ION EXCHANGE RESINS

by

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Presented at

Alberta Government Public Works, Supply and Services
Water Treatment Coordinator's Meeting
Calgary, Alberta

20 October 1983

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1.0 INTRODUCTION

All too often, full utilization of an ion exchange system is not realized because of the lack of a proper understanding of the properties of the ion exchange material and the experience necessary to handle certain unforseen events.

Therefore, such topics as types of ion exchange resins, manufacture of ion exchange resins, preservation of ion exchange resins, conditioning of ion exchange resins, fouling of ion exchange resins, regeneration of ion exchange resins, and sampling of ion exchange resins are covered herein.

2.0 TYPES OF ION EXCHANGE RESIN

The two main types of ion exchange resin, namely cation and anion resin, are futher subdivided into their sub-groups of strong and weak acid cation resin and strong and weak base anion resin.

The strong acid cation resin, a typical example of which is Rohm & Haas IR-120, may be regenerated with brine or with a strong acid such as sulfuric or hydrochloric acid. If regenerated with brine, strong acid cation resin will replace the calcium and magnesium ions in the raw water with sodium ions, a process which does not reduce the dissolved solids in the raw water, but does soften the raw water. If regenerated with acid, strong acid cation resin will replace all of the cations in the raw water with hydrogen ions, thus reducing the dissolved solids in the raw water.

Weak acid cation resin, a typical example of which is Rohm & Haas IRC-84, is regenerated with a weak acid solution. Thus, in a multi-stage demineralization plant, the "spent" regenerant from the strong acid cation unit can be used to regenerate the weak acid cation unit. The higher regeneration efficiency and exchange capacity of the weak acid cation resin over the strong acid cation resin makes the former resin more economical for use in waters having high percentages of hardness and alkalinity. Thus, when used together, the weak acid cation resin is used to remove the hardness associated with alkalinity and the strong acid cation resin is used to remove the balance of the hardness as well as the rest of the cations - in both cases the cations in the raw water are replaced with hydrogen ions.

Strong base anion resins are sub-divided into Type I and Type II, typical examples of which are Rohm & Haas IRA-402 and IRA-410 respectively.

When both types of resins are regenerated with caustic, the sulfates, chlorides, alkalinity, carbon dioxide, nitrates, silica, weak organic acids and all other anions in the influent water are exchanged for hydroxyl ions. However, type I strong base anion resin is a more strongly basic resin than is type II, but it has less exchange capacity for strong acids than type II. Therefore, type I strong base anion resin provides for better silica and carbon dioxide removal but is less efficient chemically.

When types I and II strong base anion resins are regenerated with brine, the anions of strong and weak acids in the influent water are exchanged for chloride ions. However, like the caustic regenerated resins, the chloride regenerated type I has a higher exchange capacity for weak acids (ie., as in chloride form dealkalizers), but the chloride regenerated type II resin is more efficient chemically.

Weak base anion resins, a typical example of which is Rohm & Haas IRA-45, may also be regenerated with caustic.

When regenerated with caustic, weak base anion resins, referred to as "working organic traps", have a high capacity to remove chlorides, sulfates and organics.

When regenerated with brine, strong base anion resins, referred to as "non-working organic traps", have a higher capacity to remove organics than do the weak base resins.

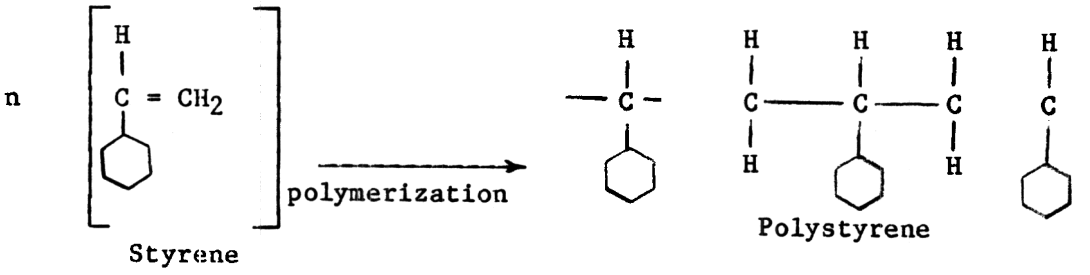
In addition to these various sub-groups of anion and cation exchange resin, there are two master groupings, namely gel resins and macroporous resins.

The advantages of the macroporous resins over the gel resins of higher porosity (ie., 30% for macroporous vs. less than 1% for gel) and higher surface area (ie., 100 m^2/g for macroporous vs. less than 1% for gel) make macroporous resins more suitable for applications involving treatment of highly organic bearing waters, waters containing oxidizing agents, and where mechanical attrition (ie., as in external regeneration) is a problem. However, these advantages are somewhat off-set by the 20% lower exchange capacity of the macroporous resins.

