INTEGRATING CORROSION TESTING INDUSTRIAL PROTOCOLS INTO A HIGH SCHOOL / TWO-YEAR COLLEGE CHEMISTRY CURRICULUM

by Michael Angelo D’Agostino

The thesis described herein involves research in the domain of Curriculum Development and Testing in chemical education as partial fulfillment of the requirements for the M.S. program in chemistry at Miami University with an emphasis on chemical education.

The research undertaken was the development of chemistry instructional materials which integrate authentic industrial chemical technology practices into a high school/two-year college curriculum. The study of iron corrosion was used to create eight activities, including the following areas: economic impact, relative reactivity of iron, oxidation reduction, factors affecting corrosion, qualitative and quantitative analysis, and the testing and evaluation of corrosion control coatings using simulated American Society of Testing and Materials (ASTM) protocols for corrosion testing. Additionally, chemical tests were conducted to explore the underlying mechanisms involved in the corrosion of iron. All activities were developed to integrate with National Science Education Standards (NSES), Voluntary Industrial Standards (VIS), and American College Testing (ACT) Workkeys Standards.
INTEGRATING CORROSION TESTING INDUSTRIAL PROTOCOLS INTO A HIGH SCHOOL/TWO-YEAR COLLEGE CHEMISTRY CURRICULUM

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Department of Chemistry and Biochemistry

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DEDICATION

To Emilie
the love of my life
and the best Mama anywhere

To Emerson and Margo
for you bring genuine happiness
the love only children can bring

Life is all about the journey
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To Emilie, my wife, whose unfailing flexibility and support made this pursuit possible.

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To fellow science faculty at Madeira High School, Christy Barton, Davis Tapp, and Margaret Miller, who heard much complaining about corrosion over the years.

To my Madeira students of 2004-2005, who were subjected to a sleep deprived, somewhat incoherent chemistry instructor during the final phase of this project.

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To the countless high school teachers and students from around the country who provided important constructive feedback in the development of *The Corrosion Unit* in all stages of its development.

To the Board of Education of Madeira City Schools who graciously provided technical resources such as computer hardware, color laser printers, instrumentation, and projection equipment throughout the duration of this pursuit.

And finally, to my parents Michael and Evelyn D’Agostino who taught me the value of hard work, perseverance, and education.
Chapter 1: Introduction

1.1 Statement of the problem

The research problem undertaken in this work was the development of a standards-based instructional unit that could be integrated into existing curriculum of core chemistry and chemistry technology courses at high school and two-year college levels. Additionally, thus unit had to be designed to provide contextual understanding of chemistry content while including authentic industrial practices used in the discipline of chemical technology, workplace readiness skills, and current pedagogical strategies.

The rationale for undertaking this research was to provide high school and two-year college chemistry educators with instructional materials they could use to better prepare their students to subsequently assume jobs in today’s highly technical workforce. Giddens and Stasz (1) describe the skills technical workers utilize in their daily tasks. “Today’s technical workers are expected to draw information from a range of…disciplines and to use that knowledge to perform tasks and solve unique problems…without direct oversight or much advice. Technical workers are synthesizers of information,…their jobs are as likely to demand creativity and intellectual dexterity as the ability to follow instructions or work precisely.” Stasz also states, “…students learn to work in a classroom where skills and attitudes are taught in the context of complex, realistic tasks.” She continues by
noting, “…teachers are not currently trained to design and conduct classrooms based on a culture of authentic practice,” and that teachers need opportunities to enhance their experience with authentic practice within their discipline (2). Instructional materials of this type linked specifically to the corrosion field did not previously exist. The result of this work was the creation of a unit titled *The Corrosion Unit* which is provided in its entirety in the Appendix.

### 1.2 Goal and Objectives

The goal of developing *The Corrosion Unit* was to provide instructional materials that could be integrated into existing chemistry curriculum in high school, two-year college and chemistry technology programs. Thus, not separating important job skills from their direct application in the chemistry-based workplace.

This goal was accomplished through the following objectives:

1. To use the process of corrosion of iron as a framework to present the contextual basis for the chemistry.

2. To develop activities that address the National Science Education Standards (NSES) (3), The American Chemical Society (ACS) Voluntary Industrial Standards (VIS) (4), and American College Testing (ACT) Workkeys Standards (5).
3. To include authentic complex tasks that integrate workplace applications of chemistry content, process skills and procedures based on American Society of Testing and Materials (ASTM) standards (6).

4. To apply constructivism to the design and development of the unit by following a learning cycle approach, embedding cooperative learning strategies and using case-study scenarios that were built from actual workplace situations as a part of the activities.

5. To utilize the Center for Chemistry Education (CCE) Continuous Quality-Control Materials Development Protocol to ensure a well-tested, safe unit (7).

1.3 Overview of the Corrosion Unit

*The Corrosion Unit* was developed as part of the *Using Chemistry* project which was funded by the National Science Foundation (8) under the guidance of Miami University’s Center for Chemistry Education (CCE). In developing *The Corrosion Unit*, the established planning method known as a learning cycle was used to create opportunities for students to confront what they presently know, formulate hypotheses about new observations, work on tasks that promote questions and allow students to make new discoveries based on these experiences (9). Learning cycles are based on the
constructivist philosophy of learning that holds that students construct their own knowledge based on their existing experiences and beliefs (10). The learning cycle protocol for this unit is as follows:

- **Awareness:** Students are engaged in one or more simple activities to help the students make connections between past and present learning experiences. This lays the groundwork for subsequent experiences and stimulate their involvement in the lesson. In this unit, this translated into having the students conduct a web exploration about the economic impact of corrosion and also witness a video presentation about a corrosion induced bridge collapse.

- **Exploration:** Several experiences are provided for students to do which build a base of common experiences to initiate the development of their understanding. For this unit, students explored accelerated rusting of steel wool and a metal activity series exercise.

- **Concept Introduction:** Two multi-part activities are included in this phase of the learning cycle to introduce students to the chemical mechanisms of corrosion and provide them experience using standard industrial testing protocols.

- **Concept Application:** Students apply knowledge gained from the previous activities to the research component of the unit.
Specifically, this became the task of testing various coatings for steel utilizing new standard industrial testing protocols, and obtaining data to help them answer whether a bridge should be closed because of unsafe conditions caused by corrosion processes.

The complete corrosion unit, which includes a prologue and eight classroom activities is in the Appendix and their titles are identified by bold text throughout this thesis. Each activity develops a category of the learning cycle utilizing both a student handout and instructor notes. A summary of the learning cycle category and the most significant skills and content within each activity are noted in the table on the following page. Skills (teamwork, communication, observation) and content (conducting chemical investigations, identifying chemical changes) common to multiple activities have been omitted from the table. A detailed listing of all featured skills and key scientific concepts are provided in the instructor notes section of each activity in the Appendix.
**Overview of The Corrosion Unit**

<table>
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<th>The Prologue</th>
<th>provides an overview for the teacher of the chemistry of rusting including the various reaction mechanisms</th>
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| Awareness | Web Research: What’s Up With Corrosion? | *skills*: web research, presentation, locating and organization of information  
*content*: corrosion control methods, economic impact |
| Awareness | Corrosion Collapse | *skills*: listening, applied technology, recall of information  
*content*: societal impact of undetected corrosion, scientific method |
| Exploration | The Hungry Flask | *skills*: obtaining evidence, formulation of a theory  
*content*: simplified view of rusting, elements vs. compounds, oxygen’s role in rusting |
| Exploration | The Reactivity of Metals | *skills*: data analysis, identifying trends  
*content*: activity series, ionic compound formulas, single replacement reactions |
| Concept Introduction | Relative Rusting | *skills*: observation, writing based on experimental results  
*content*: chemical indicators, galvanization, rusting reaction mechanisms |
| Concept Introduction | Working as a Lab Technician for ElectroChem Labs, Inc. | *skills*: use of standard procedures, measurement, use of typical forms  
*content*: corrosion testing in accelerated environment, corrosion terms, safety |
| Concept Application | Coatings | *skills*: use of standard procedures, data analysis  
*content*: coating control methods, m/v solution concentration |
| Concept Application | Should the Bridge Be Closed? | *skills*: extraction, filtration, dimensional analysis, using computer probe  
*content*: solution concentration, gravimetric analysis, concrete |

Refer to the Appendix for the complete unit
Chapter 2: Review of the Literature

The technology education field has undergone numerous changes in scope, mission, and teaching philosophy in its one hundred-year history. This field needs to meet the demands of the ever-changing technological society as well as keep pace with industrial expansion (11). Effectively preparing students for post-secondary technology education and the technical workplace requires a modeling of how content knowledge and general workplace skills are integrated. Bailey (12) describes how integration of both academic and industry skill standards has a positive influence of education as a whole. It is crucial for an educational approach to integrate work place readiness skills (e.g. teamwork, communication, interpersonal skills, computer skills) with content that parallels the nature of today’s work environment (11). Subsequently, the National Science Foundation (NSF) and other funding agencies have funded the development of curriculum materials which address this integration.

The Corrosion Unit utilized standards provided from three different organizations: The National Research Council (NRC), The American Chemical Society (ACS), and The American College Testing (ACT) Service who developed Workkeys in direct response to the Secretary of Labor’s Commission on Achieving Necessary Skills (SCANS) (13). The following is an overview of each of these sets of standards.
• The NRC’s National Science Education Standards (NSES) are academic benchmarks upon which all science curricula (including chemistry) are measured. Content standards are written for given grade level blocks including K-4, 5-8, and 9-12. Each block includes specifics on Science as Inquiry, Science and Technology, Science in Personal and Social Perspectives, and the three content areas of Physical Science, Life Science, and Earth and Space Science. These standards set out to “create a scientifically literate populace in which interlocking communities of teachers and students learn science” (3).

• The ACS’s Voluntary Industrial Standards (VIS) include skills and knowledge specifically needed for competency within the chemistry process and chemical technician industries (4). The VIS standards for chemical lab technicians were a principle focus of this research. These standards include: Working in the Chemical Process Industries; Maintaining a Safe and Clean Lab Adhering to Safety/Health and Environmental Regulations; Sampling and Handling Chemical Materials; Measuring Physical Properties; Performing Chemical Analysis; Performing Instrumental Analysis; Planning, Designing and Conducting Experiments; and Synthesizing Compounds.

• ACT’s service Workkeys provide criteria upon which workplace skills are measured against quantified levels of readiness required for particular jobs
(5). Categories within these Workkeys concentrate on workplace readiness skills which include: Applied Mathematics, Applied Technology, Listening, Locating Information, Observation, Reading for Information, Teamwork and Writing. It is important to note that skills specific to chemistry or chemistry technology are not included in the set of Workkeys standards. Prior to the Using Chemistry project, no instructional materials had been developed to integrate these targets into a chemistry core subject area.

Before The Corrosion Unit was developed, existing industrial-based teaching materials for the high school and two-year college levels were reviewed. The ones which provided to be of most usefulness to this project were the ACS’s Science in a Technical World (14), Science That Counts in the Workplace (15), Science in Our World Series (16), Science & Technology in Society (17), The Salter’s Chemistry Course (18), The Palette of Color Monograph Series (19), The PACT Chemical Technology Resources(series) (20) and Bringing Industry-based Science Into the Classroom (21). While these materials provided a foundation for The Corrosion Unit, the subject and use of corrosion in workplace education materials was unique.

As per objective #4 in section 1.2, The Corrosion Unit was written to implement a learning cycle approach which is based on constructivist theory.
“Constructivist theories of knowledge are based on… (this) assumption: Knowledge is constructed in the mind of the learner (22).” Students use such an approach to make sense of information as they construct their own knowledge. In *The Corrosion Unit*, with the architecture of a learning cycle established, it enables the learner to take small steps in moving forward with their learning. Here, the instructor is not a “teacher,” but rather a facilitator of learning.

Cooperative learning, defined as “students working together in groups small enough that everyone can participate in a collective task that has been clearly assigned” (23), is another key pedagogical strategy is used in this research. Cooperative learning strategies promotes higher academic achievement (24) and students that are involved in these methods reported more positive attitudes towards their classes (25).
Chapter 3: Chemistry of the Unit

3.1 Simplified View of Rusting

This chapter provides a summary of the chemical process of rusting. The Prologue for the unit (found in the Appendix) gives a more detailed accounting of this chemistry including full half reactions, step by step processes, and a discussion of the stability of ferric (Fe\textsuperscript{3+}) ion vs. ferrous (Fe\textsuperscript{2+}) ion.

Novice learners consider rusting to be the combination reaction between iron in its metallic form with oxygen, forming iron(III) oxide (ferric oxide).

\[
\begin{align*}
\text{H}_2\text{O} \\
\text{Fe(s) + O}_2\text{(g)} & \rightarrow \text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O(s)} \\
\text{Iron} & \quad \text{oxygen} \\
\end{align*}
\]

Actually, rusting is not as straightforward as the above reaction implies, nor is rust a single product. Rusting is inherently a random process (26), yet some predictability in mechanism can be established when pH and the existence of a competing species (chloride) are controlled. Subsequent sections of this chapter present what is known about these reaction mechanisms.

3.2 Rusting in the presence of oxygen at pH ≥ 5

The most prevalent case of iron rusting occurs when iron in the presence of oxygen is exposed to an aqueous solution with a pH ≥ 5. The oxygen source for this reaction is atmospheric oxygen dissolved in the water. Metallic iron (Fe\textsuperscript{0}) is oxidized to the ferrous (Fe\textsuperscript{2+}) ion at the anode. Oxygen gas (O\textsubscript{2}\textsuperscript{0}) is reduced to
oxide ion \((O^{2-})\) in the form of hydroxide anions \((OH^-)\). The ferrous ion \((Fe^{2+})\) reacts with the hydroxide \((OH^-)\) to form ferrous hydroxide \(Fe(OH)_2\) which precipitates as an intermediate product of this reaction as shown in the following equation:

\[
2Fe(s) + O_2(g) + 2H_2O(l) \rightarrow 2Fe(OH)_2(s).
\]

Note that while the insolubility of the ferrous hydroxide \((K_{sp} = 8 \times 10^{-16})\) drives the overall reaction to the right, the inherent instability of the ferrous hydroxide under these conditions causes it to further oxidize to ferric hydroxide \((27)\):

\[
4Fe(OH)_2(s) + O_2(g) + 2H_2O(l) \rightarrow 4Fe(OH)_3(s)
\]

Over time, the powdery ferric hydroxide precipitate forms the more commonly observed hydrated ferric oxide crystals as presented in the following hydration equation.

\[
Fe(OH)_3(s) \rightarrow Fe_2O_3 \cdot xH_2O(s)
\]

This hydrated crystal is considered “rust.” The degree of hydration of the iron oxide affects the color of the rust, which may vary from yellow, to the familiar reddish brown, to black \((28)\).

### 3.3 Iron oxidation in the presence of oxygen at pH < 5

A second corrosion mechanism iron can undergo occurs when oxygen is present in a more acidic aqueous environment. Two different and competing pathways are proposed within this acidic environment. Both are observed in vitro.
In both pathways the oxidation of metallic iron to the ferrous ion (Fe$^{2+}$) is identical to that discussed in section 3.2. It is the cathodic reaction that varies.

**Pathway A (no hydrogen gas formed)** At the cathode, oxygen gas (O$_2$) is reduced to oxide ion (O$^{2-}$) which reacts with the available acidic protons (H$^+$) (hydrogen cations) to form water (29). The pH of the solution will increase as this reaction proceeds forward due to the consumption of these hydrogen cations. The reaction can be summarized as

$$2\text{Fe(s)} + \text{O}_2(\text{g}) + 4\text{H}^+(	ext{aq}) \rightarrow 2\text{Fe}^{2+}(	ext{aq}) + 2\text{H}_2\text{O(l)}.$$  

The formation of water (a molecular compound) drives this reaction to the right in accordance with LeChatlier’s principle. But, the hydroxide (OH$^-$) concentration is not high enough to form insoluble Fe(OH)$_2$ as is described in pH $\geq$ 5 mechanism. Instead of forming the familiar red/brown precipitate, *only* soluble ferrous ions and water are produced which accounts for the fact that when iron is present in an acidic solution, it remains shiny, but loses significant mass over time.

**Pathway B (formation of hydrogen gas)** Unlike pathway A, it is the hydrogen cations (H$^+$) that are reduced producing hydrogen gas (H$_2$) at the cathode. This pathway is confirmed by the appearance of bubbles (30).

$$\text{Fe(s)} + 2\text{H}^+(	ext{aq}) \rightarrow \text{Fe}^{2+}(	ext{aq}) + \text{H}_2(\text{g})$$
As with pathway A, the pH of the solution rises as this reaction proceeds. The formation of hydrogen gas drives this reaction to the right in accordance with LeChatlier’s principle.

3.4 Rusting in the presence of chloride at pH ≥ 5

It is a well known fact that iron corrodes much more quickly in saline environments such as those caused by road salt or ocean spray. This observation is typically correlated to simple ionic, conductive character of a sodium chloride solution assisting in the electron exchange characteristic of an oxidation reduction reaction, but this is NOT the case. When chloride is present it participates in a favored, third possible mechanism for corrosion (31). This mechanism holds great significance in the corrosion of reinforcing steel within the concrete roadbeds subjected to deicing chemicals containing chloride (Cl⁻).

Once the ferrous ions (Fe^{2+}) are produced, they then react with available chloride anions to form a soluble intermediate iron chloride complex (FeCl₄^{2-}) (31,32) which subsequently reacts via one of the two following pathways to form insoluble ferrous hydroxide (Fe(OH)₂).

- **Pathway A (FeCl₄^{2-} to Fe(OH)₂ in the absence of Ca(OH)₂)** When calcium hydroxide (Ca(OH)₂) (or concrete) are not present, the corrosion of iron proceeds with the soluble ferrous chloride complex (FeCl₄^{2-}) reacting with moisture to form insoluble ferrous hydroxide...
(Fe(OH)$_2$), hydrogen cations (H$^+$), and chloride (Cl$^-$) anions. The reaction is summarized below.

$$\text{FeCl}_4^{2-}(aq) + 2\text{H}_2\text{O}(l) \rightarrow \text{Fe(OH)}_2(s) + 2\text{H}^+(aq) + 4\text{Cl}^-(aq)$$

**Pathway B (FeCl$_4^{2-}$ to Fe(OH)$_2$ in the presence of Ca(OH)$_2$)** This pathway occurs simultaneous to the previous pathway if calcium hydroxide (Ca(OH)$_2$) is present. The soluble ferrous chloride complex (FeCl$_4^{2-}$) reacts with calcium hydroxide (Ca(OH)$_2$) to form insoluble ferrous hydroxide (Fe(OH)$_2$), calcium cations (Ca$^{2+}$), and chloride (Cl$^-$) anions. The reaction is summarized below.

$$\text{FeCl}_4^{2-}(aq) + \text{Ca(OH)}_2(s) \rightarrow \text{Fe(OH)}_2(s) + \text{Ca}^{2+}(aq) + 4\text{Cl}^-(aq)$$

Both pathways result in the formation of solid ferrous hydroxide (Fe(OH)$_2$). As previously discussed the insoluble nature of the ferrous hydroxide drives the overall reaction to the right in accordance with LeChatlier’s principle. Also the resulting ferrous hydroxide (Fe(OH)$_2$) further oxidizes to ferric hydroxide (Fe(OH)$_3$), which then crystallizes to form the characteristic rust:

$$\text{Fe(OH)}_3(s) \rightarrow \text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O}(s).$$

The steel reinforcing bars (rebar) in a concrete roadbed are extremely susceptible to chloride (Cl$^-$) in the concrete. This is because of the chloride reactivity with the ferrous ion (Fe$^{2+}$) described herein. The consequence of this
mechanism (regardless of which pathway is followed) is profound. Once a concrete roadbed is deiced with salt, the chloride in the concrete remains for the life of the slab! Unfortunately, none of the chloride ($\text{Cl}^-$) ions involved in this accelerated corrosion process are consumed and therefore remain available to contribute to even more deterioration of the iron. Corroding rebar causes concrete to be unsafe. Because the rebar cannot be directly examined without destroying the concrete roadbed, indirect techniques must be used to determine the “health” of the rebar within concrete. These indirect techniques include X-ray, conductivity tests, and chloride ion concentration tests. The larger the chloride ion concentration in a sample of concrete, the more corrosion that is currently underway.

It should be noted that in the case of some coastal communities, the sand used to make concrete roadbeds and bridges would come from nearby beaches because importing “clean” sand was cost prohibitive. In this concrete, the chloride ion ($\text{Cl}^-$) was present from the onset thus causing the underlying rebar to begin corroding immediately. It is not surprising that structures made from this defective concrete failed quickly. In Florida, the initial attempt to correct this problem was to use stainless steel rebar at a tenfold increase in price. Unfortunately, the stainless performed only marginally better than the standard. Another attempt to alleviate this problem was to pre-wash the sand to remove the salt prior to mixing the concrete. This proved difficult in large scale. Currently,
the state of Florida uses epoxy coated rebar which seems to be performing well. Additionally, the state is experimenting with various polymer coatings as well (33).

3.5 Chemical indicators

This section provides a description of the various tests which were used in Relative Rusting, to determine the identity of ions in solution.

• **Test for the ferrous (Fe<sup>2+</sup>) ion:** The indicator potassium ferricyanide (K₃Fe(CN)₆) forms a deep-blue in the presence of the ferrous ion (Fe<sup>2+</sup>). The colored precipitate, commonly known as Prussian Blue, was formed in the following series of reactions:

\[
\begin{align*}
\text{Fe}^{2+} + [\text{Fe(CN)}_6]^{3-} & \rightarrow \text{Fe}^{3+} + [\text{Fe(CN)}_6]^{4-} \\
4\text{Fe}^{3+} + 3[\text{Fe(CN)}_6]^{4-} & \rightarrow \text{Fe}_4[\text{Fe(CN)}_6]_3 \text{ (deep blue precipitate)}
\end{align*}
\]

• **Test for the ferric (Fe<sup>3+</sup>) ion:** The indicator potassium thiocyanate (KSCN) was used to determine the presence of ferric ion (Fe<sup>3+</sup>). The two species react to produce a deep blood red, water-soluble complex as per the following reaction:

\[
\begin{align*}
\text{Fe}^{3+} + \text{SCN}^- & \rightarrow \text{FeSCN}^{2+}
\end{align*}
\]

• **Test for the hydroxide (OH⁻) ion:** Phenolphthalein indicator was used to denote the presence of hydroxide (OH⁻) by turning a pink color.
Chapter 4: Development of the Unit

4.1 Sequence

Contained in this chapter is a discussion of the global philosophy of the development of the unit, followed by a discussion of the general sequence of events encountered during the development of this unit, and finally a discussion of the details encountered during the development of The Prologue and each individual activity.

Prior to the author’s involvement in the project, A.M. Sarquis developed a corrosion unit overview for inclusion in the NSF proposal that subsequently funded the project. Two very experienced high school teachers with extensive knowledge of corrosion chemistry were also included prior to the author’s involvement. These contributors, Bleam (35) and McKinney (36) provided a vision of the unit and rough drafts of several of the activities. In the spring of 2002, their efforts were advanced to produce the prototype Using Chemistry unit on corrosion. The Corrosion Unit was the first of five such units under development, as a result, several unanticipated issues which surfaced were addressed, and subsequently incorporated into the project development template for future Using Chemistry units.

The instructional materials design protocol given to all authors in the Using Chemistry project incorporated the Wiggins’ and McTighe’s...
backward design process (37). Desired learning objectives and assessment strategies are developed first, followed by the design of activities that coincide with previously decided student learning objectives. In the case of the whole Using Chemistry project, focus was made on providing enduring understanding and important knowledge to the students. Specifically for The Corrosion Unit the enduring understanding included a knowledge of how corrosion is a natural process that is continuously occurring destroying metals used in products such as cars, roads and bridges. The important knowledge included using standard operating procedures for conducting laboratory work; chemistry content from the NSES and VIS including the basics of corrosion, oxidation-reduction, and the role of galvanization and other coatings in preventing corrosion; and workplace readiness skills.

As per the fourth objective of this research (see page 3), application of the constructivism mandated the inclusion of a learning cycle and cooperative learning using appropriate real life scenarios within activities. Because the unit was intended for integration into existing curricula, the activities were constructed to stand-alone. A reproducible student handout was provided for each activity as well as a comprehensive set of instructor notes. The student handout presented a challenge to the student via a short scenario labeled “The Problem” and brief statement of “Your Task.” After a list of needed materials was provided, students were then given a
“Procedure” to guide their work and tasks to complete as an “Assessment” of their learning. The instructor notes included a description of the “Time Required,” an outline of “Key Science, Mathematics, and Engineering Concepts” covered in that activity, a discussion of the “Prior Concepts and Skills Needed,” an expanded “materials list,” information on “Safety and Disposal,” suggestions for “Getting Ready,” “Procedure Notes,” “Assessment,” “Explanation,” “Extension Activities,” and detailed correlation to standards and appropriate references. Both documents (student handout and instructor notes) are included for each of the eight activities found in the Appendix.

The focus of the unit (corrosion), intended audience (high school & two-year college chemistry, and chemistry technology), and targeted standards to be incorporated (NSES, VIS, ACT Workkeys) were determined prior to the research reported in this thesis (8). Because the ACT Workkeys standards had not previously been correlated to chemistry, particular attention was paid to doing so. The method developed for doing this subsequently provided guidance both to the Document Production department of the CCE and to authoring teams for other Using Chemistry modules.
Work reported herein began with reviewing early outlines and thumbnail sketches, identifying gaps in the standards-based skills and content, and planning the modification of existing activities or developing new activities to address these deficiencies. It was critical to view standards from two perspectives: how they supported or built the enduring understandings previously identified for the unit, and how they developed the detailed underlying knowledge and skills needed for the individual activity. The correlation of standards to an activity is not accomplished in a single attempt; it is a cyclical revision process.

Once activities were drafted and tested by the author, the activities were then subjected to “microtesting” utilizing a target audience end user of the material according to the CCE Continuous Quality-Control Materials Development Protocol (7). Storer points out several advantages of the microtesting methodology:

1. The tester cannot rely on others to perform the task.
2. The author can observe misinterpretations of the written document which might be overlooked in a full-scale classroom test of the activity.
3. The author may observe that the procedure is not as clear in action as it is on paper.
4 Through the debriefing, the participants can provide valuable insights and suggestions to clarify the written document.

A student or group of students from Madeira High School (38) were chosen to test alpha-draft versions of all the activities from *The Corrosion Unit*. Students that were involved were both first-year and second-year high school chemistry students at various levels of proficiency. In this research, Storer’s third and forth advantage were particularly relevant. In the testing of the eight activities, students regularly provided significant intuition as to the best way to clarify different portions of the written documents. The activities were then reworked based on this feedback.

After microtesting was complete and a beta-draft of the unit was developed, it was workshop tested with high school teachers during an academy in December 2002. Teachers provided feedback during the initial academy presentation and these changes were integrated. Next, these teachers utilized selected activities in their own classrooms and reported back with follow-up comments including their student insights as well.

With this feedback, *The Corrosion Unit* was again refined and a presented at a second teacher academy held in July 2003. Suggestions from
teacher reviewers from this event were similarly implemented into the document.

The following sections provide a summary of the development process for each of the sections in *The Corrosion Unit* which is found in its entirety in the Appendix.

### 4.2 Development of The Prologue

The Prologue to the corrosion unit provides a mechanistic discussion of the corrosion of iron. This prologue was created in response to requests from teacher reviewers for such background information.

### 4.3 Development of Activity 1: Web Research: What’s Up with Corrosion?

The first activity in *The Corrosion Unit*, Web Research: What’s Up with Corrosion was created as an awareness activity to allow students to make connections between experiences they have previously had with rusting and to simulate their interest in the lesson. Corrosion has a significant impact on society, and this activity frames the ubiquitous nature of corrosion and its economic impact. Teams of students use the web to research an individual portion of the assignment. Using standard methodology, teams then develop and give an oral presentation to the whole
class. This idea of teamwork using standard methods sets the tone for the use of standard procedures, which are further developed and strengthened later in the unit.

As a prerequisite to learning about corrosion, students must understand its importance. The following web site, www.corrosioncost.com was chosen as the springboard for this activity. Several considerations were made in its selection: online resources offer engaging, interactive presentations and as a federally funded site, it would most likely be sustained. Additionally, the site was robust enough to provide information for multiple levels of instruction, yet easy to understand and navigate. Even so, the site provided important chemical insights for students’ future study of corrosion chemistry and corrosion control methods, such as coatings and corrosion inhibitors (39).

4.4 Development of Activity 2: Corrosion Collapse

A second awareness activity, **Corrosion Collapse**, is included in the unit to reinforce the importance of corrosion of bridges. The activity begins with the students viewing an eleven-minute video clip produced by the Discovery Channel (40) graphically showing the catastrophic bridge collapse of The Great Silver Bridge of Point Pleasant West Virginia (1967). Students made observations, took notes, and then completed a quiz. This
activity proved to be one of the most popular among teachers and students since it provided a highly dramatic, real-life consequence of undetected corrosion.

This activity addressed several ACT Workkeys standards including listening, observation, and note taking (5). In both the December 2002 and July 2003 teacher academies, corrosion and structural expert Professor Gary Drigel elaborated on the cause of the structural failure. His presentation addressed the concept of corrosion fatigue and the subsequent repercussion of the dramatic bridge collapse (33). Much of this information was subsequently included in the instructor notes portion of the activity.

4.5 Development of Activity 3: The Hungry Flask

Activity 3, The Hungry Flask, is the first of the exploration activities in the unit. This activity provided a simplified understanding of the rusting process which would be developed further, later in the unit.

Students conducted an experiment where they observed rusting occurring over a very short period of time. A piece of steel wool pretreated in vinegar, is transferred to an erlenmeyer flask and a balloon is placed on the mouth of the flask. Students record their observations, answer guided questions, and discuss their answers to better understand the underlying
phenomenon. Similar to other activities in this unit, depth of discussion can be tailored to the needs of particular classes.

This activity was in the CCE archives and originally published in *Science Scope* (41), then modified for an unpublished cooperative effort with Armco Steel (42). For *The Corrosion Unit*, the format, strategy, standards alignment, and procedural details were all modified to better meet the goals of *Using Chemistry*. When given to a panel of teachers for critical evaluation, even a well-tested activity can improve.

### 4.6 Development of Activity 4: The Reactivity of Metals

A second exploration activity is also included in the unit, Activity 4, **The Reactivity of Metals**, which emphasizes data analysis skills and the identification of chemical trends which are subsequently applied to the determination of the metal activity series.

In this activity, students conducted twenty separate chemical reactions on five different metals to determine their relative reactivities in single replacement reactions. These reactions were consistent with the acidic pathway in the corrosion process of metals. It might be surprising that iron, the ubiquitous construction metal showed high reactivity, but, in fact, it did. Of course, coinage metals do not possess the requisite strength, availability,
or appropriate cost for large-scale use in construction. The fact that students observed iron rapidly corroding, provided a fundamental understanding of the importance of protecting iron from conditions which promote corrosion.

The metal activity series is a common investigation performed at the high school level (43). The Reactivity of Metals included in this unit has two significant changes over what is typically done: 1) A simple integer was designated to each metal based on the number of reactions that it underwent. This allowed for an easy method of ranking the items in the set. 2) Metallic iron was included in the series used in this activity. It is important to note that iron is not included in the vast majority of metal activity series schemes, because it does not readily react with silver nitrate. It is suspected that this non-reactivity is due to corrosion inhibitor properties of trace elements (copper, sulfur, manganese, or phosphorus) present in the cold rolled steel (33).

