ASME PTC 31-2011

[Revision of ASME PTC 31-1973 (R1991)]

# High-Purity Water Treatment Systems

## Performance Test Codes

AN AMERICAN NATIONAL STANDARD

The American Society of Mechanical Engineers

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AN AMERICAN NATIONAL STANDARD

The American Society of
Mechanical Engineers

Three Park Avenue • New York, NY • 10016 USA

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# NOTICE

All Performance Test Codes MUST adhere to the requirements of PTC 1, General Instructions. The following information is based on that document and is included here for emphasis and for the convenience of the user of this Code. It is expected that the Code user is fully cognizant of Sections 1 and 3 of ASME PTC 1 and has read them prior to applying this Code.

ASME Performance Test Codes provide test procedures that yield results of the highest level of accuracy consistent with the best engineering knowledge and practice currently available. They were developed by balanced committees representing all concerned interests and specify procedures, instrumentation, equipment-operating requirements, calculation methods, and uncertainty analysis.

When tests are run in accordance with a Code, the test results themselves, without adjustment for uncertainty, yield the best available indication of the actual performance of the tested equipment. ASME Performance Test Codes do not specify means to compare those results to contractual guarantees. Therefore, it is recommended that the parties to a commercial test agree before starting the test and preferably before signing the contract on the method to be used for comparing the test results to the contractual guarantees. It is beyond the scope of any Code to determine or interpret how such comparisons shall be made.

# FOREWORD

The ASME Performance Test Codes Committee voted in December 1965 to establish a Test Code Committee for Demineralizers (PTC 31) and later approved as PTC 31 Committee's objective, the development of a Test Code that would define the procedures for the accurate testing of ion exchange equipment for determining level of performance. The name of this Committee was changed at the request of PTC Committee No. 31 from Demineralizers to Ion Exchange Equipment on June 12, 1970.

Most steam generation cycles, either for process application or utility power production, require the use of water treatment equipment. Such equipment may be a simple process application, removing only hardness constituents from water, or may be a relatively complex process employing one or more types of ion exchange resin processes and/or membrane processes each with a high degree of instrumentation and control logic. Additionally, such equipment is employed in virtually all types of nuclear steam generation cycles, processing water containing not only very high concentrations of impurities, but also treating liquids with impurity concentrations in the range of parts per billion to parts per trillion. Because performance of such process equipment directly influences the efficiency and output of steam generation cycles, a Committee was named by The American Society of Mechanical Engineers to draft a revised Performance Test Code for High-Purity Water Treatment Systems. Members of this Committee were selected on the basis that equipment manufacturers, users, and consultants as well as general interest groups were represented. A draft of this Code was distributed in September 2011 for comment and criticism by industry and other interested individuals.

This edition was approved by the PTC Standards Committee on September 2, 2011, and approved and adopted as a Standard practice of the Society by action of the Board on Standardization and Testing on October 7, 2011. It was also approved as an American National Standard by the ANSI Board of Standards Review on November 21, 2011.

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# Performance Test Codes

(The following is the roster of the Committee at the time of approval of this Code.)

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Secretary, PTC Standards Committee
The American Society of Mechanical Engineers
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New York, NY 10016-5990

**Proposing Revisions.** Revisions are made periodically to the Code to incorporate changes that appear necessary or desirable, as demonstrated by the experience gained from the application of the Code. Approved revisions will be published periodically.

The Committee welcomes proposals for revisions to this Code. Such proposals should be as specific as possible, citing the paragraph number(s), the proposed wording, and a detailed description of the reasons for the proposal including any pertinent documentation.

**Proposing a Case.** Cases may be issued for the purpose of providing alternative rules when justified, to permit early implementation of an approved revision when the need is urgent, or to provide rules not covered by existing provisions. Cases are effective immediately upon ASME approval and shall be posted on the ASME Committee Web page.

Request for Cases shall provide a Statement of Need and Background Information. The request should identify the Code, the paragraph, figure or table number(s), and be written as a Question and Reply in the same format as existing Cases. Request for Cases should also indicate the applicable edition(s) of the Code to which the proposed Case applies.

**Interpretations.** Upon request, the PTC Standards Committee will render an interpretation of any requirement of the Code. Interpretations can only be rendered in response to a written request sent to the Secretary of the PTC Standards Committee. The request for interpretation should be clear and unambiguous. It is further recommended that the inquirer submit his/her request in the following format:

| | |
|---|---|
| **Subject:** | Cite the applicable paragraph number(s) and the topic of the inquiry. |
| **Edition:** | Cite the applicable edition of the Code for which the interpretation is being requested. |
| **Question:** | Phrase the question as a request for an interpretation of a specific requirement suitable for general understanding and use, not as a request for an approval of a proprietary design or situation. The inquirer may also include any plans or drawings that are necessary to explain the question; however, they should not contain proprietary names or information. |

Requests that are not in this format will be rewritten in this format by the Committee prior to being answered, which may inadvertently change the intent of the original request.

ASME procedures provide for reconsideration of any interpretation when or if additional information that might affect an interpretation is available. Further, persons aggrieved by an interpretation may appeal to the cognizant ASME Committee or Subcommittee. ASME does not “approve,” “certify,” “rate,” or “endorse” any item, construction, proprietary device, or activity.

**Attending Committee Meetings.** The PTC Standards Committee and PTC Committees hold meetings regularly, which are open to the public. Persons wishing to attend any meeting should contact the Secretary of the PTC Committee.

# HIGH-PURITY WATER TREATMENT SYSTEMS

## Section 1
## Object and Scope

### 1-1 OBJECT

### 1-1.1

This Code defines the procedures for the accurate field testing of high-purity water treatment systems for the purpose of determining level of performance. It is based on the use of accurate instrumentation and the best analytical and measurement procedures available.

### 1-1.2

This Code is recommended for use in conducting acceptance tests of high-purity water treatment systems. If so used, any deviations from Code procedure must be agreed upon in writing. In the absence of written agreement, the Code requirements shall be mandatory.

Upon completion of tests, the report issued should provide all necessary base line data against which all future operational test results can be measured to assess deterioration of performance in the interim.

### 1-1.3

Before formulating the procedure for testing a specific process or system, the Code on General Instructions PTC 1 should be studied and followed in detail.

### 1-1.4

The Code on Definitions and Values (PTC 2) defines certain technical terms and numerical constants. Unless otherwise specified in this Code, instrumentation should comply with the sections of Supplements on Instruments and Apparatus (PTC 19 Series).

### 1-2 SCOPE

Only the relevant portion of this Code need apply to any individual case or test under consideration. In some cases the procedure is simple; however, for complex systems or complex modes of system operation, the procedures and calculations of test results require more involved provisions for testing.

### 1-2.1

This Code is applicable to the following types of high-purity water treatment systems, which are either used individually or in various combinations depending on requirements of the process:

*(a)* *membrane equipment* including but not limited to, microfiltration, ultrafiltration, nanofiltration, and reverse osmosis

*(b)* *ion exchange equipment* including, but not limited to, softeners, dealkalizers, multibed demineralizers, mixed-bed demineralizers, and condensate polishers

*(c)* *hybrid equipment* including, but not limited to, electrode ionization (EDI) and electrodialysis reversal (EDR).

### 1-2.2

This Code applies to equipment and systems that are utilized for

*(a)* the conditioning of makeup, feedwater, and condensate for steam generation

*(b)* the conditioning of process waters

### 1-2.3

This Code applies to the performance of high-purity water treatment systems at design, minimum flow rates or maximum flow rates, depending on the purpose of the test, with regard to one or more of the following:

*(a)* water quality and quantity of influent and effluent

*(b)* pressure drop, flow, and temperature

*(c)* startup, shutdown, and lay-up procedure

*(d)* operating efficiency

*(e)* media testing

*(f)* media cleaning and maintenance

*(g)* chemical purity and solution concentrations

*(h)* associated chemical equipment

## 1-3 TEST UNCERTAINTIES

### 1-3.1

Expected test uncertainties cannot be established because of the inability to control operating parameters that are the causes of the uncertainty. The following is a partial list of parameters that may impact the test uncertainty:

*(a)* seasonal variations in influent water characteristics
*(b)* aging of equipment and media
*(c)* operator interface

### 1-3.2

As stated in subsection 1.1, this Code primarily provides guidance in the design and operation of the high-purity water treatment systems. Section 5 is included to identify dominant sources of errors and their effect on the test results but not serve to validate the quality of the results.

# Section 2
# Description and Definition of Terms — Ion Exchange

## 2-1 DEFINITIONS

*acidity:* expression of the concentration of hydrogen ions present in a solution.

*alkalinity:* expression of the total basic anions (hydroxyl groups) present in a solution. It also represents, particularly in water analysis, the bicarbonate, carbonate, and occasionally, the borate, silicate, and phosphate salts that will react with water to produce the hydroxyl groups.

*alkalinity "M" (total alkalinity):* the total titratable alkalinity down to the pH of 4.3. It represents hydroxide carbonate and bicarbonate ions in the water.

*alkalinity "P" (phenolphthalein):* the titratable alkalinity down to pH 8.2. It represents the hydroxide and carbonate ions in the water.

*amine cycle:* operation of a cation exchange cycle in condensate polishers wherein the removal of specified cations from the influent condensate is accomplished by exchange with an equivalent amount of amine ion from the exchange material.

*anion:* negatively charged ion, such as chloride ($Cl^-$), sulfate ($SO_4^-$), nitrate ($NO_3^-$), carbonate ($CO_3^-$), fluoride ($F^-$), bromine ($Br^-$), carbon dioxide ($CO_2$), and silica ($SiO_2$).

*anion exchange material:* ion exchange material capable of the reversible exchange of negatively charged ions.

*anion exchange material, intermediate base:* ion exchange material containing a mixture of weakly basic and strongly basic groups. The properties of this resin are intermediate between the weak and strong base anion exchangers.

*anion exchange material, strong base:* ion exchange material containing the highly ionized quaternary ammonium functional group. It can remove both the highly dissociated strong acids (sulfuric, hydrochloric, nitric, etc.) and the weakly dissociated weak acids (carbonic, silicic, etc.).

*anion exchange material, weak base:* ion exchange material containing the polyamine functional groups. It is highly ionized in the salt form and can operate only when pH is below 7. Weakly ionized in the free base form, it has little, if any, salt-splitting capacity and can remove the anionic component of strong acids (sulfuric, hydrochloric, nitric, etc.) only.

*anions, total:* numeric sum of the anions listed above except carbon dioxide and silica, with all being expressed as calcium carbonate ($CaCO_3$) equivalents.

*anions, total exchangeable:* numeric sum of the anions listed above including the anions from carbon dioxide and silica, with all being expressed as calcium carbonate ($CaCO_3$) equivalents.

*array:* numeric description of the number of modules contained in the stages or passes in the system.

*backwash:* reverse flow of water through the media to fluidize and reclassify the bed to remove suspended matter and fines.

*breakthrough:* first appearance in the solution flowing from an ion exchange unit of unadsorbed ions similar to those that are depleting the activity of the resin bed. Breakthrough is an indication that regeneration of the resin is necessary.

*calcium carbonate ($CaCO_3$) equivalents:* preferred reporting form of ionic concentrations of impurities in water facilitating calculations because calcium carbonate has a molecular weight of 100 and concentrations can be totaled and balanced when in this form.

*cation:* positively charged ion, such as sodium ($Na^+$), calcium ($Ca^{++}$), ammonia ($NH_4^+$), magnesium ($Mg^{++}$), potassium ($K^+$), barium ($Ba^{++}$), strontium ($Sr^{++}$), iron ($Fe^{++}$), manganese ($Mn^{++}$), copper ($Cu^{++}$), and aluminum ($Al^{+++}$).

*cation exchange material:* ion exchange material capable of the reversible exchange of positively charged ions.

*cation exchange material, intermediate acid:* ion exchange material containing a mixture of weakly acidic and strongly acidic groups. The properties of this resin are intermediate between the weak and strong acid cation exchangers.

*cation exchange material, strong acid:* ion exchange material containing the highly ionized sulfonic acid functional group ($SO_3H$). It can easily split salts converting them to acids.

*cation exchange material, weak acid:* ion exchange material containing the weakly ionized carboxylic acid group COOH, which has a great affinity for the hydrogen ion. When operating in the hydrogen ($H^+$) form, it removes only that part of the total cations that is equivalent in amount to the bicarbonate alkalinity. When operating in the sodium ($Na^+$) form following regeneration with acid followed by regeneration with caustic soda (sodium hydroxide), it removes only the hardness ions $Ca^{++}$ and $Mg^{++}$.

*cations, total:* numeric sum of the cations listed above with all being expressed as calcium carbonate ($CaCO_3$) equivalents.

*cations, total exchangeable:* numeric sum of only those cations listed above to be removed, with all being expressed as calcium carbonate ($CaCO_3$) equivalents. For example, in simple softening ion exchange, this would be the sum of only the hardness ions calcium and magnesium. This is also referred to as "total hardness." However, any multivalent ion held stronger than sodium would also be removed. In naturally occurring waters these ions are usually very low in concentration relative to hardness levels and can normally be ignored.

*cellulose acetate:* cellulose material used in a reverse osmosis membrane.

*channeling:* cleavage and furrowing of the bed due to faulty design and/or operational procedures, in which the solution being treated follows the path of least resistance, runs through these furrows, and fails to contact active groups in other parts of the bed.

*cocurrent flow:* direction of flow of the regenerant is the same direction of flow as the service flow.

*colloids:* matter of very fine particle size, usually in the range of $10^{-5}$ cm to $10^{-7}$ cm in diameter. Colloids of iron and silica are frequently found in surface waters, carry electrical charges, and cause fouling of reverse osmosis membranes and pass completely through ion exchange resin beds.

*color throw:* describes the phenomenon of processed effluent being colored more than the influent, generally caused by

*(a)* deterioration of ion exchange resin stored for prolonged periods at elevated temperatures

*(b)* deterioration of ion exchange resin due to repeated freezing and thawing

*(c)* high content of iron in raw water

*(d)* microbial contamination of raw water

*(e)* leaching into the effluent of residual monomers from the manufacturing process as a result of prolonged or repeated storage

*common header interconnection:* ion exchange unit interconnection method where all units of identical type or function are interconnected by a shared influent and effluent pipe header.

*compressed regeneration schedule:* sequence designed to reduce total regeneration time by injecting dilute regenerant chemicals simultaneously into two or more dissimilar units.

*concentrate (brine, reject):* waste liquid containing all the solids and material unable to pass through the membrane.

*condensate demineralizer:* ion exchange units applied to the removal of contaminants from steam condensates. Mixed-bed units are utilized for this application at much higher flow rates per square foot of resin bed surface than mixed-bed units employed for high solids water processing.

*condensate ion exchanger:* ion exchange equipment containing cation exchange resin in the sodium or amine form that is used for the removal of contaminants from steam condensates. Generally, this equipment is designed to operate at a much higher flow rate per square foot of resin bed surface than a typical softening ion-exchanger. Mixtures/blends of steam/process condensate and makeup water may also be processed by this equipment.

*condensate polisher:* see *condensate demineralizer* and *condensate ion exchanger.*

*conductivity (cation):* amplified electrical conductance of a solution that has had all cations and amines present replaced by hydrogen ($H^+$) ions by being passed through a highly regenerated column of hydrogen form strong cation exchange resin.

*conductivity (degassed cation):* cation conductance of a solution that has also been degasified to remove any contribution of carbon dioxide to the electrical conductance of the solution.

*conductivity (specific):* electrical conductance of a solution is a general property and is not specific for any particular ion. In determining the quality of the effluent from certain ion exchange processes, it is an excellent method of assessing total salt content. The value is reported in $\mu S/cm^2$ ($\mu mho/cm^2$).

*copper:* may be soluble or a particulate oxide.

*countercurrent flow:* direction of flow of the regenerant is the reverse of the direction of flow of the service flow.

*crossflow filtration:* membrane processes use flow of the liquid across (tangential to) the membrane surface.

*crud:* particulate metallic oxides present in condensate that are filtered out in condensate polishers and powdered resin filtration systems.

*dead end:* term applied to cartridge and membrane filtration to signify flow path perpendicular to the surface of the filtration medium.

*dealkalizer:* application of acid regenerated cation or chloride regenerated anion ion exchange resins in equipment designed to reduce alkalinity in waters.

*dealkalizer (split stream):* a system of dealkalization that blends acidic and alkaline waters to achieve a desired effluent alkalinity.

*decarbonator (forced-draft):* process equipment in water treatment installations used to reduce or remove free carbon dioxide from the water passed through it by contact between water and a stripping gas, usually air.

