

Standard Recommended Practice

Monitoring Corrosion in Oil and Gas Production with Iron Counts

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Foreword

This standard recommended practice describes the use of iron counts as a corrosion-monitoring method and some common problems encountered when using this method. For several years, NACE Task Group T-1C-7 on Iron Determination examined the problems and successes experienced by oil-producing companies and service companies using iron counts as a corrosion monitor and determined that iron counts on wellhead samples can provide information on the existence of downhole corrosion and the effectiveness of inhibitor treatments. Iron counts can also give information on the corrosion activity in flowlines in waterflood systems and oil-production operations. This standard is a guide for those designing corrosion-monitoring programs as well as those carrying out the programs in the field.

This standard was originally prepared in 1992 by Task Group T-1C-7, a component of Unit Committee T-1C on Detection of Corrosion in Oilfield Equipment. T-1C was combined with Unit Committee T-1D on Corrosion Monitoring and Control of Corrosion Environments in Petroleum Production Operations. This standard was revised by Task Group T-1D-55 in 1998, and is issued by NACE International under the auspices of Group Committee T-1 on Corrosion Control in Petroleum Production.

In NACE standards, the terms *shall*, *must*, *should*, and *may* are used in accordance with the definitions of these terms in the NACE Publications Style Manual, 3rd. ed., Paragraph 8.4.1.8. *Shall* and *must* are used to state mandatory requirements. *Should* is used to state that which is considered good and is recommended but is not absolutely mandatory. *May* is used to state that which is considered optional.

**NACE International
Standard
Recommended Practice**

**Monitoring Corrosion in Oil and
Gas Production with Iron Counts**

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Section 1: General

1.1 The anomalies experienced when using iron counts as a monitor for corrosion result mostly from the varying, usually uncontrollable, conditions found in almost every production system. Because the term *iron count* refers to the concentration of iron dissolved in the water expressed as milligrams per liter (mg/L) or ppm (mg/kg), those monitoring corrosion using iron counts must specify whether the iron content is based on the total fluid produced and whether the iron is reported as soluble iron, ferrous iron, or total iron. The exact method of sampling and sample treatment required to separate and analyze for ferrous, ferric, soluble, and total iron content of a water sample is described in the analytical procedures cited in the Reference section. If techniques are employed to analyze for the individual species of iron, the final report must indicate the form of iron being reported. If only the typical total acid-soluble iron content is determined, the final report should indicate that the result is "total iron." The usual oilfield iron count is total iron content of an acid-treated sample. In order to use iron counts to monitor corrosion trends, the same species must be determined consistently for a given sampling point in a system. For comparison of systems producing varying amounts of water, a more meaningful tool is the iron production rate that takes into consideration the water flow rate at the time of sampling. The iron count is converted to an iron production rate, usually expressed in kilograms of iron per day (kg/day [lb/day]).

1.1.1 The analyst should evaluate other available test methods for iron content to determine the most suitable method regarding detection limits, accuracy, precision, and interferences. Specific analytical procedures¹⁻³ are adequately covered in other documents and are not addressed in this standard.

1.1.2 For the purposes of this standard, it is presumed that iron counts will be run on aqueous samples. Analysis of hydrocarbon samples for iron content is possible and the technique is practiced by some corrosion engineers. One suggested technique for "iron in oil" is described by Rydell and Rodewald.¹

1.2 The mechanical arrangement, physical conditions, and chemical environment in almost every system or part of a system must be evaluated under comparable conditions before the iron content of each sample can be correctly interpreted. The iron counts measured are not of any value if these variables are not considered in the interpretation.

1.3 Monitoring corrosion by the use of iron counts can be done easily, inexpensively, and quickly in the field. Iron production rates, unlike test specimen corrosion rates, can give some indication of corrosion upstream or downhole from the sampling point. Iron counts are useful when surface-monitoring devices, such as test specimens, may not reflect downhole conditions, such as when paraffin forms on test specimens. The principal reason for the historical popularity of iron counts as a standalone corrosion-monitoring method is that in many small production facilities other forms of monitoring facilities have not been installed. However, iron count measurements should be combined with other corrosion-monitoring techniques whenever possible.