To solve the problem of the apparent non-reactivity of iron in the series, a search was undertaken for an iron source that would react as predicted. The solution was to use iron filings attached to a magnetized iron wire. This source provided a positive reaction in a visually dramatic way. An additional but more commonly known challenge with the investigation was the tendency of iron hydroxide to precipitate in neutral or basic
solutions of ferrous sulfate as ferrous or ferric hydroxide. This problem was overcome by working at low pH which keeps the ferrous ion in solution.

Teachers found that the flexibility of including extension activities in The Reactivity of Metals eased its placement in their curriculum. When teaching single replacement reactions, teachers could use this activity as an introduction to the idea of activity series, or as a corollary, teachers might use it as a follow-up activity to confirm the classroom theory. The activity could also be extended to include half-cell reactions, the calculation of cell potential, or even Gibb’s free energy. The teachers also appreciated the application to “real world” problems with steel as a building material, as compared to traditional “esoteric” chemicals typically used in such an instructional scheme.

In summary, elementary chemical principles such as the involvement of oxygen in a corrosion redox reaction and iron’s surprising relative reactivity were explored in Activity 3 and Activity 4. These activities laid the foundation for the concept introduction phase which follows.

4.7 Development of Activity 5: Relative Rusting

The first of two concept introduction activities, Relative Rusting, explored the diverse pathway in which iron corrodes. The activity was
placed at the point in the unit, because successful completion was deemed commensurate with a working knowledge of the concept of iron corrosion which was needed before further, more sophisticated investigations could be undertaken.

This activity was divided into two parts to help reinforce the fact that iron rusts via multiple pathways, and to examine this process in various media. Part A presents the three different mechanisms through which iron rusts by exposing iron strips to various corrosive environments and allowing these samples to sit overnight. Through observations, guided questions, and an instructor-guided discussion, students determined that there are different chemical mechanisms involved. The role galvanic protection plays in the overview of iron rusting is explored in Part B. Both galvanized and native strips of iron are embedded in a specially designed corrosive environment set up in agar doped with a mixture of sodium nitrate and indicators to allow the identification of various products of this corrosion.

4.8 Development of Activity 6: Working as a Lab Technician for ElectroChem Labs, Inc.

Activity 6, Working as a Lab Technician for ElectroChem Labs, Inc., the second of the concept introduction phase activities, is also first of three to incorporate standard operating procedures modeled after American
Society for Testing and Materials (ASTM) Standards (44,45). In this activity, students are given a scenario in which they work as teams of chemical technicians using imposed industrial standards to examine variables that affect the rate of corrosion. Over the term of the exercise, students follow standard operating procedures to test samples of untreated steel in two different accelerated corrosion environments. Student teams quantify their experimentation by utilizing authentic practices of both the Mass Loss Method and the Area Coverage Method of evaluating corroding surfaces. The activity concludes with students doing both written and oral reports of their findings.

Throughout this activity, the instructor played the role of lab manager, assigning testing environments using industry standard forms, monitoring the teams’ progress throughout the students’ research and assessing the teams’ reports.

4.9 Development of Activity 7: Coatings

Activity 7, Coatings challenges students to apply what they have previously learned about the corrosion of iron and how to follow standard operating procedures to the task of evaluating protective coatings. This activity builds directly on the skills acquired in the previous activities. This activity was included as the first of the concept application exercises,
because it allows students to apply concepts and skills they have begun to master.

In this activity, student teams investigate the effectiveness of various non-metallic coatings in preventing corrosion. Student teams quantify their experimentation by utilizing the Scribe Creepage and the Area Coverage Method of evaluating corroding surfaces modified from ASTM standards (44, 46, 47). As in the previous exercise, the activity concludes with students doing both written and oral reports of their findings and the instructor continued to play the role of lab manager throughout.

4.10 Development of Activity 8: Should the Bridge Be Closed?

The second concept application and final activity in The Corrosion Unit is Should the Bridge Be Closed? which mimics an actual research problem that a chemistry technician in the corrosion industry might undertake. It was developed in the concept application stage of the learning cycle where students utilize significantly more advanced standard operating procedures that incorporate complex analytical techniques to determine what if anything should be done about a fictitious bridge found to have extreme spalling of its concrete roadbed. In their investigation to answer the question at hand, student teams perform analyses based on real corrosion testing protocols (47,48,49,50) including testing concrete samples using redundant
techniques of gravimetric determination and a chloride-ion specific electrode. Teams statistically compared their data between these two techniques.

The development challenge that existed with this activity was to provide appropriate testing protocols that reflected actual practice and still be accomplished at the targeted grade levels. Reinforcing bars of steel, “rebar” are placed within concrete to improve the concrete’s performance. In the industry, rebar is typically not directly examined as this would destroy the concrete roadbed, instead indirect techniques such as determining the chloride ion concentration, are used. The inference being the more chloride that was present in a sample of concrete, the greater the hidden corrosion of the underlying rebar. One of the methods selected to determine the chloride ion concentration in this activity was to use a chloride specific ion probe. This method is used by The New Mexico State Highway and Transportation Department during their “on the site” work. Although not as accurate as the laboratory tests using potentiometric methods, the results of the chloride probe are reported to be perfectly adequate for this purpose (52). The Federal Highway Agency traditionally uses a potentiometric titration to determine the chloride ion concentration, but this method was too complex for a typical high school/introductory college laboratory. A second chloride ion concentration method was included to allow cross-interpretation of data.
A common silver chloride gravimetric analysis was used for this purpose. It should be noted that the standard ASTM procedure for chloride ion concentration was with the use of a chromate titration (48). Due to the toxicity of chromate, and its prohibition from many high schools it could not be used.

An additional improvement was made in the implementation of mathematical procedures in this activity. The ASTM protocol (48) from which mathematical procedures were based did not detail how and why certain values were related to each other. Procedures often had technicians multiplying or dividing obtained data by unitless values. To help future lab technicians understand why they were performing particular procedures the mathematical analysis portions of the standard procedures were enhanced to include descriptions of the underlying meaning of the mathematical operations. (Gram to mole conversion, mole relationships from the balanced equation, etc.)
Ch 5 Conclusions

5.1 Dissemination

The rationale for undertaking the research contained in this thesis was to provide high school and two-year college chemistry educators with instructional materials they could use to better prepare their students assume jobs in today’s highly technical workforce. As a result of the imbedded charge for dissemination of the product of this research, the activities that were developed were shared with teachers via two hands-on teacher workshops sponsored by The Center for Chemistry Education Dec 6-7, 2002, and July 7-10, 2003. The final corrosion unit (see Appendix) was shared with the chemistry education community through an invited presentation as a part of the symposium on Developments in Chemical Technician Education, September 10, 2003, at the 226th American Chemical Society National Meeting, New York, NY (53). Additionally, this unit has subsequently been revised and incorporated by CCE staff into a large collection of Using Chemistry modules and are being distributed through Terrific Science Press. These additional modules include the topics of skin care, copper mining, flavors, and forensics.

5.2 Impact and Significance

Implementation of The Corrosion Unit into the high school or two-year college chemistry curriculum can provide a means of linking chemistry
content to workplace skills and while providing contextual links to students’ previous experiences. Additionally, immersion of students into real life corrosion problems explored with simulated ASTM protocols imploring high technology and analytical techniques provides marketable skills for future careers in the technological workplace.

This research project has involved countless individuals both teachers and students from around the country. Because of their involvement (both teachers and students) were not only introduced to the science of the unit, but also to various aspects of conducting a research project.

The CCE publication of the Using Chemistry modules (for which this research provided a foundational piece) provides teachers of two-year colleges and high school chemistry with a well-tested, industry connected approach to teaching chemistry content. The methods used in the development of this instructional unit can serve as a model for similar projects in the future.
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Appendix
Corrosion Activities
The chemical process of rusting is typically considered to be the combination reaction between iron in its metallic form with oxygen, forming iron (III) oxide (ferric oxide). Further research reveals that rust is not a single product, nor is its production as straightforward as initially appearing. This section will outline the various mechanisms iron will undergo in its process of rusting. It begins with the greatly simplified equation presented below.

\[
\begin{align*}
\text{Fe(s)} + \text{O}_2(g) & \rightarrow \text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O(s)} \\
\text{Iron} & \quad \text{oxygen} & \quad \text{rust}
\end{align*}
\]

This is an oxidation/reduction reaction where iron is losing electrons to oxygen. The iron is being oxidized and the oxygen is being reduced. Water and oxygen are both involved in this reaction; iron does not rust in water that has no oxygen. Likewise, iron will not rust in oil, even if it is saturated with oxygen.

Rusting is inherently a random process, yet some predictability in mechanism can be established when pH and a competing species (chloride) are controlled and examined.

The following provides an overview of the three mechanisms that may occur during the rusting process. The environment in which the rusting occurs controls the mechanism of the process. Significant differences are noted dependent on pH, and the existence of chloride.

I. Rust formation in the presence of oxygen in a neutral or basic environment

This mechanism is the typical one observed when encountering iron oxidation.

At the anode, metallic iron (Fe\(^0\)) is oxidized to the ferrous (Fe\(^{2+}\)) ion via the loss of electrons as per the following oxidation half-reaction:

\[
4\text{Fe(s)} \rightarrow 4\text{Fe}^{2+}(\text{aq}) + 8\text{e}^-
\]

---

\(^1\) unless chloride is present
At the cathode, oxygen gas ($O_2^0$) in the presence of water is reduced to $O^{2-}$ in the form of hydroxide anions.

$$8e^- + 2O_2(g) + 4H_2O(l) \rightarrow 8OH^-(aq)$$

The oxygen available for this reaction is atmospheric oxygen dissolved in the water.

*Adding the oxidation and reduction half reactions gives*

$$4Fe(s) + 8e^- + 2O_2(g) + 4H_2O(l) \rightarrow 4Fe^{2+}(aq) + 8e^- + 8OH^-(aq) \rightarrow 4Fe(OH)_2(s)$$

The reaction can be summarized as

$$4Fe(s) + 2O_2(g) + 4H_2O(l) \rightarrow 4Fe(OH)_2(s)$$

(note this is an intermediate step in the process so the stoichiometry coefficients are not yet reduced)

The above reaction shows the precipitation of ferrous hydroxide as the ferrous cations combine with hydroxide anions in the aqueous solution. Note that the insoluble nature of the ferrous hydroxide ($K_{sp} = 8 \times 10^{-16}$) drives the overall reaction to the right.

As noted above, ferrous hydroxide is an intermediate product. It is unstable in water, and will, in time, further oxidize to ferric hydroxide as shown in the following reaction:

$$4Fe(OH)_2(s) + O_2(g) + 2H_2O(l) \rightarrow 4Fe(OH)_3(s)$$

Over time, the powdery ferric hydroxide precipitate forms the more commonly observed hydrated ferric oxide crystals via the following reaction.

$$Fe(OH)_3(s) \rightarrow Fe_2O_3 \cdot xH_2O(s)$$

This hydrated crystal is considered “rust.” The degree of hydration of the iron oxide affects the color of the rust, which may vary from yellow, to the familiar reddish brown, to black.
II. Iron oxidation in the presence of oxygen in an acidic environment

The second mechanism iron can undergo in the process of corrosion occurs when oxygen is present in an acidic environment. Two different and competing pathways are proposed within this acidic environment. Both are observed in vitro. Stoichiometric ratios have not been reduced to be consistent with the first mechanistic pathway.

Pathway A
As before, at the anode metallic iron \((\text{Fe}^0)\) is oxidized to the ferrous \((\text{Fe}^{2+})\) ion via the loss of electrons as per the following oxidation half reaction.

\[
4\text{Fe}(s) \rightarrow 4\text{Fe}^{2+}(aq) + 8e^-
\]

The first significant difference from the basic/neutral mechanism is observed in this next step. At the cathode, oxygen gas \((\text{O}_2^0)\) is reduced via the consumption of electrons to \(\text{O}^2-\). Acidic protons \((\text{H}^+)\) (hydrogen cations) are added to this \(\text{O}^2-\) forming water.

\[
2\text{O}_2(g) + 8\text{H}^+(aq) + 8e^- \rightarrow 4\text{H}_2\text{O}(l)
\]

The oxygen available for this process is typically atmospheric oxygen which has dissolved in the acidic solution. It should be noted that the pH of the solution will increase as this reaction proceeds forward due to the consumption of these hydrogen cations.

Adding the oxidation and reduction half reactions gives

\[
4\text{Fe}(s) + 8e^- + 2\text{O}_2(g) + 8\text{H}^+(aq) \rightarrow 4\text{Fe}^{2+}(aq) + 4\text{H}_2\text{O}(l) + 8e^-
\]

The reaction can be summarized as

\[
4\text{Fe}(s) + 2\text{O}_2(g) + 8\text{H}^+(aq) \rightarrow 4\text{Fe}^{2+}(aq) + 4\text{H}_2\text{O}(l)
\]

The above reaction shows metallic iron, oxygen, and hydrogen cations react to form soluble ferrous ions and water. It should be noted that the formation of water (a molecular compound) drives this reaction to the right in accordance with LeChatlier’s principle. Unlike the previous neutral and basic pathway, \(\text{OH}^-\) concentration is not high enough to form insoluble \(\text{Fe(OH)}_2\). In a radical departure from the basic/neutral mechanism, instead of forming the familiar red/brown precipitate, only soluble ferrous ions and water are produced. When iron is present in an acidic solution, it remains shiny, but loses significant mass
over time. Additionally, the pH of the solution rises.

**Pathway B**
As before, at the anode metallic iron (Fe$^0$) is oxidized to the ferrous (Fe$^{2+}$) ion via the loss of electrons. This oxidation half reaction is identical to Pathway A.

$$4\text{Fe}(s) \rightarrow 4\text{Fe}^{2+}(aq) + 8e^-$$

At the cathode, the hydrogen cations (H$^+$) are reduced via the consumption of electrons producing hydrogen gas (H$_2^0$).

$$8\text{H}^+(aq) + 8e^- \rightarrow 4\text{H}_2(g)$$

This pathway is consistent with the appearance of bubbles, and is seen in examples such as Mg in HCl, Zn in H$_2$SO$_4$, and countless others. As before, the pH of the solution rises as this reaction proceeds.

*Adding the oxidation and reduction half reactions gives*

$$4\text{Fe}(s) + 8\text{H}^+(aq) + 8e^- \rightarrow 4\text{Fe}^{2+}(aq) + 8e^- + 4\text{H}_2(g)$$

The reaction can be summarized as

$$4\text{Fe}(s) + 8\text{H}^+(aq) \rightarrow 4\text{Fe}^{2+}(aq) + 4\text{H}_2(g)$$

In the above reaction, the iron metal combines with hydrogen cations to form soluble ferrous ions and hydrogen gas. The formation of hydrogen gas drives this reaction to the right in accordance with LeChatlier's principle.

**III. Rust formation when chloride is present in a neutral or basic environment**

The third mechanism to be discussed includes the accelerated corrosion pathway observed in saline environments. It is readily observed that iron corrodes much more quickly in saline environments such as those caused by road salt or ocean spray. This observation is typically correlated to simple ionic, conductive character of a sodium chloride solution assisting in the electron exchange characteristic of an oxidation reduction reaction. This is NOT the case. When chloride is present it participates in a favored, alternative mechanism for corrosion. This holds great significance in the corrosion of reinforcing steel within the concrete roadbeds subjected to deicing chemicals containing Cl$^-$. 
As in each case presented thus far, at the anode metallic iron (Fe⁰) is oxidized to the ferrous (Fe²⁺) ion via the loss of electrons as per the following half reaction.

\[
\text{Fe}(s) \rightarrow \text{Fe}^{2+}(aq) + 2e^-
\]

At the cathode hydrogen cations are reduced to hydrogen gas via the consumption of electrons in this reduction half reaction².

\[
2\text{H}^+(aq) + 2e^- \rightarrow \text{H}_2(g)
\]

Adding the oxidation and reduction half reactions gives

\[
\text{Fe}(s) + 2\text{H}^+(aq) + 2e^- \rightarrow \text{Fe}^{2+}(aq) + 2e^- + \text{H}_2(g)
\]

The reaction can be summarized as

\[
\text{Fe}(s) + 2\text{H}^+(aq) \rightarrow \text{Fe}^{2+}(aq) + \text{H}_2(g)
\]

The oxidized iron (Fe²⁺) then reacts with available chloride anions to form a soluble intermediate iron complex (FeCl₄⁻²).

\[
\text{Fe}^{2+}(aq) + 4\text{Cl}^-(aq) \rightarrow \text{FeCl}_4^{2-}(aq)
\]

Insoluble ferrous hydroxide (Fe(OH)₂) will then directly form via one of two different mechanisms. The deciding factor is whether or not calcium hydroxide, Ca(OH)₂ (a component of concrete) is present.

**Pathway A (without concrete or Ca(OH)₂ present)**

The soluble ferrous chloride complex (FeCl₄⁻²) then reacts with moisture to form insoluble ferrous hydroxide, hydrogen cations and chloride anions.

\[
\text{FeCl}_4^{2-}(aq) + 2\text{H}_2\text{O}(l) \rightarrow \text{Fe(OH)}_2(s) + 2\text{H}^+(aq) + 4\text{Cl}^-(aq)
\]

² One might suspect an alternative oxygen/water reduction pathway to occur additionally, but this is NOT observed.

\[
4e^- + \text{O}_2(g) + 2\text{H}_2\text{O}(l) \rightarrow 4\text{OH}^-(aq)
\]

It should be noted that hydrogen cations are generated in the formation of Fe(OH)₂ below in Pathway A. This provides the preferential hydrogen cation reactant as required by the actual reduction half reaction.
**Pathway B (within a concrete slab)**

This pathway occurs simultaneous to the previous pathway if concrete (or calcium hydroxide) is present. The soluble ferrous chloride complex (FeCl$_4^{2-}$) then reacts with calcium hydroxide to form insoluble ferrous hydroxide, calcium cations and chloride anions.

$$\text{FeCl}_4^{2-}(aq) + \text{Ca(OH)}_2(s) \rightarrow \text{Fe(OH)}_2(s) + \text{Ca}^{2+}(aq) + 4\text{Cl}^-(aq)$$

The two reactions above show the precipitation of ferrous hydroxide as the ferrous chloride complex combines with either water or calcium hydroxide. Note that the insoluble nature of the ferrous hydroxide (its $K_{sp}$ is $8 \times 10^{-16}$) drives the overall reaction to the right in accordance with LeChatlier's principle.

*As noted above, ferrous hydroxide is an intermediate product. It is unstable in water. If oxygen is available for further oxidation, this inherently unstable ferrous hydroxide will with time further oxidize to ferric hydroxide as shown in the following reaction:*

$$4\text{Fe(OH)}_2(s) + \text{O}_2(g) + 2\text{H}_2\text{O}(l) \rightarrow 4\text{Fe(OH)}_3(s)$$

As before, over time, the powdery ferric hydroxide precipitate forms the more commonly observed hydrated ferric oxide crystals via the following reaction.

$$\text{Fe(OH)}_3(s) \rightarrow \text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O}(s)$$

As before, this hydrated crystal is considered "rust." The degree of hydration of the iron oxide affects the color of the rust, which may vary from yellow, to the familiar reddish brown, to black.

Moisture is required not only to support the cathodic reactions, but also to enhance the electrical conductivity. This third mechanism explains the enhancement of corrosion in a chloride environment in comparison to that of a similar ionic concentration without chloride present.

Unfortunately, none of the chloride ions involved in this accelerated process are consumed and therefore remain available to contribute to more corrosion. The consequence of this is profound. Once a concrete roadbed is deiced with salt, the chloride in the concrete remains for the life of the slab!

The steel reinforcing bars (rebar) in a concrete roadbed are extremely susceptible to chloride in the concrete. Corroding rebar causes concrete to be unsafe. Because the rebar cannot be directly examined without destroying the concrete roadbed, indirect techniques must be used to determine the “health” of
the rebar within concrete. These indirect techniques include X-ray, conductivity tests, and chloride ion concentration tests. The larger the chloride ion concentration in a sample of concrete, the more corrosion that is currently underway.

It should be noted that in the case of some coastal communities, the sand used to make concrete roadbeds and bridges would come from nearby beaches. In this concrete, the chloride ion would be present initially, and would corrode the underlying rebar from the beginning. Structures made from this defective concrete would fail quickly. In the case of Florida, the initial attempt to correct this problem was to use stainless steel rebar at a tenfold increase in price. Unfortunately, the stainless performed only marginally better than the standard. Another attempt to correct this problem was to wash the sand (prior to mixing the concrete) to remove the salt. This proved difficult in large scale, and importing “clean” sand was cost prohibitive. Currently, the state of Florida uses epoxy coated rebar and this seems to be performing well. Additionally, they are experimenting with various polymer coatings as well.

References


http://www.tfhrc.gov/////structur/corros/history.htm


Personal Communication Gary Drigel, Professor in Engineering Technology, Miami University, Middletown Campus.
The Problem
The corrosion of materials in society affects many aspects of your life. You trust your life that the stadiums, concert arenas, bridges, and buildings which you use have not been subjected to structurally damaging corrosion. It is time to learn more about the cost to control its potentially devastating effects.

Your Task
As a team, prepare a brief computer supported, oral presentation on how corrosion affects a category of society.

Materials
Per group
• minimum 1 computer with internet browser and presentation software

Per class
• computer projector

Procedure
Part A: Background Research
The overall goal of this activity is to understand the pervasiveness of corrosion on many aspects of your life. You will work in teams to research, discuss, and develop a presentation that describes the impact of corrosion in a particular category of society. Additionally, you will address its financial impact. You will NOT need to define corrosion or discuss the chemistry of corrosion. These topics will be addressed later in the unit. Your instructor will select which category of society you will study. You are to look at the appropriate web site as source material for addressing that category. Your presentation should concentrate on addressing answers to the following questions:

What areas of our economy are affected by corrosion?

What is the importance of the above area to our society? (This is not directly addressed, you need to base this on your experience)

What are the individual costs of corrosion in each sector, and the total cost in the category?
**Navigation Notes:** Upon opening your site you will see a list of folder tabs across the top, corresponding to the various categories of study: utilities, transportation, infrastructure, etc. The specific folder of your assigned category should be open. This page provides generic information. Within this folder, listed on the right side, are individual sectors addressing specific aspects within the category. These should be investigated as well. In some cases you can download a large pdf file that describes a sector in significantly more detail than the site. Considering this, do not attempt to address every aspect of a category or sector.

One group will be assigned *Corrosion Control Methods*. This presentation does not follow the above standards. See below for specific instructions for that category.

One group will be assigned *Career Comparison*. This presentation does not follow the above standards. See below for specific instructions for that category.

Utilities

http://www.corrosioncost.com/utilities/index.htm

Transportation

http://www.corrosioncost.com/transportation/index.htm

Infrastructure

http://www.corrosioncost.com/infrastructure/index.htm

Government


Production and Manufacturing

http://www.corrosioncost.com/prodmanu/index.htm
If you are assigned the category of **Corrosion Control Methods**, there is a different set of questions to guide you in formulating your presentation.

Provide brief descriptions of the different methods/techniques used to battle corrosion.

How do they work?

What do the above methods/techniques cost to implement?

**Navigation Notes:** In addition to the previous navigation notes, in your presentation, do not consider the sectors of research and development, corrosion control services, and education and training. Once again, do not attempt to address every aspect of a category or sector.

Corrosion Control Methods


If you are assigned the category of Career Comparison, there are different guidelines you are to use in formulating your presentation.

Compare a wide variety of careers related to corrosion. In your presentation, include lab technicians, field technicians, and engineers. Compare the amount of education, training and working conditions typical of these careers. Compare the different organizations that are offering these jobs (engineering firms, government, painting contractors etc and salaries. To get started, search nationwide job ads. The National Association of Corrosion Engineers (NACE International) has a career center online at [http://www.nace.org](http://www.nace.org)

Part B: The Presentation:

As a team, prepare and present a brief presentation of your findings. Your presentations need to include:

1. five slides with at least one illustration (clip art / photo / original sketch / graphic) on each

2. accurate information
3. zero grammatical or spelling errors

4. equal contribution from team members

5. responses to all questions, but integrated into your presentation (not a question-answer format)

6. an appropriate elaboration to create interest

7. approximately 10 minutes of information

Assessment

Class Presentation Rubric

<table>
<thead>
<tr>
<th>Maximum Points</th>
<th>Item</th>
<th>Points earned</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>Required number of slides</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Accuracy</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Appearance/Illustrations</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Equal member contribution</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Length requirement</td>
<td></td>
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</tbody>
</table>
Instructor Notes

Web Research: What’s Up With Corrosion?

This is the first activity in this module, and is designed to give students an appreciation of the impact corrosion has on society. Each student, working as part of a team, will research a given portion of the assignment, coordinate this with their team, and then present to the class. By requiring students to present their work in a standard but creative way, students will be oriented to the idea of using their best creative talents within a framework of standard procedures. Encouraging students to be active learners at the beginning of this module sets the tone for later work and in this manner, students introduce to each other the concept and importance of corrosion. To the teacher, contained elsewhere is The Prologue detailing the specific chemical mechanisms of iron corrosion. To the students, the specific chemistry of corrosion will be addressed in later activities.

Time Required

Setup: none
Research: 50 minutes for students to gather information from website
Design: 50 minutes for students to prepare Microsoft PowerPoint® presentation
Presentations: 10 minutes per group
Cleanup: none

Key Science Concepts

Chemistry
• anodic protection
• chemical reactions
• reaction rates

Other Concepts
• economic impact of corrosion on society
• inhibition

Engineering and Technology
• protective steel coatings

Prior Concepts and Skills Needed

Students should have a familiarity with the concept of chemical changes and that what is observed on the macro level is based on interactions at the atomic level. Students should understand internet navigation and also have familiarity with Microsoft PowerPoint® or other appropriate presentation software.
Materials

Per group
• minimum one computer with internet browser and presentation software

Per class
• computer projector

Getting Ready

Instructors should review the viability of the web sites discussed in this activity prior to implementation. They are maintained by a third party and the author makes no claims as to their accuracy or availability.

If students are not familiar with electronic presentation software such as Microsoft PowerPoint®, a primer should be given.

Students should be divided into groups of four and these groups should remain intact for the duration of this module.

Procedure Notes

Each team is assigned a web site to study, which explores the economic impact of how corrosion affects specific industries. One group is assigned a unique site that discusses corrosion control methods. Their questions and presentation is in a slightly different methodology, as outlined in the student copy of this activity.

The teams then prepare brief presentations for the class. The instructor is to provide technical support and clarify the assignment as necessary, but the students are to be active participants in the formulation of their presentation.

In this activity, students develop for themselves a “picture” of corrosion. They gain an appreciation of the magnitude of the problem and some of the solutions that technology provides. Students also begin to understand the nuances that group work involves and the importance of presenting within the framework of standard expectations.

Explanation

Whether it is with their bicycle, car or wagon, corrosion is so common that students will likely have some familiarity before they enter the classroom. The chemical process of rusting is specifically the combination reaction between iron in its metallic form with oxygen forming iron(III) oxide. A greatly simplified equation is below. Detailed aspects of the full mechanism are described elsewhere in The Prologue. Student investigations of these activities are addressed in other activities.
H₂O
Fe(s) + O₂(g) → Fe₂O₃• x H₂O(s)
Iron    oxygen    rust

The degree of hydration of the iron oxide affects the color of the rust, which may vary from yellow, to the familiar reddish brown, to black.

This is also a redox reaction where iron is losing electrons to oxygen. The iron is being oxidized and the oxygen is being reduced.

Water and oxygen are both involved in this reaction; iron does not react in water that has no oxygen. Similarly, iron will not rust in an oil even if it is saturated with oxygen.

See The Prologue for a detailed discussion of the various mechanisms involved when iron undergoes corrosion.

Extension Activities

The following sites can be used to provide additional resources and information for students with regard to the topics provided.

Corrosion Consultancy (commercial site) - Provides information about various types of corrosion and its control.

http://www.xs4all.nl/~cdewaard/

What are the types of corrosion?

How is corrosion controlled?

What is a galvanic cell?

National Association of Corrosion Engineers (professional site) - provides details on the general condition of bridges in the U.S. and the magnitude of the bridge corrosion problem.

http://www.nace.org/nace/content/publicaffairs/media/bridge.asp
National Association of Corrosion Engineers (professional site) - provides information on the cost of bridge corrosion.

http://nace.org/NACE/content/PublicAffairs/media/costcor.asp

National Association of Corrosion Engineers (professional site) - provides information about how corrosion affects the environment.

http://nace.org/nace/content/publicaffairs/media/ccenv.asp

Corrosion Doctors (commercial site) – provides information about the factors which affect pipeline corrosion and the costs involved.

http://www.corrosion-doctors.org/Pipeline/Introduction.htm#corrosion

CorrosionSource – provides comparisons between the corrosion of various metals.

http://www.clihouston.com/howmetals.htm

Federal Highway Administration - provides information related to roads, bridges and corrosion.

http://www.tfhrc.gov/focus/jan01/quality_safety_reliability.htm

The Road Information Program (TRIP) - provides statistical information about America’s highways and bridges.

http://www.tripnet.org/nationalfactsheet.htm

Rebuild America Coalition – provides information about local bridges in your state.

http://www.rebuildamerica.org/reports/#resources
Federal Highway Administration – provides information related to health and safety issues involved in bridge maintenance.

http://www.tfhrc.gov/hnr20/bridge/repair/ch1/set.htm

Corrosion Doctors (commercial site) – provides information as to why seawater promotes corrosion.

http://www.corrosion-doctors.org/Seawater/Introduction.htm#U.S.

**Addressing the National Science Education Standards**

- 188: In communication within and between groups of students, students report clearly their scientific discoveries.
- 199: Through their research students see corrosion as a ubiquitous phenomenon.
- 202: Through their research students have an understanding of the time frame for corrosion to occur.
- 284: Corrosion is seen as a naturally occurring process.
- 286: Students understand that technology is used to help solve corrosion problems.
- 289: Corrosion is seen as a slow and progressive change.
- 292: Students understand the economic impact corrosion has on society

**Addressing the Voluntary Industry Standards**

- **L1.01.07** Assigned groups will uncover the environmentally hazardous materials historically used for corrosion protection.
- **L1.04.01** Students will gain experience working as a member of a team reporting their findings.
- **L4.05.01** Students will observe the long term economic impact on society caused by implementing iron as a structural building material.
- **L5.13.01** In their research students will describe the rate of corrosion reactions.
- **L7.05.03** Students prepare and present an oral report.
Addressing the SCANS Skills
Using Workkeys Targets

Locating Information

Level 4- The graphical information found within this activity’s web sites have students summarize and compare information.

Reading for Information

Level 5- The web site information contains jargon, technical terms and acronyms. Additionally, students apply instructions from the activity handout to their presentations.

Teamwork

Level 3- Students identify team goals and work to accomplish these goals

References

http://www.corrosioncost.com

http://www.nace.org
The Roebling Suspension Bridge connects Covington, KY to Cincinnati, OH. It was built in 1862 and still stands today. The load of the road deck and traffic is transferred to the two stone towers and the riverbank via the connecting cables. This bridge that spans the Ohio River confirmed new design principles Roebling intended to use in the construction of the mighty Brooklyn Bridge. Thus the two bridges have uncanny similarity in design.

