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**Corrosion of metals and alloys —
Corrosion and fouling in industrial
cooling water systems —**

Part 1:
**Guidelines for conducting pilot-scale
evaluation of corrosion and fouling
control additives for open recirculating
cooling water systems**

*Corrosion des métaux et alliages — Corrosion et entartrage des circuits
de refroidissement à eau industriels —*

*Partie 1: Lignes directrices pour l'évaluation pilote des additifs
anticorrosion et antitartre pour circuits de refroidissement à eau à
recirculation ouverts*



Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16784-1 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

ISO 16784 consists of the following parts, under the general title *Corrosion of metals and alloys — Corrosion and fouling in industrial cooling water systems*:

- *Part 1: Guidelines for conducting pilot-scale evaluation of corrosion and fouling control additives for open recirculating cooling water systems*
- *Part 2: Evaluation of the performance of cooling water treatment programmes using a pilot-scale test rig*

Introduction

Environmental requirements, water shortages, and business pressures have forced industrial plants and power stations to operate with longer production runs, reduced maintenance outages, fewer operating personnel, and increased stress on cooling water systems. Similarly, commercial refrigeration (heating, ventilating, and air conditioning [HVAC]) systems have experienced increased heat loads and requirements for a long-term, continuous, cooling water supply to computer facilities, large retail establishments, campuses, and office complexes.

Under these increasingly severe conditions, cooling water chemical treatment programmes are expected to maintain optimum operating efficiency and, at the same time, protect the economic life of the equipment by inhibiting corrosion, mineral scaling, microbiological fouling, and miscellaneous deposition on heat-transfer surfaces.

Cooling system design and operating characteristics vary widely, within individual plants, from site to site, and worldwide. Thus, selection and optimization of water treatment programmes must be a site-specific process. In most systems, optimized cooling water chemical treatment is the key to successful long-term operations. The subject of this part of ISO 16784 is, therefore, the establishment of criteria for the pilot-scale evaluation of the performance of cooling water additives under field-specific operating conditions.

This part of ISO 16784 is intended for use by cooling system owners/operators, water treatment companies and others who must evaluate the performance of cooling water additives under field-specific operating conditions.

This part of ISO 16784 was developed on the basis of NACE RP0300 [4].

Corrosion of metals and alloys — Corrosion and fouling in industrial cooling water systems —

Part 1: Guidelines for conducting pilot-scale evaluation of corrosion and fouling control additives for open recirculating cooling water systems

1 Scope

This part of ISO 16784 applies to corrosion and fouling in industrial cooling water systems.

This part of ISO 16784 covers the criteria that must be defined and implemented in a pilot-scale testing programme to select water treatment programmes for use in specific recirculating cooling water systems.

This part of ISO 16784 covers only open recirculating cooling water systems. Closed cooling systems and once-through cooling water systems are specifically excluded.

This part of ISO 16784 applies only to systems incorporating shell-and-tube heat exchangers with standard uncoated smooth tubes and cooling water on the tube side. Heat exchangers with shell-side water, plate and frame and/or spiral heat exchangers, and other heat exchange devices are specifically excluded. However, when the test conditions are properly set up to model the surface temperature and shear stress in more complex heat-transfer devices, the test results may predict what may occur in an operating heat exchanger of that design.

The test criteria established in this part of ISO 16784 are not intended to govern the type of bench and pilot-scale testing normally carried out by water treatment companies as part of their proprietary product-development programmes. However, water treatment companies may choose to use the criteria in this part of ISO 16784 as guidelines in the development of their own product-development test procedures.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8044:1999, *Corrosion of metals and alloys — Basic terms and definitions*

ISO 16784-2, *Corrosion of metals and alloys — Corrosion and fouling in industrial cooling water systems — Part 2: Evaluation of the performance of cooling water treatment programmes using a pilot-scale test rig*

3 Terms, definitions, symbols and abbreviated terms

For the purposes of this document, the terms and definitions given in ISO 8044:1999 and the following abbreviations and symbols apply.

ASTM: ASTM International

BOD: Biological oxygen demand

COD: Chemical oxygen demand

HVAC: Heating, ventilating, and air conditioning

LPR: Linear polarization resistance

MIC: Microbiologically influenced corrosion

NACE: NACE International

PVC: Polyvinyl chloride

s/V ratio: Surface-to-volume ratio

UNS: Unified Numbering System

4 Types of testing

4.1 Laboratory and off-site testing

4.1.1 Laboratory testing, or testing at alternative off-site locations, may in some cases be necessary for selecting cooling water chemical treatment programmes. This type of testing could be used for new construction start-up programmes, when operating systems are not available, or for evaluating alternative treatment programmes. In such cases, the evaluation should include site-specific design criteria and environmental regulations that affect the cooling water system. Site-specific water supplies should be used whenever possible. All criteria in this part of ISO 16784 relating to water compositions, test unit configuration, heat exchanger design, and operating conditions should be followed insofar as possible.

