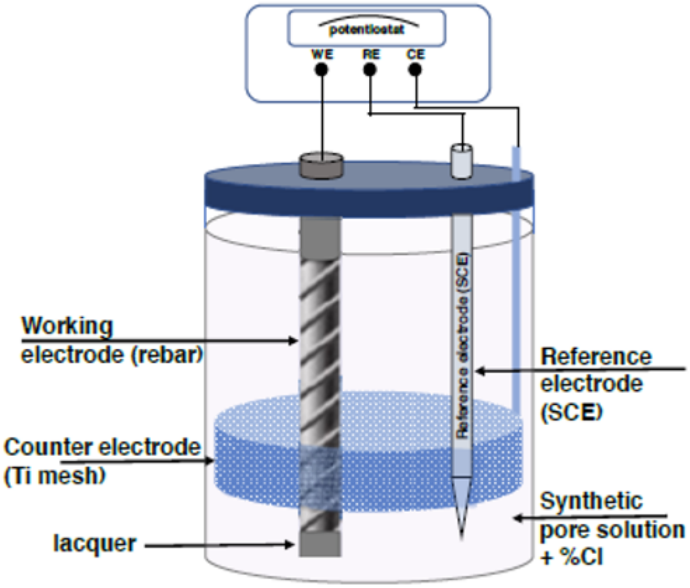


Figure 20: Three-probe electrochemical cell with a mixed metal-oxide titanium mesh counter electrode and a saturated calomel reference electrode (SCE)

minute with a magnetic stirrer to completely dissolve the solutes. Chlorides were added at five different percentages by mass of solution; 1.2, 1.4, 1.6, 1.8, and 2 %. Three replicates were tested for each of the five chloride level solution compositions. After the corrosion potential of the steel bars stabilized, indicating equilibrium had been reached with the added chlorides and sulphates, an anodic potentiodynamic polarization test was performed at a scan rate of 0.01 mV/s. Each bar was scanned from its corrosion potential in the anodic direction to +0.5 V_{SCE} (or to the set current limit of 5 mA) and, if the bar did not display any pitting potential, reversed in the cathodic direction to observe any hysteresis loop. The equilibrium corrosion potentials and measured pitting potentials decrease linearly with increasing chloride contents in the solutions. Extrapolation of obtained values were used to determine the solution chloride content amount at which the equilibrium potential of the steel would be equal to its pitting potential. In theory this indicates the critical solution chloride content at which chlorides will spontaneously initiate corrosion by damaging the passive layer. The obtained critical solution chloride content was then converted back to the critical chloride content as % by mass of cement using the initially established relation.

4. Results and discussion

4.1. Fresh and hardened properties of mortar

4.1.1. Mortar flow and strength

Table 5 presents the average compressive strength and flow of the three mortar mixtures with s/c 1.375, 2.0, and 2.75 described in Chapter 3 Table 4. Compressive strengths shown are the average of three cubes tested after 7 and 28 days of curing. All mixtures had a constant w/c of 0.42 and the compressive strength decreased with an increase of s/c due to decreasing cement contents in the mixtures. Similarly, the flow of mixtures were decreasing with increasing s/c due to decrease in paste content and increase of sands in the mixtures. The mixture with s/c of 1.375 was used for both OC$_{crit}$ and ASTM type samples and was a highly flowable mixture. The flow of this mixture was greater than the standard flow table at 25 drops. Therefore, its flow is reported only at 10 drops similar to the procedure described in ASTM C185. This highly flowable mixture was necessary to cast the OC$_{crit}$ samples with 0.19 in (0.5 mm) thin cover. However, this high flowability caused settlement issues with larger ASTM type samples and mixtures with higher s/c values were used to cast additional ASTM type samples.

Table 5: Compressive strength and Flow of mortar mixtures

	Compressive strength (psi/ Mpa)		**Flow (%)**	
s/c	7 Days	28 Days	10 drops	25 drops
1.375	7558/ 52	10287/ 72	106	-
2.0	6086/ 42	7693/ 53	63	103
2.75	5516/ 38	6961/ 46	-	30

4.1.2. pH

Figure 21 shows the change of pH measured through in-situ leaching method over time for samples of the three mortar mixtures. For all mortar mixtures pH started around 13.37 and came to an equilibrium around 13.7 within a month. These results are consistent with results reported by Sagues et al., which described this test method [45]. The pH data obtained through the leaching method are also similar to the pH values measured through pore water extraction (13.07 to 13.4) at University of Waterloo. Analysis of extracted pore water solutions are reported later together with the in-solution test results in this chapter.

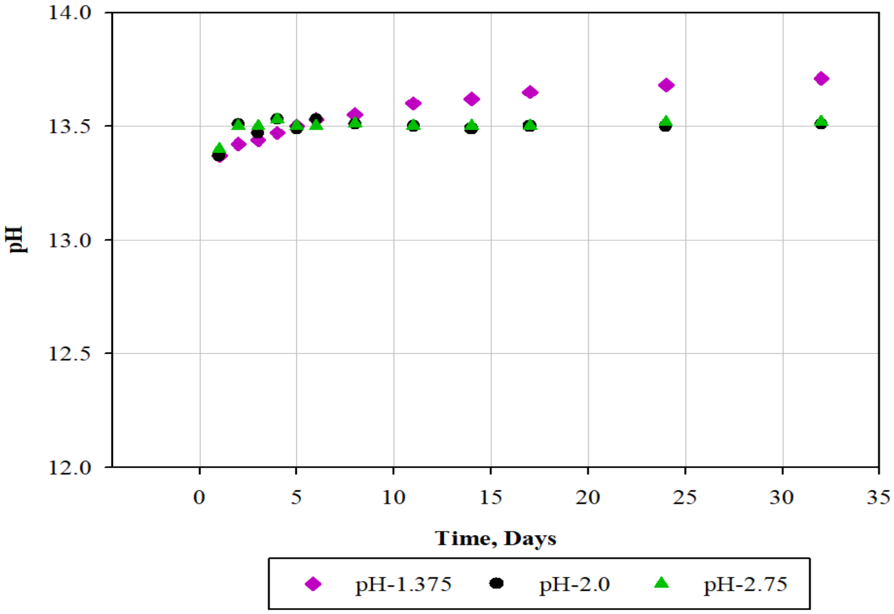


Figure 21: pH Evolution with time in Mortars

4.1.3. Resistivity

Electrical resistivity of concrete and mortar samples can be correlated to their microstructure and pore volume fraction. Studies have shown that the chloride diffusion coefficient and water permeability of concrete and mortar can be predicted based on measured conductivity of their pore solution [55]. Table 6 shows the measured surface and bulk resistivity values for the mortar mixtures with different s/c ratios at 28 days in saturated conditions. Previous studies have shown that bulk resistivity measurements are typically lower than surface resistivity measurements but correlated based on sample geometry [56, 57]. However, in this study the bulk resistivity values didn't show an increase with increasing s/c ratio although an increase was observed with surface resistivity measurements. The measured surface resistivity of 5.8 kΩ.cm for the mortar mixture with s/c of 1.375 is in agreement with earlier published mortar surface resistivities in the literature and indicates a high permeability [58]. This high permeability may explain observed time to activation values that were less than a month for the OC$_{crit}$ samples. The OC$_{crit}$ samples were only cast using the mortar with s/c of 1.375. The increase in observed mean time to activation values for ASTM type samples that were cast with increasing s/c mortars can be explained by the increase of surface resisitvity of these mixtures.

Table 6: Surface and Bulk resistivity of mortar mixtures

Mortar	Surface Resitivity(kΩ.cm)	Bulk conductivity(mS/m)	Bulk resistivity(kΩ.cm)
1.375	5.8	21.75	4.59
2.00	7.2	21.87	4.57
2.75	8.7	21.65	4.62

4.2. OC$_{crit}$ Test Results

The OC$_{crit}$ test was performed at 3 different laboratories; University of Missouri - Kansas City (UMKC), Oregon State University (OSU), and CTL Group. The test was performed twice at UMKC to evaluate the variance of two tests performed at the same lab with the same materials. University of Kansas also participated in this test, however was not able to provide results at the time of writing of this report. This section provides the results obtained in each lab, and a comparison of the results at the end of this section.

4.2.1. UMKC Test 1

A set of 12 samples were cast for the first test. Proper consolidation of the OC$_{crit}$ samples is critical to prevent any large voids on the surface of the thin mortar cover and proper casting of these samples requires practice. When the first set of samples were demolded, three of the samples had large holes on the surface of thin mortar cover caused by entrapped air and improper compaction. After discussion with the OSU team, it was decided to test these samples together with the rest of the samples after repairing the holes using a two-part concrete epoxy as shown in Figure 22.

Figure 22: Repaired specimen with concrete epoxy

Figure 23 shows the daily steel potentials measured against a Cu-CuSO$_4$ electrode over time until their activation. This figure shows only the samples without any surface defects. Testing was stopped for each sample when they exhibited a potential less than -350 mV for two consecutive readings. Figure 24 shows the steel potentials of the repaired specimens. All samples, including the repaired ones, had a passive potential in the range of -100 to -200 mV at the beginning of the testing. Testing of the first sample that showed activation was stopped at 9 days and one of the repaired samples had the longest test duration with 21 days.

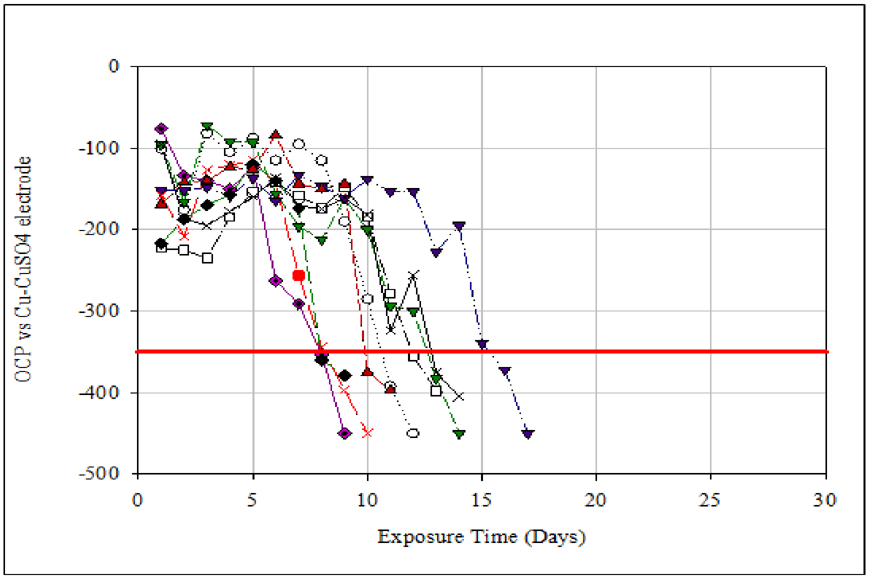


Figure 23: E_{corr} of steel reinforcement, UMKC1

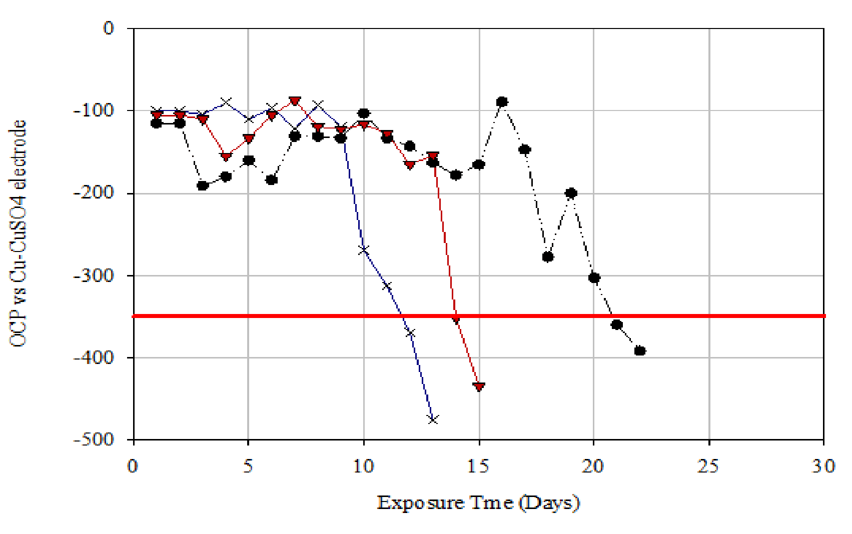


Figure 24: E_{corr} of steel reinforcement , UMKC1-Repaired Sample

At the completion of testing, the thin mortar cover of all samples was broken and ground to determine their total acid soluble chloride content. The epoxied section of the thin mortar cover of repaired samples was discarded before grinding for chloride analysis. Table 7 shows the acid soluble total chloride content as % by weight of cement of all the samples and their time to activation in days. The R letter next to the sample numbers 2, 6, and 12 indicate that these were the repaired samples. When the thin mortar cover section of samples was broken for analysis, researchers also observed that the steel of two samples, sample 1 and 7, were off center as shown in Figure 25. The chloride content of all the samples varied between 0.460 and 1.058 % by mass of cement with an average of 0.753% and standard deviation of 0.172%. The two samples with off center steel showed the lowest two chloride contents in the data. Bending of the bottom copper wire during casting was believed to be the cause of steel being off center in these two samples. Therefore stainless steel threaded rods were used instead of copper wires for the second set of samples as described in chapter 3.

Table 7: Chloride content by mass of cement and time to activation for UMKC1

Lab	**%Cl (cement)**	**Time to act (days)**
1	0.511	9
2	1.024	21
3	0.700	12
4	0.709	14
5	0.644	16
6	0.702	12
7	0.460	11
8	0.837	13
9	0.749	9
10	0.889	14
11	0.749	11
12	1.058	14
Average	0.753	13
St. Dev	0.172	3
Max	1.058	21

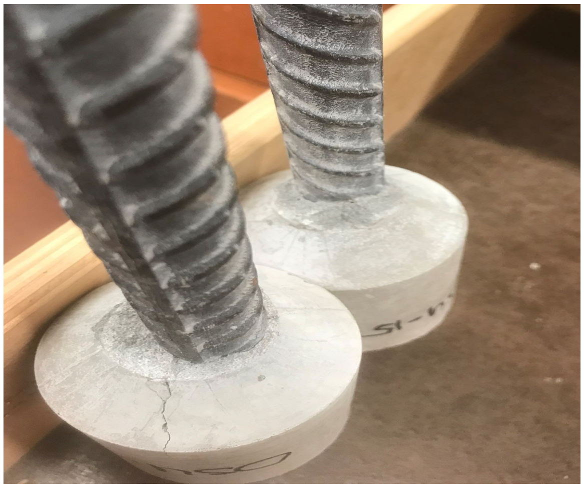


Figure 25: Bad samples with off center steel specimen

4.2.2. UMKC Test 2

10 samples were cast for the second test and no surface defects were observed on any of the samples. Figure 26 shows the potential of the samples over time. Similar to the first set of samples, all samples were in the passive range at the beginning of testing and they activated between 8 and 16 days of exposure.

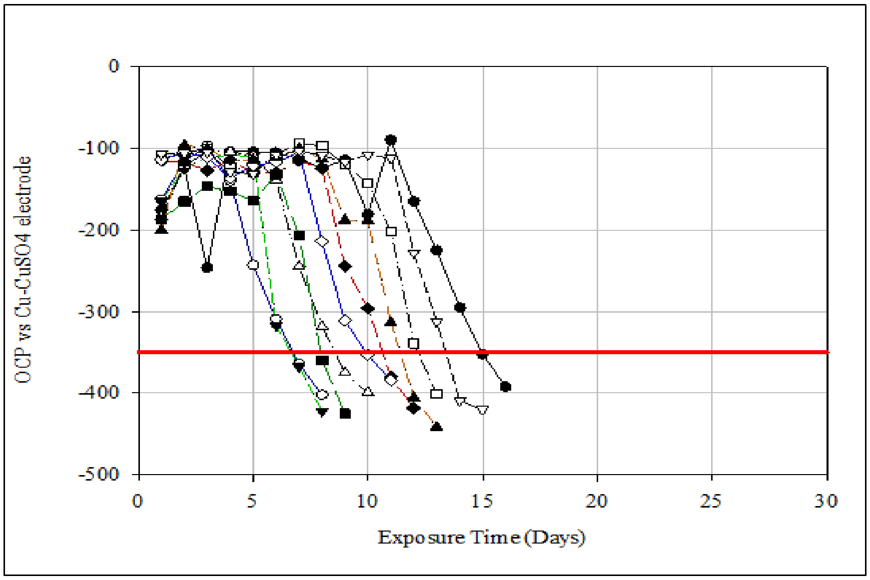


Figure 26: E$_{corr}$ of steel reinforcement, UMKC2

Table 8 shows the chloride content of samples as % by weight of cement and their time to activation in days. The average, standard deviation, minimum, and maximum values are also shown. The average chloride content was 0.806 % by weight of cement, which is slightly higher than the average of the first set of samples.

Table 8: Chloride content by mass of cement and time to activation for UMKC2

Lab	%Cl (cement)	Time to act (days)
1	1.179	16
2	0.788	8
3	0.609	8
4	0.690	10
5	0.850	9
6	0.735	13
7	0.696	12
8	0.676	11
9	0.894	13
10	0.945	15
Average	0.806	12
St. Dev	2.655	3
Max	1.18	16
Min	0.609	8

Figure 27 shows the distribution of chloride contents as % by weight of cement for samples in test 1 and 2. Test 1 samples are shown as three distributions where the first distribution shows all the samples, the second distribution shows the samples without the bad samples with off center steel, and the third distribution shows the samples without the bad and repaired samples. Figure 28 shows the distribution of time to activation of samples in test 1 and 2. Initiation of corrosion was visually confirmed for all OC_{crit} samples after breaking their mortar cover. The pictures of the test 2 samples before and after testing are provided in Appendix C of this report.

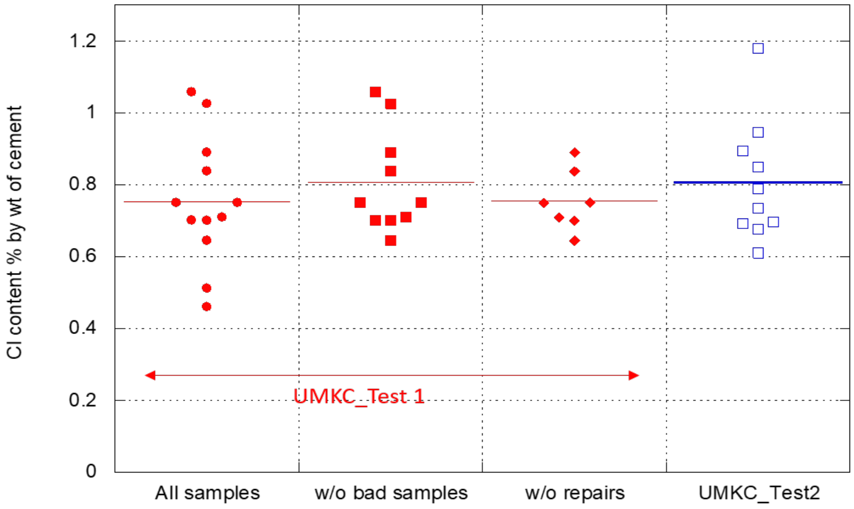


Figure 27: Distribution of chloride contents for test 1 and 2 samples

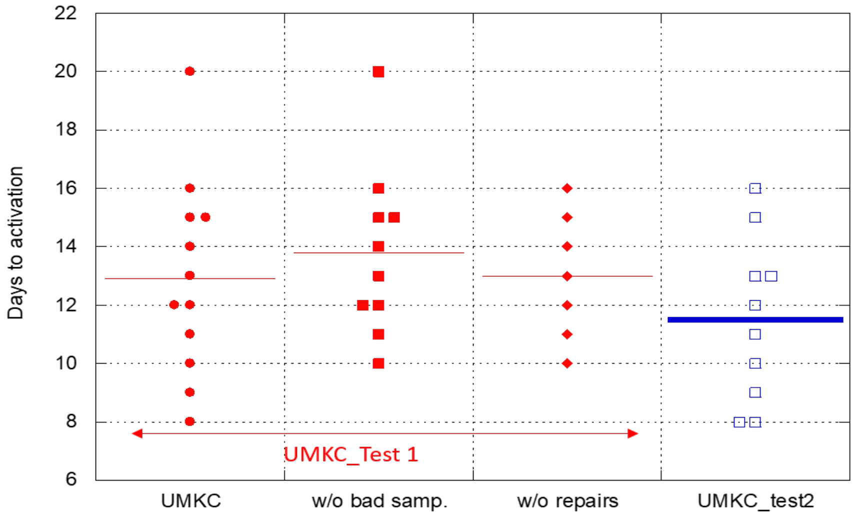


Figure 28: Distribution of time to activation values for test 1 and 2 samples

4.2.3. Oregon State University (OSU) Results

A set of 10 samples were tested at OSU laboratories using the materials shipped from UMKC. Figure 29 shows the potential values measured against a Cu-CuSO$_4$ electrode over time. Although the measured potential values showed a similar trend, the initial potential values observed at OSU and UMKC were different. The initial potential of samples at OSU was lower, between -200 and -300 mV range, compared to the initial potential values at UMKC tests, which were between -100 and -200 mV. The first sample activated at 8 days similar to the test results at UMKC but the time to activation of the last sample was 25 days, which is longer compared to 16 days observed at UMKC.

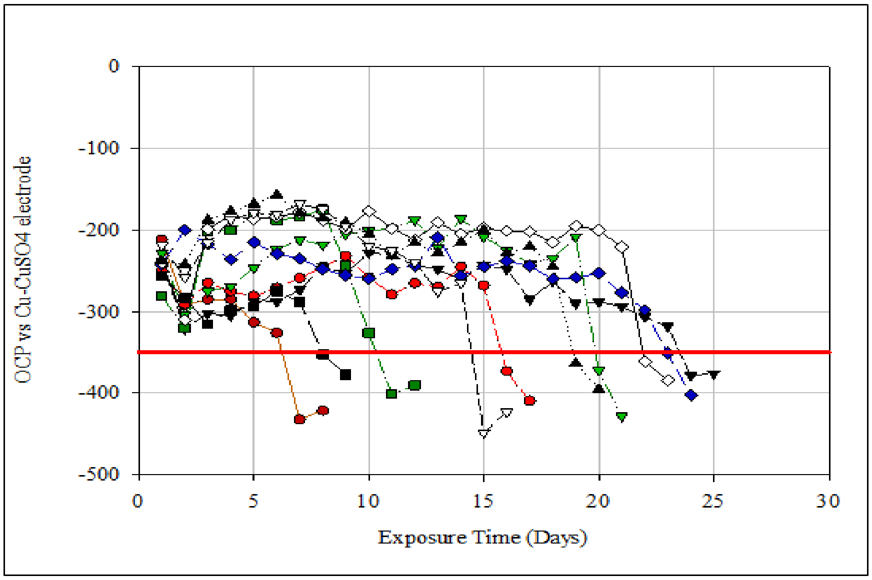


Figure 29: E_{corr} of steel reinforcement, OSU

Table 9 shows the chloride content of samples as % by weight of cement and their time to activation in days. The average, standard deviation, minimum, and maximum values are also shown. The average chloride content was 0.607 % by weight of cement, which is slightly lower than the average of the UMKC results.

4.2.4. CTL Group Results

CTL was the third laboratory to perform the OC_{crit} test within the round robin test program. Figure 30 shows the potential values measured against a $Cu\text{-}CuSO_4$ electrode over time. The initial potential values were between -200 and -300 mV similar to the OSU samples. Four of the

Table 9: Chloride content by mass of cement and time to activation for UMKC2

Lab	**%Cl (cement)**	**Time to act (days)**
1	0.255	8
2	0.620	17
3	0.835	25
4	0.805	21
5	0.363	9
6	0.419	12
7	0.751	24
8	0.872	23
9	0.681	20
10	0.465	16
Average	0.607	18
St. Dev	0.218	6
Max	0.87	25
Min	0.255	8

samples activated earlier between 12 and 15 weeks similar to previous test results and the rest of the samples activated between 24 and 29 days. Table 10 shows the chloride content of samples as % by weight of cement and their time to activation in days. The average, standard deviation, minimum, and maximum values are also shown. The average time to activation was 21.7 days which is significantly higher than other laboratories. Also, the highest standard deviation in time to activation was observed in CTL results.

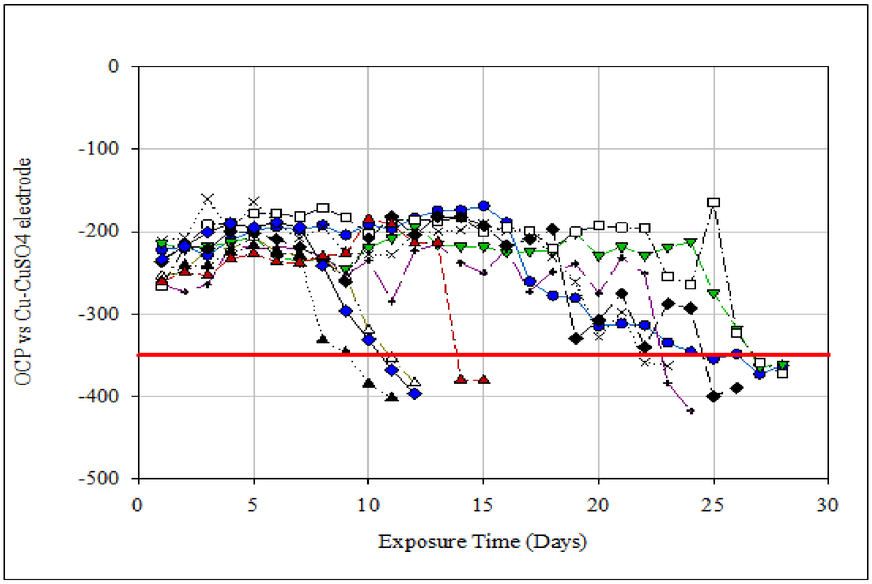


Figure 30: E_{corr} of steel reinforcement, CTL

Table 10: Chloride content by mass of cement and time to activation for UMKC2

Lab	%Cl (cement)	Time to act (days)
1	0.663	29
2	0.708	24
3	1.027	29
4	0.556	13
5	0.762	25
6	0.829	29
7	1.030	27
8	0.948	13
9	0.703	12
10	0.919	16
Average	0.815	22
St. Dev	0.162	7
Max	1.03	29
Min	0.556	12

4.2.5. Comparison of OC_{crit} results

Table 11 shows the mean, standard deviation, minimum, and maximum of chloride content values expressed as % by weight of cement collected from the four sets of OC_{crit} data.

Table 11: Summary of OC_{crit} test chloride content results

Chloride content % by weight of cement				
Statistics	**UMKC1**	**UMKC2**	**OSU**	**CTL**
Mean	0.753	0.806	0.607	0.815
Standard Dev.	0.172	0.168	0.218	0.162
Max	1.058	1.179	0.872	1.03
Min	0.46	0.609	0.255	0.556

As an initial step of statistical analysis, the distribution of each data set was evaluated to see if they are normally distributed. Figure 31 shows that all four data sets were normally distributed using a 95% confidence interval.

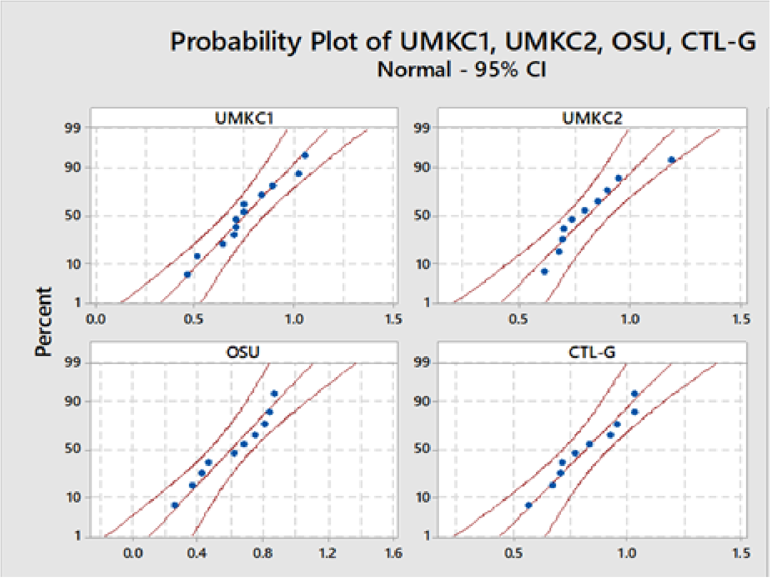


Figure 31: Distribution of data (%Cl by mass of cement) in different laboratories

After confirming that the data sets were normally distributed, they were compared using Tukey pairwise comparison test. Tables 12 and 13 show the results of analysis of variance and Tukey pairwise comparison. As the grouping section shows, all test results were placed in the same group, i.e. the differences between them were not statistically significant. The data in four different sets could all have come from one data set of a test performed in one lab with the same materials. Figure 32 shows the same data graphically by showing the 95% confidence intervals for

Table 12: Analysis of variance of % chloride content

Analysis of Variance					
Source	DF	Adj SS	Adj MS	F-Value	P-Value
Factor	3	0.2783	0.9277	2.77	0.055
Error	38	1.2737	0.03352		
Total	41	1.552			

Table 13: Tukey pairwise comparison for % chloride content

Grouping information using Tukey method and 95% condfidence			
Factor	N	Mean	Grouping
CTL group	10	0.815	A
UMKC2	10	0.806	A
UMKC1	12	0.753	A
OSU	10	0.607	A

differences of means. If the intervals contain zero, it shows that the means were not statistically significantly different from each other and all of the intervals contain zero.

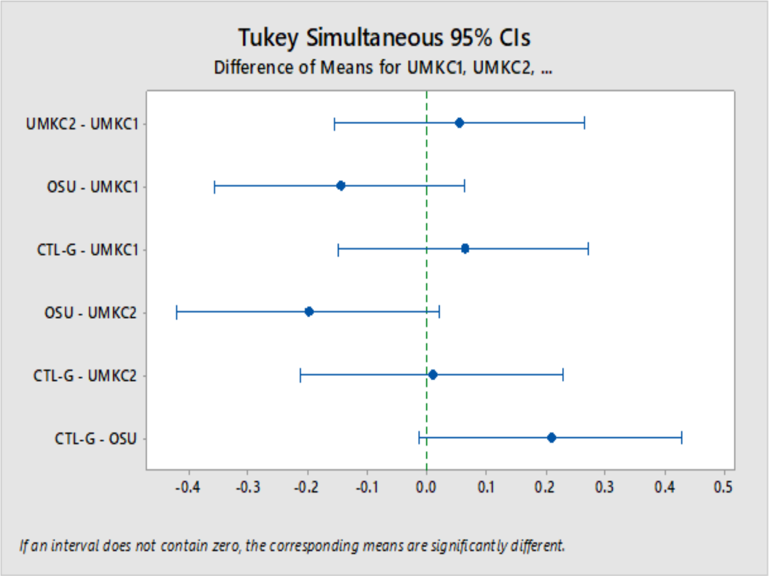


Figure 32: Tukey post-hoc test for %Cl content by mass of cement

Because the analysis shows that the means are not statistically significantly different, all of 42 data points can be combined together. Figure 33 shows that combination of all data points was also normally distributed with a mean chloride content of 0.745% by weight of cement and a standard deviation of 0.195.

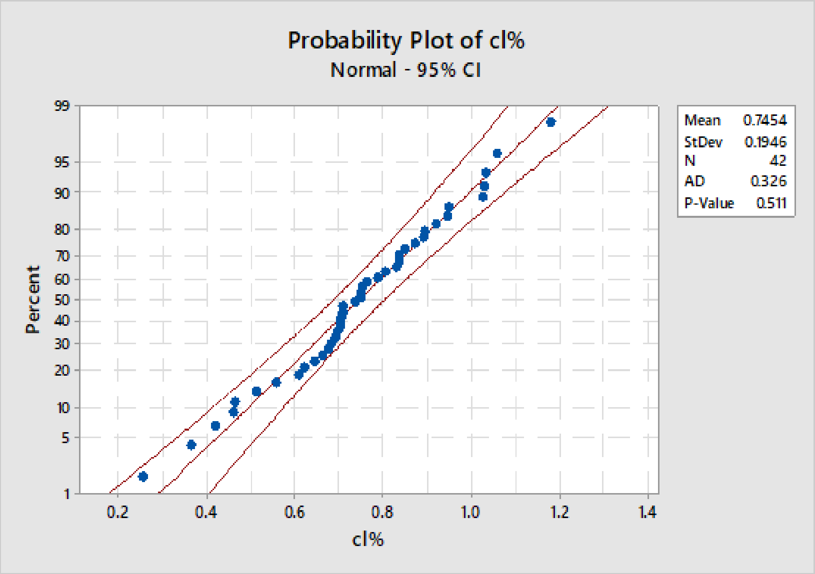


Figure 33: Distribution of all data (%Cl by mass of cement)

As described in Chapter 3 section 3.2 the OC_{crit} test method also provides an equation to convert the average chloride content determined for the whole thin mortar cover, Cl_{test}, to the critical chloride content, Cl_{crit}, at the steel mortar interface. Figure 34 shows the distribution of Cl_{crit} obtained from the four sets of data calculated using equation 1. Table 14 shows the calculated mean Cl_{crit} values and their average time to activations in days. Based on this data the OC_{crit} test method shows that for the steel and mortar combination used in this study the critical chloride content, Cl_{crit} was 0.416 % by weight of cement with a standard deviation of 0.111.

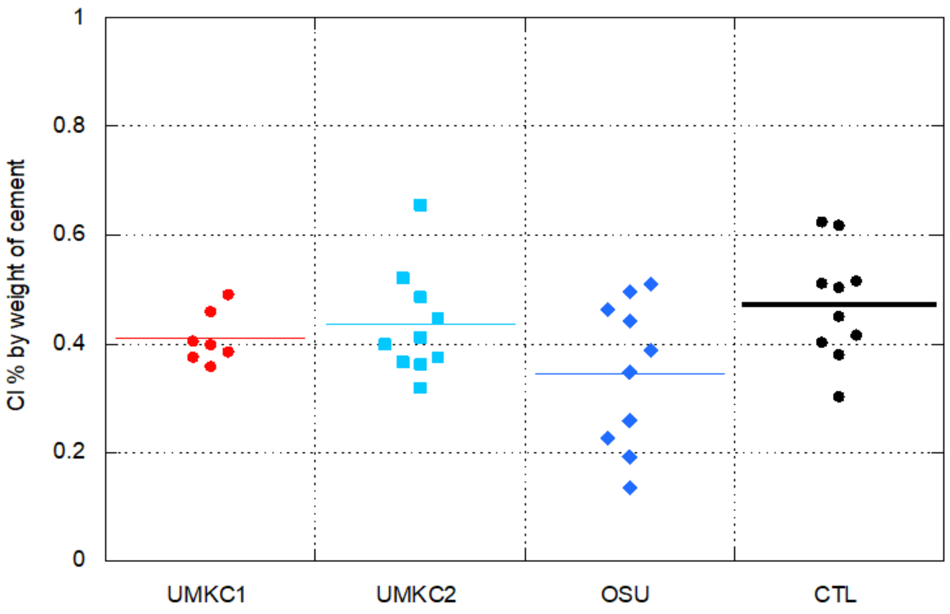


Figure 34: Distribution of Cl_{crit} data for each laboratory test set

Table 14: Mean Cl_{crit} data calculated for each data set

Lab	Mean Cl test % by weight of cement	Average time to activation(days)	Mean Clcrit % by wight of cement
UMKC1	0.753	13	0.409
UMKC2	0.806	12	0.434
OSU	0.607	18	0.341
CTL	0.815	22	0.471

A similar statistical analysis was performed for the time to activation data of four laboratories. Figure 35 shows that the time to activation data of each laboratory also fit to a normal distribution. However, analysis of variance and Tukey pairwise comparisons as shown in Tables 15 and 16 indicate that the time to activation data of CTL group was statistically significantly different from the UMKC test data. Analysis shows that OSU, UMKC 1, and UMKC 2 time to activation data were not statistically significantly different from each other. Similarly, CTL data and OSU data comparison shows that they were not significantly different. However, due to observed large time to activation data in CTL group and relatively short time to activation data observed at UMKC tests, these groups are significantly different from each other at 95% confidence level.

