

Soil Sampling and Testing Methods to Evaluate
the Corrosivity of the Environment for Buried
Piping and Tanks At Nuclear Power Plants



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PRODUCT DESCRIPTION

Background

The corrosion behavior of buried assets at nuclear power plants (NPPs), like at many other industrial locations, is heavily influenced by the environment in which they are placed. Many recognized soil parameters have an influence on buried structures. The methods by which these parameters are measured have varied. As a result, the conclusions drawn from these methods have differed. Information related to analyzing soils and the influence on buried structures is scattered. Comprehensive guidance to determine how soil samples should be collected, analyzed, and interpreted has not been adequately compiled. This work included a review of related research, codes, and standards to provide guidance on suitable methods for soil sample collection, suitable testing methods, and optimal approaches to analyze the data.

Objectives

The objectives of this report are as follows:

- To provide guidance on identifying suitable soil sampling locations
- To provide guidance on how to determine and quantify the soil parameters that affect the corrosivity of the environment for buried pipes and tanks at NPPs
- To provide guidance on interpreting the results of the soil testing

Approach

Previous research in soil testing, both within the nuclear industry and in other utility industries, and standards related to underground structure maintenance were reviewed to develop guidance on soil testing for corrosivity at NPPs. This was accomplished by doing the following:

- Using industry experience to identify soil sampling locations
- Performing a literature review to determine what soil parameters and characteristics affect the corrosivity of buried pipe
- Identifying any applicable codes and standards that are used to characterize soils for buried piping at NPPs
- Providing recommendations for interpretation of the test results, including the effects on cathodic protection and selection of coatings, piping, and tank materials
- Developing generic procedures to perform soil testing for the identified parameters

Applications, Value, and Use

The scope of this document includes buried metallic piping and tanks and the relationship that soil characteristics have on corrosion. This document was prepared as a guidance document and source of information for performing soil testing at NPPs. It should not be considered to provide minimum or maximum testing requirements for soil testing or to supersede any established federal, state, or local regulations.

Keywords

Buried pipe

Buried tank

Cathodic protection (CP)

Corrosion

Sample handling

Soil testing

ABSTRACT

The degradation of buried assets at nuclear power plants due to external corrosion is heavily dependent on the characteristics of the environment in which they are placed. Because of access and inspection limitations, the monitoring of buried assets can be more challenging than monitoring aboveground assets within the same system. A need arises to accurately characterize the corrosivity of the soil around buried assets to assess and maintain plant integrity.

Understanding which soil parameters affect soil corrosivity, and how to properly analyze these parameters, is key to developing prevention and mitigation strategies. Proper analysis of soil parameters requires proper site selection, sample collection, and sample measurement. Industry guidance on how to collect these data is scattered and not comprehensive. This document provides a foundation in soil testing by summarizing the available industry guidance.

EXECUTIVE SUMMARY

Based on a literature review, the following soil parameters were found to have the most significant effect on the corrosivity of soil on buried structures: soil resistivity, reduction–oxidation potential, chemical constituents (for example, chlorides, sulfides), acidity and alkalinity, moisture content, microbial content, soil particle size distribution, bulk density, and hydraulic conductivity. The corrosion behavior of buried piping and tanks has been characterized based on these parameters through decades of research from several different industries. However, the methods to properly analyze and measure these parameters are not standard throughout the nuclear industry. This report provides guidelines for locating sample sites and collecting, handling, shipping, and analyzing samples for soil testing.

Backfill used at nuclear power plants (NPPs) is typically engineered backfill or flowable fill, which will have different properties from the native soil. Soil sample locations should therefore be selected at or near buried metallic structures. Soil sample locations can be selected using statistical analysis or a knowledge-based approach based on known potential problem areas. Samples can be collected using mechanical excavations, which can be achieved using an auger, by drilling, or by hand digging. Proper labeling and shipping procedures are required to maintain sample integrity, as well as to guide a comprehensive review of the results. There are shipping restrictions on soil samples that are contaminated with radioactive material and/or hydrocarbons, and for samples being transferred outside local areas.

There are many ways to accurately characterize and measure the given soil parameters. Commonly used industry standards include those of the American Society of Testing and Materials International (ASTM International) and International Organization for Standardization (ISO), Standard Methods¹, and Environmental Protection Agency (EPA) methods. Any testing should be completed using established quality assurance and quality control procedures to ensure testing integrity. Third-party labs should be vetted using reference standards, by reviewing the lab quality assurance/quality control procedures, or by a qualified testing authority.

The corrosivity of the soil affects material selection, coating selection, and cathodic protection system design and operation. Carbon steel is the primary material of construction for buried metallic assets at NPPs. It can be difficult to assign an exact corrosion rate to a given soil owing to the complex interaction of the different soil parameters. In addition, different materials will have different corrosion behavior. To predict soil corrosivity, methods have been developed ranging from qualitative guidelines, to semi-quantitative guidelines, to mathematical modeling. Providing adequate data to input into these analysis methods is essential for establishing proper integrity management strategies.

¹ Standard Methods is a joint publication of the American Public Health Association, the American Water Works Association, and the Water Environment Federation.

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1

INTRODUCTION

Leaks in buried piping systems for both safety-related and non-safety related systems presents a challenge to safe operation of nuclear power plants (NPPs). Buried systems provide an added challenge related to integrity management due to the difficulty to monitor and access these systems. The release of licensed radioactive material poses an environmental threat to local groundwater systems. The release of other process streams, including fuels, wastewater, and process chemicals can also pose an environmental threat. The release of process material also affects plant economics for both repair costs and production costs associated with possible down times. Public perception has also been identified as a major potential consequence of releases, as public perception of nuclear safety can be heavily influenced by leak events. Several initiatives have been established to better understand, inspect, and mitigate degradation of buried metallic systems within EPRI, NACE International, and nuclear regulatory organizations.

Soil testing for buried metallic structures has been identified as an essential area of study as part of a continued effort to better understand and mitigate the degradation of buried structures to support activities called out by the Nuclear Energy Institute's (NEI) Nuclear Strategic Issues Advisory Committee (NSIAC), "Buried Piping Initiative", EPRI report 1021175 *Recommendations for an Effective Program to Control the Degradation of Buried and Underground Piping and Tanks* [1], and the Nuclear Regulatory Commission NUREG-1801 "Generic Aging Lessons Learned (GALL) Report" [2], among others. Soil analysis is required to understand the external degradation characteristic of buried metallic structures. A need has been identified to provide guidance on how to collect reliable soils analysis data that can be used as part of an integrity assessment for an effective program to control the degradation of buried piping and tanks.

Corrosivity of buried assets found at NPPs is heavily influenced by the environment in which it is placed. There have been many recognized soil parameters that have an influence on buried structures. The methods by which these parameters are studied have varied, and as a result, the conclusions drawn from these methods have differed in some cases. Information related to analyzing soils and the influence on buried structures is scattered. Single sources do not provide comprehensive guidance to determine how soil samples should be collected, how they are to be analyzed, and how that analysis may be interpreted. The work presented herein includes a review of related research, codes, and standards to provide guidance on suitable methods for soil sample collections, suitable testing methods, and optimal approaches to analyze the data.

1.1 Scope and Purpose of this Report

Industry information and guidance related to soils testing for soil-side corrosivity of buried metallic structures has historically been scattered and fragmented. Reliable soil data is required to accurately predict degradation behavior due to external corrosion, in conjunction with other factors. The work herein provides guidelines for collecting and analyzing soils data for corrosion analysis of buried metallic structures.

1.2 Key Points

Throughout this report, key technical information is summarized in “pop outs”. Pop outs are bold lettered boxes that succinctly restate information covered in detail in the surrounding text, making the key points easier to locate. The primary intent of a pop out is to emphasize information that will allow individuals to take action for the benefit of their plant. Appendix A contains a compilation of all of the key points. The compilation in the appendix restates each key point and provides reference to its location in the body of the report. By reviewing this listing, users of this guide can determine whether they have taken advantage of key information that the author believes would benefit their plants.



Key Technical Point

Targets information that will lead to improved data collection and analysis.

2

EFFECT OF PHYSICAL AND CHEMICAL SOIL PROPERTIES ON THE CORROSION RATE OF BURIED CARBON STEEL PIPING

2.1 General Overview of Metallic Corrosion in Soil

This report provides a thorough discussion of the degradation of buried metallic structures in soil. The following EPRI reports provide a detailed discussion of corrosion, corrosion mechanisms, and the degradation of buried steel structures:

- *Service Water Corrosion and Deposition Sourcebook*. TR-103403 [3].
- *Service Water Piping Guideline*. 1010059 [4].
- *Balance of Plant Corrosion-The Underground Piping and Tank Reference Guide: Revision 1*. 1021470 [5].
- *A Literature Review of Soil-Side Corrosion Rates for Buried Metallic Piping*. 1025256 [6].
- *Recommendations for an Effective Program to Control the Degradation of Buried and Underground Piping and Tanks (1016546, Revision 1)*. 1021175 [1].
- *Interim Guidelines for Determining the Corrosion Rate for Use in Fitness for Service Evaluations of Buried Pipe*. 3002003057 [7].

2.1.1 Corrosion Fundamentals

Corrosion is based on the principle that all metals are more thermodynamically stable in the oxide state. Therefore, metals will oxidize from the metastable metallic state to the oxide state given enough time. For metal loss to occur a corrosion cell is required.

A corrosion cell consists of an anode, cathode, electrolyte (soil or groundwater in this case), and a metallic electron path. The metallic electron path is provided by the buried metallic structure itself. The anode and cathode exist at different locations on the metal surface due to differing conditions along the metal surface. Anodes are regions on the surface of an alloy that support oxidation reactions. Oxidation reactions in a corrosion cell result in metal loss where metal ions are liberated from the alloy into the electrolyte and electrons flow through the alloy toward cathodes. Cathodic sites, or cathodes, on an alloy surface support reduction reactions. Reduction reactions consume electrons and reactive species from the environment, like dissolved oxygen or protons, and liberate new compounds or ions. Both reactions must occur for corrosion to occur and the kinetics of the slower reaction will determine the corrosion rate. For buried metallic structures, anodic and cathodic regions can exist on the same structure and are formed based on the metal properties and local environment adjacent to the alloy [8, 9, 10].

An electromotive force between the anode and cathode provides the driving force for corrosion cell reactions. The potential difference between the anode and cathode results in direct current flow. The magnitude of the current flow is directly proportional to metal loss according to Faraday's Law:

$$\frac{m}{a * t} = \frac{i * M}{n * F}$$

Where:

m = the mass loss of the corroding metal, lb (grams)

a = the exposed surface area of the metal, in² (cm²)

t = the time of exposure, seconds

i = the current density, A/in² (A/cm²)

M = the atomic weight of the metal, lb/mol (grams/mol)

n = the number of electrons transferred

F = Faraday's constant (96,500 Coulombs/mol of e⁻)

For electron flow to occur, an equivalent ionic current must flow from the anode to the cathode through an electrolyte. In most cases the cathodic half reaction, or reduction reaction, determines the reaction rate. Reduction reactions involve the addition of an electron to a species. The reduction of oxygen in neutral or alkaline environments and the reduction of the hydrogen ion in acidic environments are the most common reduction reactions in soil. As a result, soils analysis related to corrosion mainly focuses on measuring the amount of oxygen, hydrogen ions, and other ionic species that can make the corrosive environment more aggressive. Soils analysis is also based on determining how well the corrosive species can move through the soil, detecting additional sources of corrosive species (microbes), and detecting species that make the environment less aggressive.

2.1.2 General and Localized Corrosion of Buried Components

When exposed to soil, buried metallic structures will experience corrosion activity. Corrosion will occur at exposed metallic locations that are more anodic than adjacent, electrically continuous areas. These adjacent, electrically continuous areas can be on the same pipe or a nearby buried metallic structure. The soil serves as the electrolyte for the corrosion cell. Anodic and cathodic areas can be mobile on the pipe surface due to local variations in corrosion activity and changes in the environment at different areas on the pipe. The movement of the anodic and cathodic areas will result in general metal loss over time [11]. Common environmental factors that create anodic and cathodic regions are oxygen concentration differences, dissimilar soils, soil contamination from ground runoff, and corrosion product formation [10]. Metals exposed to certain environments can form a passive film layer that inhibits further corrosion activity from occurring. This passive film is a result of an initial corrosion reaction and deposition of corrosion products on the metal. The film layer can have varied chemical composition, structure, and thermodynamic stability and, as a result, the passive film on certain alloys is more resistant to corrosion than others [11].

Localized corrosion occurs in one location where the anodic area of a buried metallic structure remains stationary. The most common form of localized corrosion is pitting or crevice corrosion. A pit is a narrow, deep area of metal loss caused by damage or breakdown of the passive film on the metal surface. This breakdown can be caused by mechanical damage or by interaction of the passive film with certain chemical species (for example, chlorides for steels). The pit will grow as the local environment underneath the film breakdown remains more anodic than the surrounding areas. The corrosion rate within a pit will often be much higher than the general corrosion ongoing at the surface. The high corrosion rate can be attributed to a high cathode-to-anode surface area ratio, unfavorable anodic reaction kinetics, and the presence of several supporting cathodic reactants. This can lead to pinhole leaks.

One way to prevent corrosion activity is to isolate the buried metallic structure from the soil, thus isolating it from the electrolyte and retarding the corrosion reaction. This is the intent of applying coatings to the external surfaces of buried metallic structures. The coatings minimize the amount of metal surface area in contact with soil and, in effect, minimize corrosive species from interacting with the pipe surface [5].

Another corrosion mitigation method that is used to minimize corrosion on coated metal structures is to use cathodic protection (CP). Cathodic protection is used in conjunction with coatings to protect buried assets. It is utilized for bare buried piping as well. Cathodic protection can polarize the anodic sites on a buried metallic structure near the level of the cathodic sites, thus greatly reducing the driving force responsible for formation of corrosion cells. Cathodic protection systems use sacrificial anodes, direct current via rectifiers, or a combination of both to make the protected buried metallic structure more cathodic [10].

2.1.2.1 Galvanic Corrosion

Galvanic corrosion occurs due to two metals of differing corrosion potentials interacting electrochemically. Common galvanic couples are magnesium connected to steel and zinc connected to steel. The alloy with a more active corrosion potential (for example, magnesium or zinc) will become the anode when it is connected to a more noble metal and immersed in an electrolyte. A common galvanic cell in NPPs is the cell that can form between buried active metals, such as steel and aluminum, and the more noble copper grounding system. Soil properties like resistivity can determine how effective currents flows in a galvanic cell.

Galvanic corrosion cells can also occur on the same metallic structure due to different corrosion potentials as a result of different compositions of the soil/environment, or different passivation levels of the metal itself. New sections of steel pipe are more active compared to older sections due to the extent of surface passivation. This can cause new sections to corrode due to interaction with the old piping section [12].

2.1.2.2 Dealloying

Dealloying is a form of degradation where an element of an alloy is removed due to an interaction with the environment, which leaves behind an altered alloy structure. Dealloying is a common form of degradation in copper alloys as dezincification and in cast iron as graphite corrosion. Zinc is the primary alloying element that makes up brass with copper. Dezincification occurs in alloys with greater than 15 percent zinc. Dezincification is more common in slightly acidic soils. The loss of the zinc ion results in a loss of strength in the alloy. Graphite corrosion in cast irons is the result of selective dissolution of the iron matrix in the alloy, which leaves behind only graphite. The result is a porous structure made of a network of graphite. This weakens the mechanical integrity of the alloy. Aluminum bronze alloys are also subject to dealloying through the removal of the aluminum atoms from the metal matrix. This dealloying can occur due to sulfate-reducing bacteria (SRB).

2.1.2.3 Differential Aeration Corrosion Cell

A differential corrosion cell can form on an electrically continuous structure if one region of the structure is exposed to a different environment than another region. One common differential corrosion cell occurs when the oxygen concentration varies along the surface of a buried or partially buried metallic structure. This is called a differential aeration corrosion cell. The metallic area that is exposed to a lower concentration of oxygen becomes anodic to regions with a higher oxygen concentration and the anodic area corrodes [8]. Non-homogenous soils can also create differential aeration cells. The physical and chemical soil properties in one location on the buried metallic structure can create a potential difference compared to another location on the same structure, leading to the formation of a corrosion cell. Disbonded coatings can also create local aeration cells where the pipe accessibility to aerated ground water changes along the length of the pipe.

2.1.2.4 Microbiologically Influenced Corrosion

Microbiologically Influenced Corrosion (MIC) is an aggressive form of corrosion activity that is caused either directly or indirectly by microorganism. Microorganisms, including bacteria and fungi, are normally abundant in soil and groundwater. Environmental conditions in the soil (for example, pH redox potential, temperature, salinity, nutrients, availability of oxygen) establish the types of microorganisms that thrive in any particular environment. Wet, dense clay or water-saturated organic soils void of oxygen promote the growth of anaerobic bacteria such as SRB. This is why some anaerobic soils or muck have a foul odor (from sulfide production). When microorganisms can have access to the pipe surface through damaged coating, or on bare pipe, MIC can occur. Microbiologically Influenced Corrosion is caused by the presence and activity of sessile microbes in a biofilm on a structure. Although microbes are present in all soil types their interaction with the environment at the interface between the soil and metallic structure can cause the local environment to be more corrosive than the bulk soil environment. These microorganisms can either be anaerobic or aerobic. Microorganisms are then classified into phenotypes based on their interaction with the metal surface. The most common groups of microbes associated with MIC are SRB, acid producing bacteria (APB), and metal oxidizing bacteria (MOB) [10, 13].

2.1.2.5 Stress Corrosion Cracking

Stress corrosion cracking (SCC) is a form of material degradation where both mechanical and corrosion degradation occurs. In most cases of SCC, an alloy that would normally be ductile in service can crack or fail by brittle fracture. The loss of ductility can be attributed to local hydrogen absorption or metal dissolution in the alloy near the crack tip. Stress Corrosion Cracking can have a very distinctive morphology that consists of many small cracks clustered together in what is known as a colony. This type of cracking requires specific operating and environmental conditions to occur on buried metallic structures. Operating conditions of buried piping systems in NPPs are typically well below the stress level where SCC could occur and therefore this report will not go into detail on this phenomenon [5]. It is possible to find SCC on buried piping systems near areas of mechanical damage, which results in unfavorable residual stress in the structure, and near areas of disbonded external coating that are shielded from CP.

The environmental conditions for SCC are different for different materials. For example in carbon steel, SCC has been observed in high pH and near neutral environments typically due to the presence of carbonates/bicarbonates. For stainless steels, SCC can be caused by high chloride environments.