4.1.2 No laboratory or off-site testing programme can completely duplicate plant conditions. Site-specific factors, such as process leaks, microbiological growth, corrosion products, airborne contamination, etc., may affect the operation of cooling water systems and the performance of chemical treatment programmes in ways that override the results of laboratory or off-site testing programmes.

4.2 On-site testing

4.2.1 Whenever possible, water treatment programmes should be evaluated on site, using plant water supplies and actual design and operating conditions, particularly those that cannot be duplicated in the laboratory. Criteria for these effects are discussed in 9.1.2.

4.2.2 Specific attention shall be given to site-specific rules and environmental regulations that may affect the types of chemical products that can be used, the allowable amount and composition of blowdown water, and air quality regulations affecting cooling tower discharge.

4.3 On-line testing

Whenever possible, all off-site, laboratory, and on-site pilot-scale testing should be validated by monitoring actual performance results on-line. Pilot units can be adapted for on-line work by using a sidestream from the plant circulating cooling water as feedwater, bypassing the pilot unit cooling tower. Such on-line testing serves to validate the off-line/laboratory tests. Cooling systems may be evaluated on-line; however, the data collected will be the result of the combination of any existing treatment and all additional chemicals that were added for the evaluation period. On-line testing in this way can be useful for optimizing the treatment programme to meet specific plant requirements. For example, small quantities of a treatment chemical may be added just

ahead of the test heat exchanger to measure the effects of increasing additive dosage, or the possible synergistic effects of a new chemical added to the existing treatment programme.

5 Test unit design parameters

5.1 General

Careful evaluation of the mechanical design and operation of each cooling water system is a necessary prerequisite for designing a pilot-scale water treatment product-evaluation programme. It may not be practical to simulate a specific critical plant heat load or water flow pattern exactly. Contamination in a pilot cooling tower may not develop in the same way as in the plant systems; compromises may therefore be necessary. In all such cases, plant design and operations must be followed as closely as possible, and deviations must be noted in the test reports.

5.2 Construction materials

5.2.1 Cooling towers

5.2.1.1 Small cooling tower basins may be made of uncoated, plastic-coated, galvanized low-carbon steel, or stainless steel. Large tower basins are usually concrete. Splash fill may be wood, ceramic, or plastic. It is not important that the pilot cooling tower duplicates the design of the plant towers. However, if the plant system contains galvanized steel, galvanized steel should be included as a non-heat-transfer test material in the pilot system.

5.2.2 Special requirements for film fill

5.2.2.1 If the plant cooling towers contain film fill, a section of this fill (if available) should be used in the pilot tower. Film fill consists of closely packed layers of lightweight plastic material, normally PVC, arranged in a honeycomb-like structure. This maximizes the surface area over which water must flow, and thereby improves evaporation efficiency. However, the increased surface area also encourages deposit formation in the fill.

5.2.2.2 Deposits may consist of mineral scales formed by evaporation of water, corrosion products and silt carried into the tower, and microbiological deposits. Biofilms tend to act as a “glue” that encourages other deposits to adhere to the fill. Because the space between adjacent layers of fill is often quite small, deposited material may “bridge” the fill and block water flow. This is a serious problem, because film fill cannot be cleaned chemically unless water can flow through all parts of the fill.

5.2.2.3 Mechanical cleaning, including water lancing, often damages the lightweight fill material. In addition, the weight of a significant deposit in the film fill can mechanically damage it. Hence, one performance requirement of any cooling water chemical treatment programme intended for use in a film-fill cooling tower shall be to prevent bridging of the fill.

5.2.2.4 The condition of film fill in an operating cooling tower can be monitored by using a “fill test box.” This is simply a section of fill, roughly a 0.6 m (2 ft) cube, enclosed in a supporting box open at the top and bottom. The box is exposed to the “rain” falling below the fill in the cooling tower, in an accessible location. A slippery feeling on the fill surfaces, or appearance of a visible deposit layer, indicates fouling conditions in the fill.

5.2.2.5 A fill test box is a very useful qualitative monitoring tool in an operating cooling tower, but because of space and size limitations, it may not be practical in a pilot cooling tower. In such cases, it is best to design the pilot cooling tower so that the actual tower fill can be accessed conveniently for visual and physical inspection.

5.2.3 Non-heat-transfer metal surfaces

5.2.3.1 Circulating water lines may be carbon steel, copper, brass, fiberglass, polyethylene or cement-lined. Unless process-side conditions dictate otherwise, heat exchanger shells are usually made of carbon steel.

5.2.3.2 All corrosion-prone metals that are present in the operating system should be included as non-heat-transfer test coupons in the pilot study. This is important for two reasons: localized corrosion of piping systems can lead to unexpected failures; and corrosion product deposits can accumulate on heat-transfer surfaces, leading to losses in efficiency and opportunities for underdeposit corrosion. Water treatment chemicals can only provide corrosion protection when the chemicals can reach the metal surfaces. Unprotected metal areas beneath deposits thus become potential sites for underdeposit corrosion.