**The Problem**
During rush hour traffic, a bridge collapses, and automobiles fall helplessly into the cold water below. What happened to cause such a tragedy? Let's investigate the underlying problem with the bridge that brought about such a catastrophic loss of life.

**Your Task**
In today's fast-paced world, events go on constantly around you. Some of these actions get noticed, and some go unrecorded. It is a developed skill to be able to remember, record, and filter important information. In this activity, you will observe an eleven minute videotape with pen and paper in hand, sharpening your listening and observation skills. Afterwards, you will be tested on your abilities to recall auditory and visual information about corrosion's role in a calamitous bridge collapse. As this activity is at the beginning
of your study of corrosion, it is important to emphasize that you will be tested on the
details of the event, not the understanding of the underlying chemistry.

Materials

“Collapse” volume entitled Failure by Design, produced by The Learning Channel.

Procedure

As you view the video of a famous bridge collapse, you are to record any factual
information that you deem important. You will be only be allowed to view the video
once. Upon completion of the video, you will be tested on your ability to recall the
details. You will not be able to talk or share information with others during this exercise.
Also it is very unlikely that you will be able to write down everything you see and hear,
so attempt to filter out items of importance, while omitting trivial details. Be careful not to
get hung up on a particular point and miss future information.
Assessment

The following questions are generally in chronological order. You may only refer to the notes you took during the viewing of the videotape.

Answer the questions to the best of your ability. Spelling and grammar does count. Your teacher will provide details about the scoring system to be used. Complete sentences are only required for question 17.

1. What was the name of the town featured in the video?

2. This town was in which state?

3. What is the name of the river in which the bridge crossed?

4. What year was the bridge built?

5. What year did the bridge fall?

6. In what month did the bridge fall?

7. What material was the bridge made out of?

8. What was the nickname for the bridge?

9. What type of bridge design was used in building this bridge?
10. How many dead bodies were pulled from the river?

11. What was the total number of cars and trucks that fell into the river?

12. What holiday season was approaching at the time of the bridge failure?

13. Prior to the metals expert's evaluation of this collapse, what was the most popular theory held about the reason for this catastrophe?

14. Draw a picture of the piece that broke.

15. How many years did the bridge stand before collapsing?

16. There is a bridge that still exists next to where the collapsed structure originally stood. What kind of vehicles does this span carry?

17. In a few sentences describe in your own words what caused the collapse of the bridge.
Corrosion Collapse

Industry demands potential employees have good listening, observation, writing, and question answering skills. Through this activity, students exercise these skills as they learn about the cause of a famous catastrophic bridge collapse.

This activity combines some relevant background on corrosion and bridge design with several important workplace readiness skills. In addition to being qualified in basic scientific and mathematical skills.

Time Required

Setup: 5 minutes to cue video clip
Viewing of videotape: 11 minutes
Quiz: 15 minutes
Optional Discussion: 10 minutes

Key Science Concepts

Chemistry
• corrosion of iron
• materials sciences
• properties of materials

Other Science Concepts
• physics: treatment of load bearing forces in a suspension bridge
• physics: gravity

Engineering and Technology
• foundry of high carbon high strength steel
• bridge design and construction

Materials

“Collapse” volume entitled Failure by Design, produced by The Learning Channel. See reference for details. (original broadcast version, NOT Discovery Channel School)

Getting Ready

The videotape cited above has an eleven minute clip that describes the Point Pleasant
bridge collapse. It needs to be cued prior to class. The whole collapse series is a four volume set, with the Point Pleasant clip roughly 30 minutes into the volume entitled *Failure by Design*.

** It should be noted that the version released in the Discovery Channel School series is only 6 minutes long and the questions in this activity need to be modified accordingly. On this tape, the clip is 9:30 from the beginning.

Teachers could conduct a brainstorming activity and have students contemplate possible catastrophic events due to corrosion and metal failure, prior to watching the videotape. This would set the stage for the disastrous event that is about to be presented.

**Procedure Notes**

Provide students the details of the problem and the tasks to be carried out in this activity either verbally or by passing out page 1 of the student handout. Do not hand out the student questions until after the video is viewed. Emphasize the fact that a quiz will be given immediately following the video and that students will not be able to share their information. After the video is complete, handout the quiz and review the grading scale you will use. We recommend questions 1-16 be assigned 3pts and question 17 being assigned 10 pts. Additionally, we recommend that partial credit be as allowed per the rubric that follows.

Lastly, a brief discussion of the activity is suggested after the quiz.

**Assessment**

Grading rubric

3pts per question

In all cases grammatical/spelling errors an additional –1pt.

Wild guesses earn zero points.

1. **What was the name of the town featured in the video?**

   Point Pleasant = 3pts, Incorrect/name such as Pleasant point or Point Crescent = 2 point.

2. **This town was in which state?**

   West Virginia = 3 pts. Virginia = 2pts, Ohio = 1pt
3. What is the name of the river in which the bridge crossed?

Ohio, The Ohio, The Ohio River, Ohio River = 3pts

4. What year was the bridge built?

1927 = 3pts, +/- 1 year = 2pts, +/- 5 years = 1pt

5. What year did the bridge fall?

1967 = 3pts, +/- 1 year = 2pts, +/- 5 years = 1pt

6. In what month did the bridge fall?

December = 3pts, January = 1pt.

7. What material was the bridge made out of?

High Carbon, High Strength Steel = 3 pts. High Carbon Steel or High Strength Steel = 2 pts. Steel or Iron or Metal = 1 point.

8. What was the nickname for the bridge?

The Silver Bridge or The Great Silver Bridge = 3 pts.

9. What type of bridge design was used in building this bridge?

Suspension = 3 points, “Elegant Chain Link Design” (or a similar variation) = 2 pts.

10. How many dead bodies were pulled from the river?

46 bodies = 3pts, +/- 1 bodies = 2pts. +/- 3 bodies = 1 pt.

11. What was the total number of cars and trucks that fell into the river?

37 Cars and trucks (vehicles) = 3 pts, +/- 1 vehicle = 2pts, +/- 5 vehicles = 1pt.

12. What holiday season was approaching at the time of the bridge failure?

Christmas = 3pts, Winter = 1 pt.

13. Prior to the metals expert’s evaluation of this collapse, what was the most popular theory held about the reason for this catastrophe?

Overloading / too much weight = 3 pts. Bridge being hit by a barge = 2 pts. Bridge not
14. Draw a picture of the piece that broke.

15. How many years did the bridge stand before collapsing?

40 years or 41 years = 3pts (It was finished in the early part of 1927 and eventually fell in December 1967), +/- 1 year (starting from either 40 years or 41 years) = 2pts, +/- 5 years = 1pt

16. There is a bridge that still exists next to where the collapsed structure originally stood. What kind of vehicles does this span carry?

Trains, Rail Cars, Railroad = 3 pts.

10 point question below

17. In a few sentences, describe in your own words what caused the collapse of the bridge.

A hairline crack in one structural eye bar (in the main suspension span) existed since the original casting in 1926. Forty years of corrosion weakened the structural member and eventually the eye bar fractured. This caused a catastrophic failure. = 10 pts

A part of the bridge was cracked and rusty = 2 pts

Explanation

The Point Pleasant Bridge failure was of a type known as corrosion fatigue, and a number of factors were implicated. First and most important, the manufacturing defect of a pre-existing crack existed when the eye bar was originally cast at the foundry. This internal cracking resulted from inclusions and forging defects in the metal. Secondly, corrosion played a role in causing the eventual failure of the metal. It is noted that the close proximity of a rail bridge carrying coal-burning engines exposed the bridge to
SO₂, NO₂, and CO₂. These compounds, in the presence of water, form acids which increased the rate of corrosion. Additionally, inherent movement and change of load (based on traffic) caused a type of metal fatigue known as “cyclic loading”. This would then cause such a crack to grow deeper, slowly revealing new surfaces where additional corrosion would occur. Lastly, corrosion in conjunction with an applied stress makes metal fatigue occur more rapidly. This is known as stress corrosion cracking.

**Extension Activity**

Although the technical nature of the Point Pleasant bridge collapse is not addressed, the fictional sci-fi/thriller movie “The Mothman Prophecies,” was loosely based on the events in Point Pleasant, West Virginia that allegedly occurred up to and including the silver bridge collapse. The movie is rated R.

**Addressing the National Science Education Standards**

- **180**: Students experience the revision of the collapse theory when new evidence is presented.
- **181**: Students analyze and compare the original explanation of the collapse to the one as provided by the metals expert.
- **182**: Students witness the defense of a scientific argument provided by the metals expert. This presentation is one that can then be modeled in future presentations.
- **184**: Students witness a scientist conducting an investigation to determine the flaw in the bridge, so as to prevent such a catastrophe from happening again.
- **199**: Students witness that chemical reactions such as corrosion occur all around us.
- **202**: Students are exposed to rusting both as a fast process (as in the surface rust of the broken eyebar) and as a slow process (which took 40 years to weaken The Silver Bridge).
- **266**: It was through the development of new high-strength steel in the 1920s that a suspension bridge of chain link design could be constructed.
- **268**: A bridge is an example of a technological achievement as compared to a scientific inquiry
- **290**: A collapsing bridge is an example of a human induced hazard.
- **294**: The Silver Bridge was an unproven chain link design, which proved to be subject to failure
- **301**: The new high-strength steel used in the construction of the Silver Bridge was originally believed to possess the proper strength characteristics, but proved to have a dangerously weak interior.
Addressing the Voluntary Industry Standards

L1.02.06 Students are introduced to the type of work done by a metal analysis technician as he describes his investigation of the collapse.

L4.01.01 Students are exposed to the importance of measurement in chemistry as they witness the metal analysis technician discuss the depth of the original crack.

L5.14.01 Students witness the importance of chemical analysis in the investigation of the bridge collapse

Addressing the SCANS Skills

Using Workkeys Targets

Applied Technology

Level 3- Students gain a basic appreciation for the manner in which a suspension bridge transfers the load of the bridge deck first to the main span and subsequently how the main span then transfers the load to the towers.

Listening

Level 4- In watching the video, students can convey the central idea of the spoken communication. Additionally, students can convey the important information and relationships among these pieces of information.

Locating Information

Level 3- Students can recognize and locate the piece of the bridge that broke, causing the catastrophe.

Observation

Level 5- Students demonstrate their ability to focus their attention on important aspects of the information presented. As this is an introductory activity the information is unfamiliar. Additionally, students must use selective attention in order to ignore irrelevant information.

Writing

Level 4- Students are expected to write responses to questions that are clear and consistent with standard business English.
References

Collapse video series, Failure by Design. A Darlow Smithson Production for Channel 4 Television and The Learning Channel 2000

Gary Drigel, Professor in Engineering Technology, Miami University, Middletown Campus. drigelgs@muohio.edu
The Hungry Flask

The Problem

We have all encountered rust at one time or another. It wreaks havoc with automobiles, bicycles, and the steel industry. But is the iron the only thing to change when rust is formed? In this activity you will investigate rusting.

Your Task

In this activity you will prepare an appropriate environment which will allow steel wool to rust quickly.

Materials

Per group

- egg-sized piece of steel wool
- 50–100 mL vinegar
- 250-mL wide mouth Erlenmeyer flask or small-mouthed jar
- 250-mL beaker
- large (11-in) latex balloon
- tongs or large forceps
- latex gloves

Safety and Disposal

As instructed by your teacher, follow appropriate safety procedures, including the use of personal protective equipment such as goggles and an apron. Be careful handling the steel wool. Painful steel splinters can lodge in skin. Do not leave the steel wool in the reaction flask for long periods of time; it will permanently discolor the glass. The reacted steel wool can be rinsed with water and placed in the trash when completed.

Procedure

1. Obtain a clean latex balloon from your instructor. Stretch it several times and blow it up and release the air several times. This will relax the latex.

2. Put on a pair of latex gloves to avoid splinters while handling the steel wool.

3. Place an egg-sized piece of steel wool into a 250-mL beaker. Do not compress the wool, so the vinegar will penetrate easily to the center. Pour enough vinegar over the steel wool to cover it, and allow it to sit for about 4 – 5 minutes.

Note: A large quantity of steel wool is not required for this reaction. A small piece is adequate.
4. Using tongs, remove the steel wool from the beaker and carefully shake out the excess vinegar. Do not squeeze the steel wool into a tight ball. This will slow down the reaction. The shape of the steel wool should be approximately the same as when it was dry. Gently place the steel wool into a 250-mL Erlenmeyer flask or small-mouthed jar.

5. Quickly cover the mouth of the flask with the pre-stretched, deflated balloon from step 1.

6. Observe the flask/balloon system for 20 – 40 minutes while answering questions 1 – 12 in the Assessment section.

7. Compare your balloon observations to the rest of the class and answer question 13.

8. When your instructor indicates, remove the balloon, and record your observations during this procedure.

9. After the class discussion answer question 14.

10. Invert the flask over a trashcan. Using forceps, remove, rinse with water and discard the steel wool.

11. Rinse the flask with water.

**Assessment**

Answer the following questions in complete sentences. In all cases, justify your answer. For example, simply responding yes or no to question #3 will not earn you credit.

1. Describe all the changes you observed.

2. What evidence exists that a chemical reaction is taking place?

3. Was a new substance produced?

4. Was anything used up? Explain your answer.

5. What do you think the substance produced might be?


7. What is the common name for the process you just observed?

8. What do you think caused the reaction?

9. Describe what is happening at the atomic level.

10. Why do you think steel wool was chosen instead of an iron nail?

11. Write the chemical equation for the reaction occurring in the above system including states of matter.

12. How would the mass of the solid in the flask compare with the initial mass of the
steel wool before the reaction occurred?

13. Discuss possible reasons for differences between lab groups.

14. When the instructor indicates, remove the balloon and record your observations.
Instructor Notes

The Hungry Flask

Students conduct an experiment where they observe steel wool rusting in a short period of time. They discuss their observations and ideas in order to better understand the underlying phenomenon.

Time Required

Setup: 10 minutes
Procedure: 20-40 minutes
Discussion: 10-20 minutes
Cleanup: 10 minutes

Key Science Concepts

Chemistry
• chemical change
• combination reactions
• compounds
• conservation of mass
• Dalton’s law of partial pressure
• electrolytes
• elements
• gas pressure
• kinetic molecular theory
• reaction kinetics (using steel wool vs. an iron nail)
• redox reactions
• spontaneous reactions

Prior Concepts and Skills Needed

The depth expected final discussion dictates the depth of the activity, and thus the prior knowledge. At a minimum, students should be familiar with kinetic molecular theory, basic gas properties, chemical reactions, states of matter, and conservation of mass. If your discussion will include equations and predicting products, bonding concepts are also required.

Materials

For Getting Ready
• Heavy duty scissors
• Grade 00 steel wool

**For the Procedure**

Per group

• egg-sized piece of steel wool
• 50–100 mL vinegar
• 250-mL wide mouth Erlenmeyer flask or small-mouthed jar
• 250-mL beaker
• large round latex balloon (11-in diameter is recommended)
• tongs or large forceps

**Safety and Disposal**

It is your responsibility to review appropriate safety procedures with your students, including the use of personal protective equipment. The used steel wool can be disposed in a solid waste container after being rinsed and the used vinegar solution can be rinsed down the drain.

**Getting Ready**

Cut the steel wool into egg-sized pieces ahead of time to minimize the risk of students getting iron splinters. This will also maximize classroom observation time.

**Procedure Notes**

Regarding the steel wool, it is important to find steel wool that has not been treated with soap (as in a scouring pad) or oil (that would prevent corrosion). The steel needs to be in contact with the air. The finer the grade of the steel wool (00 vs. 1), the greater the surface area and the quicker and more impressive the reaction. It takes about 30 minutes for grade 00 steel wool to force a pre-stretched, 11-inch diameter balloon into the flask.

Students place steel wool and vinegar in a beaker for a short time. They then transfer the steel wool to an Erlenmeyer flask and cover with a balloon. For the majority of the class period, students observe the system. Be sure to allow time for recording observations and cleaning up.

Students will conduct the activity and, in complete sentences, answer the list of guided questions 1-13. Collect the students’ answers and lead a class discussion, eliciting new observations and questions that the students may have. During this time, students can continue to make observations and can observe their system throughout the discussion.
Students return to their experiment, answering question, #14. You may choose to leave one of the setups intact overnight for follow-up observations on the following day.

The student responses to question #14 can also be collected and answers discussed as above. It is important to ensure that you provide an environment where students do much of the talking. As you are most aware of how this procedure fits within your curriculum, adjust the lab experience accordingly. It is important to keep this as a student-centered environment and lead the students to the correct conclusions. Students should be able to generate the answers independently as reasonably possible. The discussion should be a brainstorming session, with the students deciding which of the proposed answers are unreasonable.

It is important that students understand that the “consumption” of the oxygen is actually a chemical reaction where the oxygen combines with the iron to form the solid iron oxide (rust). A discussion of conservation of mass at this time is appropriate.

**Assessment**

1. Describe all the changes you observed.

A red material formed on the steel wool and the balloon was pushed into the flask.

2. What evidence exists that a chemical reaction is taking place?

Color changes are often indicative of a chemical reaction. Additionally, the balloon pushed into the flask demonstrates the pressure inside the flask is less than the atmospheric pressure (initially the pressure inside and outside the flask were the same). This would happen if the system was cooled substantially (physical change) or if a chemical reaction occurred that used up some of the gas inside the flask. You can rule out the physical change option because the flask did not cool. In fact, if any temperature change is observed it was that the flask may have warmed slightly; this would cause the balloon to inflate rather than deflate. Thus you can conclude that the change is based on a chemical reaction.

3. Was a new substance produced?

A new substance was produced. It has a red-brown color.

4. Was anything used up? Explain your answer.

A gas (oxygen) was consumed in the formation of the solid rust. Oxygen is the only gas in our atmosphere that will react under these conditions.

5. What do you think the substance produced might be?

The new substance is rust, also referred to as iron(III) oxide, or as ferric oxide.

Students would find it difficult to know if or when the reaction was finished. This reaction slowly reacts with the oxygen from the container, and one would need to observe the setup for a long period of time to ensure that the balloon was not pushed further into the flask.

7. What is the common name for the process you just observed?

The common name of the process is rusting. It is also called corrosion, oxidation or reduction.

8. What do you think caused the reaction?

This reaction occurred because the correct conditions existed in the flask. The necessary conditions are that iron (from the steel wool), oxygen (which is dissolved in the water from the air), and water (which is provided by the vinegar which is typically 5% acetic acid and 95% water) were all present. The acetic acid was also helpful in speeding up the reaction so that the reaction would occur within the class period. However, it should be noted that oxidation would have still occurred without the acetic acid (although at a much reduced rate).

9. Describe what is happening at the atomic level.

The molecular oxygen from the air chemically reacted with the iron atoms to form a new compound, rust. Water (from the vinegar) provides the vehicle for this reaction to occur. (As a note to teachers: there is an excellent discussion of the role of water in the full redox mechanism of this reaction which can be found in the "The Chemistry of Corrosion" article referenced at the end of this activity.)

10. Why do you think steel wool was chosen instead of an iron nail?

As compared to an iron nail, steel wool has a large surface area and thus the rate of the reaction is substantially increased.

11. Write the chemical reaction occurring in the above system including states of matter.

\[
\text{H}_2\text{O} \quad \text{Fe}(s) + \text{O}_2(g) \rightarrow \text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O}(s)
\]

Iron oxygen rust  

Water is required for the above reaction to occur.

12. How would the mass of the solid in the flask wool compare with the initial mass of the steel wool before the reaction occurred?

After the reaction, the mass of the solid is greater than that of the original steel wool as it is no longer just steel wool. It is now rust and unreacted steel wool. The mass of oxygen is now included as rust. The steel wool also weighs more because it is wet, but this is not an acceptable reasoning. Once the steel wool dries this additional mass has
13. Discuss possible reasons for differences between lab groups.

Corrosion is inherently a random occurrence. Different conditions such as the degree of prestretching / preinflating, a different amount of exposed surface, temperature, oil on the steel wool from someone handling it, can all account for different observations between lab groups.

14. When the instructor indicates, remove the balloon and record your observations.

Air from the atmosphere was pushed into the flask because gases move from higher to lower pressure. The atmospheric pressure was greater than the air pressure in the flask as some of the oxygen gas in the flask was used up in the rusting reaction. This causes the balloon to return to its original size.

Explanation

![Figure 1. Chronology of hungry flask experiment during a 30 minute reaction](image)

We have all probably seen rust and know it by its characteristic red-brown color, flaky texture, and the pitting on the original metal. We may even know rusting as the oxidation of iron. However, most of our experience with this process concentrates on the corrosion and the weakening of the metal in the presence of water, and not on oxygen as the other key reactant needed for the reaction to occur. This activity provides evidence of
the consumption of oxygen during the rusting process.

As the iron in the steel wool combines with the oxygen in the air (and in the presence of water), rust (iron(III) oxide) is formed. As the oxygen in the flask is consumed in the reaction, the air pressure inside the flask decreases (Dalton's law of partial pressure), causing the greater atmospheric pressure outside the flask to push the balloon down into it. The large surface area of the steel wool allows the consumption of oxygen to occur rather quickly in the system. The vinegar provides an electrolytic solution which improves ion transfer (as compared to distilled water).

This could be an excellent starting place for introducing various aspects of chemistry. Use this activity as a springboard for introducing Dalton's Law of partial pressure, balancing equations, discussing combination reactions, redox reactions, and the concept of limiting reagents.

**Extensions**

1. Conduct the same experiment but substitute water for the vinegar. The rate of the chemical reaction will decrease dramatically. Over time, the students would compare their acidic (vinegar) reaction to the water reaction. The vinegar apparatus would show change much more quickly. But in contrast, in a larger amount of time, the balloon from the water apparatus would be also be drawn into the flask. Student could then determine that the amount of oxygen that has been consumed is consistent within the two systems.

2. Conduct the experiment using a 250-mL graduated cylinder with the acidic-treated steel wool lodged in the bottom. Fill this graduate with colored vinegar, and quickly invert it into a large beaker that also contains colored vinegar. Draw a line at the level of the vinegar inside the graduated cylinder. Over time this level will rise as the oxygen is consumed.

3. Use this experiment to explain conservation of mass. A misconception students may have is that the oxygen is lost. It is not lost; it is now part of the compound iron oxide. An empirical proof utilizing the above system would be difficult to accomplish. One suggestion is to run a parallel experiment by allowing a known mass of steel wool to rust, dry out and remass. Additionally, a similar combination reaction is allowing magnesium to combine with oxygen in a covered crucible (under high heat) to show a similar mass gain.

4. Run this experiment again using an iron nail instead of the steel wool. Several days will be required.
Addressing the National Science Education Standards

• 180: Students use evidence from their observations to explain the “hungry flask”.
• 181: Within the class discussion, students analyze and decide which explanation of the “hungry flask” is best.
• 183: Using their conceptual principles, students inquire about how the system functions.
• 184: Students test their prior theories about corrosion as they witness it first hand at accelerated speed.
• 187: Students present their explanations that abide by scientific criteria.
• 199: Many students have witnessed the rusting of a steel scouring pad.
• 200: Students witness a reaction that consumes a gas.
• 202: Students gain an appreciation of a typical reaction rate.
• 290: In this activity, iron corroded quickly. This justifies to students the need to protect society’s structural steel such as bridges.
• 299: Students explain the natural world.
• 300: Student explanations meet scientific criteria

Addressing the Voluntary Industry Standards

L1.04.05 Students demonstrate critical thinking skills in assessing the presented phenomena.

L2.05.04 Students manipulate erlenmeyer flasks and tongs in executing this experiment.

L3.01.08 Atoms of iron combine with oxygen to form the compound iron(III) oxide.

L3.01.10 The chemical equation describing the corrosion of iron is written and balanced.

L3.02.03 The formula for the common inorganic material, rust (iron(III) oxide), is written correctly

L4.03.04 Students set up the required apparatus.

L5.01.02 & L5.08.04 Students write equations for oxidation reduction reactions.

L5.13.01 Rate of reaction is explored in this activity.

L7.04.02 Students record their observations.

Addressing the SCANS Skills

Using Workkeys Targets

Applied Technology
Level 3- Students must understand the notion of a contained gaseous system and pressure.

**Observation**

Level 5- Through their use of selective observation, students must ignore irrelevant information (such as the condensation forming on the inside of the flask) to focus on the important aspects of the system (red rust forming and gas pressure decreasing).

**References**


The Reactivity of Metals

The Problem
Metals undergo the process of corrosion by utilizing several different classes of chemical reactions. It can be noted that under identical circumstances, certain metals are more likely to corrode (react) than others. The study of single replacement reactions provides a powerful tool known as a metal activity series. In acidic environments, metals typically react by single replacement reactions.

Your Task
You will conduct a series of experiments which will determine the relative reactivities of several metals. Some metals exhibit evidence of reaction, others do not. In essence, your laboratory data will be used to build an activity series. Your goal is to determine where iron's reactivity (the building metal of choice), compares to other metals. The answer may surprise you!

Materials
Per group
- 20 reaction vessels, (a microplate, small test tubes or small beakers)
- test tube rack
- 4 zinc strips (Zn)
- 4 silver strips (Ag)
- 4 magnesium strips (Mg)
- 4 copper strips (Cu)
- 3 iron strips (Fe)
- 1 iron wire 18 guage
- iron filings
- magnet
- 0.1 M cupric sulfate (CuSO₄)
- 0.1 M ferrous sulfate (FeSO₄)
- 0.1 M magnesium sulfate (MgSO₄)
- 0.1 M silver nitrate (AgNO₃)
- 0.1 M zinc chloride (ZnCl₂)
- sheet of white paper

Safety and Disposal
As instructed by your teacher, follow appropriate safety procedures, including the use of personal protective equipment such as goggles and an apron. Be careful
with the silver nitrate (AgNO₃) as it stains skin. In general, solids can be rinsed
and placed in the trash. Dispose of waste liquids as instructed by your teacher.
Solutions containing Ag⁺ and Cu²⁺ should be collected in separate waste
containers for special handling before disposal.

Procedure

Answer the following background questions.

Q1. Do metals typically form cations or anions?
Q2. When magnesium forms an ion what is the formula of the most
common ion?
Q3. Silver and zinc typically form only one ion each. What are they?
Q4. Copper typically forms two common ions. What are they? In this
activity copper(II) is the ion formed.
Q5. Iron typically forms two common ions. What are they? In this
activity iron(II) is the ion formed.

Experiment procedure

1. Review the attached data table, you will conduct 20 different
   experiments.
2. The metal (left column), will be dropped into the aqueous metal ion
   solution (top row). Use only enough solution to cover the metal
   completely.

   Note: A metal does not need to be tested in the solution of that
   metal. For example copper, (Cu) does not need to be tested in
copper sulfate (CuSO₄)

3. Repeat step 2 for 19 of the reactions with the exception of iron (Fe) with
   the 0.1M silver nitrate (AgNO₃).
4. Use the unique procedure on the following page when reacting the iron
   (Fe) with the 0.1M silver nitrate (AgNO₃).
5. Allow all reactions to proceed for 10 minutes before making observations. A piece of white paper under the reaction vessel will improve observations.

6. In each box you need to:
   a) Record your observations.
   b) Write the complete, balanced equation, indicating states of matter. If no reaction occurs, write NR (no reaction) on the product side.

7. Record the number of reactions that occurred for each row of metals. Each line should be given an single unique integer 0-4. (no number is repeated)

**Discussion and Conclusions**

By following these instructions you will determine an “activity series” for the metals you tested.

Q6. Which metal had 4 reactions?

Q7. Which metal had 3 reactions?

Q8. Which metal had 2 reactions?
Q9. Which metal had 1 reaction?

Q10. Which metal had 0 reactions?

These metals are now listed in order of decreasing activity. The most reactive metal is on top. The least reactive metal is on the bottom. Activity series works like “king of the hill.” The “strongest” (most reactive) metal can replace the metal ion of any weaker metal. The “weakest” (least reactive) metal can not replace any other metal ions. A metal can replace any metal ion below it.

Q11. What is the least reactive metal?

Q12. Precious metals are by definition metals utilized in the jewelry industry. How does metal reactivity relate to defining a precious metal?

Q13. Where is iron in the series?

Q14. Why do engineers select more reactive metals to build bridges and other structures rather than the less reactive ones?

Q15. Could a solution of magnesium chloride be safely stored in a steel (iron) drum?

**Assessment**

Complete the attached data table.
<table>
<thead>
<tr>
<th>Solutions</th>
<th>ZnCl₂</th>
<th>AgNO₃</th>
<th>MgSO₄</th>
<th>FeSO₄</th>
<th>CuSO₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
</tr>
<tr>
<td>Ag</td>
<td>eq</td>
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</tr>
<tr>
<td>Mg</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
</tr>
<tr>
<td>Fe</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
</tr>
<tr>
<td>Cu</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th># of reactions</th>
<th>Zn</th>
<th>Ag</th>
<th>Mg</th>
<th>Fe</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
<td>eq</td>
</tr>
</tbody>
</table>
The Reactivity of Metals

In this activity the students will conduct a series of single replacement reactions in order to elucidate the activity series (also known as the electrochemical series) for five different metals. Two of the metals tested are more reactive than iron, (the focus metal of this corrosion unit) two of the metals tested are less reactive than iron. Extension activities are provided to address the concepts of half-reactions and spontaneous reactions.

Students may want to know why society builds with iron, rather than less reactive metals. There is a two-fold answer. Metals with low reactivity typically fall into the class of metals known as precious (or coinage) metals. Their significant cost prohibits large-scale use. These precious metals are also typically softer than iron and thus do not have the structural properties required of a material that must support enormous weight.

Alternatively, if students already have familiarity with the activity series of metals, this lab could be used with students speculating and predicting which reactions should and should not occur, and then compare their actual data to their theoretical predictions.

Time Required

Setup: 50 minutes
Lab procedure: 50 minutes
Discussion, Conclusion, Assessment: 40 min
Cleanup: 15 minutes

Key Science Concepts

Chemistry

• activity series
• balancing equations
• corrosion inhibition
• *half reactions
• *net ionic equations
• *redox equations
• single-replacement reactions
• *standard reduction potential
• states of matter (solid vs aqueous)
*indicates extension activity concept
Engineering and Technology

- cost analysis for iron as a building material

Prior Concepts and Skills Needed

Students need to understand the following concepts prior to this activity, (or conversely you can use this activity to instruct the following concepts):

activity series of metals

evidence of chemical change

atoms vs ions

aqueous solutions

balancing equations

chemical equation nomenclature (i.e. products, reactants, →)

chemical element symbols

states of matter

single replacement reactions

redox reactions

Materials

For Getting Ready

- steel wool
- zinc strips (Zn)
- silver strips (Ag)
- magnesium strips (Mg)
- copper strips (Cu)
- iron strips (Fe)
- cupric sulfate (CuSO₄ • 5H₂O)
- ferrous sulfate (FeSO₄ • 7H₂O)
- magnesium sulfate (MgSO₄ • 7H₂O)
- silver nitrate (AgNO₃)
- zinc chloride (ZnCl₂)
For the procedure

- 20 reaction vessels, (a microplate, small test tubes (13mm x 100 mm or other suitable tubes) or small (10 mL) beakers)
- test tube rack
- 4 zinc strips (Zn)
- 4 silver strips (Ag)
- 4 magnesium strips (Mg)
- 4 copper strips (Cu)
- 3 iron strips (Fe)
- 1 iron wire
- Iron filings
- magnet
- 0.1 M cupric sulfate (CuSO₄)*
- 0.1 M ferrous sulfate, acidified (FeSO₄)* (typically acidified with HCl)
- 0.1 M magnesium sulfate (MgSO₄)*
- 0.1 M silver nitrate (AgNO₃)*
- 0.1 M zinc chloride (ZnCl₂)*
- sheet of white paper

*The amount of each of these solutions required will depend upon the size of the reaction vessels used by students. You can estimate you will need four times the volume of the typical reaction vessel to be used multiplied by the number of groups performing the procedure.