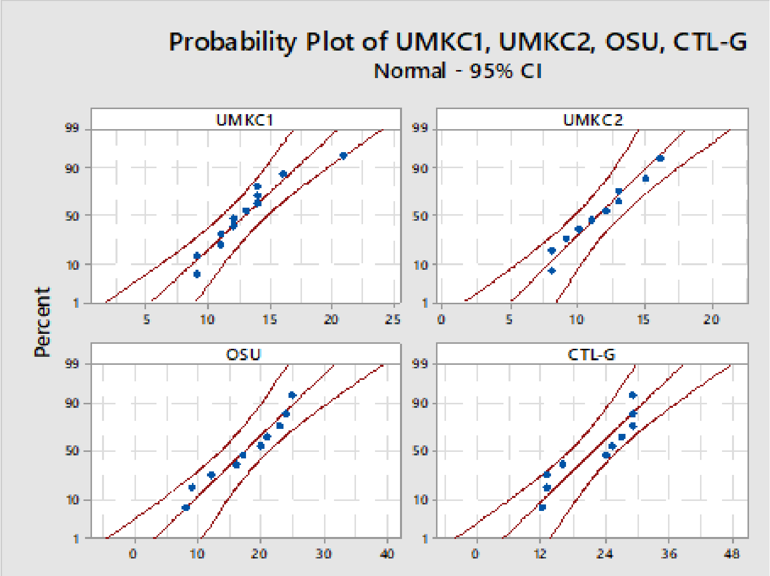


Figure 35: Normal distribution of time to activation in different labs

Table 15: Analysis of Variance for time to activation comparison

Analysis of Variance					
Source	DF	Adj SS	Adj MS	F-Value	P-Value
Factor	3	656	218.66	8.2	0.000
Error	38	1031.1	26.66		
Total	41	1669.1			

Table 16: Tukey pairwise comparison for time to activation

Grouping information using Tukey method and 95% condfidence			
Factor	N	Mean	Grouping
CTL group	10	21.70	A
UMKC2	10	17.50	A B
UMKC1	12	13.00	B
OSU	10	11.50	B

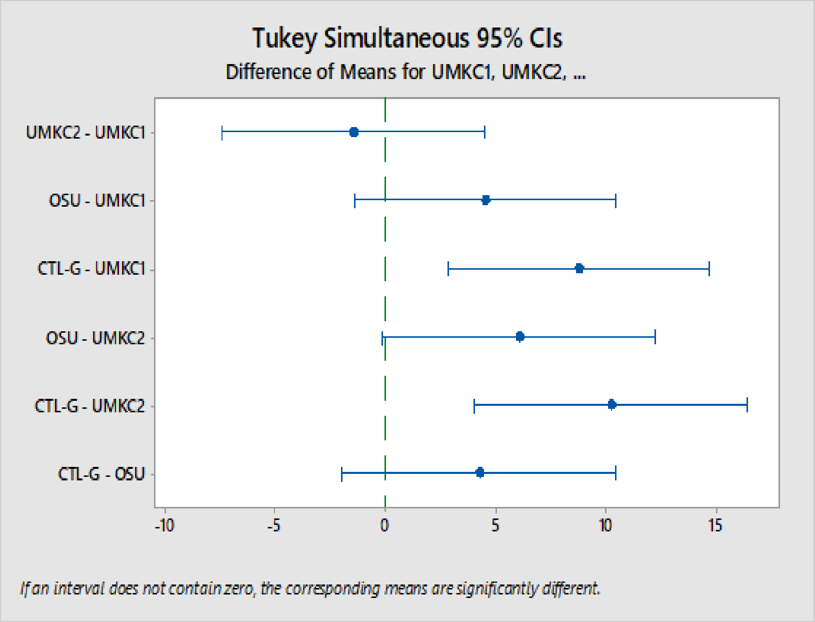


Figure 36: Tukey post-hoc test for time to activation

It should be further investigated why the time to activation for some samples was longer at OSU and CTL laboratories compared to UMKC and why the initial steel potential values were lower. However, analysis of chloride contents show that the OC_{crit} test method can generate repeatable and statistically not significantly different results for the same set of materials when repeated in the same laboratory and between different laboratories.

4.3. ASTM type Test Results

The ASTM type test was performed at 4 laboratories; University of Missouri - Kansas City (UMKC), Tourney Consulting, University of Kansas (KU), and CTL group. As explained earlier, to be able to compare the results of OC$_{crit}$ test and the ASTM type test, the mortar mixture with w/c of 0.42 and s/c of 1.375 was used for both tests. Due to the size of the ASTM type test samples, mixing and casting a set of 10 samples from the same batch, using standard benchtop laboratory mixers is not possible. At first trial at the UMKC laboratory, a large masonry mortar mixer was used to cast a large batch of mortar enough for 12 ASTM type samples. However, due to the size of the mixer, casting had to be done outside in a non-controlled environment. The samples were cast upside down and after about an hour settlement cracks were observed as shown in Figure 37a. After refinishing the surface, samples were covered with plastic sheets and wet burlap for 24 hours and then were moved to a 100% relative humidity curing room. The test calls for drying of the samples for 14 days at 50% relative humidity environmental chamber before covering the sides with epoxy and exposing them to the chloride solution. When the first set of 12 samples were exposed to the chloride solution, shrinkage cracks were observed on the surface as shown in Figures 37b and 37c. One day after exposing the samples to the chloride solution, the potential of all the samples were lower than -350 mV vs. Cu-CuSO$_4$ electrode which showed initiation of corrosion. Therefore this first set of 12 samples were discarded at UMKC without collecting chloride content samples.

(a) Mortar settlement (b) Plastic shrinkage (c) Plastic shrinkage

Figure 37: Challenges in ASTM type

A set of 10 samples was also cast at Tourney Consulting laboratories when the first set of UMKC samples were being cured. After the curing and drying period, these samples were exposed to the chloride solution and initial potential measurements showed that the samples were at passive state. Based on this information, UMKC cast a second set of 10 samples but this time samples were cast in laboratory using benchtop mortar mixers in multiple batches. Flow of each batch was measured as a quality control measure between batches. When the second set of samples were exposed to chloride solution, the potential readings of all the samples were above -350 mV vs. Cu-$CuSO_4$ with the exception of one sample. Following these casts KU was recommended to cast a set of 10 samples with the same mortar in laboratory. However, they also observed shrinkage cracks and the samples indicated corrosion initiation at the first measurement similar to the UMKC's initial experience. After discarding two sets of samples (UMKC and KU), UMKC cast two more sets of 5 samples using mortars with s/c of 2.0 and 2.75 keeping the w/c at 0.42. These samples were at the passive state when initially exposed to the chloride solution and didn't exhibit any settlement cracks or excessive bleeding during casting due to their lower flow values. Based on this observation, CTL was recommended to cast their samples using a mortar with s/c of 2.0 and w/c of 0.42. Results of these tests and their comparison are provided below.

4.3.1. UMKC and Tourney Consulting Tests using Mortar with s/c 1.375

Figure 38 shows the potential measurements for the top steel bars of ASTM type samples cast at UMKC over time. The potential of all samples were higher than -350 mV vs.Cu-CuSO$_4$ 24 hours after initial exposure to chloride solution except one sample. Although, the potential of this sample increased to a value above -350 mV at the second weekly cycle, it was below this limit value at the third and fourth cycles. Eight out of the ten samples activated within first five weekly cycles and the remaining two samples satisfied the activation criteria at weeks 9 and 12. Macro-cell current readings were close to zero throughout the whole testing period for all samples. Samples that satisfied the activation criteria were cut to collect mortar powder samples for chloride content analysis as described in Chapter 3.

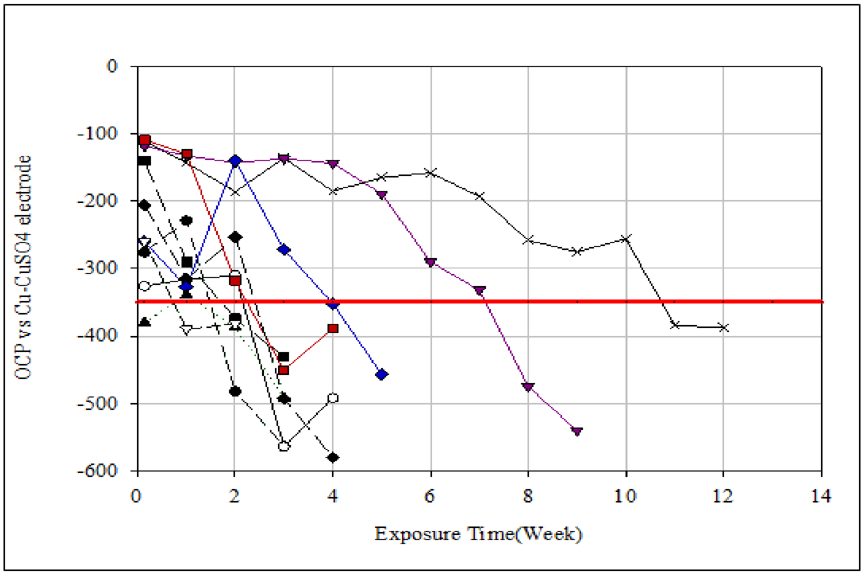


Figure 38: E$_{corr}$ vs time of specimens with s/c 1.375, UMKC

Table 17 shows the measured total acid soluble % chloride content values by weight of cement and the time to activation of samples. The mortar covers of two of the samples, 3 and

10, were broken during the saw cutting and their chloride content could not be determined. The average % chloride content by weight of cement was 0.442 with a standard deviation of 0.131. Testing was completed with 12 weeks with an average time to activation of 5 weeks. Figure 39 shows that the chloride cotent data fit to a normal distribution using a 95 % confidence interval. It should be noted that this critical chloride content distribution is statistically not significantly different from the combined results obtained from the OC$_{crit}$ test results.

Table 17: Chloride content and time to activation of mixture with s/c 1.375, UMKC

Sample no.	%Cl (Cement)	Time to act (Weeks)
1	0.198	3
2	0.447	4
3	Broken	12
4	0.640	9
5	0.344	3
6	0.411	4
7	0.523	4
8	0.495	5
9	0.475	4
10	Broken	3
Average	0.442	5
Stan dev.	0.131	3
Max	0.640	12
Min	0.198	3

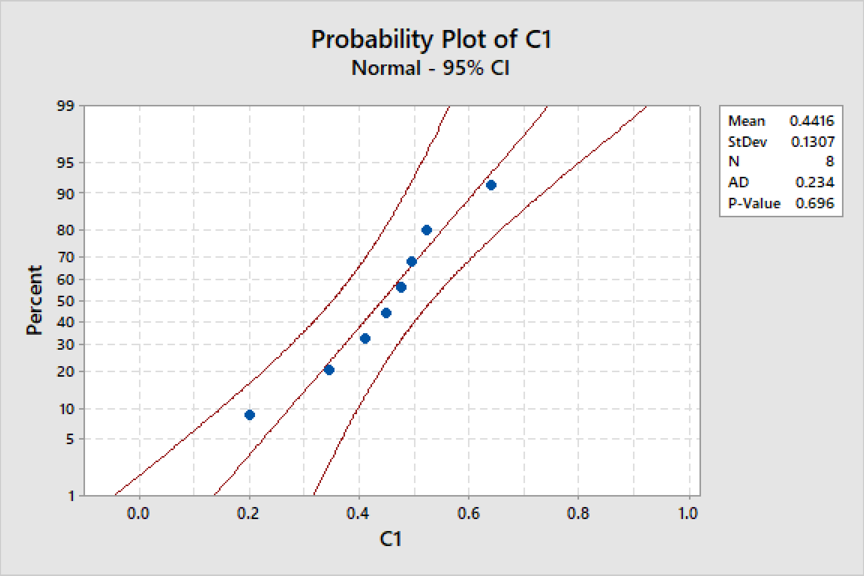


Figure 39: Chloride content distribution, UMKC

Figure 40 shows the half cell potential values of the samples tested at Tourney Consulting laboratory over time. The potential values were measured using a Cu-$CuSO_4$ electrode. The activation criteria proposed for the round robin test was a drop greater than 100 mV to a value less than -230 mV vs. Ag-$AgCl_2$ (corresponds to -350 mV vs. Cu-$CuSO_4$) or a macro-cell current greater than 1 μA. The test was supposed to be stopped when either of these events were observed for two consecutive weeks. However, at this test group, testing of samples was stopped when a large drop in half cell potential was observed even if the potential was not below -350 mV. Only three of the samples (3, 8, and 9) satisfied the criteria proposed for the round robin test. Sample 5 never activated based on either criteria. Figure 41 shows the corresponding macro-cell current values observed over time. For most of the samples a drop in half-cell potential was accompanied with an increase in the macro-cell current. With the exception of sample 7, all samples activated within 12 weekly cycles.

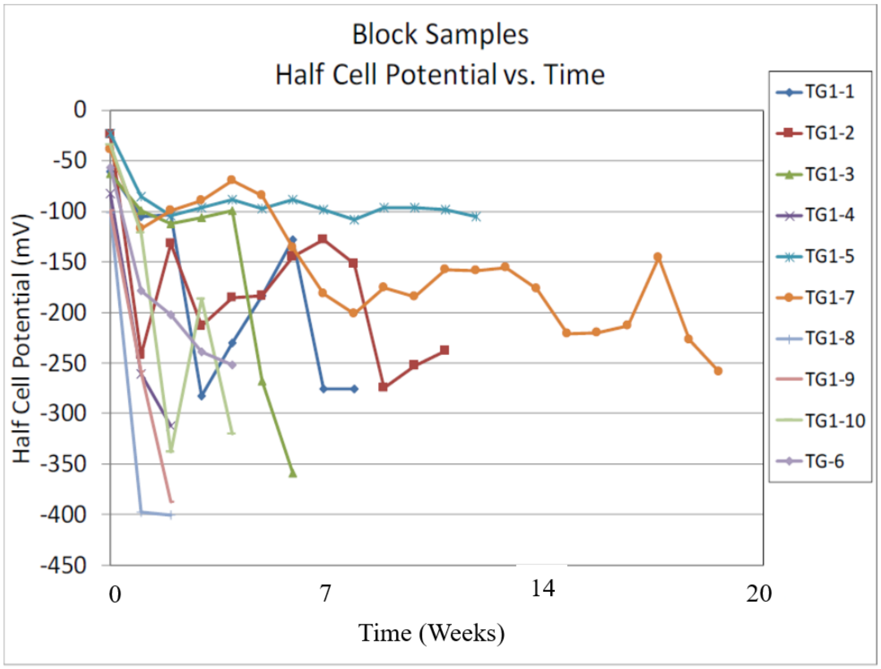


Figure 40: E_{corr} vs time of mortar with s/c 1.375, Tourney Consulting

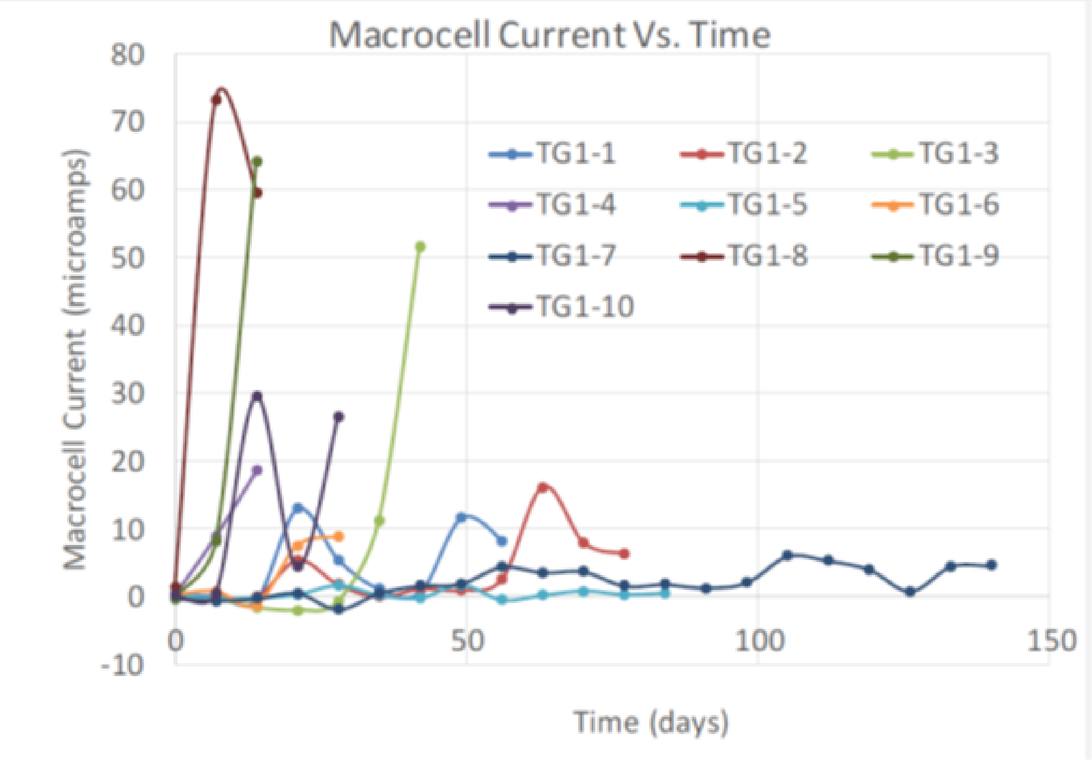


Figure 41: Macro-cell corrosion current vs time, Tourney Consulting

Table 18 shows the measured total acid soluble % chloride content values by weight of cement and the time to activation of samples. The average chloride content without sample 5, which never activated, was 0.148 % by weight of cement with a standard deviation of 0.060. The average chloride content of only the three samples that met the round robin test criteria (highlighted with blue) was 0.154 % by weight of cement with a standard deviation of 0.053. These values are not significantly different, which indicates that the used activation criteria instead of the round robin test criteria may not have a significant effect on the test results. Figure 42 shows that the chloride contents were normally distributed.

Table 18: Chloride content and time to activation of mixture with s/c 1.375, Tourney Cansulting

Sample no.	%Cl (Cement)	Time to act (Weeks)
1	0.179	8
2	0.142	11
3	0.198	6
4	0.050	2
5	0.088	12
6	0.231	4
7	0.181	20
8	0.170	2
9	0.095	2
10	0.083	4
Average (without sample 5)	0.148	7
Stan dev.	0.060	6
Max	0.231	20
Min	0.050	2

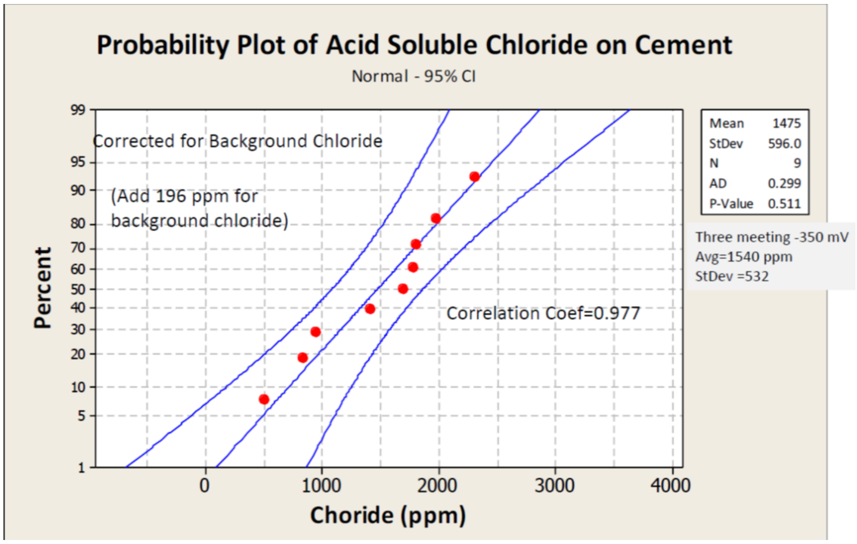


Figure 42: Probability Plot for Chloride Contents of Samples, Tourney

Evaluating the chloride content distributions for the ASTM type samples that were cast using the mortar with s/c of 1.375 at UMKC and Tourney Consulting, it is clear that these results are statistically significantly different. The chloride distribution obtained at Tourney Consulting laboratory for the same materials was significantly lower compared to the results obtained at UMKC laboratory. Observed settlement cracks and excessive bleeding during casting may have contributed significantly to the variability of the results. Use of a mortar mixture with such high flow value for ASTM type sample sizes was found to be problematic. At the conclusion of testing Tourney Consulting suggested that the activation criteria for future testing should be changed to either a large drop in potential accompanied with at leasat 5 μA macro-cell corrosion or a potential below -350 mV vs. Cu-CuSO$_4$. The test procedure proposed to the task group also required linear polarization testing of samples twice after they indicated activation but these values were not used as part of the activation criteria. Appendix C shows the pictures of ASTM type samples or steel specimens at both laboratories at the conclusion of testing and Appendix D shows the linear polarization data for samples tested at UMKC.

4.3.2. UMKC and CTL Group Tests using Mortar with s/c 2.0

The mortar mixture with s/c of 2.0 and w/c of 0.42 was tested with the same steel using the ASTM type setup at UMKC and CTL Group laboratories. Due to lower flow value of this mixture, settlement cracks and excessive bleeding were not observed when casting these samples. However, at both laboratories shrinkage cracks were observed on the surface of some samples after exposure to chloride solution. These cracks were not as large as the ones observed on samples cast with s/c of 1.375 and were not visible at the end of moist curing period. The recommended drying period of 14 days at 50% relative humidity may be too harsh for these larger ASTM type mortar samples.

Figure 43 shows the half cell potential values of the five samples tested at UMKC over time. All samples had a potential value higher than -350 mV after 24 hours of exposure to chloride solution. Four samples showed activation within 12 weekly cycles and the remaining sample never satisfied the activation criteria. Although this sample showed a sudden decrease in potential after 17 cycles of approximately 200 mV, its potential remained above 350 mV and it slowly became passive over time. This activation and repassivation is a common problem with ASTM G109 samples as well, which is the basis of the mortar test method evaluated in this study [23].

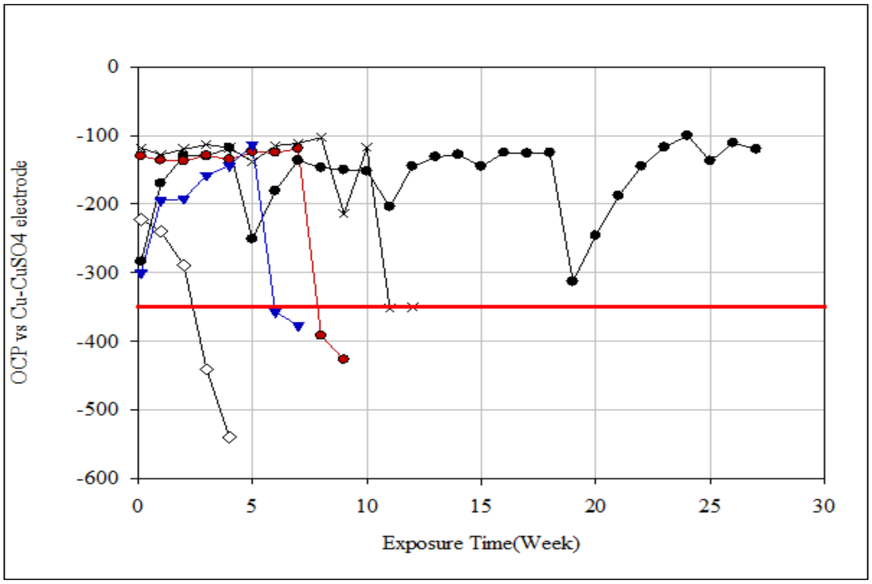
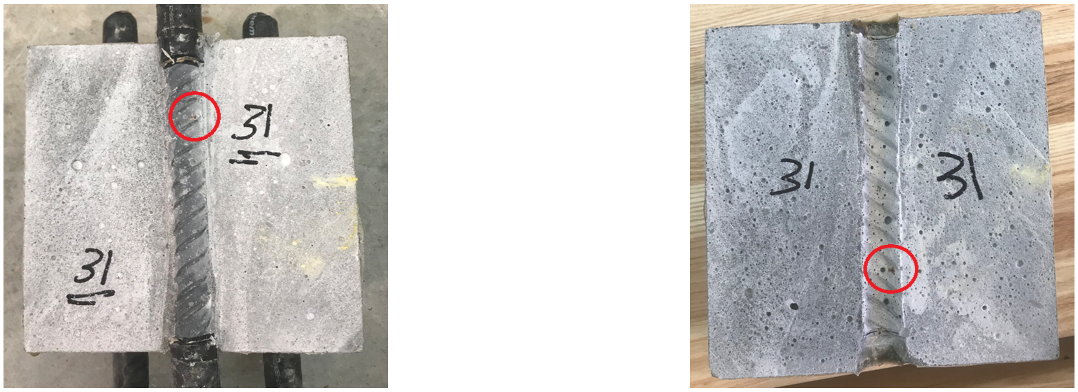


Figure 43: E_{corr} vs time of mortar with s/c of 2.0, UMKC

After 27 weekly cycles of testing, the sample that did not satisfy the activation criteria was cut to visually inspect corrosion. Figures 44a and 44b show the bottom part of the sample with the steel bar and the mortar cover with the trace of the steel bar. The red circles mark the area where corrosion products were visually observed on the steel and in the mortar at the same location. This may indicate that a large drop in the measured potential may be a better activation criteria for ASTM type samples. Determining the amount of large drop to count as activation criteria may be difficult. Analysis of CTL group data, provided later, showed the drop in the observed potential may take place incrementally over time.



(a) (b)

Figure 44: Steel concrete interface of inactivated specimen

Table 19 shows the measured total acid soluble % chloride content values by weight of cement and the time to activation of samples. The average chloride content of the four activated samples was 0.679 % by weight of cement with a standard deviation of 0.186. The average time to activation was 8 weekly cycles.

Table 19: Chloride content and time to activation of mixture with s/c 2.0, UMKC

Sample no.	%Cl (Cement)	Time to act (Weeks)
1	0.886	27
2	0.811	9
3	0.556	7
4	0.486	4
5	0.864	12
Average (without sample 1)	0.679	8
Stan dev.	0.186	3
Max	0.864	12
Min	0.486	4

Figure 45 shows the half cell potential values of ten samples tested at CTL group using the mortar with s/c of 2.0 over time. One of the samples had a lower initial potential value compared to the rest of the samples and showed activation quickly after 2 cycles of testing. The rest of the samples activated between 4 and 10 weeks with the exception of two samples that never satisfied the activation criteria during testing and were stopped after 12 weekly cycles. As stated earlier, this data shows that the potential drop for some of the samples was not sudden and happened gradually over time. This indicates that if a drop in potential will be used as an activation criteria, defining the magnitude of the drop may not be a simple task.

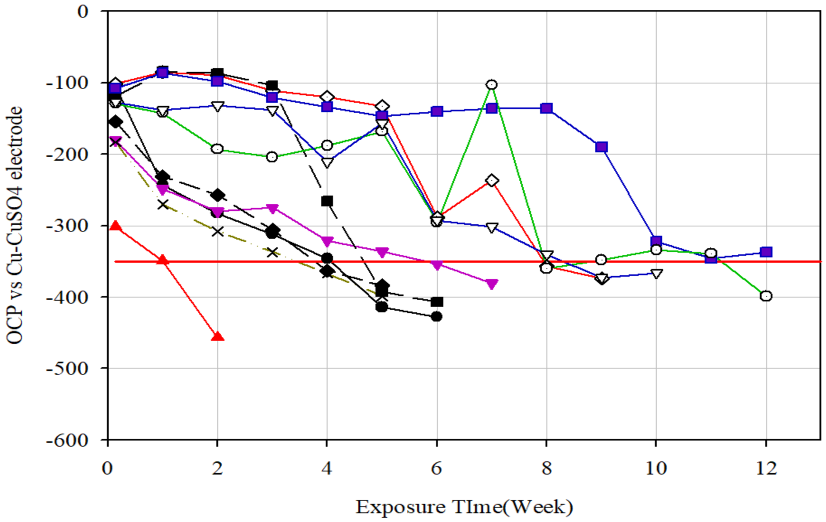
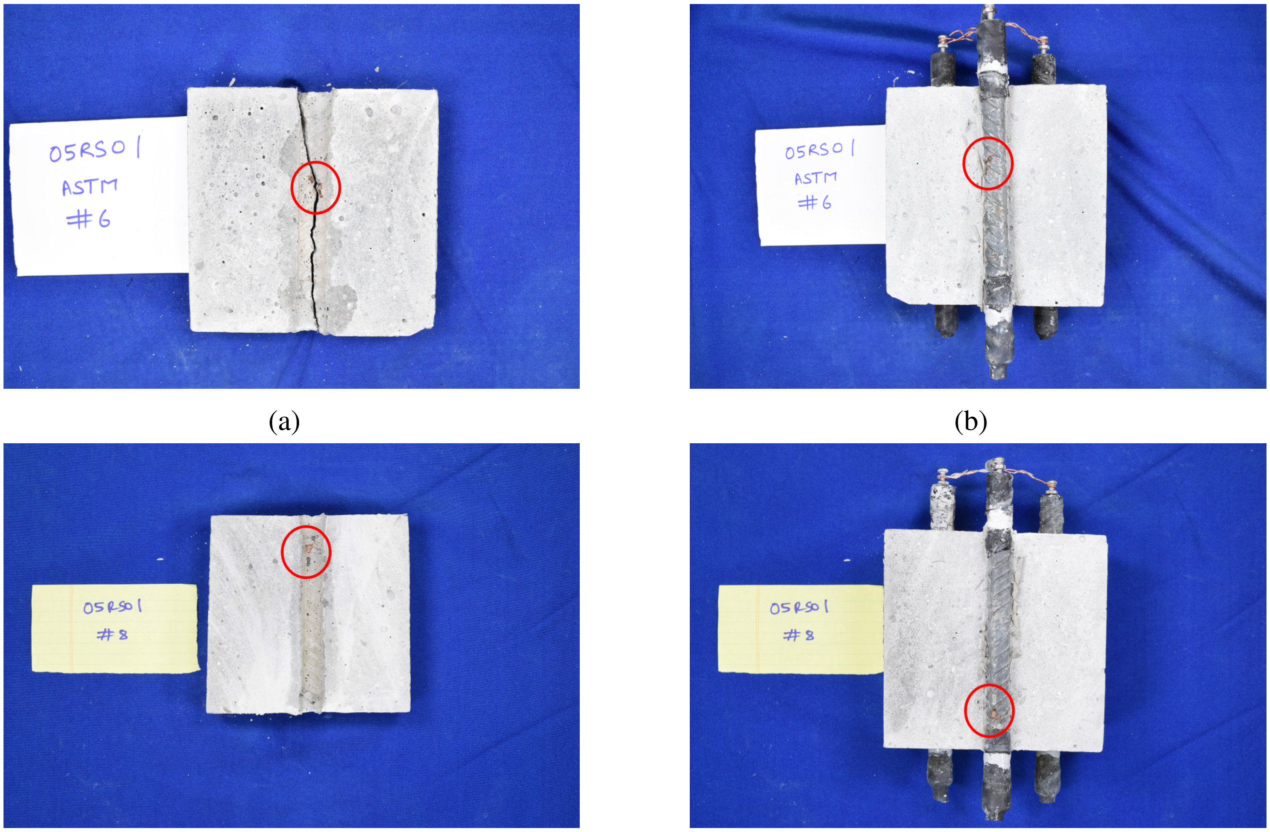


Figure 45: E_{corr} vs time of mortar with s/c of 2.0, CTL group

Figure 46 shows the 2 samples that did not satisfy the activation criteria during the 12 weeks of testing. Visual inspection of the samples showed clearly that corrosion initiated in both samples.



(a) (b)

(c) (d)

Figure 46: CTL samples 6 and 8 that did not activate

Table 20 shows the measured total acid soluble % chloride content values by weight of cement and the time to activation of samples. The average chloride content of the four activated samples was 0.334 % by weight of cement with a standard deviation of 0.257. The average time to activation was 6 weekly cycles.

Table 20: Chloride content and time to activation of mixture with s/c 2.0, CTL

Sample no.	%Cl (Cement)	Time to act (Weeks)
1	0.379	6
2	0.886	10
3	0.363	7
4	0.251	5
5	0.208	6
6	0.326	12
7	0.089	5
8	0.462	12
9	0.082	2
10	0.417	9
Average (without samples 6 and 8)	0.334	6
Stan dev.	0.257	3
Max	0.886	10
Min	0.082	2

Evaluating the chloride content distributions for the ASTM type samples that were cast using the mortar with s/c of 2.0 at UMKC and CTL Group, it is clear that these results are statistically significantly different. The mean value of chloride distribution obtained at CTL Group laboratory (0.334%) for the same materials was approximately half of the mean value obtained at UMKC laboratory (0.679%).

4.3.3. UMKC Test using Mortar with s/c 2.75

A set of five samples were cast at UMKC using a mortar with s/c of 2.75 to evaluate after observing settlement and excessive bleeding issues with earlier samples that were cast using a mortar with s/c of 1.375. Samples using this mortar were only cast UMKC laboratory and it is not possible to compare it to any other results in the program. Figure 47 shows the half-cell potentials of the samples measured against a Cu-CuSO$_4$ electrode over time. Potential of all samples was above -300 mV but one of the samples showed activation very quickly in 2 cycles. One of the samples satisfied the activation criteria after 50 weeks of testing. Another sample never satisfied the activation criteria after 53 weeks. The potential of this sample dropped below -350 mV twice but not consecutively. Similar to earlier results obtained with different mortars, slow drop of potentials and repassivation seems to be an issue in determining corrosion initiation.

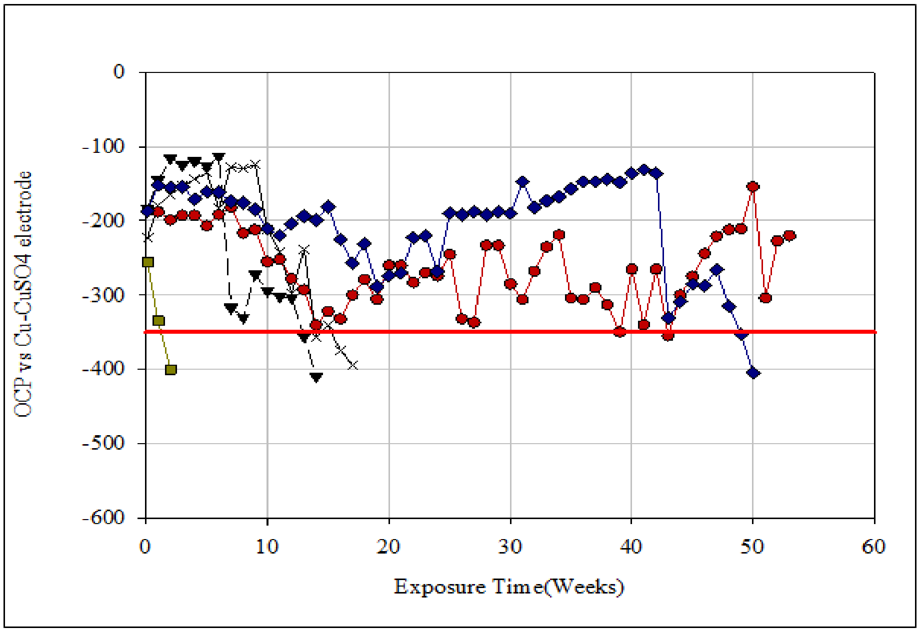


Figure 47: E$_{corr}$ vs time of mortar with s/c of 2.75, UMKC

Figure 48 shows the cut ASTM type sample that did not satisfy the activation criteria for 53 weeks. Similar to earlier findings, visual inspection clearly shows activation of corrosion and accumulation of corrosion products.

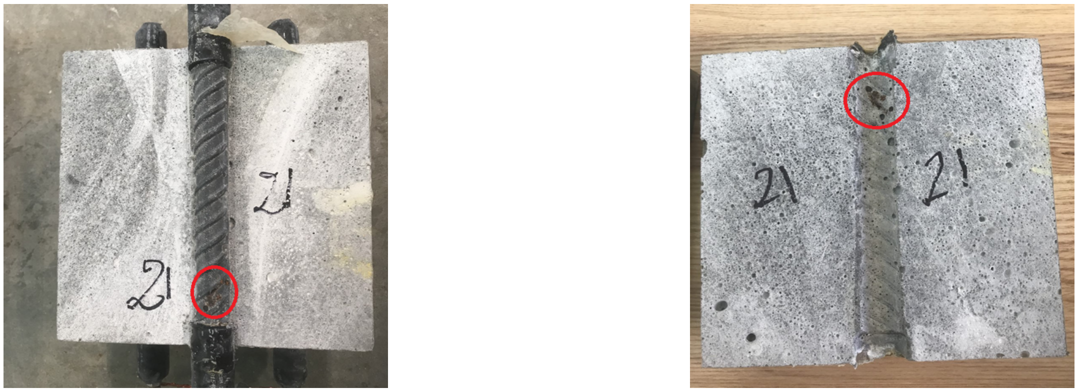

(a) (b)

Figure 48: Inactivate sample after 53 weeks of exposure

Table 21 shows the measured total acid soluble % chloride content values by weight of cement and the time to activation of samples. The average chloride content of the four activated samples was 1.048 % by weight of cement with a standard deviation of 0.127. Although the average time to activation exhibited a large variation from 2 to 50 weeks, variability of measured chloride contents is not very large with a coefficient of variation of only 12%.