5.2.4 Heat exchangers

5.1.4.1 Heat exchanger design is generally focused on process-side requirements and on the actual process involved (liquid cooling, gas cooling, or condensing). Process heat exchangers are designed to control the temperature of a process fluid under the most severe expected conditions, that is, the warmest cooling water and the maximum production rate.

5.2.4.2 Heat exchangers are designed with a built-in *fouling factor*¹⁾ that allows the unit to produce the desired process temperature control with some loss of efficiency due to either water- or process-side fouling of the tubes. For these reasons, process heat exchangers are often oversized. To achieve the desired process-side outlet temperature control, operators throttle the water flow in response to ambient conditions, production demands, and the degree of fouling in the heat exchanger. Reducing the water flow rate through the tubes increases the surface temperature and provides more opportunity for suspended solids to settle on the tube surfaces and for mineral scale deposits to form. Both of these effects lead to losses in heat-transfer efficiency and increased opportunities for corrosion of the tubes. See also 9.3.1.

5.2.4.3 One very important function of the chemical water treatment programme is to minimize corrosion and deposit formation of all kinds on heat exchanger surfaces. In designing a pilot-scale testing programme, one critical set of parameters involves the configuration of the heat-transfer section. Heat-transfer tubes may be made of carbon steel, copper, copper alloys, or UNS²⁾ S30400 and S31600 (types 304 and 316 stainless steels). If required in petrochemical plants or other locations with severe process-side conditions, heat-transfer tubes may include a wide variety of other alloys and a few nonmetallic materials.

5.2.4.4 Care should be taken in the selection of the heat exchanger to be modelled. The most appropriate heat exchanger is the one with a combination of the highest surface temperature and the lowest velocity, within reason. Some judgment may be required in the selection process.

5.2.4.5 Petrochemical plants sometimes include vertically oriented shell-and-tube heat exchangers. Because of process requirements, water is often on the shell side in such exchangers. Shell-side water creates particularly severe corrosion and fouling problems that cannot be satisfactorily simulated in the type of pilot-scale equipment covered by this part of ISO 16784. This is especially true of vertical shell-side water heat exchangers.

NOTE As stated in the fourth paragraph of the Scope, shell-side heat exchangers are specifically excluded from this part of ISO 16784.

5.2.4.6 Many plant heat exchangers include multi-tube and multi-pass designs. Such designs are difficult to simulate in a pilot-scale unit. This part of ISO 16784 refers to single-tube, single-pass designs with parameters selected to simulate the conditions under study in the plant exchanger.

1) *Fouling factor* or *fouling thermal resistance* refers to the measured resistance to heat transfer caused by a deposit on a heat-transfer surface. *Fouling factor* is also used in heat-exchanger design to increase the heat-exchanger surface area to compensate for the thermal inefficiency expected to occur due to a deposit on the heat-transfer surface. The term *fouling factor* is commonly used for both. However *fouling thermal efficiency* may be substituted for the measured fouling factor.

2) Metals and Alloys in the Unified Numbering System (latest revision), a joint publication of the American Society for Testing and Materials (ASTM) and the Society of Automotive Engineers Inc. (SAE), 400 Commonwealth Dr., Warrendale, PA 15096.

6 Operating parameters

6.1 General

For any given heat exchanger design, the kinetics of fouling and corrosion are controlled by four parameters: surface temperature, water velocity, residence time, and water quality. Because it is not possible, in a small pilot-scale unit, to duplicate all of the characteristics of an operating heat exchanger, compromises must be made in controlling each of these parameters.

6.2 Surface temperature

6.2.1 The surface temperature of the heat-transfer surface controls the rate of temperature-driven corrosion and fouling reactions. The surface temperature, in turn, is a function of the heat flux, metallurgy, water flow, and the degree of water- and process-side fouling of the tubes.

6.2.2 During testing of water treatment programmes under the most severe conditions that can realistically exist in a specific plant, the surface temperature of the heated tube sections in the pilot unit should match the highest surface temperature in the operating heat exchanger. This temperature can be estimated from measured water- and process-side flows and temperatures, and the design data for the heat exchanger.

6.3 Water velocity

6.3.1 Water velocity through the heat exchanger tubes determines the rate of transfer of dissolved and suspended matter between the bulk cooling water and the water film in contact with the tube wall. These materials can include scaling ions (e.g. calcium and bicarbonate), dissolved ions (the corrosive species in most cooling water systems), foulants including suspended solids, and the chemical additives designed to control fouling and corrosion.

6.3.2 Increasing water velocity normally helps to control both fouling and corrosion. Flow rates between about 1.0 m/s and 2.5 m/s (3 and 8 ft/s) are common. Excessive velocity may cause flow-assisted corrosion, depending on the tube metallurgy. Lower velocities may be required to closely simulate specific plant heat exchangers operating with velocities lower than 1 m/s (3 ft/s).