Safety and Disposal

It is your responsibility to review appropriate safety procedures with your students, including the use of personal protective equipment.

Silver nitrate stains skin and some fabrics. 3% (w/v) hydrogen peroxide can sometimes be used to remove this discoloration.

Check your local ordinances regarding the waste metal ion solutions. The Flinn Chemical and Biological catalog recommends that solutions containing Ag⁺ be treated with 1M NaCl(aq) to precipitate AgCl before disposing of the solid in a solid waste container. (See the Flinn reference for complete details. This is also available on their web site.)

You may want to recycle the silver, as it is expensive and not affected in these reactions.
Getting Ready

Strips of metal must be shiny prior to use. Use steel wool to get a clean surface.

The iron sulfate as purchased commercially is typically "acidified". If you chose to make your own 0.1 M iron sulfate solution, include 10 mL of 6M HCl to every 1L of solution.

Author Note: The reason for the acidification of the iron sulfate is that iron is somewhat reactive in the ferrous (2+) oxidation state. In neutral or basic solutions it will precipitate over time as ferrous hydroxide, which oxidizes to ferric (3+) hydroxide creating the orange precipitate. A low pH lowers the hydroxide ion concentration and thus causes the ferrous ion to remain in solution.

Procedure Notes

In this activity students conduct 20 single replacement metal/metal ion reactions. They compile their observations as data and then determine the relative reactivities of the five metals. Since each lab group is performing 20 reactions, a systematic, organized approach is necessary. If time is sufficient, students should write their chemical equations as they perform the numerous experiments.

If time is limited, students could perform their reactions on the first day and write their reactions on the second day. Additionally, each group could be assigned a metal, perform and write these reactions and then share their data with the class.

If students are using a microplate, they may need to turn the samples over using forceps, or pick the plate up to observe the underside in order to improve their observations.

Answers to the Questions in the Procedure

Q1. Do metals typically form cations or anions?

Metals typically lose electrons to form cations.

Q2. When magnesium forms an ion what is the formula of the most common ion?

Mg^{2+}

Q3. Silver and zinc typically form only one ion each. What are they?

By utilizing resource material, students should conclude that silver forms a 1+ ion (Ag^{1+}), and zinc forms a 2+ ion (Zn^{2+}).

Q4. Copper typically forms two common ions. What are they? In this activity copper(II) is the ion formed.

As above, by utilizing resource material, copper forms the 1+ cuprous ion (Cu^{1+}) and the
2+ cupric ion (Cu^{2+}).

Q5. Iron typically forms two common ions. What are they? In this activity iron(II) is the ion formed.

Note: In non oxidizing acids, iron forms the 2+ ferrous ion (Fe^{2+}). However in the presence of air, Fe^{2+} (aq) tends to oxidize to the 3+ ferric ion (Fe^{3+}(aq)) with a positive EMF of 0.46 V. Over an extended time the 3+ ferric ion (Fe^{3+}) will form.

Assessment

Consult attached data table for observations and complete balanced equations.

Explanation

In this activity students conduct 20 single replacement experiments. Ten reactions proceed spontaneously with a positive cell voltage and a negative change in Gibbs free energy. Ten of the combinations do not proceed as spontaneous reactions as they have a negative cell voltage and thus a positive Gibbs free energy.

Author Note: In most high school activity series schemes, iron is deleted because it does not react well in silver nitrate. This reaction although it has a positive cell potential simply does not occur. Unfortunately, this whole unit is structured around iron, so it could not be omitted. The difficulty with this reaction is due partly to the difference between iron and steel. In the manufacturing of steel, elements such as copper, sulfur, manganese, and phosphorus exist in addition to iron. I speculate that one of these elements act as an inhibitor to the reaction. I found reasonable success with iron filings (as compared to cold rolled steel, iron wire, and iron powder). The magnetism of the iron wire allows for enhanced visualization. It should be noted that in the above example, the iron wire typically does not show the reaction whereas the iron filings should. The concept of a corrosion inhibitor (or in the case of biology, competitive inhibition) can be addressed at this time as well.

Instructors that seem to find success using paper clips, nails, and staples typically have the coating (either cadmium or zinc) undergo the replacement, NOT the base metal iron.

Of the 20 possible reactions tested, only the following reactions proceed. The remaining examples do not occur.

\[
\text{Zn(s) + CuSO}_4(aq) \rightarrow \text{ZnSO}_4(aq) + \text{Cu(s)}
\]

\[
\text{Zn(s) + FeSO}_4(aq) \rightarrow \text{ZnSO}_4(aq) + \text{Fe(s)}
\]

\[
\text{Zn(s) + 2AgNO}_3(aq) \rightarrow \text{Zn(NO}_3)_2(aq) + 2\text{Ag(s)}
\]
Mg(s) + CuSO₄(aq) → MgSO₄(aq) + Cu(s)
Mg(s) + FeSO₄(aq) → MgSO₄(aq) + Fe(s)
Mg(s) + 2AgNO₃(aq) → Mg(NO₃)₂(aq) + 2Ag(s)
Mg(s) + ZnCl₂(aq) → MgCl₂(aq) + Zn(s)
Fe(s) + CuSO₄(aq) → FeSO₄(aq) + Cu(s)
Fe(s) + 2AgNO₃(aq) → Fe(NO₃)₂(aq) + 2 Ag(s)
Cu(s) + 2AgNO₃(aq) → Cu(NO₃)₂(aq) + 2 Ag(s)

**Discussion and Conclusion**

Q6. Which metal(s) had 4 reactions?

Mg

Q7. Which metal(s) had 3 reactions?

Zn

Q8. Which metal(s) had 2 reactions?

Fe

Q9. Which metal(s) had 1 reaction?

Cu

Q10. Which metal(s) had 0 reactions?

Ag

Q11. What is the least reactive metal?

Ag

Q12. Precious metals are by definition metals utilized in the jewelry industry. How does metal reactivity relate to defining a precious metal?

Precious metals have little reactivity and thus exist in their (non reacted) metallic state for a long time! (As in the still shiny gold burial masks found in Tutankhamen’s tomb in the 1920s.)
Q13. Where is iron in the series?

Iron is the third most reactive metal. Two metals are more reactive, two metals are less reactive.

Q14. Why do engineers select more reactive metals to build bridges and other structures rather than the less reactive ones?

Precious metals are typically both too expensive and too soft for practical use in construction.

Q15. Could a solution of magnesium chloride be safely stored in a steel (iron) drum?

Yes. Magnesium is above iron in the activity series.

**Extension activities**

**Net ionic equations**

Elements exist as neutral atoms with their number of protons and electrons being equal. Thus Zn(s) as a metal has no net charge (Zn\(^0\)). When aqueous solutions are written, it is the dissociation of the ions that promoted dissolving. CuSO\(_4\)(aq) for example has independent ions in solution, Cu\(^{2+}\) and SO\(_4^{2-}\). Chemists often rewrite single replacement reactions as ionic equations as they better describe the underlying phenomenon.

\[
\text{Zn(s) + CuSO}_4(aq) \rightarrow \text{Zn(SO}_4)(aq) + \text{Cu(s) (A molecular equation) becomes}
\]

\[
\text{Zn}^{0} + \text{Cu}^{2+} + \text{SO}_4^{2-} \rightarrow \text{Zn}^{2+} + \text{SO}_4^{2-} + \text{Cu}^{0}. \quad \text{(A full ionic equation)}
\]

What species exists on both sides?

SO\(_4^{2-}\). This is called a spectator ion and is eliminated from the above equation. We are then left with

\[
\text{Zn}^{0} + \text{Cu}^{2+} \rightarrow \text{Zn}^{2+} + \text{Cu}^{0}
\]

This is known as a “net ionic equation.” A net ionic equation includes only the species involved in a reaction.

**Redox Half-Reactions and spontaneity**

Students can take their equations from the above exercise and determine the appropriate redox half reactions.

\[
\text{Zn}^{0} \rightarrow \text{Zn}^{2+} + 2e^{-} \quad \text{(oxidation)}
\]
An additional extension activity would be to take each half-reaction, look up the reduction potentials and prove the spontaneous reactions have a $+E^\circ$ (voltage). Conversely the non spontaneous reactions have a $-E^\circ$ (voltage). In the zinc half reaction the reduction potential would be written as $-0.76$ V. Because it expressed below as an oxidation, its numerical sign is reversed.

\[ \text{Zn}^0 \rightarrow \text{Zn}^{2+} + 2e^- \quad \text{(oxidation)} \quad +0.76\text{V} \]
\[ \text{Cu}^{2+} + 2e^- \rightarrow \text{Cu}^0 \quad \text{(reduction)} \quad +0.34\text{V} \]

\[ E^\circ = +1.10\text{V} \]

**Addressing the National Science Education Standards**

- 178 Students conduct a scientific investigation in the area of single replacement chemical reactions.
- 180 Students explore the conceptual model of metal reactivity. Based on evidence from their investigation, they generate a partial activity series.
- 181 As students grapple with understanding the concept of metal reactivity, they may have to analyze and discount alternative explanations for the behavior seen.
- 183 Students inquire about the function of a physical system.
- 187, 300 Students utilize consistent logic, evidence and current scientific knowledge to generate their activity series.
- 199 The above activities are examples of chemical reactions, that will form the basis for cathodic protection of pipelines, ship hulls, and other iron objects.
- 201 Electrons are transferred in the above redox reactions.
- 202 Although not instantaneous, the above reactions occur within a ten-minute time frame.

**Addressing the Voluntary Industry Standards**

L.1.04.01 Working as a member of a lab team, students perform experiments, analyze data, and report results.

L.1.04.07 Reactivity decisions are based on data and observations.

L.1.04.08 Trends in reactivity are observed.

L.2.04.03 Good housekeeping is demonstrated by maintaining a clean and safe workplace.
Class 2.05.04 Glassware is manipulated.

L3.01.01 Both elements and compounds are utilized in this laboratory activity.

L3.01.10 Chemical reactions are written and balanced.

L3.01.13 Ionic bonding is addressed.

L3.02.01 Element symbols and groups from the periodic table are used in this activity.

L3.02.03 Formulas for common inorganic aqueous metal salt solutions are written.

L3.03.08 Reaction vessels are appropriately cleaned after being used.

L4.02.05 Several physical properties (tensile strength, hardness) of metals are discussed.

L4.04.02 Data is presented graphically.

L4.05.01 The connection between the economic impact and physical properties of materials are addressed in this lab.

L5.01.02 & L5.08.03 Redox reactions are written and balanced.

L5.08.02 Students apply the oxidation state of common ions in writing chemical formulas.

L5.08.04 Redox potentials are used to predict the outcome of oxidation-reduction reactions.

L7.03.02 Lab teams develop a strategy for conducting the above experiment.

L7.04.02 Experiments are observed.

**Addressing the SCANS Skills**

Using Workkeys Targets

**Observation**

Level 4-Students must select and attend to the important details in the reaction vessel, to establish whether a reaction has occurred.

**Reading for Information**

Level 3-Students must read the procedure to perform the experiment in the proper sequence.

**Teamwork**

Level 4-Students must organize their tasks and a time schedule, to
effectively complete the activity.

Writing

Level 3-Students will clearly write the observations they witness.

References


## Solutions

<table>
<thead>
<tr>
<th>Metals</th>
<th># of reactions</th>
<th>CuSO$_4$</th>
<th>FeSO$_4$</th>
<th>MgSO$_4$</th>
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- **CuSO$_4$**: Copper(II) sulfate
- **FeSO$_4$**: Iron(II) sulfate
- **MgSO$_4$**: Magnesium sulfate
- **AgNO$_3$**: Silver nitrate
- **ZnCl$_2$**: Zinc chloride

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Student Handout

Relevant Rusting

The Problem
Corrosion occurs all around us, whether it is the red wagon in our garage, the chicken wire fence around the ball field, or the car we drive. In this investigation you will explore corrosion.

Your Task
Use your power of observation coupled with your knowledge of chemistry to determine the chemical reactions occurring when a piece of iron rusts and the effect of galvanization on iron. You will write two paragraphs summarizing your conclusions. The information you will use includes the data from your experiments, the answers to several guided discussion questions, and finally the input from your instructor.

Materials

Part A, Per group
- 12 test tubes
- test tube rack
- pen for labeling test tubes
- eight iron strips
- 0.1 M sodium chloride (NaCl)
- 0.1 M sodium nitrate (NaNO₃)
- 0.1 M sulfuric acid (H₂SO₄)
- 0.02 M ferrous sulfate (FeSO₄) acidified
- 0.02 M ferric chloride (FeCl₃) acidified
- 0.1 M potassium ferricyanide (K₃Fe(CN)₆) in dropper bottle
- 0.1 M potassium thiocyanate (KSCN) solution in dropper bottle
- distilled / deionized water
- hand lens (magnifying glass)
- pH paper, hydron (pH4-pH10) or blue and red litmus
- steel wool
- test tube rack

Part B, Per group
- 2 petri dishes with covers
- 1.5 g powdered agar
- 100 mL 0.1 M sodium nitrate (NaNO₃)
- distilled / deionized water
- beaker tongs
• burner / hot plate
• glass stir rod
• 0.1 M potassium ferricyanide (K₃Fe(CN)₆) solution in dropper bottle
• phenolphthalein indicator in dropper bottle
• 10-cm copper wire
• 10-cm zinc wire or thin zinc strip
• steel wool
• 3 6-cm iron strips

Safety and Disposal

As instructed by your teacher, follow appropriate safety procedures, including the use of personal protective equipment such as goggles and an apron.

Dilute solutions of sulfuric acid should be handled with extreme care.

After the experiment the solutions should be treated as follows. Slowly pour the liquid into the drain ensuring the iron sample remains in the tube. Then over a trash can shake the metal out of the tube. Next, rinse the tube with water.

Procedure

Day 1-Part A

1. Obtain 12 test tubes and a test tube rack.

2. Label the tubes 1-12.

3. Label test tubes 1 and 2 as water. Label test tubes 3 and 4 as 0.1M NaCl. Label test tubes 5 and 6 as 0.1M NaNO₃. Lab test tubes 7 and 8 as 0.1M H₂SO₄.

4. Label test tubes 9 and 10 as 0.02M FeSO₄. Label test tubes 11 and 12 as 0.02M FeCl₃. Set these aside. These solutions will be used on Day 2 as controls.

   Special Note: To eliminate the formation of air bubbles, the metals must be dropped into the liquids.

5. Fill each test tube with enough of the appropriate liquid so that the iron strip will be submerged.

6. Obtain eight iron strips, clean and shine them with steel wool.

7. Place one strip of the cleaned iron from Step 6 into each of the test tubes labeled 1-8.

8. Use pH paper to determine the pH of each of the test liquids and the control. Record the data in data table 1.
9. Let the systems sit undisturbed overnight.

Day 1 - Part B

1. Heat 100 mL of 0.1M sodium nitrate (NaNO₃) to boiling in a 250-mL beaker. Add, while stirring with a glass rod, 1.5 g of powdered agar. Heat and stir the mixture until the agar forms a suspension. Do not burn the agar.

2. Add 10 drops of 0.1 M potassium ferricyanide (K₃Fe(CN)₆) and 10 drops of phenolphthalein indicator to the agar suspension. Stir to mix thoroughly.

3. Clean three 6-cm iron strips with steel wool. Place one by itself in a petri dish.

4. Use the steel wool to polish a 10-cm piece of copper wire, and a 10-cm piece of zinc wire (or strip).

5. Tightly wrap the polished copper wire from step 4 around one of the unused, cleaned iron strips. Be sure to spread the wire along the length of the strip. The wire should not be bunched at one end. Similarly, tightly wrap the piece of zinc wire (or strip) around the remaining cleaned iron strip.

6. Place both of these wrapped iron strips in another empty petri dish. Do not allow these strips to touch each other.

7. Using beaker tongs, carefully pour the warm agar suspension into both petri dishes. The iron strips need to be covered by agar to a depth of about 1-mm. If the iron is buried any deeper, the view of the corrosion process will be obscured by the opacity of the agar. Any bubbles that form while pouring should be popped with a stir rod before the agar hardens.

8. Cover both petri dishes w/ their covers. Record your observations and sketch the appearance of the contents of each in Figure 1. Let the systems sit undisturbed overnight.

Day 2-Part A
1. Using a hand-lens and your naked eye, observe each of the test tubes from day 1 against a white background. In the data table, record observations. Be critical in trying to identify differences in the appearance of the precipitates formed.

2. Carefully place 2 drops of 0.1 M potassium ferricyanide (K₃Fe(CN)₆) into the test tubes labeled 9 (FeSO₄), and 11 (FeCl₃). Record results in the attached data table from this control experiment. If a blue precipitate is produced, this is the POSITIVE test for the ferrous cation (Fe²⁺) (implying that it exists).

3. Apply the above method to the remaining odd-numbered test tubes and record the results in the attached data table.

4. Carefully place 2 drops of 0.1 M potassium thiocyanate (KSCN) to the test tubes labeled 10 (FeSO₄) and 12 (FeCl₃). If a light red solution is produced, this is a NEGATIVE test for the ferric cation (Fe³⁺) (implying it does NOT exist). If a deep blood red solution forms, this is the POSITIVE test for Fe³⁺.

5. Apply the above method to the remaining even-numbered test tubes and record the results on the attached data table.

Part B

1. Observe the petri dishes against both a white and a black background. Record your observations and sketch the appearance of the contents of your dishes in Figure 1. Be sure to show the location and color the various regions.

**Discussion Questions**

**Part A**

1. What is the oxidation state of iron in FeSO₄?

2. Describe what happens when you added K₃Fe(CN)₆ to FeSO₄.

3. Describe what happens when you added K₃Fe(CN)₆ to FeCl₃.

4. What is the oxidation state of iron in FeCl₃?

5. Describe what happens when you added KSCN to FeSO₄.

6. Describe what happens when you added KSCN to FeCl₃.

7. Examine the data from part A. Did all the iron strips corrode in the same manner? Explain. How many different types of corrosion you observed in the eight different reaction tubes?

8. Did the ferric cation (Fe³⁺) exist in any of the corroding samples? How do you
Part B

1. Did either the copper or zinc appear to protect the iron strip against corrosion? Explain.

2. Propose the relative reactivities of the three different metals in this experiment. Explain.

3. Is it correct to say that corrosion did not take place in the nail wrapped with zinc? Explain.

Conclusions

It is your job to compile the information from your data table, the information from the above discussion questions, and the information from your teacher’s discussion to write two summarizing paragraphs. The first paragraph needs to discuss the reactions from part A. The topics you need to include are:

The precipitates formed

Soluble ions

Evidence of corrosion

The second paragraph needs to discuss the reactions from part B. You need to include:

What your sketch of the first dish implies including the colors of the indicators and any precipitates formed.

What your sketch of the second dish implies including the colors of the indicators and precipitates formed.

How galvanization works

Assessment

Completed data table (30 pts)

Discussion questions (30 pts)

Conclusion summary paragraphs (40 pts)
Figure 1. Use the circles above to draw what you saw in your petri dishes on day 1. On day 2 revise these drawings to reflect the changes observed overnight.
<table>
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<tr>
<th>test tube</th>
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Instructor Notes

Relevant Rusting

In this activity, students observe numerous chemical reactions affiliated with the corrosion process. Iron does not undergo a single pathway when it rusts. There are three different mechanisms present in this exercise. Through observation, students will elucidate the different underlying chemical mechanisms. In a post lab discussion, the teacher will build on the conclusions made by the students, establishing the chemistry of corrosion.

Time Required

Setup: 30 minutes to prepare solutions + 20 minutes to gather equipment + time required to cut Fe strips, Cu strips, Zn strips.

Day 1: 50 minutes for students to set up and observe part A and part B

Day 2: 70 minutes for students to observe, record data and answer questions part A and part B (can be split)

Day 3: 50 minutes for teacher to discuss student data

Cleanup: 30 min

Key Science Concepts

Chemistry
- aqueous solutions
- cathodic protection (galvanization)
- compounds
- equilibrium
- pH
- reaction mechanisms
- redox reactions
- solubility
- solvation
- spontaneous reactions
- types of reactions

Materials

Part A, Per group
- 12 test tubes
- pen for labeling test tubes
- eight iron strips (available from Flinn Scientific, or cold rolled steel, cannot be coated with zinc (galvanized), or oil. Iron nails may be substituted.)
• hand lens (magnifying glass)
• 0.1 M sodium chloride (NaCl)*
• 0.1 M sodium nitrate (NaNO₃)*
• 0.1 M sulfuric acid (H₂SO₄)*
• 0.02 M ferrous sulfate (FeSO₄)*
• 0.02 M ferric chloride (FeCl₃)*
• 0.1 M potassium ferricyanide (K₃Fe(CN)₆) in small dropper bottle**
• 0.1 M potassium thiocyanate (KSCN) in small dropper bottle**
• distilled/deionized water
• pH paper
• steel wool (used for cleaning iron strips. Cannot contain soap, as in a scouring pad)
• test tube rack

Part B Per group
• 2 petri dishes with covers
• 1.5 g powdered agar (not nutrient agar)
• 100 mL 0.1 M sodium nitrate (NaNO₃)
• distilled/deionized water
• beaker tongs
• burner/hot plate
• glass stir rod
• 0.1 M potassium ferricyanide (K₃Fe(CN)₆) in small dropper bottle***
• phenolphthalein indicator in dropper bottle***
• 10-cm copper wire (copper wire is often used for winding motor armatures. In this case, the copper wire is coated with an almost imperceivable clear coating and will seriously adversely affect this activity. Check your copper for this.)
• 10-cm zinc wire or thin zinc strip
• steel wool (used for cleaning iron strips. Cannot contain soap, as in a scouring pad)
• three 6-cm iron strips (available from Flinn Scientific, or cold rolled steel, cannot be coated with zinc (galvanized), or oil. Iron nails may be substituted.)

*The exact amount required of these solutions will depend on the size of the reaction vessels used. You can minimally estimate the volume as being twice the volume of the test tubes to be used multiplied by the number of groups performing the experiment.

**You can minimally estimate needing 2 drops of each of these solutions per group performing the experiment.

***You can estimate the minimum volume needed of this solution as being about 0.5mL per group performing the experiment.
Safety and Disposal

It is your responsibility to review appropriate safety procedures with your students, including the use of personal protective equipment.

After the experiment the students should treated the solutions as follows. First they should slowly pour the liquid into the drain ensuring the iron sample remains in the tube. Then over a trash can they should shake the metal out of the tube. Finally the tubes should be rinsed with water.

The petri dishes should be left open to dry, and then the agar will harden before disposal.

Procedure Notes

In part A, students will subject iron strips to various corrosive environments. Through their observation of experimentation, guided questions, and an instructor guided discussion, students will determine that there are at least two (actually three) different chemical mechanisms iron is undergoing during the process of corrosion. They will write a paragraph summarizing their conclusions.

In part B, students will subject protected and unprotected iron to a corrosive environment within an agar petri dish. Through the use of indicators in their experimentation they will witness corrosion and the protective process of galvanization. As above, this experimentation is followed up with guided questions, an instructor guided discussion, and lastly the students will again write a paragraph summarizing their conclusions.

If your class is large, you might want to divide the work up by having half of the class doing Part A and the other Part B. If you do this, be sure that students share the observations as they proceed so that the entire class gets the benefit of the observations and thus can contribute to the class discussion on the topic.

Discussion Questions

Part A

1. What is the oxidation state of iron in FeSO₄?

   2+

2. Describe what happens when you added K₃Fe(CN)₆ to FeSO₄.

   A blue precipitate (Prussian blue) is formed. This is the positive test for Fe²⁺. See explanation for more details.

3. Describe what happens when you added K₃Fe(CN)₆ to FeCl₃.
No evidence of chemical change.

4. What is the oxidation state of iron in FeCl₃?

3+

5. Describe what happens when you added KSCN to FeSO₄.

A light red solution is formed. This is not a strong indication of the Fe³⁺ ion. A small amount is present due to the oxidation of the Fe²⁺.

6. Describe what happens when you added KSCN to FeCl₃.

A deep blood-red solution is formed. This is the positive test for Fe³⁺. See explanation for more details.

7. Examine the data from part A. Did all the iron strips corrode in the same manner? Explain. Propose how many different types of corrosion you observed in the eight different reaction tubes.
Students should observe at least two different types of corrosion. At a minimum, tubes 1-6 contain a rust colored precipitate and tubes 7&8 do not. Additionally the presence of Fe $^{2+}$ is confirmed in tube 7, but no where else. This proves at least 2 different chemical pathways. This is the whole picture:

Tubes 1&2 (water) the typical rust-colored precipitate is noticed.

Tubes 5&6 (0.1 M NaNO$_3$) has the same rust colored precipitate as above, but possibly a larger quantity due to the increased conductivity in an ionic solution.

Tubes 3&4 (0.1M NaCl) has a slightly more yellow precipitate than above. It is also less adhesion to the metal. This may be difficult to notice by all but the most careful observers. The next activity (Working as a Lab Tech for ElectroChem Inc.) has this reaction in a much larger scale, and will provide greater chance for detection of this second mechanism.

Tubes 7&8 (0.1 M H$_2$SO$_4$) Provide the third mechanism. No rust colored precipitate is observed. These tubes may have bubbles on their metal surfaces. This is the production of hydrogen gas. Additionally the positive test for Fe $^{2+}$ is seen only in tube 7. This positive test in addition to the radical difference in appearance from tubes 1-6 implies a new mechanism.
8. Did the ferric cation (Fe$^{3+}$) exist in any of the corroding samples? How do you know?

No. The blood-red solution observed in the control experiment was not observed in tubes 1-8.
Part A Control Tubes on Day 2 after indicators added

Part B

1. Did either the copper or zinc appear to protect the iron strip against corrosion? Explain

The iron strip corroded with the copper. The iron strip appears unchanged with the zinc.

2. Propose the relative reactivities of the three different metals in this experiment. Explain.

Zn is more reactive than Fe and Cu

Fe is more reactive than Cu

Cu is least reactive

With both Zn and Fe present, the Zn reacted preferentially.

With both Fe and Cu present the Fe reacted preferentially.

The Cu exhibited no evidence of corrosion, thus being the least reactive metal.

3. Is it correct to say that corrosion did not take place in the nail wrapped with zinc? Explain.

No, the phenolphthalein indicator is turning pink indicating a base being formed. There is a white precipitate, Zn(OH)$_2$ forming near the zinc. There is no corrosion of the iron, but corrosion of Zinc is occurring. See explanation for more details.
Oxidation of iron seen as rust-colored
Fe(OH)$_2$(s)/Fe(OH)$_3$(s) in the anodic region.

Darkening of the agar is the positive test for the Fe$^{2+}$ cation in the anodic region.

Phenolphthalein indicator turns pink indicating the presence of OH$^{-}$ in the cathodic region.

Phenolphthalein indicator turns pink indicating the presence of OH$^{-}$ in the cathodic region.
Part A

In Part A of this experiment, students explore the rusting of iron metal in various environments. This discussion is presented in the order in which the students conducted...
their procedure.

**pH tests:**

Students should see that you can only have a soluble iron ion (either ferrous or ferric) in acidic pH. Or expressed in the opposite fashion, a precipitate of iron will only exist in neutral or basic pH.

**Control experiments:**

Tube 9 & 10 contain Fe²⁺ and Tube 11 & 12 contain Fe³⁺.

Students add the indicator K₃Fe(CN)₆ (potassium ferricyanide) to tubes 9 and 11.

The presence of a deep blue complex ion (a positive test) indicates the presence of Fe²⁺. The color precipitate, also called Prussian Blue is formed through the following sequence of reactions:

$$Fe^{2+} + [Fe(CN)_6]^{3-} \rightarrow Fe^{3+} + [Fe(CN)_6]^{4-}$$

$$4Fe^{3+} + 3[Fe(CN)_6]^{4-} \rightarrow Fe_4[Fe(CN)_6]_3 \text{ (deep blue precipitate)}$$

This positive test is seen in tube 9.

Students add the indicator KSCN (potassium thiocyanate) to tubes 10 and 12.

The presence of a deep blood-red solution (a positive test) indicates the presence of Fe³⁺. This complex is soluble in water.

$$Fe^{3+} + SCN^- \rightarrow FeSCN^{2+}$$

This positive test is seen in tube 12.

**The summary of the ferrous ion tests are below.**

In the test tube labeled water, a negative test result is seen, as all the Fe²⁺ is combined to form insoluble Fe(OH)₂. In the test tube labeled NaCl, a negative test result is seen, as all the Fe²⁺ is in the form of either (FeCl₄)²⁻ or Fe(OH)₂. In the test tube labeled NaNO₃, all the Fe²⁺ is combined to form insoluble Fe(OH)₂ (as in the water case). And finally, a positive test is seen as there is Fe²⁺ formed in the H₂SO₄.

In general, ferrous (Fe²⁺) ion is much less stable than ferric (Fe³⁺) ion. Iron has an electron configuration ending in 4s²3d⁶. In the formation of a ferric ion the electron configuration contains an empty 4s orbital and a five electrons in the 3d orbital. In the
formation of a ferrous ion, the electron configuration contains an empty 4s orbital and six electrons in the 3d orbital. The ferric case with a half-filled d orbital is more stable.

Additionally, Fe$^{2+}$ is generally a clear, colorless ion and Fe$^{3+}$ is an amber colored ion. Under many circumstances Fe$^{2+}$ converts to Fe$^{3+}$. (especially in neutral / basic environments) This is why the FeSO$_4$ is purchased as an acidified version, and the only positive test for Fe$^{2+}$ (in addition to the control, FeSO$_4$) was in the sulfuric acid.

The summary of the ferric ion tests are below.

The ferrous hydroxide precipitate (Fe(OH)$_2$) will oxidize to a ferric hydroxide (Fe(OH)$_3$) precipitate over time. Although Fe$^{3+}$ is present in test tubes 2,4,6,8, and 12, in all but test tube 12, the Fe$^{3+}$ is in the form of an insoluble precipitate. This is why the only positive test for Fe$^{3+}$ was the control, FeCl$_3$.

In the test tubes containing water:

The typical rust-colored precipitate begins to form on the iron and may fall to the bottom of the test tube. The mechanism for this process is provided in The Prologue to the entire corrosion unit. See I. Rust formation in the presence of oxygen in a neutral or basic environment, for specific details.

In the test tubes containing 0.1 M NaCl:

The typical rust-colored precipitate begins to form on the iron and may fall to the bottom of the test tube. The rate may be increased compared to the water because electrochemical processes will increase in an ionic electrolyte. Additionally, a different, competing mechanism is also present. The mechanism for this process are provided in The Prologue. See III. Rust formation when chloride is present in a neutral or basic environment, for specific details. Both this new mechanism and the one above are found within this test tube. Students may recognize a different appearance in this environment.