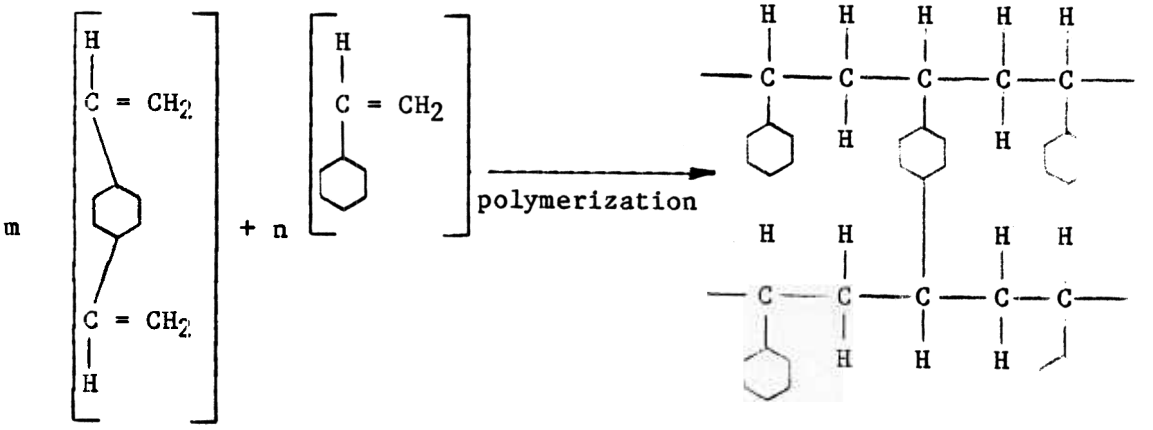
3.0 MANUFACTURE OF STRONG ACID CATION AND STRONG BASE ANION EXCHANGE RESINS

The first step in the synthesis of modern-day strong acid cation and strong base anion resins, a polymerization reaction, is common to both types of resin.

The most common base material is styrene, the polymerization reaction of which is shown overleaf.



However, in order to increase the stability of the resin, divinylbenzene is added to the polymerization reaction as shown below.



By varying the amount of divinylbenzene, typically in the range of 4-20%, the degree of cross-linking can be controlled. This affects the pore size and stability of the resins (ie., the more highly cross-linked resins have firstly, smaller pore size, thus resulting in higher pressure drop, reduced capacity, and lower resistance to organic fouling and secondly, increased resistance to high temperatures, oxidants, and mechanical shear).

In any case, the polymerization reaction is a batch process. Styrene and divinylbenzene, both in liquid form, are added to a reactor where they are agitated with water and dispersed into small globules. Benzoyl peroxide is added to initiate polymerization at the monomer's double bonds. In order to control particle size and prevent globules from agglomerating into a big unmanageable mass, small amounts of suspension stabilizers are added; gelatin, starch, bentonite, or talc are commonly used to form a protective layer on the surface of the globules so as to prevent their agglomeration upon collision. Other variables such as speed of mixing, temperature, pH, and catalyst concentration are also used to control the diameter of each bead, typically in the range of 0.30-1 mm.

Polymerization is carried out at a temperature of about 175°F for approximately two hours, after which time the resulting beads are filtered, washed, and dried.

At this point in the process, the copolymer is used to produce either strong acid cation resin or strong base anion resin.

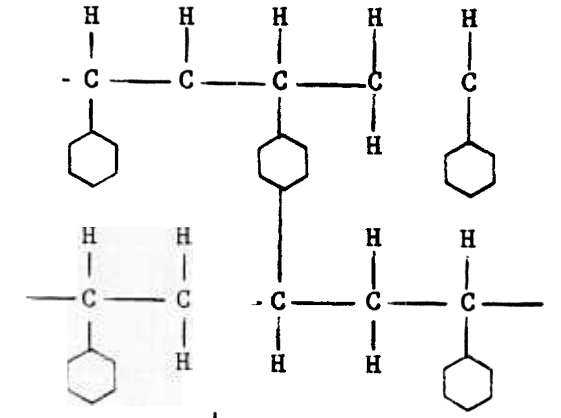
In the synthesis of strong acid cation resin, the cured beads enter an agitated kettle where they are mixed with concentrated sulfuric acid. Sulfonation takes place in the styrene component after several hours at a temperature of about 200°F. Excess acid is removed by filtration and washing. In order to stabilize the cation resin against degradation and pickup of impurities during packaging and shipment, the sulfonated styrene-divinlybenzene beads are changed to the sodium form by neutralization with caustic soda.

The reactions for the production of strong acid cation resin are shown overleaf.

Chloromethylation and amination are the two steps necessary to introduce anionic exchange groups into the cross-linked copolymer.

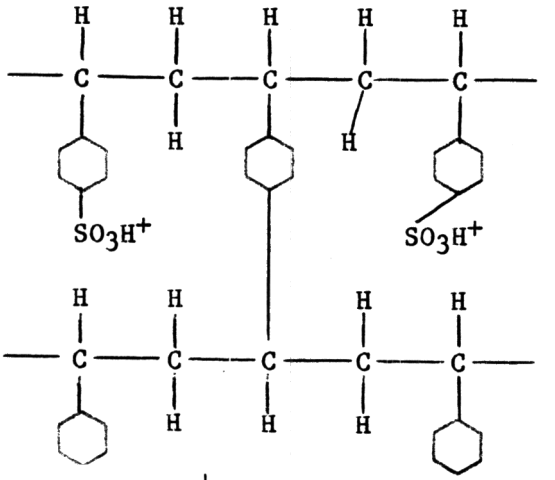
Dry copolymer beads are sent to a reactor and chloromethylether is added. Alkylation takes place at the styrene rings in the presence of diethyl ether, aluminum trichloride, and freezing temperature. After being filtered, washed and dried, the beads next go to the amination reactor where they swell in benzene solvent and are cooled to room temperature. A steady stream of trimethylamine gas flows into this mixture while the temperature is increased. After several hours at 68°F, the reaction is completed, and the quarternary-ammonium resin (strongly basic anion exchange resin) is filtered and washed.