Table 21: Chloride content and time to activation of mixture with s/c 2.75, UMKC

Sample no.	%Cl (Cement)	Time to act (Weeks)
1	1.219	53
2	0.906	17
3	Broken	14
4	1.149	50
5	1.091	2
Average	1.048	21
Stan dev.	0.127	21
Max	1.149	51
Min	0.906	2

4.3.4. Comparison of ASTM type results

Table 22 shows the average, standard deviation, maximum, and minimum values for chloride content and time to activation values measured at different labs for mortars with different s/c values. Although mean time to activation values did not show a large variation, the mean chloride contents measured at different labs for the same steel-mortar system were statistically significantly different from each other. This indicates that two laboratories testing the exact same mortar-steel system can produce completely different results that may lead to contradictory design or maintenance decisions. Although the mean chloride content measured for the mortar with s/c of 2.75 was significantly higher compared to the other data sets, it is not possible to state that the critical chloride content was increasing with increasing s/c. Comparison of UMKC chloride content data for mortar with s/c of 1.375 and CTL group data for mortar with s/c of 2.0 shows that they were not statistically significantly different. It should be noted that the chloride content distribution for each data set was normally distributed in itself and didn't show a large coefficient of variation.

Table 22: Comparison of ASTM type test results

Mortar	**s/c 1.375**				**s/c 2.0**				**s/c 2.75**	
Lab	**Tourney C.**		**UMKC**		**CTL Group**		**UMKC**		**UMKC**	
	%[1]	Weeks [2]	%	Weeks	%	Weeks	%	Weeks	%	Weeks
Average	0.148	7	0.442	5	0.334	6	0.679	8	1.048	21
Stand. Dev	0.06	6	0.131	3	0.257	3	0.186	3	0.127	21
Max	0.231	20	0.64	12	0.886	10	0.864	12	1.149	51
Min	0.05	2	0.198	3	0.082	2	0.486	4	0.906	2

[1] Cl% by weight of cement

[2] Time to activation

4.4. In-solution Test - University of Waterloo

The type I/II cement used in the round robin test was used to prepare cement pastes at w/c of 0.4 with 0, 0.5, 1, 2, and 3% Cl by mass of cement admixed chlorides. The pore solution of the prepared cement pastes were extracted and analysed to prepare the synthetic pore solution used for this test method. Analysis was performed using ion chromatography (IC) and inductively coupled plasma (ICP). Results of pore water analysis are provided in Appendix E.

The compositions of the prepared synthetic pore solutions based on the analysis are shown in Table 23 together with the pH values of the expressed pore solutions. Figure 49 was also prepared based on the analysis which shows the correlation of admixed chlorides in cement paste and chlorides measured in solution.

Table 23: Pore Solution compositions based on the average values of data from the expressedpore solutions

pH	%Cl in cement	Mass of compound in solution									
		KOH		CaSi4.2H2O		NaOh		Ca(OH)2		NaCl	
		(g/L)		(g/L)		(g/L)		(g/L)		(g/L)	
13.80	0%NaCl	13.8	16	1	1	5.3	5.8	0.2	0.1	0.2	0
13.07	0%NaCl	15.8		0.9		5.7		0.1		0	
13.20	0%NaCl	17.4		1.1		6.4		0.1		0	
13.75	0.5%NaCl	16.5	16	2.4	2.4	15.8	15	0.1	0.1	1.1	1.1
13.80	0.5%NaCl	16		2.8		15.4		0.1		1.2	
13.85	0.5%NaCl	15.1		1.9		14.3		0.1		1.1	
14.00	1%NaCl	15.8	14	3.3	3.3	23	21	0.1	0.1	4.7	4.7
13.99	1%NaCl	12.9		3.4		18.9		0.1		4.7	
14.10	1%NaCl	13.5		3.2		19.6		0.1		4.7	
14.08	2%NaCl	14.4	15	6.1	6.5	37.3	39	0.1	0.1	17.8	18
14.13	2%NaCl	15.8		6.6		41.3		0.1		17.8	
14.10	2%NaCl	14.6		6.9		38.2		0.1		18.1	
14.10	3%NaCl	16.3	17	11.3	11	60	61	0.1	0.1	32	33
14.10	3%NaCl	16.8		11		61.6		0.1		33.4	
14.11	3%NaCl	16.3		11.2		60.5		0.1		32.8	

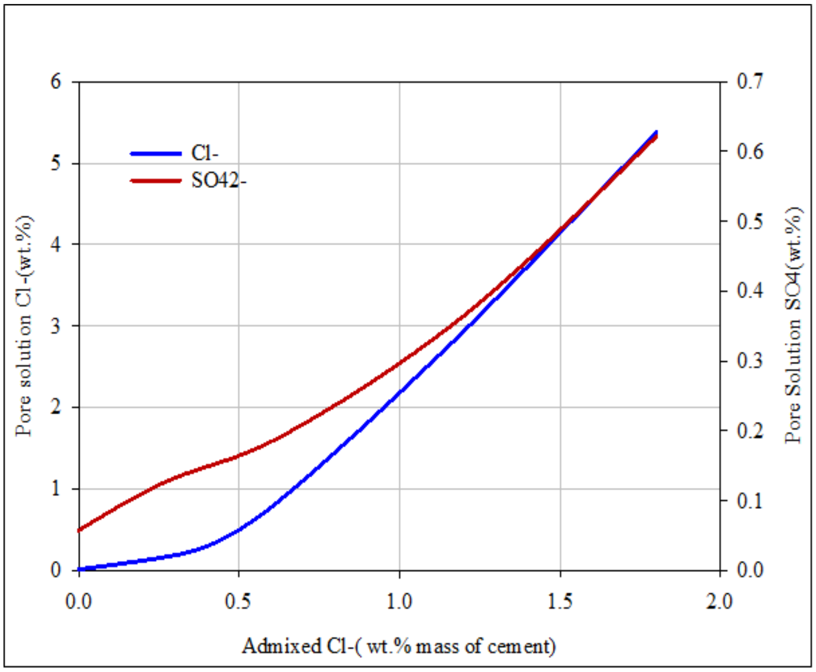


Figure 49: Chloride and sulfate contents of the expressed pore solution as a fundtion of the admixed chloride of the cement paste

Steel specimens obtained from UMKC were allowed to passivate in their respective testing solutions and after the bars have reached full passivation (determined by a non-varying corrosion potential), chlorides and sulphates were added. In solution chloride concentrations varied between 1.2 and 2 % by weight of solution. Anodic polarization tests were performed to determine the pitting potentials of steel specimens at different chloride concentrations after their potentials stabilized. Figure 50 shows the anodic polarization curves at different solution chloride concentrations.

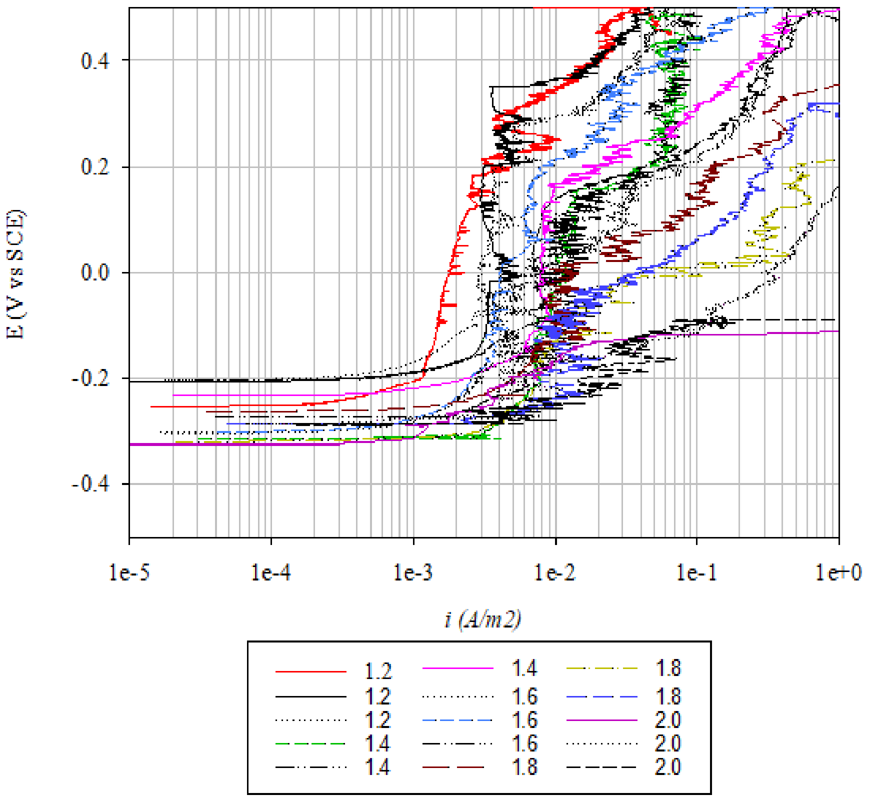


Figure 50: Cyclic polarization curves at five different solution chloride concentrations

Stabilized corrosion potentials of steel specimens before anodic polarization and their pitting potentials are plotted in Figure 51. This figure shows that the potential of steel specimens were linearly decreasing as the solution chloride content increased. The pitting potentials obtained through anodic polarization were also decreasing linearly with increasing solution chloride concentrations. Theoretically, these lines intersect at a solution chloride content value, where the steel potential will be equal to the pitting potential. At this theoretical solution chloride concentration, pitting of steel would initiate without the need of anodic polarization, i.e. this chloride concentration will be the critical chloride content, Cl_{crit}. Figure 51 indicates this would occur in cement/concrete with a pore solution chloride concentration of 2.35% Cl^-. Using Figure 49 this solution chloride content can be converted back to chloride concentration as % by weight of cement. Analysis shows that critical chloride concentration is 1.1 % by weight of cement.

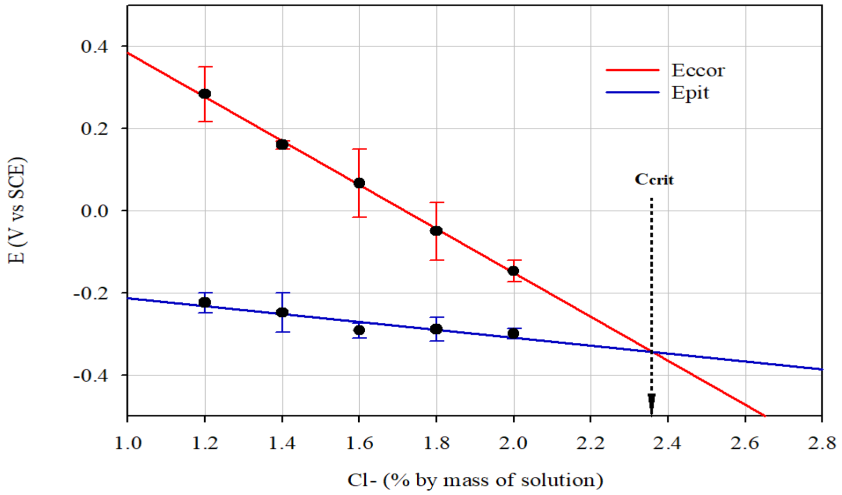


Figure 51: Steel potentials plotted vs. pitting potentials at different chloride contents

This theoretical Cl_{crit} value of 1.1 % obtained from in solution test is much higher compared to the 0.416 % obtained using the OC_{crit} data. Although the determined 1.1% value is in agreement with previous studies in the literature that evaluated critical chloride threshold in solution tests [14], its validity is questionable since this test method does not imitate the variability of the steel-mortar interface. Initial thinking of TG1 was that the result of this test could be used to help make a recommendation in case both evaluated test methods in the round robin test gave different but consistent results. However, since results obtained from the ASTM type test setup do not agree for the same steel-mortar system between different labs, the results of this in-solution test will not be used to make a recommendation. It should be noted that the theoretical concept of this test method can be applied to test setups using mortar or concrete as the alkaline medium and further evaluation in the future would be interesting.

5. Summary and Conclusions

Lack of a standard critical chloride content, C_{crit}, test is a significant issue that is causing confusion in the concrete industry, among engineers and researchers, and owners of reinforced concrete structures. Variety of test methods used in the literature to determine C_{crit} values contributes to the large scatter of published values. These publishd values vary between 0.1 and 3.1% by weight of binder [4]. Researchers cannot compare C_{crit} values obtained from their research to a standard value. Practitioners evaluating condition of reinforced concrete structures for maintenance and rehabilitation cannot make a reliable assessment based on measured chloride contents. Designers cannot estimate the service life of their design without a reliable estimate of C_{crit} value. Development of a standard critical chloride threshold test with a complete data set regarding its variability was the objective of this study.

A task group was established by the ACI committee 222 on corrosion of metals in concrete to initiate and support this effort. The task group evaluated various C_{crit} test methods in the literature including a similar earlier study performed by a RILEM committee and developed a framework for a standard C_{crit} test method. Two mortar based C_{crit} test methods were developed based on the task group framework. These two methods, OC_{crit} and ASTM type methods, were evaluated in a round robin test program with the participation of multiple laboratories. All testing was performed using materials procured from one source and distributed to the participating laboratories. A third in-solution test was also performed using materials that were evaluated in the round robin test.

5.1. OC_{crit} Test Method

Our findings and conclusions for the OC_{crit} test method are as follows;

1. Two test sets performed in the same laboratory on the same steel-mortar combination were statistically not significantly different and resulted in a mean C_{crit} of 0.425% by weight of cement and a COV of 18.8%. Analysis of data between different laboratories also showed that the differences in means were not statistically significant. The mean value of combined data was 0.416% by weight of cement with a COV of 26% and Figure 52 shows the distribution of results.

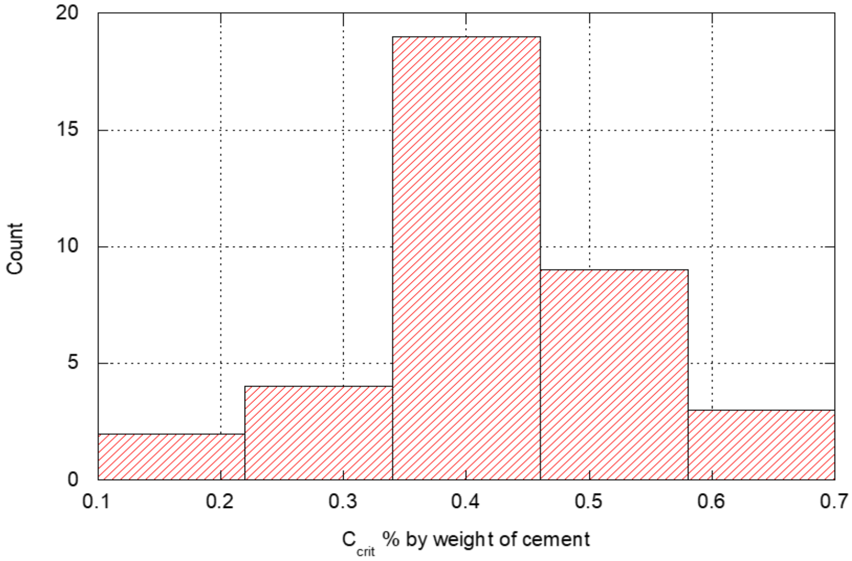


Figure 52: Histogram of C_{crit} data obtained in 4 different test sets

2. Although time to activation within the two tests performed in one lab were similar, time to activation was different between different laboratories. Statistical analysis showed that the time to activation at CTL Group laboratory was significantly longer compared to the other labs. The initial potential values of samples also varied between different laboratories. However, these differences did not cause a difference in the measured C_{crit} values.

3. Casting of OC_{crit} test samples using a three part mold is a complicated process that requires practice to produce samples with a 0.19 in (5 mm) thin cover. Initial trials showed poor

consolidation of samples may results in surface defects.

4. OC$_{crit}$ test monitors corrosion by measuring steel potentials at 24 hour intervals using a multimeter and a reference electrode. All samples tested in this program activated within a month and corrosion was visually confirmed under the thin mortar cover of samples.

5. Collection of mortar powder is a simple and uniform process that contributes to the low variability of results. A statistically developed equation is used to correlate the test results to the C$_{crit}$ value at the steel-mortar interface. The validity of this equation should be further evaluated for different cementitious systems.

5.2. ASTM type Test Method

Our findings and conclusions for the ASTM type test method are as follows;

1. Testing of mortar mixtures with s/c of 1.375 and 2.0 at two laboratories each, showed statistically significantly different results. This study strictly controlled variation of materials and test setup preparations. The fact that the results were significantly different at different laboratories for the same steel-mortar system indicates that this test method needs further improvement to reduce variability.

2. Casting of these larger size samples with a highly flowable mortar caused settlement and excessive bleeding issues. Although casting samples with less workable mortar mixtures eliminated settlement and bleeding, shrinkage cracks were observed on most of the samples cast with different mortar mixtures. The required 14 days of drying at 50% relative humidity environment and the large size of ASTM type samples may be the cause of observed cracking.

3. Used activation criteria failed to identify initiation of corrosion at some samples. Although these samples did not satisfy the activation criteria, initiation of corrosion was visually confirmed at the end of the testing period. Incremental rather than large changes in steel potentials and repassivation of steel samples are believed to have contributed to the observed variability.

4. The drilling procedure proposed in the standard for mortar powder collection was modified after discussions with TG1. Initial results showed that the chloride contents of samples collected using this procedure were too low. A procedure similar to the one used for ASTM G109 was used instead of the proposed procedure. Based on variability of one directional diffusion profile and the size of the samples and mortar cover, the process of mortar powder collection may have contributed to the variability of obtained results.

5.3. In-solution Test Method

The in-solution testing of the same cement and steel combination resulted in a critical chloride content estimation of 1.1% by weight of cement. This value is significantly higher compared to the results obtained from the mortar tests, evaluated in the round robin test. These results show that even if the same materials are tested, the test method can significantly affect the obtained C$_{crit}$ values. The steel mortar interface in solution is much more uniform compared to the mortar tests and this uniformity may have affected the results. In addition to the alkaline medium used, the concept of C$_{crit}$ determination was also very different from the mortar tests. The mortar tests exposed the system to chlorides and monitored for a change in certain properties, such as the steel potential or macro-cell corrosion current, to detect the corrosion initiation. The in-solution test changed the steel potential externally to initiate corrosion at different chloride contents and then estimated the chloride content at which corrosion initiation would be instantaneous without external manipulation.

5.4. Recommendations

During the ACI Fall 2020 virtual convention, these results were presented to the ACI committee 222 and the task group recommended the OC$_{crit}$ test as a standard critical chloride threshold test. The round robin test established the variability that should be expected when using the OC$_{crit}$ test in one or multiple laboratories. Since this data and information on basic variability is now established, further research can be performed to quantify the effect of other parameters on the variability of critical chloride content. One such important parameter is the source of materials. Materials that meet the same technical specifications may exhibit very different corrosion performances, e.g. steels that meet the requirements of ASTM A615 procured from different manufacturers from different parts of the country may have very different surface conditions affecting their corrosion resistance.

Further research and development of the OC$_{crit}$ test method to establish this as a standard critical chloride test method is recommended. Evaluation of the expected variability due to different parameters, such as source of materials, different steel types, use of SCMs, is necessary for this purpose. After determining expected added variability of these parameters, we recommend the development of the OC$_{crit}$ test method as an ASTM standard.

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A. Appendix A: OC$_{crit}$ Test Standard

A proposal for

Standardized Testing Protocol for Determination of Corrosion Initiation and Critical Chloride Threshold of Steel Reinforcement Embedded in Cementitious Materials

Submitted to

ACI 222-TG1

by

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1. Introduction

Chloride-induced corrosion of steel reinforcement in concrete results in a reduction in service life of reinforced concrete (RC) structures. The corrosion of steel reinforcement is considered to initiate when the chloride concentration at the steel-concrete interface reaches a certain level, commonly referred to as critical chloride threshold, C_{crit}. The C_{crit} values reported in the literature exhibit wide variation, with values differing by at least two orders of magnitude [1,2]. An important factor contributing to this signification variation in C_{crit} is the use of different test setups and procedures by different researchers and test agencies. Many of these test methods require significant time and labor to complete the test, which make these tests less likely to be used. Hence, a standard test procedure is required that is fast and economical, and the C_{crit} values determined through this test procedure must be representative of, or at least correlated with, the C_{crit} values in actual structures. The standard test method should be simple, repeatable, economical, and timely. This document provides a [preliminary] standard test procedure to assess the C_{crit} for steel reinforcement embedded in mortar.

2. Scope

2.1 This test is intended to determine the C_{crit} values for steel reinforcement embedded in mortar and subjected to chloride exposure.

2.2 The values stated in SI units or inch-pound units are to be regarded separately as the standard.

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1) Alonso, C., Castellote, M., and Andrade, C., "Chloride Threshold Dependence of Pitting Potential of Reinforcements," *Electrochimica Acta*, V. 47, 2002, pp. 3469-3481.

2) Angst, U., Elsener, B., Larsen, C. K., and Vennesland, Ø, "Critical Chloride Content in Reinforced Concrete—A Review," *Cement and concrete research*, V. 39, No. 12, 2008, pp. 1122-1138.

2.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

3. Referenced Documents

3.1 *ASTM standards*

A615/A615M Specification for Deformed and Plain Carbon-Steel Bars for Concrete Reinforcement

C109/C109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

C150/C150M Specification for Portland Cement

C185 Test Method for Air Content of Hydraulic Cement Mortar

C305 Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

C470/C470M Specification for Molds for Forming Concrete Test Cylinders Vertically

C511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes

C778 Standard Specification for Standard Sand

C876 Test Method for Corrosion Potentials of Uncoated Reinforcing Steel in Concrete

C1152/C1152M Test Method for Acid-Soluble Chloride in Mortar and Concrete

C1218/C1218M Standard Test Method for Water-Soluble Chloride in Mortar and Concrete

C1437 Standard Test Method for Flow of Hydraulic Cement Mortar

C1602/C1602M Standard Specification for Mixing water Used in the Production of Hydraulic Cement Concrete

D632 Specification for Sodium Chloride

G1 Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens.

G33 Practice for Recording Data from Atmospheric Corrosion Tests of Metallic-Coated Steel Specimens

G193 Standard Terminology and Acronyms Relating to Corrosion

4. Terminology

4.1 Definitions specific to this standard:

4.2.1 Open-circuit potential, OCP—The potential of steel reinforcement measured under open-circuit conditions relative to a reference electrode (also known as electrochemical corrosion potential or free corrosion potential).

4.2.3 Corrosion initiation— An OCP value more negative than -350 V for two consecutive days is considered as preliminary criteria for corrosion activation for round-robin test.

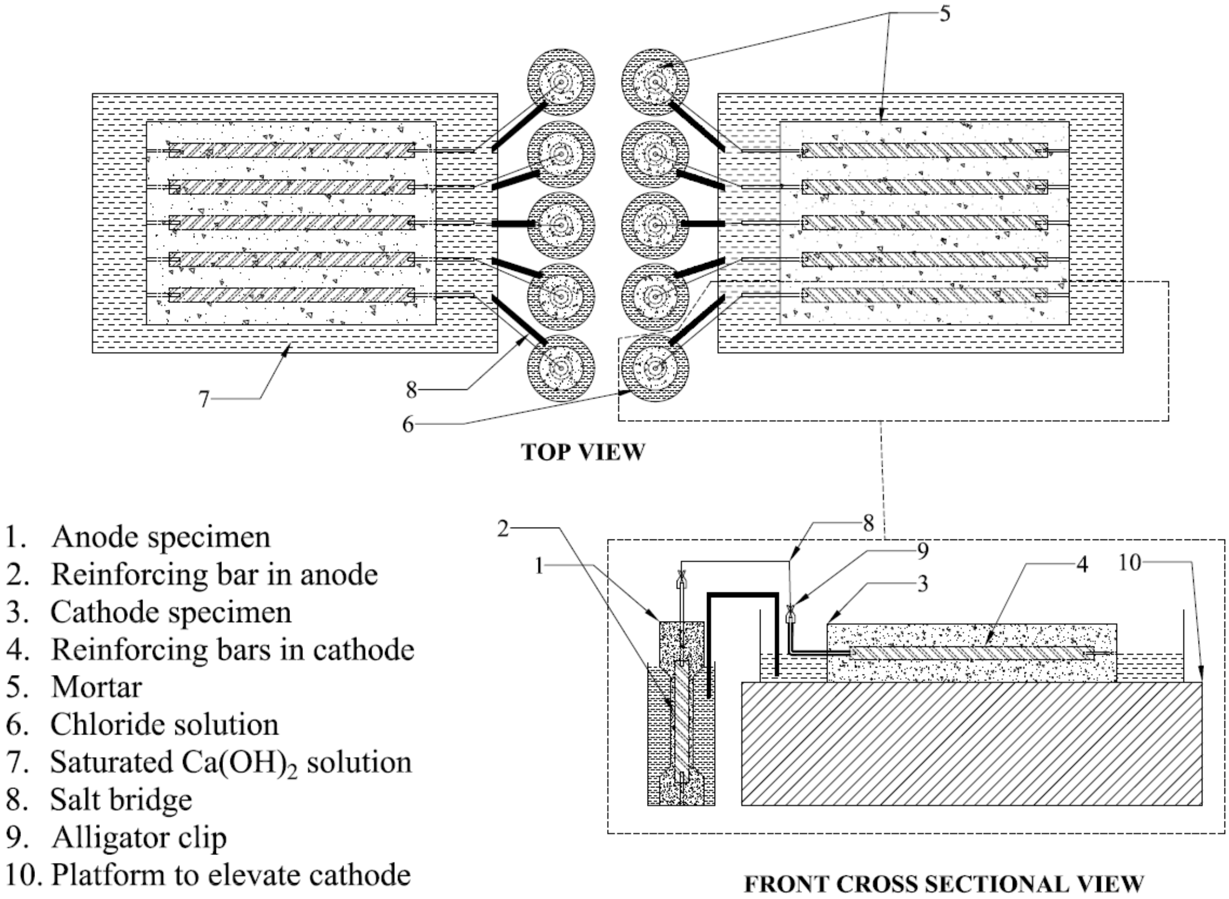
4.2.5 Critical chloride threshold, C_{crit}—The chloride concentration of the mortar, expressed as percent by mass of cement [or cementitious material], that results in the initiation of corrosion of steel reinforcement (for validation testing, this value will be for steel reinforcement meeting ASTM A615 requirements). For the validation testing, both ASTM C1152 and ASTM C1218 test standards will be used to measure chloride concentration of mortars from the specimens.

5. Summary of Test Method

5.1 The proposed test setup mimics a macro-corrosion cell, that is, the anode and cathode specimens are connected by insulated wires and the electrolytes surrounding the anode and cathode are connected through a salt bridge to complete the corrosion cell (see Figure 1). A mortar mixture made of standard sand is used for preparing the anode and cathode specimens. A dog-bone shaped mortar specimen with "as-received" steel reinforcement completely embedded in mortar is the anode (see Figure 2). Ten identical anode specimens are tested to capture the inherent variability associated with the reinforcing steel-mortar system. A prism shaped mortar specimen, with ten equally-spaced reinforcing steel samples completely embedded in mortar, is used as the cathode.

5.2 After the specified curing time, the mortar specimens shall be immersed in chloride exposure solutions. Electrical connections are established between cathode and anode specimens using conducting wires and salt bridges. The cathode specimen is immersed in saturated $Ca(OH)_2$ solution. The anode specimens are subjected to a continuous chloride exposure until corrosion initiation. Open-circuit potential of anode specimens are recorded on daily basis.

5.3 After corrosion initiation, the anode specimen is disconnected from the cathode and allowed to dry for [24] hours. The thin mortar cover, directly over the steel reinforcement, is crushed, grinded and then tested for acid-soluble chlorides (ASTM C1152). Research is ongoing to determine the correlations between the chloride concentrations determined from testing and C_{crit}. For round-robin testing, the mean and standard deviation of the chloride concentrations determined from testing will be reported.



1. Anode specimen
2. Reinforcing bar in anode
3. Cathode specimen
4. Reinforcing bars in cathode
5. Mortar
6. Chloride solution
7. Saturated $Ca(OH)_2$ solution
8. Salt bridge
9. Alligator clip
10. Platform to elevate cathode

Figure 1 – Schematic of the test setup

6. Significance and Use

6.1 Chloride-induced corrosion of steel reinforcement in concrete is a well-known issue and several factors, including the material type (steel and concrete/mortar), environmental conditions, and testing parameters have been reported to influence the corrosion process [1,2]. This test procedure is developed to standardize a laboratory procedure for quantifying the amount of chlorides required to initiate corrosion of steel reinforcement embedded in mortar (i.e., C_{crit}). It is anticipated that this information will be correlated with C_{crit} values found in actual structures.

6.2 The C_{crit} for the reinforcing steel-cementitious system used in this study is anticipated to follow a certain statistical distribution due to the heterogeneous nature of reinforcing steel surface, the heterogeneous nature of the steel-cementitious interface, and the heterogeneous nature of the cementitious system adjacent to the steel reinforcement. Characterizing the statistical distributions for C_{crit} for this standard system is necessary to establish a baseline for assessing C_{crit}. However, the eventual objective is to understand the influence of these parameters on C_{crit} and service life. Hence, this test is anticipated to be eventually used for comparing the relative performance of different reinforcing steel-cementitious systems.

7. Apparatus

7.1 *Mortar mixer*—A mechanical mixer meeting ASTM C305 requirements shall be used.

7.2 *Vibrating table*—A vibrating table meeting ASTM C192 requirements shall be used.

7.3 *Voltmeter*—A high impedance voltmeter or a multi-meter that is capable of measuring the half-cell corrosion potential of anode.

7.4 *Copper-copper sulfate reference half-cell electrode with porous tip* (see Test Method ASTM C876, Paragraph 5.1.1.1 for detailed description).

7.5 *Ultrasonic cleaner*—The ultrasonic cleaner bath shall be big enough to clean reinforcing bar samples of 381 mm (15 in.) lengths.

8. Materials

8.1 *Cement*—A Type I/II cement confirming to ASTM C150 specifications shall be used for validation testing. The chemical analysis and physical properties, as reported in the cement-mill test report, shall be reported. For future testing, if the cement is not a Type I/II, note the cement type and provide chemical analysis and physical property data.

8.2 *Water*—The water used for preparing mortar shall meet ASTM C1602 requirements.

8.3 *Mortar*—For the round-robin test, the mortar shall be developed using ASTM C778 graded sand and by using a sand to cement ratio of 1.375 and a water to cement ratio of 0.42. In future studies, details of the mortars that do not meet these requirements shall be reported.

8.4 *Reinforcing steel*—Use No.16 (No.5) steel reinforcing bars meeting ASTM A615/A615M requirements for validation testing. All samples shall be ribbed and obtained from the same heat. If the influence of the type of steel reinforcement on C_{crit} is being assessed, the complete details (e.g. chemistry, tensile strength, ultimate strength etc.) of reinforcement shall be reported. The mill scale on the steel reinforcement shall not be removed and all samples shall be free of corrosion.

8.5 *A #29 drill bit (size: 0.136 inches or 3.45 mm)*

8.6 A #32 drill bit (size: 0.116 inches or 2.95 mm)

8.7 *A 5-40 tap and die set*

8.8 *Straight copper wires*—Two types of copper wires, having different dimensions, shall be used for preparing anode specimens. The first type of cooper wire shall a diameter of 3.26 mm (0.13 in.) and a length of 95.25 mm (3.75 in.). The second type of copper wire shall have a 3.26 mm (0.13 in.) and a length of 43.18 mm (1.70 in.).

8.9 *Heat-shrink tube*—The tube shall have an inner diameter of 4.8 mm (0.1875 in.) and a shrink ratio of 2:1. The material of the tube shall be resistant to high pH exposure.

8.10 *Prefabricated specimen molds*—Figures A1 to A3 show details of different prefabricated specimen molds used for preparing anode specimens. Specimen molds A, B, and C are referred herein as SM-A, SM-B, and SM-C respectively.

8.11 *Prefabricated funnel mold*—Figure A4 shows details of prefabricated funnel mold D used in placing mortar into specimen molds. This mold is herein referred as FM-D.

8.12 *Prefabricated alignment molds*— Figure A5 shows the details of alignment molds E and F used for centering the anode-reinforcing bar during specimen fabrication. The alignment molds E and F are herein referred as AM-E and AM-F, respectively.

8.13 *Volume molds*—Figure A6 shows the details of volume molds G and H used for obtaining mortar of known volumes. The volume molds G and H are herein referred as VM-G and VM-H, respectively.

8.14 *50.8 mm x 50.8 mm x 50.8 mm [2 in. x 2 in. x 2 in.] molds*—To test compressive strength of mortar.

8.15 *M6 x 1 x 127 [¼-20 x 5] screws*

8.16 *M6 x 1 x 38 [¼-20 x 1.5] countersunk screws*

8.17 *Standard ¼-20 nuts*

8.18 *M12 x 1.75 x 241 [½-13 x 9.5] countersunk screws*

8.19 *Standard ½-13 nuts*

8.20 ACS grade NaCl meeting ASTM D632 requirements.

8.21 ACS grade $Ca(OH)_2$

8.22 *Salt bridge*—This is used to provide ionic path between anode and cathode.

8.23 *Form release oil*

8.24 *Insulated wires*

9. Specimen Preparation

9.1 *Anode specimen*

9.1.1 *Reinforcing bar*—Cut No.16 [No.5] reinforcing samples to 140 mm [5.5 in.] lengths. Samples shall be cut with any method available, however, the heating of reinforcement shall be prevented and the surface of the ends of the reinforcing bar after cutting shall be planar and perpendicular to the longitudinal axis of the reinforcement. Obtain 10 samples, one sample for each anode specimen.

9.1.1.1 For each anode reinforcing bar sample, drill a hole of 12.2 mm (0.5 in.) depth using a #29-drill bit on one end (see Note 1). This end of reinforcing bar is herein referred as end A. On the opposite end of reinforcing bar, use a #32-drill bit to drill a hole of at least 6.35 mm (0.25 in.) depth (herein referred as end B). Using a 5-40 tap bit, tap the hole on reinforcing bar end B. Clean the reinforcing bar samples in ethylene in an ultrasonic bath; samples shall be maintained in ultrasonic cleaner for a sufficient duration to remove all foreign materials from surface. Using a 5-40 die, cut threads on one end of a straight copper wire of 3.26 mm (0.13 in.) and a length of 95.25 mm (3.75 in.). The length of the threaded region shall be 6.35 mm (0.25 in.). Screw the threaded end of copper wire into end B of the reinforcing bar.

Note 1—Most drill bits, used for drilling holes in hard metals, have a point angle (θ). To drill a hole of diameter *d* and depth *x* using a drill bit with point angle *θ*, one must drive the tip of the drill bit to a depth of $x + d\big/\big(2\tan\left(\theta/2\right)\big)$ from the surface. This correction shall be considered for drilling holes for all reinforcing bars.

9.1.2 *Molds*—SM-A, SM-B, and SM-C are used to fabricate the anode specimen. Each mold has two parts. Apply a thin layer of form release oil on the inner surface of the mold pieces. Using M6 x 1 x 127 [¼-20 x 5] screws and nuts, connect both parts of molds A, of mold B, and of mold C.

9.1.3 *Mortar mixing and testing*—Follow the mixing guidelines specified in ASTM C305 for preparing mortar. Immediately after preparing the mortar, test the air content and flow of the mortar following ASTM C185 and ASTM C1437 specifications. Using the 50.8 mm x 50.8 mm x 50.8 mm [2 in. x 2 in. x 2 in.] molds, cast mortar specimens for testing 7-day and 28-day compressive strength of the mortar cubes. These specimens shall be moist cured for 6 or 27 days (1 less day than the test period) and shall be tested per ASTM C109/C109M requirements. An additional three specimens shall be prepared to measure the background chloride concentration of mortar per ASTM C1152.

9.1.4 *Fabrication of anode specimen*—Figure 2 identifies the different regions of anode specimen prepared using SM-A, SM-B, and SM-C. Guidelines on fabricating anode specimen follow.

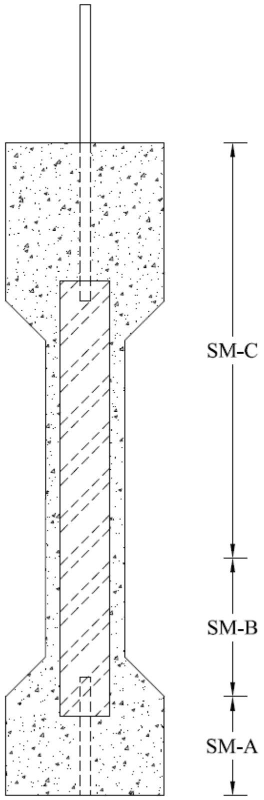


Figure 2 – Different regions of the anode specimen

9.1.4.1 **Step 1:** Using duct tape, cover the center bottom hole of SM-A. Consider a straight copper wire having a diameter of 3.26 mm (0.13 in.) and a length of 43.18 mm (1.70 in.). Insert one end of the copper wire into end A of the anode steel reinforcing bar. Insert the other end of the copper wire into the bottom of SM-A. Align the reinforcing bar approximately to the center of the mold. The reinforcing bar shall be held by hand in position (using gloves) to avoid relative motion between the reinforcing bar and SM-A. Care must be ensured for not exposing the reinforcing bar to foreign materials. Approximately fill half volume of SM-A with mortar. Place SM-A on the vibrating table until air voids visible to the surface are minimized (anticipated to be not more than

5 seconds). Slowly fill the remaining portion of the SM-A with mortar centering the reinforcing bar. Vibrate the setup until no air voids are noticeable on surface (not more than 15 seconds). Clean the top surface of SM-A by removing any extra mortar. Figure 3 shows the specimen setup after fabrication step 1.