*degasifier (vacuum):* process equipment in demineralizer installations used to reduce or remove the free carbon dioxide and air content from the water passed through it by subjecting the water to a vacuum.

*demineralization:* the removal of all dissolved ions from water.

*distributor (inlet):* mechanical system in conventional ion exchange units, generally located at the top of an exchanger, through which the influent enters the vessel for processing. When located at the top of a vessel, it may also act as a collector of the backwash water that is discharged to waste. In addition, it serves to prevent disturbance of the top of the resin bed due to impingement by introducing the influent evenly over the surface of the resin bed.

*distributor (interface, mid-collector, or buried collector):* used primarily in mixed-bed demineralizers. As implied, located at the interface between the beds of cation and anion resins that is formed after the backwash separates and classifies the two resins; collects the spent alkali and acid that has passed through the cation and anion resin layers; can be also used for distributing acid if it is to be passed downward through the cation resin bed and usually consists of a header and laterals, equipped with screens or strainers to retain resin, similar in design to the regenerant distributor. This type of distributor may also be used in other types of ion exchange equipment, such as stratified-bed units.

*distributor (outlet or underdrain):* mechanical system generally located at the bottom of an exchanger through which the processed liquid (effluent) is collected from the resin bed on a uniform basis with respect to the entire cross section of the exchanger. When located at the bottom of a vessel, it may also serve as the distributor for resin backwash water and resin mixing air.

*distributor (regenerant):* mechanical system through which the regenerant solution is introduced uniformly to the entire cross section of the exchanger for the maximum utilization of the regenerant solutions.

*distributor (subsurface):* mechanical system commonly used in cation condensate polishers, under the surface of a bed of resin or media, through which backwash solution is introduced uniformly to the entire cross section of the exchanger to loosen and remove particulates from the upper portion of the resin and to reduce bed differential pressure.

*efficiency:* effectiveness of the operational performance of an ion exchanger. Efficiency in the adsorption of ions is expressed as the quantity of regenerant required to affect the removal of a specified unit weight of adsorbed material (e.g., pounds of acid per kilograin of salt removed).

*effluent:* liquid, solid, or gaseous product discharged or emerging from a process; in this Code, treated water discharged from an ion exchange system.

*electrodeionization (EDI):* membrane process that combines electrodialysis (ED) with self-regenerating ion exchange resins installed between the membranes.

*electrodialysis (ED):* membrane process that discriminates between particles on the basis of their electrical charge by preventing the substance to be concentrated from passing through the membrane.

*electrodialysis reversal (EDR):* modification of ED in which the electrical field is periodically reversed to reduce material buildup.

*electrodialysis stack:* device that holds an array of membranes between two electrodes in such a way that the solutions being processed are separated.

*endpoint:* point of the service run of an ion exchange unit where salt, acid, or base leakage is sufficiently high to render the processed water unsuitable for a specific use. Determination of this value may be by conductivity, pH, or other measurements, and action required will generally be to remove the unit or system from service at this point.

*equivalents per liter (eq/L):* concentration of ion equivalents that can be removed per liter of solution. An equivalent is the molecular weight in grams of the compound divided by its electrical charge or valence. For example, a resin with an exchange capacity of 1 eq/L could remove 37.5 g of divalent zinc ($Zn^{+2}$, molecular weight of 65) from solution. Since 1 eq/L is equal to 21.8 $kgr/ft^3$, equivalents per liter can be converted to kilograins per cubic foot by multiplying by 21.8.

*exchanger:* pressure vessel containing ion exchange material through which liquid to be processed is passed. In some process applications, the exchanger may contain mixtures of cation and anion resins, or even mixtures of different cation or anion resins, such as stratified-bed units.

*exhaustion:* state in which the resin is no longer capable of useful ion exchange; the depletion of the exchanger's supply of available ions. The exhaustion point is determined arbitrarily in terms of

(*a*) value in parts per million of ions in the effluent solution

(*b*) the reduction in quality of the effluent water determined by a conductivity bridge that measures the electrical resistance of the water, or any other suitable criteria such as leakage of radionuclides.

*external regeneration:* procedure in which the ion exchange resin is sluiced or transferred (hydraulically, pneumatically, or by gravity) after exhaustion into another vessel wherein regeneration occurs.

*filter – nonprecoat:* mechanical device primarily used for removal of particulate oxides from condensate or other liquids as pretreatment ahead of many types of high purity water treatment processes. The device may contain permanent elements or media that is mechanically or hydraulically cleaned every service cycle or disposable media that is replaced every service cycle.

*filter – precoat:* mechanical device primarily used for removal of particulate oxides from condensate. The device may contain tubular elements that are precoated with materials that can include various fibers, activated carbon, powdered ion exchange resins, or mixtures of those materials. The precoat material is replaced every service cycle.

*flux:* measure of the rate at which the permeate passes through the membrane per unit area of membrane, for example, gal/ft$^2$/day (gfd).

*freeboard:* in certain ion exchange units and granular media filters, the space provided between the media/water interface and the water/collector/distributor to allow for expansion of the resin or media bed during backwashing, commonly expressed dimensionally or as a percentage of media depth.

*free mineral acidity (FMA):* amount of acidity present in water when leakage is subtracted from TMA; FMA = TMA – leakage.

*hardness:* scale-forming and lather-inhibiting qualities possessed by water, high in calcium and magnesium ions.

*hardness, permanent:* caused by the presence of calcium sulfate, magnesium sulfate, and the chlorides of these two metals that cannot be removed by boiling.

*hardness, temporary:* caused by the presence of magnesium or calcium bicarbonate; referred to as temporary because the hardness may be removed by boiling the water to convert the bicarbonates to the insoluble carbonates.

*hardness, total:* numeric sum of the hardness ions calcium and magnesium, both expressed as calcium carbonate ($CaCO_3$), that is used to determine work loading in simple ion exchange softening.

*hardness, total acidified:* numeric sum of the hardness ions calcium and magnesium, both expressed as calcium carbonate ($CaCO_3$), in a sample that has been acidified to dissolve unreacted lime carryover in the effluent of lime softening processes.

*hardness as calcium carbonate:* expression ascribed to the value obtained when the hardness-forming salts are calculated in terms of equivalent quantities of calcium carbonate, a convenient method of reducing all salts to a common basis for comparison.

*hollow fiber modules:* membrane material spun into hollow, hair-like fibers 200 μm diameter or less.

*hybrid systems (EDI, EDR):* may combine elements of both ion exchange and membrane technologies, which will typically consist of all equipment, including service vessels, media, all piping, pumps, valves, controls, and instrumentation.

*hydrogen cycle:* operation of a cation exchange cycle wherein the removal of specified cations from the influent water is accomplished by exchange with an equivalent amount of hydrogen ion from the exchange material.

*influent:* liquid, solid, or gaseous material being introduced into a process; in this Code, untreated or partially treated water introduced for processing into an ion exchange system.

*inside out:* term used in cartridge and membrane filtration to describe flow path of liquid from internal passage to the outside.

*in situ:* see *regeneration* (in place, in situ)

*ion exchange:* reversible process by which ions are interchanged between a solid and a liquid with no substantial structural changes of the solid.

*ion exchange capacity (operating):* portion of the total ion exchange capacity of an ion exchange resin bed that can be achieved in a practical ion exchange operation. Commonly expressed in kilograins per cubic foot (kgr/ft$^3$) or equivalents per liter (eq/L).

*ion exchange capacity (volume):* also referred to as total exchange capacity (TEC) or wet volume capacity, it is the theoretical number of exchangeable ions per unit volume or weight of resin. The TEC can only be attained by a total and complete regeneration that is not employed in practice due to the prohibitive regenerant costs that would be required. TEC is typically expressed in terms of kilograins capacity per cubic foot (kgr/ft$^3$) of resin material.

*ion exchange capacity (weight basis):* also referred to as dry volume capacity, the number of milliequivalents of exchangeable ions per dry gram of ion exchange material in its standard form.

*ion exchange material:* insoluble solid that has the ability to exchange reversibly certain ions in its structure or attached to its surface as functional groups with ions in a surrounding medium.

*ion exchange resin:* synthetic organic ion exchange material, usually in spherical form. (See also *anion exchange* and *cation exchange materials*.)

*ion exchange resin – powdered:* finely divided particles of crushed ion exchange resin beads used as a disposable precoat filter material in condensate purification systems.

*ion exchange systems:* typically consist of all equipment, including service vessels, ion exchange resins, all piping, pumps, valves, controls, and instrumentation and may be designed for

(*a*) in-place resin regeneration that is performed in the service vessel.

(*b*) external regeneration that is accomplished in specifically designed regeneration vessels that are part of the particular ion exchange system.

(*c*) external regeneration that is accomplished in specifically designed regeneration vessels off site.

(*d*) nonregenerable. These resins are normally disposed of after their service capacity has been utilized.

*ion exchange unit (conventional):* unit in which backwash freeboard is included above the resin bed and that is backwashed before every regeneration or very frequently.

*ion exchange unit (packed bed):* unit, which is essentially completely full of resin along with an inert resin layer, and that is regenerated in a countercurrent fashion. The system may or may not be designed to include in situ backwash and may or may not require infrequent, external backwash.

*iron:* may be soluble or a particulate oxide.

*kilograins per cubic foot (kgr/ft$^3$):* concentration of ions that can be removed by a cubic foot of ion exchange resin. Kilograins per cubic foot can be converted to equivalents per liter by dividing by 21.8.

*leakage:* amount of influent ion or contaminant present in the processed, effluent water. Generally, a function of regenerant level of the ion exchange resin material, and also a function of the flow dynamics within the exchanger. May be expressed as a percentage of inlet concentration, or in dimensionless part-per-million (ppm) or part-per-billion (ppb) ion or salt terms.

*membrane systems:* typically consist of all equipment, including service vessels, membranes, all piping, pumps, valves, controls, and instrumentation.

*membranes:* thin barriers or films of material that allow certain substances to pass through.

*microfiltration:* process that discriminates between particles on the basis of size using a membrane with a usual pore size of 0.45 μm.

*micron:* measurement of size equivalent to 1 μm.

*millipore filter test:* widely accepted visual indication, established by a special filtration test, used to determine concentration and nature of suspended metal oxides. Typically used on condensate samples. See Mandatory Appendix II.

*mixed-bed unit:* use of intimately mixed hydrogen form cation resins and hydroxide-form anion resins in a single ion exchange vessel; this type of equipment usually provides the highest quality treated water.

*mixed resin unit (special ionic form):* use of intimately mixed cation and anion exchange resins in forms other than hydrogen and hydroxide. Such units are used in some applications for treatment of contaminated steam condensate. Other applications find use in treatment of nuclear primary coolant and nuclear waste management flows, performing soluble radionuclide removal as well as filtering capability for particulate removal.

*multibed systems:* combination of cation and anion exchangers, usually in series flow and sometimes in combination with mixed-bed exchangers, for treating and processing water and/or dilute aqueous solutions.

*nanofiltration:* membrane liquid separation technology that is positioned between reverse osmosis and ultrafiltration. While RO can remove the smallest of solute molecules, in the range of 0.0001 μm in diameter and smaller, nanofiltration (NF) removes molecules in the 0.001 μm range. NF is a polypiperazine amide membrane and is essentially a lower-pressure version of reverse osmosis where the purity of product water is not as critical as pharmaceutical grade water, for example, or the level of dissolved solids to be removed is less than what is typically encountered in brackish water or seawater. As such, nanofiltration is especially suited to treatment of well water or water from many surface supplies. NF membranes are often referred to as softening membranes because of their ability to remove divalent ions such as calcium and magnesium but allow a large percentage of monovalent ions such as sodium to pass through.

*off site regeneration processes:* ion exchange unit regeneration performed off site to transfer regeneration waste disposal to a suitable facility. This is most easily accomplished by transfer of both the containment vessel and resin to the offsite facility but may also involve transfer of resin only.

*operating capacity:* true measure of a resin's ability to remove a given number of ions from solution. It is defined as the number of exchangeable ions available under a given set of conditions. These conditions are variable from one case to the next, and consist of such parameters as influent composition, effluent requirements, flow rate, concentration of regenerant used (i.e., degree of regeneration), temperature, exhaustion and rinse end points, resin selectivity, and particle size. The operating capacity is commonly expressed in ratios, in which the numerator is the weight of ions removed and the denominator is the volume of the bed [i.e., pounds per cubic foot (lb/ft$^3$), kilograins per cubic foot (kgr/ft$^3$), or equivalents per liter (eq/L)].

*operating cycle:* ion exchange process consisting of a regeneration phase and a service run.

*organic trap:* application of selected ion exchange resins to remove large organic molecules from water to be treated. This selection can be made in lieu of gravity separation and flocculation equipment, such as conventional clarifiers, to remove organics.

*osmosis:* spontaneous transport (diffusion) of solvent across a semipermeable membrane that separates two solutions of different concentration.

*osmotic pressure:* pressure that must be applied to a more concentrated solution to halt flow through a semipermeable membrane from the less concentrated solution into the more concentrated solution.

*outside in:* term used in cartridge and membrane filtration to describe flow path of liquid from external passage to the inside.

*packed bed ion exchange:* see *ion exchange unit (packed bed).*

*particulate matter:* nonliquid matter, exclusive of gases, that is heterogeneously dispersed in water.

*pass(es):* description of the system configuration in which the permeate from one module is the feedwater to a subsequent pass operating in series when permeate from the system is taken to service from the final pass only.

*permeate:* purified liquid exiting the system.

*pH:* expression of the acidity of a solution; the negative logarithm of the hydrogen ion concentration (pH 1, very acidic; pH 14, very basic; pH 7, neutral).

*pressure differential:* difference between the inlet and outlet pressures, measured across the media.

*pressure vessel (media):* device operating at greater than atmospheric pressure containing one or more types of media commonly used for filtration, ion exchange, or other processes.

*pressure vessel (module):* device containing one or more membrane elements.

*recovery:* measure (%) of how much of the feed stream is recovered as permeate.

*recycle:* the portion of the concentrate stream that is reclaimed by injection into, and blending with, the feedwater to the system.

*regenerant:* solution used to restore the activity of an ion exchanger. Acids are employed to restore a cation exchanger to its hydrogen form; brine solutions may be used to convert the cation exchanger to the sodium form. The anion exchanger may be rejuvenated by treatment with an alkaline solution.

*regeneration:* replacement of contaminating ions on the resin exchange sites, removed from the influent water, with desirable ions present in the regeneration chemical, thereby restoring the resin to acceptable working condition.

*regeneration (in place, in situ):* procedure in which the regenerant solutions are brought to the exhausted resin in the service vessels, and regeneration is conducted.

*regeneration efficiency:* regeneration efficiency, a measure of regenerant utilization, is commonly expressed either as pounds of regenerant per kilograin of exchange capacity as $CaCO_3/ft^3$, or as a percentage of what would be the stoichiometric quantity (one equivalent of regenerant per equivalent of ions exchanged).

*regeneration level/dosage:* weight of regenerant used per unit quantity of ion exchange material in a single regeneration. It is usually expressed in terms of pounds of regenerant (as 100%) per cubic foot of fully hydrated ion-exchange material, in a specific ionic form, measured after backwash and draining.

*regeneration phase:* cycle consisting of minimum of four steps: resin separation (backwashing) to clean the bed, introduction of the regenerant, slow rinse or displacement to push the regenerant slowly through the bed, and finally fast rinse to remove all traces of regenerant from the resin. In packed bed ion exchange systems, backwash may be infrequent or employ special procedures and/or special external vessels.

*rejection:* measure (%) of the fraction of salts that does *not* pass through the membrane.

*resin fouling:* phenomenon in which weak organic acids are absorbed by strong base anion resins and are not fully removed during regeneration; instead accumulate in, block the exchange sites, thus diminishing the capacity of the resins. In addition, the effluent quality deteriorates resulting in leakage of the organic acids, greater leakage of total dissolved solids, equivalent higher conductivity and lower pH values. Larger rinse volumes requiring more time may also be incurred during the regeneration phase. Fouling of ion exchange resins may also be caused by impure grade of regenerants, high levels of influent turbidity during the service run, precipitation of slightly soluble salts, iron or manganese and bacteria, iron in the water being processed (also during the service run).

*resin separation (backwash):* separation of the anion resin from the cation resin in mixed bed or layered bed units by backwashing. Separation due to the difference in specific gravities of the anion resin and cation resin, normally the anion resin being lighter will float to the top of the cation resin.

*resin trap:* strainer used to prevent discharge of resins from exchanger vessels into the vessels' effluent if the outlet distributor fails.