1.4 Generally, iron counts from fluids containing dissolved sulfides or dissolved oxygen are not reliable because of precipitation of iron sulfide in the system. The use of iron counts as a corrosion-monitoring tool must be validated for each specific case.

1.4.1 Proper safety precautions are required when dealing with sour systems.⁴

Section 2: Sampling

2.1. Iron counts are used for monitoring the iron content of the water phase at different points in a flowing system, thereby indirectly indicating the effectiveness of corrosion control. The results are useful if they are representative of the iron content of the flowing fluid. Solids, including old or fresh corrosion products in the form of iron compounds, can accumulate in a sampling point or trap under static conditions. Corrosion of the sample point may also contribute to the iron count.

2.1.1 The sample point in an oilfield system usually consists of a tee or nipple and valve welded onto a pipeline or vessel. The fitting may not be used exclusively for sampling; rather, many access fittings are originally installed to monitor pressure or other parameters in the system. In horizontal lines carrying water and hydrocarbon in stratified layers, the ideal location for sample collection is on the bottom of the line. If the flow in a system is annular, a representative sample can be obtained from a sample point at any position along the flowing stream. It is important to obtain a representative sample of the aqueous phase, even if this requires the use of special sample access fittings. To obtain a representative sample of the flowing water, it is necessary to blow down the sample fitting to remove any accumulated solids and stagnant water before obtaining a sample for analysis. The following sampling procedure shall be used to obtain samples that are representative of the flowing stream.

2.1.2 After the sample fitting is purged to a suitable waste container, conditions are correct for obtaining a reasonably representative sample of fluid for iron analysis.

2.1.2.1 If a steady flow of liquids exists in the system because of turbulent flow or a relatively high volume of liquids passing through the system, a sample shall be drawn directly into a suitable sample container made of corrosion-resistant or iron-free materials. The container may be a glass or plastic bottle if the system pressure permits safe collection of the sample. After purging the sample line, and while obtaining the desired sample, the valve on the sample line shall not be adjusted to either increase or decrease the flow. Any physical adjustments that disrupt the flow rate may dislodge iron precipitates from the sample point and cause them to enter the sample container.

2.1.2.2 If the flow in a low-pressure system is very slow or if small quantities of free water are present, a sample shall be collected over an extended period of time as described in Paragraph 2.1.3. This can be easily determined by observing the presence or absence of free water in a quickly obtained sample collected from a system in a glass or plastic container.

2.1.3 The sampling time period must be extended if sufficient aqueous fluid for analysis is not readily obtained. A corrosion-resistant sample receiver with a pressure rating consistent with the maximum system pressure should be installed at the six o'clock position of the line (see Figure 1). Caution should be used to avoid galvanic attack between the sample receiver and the system by use of an insulating flange between dissimilar materials of construction. The container should be suitably cleaned and free of any foreign matter. The sample fitting must have been purged as described in Paragraph 2.1.1 prior to installation of the sample receiver. The bottom valve must remain closed and both the valve on the sample fitting and the top of the sample receiver must remain open during the sample collection period.

2.1.4 Sufficient time must be allowed for water to collect in the sample receiver. In some systems this may be accomplished in a few minutes, while it may require from 12 to 24 hours in gas well flow lines when intermittent slugs of water are produced.

2.1.5 The sample receiver shall be isolated from the system by closing both the fitting and top receiver valves. The sample receiver shall be removed from the line. Care should be taken to bleed pressure slowly when the sample receiver is moved from the sample access fitting. If the system is sour and the receiver fittings contact H₂S gas, the precautions detailed in Appendix A must be followed.

2.1.5.1 A sample of the collected water may be either transferred from the receiver to a glass or plastic container for transport to a laboratory or drawn directly from the receiver to a container for field analysis. If the sample is not analyzed immediately, to retain all iron in solution, hydrochloric acid shall be added to the sample container as outlined in Paragraph 2.1.10. Acid addition dissolves suspended iron particles, which can result in artificially high iron counts.