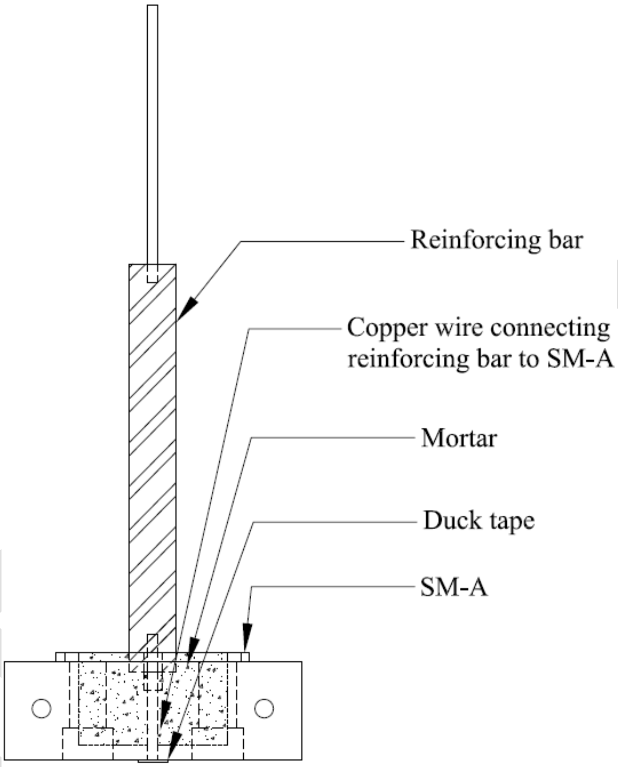


Figure 3 – A schematic of the specimen setup after fabrication step 1

9.1.4.2 **Step 2:** Using the M6 x 1 x 38 [¼-20 x 1.5] countersunk screws, attach SM-A and SM-B together. After connecting SM-B to SM-A, place FM-D over top of SM-B. Align the reinforcing bar by sliding AM-E over reinforcing bar and fixing AM-E on SM-B. The purpose of using the combined set up of FM-D and AM-E is to align the reinforcing bar along the longitudinal axis of the combined setup of the SM-A and SM-B, and to better control the placement of mortar into SM-B. Fill the volume of VM-G with mortar up to the specified level. This volume of mortar is the volume needed to fill SM-B. Place approximately half of the measured mortar from VM-G into FM-D. Vibrate the setup until all mortar inside FM-D flows down into SM-B. Then vibrate the setup for about 15 seconds to release any entrapped air-voids. Place the mortar remaining in VM-G into SM-B. Vibrate the setup until all mortar inside FM-D flows down into SM-B and then vibrate the setup for about 15 seconds. The reinforcing bar shall be held firmly during vibration to avoid any relative motion between the reinforcing bar and the mold setup. Detach FM-D from SM-B and clean the top surface of SM-B from extra mortar. Figure 4 shows the specimen setup after fabrication step 2.

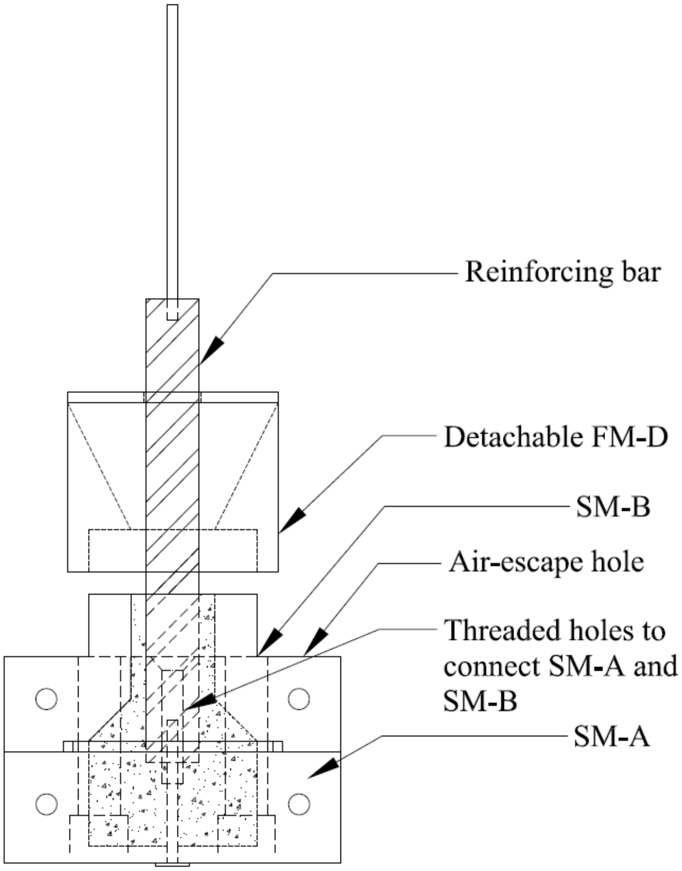


Figure 4 – A schematic of the specimen setup after fabrication step 2

9.1.4.3 **Step 3:** Detach FM-D (and AM-E) from SM-B and set FM-D off to side. Place SM-C on top of SM-B such that the copper wire. Using the M12 x 1.75 x 241 [½-13 x 9.5] screws, fix SM-C to the top of SM-B. Using AM-F, align the reinforcing bar such that the copper wire on end

B of the steel reinforcing bar passes through the center hole of the mold. The space between the anode-reinforcing bar and the inner surface (i.e., low cover area) of SM-C shall be filled with mortar in two layers. Fill VM-H with mortar up to the specified level. Approximately place half of the measured mortar into SM-C. Vibrate the set-up until all mortar slides into the ‘thin-cover’ region between SM-C and reinforcing bar. The copper wire attached to end B of the reinforcing bar shall be held firmly to avoid during vibration to avoid any relative movement between the reinforcing bar and the molds. Slowly remove AM-F. Using a thin round-ended cylindrical rod [preliminary dimensions: 1.6 mm (0.06 in.) diameter and 203 mm (8 in.) height, rod and consolidate the mortar. Distribute the strokes uniformly over the cross section of the mold. Following this, vibrate the setup for 15 seconds. Transfer the mortar remaining in VM-H into SM-C. Place AM-F on SM-C. Vibrate the setup until all mortar slides into the ‘thin-cover’ region. Following this, vibrate the setup for 15 seconds to remove any entrapped air voids. Fill approximately half of the empty space in SM-C with mortar and vibrate the setup for 15 seconds. Overflow the remaining space of SM-C with mortar and vibrate the setup for 15 seconds. Strike-off extra mortar from the top surface of SM-C. Figure 5 shows the specimen setup after fabrication step 3.

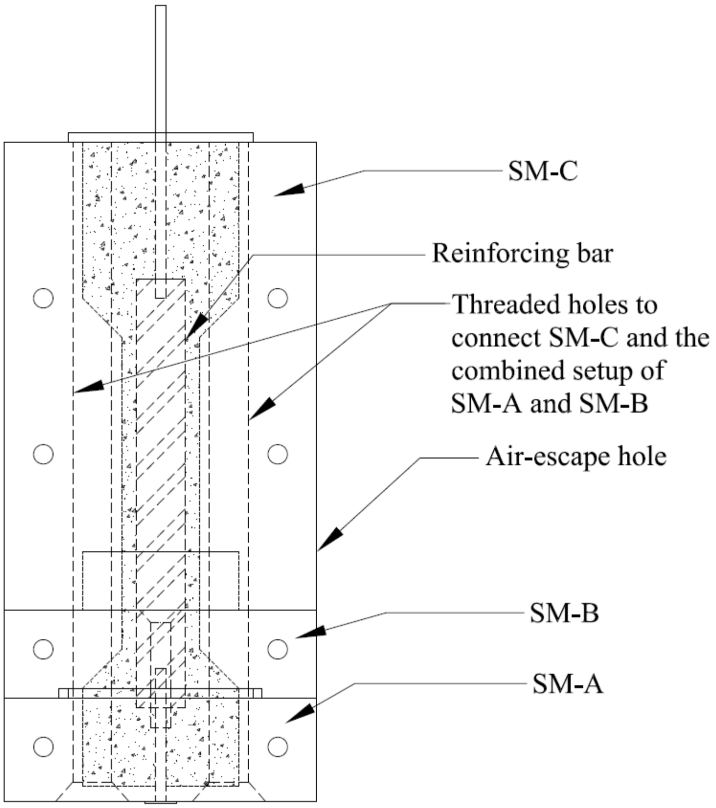


Figure 5 – A schematic of the specimen setup after fabrication step 3

9.1.4.4 **Step 4:** Seal the top surface of SM-C with a material that prevents evaporation (e.g., plastic wrap). Twenty-four hours after casting, remove the hardened specimen from the molds. For validation testing, where the mortar specimens are made of ordinary portland cement, the specimens will be cured in a moist room for 27 days. Moist room shall meet ASTM C192-16 requirements. After curing, remove the protruding copper wire from the bottom end of the mortar specimen using a dremel tool or similar. A schematic of the fabricated anode specimen is shown in Figure 6.

Note 2—Mortar specimens made of supplementary cementitious materials may be damaged if the specimen molds are removed too early after casting. Additional time may be required for such specimens before removing the specimen molds. Any additional time allowed must be reported. Specimens containing SCMs may also have to be cured for longer periods due to the slower hydration.

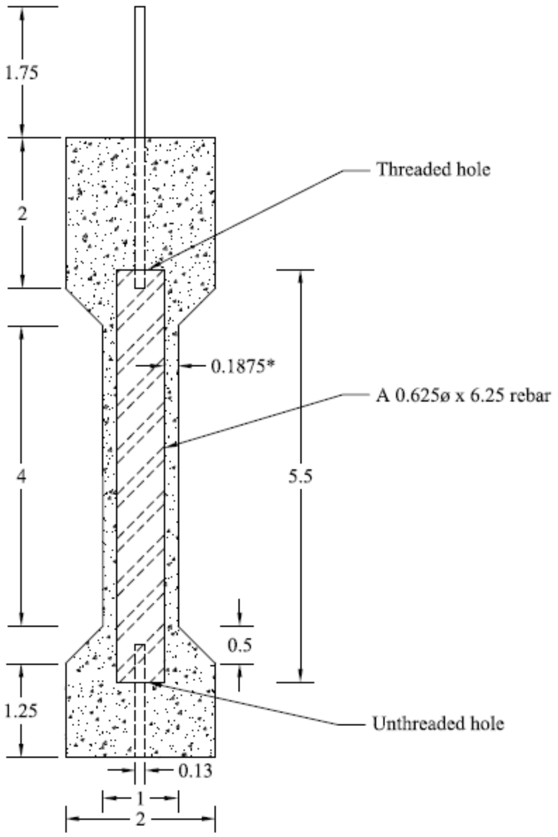


Figure 6 – Longitudinal cross section of the anode specimen
(all dimensions in inches and not to scale;*- cover based on the nominal diameter of the reinforcing bar)

9.2 *Cathode specimen*

9.2.1 *Reinforcing bar*—Cut No.16 [No.5] reinforcing samples to 297 mm [11 inch] lengths. Samples shall be cut with any method available, however, the heating of reinforcement shall be prevented and the surface of the ends of the reinforcing bar after cutting shall be planar and perpendicular to the longitudinal axis of the reinforcement. Clean and cut ten No.16 [No.5] reinforcing bar samples of 297 mm [11 inch] length each. Note that each cathode specimen has ten reinforcing bars.

9.2.1.1 For each cathode reinforcing bar sample, drill a hole of 12.2 mm (0.5 in.) depth using a #29-drill bit on one end. This end of reinforcing bar is herein referred as end C. On the opposite end of reinforcing bar, use #32-drill bit to drill a hole of at least 6.35 mm (0.25 in.) depth (herein referred as end D). Using a 5-40 tap bit, tap the hole on reinforcing bar end D. Clean the reinforcing bar sample in ethylene using an ultrasonic bath for sufficient duration to remove all foreign materials from surface. Using a 5-40 die, cut threads on one end of a straight copper wire of 3.26 mm (0.13 in.) diameter and an approximate length of 114.3 mm (4.5 in.). The minimum length of the threaded region shall be 6.35 mm (0.25 in.). Screw the threaded end of copper wire into end D of the reinforcing bar. Bend the copper wire 90 degrees at approximately 69.85 mm (2.75 in.) from the end D of the reinforcing bar. Using a heat-shrink tube, shrink-wrap the tubing onto the copper wire. Avoid the tubing for a small region at the end of the copper wire for electrical connections.

9.2.2 *Molds*—Plyform wood pieces with appropriate dimensions may be used to build a simple mold for the fabrication of the cathode specimen. Plyforms shall be 19 mm [0.75 inch] thick and should have a smooth surface on one side. The smoother sides of plyforms will form the internal surface of the mold. The dimensions of the inner surfaces of the mold should match the dimensions of the cathode specimen.

9.2.3 *Mortar Mixing and Testing*—The mortar material used for fabrication of anode and cathode specimens and testing of all mortar specimens shall be from the same batch.

9.2.4 *Fabrication of cathode specimen*—Figure 7 shows a schematic of the cathode specimen. The center-to-center spacing of reinforcing steel bars shall be 41.3 mm [1.625 in.] and a 25.4 mm [1 in.] mortar cover shall be provided for all bars. Coppers wires of 3.26 mm (0.13 in.) diameter and 63.5 mm (2.5 in.) lengths shall be used to align reinforcing bars in the mold. Care must be taken to ensure specified cover of the reinforcing bars while placing the mortar into the mold and vibrating the mold setup. The curing conditions for the cathode specimen shall be the same as the curing conditions of the anode specimen.

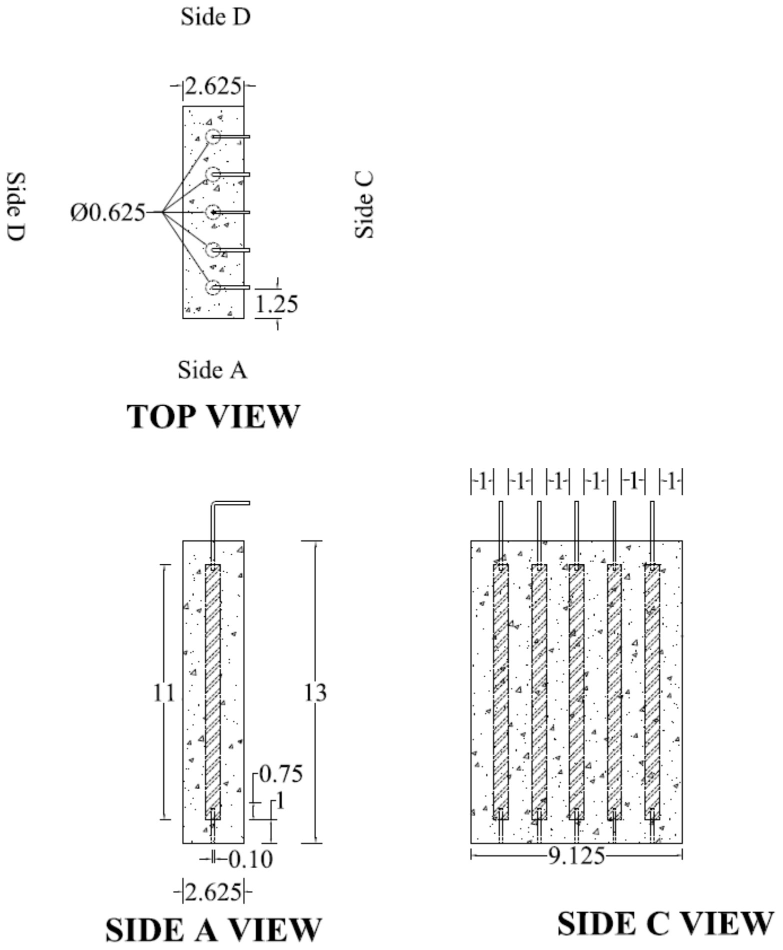


Figure 7 – A schematic of the cathode specimen
(all dimensions in inches and not to scale)

9.3 *Specimens for Resistivity and pH Testing*

9.3.1 The C_{crit} of a steel-cementitious system could be influenced by the resistivity of the mortar and the pH of the mortar pore solution. Therefore, it is necessary to establish correlations between these parameters and C_{crit}. Pore solution extraction will be used to generate samples for pore solution resistivity and pH (and possibly alkali content). The samples for extracting pore solution shall be 50.8 mm (2 in.) diameter by 101.2 mm (4 in.) height mortar specimens. pH testing shall be performed using pore solution extraction technique (see reference 3) and the leaching techniques (see references 4 and 5). The resistivity of mortar shall be investigated by testing 101.6 mm (4 in.) diameter by 203.2 mm (8 in.) specimens with a bulk resistivity meter.

10. Test procedure

10.1 *Test setup*

10.1.1 The schematic of the macro-corrosion cell setup for one anode specimen is shown in Figure 8. Each anode specimen shall be placed in a separate cylindrical-shaped plastic container during the period of chloride exposure. The plastic container shall be closed on one end and shall have a diameter of 76.2 mm (3 in.) and a height of 158.75 mm (6.25 in.). Place the anode specimens in the container and fill the container with a [2% chloride + saturated $Ca(OH)_2$] solution (herein referred as the exposure solution). The exposure solution shall be prepared by mixing dissolving [3.3 grams of ACS grade NaCl and 0.3 grams of $Ca(OH)_2$ in 100 grams of distilled water]. The level of the exposure solution shall be 50.8 mm (2 in.) below the top mortar surface of the anode specimen and this level must be maintained at all times. The exposure solution shall be replaced with new solution every week.

10.1.2 The anode specimens shall be placed in the anode solution immediately after taking them out from mist room. The samples shall be in saturated state before chloride exposure.

10.1.2 Procure a plastic container large enough to hold the cathode specimen. The container shall be resistant to degradation to high pH solution. Place the cathode specimen in the container

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3) Trejo, D., Shakouri, M., Vaddey, N., Isgor, O.B., “Development of Empirical Models for Chloride Binding in Cementitious Systems Containing Admixed Chlorides,” *Construction and Building Materials*, V.189, 2018, pp. 157-169.

4) Cáseres, L., Sagüés, A.A., Kranc, S.C., Weyers R.E., “In situ Leaching Method for Determination of Chloride in Concrete Pore water,” *Cement and Concrete Research*, V.36, 2006, pp. 492-503.

5) Arya, C., Buenfeld, N., and Newman, J., “Assessment of Simple Methods of Determining the Free Chloride Ion Content of Cement Paste,” *Cement and Concrete Research*, V.17, No.6, pp. 907-918.

and fill the container with saturated $Ca(OH)_2$ solution. The solution level shall be 33.78 mm [1.33 in.] below the top surface of the cathode. This solution level must be maintained at all times. Elevate the cathode container such that the level of solution in cathode is higher than the level of the solution in anode (see figure 8).

10.1.3 Use a salt bridge to establish the electrical contact between the electrolytes surrounding the anode and cathode specimens. A plastic pipe having an inner and outer diameter of 9.53 mm (0.38 in.) and 12.7 (0.5 in.) shall be used. The conductive material for the salt bridge shall be prepared following the procedure reported in Kahrs et al. (2001)[6]. The copper wires protruding from anode and cathode specimens shall be connected using insulated wires and alligator clips. The salt bridges shall be replaced every three weeks.

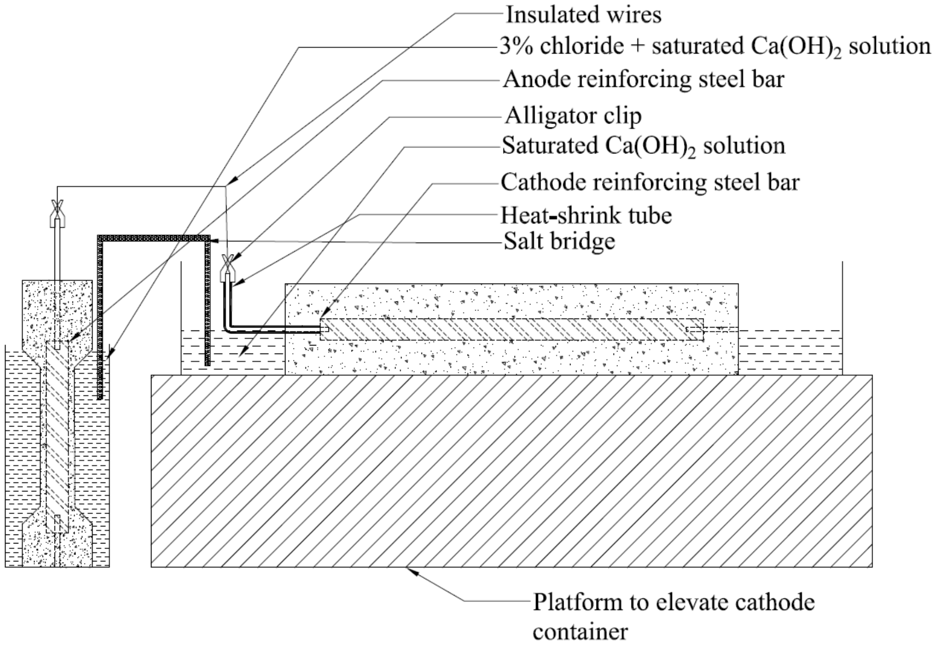

Figure 8 – Illustration of corrosion setup for an anode specimen

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6) Kahrs, J. T., Darwin, D., and Locke, C. E., "Evaluation of Corrosion Resistance of Type 304 Stainless Steel Clad Reinforcing Bars," University of Kansas Center for Research, Inc., Lawrence, Kansas, 2001.

10.2 *Electrochemical measurements*

10.2.1 The OCP of the reinforcing bar of anode specimen shall be taken at 24-hour intervals as discussed in 10.2.2. The first OCP reading shall be taken immediately after taking the specimen out of the fog room and before dipping the specimen in chloride solution. OCP readings shall be recorded Monday through Friday. The measurements shall not be taken during the weekend, however, the specimens shall remain exposed to chloride solution.

10.2.2 Determine the OCP of the reinforcing bar in accordance with Test Method ASTM C876. The corrosion potential is determined by attaching the common (black) terminal of the voltmeter to the half-cell electrode and the active (red) terminal of the voltmeter to the copper wire from the reinforcing bar of the mortar specimen. The connection between the voltmeter and the reinforcing bar shall be as close to mortar as possible. The tip of the copper-copper sulfate electrode shall be placed on the center surface of the anode specimen. A pencil cell electrode with a 9.5 mm (3/8 in) tip shall be used. One reading shall be recorded per specimen every day at the center of the '4-inch' section between the dog-bone ends of the specimen (i.e., 114.3 mm (4.5 in.) from the top end of the specimen). The location used for recording the first reading shall be marked and the same location shall be used for all future readings.

10.3 *Corrosion initiation*

10.3.1 An OCP value more negative than –350 mV against the copper-copper sulfate electrode for two consecutive days will be used as corrosion activation criteria for round-robin test. If the first OCP reading, that is more negative than -350 mV, is recorded on Friday, the specimen shall be kept exposed to chloride solution during the weekend. The specimen shall be considered activated if the OCP reading on the following Monday is more negative than -350 mV. All anode specimens shall be treated independently to determine corrosion activation.

10.4 *Chloride testing and reinforcing bar assessment*

10.4.1 Immediately after the anode specimen is activated, the specimen shall be removed from the chloride solution and detached from the macro-cell setup. Allow the specimen to dry for [24 hours]. Following this, remove the mortar directly above the reinforcing bar and between the dog-bone ends of the specimen (refer to Figure 5). The obtained mortar pieces should be ground into a fine powder such that the entire sample passes through a No.20 sieve. Determine the acid-soluble chloride concentration per ASTM C1152.

10.4.2 Examine the steel reinforcement bars for extent of corrosion and measure and document the corroded area. Record the percentage of corroded area, as described in G33.

11. Calculation

11.1 Transform the readings obtained from ASTM C1152 and ASTM C11218 testing from percent by mass of sample to percent by mass of cement [or cementitious material] by using appropriate conversion factors as follows,

$$\left[Cl^{-}\right]_{test,cem} = \left[Cl^{-}\right]_{test,mor} \times \frac{M_m}{M_c}$$

where $\left[Cl^{-}\right]_{test,cem}$ represents the chloride concentration as a percentage of cementitious material mass, $\left[Cl^{-}\right]_{test,mor}$ represents the chloride concentration as a percentage of mortar mass determined through testing, M_m represents the mass of the mortar per unit volume of mortar and M_c represents the mass of cement [or cementitious material] per unit volume of mortar. The M_m and M_c shall have the same units. For round robin testing, determine and report the mean and standard deviation of the ten $\left[Cl^{-}\right]_{test,cem}$ readings obtained from the different specimens.

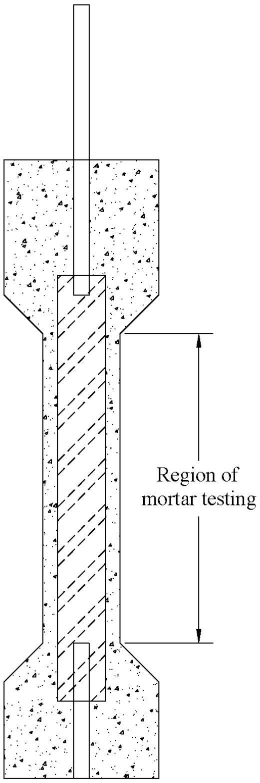
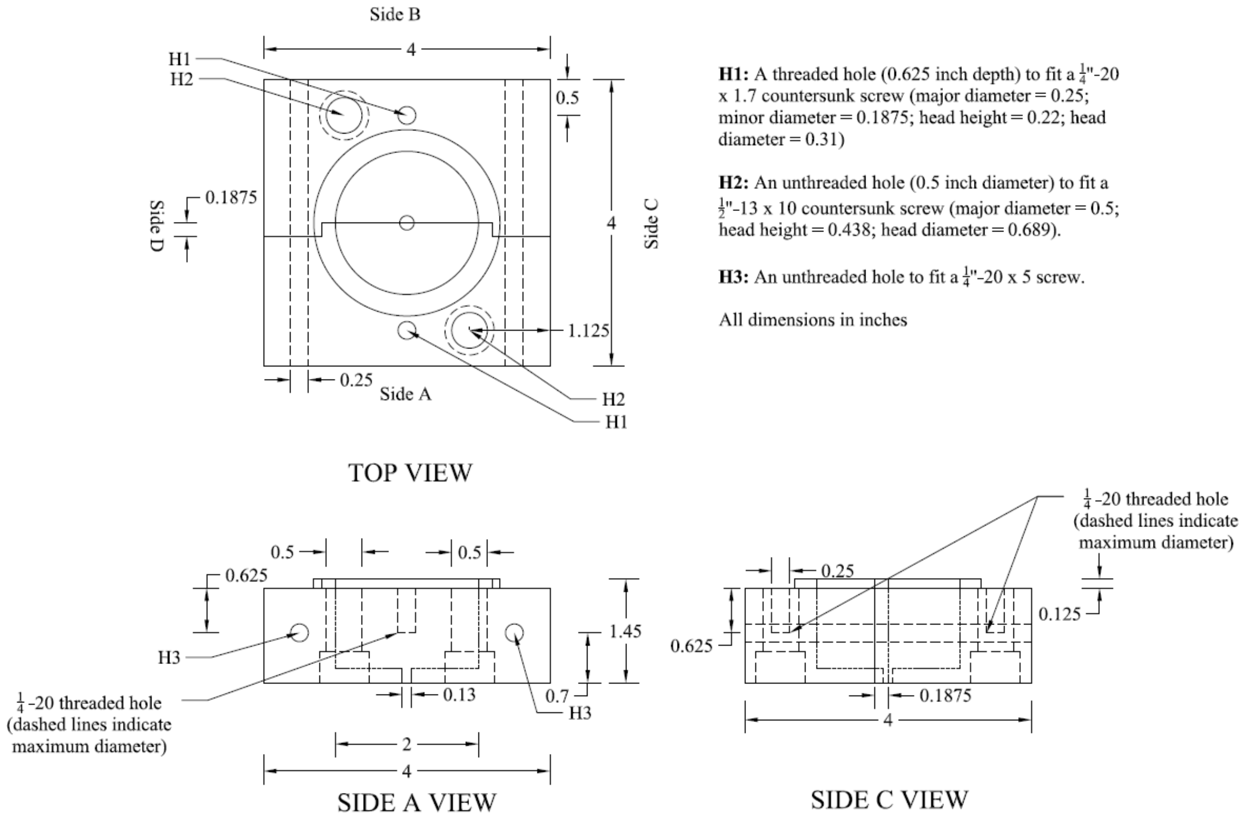


Figure 9 – Region of anode specimen considered for chloride analysis

12. Precision and Bias

12.1 Information on the precision and bias will be reported after the data generated from inter laboratory tests is analyzed.

Appendix



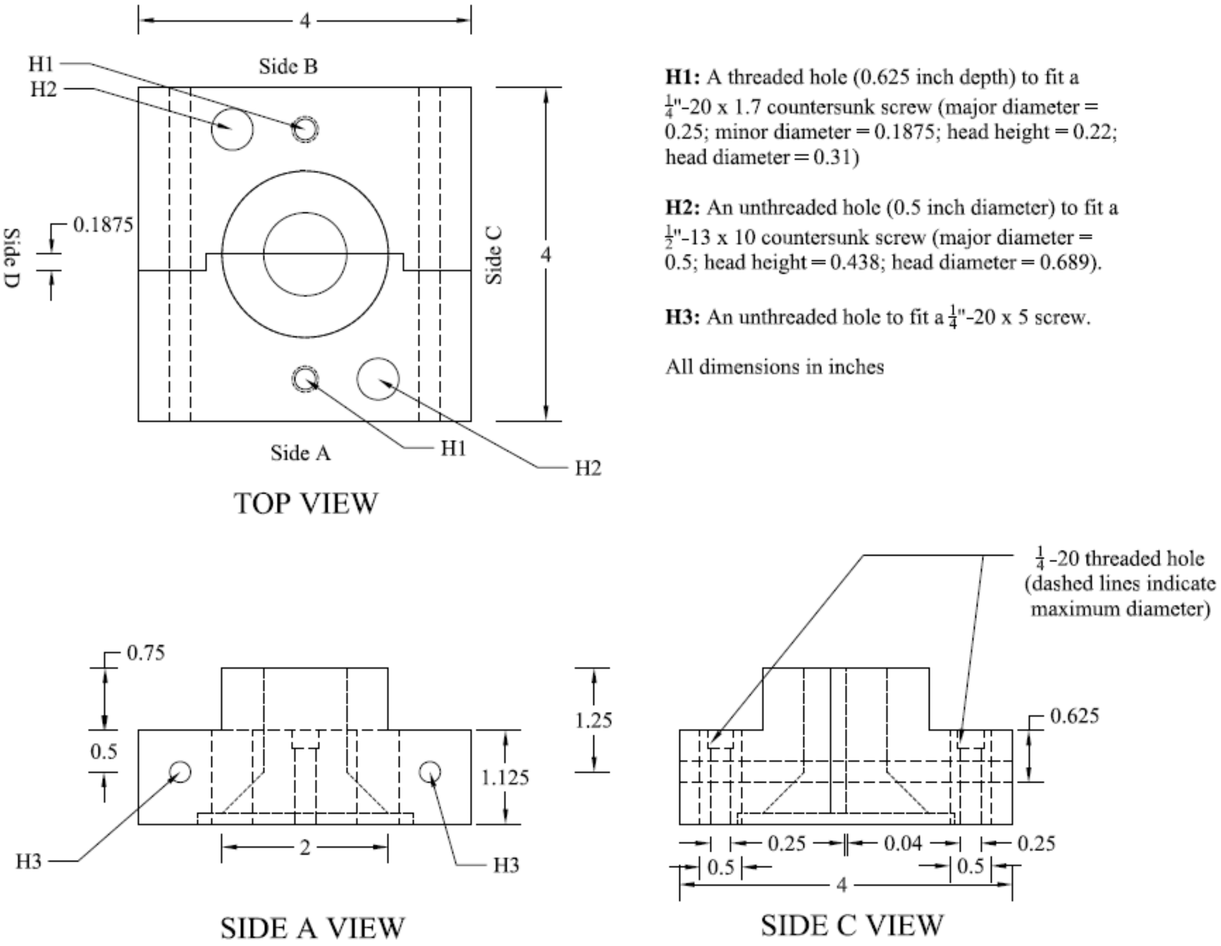
H1: A threaded hole (0.625 inch depth) to fit a $\frac{1}{4}$"-20 x 1.7 countersunk screw (major diameter = 0.25; minor diameter = 0.1875; head height = 0.22; head diameter = 0.31)

H2: An unthreaded hole (0.5 inch diameter) to fit a $\frac{1}{2}$"-13 x 10 countersunk screw (major diameter = 0.5; head height = 0.438; head diameter = 0.689).

H3: An unthreaded hole to fit a $\frac{1}{4}$"-20 x 5 screw.

All dimensions in inches

Figure A1 – Schematic for specimen mold A
(All dimensions in inches and not to scale)



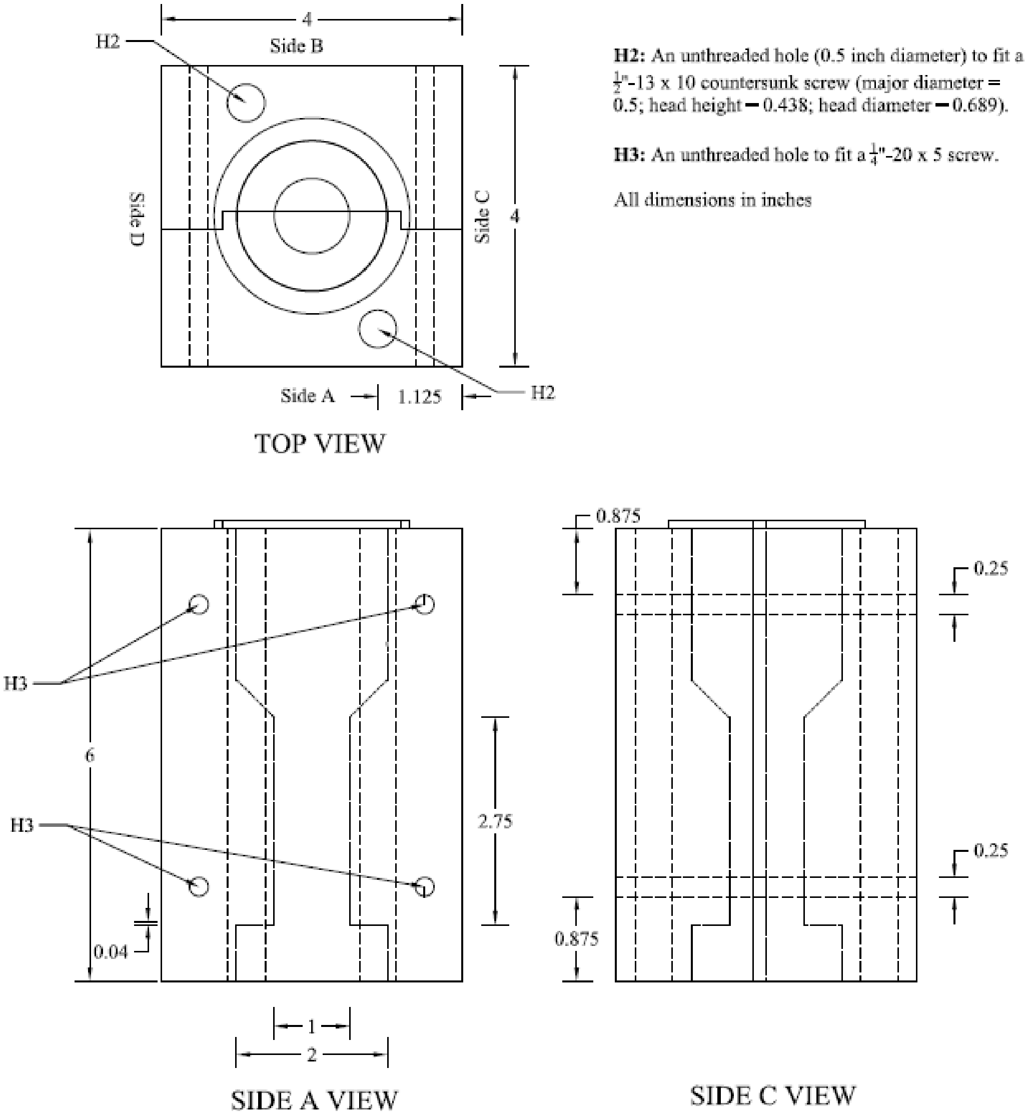
H1: A threaded hole (0.625 inch depth) to fit a ¼"-20 x 1.7 countersunk screw (major diameter = 0.25; minor diameter = 0.1875; head height = 0.22; head diameter = 0.31)

H2: An unthreaded hole (0.5 inch diameter) to fit a ½"-13 x 10 countersunk screw (major diameter = 0.5; head height = 0.438; head diameter = 0.689).

H3: An unthreaded hole to fit a ¼"-20 x 5 screw.

All dimensions in inches

Figure A2 – Schematic for specimen mold B
(All dimensions in inches and not to scale)



H2: An unthreaded hole (0.5 inch diameter) to fit a $\frac{1}{2}$"-13 x 10 countersunk screw (major diameter = 0.5; head height – 0.438; head diameter – 0.689).