*resistivity:* the electrical resistance of a solution is a general property, and is not specific for any particular ion. A measurement of resistivity can determine the quality of the effluent from certain ion exchange processes and is an excellent method of assessing total salt content. The value is reported in $M\Omega/cm^2$.

*reverse osmosis:* essentially the same process as osmosis except that pressure is applied to the more concentrated solution to force solvent flow through the semipermeable membrane into the less concentrated solution.

*rinse:* part of the regeneration phase of an ion exchange process in which water is passed through the bed of ion exchange material to remove the residual regenerant solution.

*rinse requirement:* measure of the volume of water used during regeneration before a resin can be put into service. Rinse requirements may be influenced by impure grade of regenerants, hydraulic channeling, contamination fouling, and deterioration of the resin.

*salt passage:* measure (%) of the dissolved salts passing through the membrane.

*semipermeable membrane:* membrane that allows some substances to pass through.

*sequential regeneration schedule:* units of dissimilar types in a system are regenerated together, but one unit is fully regenerated before the other unit starts regeneration.

*service run:* part of the operating cycle of an ion exchange process in which the influent is passed through a bed of the ion exchange material to exchange specific ions from the water for an equivalent amount of specific ions from the ion exchange material.

*silt density index:* widely accepted numerical indication, established by a special filtration test, used to predict colloidal particle fouling of membranes.

*sodium cycle:* operation of a cation exchange cycle wherein the removal of specified cations from the influent water is accomplished by exchange with an equivalent amount of sodium ion from the exchange material.

*softener:* ion exchange equipment containing cation exchange resin used for the removal of hardness ions (calcium and magnesium), exchanging them for sodium ions.

*spiral wound modules:* two layers of semipermeable membrane separated by a porous, woven fabric support and formed into an envelope that is then wound around a central perforated tube.

*stage(s):* description of the system configuration in which the concentrate from one module is the feedwater to a subsequent stage when the permeate from each is combined as a single stream.

*starvation process:* process used with weak acid cation resin regeneration to minimize production of free mineral acidity in the treated effluent. This is accomplished by the use of virtually stoichiometric regeneration dosages adjusted only for the alkalinity concentration in the acid dilution water.

*stoichiometric:* see *regeneration efficiency.*

*strainer plate:* internal device, installed at the top or bottom of an ion exchange unit, or as a dividing plate between two compartments, equipped with special strainers and that acts as a distributor and/or collector of service liquid and regenerant solutions in packed bed ion exchange units.

*stratified (layered) bed unit:* application of layers of weak-acid and strong-acid cation exchange resins, or weak-base and strong-base anion exchange resins in ion exchange vessels. This application is typically made to improve treatment efficiency.

*suspended solids:* matter such as clay, silt, finely divided organic matter, plankton, and other microscopic organisms that give rise to turbidity.

*theoretical free mineral acidity (TMA):* free mineral acidity that would result from the conversion of all of the anions of strong acids in solution to their respective free acids.

*thin film composite:* membrane consisting of a thin semipermeable skin on a thicker porous backing.

*thoroughfare regeneration:* regeneration of a weakly functional resin using waste dilute regenerant chemical from a strongly functional resin in a downstream position. In some cases the waste regenerant from the strongly functional resin is further diluted before injection into the upstream unit.

*total organic carbon (TOC):* the amount of carbon bound in an organic compound often used as a nonspecific indicator of water quality, expressed as milligrams per liter (mg/L).

*train interconnection:* ion exchange unit interconnection method where all units of dissimilar type or function are interconnected, operated, and regenerated as a discrete set.

*tubular element:* membrane is inserted into, or coated onto, the inside surface of a porous tube that may range in size from 30 mm to 250 mm ($\frac{1}{8}$ in. to 1 in.) diameter.

*turbidity:* an expression of the optical properties of a sample that causes light rays to be scattered and absorbed rather than transmitted in straight lines through the sample. This is generally due to the presence of suspended matter.

*ultrafiltration:* process like microfiltration, which discriminates between particles on the basis of size using a membrane with a pore size range of 0.0015 μm to 0.2 μm.

*ultra pure water:* water with a specific conductance less than 1.0 μS/cm$^2$ or greater than 10 MΩ/cm$^2$.

*units of measurement:* units used to express concentrations, based on number, volume, or weight.

*equivalents per liter (eq/L):* concentration of ion equivalents that can be removed per liter of solution. An equivalent is the molecular weight in grams of the compound divided by its electrical charge or valence. For example, a resin with an exchange capacity of 1 eq/L could remove 37.5 g of divalent zinc ($Zn^{+2}$), molecular weight of 65, from solution. Since 1 eq/L is equal to 21.8 kgr/ft, equivalents per liter can be converted to kilograins per cubic foot by multiplying by 21.8.

*kilograins per cubic foot (kgr/ft$^3$):* the weight of ions, expressed as calcium carbonate ($CaCO_3$) equivalents that can be removed by a cubic foot of ion exchange resin. Kilograins per cubic foot can be converted to equivalents per liter by dividing by 21.8 or to pounds of calcium carbonate ($CaCO_3$) equivalents by dividing by 7.0.

*milligrams per liter (mg/L):* number of thousandths of a gram of a substance in one liter of water. To convert from moles to milligrams, multiply the number of moles by the atomic or molecular weight of the substance and multiply by one thousand.

*moles per liter:* number of particles of a substance, expressed in moles, in one liter of water. One mole is $6.02 \times 10^{23}$ particles. Moles per liter (molarity) is abbreviated M. Thus, $10^{-7}$ M $H^+$ equals $6.02 \times 10^{16}$ hydrogen ions per liter.

*parts per million (ppm):* parts by weight of an ion or species in a given weight of solution. Thus, 0.025 g of calcium ion in one liter of water is 25 ppm, or 25 parts by weight in 1,000,000 parts by weight of solution.

*parts per billion (ppb):* parts by weight of an ion or species in a given weight of solution. Thus, 0.000025 g of calcium ion in one liter of water is 25 ppb, or 25 parts by weight in 1,000,000,000 parts by weight of solution.

*parts per trillion (ppt):* parts by weight of an ion or species in a given weight of solution. Thus, 0.000000025 g of calcium ion in one liter of water is 25 ppt, or 25 parts by weight in 1,000,000,000,000 parts by weight of solution.

*percent (volume/volume):* percent of total solution volume contributed by the dissolved species that has been added to the solution. Thus, 40% (v/v) methanol is 40 mL of methanol in enough water to bring the solution to 100 mL.

*percent (weight/weight):* percent of total solution weight contributed by the dissolved species. Thus, 10% $KNO_3$ (w/w) is 10 g of $KNO_3$ in 90 g of water. If not otherwise specified, percent implies percent by weight.

*valence:* all ions carry a charge that may be positive or negative. The amount and direction of the charge is the valance of the ion. A positively charged ion is a cation while a negatively charged ion is an anion. For example, calcium is a divalent cation bearing two positive charges while sodium is monovalent bearing one positive charge.

*zeolite:* group of complex hydrated aluminum silicates, either naturally occurring or synthetically manufactured with limited cation exchange properties.

## 2-2 REFERENCES

Unless otherwise specified, the standards referenced shall be the most recent at the time of order placement.

CRTD-Vol. 81, Consensus on Operating Practices for the Sampling and Monitoring of Feedwater and Boiler Water Chemistry in Modern Industrial Boilers. An ASME Research Report.

ASME Handbook on Water Technology for Thermal Power Systems

Publisher: The American Society of Mechanical Engineers (ASME), Three Park Avenue, New York, NY 10016-5990; Order Department: 22 Law Drive, P.O. Box 2900, Fairfield, NJ 07007-2900 (www.asme.org)

ASTM D511, Standard Test Methods for Calcium and Magnesium in Water

ASTM D512, Standard Test Methods for Chloride Ion in Water

ASTM D513, Standard Test Methods for Total and Dissolved Carbon Dioxide in Water

ASTM D516, Standard Test Method for Sulfate Ion in Water

ASTM D596, Standard Guide for Reporting Results of Analysis of Water

ASTM D857, Standard Test Method for Aluminum in Water

ASTM D858, Standard Test Methods for Manganese in Water

ASTM D859, Standard Test Method for Silica in Water

ASTM D888, Standard Test Methods for Dissolved Oxygen in Water

ASTM D992, Standard Method of Test for Nitrate Ion in Water

ASTM D1067, Standard Test Methods for Acidity or Alkalinity of Water

ASTM D1068, Standard Test Methods for Iron in Water

ASTM D1125, Standard Test Methods for Electrical Conductivity and Resistivity of Water

ASTM D1126, Standard Test Method for Hardness in Water

ASTM D1179, Standard Test Methods for Fluoride Ion in Water

ASTM D1193, Standard Specification for Reagent Water

ASTM D1246, Standard Test Method for Bromide Ion in Water

ASTM D1252, Standard Test Methods for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Water

ASTM D1253, Standard Test Method for Residual Chlorine in Water

ASTM D1293, Standard Test Methods for pH of Water

ASTM D1426, Standard Test Methods for Ammonia Nitrogen in Water

ASTM D1687, Standard Test Methods for Chromium in Water

ASTM D1688, Standard Test Methods for Copper in Water

ASTM D1691, Standard Test Methods for Zinc in Water

ASTM D1782, Standard Test Methods for Operating Performance of Particulate Cation-Exchange Materials

ASTM D1783, Standard Test Methods for Phenolic Compounds in Water

ASTM D1886, Standard Test Methods for Nickel in Water

ASTM D1890, Standard Test Method for Beta Particle Radioactivity of Water

ASTM D1943, Standard Test Method for Alpha Particle Radioactivity of Water

ASTM D2036, Standard Test Methods for Cyanides in Water

ASTM D2187, Standard Test Methods for Physical and Chemical Properties of Particulate Ion-Exchange Resins

ASTM D2330, Standard Test Method for Methylene Blue Active Substances

ASTM D2460, Standard Test Method for Alpha-Particle-Emitting Isotopes of Radium in Water

ASTM D2580, Standard Test Method for Phenols in Water by Gas-Liquid Chromatography

ASTM D2687, Practice for Sampling Particulate Ion-Exchange Materials

ASTM D2791, Standard Test Methods for On-line Determination of Sodium in Water

ASTM D3081, Method of Test for Selenium in Water

ASTM D3082, Standard Test Method for Boron in Water

ASTM D3087, Standard Test Method for Operating Performance of Anion-Exchange Materials for Strong Acid Removal of Quantities of Copper in High Purity Water Ueed in the Electronics Industry

ASTM D3375, Standard Method for Column Capacity of Particulate Mixed Bed Ion Exchange Materials

ASTM D4195, Standard Guide for Water Analysis for Reverse Osmosis and Nanofilter Application

ASTM D4266, Standard Test Methods for Precoat Capacity of Powered Ion-Exchange Resin

ASTM D4456, Standard Methods for Physical and Chemical Properties of Powdered Ion-Exchange Resins

ASTM D4548, Standard Test Method for Anion–Cation Balance of Mixed Bed Ion-Exchange Resins

ASTM D4582, Standard Practice for Calculation and Adjustment of the Stiff Davis Stability Index for Reverse Osmosis

ASTM D4993, Standard Practice for Calculation and Adjustment of Silica ($SIO_2$) Scaling for Reverse Osmosis

ASTM D5042, Standard Test Method for Estimating the Organic Fouling of Particulate Anion Exchange Resins

ASTM D5090, Standard Practice for Standardizing Ultrafiltration Permeate Flow Performance Data

ASTM D5091, Standard Guide for Water Analysis for Electrodialysis/Electrodialysis Reversal Applications

ASTM D5217, Standard Guide for Detection of Fouling and Degradation of Particulate Ion Exchange Materials

ASTM D5627, Standard Test Method for Water Extractable Residue form Particulate Ion-Exchange Resins

ASTM D6161, Standard Terminology Used for Microfiltration, Ultrafiltration, Nanofiltration and Reverse Osmosis Membrane Processes

ASTM D6302, Standard Practice for Evaluating the Kinetic Behavior of Ion Exchange Resins

ASTM D6529, Standard Test Method for Operating Performance of Continuous Electrodeionization Systems on Feeds from 50 – 1000 μS/cm

ASTM D6908, Standard Practice for Integrity Testing of Water Filtration Membrane Sytems

Publisher: American Society for Testing and Materials (ASTM International), 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959 (www.astm.org)

# Section 3
# Guiding Principles

## 3-1 ADVANCE PLANNING FOR TEST

### 3-1.1

The parties to any test under this Code shall reach agreement as to the location of instrument connections, position of valves, and arrangement of piping. When the interests of more than one party are involved, the parties to the test shall come to an agreement in writing and in advance of the test.

### 3-1.2

Items on which agreement shall be reached are as follows:

*(a)* basis of guarantee conditions, test objective(s), duration of test, preliminary tests, methods of computing results, and expense of testing program.

*(b)* need for, and extent of, equipment inspection before test and after test.

*(c)* deviations, if any, from Test Code procedures.

*(d)* personnel, including supervisor, conducting and observing test.

*(e)* that test objectives(s) can be realized under feasible test conditions with the existing installations.

*(f)* whether the system is to be tested "as found" or after being conditioned for test. If the latter, the extent of the conditioning of the equipment required to make apparatus ready for test.

*(g)* arrangement of test apparatus, instrumentation, and flow and cycle diagrams showing test points and data to be obtained. To avoid costly modifications, it should be noted that the instrumentation needed for a code test usually exceeds that normally provided for operation. The utilization of this Test Code should be discussed at such time that the required connections and valves may be provided for in the purchaser's piping and equipment design.

*(h)* conditions to be held constant during test and the permissible deviations there from. Methods of extrapolating test data and results to the design conditions, if deviations greater than those previously agreed upon occur.

*(i)* whether influent water chemistry and characteristics are comparable to the design conditions, if not, adjust operating parameters.

*(j)* whether influent is to be treated, and if so, the treatment methods to be used.

*(k)* analytical methods and limits of reporting accuracy for influent and effluent composition. Refer to Section 4, Instruments and Methods of Measurement.

*(l)* the choice of instruments and the probable limits of accuracy of the test results considering test procedures and probable accuracy of instruments selected, as given in the Supplements on Instruments and Apparatus.

*(m)* procedures for inspecting the high-purity water treatment equipment to be sure overall system design, type and amount of media are as previously agreed. See Mandatory Appendix II on membranes and Nonmandatory Appendix C on resins.

## 3-2 GENERAL DESCRIPTION OF TEST REQUIREMENTS

*(a)* Before conducting a test, all parties should have the right to examine the equipment to ensure that it is in the condition agreed upon, to run a brief preliminary test to give assurance that the equipment is capable of reliable and continuous operation, and to be satisfied that it is in condition to undergo performance testing.

*(b)* If the equipment is to be tested in the "as found" condition, the following should be recorded if applicable:

  *(1)* the length of time the equipment has been in service

  *(2)* the number of operational cycles the equipment has completed in the case of ion exchange systems

  *(3)* the total volume of effluent produced since placed in service

  *(4)* any unusual conditions that occurred prior to the test

  *(5)* any reported system malfunctions or production of off specification water

*(c)* All control devices and measuring instruments should be calibrated and should be checked for proper installation and operation as indicated in Section 4.

## 3-3 PRELIMINARY TESTS

A preliminary test of at least one complete cycle, or predetermined time frame (e.g., RO or UF cycle could be 7-day to 30-day cycle between cleaning) after establishment of steady state conditions should be carried out to determine whether the equipment is operating satisfactorily and whether test procedures given herein, or agreed upon, are feasible. During this test it should be ascertained that the equipment, controls, sample points, and instrumentation are functioning to ensure the recording of valid test data. Data obtained from this preliminary test should be used to determine if calibration procedures are acceptable. When preliminary inspection and test indicate that the limiting conditions for which the equipment was designed cannot be met, the parties to the test shall agree upon alternate conditions before the official test. The probable effect of these alternate conditions upon the performance of the apparatus shall also be agreed upon before the test unless it is agreed that one of the objects of the test will be to determine this effect.

## 3-4 FREQUENCY OF OBSERVATIONS

Test measurements necessary to obtain the data previously agreed upon shall be taken at sufficient intervals during the test periods to describe the performance of each step of the cycle. Frequency of readings shall be agreed upon in advance. Raw water quality and the influent composition to the equipment to be tested should be monitored during the testing. If any of these compositions are other than what was agreed upon earlier, as a result of a change of influent water quality or malfunctioning of mechanical equipment, corrective measures should be taken.

## 3-5 DURATION OF TEST RUNS

The tests shall be of duration necessary to determine performance and to average out normal variations.

### 3-5.1 Ion Exchange Equipment

It is recommended that the test consist of at least three exhaustion and regeneration cycles to allow the resin to reach operating equilibrium condition.