The reactions for the production of strong base anion resin are shown overleaf.



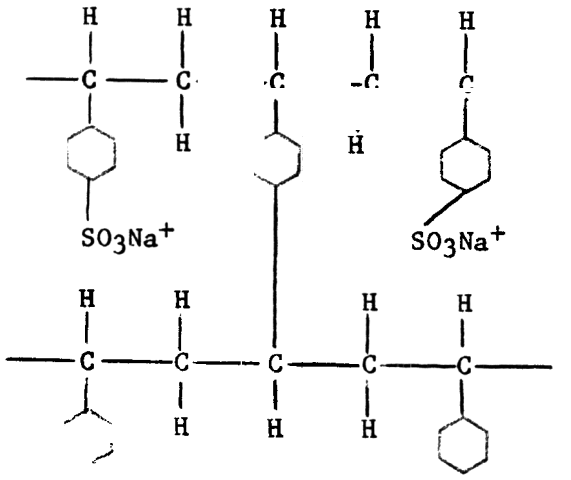
Divinylbenzene/Polystyrene Copolymer

SULFONATION

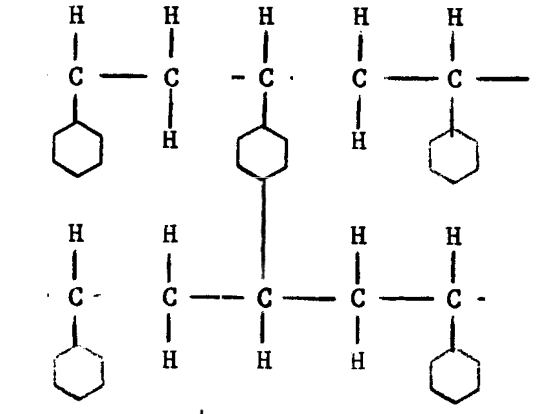


Strong Acid Cation Exchange Resin (H form)

NaOH

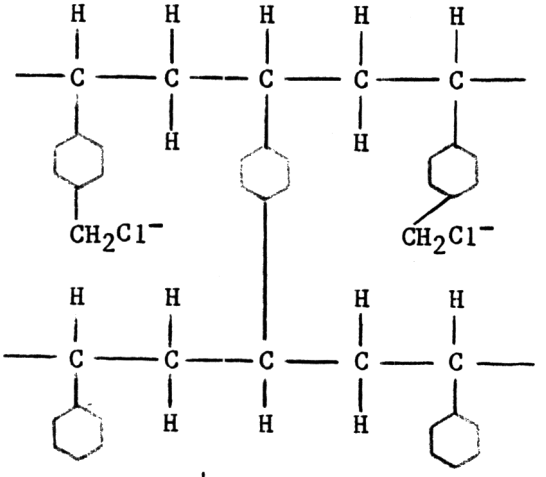


Strong Acid Cation Exchange Resin (Na Form)



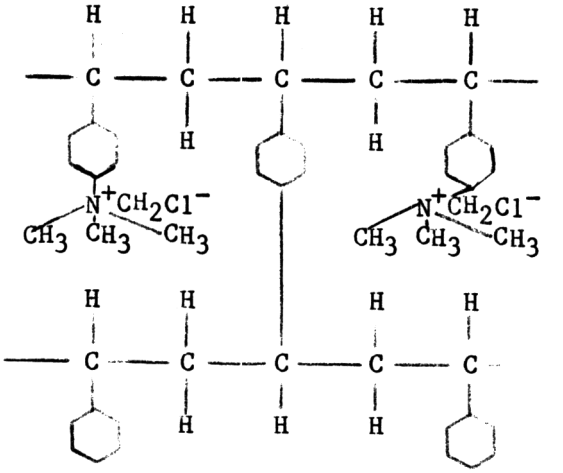
Divinylbenzene/Polystyrene Copolymer

Chloromethylation



Chloromethylated Divinylbenzene/ Polystyrene Copolymer

trimethylamine gas



Strong Base Anion Exchange Resin (Cl form)

4.0 PRESERVATION OF ION EXCHANGE RESINS

In order to ensure that ion exchange resins retain their physical and chemical properties intact, certain essential rules must be observed in their handling.

These rules relate to prevention of dehydration, protection from high temperatures or frost, and protection against the growth of biological organisms.

4.1 PROTECTION AGAINST DEHYDRATION

Ion exchange resins are always supplied with a certain moisture content, specific for each resin but generally within the range of 40-60%, in order to maintain the relative elasticity of each bead since dry beads are liable to burst when they come into contact with water.

For protection of resin in the original packages, frequently check the moisture content of resins in packages which have been opened and do not hesitate to renew damaged bags, remembering to seal the new packs carefully; if necessary, spray the resins with water before sealing the bags. Also, avoid storage in locations exposed to the sun and temperatures greater than 40°C (104°F).

Units which are out of service for extended periods should be kept filled with water (ie., filtered water for cation resins; softened or decationised water for anion resins). If the unit has to be drained it is important that it is kept perfectly tight afterwards in order to prevent evaporation of the internal moisture.