2.1.2.6 Corrosion Rate for Soil-Side Corrosion

Many studies have been completed to determine the soil-side corrosion rate for buried materials, carbon steel in particular. The EPRI technical report “Interim Guidelines for Determining the Corrosion Rate for Use in Fitness for Service Evaluations of Buried Pipe” [8], completed a statistical analysis of corrosion data in different environments to determine a soil-side corrosion rate. The average rate among all soil types was 3.5 mpy (0.9 mm/year) for pitting and 0.7 mpy (0.2 mm/year) for general corrosion. However, in poorly aerated soils the average pitting and general corrosion rates rose to 8.2 mpy (2.1 mm/year) and 2.2 mpy (0.6 mm/year), respectively. ANSI/NACE² SP0502-2010 “Pipeline External Corrosion Direct Assessment Methodology” [14] provides a default pitting rate of 16 mpy (4 mm/year) for buried pipelines. This is based on the upper 80 percent confidence level of maximum pitting rates for long term (up to 17 years) underground corrosion rates of bare, unprotected carbon steel in a variety of soils. This value is viewed as conservative compared to other studies but it does not capture the high localized corrosion rates associated with MIC.

For carbon steel in NPPs, rates can be increased due to galvanic corrosion as a result of frequent use of dissimilar metals used in the facilities. The most common case is copper grounding systems interacting with buried carbon steel structures [6].

Soil testing, at a minimum, provides users the ability to determine the relative corrosivity of soil and provides data to determine the potential damage mechanisms for buried metallic structures. The data from soils testing is used in risk management and decision making process for mitigating actions for buried metallic structures, such as in the Guideline for the Management of Underground Piping and Plant Integrity, NEI 09-14 [15].

² American National Standards Institute(ANSI)/National Society of Corrosion Engineers (NACE)

2.2 Parameters Used to Determine Soil-Side Corrosivity of Buried Pipe

Many studies have been completed to determine which soil characteristics affect the corrosivity of soil for buried metallic structures. No single characteristic can be used to determine the corrosivity of soil for buried metallic structures. Rather there are many parameters that affect the corrosivity of the soil. These parameters interact with one another to create many different environments for buried metallic structures. In addition, the same location on a buried metallic structure is likely to see changes in the local parameters over time and throughout seasonal changes.

As a result, it is difficult to assign a corrosion rate for a buried metallic structure without gathering local corrosion engineering data. However, many studies have been completed and associated models and guidelines have been created to characterize the corrosivity of soil for buried metallic structures. A literature review was completed to determine what parameters affect soil corrosivity. Many sources agreed on several main parameters. The following list contains the most prevalent parameters used to determine soil-side corrosivity of buried metallic structures used in NPPs based on the findings of the literature review:

- Soil resistivity
- Degree of aeration
 - Oxygen levels
 - Redox potential
- Chemical constituents (for example, chlorides, carbonates, sulfates, nitrates)
- Acidity/alkalinity
- Moisture content
- Microbial content
- Soil classification and particle distribution
- Mechanical properties
 - Bulk density
 - Hydraulic conductivity

However, the sources often differ on the effect the parameter has on soil corrosivity. For example, the GALL Report, Rev 2 identified moisture content, pH, ion exchange capacity, density, and hydraulic conductivity as the soil properties that can affect degradation kinetics [2]. The EPRI report, *A Literature Review of Soil-Side Corrosion Rates for Buried Metallic Piping* [6], identified important parameters to be moisture content, degree of aeration, pH, redox potential, resistivity, soluble ionic species, and microbiological activity. A commonly used industry standard to determine relative soil corrosivity is contained in the ANSI/AWWA³ C105 [16]. The AWWA C105 corrosivity table uses a point system based on parameter values for resistivity, sulfides, pH, moisture, and redox potential to determine whether or not soil is corrosive. The AWWA C105 table was created for ductile iron piping systems. The ERPI BPWORKSTM software [17] includes chlorides, sulfides, and soil consortia in the corrosivity tables.

³ American Water Works Association (AWWA)

The studies and guidelines place varying importance on different parameters and the resulting interactions between these parameters. These interactions are summarized in Figure 2-1.

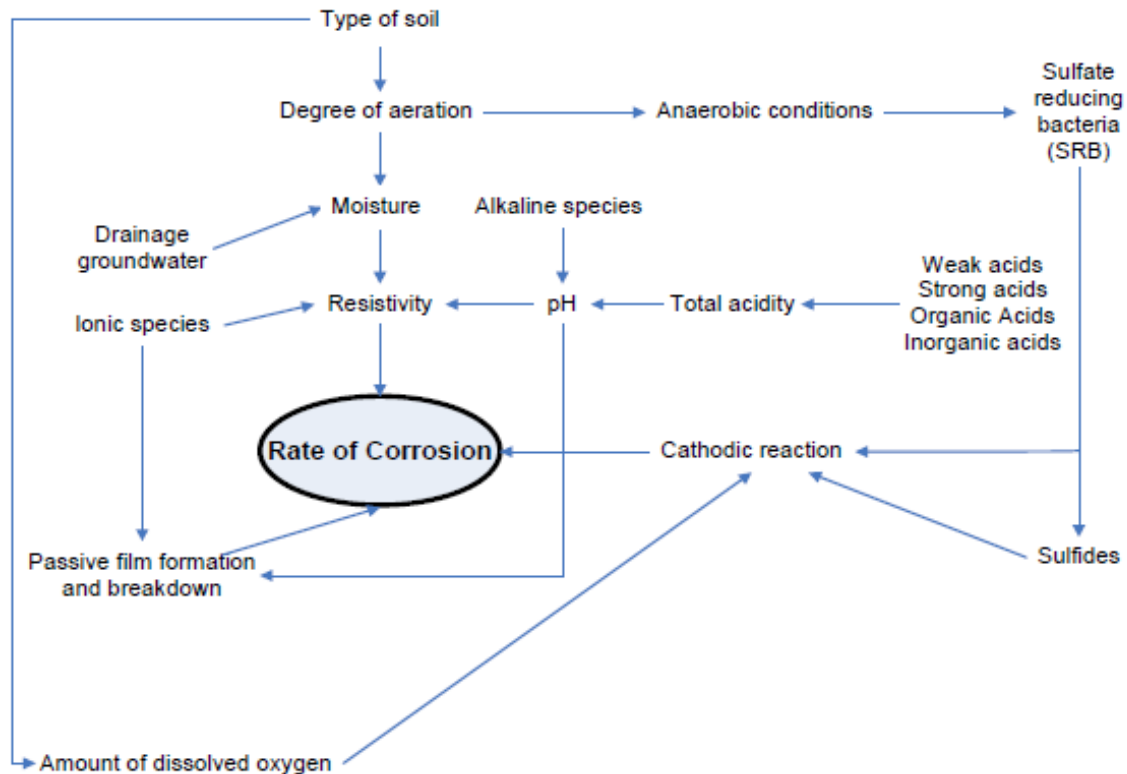


Figure 2-1
Relationship of parameters affecting soil corrosivity⁶

Due to the many influences on soil corrosivity and the complex interactions of the different parameters there are no exact equations that can predict the corrosivity of soil based on given parameters. In addition, there are commercially available models that use quantitative inputs (for example, soil test results) to estimate corrosion of steel. However, studying the most relevant parameters related to soil corrosivity provides the ability to understand the relative corrosivity of the soil and provide mitigating strategies based on the data collected.

2.2.1 Soil Resistivity

Soil resistivity is a bulk soil property that can provide a general ranking of the corrosivity of soils. Soil resistivity is a measure of one electrical component of the corrosion cell and provides a measure of the ability of corrosion currents to flow through the soil (the electrolyte) [18]. Soil resistivity is a function of the amount of dissolved salts, moisture content, soil compaction, and soil composition [19]. Soil resistivity alone does not indicate whether or not a soil is corrosive, however analyzing the soil resistivity with other factors can provide a relative likelihood of corrosion activity. Again, soil resistivity is only a measure of an electrical component of the corrosion cell. Therefore, measuring the soil resistivity is analogous to measuring the resistivity of the electrolyte in the system. Two soils with the same resistivity can have a very different corrosivity depending on other parameters. However, soil resistivity provides a reasonable

starting point to establish the corrosivity or potential corrosivity of a soil. General soil corrosivity levels based on soil resistivity levels have been established by many sources. For example, EPRI report, *A Literature Review of Soil-Side Corrosion Rates for Buried Metallic Piping* [6], presented the classifications as shown in Table 2-1 for carbon steel.

Table 2-1
Soil resistivity classifications for carbon steel

Resistivity [Ω -cm]	General Classification Category
< 500	Extremely Corrosive
500 - 1,000	Very Corrosive
1,000 - 10,000	Corrosive
10,000 - 20,000	Moderately corrosive
> 20,000	Progressively less corrosive to negligible

Soil resistivity also affects the performance of the CP system. The soil resistivity directly effects how the current used in these systems moves through the soil. Therefore, there are significant performance and design implications of CP systems based on soil resistivity. The soil resistivity will also affect the interpretation of aboveground survey data that are collected from buried piping. Soil resistivity will change based on the moisture content of the soil and the concentration of ions in the soil at the time of testing. Resistivity changes with moisture content, as soluble ionic species become dissolved in the electrolyte when soils are water-wet. It is necessary to take soil resistivity readings multiple times throughout the year to determine what range of resistivities the metallic structures in contact with the soil experience [10]. A supplementary data set can also be collected in the laboratory where the soil moisture content can be carefully controlled, adjusted, and measured. In this instance, laboratory adjustments must be carefully controlled by using deionized water that will not introduce changes to the soil ionic content.

2.2.2 Degree of Aeration

2.2.2.1 Oxygen Levels

The oxygen concentration in soil affects the corrosivity of the soil in two ways. The two important ways that oxygen can affect steel corrosion in soil are the amount of dissolved oxygen in the soil electrolyte and the oxygen distribution throughout the soil. Oxygen itself acts as an oxidizer to drive the corrosion process. The oxygen is an important cathodic reactant in the electrochemical reaction. Most typically the cathodic reaction (that is, the reduction of dissolved oxygen) is the rate-limiting reaction. Therefore a change in the oxygen concentration of the soil can have a significant effect on the overall corrosion rate.

A differential aeration cell can also develop within the soil. This cell is created due to areas with different oxygen concentrations across an electrically continuous metallic structure. Areas of lower oxygen concentration will become more anodic than areas of higher oxygen concentration. This results in a potential difference which causes corrosion to occur at the more anodic area. The cell can occur in soils where oxygen concentrations usually decrease with increasing depth,

at locations above and below the water table, or areas underneath paved roads or structures. For soils underneath paved roads or structures, the oxygen concentration underneath these structures is typically lower than the oxygen concentration in the adjacent soil. This results in corrosion of the structure in the oxygen-depleted area [10, 14].

The aeration characteristics of a soil are dependent upon physical characteristics such as the particle size, particle size distribution, and unit weight. In high-plastic clays, a reduction in moisture content results in cracks that provide effective channels for oxygen in the air to reach buried metal. Disturbed soils, such as backfill which is not properly compacted, can result in oxygen being more readily available initially. Over-compaction of backfill can have an opposite effect causing a lower oxygen concentration. In some instances, atmospheric oxygen can become trapped in isolated pockets or cells creating the potential for localized anodic regions [20].

2.2.2.2 Redox Potential

The reduction-oxidation potential of an electrolyte, or Redox potential, is a measure of the ability of electrochemical reactions to acquire or donate electrons. Redox potentials are often measured using a platinum substrate and a reference electrode, such as a silver-silver chloride reference cell or saturated calomel reference cell. The redox potential is simply the corrosion potential of platinum in a given environment. Platinum is a catalytic material with a very high exchange current density, so reduction-oxidation reactions occur freely on the surface of platinum without metal loss, because platinum is a noble metal.

In the underground metallic corrosion cell, soil contains reducible species. In the case of soils it is essentially a measure of the degree of aeration of the soil. This is due to the fact that oxygen is typically the strongest electron acceptor in soil. A high redox potential indicates a high oxygen concentration. A low redox potential indicates a low oxygen concentration. Low redox potentials (<100 mV) indicate conditions which are conducive to anaerobic MIC activity. Redox potential can be difficult to measure as disturbed soil will have increased oxygen levels and redox potentials will typically remain unstable for a period of time [21, 22]. Ions of manganese, iron, and sulfur can have a significant impact of the redox potential of electrolytes.

2.2.3 Chemical Constituents

2.2.3.1 Chlorides and Halogens

Chlorides and other halogens such as bromide, iodide, and fluoride affect the corrosivity of soil in multiple ways. Chloride is the most commonly encountered halogen ion in soil environments. Halogens prevent the formation of a passive film on metal surfaces. Halogens can contribute to general corrosion, pitting corrosion, and SCC in a variety of materials. They also increase the soluble ion content of the soil, thus reducing the soil resistivity [23]. Chlorides and bromides can be found naturally in soils from brackish water and from geological sea beds. Chlorides can also be artificially added to the soil from external sources such as de-icing salts from roadways or contamination from local plant process streams. Table 2-2 presents the relative corrosivity of soils based on the chloride ion content for carbon steel.

Table 2-2
Chloride content effect on soil corrosivity for carbon steel

Chloride Concentration [parts per million (ppm)]	Relative Corrosivity
> 5,000	Severe
> 1,500 - 5,000	Considerable
> 500 - 1,500	Corrosive
< 500	Threshold

2.2.3.2 Sulfates/Sulfides

Sulfates do not actively contribute to the corrosion of buried metallic structures. Sulfates instead act as nutrients for SRB. These bacteria reduce the sulfates into sulfides, which can be highly corrosive [24]. Sulfate testing alone does not solely indicate that MIC is occurring. Other testing, such as MIC culture testing, must be completed in conjunction with sulfate testing to determine the influence of MIC on the buried metallic structure. Sulfates can degrade concrete structures, as well [22]. Table 2-3 presents the relative corrosivity of soils based on the sulfate ion content for carbon steel.

Table 2-3
Sulfate content effect on soil corrosivity for carbon steel

Sulfate Concentration [ppm]	Relative Corrosivity
> 10,000	Severe
>1,500 -10,000	Considerable
> 150- 1,500	Positive
0-150	Negligible

2.2.3.3 Other Ions

There are other soluble cations and anions that should be measured when examining soil corrosivity. These other soluble ions contribute to the corrosivity of the soil by creating compounds that can either raise or lower the pH [25], can contribute to the current carrying capacity of the soil, can contribute to the formation of protective products, and can participate in the electrochemical reaction at the metal surface [23]. The presence of one ion versus another does not necessarily mean that a soil is more corrosive versus another; however, the presence of different soluble ions can assist in better understanding parameters such as soil resistivity and pH. Also, different protective products can form on pipe surfaces, depending on the different soluble ions present in the soil.

Soluble ions that are relevant to soil corrosivity are sodium, potassium, magnesium, calcium, carbonates, bicarbonates, nitrites, and nitrates. The cations listed tend to increase the pH in a system by forming basic compounds [25]. The anions tend to lower the pH by forming acidic compounds. However, this is not always the case. Ions such as calcium, magnesium, and carbonates commonly contribute to the formation of CP products that aid in preventing

corrosion activity. Carbonates and hydroxide ions are film-forming compounds on steel. Some ions, such as nitrates, can also assist in mitigating corrosion by competing with chlorides for sites on the passive film barrier, and prevent the breakdown of the barrier [11]. The presence of a large amount of ionic compounds in a solution will increase the current carrying capacity of the soil and thus decrease the resistivity. It is difficult to draw conclusions by the presence of one or more of the soluble ions listed in this section. However when coupled with other soil parameters, understanding what ions are present will aid in understanding the observed corrosion behavior of the associated buried metallic structure.

2.2.4 Acidity and Alkalinity

The pH of a soil is a measure of the acidity or alkalinity of a soil. The pH is the negative logarithm of the hydrogen ion concentration; the lower the pH the higher the hydrogen ion concentration. The concentration of hydrogen ions will affect the rate of the chemical reactions at the anodic and cathodic areas of the metal surface thereby changing the rate of corrosion. For buried carbon steels and stainless steels, acidic environments will increase the rate of corrosion. Alkaline environments are less corrosive for steel [19]. Aluminum and other amphoteric metals experience an increased corrosion rate at both high and low pH values as seen in Figure 2-2. The pH of the soil has an effect on the CP system as well. Acidic environments have a depolarizing effect on buried structures [26]. This increases the current demands of the CP system to effectively mitigate corrosion activity.

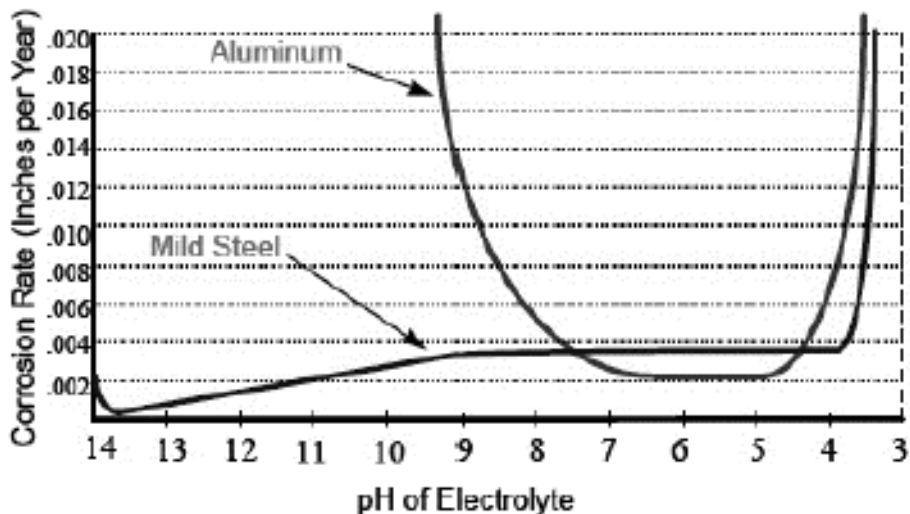


Figure 2-2
Effect of pH on the corrosion of aluminum and steel⁵

Total acidity is also a relevant parameter for determining the corrosivity of soil. Total acidity is a measure of the quantity of ionizable hydrogen in a sample, whereas pH is the concentration of H^+ ions in solution. It represents the amount of ionizable hydrogen available in a sample that can participate in an acid reaction [27]. Due to the low dissociation of most acids in soils, the pH value may not represent the potential effect of acidic compounds on corrosion activity [19]. In some cases the total acidity may provide a more relevant measure of the corrosivity of a soil as a result of the acidic species present.