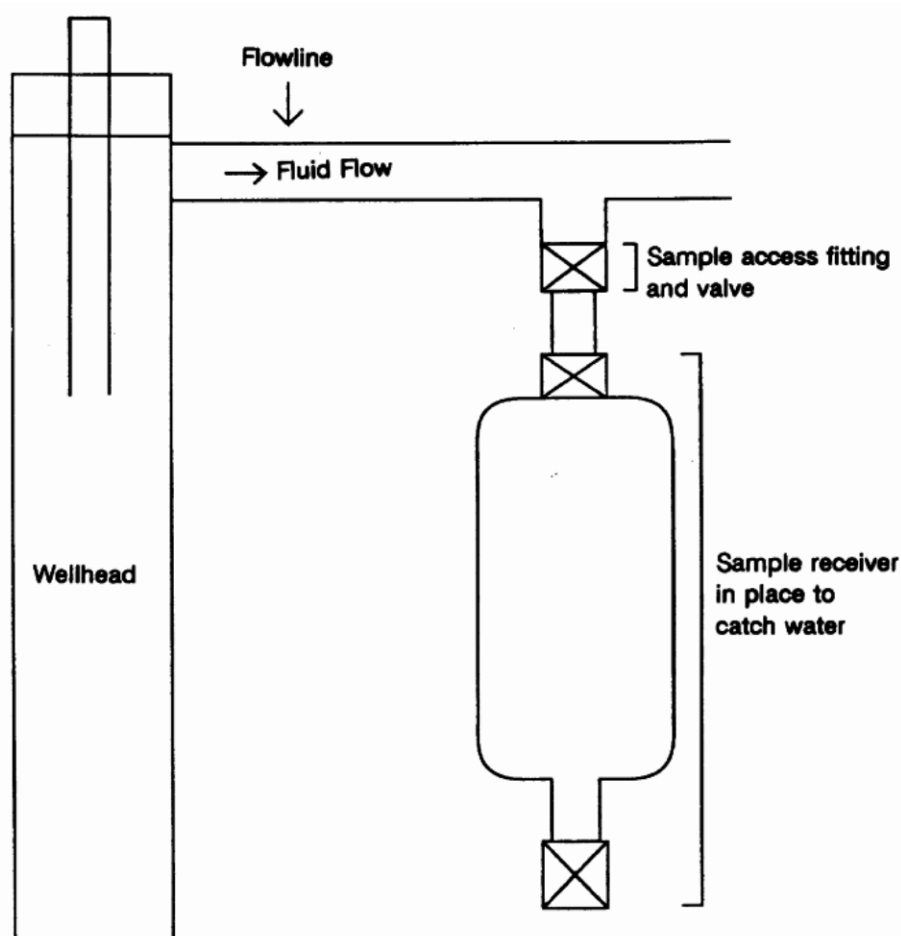


Figure 1
Typical Double-Ended Sample Receiver and Connection on the Bottom of a Flowline

2.1.6 Iron counts may also be obtained on water samples from waterflood or other water systems. The flowing stream often carries solids such as sand or silt, corrosion products, or microbiologically generated material, which tend to accumulate at the bottom of the line. Light material such as oil, gas, and some types of microbiologically generated material can accumulate in the top of the line. In such cases, side-of-line sampling may be advantageous as an alternative to bottom-of-line sampling, if iron counts representative of the bulk flowing stream are required.

2.1.7 A sample of emulsion with no free water requires treatment by heat, centrifuge, or use of chemicals to break the emulsion. It is generally accepted that free water has the same mineral content as emulsified water; therefore, only water sufficient to run the analysis need be separated.

2.1.8 Dissolved iron has a strong tendency to precipitate as a hydroxide, sulfide, or carbonate in an aqueous system, depending on the pH and the corrodent present. Oxygen can oxidize ferrous salts to less-soluble ferric salts, increasing the level of solids suspended or deposited even when other corrodents are present. A freshly formed precipitate may be carried by high fluid velocity from its origin to a less turbulent point in the system, where conditions such as reduced temperature or pressure may cause coagulation or flocculation. Because precipitation removes the iron from solution, the amount of dissolved iron may be lower at points further downstream. In such cases, a lower iron count might not necessarily indicate a reduced level of corrosion.