H3: An unthreaded hole to fit a $\frac{1}{4}$"-20 x 5 screw.

All dimensions in inches

Figure A3 – Schematic for specimen mold C
(All dimensions in inches and not to scale)

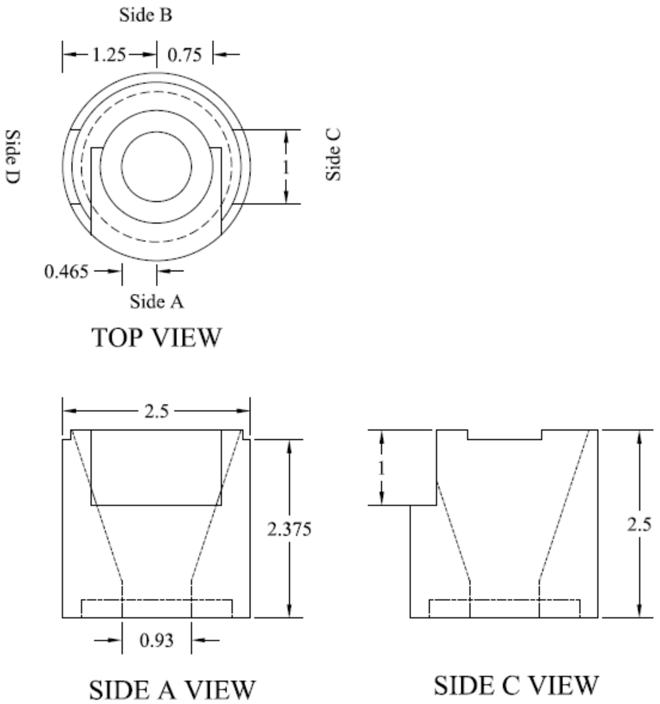
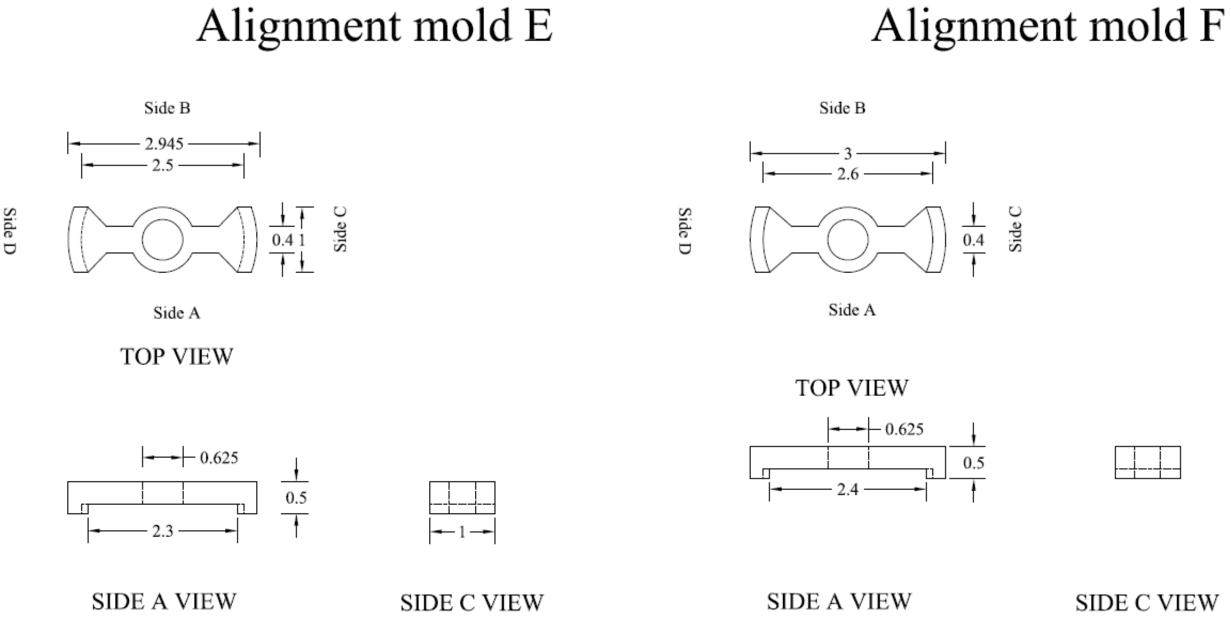


Figure A4 – Schematic for funnel mold D
(All dimensions in inches and not to scale)


Alignment mold E

Alignment mold F

Figure A5 – Schematics for alignment molds
(All dimensions in inches and not to scale)

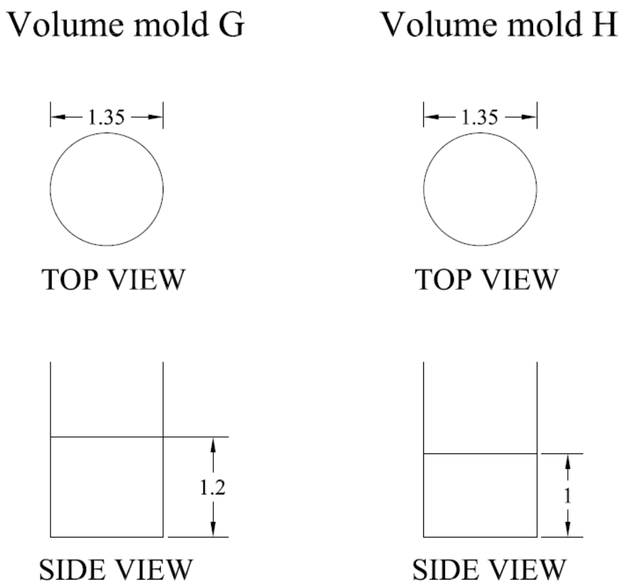


Figure A5 – Schematics for volume molds G and H
(All dimensions in inches and not to scale)

B. Appendix B: ASTM type Test Standard

Designation: X XXXX-XX

Work Item Number: WKXXXXXX
Date: 11/16/2015

Proposed Standard Test Method for Determining the Chloride-Ion Threshold for Corrosion of Reinforcing Steel in Concrete[1]

1. Scope

1.1 This test method covers the laboratory determination of the chloride-ion threshold concentration for corrosion of reinforcing steel in concrete.

1.2 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as the standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*[2]

A 615/A 615M Specification for Deformed and Plain Carbon-Steel Bars for Concrete Reinforcement

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[1] This test method is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.14 on Corrosion of Metals in Construction Materials.

Current edition approved XXX. XX, XXXX. Published XXX XXXX.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

C 125 Terminology Relating to Concrete and Concrete Aggregates

C 150/C 150M Specification for Portland Cement

C 185 Test Method for Air Content of Hydraulic Cement Mortar

C 192/C192M Practice for Making and Curing Concrete Test Specimens in the Laboratory

C 470/C 470M Specification for Molds for Forming Concrete Test Cylinders Vertically

C 511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes

C 876 Test Method for Corrosion Potentials of Uncoated Reinforcing Steel in Concrete

C 881/C 881M Specification for Epoxy-Resin-Base Bonding Systems for Concrete

C 1152/C 1152M Test Method for Acid-Soluble Chloride in Mortar and Concrete

C 1218/C 1218M Test Method for Water-Soluble Chloride in Mortar and Concrete

D 632 Specification for Sodium Chloride

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurement

G 59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements

G 109 Test Method for Determining Effects of Chemical Admixtures on Corrosion of Embedded Steel Reinforcement in Concrete Exposed to Chloride Environments

G 193 Standard Terminology and Acronyms Relating to Corrosion

L. Cáseres, A.A. Sagués, S.C. Kranc, R.E. Weyers, “In Situ Leaching Method for Determination of Chloride in Concrete Pore Water,” Cement and Concrete Research 36 (2006) 492-503. (Sagués and Weyers).

3. Terminology

3.1 *Definitions*—Refer to Terminologies C 125 and G 15 for definitions of terms used in this test method.

3.2 *Definitions of Terms Specific to this Standard:*

3.2.1 *chloride-ion threshold concentration*, *n*—mean acid-soluble chloride-ion content at the depth of reinforcement required to initiate and sustain corrosion (mass of chloride ion/mass of cement, %).

3.2.2 *corrosion initiation, n*—event at which the rate of metal dissolution increases and remains at a high level.

4. Summary of Test Method

4.1 A sufficient quantity of reinforcing steel to complete the test series is obtained from a single heat. The reinforcing steel is cut into sections, inspected for pre-existing corrosion, one end of each bar is drilled and tapped, then it is cleaned with solvent to remove drilling and cutting lubricants. The ends are prepared and placed horizontally in molds. Multiple test specimens are cast for each treatment of interest. A standard mortar mixture is used for the reference mixture, and the only alteration allowed to the standard mixture is the incorporation of the material being evaluated. Specimens are moist cured and then placed in a standard laboratory drying environment for preparations before the start of the test.

4.2 Each test specimen is exposed to cyclic ponding with a chloride solution and drying on a weekly basis. Multiple electrochemical measurements are taken on a regular basis, and corrosion initiation is indicated by a change in any, or all, of these measurements.

4.3 Once corrosion initiation occurs, the corroding specimen is removed from the cyclic ponding environment and a destructive examination of the specimen is performed. Powder samples are obtained from the specimen at the reinforcing level during the examination. The chloride-ion content of the powder sample is determined for each test specimen.

4.4 The mean and standard deviation for each treatment is calculated and reported along with visual observations.

5. Significance and Use

5.1 Corrosion of reinforcing steel in concrete is a complex process that depends on the materials used in the test, environmental conditions, specimen type, and test parameters. A complicating factor is that an induction time is part of the corrosion process. Many procedures used to accelerate corrosion electrochemically alter the surface of the steel and influence the test result. This test procedure was developed to standardize a laboratory procedure that produces chloride-ion threshold concentration values similar to those found in structures.

5.2 Because of the inherent variability in corrosion testing, it is necessary to characterize and compare the distribution of test data. This is accomplished by testing numerous specimens and reporting the mean and standard deviation for each treatment. Also, because of the variability inherent with reinforcing steel manufacturing processes, a set of control specimens is required for each test series. The chloride-ion threshold value determined from this procedure is thereby a relative comparison between data populations.

5.3 This test method is intended for use in evaluating the relative performance of corrosion protection systems such as reinforcing metals, chloride corrosion-inhibiting admixtures, and supplementary cementitious materials.

5.4 The specimen geometry and test procedure make this test inappropriate for evaluating the performance of barrier systems such as traffic membranes and epoxy-coated reinforcing steel.

5.5 The corrosion measurements produced by this test procedure are intended to indicate corrosion initiation in the test specimen and do not directly translate to the corrosion rates that occur in structures.

5.6 This test uses a standard mortar made with standard chloride-free sand to allow low cover depth to the steel. This reduces the complications associated with coarse aggregate, but increases the cementitious content of the mixture. Adjustments to the mixture proportions to incorporate alternate cementitious materials or additives must not alter the specified w/cm, the paste volume, nor the curing and storage conditions.

6. Apparatus

6.1 *Mixer*—Use a mortar mixer with a minimum total capacity of 85 L [3 ft^3] that is mechanically operated (see NOTE 1).

NOTE 1—An electric-driven motor is preferred in the laboratory to avoid noise and exhaust fumes.

6.2 *Potentiostat*—A potentiostat capable of varying potential at a constant scan rate and measuring the current is needed. A suitable device is described in Reference Test Method G 5.

6.3 *Voltmeter*—A voltmeter suitable for measuring the half-cell corrosion potential and the voltage across the 1-Ω resistors that is used to calculate the macrocell current. For the half-cell corrosion potential measurements, the voltmeter shall have ±3 % end-of-scale accuracy at the voltage ranges in use, and the input impedance shall be no less than 10 MΩ when operated at a full scale of 100 mV. The divisions on the scale used shall be such that a potential difference of 0.01 mV or less can be read without interpolation. (See NOTE 2)

NOTE 2—The precision of the voltmeter is defined by the macrocell requirement and not the half-cell requirements.

6.4 Silver Chloride Reference Electrode with Porous Ceramic Junction – Saturated potassium chloride electrolyte filling solution shall be used as the standard. (NOTE 3)

NOTE 3 – If a silver chloride reference electrode with an alternative electrolyte concentration is used, convert measurements to saturated electrolyte prior to reporting.

7. Materials

7.1 *Mortar Materials*

7.1 A standard mortar is used to provide a uniform chloride profile in this test. The standard mortar for this procedure is made with Specification C 150/C 150M Type 2 cement. The mortar sand and mixture proportions are the same as defined in Test Method C 109/C 109M. If it is necessary to deviate from the standard mortar, mixture proportions shall be adjusted on a volumetric basis, keeping the aggregate and paste volume constant. Example mixtures with equivalent paste volumes are provided in Table 1.

7.1.1 The volume of mortar required for each treatment is approximately 50 L [1.75 ft^3].

7.2 *Chemical Admixtures*

7.2.1 The only chemical admixture allowed in the mortar is a chemical admixture that is the subject of a test. The mixing water shall be adjusted to account for any water present in the quantity of admixture used.

7.2.2 Add the chemical admixture at the manufacturer recommended dose based on the mass of cementitious material in the mortar (see NOTE 4).

NOTE 4—The standard mortar used in this test contains more cement per unit volume than typical concrete. So, if the dose rate listed on the manufacturer's literature has units of admixture volume per unit volume of concrete (L/m^3 or $gal./yd^3$), calculate the equivalent admixture volume per mass of cement assuming the concrete contains 355 kg/m^3 [600 lb/yd^3] of cement.

7.3 *Reinforcing Steel*

7.3.1 Use reinforcing bars meeting the requirements of Specification A 615/A 615M. All bars in a test series shall come from the same heat.

7.3.2 Use No. 13 [No. 4] reinforcing bars 230 mm [9 in.] long. Only use sections without corrosion and clean them in xylene or hexane after cutting and machining operations. The mill scale is not removed.

7.3.3 Epoxy for coating mortar shall be an epoxy sealer of Type III, Grade 1, Class C in accordance with Specification C 881.

7.3.4 PVC ponding reservoir (Optional) that has 115-mm [4.5-in] inner-dimension by 25 mm (1 in.) high (min.).

7.3.5 *Resistors*—10-Ω (±5 %).

7.3.6 *Sodium Chloride*— Conforming to D 632 Specification for Sodium Chloride

7.3.7 *Salt Solution*—3 parts by mass sodium chloride to 97 parts by mass water

7.3.8 *Silicon Caulk*—As used in G 109

7.3.9 Specimen molds are 150-mm [6-in.] by 150-mm [6-in.] by 140 mm [5.5 in.] high. (NOTE 5) Wood molds shall be made non-adsorbent by coating the interior with a penetrating sealer such as tung oil, teak oil, polyurethane, or epoxy. Mold release agents such as silicone or Teflon-based products are acceptable. Do not coat forms with petroleum-based oils or form release oil. Petroleum-based products leave a residue on concrete surfaces that can reduce the uptake of the chloride-ion ponding solution.

8. Specimen Preparation

8.1 *Rebar*—The outside 50 mm [2 in.] from each end shall be protected as described in Sections 5 and 6 of Test Method G 109.A grade 316 stainless steel machine screw with nut shall be inserted in one end. This produces an uncoated test section 125 mm [5.0 in.] long with an area of approximately 5106 mm^2 [7.86 in.^{2}].

8.2 *Molds*—The mold shall be prepared inverted so that the base of the mold is the future ponding surface. The bottom of the mold shall include a non-absorptive insert to form the integral mortar dam. (NOTE 5) Three sets of holes are drilled in the specimen mold to insert a single reinforcing bar at the bottom level (test bar) and two on the top level (cathode bars), as cast. The clear distance from the ponding surface insert to the test bar is 13 mm [0.5 in.], and the distance between bars is 57 mm [2.25 in.] as indicated in FIG 1.

NOTE 5 – The specimen is cast inverted to minimize the influence of finishing, bleeding, and settlement on the ponded surface. Alternatively the base of the mold may be flat and a reservoir can be adhered to the ponding surface after curing.

NOTE 6—Tape or a sealer might be necessary to keep the mortar from coming out of the hole.

8.3 *Mixing*—Introduce the materials for each batch into the mixer in the following manner:

8.3.1 Place all the mixing water into the mixer.

8.3.2 Start the mixer while adding the sand to the mixer.

8.3.3 Add the cement into the mixer slowly.

8.3.4 Continue mixing for 5 min.

8.4 *Casting and Curing*

8.4.1 Cast at least ten specimens for each treatment in a test series.

8.4.1.1 Cast two blank cylinder specimens 7.6 cm diameter by 7.6 cm high, to be used for pH determination according to method by Sagués and Weyers.

8.4.2 Fill each mold in three layers and consolidated according to Practice C 192/C 192M. Cover the specimens with moist burlap and plastic. Remove the molds at one-day age. Moist cure the specimens in a room meeting the requirements of Specification C 511 for 27 additional days.

8.4.3 Record the following properties of the plastic mortar in accordance with the referenced standard:

8.4.3.1 *Test Method C 109 /C 109M*—25-drop flow.

8.4.3.2 *Test Method C 185*—Mass per 400 mL of mortar.

8.4.3.3 *Test Method C 185*—Air content.

8.4.4 Record the following properties of the hardened mortar in accordance with the referenced standard:

8.4.4.1 *Test Method C 109/C109M* – Cube compressive strength at 28 days age.

8.4.4.2 *Test Method C 1760* - Bulk resistivity at 28 days age.

8.4.4.3 Test Method C 1152/C 1152M and Test Method C 1218/C 1218M – Initial chloride content (after 28 days of moist curing)

8.4.4.4 At 14 days of curing drill 3 other holes equally spaced around the center that are all 3.6 mm in diameter and 35 mm deep as in Sagues and Weyers reference.

8.4.4.4.1 Attach acrylic washers, fill with boiled deionized water and seal with rubber stoppers.

8.4.4.4.2 Return to wet curing and keep in wet curing until five of ten specimens undergoes corrosion.

8.4.4.3 Measure pH using calibrated microelectrodes as noted in Sagués and Weyers.

8.5 *Preparations for Ponding*

8.5.1 Remove the specimens from moist curing at 28-days age. Place the specimen in a standard drying environment (23 ± 2°C [73 ± 3ºF], 50 % RH ± 4 %) for the next 14 days.

8.5.2 Determine the initial chloride content from three retained mortar samples from each test series in accordance with Test Method C 1152/C 1152M and Test Method C 1218/C 1218M. Calculate and report the mean initial chloride content for each treatment in a test series.

8.5.3 If cast without an integral mortar dam, adhere a 114-mm [4.5-in.] by 114-mm [4.5-in.] by 25-mm [1-in.] (min) high ponding reservoir with silicone caulk (see FIG 1) after 28-day age. This reservoir is centered on the top of the specimen.

8.5.4 Coat the perimeter and the top of the specimen outside of the reservoir using an epoxy-based paint.

8.5.5 Attach a 10-Ω (1% precision) resistor between the top and bottom rebars so that macrocell current can be measured.

9. Procedure

9.1 Each specimen is treated individually in this test procedure. When corrosion testing is complete for all specimens in a treatment, the statistics for each treatment are calculated and reported.

9.2 *Ponding*

9.2.1 Place specimens on spacers or an open gridded shelf so there is at least 2.5 cm [1 in.] of air space below the specimen.

9.2.2 Maintain specimens at ambient conditions of 23 ± 4°C [73 ± 7ºF].

9.2.3 Fill the ponding reservoir with 5% sodium chloride on a weekly schedule consisting of four days wet and three days dry. Take care to minimize any splashing of salt solution down the sides and onto the spacers to avoid chloride ingress through the bottom of the specimen.

9.3 *Electrochemical Measurements*

9.3.1 Determine the initial half-cell corrosion potential, and macrocell current during the first wet cycle after at least 24 h of ponding. Thereafter measure and record the half-cell corrosion potential and macrocell current for each weekly cycle on the last day of ponding.

9.3.2 Determine the corrosion potential of the top bar in accordance with Test Method C 876.

NOTE 7—The half-cell potential is determined by attaching the common (black) terminal of the voltmeter to the half-cell electrode and the active (red) terminal of the voltmeter to the top rebar of the mortar specimen.

9.3.3 The macrocell current is determined by attaching the common (black) terminal of a voltmeter to the bottom bars and the active (red) terminal of the voltmeter to the top bar across the 10-Ω resistor. The macrocell current, in amperes, is the voltage drop across the resistor, in volts, divided by the resistance (10 Ω).

9.3.4 – Conduct a linear polarization scan during the first ponding cycle, upon corrosion initiation during the wet cycle after at least 24 hours of ponding. Additional intermediate scans may be conducted, but are not required.

9.3.4.1 The results of the potentiostat scan will yield a plot of raw data that typically looks like FIG 2.

9.3.4.2 Polarization resistance is measured at the end of the first cycle then right before removal of the specimen, when macrocell data indicates corrosion has initiated, or if there is a decrease in the corrosion potential by more than 100 mV and it is more negative than --230 mV vs. saturated silver chloride electrode at 25 degrees C. The polarization resistance is calculated from the slope of volts versus current over the voltage range of E_{corr} ± 10 mV. This will yield a slope having the units of V/amps or ohms unless the rebar area has been already taken into account. To find the actual polarization resistance, the slope of the voltage-versus-current plot needs to be multiplied by the rebar area, which is 51.1 cm^2 [7.86 $in.^2$] (see 7.3.3). The polarization resistance will be in units of Ω-cm^2. Figure 3 illustrates the line fit over E_{corr} ± 10 mV for the data plotted in FIG 2.

9.3.4.3 If the output from the potentiostat and its software is reported in current density (amp/cm^2) as opposed to current (amps) and assuming that the 51.1-cm^2 [7.86-$in.^2$] area of the rebar is the input used to calculate the current density, then the polarization resistance in units of Ω-cm^2 is the slope of E_{corr} ± 10 mV versus current density. In such a case, the rebar area is already taken into account. The same data presented in FIGS 2 and 3 is plotted in FIG 4 in terms of current density versus potential. FIG 4 illustrates the slope of the curve over E_{corr} ± 10 mV is the polarization resistance in units of Ω-cm^2.

9.3.5 Samples are defined to be corroding when the macrocell, half-cell, or polarization resistance measurements show a continuous change in properties over two consecutive weeks of testing.

NOTE 8—Typically, all three measurements will indicate corrosion initiation at the same time. Typical measurements indicating corrosion activity: macrocell current (I_c) > 1 μA and half-cell potential (E_c) < –-230-mV SCE,.

9.3.6 When any of the weekly electrochemical measurements indicate corrosion activity in a specimen, obtain a complete set of measurements including polarization resistance, and continue ponding for one week. If any of the electrochemical measurements indicate corrosion activity after the second continuous week of testing, the specimen is removed from the ponding cycle and an autopsy is performed.

9.3.6.1 Obtain the polarization resistance in accordance with Test Method G 59 and the following modifications. Place the counter electrode and reference electrode in the reservoir. Adjust the potential over the range ±20 mV from the free corrosion potential. The scan rate shall be 0.167 mV/s. The polarization resistance, R_p, is calculated as the change in potential divided by the change in current over the range ±10 mV from the free corrosion potential.

9.4 *Autopsy*

9.4.1 Obtain a 15-20-g [0.35-oz] powder sample for chloride-ion analysis according to Test Method C 1152/C 1152M and Test Method C 1218/C 1218M from along-side the top reinforcing bar (see schematic in FIG 1). This sample shall be taken within 1 day of ending the test. Take care to collect the powder sample only from the region under the reservoir. Obtain the powder sample by either drilling into the side with a 13-mm [½-in.] diameter drill bit and collecting the powder. Dry and pulverize the powder sample to pass the No. 20 sieve in accordance with Test Method C 1152/C 1152M.

9.4.2 Lay the specimen on its side and cut the specimen with a water-cooled masonry saw parallel to the top reinforcing bar on opposite sides as shown in FIG. 5. Tap the top edge of the specimen to promote a crack between the saw cuts around the reinforcing bar. Remove the top reinforcing bar and any mortar adhered to the bar. .

9.4.3 Report the location of visible corrosion and estimate the percentage of corroded area on the rebar as described in Test Method G 109 (NOTE 4).

NOTE 9—It is recommended to record visual observations using photographs of the reinforcing bars that are taken within 2 h of removal from the test specimen.

9.5 Obtain the chloride content of the powder sample in accordance with Test Method C 1152/C 1152M.

9.6 If acid-soluble chloride profiles are desired, dry grind the specimen in layers of sufficient thickness to obtain a minimum 10 g [0.35 oz] of powder for each layer. The chloride profile shall include the ponded surface and extend to a depth sufficient to reach the initial chloride content. The chloride content obtained for the layers spanning the reinforcing bar trace (12- to 25-mm [0. 5- to 1.0-in.] depth) are averaged for the chloride threshold determination.

10. Calculation

10.1 Calculate the chloride threshold of each specimen as the net chloride content obtained from the reinforcing bar trace after subtraction of the initial chloride content, if any.

11. Report

11.1 *Reporting Requirements*

11.1.1 The name of the laboratory and individual for responsible supervising the work;

11.1.2 The mixing date;

11.1.3 The date cyclic ponding began;

11.1.4 The unit weight of the mortar as determined by Test Method C 185;

11.1.5 The air content of the mortar as determined by Test Method C 185;

11.1.6 The 25-drop flow of the mortar as determined by Test Method C 109/C 109M;

11.1.7 The 28-day compressive strength of the mortar by Test Method C 109/C 109M;

11.1.8 The bulk resistivity of the mortar by Test Method C 1760;

11.1.7 The mortar materials and quantity used, cement brand and type, and standard sand manufacturer;

11.1.8 The manufacturer, product name, and the quantity of material added to the mortar for each product tested;

11.1.9 Any deviations from the procedures described in this test method;

11.1.10 A table providing the individual chloride content values obtained initially, and upon corrosion initiation, with the calculated mean, and standard deviation values for each test series;

11.1.11 A plot showing the individual specimen corrosion potentials as a function of time;

11.1.12 A plot showing the individual specimen macrocell currents as a function of time;

11.1.13 A table showing the individual specimen polarization resistance results from the initial and final measurements ;

11.1.14 The individual specimen, average, and standard deviation of corroded bar areas at corrosion initiation; and

12. Precision and Bias

12.1 *Precision*—The repeatability standard deviation and reproducibility of this test method are being determined and will be available on or before XXXX, YYYY.

12.2 *Bias*—No information can be presented on the bias of this procedure because the chloride threshold value is determined by this test, and an accepted reference material is not available.

13. Keywords

13.1 chloride ion; chloride threshold; concrete; corrosion; corrosion initiation

TABLE 1*a* Example Mixtures with Equivalent Paste Volume—SI Units

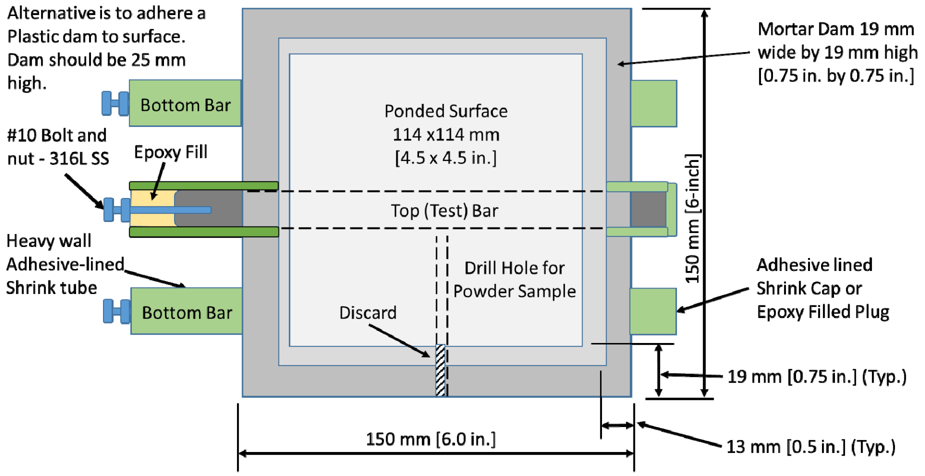
	Specific Gravity	**Standard w/cm = 0.485**	**Lower w/c w/cm = 0.40**	**20 % Ash w/cm = 0.485**	**8 % SF w/cm = 0.485**	**35 % Slag w/cm = 0.485**

Cement, kg/m^3	3.15	500	559	392	456	321
Fly ash, kg/m^3	2.40	---	---	98	---	---
Silica fume, kg/m^3	2.20	---	---	---	40	---
Slag, kg/m^3	2.90	---	---	---	---	173
Water, L/m^3	1.00	242	224	238	240	240
Sand, kg/m^3	2.60	1375	1375	1375	1375	1375
Air Content, %	---	7.0	7.0	7.0	7.0	7.0

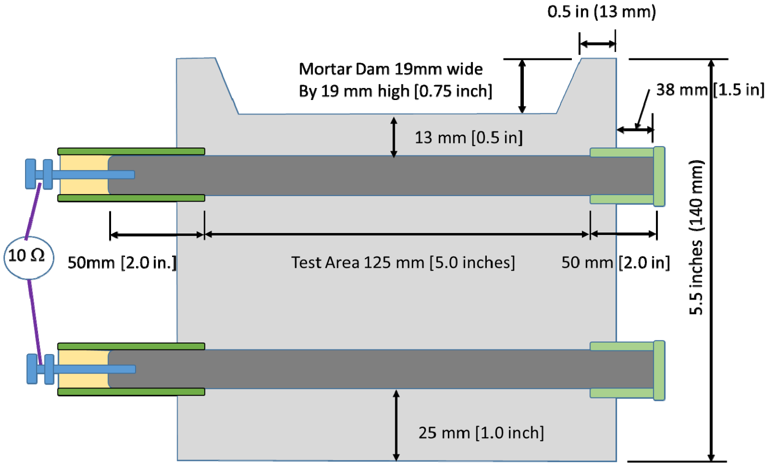
TABLE 1b Example Mixtures with Equivalent Paste Volume—U.S. Customary Units

	Specific Gravity	**Standard w/cm = 0.485**	**Lower w/c w/cm = 0.40**	**20 % Ash w/cm = 0.485**	**8 % SF w/cm = 0.485**	**35 % Slag w/cm = 0.485**
Cement, lb/yd^3	3.15	843	942	661	769	541
Fly ash, lb/yd^3	2.40	---	---	165	---	---
Silica fume, lb/yd^3	2.20	---	---	---	67	---
Slag, lb/yd^3	2.90	---	---	---	---	292
Water, lb/yd^3	1.00	409	377	401	405	404
Sand, lb/yd^3	2.60	2318	2318	2318	2318	2318
Air Content, %	---	7.0	7.0	7.0	7.0	7.0

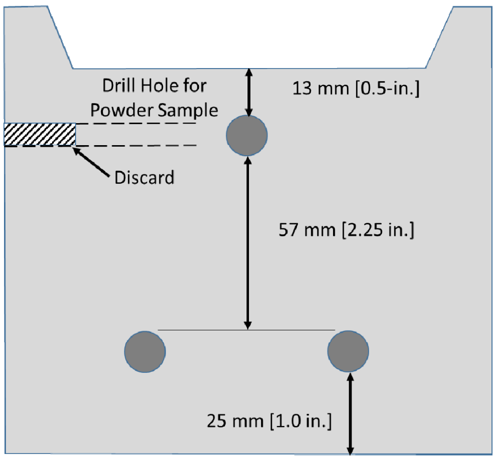
15mm



Top View Exposed to Chloride Solution Showing Top Bar End Protection



Longitudinal Section



Transverse Section

FIG 1 Sketch of Test Specimen

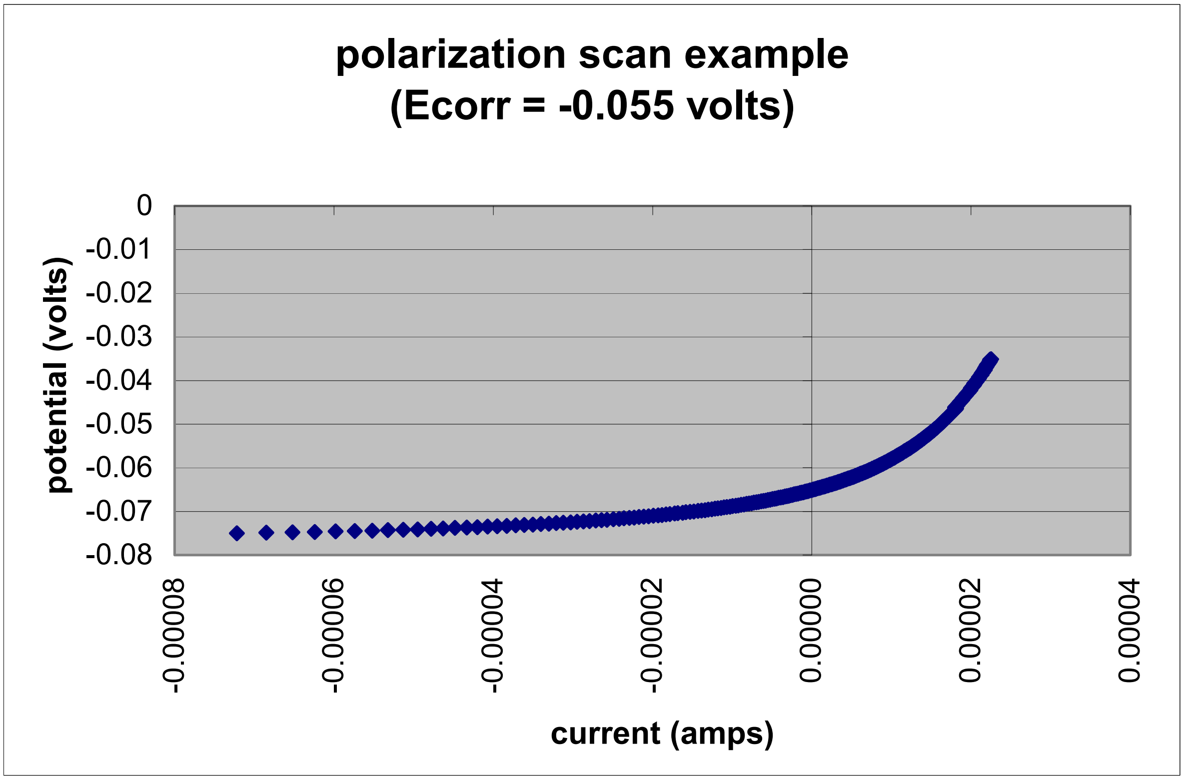


FIG 2 Representative Raw Data from a Polarization Scan

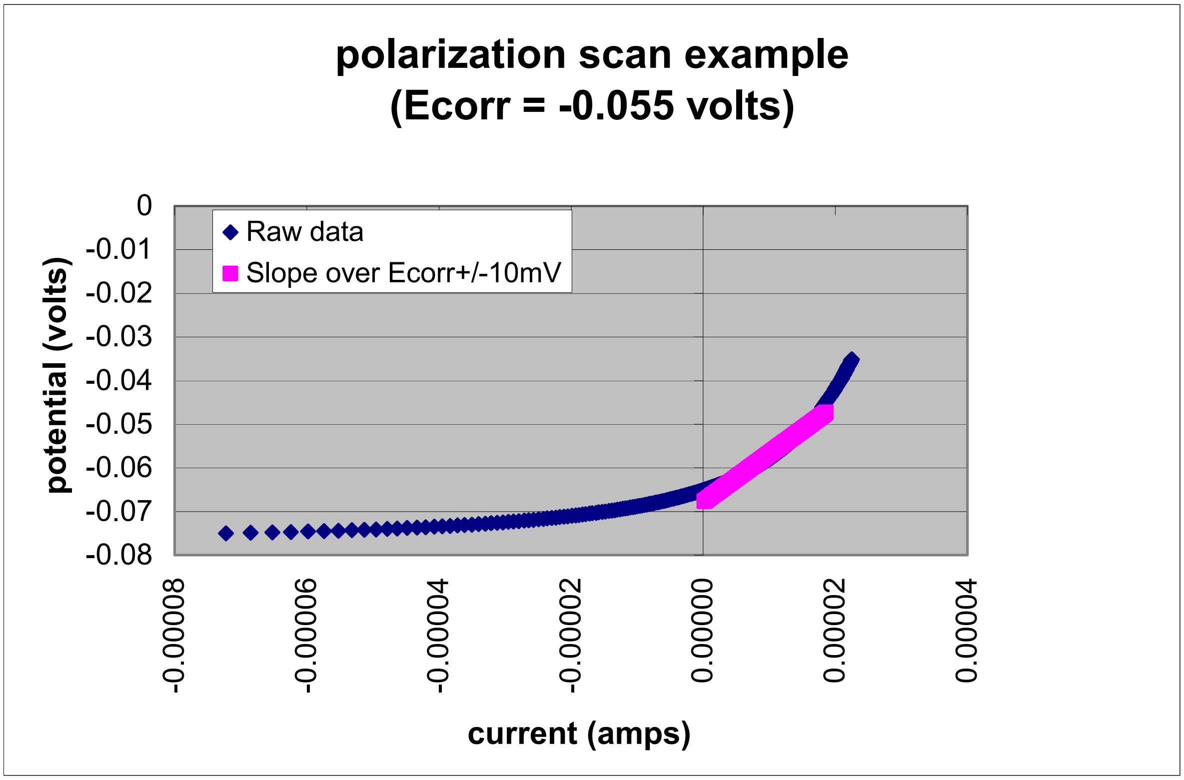


FIG 3 Based on the Raw Data Shown in FIG 4, a Line Fit Over E_{corr} ±10 mV Yields a Slope Equal to 1106 Ω; the Polarization Resistance (*Rp*) Equals the Slope Times the Rebar Area, for example, *Rp* = 1106 Ω * 47 cm^2 = 51997 Ω cm^2 = 51.997 kΩ cm^2

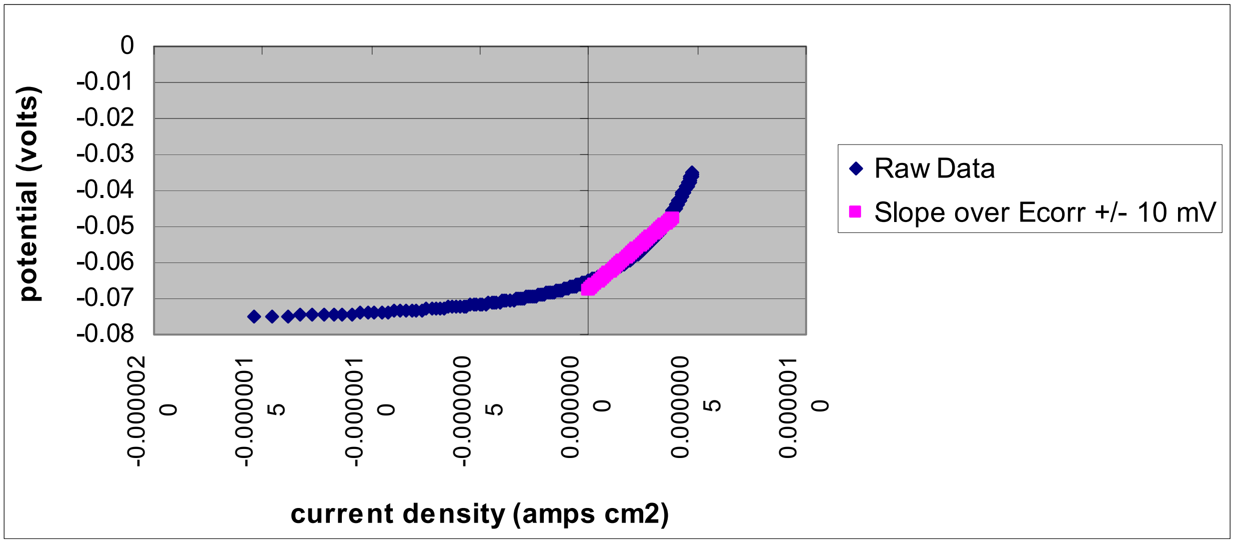


FIG 4 An Alternative Polarization Scan Takes the Rebar Area into Account and Presents the Raw Data as Current Density Versus Potential; Based on the Raw Data Shown, a Line Fit over E_{corr} ± 10 mV Yields a Slope Equal to 51567 Ω cm^2; the Polarization Resistance (*Rp*) Equals this Slope and No Further Calculations Are Needed, for this example, *Rp* = 51567 Ω cm^2 = 51.567 kΩ cm^2

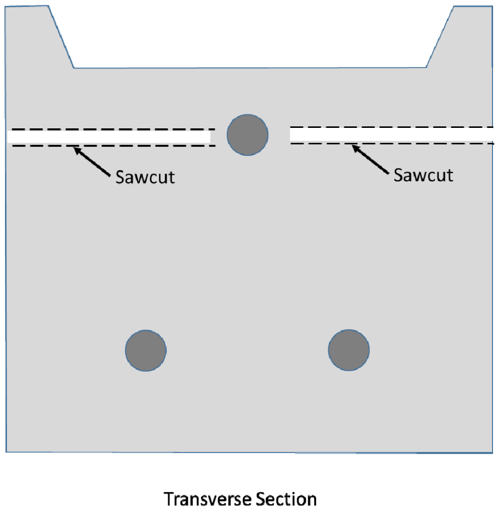


FIG 5 Illustration of Saw Cuts to Break Open Corroding Specimen and Free the Rebar for Visual Examination

Modification in appendix ASTM Type

1- Mixer [6.1]

   Instead of a 8.5 L mixer , a 12 Qt (11.4 L) laboratory mixer was used.