2.2.5 Moisture Content

The water in the soil increases the ability of the soil to behave as the electrolyte of the corrosion cell. The water is a carrier for the soluble ions and corrosive species. As moisture content increases soil resistivity will typically decrease, because electrical current can be carried more easily through the soil. The decrease in soil resistivity will further increase the potential corrosivity of the soil. The moisture content of soils fluctuates with seasonal, geographical, and other environmental changes. A change in the groundwater level for any number of reasons will change the soil moisture content. For example, rainfall will cause a temporary increase in the moisture content. Cold weather may cause some of the water in the ground to freeze reducing the ability of ions to move freely through the solution. Due to these changing factors it is important to take into consideration the time of the year during which a soil sample is collected, and may make it necessary to collect multiple samples during different meteorological conditions. In general, fully saturated soils will provide the worst case conditions for soil corrosivity. However, there are exceptions. For example, the concentration of ionic species can increase greatly at a location that has experienced a loss of moisture, and this local increase in concentration can lead to increased corrosivity. Moisture content, as with other soil properties, will change with depth. Therefore it is important to take into consideration the depth of soil sample origin when correlating soil data to expected corrosion behavior.

2.2.6 Microbial Content

Microbiologically influenced corrosion is an aggressive form of corrosion caused by the interaction of microorganisms and metals. It does not occur as one form of corrosion, but rather can occur as one of many forms including accelerated pitting attack, crevice corrosion, differential aeration cells, metal concentration cells, selective de-alloying, under-deposit corrosion or enhanced galvanic corrosion. Even corrosion resistant alloys are not immune to MIC. The presence of microbes that commonly contribute to MIC does not mean that MIC is occurring, but it means that there is a potential for MIC to occur. When trying to identify whether or not MIC is occurring it is important to have a comparison between native soil and the area immediately adjacent to where suspected MIC activity or damage is occurring. This comparison should be completed at the time of examination, but historical data from a soils testing can also be used for comparison. Determining whether or not there is active MIC on a buried metallic structure is a complex process. All soils contain MIC bacteria. Only under the proper conditions will the bacteria result in MIC activity on the metal surface. Confirmation of MIC requires examination of the metal surface and positive identification of MIC microorganism at a location of metal loss. A soil testing program will focus on the examining for the potential for MIC activity. Soil testing can be used to find favorable environments for MIC and identify increased bacteria concentrations in the soil. Identifying areas where bacteria counts at the metal surface are greater than the surrounding area can provide evidence that a favorable environment for MIC has developed [13].

Microbiologically influenced corrosion can be caused by many different types of microorganisms. These microorganisms can either be anaerobic or aerobic. Anaerobic bacteria include SRB, some APB, MOB, and metal-reducing bacteria (MRB). Aerobic bacteria include some APB, some MRB, and exopolysaccharide (EPS) producing bacteria. Several different types

of fungi can also contribute to MIC. Overall, SRB and APB have been attributed to the majority of failures related to MIC. Sulfate-reducing bacteria are the most common contributor to MIC activity in un-aerated, anaerobic soils. Acid-producing bacteria are more common in sandy soils, and high clay soils provide a favorable environment for both SRB and APB.

All of these different bacteria groups can also exist as a consortia leading to complex interactions and formation of biofilms. Microbiologically influenced corrosion bacteria can contribute to corrosion in many different ways. The bacteria can directly contribute to corrosion activity by producing corrosive species such as sulfides, organic acids, or carbon dioxide. They also can create oxygen barriers that create differential aeration cells on the metal surface that contribute to corrosion. Due to the complex interaction of the MIC bacteria and the different environments that can be produced, the same bacteria phenotype may cause different damage on the same material. For example, APB will produce different acidic compounds based on whether they are in an anaerobic or aerobic environment. The same bacteria phenotype may also cause different damage on different materials. For example, MIC due to SRB can be observed as pitting on carbon steel, yet it can cause graphitization in cast iron.

2.2.7 Soil Classification and Particle Size Distribution

Soils can be classified based on composition. Soils are typically made of up to three main mineral particle types – sand, silt, and clay. These are defined by particle size. The United States Department of Agriculture (USDA) soil classification system classifies soil particles according to size (see Table 2-4 [28]).

Table 2-4
Soil particle USDA classification

Particle Classification	Particle Size [mm]
Very Coarse Sand	2.0 - 1.0
Coarse Sand	< 1.0 - 0.5
Medium Sand	< 0.5 - 0.25
Fine Sand	< 0.25 - 0.10
Very Fine Sand	< 0.10 - 0.05
Silt	< 0.05 - 0.002
Clay	< 0.002

Soils are then classified based on the mixture of these different particle sizes. Figure 2-3 shows how soils are classified based on relative percentages of sand, silt, and clay.

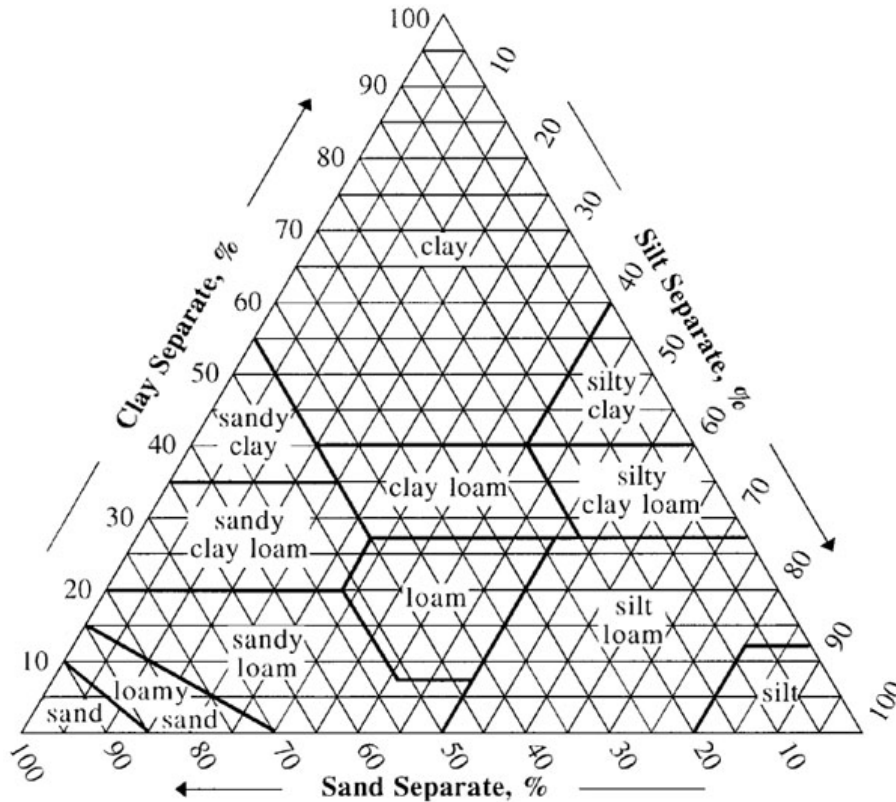


Figure 2-3
USDA soil composition diagram [5]

The corrosivity of a soil cannot be determined solely based on its classification or particle size distribution; however, important information related to potential for corrosion activity can be gathered. Soil classifications provide insight into how well moisture, nutrients, air, and electrical currents move through the soil, which in turn determines how conducive a soil is to corrosion activity. These parameters also determine how conducive a soil is to microbiological growth. In general, finer-particle clays and loam (see Figure 2-3) soils are more corrosive. This is due to the ability of these soils to retain moisture, nutrients, and chemical constituents more easily than sandy soils. Soils that are able to retain moisture and ionic species generally have a lower resistivity. Soils with finer particles, such as clays, are able to hold more moisture due to the increased surface area-to-volume ratios of the particles and its crystalline structure allows for a high degree of water absorption [29]. As stated previously, APB is more common in sandy soils, and soils with high clay content provide a favorable environment for both SRB and APB.

The Unified Soil Classification System (USCS) is also used to classify soils for engineering and design purposes. The soils are classified based on particle-size characteristics, liquid limits, and plastic limits [30].

2.2.8 Mechanical Properties

The mechanical properties of soil influence the corrosivity of a given soil in two ways. Some mechanical properties of the soil influence the movement of ions, gases, and liquids through the soil. The hydraulic conductivity of a soil refers to the ability of liquids, mainly water in this case, to move through the soil. The density and compaction of the soil also influence how well currents and ionic species move through the soil. Second, the mechanical properties of the soil aid in determining the likelihood of coating damage. If the soil is prone to expansion or contraction or behaves as a rigid solid, coating damage is more likely due to soil stresses. Coating damage then allows corrosion activity to occur under the appropriate conditions, especially in cases where the disbonded coating shields the pipe from cathodic protection. Coating damage can be accelerated when low compaction and density at the pipe surface can allow loose stone/gravel to migrate through the soil and scratch and penetrate the coating.

The hydraulic conductivity of a soil depends mainly on the size, shape, and distribution of the pores within the soil [31]. Insight about these values can be gained by examining the particle size distribution and bulk density of the soil. Typical ranges of saturated hydraulic conductivity are presented in Table 2-5.

Table 2-5
Soil texture effect on hydraulic conductivity

Soil Texture	Hydraulic Conductivity (K) [m/day]
Gravelly coarse sand	10 - 50
Medium sand	1 - 5
Sandy loam, fine sand	1 - 3
Loam, well-structured clay loam and clay	0.5 - 2
Very fine sandy loam	0.2 - 0.5
Poorly structured clay loam and clay	0.002 - 0.2
Dense clay (no cracks, no pores)	< 0.002

Loam is soil that is made up of sand, silt, and clay. These three constituents can exist in different proportions resulting in different loams, such as clay loam or sandy loam, see Figure 2-3. The differing proportions change the structure and behavior of the loam soils. For example, a sandy loam will have larger average particles, than loam.

In general the smaller the particle size, the smaller the pore size, the lower the hydraulic conductivity. That means the more coarse sandy soils will have a higher hydraulic conductivity than the fine particle clay soils.

The bulk density of a soil can be used to determine the porosity and therefore the potential degree of aeration of a soil. Measuring the bulk density at different depths can provide an indication of whether or not differential aeration cells can form due to differences in soil layers. The bulk density can be measured directly from core samples or measured from re-compacted samples. Re-compacted soil samples can provide an indication of in-situ material properties; however, the condition tested does not exactly match the true environmental condition around the buried metallic structure. Therefore it is preferable to use an as-found sample.

3

MATERIALS OF CONSTRUCTION FOR BURIED PIPING AND TANKS AT NUCLEAR POWER PLANTS

3.1 Materials Used for Piping

There are several different materials that are commonly used in NPPs for buried metallic structures. The materials of construction that are used can depend on the service and economics of construction. The most common material of construction for buried metallic structures in NPPs is carbon steel or coated carbon steel. Several other materials that are commonly used include cast iron, ductile iron, stainless steels, copper alloys, and aluminum alloys.

3.1.1 Carbon Steel

Carbon steel has historically been the material of choice for buried piping and tanks. Carbon steel is susceptible to corrosion in soil if not properly isolated or protected. Carbon steel can be protected from corrosion in soil by using coating systems and/or CP systems. Coating systems work to isolate the material and CP systems drive the exposed steel of the buried structure to be more cathodic in the environment. Leaks have been reported on carbon steel systems at NPPs and the leaks were normally associated with coating failures and corrosion of the steel substrate [9]. Carbon steel can also be galvanized by applying a layer of zinc to the carbon steel. The zinc acts as a sacrificial anode to the carbon steel. The zinc is usually applied by dipping the carbon steel in molten zinc.

3.1.2 Stainless Steel

Stainless steel has the potential to offer increased resistance to corrosion in soil compared to carbon steel. Some stainless steels are able to form a more robust passive film that offers increased environmental resistance [32]. This applies to the base material, yet may not include welds and heat affected zones where alterations to the metal microstructure alter the susceptibility to corrosion. Austenitic stainless steels (300 series stainless steels) can be susceptible to corrosion due to the presence of chlorides. Austenitic stainless steels are typically not well suited for anaerobic or low pH soils. Stainless steels form a robust passive film due to the presence of chromium, and in some cases molybdenum, in the alloy whereas the iron oxide film on carbon steels is not a passive film. However, the passive film on stainless steels can be broken down by aggressive species and under crevices. This leads to localized attack. Austenitic stainless steels are also susceptible to MIC in certain environments.

Super austenitic stainless steels are also used in NPPs. Super austenitic stainless steels have high molybdenum content (less than six percent) as well as increased nickel and nitrogen levels compared to 300-series stainless steels. The increased levels of chromium, molybdenum, nitrogen, and copper increase the pitting resistance of the material. Super austenitic stainless steels offer higher corrosion resistance to general, local, and MIC attacks. Many are also suitable for chloride environments approaching 7,500 ppm. These materials are resistant to corrosion in soil.

3.1.3 Aluminum Alloys

Aluminum is a common material for condensate systems. It is commonly used in systems where low iron ion content is a concern. Aluminum forms a passive film in aerated, aqueous environments. It resists general corrosion in soil due to the tough aluminum oxide layer that forms. Some soils, however, are corrosive to aluminum. These soils are non-draining, anaerobic (lack oxygen), and consists of clay-sized particles [33]. Aluminum is an amphoteric material meaning that it corrodes in both low and high pH environments. This must also be considered when using aluminum especially in relation to the use of CP. Cathodic protection, especially in overprotection conditions, can result in an alkaline environment forming around the protected metal. This can lead to corrosion of aluminum when overprotected [9]. Aluminum can also be subject to attack by chlorides and de-alloying. Leaks have been reported in buried aluminum piping systems, such as aluminum bronze systems.

3.1.4 Copper Alloys

Copper alloys are used in a limited capacity at NPPs. Similar to aluminum alloys, selection of copper alloys is often based on the requirements of the intended service. Copper alloys can offer some increased corrosion resistance compared to carbon steel in soils [34]. Copper is susceptible to corrosion due to high sulfide concentrations. Dezincification can also occur in copper alloys with a zinc content greater than 15 percent.

3.1.5 Cast Iron

Cast iron has been used historically for service water and drainage piping. Cast iron refers to one of several different types of alloys comprised mainly of iron, carbon, and silicon. The carbon content is higher than that of steels. Cast iron is more brittle than steel. Leaks have been reported in cast iron piping. Degradation of cast iron piping is commonly in the form of general corrosion, pitting, galvanic corrosion, MIC, or graphite corrosion. Graphite corrosion is the result in the selective attack of the iron-rich phases in the material by the environment, which leaves behind a brittle graphite matrix.

3.1.6 Ductile Iron

Ductile iron has been used in many of the same applications as cast iron historically. Ductile iron differs from other cast irons in that it has a much higher fracture toughness, and is therefore much less brittle. Ductile iron has spheroidal or nodular graphite inclusions, which gives it more fracture resistance. The corrosion behavior of ductile iron is very similar to that of cast iron.

3.1.7 Metal Reinforced Concrete

Metal reinforced concrete covers a wide variety of designs, however the two most common are prestressed concrete cylinder pipe (PCCP) and reinforced concrete pipe (RCP). Prestressed concrete cylinder pipe is a composite of concrete and a steel cylinder along with prestressed wires that put the concrete in compression. Reinforced concrete pipe can either have steel reinforcing bars or an embedded steel cylinder, or both. These types of piping are common for cooling water systems in NPPs. Degradation mechanisms for these types of piping can attack both the metal matrix and the concrete. Corrosive environments for this type of piping includes high sulfate (greater than 2,000 ppm), high chloride (greater than 400 ppm), and acidic soils (pH less than 5) [5]. The chlorides are a greater concern for the steel matrix and the sulfates result in the breakdown of most types of concrete.

3.1.8 Asbestos-Cement

Historically, asbestos-cement was used in water and drainage systems. Asbestos was mixed with the cement to increase the tensile strength of the concrete. This material is no longer installed due to the recognized health concerns of asbestos, however it still is in use from previous installations. Similar to the degradation mechanism of the concrete portions of PCCP, asbestos-cement can be subject to degradation in high sulfate and acidic environments.

3.2 Use of Coatings in Buried Service

As stated previously, metals will undergo corrosion if exposed to soil. As a result, most buried metallic structures are coated with an organic coating or coating system. The EPRI “Balance of Plant Corrosion-The Underground Piping and Tank Reference Guide” [5] provides a more detailed discussion of the different types of coatings used for buried metallic piping. Coatings are selected primarily based on the ability of the coating to isolate the buried metallic pipe surface from the environment, the geometry of the structure, and maintenance requirements. Coatings must be able to stay well adhered to the pipe surface, allow CP currents to flow through them when disbonded, handle soil stresses, and be resistant to mechanical damage to be effective.

An isolated location where a hole develops in the coating and the metal substrate is exposed to the soil is known as a holiday. Coating holidays should be expected during the operating life of the buried metallic structure. Protection of metal at coating holidays is accomplished with proper application of CP.

Coatings degrade over time and also interact in positive and negative ways with the CP system. Fusion Bonded Epoxy (FBE) has shown to have superior resistance to degradation and cathodic disbondment compared to coal tar coatings in many environments. A negative consequence of coating degradation is a phenomenon known as CP shielding. Shielding occurs when CP current is unable to reach areas underneath disbonded coating. This results in the formation of anodic regions in the shielded areas and can lead to corrosion activity. Fusion Bonded Epoxy coatings allow a small amount of CP current to pass through the coating and reach the metal substrate. This can reduce the effects of corrosion under areas of disbonded FBE coatings.

4

CATHODIC PROTECTION OF BURIED METALLIC STRUCTURES

In addition to this section, the EPRI report *Cathodic Protection Application and Maintenance Guide* [26] provides a more complete discussion on CP and monitoring a CP system.

Cathodic protection is used to minimize the degradation of buried metallic structures by making the structure the cathode in the corrosion cell. There are two main forms of CP, sacrificial and impressed current. Sacrificial anode CP uses anodes made of materials that are more anodic than the buried structure and makes them electrically continuous to the buried structure. Therefore, when the buried metallic structure is exposed to the environment, the sacrificial anode will corrode (hence the term “sacrificial”) and the buried structure will be protected from corrosion. Over time, the sacrificial anodes need to be replaced due to consumption. The common components of an impressed current CP system are a power source, a transformer, a rectifier (converts alternating current (AC) to direct current (DC)), and an anode bed. Direct current flows from the anode bed toward the buried metallic structure through the soil. This polarizes the buried metal to cathodic potentials. The performance of the CP system is dependent on the ability of the current to reach the buried structure and decrease its potential. Therefore, soil resistivity, soil composition, structure age, anode bed resistance to earth, coating condition, and other factors can have a measurable effect on the performance of the CP system. The performance of the CP system can be monitored by measuring pipe-to-soil potentials and rectifier outputs. The criteria for effective CP according to NACE SP0169 “Control of External Corrosion on Underground or Submerged Metallic Piping Systems” [35] are:

1. Pipe-to-soil potential of the steel buried in soil is -0.850 V CSE (Copper/Copper Sulfate) or more electronegative with the CP system on (taking IR drop into consideration).
2. Polarized potential of the steel buried in soil is -0.850 V CSE or more electronegative, also known as an instant off potential.
3. The structure is polarized more than 100 mV from its native corrosion potential.
4. Engineering evaluation supporting acceptability of other criteria based upon observed corrosion rates and performance requirements.