### 3-5.2 Condensate Polishing

It is recommended that the test consist of both high and low flows, where the service cycle is normally of a very long duration. The parties involved shall agree on the number of test cycles sufficient to establish performance.

### 3-5.3 Reverse Osmosis, Submicron and Ultrafiltration Equipment

It is recommended that the test consist of a 24-hr to 48-hr cycle.

## 3-6 GENERAL DESCRIPTION OF TEST PROCEDURES

### 3-6.1 Ion Exchange Testing

Ion exchange systems, as described in the introductory statements, require certain measurements during a performance/acceptance test. Although it is not possible to detail all measurements desired on specific ion exchange systems, it is the intent of this part of the Test Code to indicate those test procedures that are generally required to establish system performance.

Differential pressure, flow, temperature measurements, and resin volumes are primarily used for the determination of hydraulic characteristics of ion exchange systems. They may be utilized to determine overall system characteristics, or may be used to measure the performance of various subcomponents of the ion exchange system (e.g., individual differential pressure drop across a single tank in a multi-tank system). Similarly, flow measurements may be applied to determine backwash and rinse properties of individual subcomponents. Flow and time or volume measurements may also be used to verify chemical properties of ion exchange systems. Certain hydraulic properties, such as flow distribution within the ion exchange vessels, are not included in this part of the Test Code. Chemical test methods as outlined in para. 3-6.2 are more sensitive in the determination of such hydraulic characteristics than basic flow and differential pressure measurements.

**3-6.1.1 Differential Pressure.** The measurement of differential pressure (pressure drop) across the ion exchange system, or subcomponents thereof, is normally accomplished by the use of line-mounted pressure gages, dual gages, or transmitters of various mechanical, pneumatic, or electronic designs, as described in Section 4.

The rate of particulate fouling of ion exchange resin beds as well as degradation, such as oxidation of resins, can be determined by the use of differential pressure measurements. Mechanical restrictions of flow would also be a source of an increased pressure differential. Effectiveness of resin bed backwash operations can also be determined by measuring pressure drop across the resin bed both before and after the backwash cycle provided sufficient time is allowed for bed compaction to occur.

**3-6.1.2 Flow.** Evaluation of various flow streams can be accomplished by the use of suitable measurement devices as outlined in Section 4. Flow rate and volume measurement may be applied to determine backwash, regeneration, rinse, and service parameters of ion exchange subcomponents. Backwash properties shall be observed to determine backwash effectiveness in the time prescribed. Backwash effluent shall be sampled periodically during the backwash cycle and observed for clarity and the presence of resin.

NOTE: Packed bed ion exchange units are not backwashed in situ so this part of the evaluation is not applicable. Suitable types of flowmeters can also be used to measure the total volume of water treated. The capability to measure ion exchange process flow rates and volume will establish, when coupled with suitable chemical analyses, overall ion exchange system performance.

**3-6.1.3 Temperature.** Section 4 refers to temperature measurement methods suitable for use in ion exchange equipment. Temperature measurements are important

### Table 3-6.1.4-1 Resin Volume Change Chemical Form-to-Form

| Resin Type | Resin Volume Increase Form-to-Form |
|---|---|
| SAC – gel – 8% crosslink | Na → H = +5% |
| SAC – gel – 10% crosslink | Na → H = +5% |
| SAC – macroporous | Na → H = +4% |
| WAC – acrylic | H → Ca = +15% |
| SBA type I DVB gel | Cl → OH = +15% |
| SBA type I – porous DVB gel | Cl → OH = +20% |
| SBA type II DVB gel | Cl → OH = +10% |
| SBA type I acrylic | Cl → OH = +15% |
| SBA type I macroporous | Cl → OH = +15% |
| SBA type II macroporous | Cl → OH = +10% |
| WBA DVB macroporous | Free amine → HCl = +20% |
| IBA epoxy amine | Free amine → HCl = +20% |

GENERAL NOTE: This table may not include some types of resin. In addition, the expansion quoted usually occurs only when all active sites have been converted to one form or the other. In normal operation the expansion may be ≈ 50% of that indicated in the table.

during the determination of proper backwash flow rates on ion exchange tanks so that optimum backwashing takes place. Additionally, regenerant solution and water sample temperatures must be measured during the determination of process efficiency, since abnormal regenerant temperatures will result in resin capacity deviations. Variations in sample temperatures will also influence conductivity, pH, and various colorimetric analyses. Samples should be temperature conditioned or, alternately, temperature compensation utilized.

**3-6.1.4 Resin Volume in a Nonpacked Bed System.** Ion exchange resin beads swell and shrink when passing from the regenerated form to the exhausted form. The degree and direction of physical change varies by resin functionality, chemical structure, physical structure, bead size, and size distribution.

Resin column measurements are made to determine if the ion exchange unit contains the proper quantity of resin. The chemical form of the resin at the time measurement is made is very important and the resin manufacturer's data sheet should be consulted on this point. For an example, see Table 3-6.1.4-1. Also, resins will undergo a certain amount of irreversible expansion during the first operational cycle; therefore, it is desirable to place the unit through one operational cycle before the resin volume measurement is made.

The following method for resin volume measurement is one of many that can be utilized for this purpose; in any method, due consideration must be given to vessel head volume and internal distributor displacement volume.

*(a)* Backwash unit so that resin bed is expanded at least 50%, for a 10-min period.

With the vessel at atmospheric pressure at a flow rate not to exceed 1 gpm/ft$^2$, drain the water level in the unit down to where it is 6 in. above the resin. Measurement of the resin bed depth should be made, being careful not to jar the vessel or disturb the resin bed. With the measurement of resin bed depth, knowing the inside diameter of the unit, the resin volume can be calculated. Conversely, freeboard volume may be calculated and this value subtracted from the entire tank volume, which will then give the gross resin volume. Subtraction of distributor displacement volume (available from the equipment manufacturer) then completes the calculation to produce net resin volume.

**3-6.1.5 Resin Volume in a Packed Bed System.** Every packed bed unit must be designed with a specific maximum allowable freeboard if the unit is to meet the treated water purity requirement. To do so, the changes in resin size and volume must be dependably predicted and the design freeboard maintained in the unit at all times.

Each packed bed design has specific requirements for the installation and maintenance of the required resin volume and it is not possible to provide details of all the various designs in this document.

Therefore, specific procedures for confirming resin bed height and volume must be provided by individual equipment manufacturers.

Should a unit or system fail to meet specified purity either at startup or in subsequent operation, resin volume and degree of actual swelling/shrinking should be determined.

### 3-6.2 Regenerant Quality and Purity

The quality and purity of regenerants used in ion exchange operations are critical to both the system's effluent quality as well as the operating life of the resin. Many different regenerants can be used depending on the application. As a rule, the purer the water the system is expected to produce, the purer the regenerants need to be to produce the expected water quality. Mandatory Appendix I lists the more common regenerants used in ion exchange operations and the maximum contaminant levels in the regenerant required for high-purity water production. Low-purity regenerant chemicals will also reduce the operating life of the resin by causing premature fouling and/or chemical degradation of the resin.

Whenever dealing with high-purity regenerant chemicals it is always a good operating procedure to demand a Certificate of Analysis (COA) from the supplier for the specific batch or lot number that is being received.

### 3-6.3 Common Impurities That May Require Measurement During Ion Exchange System Performance Testing

See Table 3-6.3-1.

### 3-6.4 Chemical Measurements During Ion Exchange System Performance Testing

See Table 3-6.4-1.

## 3-7 MEMBRANES TESTING

### 3-7.1 Physical Inspection of Element

This is a nondestructive test to observe the physical integrity of the element and visually identify potential foulants. This test is used to help the technician determine what steps to take in fully analyzing element performance. All elements are visually inspected noting any differences from new product. (Examples would be cracked end caps, discolored scrolls, fiberglass discoloration, etc.)

### 3-7.2 Performance Testing

This test involves the determination of element operating performance, with a comparison to typical properties and original production test data (when available, not applicable to dry product). This is also a nondestructive test, used to determine how the element is performing at standard test conditions.

### 3-7.3 Element Autopsy as a Destructive Test

This requires the element to be cut lengthwise to allow the membrane to be unrolled. Two cuts must be made, on opposite sides, just deep enough to penetrate the element casing. The element is unrolled carefully so as to not damage the membrane surface. The surface is fully examined and sprayed with cleaning chemicals to check for reaction.

### 3-7.4 Oxidative Testing: Dye Test/Fujiwara Test

Two tests can be performed to determine if the membrane has been oxidized. A quick test is to drop a small quantity of a dye solution on the membrane surface. The dye will adhere readily to support material that has been exposed, due to damage in the membrane barrier layer. These damaged areas will appear as bright pink spots. The dye will not adhere to undamaged membrane. Another form of oxidative testing is a Fujiwara test. Using a test solution of pyridine and sodium hydroxide, small coupons of membrane are put in the solution and heated. A positive response is indicated by a red or pink color in the pyridine layer. This shows the membrane has been oxidized by chlorine or halogens.

### 3-7.5 Electron Spectroscopy for Chemical Analysis or X-Ray Fluorescence Testing

These tests involve the identification of impurities which have been organically bound to the membrane surface. It is predominantly used to identify the source of oxidation. Because the composition of the membrane is known, anything else remaining on the surface indicates that oxidation of the membrane has occurred. This is a more thorough test than the oxidative dye test or Fujiwara test, and therefore, requires a longer lead time.

### 3-7.6 Analytical Testing

Two types of analytical testing can be done including inorganic testing, such as inductively coupled plasma (ICP) emission spectroscopy, which helps identify specific metals on the membrane surface, as well as silica testing. Organic testing is measured by total organic carbon (TOC) readings from foulants extracted from the surface. Organic testing is used only for total TOC count. Specific organics cannot be identified.

### 3-7.7 Flat Cell Testing

This test is performed after the destructive autopsy. Samples are cut from fouled membrane and placed in a plate and frame apparatus. These are compared against new membrane control samples. Although the comparison is not exact, it does provide helpful information on the fouled membrane.

## Table 3-6.3-1 Ion Exchange System Performance Testing

| Dissolved | Undissolved | Gaseous |
|---|---|---|
| **Cations** | | |
| Aluminum | Bacteria | Ammonia |
| Ammonium | Color | Carbon dioxide |
| Calcium | Colloidal materials | Chlorine |
| Magnesium | Condensate corrosion products | Hydrogen sulfide |
| Copper | Organic materials | Oxygen |
| Hydrogen (pH) | Suspended materials, such as turbidity, silt, and dirt | ... |
| Iron | ... | ... |
| Manganese | ... | ... |
| Potassium | ... | ... |
| Sodium | ... | ... |
| **Anions** | | |
| Alkalinity | ... | ... |
| Bicarbonate | ... | ... |
| Carbonate | ... | ... |
| Chloride | ... | ... |
| Fluoride | ... | ... |
| Hydroxide | ... | ... |
| Nitrate | ... | ... |
| Phosphate | ... | ... |
| Silica | ... | ... |
| Sulfate | ... | ... |
| Sulfide | ... | ... |
| Sulfite | ... | ... |

GENERAL NOTES:

(a) Hardness may be measured directly in terms of $CaCO_3$ or may be calculated from Ca and Mg ion concentration.

(b) Alkalinity may be measured directly in terms of $CaCO_3$ or may be calculated from bicarbonate, carbonate and hydroxide ion concentration.

## Table 3-6.4-1 Chemical Measurements During Ion Exchange System Performance Testing

| Ion Exchange System | Typical Measurements | |
|---|---|---|
| | Influent | Effluent |
| Sodium ion exchange | Total calcium and magnesium (1) | Total hardness |
| | Chlorine (1) | |
| | Turbidity (1) | |
| | Total cations (1) | |
| | Iron (1) | |
| | Manganese (1) | |
| | Oxygen (2) | |
| Sodium and hydrogen ion exchange – blended effluents | Total calcium and magnesium (1) | Total hardness |
| | Chlorine (1) | Alkalinity (1) |
| | Alkalinity (1) | pH |
| | Turbidity (1) | |
| | Total cations (1) | |
| | TMA | |
| | Iron (1) | |
| | Manganese (1) | |
| | Oxygen (2) | |
| | pH | |
| Sodium (SAC) and chloride (SBA) ion exchange | Total calcium and magnesium (1) | Total hardness |
| | Chlorine (1) | Alkalinity (1) (P & M) |
| | Alkalinity (1) | pH |
| | Turbidity (1) | Chloride |
| | Total cations and anions (1) | |
| | TMA | |
| | Iron (1) | |
| | Manganese (1) | |
| | Oxygen (2) | |
| | pH | |
| | Chloride | |
| | Nitrate | |
| | Sulfate | |
| | Color | |
| | Organic matter | |
| Hydrogen exchanger – weak or strong acid cation resins | Total cations (1) | Total hardness |
| | Chlorine (1) | Sodium |
| | Color | pH |
| | Turbidity | FMA |
| | pH | Conductivity |
| | | $CO_2$ |

## Table 3-6.4-1 Chemical Measurements During Ion Exchange System Performance Testing (Cont'd)

| Ion Exchange System | Typical Measurements | |
|---|---|---|
| | Influent | Effluent |
| Demineralization with weak base anion resin | Total anions (1) | |
| | Sodium | Sodium |
| | Alkalinity (1) | Alkalinity |
| | Color | Conductivity (1) |
| | TMA | pH (1) |
| | Organic matter | |
| | Turbidity (1) | |
| | Phosphate | |
| | pH (1) | |
| | Iron | |
| Demineralization with strong base anion resin | Total anions (1) | Total hardness |
| | Sodium (1) | Sodium (1) |
| | Alkalinity | Alkalinity |
| | Color | Conductivity (1) |
| | TMA | pH |
| | Organic matter | Silica (1) |
| | Turbidity (1) | Total anions (1) |
| | Phosphate | |
| | pH | |
| | Silica (1) (reactive and total) | |
| | Conductivity (1) | |
| | Iron | |
| Condensate polishing using sodium form strong acid cation resin | Total hardness | Total hardness |
| | Conductivity | Suspended solids (3) |
| | Iron (1) (soluble) | Iron (1) (soluble) |
| | Iron (1) [suspended (4)] | Iron (1) [suspended (4)] |
| | Oxygen (2) | Conductivity (1) |
| | Suspended solids | Copper (5) |
| | Amines | |
| | Ammonia | |
| | Total cations | |
| | Copper (5) | |
| Mixed bed – applies to demineralizer, and condensate polishing, hydrogen/hydroxide, or ammonium/hydroxide cycles | Total hardness | Ammonia |
| | Conductivity | Sodium (1) |
| | Iron (1) (soluble) | Iron (1) (soluble) |
| | Iron (1) [suspended (4)] | Iron (1) [suspended (4)] |

### Table 3-6.4-1 Chemical Measurements During Ion Exchange System Performance Testing (Cont'd)

| Ion Exchange System | Typical Measurements | |
|---|---|---|
| | Influent | Effluent |
| | Copper (6) | Copper |
| | Oxygen (2) | Chloride |
| | Suspended solids | Conductivity (1) |
| | Amines (6) | Silica (1) |
| | Ammonia (6) | pH |
| | Total cations | |
| | Chloride | |
| | Silica | |
| | Boron (6) | |
| | Turbidity | |
| | Sodium | |
| | pH | |
| | Hydrazine (6) | |
| Nuclear coolant ion exchange (applies to lithium/hydroxide, ammonium/hydroxide, potassium/hydroxide, and hydrogen/hydroxide mixed bed resins, and those in which cation resin contains one of these metals and the anion is in the borate form) | Activity level (1) | Activity level (1) |

NOTES:
(1) Major importance for the type of ion exchange system listed.
(2) Also, major importance if system operating above 100°F (35°C).
(3) Analytical procedure given in Section 4, Instruments and Methods of Measurement.
(4) For suspended iron, see Mandatory Appendix II.
(5) For systems containing copper and copper alloys.
(6) These are specific for condensate polishing applications.

## 3-8 REVERSE OSMOSIS (RO) OPERATING PERFORMANCE

The performance of a reverse osmosis (RO) or nanofiltration (NF) system is typically determined by salt rejection and permeate flow. Loss of salt rejection and loss of permeate flow are the most common problems encountered in RO and NF. Plugging of the feed channels associated with pressure drop increase is another typical problem. An immediate decline in performance indicates a defect or misoperation of the plant. Factors including the quality of the treated water/product required, e.g., the type of membrane process being used, the pretreatment being utilized, the presence of certain organic substances, oxidants and metals (such as Fe, Mn, Cr, Cl$^2$, and others), temperature and extreme hydraulic conditions must all be considered.