4.2 PROTECTION AGAINST FROST

Tests have shown that resins subjected to temperatures as low as -40°C (-40°F) are not adversely affected. However, there is an element of risk if resins are stored in locations subject to substantial and frequent fluctuations in temperatures (ie., alternate freezing and thawing).

Therefore, it is advisable to store resin in locations protected from low temperatures, or alternately to soak the resin in a saturated solution of sodium chloride.

4.3 PROTECTION AGAINST BIOLOGICAL GROWTHS

Micro-organisms (ie., algae, bacteria, etc.) tend to proliferate in units shut down for prolonged periods if the conditions

are favourable (ie., presence of organic matter, mineral salts, pH, temperature, etc.). A complex system arises which may choke the unit or form a solid mass inside it.

Biological growths may be prevented by storing cation resin in a 5% solution of formaldehyde, 10 grams of pure formaldehyde being used for each litre of resin, and storing anion resin in a 1% solution of a quarternary ammonium solution, 2 grams being used for each litre of resin.

5.0 CONDITIONING OF ION EXCHANGE RESINS BEFORE USE

Standard resins may contain minute quantities of soluble impurities which should be removed prior to use.

The degree of conditioning is determined by the end use of the ion exchange resin.

For general purpose use, a simple rinse with several bed volumes of water is adequate. In the more critical applications, cyclic washing with dilute acid and alkali removes all but trace residuals of soluble components.

Irrespective of the method of cleansing selected, new resins must initially be regenerated with twice the normal amount of regenerant prior to service.

6.0 FOULING OF ION EXCHANGE RESINS

Although ion exchange resins are considered to be a capital investment, they suffer both physical and chemical degradation and their useful life is usually less than the life of the equipment.

Anion resins are normally the least stable of all ion exchange resins and their useful life varies from 500,000 to 2,000,000 gallons of treated water per cubic foot of resin. By contrast, the useful life of cation exchange resins varies from 2,000,000 to 10,000,000 gallons of treated water per cubic foot of resin.

In discussing the problem of the deterioration of ion exchange resin, one must consider the fact that the deterioration is manifested by many criteria, each of which must be considered separately.

6.1 DECROSSLINKAGE

As indicated in Section 3.0, divinyl benzene (DVB) crosslinks in the polymer structure of ion exchange resins impart to the beads their shape and physical strength. Destruction of these crosslinks causes the beads to deform, thus causing the resin bed to compact and impair the flow distribution. To relieve the resultant pressure drop increase, channeling occurs.

Severe cases of decrosslinkage are indicated by the "mushy" feel of the resin. However, increases in moisture content of the resin are also an indication of decrosslinkage.

In addition to high temperatures, the limits of which are indicated in Section 7.2, the most frequently encountered DVB decrosslinker is free chlorine. In order to avoid oxidation, the free chlorine concentration in the influent of a hydrogen form cation exchanger should be less than 0.3 ppm. However, the presence of trace quantities of catalysts such as iron and copper reduces this limit to 0.1 ppm free chlorine. For sodium form cation exchangers, the maximum influent free chlorine concentration is 0.5 ppm.

Free chlorine may be eliminated by feeding a reducing agent such as catalyzed hydrazine or sodium sulfite, or alternately the use of an activated carbon filter.

6.2 IRON FOULING

Iron is the most frequently encountered foulant of cation resins. Fouling of anion resins by iron may also occur when it is complexed with certain organic species, when it is in excessive amounts in the caustic regenerant, or when the anion resin is regenerated with brine in an unlined carbon steel vessel.

Fouling of cation resin by iron occurs by the following mechanisms:

a) suspended, insoluble particles such as corrosion products, are removed by the filtering action of the resin bed;

b) soluble ferrous iron is oxidized to the insoluble ferric form before entering the ion exchanger and removed by filtration;

c) soluble ferrous iron is oxidized to the insoluble ferric form during the normal ion exchange process and precipitated within the resin bed.

Iron introduced by the first mechanism may be removed by thorough backwashing. However, total removal is not assured. The resultant build-up of suspended solids can cause an increased pressure drop across the bed and eventually lead to channeling and subsequent leakage of unwanted ions.

The second mechanism introduces insoluble ferric hydroxide into the unit and coats individual beads. Consequently, the exchange capacity of the unit is reduced, thus resulting in short service runs. Backwashing may be of limited value in removing this adherent foulant.

Fouling by the third mechanism poses the most serious problems. Ferrous ions in the influent will exchange onto the cation resin in the same manner as hardness ions, except that they tend to be more tightly held by the resin, thus making their removal during regeneration more difficult. Exposure to dissolved oxygen in the backwash water causes them to oxidize and precipitate within the structure of the resin beads, thus reducing both exchange capacity and regeneration efficiency, the net effect of which is shortened service runs. This type of fouling can only be removed by chemically cleaning the resin. Visual inspection of the resin can reveal iron fouling as it will exhibit an increasingly dark brown colour until it appears almost black.

When iron fouling of cation resin is considered to be due to the first and second mechanisms, the resin may be cleaned either with hydrochloric acid or with sodium hydrosulfite per Appendix A; however, iron fouling due to the third mechanism may only be removed with the hydrochloric acid procedure.