Additionally, recent drafts of industry guidance in LR-ISG-2015-01, “Changes to Buried and Underground Piping and Tank Recommendations” [36], has been released for public comment. The draft version of this document recommends less stringent criteria for higher resistivity soils. For soils with a resistivity between 10,000 ohm-cm to 100,000 ohm-cm a -750 V CSE is recommended. For soils with a resistivity greater than 100,000 ohm-cm a threshold of -650 V CSE is recommended. For copper and aluminum alloys the acceptance criteria for the CP will have a minimum of a 100 mV polarization.

The CP system output can be adjusted or modified when areas of buried metallic structures are found not to be meeting specific criteria. The CP system can cause certain products to form on the buried metallic structure surface. One common example is calcium carbonate. The CP system will change the pH of the electrolyte near the pipe surface and increase the pH due to the presence of cathodic reaction products. Soils testing can detect the presence of reactions products that commonly form due to CP by measuring an increased pH compared to native soil and increased concentrations of calcium or carbonates on or near the surface of the structure.

5

CHARACTERIZATION OF SOILS AT NUCLEAR POWER PLANTS

Soils at NPPs are mainly engineered backfill or flowable fill. The backfill can be repurposed from the original site location or nearby locations, or it may be foreign backfill. Engineered backfills have a controlled composition and size distribution. Flowable fill, also commonly referred to as controlled low strength material (CLSM), is flowable at the time of pouring and then hardens into a compact material with a much lower strength than concrete. Flowable fill has permeability similar to or less than that of compacted soil. It should be noted, however, that many NPPs used leftover structural concrete, unqualified mixtures, and other structural concrete as backfill. This material, while still cementitious, can have a much lower permeability and can create significant challenges in classification/corrosion performance.

Plant requirements are designed to ensure that the soils used have the proper engineering properties to suit the intended purpose. Backfills used for overall cutting/filling, structural fill for large structures, bedding materials for pipe trenches, hydraulic barriers in ponds/containments, and cosmetic applications will vary greatly. When defining soil characteristics ASTM⁴ D2487-11 “Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)” [37] is used to classify soils used for engineering purposes based on particle-size characteristics, compressibility, liquid limits, and plastic limits. Liquid and plastic limits are also known as Atterberg Limits and provide the level of moisture demarcating changes in solid, semi-solid, and liquid phases of soil. The required values will be unique to each plant design and geological conditions. A list has been compiled of the standards and codes that have historically and are currently used for soil characterization at NPPs. These standards and codes are presented in Table 5-1.

⁴ American Society of Testing and Materials International (ASTM)

Table 5-1
Standards used to characterizes soils used at NPPs

Standard Number	Standard Title
Standards used to determine plastic and liquid limits	
ASTM D4318-10	Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils [38]
Standards used to determine particle size distribution and density of soils	
ASTM D422-63 (2007)	Standard Test Method for Particle-Size Analysis of Soils [39]
ASTM D1556	Standard Test Method for Density and Unit Weight of Soil in Place by Sand-Cone Method [40]
ASTM C136-14	Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates [41]
ASTM D6913-04 (2009)	Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis [42]
ASTM D6938-10	Standard Test Method for In-Place Density and Water Content of Soil and Soil-Aggregate by Nuclear Methods (Shallow Depth) [43]
ASTM D2937-10	Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method [44]
Standards used to determine soil moisture content	
ASTM D2216-10	Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass [45]
ASTM D4643-08	Standard Test Method for Determination of Water (Moisture) Content of Soil by Microwave Oven Heating [46]
Standards used to characterize flowable fills used at NPPs	
ASTM D5971-13	Standard Practice for Sampling Freshly Mixed Controlled Low-Strength Material [47]
ASTM D6023-07	Standard Test Method for Density (Unit Weight), Yield, Cement Content, and Air Content (Gravimetric) of Controlled Low-Strength Material (CLSM) [48]
ASTM D6024-07	Standard Test Method for Ball Drop on Controlled Low Strength Material (CLSM) to Determine Suitability for Load Application [49]

Also, industry documents provide guidance on the required backfill requirements. The GALL report [2] provides guidance on acceptable backfill. The GALL indicates Aging Management Programs (AMPs), and GALL AMP XI.M41 states that backfill is to be in accordance with NACE SP0169 (2013) [35].

5.1 Special Considerations for Nuclear Power Plants

Piping systems backfilled using flowable fill typically experience lower corrosion rates and may be more difficult to excavate than piping systems backfilled using engineered backfill. As a result, piping systems that are backfilled using engineered backfill should generally be given a higher inspection priority than comparable systems that are completely backfilled using CLSM, as per the GALL Report [2].

Nuclear power plants have more complex underground systems than compared to transmission pipelines or sewer systems. Nuclear power plants have underground piping that extends to deeper depths than many industries (up to 50 ft). Nuclear power plants also have piping in close proximity to one another. This can have an influence on the soil characteristics and can also make it difficult to collect soil data as drilling or digging can be unsafe due to the piping being in close proximity. It may also complicate CP of the piping due to effects such as shielding.

Failures have been reported due to inadequate backfill at NPPs. If historical records do not exist confirming the quality of the backfill material used, it is recommended to perform soils analysis to assist in soil characterization.

6

SELECTING SAMPLING LOCATIONS

6.1 Soil Sampling Locations

The intention of most soil sampling activities is to determine the composition of the soil that is or will be in contact with buried structures. The number and location of soil samples to be collected is based on the variability of the soil, accessibility of sampling locations, and the intended use of the information. Soil sampling can either be done using a statistical approach or a knowledge based approach. A statistical approach selects sites either systematically or randomly. A knowledge based approach selects specific locations based on known soil conditions, the locations of buried structures, and areas of known susceptibility. Use of random sampling is better suited for understanding large areas of soil that are generally uniform or where variability is limited. Soil sampling, for the purposes of corrosion monitoring, should be focused on the local conditions near buried structures. In most cases, underground piping at an NPP covers only a small portion of the total land area at a facility. Therefore a knowledge based approach is recommended where specific locations are chosen along buried structures. Corrosion engineering methods, such as indirect piping inspections, may also help guide the decision making process when selecting soil sampling locations.



Key Technical Point

A knowledge based approach to selecting sampling locations is recommended when possible. This method combines background information such as site drawings and materials of construction with corrosion susceptibility to select areas that are likely to have corrosion issues.

A sampling plan should be developed to determine where samples should be collected. These locations should be selected such that both representative and worst case conditions are included. There are many factors to consider when selecting a location to collect samples. These include:

- Pipe segments identified as having a high likelihood of failure or locations where external corrosion has been previously reported
- Locations of known changes in soil composition
- Locations where local surface conditions may change:
 - Areas underneath roads, parking areas, and buildings may have different conditions than uncovered areas
 - Areas where leaching or runoff occur such as roadways or storage areas

- Salt treatment of roads and parking lots can lead to increased chloride concentrations due to runoff
- Areas of poor or excessive drainage
- Seasonal variations in soil conditions
- Areas where plant records do not include the type of backfill used, or where the accuracy of plant records is uncertain
- Areas of concern based on cathodic protection surveys
- Areas that are known to have been previously exposed (that is, were uncovered)
- Areas where ground water fluctuations have been observed
- Areas where contamination is possible or expected

When collecting the sample the following should be considered:

- Soil samples should be collected at the location most representative of the local soil conditions near the underground pipe or tank.
- Samples should be collected at the same depth as the buried structure.
- Samples should be a representative of the backfill nearest to an area of corrosion on the buried structure.
- Samples of undisturbed native soil can also be collected and they should be collected from approximately the same depth as the structures of interest.
- Care should be taken to not damage buried piping or other buried structures when collecting soil samples [1].

Soil sample collection should be performed any time an excavation is performed that exposes buried structures. The soil sample should not be obtained from the spoils pile. If the excavation was performed due to the presence of external corrosion or a coating holiday, one sample should be collected at the area of corrosion or coating damage. A second sample should be collected at an area on the pipe or tank that does not have corrosion or coating damage, preferably at either end of the excavation.



Key Technical Point

When collecting samples from excavations, it is necessary to scrape away several inches of soil prior to collecting the sample. The edges of the excavation will have spent time exposed to the air prior to sampling.

When sampling from areas where the piping is still buried, locations may be selected by first performing soil resistivity measurements every 100 feet. Soil samples should be collected from locations where significant changes in soil resistivity are observed. The soil sample should be collected at the same depth as and within ten horizontal feet of the buried piping and within the backfill.

Obtaining soil samples representative of the environment in contact with buried structures also requires consideration of the timing of sample collection. Outside temperature changes, precipitation, and other seasonal variations occur over time and they can affect the corrosive properties of soil. The variation in temperature and rainfall will also vary not just with time but with geographic location. Sample collection should be performed in a manner that provides a complete picture of the range of conditions that are likely and potentially to be encountered, as described in this document.



Key Technical Point

Sampling is sometimes completed as a result of maintenance excavations, drilling, or other subsurface operations.

6.2 Labeling Sampling Locations

Soil sampling locations should be identified graphically on site drawings/plans, and also at the physical sampling location. In the case of soil samples collected during excavations, graphical identification of the location should suffice. There should be enough information about the site to allow an individual with no knowledge of previous sample collections to go and find the sampling locations. The following documentation should be made of each sampling location:

- Site identification
- Specific location, global positioning system (GPS) or site coordinates
- Date
- Location marked on a site drawing
- Photographs of the dig location and nearby aboveground features

The physical location can be marked if repeated testing is to be completed at a site. Repeated sampling allows for a comparison of the soil properties over time, which can be used to better predict the corrosion behavior of the buried asset in the soil, or can validate or discredit assumptions made in corrosion predications. Survey stakes can be used, however this is not recommended as soil sampling is typically completed at intervals of several months at a minimum. Survey stakes may not last that long and can be moved or knocked over, especially in congested areas. It is recommended to use a more robust identifier such as a wood or metal post that is either tagged with the relevant soil data, or has a placard. If using a post, ensure the post meets site requirements for coloring, marking, protection, and so on. Relevant data includes site identification and sample depth. Modern radio frequency identification (RFID) tags may also be an option for local data storage and site marking.

Soil samples can be obtained from test wells being drilled for the ground water sampling such as that required in NEI Groundwater Initiative, NEI 07-07 [50]. This should be coordinated with station personnel involved in the ground water program. Care should be taken when sampling from these locations as samples may be contaminated, and may not be reflective of the properties

from locations where piping has been buried. Data correlation can be completed using the data collected as part of an NEI 07-07 program to help identify which chemical constituents exist in the soil and ground water. The chemical constituents measured in the groundwater sampling and a soil testing program should be compared.

6.3 Locating Underground Structures

This section outlines a few common techniques for locating underground structures. Additional guidance is provided in the EPRI report *Balance of Plant Corrosion-The Underground Piping and Tank Reference Guide: Revision 1* [5]. The first step to locating underground structures should consist of a review of the most recently updated plant drawings. This can provide a starting point to locate buried structures. These drawings should also be reviewed with any vendor completing locating or excavating activities.



Key Technical Point

Locating underground structures accurately is essential to the soil sampling process. Conclusions drawn from a particular soil sample must be taken in the context of where it was collected in relation to the underground structure.

6.3.1 Ground Penetrating Radar

Ground penetrating radar (GPR) utilizes waves of energy to detect underground structures. The frequency of the waves range between radio waves and microwaves. The waves penetrate the soil and are reflected by objects in the soil. Most GPR equipment consists of a single component on wheels that is either pulled or pushed. The equipment can operate at different frequencies. They are selected based on the intended target, the depth of the target, and the electrical properties of the soil. The electrical conductivity of the soil affects the ability of the GPR to penetrate the soil, the higher the conductivity of the soil, the lower the penetration ability of GPR. For most utility applications, frequencies range from 400 MHz to 500 MHz, yet can go as low as 200 MHz. Lower frequencies are better suited to penetrate to greater depths. However, there is a lower resolution for lower frequency waves, so the intended target must be larger. In this case the diameter of the pipe must be large enough to detect with the wave frequency selected. Service providers will be able to recommend the proper frequency based on the expected depth and diameter of the buried metallic structure in question [51].

6.3.2 Electromagnetic Line Location

Electromagnetic line location (ELL) has been used in the pipeline and other utility industries for decades. The method utilizes a transmitted or induced electromagnetic signal applied to a target line and then a locator is used to detect the electromagnetic field being generated by the target pipe. Line locators can be very basic and only provide an audible sound pitch when the locator is moved over a line. They can also be very complex providing depth, current magnitude, current direction, and pipe orientation. Locators can be adjusted to operate at different frequencies based on the application. This method also has limitations, especially in facilities where many objects are electrically continuous. If there are many electrically continuous objects in one location, each will have its own electromagnetic field and the locator will have difficulty detecting the peaks

and nulls in the electromagnetic signals that are required to detect the piping. This method can be completed by directly connecting to a buried pipe to apply the signal or can be applied indirectly by placing the transmitter over the buried pipe. The latter method induces a current on the buried pipe.

Another related method to ELL, is the use of acoustic waves to detect a buried structure. This method works in a similar way where a sound is transmitted through a pipe and a detector will detect the sound of the buried structure.

6.3.3 Probing

Probing is the most basic form of line locating buried piping. Probing utilizes a metal or fiberglass rod that is pushed into the ground in an effort to make a physical connection with the pipe. The probing depth is limited by the length of the probe bar; therefore this technique is only practical for piping that is shallower than 10-12 feet. Care must be taken when using a probe bar as over exertion with the probe rod can cause damage to coatings or piping. In addition, there are many possible errors with the method, and unless it is used for crude locating, multiple measurements must be made in the same area to confirm the location of the buried metallic structure.

7

PROCEDURES FOR SOIL TESTING

7.1 Sampling Methods

7.1.1 Excavation

Excavations provide an excellent opportunity to collect soil samples for corrosion testing, whether or not the excavation is related to a corrosion assessment. Soil samples are typically collected by hand during excavations. It is recommended to collect the soil sample using an uncontaminated shovel made of stainless steel or plastic, or using a hand auger if excavation geometry permits. It is not recommended to use chrome plated equipment. The chrome can contaminate the sample being collected. Chrome plating is common especially for trowels and smaller shovels. The sample should be collected at the same depth as the pipe and from the backfill. A few inches of soil should be removed and discarded from the excavation wall at the given location and sampling should be completed at the area behind the discarded soil. The soil nearer to the wall likely spent considerable time exposed to atmosphere. Therefore it is necessary to core or hand excavate further after the overall excavation process has been completed. It is important to ensure that the sample is collected in a safe manner and that the sample collection does not negatively impact the integrity of the excavation wall, and ensure that sample is collected in a manner that will not subject the collector to potential collapse [52]. If an excavation requires shoring, the sample can be collected from the excavation floor at the depth of the pipe. In this case rather than waiting until the entire pipe is exposed, the excavation can be interrupted as soon as the pipe depth is reached. The shoring can then be put into place and the sample can be collected within the excavation floor rather than the wall.

It is not recommended to collect samples from excavations using hydro-lancing. This process changes the soil moisture and adds foreign water to the soil. If this cannot be avoided, it is recommended to collect a sample of the water used for the lancing. This will provide a baseline of concentration for the chemical constituents which have been added to the soil during excavation.

If collecting samples as part of a corrosion assessment, a sample should be collected from the excavation wall as described above. This sample will be used as a comparison to the sample taken at the anomaly under investigation. A second sample should be collected at the anomaly under investigation [14]. Again, this sample is typically collected by hand. The goal is to remove the soil as close as possible to the pipe without causing damage to the pipe surface. Clean, sterile equipment, including gloves, tools, and storage containers should be used when practical.

7.1.2 Auger/Drilling/Coring

If using an auger, ensure that the sample is taken close to the pipe in order to collect a backfill sample, however not so close that contact could be made with the pipe. Considerations should be made to ensure that no contamination of the soil sample occurs from drilling or using an auger [53].

A hand auger or power driven auger may be used to collect soil samples. A hand auger can be used for piping that is closer to the surface. A hand auger system will typically consist of an auger, “T” handle, extension rods, and a thin-wall tube sampler. An acetate sleeve should also be inserted prior to removal to preserve the soil sample. With this method a hole can be made to the desired depth using an auger and then a thin-walled tube sampler can be used to extract a sample from the desired depth. Bucket augers are recommended for soil sampling, as they can collect a larger volume of soil quickly. Shelby-tubes and split spoon samplers can also be used depending on the allowable level of disruption to the soil. The core produced from this method can be placed into a recommended soil container and homogenized. However, the top inch of the core should be discarded. This discarded soil will typically not be soil collected from the area of concern. Posthole augers are not recommended for sample collections as it is difficult to differentiate soil removed from different depths.

Drilling utilizes a drill core, handle, and an acetate core. For shallow depths, a drive hammer can be used, or for soft soils a “T” handle can be utilized. Once the desired depth has been reached, the sample should be sheared off by rotating the sampler. The soil sample can then be extracted. Even hydraulic drilling is usually limited to partially weathered rock. Sites with near-surface bedrock may experience auger refusal when attempting to recover a soil sample. Dewatering may be necessary to complete the assessment. Groundwater from other depth levels has the potential to contaminate the sample. Figure 7-1 shows an example of a drilling operation for soil sampling.



Figure 7-1
Soil testing using a drill rig [54]

7.1.3 Hand Digging

Hand dug excavations can also be completed solely for the purpose of soil sample collection. While excavating, soil from shallower depths may fall to the bottom of the excavation. Care should be taken to remove any soil that is not from the depth of concern. Again, it is recommended that a stainless steel or plastic shovel be used for the sample collection.

7.2 Handling of the Sample

The amount of soil to be collected should be determined prior to collecting the sample. Check with the laboratory where the sample(s) is being sent to determine how much soil will be needed to complete the desired tests. It is important to note that these amounts, for some tests, do not include rocks or gravel in the soil, so extra material may need to be collected for rocky soils. Rock and gravel should not be excluded during sample collection because they are often characterized during physical soil characterization tests, such as sieving tests. Rocks and gravel can have an impact on the soil corrosivity. The amount of organic matter in the sample should also be minimized. Any large roots and grass should be removed. If any insects are noticed in the sample, they should be removed.

Handling of soil samples should be kept at a minimum and be completed as quickly as possible to minimize moisture loss and air exposure. It is recommended to wear rubber, latex (non-powdered), or nitrile gloves while collecting samples. Wearing gloves also minimizes tool contamination. Fresh gloves are recommended for each step of the process, or when handling different samples.