### 3-8.1 RO Proper Operation

Proper operation of the RO or NF system requires a number of control instruments listed below. The accuracy of all instruments is critical. They must be installed and calibrated according to manufacturer's instructions.

*(a)* pressure gages that are required to measure the pressure drop across the cartridge filter, the pressure on the pump inlet line and discharge line, the feed pressure to the membrane element(s), the pressure drop between feed and concentrate of each stage, and eventually the pressure in the permeate line. Liquid-filled gages should contain membrane-compatible fluids such as water in place of oils or other water-immiscible liquids.

*(b)* flowmeters to measure concentrate and total permeate flow rate, also permeate flow rate of each stage.

*(c)* water meters in the permeate and feed line to log the total water volume treated and produced.

### Table 3-8.2-1 K-Factor for Reverse Osmosis Calculations

| Water | $EC_{25}$ (µS/cm) | K |
|---|---|---|
| Permeate | − 1 | 0.50 |
| | 30–80 | 0.55 |
| Seawater | 4,500–6,000 | 0.70 |
| Concentrate | 6,500–8,500 | 0.75 |

### Table 3-8.2-2 SDI Indices

| SDI Test Value | Fouling Potential Estimate |
|---|---|
| SDI <1 | Several years without colloid fouling |
| SDI <3 | Several months between chemical cleanings |
| SDI 3–5 | Particulate fouling likely. Frequent cleaning |
| SDI >5 | Unacceptable for membranes. Additional pretreatment required. |

GENERAL NOTES:
(a) Varies with module design. Spiral-wound modules generally require <5 SDI whereas hollow fine-fiber modules require <3 SDI.
(b) Target SDI after filtration 3–5 or less. Surface or seawater may have SDI values of ≤200 and will require complex pretreatment.

*(d)* hour meter to log the total operating time.

*(e)* pH meter in the feed line after acidification to measure carbonate scaling potential.

*(f)* conductivity meters in the feed line, concentrate line, and permeate line to determine permeate quality and salt rejection.

*(g)* sample ports on the feed, concentrate, and permeate (total permeate and permeate of each stage) to enable evaluation of system performance. A sample port on each pressure vessel permeate outlet is recommended to facilitate troubleshooting.

### 3-8.2 Data Recording

The following data must be recorded and logged into an appropriate log sheet at least once per 8 hr:

*(a)* date, time, and hours of operation.

*(b)* pressure drop per pretreatment cartridge filter and per RO stage.

*(c)* feed, permeate, and concentrate pressure of each stage.

*(d)* permeate and concentrate flows of each stage.

*(e)* conductivity of the feed, permeate, and concentrate streams for each stage. Permeate conductivity of each pressure vessel weekly.

*(f)* TDS of feed, permeate, and concentrate streams for each stage. The TDS is calculated from the water analysis. It can also be calculated from the conductivity (at 25°C) $EC_{25}$ and an appropriate K-factor

$$\mathrm{TDS} = \mathrm{K}EC_{25}$$

The K-factor has to be determined for each specific stream. Typical K-factors are shown in Table 3-8.2-1.

*(g)* pH of the feed, permeate, and concentrate streams.

*(h)* silt density index (SDI) and turbidity of the RO feed stream. SDI is a numerical index only of the fouling potential of membranes due to the presence of suspended solids. It is not a measurement of amount and may include organic colloids, iron corrosion products, precipitated oxides, algae, and fine particulate matter. There is no direct correlation between turbidity or suspended solids measurements and SDI. It is an accepted method of predicting expected membrane fouling potential[1] (ASTM D4189-07). See Table 3-8.2-2.

*(i)* water temperature of the feed stream.

*(j)* Langelier saturation index (LSI) of the concentrate stream from the last stage (for concentrate streams <10,000 mg/L TDS).

*(k)* Stiff and Davis Stability Index (S&DSI) of the concentrate stream from the last stage (for concentrate streams >10,000 mg/L).

*(l)* calibration of all gages and meters based on manufacturer's recommendations as to method and frequency but no less frequent than once every 3 months.

*(m)* any unusual incidents; for example, upsets in SDI, pH, and pressure and shutdowns.

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[1] Lenntech.

*(n)* complete water analysis of the feed, permeate, and concentrate streams and the raw water at startup and every week thereafter. The water analysis shall include

- calcium
- magnesium
- sodium
- potassium
- strontium
- barium
- iron (total, dissolved, and ferrous)
- aluminium (total and dissolved)
- alkalinity (bicarbonate, carbonate, hydroxyl)
- sulfate
- chloride
- nitrate
- fluoride
- phosphate (total)
- silica (total and dissolved)
- total dissolved solids
- conductivity
- pH
- TOC
- silt density index (SDI). See Table 3-8.2-2.

### 3-8.3 Data Recording of Operating Characteristics of the Pretreatment Equipment

Since the RO system performance depends largely on the proper operation of the pretreatment, the operating characteristics of the pretreatment equipment should be recorded. Specific recommendations for all record keeping cannot be given, because pretreatment is site dependent. Typically, the following items must be recorded:

*(a)* total residual chlorine concentration in the RO feed (daily, unless known to be completely absent)

*(b)* discharge pressure of any well or booster pumps (twice a day)

*(c)* pressure drop of all filters (twice a day)

*(d)* consumption of acid and any other chemicals (daily, if used)

*(e)* calibration of all gages and meters based on manufacturers' recommendations as to method and frequency but no less frequent than once every 3 months.

*(f)* any unusual incidents; for example, upsets and shutdowns as they occur

## 3-9 MEMBRANE PLANT PERFORMANCE NORMALIZATION

### 3-9.1 Normalized Permeate Flow: Reverse Osmosis Normalization

The performance of an RO system is influenced by the feedwater composition, feed pressure, temperature, and recovery. For example, a feed temperature drop of 4°C will cause a permeate flow decrease of about 10%.

This, however, is a normal phenomenon. To distinguish between such normal phenomena and real performance changes, the measured permeate flow and salt passage have to be normalized. Normalization is a comparison to a given reference performance while the influences of operating parameters are taken into account. The reference performance may be the designed performance or the measured initial performance.

Normalization with reference to the initial system performance is useful to show up any performance changes between Day 1 and the actual date. This procedure is strongly recommended, because it allows an early identification of potential problems (e.g., scaling or fouling) when the normalized data are recorded daily.

Corrective measures are much more promising when taken early. Computer programs are available for normalizing operating data and graphing parameters including normalized permeate flow and salt passage as well as pressure drop. Alternatively, the measured plant performance at operating conditions can be transferred to standard (reference) conditions by the calculations in para. 3-9.2.

### 3-9.2 Normalized Permeate Flow: Flow Normalization

The performance of an RO/NF system is influenced by the feedwater composition, feed pressure, temperature and recovery. For example, a feed temperature drop of 4ºC will cause a permeate flow decrease of about 10%. This, however, is a normal phenomenon.

To distinguish between such normal phenomena and performance changes due to fouling or problems, the measured permeate flow and salt passage have to be normalized. Normalization is a comparison of the actual performance to a given reference performance while the influences of operating parameters are taken into account. The reference performance may be the designed performance or the measured initial performance.

Normalization with reference to the designed (or warranted) system performance is useful to verify that the plant gives the specified (or warranted) performance. Normalization with reference to the initial system performance is useful to show up any performance changes between Day 1 and the actual date.

Plant performance normalization is strongly recommended because it allows an early identification of potential problems (e.g., scaling or fouling) when the normalized data are recorded daily. Corrective measures are much more effective when taken early. The measured plant performance at operating conditions can be transferred to standard (reference) conditions by the calculations specified in paras. 3.9.2.1 through 3.9.2.3.

#### 3-9.2.1 Normalized Permeate Flow

$$Q_s = \frac{Pf_s - \dfrac{\Delta P_s}{2} - Pp_s - \pi fc_s}{Pf_o - \dfrac{\Delta P_o}{2} - Pp_o - \pi fc_o} \times \frac{TCF_s}{TCF_o} \times Q_o \qquad (1)$$

where

$P_f$ = feed pressure

$\dfrac{\Delta P}{2}$ = one half device pressure drop

$Pp$ = product pressure

$\pi fc_s$ = osmotic pressure of the feed-concentrated mixture

TCF = temperature correction factor

$Q$ = product flow

Subscript $s$ = standard condition

Subscript $o$ = operating condition

The temperature correction factor follows the formula:

$$\text{TCF} = \text{EXP}\,[2\,640 \times \{1/298 - 1/(273 + T)\}]$$
$$\text{for } T \geq 25^{\circ}\text{C}$$

$$\text{TCF} = \text{EXP}\,[3\,020 \times \{1/298 - 1\,(273 + T)\}]$$
$$\text{for } T \leq 25^{\circ}\text{C}$$

where

$T$ = temperature, °C

As standard conditions, take either the design values or the conditions at initial performance as given in the startup report, so that a fixed reference point is available.

*(a)* For the osmotic pressure, different formulas are available in the literature. A valid and practical short approximation is

*(1)* For $Cfc < 20\,000$ mg/L

$$\pi fc = \frac{Cfc \times (T + 320)}{491\,000}\ \text{bar}$$

*(2)* For $Cfc > 20\,000$ mg/L

$$\pi fc = \frac{0.0117 \times Cfc - 34}{14.23} \times \frac{T + 320}{345}\ \text{bar}$$

where

$Cfc$ = concentration of the feed-concentrate.

*(b)* $Cfc$ can be calculated from following approximation:

$$Cfc = Cf \times \frac{\ln \dfrac{1}{1-y}}{Y}$$

where

Y = recovery ration

$= \dfrac{\text{product flow}}{\text{feed flow}}$

$Cf$ = TDS feed mg/L

**3-9.2.2 Normalized Permeate TDS.** The normalized permeate TDS is calculated from

$$Cp_s = Cp_o \times \frac{Pf_o - \dfrac{\Delta P_o}{2} - Pp_o - \pi fc_o + \pi p_o}{Pf_s - \dfrac{\Delta P_s}{2} - Pp_s - \pi fc_s + \pi p_s} \times \frac{Cfc_s}{Cfc_o} \tag{2}$$

Terms not yet defined under para. 3-9.2.1 are

$Cp$ = product concentration as ion in mg/L

$\pi p$ = osmotic pressure of the permeate in bar

### 3-9.2.3 Example of Flow Normalization

*Values of startup (feedwater analysis in mg/L):*

Ca: 200
Mg: 61
Na: 388
$HCO_3$: 152
$SO_4$: 552
Cl: 633

Temperature: 59°F (15°C)
Pressure: 363 psi (25 bar)
Flow: 660 gpm (150m$^3$/h)
Recovery: 75%
Pressure drop: 44 psi (3 bar)
Permeate pressure: 14.5 psi (1 bar)
Permeate TDS: 83 mg/L

*Values after 3 mo: (feed water analysis in mg/L):*

Ca: 200
Mg: 80
Na: 480
$HCO_3$: 152
$SO_4$: 530
Cl: 850

Temperature: 50°F (10°C)
Pressure: 406 psi (28 bar)
Flow: 559 gpm (127 m$^3$/h)
Recovery: 72%
Pressure drop: 58 psi (4 bar)
Permeate pressure: 29 psi (2 bar)
Permeate TDS: 80 mg/L

*(a)* For the standard conditions we have

$Pf_s$ = 363 psi (25 bar)

$\dfrac{\Delta P_s}{2}$ = 22 psi (1.5 bar)

$Cf_s$ = 1 986 mg/l

$$Cfc_s = 1\,986 \times \frac{\ln \dfrac{1}{1-0.75}}{0.75} = 3\,671\ \text{mg/l}$$

$\pi fc_s$ = 36.3 psi (2.5 bar)

$$TCF_s = \text{EXP}\,[3\,020 \times \{1/298 - 1/(273 + 15)\}] = 0.70$$

*(b)* For the operating conditions we have

$$Pf_o = 406 \text{ psi (28 bar)}$$

$$\frac{\Delta P_o}{2} = 29 \text{ psi (2 bar)}$$

$$Cf_o = 2\,292 \text{ mg/l}$$

$$Cfc_o = 2\,292 \times \frac{\ln \frac{1}{1-0.72}}{0.72} = 4\,052 \text{ mg/L}$$

$$\pi fc_o = 39.4 \text{ psi (2.72 bar)}$$

$$TCF_o = \text{EXP}\,[3\,020 \times \{1/298 - 1/(273 + 10)\}] = 0.58$$

Substituting these values in eq. (1) gives

$$Q_s = \frac{25 - 1.5 - 1 - 2.5}{28 - 2 - 2 - 2.7} \times \frac{0.70}{0.58} \times 127$$
$$= 636 \text{ gpm normalized flow } (144 \text{ m}^3\text{/h})$$

Compared to the startup conditions, the plant has lost 4% capacity. This is a normal value after a period of 3 months. Cleaning is not yet necessary.

The normalized permeate TDS is derived from eq. (2)

$$Cp_s = \frac{28 - 2 - 2 - 2.72 + 0.06}{25 - 1.5 - 1 - 2.5 + 0.05} \times \frac{3\,671}{4\,052} \times 80$$
$$= 77 \text{ mg/L}$$

Compared to the initial 83 mg/L, the salt rejection has slightly improved. Such behavior is typical for the initial phase.

# Section 4
# Instruments and Methods of Measurement

## 4-1 INSTRUMENTS AND METHODS OF MEASUREMENT

This Code presents the mandatory requirements for instruments, methods and precautions of measurement that shall be employed. The Performance Test Code Supplements on Instruments and Apparatus provide general and authoritative information concerning instruments and their use, and should be consulted if sufficient information is not included in this Code.

By mutual agreement of the parties to the test, instrument devices and systems such as those using electronic devices or mass-flow techniques may be used as alternative to the mandatory Code instrument requirements, provided that the application of such devices and systems has demonstrated accuracy provided by the instrument manufacturer. Reference is made herein of the latest revision of the ASME Performance Test Code PTC 19, Supplements on Instruments and Apparatus:

– PTC 1, General Instructions
– PTC 19. 2, Pressure Measurement
– PTC 19.3, Temperature Measurement
– Supplement to PTC 19.5, Chapter 1, Part 5, Measurements of Quantity of Materials

## 4-2 MECHANICAL MEASUREMENTS

Measurements may be made to determine flow rates for all liquid streams in a system, including but not limited to regeneration of ion exchange units, or as a part of a pressure differential test. Normally, flow measuring (indicating and or transmitting) devices that may be used for these measurements are provided in the appropriate lines of a system. If weight determinations are to be made, it will also be necessary to determine relative density and temperature of the fluid being measured and the necessary compensating corrections applied to obtain accurate weights.

## 4-3 FLOWMETER ABSENSE

In the absence of a flowmeter, concentrated regenerant flow rates and total volume of concentrated regenerant used in regenerating ion exchange units may be determined using a calibrated volume tank or container of known volume. The rate of draw down, the total level change, and time increment will provide the data necessary for calculation. Alternately, volumetric flow measurement of regenerant pumping rates and duration with chemical analyses may be employed.

## 4-4 TEMPERATURE MEASUREMENT

Temperature measurements will be required to

*(a)* determine correct backwash rates for ion exchange units

*(b)* determine that resins and membranes are being operated within specified temperature limitations

*(c)* determine that regenerant temperature is within the specified range

*(d)* establish need for correction factors for pH, conductivity, relative density, or other tests that are temperature sensitive

*(e)* determine temperature correction factor for normalized flow rates

It should be noted that the viscosity of water varies considerably with temperature increasing as temperature declines, and this is a very important consideration in backwash rates for ion exchange units.

If temperature-measuring instruments are provided at suitable locations in the system, they may be used for the tests. All temperature-measuring devices may be tested and correction factors determined by methods given in PTC 19.3 (1961) if the parties to the test deem it necessary. If any additional instruments are required, it is preferred that they be installed in thermometer wells installed in the pipelines where temperature measurement is required. In systems operating at less than 200ºF, temperatures may be measured at outlet points by immersing the sensing element in a container and allowing the water to overflow from this container into another surrounding container from which it may finally flow to waste.

## 4-5 LOSS OF PRESSURE MEASUREMENT

Measurement of pressure loss in a system may be made to

*(a)* determine physical or chemical condition of ion exchange resin beds or membrane elements

*(b)* determine condition of an entire unit

*(c)* determine overall condition of an entire train of two or more ion exchange units operating in series

*(d)* determine effectiveness of backwash

*(e)* check for biological fouling of membranes in the lead array

*(f)* check for fouling or compaction of membrane elements.