In the test tubes containing 0.1 M NaNO$_3$:

The typical rust-colored precipitate begins to form on the iron and may fall to the bottom of the test tube. The mechanism is identical to that of water. A larger quantity of precipitate may be noted. (Electrochemical processes increase in rate in an ionic solution)

In the test tubes containing H$_2$SO$_4$:

No rust is noted, but the appearance of bubbles (H$_2$) may occur. Students may report that no corrosion has occurred, but in this case a substantially different mechanism is in play.
from that observed in the other test tubes. The mechanism for this process are provided in The Prologue. See II. Iron oxidation in the presence of oxygen in an acidic environment, for specific details.

**Part B**

In part B of the experiment, students explored the rusting process in an agar media. In petri dish 1, corrosion of the iron has occurred. The cathode and anode regions are clearly seen. The cathode is the region where the phenolphthalein indicator within the agar has turned pink.

At the cathode, oxygen gas \( \text{O}_2^0 \) in the presence of water is reduced to \( \text{O}^{2-} \) in the form of hydroxide anions.

\[
8e^- + 2\text{O}_2(g) + 4\text{H}_2\text{O}(l) \rightarrow 8\text{OH}^-(aq)
\]

The oxygen available for this reaction is atmospheric oxygen dissolved in the water.

The formation of the intermediate hydroxide (OH\(^-\)) (from the oxygen and the water) is seen, prior to its migration to the anode. The anodic region is where the rust has formed. Additionally the anodic region should also contain a darkening of the agar. This is the formation of \( \text{Fe}^{2+} \) and the positive test of the indicator \( \text{K}_3\text{Fe}(<CN>)_6 \) potassium ferricyanide.

In petri dish 2, corrosion has occurred in both cases. The copper-wrapped iron strip should have corrosion identical to that described above in petri dish 1. Copper is a less active metal than iron, and provides no galvanic protection. Contrary to copper, zinc is a more reactive metal and more likely to oxidize than iron. Iron is thus uncorroded in this case. This is characteristically known as galvanization or cathodic protection. The zinc is the sacrificial anode, (iron is no longer the anode. In fact, iron still gives up electrons to the system, but because zinc is a more easily oxidized than iron, the zinc immediately transfers electrons to the iron ions, reducing them back to neutral iron atoms.) No darkening of the agar exists, (no \( \text{Fe}^{2+} \) is indicated by the \( \text{K}_3\text{Fe}(<CN>)_6 \) potassium ferricyanide.) A white precipitate is forming around the zinc strips, this is the formation of zinc hydroxide \( \text{Zn(OH)}_2 \). This is certainly a preferred species as compared to \( \text{Fe(OH)}_2 \). The iron has been protected from corrosion.

**Addressing the National Science Education Standards**

- 178 Students conduct scientific investigations to explore corrosion mechanisms.
- 201 In participating in the teacher directed discussion, students see how all corrosion reactions involve the transfer of electron (redox).
• 299 Students witness the logical explanations during the teacher directed discussion.
• 300 Students provide a scientific explanation for their observations in their concluding paragraphs.
• 301 After witnessing the protection of iron in the process of galvanization, students’ scientific knowledge about corrosion will be subjected to change.

**Addressing the Voluntary Industry Standards**

L1.04.03 Students demonstrate problem solving in elucidating what their data implies about corrosion pathways.

L1.04.05 Students demonstrate critical thinking skills in processing their data.

L1.04.06 Students need to set up 2 parallel experiments with differing objectives in the same day.

L1.04.07 Students need to make decisions about corrosion mechanisms based on their data and observations.

L2.04.02 Students are expected to abide by safety protocols in the performance of this activity.

L2.05.04 In this lab students manipulate various types of glassware.

L2.05.04 Students demonstrate a basic awareness of electrical safety in using the hot plate to prepare agar.

L3.01.08 Students demonstrate how atoms combine to form molecules.

L3.02.02 Students gain familiarity with the names ferrous and ferric in describing iron’s ionic charges.

L3.02.03 Formulas are written for common inorganic compounds.

L3.03.08 Students are expected to clean their laboratory glassware upon completion of this activity.

L3.05.05 The issue of homogeneity is addressed in the preparation of the agar.

L5.01.02 Students describe chemical reactivity by writing and balancing equations involving oxidation-reduction reactions.

L5.01.03 Color is addressed in the use of indicators.

L5.01.05 Insoluble precipitates are addressed in this activity.

L5.06.01 pH scale (acid/base) is explored and connected to corrosion.

L5.08.02 & L5.08.03 & L5.08.04 Redox reactions are explored, oxidation states discovered, and the gaining and loss of electrons are seen in the various half reactions involved in the corrosion of iron.
L5.11.02 Precipitation reactions are observed.
L7.03.06 Students conduct a control experiment.
L7.04.02 Observations are recorded.
L7.05.02 Results of experimentation are summarized in a conclusion paragraph.
L8.02.03 LeChatlier's principle is addressed in discussing competing mechanisms.

**Addressing the SCANS Skills**

**Using Workkeys Targets**

**Listening**

Level 5- The teacher conducts a discussion which contains information that students must connect to their data and observe the relationships between various aspects of the experimentation.

**Observation**

Level 6 – Students must use their observations to evaluate their data and make chemical predictions about corrosion pathways.

**Teamwork**

Level 4- Students work as a team to gather and analyze their data.

**Writing**

Level 5- Students are expected to write a cohesive paragraph based on information gathered from various sources including experimentation, guided questions, and teacher led discussion.

**References**


<table>
<thead>
<tr>
<th>test tube</th>
<th>solution</th>
<th>pH</th>
<th>Fe^{2+} present</th>
<th>observations</th>
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<tr>
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<td>water</td>
<td>neutral</td>
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<td>H₂SO₄</td>
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<td>FeSO₄</td>
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<td>no rust present.</td>
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<tr>
<td></td>
<td>FeCl₃</td>
<td>controls</td>
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<td>no rust present.</td>
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</table>

**Data Table 1**
Student Handout

Working as a Lab Technician for ElectroChem Labs, Inc.

The Problem
You and several of your colleagues are new employees with ElectroChem Labs, an independent analytical testing laboratory. ElectroChem Labs works extensively with standard lab procedures. Such procedures have been established by the American Society for Testing and Materials (ASTM). Independent laboratories from around the country (including ElectroChem) adhere to these standard techniques in order to obtain reproducibility of their results. Electrochem Labs has recently won the contract for all the analytical testing of The Bridge Analysis and Redevelopment Corporation (BARC). Your immediate supervisor has assigned the BARC account to your newly-formed team. The quality of the analytical work you perform for this client makes the first impression of your competence in your new job!

Your Task
Your team will receive a Request for Sample Analysis form from the Bridge Analysis and Redevelopment Corporation. BARC is interested in the corrosion of steel (a form of iron metal) in various environments. Because the rate of corrosion under normal conditions is low, most of the testing environments are in an accelerated mode. Testing labs regularly use this approach to obtain results in one week that are equivalent to a substantially longer time period. Fortunately, such standard procedures have been already established for measuring the corrosion of iron metal. Your team will investigate the rate of corrosion of an uncoated metal specimen in two different accelerated environments, be assessed on your ability to implement these standards procedures, and provide a written and oral report on your findings.

The possible accelerated environments are:

• Water
• Heated water
• Salt solution
• Acidic solution

Materials
Per class
• steel samples
• electronic balance
• deionized or distilled water
• digital camera / computer/ projector for oral presentations (optional)

Per group

*Dependent upon the assigned standard procedures, see standards for details*

**Safety and Disposal**

As instructed by your teacher, follow appropriate safety procedures, including the use of personal protective equipment such as goggles and an apron. No special disposal procedures are required.

**Procedure**

*NOTE: this activity and the ones that follow are presented in a different manner as compared to the other activities in this unit. It involves the use of an approved standard method that must be followed explicitly as written to ensure validity of the results. Industrial, independent, and government laboratories throughout the U.S. adhere to these standard methods as a means of guaranteeing accuracy and reproducibility of their results.*

*Being able to read and following approved ASTM (American Society for Testing and Materials) methods is an important workplace skill. Part of your assessment for this activity will involve your ability to complete the standard methods provided.*

1. Familiarize yourself with the attached standard procedures for the preparation, testing, and evaluation of test specimens (A-1, A-2a-A2e, and A-3).

2. You will be given a “Request for Sample Analysis” document. It specifies environments you will test iron corrosion. Familiarize yourself with the environments that you will be responsible for testing as specified by the Bridge Analysis and Redevelopment Corporation’s Request for Sample Analysis.

3. Write a Lab Analysis Work Order. This is the document ElectroChem Labs generates for each experiment the company performs.


5. Test your sample in the assigned accelerated environment by referring to the appropriate version of Standard Procedure A-2: Standard Method(s) for Testing Uncoated Metal Specimen(s) for Corrosion (in varying environments).

*NOTE: The various environments will cause subtle differences in the appearances of the corroding metals.*

7. Prepare both written and oral reports on your findings.

**Discussion and Conclusions**

Each testing team should prepare a report that includes the following:

Request for Sample Analysis (10pts)

Lab Analysis Work Order (10pts)

Goals of your experiment - This includes a statement of the testing objective and the type of environments tested. (5pts)

Background chemistry - Display evidence of your knowledge about the chemistry of corrosion. (10 pts)

Experiment design - Include the source of the procedures and a brief synopsis of what was done. Diagrams are appropriate. (10 pts)

Experimental results – This includes appropriate organization of your data presenting in more than one format, one of which must be graphical. (10 pts)

Error analysis – Discuss what did go wrong, what could have gone wrong, and how this would affect your data. (10 pts)

Conclusion – A summary of the team’s findings. (5 pts)

Future Experimentation – Describe, in detail, a follow up experiment that your team could conduct based on your results. (5 pts)

Team Analysis – Describe how well your team functioned. (5 pts)

**Assessment**

Your grade on this activity includes:

- the written report on your findings (as above) (80 points)

- adherence to standard procedures (as below) (maximum 44 points)
Adherence to Standard Procedures Rubric

Unacceptable- flaw in a critical aspect of procedure or more than three flaws present

Adequate- flaw in three non-critical aspects of procedure

Average- flaw in two aspects of procedure

Good- flaw in one aspect of procedure

Excellent- procedure performed without flaw
**Table of Adherence to Standard Procedures**

<table>
<thead>
<tr>
<th>Standard</th>
<th>Procedure</th>
<th>Grade</th>
</tr>
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<tbody>
<tr>
<td>A - 1</td>
<td>Labeling specimens</td>
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<td>A - 1</td>
<td>Cleaning specimens</td>
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<td>A - 1</td>
<td>Preparing specimens</td>
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<td>Apparatus</td>
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<td>A - 3</td>
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<td>A - 3</td>
<td>Recorded Properly</td>
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<tr>
<td>A - 3</td>
<td>Mass Loss Cleaning</td>
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<tr>
<td>A - 3</td>
<td>Area Coverage</td>
<td></td>
</tr>
<tr>
<td>A - 3</td>
<td>Calculations</td>
<td></td>
</tr>
</tbody>
</table>

Grade:

- 0 = Unacceptable
- 1 = Adequate
- 2 = Average
- 3 = Good
- 4 = Excellent

*10 minute oral report of your findings presented to the class. This should include the indication of corrosion by both the mass loss and area coverage methods. Digital photos may be used to describe observations. (30 pts)*
Instructor Notes

Working as a Lab Technician for ElectroChem Labs, Inc.

In this activity, students are given a scenario in which they are working as chemical technicians. Working in teams, students examine some of the variables that affect the rate of corrosion, all within the framework of industrial standards. Over one week, the students test samples of untreated steel in two different accelerated corrosion environments and provide both a written and an oral report detailing their findings. As the instructor, your role will be that of lab manager at ElectroChem. You assign testing environments using a standard “Request for Sample Analysis” form, monitor team progress throughout the students’ research, and assess the teams’ reports.

Time Required

Setup: 30 minutes
Standard Procedure A-1: 40 min
Standard Procedure A-2a through A-2d: 20 min
Reaction: 3-4 days or longer
Evaluation: A-3 50 minutes
Cleanup: 30 minutes

Key Science Concepts

Chemistry

• combination reactions
• single replacement reactions
• electrochemistry
• electrolytes
• measurement
• reaction rates
• redox reactions
• solution concentration (m/v %)

Mathematics

• mass/volume percentage
Engineering and Technology

- American Society for the Testing of Materials (ASTM) standards

Prior Concepts and Skills Needed

Students should have an understanding of the corrosion process and how ionic or acidic environments promotes electron transfer in such a reaction.

Materials

For Getting Ready
For Salt Water Immersion Environment

- sodium chloride, (NaCl) reagent grade is preferred although not necessary. Household table salt contains an anticingaking agent which clouds the water upon dissolving. If used, the concentration will be slightly below 3%, but this is not critical. Kosher salt can also be used as a substitute
- overhead transparency

For the Procedure
Per class

- adequate steel strips 3-inch x 1/2-inch or other samples of cold rolled (uncoated) steel
  Flinn Scientific sells 6-inch x 1/2-inch strips that can easily be cut in half. Nails will also work, but they cannot be galvanized and will cause difficulty with the area coverage method of evaluation.
- distilled or deionized water
  Deionized water will work better because the dissolved oxygen required for rusting has not been removed (as in boiling). Distilled water that is in an open environment has likely dissolved the requisite oxygen. Fresh distilled water can be shaken to dissolve oxygen.
- digital camera, computer, and/or projector
- glass stir rods, wooden dowel rods, or pencils
- Parafilm® or Saran® Wrap
  The Parafilm® must be of the large roll type. The standard 4-inch roll will not cover the 600 mL beakers sufficiently.
• spatula with flat side (If spatulas are not available, a flat head screwdriver or other similar instrument will work.)

• steel wool grade 00

• evaluation grids (found at the end of Standard Procedure A-3 for the Area Coverage Method)

For Salt Water Immersion Environment
• sodium chloride (NaCl)
Reagent grade is preferred although not necessary. Household table salt contains an anticaking agent which clouds the water upon dissolving. If used, the concentration will be slightly below 3%, but this is not critical. Kosher salt can also be used as a substitute

For Acid Immersion Environment
• vinegar

Additional materials are needed and are group dependent. See standard procedures A-1, A-2a, A-2b, A-2c, A-2d, A-3 for details.

Safety and Disposal
It is your responsibility to review appropriate safety procedures with your students, including the use of personal protective equipment. No special disposal procedures are required.

Getting Ready
Divide the class into teams. Have the students name their teams as an important means of identifying samples later on.

Make a 3% (m/v) NaCl solution by dissolving 30 g sodium chloride (NaCl) in sufficient distilled or deionized water to make 1 L of solution.

For students to evaluate their corrosion using the Area Coverage Method (Standard Procedure A-3), make transparent plastic grids. A master is provided at the end of Standard Procedure A-3. Copy the master onto a clear transparency cut and distribute the grids to the teams.

Fill out the “Request for Sample Analysis” documents (or use the ones provided) and have them ready at the beginning of the lab.
Procedure Notes

The students are given an overview of their procedure in the student handout. They must also consult the three “standard procedures” in order to formulate their plan of action. The instructor should play a supportive role, but interpretations of the standard procedures should be made by the student team without guidance and support. Students may initially resist the open ended nature of this experiment.

Students will prepare their samples, prepare an accelerated environment, test their samples, and after a specified time (approximately one week) evaluate their samples. Students should make observations of their samples daily within the week period of time. The area coverage method should be done prior to the mass loss method. If you would like to run the experiment for a week, a timetable would be:

Monday – prepare samples

Tuesday, Wednesday- observations only

Thursday- observations and drying

Friday- evaluation of corrosion

Mon- students present their results to class

Tuesday, Wednesday and Thursday will not be full lab days. If more than one week can be dedicated to this project, the degree of corrosion will be greater and thus the data would be more definitive.

Assessment

Students are assessed on their final report, their adherence to standard procedures, and their oral presentation. These are detailed in the student handout. It is important for the instructor to record student work during the activity and evaluate their product later at a specified time. In this manner, a student cannot claim, “But our samples were not ready yet.” Additionally, you may want to assess students on their lab technique throughout this activity.

Guidelines for grading adherence to standard procedures:

Unacceptable - team shows no evidence of awareness of procedure in their work

Adequate – team shows evidence that they are aware of the standard procedure and have made a minimal attempt to adhere to it.
Average – team demonstrates in much, but not all of their work an awareness of and adherence to the procedure

Good – team demonstrates in most observed situations adherence to most but not all of the procedure

Excellent – team demonstrates in all observed situations strict adherence to the procedure

**Explanation**

Throughout this activity, the students experience an industrial laboratory environment. They begin to see some of the variables involved in the corrosion of iron. Fundamentally, iron will corrode in the presence of oxygen, but typically this only happens in the presence of water (it is the oxygen dissolved in the water that causes the rusting). Redox reactions require electron exchange. A solvent with dissolved ions tends to promote this exchange, thus the acidic and salt immersion environments should have a larger degree of corrosion. See *The Prologue* for a full description. The mechanistic differences in the corrosion pathways of iron in salt water is discussed in section III, vinegar (acetic acid) is discussed in section II, and water is discussed in section I.

If this activity is completed after *Relevant Rusting*, the students should notice great similarity to the reactions performed earlier. This provides solid reinforcement of their previous knowledge, and these examples are in a much larger scale. In water and hot water your students should notice typical rusting. In the salt environment the rust is a little more porous and a slightly lighter color. This is due to a chloride intermediate being formed. In the vinegar environment, it will appear that no corrosion occurs until after the material is removed from the container. The acidic mechanism produces a soluble iron ion so the surface appears clean (but significant mass is lost in the process).

As temperature increases, so does the rate of reaction, and thus one would expect increased corrosion in the heated water environment. Yet, gas solubility decreases with temperature and thus the quantity of oxygen available for corrosion could be diminished. The hot water (30°C) environment, however, maintains ample oxygen for accelerated corrosion and it is observed in this technology.

According to Dr. Sheldon Dean, of Dean Corrosion Technology, the somewhat tedious ASTM standard procedures are central to the nature of corrosion testing. Even so, corrosion is a somewhat random event and it is extremely difficult to reproduce experimental data.
Extension Activity

Although this activity does not directly investigate the various mechanisms iron undergoes in the corrosion process, the accelerated environments can be tested using complexes of iron in the same manner as described in part A of the previous activity in the corrosion series, **Relevant Rusting**. The testing should occur at the same time the specimens are removed for drying.

Addressing the National Science Education Standards

• 177 With background knowledge of corrosion on hand, students connect the design of the experiment to the variable given
• 178 When setting up their corrosion environment, students conduct an investigation. Students then present their results using cohesive arguments.
• 179, 186 Mathematics are utilized in the area coverage method of corrosion determination. Additionally, graphical representation of results are conveyed.
• 182, 264 Scientific arguments are communicated and defended in their reports.
• 184 Students conduct experiments to explain observed corrosion.
• 187 Logic and evidence are considered in drafting the final report.
• 188: In communication within and between groups of students, students report clearly their scientific discoveries.
• 199 The ubiquity of corrosion is explored.
• 202 Students gain an appreciation for the varying rates of corrosion in different environments.
• 260 The final report requires students to design future experimentation.
• 263 Students evaluate their experiments to determine if they meet the designated standard procedures.
• 284 Corrosion is a basic process that affects humans.
• 289 Corrosion is a slow and progressive process that negatively affects society.
• 290 Corrosion is a natural process that requires humans to assess its potential danger and risk.
• 293 Although iron is subject to corrosion, suitable non-corroding building materials are slow to market due to economic considerations.
• 299 Through a students use of standard procedures, they gain an understanding of the empirical standards typified in science.
• 300 In the final written report, explanations must meet scientific criteria.

Addressing the Voluntary Industry Standards

L1.03.02 Students take on the role of process technicians.
L1.04.01 Students work as members of a larger team.
L1.04.08 Students observe the details and trends of an object corroding throughout the duration of the experiment.
L2.04.02 Students demonstrate the appropriate use of eye protection.
L2.04.03 Students maintain a clean and safe workplace.
L2.05.01  Students handle common lab equipment.
L2.05.07  Through the proper use of temperature controlled hot plates, students show awareness of electrical safety.
L3.01.08  Students display their knowledge of atoms combining to form molecules in the formation of iron(III) oxide (Fe$_2$O$_3$).
L3.01.10  Students write and balance the equations applicable to corrosion.
L3.02.03  Students write the name and formula for the common inorganic substance ferric oxide (iron(III) oxide).
L3.03.08  Students are expected to properly clean their testing apparatus upon completion of the activity.
L4.01.01  Both the mass loss method and the area coverage method display the importance of measurement in chemistry.
L4.03.01  The standard procedures in this activity are based on ASTM standards.
L4.03.04  Students carry out standard stepwise procedures in this activity.
L4.04.02  Data is presented graphically in the final report.
L4.06.04  A final report is required which includes principles of the method, conclusions, next steps, and follow up.
L5.01.02  Chemical reactivity is described using oxidation-reduction reactions.
L5.02.01  Familiarity is gained with the sections of published standard methods of chemical analysis.
L5.02.03  Familiarity is gained with associations that provide standard methods of chemical analysis.
L5.03.03  Solution concentration is expressed as % (m/v).
L5.13.01  Rates of chemical reactions are explored.
L7.02.04  Teamwork is explored in this activity.
L7.04.02 & L7.04.07  Corrosion progress is recorded throughout the duration of this experiment.
L7.04.03  Data is expressed graphically.
L7.05.02  A written report documents results of the experiment.
L7.05.03  Students prepare and present an oral report.

**Addressing the SCANS Skills**

**Using Workkeys Targets**

**Applied Mathematics**

- **Level 3-** Students perform a translation from a verbal setup to a math equation (area coverage method).
- **Level 4-** Students perform a ratio calculation (area coverage method).

**Applied Technology**
Level 3- Students understand the operation of simple electrical heaters.

Locating Information
Level 6- When using standard procedure documents, students apply information to only their described experiment.

Reading for Information
Level 5- Students will recognize the application of technical terms to stated situations. Additionally, students will recognize the application of instructions from the document to new situations that are similar. Standard procedures with multiple steps must be followed.

Teamwork
Level 3- Students recognize the need for trust and dependability in their team environment.
Level 5- Students determine the best use of their team’s talents to accomplish goals.

References
“The Prologue”
“Relevant Rusting” activity
Sheldon Dean, Dean Corrosion Technologies, Philadelphia PA. Sheldondean@deancorrtech.com
# Request for Sample Analysis

<table>
<thead>
<tr>
<th>Name of Contact Person</th>
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<tbody>
<tr>
<td>Name of Laboratory doing Analysis</td>
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<td>St. Address</td>
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<tr>
<td>City, State, Zip Code</td>
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<tr>
<td>Phone No.</td>
<td>Fax No.</td>
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</table>

Date of Request

Description of problem

Description of specimen materials

Specific analyses to be performed including requirements within standards

Date results required: ____________
Request for Sample Analysis

D'Agostino, Mike
Name of Contact Person

Electrochem Labs Inc
Name of Laboratory doing Analysis

4717 Precipitation Drive
St. Address

Labland, OH 26817
City, State, Zip Code

301
Request No.

1-800-355-5585 1-800-355-1079
Phone No. Fax No.

20 Oct 2004
Date of Request

The Bridge Analysis and Redevelopment Corporation (BARC) has contracted AMS Steel in the development of a corrosion resistant alloy for possible use in future bridge construction.

Description of specimen materials
3 inch X 1/2 inch sample of new proprietary alloy (attached)

Specific analyses to be performed including requirements within standards
Accelerated corrosion test in water environment to simulate the effect of snow / rain on bridge base metal A-1, A-2a, A-3

Date results required: 15 Nov 04
Request for Sample Analysis

D’Agostino, Mike  
Name of Contact Person

Request Sent to:

Electrochem Labs Inc  
Name of Laboratory doing Analysis

4717 Precipitation Drive  
St. Address

Labland, OH 26817  
City, State, Zip Code

1-800-355-5585  
Phone No.

1-800-355-1079  
Fax No.

302  
Request No.

20 Oct 2004  
Date of Request

Date results required: 15 Nov 04

The Bridge Analysis and Redevelopment Corporation (BARC) has contracted AMS Steel in the development of a corrosion resistant alloy for possible use in future bridge construction.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Accelerated corrosion test in heated water environment to simulate the effect of summer rains on bridge base metal A-1, A-2b, A-3
Request for Sample Analysis

D'Agostino, Mike
Name of Contact Person

Request Sent to:

Electrochem Labs Inc
Name of Laboratory doing Analysis

4717 Precipitation Drive
St. Address

Labland, OH 26817
City, State, Zip Code

1-800-355-5585 1-800-355-1079
Phone No. Fax No.

303
Request No.

20 Oct 2004
Date of Request

The Bridge Analysis and Redevelopment Corporation (BARC) has contracted AMS Steel in the development of a corrosion resistant alloy for possible use in future bridge construction.

Description of specimen materials

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Specific analyses to be performed including requirements within standards

Accelerated corrosion test in salt water to simulate the effect of sea spray / deicing salt on bridge base metal A-1, A-2c, A-3

Date results required: 15 Nov 04
**Request for Sample Analysis**

<table>
<thead>
<tr>
<th>Name of Contact Person</th>
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The Bridge Analysis and Redevelopment Corporation (BARC) has contracted AMS Steel in the development of a corrosion resistant alloy for possible use in future bridge construction.

**Description of specimen materials**

3 inch X 1/2 inch sample of new proprietary alloy (attached)

**Specific analyses to be performed including requirements within standards**

Accelerated corrosion test in acid environment to simulate the effect of acid rain on bridge base metal A-1, A-2d, A-3

**Date results required:** 15 Nov 04
NOTE: Complete description of tests, procedure, and findings on attached sheet(s)
1. **Scope**
1.1. This method covers procedures for preparing and labeling solid uncoated metal specimens to be tested for corrosion.
1.2. This standard does not attempt to address safety concerns, if any, associated with its use.

2. **Referenced Documents**
2.2. Standard Procedures A-2a – A-2d: *Standard Method(s) for Testing Uncoated Metal Specimen(s) for Corrosion (in varying environments)*

3. **Materials**
- metal specimen
- latex gloves
- acetone
- grade 00 steel wool
- glass stir rod
- masking tape
- permanent marker
- 600 mL beaker
- paper towel
- electronic balance
- ruler

4. **Methods for Handling and Cleaning Specimens for Test**
4.1. Samples should be handled with latex gloves to eliminate the transfer of skin oils to the surface of the test sample.
4.2. Samples should be placed on a clean surface prior to mechanical cleaning. Ten strokes should be applied using grade 00 steel wool. Significant pressure should be used in one direction followed by wiping with a dry paper towel. Both sides of each specimen should be cleaned in the same manner.
4.3. A final chemical rinse with acetone should be performed, followed by wiping with a dry paper towel.

5. **Initial Measurement Required**
5.1. Specimens that are to be evaluated for corrosion damage require a measurement be made prior to corrosion testing. An initial mass of each specimen should be measured to greatest degree of precision readily available.
6. Methods for Labeling Specimens for Test
6.1. For laboratory corrosion tests that simulate exposure to a natural environment, a surface similar to the one that would exist in the actual use will yield the best results.
6.2. It is assumed in this standard procedure that specimens are metal strips measuring approximately 3 inches x 1/2 inch. If specimens of other forms are used, the method of labeling should be as close to that specified here as possible.
6.3. Each specimen should be marked with a unique designation. Teams of technicians should be assigned a designation for their team as follows. Each team uses the first 3 letters of the last name of one member as this unique designation. These three letters are then followed by A-2a, A-2b, A-2c, A-2d, or A-2e, depending on the accelerated environment in which to be tested.
6.4. In labeling a specimen, on one end of the specimen, a strip of masking tape should be affixed to both sides so that the tape covers 1 cm of the specimen. With a permanent fine point marker, the designation (from 6.3) should be written clearly on this tape twice, once on each side of the strip.

7. Method of Preparing Specimens for Testing
7.1. Label each specimen with a unique designation. (See section 6 above)
7.2. Specimens will be hung vertically in the test environment using this method. For each specimen, a 14 cm length of masking tape is first affixed to the sample covering one side of the label. It is then looped over a glass rod at the midpoint of the tape with the loose end terminating over the reverse side label.
7.3. The rod and specimen should then be set into the 600 mL beaker using the beaker’s pouring spout for stability. The specimen should be adjusted in height so that the specimen nearly touches the bottom of a 600 mL beaker. This is accomplished by spinning the rod. Additional masking tape is used to adjust for final height.
7.4. Relabel specimen on the supporting tape as established in section 6.
7.5. If more than one test specimen is suspended by this method in the same container, each specimen must be kept the same distance from other specimens and the container.
Standard Procedure A-2a:  
Standard Method for Testing Uncoated Metal Specimens for Corrosion  
In Distilled or Deionized Water

1. Scope:
1.1 This practice describes the apparatus and conditions required to conduct corrosion testing on metal surfaces using an accelerated immersion test in distilled or deionized water.  
1.2 This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents:
2.2 Standard Procedure A-1: Standard Method for Preparing, Cleaning, and Labeling Corrosion Uncoated Test Specimens

3. Materials
labeled metal specimen (as prepared in standard procedure A-1)  
apparatus (as described in step 4.)  
approximately 500 mL distilled or deionized water immersion solution  
permanent marker  
parafilm

4. Apparatus
4.1 The apparatus for this test consists of a glass or plastic container of sufficient volume and depth to allow a metal specimen test strip to be lowered vertically into it so that at least 90% of the strip can be submerged in the testing liquid placed in it. The specimen should be able to be covered by the testing liquid up to but not including the labeling/mounting tape. (see Standard Procedure A-1 section 6) Ideally a standard 600 mL laboratory beaker will be used, but any plain glass or plastic container is acceptable.  
4.2 If a single test requires the use of more than one container, all containers used for the test must be identical.  
4.3 Each container must be labeled with the same designation as the test specimens contained in it.  
4.4 Each test specimen is to be suspended in the test environment by a standard method. (See Standard procedure A-1, Section 7)  

5. Preparation of immersion solution
5.1 Tap water should be singly distilled or deionized in a standard chemistry water purification apparatus.
5.2. Well water should be doubly distilled or deionized in a standard chemistry water purification apparatus.
5.3. The prepared water must sit in an open container overnight to absorb ample atmospheric gases.
5.4. The prepared water must be at room temperature prior to using.

6. Conditions
6.1. Test environment solutions must be prepared in sufficient quantity to permit completion of all similar tests.
6.2. Suspended test strips may not touch the sides of the test container.
6.3. Suspended test strips may touch the bottom of the test container, but should remain perpendicular to the supporting rod.
6.4. Prior to pouring the immersion solution into the apparatus, the tester should note the height at which the tape label on the specimen begins.
6.5. This height should be marked on the outside of the apparatus in permanent marker.
6.6. The specimen should be removed from the apparatus.
6.7. The immersion solution should be added to the apparatus to a height no higher than that labeled in step 6.5 (typically 300-400 mL).
6.8. The specimen should be returned to the apparatus, ensuring the label is above the immersion solution.
6.9. Suspended test strips in the same container must be placed equally distant from each other.
6.10. All specimens in a single test must be stored under the same conditions for the duration of the test.
6.11. All environments once prepared should be sealed with parafilm.
6.12. Specimens should remain untouched during the testing.
6.13. When specimens are removed for testing, all specimens should be removed at the same time.