2.1.9 Increases in sulfide concentration resulting from an increased level of sulfate-reducing bacterial activity can reduce the iron count by the deposition of insoluble iron sulfide. The fluid temperature can vary significantly during the day, especially if the piping is not insulated or buried and is in desert climates; this also can affect the level of microbiological activity in the system. If the precipitated iron settles near the sample point, opening the sample valve can sweep precipitated material into the sampler. This can result in measuring an iron count that is not representative of the flowing stream. In critical tests, the aqueous sample should be filtered to remove precipitated iron particles prior to adding acid, thus ensuring that only soluble iron is measured in the analytical procedure.

2.1.10 Acid must be added to the sample to hold the dissolved iron in solution and preserve the sample for the analyst. The sample container should resist corrosion by the acid-treated solution. Acid is frequently added prior to drawing the sample from the system or prior to transfer from the double-valved sample receiver. Reagent grade hydrochloric acid should be used unless specific conditions dictate use of another acid.

2.1.10.1 Ten drops of 10% acid are recommended for a 100-mL (3.4-oz fluid) sample. If the sample contains water in which precipitated iron particles are suspended, this acid treatment dissolves the particles.

2.2 For a given corrosion-monitoring program, the sampling procedure should be stipulated and followed.

Section 3: Analysis

3.1 Preparation of Sample

3.1.1 The sample should be oil-free and solids-free for any of the usual analytical methods.

3.1.2 Water Separation

3.1.2.1 When a sample is found to be completely emulsified with no free water, one of the following methods may be used to separate free water:

3.1.2.1.1 The sample may be heated to break the emulsion.

3.1.2.1.2 A portion of the emulsion may be separated by centrifuging to obtain sufficient water for the particular analytical procedure selected.

3.1.2.1.3 A small quantity of iron-free demulsifier may be added to a sample followed by heat and vigorous agitation and centrifugation to hasten water separation.

3.2 Analytical Methods

3.2.1 Several methods for iron analysis found in the publications listed in the Reference section may be used with this standard. The following methods are subject to possible interferences; the literature references should be consulted.¹⁻³

3.2.1.1 The most often-used method is the orthophenanthroline colorimetric method; however, other methods mentioned in this standard are also widely used. Colorimetric methods have been adapted for field use by several companies that have developed compact portable kits for immediate analysis at the sample site. The results obtained using the field kits and instructions provided have been found reliable for determination of iron count.

3.2.1.2 An atomic absorption spectrophotometric method is often used when samples are analyzed in a laboratory.

3.2.1.3 Dichromate and ethylenediaminetetraacetic acid (EDTA) titration are two volumetric methods that have been used in laboratory analysis (API⁽¹⁾ RP 45⁵).

⁽¹⁾ American Petroleum Institute (API), 1220 L St. NW, Washington, DC 20036.

Section 4: Interpretation

4.1 Iron counts may be considered a good monitoring method only when a number of iron counts have been gathered from the same sample point in the same manner and analyzed by the same analytical method.⁶⁻⁹ Variations in flow rate in a given system can lead to fluctuation in iron counts. Therefore, production or flow rates should be reported for use in interpreting iron count data. The use of iron count (mg/L) data is only relevant to changes in corrosion activity if the flow rate in a system is constant. If the flow rate varies, the iron count shall be converted to an iron production rate (kg/day [lb/day]) to detect changes in the system.

4.2 Flowlines and Pipelines

4.2.1 Iron counts are used for monitoring corrosion at different points in a flowing system. The results indicate the effectiveness of the corrosion control system; results from different points can only be compared usefully if they are representative of the

iron content of the flowing fluid. Solids, including old or fresh corrosion products in the form of iron compounds, can accumulate in a sampling point under static conditions. The sampling procedure described in Section 2 should be used to obtain samples that are representative of the flowing stream.