Prior to testing, it will be necessary to agree on the flow rate or flow rates at which the test will be made, and to establish whether the test will be made before or after backwashing or cleaning. Suitable pressure-sensing connections are to be made available. For the preferred methods of sensing connections, refer to the latest revision of PTC 19.2.

## 4-6 PRESSURE LOSS ACROSS A SINGLE UNIT/ARRAY OR TRAIN OF MULTIPLE UNITS/ARRAYS MEASUREMENT

Pressure loss of the magnitude expected across a single unit/array or train of multiple units/arrays may be measured by a diaphragm or bellows-type differential gage or by a pair of matched, calibrated Bourdon tube-type pressure gages. If a pair of gages is used, they should be mounted at the same elevation to avoid the necessity of correction for different static head and to allow for simultaneous reading of inlet and outlet pressures. Ranges of the pressure gages should be appropriately selected. Gages should be calibrated with a dead-weight tester before the test period. Decreased temperature increases viscosity and pressure differential across resin beds, and $\Delta P$ variation is linear with flow rate. However, viscosity has little effect on $\Delta P$ in system mechanical components, while $\Delta P$ varies as the square of flow rate.

Pressure loss measurement may be made across a resin bed to indicate the rate of particulate fouling or of backwash effectiveness. Pressure loss tests should be made immediately before and after backwash using the same flow rates for each pressure loss test.

## 4-7 PRESSURE LOSS INDICATION ACROSS A RESIN BED MEASUREMENT

For pressure loss indication across a resin bed only, it is necessary to have pressure connections available in the ion exchange vessel external inlet and outlet piping between the vessel and all isolating and operating valves, flowmeters, resin traps, etc. An acceptably accurate measurement of pressure loss through the resin bed only can be made by calculating pressure drop in the small amount of piping included and allowing 1 psi (6.9 kPa) for internal collectors and distributors.

Class AA bellows, diaphragm, or Bourdon tube-type gages may be used. If these gages are to be used, they should be calibrated before the test by means of a dead-weight tester in accordance with the latest revision of PTC 19.2. The calibration chart for each shall be made a part of the test records.

## 4-8 CHEMICAL MEASUREMENTS

The necessary instruments and mandatory rules for making measurements are prescribed here in. Specific references are made to ASME Power Test Codes, to the Supplements on Instruments and Apparatus, PTC 19.11-1970, and to other publications describing detailed methods and apparatus that shall be employed in testing ion exchange equipment under the Code.

## 4-9 SAMPLING OF WATER FROM INFLUENT AND EFFLUENT OF WATER TREATMENT EQUIPMENT

Sampling of water shall be in accordance with the method outlined in ASTM D510-68, Methods of Sampling Industrial Water, and subsection 2.2 of ASME PTC 19.11-1970. These methods cover the sampling of industrial water and include

*(a)* sampling for chemical or physical tests
*(b)* sampling for biological tests
*(c)* sampling for radioactivity determination
*(d)* sampling for continual analysis

Normal variations in processes and in equipment from plant to plant preclude the possibility of specifying methods of sampling that are applicable in all cases. However, definite principles have been established that are applicable in general and in most specific cases. Where modifications or changes in these procedures may be necessary under specific circumstances, they may be made by mutual agreement of the parties concerned.

A convenient method of obtaining samples of service, backwash, and rinse waters in an ion exchange system is by installation of sample valves in pressure gage piping. A gage and isolating valve may be mounted in the top run of tee connection while a sample valve is mounted on the bottom run of tee connection. The tee side connection is piped to the sample point. Samples drawn from such points are generally acceptable for noncritical tests where constituent concentrations are higher and isokinetic sampling is unnecessary.

Results in all cases shall be reported in accordance with ASTM D596, Standard Method of Reporting Results of Analysis of Water.

Reference is made to ASME PTC 19.11-1970, Instruments and Apparatus Supplement, Water and Steam in the Power Cycle (Purity and Quality, Leak Detection and Measurement), and to other publications describing detailed methods and apparatus that shall be employed in testing under this Code.

For a more complete listing of constituents to be measured with respect to particular ion exchange systems, refer to Tables 3-6.3-1 and 3-6.4-1.

Chemical and biological tests for measuring influent and effluent constituents may be found in ASME PTC 19.11 and in American Society for Testing Materials (ASTM) Standards, Part 23, Water; Atmospheric Analysis. A list of suggested ASTM methods is detailed in Mandatory Appendix II.

## 4-10 FIELD SAMPLING OF MEDIA

Field sampling of ion exchange materials shall be done in accordance with the method outlined in ASTM D2687-95, Standard Practices for Sampling Particulate Ion Exchange Materials. The method covers sampling of ion exchange materials as received from the manufacturer in the original shipping container and prior to any resin-conditioning procedure. The sampling of ion exchange material from a fixed bed can be performed, using the general principles outlined in the above ASTM method and suitable sampling equipment agreed upon by all parties involved.

## 4-11 FIELD MEASUREMENT OF RESIN VOLUME

Refer to para. 3-6.1.4.

## 4-12 ANALYSIS OF ION EXCHANGE MATERIALS

*(a)* The analytical methods normally employed to determine physical and chemical properties of ion exchange resins are generally conducted by laboratories. They are not easily adapted or modified to field applications. The following tests will adequately determine acceptability (or rejection) of resins:

– moisture
– density
– percent whole beads
– strong acid exchange capacity
– strong base exchange capacity
– weak acid exchange capacity
– weak base exchange capacity
– particle size analysis
– foulants

The procedures for sampling and testing are described in ASTM D1782-95, ASTM D2187-94, ASTM D2687-95, and ASTM D3087-91.

*(b)* The analysis of ion exchange resins shall be done in accordance with the methods outlined in ASTM D1782-95, Standard Test Methods for Operating Performance of Particulate Cation-Exchange Materials.

*(1)* The methods cover
– pretreatment
– water retention capacity
– backwashed and settled density
– particle size distribution
– total capacity of cation exchange resins

*(2)* Depending on the type of ion exchange material and its specific application, it may be necessary to measure other chemical or physical properties, such as
– total and salt-splitting capacity of strong base anion exchange resins
– total capacity of chelating resins
– total capacity of weak and intermediate acid cation ion exchange
– total capacity of weak-base anion exchange resins
– microscopic examination
– physical attrition

In these cases, the analyses can be performed using the methods agreed upon by all parties involved.

The ion exchange resin should be analyzed prior to operation and samples taken of each batch and retained for future reference. This will provide a record of the properties of the as-received unused material and serve for comparison purposes.

*(c)* Membranes cannot be tested and examined prior to service in a manner comparable to ion exchange resins. As furnished they are guaranteed to have the stated membrane area, have an initial minimum permeate flow capacity, and have an initial minimum salt rejection.

Detailed records should be kept of date of receipt, any applicable product numbers, serial numbers, storage provisions, date placed in service and any and all operating problems along with records of influent water analysis or treatment methods.

## 4-13 SAMPLING FOR SUSPENDED SOLIDS

Crud or suspended solids is of primary interest with respect to condensate demineralizers, while ion exchange capacity is more applicable to make-up demineralizers. Although a wall (flush) sample tap can give representative samples in some situations, a multiport or single-port nozzle (as illustrated in ASTM D3370, Section 7) is recommended to obtain a truly representative sample, especially in applications where particulates in the stream are of interest. As noted in ASTM D3370, isokinetic sampling should be employed to obtain a representative sample in any two-phase system, which includes steam/water mixtures as well as water/particulates. The amount of deposition in a sample line is proportional to the length of the sample tubing. Therefore, sample lines should be maintained as short as practical to ensure that the suspended particulate is present in representative proportion in the collected sample to the main fluid.

Turbulent flow (Reynolds number $> 4,000$) should be maintained over the entire length of the sampling line including the sample cooler, if any. To maintain as high a degree of turbulence as possible at a given sample flow rate, the sample cooler, if required, should be located as near as possible to the sample line termination. Minimum flow rates required for turbulent flow can be easily calculated, and are a function of line size (ID) and sample temperature. Studies on particle transport have indicated that velocities in the sample lines near 5 ft/sec are optimal for preventing deposition on and erosion of sample tube walls. See Table 4-13-1. Within the requirements to maintain turbulent sample line flow and isokinetic sample flow rate through the sample probe, sample line size can be set by other system design conditions.

## Table 4-13-1 Purge Times Required for Representative Sampling

| Line Size, in. | Wall Thickness, in. | ID, in. | Recommended Purge Time in Seconds Per Lineal Foot at 500 mL/min | Recommended Flow Required to Achieve, 5 ft/sec (mL/min) | Recommended Flow Required to Achieve, 5 ft/sec (gal/min) |
|---|---|---|---|---|---|
| $^1/_4$ Tubing | 0.035 | 0.180 | 1.8 | 1,501 | 0.40 |
| | 0.042 | 0.166 | 1.6 | 1,277 | 0.34 |
| | 0.049 | 0.152 | 1.3 | 1,070 | 0.28 |
| | 0.058 | 0.134 | 1.0 | 832 | 0.22 |
| | 0.065 | 0.120 | 0.8 | 667 | 0.18 |
| $^3/_8$ Tubing | 0.035 | 0.305 | 5.2 | 4,310 | 1.14 |
| | 0.042 | 0.291 | 4.8 | 3,924 | 1.04 |
| | 0.049 | 0.277 | 4.3 | 3,555 | 0.94 |
| | 0.058 | 0.259 | 3.8 | 3,108 | 0.82 |
| | 0.065 | 0.254 | 3.4 | 2,989 | 0.79 |
| $^1/_2$ Tubing | 0.035 | 0.430 | 10.3 | 8,567 | 2.26 |
| | 0.042 | 0.416 | 9.7 | 8,018 | 2.12 |
| | 0.049 | 0.402 | 9.0 | 7,488 | 1.98 |
| | 0.058 | 0.384 | 8.3 | 6,832 | 1.81 |
| | 0.065 | 0.370 | 7.7 | 6,343 | 1.68 |
| | 0.072 | 0.356 | 7.1 | 5,872 | 1.55 |
| | 0.083 | 0.334 | 6.3 | 5,169 | 1.37 |
| $^1/_2$ Pipe | Schedule 40 | 0.622 | 21.6 | 17,926 | 4.74 |
| $^3/_4$ Pipe | Schedule 40 | 0.824 | 37.8 | 31,459 | 8.31 |
| 1 Pipe | Schedule 40 | 1.049 | 61.3 | 50,985 | 13.47 |

All sample lines should be flushed thoroughly prior to sampling. Sample lines should be maintained with continuous flow through the system, if possible. If continuous flow is not practical, the lines should be flushed for a minimum of three sample line volumes prior to collection of the sample. The valves should be then returned to a "set-point" position with respect to sample flow requirements. All sample valves should be of the type designed with a minimum of internal pressure drop or flow disturbance. The same parameters concerning sampling for suspended solids should be used at the influent and effluent of the ion exchange equipment or system.

*(a)* A 0.45 µm filter pad comparison chart is a rapid means for estimation of total iron present in a particulate form (crud) or mixture. The color produced depends not only on the concentration, but also on the particle size distribution, morphology, and composition of the suspended solids. Reference is made here to Mandatory Appendix II of this Test Code, which details methods of preparing filter pad comparison charts.

*(b)* The capacity of ion exchange resin for the removal of crud will depend on the concentration of the influent suspended material. In general, a greater capacity is obtained at higher influent concentrations (0.5 ppm to 2.0 ppm) than at lower influent concentrations (<0.1 ppm). The capacity will depend on the composition of the crud. In general, a higher capacity is obtained for magnetite than hydrated iron oxides. Other parameters affecting this are the number of regenerations, regeneration level, backwash flow rate, and the operational flow rate. In general, higher flow rates (>25 gpm/ft$^2$) produce higher capacities (or removal efficiency) for crud than low flow rates (<25 gpm/ft$^2$).

## 4-14 ION EXCHANGE OPERATING CAPACITY

Ion exchange operating capacity is that portion of the total ion exchange capacity of a resin that can be utilized in a practical ion exchange operation. There are a large number of service and regeneration factors that influence the capacity that can be achieved. This "capacity" is commonly expressed in kilograins of calcium carbonate ($CaCO_3$) per cubic foot (kgr/ft$^3$) or equivalents per liter (eq/L) of resin. The equipment manufacturer normally calculates the resin working capacity and resin volume required to meet the plant requirements based on a given water analysis.

Operating capacity decline can be the result of chemical and/or mechanical deterioration. Factors including the quality of the treated water/product required, e.g., the type of ion exchange process being used, the frequency of regeneration, the adequacy of the of removal of resin fines and accumulated solids, the hydraulic load on the resin, the resin type, the presence of certain organic substances, oxidants and metals (such as $Cl_2$, Fe, Mn, Cr, and others), temperature, and extreme hydraulic conditions must all be considered.

*(a)* The influent and effluent ammonia concentration must be determined for a unit operating in the ammonia cycle and using ammonia treatment or operating in an ammonia environment or plant.

*(b)* Dissolved solids determination by electrical conductivity alone cannot be used in all cases to determine operating ion exchange capacity. In some cases, the dissolved solids must be calculated from the complete water analysis. For example, weakly ionized dissolved solids such as silica and boric acid contribute little to electrical conductivity. The amount present, as found by separate determination, should be added to the dissolved solids calculated from conductivity to obtain the total dissolved solids.

*(c)* Faulty pretreatment can lead to increased influent suspended solids, which cause higher pressure drop and potential blockage. This initiates flow channeling, thereby reducing the operating capacity. Inadequate pretreatment can also lead to slippage of oxidizing agents in the feedwater, such as free $Cl_2$ or polyelectrolytes, thereby damaging the resin and impairing plant performance. Performing periodic analyses of the incoming water according to the appropriate ASTM methods is recommended. The frequency will depend on the stability of the water source and can only be determined by experience.

*(d)* Feed temperature can also have a profound effect on the operating capacity of weak acid cation, weak base, and Type II strong base anion resins. High temperatures can degrade the resin, especially acrylic and Type II anions. Temperatures should be closely monitored at minimum, in the influent to the ion exchange system.

*(e)* A change in the ionic composition of the water can also affect operating capacity, even if the total ionic content remains unchanged. For example, an increase in sodium to total cations ratio may lead to sodium leakage from the cation if the acid regeneration level is not adjusted to compensate. Similarly, an increase in free mineral acidity (FMA) will shorten the throughput of an anion resin after a decarbonator. As a result, specific ion electrodes or other analytical tools should be utilized in the determination of certain ions that require measurement, and system adjustments should be made as appropriate.

*(f)* Pumps and measuring equipment should be checked and, if faulty, either restored to good working order or replaced. In the case of poor water quality, valves should be checked to ensure there is no leakage of regenerant into the service water, or raw water into the treated water. Flow distribution problems and/or inefficient regeneration are caused by improper, blocked, or broken internal distribution systems.

*(g)* Electrical conductivity and/or specific ion measurements alone may be impractical for determining changes in the ion exchange resin characteristics, such as degradation or fouling that can cause a significant decline in operating capacity. Whenever resin fouling or bead breakage is suspected, a representative resin sample, withdrawn according to recommended procedures, should be tested per the appropriate ASTM procedures. If necessary, the ion exchange resins should either be cleaned according to recommended procedures or replaced.

*(h)* In other cases, measurement of electrical conductivity may also be impractical for determining operating ion exchange capacity or exhaustion. These are instances where the electrical conductivity does not change significantly, regardless of the concentration of dissolved solids. An example is the determination of the lithium or ammonium capacity of a reactor coolant purification ion exchanger operating in the hydrogen-borate form with a high concentration of boric acid present in the coolant. Another example is the determination of the operating capacity of a reactor demineralizer for "trace" concentrations of radioactive ions. In such cases, the determination of capacity should be made using the appropriate analytical procedure.

*(i)* The testing of ion exchange resin for removal of trace [ppb (μg/L), ppt (pg/L)] quantities of soluble radio nuclides should reflect the fact that concentration is the primary factor to be considered. For example, concentrated test solutions will indicate higher capacities than will the operational case. The calculation necessary to arrive at the value of the regeneration efficiency should be done in accordance with subsection 2-1.

*(j)* Determination of anion effluent silica alone cannot be used in all cases to determine the end of anion operating exchange capacity. In some cases, the sodium levels from the cation unit must be determined by the use of a sodium analyzer or other analytical technique on the effluent of the cation exchanger (i.e., the influent of the anion exchanger). Increased influent sodium to the anion exchanger will result in increased silica leakage.