Iron fouling of anion resins may only be removed with the hydrochloric acid procedure (Note: caustic regenerated strong base anion resins must first be exhausted with brine since the acid-base reaction would produce heat that could be destructive to the resin and protective vessel lining) or alternately with the brine squeeze procedure per Appendix A if the iron is complexed with organic matter. Under no circumstances should anion resin be cleaned with sodium hydrosulfite.

6.3 MAGNESIUM HYDROXIDE FOULING

Magnesium hydroxide fouling of cation resin can be due to floc carryover from a lime softening process or cross regeneration of deionizers (ie., erroneous introduction of caustic to the cation resin and acid to the anion resin).

In anion units, magnesium hydroxide fouling is often caused by failure to use softened water for preparation of the dilute regenerant or when the capacity of the upstream cation unit is exceeded.

Decreased capacity, increased pressure drop, and magnesium hydroxide leakage throughout the service run are all symptoms of magnesium hydroxide fouling.

Because magnesium hydroxide tends to accumulate as a "plug" near the bottom of the bed, agitation by air lancing during acid cleaning is of great value.

6.4 CALCIUM SULFATE FOULING

Calcium sulfate fouling is frequently encountered in cation units where sulfuric acid is used as the regenerant. As the hydrogen ions replace calcium from the exhausted bed, a supersaturated solution of calcium sulfate is produced which under the proper conditions will precipitate.

By maintaining the regenerant temperature below 85°F, the regenerant flow rate at 1 USGPM/ft^3 or greater, and the regenerant concentration at less than 5% H_2SO_4, this problem can usually be avoided.

When calcium sulfate precipitation occurs, a white, powdery solid may be observed in the regenerant effluent and calcium leakage throughout the service cycle will be measurable.

Failing its removal with backwashing and soft water washing, calcium sulfate fouling may be removed with hydrochloric acid.

6.5 ORGANIC FOULING

High molecular weight organics such as humic, fulvic, and tannic acids, which form from decomposing vegetable matter, are the major organic foulants that tend to affix themselves to exchange sites on and within anion resins. Lack of porosity of gel resins prevents effective elution of the organic molecules, thus impairing the normal ion exchange process. Without their removal, these weak organic acids decrease the pH and increase the conductivity of the deionized water, thus reducing demineralizer capacity and quality, and causing long rinse times for anion regeneration.

Removal of organic fouling can only be realized by performing the brine squeeze procedure indicated in Appendix A. This procedure utilizes the fact that anion resins in the hydroxide form are usually swollen to a volume about 20% greater than the resin is in the chloride form. Thus, by alternately expanding and contracting the resin bed with caustic and brine, respectively, the organic foulants are squeezed out of the resin beads.

7.0 REGENERATION OF ION EXCHANGE RESINS

Proper performance of the ion exchange regeneration procedure is very important if optimum operation of the ion exchange system is to be obtained.

Periodic reviews of regeneration introduction times, flow rates, dosages, etc. must be performed. The following guidelines will assist in maintaining optimum performance and trouble free operation.

7.1 BACKWASH

Backwash water flow rate, temperature, and duration are very important, as is the type of resin being backwashed.

If the backwash rate is too low, the suspended material that has accumulated on the resin bed (ie. crud and resin fines) will not be removed no matter how long the backwash step is continued. If the backwash rate is too high, either the resin will be washed out of the vessel, or the resin bed will be forced up against the backwash collector, thus causing the bed to act as a filter and preventing the suspended material from escaping.

If the backwash water temperature is too low, the water is denser and more viscous, thus having a greater lifting effect. Therefore, a lower backwash rate is used at lower backwash water temperatures and a higher backwash rate is used at higher temperatures.

Normally, ion exchange resins are backwashed for 10 minutes. However, the proper backwash time is that which is required to remove most of the suspended material, so that the wash water appears practically clear.

Anion resin is typically 15% lighter than cation resin. Therefore, backwash water flow rates are higher for cation resin than for anion resin.

7.2 REGENERANT INTRODUCTION

The regenerant used must be readily soluble, relatively inexpensive, and not cause precipitates to form when reacting with the ions removed from the exchanger. Specifications of typical regenerants are indicated in Tables 1, 2, and 3 of Appendix A.

Parameters to be considered when reviewing regenerant setpoints include regenerant concentration, level, flow rate (or alternately, contact time), and temperature.

The regenerant level, usually expressed as pounds of regenerant per cubic foot of resin, is dependent on other variables such as influent water quality, acceptable leakage, and regenerant quality and concentration.

The regenerant concentration affects the exchange capacity and integrity of ion exchange resin. If the regenerant concentration is too low, incomplete regeneration and reduced capacity will result. If the regenerant concentration is too high, the resin beads will be damaged when the regenerant is acid or caustic, calcium sulfate may be precipitated within the resin bed when the regenerant is sulfuric acid, or the exchange capacity may be reduced when the regenerant is brine. Typically, the regenerant concentrations are 10% NaCl for softener regenerations, 5% NaCl for regeneration of chloride form dealkalizers, 4% NaOH for regeneration of strong base anion resin, 1-5% H_2SO_4, depending on the influent calcium concentration, for regeneration of strong acid cation resin, and less than 1% H_2SO_4 for regeneration of weak acid cation resin.