6.4 Residence time

In reference to heat exchangers, residence time is the time that water is exposed to the heat-transfer surfaces in a specific exchanger, during each cycle through the cooling system. This cannot be exactly duplicated in a small pilot unit. However, the effect of residence time per unit of heat-transfer length is simulated by matching surface temperature and flow velocity to field conditions as closely as possible.

7 Water quality

7.1 General

This clause discusses the effects of the quality and availability of make-up water for open cooling water system operation, performance, and control, emphasizing problems that must be considered when designing specific pilot cooling water test facilities. The quality of the available make-up water may vary seasonally, or may be drawn from several different sources. Such variations should be considered.

7.2 Natural versus synthetic water supplies

For practical reasons, most water treatment product development work is performed in the laboratory, using waters that have been formulated ("synthesized") to resemble typical natural waters. However, the closer a product development project comes to actual field use, the more important it is to test on-site, using actual field water supplies. The reasons for this are given in 7.2.1 to 7.2.3.

7.2.1 Laboratory waters cannot duplicate the organic content of natural waters. Simple organic compounds are sometimes added in the laboratory to compensate for this, but without much success. Natural lignins and tannins and process contaminants are site-specific. These materials can have important effects, especially on

the tendency of a water to precipitate mineral scales. Similarly, microbiological contamination from water and airborne sources cannot be duplicated in the laboratory.

7.2.2 For convenience, laboratory waters are synthesized by dissolving various salts to form stock or mother solutions, which are then mixed as needed to form test waters. Waters made in this way always have higher contents of dissolved solids, and therefore higher conductivities, than natural waters that obtain their mineral contents by slow dissolution of oxides and carbonates. For this reason, synthetic waters are generally more corrosive than the corresponding natural supplies. This difference can significantly affect product performance, especially if the intended application is in a soft water supply, with a low content of dissolved-solids.

7.2.3 Natural waters are sometimes shipped to laboratories for cooling water testing. This may be a partial solution to the problems described above. However, alkalinity relationships, pH, and the microbiological community in the water all change on standing, so that even drummed natural waters often are not fair representations of field conditions.

7.3 Fresh water

Soft, medium, and hard water supplies create specific problems when cycled in cooling tower systems. These problems shall be solved by water treatment programmes matched to the chemistry of the circulating water. Water quality and availability vary widely worldwide. Mineral content, organic matter, other contaminants, and cycles of concentration all affect water treatment operations.

7.4 Seawater and brackish water

High levels of dissolved solids make these water supplies corrosive and require specially formulated treatment programmes. Alkalinity and hardness are often low, so that mineral scaling may be less significant than in some fresh water supplies. Seawater is normally used in once-through systems, but brackish water may be concentrated to conserve water and minimize blowdown. Microbiological contamination shall be considered in pilot testing with these waters, and metals shall match site conditions.

7.5 Recycle/reuse water

Use of secondary treated sewage effluent as cooling tower make-up is recognized as an effective way to conserve fresh water and minimize wastewater discharge. Special problems introduced by these water sources include ammonia, high levels of phosphate, other inorganic salts, and biological and chemical oxygen demand (BOD and COD). These waters are particularly unstable and cannot be transported to a laboratory for reliable testing.

Use of in-plant recycled wastewater as cooling tower make-up is also recognized as an effective way to conserve fresh water and minimize wastewater discharge. Special problems introduced by these water sources include a wide variety of inorganic and organic constituents and biological and chemical oxygen demand (BOD and COD), depending on the plant processes from which the wastewater is received, as well as the processes through which it is treated.

7.6 Dual and combined make-up systems

Some plants use two very different make-up water sources, for example, recycled water and a municipal supply, or a surface water and a ground (well) water. Supplies may be used concurrently, or one may be a "backup" for the other. This creates a variable circulating water composition that cannot easily be simulated. In such cases, on-site pilot-scale testing is the only practical way to evaluate cooling water additives.

8 Contamination

8.1 General

Contamination of the circulating cooling water with airborne solids and gases, microbiological matter, and process materials can severely affect system performance and operating efficiency. Contaminants can create fouling and corrosion problems that may seriously degrade the performance of cooling water chemical additives. In most cases, system contaminants cannot be tested off site. It is very important, therefore, to

recognize the presence of contaminants in the system for which additives are being evaluated. The test protocol must include these contaminants and evaluate their effects on additive performance.

8.2 Process leaks

Process leaks are common in industrial plants. Hydrogen sulfide, ammonia, and oil in oil refineries, a wide variety of organic and inorganic chemicals in chemical plants, coke fines and mill scale in steel mills, etc., all create water treatment problems that must be managed by the water treatment programme. Contaminants may interact with normal constituents of the water, with suspended solids, and with the water treatment chemicals, to create secondary fouling and corrosion problems. These problems are site-specific and very difficult to simulate in any laboratory.