2- Mortar Materials [7.1]

   Standard mortar has not been used as recommended in the procedure. Water to cement ration(w/c) changed to 0.42, with three different s/c ratios of 1.375, 2.0, and 2.75 has been used.

3- Reinforcing Steel [7.3]

   Instead of steel No 4, steel No 5 was used to be consistent with OC_{crit}.

4- Resistors [7.3.5]

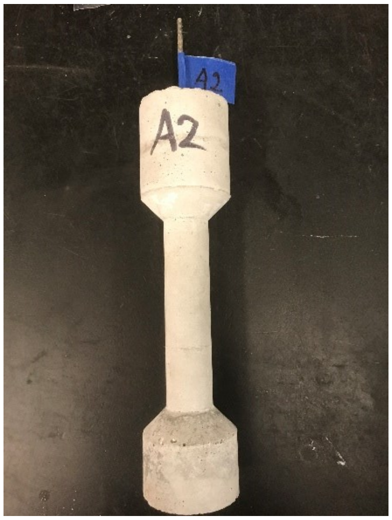
   Instead of 1-Ω (±5 %), 10 Ω resistors were used.

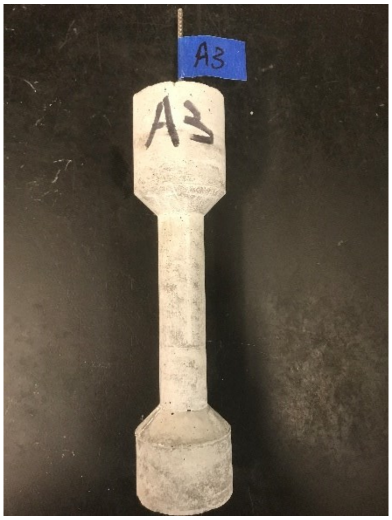
5- The proposed procedure to collect mortar samples was modified

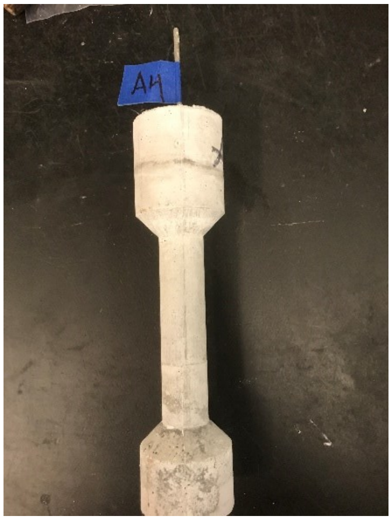
   After drying at room temperature, samples were cut at the mid-level of top steel bar using a water-cooled concrete saw. Mortar cover above the top steel bar with the trace of the steel bar was removed for chloride sampling. Procedure is described in detail in the report.

C. Appendix C: Sample Pictures

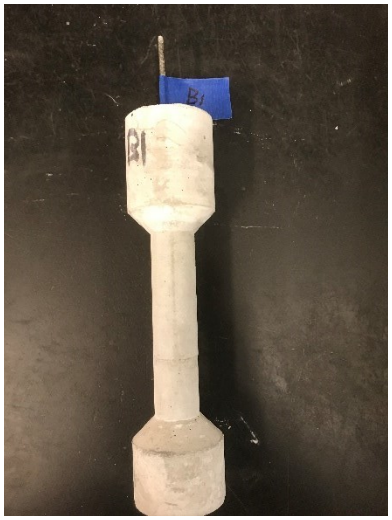

A1
OSU

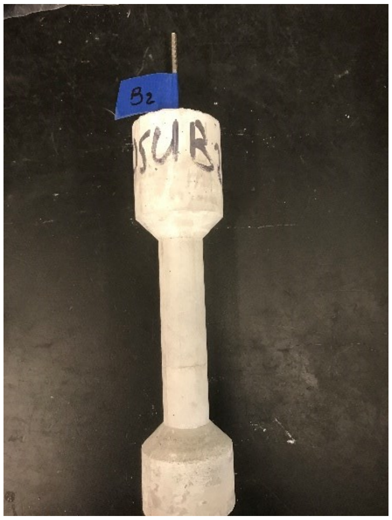

A2
A2


A3
A3

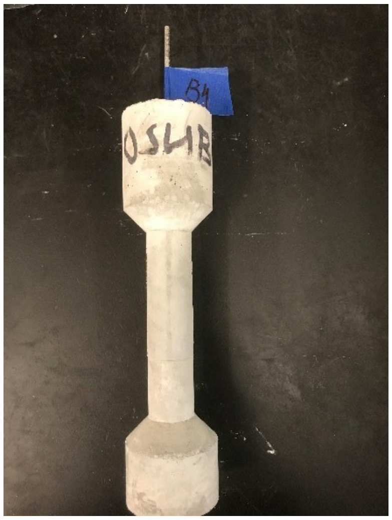

A4


A5
A5


B1
B1


B2
OSU B


B3
B3


B4
OSUB

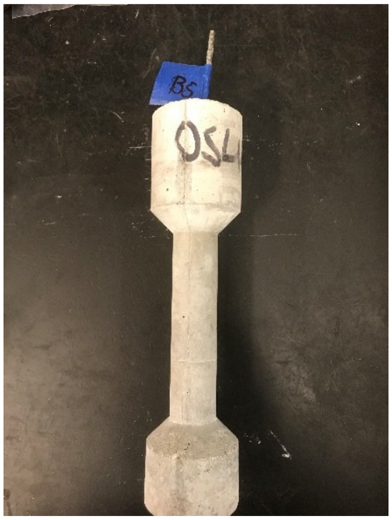
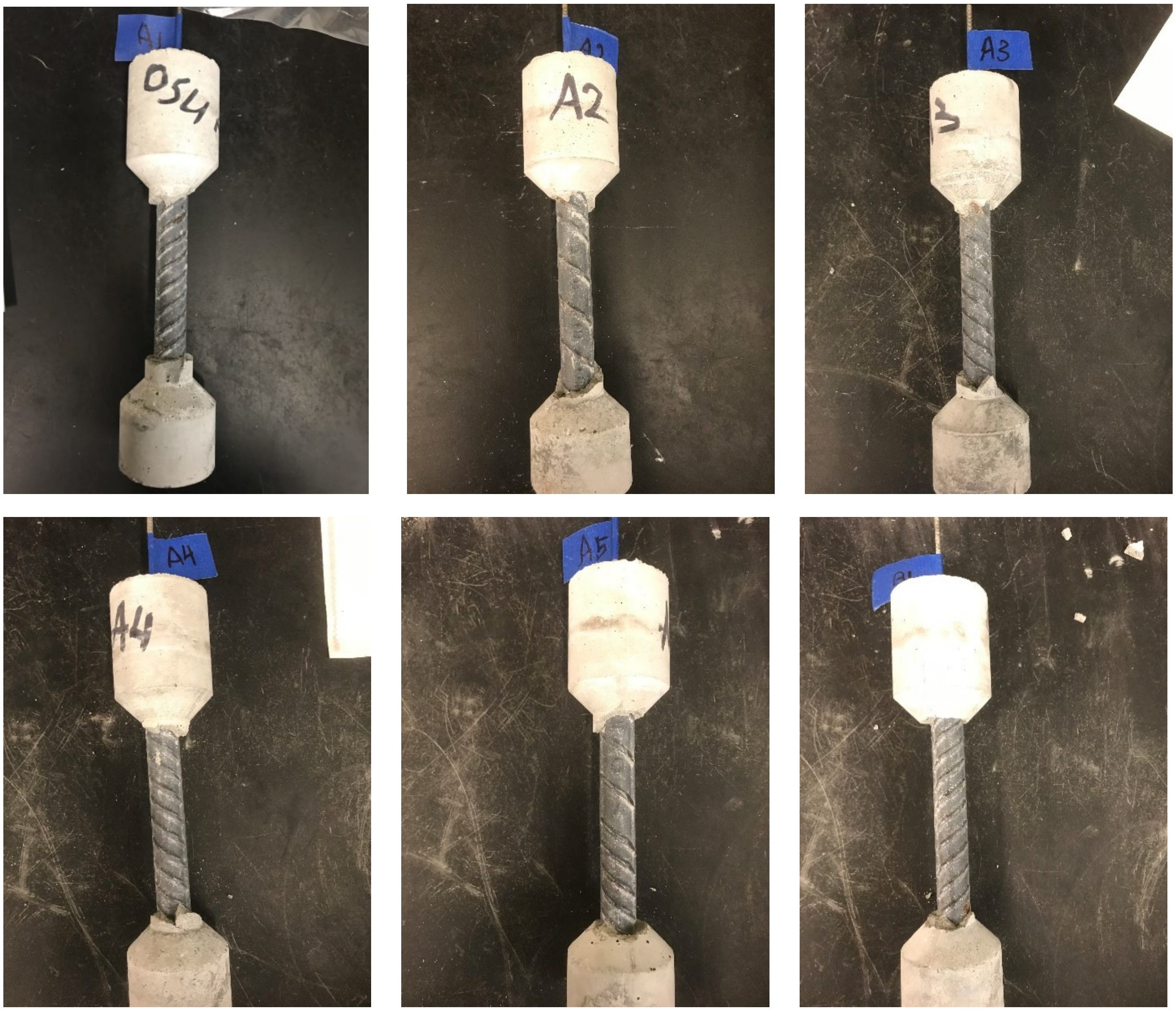


Figure 1 OCcrit specimens before sampling (UMKC)



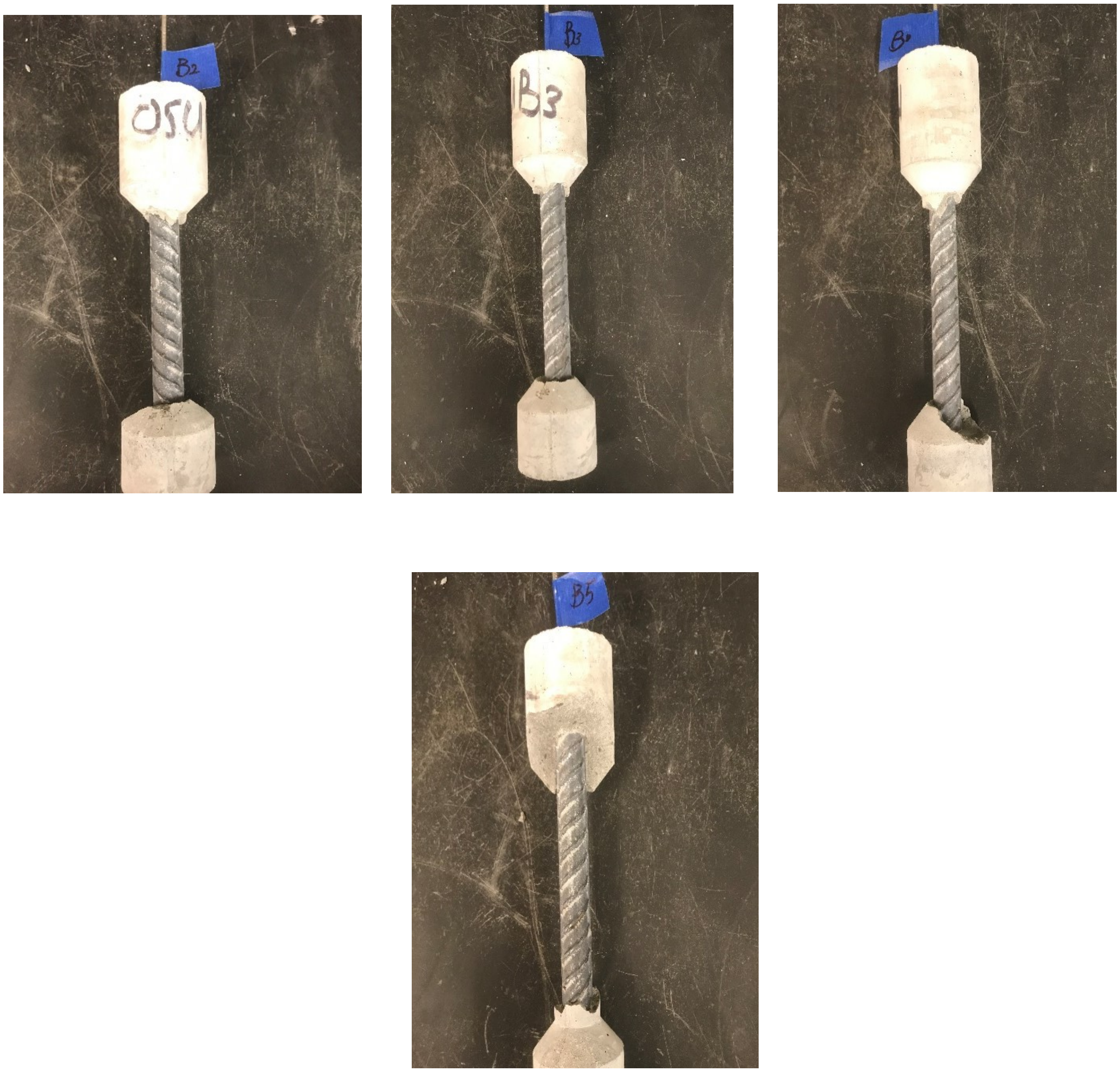
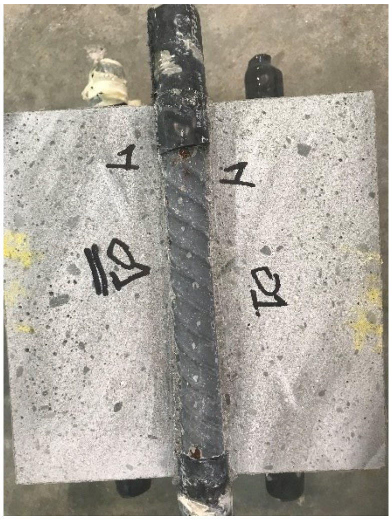


Figure 2 OCcrit specimens after sampling (UMKC)

Figure 3 ASTM type top cap of specimens (s/c of 1.375, UMKC))


1
1


2
2


3
3


4
4
4


5
5
5


6
6


7
7


Raychem
8
8

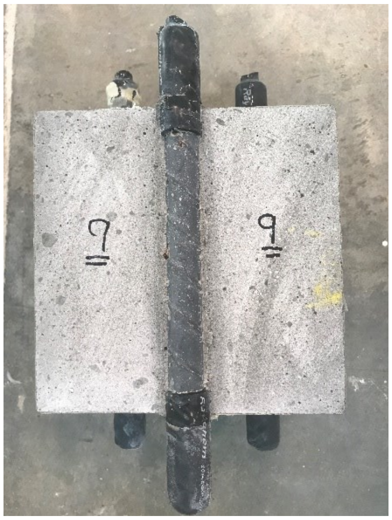

9
9

Figure 4 ASTM type steel-mortar interface (s/c of 1.375, UMKC)

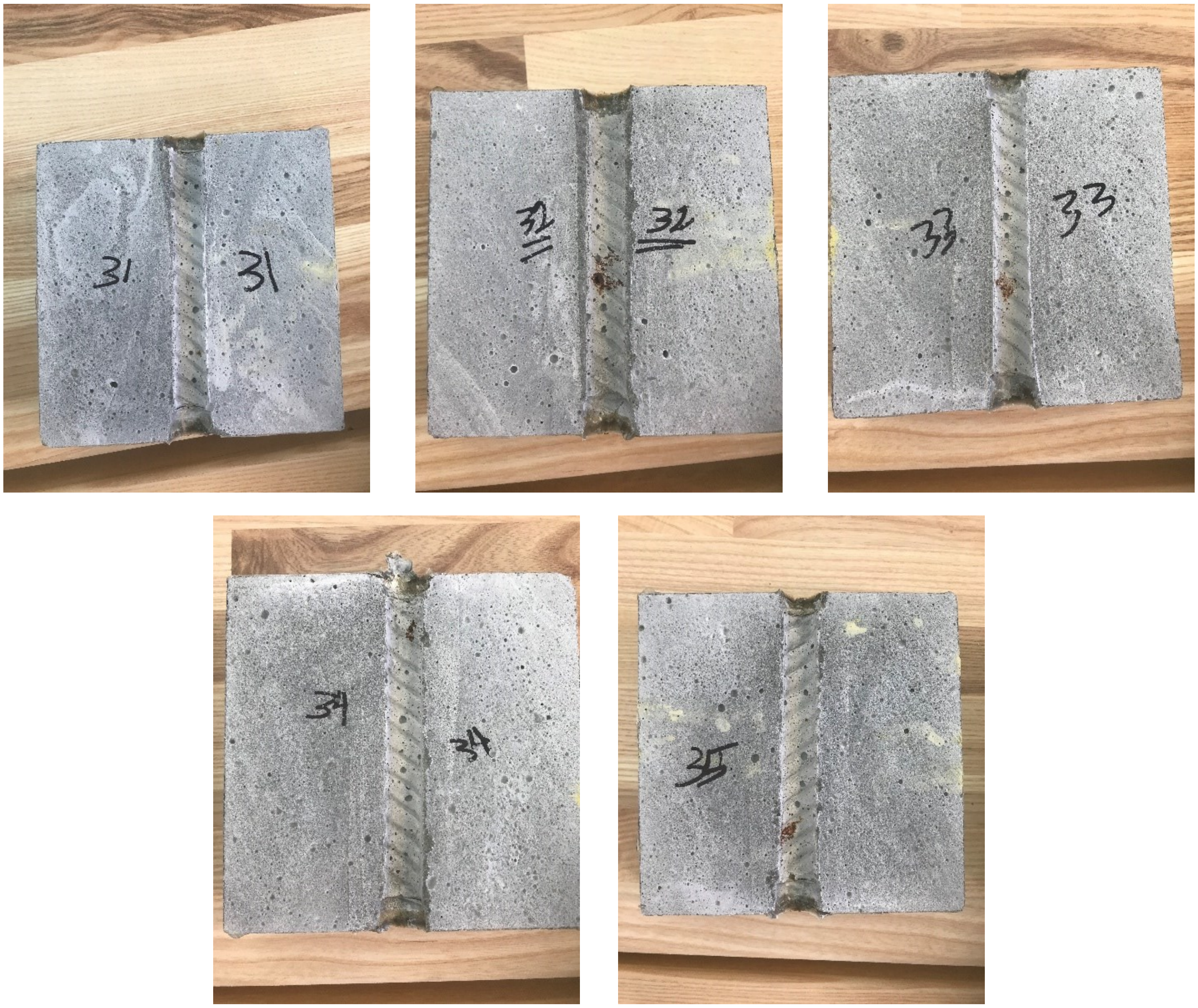


Figure 5 ASTM type top cap of specimens (s/c of 2.0, UMKC)

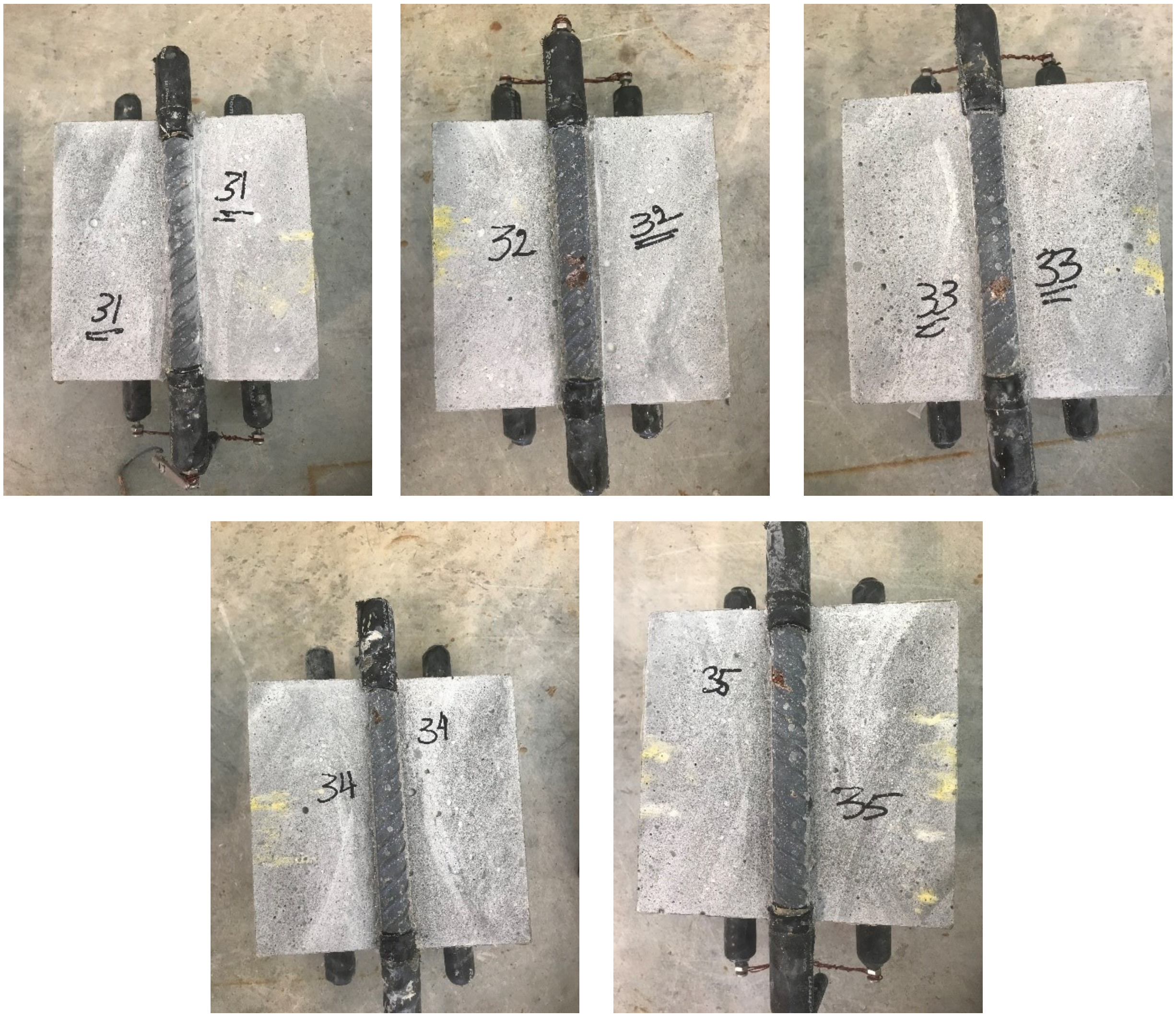
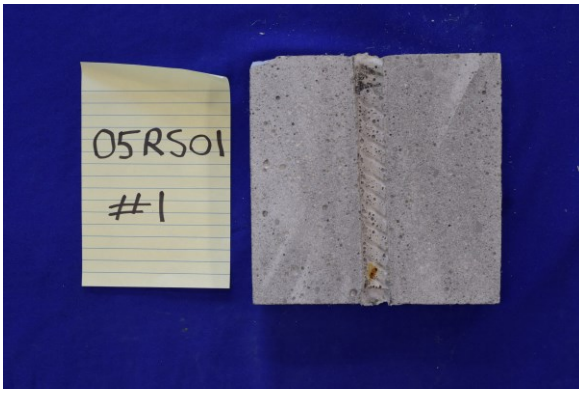
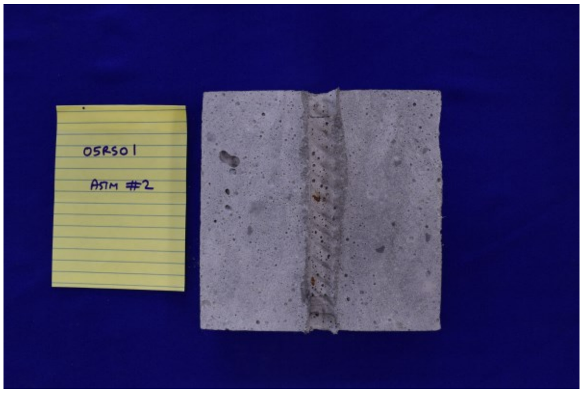


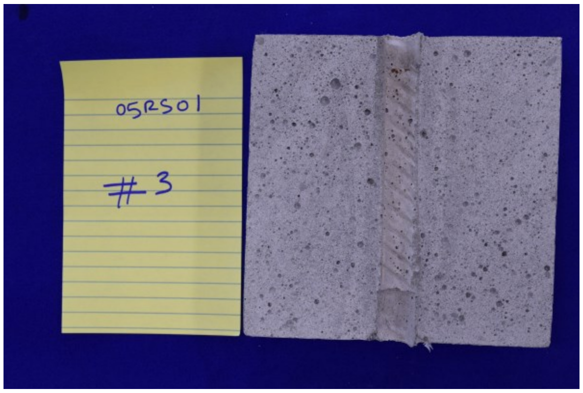
Figure 6 ASTM type steel-mortar interface (s/c of 2.0, UMKC)

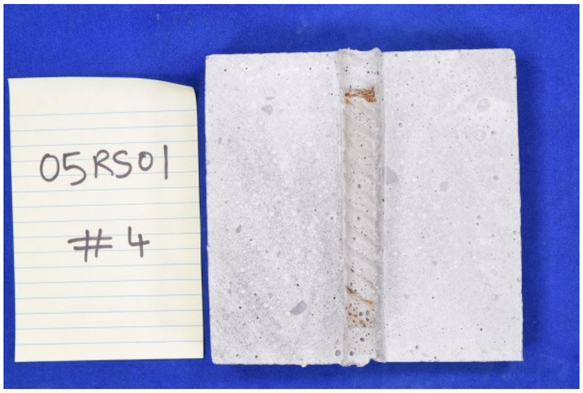
Figure 7 ASTM type top cap of specimens (s/c of 2.75, UMKC)

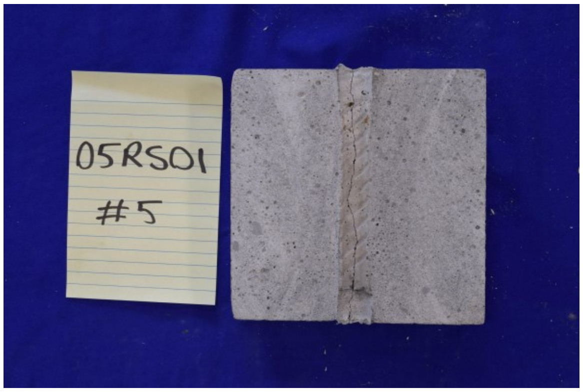
Figure 8 ASTM type steel-mortar interface (s/c of 2.75, UMKC)

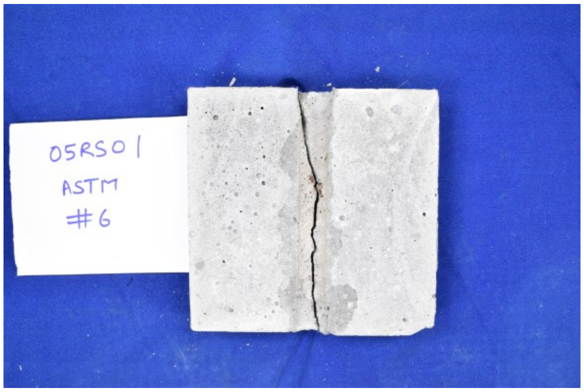

05RS01
#1


05RS01
ASTM #2


05RS01
#3


05RS01
#4


05RS01
#5


05RS01
ASTM
#6

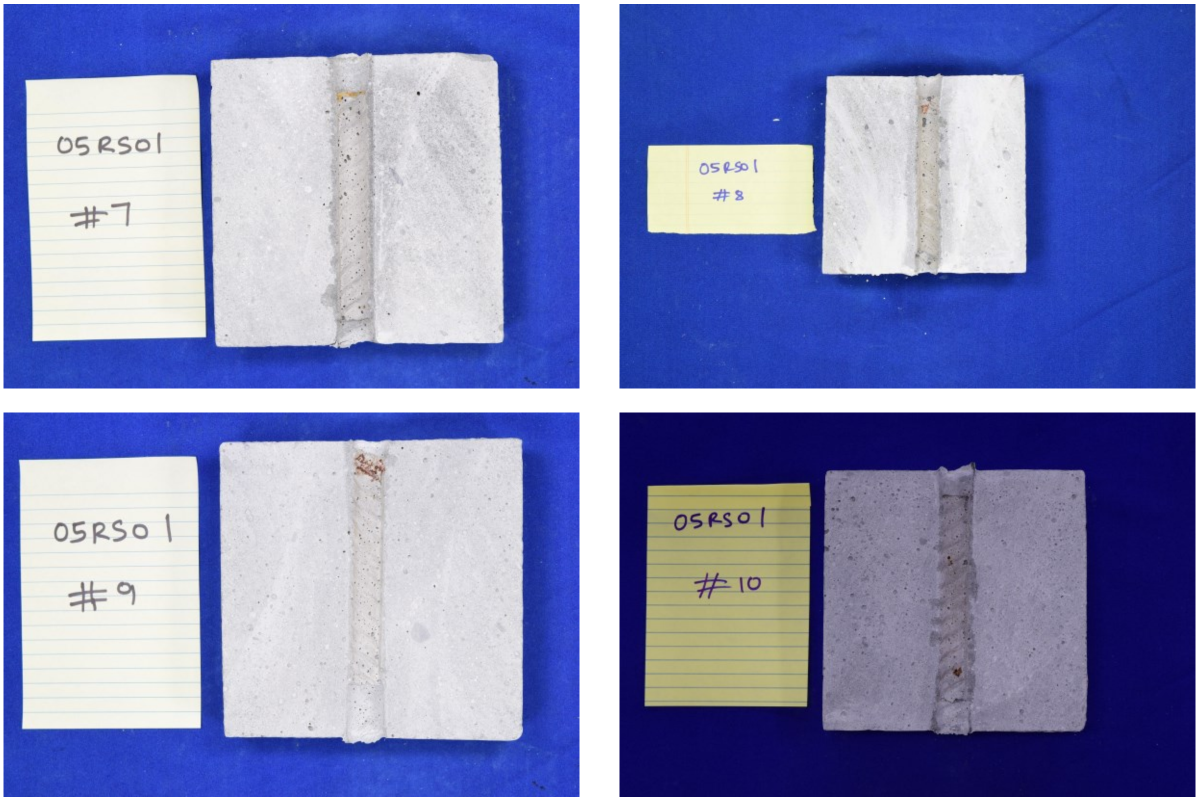
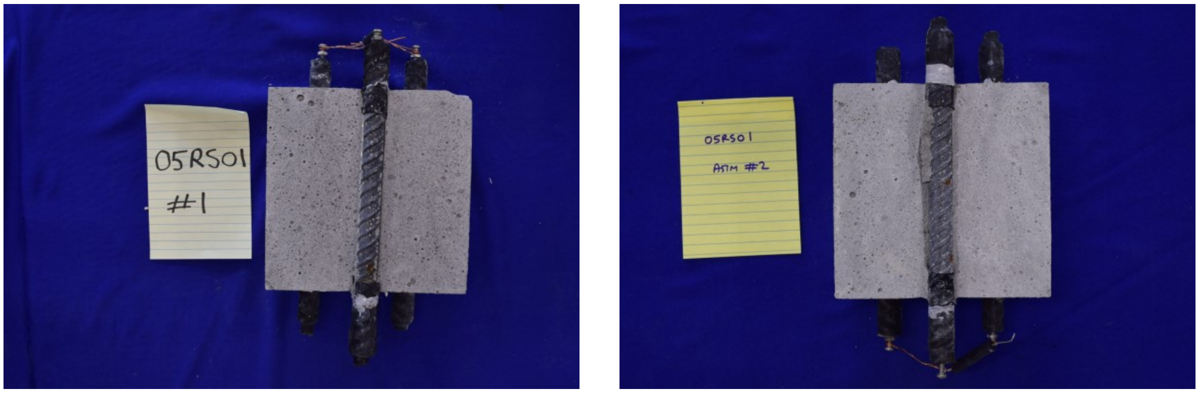


Figure 9 ASTM type top cap of specimens (s/c of 2.0, CTL)