7.3 Packaging and Shipping

After collection, samples should immediately be placed in a sealable plastic bag or rigid plastic container. It is recommended to use a gallon-sized freezer bag, and to press out all excess air from the sample. Then double bag the sample using another freezer bag. This minimizes moisture loss and exposure in the event that the original bag is compromised. If more than one gallon of soil is required for testing, it is recommended to use multiple bags. Label the bags as part of a series collected at a single location. For, example, label the bags Sample 01-1, Sample 01-2, and so on in coordination with the sample labeling/logging. If using a rigid plastic container, fill the container completely with soil to avoid unnecessary air exposure. Seal the top of the container with adhesive tape. The sample should then be placed in a leak-proof container for transport or shipping. This is typically an insulated plastic cooler. It is recommended to store the samples near 39.2°F (4°C) to minimize sample changes between collection and testing [55]. Soil properties change over time. Testing should be completed within 2-3 days if completing microbial testing. Samples can be stored if sealed and stored at the recommended temperature, however it is recommended to complete testing as soon as possible after collection. Dry ice should never be used to keep samples cool as dry ice is a hazardous substance for shipping.



Key Technical Point

It is recommended to use freezer packs or gel packs to keep the soil samples cool during transportation, as using melting ice has the potential to contaminate a sample that was not properly sealed.

7.4 Labeling Requirements

Each soil sample should be labeled individually. If double bagging, it is recommended to label each bag. The label should be physically written on the transporting item. This lessens the chance of the soils being misidentified during later testing steps. At a minimum the following should be included on the label:

- Company and plant/facility name
- Name of the person/organization collecting the sample
- Location:
 - State and County
 - Northing/Easting
 - GPS coordinates
- Date
- Depth of sample

- Weather conditions
- Identification of associated plant/facility equipment, location in relation to the pipe (12 o'clock, 3 o'clock, girth weld, stationing, distance to nearest reference point along the pipe, and so on)
- Sample number if taken as part of a series (1 of 4 for example)
- Backfill or residual soil, and/or if taken directly next to the buried structure
- Sample collection method (excavation, hand dug, auger, or other)
- Soil type estimate (sand, clay, and silt)

7.5 Shipping Samples

7.5.1 Chain of Custody

A chain of custody (COC) is essential to ensure sample integrity through the entire sample collection and analysis process. A COC is a regulatory requirement for environmental samples and almost all soil testing laboratories will require one to receive samples regardless the federal soil shipping requirements for regulated and unregulated soils. A COC helps ensure that the sample is not tampered with and helps ensure the proper analysis is completed on the correct samples. A COC form will be initiated by the sample collector and sent with the samples to any other location where analysis is completed. A COC form will typically be a carbon copy form with 2-3 copies. A copy should accompany the samples and it is recommended to retain a copy as the owner of the soil sample. The COC form should be able to identify the samples collected, provide information on when and where the samples were collected, list the analyses to be completed, and establish any special requirements. The COC forms can be tailored to fit specific needs; however the following information should be included at a minimum:

- Owner information. The contact information of the individual responsible for the sample collection and analysis from within the facility organization.
- Sample identification. The facility, sample number/identification, date, number of containers, sample type (soil, water from soil extractions), sampler, and so on.
- Requested analysis. Itemize what analysis is to be completed on each sample (soil resistivity, pH, anion/cation content).
- Sign offs for each step. Steps requiring a signature:
 - Sample collection
 - Acceptance by transporter
 - Sample received by laboratory
- A location for notes and comments.

Laboratories will often have their own COC forms that are required in order to receive samples. It is recommended to attach tracking identification tags to COC forms as an added tracking system.

7.5.2 Shipping Restrictions

7.5.2.1 Radioactive Contamination

Soil samples must be unconditionally released from the site. Samples must be scanned to verify whether there is radioactive material present. Shipping regulations exist from the Department of Transportation (DOT), Nuclear Regulatory Commission (NRC), Department of Energy (DOE), United States Postal Service, and State Agencies [56]. If the sample does contain radioactive material, the sample can only be sent to a laboratory equipped to handle radioactive material. Consultation should be gathered for shipping radioactive material.

Nuclear power plants must consider, more than most facilities, the possibility of collecting soil samples that are contaminated with permitted/monitored substances, particularly radionuclides. Prior to shipping samples off-site, NPPs must pre-screen the specimen to verify whether it is contaminated. If so, this may limit evaluation to on-site testing or to laboratories that are specifically suited to evaluate contaminated samples. This increased control will add to permitting/review required.

7.5.2.2 Quarantine Regulation

Shipping and movement of soils is regulated by the US Department of Agriculture (USDA) Animal and Plant Health Inspection Services (APHIS) under the Plant Protection Act of 2000 [57]. Soil samples have the ability to contain organisms that may adversely affect a local environment if transported to a new location. As such the movement of soils is restricted. As a result, importation of soil into the United States from foreign sources is prohibited, and movement within the continental US is restricted unless authorized by APHIS under specific conditions, safeguards, and controlled circumstances described in a permit and/or compliance agreement. Some soil movement is regulated and some soil movement is not. Soil samples are regulated based on where they are collected and where they will be shipped. A laboratory must have a "Permit to Receive Soil and Compliance Agreement" via a Plant Protection Quarantine office in order to receive regulated soils. A list of currently approved facilities is provided on the USDA APHIS website [57].

The goal of the regulations is to prevent insects and other environmental pests from moving from one location to another via soil samples. Therefore, quarantine zones are set up for individual pests. If a soil sample is moved from a quarantine zone to outside the quarantine zone, the soil sample movement will be regulated. The majority of quarantine zones are based on states and in some cases counties within states. It can be difficult to determine whether or not a soil sample will be regulated. As a shipper, the following steps can be completed to ensure that there are no violations:

- Contact a local Plant Protection Quarantine (PPQ) office and determine what areas will be regulated for the facility where the soil samples are to be collected.
- Check with the laboratory where the samples are to be sent. The laboratory will know what samples are regulated for them to receive if they have an APHIS Permit to Receive Soil and Compliance Agreement.

For domestic samples, if movement of the soil sample does not require crossing a quarantine zone, the soil sample movement will not be regulated and the soil sample can be shipped in a normal manner.

Soil samples being imported into the United States must meet several requirements. The exact requirements may vary based on the permit for the specific laboratory. The following will be required at a minimum:

- The exterior of the packing must read “Contents: Soil Samples”.
- The USDA PPQ Form 550 and copy of the “Permit to Receive Soil” must be affixed to the exterior of the container.
- A laboratory request label must also be affixed to the exterior.

The laboratory receiving the soils will need to be contacted prior to shipping the soil samples. The laboratory will be able to provide the legal requirements to have the soil shipped there. Be aware, that there can be up to 20-day notification times required so laboratories should be notified well in advance of soil sample collection.

7.6 Testing Procedures

Proper quantification of the identified parameters is essential to accurately characterize the soil corrosivity at an NPP facility. When possible, soil samples should be tested in the as-collected condition, as well as the saturated condition. For most parameters there are multiple test methods that can be used to accurately quantify the given parameter. In general, each parameter should be tested using the appropriate ASTM International standard, ISO⁵ Standard, EPA Test Method, or Standard Method.

Testing should be completed by qualified individuals, who have received the proper training, education, and if necessary certifications to complete the testing. Regardless of the parameter being analyzed, it is important to vet the work of third party contractors and laboratories. Laboratories for soil testing should have its own internal quality assurance/quality control (QA/QC) programs. Reviewing and understanding these procedures can help with understanding the quality of the data being provided. It is also recommended to send the laboratory reference samples with known concentrations of certain parameters [58]. These reference samples can be purchased from commercial vendors or created by a qualified individual. The advantage of using a test method from one of the sources listed above is that the test will list the precision, threshold, and/or repeatability statistics of the test method.

The number of different relevant parameters tested will vary from facility to facility. The different tests to be performed is based on the underground piping and tank materials, the coating systems used, whether or not CP is used, local soil conditions, and identified potential damage mechanisms. For facilities where soil testing has not been used historically, it is recommended to test for a broader set of parameters initially.

This section provides a general description of each soil parameter and how to test for the soil parameter. Appendix C provides the general procedures for the processes summarized in this section.

⁵ International Organization for Standardization (ISO)

The standards listed in this document are the standards commonly utilized in the industry. The goal of this document is to present the procedures that can be used to develop internal soil testing procedures. The standards used in this document to explain the testing of a given parameter are provided as an example or guide to develop an internal procedure. Standards are also listed to facilitate a discussion with external testing laboratory. The decision to use a given reference procedure should be based on company policy, local regulatory requirements, and in consultation with a corrosion engineer.

7.6.1 Soil Resistivity

Soil resistivity can be measured both in the field and in the laboratory using ASTM G57-06 (2012) “Standard Test Method for Field Measurement of Soil Resistivity” [59], using the Wenner Four-Electrode Method. The most applicable method to testing in NPPs is the soil box method. The soil box method and the four-pin method, discussed below, use the same principles. The soil box method is preferable to the four-pin method in most applications for NPPs. This is because the soil box method measures the local resistivity of the sample collected, ideally in the backfill adjacent the pipe. The four-pin method measures the average soil resistivity over a larger area and is affected by changing soil types and other buried structures. For both methods, a current is passed through two outer electrodes and the potential difference between two inner electrodes is measured. Using Ohm’s Law ($V=IR$) the resistance “R” can then be calculated. Figure 7.2 shows a diagram of a typical soil box. Taking the system geometry into account, the resistivity can then be calculated.

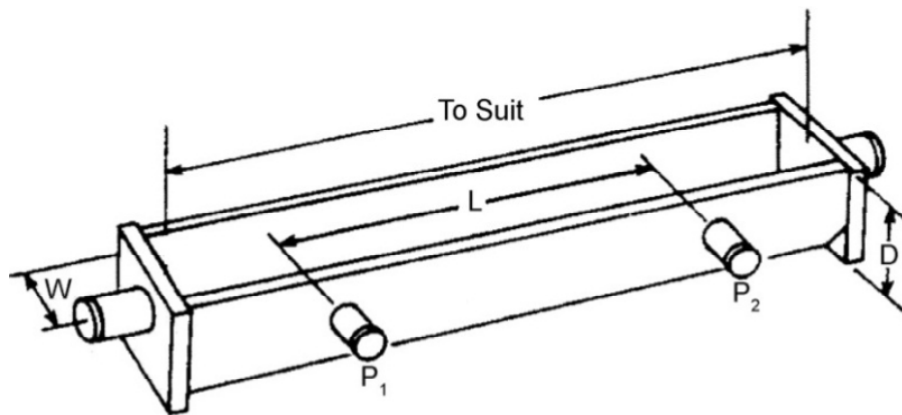


Figure 7-2
A diagram of a soil box used to measure soil resistivity [1]

The equation for calculating the soil resistivity using the four-electrode soil box method is presented below:

$$\rho = R \cdot A / L$$

Where:

ρ = Resistivity, ohm·in (ohm·cm)

R = Resistance, ohm

A = cross sectional area of the container perpendicular to the current flow, in² (cm²),
where $A = 2W \times D$ in Figure 7-2

L = inner electrode spacing, in (cm)

In addition to the four-electrode method, ASTM G187-12a “Standard Test Method for Measurement of Soil Resistivity Using the Two-Electrode Soil Box Method” [60] can be used to measure the soil resistivity. The two-electrode method does not utilize the two inner pins used in the four-electrode method. Yet another method is the single probe method. There are several commercially available probes to be used. This method can also be used when it is impractical to use the four-pin method due to other piping in the area [14].

Another common in-situ method, described in 6.1, utilizes four pins placed into the ground, see Figure 7-3. The four-pin method uses four electrodes placed in straight line, perpendicular with the pipe, with an equal distance between them.

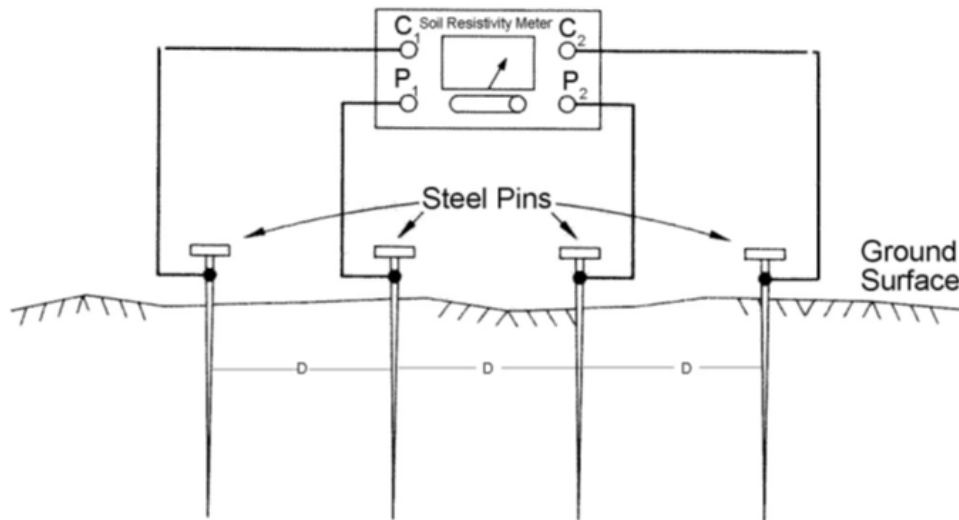


Figure 7-3
Pin placement for soil resistivity measurements using four-pin method [1]

The outer pins act as the current carrying electrodes, and the difference of electrical potential is measured between the two inner pins. The resistivity of the soil can then be calculated using the following equation:

$$\rho = 2\pi aR$$

Where:

ρ = Resistivity, ohm·ft (ohm·cm)

a = pin spacing, ft (cm)

R = Resistance, ohm

The resistance can either be measured using a separate current source, ammeter, and voltmeter, or by using a galvanometer/soil resistivity meter. The resistivity measured is the average resistivity in a hemisphere centered at the middle of the pin arrangement with a radius equal to the pin spacing, see the outlined area “d” in Figure 7-4.

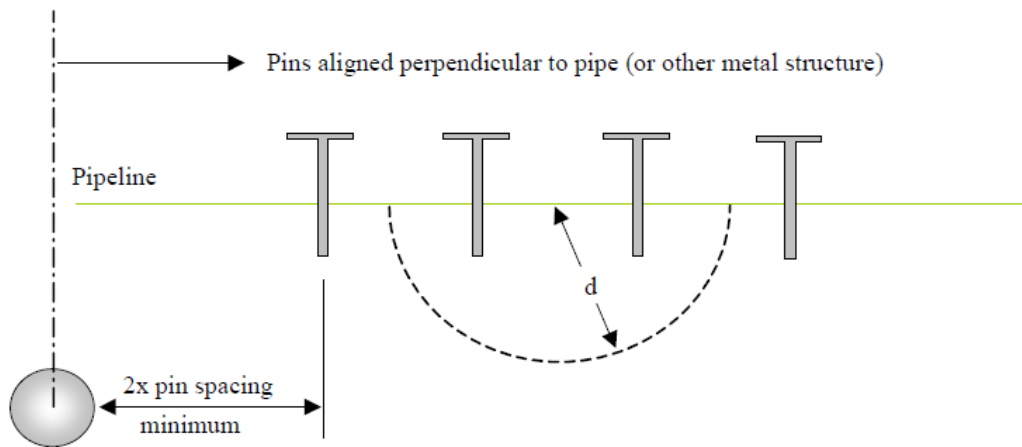


Figure 7-4
Required pin orientation for soil resistivity measurements using four-pin method [1]

Additional guidance for completing field resistivity measurements is presented in NACE Standard SP0502-2010 “External Corrosion Direct Assessment Methodology” [14]. It is important to keep a horizontal separation of at least twice the pin spacing from any buried metallic structure, including the buried structure of interest. It is also important to select an area that is representative of the soil along the length of the pipe. If there is a section where large rocks are known to be present, this area will have different resistivity properties than adjacent areas.



Key Technical Point

The soil resistivity for a sample should be measured for both an as-received sample and for a water-saturated sample. The saturated sample usually has the lowest resistivity for a given soil sample.

The resistivity of soil will vary throughout the year based on the temperature and the soil moisture. The sample collected will only present the soil moisture at the time of collection. Measuring a saturated soil resistivity will provide a worst case scenario that can be used to estimate corrosion rates during wet periods. ASTM G57-06 (2012) contains guidelines on saturating soil for soil resistivity measurements [59]. It is important to use deionized water for the saturation of a soil sample to prevent the introduction of new ionic species to the sample.

7.6.2 Redox Potential

The redox potential can be measured both in the field and in the laboratory following ASTM G200-09 (2014) “Standard Test Method for Measurement of Oxidation-Reduction Potential (ORP) of Soil” [61]. Redox potential can be measured using any commercially available probe designed for redox potential measurements. As stated previously, it is difficult to measure an accurate redox potential from soil that has been disturbed and exposed to oxygen. Therefore, a more accurate reading can be obtained by taking a reading in undisturbed soil several inches deep from the wall of the pipe excavation.

7.6.3 Chemical Constituents

The most common way to determine the soluble ionic content is to perform a comprehensive water analysis on an extracted sample.

7.6.3.1 Soluble Ion Extraction

Soluble ion extraction procedures can vary based on the type of analysis being completed and the requirements of that analysis. Many laboratories will offer in house extraction as part of their services. If completing the process in house there are a few general principles behind the process. The goal is to remove the soluble ions from the soil and create a solution that will be acceptable to perform the desired testing.

The first step of the process is to remove the ions to be measured by mixing the as-collected soil sample with deionized water. This should be done by removing a specified mass of a soil sample and mix it with a known volume of deionized water. This process is known as dilution. Analytical tests require various levels of dilution of the soluble species in the sample in order to measure the ion content accurately. The degree of dilution must be measured carefully in order to obtain accurate results from the soluble species analyses.

The second step is to ensure that the soil and water sample is thoroughly mixed. This can be completed by using an agitator, stirring apparatus, or any other mixing method. The preferred methods are the ones that limit air exposure of the sample during mixing.

After the sample is thoroughly mixed, the sample is then filtered in order to remove suspended solids. Certain analytical test techniques specify a maximum particle size remaining suspended in the electrolyte to ensure an accurate measurement without interference from the solids. It is recommended to use a series of increasingly finer filters to clarify the electrolyte. The first filter step can be performed by passing the electrolyte through a 2 mils (50 microns) filter. This will aid in processing time as larger soil particulates often clog fine filters during the clarification process.



Key Technical Point

Sample exposure and contamination must be minimized during extraction to provide the most accurate results.

7.6.3.2 Analytical Methods

There are several analytical methods that can be used to test for the given chemical constituents. Each method has its advantages and disadvantages. The test method used can depend on time, cost, availability of local labs and equipment, accuracy desired, and regulatory requirements. Table 7-1 lists the recommended methods for each ionic constituent or property.