8.3 Biological matter

Biological contamination from the make-up water, process leaks, and airborne sources shall be considered in designing every water treatment programme. Loss of heat transfer, flow restrictions, and microbiologically induced underdeposit corrosion shall all be considered. Microbiological matter is often the "glue" that binds other deposits to metal surfaces, so that microbiological control becomes an essential part of any water treatment programme. These problems are especially difficult to simulate because they are site-specific and because the microbiological sources, even if they can be transported, are unstable and not reproducible in a laboratory.

8.4 Airborne solids and gases

For obvious reasons, the air in a controlled laboratory environment cannot be the same as the air blowing through a cooling tower in an operating plant. In an oil refinery, for example, ammonia and hydrogen sulfide gases can affect pH control and microbiological activity. Dirt, coke fines, and other airborne material add suspended solids. Exhaust fans can blow kitchen fumes into commercial HVAC cooling towers. These and similar contaminants can have major effects on the performance of cooling water treatment programmes. Identification of specific airborne contamination is a vital part of any pilot test programme, and this contamination cannot easily be simulated in a laboratory.

9 Parameters to be evaluated in pilot test units

9.1 Corrosion

9.1.1 General comment

Corrosion is a principal determinant of the economic life of water-handling equipment. Economic life, in turn, is a key design parameter for equipment used in cooling water systems. Operating systems may contain several different materials of construction. The corrosion criteria to be evaluated for each of the critical metallurgies should be determined. Water treatment programmes must be selected to protect metals exposed to water in the system that can be expected to corrode. The pilot testing programme must include corrosion measurements on each of these metals, including where applicable, on both heat-transfer and non-heat-transfer surfaces.

9.1.2 Criteria for corrosion evaluations

The criteria used for evaluating corrosion inhibitor performance are industry-dependent. For example, a carbon steel corrosion rate of 0.075 mm/year (3.0 mpy) would be considered unusually low in many steel mills. In an oil refinery, this same corrosion rate might be in the normal range, while in a clean HVAC system it would be unacceptably high. Thus, a corrosion inhibitor programme required for good performance in an HVAC system might be "overkill" and not cost-effective in a steel mill. The pilot test parameters shall be matched both to the industry type and to the specific plant site.

9.1.3 Types of corrosion damage

Serious corrosion damage in open cooling water systems is usually the result of localized attack, including pitting, crevice attack, etc. Damage from general surface corrosion, in the absence of major upsets such as

acid leakage into the water, is unusual. Evaluation should therefore include both localized and general corrosion on all metals to be protected.

9.1.4 Microbiologically influenced corrosion (MIC)

MIC occurs in an environment developed by certain types of microorganisms that make the alloys susceptible to attack. These organisms can generate organic and inorganic acids as part of their metabolic processes, and this can lead to rapid pitting attack on steel and other metals. MIC is especially difficult to study in pilot-scale test units because the deposits that harbor the microorganisms do not form readily under these conditions. MIC is best studied in the field. If a specific system under evaluation is shown to be subject to MIC, the chemical treatment programme shall include methods for removing deposits and killing microorganisms in these deposits. If this is not done, chemical treatment programmes that are effective in a pilot unit might not perform well in the field.

9.1.5 Methods for evaluating corrosion in pilot-scale test units

9.1.5.1 Heat-transfer specimens

Heat-transfer specimens in the pilot test unit should be used for both corrosion and fouling evaluations. The test specimens should be easy to remove so that they can be photographed and weighed. Corrosion patterns should be noted and pit depths measured when appropriate.

9.1.5.2 Corrosion coupons

Coupons provide a measure of the average corrosion rate over the period of exposure. They also accumulate deposits and can show the presence of underdeposit corrosion. Coupons should be handled and evaluated in accordance with NACE Standard RP0497³⁾ and ASTM G 4⁴⁾. Coupons, while useful for evaluation of system corrosiveness, fail to replicate the hydrodynamic and thermal conditions of heat exchanger tubes. Coupon data should be considered indicative only.

9.1.5.3 On-line measurements of general corrosion

Linear polarization resistance (LPR) instruments measure the corrosion current flowing between two electrodes exposed in the cooling water, with a small applied voltage. (Both two-electrode and three-electrode LPR systems are available; electrodes may also be used as corrosion coupons.) From this current, the instrument calculates an “instantaneous” corrosion rate, i.e. the rate at the moment of measurement. Such data are useful for showing effects of additives and dosage changes and changes in process variables such as pH. Combined with coupon results, LPR data can be useful in identifying the corrosive species in specific systems and studying the performance of corrosion inhibitors. Such devices may be calibrated against corrosion coupon studies performed over the same exposure period. Care should be taken to perform several such studies to validate the calibration. Similar data can be generated using galvanostatic methods.

More recent techniques, such as electrochemical noise, may be adopted but need to be validated for the application.