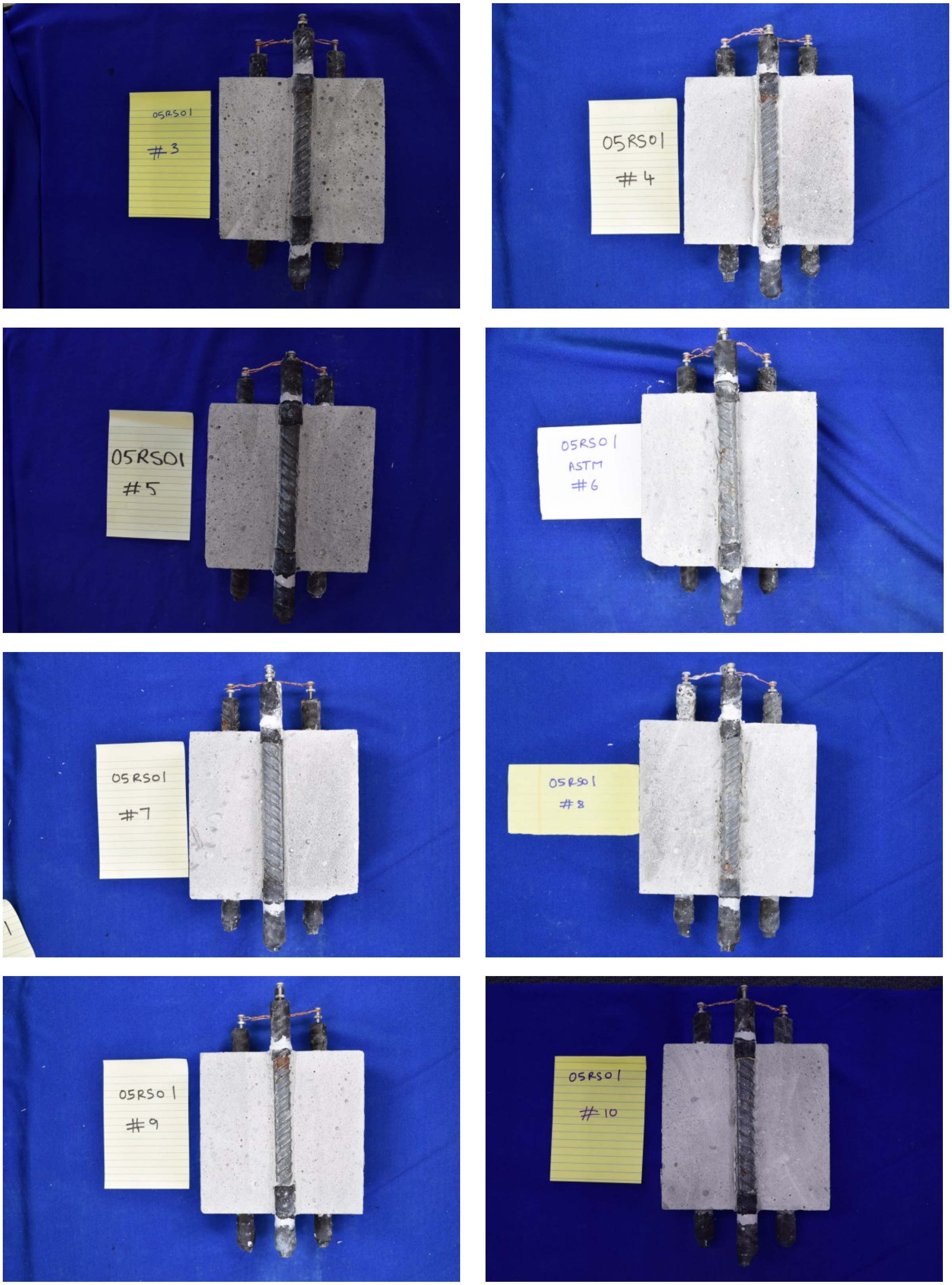


Figure 10 ASTM type steel-mortar interface of specimens (s/c of 2.0, CTL)

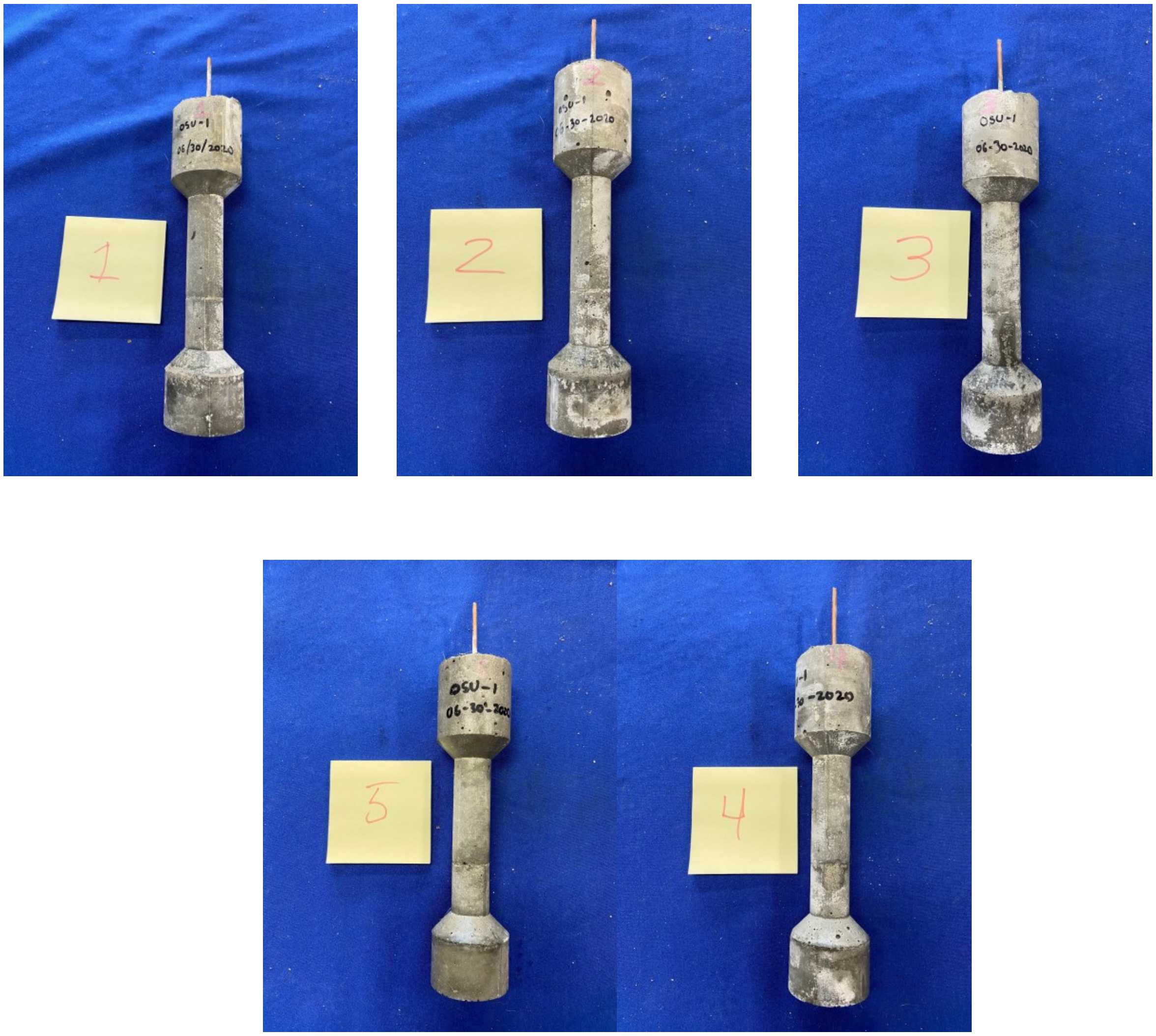


Figure 11 OCcrit specimens before testing (CTL)


1
1
OSU-1
06/30/2020


2
2
06-30-2020


3
OSU-1


4
OSU-1
30-2020


5
06-30-2020


6


7


8


9

Figure 12 OCcrit specimens after sampling (CTL)

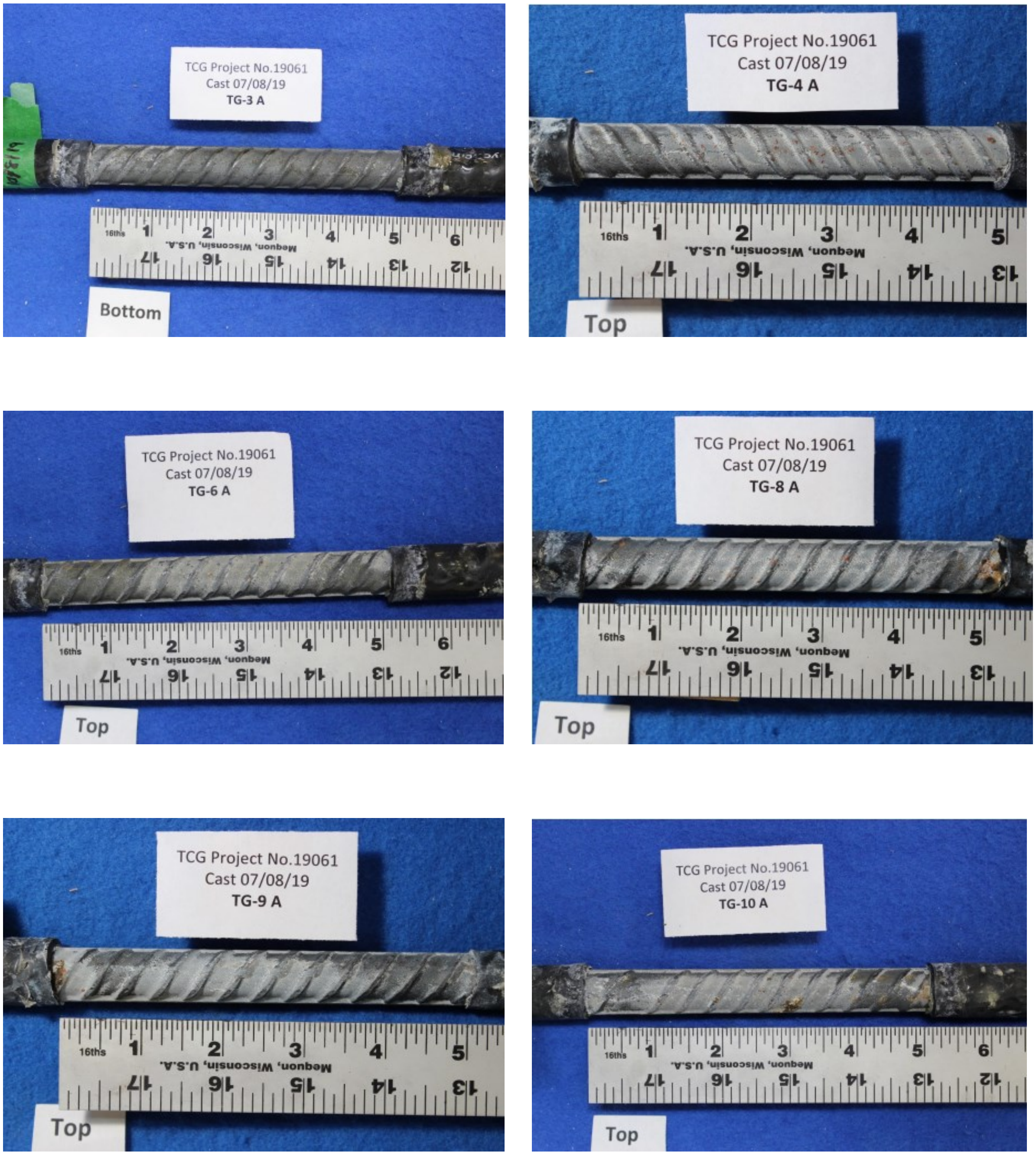
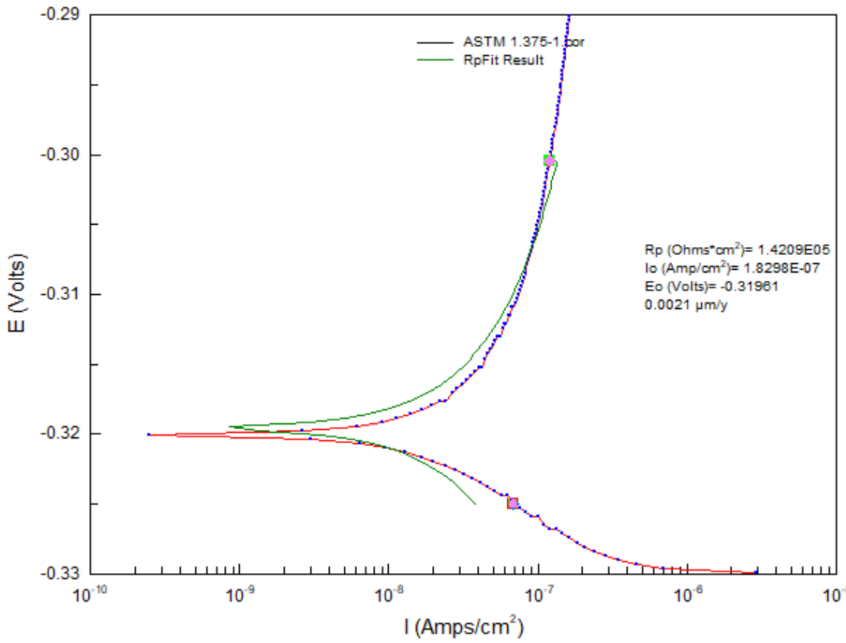
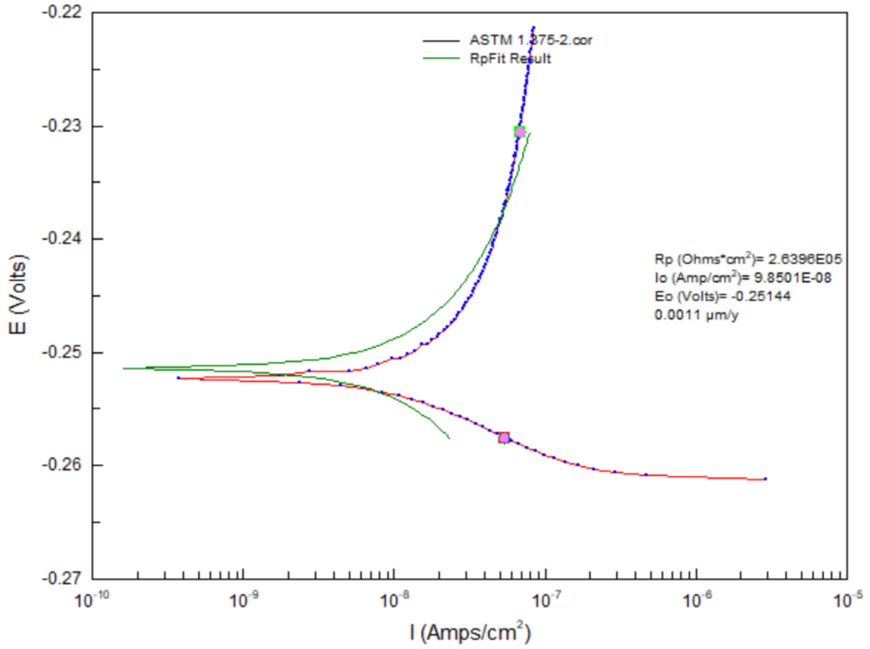
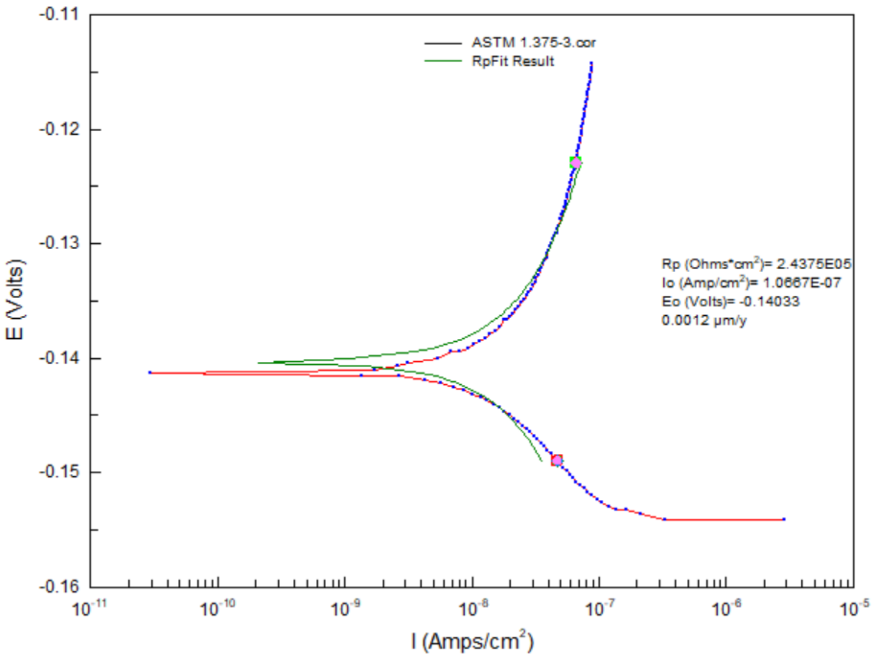


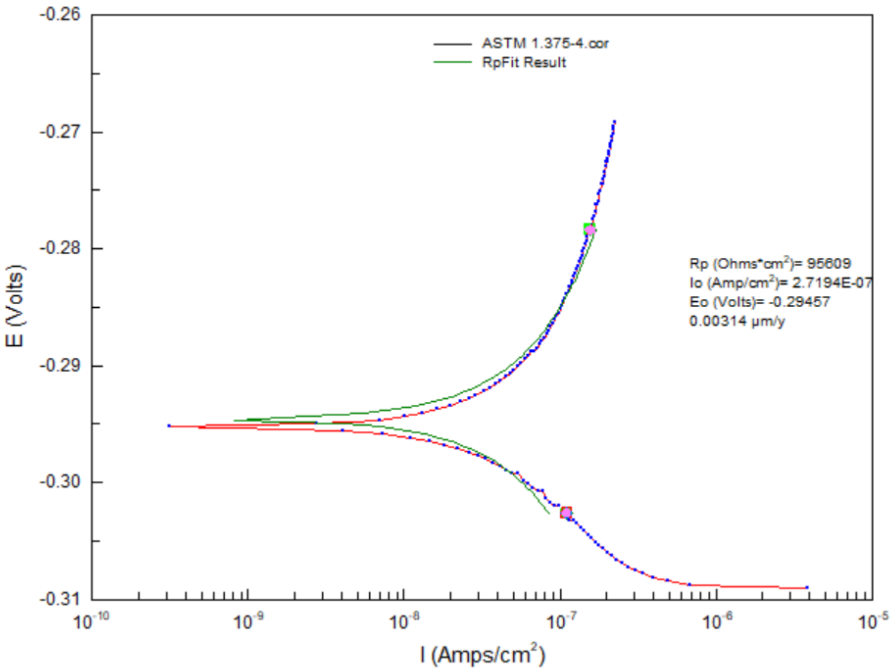
Figure 13 ASTM type steel specimens (s/c of 1.375, Tourney)

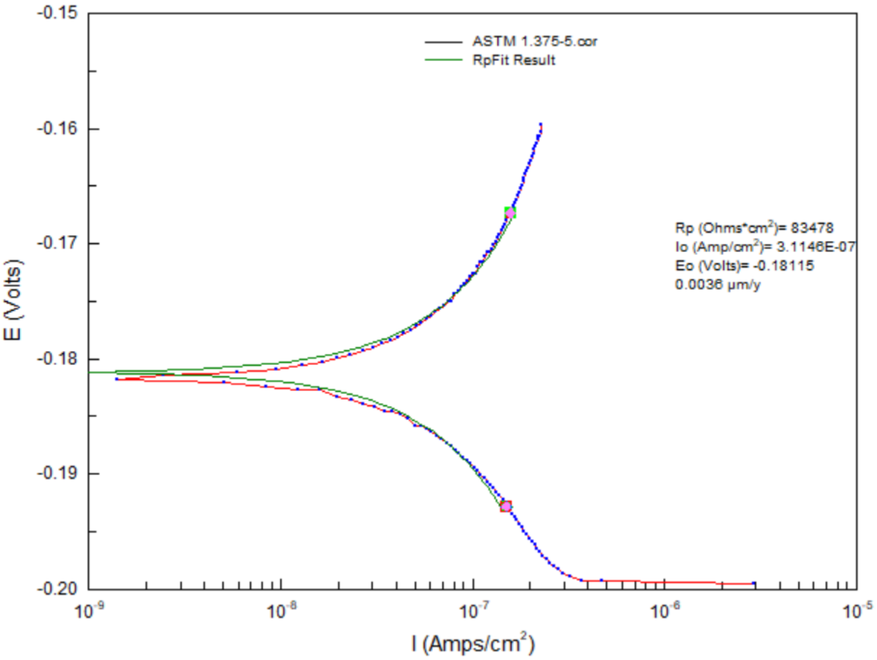
D. Appendix D: UMKC ASTM type Test LPR Data

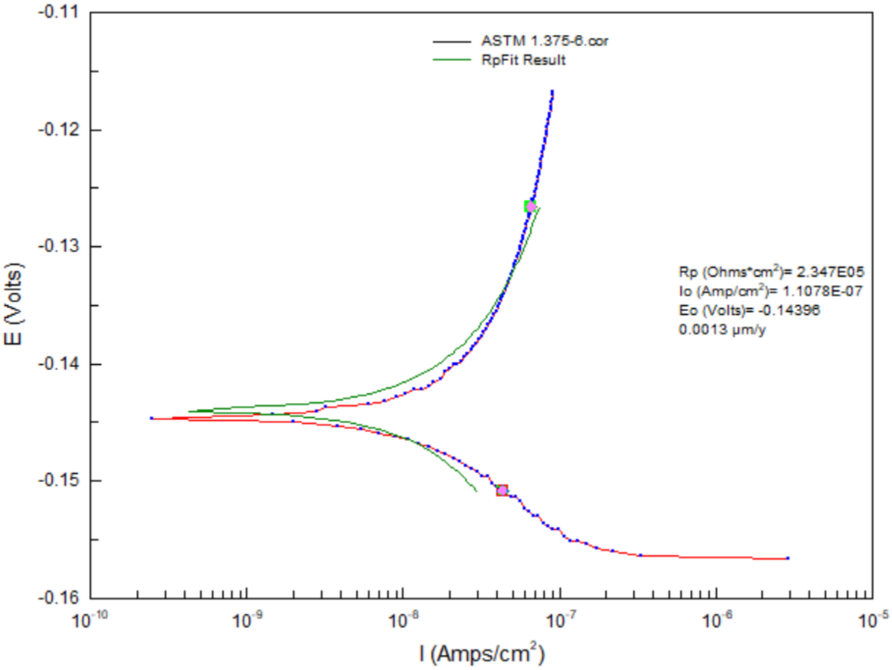

ASTM 1.375-1.cor
RpFit Result
Rp (Ohms*cm^2)= 1.4209E05
Io (Amp/cm^2)= 1.8298E-07
Eo (Volts)= -0.31961
0.0021 µm/y
E (Volts)
-0.29
-0.30
-0.31
-0.32
-0.33
I (Amps/cm^2)
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}

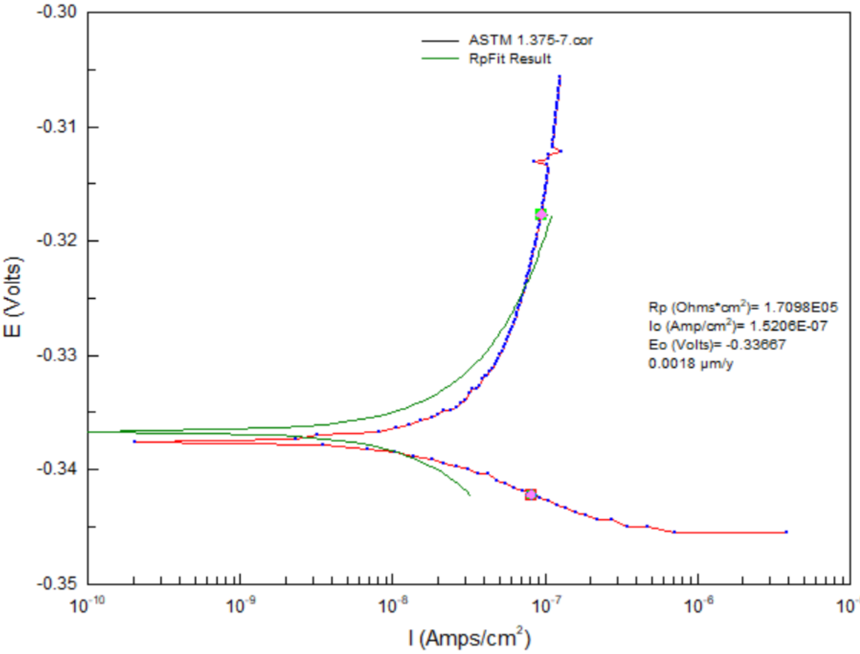

ASTM 1.375-2.cor
RpFit Result
Rp (Ohms*cm^2)= 2.6396E05
Io (Amp/cm^2)= 9.8501E-08
Eo (Volts)= -0.25144
0.0011 µm/y
E (Volts)
-0.22
-0.23
-0.24
-0.25
-0.26
-0.27
I (Amps/cm^2)
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}

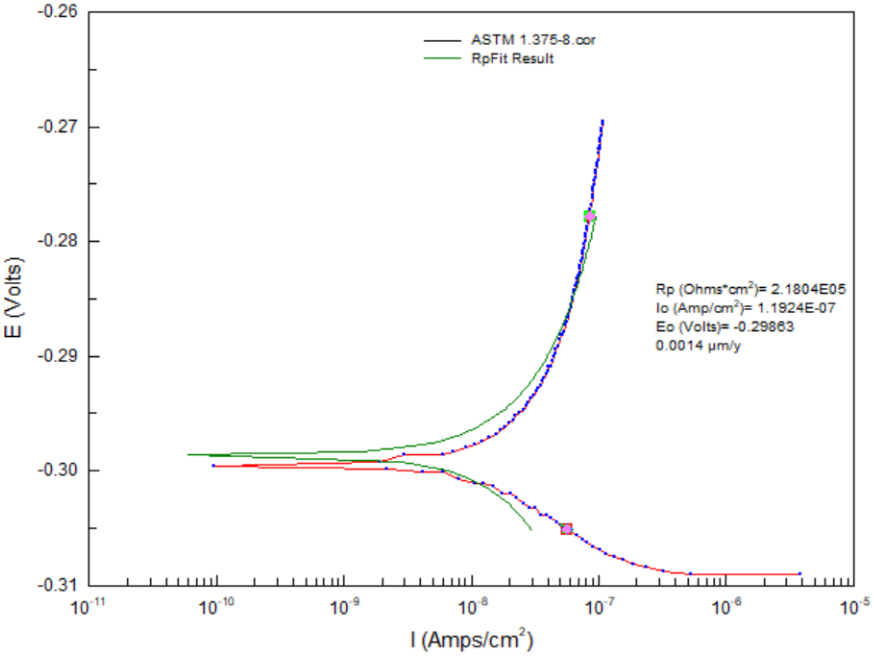

-0.11
-0.12
-0.13
-0.14
-0.15
-0.16
E (Volts)
ASTM 1.375-3.cor
RpFit Result
Rp (Ohms*cm^2)= 2.4375E05
Io (Amp/cm^2)= 1.0667E-07
Eo (Volts)= -0.14033
0.0012 µm/y
10^{-11}
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}
I (Amps/cm^2)


-0.26
-0.27
-0.28
-0.29
-0.30
-0.31
E (Volts)
ASTM 1.375-4.cor
RpFit Result
Rp (Ohms*cm^2)= 95609
Io (Amp/cm^2)= 2.7194E-07
Eo (Volts)= -0.29457
0.00314 µm/y
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}
I (Amps/cm^2)


ASTM 1.375-5.cor
RpFit Result
Rp (Ohms*cm^2)= 83478
Io (Amp/cm^2)= 3.1146E-07
Eo (Volts)= -0.18115
0.0036 µm/y
E (Volts)
I (Amps/cm^2)
-0.15
-0.16
-0.17
-0.18
-0.19
-0.20
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}


ASTM 1.375-6.cor
RpFit Result
Rp (Ohms*cm^2)= 2.347E05
Io (Amp/cm^2)= 1.1078E-07
Eo (Volts)= -0.14396
0.0013 µm/y
E (Volts)
I (Amps/cm^2)
-0.11
-0.12
-0.13
-0.14
-0.15
-0.16
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}


ASTM 1.375-7.cor
RpFit Result
Rp (Ohms*cm^2)= 1.7098E05
Io (Amp/cm^2)= 1.5206E-07
Eo (Volts)= -0.33667
0.0018 µm/y
E (Volts)
-0.30
-0.31
-0.32
-0.33
-0.34
-0.35
I (Amps/cm^2)
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}


ASTM 1.375-8.cor
RpFit Result
Rp (Ohms*cm^2)= 2.1804E05
Io (Amp/cm^2)= 1.1924E-07
Eo (Volts)= -0.29863
0.0014 µm/y
E (Volts)
-0.26
-0.27
-0.28
-0.29
-0.30
-0.31
I (Amps/cm^2)
10^{-11}
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}

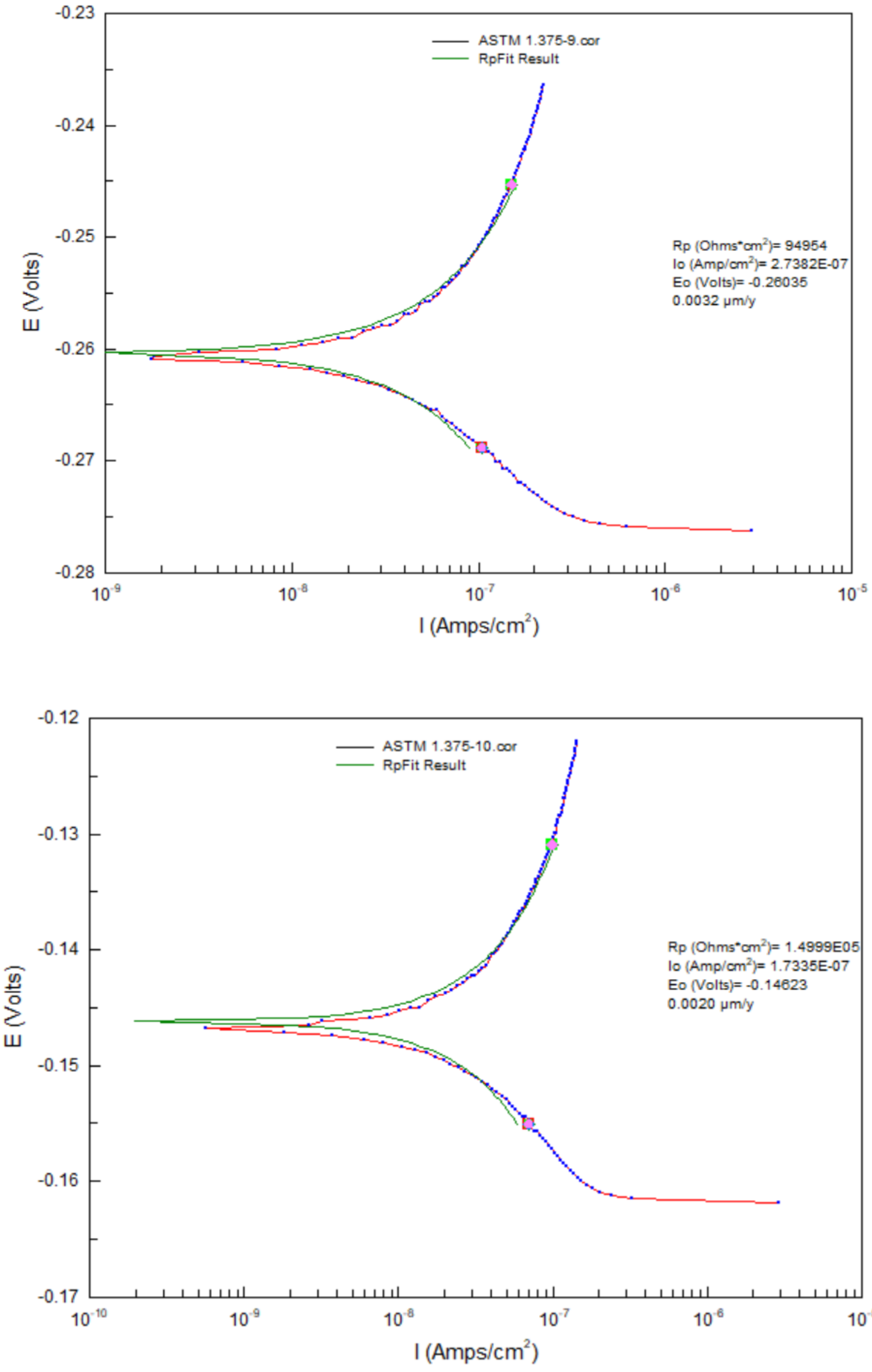
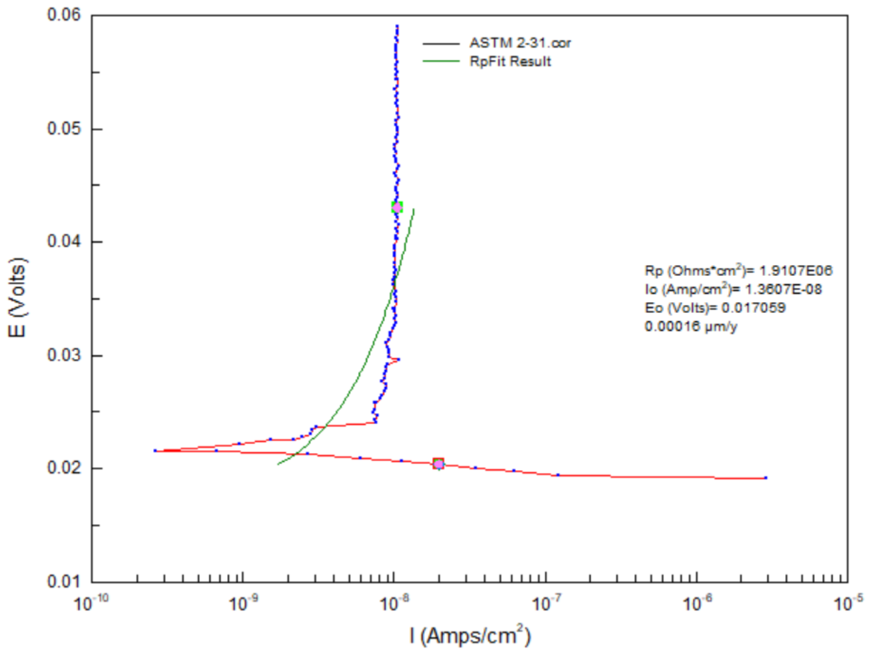
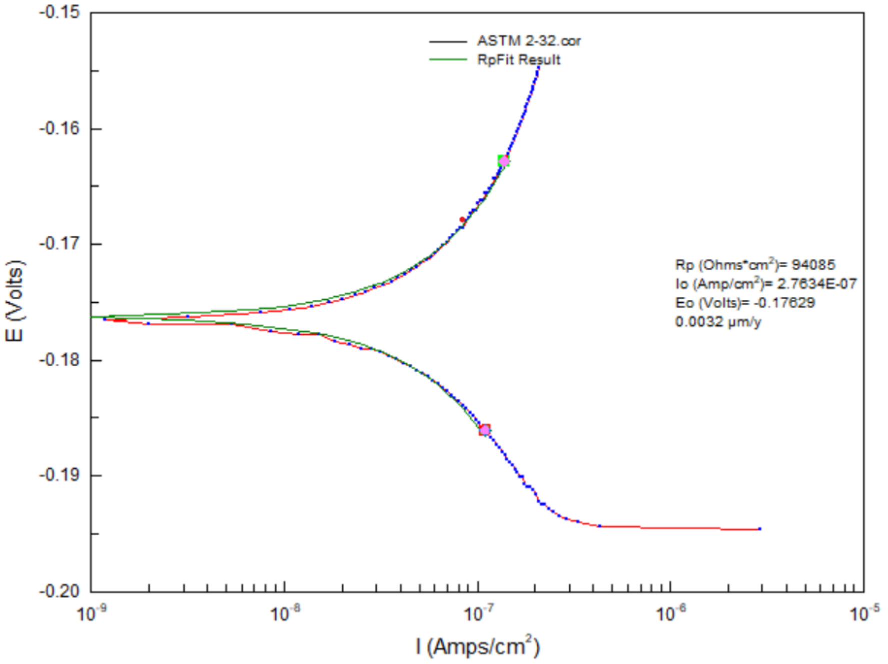
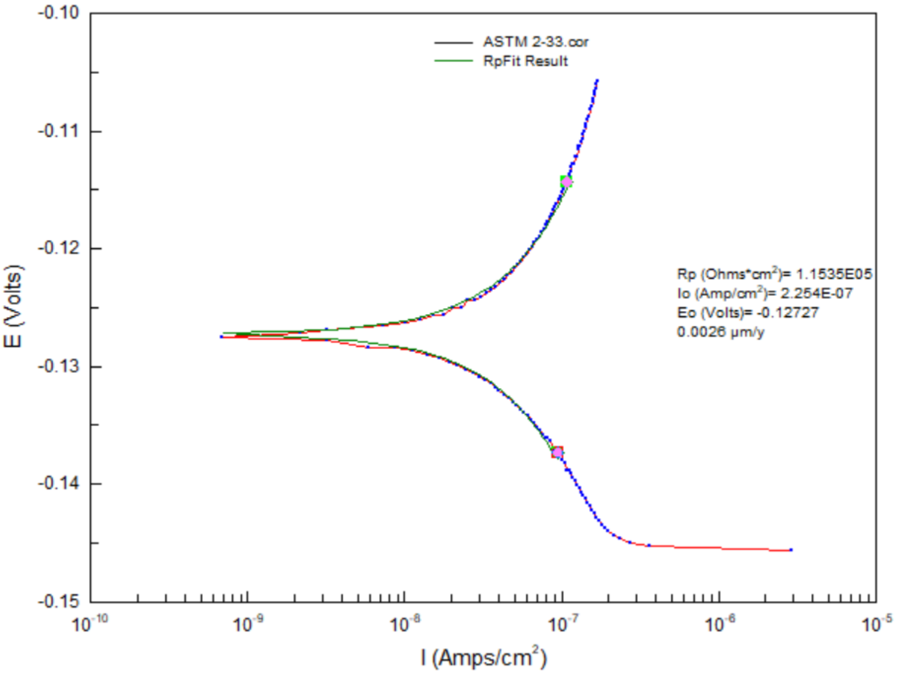
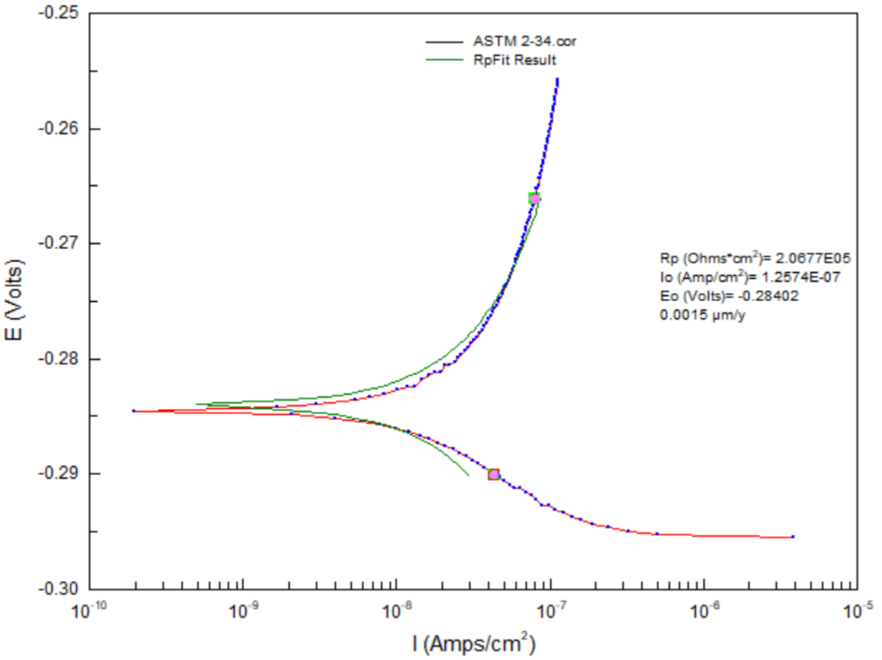