Titration

Titration determines the concentration of an ion in a solution by adding a known amount of a reagent and measuring how much of that reagent is required to complete a chemical reaction to a given endpoint. By knowing the volume and concentration of reagent required to complete the given reaction, the concentration of the ionic reactant can be calculated. The end point of a reaction is commonly detected by observing a color change of the solution, fluorescence, change in pH to a given level, formation of a precipitate, or a change in clarity of the solution (turbidity). The reagent required to complete the titration will change based on the ion being measured, and may change for different analytical methods of the same ion. Some titration methods involve additives of buffered reagents or indicators. Refer to the specific procedure for the ion being detected for what reagents, buffer solutions, or indicators are required.

Titration is commonly used to measure the hardness and alkalinity of a solution; however, the method can be used to determine the concentration of most ions of concern for corrosivity of soils. Titration requires limited equipment and investment compared to other methods. However to achieve the level of accuracy required, the method requires well trained technicians, proper procedures, and a written quality control program.

Colorimetric Spectrophotometry

Colorimetric spectrophotometry determines the concentration of a species in a solution by forming a colored complex. The color in the solution is created by mixing the sample and a reagent. The colored complex is analyzed by passing a light beam through the sample. The concentration of the target ionic species bound to the complex is proportional to the amount of light absorbed by the sample at a given wavelength.

This method can be completed using a commercially available colorimetric spectrophotometer. Each unit requires specific procedure and reagents to complete the analysis of given ions. Some colorimeters only measure one ionic species, such as chloride. It is also important to ensure that the colorimeter can measure the species of interest to the desired level of accuracy.

Ion Chromatography (IC)

Ion chromatography (IC) determines the concentration of ions of interest by first separating them and then detecting each individual ion. The process begins by adding a clarified water sample to an eluent stream. The stream is pumped through a column that separates the ions based on the ion affinity to the column substrate. The greater the affinity an ion has for the substrate in the column

the longer it takes for the ion to move through the column. This principle allows ions to reach the end of the column at different times. The amount of time the ion takes to move through the column is known as the retention time. Modern IC machines incorporate a detector to determine which ion is present. Common methods are conductivity detection, electrochemical detection, and spectroscopic detection methods [62].

Inductively Coupled Plasma/Mass Spectrometry

Inductively coupled plasma/mass spectrometry (ICP/MS) can be used to detect metallic ions in a sample. A sample is nebulized and exposed to a high temperature ionized argon gas plasma stream. The argon gas is ionized by high frequency radio waves. The sample interaction with the plasma will result in temperatures high enough to completely dissociate molecules in the sample. The dissociated ions are then measured using a mass spectrometer [58].

Table 7-1
Soil testing procedures for chemical constituent analysis

Parameter	ASTM	ISO	Standard Method
Carbonates	D1067 [63]	ISO 9963-1:1994(en) [64]	SM2320B [65]
Bicarbonates	D1067		SM2320B
Chlorides	D512 [66]	10304-1:2007(en) [67]	SM4500 [68]
Sulfates	D516 [69]	10304-1:2007(en)	SM4500
Sulfides	–		SM3500D [70]
Nitrates	D3867 [71]	10304-1:2007(en)	SM4500
Nitrites	D3867	10304-1:2007(en)	SM4500
Calcium	D511 [72]	14911:1998 [73] and 17294:2003 [74]	SM3500
Magnesium	D511	14911:1998 and 17294:2003	SM3500
Potassium	D3561 [75]	14911:1998 and 17294:2003	SM3500
Sodium	D3561	14911:1998 and 17294:2003	SM3500

Note: The ISO Standards provided are the methodologies used to determine the soluble contents of soil extracts.

7.6.4 Acidity

7.6.4.1 pH

ASTM D4972-13 “Standard Test Method for pH of Soils” [76] and AASHTO⁶ T289-91 (2013) “Standard Test Method for Determining pH of Soil for Use in Corrosion Testing” [77] provide a similar method to measure soil pH. ASTM D4972-13 was developed for testing in any industry. Both methods involve creating soil suspensions and measuring the pH of the suspended soil mixture. ASTM D4972-13 is the industry recommended method for pH testing in soil as it also requires a suspension in calcium chloride solution, in addition to testing in water. The calcium chloride interacts with the soil producing a lower pH measurement than water. However, this measurement is potentially more accurate based on the metallic compounds present in the soil.

ASTM G51-95 (2012) “Standard Test Method for Measuring pH of Soil for Use in Corrosion Testing” [78] is another available method. This method uses a reference calomel electrode and a glass electrode suitable for field use and a portable pH meter. After calibrating the pH apparatus using buffer solution, the pH of the soil is measured by inserting the electrodes into the soil. Measurements should be taken at three different locations for a given depth and averaged. It is recommended to measure the pH of the soil at the same depth of the buried metallic structure. This can be completed by collecting a sample of the soil from that depth by boring or excavating and then bringing the sample to the surface for testing. This method can also be completed in the laboratory if necessary.

ASTM G51-95 (2012) recommends packing the sample in dry ice if the pH is not measured within 24 hours of taking a soil sample to minimize any change in the soil sample. This can be done if the sample is going to be tested at a later date on site, however as discussed previously, dry ice should not be used on samples to be shipped.

Both methodologies have advantages and disadvantages. ASTM D4972-13 is a simple test. In some cases a change in pH will occur due to the poor buffering capacity of soils. However, research has shown that drying out the soil prior to testing will cancel out this effect. The ASTM G51-95 (2012) method assumes ideal conditions for the measurements, which are often not encountered in the field. The methodology used in ASTM D4792-13 was developed in response to these challenges [21].

7.6.4.2 Total Acidity

In addition to the pH, the total acidity should be measured. A recommended procedure for measuring total acidity is Standard Method 2310B [79]. This method uses titration to determine the total acidity of a solution. This test is completed on a solution extracted from the soil sample.

⁶ American Association of State Highway and Transportation Officials (AASHTO)

7.6.5 Moisture Content

Moisture content can be measured using simple drying and weighing techniques. The principle is that soil is measured before and after drying to calculate the percent moisture in the sample. The key to accurate testing is following proper handling and shipping instructions to maximize moisture retention of the sample during shipment and minimizing exposure that may add or take away moisture from the sample. The following procedures are recommended for measuring moisture content of a sample:

- ASTM D2216-10 “Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass” [45]
- AASHTO T 265-12 “Standard Method of Test for Laboratory Determination of Moisture Content of Soils” [80]
- EPA Method 1684 “Total, Fixed, and Volatile Solids in Water, Solids, and Biosolids” [81]

ASTM D2216 and AASHTO T265 yield moisture content as a ratio of water to solids. The EPA method calculates the mass percentage of water. This value is more useful when attempting to calculate the amount of water needed for proper water extraction for soluble ion testing. Other, more advanced methods of determining soil moisture have been developed; however if done correctly, the drying and weighing differential methods can provide an accurate value of the moisture content of the soil for the purposes of corrosivity analysis.

7.6.6 Microbial Content

There are many different types of tests that can be used to assess the types, numbers, and activities of bacteria in soils. NACE TM0106-2006 “Detection, Testing, and Evaluation of Microbiologically Influenced Corrosion (MIC) on External Surfaces of Buried Pipelines” [13] provides guidelines for sample collection and testing. Liquid culture media are commonly used to quantify different groups of microorganisms in soils, and from biofilms/corrosion on the pipe. Some bacteria cannot be grown in culture media, so results from this method may under report the numbers of bacteria in the sample. Genetic methods can also be used to quantify functional groups and specific organisms in a sample. Genetic methods identify and count microorganisms using rDNA⁷. Most genetic methods count both living and dead bacteria and provide a more accurate picture of microbial diversity in soils. Microbial numbers and diversity may vary significantly between samples from:

- Uncorroded pipe wall
- Beneath coating defects, blisters, or disbondment
- Corrosion pits on the pipe wall
- Soil directly in contact with the pipe
- Soils from 1 inch to 5 feet (or more) away from the pipe
- Backfill
- Side wall of ditch at different depths
- Ground water

⁷ Ribosomal Deoxyribonucleic Acid (rDNA) is genetic code for a specific cellular component within the bacteria

Samples for bacteria testing should be collected using sterile instruments to avoid contamination. Samples should be collected immediately after the pipe is exposed as air, sunlight, water, and other factors will quickly cause changes that affect the microbiological test results. For genetic testing, samples should generally be stored on dry ice until they arrive at the testing lab.

Bacteria testing can be completed in accordance with NACE TM0194-2014 “Standard Test Method Field Monitoring of Bacterial Growth in Oil and Gas Systems” [82], which provides guidelines on culture testing. The scope of NACE TM0194 goes beyond MIC on the external surface; however, the principles of culture testing are the same. There are several commercially available kits for bacteria culture testing. Aerobic, anaerobic, sulfate-reducing, acid-producing, and iron-reducing bacteria are typical groups of microbes that are tested using media.

7.6.7 Soil Classification and Particle Distribution

In part, soils are classified by the size of the particles that make up the soil. The particle size distribution of the soil can provide an indication as to how well moisture, air, and electrolytes will move through the soil. ASTM D422-63(2007) “Standard Test Method for Particle Analysis of Soils” [39] can be used to determine the particle size distribution. The test method uses two different phases of analysis to determine the different sizes of particles and corresponding percentage of the total soil mass. The first phase uses sieves to collect the larger particles. Sieves of different sizes capture particles of decreasing size. The weight of the particles left in a sieve gives the amount of particles that are nominally larger than the sieve size, but smaller than the sieve size above it. For smaller particles, a sedimentation process is used based on Stoke’s Law. Smaller particle sizes are defined as those which pass the #200 sieve. The test will distinguish between particle sizes which are classified as silts and clays. This is the second phase for soil grain-size analysis.

A hydrometer is used to measure the specific gravity of the silt and clay particles in a water suspension. The rate at which the particles sink is a function of its diameter. Between the sieve and sediment analysis the particle distribution can be determined ranging from particles greater than 3 in. (75 mm) to 0.04 mils (1 micron). ISO/TS 17892-4 “Geotechnical investigation and testing-laboratory testing of soil-Part 4: Determination of particle size distribution” [83] provides a procedure for the same general process.

7.6.8 Mechanical Properties

The bulk density of a soil can be determined using ASTM D7263-09 “Standard Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens” [84]. The bulk density of a soil can be used to determine the porosity and therefore the potential degree of aeration of a soil. This method has the advantage of providing methodologies for both intact and re-compacted soil samples. The intact method provides a more accurate measurement than re-compacted soil. The intact method requires the use of thin-wall tubes (Shelby tube), or similar method for extraction.

The hydraulic conductivity of a soil can be measured using ASTM D7664-10 “Standard Test Methods for Measurement of Hydraulic Conductivity of Unsaturated Soils” [85]. This standard provides several different methodologies to measure the hydraulic conductivity of the soil. Determining which methods to be utilized and analysis of those results requires an individual with knowledge of soil engineering properties.

7.6.9 Summary of Soil Testing Procedures

Table 7-2 provides a summary of the soil testing procedures discussed in this section.

Table 7-2
Summary of soil testing procedures

Testing Parameter	Testing Procedure(s)
Soil resistivity	ASTM G57-06 (2012) ASTM G187-12
Redox potential	ASTM G200-09 (2014)
Chemical constituents	See Table 7-1
pH	ASTM D4972-13 AASHTO T289-91 (2013) ASTM G51-95 (2012)
Total acidity	Standard Method 2310B
Moisture content	ASTM D2216-10 AASHTO T 265-12 EPA Method 1684
Bacterial content	NACE TM0106-2006
Mechanical properties: • Bulk density • Hydraulic conductivity • Soil classification and particle distribution	ASTM D7263-09 ASTM D7664-10 ASTM D422-63(2007)e2 ISO/TS 17892-4

7.7 Seasonal Variations

Soil characteristic can vary significantly over time and with changes in season. Throughout the year the amount of precipitation and temperature will vary significantly. Increased precipitation will cause more moisture to ingress into the soil. Not only will this increase soil moisture, it can also bring additional soluble ions into the soil and alter the soil chemistry. The change in moisture also has an effect on the mechanical characteristics of the soil. The seasonal variability occurs mainly in clay soils with swelling and shrinking properties. The swelling or shrinking follows the periodicity of the wet and dry seasons [31]. The swelling and shrinking can result in cracking of the soil allowing water and ions to move through the soil. Soil stress can also affect the coating performance and the swelling and shrinking of soil can increase the rate of coating degradation⁸. The temperature of the soil also changes the characteristics of the soil. One obvious change is when soils freeze. Frozen soils will not transport water, nutrients, ions, and currents in the same manner as thawed soils. An increased temperature can lead to increase solubility of some ions and a decreased solubility of other ions.

⁸ The movement of the soil causes disbondment and wrinkling, which can lead to cracking of the coating and moisture ingress.

Both the amount of water and the phase of the surface water have a large effect on the soil resistivity throughout the year. For example, when surface water is frozen the resistivity will be much higher. One study indicated almost an order of magnitude difference between seasonal soil resistivity measurements [86]. The changes on the surface will also affect the environment in the soil at the pipe depth. The magnitude of these changes will depend on the soil properties such as the drainage, hydraulic conductivity, and particle size distribution.

Due to the seasonal variability of soil characteristics, soil testing should occur at different times of the year. The time of collection should be closely tracked. When analyzing data from soil samples the climatological conditions at the time of collection should be recorded. It is recommended to collect soil samples during both wet and dry seasons and comparing the results. Corrosion rates in soils can vary throughout the year and understanding soil characteristics over the course of an entire range of conditions can lead to better corrosion rate prediction. Soil resistivity in particular can vary by as much as an order of magnitude based on soil conditions.

One approach to meet the challenges of seasonal variability is to analyze a soil in the “as-found condition” and also test the soil in the saturated condition. Saturated samples are soils that cannot take on any additional water. Deionized water can be added to a soil sample until the point when free liquid begins to form. This type of testing is common with soil resistivity and moisture content. This type of testing can provide insight into the soil conditions during extremely wet weather patterns.

8

DETERMINING QUALITY OF TEST RESULTS

The validity of the data collected is dependent both on the ability of the sample collected to represent the conditions experienced by the buried metallic structure and the ability of the test utilized to accurately measure the parameter of interest. The sample should be representative of the environment when the procedures for collecting, shipping, and storing the sample are followed. It is also important to note that even if samples are collected properly, the sample collected only represents the condition the buried metallic structure is experiencing at the time of collection. As further described above, the soil conditions will often change throughout the year, and conclusions drawn from the sample collected should be made with an understanding that the data is a snapshot of the conditions at one point in time from a single location. Analyzing soil samples throughout the year can improve the understanding of the impact of seasonal variability on soil corrosivity.

The ability to accurately measure the parameter being tested in a soil sample is determined by the analytical techniques, skill of the technician, hardware type, and adherence to procedures. Standards such as those from ASTM International and ISO typically specify a desired level of accuracy for a specific test. The specified accuracy in a standard can provide a guideline to determine the quality and accuracy of analytical results. Data that do not meet the desired level of accuracy should not be used and the test should be repeated using new samples. Also, laboratories will typically have their own internal QA/QC procedures to specify the precision of the results. Precision is the closeness of agreement between two randomly selected individuals or test results. For example, the standard deviation of the error of a method can be used to measure the precision of a set of test results [87]. It is always advisable to request a QC report with standardized sample results collected by a third party laboratory. The procedures in Appendix C discuss test elements that may affect test accuracy for a given soil parameter.



Key Technical Point

The interpretation of a given test result will be based on the standard selected/procedure developed to quantify that particular parameter.

9

EFFECT ON CATHODIC PROTECTION, COATINGS, AND MATERIALS OF CONSTRUCTION FOR BURIED STRUCTURES

9.1 Effect on Cathodic Protection

Soil properties have two main effects on the CP system. The soil properties can determine the electrical path for CP currents and soil properties often determine the corrosion potential of the steel in soil. These two factors determine the current demand of the buried structure on the CP system to mitigate corrosion. For example, if the properties of the soil make the corrosion potential of steel more active (more electronegative) and create a situation where the polarization resistance of the structure is high, less CP current will be required to maintain the required cathodic potential to minimize corrosion. Cathodic protection current demand can be high when the steel corrosion potential is noble (more electropositive) and the polarization resistance is low. Increased CP current demand can affect the maintenance and design of the system due to anode consumption and other hardware maintenance issues. All of the following can have an effect on CP current demand [35]:

- Type of soil, including backfill and soil between pipe and anode
- Soil resistivity
- Soluble salt content
- Moisture content
- Acidity/alkalinity
- Dissolved gases content
- Presence of MIC producing bacteria
- Temperature

Some of these properties are interrelated. For example, the type of soil, soil moisture, and soluble ion content will have an effect on the soil resistivity. The soil resistivity has a direct impact on the electrical current demand. From a CP perspective, the soil resistivity is more important in the anode groundbed design, as it is used for calculating anode-to-earth resistance. That being said, buried metallic structures that are exposed to soils with lower soil resistivity (that is, soils that are more corrosive), will tend to have a higher current demand for CP. Table 9-1 [26] shows an example of this phenomenon.

Table 9-1
Current requirements of CP for buried steel at different soil resistivities

Soil Resistivity [ohm-cm]	Current Density [mA/ft²]
50-500	0.1-0.2
500-1,500	0.05-0.1
1,500-4,000	0.01-0.05

These current demands do not consider the influence of other buried metallic structures, such as grounding systems. However, the general trend can be seen in Table 9-1. Accurate characterization of soil resistivity is an essential part of the design of CP systems. Knowing the average soil resistivity will allow designers and engineers to better select the type of system, location of ground beds⁹, type of ground beds, and the expected life of the ground beds. As discussed previously, the soil resistivity will change throughout the year. Understanding this phenomenon throughout the year can also affect the CP system design and operation. It may be necessary to change current output on rectifiers¹⁰ throughout the year as the soil resistivity changes.

The pH and dissolved oxygen content also have an effect on the current demand. Lower pH and increased dissolved oxygen content will increase the current demand. This is due to the increased driving force for corrosion activity by supplying cathodic reactants to the electrochemical cell. As stated in 2.1.1, the rate determining reaction in a corrosion reaction is most often the cathodic reaction. Therefore, if there are more cathodic reactants, there will be greater chance for corrosion activity. If MIC is identified on a buried metallic structure, the required pipe-to-soil potential could be increased to -0.950 V CSE to prevent corrosion activity [35]. In order to make the pipe-to-soil potential more electronegative, the current output would need to be increased if all other system parameters remain the same.

9.2 Effect on Coatings

Soil properties, in relation to coatings and corrosion, can determine whether a CP and coating system is required on the buried structure. Coatings are the first line of defense in separating the metal surface from the environment. Cathodic protection provides a second line of defense in instances where there is damage to the coating and the metal surface is exposed to the environment. The soil properties will also affect how coating systems perform in a given environment. The GALL Report, Section XI.M41 [2] provides guidelines for preventative action of buried metallic structure degradation. Table 9-2 summarizes the guidelines for use of coating based on material selection. Stainless steel, for example, is recommended to have coating in chloride containing environments.