4.2.2 High iron counts in wells with low water production are not necessarily indicative of severe corrosion; low iron counts in wells with high water production are not necessarily indicative of mild corrosion. Water production rate together with the iron count can provide the iron production rate of the system, which is indicative of the corrosion activity. The formulas for converting iron count to iron production rate in kilograms or pounds of iron removed per day are shown in Equations (1) and (2).

$$\text{kilograms of iron / day} = (\text{iron count, mg / L}) \left(\frac{1 \text{ g}}{1,000 \text{ mg}} \right) \left(\frac{1 \text{ kg}}{1,000 \text{ g}} \right) \left(\frac{1,000 \text{ L}}{\text{m}^3} \right) (\text{m}^3 / \text{day}) = \frac{(\text{mg / L}) (\text{m}^3 / \text{day})}{1,000} \quad (1)$$

or

$$\begin{aligned} \text{pounds of iron / day} &= (\text{iron count, mg / L}) \left(159 \frac{\text{L}}{\text{bbl}} \right) \left(\frac{1 \text{ g}}{1,000 \text{ mg}} \right) \left(\frac{1 \text{ lb}}{453.6 \text{ g}} \right) (\text{water production, } \frac{\text{bbl}}{\text{day}}) \\ &= 0.00035 (\text{mg / L iron}) (\text{bbl / day}) \end{aligned} \quad (2)$$

Figure 2 is a nomograph showing the amount of iron removed per day based on iron count and amount of produced water.

4.3 Correlation with Corrosion

4.3.1 Iron counts are a measure of the iron contained in the aqueous fluid at the point of sampling. Neither iron counts nor iron production rates predict the location or type of corrosion in the sampled system. Trends or changes in iron production rates are used to detect changes in corrosion rates or to monitor inhibition programs.

4.3.1.1 An iron production rate increase is a warning of an increased corrosion rate. Low iron production is not a guarantee that a system is under control because pitting may be active even when iron counts are only 2 or 3 mg/L. Supplemental corrosion control should be considered for internally coated piping if even comparatively low iron production rates are observed.