7. Evaluation of Corrosion
Standard Procedure A-2b:
Standard Method for Testing Metal Specimens for Corrosion In Heated Water

1. Scope:
1.1 This practice describes the apparatus and conditions required to conduct corrosion testing on metal surfaces using an accelerated immersion test in heated distilled or deionized water.
1.2 This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents:
2.2 Standard Procedure A-1: Standard Method for Preparing, Cleaning, and Labeling Corrosion Uncoated Test Specimens

3. Materials
labeled metal specimen (as prepared in standard procedure A-1)
apparatus (as described in step 4.)
approximately 500 mL distilled or deionized water immersion solution
fish tank heater or temperature controlled hot plate
partial immersion thermometer
parafilm

4. Apparatus
4.1 The apparatus for this text consists of a glass or plastic container of sufficient volume and depth to allow a metal specimen test strip to be lowered vertically into it so that at least 90% of the strip can be submerged in the testing liquid placed in it. The specimen should be able to be covered by the testing liquid up to but not including the labeling/mounting tape.(see Standard Procedure A-1 section 6) Ideally a standard 600 mL laboratory beaker will be used, but any plain glass or plastic container is acceptable.
4.2 If a single test requires the use of more than one container, all containers used for the test must be identical.
4.3 Each container must be labeled with the same designation as the test specimens contained in it.
4.4 Each test specimen is to be suspended in the test environment by a standard method. (See Standard procedure A-1, Section 7)

5. Preparation of immersion solution
5.1 Tap water should be singly distilled or deionized in a standard chemistry
water purification apparatus.
5.2. Well water should be doubly distilled or deionized in a standard chemistry water purification apparatus.
5.3. The prepared water must sit in an open container overnight to absorb ample atmospheric gases.
5.4. The solution should be prepared in the testing apparatus container.
5.5. The solution should be subjected to heat either by placing on a hot plate, or by dropping in a fish tank heater.
5.6. The thermometer choice is left to the discretion of the tester, but must be of partial immersion type.
5.7. The thermometer must be mounted so as not to contact any heating surface.
5.8. The prepared water must be at a constant temperature of 30°C (+/- 5°C) prior to using.

6. Conditions
6.1. Test environment solutions must be prepared in sufficient quantity to permit completion of all similar tests.
6.2. Suspended test strips may not touch the sides of the test container.
6.3. Suspended test strips may touch the bottom of the test container, but should remain perpendicular to the supporting rod.
6.4. Prior to pouring the immersion solution into the apparatus, the tester should note the height at which the tape label on the specimen begins.
6.5. This height should be marked on the outside of the apparatus in permanent marker.
6.6. The specimen should be removed from the apparatus.
6.7. The immersion solution should be added to the apparatus to a height no higher than that labeled in step 6.5 (typically 300-400 mL).
6.8. The specimen should be returned to the apparatus, ensuring the label is above the immersion solution.
6.9. Suspended test strips in the same container must be placed equally distant from each other.
6.10. All specimens in a single test must be stored under the same conditions for the duration of the test.
6.11. All environments once prepared should be sealed with parafilm.
6.12. Specimens should remain untouched during the testing.
6.13. When specimens are removed for testing, all specimens should be removed at the same time.

7. Evaluation of Corrosion
Standard Procedure A-2c:
Standard Method for Testing Uncoated Metal Specimens for Corrosion
In a 3% Saline Environment

1. Scope:
1.1 This practice describes the apparatus and conditions required to conduct
corrosion testing on metal surfaces using an accelerated immersion test in 3%
salt water. This simulates the effect of a saline environment due to road salt or
sea spray.
1.2 This standard does not attempt to address safety concerns, if any, associated
with its use.

2. Referenced Documents:
Apparatus
2.2 Standard Procedure A-1: Standard Method for Preparing, Cleaning, and Labeling
Corrosion Uncoated Test Specimens

3. Materials
labeled metal specimen (as prepared in standard procedure A-1)
apparatus (as described in step 4.)
electronic balance
500 mL volumetric flask, graduated cylinder, graduated beaker or graduated
Erlenmeyer flask
approximately 500 mL distilled or deionized water in
15 g sodium chloride (NaCl)
stir rod
permanent marker
parafilm

4. Apparatus
4.1. The apparatus for this text consists of a glass or plastic container of sufficient
volume and depth to allow a metal specimen test strip to be lowered vertically
into it so that at least 90% of the strip can be submerged in the testing liquid
placed in it. The specimen should be able to be covered by the testing liquid up
to but not including the labeling/mounting tape.(see Standard Procedure A-1
section 6) Ideally a standard 600 mL laboratory beaker will be used, but any plain
glass or plastic container is acceptable.
4.2. If a single test requires the use of more than one container, all containers used for
the test must be identical.
4.3. Each container must be labeled with the same designation as the test specimens
contained in it.
4.4. Each test specimen is to be suspended in the test environment by a standard
method. (See Standard procedure A-1, Section 7)
4.5. For pre-test preparation of specimens, see Standard Procedure A-1: Standard
Method for Preparing, Cleaning, and Labeling Uncoated Corrosion Specimens.
5. Preparation of immersion solution
5.1. Tap water should be singly distilled or deionized in a standard chemistry water purification apparatus.
5.2. Well water should be doubly distilled or deionized in a standard chemistry water purification apparatus.
5.3. The purified water must sit in an open container overnight to absorb ample atmospheric gases.
5.4. Dissolve 15 g of sodium chloride (NaCl) in enough water to make 500 mL of solution.
5.5. The prepared salt water must be at room temperature prior to using.

6. Conditions
6.1. Test environment solutions must be prepared in sufficient quantity to permit completion of all similar tests.
6.2. Suspended test strips may not touch the sides of the test container.
6.3. Suspended test strips may touch the bottom of the test container, but should remain perpendicular to the supporting rod.
6.4. Prior to pouring the immersion solution into the apparatus, the tester should note the height at which the tape label on the specimen begins.
6.5. This height should be marked on the outside of the apparatus in permanent marker.
6.6. The specimen should be removed from the apparatus.
6.7. The immersion solution should be added to the apparatus to a height no higher than that labeled in step 6.5 (typically 300-400 mL).
6.8. The specimen should be returned to the apparatus, ensuring the label is above the immersion solution.
6.9. Suspended test strips in the same container must be placed equally distant from each other.
6.10. All specimens in a single test must be stored under the same conditions for the duration of the test.
6.11. All environments once prepared should be sealed with parafilm.
6.12. Specimens should remain untouched during the testing.
6.13. When specimens are removed for testing, all specimens should be removed at the same time.

7. Evaluation of Corrosion
Standard Procedure A-2d:
Standard Method for Testing Uncoated Metal Specimens for Corrosion
In an Acidic Environment

1. Scope:
1.1 This practice describes the apparatus and conditions required to conduct corrosion testing on metal surfaces using an accelerated immersion test in a 5% (v/v) acetic acid (household vinegar) solution.
1.2 This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents:
2.2 Standard Procedure A-1: Standard Method for Preparing, Cleaning, and Labeling Corrosion Uncoated Test Specimens

3. Materials
labeled metal specimen (as prepared in standard procedure A-1)
apparatus (as described in step 4.)
approximately 500 mL household vinegar or a 5% (v/v) acetic acid (CH3COOH)solution
permanent marker
parafilm

4. Apparatus
4.1. The apparatus for this text consists of a glass or plastic container of sufficient volume and depth to allow a metal specimen test strip to be lowered vertically into it so that at least 90% of the strip can be submerged in the testing liquid placed in it. The specimen should be able to be covered by the testing liquid up to but not including the labeling/mounting tape.(see Standard Procedure A-1 section 6) Ideally a standard 600 mL laboratory beaker will be used, but any plain glass or plastic container is acceptable.
4.2. If a single test requires the use of more than one container, all containers used for the test must be identical.
4.3. Each container must be labeled with the same designation as the test specimens contained in it.
4.4. Each test specimen is to be suspended in the test environment by a standard method. (See Standard procedure A-1, Section 7)

5. Preparation of immersion solution
5.1. White household vinegar is the recommended solution.
6. Conditions
6.1. Test environment solutions must be prepared in sufficient quantity to permit completion of all similar tests.
6.2. Suspended test strips may not touch the sides of the test container.
6.3. Suspended test strips may touch the bottom of the test container, but should remain perpendicular to the supporting rod.
6.4. Prior to pouring the immersion solution into the apparatus, the tester should note the height at which the tape label on the specimen begins.
6.5. This height should be marked on the outside of the apparatus in permanent marker.
6.6. The specimen should be removed from the apparatus.
6.7. The immersion solution should be added to the apparatus to a height no higher than that labeled in step 6.5 (typically 300-400 mL).
6.8. The specimen should be returned to the apparatus, ensuring the label is above the immersion solution.
6.9. Suspended test strips in the same container must be placed equally distant from each other.
6.10. All specimens in a single test must be stored under the same conditions for the duration of the test.
6.11. All environments once prepared should be sealed with parafilm.
6.12. Specimens should remain untouched during the testing.
6.13. When specimens are removed for testing, all specimens should be removed at the same time.

7. Evaluation of Corrosion
Standard Procedure A-3:  
Standard Method for Evaluating the Corrosion of Uncoated Specimens

1. Scope:
1.1. This practice describes two separate methods for evaluating corrosion damage on uncoated metal specimens: the Mass Loss Method, and the Area Coverage Method.
1.2. This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents:
2.3 Standard Procedure A-1: Standard Method for Preparing, Cleaning, and Labeling Uncoated Corrosion Specimens
2.4 Standard Procedure A-2a – A-2d: Standard Method(s) for Testing Uncoated Metal Specimen(s) for Corrosion (in varying environments)

3. Materials
- electronic balance
- steel wool
- spatula with flat end or flat head screwdriver
- evaluation grid (attached)
- 600 mL beaker

4. Significance and Use
4.1. This procedure is utilized to evaluate the effect of corrosion on an uncoated metal, such as metal subjected to Standard Procedure A-2a – A-2d: Standard Method(s) for Testing Uncoated Metal Specimen(s) for Corrosion (in varying environments) or other accelerated tests
4.2. This procedure can also be used to evaluate the effect of corrosion on uncoated metal in an environmental (non accelerated) test.
4.3. Testers will be required to decide which of the methods of evaluation to employ in their testing.
4.4. The method of evaluation by area coverage requires the testers to judge the portion of the grid square affected by corrosion (see section 6)

5. Initial Measurement Required
5.1. Specimens that are to be evaluated for corrosion damage utilizing the Mass Loss Method (section 7), require a measurement be made prior to corrosion testing. An initial mass of each specimen should be measured to the greatest degree of precision readily available. (minimum \( \pm 0.01 \) g)
6. Drying
6.1. Upon the passage of the required time as specified by the client, the samples should be removed from their environment.
6.2. The parafilm should be removed as gently as possible
6.3. The glass rod, tape and specimen should be lifted in a gentle manner, from the testing environment, and transferred to an empty reaction vessel of the same size.
6.4. The system should remain in ambient conditions for 24 hours.
6.5. Upon completion of drying, the tape, tape label, and glass rod should be removed.

7. Mass Loss Method
7.1. This method can be used under most circumstances, regardless of the extent of corrosion.
7.2. If the Area Coverage Method is to be used in addition to the mass loss method, the Area Coverage Method must be performed first.
7.3. Method of Cleaning – In order to determine corrosion damage using the mass loss method, the corrosion products must be removed from the specimen. This should be done in two steps. In the first step, the bulk of the corrosion products should be removed by abrading the surface gently with the flat edge of a laboratory spatula, flat head screwdriver or other similar device. Care must be taken not to remove any of the base metal. In the second step, the surface of the specimen should be abraded with steel wool until the surface of the specimen is returned to its original appearance or as near to that condition as possible.
7.4. The specimen should then be weighed using the same balance as 5.1 and the mass loss calculated and recorded. The mass loss should be expressed as a %.
7.5. The specimen should be relabeled as in Standard Procedure A-1: *Standard Method for Preparing, Cleaning, and Labeling Uncoated Corrosion Specimens*

8. Area Coverage Method
8.1. This method should be used when the extent of corrosion is limited in area.
8.2. Remove the specimen from the drying environment. Place the specimen in a horizontal position on a stable surface. Hold the transparent plastic grid (see section 9) against the specimen. Record the total number of boxes constituting the full specimen surface area. Include the area under the tape label. Next record the corroded surfaces. If a square is completely covered with corroded surface, this is counted as 1 box. If a square is three quarter covered, this is counted as 3/4 box. If a square is half covered this is counted as 1/2 box. If a square is one quarter covered this is counted as 1/4 box. Repeat the process for the opposite side of the specimen. Determine the total surface area containing signs of corrosion. Express these results as a percentage of the total specimen surface area.
8.3. The specimen should be relabeled as in Standard Procedure A-1: *Standard Method for Preparing, Cleaning, and Labeling Uncoated Corrosion Specimens*
9. Evaluation Grid
9.1. Grid is attached.
9.2. Grid should be duplicated in a 1:1 ratio and transferred to a transparent overhead acetate.
The Problem
The Bridge Analysis and Redevelopment Corporation (BARC) is in the process of preparing bids for local, state, and federal highway departments in order to secure future painting contracts. They want to include in their bid data about types of coatings that inhibits corrosion in steel structures like bridges. BARC was very impressed with the quality of the previous work ElectroChem Inc. performed and has once again contracted Electrochem (the company you work for). Your project is to gain familiarity with the appropriate standard lab procedures. These procedures have been modified from the American Society for Testing and Materials (ASTM). Independent laboratories from around the country adhere to these standard techniques in order to obtain reproducibility of their results. Such standards have been established for measuring the effectiveness of surface coatings.

Your Task
Your team will receive a Request for Sample Analysis form from the Bridge Analysis and Redevelopment Corporation (BARC). BARC is interested in the effectiveness of various coatings to prevent the corrosion of steel. You will test in an accelerated mode which mimic methods used by testing labs to obtain results in one week that are equivalent to results from substantially larger time tests. The standard procedures you will follow have been adapted from ASTM methods for measuring the corrosion of coated iron metal. Your team will investigate the ability of two different coatings to protect the base metal from corrosion.

You will be assessed on your ability to implement these standard procedures, and provide a written and oral report on your findings. This report needs to contain sufficient detail to substantiate BARC’s bid for government work.

The possible coatings are:

• Pam (or other oil spray)
• hair spray
• WD-40
• melted candle wax
• dry candle wax
Materials

Per class

• steel samples
• deionized or distilled water
• digital camera / computer/ projector for oral presentations (optional)
• hairdryer (optional)

Per group

Dependent upon the assigned standard procedures, see standards for details

Safety and Disposal

As instructed by your teacher, follow appropriate safety procedures, including the use of personal protective equipment such as goggles and an apron. No special disposal procedures are required.

Procedure

1. Your instructor will give your team a “Request for Sample Analysis” document which specifies which coating you will test. Familiarize yourself with the standard procedures for the preparation (B-1), coating (B-2a – B2d), testing (B-3), and evaluation of test specimens (B-4). Note that you are responsible only for test the coating you have been assigned.

2. Write a lab analysis work order. This is the document ElectroChem Labs generates for each experiment the company performs.


4. Coat your sample referring to the appropriate version of Standard Procedure B-2: Standard Method(s) for Application of (various) non-metallic Coatings to Uncoated Specimen(s) Subjected to Corrosion Environments.

5. Test your sample by referring to Standard Procedure B-3: Test Method for Coated Specimens subjected to Corrosive Environments.

6. After a period of time, (as specified on your Request for Sample Analysis) evaluate the corrosion on your metal strips. Refer to Standard Procedure B-4: Evaluation of
Coated Specimens Subjected to Corrosive Environments.

7. At the end of your team’s work you will prepare both written and oral reports on your findings.

Discussion and Conclusions

Each testing team should prepare a report that includes the following:

Request for Sample Analysis (10pts)

Lab Analysis Work Order (10pts)

Goals of your experiment - This includes a statement of the testing objective and the type of environments tested. (5pts)

Background chemistry - Display evidence of your knowledge about the chemistry of corrosion. (10 pts)

Experiment design - Include the source of the procedures and a brief synopsis of what was done. Diagrams are appropriate. (10 pts)

Experimental results – This includes appropriate organization of your data presenting in more than one format, one of which must be graphical (10 pts)

Error analysis – Discuss what did go wrong, what could have gone wrong, and how this would affect your data. (10 pts)

Conclusions – A summary of the teams finding. (5 pts)

Future Experimentation - Describe in detail a follow up experiment that your team could conduct based on your results. (5 pts)

Team Analysis – Describe how well your team functioned. (5 pts)

Assessment

Your grade on this activity includes:

• the written report on your findings (as above) (80 points)

• your presentation to the class (50 points)

• adherence to standard procedures (as below) (maximum 40 points)
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<th>Procedure</th>
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<td>B - 1</td>
<td>Cleaning specimens</td>
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<td>B - 1</td>
<td>Preparing specimens</td>
<td>2 = Average</td>
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<td>B - 2</td>
<td>Coating Application</td>
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<tr>
<td>B - 3</td>
<td>Scribe Mark</td>
<td>4 = Excellent</td>
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<td>B - 4</td>
<td>Scribe Creepage</td>
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*10 minute oral report of your findings presented to the class. This should include the indication of corrosion by both the scribe creepage and area coverage methods. Digital photos may be used to describe observations. (30 pts)*
Instructor Notes

Coatings

This activity is a continuation of the scenario from Working as a Technician for ElectroChem Inc. Students continue to work as chemical technicians. Working in teams, students examine various coatings all within the framework of utilizing more complex industrial standards. The students investigate the effectiveness of various non-metallic coatings in preventing corrosion over one week and subsequently provide both a written and oral report detailing their findings. In the real world, the coatings would be paints and other substances designed to create a barrier between the metal and corrosive factors. Testing the effectiveness of paints within the time frame of a typical chemistry course would be prohibitive. Therefore, this activity utilizes coatings that are less effective at preventing corrosion but are much more familiar and available to students. Additionally, they are tested in an accelerated environment. As the instructor, your role will be that of lab manager at ElectroChem. You assign different coatings to be tested using a standard “Request for Sample Analysis” form, monitor team progress throughout the students’ research, and assess the teams’ reports.

Time Required

Setup: 30 minutes
Standard Procedure B-1: 30 minutes
Standard Procedure B-2a—B-2d: 15 minutes ( + drying time if necessary)
Test Method B-3: 30 minutes
Reaction: 3-4 days or longer
Evaluation B-4 30 minutes
Cleanup: 30 minutes

Key Science Concepts

Chemistry

- measurement
- combination reactions
- solution concentration (m/v %)
- electrolytes
- reaction rate
- redox reactions
- electrochemistry
Mathematics
- mass/volume percentage
- mean

Engineering and Technology
- American Society for the Testing of Materials (ASTM) standards
- Metal Coatings

Prior Concepts and Skills Needed
Students should have conducted the activity entitled, “Working as a Lab Tech”. This activity builds on the skills learned within that activity. Once the learning curve has been mastered, this second reiteration should flow much more smoothly, take less time, and be very receptive to students. In terms of content, students should have an understanding of the corrosion process, how an ionic or acidic environment promotes electron transfer in such a reaction.

Materials

For Getting Ready
- overhead transparency

For the Procedure
Per class
- adequate steel strips 3-inch x 1/2-inch or other samples of cold rolled (uncoated) steel.
  Flinn Scientific sells 6-inch x 1/2-inch strips that can easily be cut in half. Nails will also work, but they cannot be galvanized and will cause difficulty with the area coverage method of evaluation.
- distilled or deionized water.
  Deionized water will work better because the dissolved oxygen required for rusting has not been removed (as in boiling). Distilled water that has sat in an open environment or has been vortexed will provide the requisite oxygen.
- digital camera, computer, and/or projector
- glass stir rods, wooden dowel rods or even pencils will also work
- hairdryer
  If decreased drying time is desired, a hairdryer is recommended.
• Parafilm or Saran Wrap
The Parafilm must be of the large roll type. The standard 4-inch roll will not cover the 600 mL beakers sufficiently.

• steel wool, grade 00

• evaluation grids (found at the end of Standard Procedure B-4 for the Area Coverage Method)

• sodium chloride (NaCl)
Reagent grade is preferred although not necessary. Household table salt contains an anticaking agent which clouds the water upon dissolving. Upon using the specified 12g of household table salt, the concentration will be slightly below that of 3%, but this is not critical.

• 3% (m/v) salt solution if you prefer not to have your students make it themselves. Each group will use approximately 450 mL.

Additional materials are needed and are group dependent. See standard procedures B-1, B-2a, B-2b, B-2c, B-2d, B-3, B-4 for details

Safety and Disposal
It is your responsibility to review appropriate safety procedures with your students, including the use of personal protective equipment. No special disposal procedures are required.

Getting Ready
Divide the class into teams. Have the students name their teams as an important means of identifying samples later on.

For students to evaluate their corrosion using the Area Coverage Method (standard procedure B-4), make transparent plastic grids. A master is provided at the end of Standard Procedure B-4. Copy the master onto a clear transparency, cut and distribute the grids to the teams.

Fill out the “Request for Sample Analysis” documents (or use the ones provided) and have them ready at the beginning of the lab. You may want to affix the metal sample directly to the “Request for Sample Analysis” document.
Procedure Notes

It is crucial that prior to beginning the lab procedure, the students have established a name for their team, abbreviated with letters, so samples can be easily identified by code. Likewise it is advised that the instructor has filled out the “Request for Sample Analysis” documents ready to distribute upon beginning lab.

The students are given an overview of their procedure in the student handout. They must also consult the four “standard procedures” in which to formulate their plan of action. The instructor should play a supportive role, but interpretations of the standard procedures should be made by the student team without guidance and support.

Students will prepare their samples, coat their samples, test their coated samples in an accelerated environment, and after a specified time (approximately one week) evaluate their samples. Students should make observations of their samples daily within the week period of time. It does not matter whether the scribe creepage method or the area coverage method is done first. If you run the experiment for a week, a timetable would be:

Week Prior – give assignments, all teams review their tasks and production time table

Monday – prepare, coat, and test samples (~40 min)

Tuesday– observations only (~5 min)

Wednesday– observations only (~5 min)

Thursday– observations and drying (~15 min)

Friday– evaluation of coating (~40 min)

Mon– students present their results to class

If more than one week can be allowed for the corrosion to occur, the data would be more definitive.

Assessment

Students are assessed on their final report, their adherence to standard procedures, and their oral presentation. These are detailed in the student handout. It is important for the instructor to record student work during the activity and evaluate their product later at a specified time. In this manner, a student cannot claim, “But our samples were not ready yet.” Additionally, you may want to assess students on their lab technique throughout this activity.
Guidelines for grading adherence to standard procedures:

Unacceptable - team shows no evidence of awareness of procedure in their work

Adequate – team shows evidence that they are aware of the standard procedure and have made a minimal attempt to adhere to it.

Average – team demonstrates in much, but not all of their work an awareness of and adherence to the procedure

Good – team demonstrates in most observed situations adherence to most but not all of the procedure

Excellent – team demonstrates in all observed situations strict adherence to the procedure

Explanation

Throughout this activity, the students experience an industrial laboratory environment. They begin to see some of the variables involved in protecting the corrosion of iron. Ease of application, surface adhesion, cost, and effectiveness all play into the rating of a coating.

All of the materials used as coatings in this activity were chosen because of their high failure rate. If the students were to investigate commercial coatings such as paints, they would not provide the adequate data within a reasonable period of time. (In other words, commercial paints are quite effective in minimizing corrosion).

According to Dr. Sheldon Dean, of Dean Corrosion Technology, the somewhat tedious ASTM standard procedures are central to the nature of corrosion testing. Even so, corrosion is a somewhat random event and it is extremely difficult to reproduce experimental data.

Addressing the National Science Education Standards

• 177 With background knowledge of corrosion on hand, students connect the design of the experiment to the variable given.
• 178 When setting up their corrosion environment, students conduct an investigation. Students then present their results using cohesive arguments.
• 179, 186 Mathematics are utilized in the area coverage method of corrosion and scribe creepage determination. Additionally, graphical representation of results are conveyed.
• 182, 264 Scientific arguments are communicated and defended in their reports.
• 187 Logic and evidence are considered in drafting the final report.
• 188 In communication within and between groups of students, students report clearly their scientific discoveries.
• 199 The ubiquity of corrosion is explored.
• 202 Students gain an appreciation for the varying rates of corrosion utilizing different coatings.
• 260 The final report requires students to design future experimentation.
• 263 Students evaluate their experiments to determine if they meet the designated standard procedures.
• 284 Corrosion is a basic process that affects humans.
• 289 Corrosion is a slow and progressive process that negatively affects society.
• 290 Corrosion is a natural process that requires humans to assess its potential danger and risk.
• 293 Although iron is subject to corrosion, suitable non-corroding building materials are slow to market due to economic considerations.
• 299 Through a students use of standard procedures, they gain an understanding of the empirical standards typified in science.
• 300 In the final written report, explanations must meet scientific criteria.

**Addressing the Voluntary Industry Standards**

L1.03.02 Students take on the role of process technicians.

L1.04.01 Students work as members of a larger team.

L1.04.08 Students observe the details and trends of an object corroding throughout the duration of the experiment.

L2.04.02 Students demonstrate the appropriate use of eye protection.

L2.04.03 Students maintain a clean and safe workplace.

L2.05.01 Students handle common lab equipment.

L3.01.08 Students display their knowledge of atoms combining to form molecules in the formation of iron(III) oxide (Fe₂O₃).

L3.01.10 Students write and balance the equations applicable to corrosion.

L3.02.03 Students write the name and formula for the common inorganic substance ferric oxide (iron(III) oxide).

L3.03.08 Students are expected to properly clean their testing apparatus upon completion of the activity.

L4.01.01 Both the scribe creepage and the area coverage method display the importance of measurement in chemistry.
The standard procedures in this activity are based on ASTM standards.

Students carry out standard stepwise procedures in this activity.

Data is presented graphically in the final report.

A final report is required which includes principles of the method, conclusions, next steps, and follow up.

Chemical reactivity is described using oxidation-reduction reactions.

Familiarity is gained with the sections of published standard methods of chemical analysis.

Familiarity is gained with associations that provide standard methods of chemical analysis.

Solution concentration is expressed as % (m/v).

Rates of chemical reactions are explored.

Teamwork is explored in this activity.

Each student group has both a variable condition and a control experiment.

Corrosion progress is recorded throughout the duration of this experiment.

Data is expressed graphically.

A written report documents results of the experiment.

Students prepare and present an oral report.

**Addressing the SCANS Skills**

**Using Workkeys Targets**

**Applied Mathematics**

Level 3- Students perform a translation from a verbal setup to a math equation (area coverage method and scribe creepage).

Level 4- Students perform a ratio calculation (area coverage method). Students perform a mean calculation (scribe creepage)

**Applied Technology**

Level 3- Students understand the function of simple coatings such as paints.

**Locating Information**
Level 6- When using standard procedure documents, students apply information to only their described experiment.

Reading for Information

Level 5- Students will recognize the application of technical terms to stated situations. Additionally students will recognize the application of instructions from the document to new situations that are similar. Standard procedures with multiple steps must be followed.

Teamwork

Level 3- Students recognize the need for trust and dependability in their team environment.

Level 5- Students determine the best use of their team’s talents to accomplish goals.

References


ASTM Standard D1014-95 Standard Practice for Conducting Exterior Exposure Test of Paints on Steel


“What's up with corrosion” activity

Sheldon Dean, Dean Corrosion Technologies, Philadelphia PA. Sheldondean@deancorrtech.com
The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate oil cooking spray as a metallic coating. B-1, B2a, B-3, B-4

Date results required: 15 Dec 04
The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate hair spray as a metallic coating.
B-1, B2a, B-3, B-4

Date results required: 15 Dec 04
The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate WD-40 spray as a metallic coating.  
B-1, B2a, B-3, B-4

Date results required: 15 Dec 04
The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate melted candle wax as a metallic coating. B-1, B2c, B-3, B-4

Date results required: 15 Dec 04
The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate dry candle wax as a metallic coating. B-1, B2d, B-3, B-4

Date results required: 15 Dec 04
The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate Elmer's glue as a metallic coating.

B-1, B2b, B-3, B-4

Date results required: 15 Dec 04
The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate rubber cement as a metallic coating. B-1, B2b, B-3, B-4

Date results required: 15 Dec 04
Request for Sample Analysis

D'Agostino, Mike  
Name of Contact Person

Request  
Sent to:

Electrochem Labs Inc  
Name of Laboratory doing Analysis

4717 Precipitation Drive  
St. Address

Labland, OH 26817  
City, State, Zip Code

1-800-355-5585  
Phone No.

1-800-355-1079  
Fax No.

408  
Request No.

20 Nov 2004  
Date of Request

The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate petroleum jelly as a metallic coating. B-1, B2b, B-3, B-4

Date results required: 15 Dec 04
The Bridge Analysis and Redevelopment Corporation (BARC) is a finalist for receiving the bridge painting contract for 45 bridges in a coastal county of Florida. Data is required to determine the most effective coating of AMS Steel's new alloy in Florida's salty air.

3 inch X 1/2 inch sample of new proprietary alloy (attached)

Investigate lip balm as a metallic coating. B-1, B2d, B-3, B-4

Date results required: 15 Dec 04
Request for Sample Analysis

Name of Contact Person

Name of Laboratory doing Analysis

St. Address

City, State, Zip Code

Phone No.  Fax No.

Request No.

Date of Request

Description of problem

Description of specimen materials

Specific analyses to be performed including requirements within standards

Date results required: ____________
1. Scope
1.1. This method covers procedures for preparing and labeling solid metal specimens to be subsequently coated and then tested for corrosion.
1.2. This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents:
2.2. Standard Procedures B-2a–B2d: *Standard Method(s) for Application of (various) Non-Metallic Coatings to Specimens Subjected to Corrosive Environments*
2.4. Standard Procedure B-4: *Evaluation of Coated Specimens Subjected to Corrosive Environments*

3. Materials
metal specimen
latex gloves
acetone
grade 00 steel wool
glass stir rod
masking tape
permanent marker
600 mL beaker
paper towel
electronic balance
ruler

4. Methods for Handling and Cleaning Specimens for Test
4.1. Samples should be handled with latex gloves to eliminate the transfer of skin oils to the surface of the test sample.
4.2. Samples should be placed on a clean surface prior to mechanical cleaning. Ten strokes should be applied using grade 00 steel wool. Significant pressure should be used in one direction followed by wiping with a dry paper towel. Both sides of each specimen should be cleaned in the same manner.
4.3. A final chemical rinse with acetone should be performed, followed by wiping with a dry paper towel.

5. Initial Measurements
5.1. Specimens that are to be coated and evaluated for corrosion damage do not require an initial mass measurement.
6. Methods for Labeling Specimens for Test

6.1. For laboratory corrosion tests that simulate exposure to a natural environment, a surface similar to the one that would exist in the actual use will yield the best results.

6.2. It is assumed in this standard procedure that specimens are metal strips measuring approximately 3 inches x 1/2 inch. If specimens of other forms are used, the method of labeling should be as close to that specified here as possible.

6.3. Each specimen should be marked with a unique designation. Teams of technicians should be assigned a designation for their team as follows. Each team uses the first 3 letters of the last name of one member as this unique designation. These three letters are then followed by B-2a, B-2b, B-2c, or B-2d, depending on the type of coating in which to be tested.