9.2 Fouling

9.2.1 General comment

Fouling is defined as the deposition of any material on a heat-transfer surface. Fouling reduces heat exchanger performance by insulating the heat-transfer surface from the water. Fouling also increases the pressure drop through the heat exchanger, thus restricting water flow. Fouling of the tube surface determines the efficiency with which heat is transferred through a heat exchanger. Fouling creates potential sites for underdeposit corrosion, including MIC. Possible process-side fouling must be considered when evaluating the performance of water treatment programmes in specific-field heat exchangers. Virtually all heat exchangers are designed with an allowance for fouling, as explained in 5.2.4.2. Thus, any evaluation of a cooling water treatment programme should include provisions for quantifying fouling. Measured fouling must be compared

3) Available from NACE International, 1440 South Creek Drive, Houston, TX 77084-4906 USA.

4) Available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959 USA.

against the design allowance for the heat exchanger, taking into account any deviation of test parameters from actual operating and design conditions. Corrosion coupons should be evaluated in accordance with 9.1.5.2.

9.2.2 Types of water-side fouling

Water-side foulants include mineral scales such as calcium carbonate and calcium phosphate, corrosion products, microbiological material, general waterborne and airborne solids, and, in some cases, process contaminants. The specific mix of foulants in an operating system can affect the choice of fouling inhibitors included in the water treatment programme.

9.2.3 Sources of test waters

9.2.3.1 Off-site testing with synthetic make-up water

For off-site testing with synthetic waters, as explained in 4.1, the heated surface skin temperature and water flow velocity in the pilot unit shall be matched to field conditions as closely as possible. By properly preparing a synthetic water for testing, it may be possible to simulate the scaling tendency of the field system. It is generally not possible, however, to simulate corrosiveness, microbiological fouling, waterborne suspended solids, airborne contamination, and process contamination in a synthetic water. All these factors influence both the nature and amount of fouling that occurs in field situations, and the performance of scale inhibitors, dispersants, and microbiocides. For this reason, any data obtained in off-site tests with synthetic waters shall be considered preliminary until they have been confirmed either in on-site pilot tests or in field use.

9.2.3.2 On-site testing with plant make-up water

On-site fouling evaluations provide an opportunity to study the interactions of various deposits and the effects of ongoing corrosion on fouling reactions. It is known, for example, that corrosion reactions raise the pH of surface films on cathodic areas of the corroding metal. This, in turn, encourages mineral scale precipitation. Microbiological “glue” increases all types of fouling and makes control more difficult. Realistic comparisons of fouling inhibitors should always be conducted in the field, under pilot system conditions that simulate operating heat exchangers as closely as possible.

9.2.4 Pilot-scale methods for evaluating fouling (see also ISO 16784-2)

9.2.4.1 Heat-transfer methods

The central focus of a pilot-scale cooling water test unit is usually a heated tube surface that simulates a field heat exchanger. In one design, the tube is heated electrically from the inside. Water flows through the annular space between the outside of the tube and a surrounding glass or plastic tube. Thus, the outer surface of the heated tube simulates the inner surface of a “tube-side” heat exchanger. Another design uses an external heater with water flowing within the tube. In either case, the tube should be commercial heat exchanger tubing matched as closely as possible to the field system, and should be usable without extensive machining or other treatment.

Fouling should be evaluated throughout the test by measurement of heat-transfer coefficients and fouling factors. At the conclusion of a pilot test, fouling of the heated tube surface can be evaluated by visual examination of the tube surface (including a photographic record), by the weight of the deposit removed from the surface and by analyses of the deposits. It is important to be able to assemble the unit easily and to disassemble it without disturbing deposits on the tube surface.

9.2.4.2 Non-heat-transfer methods

Fouling of non-heat-transfer surfaces can also be important in operating cooling systems. Deposits can restrict water flow in small-diameter tubes and create sites for underdeposit corrosion. Inspections of corrosion coupons and coupon racks provide visual evidence of deposit formation. Coupons made from corrosion-resistant stiff wire mesh (e.g. nichrome, stainless steel, and titanium) can be used to accumulate suspended solids and microbiological deposits.

Pressure drop across a length of small-diameter tubing can provide a measure of fouling. It is particularly sensitive to microbiological fouling, and is most appropriately used to measure this type of fouling. If desired, such a device can be incorporated as an optional part of a pilot-scale testing unit.

9.3 Practical problems in operating systems — Multiple combinations of problems

9.3.1 Multiple problems

In operating cooling systems, none of the water treatment problems described in this part of ISO 16784 occur in isolation. Corrosion increases the pH of the surface film and encourages mineral scaling. Deposits of all kinds create sites for underdeposit corrosion. Biofouling produces a “glue” that helps to bind deposits to surfaces. Thus, to be realistic, a pilot testing programme intended to evaluate the performance of cooling water treatment programmes for a specific site should include all of the chemical, operating, and environmental factors that, working together, produce the cooling water environment at that site.