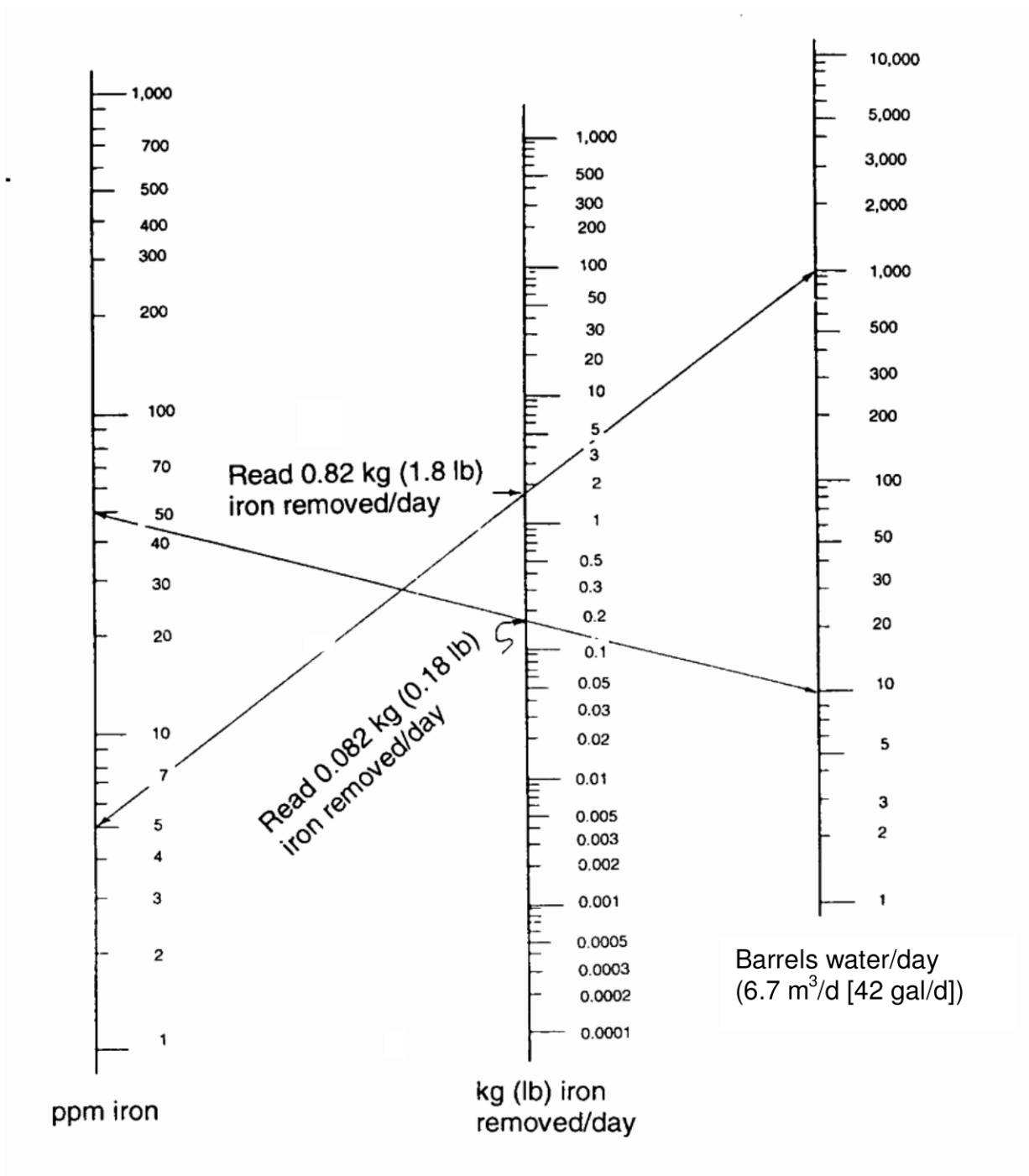


FIGURE 2

Nomograph Showing Kilograms (Pounds)⁽²⁾ of Iron Lost per Day in a Water Distribution System
Iron-loss values are found by relating measured values of iron concentration in the water to flow rate through the system. (Reprinted from NACE Publication TPC #5 [latest revision], Corrosion Control in Petroleum Production [Houston, TX: NACE]).

⁽²⁾ Metric conversions 1 lb = 0.454 kg
 1 bbl/d = 159 L/d = 0.159 m³/d

4.3.1.2 Corrosion of steel may produce other ions besides iron. Analyses of waters for manganese have been used to indicate that the iron results from the corrosion of steel.¹⁰ The concentration of manganese in iron alloys used in oilfield downhole equipment is typically 0.5 to 1.5%. Therefore, the supposition is that the ratio of manganese to iron in produced water should be about 1:100 if all the iron and manganese result from corrosion and no precipitation has occurred from the water. If the iron content of a liquid sample is much more than one hundred times the manganese content, extraneous or noncorrosion-related iron may be present in the formation water. A manganese content greater than about 1% of the iron content suggests that iron has deposited as scale, or is present in the hydrocarbon phase, or that manganese is produced from the formation. If the iron is deposited as a scale, the iron count would indicate an erroneously low corrosion rate. There is no correlation between manganese count and pitting. Use of manganese analyses is not documented; such usage must be evaluated on a case-by-case basis.

4.4 Analysis of Data

4.4.1 Presence of Background Iron Content of Produced Water

Some produced waters contain naturally occurring dissolved iron. This iron is detected when running iron counts in production systems and can be mistaken for iron produced by corrosion. The presence of iron in produced water must be viewed along with the other indicators of corrosion to determine whether iron count values are significant. The probable occurrence of corrosion should always be confirmed by equipment inspection, downhole caliper surveys, and review of failure records before establishing parameters for using iron counts as an indicator of corrosion.

4.4.2 Contamination of Water

4.4.2.1 Acidizing treatments in oil wells can result in a temporary or short-term increase in the formation water iron count. Acidizing can remove the protective films of corrosion product

and inhibitor on tubing, leaving a fresh metal surface that can corrode at a high rate. The dissolution of corrosion products can result in high iron counts that are not necessarily an indication of a short-term increase in the corrosion rate. Following acid treatments, the iron counts should return to normal levels within a few days, although in rare instances it can take up to several months for iron levels to return to normal.