6.4. The identity of the specific coating is to be added to the label, as determined by the team of technicians.

6.5. In labeling a specimen, on one end of the specimen, a strip of masking tape should be affixed to both sides so that the tape covers 1 cm of the specimen. With a permanent fine point marker, the designation (from 6.3 and 6.4) should be written clearly on this tape twice, once on each side of the strip.

7. Method of Preparing Specimens for Testing

7.1. Label each specimen with a unique designation. (See section 6 above.)

7.2. Specimens will be hung vertically in the test environment using this method. For each specimen, a 14 cm length of masking tape is first affixed to the sample covering one side of the label. It is then looped over a glass rod at the midpoint of the tape with the loose end terminating over the reverse side label.

7.3. The rod and specimen should then be set into the 600 mL beaker using the beaker’s pouring spout for stability. The specimen should be adjusted in height so that the specimen nearly touches the bottom of a 600 mL beaker. This is accomplished by spinning the rod. Additional masking tape is used to adjust for final height.

7.4. Relabel specimen on the supporting tape as established in section 6.

7.5. If more than one test specimen is suspended by this method in the same container, each specimen must be kept the same distance from other specimens and the container.

7.6. Refer to Standard Procedure B-2a – B-2d: Standard Method(s) for Application of (various) Non-Metallic Coatings to Specimens Subjected to Corrosive Environments according to the work order assigned.

1. Scope
1.1. This method describes the methods of applying aerosol spray nonmetallic coatings to a metal surface prior to corrosion testing.
1.2. Such coatings include Pam or (other oil spray), hair spray, and WD-40.
1.3. This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents
2.1. ASTM Standard D1014 – 95 Standard Practice for Conducting Exterior Exposure Tests of Paints on Steel
2.2. Standard Procedure B-1: Standard Method for Preparing, Cleaning, and Labeling Coated Corrosion Specimens
2.3. Standard Procedure B-3: Test Method for Coated Specimens Subjected to Corrosive Environments
2.4. Standard Procedure B-4: Evaluation of Coated Specimens Subjected to Corrosive Environments

3. Significance and Use
3.1. The objective of this standard is to ensure that protective coatings applied to a metal specimen are applied as uniformly as possible.
3.2. If appropriate, coatings should be allowed to dry before proceeding to Standard Procedure B-3: Test Method for Evaluating Coated Specimens Subjected to Corrosive Environments

4. Materials
labeled metal specimen (as prepared in Standard Procedure B-1)
Pam or (other oil spray) or hair spray or WD-40
index card

5. Coating Test Specimens
5.1. Testers must practice uniform application techniques prior to attempting this procedure.
5.2. In the interest of overspray, testers must determine a suitable location for the application.
5.3. The two wide surfaces, and 3 narrow edges of the specimen are all to be coated.
5.4. When coatings are applied by spraying, the sprayer should be held at least 15 cm and no more than 20 cm from the surface to be coated. The specimen surface should be vertical, and the sprayer should be held parallel to the specimen surface. It is crucial that an index card of appropriate size is held or mounted to protect the attaching/labeling tape. If this tape is subjected to coating, there is great likelihood for failure of the mounting apparatus during the duration of the experiment. The sprayer should then be activated in a practice mode to familiarize the tester with spray velocity, size of spray etc. Prior to releasing the spray, the tester should begin to move the
sprayer up and down across the specimen surface to ensure uniformity of coating. Next, the tester should activate the spray and apply a thin coating without dripping, or overspraying onto the mounting material.

5.5. Repeat for the other surfaces.
5.6. If appropriate, the coating should be allowed to air dry.
5.7. A discussion of the ease or difficulty in the application should be made in the final report.

6. Testing Specimen
6.1. Refer to Standard Procedure B-3: *Test Method for Evaluating Coated Specimens Subjected to Corrosive Environments*
Standard Procedure B-2b: 
Standard Method for Application of a Liquid Non-Metallic Coating to Specimens Subjected to Corrosive Environments

1. Scope
1.1. This method describes the methods of applying liquid nonmetallic coatings to a metal surface prior to corrosion testing.
1.2. Such coatings include Elmer’s glue, rubber cement, and petroleum jelly
1.3. This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents
2.1. ASTM Standard D1014 – 95 Standard Practice for Conducting Exterior Exposure Tests of Paints on Steel
2.2. Standard Procedure B-1: Standard Method for Preparing, Cleaning, and Labeling Coated Corrosion Specimens
2.3. Standard Procedure B-3: Test Method for Coated Specimens Subjected to Corrosive Environments
2.4. Standard Procedure B-4: Evaluation of Coated Specimens Subjected to Corrosive Environments

3. Significance and Use
3.1. The objective of this standard is to ensure that protective coatings applied to a metal specimen are applied as uniformly as possible.
3.2. If appropriate, coatings should be allowed to dry before proceeding to Standard Procedure B-3: Test Method for Evaluating Coated Specimens Subjected to Corrosive Environments

4. Materials
labeled metal specimen (as prepared in Standard Procedure B-1)
Elmer’s glue or rubber cement or petroleum jelly
wood popsicle stick

5. Coating Test Specimens
5.1. The two wide surfaces, and 3 narrow edges of the specimen are all to be coated.
5.2. A standard application device shall be built. A wood splint of sufficient rigidity, should be chosen. It needs to be cut to have the application surface most closely resemble the specimen surface geometry. (If one is surface coating a conical surface, a conical surface should be cut out of the wood splint. If the tester is coating a flat surface a flat application surface should be cut out of the wood splint.) The objective is to coat the surface with a uniform coating. The sample should be held by the labeling portion (Standard Procedure B-1 Section 6), at a 45 degree angle with the opposite end resting against a solid surface. The application device should contain a similar quantity of coating for each application and the application device should be held at an appropriate angle to the specimen surface and drawn in a single direction across the surface. If a
second, third, fourth (or greater) pass is necessary, the tester should maintain the objective of a uniform coating on all specimens.

5.3. Repeat for the other surfaces.
5.4. If appropriate, the coating should be allowed to air dry.
5.5. A discussion of the ease or difficulty in the application should be made in the final report

6. Testing Specimen
6.1. Refer to Standard Procedure B-3: *Test Method for Evaluating Coated Specimens Subjected to Corrosive Environments*
Standard Procedure B-2c:  
Standard Method for Application of a Solidifying Liquid Coating to Specimens Subjected to Corrosive Environments

1. Scope
1.1. This method describes the methods of applying solidifying liquid nonmetallic coatings to a metal surface prior to corrosion testing.
1.2. Such coatings include melted candle wax.
1.3. This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents
2.1. ASTM Standard D1014 – 95 Standard Practice for Conducting Exterior Exposure Tests of Paints on Steel
2.2. Standard Procedure B-1: Standard Method for Preparing, Cleaning, and Labeling Coated Corrosion Specimens
2.3. Standard Procedure B-3: Test Method for Coated Specimens Subjected to Corrosive Environments
2.4. Standard Procedure B-4: Evaluation of Coated Specimens Subjected to Corrosive Environments

3. Significance and Use
3.1. The objective of this standard is to ensure that protective coatings applied to a metal specimen are applied as uniformly as possible.
3.2. If appropriate, coatings should be allowed to dry before proceeding to Standard Procedure B-3: Test Method for Evaluating Coated Specimens Subjected to Corrosive Environments

4. Materials
labeled metal specimen (as prepared in Standard Procedure B-1)
candle
match

5. Coating Test Specimens
5.1. The two wide surfaces, and 3 narrow edges of the specimen are all to be coated.
5.2. A standard application device shall be built. A wood splint of sufficient rigidity, should be chosen. It needs to be cut to have the application surface most closely resemble the specimen surface geometry. (If one is surface coating a conical surface, a conical surface should be cut out of the wood splint. If the tester is coating a flat surface a flat application surface should be cut out of the wood splint.) The objective is to coat the surface with a uniform coating. The sample should be held by the labeling portion (Standard Procedure B-1 Section 6), at a 45 degree angle with the opposite end resting against a solid surface.
5.3. The solid should be melted in an appropriate fashion. (In the case of the candle, it should be lit and tilted to allow wax to fall onto the base metal.) Working quickly, the
application device should be held at an appropriate angle to the specimen surface and
drawn in a single direction across the surface.
5.4. Repeat for the other surfaces.
5.5. The coating should be allowed to solidify
5.6. A discussion of the ease or difficulty in the application should be made in the final report

6. Testing Specimen
6.1. Refer to Standard Procedure B-3: Test Method for Evaluating Coated Specimens
    Subjected to Corrosive Environments
Standard Procedure B-2d:  
Standard Method for Friction Application of a Non-Metallic Coating to Specimens  
Subjected to Corrosive Environments

1. Scope
1.1. This method describes the methods of friction application of a nonmetallic coatings to a  
metal surface prior to corrosion testing.  
1.2. Such coatings include dry candle wax (paraffin) or lip balm.  
1.3. This standard does not attempt to address safety concerns, if any,  
associated with its use.

2. Referenced Documents
of Paints on Steel  
2.2. Standard Procedure B-1: Standard Method for Preparing, Cleaning, and Labeling  
Coated Corrosion Specimens  
Environments  
2.4. Standard Procedure B-4: Evaluation of Coated Specimens Subjected to Corrosive  
Environments

3. Significance and Use
3.1. The objective of this standard is to ensure that protective coatings applied to a metal  
specimen are applied as uniformly as possible.  
3.2. If appropriate, coatings should be allowed to dry before proceeding to Standard  
Procedure B-3: Test Method for Evaluating Coated Specimens Subjected to Corrosive  
Environments

4. Materials
labeled metal specimen (as prepared in Standard Procedure B-1)  
dry candle wax (paraffin) or lip balm

5. Coating Test Specimens
5.1. The two wide surfaces, and 3 narrow edges of the specimen are all to be coated.  
5.2. When coatings are applied in a friction application, the sample should be held by the  
labeling portion, at a 45 degree angle with the opposite surface resting against a solid  
surface. Ten rubs should be applied in a single direction with the underlying goal of  
uniform application of coating. Both sides of the specimen should be similarly coated.  
5.3. Repeat for the other surfaces.  
5.4. If appropriate, the coating should be allowed to air dry.  
5.5. A discussion of the ease or difficulty in the application should be made in the final report

6. Testing Specimen
6.1. Refer to Standard Procedure B-3: Test Method for Evaluating Coated Specimens  
Subjected to Corrosive Environments
Standard Procedure B-3: Test Method for Coated Specimens Subjected to Corrosive Environments

1. Scope:
1.1 This practice covers the testing of coated specimens under atmospheric and accelerated exposure tests
1.2 This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents:
2.2 Standard Procedure B-1: Standard Method for Preparing, Cleaning, and Labeling Coated Corrosion Specimens
2.3 Standard Procedure B-2a–B2d: Standard Method(s) for Application of (various) Non-Metallic Coatings to Specimens Subjected to Corrosive Environments
2.4 Standard Procedure B-4: Evaluation of Coated Specimens Subjected to Corrosive Environments

3. Materials
Labeled and coated metal specimen (as prepared in Standard Procedure B-1,B-2)
scribe nail
apparatus (as described in step 5)
approximately 500 mL of 3% (m/v) Sodium Chloride (NaCl) immersion solution. (as prepared in step 8)
permanent marker
parafilm
distilled water
500 mL volumetric flask, graduated cylinder, graduated beaker or graduated Erlenmeyer flask
15 g sodium chloride (NaCl)
stir rod

4. Application of Coatings:
See Standard Procedure B-2a–B2d: Standard Method(s) for Application of (various) Non-Metallic Coatings to Specimens Subjected to Corrosive Environment

5. Apparatus
5.1 The apparatus for this text consists of a glass or plastic container of sufficient volume and depth to allow a metal specimen test strip to be lowered vertically into it so that at least 90% of the strip can be submerged in the testing liquid placed in it. The specimen should be able to be covered by the testing liquid up to but not including the labeling/mounting tape.(see Standard Procedure B-1 section 6) Ideally a standard 600 mL laboratory beaker will be used, but any plain glass or plastic container is acceptable.
5.2 If a single test requires the use of more than one container, all containers used for the
test must be identical.
5.3 Each container must be labeled with the same designation as the test specimens
contained in it.
5.4 Each test specimen is to be suspended in the test environment by a standard method.
(See Standard procedure B-1, Section 7)
5.5 For pre-test preparation of specimens, see Standard Procedure B-1: Standard Method
for Preparing, Cleaning, and Labeling Coated Corrosion Specimens

6. Scribed Specimens
6.1. A standard finishing nail (the scribing tool) of agreed upon size will be used to scribe a
uniform mark in the coatings
6.2. The test specimen should be held by the labeling portion (as specified in Standard
Procedure B-1 section 6 ) with the opposite end resting firmly on a solid surface. It
should be held at an appropriate angle to prevent the coating from contacting the solid
surface. Only the masking tape used for labeling) should be in contact with the tester’s
hand.
6.3. The scribing tool should be held relative to the specimen surface and brought into
contact with the surface just below the masking tape label. With a smooth continuous
motion and significant pressure, a single scribe mark should be made down the center
of the specimen along its length. Both sides should be scribed.
6.4. In making the scribe mark the pressure exerted should be sufficient to make a mark on
the substrate metal surface under the coating.
6.5. If the coating adhesion fails catastrophically due to this scribe procedure, testing should
continue, but this failure type noted in the final report.

7. Preparation of immersion solution
7.1. Tap water should be singly distilled or deionized in a standard chemistry water
purification apparatus.
7.2. Well water should be doubly distilled or deionized in a standard chemistry water
purification apparatus.
7.3. The purified water must sit in an open container overnight to absorb ample atmospheric
gases.
7.4. Dissolve 15 g of sodium chloride (NaCl) in enough water to make 500 mL of solution
7.5. The prepared salt water must be at room temperature prior to using.

8. Conditions
8.1. Test environment solutions must be prepared in sufficient quantity to permit completion
of all similar tests..
8.2. Suspended test strips may not touch the sides of the test container.
8.3. Suspended test strips may touch the bottom of the test container, but should be remain
perpendicular to the supporting rod.
8.4. Prior to pouring the immersion solution into the apparatus, the tester should note the
height at which the tape label on the specimen begins.
8.5. This height should be marked on the outside of the apparatus in permanent marker.
8.6. The specimen should be removed from the apparatus.
8.7. The immersion solution should be added to the apparatus to a height no higher than that labeled in step 9.5 (typically 300-400 mL).
8.8. The specimen should be returned to the apparatus, ensuring the label is above the immersion solution.
8.9. Suspended test strips in the same container must be placed equally distant from each other.
8.10. All specimens in a single test must be stored under the same conditions for the duration of the test.
8.11. All environments once prepared should be sealed with parafilm.
8.12. Specimens should remain untouched during the testing.
8.13. When specimens are removed for testing, all specimens should be removed at the same time.

9. Evaluation of Corrosion
See Standard Procedure B-4: Evaluation of Coated Specimens Subjected to Corrosive Environments
Standard Procedure B-4: Evaluation of Coated Specimens
Subjected to Corrosive Environments

1. Scope:
1.1 This practice covers the evaluation of a corrosion failure along a scribe mark. Two methods are included here: area coverage and scribe creepage.
1.2 This standard does not attempt to address safety concerns, if any, associated with its use.

2. Referenced Documents:
2.2. Standard Procedure B-1: Standard Method for Preparing, Cleaning, and Labeling Coated Corrosion Specimens
2.3. Standard Procedures B-2a–B2d: Standard Method(s) for Application of (various) Non-Metallic Coatings to Specimens Subjected to Corrosive Environments
2.4. Standard Procedure B-3: Test Method for Coated Specimens Subjected to Corrosive Environments

3. Materials
evaluation grid (attached)
ruler
600 mL beaker

4. Significance and Use
4.1. Testers will be required to decide which of the methods of evaluation to employ in their testing.
4.2. The method of evaluation by area coverage requires the testers to judge the portion of the grid square affected by corrosion.
4.3. The method of evaluation by creepage due to surface failure requires the testers to measure the distance corrosion appears from the scribe mark, and correlate a rating code (see section 9).
4.4. The application of commonly available nonmetallic coatings may not provide adequate surface adhesion between coating and substrate metal which may result in corrosion beyond the normal limits along a scribe mark.

5. Application of Coating
See Standard Procedures B-2a–B2d: Standard Method(s) for Application of (various) Non-Metallic Coatings to Specimens Subjected to Corrosive Environments

6. Testing of Coating
See Standard Procedure B-3: Test Method for Coated Specimens Subjected to Corrosive Environments

7. Drying
7.1. Upon the passage of the required time as specified by the client, the samples should be removed from their environment.
7.2. The parafilm should be removed as gently as possible.
7.3. The glass rod, tape, and specimen should be lifted in a gentle manner, from the testing environment, and transferred to an empty reaction vessel of the same size.
7.4. The system should remain in ambient conditions for 24 hours.
7.5. Upon completion of drying, tape, tape label, and glass rod should be removed.

8. Area Coverage Method
8.1. This method should be used when the extent of corrosion is limited in area.
8.2. Remove the specimen from the drying environment. Place the specimen in a horizontal position on a stable surface. Hold the transparent plastic grid (see section 10) against the specimen. Record the total number of boxes constituting the full specimen surface area. Include the area under the tape label. Next record the corroded surfaces. If a square is completely covered with corroded surface, this is counted as 1 box. If a square is three quarter covered, this is counted as 3/4 box. If a square is half covered this is counted as 1/2 box. If a square is one quarter covered this is counted as 1/4 box. Repeat the process for the opposite side of the specimen. Determine the total surface area containing signs of corrosion. Express these results as a percentage of the total specimen surface area.
8.3. The specimen should be relabeled as in Standard Procedure A-1: Standard Method for Preparing, Cleaning, and Labeling Uncoated Corrosion Specimens

9. Scribe Creepage
9.1. Remove the specimen from the drying environment.
9.2. Visually inspect the specimen along the scribe line for signs of corrosion or loss of coating on the specimen due to corrosion.
9.3. In at least five locations equidistant along the scribe line, measure the maximum distance between the scribe line and signs of corrosion or coating loss (called the "creepage") and the minimum distance in millimeters.
9.4. Creepage at each location should be noted in both directions from the scribe line, but at any location along the scribe line only one maximum creepage and one minimum creepage should be recorded in millimeters.
9.5. From the data in 9.4, mean creepages should be calculated for each location.
9.6. From the data in 9.5 a single mean should be calculated.
9.7. Coating failure may be reported either as mean creepage or by rating index from the table below.
9.8. Repeat for other side.

Rating of Failure at Scribe Mark
Representative Mean Creepage from scribe
   Based on 7 mm sample width, scribe line down center

<table>
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<tr>
<th>Millimeters</th>
<th>Rating Number</th>
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<tr>
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</tr>
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**10. Evaluation Grid**

10.1. Grid is attached.

10.2. Grid should be duplicated in a 1:1 ratio and transferred to a transparent overhead acetate.
Should the Bridge Be Closed?

Background
Concrete is a material that has been used for centuries. For example, the Coliseum of ancient Rome was constructed with the use of concrete. Concrete is an incredible modern-day building material – it is cheap and naturally strong with regard to its compressibility. For these reasons concrete is used extensively in residential footers and foundations where it can hold weight in a downward direction. Concrete works well when it is pushed against itself.

Concrete’s weakness is in its relatively low tensile (pulling apart) strength. Forces in this manner will cause concrete to crack. Looking at cardboard as an example, if you press down on the top of a hollow cardboard box (without a front or a back), it will bend in both horizontal and vertical directions. The “walls” bend outward and the “ceiling” bends inward. These simple forces would be catastrophic to concrete.

Concrete’s low tensile strength is overcome with the addition of rough surfaced reinforcing iron bars (rebar) latticed into a network within the form. The wet concrete is then poured and cured with rebar in it. This rebar gives concrete tensile strength. The resulting material is known as steel reinforced concrete and can be used as a roadbed. This reinforcing steel although deeply buried within concrete is subject to corrosion!

Steel reinforced concrete is a popular choice for roadbeds both on ground and on bridges. Since roadbeds are subject to rain and snow, moisture will penetrate the concrete, subjecting the roadbeds to corrosion. Unlike a bridge’s metal support structure, or superstructure, that can be easily inspected for corrosion, the roadbed requires a more creative inspection.

Why does the roadbed corrode? Once the rebar begins to react, it produces iron(III) oxide on the surface of the rod, increasing the size of the rebar and causing the concrete to expand and crack. Concrete cannot withstand the pressure. The result is flaking and peeling called spalling. As the concrete cracks, new surfaces within the concrete roadbed are exposed to the air and rain. This brings more moisture and oxygen to the rebar, which exacerbates rusting and increases the rate of deterioration of the bridge.

One species that affects rebar corrosion is the chloride ion (Cl\(^-\)). Its major effect is felt in two primary areas of the country:

- Bridge structures and roadway surfaces constantly exposed to salt air.
• Northern states where ice melter is used to dissolve ice or snow. Melter is composed of sodium chloride, potassium chloride, calcium chloride or magnesium chloride. All three of these solids dissolve in the ice or snow resulting in solutions that contain chloride ions.

Over time, the chloride ion solution oxidizes and corrodes the rebar in the roadbed. For a demonstration of this phenomenon on the Old Perley Bridge in Hawkesbury, Ontario, visit www.nrc.ca/irc/newsletter/v6no1/corrosion_e.html.

Examples like the Old Perley Bridge show that the concentration of chloride ion in the roadbed is closely correlated with corrosive damage. Therefore, determining the chloride concentration in concrete is useful when assessing a bridge’s condition.

The Problem
The Bridge Analysis and Redevelopment Corporation (BARC) received a lucrative contract from the state of Florida for bridge maintenance in 13 of its coastal counties. Upon preparing bridge #123 in Dade County, a technician noted extreme spalling of the concrete roadbed. She reported this to her supervisor, who halted the maintenance project on the bridge until additional analysis was completed.

Your Task
Your company ElectroChem Inc. was called in to test a concrete core sample from this bridge and provide guidance as to the future course of action. Should the bridge be closed?

BARC has sent you a concrete core sample. Your job is to analyze the sample for their chloride ion content and to write a report that provides details as to the condition of the bridge. You will perform two different analyses on the samples, each verifying the validity of the other. Your experimental results will provide BARC with additional information about the extent of damage to the bridge and play a key role determining the level of bridge rebuilding that will be necessary.

The web sites below describe a new chloride test kit that some federal and state highway departments are using to get faster, cheaper results from their chloride analyses. Since these tests are used at the site and even though they are subjected to contamination and validity, they provide quick insights into possible courses of action.

www.fhwa.dot.gov///winter/roadsvr/CS044.htm

www.fhwa.dot.gov///winter/roadsvr/chloride.html

BARC still wants an accredited lab, like ElectroChem, to perform analytic analyses of
concrete samples to validate the data from the new chloride test kits. You will be assessed on your ability to implement these standards, and provide a written report on your findings.

**Materials**

- Concrete sample
- See standard procedures for additional materials

**Safety and Disposal**

In Standard Procedure D-2 you will use 1M silver nitrate (AgNO₃). This material stains skin and clothing. Use appropriate protective equipment such as gloves, aprons, etc. Excess silver nitrate will be contained in the Erlenmeyer flasks labeled “silver waste” (from Standard Procedure D-2). Your instructor will provide a waste container. The solid silver chloride produced in Standard Procedure D-2 can typically be disposed of in the trash, but discuss with your instructor before doing so.

**Procedure**

Take the following steps to determine if the bridge should be closed.

1) Obtain a request for sample analysis from BARC.

2) Perform the specified tests using standard procedures.

3) Prepare a formal report on your findings.

**Assessment**

A formal report is to be provided back to BARC. It should include:

- Request for Sample Analysis (10pts).
- Lab Analysis Work Order (15pts). This includes your suggested action as determined by Standard Procedure F-1.

Your grade on this activity includes:
- The written report on your findings (65 points).

- Adherence to standard procedures (see below) (maximum 40 points).

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<tr>
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<th>Adequate</th>
<th>Average</th>
<th>Good</th>
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**Standard C-1**

Technique

**Standard C-2**

Technique

**Standard D-1**

Technique

**Standard D-2**

Technique

(Due to its length, point values are doubled. For example, excellent = 16 pts., good = 12 pts., etc.)
Instructor Notes

Should the Bridge Be Closed?

The problem of bridge corrosion is a real problem and the analysis students perform are based on real corrosion testing protocols. Chloride once present provides a faster mechanism, once it is present it cannot be removed, and corrosion is imminent.

Chloride detection is accomplished using two methods. Students will utilize both a chloride ion specific probe and the technique of gravimetric determination. If an ion specific probe is not available, consider having multiple groups do the gravimetric determination and compare results.

Time Required

Concrete Preparation: 60 minutes.
Concrete curing: One week minimum.
Standard Procedure C-1: 50 minutes.
Standard Procedure C-2: 50 minutes.
Standard Procedure D-1: 5 minutes per group (assuming only 1 probe is available).
Standard Procedure D-2: 100 minutes.
Drying time for D-2: 3 days.
Standard Procedures E-1, E-2, G-1: 40 minutes (calculations which can be performed as homework).
Completion of Final Report: 50 minutes (can be performed as homework).

Key Science Concepts

Chemistry
- deviation (precision)
- accuracy (% error)
- dimensional analysis (a.k.a. unit analysis)
- gram to mole conversions
- gram formula mass
- ion specific probes
- solution concentration
- extraction
- filtration
- molarity
- solubility
- gravimetric determination
• precipitation reactions

Prior Concepts and Skills Needed

• Ions
• solubility
• precipitation reactions
• chloride corrosion mechanism from Relevant Rusting activity
• moles
• basic stoichiometric operations
• dimensional analysis
• gram formula mass

Materials

Per class

Mortar mix (which we will become “concrete”).
Salt, sodium chloride NaCl (reagent grade will provide better data, if not available household will suffice).
3 oz Dixie cups (for production of the round “coring” of concrete).
Plastic bags to give students their sample core of concrete (adds authenticity, verses giving the students their samples in a Dixie cup).
Distilled water (for mixing into the mortar).
Mixing stick.
See standard procedures for additional materials.

Safety and Disposal

The mixing of mortar should be done with care in a fume hood. Portland Cement (the active ingredient in all concrete/mortar mixtures) is alkaline and a lung irritant. Wear gloves. Most bricklayers simply do not inhale during the preparation process. 1M silver nitrate stains skin and clothing. Ensure students are provided with appropriate protective equipment (gloves, aprons, etc.). Excess silver nitrate such as those in the erlenmeyer flasks labeled “silver waste” (from Standard Procedure D-2) can be precipitated easily using 1M NaCl. (Flinn silver nitrate disposal 11b). The solid silver chloride produced in Standard Procedure D-2 can typically be disposed of in the trashcan, but check local ordinances before doing so.

Getting Ready

The concrete needs to be prepared based on the following recipe. Use a standard mortar mix (available at home improvement centers / hardware stores) typically in 60 lb.
or 80 lb. bag. Prepare several different mixes and ensure that you code them.

Below are some suggestions. You can calculate the actual accepted values based on standard procedure H-1. The Standard Procedure F-1 ultimately determines the health of the concrete.

Sample I  a 0% NaCl mixture (by mass) (100% mortar mix) This will produce a concrete specimen with a concern level of “None” (based on Standard Procedure F-1).

Sample II roughly a 3.6% mixture of NaCl (by mass). (~5g of NaCl + 135g of mortar mix). (Scale up, scale down as needed, keeping the ratio consistent). This will produce concrete in the 2% Cl\(^-\) range. This specimen has a concern level of “Minimal”.

Sample III roughly a 13.6% mixture of NaCl (by mass). (~22 g of NaCl + 140g of mortar mix). This will produce concrete in the 8% Cl\(^-\) range. This specimen has a concern level of “Significant”.

Sample IV roughly a 22.9% mixture of NaCl(by mass). (~40 g of NaCl +135 g of mortar mix). This will produce concrete in the 13% Cl\(^-\) range. This specimen has a concern level of “Immediate”.

The dry salt/ mortar powders should be adequately mixed prior to wetting.

**NOTE: Do not inhale any powder liberated in this mixing process. Use a fume hood.**

Using the standard test for mortar. Slowly add distilled water folding the powder into the liquid. Continue mixing, adding additional distilled water sparingly. It is very easy to "blow past the end point" and go from a powdery crumbly mess to soup by adding too much water. Use the standard bricklayer test for mortar. Scoop up a small representative sample with a flat surfaced mixing tool (paint stirrer, yard stick, trowel, flat spatula) etc. Then invert the tool. The mortar should adhere to the underside of a spatula (or trowel) without crumbling or falling. Fortunately, lives are not dependent on the quality of our mortar as it does not need to support weight, Therefore, proper water quantity in this activity is not at all as crucial as it is in building construction.

Transfer the wet mortar mix to 3 oz Dixie cups, to a depth of between 1/4 – 1/2 inch.

Allow at least a week for the concrete to cure.

Remove the samples from the cups, and place them in plastic bags for the students. Be sure to code the bags. Attach the bags to a request for sample analysis form.
Procedure Notes

Students will be proceeding throughout this activity at their own pace. Unlike the previous activities that utilized standard procedures, (Working as a Lab Tech for ElectroChem, and Coatings) the teacher cannot easily look at the students’ adherence to standard procedures after class. Therefore the only way to quantify the quality of a student’s work is during the lab period. Instructors must first familiarize themselves with the standard procedures. During the lab period use a subtractive method. In a systematic fashion, wander around the room observing students’ work. If you happen to “catch” a lab group failing to follow the designated protocol make a note of it. At the end of the lab period if a lab group was not cited for an infraction they receive the perfect score of 8 points for that standard procedure. Use your judgement for the lab groups that failed to follow procedures exactly.

If you chose, the %Cl- ion values obtained by the students in Standard Procedure E-1 and E-2 can be looked at statistically using Standard Procedure G-1.

Assessment

Students should draft a formal report back to BARC. It should include:

- Request for Sample Analysis (10pts).
- Lab Analysis Work Order (15pts) This includes the finding (suggested action to be taken) as determined by Standard Procedure F-1.
- Experimental Data / Calculations (30pts) Included in this section Standard Procedures E-1, and E-2.

*If Chosen: Statistical Analysis (10 pts) Included in this section Standard Procedure G-1.*

Students grade on this activity include:

- the written report on their findings (as above) (65 points).
- adherence to standard procedures (as below) (maximum 40 points).

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**Standard C-1**
Technique

**Standard C-2**

Technique

**Standard D-1**

Technique

**Standard D-2**

Technique

(due to its length, point values are doubled. i.e. excellent =16pts, good =12pts, etc.)

**Explanation**

Chloride ion concentration is an indirect way of measuring the degree of corrosion in a concrete structure. The presence of the chloride ion accelerates corrosion by providing an alternative mechanism for iron. The formation of this chloride complex speeds up the corrosion process. Details can be found in the appendix of the first activity "Web Research: What's Up With Corrosion?"

In the gravimetric determination, the silver ion \( (\text{Ag}^+) \), dissolved in the silver nitrate solution reacts with the chloride ion \( (\text{Cl}^-) \), dissolved in the concrete sample, to produce silver chloride \( (\text{AgCl}) \), a compound with very low solubility. The AgCl precipitates out as a white cloudy substance in the flask. AgCl will continue to form with the addition of \( \text{AgNO}_3 \) as long as chloride ions are present in the sample.

\[
\text{Ag}^+ (\text{aq}) + \text{Cl}^- (\text{aq}) \rightarrow \text{AgCl (s)}
\]

\[
\text{Ksp} = [\text{Ag}^+ (\text{aq})] \times [\text{Cl}^- (\text{aq})] = 1.8 \times 10^{-10}
\]

When the endpoint for this reaction is reached, all the chloride ion in the sample will have been used up by the silver nitrate titrant that students added to the sample.