⁹ Ground beds are an arrangement of anodes that are part of the CP system. The arrangement and design of the ground bed can be changed based on the demands on the CP system.

¹⁰ A rectifier is used to convert alternating current to direct current and adjust the CP current applied to the system.

Table 9-2
Recommended use of coatings based on material and soil environment [2]

Material	Coating Utilization
Titanium	No
Super Austenitic Stainless	No
Stainless Steel	Yes, when chlorides are present
Steel	Yes
Copper	Yes
Aluminum	Yes

The soil properties that can impact coatings performance and coating selection are soil type, particle size distribution, and moisture content. Industry research has shown that the vast majority of coatings used in NPPs, up to 95 percent, have been coal tar enamels and coal tar epoxies [9]. Coal tar enamel and epoxy coatings can be susceptible to degradation due to soil stresses, poor installation practices, and temperature. Soil stresses are often much higher in expansive-clay type soils due to the ability to transfer stresses and expand and contract. The movement of the soil causes disbondment and wrinkling, which can lead to cracking of the coating and moisture ingress. Soil moisture will also play a part in this process. Other coating systems that are more susceptible to degradation due to soil stress are tape coatings. More modern coating systems such as FBE have better resistance to soil stress due to its superior bond strength and ease of quality control at the mill. In considering soil impact on the coatings, it is recommended to consider worst case locations including field-coated welds, patch repairs, pipe irregularities, concrete interfaces, and other areas where differences in coating performance combine to create heightened risks for a given soil condition as compared to uniformly coated piping. Soil particle size distribution and rock content also have an effect on coating system selection. In rocky soils, it may be necessary to apply a rock shield overwrap to prevent damage to the coating during backfill and natural soil movement.

9.3 Effect on Materials for Piping and Tanks

Plain carbon steel is the most abundant material of construction for piping and tanks in the NPP industry and beyond. As such, steel has the most information describing its corrosion characteristics in many different types of buried service over a wide range of exposure periods. Other common materials are cast iron, ductile iron, stainless steels, aluminum alloys, copper alloys, PCCP. There are very limited instances of titanium being used in buried piping in the NPP industry. As discussed previously, it can be difficult to assign an exact corrosion rate to a given soil due to the complex interaction of the different soil parameters. Figure 2-1 shows the relationship of the different soil parameters. In addition, different materials will have different corrosion behavior. This section summarizes some of the common methods of determining the corrosion behavior of the different materials commonly used in the NPP industry.

Many studies have been completed to measure the corrosion rate of steel in soils. The corrosion rates reported in these studies must be examined in the context of the exact soil conditions in which they were measured and the exposure period. The large number of variables that can affect steel corrosion rates in soils leads to great difficulty when attempting to derive precise relationships between soil properties and metal loss. Nonetheless, methods have been developed ranging from qualitative guidelines, to semi-quantitative guidelines, to mathematical modelling to predict soil corrosivity. Previous EPRI research calculated the average corrosion rate over all soil types to be 3.5 mpy (0.9 mm/year) for pitting and 0.7 mpy (0.2 mm/year) for general corrosion rates [7]. These rates varied however for different soil types. The average rates for poorly aerated soils for pitting and general corrosion were 8.2 mpy (2.1 mm/year) and 2.2 mpy (0.6 mm/year). A conservative, default pitting corrosion rate of 16 mpy (4 mm/year) on carbon steel in soil has been established for the pipeline industry in NACE SP-0502-2010 [14].

A simplified approach to estimate the corrosivity of soil is to measure selected parameters and place a qualitative assessment (or ranking) of the measured values in relation to corrosivity. Section 2 presents an example of qualitative assessments for soil corrosivity on carbon steel and other select materials. Examining each parameter individually can provide insight as to how corrosive the soil is, but this simplified approach may be misleading in some cases. For example, a very low soil resistivity does not always result in an extremely corrosive soil if film forming species are present. In other cases, a soil with a moderate soil resistivity could cause high corrosion rates on steel if the pH is low, or if the soil has a low oxygen concentration compared to adjacent areas on the buried metallic structure. Examining individual sites carefully provides more insight into the potential for corrosion activity on a case by case basis.

Soil corrosivity indexes have also been published to help engineers estimate corrosion rates of steel in soils. The indexes relate the likelihood of corrosion to several relevant parameters such as resistivity, pH, sulfides, chlorides, redox potentials, bacteria consortia, and moisture content. One example is the ANSI/AWWA C105 table for the corrosivity of soil for ductile iron pipes. The AWWA C105 values are presented in Table 9-3 [16]. A soil with a corrosivity index of 10 or greater is considered corrosive. These values are only relevant for buried cast/ductile iron structures. Another comprehensive index is the Romanoff Underground Corrosion book [19], which contains a table of corrosion rates of steel in soils throughout the United States and in selected regions around the world.

Table 9-3
ANSI/AWWA C105/A21.5 Soil corrosivity index for ductile iron in soil

Soil Characteristic	Metric/Metric Range	Points
Resistivity [ohm-cm]	< 700	10
	700-1,000	8
	1,000-1,200	5
	1,200-1,500	2
	1,500-2,000	1
	> 2,000	0
pH	0-2	5
	2-4	3
	4-6.5	0
	6.5-7.5	0*
	7.5-8.5	0
	> 8.5	3
Potential [mV]	> +100	0
	+50 to +100	3.5
	0 to +50	4
	Negative	5
Sulfides	Positive	3.5
	Trace	2
	Negative	0
Moisture	Poor drainage, continuously wet	2
	Fair drainage, generally moist	1
	Good drainage, generally dry	0

*If sulfides are present and a low or negative redox potential is measured, this value is 3.

The EPRI software BPWORKS [17] also has soil corrosivity indexes. This index includes most metallic materials that are used in NPPs. Table 9-4 presents the corrosivity index from BPWORKS. For the BPWORKS index, 15+ points is considered severely corrosive, 10-15 points is considered appreciably corrosive, 5-10 is considered moderately corrosive, and soils with 0-5 are considered mildly corrosive.

Computer models have also been developed to predict soil corrosivity based on soil properties. Some models use probabilistic relationships between parameters and corrosion rate, other models are deterministic (that is, they are only valid within the data set used to generate the model). Models can employ Monte Carlo Simulations, fuzzy clustering analysis, Weibull statistics, and Bayesian models [88, 89, 90]. General principles, questions, and parameters of interest that can be used to make reliable conclusions from a computer model are listed below [91]:

- What information is being gained from the model?
- Corrosivity ranking.
- Estimation of corrosion rate.
- Be aware of differences among data sets used in the model.
- Trend lines can be useful, however be aware of the size of the data scatter.
- Consider the size of error bands when selecting a model.
- Is the model applicable to the site conditions?
 - Does the model cover the site soil types?
 - Does the model incorporate CP?
 - For what material is the model suited?
- Be aware of boundary conditions in the model.
- Beware of applicability to welds.

Table 9-4
Soil corrosivity index from BPWORKS

Parameter	Value	Cast Iron	Carbon Steel Mild Steel Ductile Iron	Copper 90-10 CuNi	70-30 CuNi Adm Brass Al Brass Si-Bronze Al Bronze Naval Brass Muntz Metal	304 SS 316 SS Martensitic SS Duplex 2205 Duplex 2507 Super SS	Al Alloys 6061-T6 6063-T6 6063-T52 6101-T61	Titanium
Resistivity [ohm-cm]	< 500	10	10	4	4	10	10	0
	> 500 - 1,500	10	10	3.5	3.5	10	9	0
	> 1500 - 1,800	8	8	3	3	8	8	0
	> 1,800 - 2,100	5	6	1.5	1.5	5	7	0
	> 2,100 - 2,500	2	3	1	1	2	6	0
	> 2,500 – 3,000	1	2	0	0	1	5	0
	> 3,000 - 10,000	0	1	0	0	0	3	0
	> 10,000 - 20,000	0	0	0	0	0	1.5	0
	> 20,000	0	0	0	0	0	1	0
pH	0-2	5	5	4	4	5	10	0
	2-4	3	4	4	4	4	10	1
	4-5.5	2	3	3	3	3	2	0
	5.5-6.5	0	2	1	1	2	2	0
	6.5-7.5*	0	1	0	0	1	2	0
	7.5-8.5	0	0	0	0	1	2	0
	> 8.5	3	0	0	0	0	10	0

Table 9-4 (continued)
Soil corrosivity index from BPWORKS

Parameter	Value	Cast Iron	Carbon Steel Mild Steel Ductile Iron	Copper 90-10 CuNi	70-30 CuNi Adm Brass Al Brass Si-Bronze Al Bronze Naval Brass Muntz Metal	304 SS 316 SS Martensitic SS Duplex 2205 Duplex 2507 Super SS	Al Alloys 6061-T6 6063-T6 6063-T52 6101-T61	Titanium
Redox Potential [mV]	> +100	0	0	0	0	5	0	0
	> +50 to +100	3.5	0	0.2	0.2	4	0	0
	> 0 to +50	4	2	3	3	1	0	0
	Negative	5	3	0	0	0	0	0
Sulfides	Trace	0	0	12	12	3.5	1	0
	Negative	0	0	0	0	0	0	0
Chlorides [ppm]	> 5,000	0	4	1	1.5	5	4	0
	> 1,500 - 5,000	0	3	0	1	5	3	0
	> 1,000 - 1,500	0	2	0	0	5	2	0
	> 100 - 1,000	0	1	0	0	3	1	0
	< 100	0	0	0	0	1	1	0
Soil Moisture	Continuously Wet	2	3	2	2	2	3	0
	Generally Moist	1	2	1	1	1	2	0
	Generally Dry	0	1	0	0	0	0	0

Table 9-4 (continued)
Soil corrosivity index from BPWORKS

Parameter	Value	Cast Iron	Carbon Steel Mild Steel Ductile Iron	Copper 90-10 CuNi	70-30 CuNi Adm Brass Al Brass Si-Bronze Al Bronze Naval Brass Muntz Metal	304 SS 316 SS Martensitic SS Duplex 2205 Duplex 2507 Super SS	Al Alloys 6061-T6 6063-T6 6063-T52 6101-T61	Titanium
Soil Consortia (Bacteria)	Positive**	0	3	0	0	3	3	0
	Positive Other Bacteria	0	3	0	0	1	1	0
	Negative	0	1	0	0	0	0	0

* If sulfides are present and a low or negative redox potential is measured, this value is 3.

**Positive for aerobic iron oxidizing bacteria, aerobic manganese oxidizing bacteria, sulfate reducing bacteria, or sulfur oxidizing bacteria.

9.3.1 Other Alloys

The use of other alloys may be necessary to maintain structural integrity if the corrosivity of the soils is unacceptable for carbon steel. Other factors, such as the service of the underground pipe or tank, regulatory requirements, or long term economics may require the application of an alloy other than steel. GALL AMP XI.M41 [2] provides incentives for using corrosion resistant materials through fewer recommended inspections and a lower risk ranking [9]. Regardless of the driving forces for utilizing different alloys, this section describes the corrosion behavior of other common alloys as it pertains to the interpretation of soil testing.

9.3.1.1 Stainless Steels

Austenitic stainless steels have the potential to offer improved protection over carbon steel in certain environments. However, austenitic stainless steels can still corrode in soil. Austenitic stainless steels are typically not well suited for high-chloride soils, anaerobic soils, or low pH soils. Studies have shown that soils with chloride contents as low as 500 ppm can lead to localized corrosion on stainless steels [32]. The damage induced by chlorides on stainless steel is typically pitting or crevice corrosion. Welds and the heat affected zone are especially vulnerable, as seen in Figure 9-1. These locations have been sensitized due to the heating and rapid cooling process. This makes them more susceptible to corrosion activity. Stainless steels are also susceptible to localized corrosion in the presence of oxygen concentration cells. Anaerobic conditions are also conducive to MIC activity, which can lead to the degradation of austenitic stainless steels. A soil pH of 4.5 and below is typically considered corrosive for austenitic stainless steels.

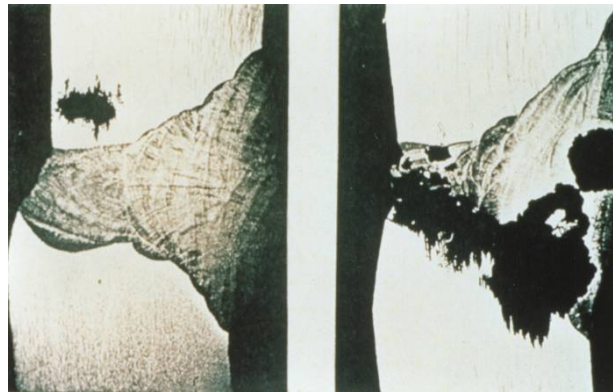


Figure 9-1
The pitting attack of the heat affect zone (left) and weld material (right) of a stainless steel alloy

Super austenitic stainless steels, alloys with less than six percent molybdenum and increased nickel and nitrogen levels, are more resistant to corrosion in soil. The increased level of chromium, molybdenum, and nitrogen increases the pitting resistance of the material. Super austenitic stainless steels offer higher corrosion resistance to general, local, and MIC attacks. Many are also suitable for high chloride environments.

9.3.1.2 Aluminum Alloys

Buried aluminum piping is commonly selected for condensate piping in plants when the iron content of the water is low. Aluminum alloys rely on an aluminum oxide film to protect the alloy from corrosion. The oxide film forms readily on aluminum and its alloys in the presence of water and oxygen, as the metal is quite reactive in many environments. Buried aluminum piping can be susceptible to damage from CP if the lines are over protected (that is, the cathodic potential is more negative than required). Cathodic reaction products will raise the pH at the external aluminum piping surface, which can result in loss of passivity and corrosion. Aluminum is also not suitable for service in soils with naturally high or low pH values. Soils that damage the passive film will likely lead to rapid corrosion of aluminum alloy in buried service. Aluminum bronze alloys are also common in service water piping. Dealloying of these alloys has been observed in the nuclear industry. In this case, it is the aluminum that is selectively removed from the metal matrix due to the interaction with the environment.

9.3.1.3 Copper Alloys

Copper alloys are also traditionally selected based on its service, such as service water [92]. Copper alloys can offer increased corrosion resistance compared to carbon steel. In historical experiments, copper alloys subjected to the same environments as carbon steel, generally had increased corrosion resistance [34]. However, copper alloys are not well suited for high sulfide environments [18]. Dezincification can also occur in copper alloys, mainly in alloys with greater than 15 percent zinc. This results a weaker metallic structure.

9.3.2 **Cast and Ductile Iron**

The corrosion behavior of cast and ductile iron pipe is very similar. The susceptibility to graphite corrosion can be different due to the more spherical grain structure of the ductile iron. However, both materials are subject to corrosion in soil. Table 9-3 shows an example of a common soil corrosivity rating index provided by ANSI/AWWA. Degradation of iron alloys in soils can be in the form of general corrosion, pitting, galvanic corrosion, MIC, or graphite corrosion

9.3.3 **Concrete Materials**

Concrete materials used for piping such as PCCP, RCP, and asbestos-cement are used typically for cooling water, service water, or drainage systems. The concrete can initially act as a protector of the steel components; however the interaction with the concrete can also complicate the steels interaction with the environment due to its shielding capabilities in some cases. Reinforced concrete systems can be subject to degradation to both the metallic and concrete component. Chloride environments of greater than 400 ppm can lead to the degradation of the metallic structure if it becomes exposed to the environment. High sulfate environments, typically greater than 2,000 ppm, can lead to the degradation of the concrete. Highly acidic environments can cause degradation as well. Soil stresses can also increase the rate of cracking and spalling of the concrete.

10

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A

LISTING OF KEY POINTS

The primary intent of the key technical points is to emphasize information that will allow individuals to take action for the benefit of their plant. The key technical point has an identifying icon, as shown below, to draw attention to it when quickly reviewing the report.



Key Technical Point
Targets information that will lead to improved data collection and analysis.

The following is a summary of the key points in this report. The listing restates each key point and provides reference to its location in the body of the report.

A.1 Key Technical Points



Key Technical Point 6.1
A knowledge based approach to selecting sampling locations is recommended when possible. This method combines background information such as site drawings and materials of construction with corrosion susceptibility to select areas likely to have corrosion issues.



Key Technical Point Section 6.1
When collecting samples from excavations, it is necessary to scrape away several inches of soil prior to collecting the sample. The edges of the excavation will have spent time exposed to the air prior to sampling.



Key Technical Point Section 6.1
Sampling is sometimes completed as a result of maintenance excavations, drilling, or other subsurface operations.



Key Technical Point 6.3
Locating underground structures accurately is essential to the soil sampling process. Conclusions drawn from a particular soil sample must be taken in the context of where it was collected in relation to the underground structure.



Key Technical Point Section 7.3
It is recommended to use freezer packs or gel packs to keep the soil samples cool during transportation, as using melting ice has the potential to contaminate a sample that was not properly sealed.



Key Technical Point Section 7.6.1

The soil resistivity for a sample should be measured both for an as-received sample and for a water-saturated sample. The saturated sample usually has the lowest resistivity for a given soil sample.



Key Technical Point Section 7.6.3.1

Sample exposure and contamination must be minimized during extraction to provide the most accurate results.



Key Technical Point Section 8

The interpretation of a given test result will be based on the standard selected/procedure developed to quantify that particular parameter.