If the regenerant contact time is too short (ie., high regenerant flow rate), there is not enough time available for the ion exchange reactions to take place (ie., removing the unwanted ions and replacing them with the regenerant ions), thus resulting in leakage. If the regenerant contact time is too long, the only potential difficulty could be with sulfuric acid regeneration, where calcium sulfate precipitation might occur.

Regenerant temperature, if too high (ie., greater than 250°F for most cation resins; greater than 100-170°F, depending on the resin and its form, for anion resins), can destroy the bead structure of the resin. At lower temperatures, but still above ambient, calcium sulfate may be precipitated in cation resin regenerated with sulfuric acid.

7.3 RINSING

The purpose of the rinse step is to remove excess regenerants and the unwanted ions taken up by the ion exchanger during the service run.

The rinse rate should not be so high as to reduce the regenerant contact time. Low rinse rates may lead to precipitation of calcium sulfate with cation resin beds which have been regenerated with sulfuric acid.

Typically, an initial rinse at 0.5-1 USGPM/ft^3 is followed by a fast rinse at 1.5 USGPM/ft^3. The total quantity of rinse water is 25-75 USG/ft^3 for cation resins and 40-90 USG/ft^3 for anion resins.

7.4 SERVICE

Depending on the end use of the treated water, rinsing of the resin bed may be required each time the ion exchanger is started up.

Once in service, anions and cations are exchanged in accordance with the ion selectivities indicated in Table 7-1 overleaf.

For example, a cation exchanger regenerated with brine (ie., sodium chloride) will exchange all cations above Na+ indicated in Table 7-1. At the end of a softening run, magnesium ions will leak out of the bed before calcium ions do.

8.0 SAMPLING OF ION EXCHANGE RESINS

The recommended procedure for the sampling of ion exchange resin for analysis consists of core sampling in accordance with ASTM D 2687-77, a simplified version of which is contained in Appendix A.

TABLE 7-1

General Order of Ion Selectivity

Cations	Anions
Fe^{+3}	CrO_4^{-2}
Al^{+3}	SO_4^{-2}
Pb^{+2}	SO_3^{-2}
Ba^{+2}	HPO_4^{-2}
Sr^{+2}	CNS^-
Cd^{+2}	CNO^-
Zn^{+2}	NO_3^-
Cu^{+2}	NO_2^-
Fe^{+2}	Br^-
Mn^{+2}	Cl^-
Ca^{+2}	CN^-
Mg^{+2}	HCO_3^-
K^+	$HSiO_3^-$
NH_4^+	OH^-
Na^+	F^-
H^+	
Li^+	

APPENDIX A

TABLE 1

Purity of Salt for Regeneration of Dealkalizers, Softeners and Sodium Form Condensate Polishers

Parameter	Purity of Salt for Regeneration of Dealkalizers	Purity of Salt for Regeneration of Softeners and Sodium Form Polishers
grade	white/evaporated	white/any source
sodium chloride, NaCl	99.9% minimum	97.0% minimum
sodium sulfate, Na_2SO_4	0.10% maximum	2.0% maximum
calcium sulfate, $CaSO_4$	0.05% maximum	0.5% maximum
magnesium chloride, $MgCl_2$	0.01% maximum	0.5% maximum
insolubles	5 ppm maximum	0.25% maximum
size	granulated to $\frac{1}{4}$"	granulated to $\frac{1}{2}$"
additives (i.e. iodides, silicates, etc.)	none	none

TABLE 2

Purity of Sulfuric Acid for
Regeneration of Cation Resin

Parameter	Purity of Sulfuric Acid
grade	technical
colour	water white to light brown, no sediment
usual shipping concentration	66 Bé (93% H_2SO_4)
iron, Fe	50 ppm maximum
chlorides, Cl	5 ppm maximum
arsenic, As	0.2 ppm maximum
copper, Cu	1 ppm maximum
manganese, Mn	0.5 ppm maximum
lead, Pb	5 ppm maximum
freezing point	-24°F maximum
organic contaminants	100 ppm O_2 maximum
inhibitors and oxidants	nil

TABLE 3

Purity of Sodium Hydroxide for
Regeneration of Strong Base Anion Resin

Parameter	Purity of Caustic
grade	rayon, flake
sodium hydroxide, NaOH	98%
sodium oxide, Na_2O	76%
sodium carbonate, Na_2CO_3	8,000 ppm
sodium chloride, NaCl	10,000 ppm
sodium sulfate, Na_2SO_4	2,500 ppm
sodium chlorate, $NaClO_3$	5 ppm maximum
iron, Fe	10 ppm
silica, SiO_2	50 ppm
calcium & magnesium, $CaCO_3$	25 ppm
copper, Cu	0.2 ppm
manganese, Mn	0.5 ppm
lead, Pb	0.5 ppm
nickel, Ni	1.0 ppm

IN-SITU CLEANING OF IRON FOULED RESIN WITH HYDROCHLORIC ACID

A. Chemicals and Materials

For acid cleaning of 100 ft^3 of resin, 375 USG of 10% inhibited hydrochloric acid (Note: non-inhibited hydrochloric acid should be used for cleaning anion resin).

For neutralization of acid waste, 127 lbs. of soda ash or 89 lbs. of hydrated lime or 96 lbs. of caustic, all per 100 USG of 10% HCl.