9.3.2 Problems with standardized conditions

Testing additives under clean, standardized conditions can produce misleading results. For example, mineral scale inhibitors that perform well when there is no corrosion in progress may be deactivated by iron or copper corrosion products in the water. Some corrosion and scale inhibitors may precipitate at high levels of hardness in the circulating water. Pilot plant testing should be designed to include as many of these factors as possible, and unavoidable deviations from plant practice should be carefully noted in the test reports.

9.4 Water treatment additives

9.4.1 Combination testing

For the reasons described in this subclause, pilot-scale test facilities should be designed to allow feeding and control of water treatment additives combined into practical programmes as recommended by the suppliers. Testing of individual components may not produce results typical of field conditions. Different combinations of additives may produce equivalent good performance, but may require different sets of operating parameters. Therefore, testing of different programmes under identical conditions may not produce the best results that can be obtained from any programme.

9.4.2 Compatibility of additives

The components of any treatment programme shall be compatible with one another and with the system. Cationic biocides may precipitate with anionic surfactants. Oxidizing biocides may enhance the activity of some non-oxidizing biocides, but may destroy some scale and corrosion inhibitors, especially when their concentration is high. Some corrosion inhibitors are effective at recommended dosages, but may precipitate and cause fouling if overdosed or used outside the recommended pH range.

10 Design of pilot-scale performance testing facilities

10.1 Objectives

A method for the evaluation of the performance of cooling water treatment programmes using a pilot-scale rig is given in ISO 16784-2. Development of additives to control specific water treatment problems and mechanisms is not covered by either part of ISO 16784. The objective of pilot plant testing incorporating this part of ISO 16784 should be to assess the performance of formulated water treatment programmes under site-specific field conditions. When the objective is to solve an existing problem in an operating cooling system, that may be done most effectively with an on-line monitor of fouling, corrosion, and chemical parameters operating on the existing cooling system. This would be undertaken initially by incrementally adding to the existing treatment chemistry. Subsequently, the existing treatment programme may be changed by incorporating the new chemistry.

10.2 The importance of simulating specific application environments

Care should be taken to simulate fouling and corrosion conditions in heat exchangers, which are critical from a water treatment standpoint, as opposed to specific process bottlenecks. The latter may be the result of design shortcomings or process problems, rather than water chemistry. Realistic test conditions are critically important. Both overstressing and understressing the water treatment programme may decrease the utility of the test results and make the test results misleading. Examples can be found in NACE Standard RP0189 (see Bibliography).

10.3 Compromises in pilot-scale performance testing

10.3.1 Heat source, heat duty and temperature

10.3.1.1 Heat source

Most pilot-scale performance test devices substitute electrically heated heat-transfer surfaces for process heat exchangers. These devices are subject to some limitations. They are steady-state devices, which eliminate all process-side variables, such as heat transfer through a process film, process side fouling, and variations in heat duty as production rates vary. Thus, the test device has a constant heat duty regardless of the waterside fouling. In actual operation, the heat duty of a heat exchanger would be reduced as the water-side fouling deposit thickens.

10.3.1.2 Heat duty

The heat duty and heat flux of a pilot-scale performance test device are substantially different from those of a process heat exchanger being modelled. The differences are the result of both size and geometry. The test device is much smaller than the process heat exchanger.

10.3.1.3 Test geometry

An annular geometry may be substituted for internal tubular flow. The heat flux in the test device is greater in order to obtain the same heat-transfer surface temperature as the outlet of the process heat exchanger. The water velocity may be different from that in the process heat exchanger, in order to replicate the shear stress of the water film against the heat-transfer surface. When an annular geometry is used, the equivalent diameter of the annular space, and that of the internal tubes in the process heat exchanger, may be significantly different.

10.3.1.4 Temperature profile

It is very difficult to simulate the water temperature profile in a large operating cooling circuit with a pilot unit. The conditions of the pilot unit should be adjusted, as closely as possible, to simulate the conditions that are under study. Possible compromises include corrosion rate versus bulk water temperature, relative surface areas, and localized chemistry.

10.3.1.5 Statistical analyses

In order to analyse test results statistically, either a device consisting of multiple parallel tubes or a series of tests on a single tube device would be required. Statistical analysis is not normally required under field conditions.

10.3.1.6 Protocol and report

The test protocol and test reports should include specific design and operating data for both the plant heat exchanger and the test unit, so that this information can be part of the water treatment decision process. The test report should include calculations demonstrating hydrodynamic similarity of the test unit to the exchanger being simulated. Differences, if any, should be described and discussed.

10.3.2 Water chemistry

10.3.2.1 On-site versus off-site water

As explained in this part of ISO 16784, whenever possible, pilot-scale testing should be performed on site using plant water supplies. A second and less satisfactory option is to ship plant water to an off-site laboratory for testing. For practical reasons, this method is frequently used. In such cases, it is important to remember that the water chemistry, particularly the alkalinity relationships and the microbiological content of the water, changes in transit and storage, so that these properties of the water are not correctly assessed in the off-site tests.