B

LIST OF ACRONYMS

AASHTO	American Association of State Highway and Transportation Officials
AC	Alternating current
AMP	Aging Management Program
ANSI	American National Standards Institute
APB	Acid producing bacteria
APHIS	Animal and Plant Health Inspection Service
ASTM	American Society for Testing and Materials
AWWA	American Water Works Association
COC	Chain of custody
CLSM	Controlled Low Strength Material
CP	Cathodic protection
CSE	Copper/copper sulfate electrode
DC	Direct current
DOE	Department of Energy
DOT	Department of Transportation
ELL	Electromagnetic line location
EPA	Environmental Project Agency
EPRI	Electric Power Research Institute
EPS	Exopolysaccharide producing bacteria
FBE	Fusion bonded epoxy
GALL	Generic aging lessons learned
GPR	Ground penetrating radar
GPS	Global positioning system
IC	Ion chromatography
ICP/MS	Inductively coupled plasma/mass spectroscopy
ISO	International Organization for Standardization

List of Acronyms

MIC	Microbiologically influenced corrosion
MOB	Metal-oxidizing bacteria
MRB	Metal-reducing bacteria
NACE	National Association of Corrosion Engineers
NRC	Nuclear Regulatory Commission
NSIAC	Nuclear Strategic Issues Advisory Committee
NEI	Nuclear Energy Institute
NPP	Nuclear power plant
NUREG	United States Nuclear Regulatory Commission Regulatory Guidance
PCCP	Prestressed concrete cylinder pipe
PPM	Parts per million
PPQ	Plant protection quarantine
QA/QC	Quality assurance/quality control
RCP	Reinforced concrete pipe
rDNA	Ribosomal Deoxyribonucleic Acid
RFID	Radio-frequency identification
SCC	Stress corrosion cracking
SRB	Sulfate reducing bacteria
USCS	Unified Soil Classification System
USDA	United States Department of Agriculture

C

SPECIFIC PROCEDURES USED FOR SOIL CORROSIVITY TESTING

C.1 Soil Resistivity

C.1.1 Referenced Procedures(s)

ASTM G57-06 (2012) “Standard Test Method for Field Measurement of Soil Resistivity” [59]

One method for the field and laboratory testing for soil resistivity uses the soil box method. The equation for calculating the soil resistivity using the soil box method is presented below:

$$\rho = R \cdot A/a$$

Where:

ρ = Resistivity, ohm·in (ohm·cm)

R = Resistance, ohm

A = cross sectional area of the container perpendicular to the current flow, in² (cm²)

a = inner electrode spacing, in (cm)

The resistance can either be measured using a separate current source, ammeter and voltmeter, or by using a galvanometer/soil resistivity meter.

C.1.2 Equipment Required

Soil box method:

- Either:
 - Current source, ammeter, and voltmeter
 - Galvanometer
- Soil box
- Wiring to connect soil box and measurement apparatuses

C.1.3 Generic Procedure

1. Calibration/Preparation:
 - a. Calibrate the electrical measurement equipment using a variable resistor. Perform the calibration over multiple orders of magnitude.
 - b. If using a soil box, ensure that the soil box is completely clean of soil.

2. Soil box method:

- a. Either an as-received sample or a saturated sample can be used.
- b. Fill the soil box such that the compaction in the soil box is as close to the collected soil as possible. In general there should be no air pockets or large rocks.
- c. Connect the electrical measurement apparatus such that the current is passed through the two outer plates and the potential is measured across the two inner pins.
- d. Perform the measurements per the selected procedure.
- e. Perform the required calculations per the selected procedure.

C.1.4 Additional Consideration(s)

Soil resistivity measurements can vary from location to location, and also vary on the degree of compaction of an individual sample. Small changes in local soil composition and local moisture can change the soil resistivity. As a result, it is recommended to collect multiple samples or test a collected sample multiple times using a fresh sample if enough soil has been collected.

Due to seasonal variations and the dependency of soil moisture on soil resistivity, it is recommended to measure the soil resistivity of a saturated sample, as this sample would represent a worst case scenario for soil resistivity.

C.2 Redox Potential

C.2.1 Referenced Procedures(s)

ASTM G200-09 (2014) “Standard Test Method for Measurement of Oxidation-Reduction Potential (ORP) of Soil [61]

The redox potential is a measure of the tendency of a material, in this case soil, to accept electrons. In the case of soils, this is essentially a measure of the oxygen content in the soil. The measurement is made using a commercially available probe. After proper calibration the probe is inserted into a soil sample to determine the redox potential of the soil.

C.2.2 Equipment Required

- Oxidation reduction potential meter
- Oxidation reduction potential electrode
- Deionized water
- Inert plastic container

C.2.3 Generic Procedure

1. Calibrate the meter using a commercially available solution.
 - a. Zero the meter as per the selected standard.
 - b. If in the field, collect the desired soil sample.
 - c. There must be enough soil in the sample such that the probe is covered in soil in all directions.
2. Perform the measurement as per the selected procedure.
3. The most negative or most positive reading must be recorded.
4. At least three readings should be taken at different locations, such that the same soil is not included in the same reading.

C.2.4 Additional Consideration(s)

As stated previously the redox potential in soil is directly associated with the oxygen concentration in the soil. Disturbing the soil will change this concentration. Regardless the amount of disturbance of the soil during removal, transport, and sampling should be limited as best as possible.

C.3 Chemical Constituents

C.3.1 Referenced Procedures(s)

See Table 7-1.

C.3.1.1 Soluble Ion Extraction

Soluble ion extraction procedures can vary based on the type of analysis being completed and the requirements of that analysis. Many laboratories will offer in-house extraction as part of their services. If completing the process in-house there are a few general principles. The goal is to remove the soluble ions from the soil and create a solution that will be acceptable to perform the desired testing.

The first step of the process is to remove the ions to be measured by mixing the soil sample with deionized water to remove the soluble ions. This should be done by removing a specified mass of a soil sample and mix it with a set amount of deionized water. This process is known as dilution. Different tests require different levels of dilution. By knowing the level of dilution of the bulk sample a laboratory technician will know the additional amount of dilution required to complete a specific step.

The second step is to ensure that the soil and water sample is thoroughly mixed. This can be completed by using an agitator, stirring apparatus, or any other mixing method. The preferred methods are the ones that limit air exposure of the sample during mixing.

After the sample is thoroughly mixed, the sample will need to be filtered such that the maximum particle size remaining is acceptable to the test method to be used. This particle size will vary based on what analysis is to be completed. Check with the analytical test procedure to determine the maximum acceptable particle size. If using physical filters it is recommended to use a series of increasingly finer filters beginning with an opening size of approximately 2 mils (50 microns) or larger. This will aid in processing time as larger soil particulates will often clog the filter.

C.3.2 Equipment Required

- Plastic sealable bottle (500 mL-1,000 mL)
- Oscillating table/stirring plate with stir bar
- Balance
- Deionized water

C.3.3 Generic Procedure

1. Calibrate the balance using a traceable weight.
2. Create an approximately 5:1 ratio of water to soil by mass.
 - a. Weigh out the required amount of soil into the plastic bottle. Use a plastic bottle that limits the amount of free space in the container. This will minimize the amount of air that interacts with the sample.
 - b. The final amount of liquid required is between 16.9 oz (0.5 L) and 33.8 oz (1 L) to complete all of the tests listed in Table 7-1. Collect soil from different locations in the soil sample and ensure that any large rocks or plant matter are removed.
 - c. Add the desired amount of deionized water to the weighed soil.
3. Label each bottle with a sample identification.
4. Thoroughly mix the sample using either an oscillating table or stirring plate.
5. Allow the solution to mix for at least 12 hours.
6. Filter the solution down to the required maximum particle size. This will typically be 0.9 mils (22 microns) or less.
 - a. Begin with a coarser filter paper as larger particles will tend to clog finer filters.
 - b. Pressure or vacuum filtering is recommended especially for finer, high clay content soils.
7. Store the filtered sample in a refrigerator until testing.

C.3.3.1 Analysis Methods

There are several different analytical methods that can be used to test for the given chemical constituents. Each method has its advantages and disadvantages. The test method used can depend on time, availability of local labs and equipment, accuracy desired, and regulatory requirements. Table 7-1 lists the recommended methods for each ionic constituent or property.

A summary of the different methods used to determine the chemical constituents is presented in Section 7.6.3.2. This procedure includes additional quality control considerations when completing chemical constituent analysis.

C.3.3.2 Quality Control Considerations

Almost all tests will have some type of interference. The interference can be caused by a false positive registered by another species, a retardation of the indicator reaction caused by another species, or an inability to distinguish signatures between two species. If concerns exist regarding the quality of the data collected it may be necessary to screen samples for a wider chemistry range to examine for possible interference species. This would include heavy metals, other halides, and any other soluble species. Any standardized test will list the potential interference species. If using commercial equipment, manufacturers should list interference species for each test in the equipment manual.

C.4 Acidity (pH)

C.4.1 Referenced Procedures(s)

- ASTM D4972-13 “Standard Test Method for pH of Soils” [76]
- AASHTO T289-91 (2013) “Standard Test Method for Determining pH of Soil for Use in Corrosion Testing” [77]

C.4.2 Equipment Required

- pH meter
- pH electrode
- Deionized water
- Beaker
- Balance
- Stir rod

C.4.3 Generic Procedure

1. Calibrate the pH meter and electrode prior to use.
2. Create a solution consisting of approximately equal parts deionized water and soil. Stir the solution well to ensure complete mixture.
3. Measure the pH of the solution using the pH meter and electrode.

C.4.4 Additional Consideration(s)

ASTM G51-95 (2012) “Standard Test Method for Measuring pH of Soil for Use in Corrosion Testing” [78] is another available method. This method uses a reference calomel electrode and a glass electrode suitable for field use and a portable pH meter. After calibrating the pH apparatus using buffer solution, the pH of the soil is measured by inserting the electrodes into the soil. Measurements should be taken at three different locations for a given depth and averaged. It is recommended to measure the pH of the soil at the same depth of the buried metallic structure. This can be completed by collecting a sample of the soil from that depth by boring and then bringing the sample to the surface for testing. The ASTM G51-95 (2012) method assumes ideal conditions for the measurements, which are often not encountered in the field.

C.5 Total Acidity

C.5.1 Referenced Procedures(s)

Standard Method 2310B Acidity [79]

A common industry-accepted method for measuring total acidity is titration. A strong base, commonly NaOH, is used as the titrant. The titrant is added in small increments to an extracted and diluted sample to establish a titration curve or a color change to determine the total acidity. Some procedures provide guidelines for pre-treating samples to achieve more accurate results.

C.5.2 Equipment Required

- pH meter
- pH electrode
- Buret
- Beaker
- Stirring apparatus, preferably a stir plate/bar
- Commercial titration systems are also available
- Titrant and indicators
- Deionized water

C.5.3 Generic Procedure

1. Calibration/Preparation:
 - a. Calibrate the pH meter and electrode prior to use.
 - b. If using a commercial titration system, calibrate to the manufacturer standard.
 - c. Ensure all glassware is clean and free of damage.
2. Perform a sample extraction from the as-received soil. The precise degree of dilution must be determined for accurate calculation of total acidity.
3. Create the necessary titration solutions as per the selected procedure.

4. Carry out the titration as per the selected procedure.
5. Perform the necessary calculations to establish the total acidity. The common output is in unites of mgCaCO_3/L .

C.5.4 Additional Consideration(s)

The main acidic component in most surface waters and soils is CO_2 . This is why the output is mgCaCO_3/L as the carbonic acid in solution is neutralized to bicarbonate.

As with other tests, the lack of preservation of the sample is the biggest potential source of error in determining an accurate measurement for the total acidity of the compounds in the soil. This parameter is especially sensitive to proper handling protocols as CO_2 and other dissolved gases that can create weak acids can leave the solution over time. As a result, it is important to limit the exposure of the soil extract to air and limit the amount of sloshing of the solution. Solubility of gases increases with decreasing temperature, therefore it is also important to keep the solution at a low temperature.

C.6 Moisture Content

C.6.1 Referenced Procedures(s)

- ASTM D2216-10 “Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass” [45]
- AASHTO T 265-12 “Standard Method of Test for Laboratory Determination of Moisture Content of Soils” [80]
- EPA Method 1684 “Total, Fixed, and Volatile Solids in Water, Solids, and Biosolids” [81]

These tests utilize an oven to evaporate the moisture in the soil. By weighing the sample before and after drying the moisture content can be calculated. The specific method utilized must ensure that the sample is completely dried to accurately measure the moisture content.

C.6.2 Equipment Required

- Balance
- Drying device (that is, oven, hot plate)
- Weighing container
- Thermocouple or thermometer

C.6.3 Generic Procedure

1. Calibration/Preparation:
 - a. Ensure the balance is calibrated using a traceable weight.
 - b. Ensure the weighing container is completely clean.
 - c. Ensure the drying oven is calibrated using either a thermometer or thermocouple.

2. The soil utilized for this procedure should be the as-received or saturated soil sample. The key to accurate measurement is minimizing the amount of moisture loss or gain after the sample is collected in the field.
3. Perform the test according to the selected procedure. Best practices includes covering the sample during the drying process, because trapped moisture within the soil particles will escape suddenly under pressure and soil particles may be lost if not contained.
4. Even if it is not required by the procedure, it is recommended to re-dry the sample for a fraction of the original drying time. If the sample weight does not change more than within the specified tolerance after a second drying cycle, this is an additional indicator that the sample has completely dried. If the sample weight has decreased more than the tolerance provided in the standard utilized, after the initial re-drying, repeat this added step. Do not over dry and excessively scorch the soil.
5. Perform the required calculations according to the selected procedure.

C.6.4 Additional Consideration(s)

It is important to understand the output of the different procedure methods. ASTM D2216-10 and AASHTO T 256 present the percent moisture as a ratio of water to solids. EPA Method 1684 presents the percent moisture as a weight percentage. Put another way, if a sample is 50 percent water by weight, the AASHTO and ASTM method will report 100 percent moisture, while the EPA method will report 50 percent moisture content. The main significance of this difference relates to dilution of the sample for soil extraction tests. For example, if diluting the sample using a weight ratio, the weight percentage of water is the relevant value.

C.7 Microbial Content

C.7.1 Referenced Procedures(s)

NACE TM0194-2014 “Standard Test Method Field Monitoring of Bacterial Growth in Oil and Gas Systems” [82]

It is recommended to complete culture testing for aerobic, anaerobic, sulfate-reducing, acid-producing, and iron-reducing bacteria at a minimum. These tests can be done both in the field and in the lab. Almost all tests are conducted using a similar procedure. The process works by inoculating media vials using a serial dilution process. First a solution is created inserting a piece of the physical sample collected into a vial of a buffered saline solution. This creates a solution containing bacteria from the soil sample. A syringe is used to pull a sample of the solutions and the sample is then inoculated into a media solution that is conducive to growth for a given bacteria type. A media solution is a solution that has the necessary nutrients for bacteria growth. A sample is drawn from the inoculated media vial, and then inserted into another vial in order to dilute the solution. By pulling a portion of each vial and inserting it into the next vial, each vial becomes more diluted than the vial before it. The idea is that in order for bacteria to grow in the more diluted vials, there had to have been a greater concentration in the initial sample. If there are not enough bacteria injected into a given vial, the bacteria will not be able to have the required numbers to sustain growth. The dilution process is completed for each vial in the kit. For example, there may be five media vials for a given bacteria type, and each time the solution is drawn from one and inserted to the next vial the sample becomes diluted. The inoculated media

vials are then stored for several days to several weeks, and then they are examined for bacteria growth. All of the media vials will have an indicator, whether a potential color change or turbidity change, that will allow for the positive identification of bacteria growth. Each vial will represent an initial concentration range in the sample. Therefore, by examining which vials are positive for bacteria growth, the initial concentration of bacteria in the soil sample can be estimated.

C.7.2 Equipment Required

- It is recommended to use a commercially available testing kit. These kits can be used in the field and in the lab. It is recommended to use a kit that can analyze solid samples.
- Optional: Balance
- Permanent marker

C.7.3 Generic Procedure

1. Calibration/Preparation:
 - a. If using a balance, ensure it is calibrated using a traceable weight.
2. Select a kit that provides testing for the types of bacteria of interest.
3. Collect a small amount of soil and place the soil in a diluting solution. (This solution should come with the kit.) This can be completed using a tongue depressor or metal scoopula.
4. To calculate the amount of bacteria present per gram of material, weigh the mass of the sample collected.
5. Perform the inoculation according to the instructions of the selected kit.
6. Label the samples with the date and time of inoculation and the sample ID.
7. Allow the sample to incubate for the required time.
8. Visually examine the vials for bacteria growth.
9. Match the most diluted vial with a positive reading to the provided chart in the kit to determine the initial bacteria concentration.

C.8 Particle-Size Analysis

C.8.1 Referenced Procedures(s)

- ASTM D422-63(2007)e2 “Standard Test Method for Particle Analysis of Soils” [39]
- ISO/TS 17892-4 “Geotechnical investigation and testing-laboratory testing of soil – Part 4: Determination of particle size distribution” [74]

The test method uses two different phases of analysis to determine the different sizes of particles and corresponding percentage of the total soil mass. The first phase uses sieves to collect the larger particles. Sieves of different mesh sizes capture particles of decreasing size. The size of the particles left in the series of sieves with larger openings gives the amount of particles that are larger than the sieve size in which they are collected, but smaller than the preceding sieve sizes above it. For particles passing the number 200 sieve, a sedimentation process is used to classify

silts and clays. A hydrometer is used to measure the specific gravity of the particles in a water suspension. The rate at which the particles sink is a function of its diameter. Between the sieve and sediment analysis the particle distribution can be determined ranging from particles greater than three inches to one micrometer.

C.8.2 Equipment Required

- Balance
- Sieves
- Graduated Cylinder
- Hydrometer
- Stirring device (if the selected procedure requires it)
- Thermometer
- Dispersing Agent

C.8.3 Generic Procedure

1. Calibration/Preparation:
 - a. Ensure the balance is calibrated using traceable weights.
 - b. Inspect the sieves to ensure that there is no damage to the mesh.
 - c. Ensure the thermometer and hydrometer have been calibrated.
 - d. Inspect all other equipment for damage.
2. The minimum amount of soil required is typically over 1.1 lb (0.5 kg), however some procedures require more.
3. The soil must be appropriately dried such that particles can separate and pass through the required sieves.
4. Perform the analysis according to the selected procedure.
5. Perform the necessary calculations to determine the particle size distribution.
6. The common output for the data is the mass percentage finer for each specified size range of the test.

C.9 Bulk Density

C.9.1 Referenced Procedures(s)

ASTM D7263-09 “Standard Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens” [84]

There are multiple methods to measure the density of soil; however the simplest way is a measurement of the soil volume and dry weight. This can be completed on a cored sample, or a re-compacted sample. Use of a cored sample is preferred because the sample volume can be easily calculated.

C.9.2 Equipment Required

- Balance
- Drying oven
- Container for drying
- Shelby tube for coring
- Measuring equipment:
 - Tape
 - Ruler
 - Caliper
- Mold and compactor (if re-compacting)

C.9.3 Generic Procedure

1. Calibration/Preparation:
 - a. Calibrate the balance using a traceable weight.
 - b. Ensure the core is free of debris prior to collecting the soil sample.
2. Measure the dimensions of the collected sample, this should be equal to the inner dimensions of the core.
3. Measure the weight of the Shelby tube sampler.
4. After the volume is measured, weigh the as-collected soil in a container.
5. Dry the soil such that all moisture has been removed within the drying tolerance of the procedure. The drying procedure used for determining soil moisture can be used.
6. As with the soil moisture procedure it is recommended to re-dry the soil for a fraction of the original dry time to ensure drying has been completed. This is confirmed by a weight reduction within the specified tolerance between the first and second drying.
7. Calculate the bulk density from the dry soil weight and soil volume.

The recommended procedure follows a similar process and also provides a methodology for wax coated samples.

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