Silver halides (as a class of compounds) are well studied substances that typically change when subjected to energy. The most classic example is the photographic use of silver bromide. In this activity, the extremely fine white crystals of silver chloride change conformation when heated. In the presence of heat they change color from the fine white precipitate to a brown/dark purple coagulated crystal. Their formula mass does not change in this transformation. This larger crystal structure coagulates and precipitates, much more readily than the original crystal. This allows for enhanced visualization of the
continued precipitation reaction in the mother liquor.

**Further Discussion**

The underlying chemistry of cement/concrete as it cures is incredibly complex, and will not be described in great detail. In summary, various silicates (tricalcium silicate and dicalcium silicate) as well as tricalcium aluminate and calcium sulfate undergo various hydrations. These hydrations (the formation of calcium silicate hydrate and calcium aluminate hydrate for example) cause the familiar crystalline structure and porosity characteristic of concrete. For more information consult the references at the end of this document.

**Justification of the Procedure**

Although uncured cement is not the same chemically as cured cement, if the original mass (before hydration) is compared to that of a dried cured sample, their masses differ by less than 2%. Therefore, in the interest of simplicity, the author recommends establishing the “accepted value” for a sample with the assumption of mass conservation. (See Standard Procedure H-1). Additionally, silver nitrate will precipitate almost all ions in solution (anything that exists in the mortar mix in addition to the sodium chloride which was doped). Fortunately, concrete is a very insoluble material (or else we would have an issue with our houses melting in the rain) and the quantity of “loose” soluble ions not accounted for in the mortar mix is small enough to be insignificant, and will not adversely affect this activity.

Corroding rebar causes concrete to be unsafe. One cannot examine the rebar directly without destroying the concrete roadbed. Therefore, indirect techniques must be used to indirectly determine the “health” of the rebar within concrete. These indirect techniques include X-ray, conductivity tests, and lastly chloride ion concentration tests. The more chloride present in a sample of concrete, the more corrosion that is underway.

Traditionally, the Federal Highway Agency takes samples from concrete bridges and sends them to a laboratory for analysis using potentiometric titration, a complex and time consuming laboratory procedure. The New Mexico State Highway and Transportation Department has found a simple, accurate, and “on the site” test for determining chloride ion concentration, utilizing a chloride specific ion probe. Although not as accurate, as the laboratory tests, the results are perfectly sufficient for the analysis. In this lab activity, we modeled the two actual procedures for determining chloride ion concentration. The use of the chloride specific ion probe is typical of the New Mexico on site procedure. A true potentiometric titration is too complex for a typical high school / introductory college laboratory, so a standard gravimetric determination was substituted in this second case.
Where does the chloride ion present in concrete come from? Chloride is found in many deicing chemicals, and in coastal regions within the environment (air, rainwater runoff etc) itself. It should be noted that in the case of Florida, the actual sand that was used to make the concrete roadbeds originally came from nearby beaches. In this concrete the chloride ion was present since the road was poured, and has been corroding ever since! Obviously, catastrophic failure of structures resulted. The initial attempt to ameliorate the problem was to specify the use of stainless steel rebar. At a cost 10 times that of standard rebar Unfortunately, it performed only marginally better than the original. Washing the sand prior to mixing concrete proved difficult in large scale, and importing sand was cost prohibitive. Currently, the state of Florida uses epoxy coated rebar and this seems to be performing well. Additionally, they are experimenting with coating rebar with various polymers as well.

It should be noted that the scale provided in Standard Procedure F-1 uses chloride concentrations much greater than anything that would be seen in a real analysis of a bridge deck. These values were chosen so as to provide adequate differentiation using techniques and skill typified of that of an inexperienced student chemist.

**Addressing the National Science Education Standards**

• 177 In this activity, students observe a real question, “Should the Bridge Be Replaced?” and conduct an appropriate investigation to answer that question.

• 179 In the analysis of their concrete sample, students use ion specific probes driven by modern software.

• 182 & 264 Students communicate to BARC the action necessary as determined by their analysis of a sample.

• 184 Students conduct this investigation for reasons of public safety.

• 186 Mathematics are used in students’ analyses.

• 188 Students must report clearly their data, for investigation by the instructor.

• 199 The chemistry of bridge analysis/maintenance affects most modern individuals.

• 201 The corrosion of iron is an example of a redox (electron exchange) reaction.

• 202 A time scale is provided in Standard Procedure F-1.

• 203 The presence of chloride ions accelerate the corrosion reaction.

• 260 A discussion of the evolution of rebar technology expose students to improvements in technical design.

• 289 The corrosion of the steel reinforcing of a bridge deck is a slow and progressive change

• 300 Evidence is used in the scientific arguments presented in the students’ final reports.

**Addressing the Voluntary Industry Standards**
L1.03.09 Students compare their results to the specifications provided in standard procedure F-1.

L1.04.01 Students work as part of a team.

L1.04.07 Students base their action plan on data obtained in the laboratory exercise.

L1.04.04 & L2.04.02 Safety is addressed throughout this activity.

L2.02.07 Labels are prepared for various glassware throughout their procedures.

L2.02.10 Concentration conversions are accomplished (g/ml $\rightarrow$ %) (mg/L $\rightarrow$ %).

L2.05.01 Chemical laboratory equipment is handled safely throughout this activity.

L3.01.09 Formula weight is addressed in this activity.

L3.01.12 Stoichiometry is applied in this activity.

L3.03.08 Students are expected to clean their laboratory station upon completion.

L3.05.01 Solutions are mixed to ensure representative samples are used in the analysis.

L3.05.05 The initial concrete sample is ground.

L4.01.02 & L4.04.01 & L7.05.01 Precision and accuracy are addressed in the statistical data portion (Standard Procedure G-1).

L4.01.14 Errors in Deviation and %error are calculated from data students obtain in the lab.

L4.03.01 The Standard Procedures in this activity are based on ASTM protocols.

L4.03.04 Students carry out stepwise procedures, such as performing tests according to procedures, calculating results, maintaining the instrument for future use, and documenting the above behavior. Additionally, students may participate in the calibration of the Cl-specific ion probe.

L4.06.04 A final report is written with the follow-up based on the outcome of tests performed.

L5.01.04 Students witness the formation of silver chloride, an insoluble precipitate.

L5.02.01 Students experience different sections of published standard methods, including scope, safety considerations, calibrations, procedure, and results.

L5.02.03 ASTM is identified as an association that develops analytical methods.

L5.03.02 Mole quantities are used in this exercise.

L5.03.03 Various ways of expressing solution concentrations are experienced in this exercise.

L5.11.02 The production of silver chloride is an important precipitation reaction used in
analytical methods.

L5.11.05 A multistep gravimetric procedure is conducted in this activity.

L7.05.02 A written report is generated providing results, conclusions, and the next steps.

**Addressing the SCANS Skills**

**Applied Mathematics**

Level 6 – Students solve solution ion concentrations involving multiple step calculations and conversions.

Level 7-Students calculate using basic statistical concepts (% error, deviation).

**Applied Technology**

Level 4- In performing an extraction, a gravimetric determination and utilizing a computer driven probe, students apply principles underlying the operation of several physical systems.

**Locating Information**

Level 5- In calculating their solution concentrations, students sort through information presented in several locations (different standard procedures).

**Observation**

Level 6- In the gravimetric determination, the morphing of the silver chloride into a more useful conformation force students to visualize on a microscopic level how the task of precipitation is involved with basic limiting reagent concepts.

**Reading for Information**

Level 5- Students use complex standard procedures and have to apply the instructions from their document to their situation.
Teamwork

Level 4- Students work as part of a team, providing and accepting direction from others.

Writing

Level 5- As the report in this activity is the final communication a lab group has created, the expectation is that the students analysis is clear and without mechanical and grammatical errors.

References

Gary Drigel Professor in Engineering Technology, Middletown Campus 727-3237 drigelgs@muohio.edu


http://www.fhwa.dot.gov///winter/roadsvr/cs044.htm


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

C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete

C 114 Test Methods for Chemical Analysis of Hydraulic Cement

E 832 Specification for Laboratory Filter Papers

Flinn Chemical & Biological Catalog Reference Manual 2002 Laboratory Chemical Disposal Procedure #11, Silver compounds pp986-988.

Prologue
# Request for Sample Analysis

<table>
<thead>
<tr>
<th>Name of Contact Person</th>
<th>Name of Laboratory doing Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Request No.</td>
</tr>
<tr>
<td></td>
<td>St. Address</td>
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<tr>
<td></td>
<td>City, State, Zip Code</td>
</tr>
<tr>
<td>Phone No.</td>
<td>Fax No.</td>
</tr>
<tr>
<td></td>
<td>Date of Request</td>
</tr>
</tbody>
</table>

**Description of problem**

**Description of specimen materials**

**Specific analyses to be performed including requirements within standards**

**Date results required:** ____________
Lab Analysis Work Order

ElectroChem Labs, Inc.
4717 Precipitation Drive
Labland, USA  26817
Ph. (800) 355-5585
tax: (800) 355-1079

Company Representative  
Requested By

Company Name
St. Address
City, State, Zip Code
Phone No.  Fax No.

Description of Work Requested

Date Work Begun

Description of Work Done

Date Work Completed

Summary of Findings

Team

NOTE: Complete description of tests, procedure, and findings on attached sheet(s)
Standard Test Method C-1 Preparation of Concrete Core Samples

1. Scope
1.1 This test method provides for the sampling and preparation of concrete for water-soluble ion tests.
1.2 This standard does not claim to address all safety concerns regarding its use. It is the user’s responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents
2.1 American Society for Testing and Materials (ASTM) Standards:
   C 1218 Standard Test Method for Water-Soluble Chloride in Mortar and Concrete
   C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
   C 114 Test Methods for Chemical Analysis of Hydraulic Cement
2.2 Standard Test Method C-2 Extraction of Cl⁻ From Concrete Samples
2.3 Standard Test Method C-0 Coring of Concrete Samples
2.4 Standard Test Method E-1 Mathematical Treatment of [Cl⁻] via Specific Ion Probe
2.5 Standard Test Method E-2 Mathematical Treatment of [Cl⁻] via Gravimetric Determination

3. Significance and Use
3.1 A coring of concrete using a 2 inch coring bit and a hammer drill utilizing Standard test method C-0 for Coring of Concrete Samples should be completed prior to implementing this procedure.

4. Materials
   latex gloves
   clay triangle
   ring stand
   electronic balance
   a 2 inch core sample roughly 20 g.
   porcelain evaporating dish
   mortar and pestle
   250 mL beaker
   permanent marker
   stir rod with rubber policeman

5. Procedure
5.1 Put on a pair of latex gloves.
5.2 Obtain a evaporating dish of suitable size.
5.3 Heat evaporating dish in a Bunsen burner flame for 2 min.
5.4 Allow for cooling time.
5.5 Mass evaporating dish ___________ g.
5.6 Reheat evaporating dish in a Bunsen burner flame for 2 min.
5.7 Allow for cooling time.
5.8 Mass evaporating dish ___________ g (this is Value X).
5.9 If mass from steps 5.5 and 5.8 is within +/-0.01g proceed to step 5.11.
5.10 If mass from steps 5.5 and 5.8 is not within +/- 0.01 g repeat steps 5.6-5.8.
5.11 Transfer sample to mortar.
5.12 Crush sample with pestle so particle size is no larger than a grain of short grain rice.
5.13 Using the stir rod with rubber policeman, transfer sample to evaporating dish.
5.14 Heat 3 min.
5.15 Allow for cooling time.
5.16 Record mass of evaporating dish + sample ________ g.
5.17 Heat 2 min.
5.18 Allow for cooling time.
5.19 Record mass of evaporating dish + sample ________ g (this is Value Y).
5.20 If mass from steps 5.16 and 5.19 is within +/-0.01g proceed to step 5.22.
5.21 If mass from steps 5.16 and 5.19 is not within +/- 0.01 g repeat steps 5.17-5.19.
5.22

\[ \text{Value } Y \ _______ \text{(step 5.19)} \]

\[ - \text{ Value } X \ _______ \text{(step 5.8)} \]

\[ \text{__________________________} \]

\[ \text{Value } A \ _______ \text{ (you will use this in Standard Procedures E-1, E-2)} \]

5.23 Thoroughly wash a 250-mL beaker, rinse with tap water, then with distilled water. (You
do not need to dry this beaker.)
5.24 This beaker should be marked with a unique designation. Teams of technicians should
be assigned a designation for their team as follows. Each team uses the first 3 letters of
the last name of one member as this unique designation. These three letters are then
followed by the sample number and source company.
5.25 Using a stirring rod with rubber policeman quantitatively transfer sample to the labeled
250 mL beaker.
5.26 Consult C-2 Standard Test Method C-2 for Extraction of Cl\(^{-}\) from concrete samples.
Standard Test Method C-2 Extraction of Cl⁻ From Concrete Samples

1. Scope
1.1 This test method provides for the sampling of concrete for water-soluble chloride ion. In most cases, soluble chloride is equivalent to total chloride.

1.2 This standard does not claim to address all safety concerns regarding its use. It is the user’s responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents
2.1 American Society for Testing and Materials (ASTM) Standards:
   C 1218 Standard Test Method for Water-Soluble Chloride in Mortar and Concrete
   C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
   C 114 Test Methods for Chemical Analysis of Hydraulic Cement
   E 832 Specification for Laboratory Filter Papers
2.2 Standard Test Method C-1 Preparation of Concrete Core Samples

3. Significance and Use
3.1 The authors assume the amount of water-soluble chloride in most concrete systems is equal to the total amount of chloride in the system.
3.2 This procedure does not address other water-soluble ions in the concrete.
3.3 Water-soluble chloride, when present in sufficient amount, is capable of leading to initiation or acceleration of the corrosion of metals, such as steel, embedded in or contacting a cement system such as mortar, grout, or concrete. Thus, its determination shall be required to evaluate the potential of a cement system for undergoing such reactions or to investigate cement systems where such reaction has already occurred. However, it must be recognized that water-soluble chloride determined at some particular time in the life of a cement system is capable of being substantially different than that at another time; for example, the service environment is capable of resulting in a higher water-soluble chloride content due to changes in solubility or a lower one due to leaching.
3.4 It is possible for test conditions to affect water-soluble chloride determinations. Use caution when comparing results from this test method with those from other test methods.

4. Materials
   sample for standard procedure C-1
   labeled beaker from standard procedure C-1
   distilled water
   100 mL graduated cylinder
   large funnel
   filter paper (Fisher P5 24 cm medium porosity slow flow or equivalent)
graduated 250 mL erlenmeyer
2 150 mL beakers
paper towel

5. Preparation
5.1 See standard procedure C-1 for preparation of concrete core samples.

6. Procedure
6.1 Add 100 mL of distilled water to the labeled beaker.
6.2 Use the stir rod to stir the mixture thoroughly for 5 minutes to dissolve the chloride ions present. Be careful not to spill any of the contents.
6.3 Label a graduated 250 mL erlenmeyer with the same nomenclature as the labeled beaker.
6.4 Fold a piece of filter paper and place it in the funnel. Place the funnel into the labeled erlenmeyer.
6.5 Swirl up the beaker contents and quickly transfer both solid and liquid to the filter paper. Use a spray bottle of distilled water to rinse all solid material from the beaker into the filter paper.
6.6 Let filtration proceed until its rate slows to 1 drop per 30 sec or slower.
6.7 Using distilled water, spray down solid in filter paper to bottom of cone.
6.8 Add an additional 100 mL of distilled water to filter paper.
6.9 Let filtration proceed until final volume of liquid reads 200 mL (as determined by the line on the graduated erlenmeyer flask).
6.10 Remove funnel (it will still be dripping).
6.11 Dispose of the filter paper and concrete mixture in a trash can.
6.12 Cap erlenmeyer with a stopper.
6.13 Swirl erlenmeyer to mix solution homogeneously. If remaining steps in procedure cannot occur at this time, this is a good stopping point.
6.14 Obtain two 150 mL graduated beakers.
6.15 Wash beakers and do a final rinse with distilled water.
6.16 Dry with paper towel.
6.17 Label both beakers with the same nomenclature as on the erlenmeyer.
6.18 To one beaker 's label add "to Cl\(^{-}\) ion specific probe".
6.19 To the other beaker's label add "to gravimetric determination".
6.20 Carefully pour erlenmeyer solution into both beakers, split equally with 100 mL of in each beaker.
6.21 With the beaker labeled "to Cl\(^{-}\) ion specific probe" proceed with standard procedure D-1, *Determining Soluble Cl\(^{-}\) Concentration Using an Ion-specific Probe*.
6.22 With the beaker labeled "to gravimetric determination" proceed with standard procedure D-2, *Determining Soluble Chloride Ion by Gravimetric Analysis of Precipitated Silver Chloride*.
Standard Test Method D-1 Determining Soluble Cl⁻ Concentration Using an Ion-specific Probe.

1. Scope
1.1 This test method provides for the sampling and analysis of water soluble chloride ion. In most cases, soluble chloride is equivalent to total chloride.
1.2 This standard does not claim to address all safety concerns regarding its use. It is the user’s responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
1.3 Values stated in this standard use SI units exclusively.

2. Referenced Documents
2.1 Standard Test Method C-2 Extraction of Cl⁻ from concrete samples
2.2 Standard Procedure E-1 Mathematical Treatment of Cl⁻ Specific-ion Probe Data
2.3 Standard Procedure F-1 Concrete Health Rating Matrix

3. Significance and Use
3.1 The authors assume the amount of water-soluble chloride in most concrete systems is equal to the total amount of chloride in the system.
3.2 Water-soluble chloride, when present in sufficient amount, is capable of leading to initiation or acceleration of the corrosion of metals, such as steel, embedded in or contacting a cement system such as mortar, grout, or concrete. Thus, its determination shall be required to evaluate the potential of a cement system for undergoing such reactions or to investigate cement systems where such reaction has already occurred. However, it must be recognized that water-soluble chloride determined at some particular time in the life of a cement system is capable of being substantially different than that at another time; for example, the service environment is capable of resulting in a higher water-soluble chloride content due to changes in solubility or a lower one due to leaching.
3.3 It is possible for test conditions to affect water-soluble chloride determinations. Use caution when comparing results from this test method with those from other test methods.

4. Materials
Cl⁻ ion specific probe (Vernier CL-DIN or equivalent)
Supporting software (LoggerPro version 2.2 or equivalent)
Labeled beaker of distilled water
Labeled beaker of waste water
Sample from standard procedure C-2
Kimwipe
Wash bottle containing distilled water

5. Procedure
5.1 Calibrate Cl⁻ ion specific probe according to manufacturer’s instructions.
5.2 Place probe into distilled water for short term storage.
5.3 Remove Cl⁻ probe from distilled water.
5.4 Carefully pat dry the electrode using a Kimwipe.
5.5 Place electrode into sample from standard procedure C-2.
5.6 Without letting the probe contact the beaker surfaces, slowly stir the sample for 10 seconds.
5.7 Let reading stabilize.
5.8 Record Cl⁻ concentration ________ mg/L (this is value B).
5.9 Remove probe from sample.
5.10 Discard sample.
5.11 Rinse probe for 30 seconds using distilled wash bottle.
5.12 Dry probe using Kimwipe.
5.13 If probe is to be used for an additional determination, return probe to beaker labeled "distilled water".
5.14 If probe is to be stored, consult manufacturer’s instruction for long term storage.
5.15 Proceed to Standard Procedure E-1 Mathematical Treatment of Cl⁻ Specific-ion Probe Data.
5.16 Proceed to Standard Procedure F-1 Concrete Health Rating Matrix.
Standard Test Method D-2 Determining Soluble Chloride Ion by Gravimetric Analysis of Precipitated Silver Chloride

1. Scope
1.1 This test method provides for the sampling and analysis of concrete for water-soluble chloride ion. In most cases, soluble chloride is equivalent to total chloride.
1.2 *This standard does not claim to address all safety concerns regarding its use. It is the user’s responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*
1.3 Values stated in this standard use SI units exclusively.

2. Referenced Documents
2.1 American Society for Testing and Materials (ASTM) Standards:
   - C 1218 Standard Test Method for Water-Soluble Chloride in Mortar and Concrete
   - C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
   - C 114 Test Methods for Chemical Analysis of Hydraulic Cement
   - E 832 Specification for Laboratory Filter Papers
2.2 Standard Test Method C-2: Extraction of Cl- From Concrete Samples

3. Significance and Use
3.1 The authors assume the amount of water-soluble chloride in most concrete systems is equal to the total amount of chloride in the system.
3.2 Water-soluble chloride, when present in sufficient amount, is capable of leading to initiation or acceleration of the corrosion of metals, such as steel, embedded in or contacting a cement system such as mortar, grout, or concrete. Thus, its determination shall be required to evaluate the potential of a cement system for undergoing such reactions or to investigate cement systems where such reaction has already occurred. However, it must be recognized that water-soluble chloride determined at some particular time in the life of a cement system is capable of being substantially different than that at another time; for example, the service environment is capable of resulting in a higher water-soluble chloride content due to changes in solubility or a lower one due to leaching.
3.3 It is possible for test conditions to affect water-soluble chloride determinations. Use caution when comparing results from this test method with those from other test methods.

4. Materials
   - 250 mL beaker
   - 250 mL erlenmeyer
   - sample from standard procedure C-2
   - 10 mL graduated cylinder
   - hot plate
boiling stones
large funnel
filter paper (Fisher P5 24 cm medium porosity slow flow or equivalent)
oven
stir rod
1 M silver nitrate (this material stains skin and clothing use caution)
dropper pipette
latex gloves
permanent pen for labeling glassware

5. Procedure
5.1 Put on latex gloves.
5.2 Thoroughly wash a 250-mL beaker, rinse with tap water, then with distilled water.
5.3 Dry with paper towel.
5.4 Label using the same nomenclature as on the sample as provided in the erlenmeyer.
5.5 Place 3 boiling stones into the beaker.
5.6 Fold and place 1 piece of filter paper into the beaker.
5.7 Mass beaker, stones and filter paper ________g (this is Value H).
5.8 Remove filter paper.
5.9 Transfer sample to the labeled beaker.
5.10 Ensure quantitative transfer by spraying the beaker with a small quantity of distilled water.
5.11 Transfer 6 mL of 1M silver nitrate to the sample.
5.12 Transfer sample to hot plate, stirring occasionally, bring sample to 90 °C.
5.13 Remove from heat.
5.14 Continue to stir. As reaction proceeds the precipitate should change from a very fine white powder to a light violet color, and coagulation should clarify the upper part of the liquid.
5.15 If light violet coagulant is not present, or fine white precipitate is still present, repeat steps 5.12-5.14.
5.16 Allow beaker to stand undisturbed to allow upper region to clarify.
5.17 Slowly transfer an additional 3 mL of silver nitrate to sample. Observe.
5.18 If precipitation appears to continue, stir to try to obtain coagulation/clarification. Additional heating may prove to be necessary (steps 5.12-5.16).
5.19 Continue adding 3 mL aliquots (steps 5.17-5.18) until the addition of silver nitrate does not appear to react.
5.20 At this point let system sit until upper layer is very translucent. While carefully observing add an additional 3 drops of silver nitrate to ensure all the chloride has precipitated. If clarity remains go to step 5.21. If a precipitate continues to form repeat steps 5.17-5.18.
5.21 Label a 250 mL erlenmeyer "silver waste".
5.22 Fold the massed piece of filter paper.
5.23 Place the filter paper into a funnel, and place above the erlenmeyer labeled "silver waste".
5.24 Decant the clear liquid off the top of the beaker by transferring it to the filter paper. Any solid accidentally transferred in this process will not be lost. It will be caught in the filter paper.

5.25 When the majority of liquid has been transferred, add an additional 50 mL of distilled water to the beaker containing the precipitate. This is to allow the excess silver nitrate to rinse clean of the precipitate.

5.26 Stir the beaker containing the precipitate and the distilled water. Allow for clarification.

5.27 When clarification is complete, decant this liquid to the filter paper as above (step 5.24).

5.28 When filtering has slowed to 1 drop / 30 sec. Rinse the solid in the filter paper first with a stream of distilled water and then an additional 25 mL of distilled water.

5.29 Dispose of the contents of the erlenmeyer labeled "silver waste" according to local ordinances.

5.30 When filtration is complete, carefully transfer the wet filter paper to the labeled beaker.

5.31 Let beaker and filter paper dry for 2 days in room temperature environment.

5.32 Record mass __________g.

5.33 Let beaker and filter paper dry for an additional day and record mass ________g.

5.34 If the values for steps 5.31 and 5.32 differ by more than .01g allow additional drying time.

5.35 After drying is complete record mass here __________g. (this is Value G).

5.36 Dispose of Silver Chloride according to local ordinances.

5.37 Consult standard procedure E-2 Mathematical Treatment of Gravimetric Data.
Standard Test Method E-1 Mathematical Treatment of [Cl⁻] via Specific Ion Probe

1. Scope
1.1 This procedure handles the mathematical analysis of Cl⁻ ion concentration in a cored concrete sample. The Cl⁻ ion concentration value is obtained from a specific ion probe.

2. Referenced Documents
2.1 American Society for Testing and Materials (ASTM) Standards:
   - C 1218 Standard Test Method for Water-Soluble Chloride in Mortar and Concrete
   - C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
   - C 114 Test Methods for Chemical Analysis of Hydraulic Cement
   - E 832 Specification for Laboratory Filter Papers
2.2 Standard Test Method C-1 Preparation of Concrete Core Samples
2.3 Standard Test Method C-2 Extraction of Cl⁻ From Concrete Samples

3. Procedure
\[
\frac{mgCl^-}{L solution} \times \frac{1 L solution}{1000 mL solution} \times \frac{100 mL solution}{1} \times \frac{1 g Cl^-}{1000 mg Cl^-} = \frac{g Cl^-}{1000 mL solution}
\]

(Insert your probe value above) (Value B from D-1)

\[
\frac{g Cl^-}{2} = \frac{g Cl^-}{Value D}
\]

(Value C) (sample was cut in half for the original analysis)

\[
\frac{g Cl^-}{Value D} \times 100 = \frac{Cl^-}{Value E} (ion probe)
\]

(Value D) (ion probe)

Proceed to Standard Procedure F-1 Concrete Health Rating Matrix.
Standard Test Method E-2 Mathematical Treatment of [Cl\textsuperscript{−}] via Gravimetric Determination

1. Scope
1.1 This procedure handles the mathematical analysis of Cl\textsuperscript{−} ion concentration in a cored concrete sample. The Cl\textsuperscript{−} ion concentration value is obtained from a gravimetric determination.

2. Referenced Documents
2.1 American Society for Testing and Materials (ASTM) Standards:
   - C 1218 Standard Test Method for Water-Soluble Chloride in Mortar and Concrete
   - C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
   - C 114 Test Methods for Chemical Analysis of Hydraulic Cement
   - E 832 Specification for Laboratory Filter Papers
2.2 Standard Test Method C-1 Preparation of Concrete Core Samples
2.3 Standard Test Method C-2 Extraction of Cl\textsuperscript{−} From Concrete Samples

3. Procedure
Mass of beaker, stones, filter paper = \( \text{(Value H from D-2)} \) g.

Mass of beaker, stones, filter paper, AgCl after drying = \( \text{(Value G from D-2)} \) g.

\( \text{___________ g (Value G)} \)

\( - \text{___________ g (Value H)} \)

\( \text{___________ g AgCl (Value I)} \)

\[
\frac{35.45 \text{ g Cl}^-}{1 \text{ mol Cl}^-} \times \frac{1 \text{ mol Cl}^-}{1 \text{ mol AgCl}} \times \frac{143.4 \text{ g AgCl}}{1 \text{ mol AgCl}} \times \frac{\text{g AgCl}}{1} = \text{___________ g Cl}^- \\
\text{(Value I)} \quad \text{(Value J)}
\]
\[
\frac{\text{g Cl}^- \times 2}{(\text{Value J})} = \frac{\text{g Cl}^-}{(\text{Value K})}
\]

(sample was cut in half for the original analysis)

\[
\frac{\text{g Cl}^- \text{(Value K)}}{\text{g sample (Value A from C - 1)}} \times 100 = \frac{\% \text{Cl}^- \text{ (gravimetric)}}{(\text{Value L})}
\]

Proceed to Standard Procedure F-1 Concrete Health Rating Matrix.
## Standard Procedure F-1
### Concrete Health Rating Matrix

<table>
<thead>
<tr>
<th>Cl⁻ % Value E or Value L</th>
<th>Concern Level</th>
<th>Cl⁻ Ions Present</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>0% - 1%</td>
<td>None</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>1% - 5%</td>
<td>Minimal</td>
<td>Yes</td>
<td>Retest in 3 years</td>
</tr>
<tr>
<td>5% - 10%</td>
<td>Significant</td>
<td>Yes</td>
<td>Replace concrete within 3 years</td>
</tr>
<tr>
<td>10% +</td>
<td>Dangerous</td>
<td>Yes</td>
<td>Replace immediately rebar failure imminent</td>
</tr>
</tbody>
</table>
Standard Test Method G-1 Error Analysis

1. Scope
1.1 This procedure handles the error analysis of Cl- concentration

2. Referenced Documents
2.1 American Society for Testing and Materials (ASTM) Standards:
   C 1218 Standard Test Method for Water-Soluble Chloride in Mortar and Concrete
   C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
   C 114 Test Methods for Chemical Analysis of Hydraulic Cement
   E 832 Specification for Laboratory Filter Papers
2.2 Standard Test Method E-1 Mathematical Treatment of [Cl-] via Specific Ion Probe
2.3 Standard Test Method E-2 Mathematical Treatment of [Cl-] via Gravimetric Determination
2.4 Standard Test Method H-1 Determination of Accepted Values

3. Procedure

Precision:

\[ |(\text{Value E from E-1}) - (\text{Value L from E-2})| = \]

% Error Ion Probe

\[ \frac{|(\text{Value E from E-1}) - (\text{Value T from H-1})|}{(\text{Value T from H-1})} = \% \]

% Error Gravimetric

\[ \frac{|(\text{Value L from E-2}) - (\text{Value T from H-1})|}{(\text{Value T from H-1})} = \% \]
Standard Test Method H-1
Determination of Accepted Values

1. Scope
1.1 This procedure handles the determination of accepted values for this exercise

2. Referenced Documents
2.1 Standard Test Method G-1 Error Analysis

3. Procedure

\[ \text{Value P} = \text{m} \text{ mortar mass dry} \]
\[ \text{Value Q} = \text{m} \text{ NaCl added} \]
\[ \text{Value R} = \text{m} \text{ total mass} \]

\[ \frac{35.45 \text{ g Cl}^-}{1 \text{ mol Cl}^-} \times \frac{1 \text{ mol Cl}^-}{1 \text{ mol NaCl}} \times \frac{1 \text{ mol NaCl}}{58.44 \text{ g NaCl}} \times \frac{\text{g NaCl}}{1} = \frac{\text{g Cl}^-}{\text{Value Q}} \]

\[ \frac{\text{g Cl}^-}{\text{Value S}} \times \frac{\text{g total mass}}{\text{Value R}} = \frac{\% \text{ Cl}^-}{\text{Accepted Value, Value T}} \]