4.4.2.2 Produced-water iron counts may be high for a period of time immediately following a shut-in period. If this occurs repeatedly, wells in the field should be slug treated with corrosion inhibitor before any anticipated shut-in periods or immediately after the wells have been shut in, in accordance with the type of treatment. After a shut-in period, if iron counts do not return to normal levels, then a remedial course of action (e.g., a well bore cleanout followed by treatment with a chemical corrosion inhibitor) should be considered.

4.4.3 Presentation of Data

Iron counts converted to iron production rates are used to monitor corrosion trends in production systems. These trends can warn of increased corrosion caused by increasing fluid corrosiveness or demonstrate the success (or failure) of a corrosion control program. Because a single iron count contains little information concerning corrosion in a system, iron production rate data should be accumulated over a period of time. A typical example of presentation of iron production rate data is shown in Figure 3.

4.4.4 Relation of Iron Count to Corrosion Rate

Actual corrosion rates can only be correlated with iron production rates in special circumstances. Both location and type of corrosion are system-dependent. In some special cases, iron count data can be used in conjunction with other system parameters to calculate a corrosion rate. One such case is the COPRA (Corrosion Rate—Production Rate) Correlation.¹¹ Use of such methods can be helpful in interpreting iron counts, but their suitability for use must be demonstrated on a case-by-case basis.