10.3.2.2 Microbiological effects

If microbiological fouling is thought to be an important consideration, on-site testing should be conducted during that period of time (season or other) when conditions, such as source-water quality, are most conducive to biofouling.

10.3.3 Product dosages — The effects of surface/volume ratio

The surface-to-volume ratio (s/V ratio) is defined as the ratio of the total surface area of metal exposed to water in the cooling system to the total volume of water in the system. In operating cooling systems, this ratio is quite small compared with that in pilot systems. That is, there is much more water in a plant system, compared with the metal surface area, than in a pilot-sized unit. This large volume of chemically treated water provides a reservoir of chemicals to replenish chemicals lost from the surface film by precipitation, reactions with solids, or destruction. For this reason, chemical dosage levels required for good fouling and corrosion protection are often lower in field practice than in pilot studies.

10.3.4 Test duration

It would be ideal to have a standard time period for such testing. However, it would be impossible to predetermine such a period that would be appropriate for every combination of water quality, treatment, and operating conditions. In open recirculating cooling water systems, the fouling factor is an asymptotic function and the test period should be long enough to reach at least the knee of the curve, if not the beginning of an asymptote. For corrosion, sufficient time is needed to assure the user that the test spans at least one or two cycles of the variability in the operating conditions. Pilot-scale tests are commonly run for two to three weeks, but this should not be taken as a standard recommendation. However, the standard exposure time for corrosion coupon studies is 90 days. Shorter periods generally yield higher corrosion rates than a 90 day test. The appropriate test duration should be determined at each location.

10.3.5 Overall design considerations

The test apparatus and procedures should be practical to the extent that time between tests is minimized and procedures and protocols for implementing the tests are easily understood and followed. In addition, required cleaning, calibration, assembly, and disassembly should be easily done by a trained technician in the field.

11 Pilot-scale facility operations

11.1 Documentation of design

11.1.1 General comments

Conditions in this simulated heat exchanger should match field conditions as closely as possible. All deviations from field practice should be identified and described, both in the test protocol and in the final report interpreting the test results. This information shall be available for use in selecting products and in comparing results of pilot tests with field data.

11.1.2 Equipment

The design and actual operation of the process heat exchanger to be modelled shall be documented before any test programme is initiated. While the physical design of a pilot-scale performance test device may be unchangeable after the device has been constructed, operating parameters, flow, and heat flux shall be adjustable to emulate the fouling and corrosion conditions in the process heat exchanger being modelled.

11.1.3 Water treatment

The water treatment regime of the pilot system shall be consistent with that in the operating system. This is especially important with respect to microbiological control via oxidizing or non-oxidizing microbiocides.

11.2 Repeatability of results and comparison with field performance

The results of any off-line pilot plant or laboratory testing should be validated against site-specific field performance. Any discrepancy should be investigated. Careful records of test results and actual performance should be maintained. If there are repeated discrepancies between the performance test results and actual operation, the operation of the performance test device should be modified until there is appropriate agreement between the performance of water treatment programmes in the pilot unit and the process heat exchanger.

11.3 Record-keeping and reports

Records of all relevant data shall be kept, including:

- a) heat exchanger to be modelled: process design sheet, actual cooling water flow, inlet and outlet temperatures, calculated surface temperature, metallurgy, etc.;
- b) pilot plant performance test device: process design, set-up conditions, cooling water flow rate, power or heat flux, surface temperature, metallurgy, etc.;
- c) significant compromises made in pilot-stage modelling of a specific heat exchanger and expected effects of these compromises on the test results;
- d) pilot plant water chemistry;
- e) all pilot test data and analyses.

Bibliography

- [1] ASTM G 4-01, *Standard guide for conducting corrosion tests in field applications*
- [2] KNUDSEN, J.G. *Apparatus and Techniques for Measurement of Fouling of Heat Transfer Surfaces*. In *Fouling of Heat Transfer Equipment*. Eds. E.C.F. Somerscales and J.G. Knudsen. Washington, DC: Hemisphere Publishing Corp., 1981
- [3] NACE Standard RP0189-2002, *On-Line monitoring of cooling waters*
- [4] NACE Standard RP0300-2003, *Standard recommended practice pilot-scale evaluation of corrosion and fouling control additives for open recirculating cooling water systems*
- [5] NACE Standard RP0497-2004, *Field corrosion evaluation using metallic test specimens*
- [6] NACE Standard TM0286-2001, *Cooling water test unit incorporating heat transfer surfaces*
- [7] NEN 7420:1994, *Industrial cooling water/Assessment of the performance of treatment programmes under standard conditions*. Bilthoven, The Netherlands: Netherlands Corrosion Centre ⁵⁾
- [8] PERRY, R.H. and D.W. Green, eds. *Perry's Chemical Engineers' Handbook of Chemical Engineering*. 7th ed, Chapter 10, York, NY: McGraw Hill, New, 1997
- [9] NACE Publication 3T199, *Techniques for monitoring corrosion and related parameters in field applications* (Houston, TX: NACE)

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