Fresh water supply nearby for rinsing down acid spills.

Protective clothing (i.e. oil skins and face shield) while handling acid.

B. Procedure

Thoroughly backwash the resin by passing raw water upward through the unit at a flow rate sufficient to expand the bed by at least 50% of its normal service height - the cleaner the resin is, the more efficient the acid cleaning will be.

Regenerate the resin in the usual manner (Note: anion resin must be rinsed with brine before acid is introduced).

Isolate the unit from the process by closing the isolating valves and breaking the main outlet line - this prevents acid entry to the process.

Remove the upper man-way cover.

(5) Lower the water level in the unit to approximately 3 inches above the resin bed.

(6) Check, for the presence of inhibitor in the acid by adding some of the acid to a beaker containing a small loose roll of steel wool, previously cleaned of oil - if the steel wool rises to the top of the acid solution (due to the evolution of hydrogen gas), the acid does not contain an inhibitor and should be returned.

(7) Add the 10% inhibited hydrochloric acid, the volume of which should be equal to the void volume of resin, slowly through the man-way and onto the top of the resin bed.

(8) Lower the acid level in the unit to a point approximately 3 inches above the top of the resin bed.

(9) If oil-free air is available, air lance the acid soaked resin bed for approximately 30 minutes using a rigid pipe, long enough to reach the bottom of the bed and connected to a flexible air hose, being careful not to disturb the gravel support bed - if oil-free air is not available, allow the resin to soak in the acid for 1 - 2 hours.

(10) Drain the acid from the unit, allowing the spent acid (amber in colour) to pass through the neutralizing material before entering the drains systems.

(11) Replace the man-way cover

(12) Rinse the unit with a downward flow of water until samples collected at the outlet of the unit are water white in colour - for cation resin, the pH of the effluent will be low due to the conversion of the exchange sites to the hydrogen form as a result of the acid.

(13) Repeat step (2) of the procedure.

(14) Check the pH of the rinse effluent - if necessary, repeat the regeneration until the effluent pH is within normal limits.

(15) Reconnect the outlet line and return the unit to service.

IN-SITU CLEANING OF IRON FOULED CATION RESIN WITH SODIUM HYDROSULFITE

A. Chemicals and Materials

(1) Sodium hydrosulfite, 1.25 lbs per cubic foot of resin.

(2) Fresh water supply nearby for rinsing down spills.

(3) Protective clothing (i.e. oil skins and face shield) while handling sodium hydrosulfite.

(4) Ventilation of area around exchanger.

(5) Sodium chloride, coarse crushed rock salt grade, 5 lbs. per cubic foot.

B. Procedure

(1) Thoroughly backwash the resin by passing water upward through the unit at a flow rate sufficient to expand the bed by at least 50% of its normal service height - the cleaner the resin is, the more efficient the cleaning will be.

(2) Regenerate the resin with brine

(3) Slow rinse the resin to a slightly salty taste in samples collected at the effluent drain.

(4) Isolate the unit from the process by closing the isolating valves and breaking the main outlet line - this prevents entry of the cleaning solution to the process.

(5) Remove the upper man-way cover.

(6) Lower the water level in the unit until the amount of water above the resin bed is equal to the void volume of the resin bed.

(7) Prepare a concentrated sodium hydrosulfite/water solution by adding the amount of hydrosulfite required to yield a 4% solution when mixed with the water above the resin bed (Note: always add hydrosulfite to water and provide adequate ventilation). Since these solutions lose their effectiveness quite rapidly, they should be utilized immediately after mixing and only the amount needed for a specific treatment should be prepared at one time.

(8) Mix the concentrated sodium hydrosulfite/water solution with the water above the resin bed.

(9) Drain from the bottom of the ion exchange unit until the sulfite solution level is just above the resin bed level.

(10) Mix the sulfite solution and resin so that the cleaning solution is evenly mixed throughout the resin bed - do not agitate with air as this will oxidize the sulfite and render it ineffective.

(11) Allow the cleaning solution to contact the resin for 12 hours if possible, otherwise a minimum contact time of 4 hours is recommended.

(12) Replace the upper man-way cover.

(13) Drain, rinse the unit downflow and backwash for 30 minutes to remove loosened foreign material.

14) Regenerate the resin using twice the normal amount of regenerant and return the unit to service after the effluent pH is normal.

IN-SITU BRINE SQUEEZING OF ANION RESIN

A. Chemicals and Materials

(1) Decationized or softened water, heated.

(2) Fine grade sodium chloride, about 14-42 lbs per cubic foot of resin, depending on the number of squeezes required.

(3) Sodium hydroxide (flake caustic), approximately 4-12 lbs. per cubic foot of resin, depending on the number of squeezes required.

(4) Glass sample bottles.

B. Procedure

(1) Thoroughly backwash the resin by passing water upward through the unit at a flow rate sufficient to expand the bed by at least 50% of its normal service height - the cleaner the resin is, the more efficient the cleaning will be.

(2) Isolate the unit from the process by closing the isolating valves and breaking the main outlet line - this prevents entry of cleaning solutions to the process.

(3) Remove the upper man-way cover.

(4) Lower the water level in the unit to 1-2 feet above the resin bed.

(5) Regenerate with caustic at the set-points shown for the first caustic regeneration in Table 1 over-leaf.