A properly performed evaluation of the operating ion exchange unit and the resins contained within will provide key information associated with loss of resin exchange capacity, loss of operating exchange capacity, probable factors responsible for lost capacity, and other irreversible damage as well as types and concentrations of foulants. Such a review will allow the investigator to determine the most acceptable pathway required to restore effluent quality performance, for example

*(1)* modify the regeneration procedures, conditions or concentrations

*(2)* adjust the run length service throughput

*(3)* replace the resin

*(4)* implement additional pretreatment of the influent water

*(5)* clean the resin using an approved chemical procedure

# Section 5
# Interpretation of Results

## 5-1 INTRODUCTION

Performance of various types of high-purity water treatment systems is interpreted as described in the following parts. Since it is unlikely that all operating conditions can be maintained exactly at the specified values, it will be necessary to correct test performance for the effect of such deviations.

### 5-1.1 Mechanical Measurements

Flow rates, differential pressure, and temperature measurements shall be interpreted from the readings in accordance with the directions given in the necessary sections and parts of this Code, which refer therein to the appropriate ASME Codes that have been selected for particular measurements.

A variety of mechanical measurements are utilized to monitor the performance and operation of high-purity water treatment systems. These include flow measurements, pressure measurements, temperature measurements, power readings, volume measurements, and monitoring of age and physical characteristic parameters.

**5-1.1.1 Flow.** Flow measuring devices including a wide variety of engineered and standard devices are some of the most critical devices for ensuring proper operation and troubleshooting of high-purity water treatment systems. Typical flow measurement devices utilized in these applications include flow orifices, flow nozzles, and turbine meters, etc. Ultrasonic flowmeters can be temporarily installed and are useful when in-line devices fail or are suspect. Whether these devices are permanently installed or temporarily mounted, most have specified installation requirements to ensure proper operation of the device and to deliver accurate data. Some flow measuring devices are required to have minimum straight lengths of pipe upstream and downstream of the device and are also required to be operated within a certain calibration range in order to ensure accuracy.

In ion exchange systems, calculations of flow per unit cross-sectional area (gpm/ft$^2$:m$^3$/m$^2$), and flow per unit volume of resin (gpm/ft$^3$:m$^3$/h/m$^2$) are utilized to evaluate operation and monitor the regeneration process.

NOTE: Both of the foregoing metric equivalents are more properly expressed as meters per hour (m/h). See Table 5-1.1.1-1 for typical operating flow specifications for ion exchange systems.

In membrane processes, routine monitoring of permeate, reject, and influent flow rates will provide the system operator with data related to system operation, membrane degradation, and potential fouling or scaling of the membranes. All flow data collected for membrane systems must be normalized or adjusted to account for variations in water temperature, pressure, and concentration. For example, the volume of permeate increases as the water temperature increases due to change in water viscosity. An increase in influent water pressure will also result in an increase in permeate flow rate, while variations in the concentration of contaminants in the influent water will change the efficiency of the membranes. Water flux rate (the flow rate of water through the membrane per unit area of membrane surface) and salt flux rate (the flow rate of dissolved contaminants through the membrane per unit area of membrane surface) are two important flow-related parameters for membrane systems. Flux rates for membrane processes vary depending on the influent water quality. See Table 5-1.1.1-2 for typical system flux rates of various membrane applications.

**5-1.1.2 Pressure.** Like flow measuring devices, pressure indicating equipment provide critical information for ensuring proper operation of or in troubleshooting problems in a high-purity water treatment system. Pressure and flow rate are directly related in ion exchange systems and membrane processes.

In resin-based ion exchange systems, the pressure drop or differential pressure across each resin bed should be continuously monitored to track system performance, provide indication of system problems, and predict the need for backwashing of the resin bed. The rate of particulate fouling of ion exchange resin beds as well as degradation, such as oxidation of resins, can be indicated by the use of these differential pressure measurements. Mechanical restrictions of flow would also be a source of an increased pressure differential. The effectiveness of resin bed backwash operations can also be determined by measuring pressure drop across the resin bed both before and after the backwash cycle provided sufficient time is allowed for bed compaction to occur between readings.

Pressure measurements are an integral part of successful membrane process system operations. Pressure gages or transmitters, on membrane systems, are required to measure the pressure drop across the cartridge filter, the pressure on the pump inlet line and discharge line, the

### Table 5-1.1.1-1 Typical Operating Flow Rates Specifications

| Parameter | Typical Specification Limit |
|---|---|
| Service | 1 gpm/ft$^3$ to 5 gpm/ft$^3$ (7.48 m/h to 37.4 m/h) [Note (1)] |
| Backwash | 2 gpm/ft$^2$ to 8 gpm/ft$^2$ (0.8 m/h to 3.3 m/h) [Note (2)] |
| Regenerant | 0.25 gpm/ft$^3$ to 2.0 gpm/ft$^3$ (31 L/h/m$^2$ to 249 L/h/m$^2$) [Note (3)] |
| Slow rinse (displacement) | 0.25 gpm/ft$^3$ to 2.0 gpm/ft$^3$ (31 L/h/m$^2$ to 249 L/h/m$^2$) [Note (3)] |
| Fast rinse | 1 gpm/ft$^3$ to 5 gpm/ft$^3$ (7.48 m/h to 37.4 m/h) [Note (1)] |

NOTES:

(1) This varies with the influent ionic concentration, the type of ion exchange resin, the regenerated operating form and influent temperature.

(2) See Nonmandatory Appendix B for calculation of water temperature correction factors.

(3) This is dependent on resin type, ionic concentration and selection of regenerant chemicals.

### Table 5-1.1.1-2 Typical System Flux Rates

| Application | Typical Specification Limit [Note (1)] |
|---|---|
| MF/UF influent flow rate | 36 gfd to 110 gfd (35 m/d to 108 m/d) |
| MF/UF backflush flow rate | Up to 185 gfd (181 m/d) |
| RO with MF/UF pretreatment | 12 gfd to 20 gfd (12 m/d to 20 m/d) |
| RO with conventional pretreatment | 8 gfd to 12 gfd (8 m/d to 12 m/d) |
| Second pass RO | 21 gfd to 25 gfd (21 m/d to 25 m/d) |

GENERAL NOTE: The abbreviation "gfd" denotes gallons per square foot of membrane surface per day.

NOTE:

(1) Depends on dissolved and/or suspended solids loading.

feed pressure to the membrane element(s), the pressure drop between feed and concentrate of each stage, and the pressure in the permeate line. Like flow rate, pressure measurements are parameters that must be normalized in membrane systems to account for variations in water temperature, pressure, and concentration and to accurately determine changes. Differential pressure in a membrane treatment system refers to the difference between the influent water and the reject water stream. Both should be routinely monitored and this parameter tracked to trend system performance and to predict the need for membrane cleanings. Reverse osmosis systems work by utilizing pressure to overcome the natural osmotic pressure of the system. In UF and MF membrane systems, however, transmembrane pressure (TMP) is the primary driving force for transport of water through the membrane. The transmembrane pressure is calculated as

Transmembrane pressure = (Influent water pressure + reject water pressure)/2 − effluent water pressure

Increasing transmembrane pressure can indicate deterioration or fouling of the UF or MF membranes.

**5-1.1.3 Temperature.** Temperature can be a crucial parameter for the success of not only the operation of high-purity water treatment systems but also the cleaning and regeneration of these systems. Both resin beads and membranes have maximum operating temperature limits that should not be exceeded during normal operation.

For ion exchange systems, temperature is particularly crucial for backwash operations, regeneration, and cleaning operations. If backwash temperature varies more than 10°F, the backwash flow rate should be adjusted to compensate for changes in water density. If the backwash water temperature increases and flow is not adjusted, the flow rate will be too low, and solids and broken beads will not be effectively removed. If the backwash water temperature decreases and flow is not adjusted, the backwash flow rate will be excessive and resin will be lost. Temperature is also an important factor in the removal of silica and oils or other organic foulants from resin beads. During regeneration of anion resin beads, the caustic solution must be warmed to approximately 95°F (35°C) or 120°F (49°C) for effective silica removal. To remove oil or other organic foulants from the cation resin beads, the cleaning solution in generally warmed to 160°F to 180°F (71°C to 82°C) to facilitate removal.

Changes in influent water temperature can impact the perceived pressure drop and flow rate values for an operating membrane treatment system. All flow and pressure data collected for membrane systems must be normalized or adjusted to account for variations in water temperature, pressure, and concentration. A standard approximation for the effect of water temperature changes on membrane processes is a decrease of 1.8% of the flow rate per degree Fahrenheit decrease.

**5-1.1.4 Power.** Electrical power related measurements such as resistance, amperage, and voltage are most commonly monitored in relation to EDI and EDR systems. Since EDI or EDR cells can be related analogously to resistors in series, continuous monitoring of these parameters provided indication of the performance of the unit. Fouling or scaling in the cells is normally indicated by an increase in resistance across the units. On constant current systems, fouling or scaling will also be observed by a corresponding increase in voltage as the system compensates to maintain the same current. The current efficiency, a ratio of the influent flow rate to the current, is also important in EDI and EDR units because it relates to the ability of the unit to remove weakly charged ions such as silica and boron. A current efficiency of 20% or less is common for these systems with a current efficiency of 10% or less optimal for targeted silica or boron removal.

**5-1.1.5 Volume (Media, Regenerant, Waste and Treated Product).** Volume is a method of mechanical measurement frequently encountered in conjunction with resin based ion exchange systems. The bed volume in an ion exchanger is the volume of active resin beads in the vessel that is set during the design of the system based on the influent water quality and the desired run length of the equipment. It is usually measured in cubic feet ($ft^3$) or cubic meters ($m^3$) of resin. The throughput volume relates to the design run length of the equipment; it is the volume of water that can be processed by an ion exchange unit before it reaches exhaustion. This parameter is typically measured in gallons or cubic meters ($m^3$), and any changes in the throughput volume can indicate a change in influent water quality, a reduction in regeneration efficiency, or deterioration or fouling of the resin.

Regenerant chemical quantities and regeneration wastewater quantities are other volume parameters that are typically tracked in ion exchange systems. Different resin types have different quantities of chemical required to regenerate a specific volume of that resin. For instance, 5 lb (2.3 kg) is a typical volume of 98% concentration sulfuric acid needed to regenerate 1 $ft^3$ (28.32 L) of strong acid cation resin. These quantities should be tracked to verify regeneration efficiency of the resin. Changes in required regenerant chemical volume can cause a reduction in regeneration efficiency or deterioration or fouling of the resin. Regeneration wastewater volume is tracked primarily due to the expenses associated with handling, treating, and disposing of chemical containing wastewaters. Often these volumes are segregated into high total dissolved solids (chemical wastewaters) and low total dissolved solids wastewaters (rinse and backwash waters) to minimize the volume requiring treatment or to allow reclamation.

**5-1.1.6 Age and Physical Characteristics.** For resin based ion exchange systems, routine representative resin samples should be collected approximately every 2 yr for laboratory analysis of the physical integrity of the resin beads. With proper care, cation resins should last 8 yr to 10 yr while anion resins typically have a useful life of 2 yr to 4 yr. Stressors such as fouling, high pressures and temperatures, freezing conditions, and high contaminant levels such as organics, iron, and chlorine can reduce the resin's useful life span. A high percentage of broken beads indicates that the resin may have been exposed to excessively high water differential pressures or freezing conditions. A high moisture content indicates degradation of the resin bead capacity. Fouling or coating of the bead surfaces with contaminants such as organics, metals, silt, or precipitants indicate the need for a resin cleaning or replacement (in severe contamination cases).

A visual inspection of membrane elements provides the operator information about potential fouling or scaling problems. With proper care, a membrane life span of 3 yr to 4 yr is typical. Visual observation of discoloration, foreign matter, or deposits indicates that membrane cleaning is required. Decrease in permeate flow rate, performance decline, increased pressure drop between the influent and reject sides, and higher salt passage are all indicators of potentially fouled membranes. Telescoping and fiberglass damage are indicators of excessive hydraulic loading or improper loading of the membrane modules in the pressure vessel. High salt passage is typically a result of mechanical damage to the permeate tube or damaged brine seals. Membrane mechanical integrity issues such as punctures, scratches, or delamination of the membrane surface also leads to high salt passage problems.

### 5-1.2 Chemical Measurements

Analytical measurements shall be performed on necessary water samples for recommended constituents for the particular ion exchange equipment or system under test, see para. 3-6.4.

The Instruments and Methods of Measurement Section of this Code Test refers to the appropriate ASTM test procedure applicable for selected constituents.

## 5-2 PERFORMANCE BENCHMARK

It is desirable that a performance benchmark be established immediately after systems are first placed in service. Should the Code Test be delayed, there should be reasonable assurance that the systems have not been damaged or become fouled with various materials during the intervening period of operation.

## 5-3 CALCULATIONS AND ANALYTICAL PROCEDURES

It is recommended that calculations and analytical procedures to obtain preliminary test results be carried out simultaneously with the conducting of the tests. Such procedures provide an opportunity for early examination of the test data and the detection and correction of any observer errors, instrument errors or failures, and other reasons to repeat tests. Shortly after the conclusion of this initial test series, it should be reasonably clear whether the test results are consistent and acceptable, or whether further testing is required.

## 5-4 EXPRESSION OF SPECIFIED PERFORMANCE AND RESULTS OF TESTS

The specified performance and the results of tests covering a series of runs and measurements may be expressed as a single value with respect to the removal of individual constituents within the water being treated. Single values for ion exchange capacity can also be calculated with respect to individual constituents. For calculation of ion exchange capacity, refer to subsection 2-1.

# Section 6
# Report of Tests

## 6-1 REPORT OF TESTS

The following outline is a guide for reporting the results of tests. Depending on the type and size of ion exchange of membrane system or equipment to be tested, certain readings may be omitted and others added. Since standardization of tests results is of great convenience to those who use this Code, it is recommended that the outlined format be used with as few modifications as possible. The test report should only include that data that is of significance in appraising the performance of the equipment or system. Any other data used to support calculations, results, etc., should be included in other sections of the report, if desired.

(a) *General Information*

(1) date of test

(2) name of Company, location of plant, and plant unit identification

(3) operating environment: housed or unhoused, ambient conditions, and any special operating conditions

(4) influent water: analysis, temperature of water used as basis of all calculations (well, river, process, source, quality and temperature of regeneration waters, etc.)

(5) method(s) of pretreatment, type, dosage, injection point of any chemicals added upstream of system being tested

(6) test conducted by [individual(s), firm, address, contact numbers]

(7) general objective of test

(b) *Description of Equipment*

(1) original equipment manufacturers' reference numbers and date of installation

(2) minimum, average and maximum effluent operating flow and throughput capacity and flow rate variability

(3) plant operating cycle: hr/day; days/week; weeks/yr. In the event of cyclic operating periods, state "return to service" practices basic type and configuration of system:

(a) ion exchange systems: types of units in each system and unit placement in sequence (i.e., weak acid cation, strong acid cation, decarbonator, strong base anion, mixed bed), sequential or compressed regeneration mode, single unit or thoroughfare regeneration, train or common header interconnection

(b) membrane systems: description of pretreatment, submicron filters, pass, and stage configuration, location(s) of feed pumps, details of recycle if included, type of chemical feed, injection points and control method

(4) number, size, and arrangement of each vessel/system:

(a) ion exchange systems; configuration – cocurrent/countercurrent, packed bed, layered bed, mixed bed, single or multiple chambers

(b) membrane systems; number of tubular modules per stage, number of elements per tubular module, number of stacks per modules, common or individual rectifiers

(5) volume of media

(a) ion exchange systems; specific type, resin manufacturer, resin product designation, operating ionic form, volume and bed depth of resin in each vessel, regenerant chemical, and dosage

(b) specific membrane model numbers, serial numbers, and manufacturer/supplier

(6) description and location of flow measuring devices

(7) type, description, and location of pressure measuring devices

(8) type, description, and arrangement of inline monitors, including location of sampling points

(9) capacity and guarantees

(a) flow rate, gpm, (minimum, maximum, average, peak)

(b) exchange capacity, kilograins (ion exchange only)

(c) throughput per cycle, gallons (ion exchange only)

(d) pressure drop, psi (bed, interstage, system)

(e) influent design analysis

(f) effluent water quality guarantee

(g) end points (measured in terms of conductivity or other applicable criteria)

(h) normalized permeate flow

(i) percent recovery per pass and system

(j) percent reject

(10) Equipment manufacturers' operating procedures and regeneration schedules, including flow rates, duration, amount and type of regenerants diluted chemical concentrations, and operating pressure.