Figure 1 LPR for Specimens with s/c of 1.375


0.06
0.05
0.04
0.03
0.02
0.01
E (Volts)
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}
I (Amps/cm^2)
ASTM 2-31.cor
RpFit Result
Rp (Ohms*cm^2)= 1.9107E06
Io (Amp/cm^2)= 1.3607E-08
Eo (Volts)= 0.017059
0.00016 µm/y


-0.15
-0.16
-0.17
-0.18
-0.19
-0.20
E (Volts)
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}
I (Amps/cm^2)
ASTM 2-32.cor
RpFit Result
Rp (Ohms*cm^2)= 94085
Io (Amp/cm^2)= 2.7634E-07
Eo (Volts)= -0.17629
0.0032 µm/y


ASTM 2-33.cor
RpFit Result
Rp (Ohms*cm²)= 1.1535E05
Io (Amp/cm²)= 2.254E-07
Eo (Volts)= -0.12727
0.0026 µm/y
E (Volts)
-0.10
-0.11
-0.12
-0.13
-0.14
-0.15
I (Amps/cm²)
10⁻¹⁰
10⁻⁹
10⁻⁸
10⁻⁷
10⁻⁶
10⁻⁵


ASTM 2-34.cor
RpFit Result
Rp (Ohms*cm²)= 2.0677E05
Io (Amp/cm²)= 1.2574E-07
Eo (Volts)= -0.28402
0.0015 µm/y
E (Volts)
-0.25
-0.26
-0.27
-0.28
-0.29
-0.30
I (Amps/cm²)
10⁻¹⁰
10⁻⁹
10⁻⁸
10⁻⁷
10⁻⁶
10⁻⁵

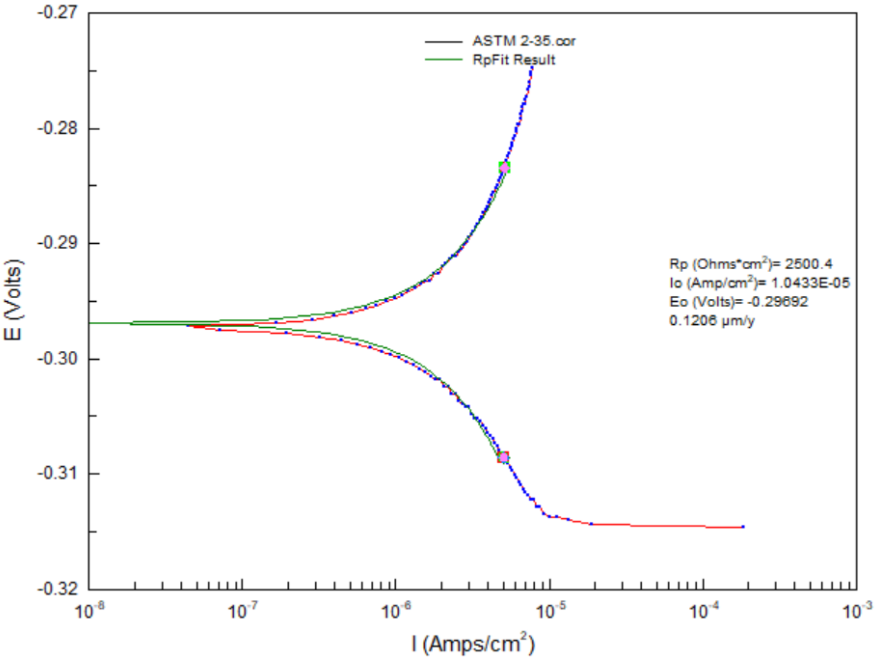
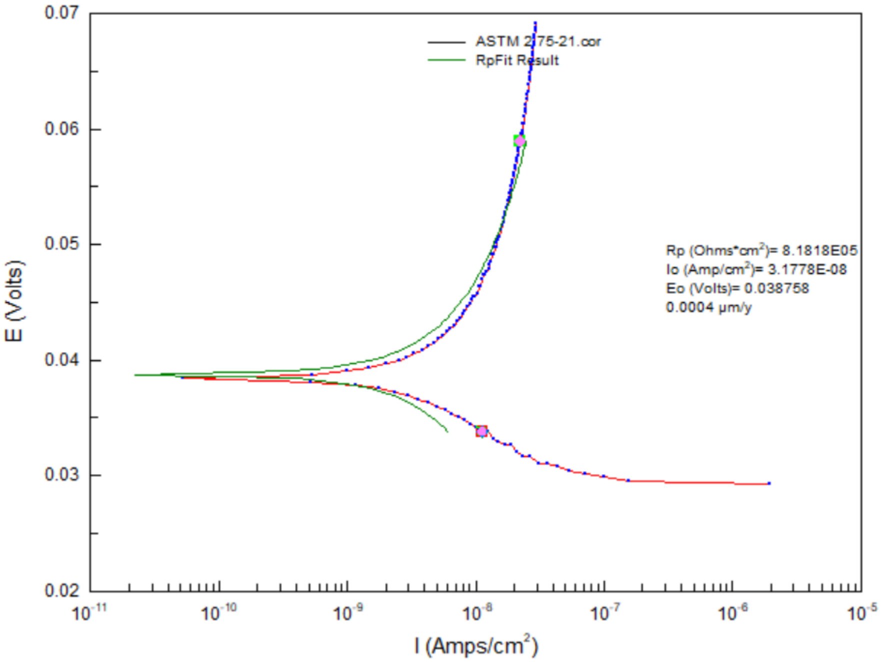
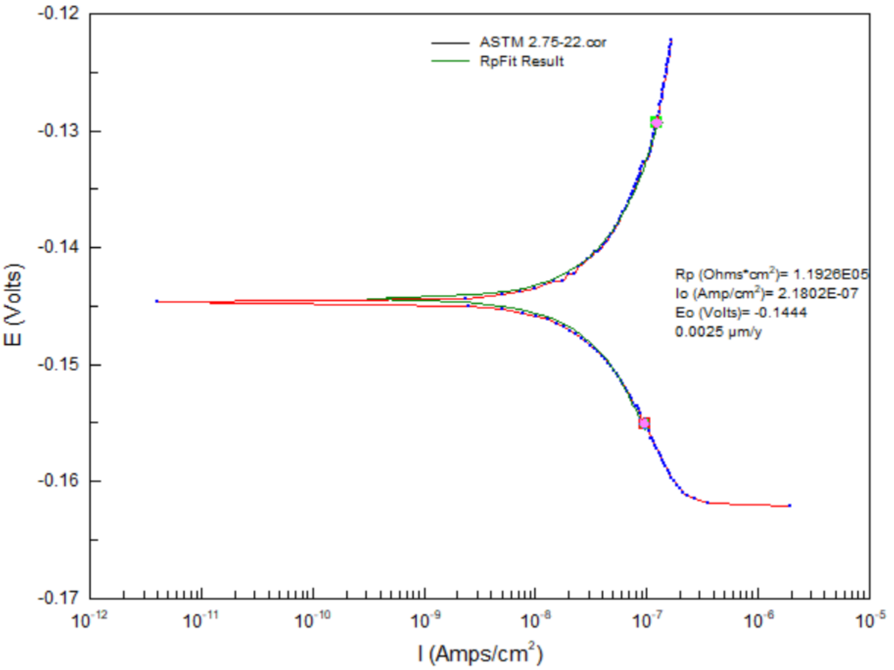


Figure 2 LPR of specimens with s/c of 2.0


ASTM 2.75-21.cor
RpFit Result
Rp (Ohms*cm^2)= 8.1818E05
Io (Amp/cm^2)= 3.1778E-08
Eo (Volts)= 0.038758
0.0004 µm/y
E (Volts)
0.07
0.06
0.05
0.04
0.03
0.02
10^{-11}
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}
I (Amps/cm^2)


ASTM 2.75-22.cor
RpFit Result
Rp (Ohms*cm^2)= 1.1926E05
Io (Amp/cm^2)= 2.1802E-07
Eo (Volts)= -0.1444
0.0025 µm/y
E (Volts)
-0.12
-0.13
-0.14
-0.15
-0.16
-0.17
10^{-12}
10^{-11}
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}
I (Amps/cm^2)

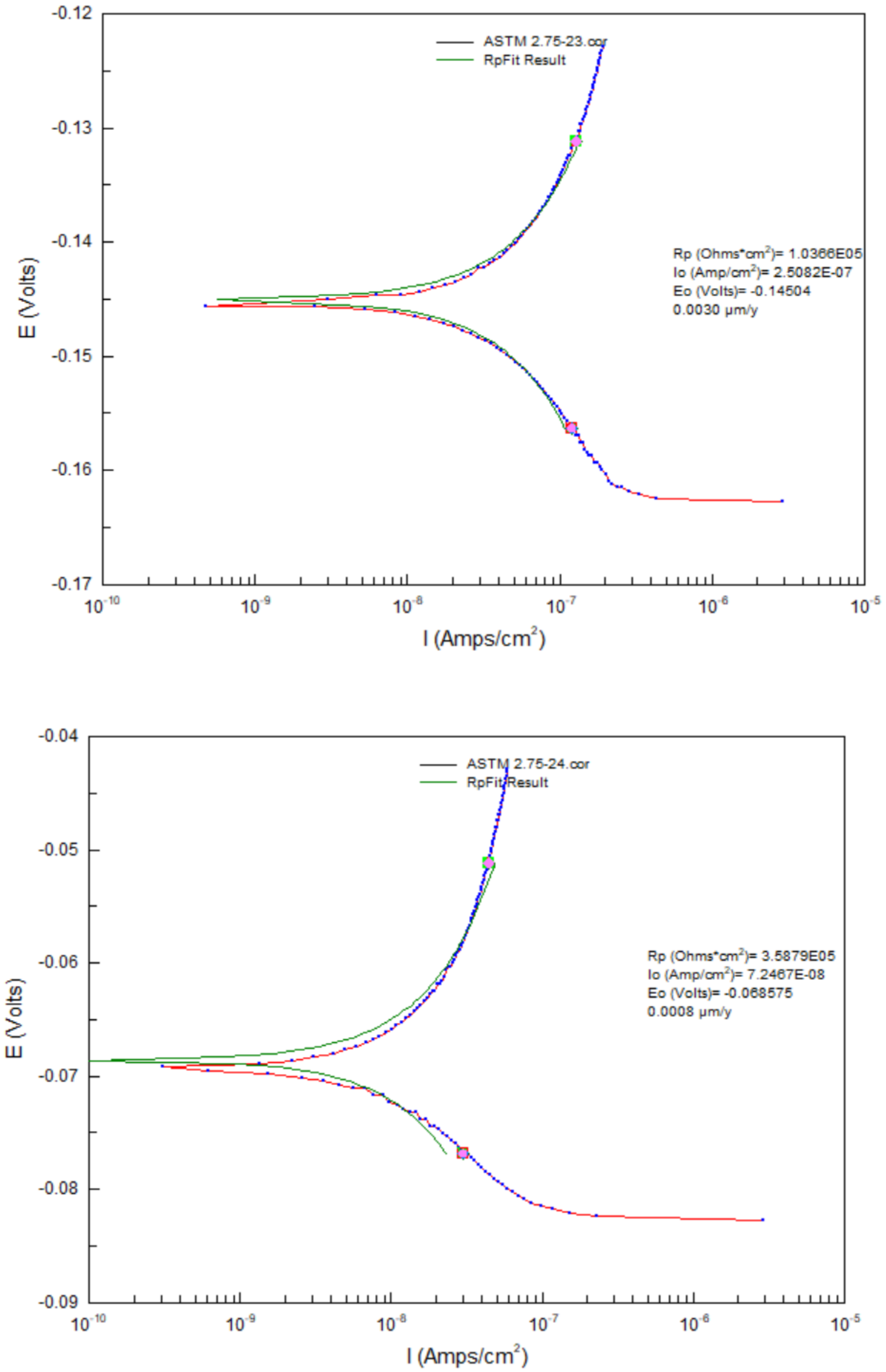
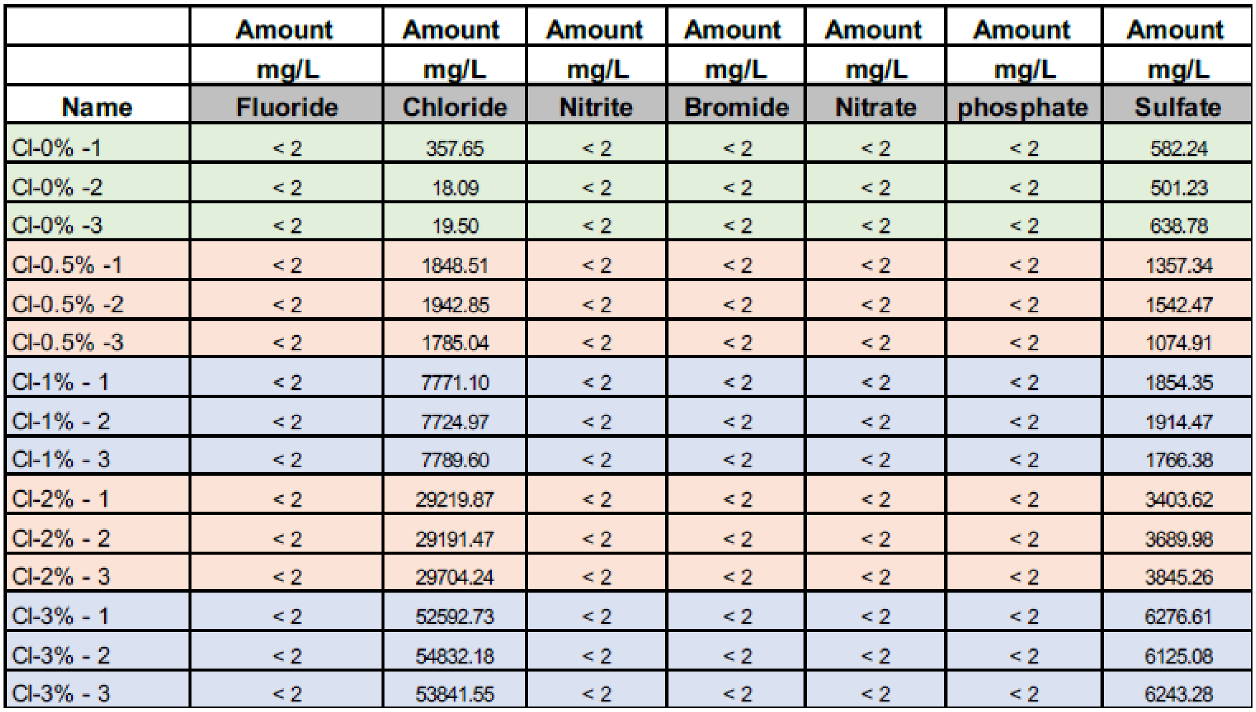


Figure 3 LPR of specimens with s/c of 2.75

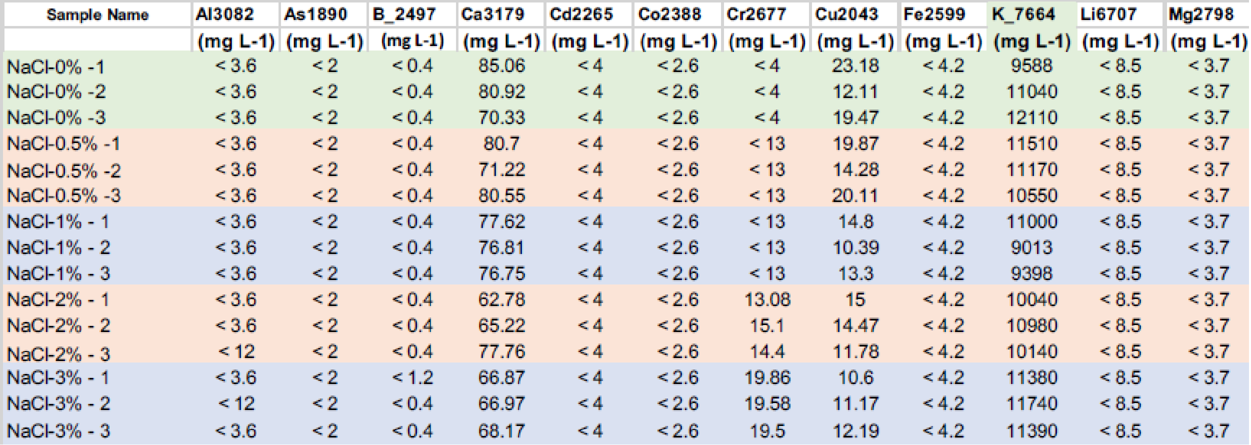
E. Appendix E: University of Waterloo Pore Solution Analysis

Table I. Anion concentrations of expressed pore solution, determined by Ion Chromatography



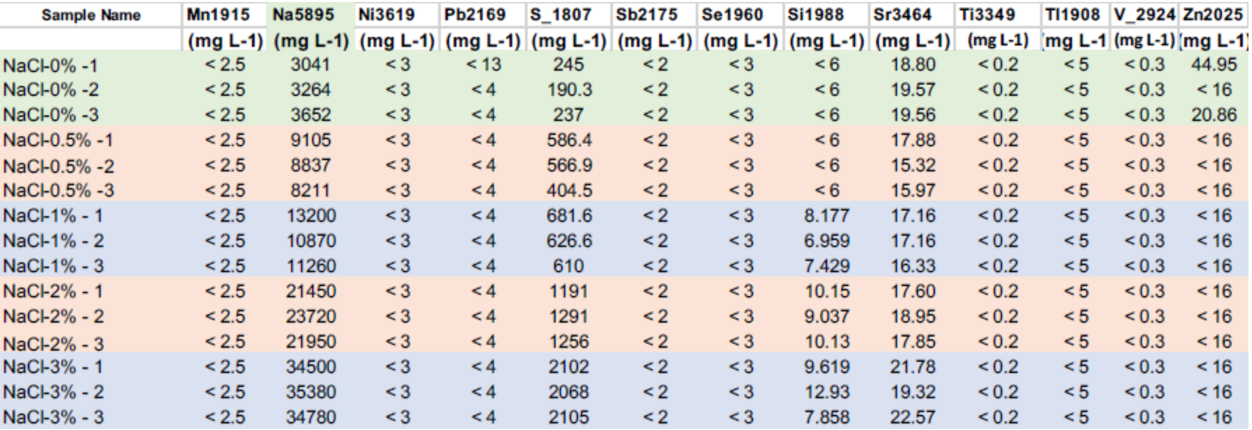
	Amount	Amount	Amount	Amount	Amount	Amount	Amount
	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
Name	Fluoride	Chloride	Nitrite	Bromide	Nitrate	phosphate	Sulfate
Cl-0% -1	< 2	357.65	< 2	< 2	< 2	< 2	582.24
Cl-0% -2	< 2	18.09	< 2	< 2	< 2	< 2	501.23
Cl-0% -3	< 2	19.50	< 2	< 2	< 2	< 2	638.78
Cl-0.5% -1	< 2	1848.51	< 2	< 2	< 2	< 2	1357.34
Cl-0.5% -2	< 2	1942.85	< 2	< 2	< 2	< 2	1542.47
Cl-0.5% -3	< 2	1785.04	< 2	< 2	< 2	< 2	1074.91
Cl-1% - 1	< 2	7771.10	< 2	< 2	< 2	< 2	1854.35
Cl-1% - 2	< 2	7724.97	< 2	< 2	< 2	< 2	1914.47
Cl-1% - 3	< 2	7789.60	< 2	< 2	< 2	< 2	1766.38
Cl-2% - 1	< 2	29219.87	< 2	< 2	< 2	< 2	3403.62
Cl-2% - 2	< 2	29191.47	< 2	< 2	< 2	< 2	3689.98
Cl-2% - 3	< 2	29704.24	< 2	< 2	< 2	< 2	3845.26
Cl-3% - 1	< 2	52592.73	< 2	< 2	< 2	< 2	6276.61
Cl-3% - 2	< 2	54832.18	< 2	< 2	< 2	< 2	6125.08
Cl-3% - 3	< 2	53841.55	< 2	< 2	< 2	< 2	6243.28

Table II. Cation concentrations of expressed pore solution determined by OES Inductively Coupled Plasma_part 1



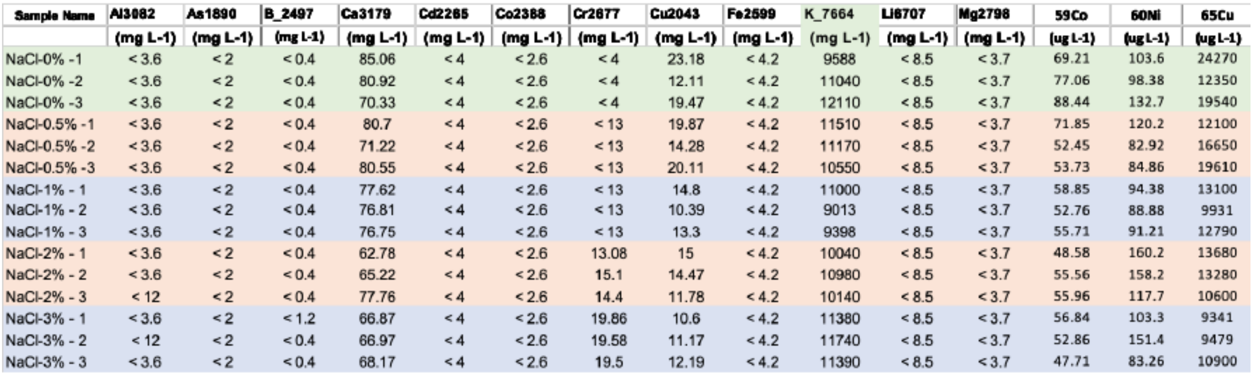
Sample Name	Al3082	As1890	B_2497	Ca3179	Cd2265	Co2388	Cr2677	Cu2043	Fe2599	K_7664	Li6707	Mg2798
	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)
NaCl-0% -1	< 3.6	< 2	< 0.4	85.06	< 4	< 2.6	< 4	23.18	< 4.2	9588	< 8.5	< 3.7
NaCl-0% -2	< 3.6	< 2	< 0.4	80.92	< 4	< 2.6	< 4	12.11	< 4.2	11040	< 8.5	< 3.7
NaCl-0% -3	< 3.6	< 2	< 0.4	70.33	< 4	< 2.6	< 4	19.47	< 4.2	12110	< 8.5	< 3.7
NaCl-0.5% -1	< 3.6	< 2	< 0.4	80.7	< 4	< 2.6	< 13	19.87	< 4.2	11510	< 8.5	< 3.7
NaCl-0.5% -2	< 3.6	< 2	< 0.4	71.22	< 4	< 2.6	< 13	14.28	< 4.2	11170	< 8.5	< 3.7
NaCl-0.5% -3	< 3.6	< 2	< 0.4	80.55	< 4	< 2.6	< 13	20.11	< 4.2	10550	< 8.5	< 3.7
NaCl-1% - 1	< 3.6	< 2	< 0.4	77.62	< 4	< 2.6	< 13	14.8	< 4.2	11000	< 8.5	< 3.7
NaCl-1% - 2	< 3.6	< 2	< 0.4	76.81	< 4	< 2.6	< 13	10.39	< 4.2	9013	< 8.5	< 3.7
NaCl-1% - 3	< 3.6	< 2	< 0.4	76.75	< 4	< 2.6	< 13	13.3	< 4.2	9398	< 8.5	< 3.7
NaCl-2% - 1	< 3.6	< 2	< 0.4	62.78	< 4	< 2.6	13.08	15	< 4.2	10040	< 8.5	< 3.7
NaCl-2% - 2	< 3.6	< 2	< 0.4	65.22	< 4	< 2.6	15.1	14.47	< 4.2	10980	< 8.5	< 3.7
NaCl-2% - 3	< 12	< 2	< 0.4	77.76	< 4	< 2.6	14.4	11.78	< 4.2	10140	< 8.5	< 3.7
NaCl-3% - 1	< 3.6	< 2	< 1.2	66.87	< 4	< 2.6	19.86	10.6	< 4.2	11380	< 8.5	< 3.7
NaCl-3% - 2	< 12	< 2	< 0.4	66.97	< 4	< 2.6	19.58	11.17	< 4.2	11740	< 8.5	< 3.7
NaCl-3% - 3	< 3.6	< 2	< 0.4	68.17	< 4	< 2.6	19.5	12.19	< 4.2	11390	< 8.5	< 3.7

Table II. Cation concentrations of expressed pore solution determined by OES Inductively Coupled Plasma_part 2



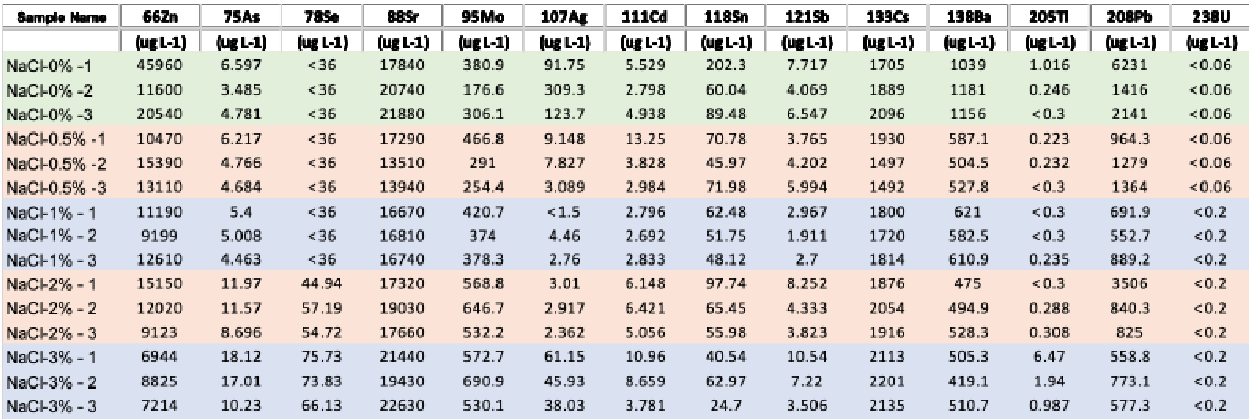
Sample Name	Mn1915	Na5895	Ni3619	Pb2169	S_1807	Sb2175	Se1960	Si1988	Sr3464	Ti3349	Tl1908	V_2924	Zn2025
	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	mg L-1	(mg L-1)	(mg L-1
NaCl-0% -1	< 2.5	3041	< 3	< 13	245	< 2	< 3	< 6	18.80	< 0.2	< 5	< 0.3	44.95
NaCl-0% -2	< 2.5	3264	< 3	< 4	190.3	< 2	< 3	< 6	19.57	< 0.2	< 5	< 0.3	< 16
NaCl-0% -3	< 2.5	3652	< 3	< 4	237	< 2	< 3	< 6	19.56	< 0.2	< 5	< 0.3	20.86
NaCl-0.5% -1	< 2.5	9105	< 3	< 4	586.4	< 2	< 3	< 6	17.88	< 0.2	< 5	< 0.3	< 16
NaCl-0.5% -2	< 2.5	8837	< 3	< 4	566.9	< 2	< 3	< 6	15.32	< 0.2	< 5	< 0.3	< 16
NaCl-0.5% -3	< 2.5	8211	< 3	< 4	404.5	< 2	< 3	< 6	15.97	< 0.2	< 5	< 0.3	< 16
NaCl-1% - 1	< 2.5	13200	< 3	< 4	681.6	< 2	< 3	8.177	17.16	< 0.2	< 5	< 0.3	< 16
NaCl-1% - 2	< 2.5	10870	< 3	< 4	626.6	< 2	< 3	6.959	17.16	< 0.2	< 5	< 0.3	< 16
NaCl-1% - 3	< 2.5	11260	< 3	< 4	610	< 2	< 3	7.429	16.33	< 0.2	< 5	< 0.3	< 16
NaCl-2% - 1	< 2.5	21450	< 3	< 4	1191	< 2	< 3	10.15	17.60	< 0.2	< 5	< 0.3	< 16
NaCl-2% - 2	< 2.5	23720	< 3	< 4	1291	< 2	< 3	9.037	18.95	< 0.2	< 5	< 0.3	< 16
NaCl-2% - 3	< 2.5	21950	< 3	< 4	1256	< 2	< 3	10.13	17.85	< 0.2	< 5	< 0.3	< 16
NaCl-3% - 1	< 2.5	34500	< 3	< 4	2102	< 2	< 3	9.619	21.78	< 0.2	< 5	< 0.3	< 16
NaCl-3% - 2	< 2.5	35380	< 3	< 4	2068	< 2	< 3	12.93	19.32	< 0.2	< 5	< 0.3	< 16
NaCl-3% - 3	< 2.5	34780	< 3	< 4	2105	< 2	< 3	7.858	22.57	< 0.2	< 5	< 0.3	< 16

Table III. Cations concentrations of expressed pore solution determined by MS Inductively Coupled Plasma_part 1



Sample Name	Al3082	As1890	B_2497	Ca3179	Cd2265	Co2388	Cr2677	Cu2043	Fe2599	K_7664	Li6707	Mg2798	59Co	60Ni	65Cu
	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(mg L-1)	(ug L-1)	(ug L-1)	(ug L-1)
NaCl-0% -1	< 3.6	< 2	< 0.4	85.06	< 4	< 2.6	< 4	23.18	< 4.2	9588	< 8.5	< 3.7	69.21	103.6	24270
NaCl-0% -2	< 3.6	< 2	< 0.4	80.92	< 4	< 2.6	< 4	12.11	< 4.2	11040	< 8.5	< 3.7	77.06	98.38	12350
NaCl-0% -3	< 3.6	< 2	< 0.4	70.33	< 4	< 2.6	< 4	19.47	< 4.2	12110	< 8.5	< 3.7	88.44	132.7	19540
NaCl-0.5% -1	< 3.6	< 2	< 0.4	80.7	< 4	< 2.6	< 13	19.87	< 4.2	11510	< 8.5	< 3.7	71.85	120.2	12100
NaCl-0.5% -2	< 3.6	< 2	< 0.4	71.22	< 4	< 2.6	< 13	14.28	< 4.2	11170	< 8.5	< 3.7	52.45	82.92	16650
NaCl-0.5% -3	< 3.6	< 2	< 0.4	80.55	< 4	< 2.6	< 13	20.11	< 4.2	10550	< 8.5	< 3.7	53.73	84.86	19610
NaCl-1% - 1	< 3.6	< 2	< 0.4	77.62	< 4	< 2.6	< 13	14.8	< 4.2	11000	< 8.5	< 3.7	58.85	94.38	13100
NaCl-1% - 2	< 3.6	< 2	< 0.4	76.81	< 4	< 2.6	< 13	10.39	< 4.2	9013	< 8.5	< 3.7	52.76	88.88	9931
NaCl-1% - 3	< 3.6	< 2	< 0.4	76.75	< 4	< 2.6	< 13	13.3	< 4.2	9398	< 8.5	< 3.7	55.71	91.21	12790
NaCl-2% - 1	< 3.6	< 2	< 0.4	62.78	< 4	< 2.6	13.08	15	< 4.2	10040	< 8.5	< 3.7	48.58	160.2	13680
NaCl-2% - 2	< 3.6	< 2	< 0.4	65.22	< 4	< 2.6	15.1	14.47	< 4.2	10980	< 8.5	< 3.7	55.56	158.2	13280
NaCl-2% - 3	< 12	< 2	< 0.4	77.76	< 4	< 2.6	14.4	11.78	< 4.2	10140	< 8.5	< 3.7	55.96	117.7	10600
NaCl-3% - 1	< 3.6	< 2	< 1.2	66.87	< 4	< 2.6	19.86	10.6	< 4.2	11380	< 8.5	< 3.7	56.84	103.3	9341
NaCl-3% - 2	< 12	< 2	< 0.4	66.97	< 4	< 2.6	19.58	11.17	< 4.2	11740	< 8.5	< 3.7	52.86	151.4	9479
NaCl-3% - 3	< 3.6	< 2	< 0.4	68.17	< 4	< 2.6	19.5	12.19	< 4.2	11390	< 8.5	< 3.7	47.71	83.26	10900

Table III. Cations concentrations of expressed pore solution determined by MS Inductively Coupled Plasma_part 2



Sample Name	66Zn	75As	78Se	88Sr	95Mo	107Ag	111Cd	118Sn	121Sb	133Cs	138Ba	205Tl	208Pb	238U
	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)	(ug L-1)
NaCl-0% -1	45960	6.597	<36	17840	380.9	91.75	5.529	202.3	7.717	1705	1039	1.016	6231	<0.06
NaCl-0% -2	11600	3.485	<36	20740	176.6	309.3	2.798	60.04	4.069	1889	1181	0.246	1416	<0.06
NaCl-0% -3	20540	4.781	<36	21880	306.1	123.7	4.938	89.48	6.547	2096	1156	<0.3	2141	<0.06
NaCl-0.5% -1	10470	6.217	<36	17290	466.8	9.148	13.25	70.78	3.765	1930	587.1	0.223	964.3	<0.06
NaCl-0.5% -2	15390	4.766	<36	13510	291	7.827	3.828	45.97	4.202	1497	504.5	0.232	1279	<0.06
NaCl-0.5% -3	13110	4.684	<36	13940	254.4	3.089	2.984	71.98	5.994	1492	527.8	<0.3	1364	<0.06
NaCl-1% - 1	11190	5.4	<36	16670	420.7	<1.5	2.796	62.48	2.967	1800	621	<0.3	691.9	<0.2
NaCl-1% - 2	9199	5.008	<36	16810	374	4.46	2.692	51.75	1.911	1720	582.5	<0.3	552.7	<0.2
NaCl-1% - 3	12610	4.463	<36	16740	378.3	2.76	2.833	48.12	2.7	1814	610.9	0.235	889.2	<0.2
NaCl-2% - 1	15150	11.97	44.94	17320	568.8	3.01	6.148	97.74	8.252	1876	475	<0.3	3506	<0.2
NaCl-2% - 2	12020	11.57	57.19	19030	646.7	2.917	6.421	65.45	4.333	2054	494.9	0.288	840.3	<0.2
NaCl-2% - 3	9123	8.696	54.72	17660	532.2	2.362	5.056	55.98	3.823	1916	528.3	0.308	825	<0.2
NaCl-3% - 1	6944	18.12	75.73	21440	572.7	61.15	10.96	40.54	10.54	2113	505.3	6.47	558.8	<0.2
NaCl-3% - 2	8825	17.01	73.83	19430	690.9	45.93	8.659	62.97	7.22	2201	419.1	1.94	773.1	<0.2
NaCl-3% - 3	7214	10.23	66.13	22630	530.1	38.03	3.781	24.7	3.506	2135	510.7	0.987	577.3	<0.2