(6) Slow rinse the resin at the set-points shown for the first caustic regeneration in Table 1.

(7) Regenerate with warm salt at the set-points shown for the salt regeneration in Table 1.

(8) Slow rinse the resin at the set-points shown for the salt regeneration in Table 1 - collect effluent samples every 5 minutes until the most intense colour is observed - retain and set aside the sample having maximum colour intensity and discard the rest.

(9) Regenerate with caustic and then rinse at the set-points shown for subsequent caustic regenerations in Table 1.

(10) Repeat steps (7), (8) and (9) until the maximum colour eluted during the brining is a small fraction of the colour observed after the first treatment - approximately 2-6 squeezes are required to purge most of the organics.

(11) Replace the upper man-way cover

(12) Repeat step 1.

(13) Regenerate the resin with twice the normal amount of salt if the unit is a chloride form dealkalizer or organic trap, or

(14) Regenerate the resin with twice the normal amount of caustic if the unit is a hydroxyl form strong base anion exchanger.

15) Return the unit to service.

TABLE 1

Set-Points For Brine Squeeze Procedure

Parameter	Units	First Caustic Regeneration	Salt Regeneration	Subsequent Caustic Regeneration
quantity of regenerant	lb/ft^3 resin	2.0	7.0	2.0
regenerant strength	%	3.5	15	5.0
quantity of dilute regenerant	lb/ft^3 resin	57	46.6	40
volume of dilute regenerant	USG/ft^3 resin	6.9	5.2	4.8
flow rate of dilute regenerant	$USGPM/ft^3$ resin	0.35	0.2	0.24
time for regeneration	minutes	20	25	20
dilute regenerant temperature	°F	normal	130-140	normal
rinse water temperature	°F	90-140	90-140	90-140
flow rate of rinse water	$USGPM/ft^3$	0.2	0.5	0.2
time for rinsing	minutes	10	15	10

PROCEDURE FOR SAMPLING OF ION EXCHANGE RESIN

1. Operate the unit to its normal exhaustion endpoint, then backwash, regenerate and rinse in the normal manner.

2. Open unit and while draining, observe and record the appearance of the top inlet distributor and regenerant distributor (i.e. are they level, are openings filled with resin, do they show any other abnormal condition). A hot process unit must be cooled before draining to prevent dehydration.

3. Drain the unit to a level well below the bed surface.

4. Observe and record the appearance of the bed surface (i.e. clean, dirty, level, hilly, mounded in center, cracks, slopes to side, pulled away from shell, or any other abnormal conditions).

5. Measure and record the bed depth.

6. If the bed surface is not normal, probe the bed with a metal rod to the supporting bed, in a number of spots to see if the supporting bed is level. Record observations.

7. Obtain a core sample from the resin bed as follows:

   (a) Add water to the unit until the surface of the bed is submerged;

   (b) Thrust a thin walled 1" copper, stainless steel, or plastic tube down into the bed to the supporting underdrain;

   (c) Cap or plug the top end of the tube;

   (d) Drain the unit;

   (e) Slowly withdraw the tube;

   (f) Transfer the resin sample to a plastic jar by adding water to the top of the tube while it is held over the mouth of the jar;

   (g) Repeat this procedure until approximately ½ - 1 pint of resin is collected;

   (h) Add water from an operating unit to the sample jar until the resin sample is submerged;

   (i) Identify the resin sample by marking the name of the institution it was collected from on the outside of the sample jar;

   (j Complete the attached data sheet;

(k) Pack resin sample (in cold weather, insulate to prevent freezing) with data sheet enclosed and mail to the following address for analysis:

Rohm & Haas
#2 Manse Road
West Hill, Ontario
M1E 3T9

Attn: Laboratory Manager

or

Alberta Government
Public Works, Supply and Services
Alberta Government Services Laboratory
11210 - 120 Street
Edmonton, Alberta
T5G 2X9

Attn: S. Seepersad

ION EXCHANGE RESIN DATA SHEET

Origin of Sample

Company Name: _____

_____

Company Address: _____

_____

_____

Company Contact: _____

_____ _____

Sample Collection

Date of Sample Collection: _____

Sampling Procedure: Core Sample Surface Sample

Exchanger Specifications

Duty (i.e. softener, sodium cycle polisher, mixed bed polisher, cation exchanger, anion exchanger, organic trap, other)

Diameter in

Area ft^2

Depth of Resin in

Volume of Resin ft^3

Manufacturer of Resin

Resin Name

Type of Supporting Bed

Freeboard in

Normal Flow Rate USGPM

Maximum Flow Rate USGPM

Volume of Water Treated Between Regenerations USG

Age of Resin Years

Influent Water

Source/Origin

Temperature °F

Total Hardness ppm $CaCO_3$

Sodium ppm $CaCO_3$

Iron ppb Fe

Alkalinity ppm $CaCO_3$

Chloride ppm $CaCO_3$ _____

Sulfate ppm $CaCO_3$

pH ppm $CaCO_3$ _____

Conductivity, micromhos/cm

Regeneration

Regenerant _____ Concentration _____ %

Amount of Regenerant: lb/cycle _____ lb/ft^3

Backwash Flow Rate _____ USGPM, Duration _____ min.

Regenerant Flow Rate _____ USGPM, Duration _____ min.

Rinse Flow Rate _____ USGPM, Duration _____ min.