(c) *Description of Test Result Documentation*

(1) operating history prior to test

(2) description of procedures used in preconditioning equipment specifically for the test

(3) general description of test procedures

*(4)* corrections for deviations from specified test conditions

*(5)* description of test methods used other than those originally specified

*(d) Observations (Mean Observations Derived From Operators' Log)*

*(1)* influent water

*(a)* source

*(b)* pressure

*(c)* temperature

*(d)* quality – individual constituents to confirm analytical data where site tests are performed or calibrated online monitors are installed

*(2)* effluent water

*(a)* pressure

*(b)* quality

*(c)* purity

*(3)* flow rates [(influent and effluent/permeate (constant/variable), regeneration wastes/concentrate, recycle)]

*(4)* volume of resin in each vessel

*(5)* type and dosage of supplementary influent chemical treatments

*(6)* amount/weight of regenerant used per regeneration cycle and raw and applied concentrations

*(7)* rinse volumes

*(8)* power consumption

*(e) Results*

*(1)* throughput per cycle for each vessel tested

*(2)* pressure drop at design flow rate across each vessel tested

*(3)* corrections to specified test conditions

*(4)* reliability of service and regeneration water supply and waste transport system

*(5)* calculated normalized permeate flow

*(6)* normalized permeate TDS

*(7)* calculated regeneration efficiency, refer to Section 2

*(f) Conclusions*

*(1)* points of compliance

*(2)* points of noncompliance

*(3)* remarks

# MANDATORY APPENDIX I
# REGENERANT PURITY REQUIREMENTS FOR ION EXCHANGE MATERIALS[1]

## I-1 SODIUM CHLORIDE (NaCl), COMMON SALT

This is generally supplied in granular or pelletized form. Rock salt, and some solar salt grades, even premium quality, containing excessive amounts of insoluble and soluble impurities can necessitate frequent cleaning of dissolving tanks or cause resin fouling. Such products are not recommended for use in ion exchange systems. Vacuum evaporated grades are recommended.

| Constituent | Minimum Specification |
|---|---|
| Purity | 98% |
| Water content | 1% |
| Insoluble matter | $< 1\,000$ mg/L |
| Sulfate ($SO_4^{--}$) | 10 000 mg/L |
| Calcium ($Ca^{++}$) and magnesium ($Mg^{++}$) | $< 500$ mg/L |
| Soluble iron ($Fe_2^{++}$) | Undetectable |
| Sand or clay | Nil |

This salt quality is suitable for use in cocurrently and countercurrently regenerated units for softening water. If the raw water contains $>$1 000 mg/L TDS, even lower levels of calcium and magnesium are recommended. Where hardness leakage from a countercurrently regenerated unit is critical (i.e. <1 mg/L $CaCO_3$), it is necessary to specify 99% salt, high-purity vacuum quality which contains <5 mg/L to 10 mg/L calcium or magnesium as $CaCO_3$.

Salt used for human consumption or for animal feed purposes generally contains iodine. This causes severe resin fouling and such grades should not be used in ion exchange systems.

When salt is used for regenerating chloride anion dealkalizers, or for counter currently regenerated softeners, the salt dissolving and dilution water must be softened and contain <1.0 mg/L calcium plus magnesium.

## I-2 SULFURIC ACID ($H_2SO_4$)

Generally supplied as a colorless to yellow-brown liquid containing 92% to 99% $H_2SO_4$ weight for weight (w/w).

| Constituent | Minimum Specification |
|---|---|
| Purity | 93% (66° Baume) |
| Iron (Fe) | 50 mg/L |
| Arsenic (As) | 1 mg/L |
| Lead (Pb) | 1 mg/L |
| Insoluble matter | Nil |

Other metals and impurities often found in commercial sulfuric acid are generally present in trace quantities only and unlikely to cause problems. This quality of acid is suitable for both weakly and strongly acidic resins for cocurrently and countercurrently regenerated units.

## I-3 HYDROCHLORIC ACID (HCl)

Generally supplied as a colorless to yellow-green liquid containing 30% to 37% HCl weight for weight (w/w).

| Constituent | Minimum Specification |
|---|---|
| Iron (Fe) | 50 mg/L |
| Sulfate ($SO_4$) | 4 000 mg/L |
| Oxidants ($Cl_2$) | 1 mg/L |
| Arsenic (As) | 1 mg/L |
| Lead (Pb) | 1 mg/L |
| Insoluble matter | Nil |
| Color | White to light yellow |

If the resin is used for treating water for potable use, or for food processing, the acid used to regenerate the resin must not contain any phenolic material. Phenolics can impart taste/smell to the treated water.

Other metals and impurities often found in commercial hydrochloric acid are generally present in trace quantities only and unlikely to cause problems. Acid that contains large amounts (100 mg/L) of iron can cause problems in the service cycle. Metals often act as catalysts in promoting undesirable side reactions. In worst cases (i.e., 5 mg/L to 10 mg/L copper), the metals can catalyze oxidation of the resin, causing the resin to swell and reduction of its volume capacity. This quality of acid is suitable for both weakly and strongly acidic

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[1] All impurity levels shown are based upon 100% w/w concentrations of regenerants and exclude any contaminant concentrations in dissolving or dilution water.

resins for cocurrently and countercurrently regenerated units.

## I-4 NITRIC ACID ($HNO_3$)

Generally supplied as a colorless to pale yellow liquid containing 52% to 96% $HNO_3$ weight for weight (w/w).

| Constituent | Minimum Specification |
|---|---|
| Nitrous oxides | 10 mg/L |
| Iron (Fe) | 5 mg/L |
| Chloride (Cl) | 5 mg/L |

**CAUTION: Nitric acid is an extremely strong oxidizing agent and can react violently with organic materials. For this reason, concentrated nitric acid must not be allowed to contact ion exchange resins.**

**Metallic impurities such as iron and nickel and free chlorine must be avoided because they speed up hazardous oxidation processes.**

**Contact between anion exchange resins and nitric acid at any concentration above 1% w/w is to be avoided at all times. Nitrate form resin is a potential explosion hazard — likely to explode at elevated temperatures, especially if in contact with nitric acid at any concentration. For use with cation exchange resins, a maximum concentration of 6% $HNO_3$ w/w at a maximum temperature of 20°C is advised.**

This quality of acid is suitable for both cocurrently and countercurrently regenerated units.

## I-5 SODIUM HYDROXIDE (NaOH) — CAUSTIC SODA

Generally supplied as liquid containing 32% to 50% NaOH weight for weight (w/w) but may also be furnished in solid forms as flakes, pellets, or blocks at 100% NaOH w/w.

The product formerly described as "rayon grade," containing low amounts of impurities and produced by the mercury cell process, is no longer available because of environmental restrictions. Caustic soda produced by other processes and containing high concentrations of chlorates and competing anions such as sodium chloride and sodium carbonate is not recommended for regeneration of strong base anion exchange resins. Caustic soda manufactured by this process may contain 3% to 18% w/w NaCl, which affects the regeneration of strong base anion resins. For example, if the caustic soda contains 2.5% NaCl, the loss of operating resin capacity to be expected for a Type I strong base anion resin is about 2% and for a Type II strong base anion exchange resin is about 5%.

The loss due to sulfate contamination is even greater. Caustic soda supplied as a solid generally contains more impurities than liquid caustic. When regenerating polishing mixed bed demineralizers and high-purity water treatment units, the use of purified grades of caustic soda may be required.

| Constituent | Strong Base Anion Minimum Specifications, mg/L | Weak Base Anion Minimum Specifications, mg/L |
|---|---|---|
| Chloride (Cl) | 3 500 | 7 000 |
| Carbonate ($CO_3$) | 5 000 | 6 000 |
| Chlorate | 1 | 1 |
| Sulfate ($SO_4$) | 1 500 | 2 500 |
| Silica ($SiO_3$) | 50 | 300 |
| Iron (Fe) | 10 | 10 |
| Mercury (Hg) | 0.1 | ... |
| Arsenic (As) | 0.1 | 0.1 |
| Nickel (Ni) | 1 | 2 |
| Lead (Pb) | 1 | 2 |
| Copper (Cu) | 0.5 | 0.5 |
| Magnesium (Mg) | 50 | 50 |
| Calcium (Ca) | 25 | 40 |
| Aluminum (Al) | 5 | 10 |

This quality of caustic stated for strong base anion resins is suitable for both cocurrently and countercurrently regenerated units.

## I-6 SODIUM CARBONATE ($Na_2CO_3$) — SODA ASH

Generally supplied as a white, anhydrous powder containing 98% to 100% $Na_2CO_3$ weight for weight (w/w).

The water soluble impurities that normally are found are generally of no importance. Sodium carbonate solution can be used for the regeneration of weak base anion resins.

| Constituent | Minimum Specification, mg/L |
|---|---|
| Chloride (Cl) | 1 500 |
| Sulfate ($SO_4$) | 150 |
| Iron (Fe) | 10 |

## I-7 AMMONIA ($NH_3$) OR AMMONIUM HYDROXIDE ($NH_4OH$)

This is generally supplied as a solution of $NH_4OH$ containing 20% w/w $NH_3$ or liquid ammonia gas in high pressure cylinders. Impurity levels are generally very low and of no consequence in ion exchange applications. Ammonia of this quality is suitable for regeneration of weak base anion resins or for converting strong base anion exchange resins to the ammoniated form.

## I-8 RECOVERED REGENERATION CHEMICALS

Because of the interest and need to reduce or eliminate plant waste discharges, it is sometimes desired to reclaim, recycle or reuse regenerant solutions or to utilize waste chemicals from a plant process stream as ion exchange resin regenerant solutions. Before making such a decision, it is necessary to fully analyze the waste chemical to determine the nature and concentration of any impurities that might affect the condition and/or performance of the resin. Some impurities might compete for the ion exchange sites and reduce capacity, while others may attack the resin itself.

# MANDATORY APPENDIX II SUSPENDED IRON OXIDE SOLIDS: MEMBRANE COMPARISON CHARTS

At this time, standard comparison charts for black iron oxide, hydrated iron oxide, and mixtures thereof, are commercially available from the Babcock & Wilcox Company, Barberton, OH.

## II-1 MAGNETITE STANDARD CHART

This procedure describes the preparation of black iron oxide ($Fe_3O_4$) and deposition of the iron oxide on a 0.45 μm membrane filter.

*(a)* Add 0.1382 g of Fisher (I 119) black iron oxide powder ($Fe_3O_4$) to clean a 2 L beaker. Add 1 L of reagent grade water. This solution contains 100 ppm iron as Fe[1].

*(b)* Stir vigorously (30 min) with a glass stirrer and stirring motor suspended above the beaker.

*(c)* Withdraw a 100 mL representative aliquot and dilute to a liter with reagent grade water (10 ppm Fe).

*(d)* Add this solution to a clean 2 L beaker and stir vigorously as above for 30 min.

*(e)* Withdraw a 100 mL representative aliquot and dilute to a liter with reagent grade water (1.0 ppm Fe).

*(f)* Add this solution to a clean 2 L beaker and stir vigorously as above for 30 min.

### II-1-1 Procedure for Membrane Stain Preparation

Take an aliquot, as desired, while the solution is being stirred and filter through a 0.45 μm, Type HA, 47 mm diameter filter paper. Use the Hydrosol Stainless Filter Assembly and Millipore (or equivalent) pump for filtering (assembly, pump, and filters are available through Millipore Corp.) The size of aliquots taken will depend on the desired amount of iron deposition equivalent to that accumulated from a 1L throughput of sample water to the filter[2].

If an iron analysis of the stain on the filter paper is desired, the paper should be treated as follows:

*(a)* Place paper disc in a 150-mL beaker and add 10 mL of reagent grade water, 2 mL perchloric acid, 2 mL sulfuric acid, and 2 mL nitric acid.

*(b)* Take the solution almost to dryness (less than 0.5 mL on a hot plate in a hood capable of handling perchloric acid fumes). In taking the solution to dryness it should be colorless. It is sometimes necessary to add more nitric acid dropwise until the solution loses its black color due to the presence of organic material.

*(c)* When the solution has been concentrated to dryness, cool, and add for convenience of handling 30 mL of reagent water and 5 mL hydrochloric acid.

*(d)* Bring the sample to a boil and digest for 5 min.

*(e)* Allow the sample to cool and dilute to a suitable volume in a volumetric flask. Determine the iron content colorimetrically by bathophenanthroline (ASTM D1068-10).

## II-2 HYDRATED IRON OXIDE CHART

This procedure describes the preparation of hydrated iron oxide ($Fe_2O_3 \times XH_2O$) and deposition of the iron oxide on a 0.45 μm membrane filter [Notes (1) and (2) of para. II-1-1 for the magnetite chart apply].

*(a)* Hydrated iron oxide ($Fe_2O_3 \times XH_2O$). Dissolve 0.7022 g of ferrous ammonium sulfate $Fe(NH_4)_2(SO_4)_2 \times 6H_2O]$ in approximately 500 mL of reagent grade water.

*(b)* Add 20 mL of sulfuric acid ($H_2SO_4$) specific gravity 1.84 and 10 mL of 30% hydrogen peroxide ($H_2O_2$). Heat to boiling. Maintain slow boiling for 30 min to ensure complete oxidation.

*(c)* Cool the solution to ambient temperature and dilute to a liter with reagent grade water (100 ppm Fe).

*(d)* Dilute 100 mL of the above 100 ppm Fe solution to a liter with reagent grade water (10 ppm Fe).

*(e)* Transfer 100 mL of the 10 ppm Fe solution to a liter beaker and adjust the pH to 10.5–11.0 using 1.0 N sodium hydroxide (NaOH). Dilute to a liter with reagent grade water (1.0 ppm Fe).

*(f)* Transfer this 1.0 ppm Fe solution to a 2 L beaker and stir vigorously with a glass stirrer and stirring motor suspended above the beaker. Cover the beaker with plastic wrap to prevent contamination.

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[1] Reagent grade chemicals and reagent grade water (ASTM D1193-06) are used for this method. Use care to avoid iron contamination from glassware and apparatus used in this method.

[2] This procedure is intended for membrane stains up to 100 ppb iron (Fe). To obtain higher concentration, the iron concentration must be increased. Check the concentration to determine that it is precisely 1.0 ppm by analysis using the bathophenanthroline method for iron (ASTM D1068-10).

### II-2-1 Procedure for Membrane Stain Preparation

Take an aliquot, as desired, while the solution is being stirred and filter through a 0.45 µm, Type HA, 47 mm diameter filter paper. Use the stainless steel filter assembly and pump for filtering. The size of aliquots taken will depend on the desired amount of iron deposition equivalent to that accumulated from a 1 L throughput of sample water to the filter.

## II-3 MIXED HYDRATED OXIDE: MAGNETITE 1:1 STANDARD CHART

This procedure describes the preparation of a mixture of iron oxides ($Fe_2O_3 \times XH_2O$ and $Fe_3O_4$) and deposition of the oxides on a 0.45 µm membrane filter[1,2] of para. II-1-1 for the magnetite chart apply].

*(a)* Hydrated iron oxide ($Fe_2O_3 \times XH_2O$). Black iron oxide ($Fe_3O_4$) mixture 1:1. Dissolve 0.7022 g of ferrous ammonium sulfate [$Fe(NH_4)_2 (SO_4)_2 \times 6H_2O$] in 500 mL of reagent grade water.

*(b)* Add 20 mL of sulfuric acid ($H_2SO_4$) sp. gr. 1.84, and 20 mL of 30% hydrogen peroxide ($H_2O_2$), and heat to boiling. Maintain boiling (slowly) for 30 min to ensure complete oxidation.

*(c)* Cool the solution to ambient temperature and adjust the pH to 10.5–11.0 using 1.0 N sodium hydroxide (NaOH). Dilute to a 1 L solution using reagent grade water (100 ppm Fe). This solution is to be combined later with the black iron oxide ($Fe_3O_4$) solution.

*(d)* Add 0.1382 g of Fisher (I 119) black iron oxide powder ($Fe_3O_4$) to a clean 2 L beaker. Add 1 L of reagent grade water. This solution contains 100 ppm iron as Fe.

*(e)* Combine the solutions in a clean 3 L beaker and stir vigorously for 30 min with a glass stirrer and stirring motor suspended above the beaker. Cover the beaker with plastic wrap to prevent contamination.

*(f)* While vigorously stirring, withdraw a 100 mL representative aliquot and dilute to 2 L with reagent grade water (5 ppm Fe solution).

*(g)* Transfer this solution to a clean 3 L beaker and stir vigorously for 30 min.

### II-3-1 Procedure for Membrane Stain Preparation

Take an aliquot, as desired, while the solution is being stirred and filter through a 0.45 µm, Type HA, 47 mm diameter filter paper. Use the stainless steel filter assembly and pump for filtering. The size of aliquots taken will depend on the desired amount of iron deposition equivalent to that accumulated from a 1 L throughput of sample water to the filter.