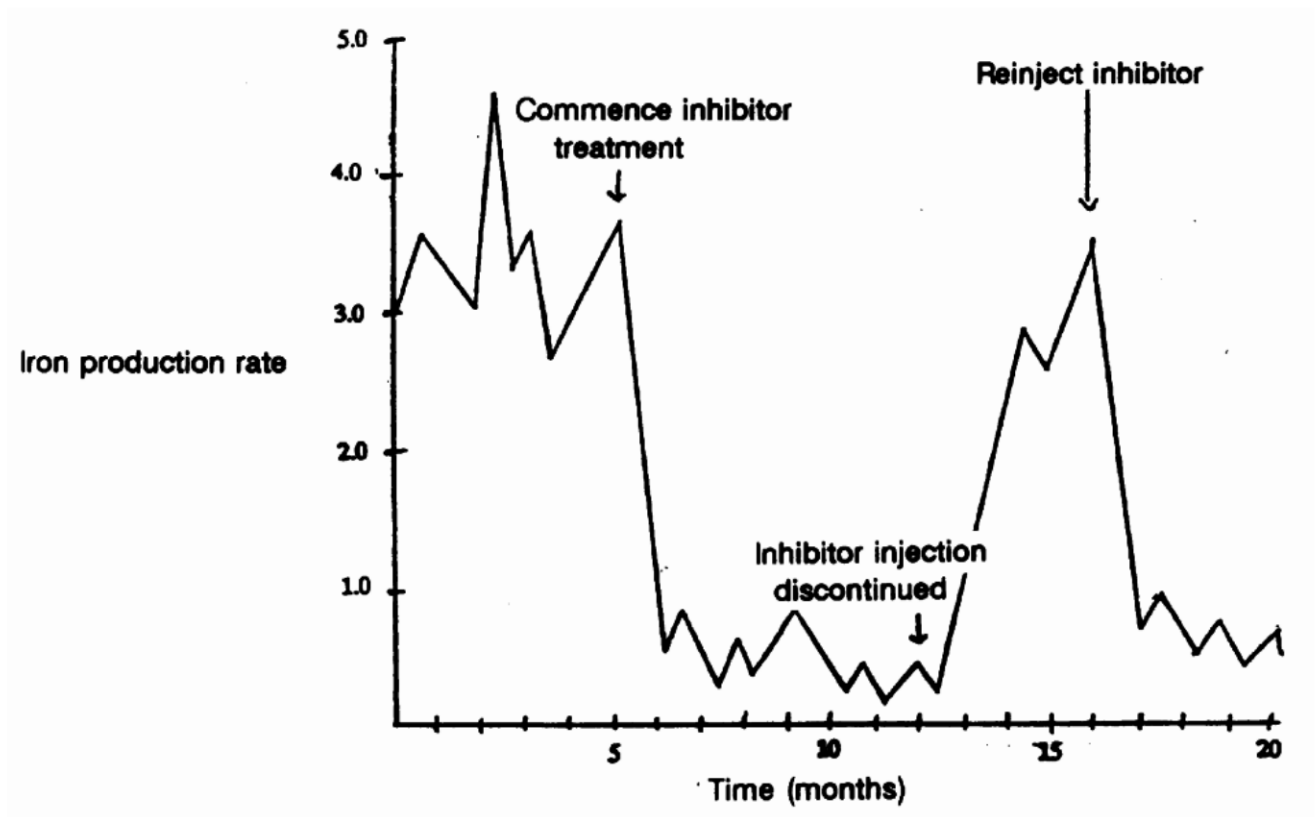


FIGURE 3
Graphical Presentation of Iron Production Rate Vs. Time Plus Pertinent Operating Information

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⁽³⁾ American Society for Testing and Materials (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

Appendix A

Safety Considerations When Handling H₂S

H₂S is perhaps responsible for more industrial poisoning accidents than any other single chemical. A number of these accidents have been fatal. H₂S must be handled with caution, and any experiments using it must be planned carefully. The maximum allowable concentration in the air for an eight-hour workday is 5 to 15 parts per million (ppm) depending on country and regulation, well above the level detectable by smell.¹² However, the olfactory nerves can become deadened to the odor after exposure of 2 to 15 minutes, depending on concentration, so that odor is not a reliable alarm system.

Briefly, the following are some of the human physiological reactions to various concentrations of H₂S. Exposure to concentrations in the range of 150 to 200 ppm for prolonged periods may cause edema of the lungs. Nausea, stomach distress, belching, coughing, headache, dizziness, and blistering are signs and symptoms of poisoning in this range of concentration. Pulmonary complications, such as pneumonia, are strong possibilities from such exposure. At 500 ppm, unconsciousness usually occurs within 30 minutes and results in acute toxic reactions. In the 700- to 1,000-ppm range, unconsciousness may occur in less than 15 minutes and death within 30 minutes. At concentrations above 1,000 ppm, a single lungful may result in instantaneous unconsciousness, with death quickly following due to complete respiratory failure and cardiac arrest.

Additional information on the toxicity of H₂S can be obtained by consulting the Material Safety Data Sheet provided by the manufacturer or distributor and from consulting sources such as *Dangerous Properties of Industrial Materials* by N. Irving Sax,¹³ *Documentation of the Threshold Limit Values*,¹⁴ and the *NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards*.¹⁵

Fire and Explosion Hazards

H₂S is a flammable gas, yielding poisonous sulfur dioxide as a combustion product. In addition, its explosive limits range from 4.0 to 46% in air. Appropriate precautions shall be taken to prevent these hazards from developing.

Experimental Suggestions

All tests shall be performed in a hood with adequate ventilation to exhaust all H₂S. The H₂S flow rates shall be kept low to minimize the quantity exhausted. A 10% caustic absorbent solution for effluent gas can be used to further minimize the quantity of H₂S gas exhausted. This solution will need periodic replenishment. Provision should be made to prevent backflow of the caustic solution into the test vessel if the H₂S flow is interrupted. Suitable safety equipment must be used when working with H₂S.

Particular attention should be given to the output pressure on the pressure regulators because the downstream pressure frequently rises as corrosion product, debris, and other obstructions accumulate and interfere with regulation at low flow rates. Gas cylinders shall be securely fastened to prevent tipping and breakage of the cylinder head. Because H₂S is in liquid form in the cylinders, the consumption of the contents should be measured by weighing the cylinder. The pressure gauge on the cylinder should also be checked frequently, because relatively little time will elapse after the last liquid evaporates until the pressure drops from 1.71 MPa (250 psi) to atmospheric pressure. The cylinder should be replaced by the time it reaches 0.52 to 0.69 MPa (75 to 100 psi) because the regulator control may become erratic. Flow should not be allowed to stop without closing a valve or disconnecting the tubing at the test vessel because the solution will continue to absorb H₂S and move upstream into the flowline, regulator, and even the cylinder. A check valve in the line should prevent the problem if the valve works properly. However, if such an accident occurs, the remaining H₂S shall be vented as rapidly and safely as possible, and the manufacturer shall be notified so that the cylinder can receive special attention.

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